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GAS-COOLED REACTOR PROGRAMS

**HIGH-TEMPERATURE GAS-COOLED REACTOR BASE-TECHNOLOGY PROGRAM
ANNUAL PROGRESS REPORT
FOR PERIOD ENDING DECEMBER 31, 1977**

F. J. Homan

Paul R. Kasten

MASTER

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F. J. Homan, Program Manager

Paul R. Kasten, Program Director

Edited by Sigfred Peterson

MASTER

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CONTENTS

SUMMARY	1
1. FISSION PRODUCT TECHNOLOGY AND COOLANT IMPURITY EFFECTS	1
1.1 FISSION PRODUCT SURVEILLANCE OF THE PEACH BOTTOM HTGR PRIMARY CIRCUIT	1
1.1.1 Fission Products in the Primary Circuit Cold Duct	1
1.1.2 Inventories of ^{134}Cs and ^{137}Cs in Peach Bottom HTGR Steam Generators at Final Shutdown	2
1.2 POSTIRRADIATION EXAMINATION OF PEACH BOTTOM HTGR FUEL ELEMENTS	4
1.2.1 Inventories of Radionuclides in Peach Bottom HTGR Driver Elements	4
1.2.2 Release of Cesium from Fuel and Transport by the Purge Gas	6
1.2.3 Fuel Particle Failure Fractions	10
1.3 MIGRATION OF ^{90}Sr IN PEACH BOTTOM HTGR FUEL ELEMENT GRAPHITE	13
1.4 REFERENCES	16
2. FUELED GRAPHITE DEVELOPMENT	19
2.1 INTRODUCTION	19
2.2 FUEL PARTICLE PROCESS DEVELOPMENT AND QUALIFICATION	20
2.2.1 Buffer Coating Studies and Partial Conversion of ThO_2	20
2.2.2 SiC Coating Studies	23
2.2.3 Particle Coating in Support of Irradiation Experiments	25
2.2.4 Triso Coating of Simulated Fission Products	26
2.2.5 Effect of Self-Radiolysis on Subsequent Chemistry of ^{233}U Loaded Weak-Acid-Resin (WAR) Kernels	26
2.2.5.1 Tapped Density	28
2.2.5.2 Diameters	28
2.2.5.3 Photographs	28
2.2.5.4 Carbonization and Conversion	29
2.2.5.5 Carbon and Uranium Concentrations	30
2.2.5.6 Shape Separation	30
2.2.5.7 Crushing Strength	30
2.2.5.8 General Conclusions	30

2.3	CHARACTERIZATION OF PYROCARBON COATINGS	31
2.3.1	Permeability Determinations Using Inert-Gas Intrusion	32
2.3.2	Permeability Determination Using Chlorine Leach Techniques	46
2.3.3	Permeability Determination Using TRIGA Activation . .	49
2.3.4	Permeability Determination Using Internal Gas Inventory Measurements	49
2.3.5	X-Ray Diffraction Determination of BAF	51
2.4	IRRADIATION TESTING IN THE HFIR TARGET FACILITY	52
2.4.1	Capsules HT-12 Through -15	53
2.4.2	Capsules HT-17 Through -19	55
2.4.3	Capsules HT-20 Through -23	55
2.4.4	Capsules HT-28 Through -30	56
2.4.5	Capsule HT-31	57
2.4.6	Capsule HT-32	60
2.4.6.1	Capsule Operation	60
2.4.6.2	Postirradiation Examination	62
2.4.7	Postirradiation Examination of HT-33	71
2.4.8	Capsule HT-34	71
2.4.8.1	Capsule Design	77
2.4.8.2	Preparation of Test Specimens	77
2.4.8.3	Operating Conditions	85
2.4.8.4	Postirradiation Examination	85
2.5	IRRADIATION TESTS IN HFIR REMOVABLE BERYLLIUM FACILITY . . .	85
2.5.1	Capsules HRB-11 and -12	87
2.5.1.1	Capsule Operation	87
2.5.1.2	Postirradiation Examination	92
2.5.2	Capsule HRB-13	98
2.5.3	Capsule HRB-14	98
2.5.4	Capsule HRB-15b	105
2.6	IRRADIATION TESTS IN THE ORR	109
2.7	CAPSULE THERMOMETRY	109
2.7.1	Thermometry Development	110
2.7.2	Thermometry in HRB Capsules	115
2.7.2.1	HRB-12	115

2.7.2.2	HRB-13	116
2.7.3	Influence of Transmutation Alloying in W-Re Thermocouples	121
2.8	NUCLEAR ANALYSIS IN SUPPORT OF IRRADIATIONS	121
2.8.1	OF-2 Neutronics Analysis	121
2.8.2	OC-1 Neutronics Analysis	127
2.8.3	Preparation of the OC-2 Dosimeters	128
2.8.4	Design Study of the MONUHRIH Experiment	128
2.9	POSTIRRADIATION PERFORMANCE ASSESSMENT	134
2.9.1	Thermal Analysis of Irradiation Capsules	135
2.9.2	Electron Microprobe Examination of HTGR Fuel Particles	135
2.9.3	Irradiated Microsphere Gamma Analyzer System	136
2.9.3.1	Failure Fraction Measurements	136
2.9.3.2	IMGA Operation	138
2.9.3.3	IMGA Analysis of OF-2 Fuel	139
2.9.4	Deconsolidation of HTGR Fuel Rods	152
2.10	FISSION PRODUCT-SiC COATING INTERACTION STUDIES	152
2.11	REFERENCES	153
3.	PRESTRESSED CONCRETE PRESSURE VESSEL DEVELOPMENT	159
3.1	INTRODUCTION	159
3.2	PCRv ANALYTICAL STUDIES	159
3.2.1	Analysis of Multicavity PCRv Elastic Response	160
3.2.1.1	Description of the PCRv	160
3.2.1.2	Description of the Analytical Model	162
3.2.1.3	Analytical Results	166
3.2.2	Analysis Methods Development	170
3.3	INSTRUMENTATION EVALUATION AND DEVELOPMENT	174
3.3.1	Review of Concrete Embedment Instrumentation	175
3.3.1.1	Strain Measurement	175
3.3.1.2	Stress Measurement	176
3.3.1.3	Moisture Measurement	176
3.3.2	Concrete Embedment Strain Instrumentation Evaluation	178
3.3.2.1	General Gage Performance	178
3.3.2.2	Gage Performance in Representative PCPV Environments	191

3.3.2.2	Gage Performance in Extreme Environments . .	192
3.3.2.4	Preliminary Conclusions from Strain Gage Study	195
3.3.3	Acoustic Emission Study	196
3.3.3.1	Instrumentation	199
3.3.3.2	Experimental Procedure	200
3.3.3.3	Cycle 1 Test Results: Pressurization to 72.4 MPa	201
3.3.3.4	Cycle 2 Test Results: Pressurization to Failure	204
3.3.3.5	Conclusions	206
3.4	CONCRETE STRUCTURAL MODEL TESTS	208
3.4.1	Structural Behavior	208
3.4.2	New Materials	215
3.4.2.1	Fiber Reinforced Concrete	219
3.4.2.2	Polymer Silica Cements	219
3.4.2.3	Shrinkage-Compensating Cements	223
3.4.3	Corrosion Behavior	224
3.4.3.1	Stressed Tendons	224
3.4.3.2	Nonstressed Tendons	225
3.4.4	Tendon Monitoring Techniques	243
3.5	LINER AND PENETRATION STUDIES	246
3.6	REFERENCES	253
4.	STRUCTURAL MATERIALS	255
4.1	INTRODUCTION	255
4.2	MECHANICAL PROPERTIES	255
4.2.1	Tensile Testing	255
4.2.2	Creep Testing of Base Materials	270
4.2.2.1	Environmental Test Facilities	270
4.2.2.2	Hastelloy X	278
4.2.2.3	2 1/4 Cr-1 Mo Steel	282
4.2.2.4	Miscellaneous Alloys	287
4.2.2.5	Changes in Carbon Content	290
4.2.3	Creep Properties of Weldments	292
4.2.4	Low Cycle Fatigue and Creep Fatigue	296
4.2.5	Fatigue Crack Growth	300

4.3	WELDING DEVELOPMENT	303
4.3.1	Weldability Testing	303
4.3.2	Weldability Testing — Alloy 800	304
4.3.3	Shielded Metal-Arc Weldments of Hastelloy X	305
4.3.4	Shielded Metal-Arc Welding of Hastelloy X to Alloy 800H	308
4.4	STEAM CORROSION	309
4.4.1	Inconel 617 Loop	310
4.4.2	Inconel 625 Loop	318
4.4.3	Heat Transfer Corrosion Tests	319
4.5	REFERENCES	320
5.	CHARACTERIZATION AND STANDARDIZATION OF GRAPHITE	323
5.1	INTRODUCTION	323
5.2	CREEP EXPERIMENTS OC-2 AND OC-3	323
5.2.1	Modification to Pressure Sensor System	324
5.2.2	Modification to Movable Thermocouple System	326
5.2.3	Status of Construction and Irradiation Schedule	327
5.3	KFA GRAPHITE IRRADIATIONS	327
5.3.1	Capsule Design and Construction	327
5.3.2	Material Evaluation of KFA Graphites	330
5.3.3	Summary and Conclusions	343
5 4	REFERENCES	343
6.	EVALUATION AND PEBBLE-BED TYPE HTR	345
6.1	INTRODUCTION	345
6.2	PBR FUEL CYCLE CALCULATIONS	345
6.3	PBR CORE PERFORMANCE AND MECHANICAL DESIGN	348
6.3.1	Heavy Metal Loadings	349
6.3.2	Pressure Drop	352
6.4	REFERENCES	355
	ORGANIZATION CHART	357

SUMMARY

1. HTGR CHEMISTRY STUDIES

The final report on surveillance activities concerning the Peach Bottom HTGR primary circuit was published. Comparisons between external gamma scan and subsequent trepanned sample data for ^{137}Cs deposition levels were fair (within 50%). Total radiocesium inventory on the steam generators was estimated to be 78 Ci at reactor shutdown based on coolant sampler data.

Postirradiation examinations of six Peach Bottom fuel elements (four end-of-life elements) were completed. Three examination reports have been published and the fourth is in press. These reports describe and record (1) the appearance of the fuel compacts, (2) dimensional alterations, (3) fission product inventories in the fuel, (4) spatial variation of fission products in the graphite, and (5) estimated fuel particle failure fractions for two fuel elements.

2. FUELED GRAPHITE DEVELOPMENT

Fuel Particle Process Development and Qualification

Studies of the buffer coatings on fertile particles and partial conversion of thorium oxide kernels to thorium carbide were continued during the reporting period. These studies were conducted at 1500°C using a coating gas of 25% MAPP gas diluted with argon. The standard deviation of the coating thickness decreased with increasing coating density. However, conversion from oxide to carbide did not take place in a controlled manner. The extent of the conversion was erratic and unpredictable, indicating the need for more process development before a product is available for irradiation testing.

A study on the effects of coating conditions on SiC coating characteristics was completed and reported. Highly dense coatings can be deposited at higher coating rates than are normally used. Coating characteristics depend on coating conditions rather than coating rate. This work needs to be extended to characterize the SiC coating microstructure as a function of coating density and coating conditions, in particular, the amount of free silicon present at the grain boundaries. This quantity is thought to influence the capacity of the SiC layer to resist attack of mobile fission products and to resist the diffusion of some fission product species.

The influence of self-radiolysis on the subsequent composition of ^{233}U loaded WAR-derived fuel particles was measured. This experiment was partially funded by the HTGR Recycle Program. The general conclusions of this work were that the two resin sources (Duolite and Amberlite) behave differently during conversion. The activation energy of fuel loaded onto

Amberlite resin is different from that of fuel loaded onto Duolite, although the experiment was not designed to determine activation energies, so this difference could not be quantified. Another conclusion was that fuel could be stored up to three months after loading without influencing the final coated-particle behavior during subsequent processing. However, storage of up to six months seriously degrades the kernel surface, generating substantial quantities of dust. At the very least this would lead to furnace contamination during processing and loss of uranium from the process stream.

Characterization of Pyrocarbon Coatings

Characterization work has centered on detecting gas permeability of PyC coatings, both as fabricated and after irradiation. Four techniques have been used, two for characterization of unirradiated coatings (Ne-He intrusion and chlorine leach) and two for characterization of irradiated coatings (internal gas pressure measurements and TRIGA activation). Eventually we plan to use all four techniques on irradiated fuel. Most of the work during this reporting period has been associated with collecting and interpreting data and trying to correlate the results from different techniques. The ultimate goal is to establish procedures that will serve both for quality assurance and inspection of unirradiated fuel and as a measure of performance for irradiated fuel.

Irradiation Testing in the HFIR Target Facility

Activities this reporting period have included postirradiation examination of several capsules recently irradiated (HT-31 through -33), additional examinations and analysis on several "old" capsules (HT-12 through -15, -17 through -19, -20 through -23, and -28 through -30), and irradiation of one additional capsule (HT-34). Work on the old capsules was associated with characterization of the PyC coatings of Biso particles for gas permeability, using the techniques described in the last section. In addition, examination of the low-enriched uranium (LEU) driver particles from earlier HT capsules was begun, because of the interest in LEU fuel for application in proliferation-resistant fuel cycles. The HT-34 capsule was designed as a comprehensive test to understand the behavior of the PyC coatings under irradiation relative to changes in gas permeability.

Irradiation Testing in the HFIR Removable Beryllium Facility

Work this period included postirradiation examination of capsules HRB-11 and -12, irradiation of capsule HRB-13, and planning and design of capsules HRB-14 and -15b. HRB-11 and -12 were dedicated to the recycle fuel chemistry specification (WAR-derived recycle fuel), and HRB-13 was dedicated to the particle shape specification. Early in the reporting period it became evident that the highly enriched uranium (HEU) fuel cycle would be replaced by low-enriched uranium (LEU) or medium-enriched uranium (MEU) cycle for early HTGRs. Therefore, the emphasis on capsules HRB-14 and -15b is on MEU fuel development. All future HRB capsules will be oriented toward the MEU fuel cycle unless emphasis should switch back to the HEU cycle.

Irradiation Tests in the ORR

Work this period included completion of a topical report on the OF-1 capsule and postirradiation examination of the OF-2 capsule. This topical report on OF-2 will be completed during the first half of 1978. A third ORR capsule is planned for irradiation beginning in FY 1980.

Capsule Thermometry

Thermometry development work has been directed toward finding a system that can be used for centerline temperature measurements in HRB capsules. Capability is required for operation at temperatures up to 1500°C for up to ten months in neutron fluences of up to 10^{26} n/m² (>0.18 MeV). All thermocouple systems used in HRB capsules to date have been unsatisfactory, because of either decalibration due to neutron-induced transmutation or chemical interaction with the fuel. The Chromel vs Alumel thermocouples used in the graphite sleeves of the HRB capsules continue to work well.

Several promising systems were identified during the reporting period for use in HRB capsules for centerline temperature measurement. These systems will be developed in 1978 and hopefully tested in a capsule in late 1979 or 1980.

Nuclear Analysis in Support of Irradiation

This work is oriented toward quantification of the irradiation environment for ORR and HFIR capsules, for both the Fueled Graphite Development Program and the Graphite Characterization and Standardization Program. During this reporting period the following activities were pursued:

- analysis of the OC-1 capsule dosimeters
- preparation of the OC-2 capsule dosimeters
- development of a program for measurement of gamma heating rates in the VXF-13 facility and HRB facilities

Postirradiation Performance Assessment

This work is associated with postirradiation examination and performance determination of HTGR fuel irradiated in HRB and ORR capsules. Work was concentrated in four areas: thermal analysis, microprobe analysis, IMGA analysis (irradiated microsphere gamma analyzer), and fuel rod deconsolidation. The deconsolidation work is associated with the IMGA analysis, in that the IMGA system is used on individual coated particles, which are recovered from the irradiated fuel rods through deconsolidation. The IMGA system is relatively new, and was applied in measurement of particle failure fractions for the first time during this reporting period. It is anticipated that the IMGA system will be a major contributor in the performance assessment of HTGR fuel in the years ahead.

Fission Product-SiC Coating Interaction Studies

The kinetics of the chemical reactions between rare-earth fission products and SiC coatings were measured. This work was done by depositing Triso coatings on kernels made from rare-earth dicarbides, and then heating these "particles" in a furnace at a known temperature and temperature gradient. The kinetics of the reactions measured under these conditions are essentially identical to the kinetics observed in UC_2 fissile particles irradiated under much less well known temperatures and gradients. The reaction that occurs is essentially a destructive distillation of the rare-earth dicarbide in the kernel, transport of rare-earth metal down the temperature gradient, and formation of the dicarbide at the SiC-PyC interface. There solution of the SiC in the dicarbide along the SiC grain boundaries completes the chemical interaction. It appears that UC_2 particles can survive four years of operation at "nominal" operating temperatures and gradients, but there is little margin to deal with overtemperature conditions.

This work has shown that rare-earth dicarbides react with SiC, while rare-earth oxides do not. This work will be extended in FY 1978 to consider chemical interactions between fission products from MEU fuel with SiC, particularly, the fission products Pd and Ag.

3. PRESTRESSED CONCRETE PRESSURE VESSEL DEVELOPMENT

The structural response of a multicavity PCRV designed for use in a commercial HTGR power plant has been analyzed with the three-dimensional elastic finite-element computer code STATIC-SAP. The results appear reasonable when compared with results of earlier analyses of the comparable Ohbayashi-Gumi multicavity PCRV. These analytical results will be used to make an independent evaluation of whether the PCRV design meets the ASTM Sect. III, Div. 2 code requirements.

A computer program, called a constitutive model driver, has been written to provide a convenient method for evaluating candidate concrete materials' constitutive equations. A Tektronix 4051 computer is used to simulate a wide variety of structural loadings. Thus far, the ADINA (1977 version) concrete model and the endochronic constitutive model have been evaluated. The endochronic model more accurately represented the behavior to ultimate stress and subsequent strain softening. Based on these findings, the endochronic model has been added to the two-dimensional materials library of the ORNL ADINA finite-element computer program.

Representative concrete embedment strain gages that are available commercially in the United States have been evaluated with respect to sensitivity, accuracy, stability, response, data acquisition requirements, cost, adequacy of calibration, and durability. Studies were also made to select a satisfactory calibration specimen and to determine the effect of concrete strength and aggregate size on gage performance. The findings indicated that the calibration factor should be determined experimentally on a representative sampling of the gages. The gages should be cast in

concrete specimens of sufficient size relative to the size of the gages, and made of basically the same concrete as would be used in the actual structure. The concrete specimens would then be tested in uniaxial compression to determine the gage calibration factor.

A multichannel acoustic emission source location system was used to monitor the internal pressure testing of a flawed thick-walled steel pressure vessel and the flexural testing of a series of post-tensioned concrete beams. The acoustic emission system correctly identified regions where significant structural distress took place. This is part of a continuing program to obtain data from various types of tests in order to develop information on the characteristic acoustical signatures produced from the overloading of complex structures.

The relative structural performance of grouted and nongrouted post-tensioned structures is being evaluated by flexural tests of concrete beams. For the same level of prestressing, the grouted beams were stronger. The grouted beams developed smaller but more numerous flexural cracks. These results confirm the capability of grouted prestressing tendons to restrict the size of cracks that could develop in a PCRV from accidental overpressurization.

Promising new concrete materials such as steel-fiber-reinforced concrete, inorganic corrosion-resistant cement, and expansive cement are being tested; however, further testing is required before any significant conclusions can be made. A bench-scale study of corrosion protection capability of concrete grout as compared with commercial corrosion protection compounds and a study of using acoustic emission to monitor prestressing tendons are described.

The results of the program to develop fracture toughness property data for representative metallic materials used in the fabrication of PCRV liners and closures are reported. The program includes drop-weight nil-ductility temperature tests, tensile tests, and various types of fracture toughness tests consisting of impact, precracked Charpy, and compact specimen tests. Also included in the program are tests of various weldment samples representing the weld metal and heat-affected zone. The 10,000-hr aging at 343°C of the first of the materials scheduled for this extended heat treatment prior to testing has been initiated.

4. STRUCTURAL MATERIALS

The principal objective of the structural materials program is to provide sufficient data for safe and economic construction of high-temperature components. Mechanical properties of selected materials are being determined in prototypical helium environments. In addition, structural stability, weldability, and steam corrosion are being studied to more fully characterize material behavior.

Aging Hastelloy X and Inconel 617 reduced their room-temperature fracture strain. Loss in room-temperature ductility was greater in Hastelloy X. Aging also influenced the tensile properties of Hastelloy X to Hastelloy X and Hastelloy X to alloy 800 weldments. Changes were greater in the Hastelloy X to Hastelloy X weldment and depended on aging temperature and type of filler metal.

Prior creep testing in HTGR helium increased yield strength and decreased total elongation in all materials except 2 1/4 Cr-1 Mo steel. This is consistent with the hypothesis that all the materials except 2 1/4 Cr-1 Mo steel were carburized in the helium environment.

Additional creep test facilities were brought onto line during the year. A total of 32 environmental and 18 air test machines are now operational. Creep data in helium on two heats of Hastelloy X have been compared with literature data, and a tentative conclusion is that the environment has little effect from 650 to 871°C. Results of creep tests of 2 1/4 Cr-1 Mo steel to date are not conclusive as to the effect of environment. HTGR creep test data appear to agree quite well with existing models. A 2%-Ti-modified Hastelloy N alloy may have much better resistance to carburization than Hastelloy Z at 871°C.

Rupture times of Hastelloy X to Hastelloy X weldments generally exceeded that of Hastelloy X base material, regardless of filler metal or welding process. Aging responses were similar to those of base material. Weldments of Hastelloy X to alloy 800H fractured in the alloy 800H base material, with little deformation in the weld. Time to rupture varied with aging treatment.

Fatigue testing of 2 1/4 Cr-1 Mo steel in HTGR helium indicated a slight increase in life in short-term continuous cycling tests. In creep fatigue tests both tensile and compressive hold periods reduced fatigue life. Analyses of creep-fatigue data in air indicate good agreement between experimental results and predictions by the run of strain-range partitioning concepts.

Temperature markedly affected the fatigue crack growth rate (FCGR) of Hastelloy X in air and helium, but frequency had only a small effect. The FCGR in helium was less than in air. The FCGR of 2 1/4 Cr-1 Mo steel in air increased with stress intensity, temperature, and inverse frequency. In steam, FCGR was similar to that in air, but it was less in vacuum and helium.

Relatively large variations in hot-cracking tendency were noted among nine heats of Hastelloy X. However, no correlation with chemical composition was found and none of the heats would be expected to exhibit cracking problems during welding. The Tigamajig test was used to detect a crack-sensitive heat of alloy 800. The ratio $(Al + Ti)/(C + Si)$ was a reasonable predictor of cracking behavior. The tensile and aging characteristics of Hastelloy X weldments made with Hastelloy X shielded metal-arc electrodes was investigated. Results were similar to those previously determined on weldments made by the gas tungsten-arc process.

Steam corrosion studies were continued in the Inconel 617 loop at 482 and 538°C. For times up to 5000 hr a nearly parabolic corrosion rate was observed. After longer times a low constant rate has been measured for all materials except alloy 800, Inconel 617, and type 304 stainless steel. Alloy 800 and Inconel 617 have corroded at such low rates that the kinetics cannot be determined from weight changes, and type 304 stainless steel oxidized nonuniformly.

A steam corrosion test under heat flux conditions was repeated such that the oxide was formed on concave surfaces, as occurs in superheaters. Oxide exfoliated under these conditions, as in the previous test service where oxide was formed on the outside surface of the test samples. Thus, heat flux appears to be an important parameter to be considered.

5. CHARACTERIZATION AND STANDARDIZATION OF GRAPHITE

The compressive creep experiments were interrupted by a reduction in operations at the ORR. This period was utilized to redesign the basic capsule to overcome difficulties encountered in the first creep experiment (OC-1) and to construct the second and third experiments. Design changes included addition of alignment devices to keep the loaded specimen columns from buckling, replacement of the LVDT load sensors by a pneumatic sensor, and improvement of the contacts for the movable thermocouple.

A new program was initiated to irradiate moderator graphites manufactured in the Federal Republic of Germany under the joint US/FRG program. These irradiations are intended to lead and simplify more extensive irradiations in Europe by utilizing the high flux of HFIR. Seven candidate graphites have been received from KFA, the blocks have been nondestructively mapped to determine volumes to be sampled, and the requisite samples have been machined and are under test. The first irradiation is planned for March 1978.

6. EVALUATION OF PEBBLE-BED HTR

This effort examines the design and mode of operation of the pebble-bed HTR concept and projects its performance. Ore utilization and fuel cost are of primary interest, with design constraints satisfied. This work is under a technical information exchange umbrella agreement with FRG. Applications for power and process heat are of interest.

A design for breeding with ^{233}U fuel was proposed by the General Electric Company. (This design was a modification of a design proposed in Germany as an advance over the AVR reactor and involves small fertile pebbles trickling between larger fueled primary pebbles.) Core physics and fuel cycle analyses were performed for this concept and also for a reactor design with two pebble types of the same size with primary emphasis on ^{233}U feed. Results of this analysis were reported in ORNL/TM-6142.

A cursory review of the pertinent mechanical design features of such a reactor system was also performed to examine some generic design constraints and/or advantages. The specific areas addressed are: limits of practical heavy metal loadings, single-versus multiple-ball pressure drops and their effects on plant efficiency, and the effect of additional axial blankets.

The heavy metal loading (i.e., mass of heavy metal per unit volume of core) is a crucial quantity in determining the size as well as the neutronic performance of the reactor system. This particular quantity depends on four independent variables: the average pebble packing fraction, the thickness of the graphite shell on the pebbles, the size of the coated fuel particle that is suspended in the inner graphite matrix of the pebble, and the packing fraction of the coated fuel particles in the inner graphite matrix.

The calculation of the pressure drop through the core of a pebble bed reactor is perhaps the most critical factor influencing the design of the system. It may also be the most uncertain factor, particularly for systems that employ two sizes of pebbles.

The pressure drop through the pebble bed is a function of a number of reactor design parameters. Of particular interest in this study is the dependence of the pressure drop on the size of the pebble or pebbles used for fuel. Five variations on a base design were studied to assess this dependence.

In the continuing study we are considering low-enriched uranium feed of nonproliferation quality, a pre-breeder to generate ^{233}U fuel, and a ^{233}U -fueled design with a high conversion ratio. One-dimensional parameter studies have been made for fully enriched ^{235}U feed and for ^{233}U feed. Two-dimensional calculations are made for selected reference conditions. Calculations have been made for a single pebble size with two feed streams, one containing ^{235}U -fueled pebbles and the other fertile pebbles.

1. FISSION PRODUCT TECHNOLOGY AND COOLANT IMPURITY EFFECTS

R. P. Wichner

1.1 FISSION PRODUCT SURVEILLANCE OF THE PEACH BOTTOM HTGR PRIMARY CIRCUIT — F. F. Dyer, R. P. Wichner, W. J. Martin, and L. L. Fairchild.

Work that involved measurements of the amounts and distributions of radionuclides in the primary coolant circuit of the Peach Bottom HTGR was completed with the publication of a final report that described the experimental methods and results.¹ Highlights of the work were presented last year,² and some of the findings were presented elsewhere.³ Since the publication of ref. 1, the amounts of radionuclides deposited on surfaces of the primary coolant duct were measured on specimens trepanned from the duct, and the amounts of ^{134}Cs and ^{137}Cs deposited in the steam generators were estimated. These results, which tend to verify previously reported plateout distributions and give a more complete picture of the plateout distributions of the cesium isotopes, are discussed below.

1.1.1 Fission Products in the Primary Circuit Cold Duct

The ductwork in the primary coolant system downstream of the steam generators, designated the cold duct, was trepanned at several axial locations following final shutdown of the reactor. This sampling operation, which was carried out by Catalytic Incorporated under subcontract to General Atomic Company (GA), is described elsewhere.⁴ Two disks from the cold duct, which were supplied to ORNL by GA, were analyzed for gamma emitters and ^{90}Sr . The specific surface activities measured on the disks are given in Table 1.1. Included in this table are results for ^{90}Sr , ^{134}Cs , and ^{137}Cs that were determined by GA on other specimens that were cut from adjacent areas on the duct at the same axial locations.⁵ Also given in Table 1 are activity values of the two cesium isotopes that were measured *in situ* by ORNL¹ and GA⁶ using external gamma scanning procedures.

Although these results are limited to two locations on the cold duct, they tend to show fairly good agreement (less than 50% differences) both between the measurements on trepan samples and external duct scans and between the ORNL and GA measurements.

Table 1.1. Specific Surface Activities of Radionuclides Found on Peach Bottom Loop 1 Cold Duct^a

Radionuclide	Location 3				Location 7			
	Trepan Specimen ^b		In situ Gamma Scan Measurement		Trepan Specimen ^b		In situ Gamma Scan Measurement	
	ORNL 4	GA	ORNL	GA	ORNL 4	GA	ORNL	GA
⁵⁴ Mn					1.9E-3			
⁶⁰ Co	6.1E-4				6.1E-4			
⁹⁰ Sr	3.1E-3	6.1E-4			2.2E-3			
¹⁰⁶ Ru					2.7E-2			
^{110m} Ag					5.5E-3			
¹²⁵ Sb					5.3E-3			
¹³⁴ Cs	2.17	2.66	3.15	2.62	1.32	1.55	1.75	2.17
¹³⁷ Cs	1.82	2.21	2.63	2.22	1.18	1.23	1.45	1.82
¹⁴⁴ Ce					0.19			
¹⁵⁴ Eu					6.8E-4			

^aLocations are defined in Fig. 1.1 in GA-A-14369. Location 3 was 0.9 to 1.2 m (3-4 ft) downstream of steam generator; location 7 was about 1.8 m (6 ft) below block valve HC-203. Blanks in table signify that either measurement was not made or nuclide was not observed.

^bFour specimens were trepanned from locations 3 and 7. Specimen 4 was supplied to and analyzed by ORNL. The GA values are mean values for the four specimens.

1.1.2 Inventories of ¹³⁴Cs and ¹³⁷Cs in Peach Bottom HTGR Steam Generators at Final Shutdown

Although gamma spectra of the steam generator in loop 1 were acquired¹ several times during the operation of core 3, we could not derive the amounts of gamma emitters in the steam generator because of its inner structural complexity. We have, however, made use of data obtained with coolant samplers (sect. 3 of ref. 1) to estimate the amounts of ¹³⁴Cs and ¹³⁷Cs that were in both steam generators at final shutdown of the reactor. The method of estimation and results are given in Appendix C of ref. 7 and are summarized below. The method of estimation was to determine the concentration of cesium in the coolant above and below the steam generator and assume that the difference was due to deposition in the steam generator.

Coolant samplers CC and CD were operated simultaneously for 1834 hr upstream and downstream, respectively, of the loop 1 steam generator between March 13 and May 28, 1974. Sampler CC had a coolant throughput of 216 kg He and sampler CD had a throughput of 319 kg. The activity of ^{137}Cs in sampler CC was 8.80 μCi , so the time-average concentration of ^{137}Cs in the coolant upstream of the steam generator was $8.80 \mu\text{Ci}/216 \text{ kg} = 0.0407 \mu\text{Ci/kg}$. Downstream the time-average ^{137}Cs concentration was $4.00 \mu\text{Ci}$ (in sampler CD)/319 kg coolant = $0.0125 \mu\text{Ci/kg}$. (These data were taken from Tables 3.1-1 and 3.2-6 of ref. 1.) From these data we can estimate the fraction deposited of the ^{137}Cs that entered the steam generator:

$$\frac{0.0407 - 0.0125}{0.0407} = 0.69 .$$

The results found from coolant samplers operated in the spring of 1973 and gamma scans of the Peach Bottom ducting in September 1973 (Fig. 2.4-1 and Table 3.2-3 of ref. 1) reveal that no significant increases of ^{137}Cs in the coolant and on the walls of ducts had occurred until after January 1974. The reactor was shut down in September 1973 and did not resume power operation until January 5, 1974. Between January 5, 1974, and October 31, 1974, the reactor operated 4697 equivalent full-power hours (195.7 EFPD). Since the CC and CD coolant samplers operated for a significant fraction of the total operating time of the reactor in 1974 (1834 hr), we assume that the concentration of ^{137}Cs in the coolant as found from the CC sampler was roughly representative of the concentration over the total operating time in 1974. From these data and the rate of coolant flow into both steam generators ($2.1 \times 10^5 \text{ kg/hr}$), we can estimate the amount of ^{137}Cs that deposited on both steam generators in 1974, namely,

$$\begin{aligned} ^{137}\text{Cs deposited} &= 0.0407 (\mu\text{Ci/kg}) \cdot 0.69 \cdot 2.1 \times 10^5 (\text{kg/hr}) \cdot 4697 \text{ hr} \\ &= 3.0 \times 10^7 \mu\text{Ci} \\ &= 30 \text{ Ci.} \end{aligned}$$

The amount of ^{137}Cs that was in the steam generators before 1974 can be estimated if we assume that the ^{137}Cs in the steam generators increased in 1974 by a factor of 4 as did the amount on the cold duct (see Fig. 2.4-1, ref. 1). If this were true, the ^{137}Cs inventory in the steam generator before 1974 was 10 Ci. We estimate therefore that the Peach Bottom steam generators at end-of-life contained approximately 40 Ci ^{137}Cs .

The activity of ^{134}Cs in the steam generators can best be estimated by assuming that the activity ratio $^{137}\text{Cs}/^{134}\text{Cs}$ in the steam generators is the same as that on the cold duct. The activity ratio found experimentally just downstream of the loop 1 steam generator was 0.83 (location 1, Table 2.3-4, ref. 1). We note from Table 1.1 that the $^{137}\text{Cs}/^{134}\text{Cs}$ ratio at location 3 was nearly the same as for location 1, which was the nearest scan location downstream from the steam generator. The activity of ^{134}Cs on both steam generators was therefore approximately $40/0.83 = 48$ Ci.

1.2 POSTIRRADIATION EXAMINATION OF PEACH BOTTOM HTGR FUEL ELEMENTS -

R. P. Wichner, F. F. Dyer, W. J. Martin, L. L. Fairchild, and
J. L. Botts.

The examination of six driver fuel elements was completed. A description of these elements, their irradiation histories, the methods used in examinations, and some highlights of results found for the first four elements examined were summarized before.² Final reports describing the examinations of the first four elements were published.⁷⁻¹⁰ Examinations of the final two elements were completed along with reports describing the findings; publication of these reports is expected soon.^{11,12} The behavior of cesium and other fission products in the elements was discussed elsewhere.¹³

1.2.1 Inventories of Radionuclides in Peach Bottom HTGR Driver Elements

The inventories of radionuclides in six Peach Bottom HTGR fuel elements were derived from axial gamma scan data as previously described.⁹ Inventories for the sleeves, spines, fuel compacts, bottom connectors, and totals for the elements are given in Table 1.2. The radionuclides observed were ^{95}Zr , ^{103}Ru , ^{106}Ru , $^{110\text{m}}\text{Ag}$, ^{125}Sb , ^{134}Cs , ^{137}Cs , ^{141}Ce , ^{144}Ce , ^{154}Eu , and

Table 1.2. Inventories of Radionuclides Observed in
Peach Bottom HTGR Driver Fuel Elements

Element	EFPD	Component	Nuclide Inventories at Time of Removal from Reactor (Ci)										
			$^{95}\text{Zr}^a$	$^{103}\text{Ru}^a$	$^{106}\text{Ru}^a$	$^{125}\text{Sb}^a$	^{134}Cs	^{137}Cs	$^{141}\text{Ce}^a$	$^{144}\text{Ce}^a$	^{154}Eu	$^{233}\text{Pa}^a$	^{110m}Ag
E06-01	384	Sleeve	2.8E-1		1.04E-2		2.14E-2	3.48E-2		3.56E-1	4.2E-3		5.5E-2
		Spine	3.1E-2		8.57E-3		1.06E-2	1.22E-2		5.5E-2	3.5E-3		1.7E-2
		Fuel	4.91E3	2.18E3	2.24E2	8.50E0	1.21E2	1.74E2	<i>b</i>	2.80E3	2.63E0	<i>b</i>	
		Bottom Connector			6.4E-4		1.1E-3	6.9E-4		9.6E-3			
		Total	4.91E3	2.18E3	2.24E2	8.50E0	1.21E2	1.74E2		2.80E3	2.64E0		7.2E-2
E11-07	701	Sleeve					3.00E1	1.71E1			1.67E0		5.59E-1
		Spine					1.46E1	8.29E0			1.52E-1		1.76E-1
		Fuel	6.45E3	2.86E3	3.15E2	1.76E1	3.36E2	3.11E2	5.69E3	3.70E3	6.57E0	4.60E4	8.49E-2
		Bottom Connector			4.8E-3		1.4E-1	1.1E-1		5.4E-2			1.0E-3
		Total	6.45E3	2.86E3	3.15E2	1.76E1	3.81E2	3.36E2		3.70E3	8.39E0		8.2E-1
E14-01	897	Sleeve					3.15E-1	2.41E-1		1.24E-1	1.73E-2		2.06E-1
		Spine					5.54E-2	4.01E-2		1.03E-1	9.42E-3		8.46E-2
		Fuel	4.39E3	1.70E3	2.29E2	1.35E1	4.15E2	4.39E2	2.70E3	2.83E3	7.45E0	2.46E4	
		Bottom Connector											
		Total	4.39E3	1.70E3	2.29E2	1.35E1	4.15E2	4.39E2	2.70E3	2.83E3	7.47E0	2.46E4	
F03-01	897	Sleeve					6.38E1	3.61E1		6.70E0	2.43E0		7.71E-1
		Spine					1.78E1	9.96E0		2.12E0	2.55E-1		1.78E-1
		Fuel	5.63E3	2.26E3	2.51E2	1.54E1	4.59E2	3.58E2	3.88E3	2.98E3	8.34E0	4.04E4	
		Bottom Connector					1.2E-2	6.4E-3					
		Total	5.63E3	2.26E3	2.51E2	1.54E1	5.41E2	4.04E2	3.88E3	2.99E3	1.11E1	4.04E4	
E01-01	897	Sleeve			≤1.9E0	3.60E-1	3.64E1	2.03E1		≤1.5E1	1.81E0		4.69E-1
		Spine			≤6.7E-1	≤1.6E-1	1.45E1	8.12E0		≤6.3E0	2.39E-1		1.5E-1
		Fuel	5.10E3	1.34E3	2.58E2	1.52E1	4.68E2	3.69E2	6.00E3	3.13E3	7.92E0	<i>b</i>	
		Bottom Connector					4.2E-2	2.8E-2					
		Total	5.10E3	1.34E3	2.58E2	1.56E1	5.39E2	3.97E2	6.00E3	3.13E3	9.97E0		
F05-05	897	Sleeve			≤3.6E0	≤5.9E-1	6.79E1	3.72E1		≤3.0E1	2.79E0		≤7.9E-1
		Spine			≤1.0E0	≤1.8E-1	1.67E1	9.23E0		≤8.0E0	2.34E-1		≤1.6E-1
		Fuel	5.52E3	<i>b</i>	3.10E2	2.01E1	4.22E2	3.46E2	<i>b</i>	3.50E3	2.69E0	<i>b</i>	
		Bottom Connector					6.10E-2	4.3E-2					
		Total	5.52E3		3.10E2	2.01E1	5.07E2	3.92E2		3.50E3	9.71E0		

^aThese nuclides were only found sporadically in the sleeve, spine, and bottom connector.

^bDecay time before measurement was too long to permit detection.

^{233}Pa . Except for elements E06-01 and F05-05, where the shorter-lived ^{141}Ce and ^{233}Pa (and ^{103}Ru for F05-05) had decayed too much to permit detection, all these nuclides were observed in the fuel. The decay time before measurement for all elements was too long to permit detection of such nuclides as ^{131}I (8-day half-life) and ^{140}Ba (12-day half-life). Generally, the nuclides ^{134}Cs , ^{137}Cs , and ^{154}Eu were in all parts of the elements. Perhaps as a result of fuel contamination, the other nuclides, except $^{110\text{m}}\text{Ag}$, were found sporadically in the graphite components. Generally, $^{110\text{m}}\text{Ag}$ was found only in the cooler regions of the spine and sleeve near the bottom of the elements.

Although $^{110\text{m}}\text{Ag}$ could not generally be detected in fuel compacts by nondestructive gamma-spectroscopy because of masking by higher level gamma emitters, it was observed in the two bottom compacts of element E11-07, where it probably had condensed from the purge gas. It was not found in the bottom connectors in which the charcoal fission product traps were located. However, the detection of very low amounts of $^{110\text{m}}\text{Ag}$ could have been masked by higher level nuclides. Although $^{110\text{m}}\text{Ag}$ could not be measured in the fuel, and the fraction of the total amounts released from the fuel could not be computed, the fraction released is thought to be rather large.

Except for two fuel elements, E06-01 and E14-01, the fraction of ^{134}Cs and ^{137}Cs released from the fuel and deposited in the sleeves and spines was appreciable. Element E06-01 was operated at power for only 384 equivalent full-power days (EFPD) and did not attain temperatures as high as some of the end-of-life (EOL) elements. Element E14-01 had a spine that contained boron carbide poison and likewise operated at lower temperatures. It is interesting that even though nearly 20% of the ^{137}Cs in three of the elements was found in the sleeves and spines, less than 1% was in the fission product traps. The release of cesium from fuel and its transport by the purge gas are discussed below.

1.2.2 Release of Cesium from Fuel and Transport by the Purge Gas

The distribution of the activity ratios of ^{137}Cs to ^{95}Zr and ^{134}Cs to ^{95}Zr in the fuel are shown in Figs. 1.1, 1.2, and 1.3 as a function of location (compact numbers) for fuel elements F03-01, E01-01, and F05-05. According to initial estimates, the highest temperature region of the fuel (around compact 16) for these elements is about 800°C higher than at compact 1 (the bottom compact).^{7,11,12} As previously argued⁹ for

Fig. 1.1 Element F03-01 Compact Inventory Ratios of $^{134}\text{Cs}/^{95}\text{Zr}$ and $^{137}\text{Cs}/^{95}\text{Zr}$ as of October 31, 1974.

fuel element E11-07, the drop in the ratios that extends from about compact 12 to 26 and the rise in the ratios centered roughly around compact 7 indicate that the cesium isotopes were significantly released from the higher temperature regions and then carried by the purge gas to the lower temperature regions, where they then condensed. Published plots^{7,11,12} of the cesium activities in the spines of these elements also indicates a tendency for these isotopes to move toward the bottom of the fuel, but similar profiles in the sleeves do not show this effect. Presumably this difference between spines and sleeves is due to the higher temperatures of the spines.

The extent to which ^{137}Cs evolved from high-temperature fuel compacts and condensed on cooler compacts was estimated by comparing the amount observed in each compact with the amount estimated to have formed. To

Fig. 1.2. Element E01-01 Compact Inventory Ratios of $^{134}\text{Cs}/^{95}\text{Zr}$ and $^{137}\text{Cs}/^{95}\text{Zr}$ as of October 31, 1974.

estimate the activity of ^{137}Cs formed in the compacts, we assumed that insignificant amounts of ^{137}Cs were lost from the whole element. The ^{137}Cs in the whole element was totaled along with the ^{95}Zr in the fuel (^{95}Zr was not evolved from the fuel) and the ratio of the total ^{137}Cs to ^{95}Zr was computed. We also assumed that the ratio of ^{137}Cs to the ^{95}Zr formed in the compacts was constant and equal to the ratio for the whole element. The ^{137}Cs activity formed in each compact was then computed by multiplying the observed ^{95}Zr activity by the overall ^{137}Cs to ^{95}Zr ratio. The difference between the observed ^{137}Cs in a compact and the amount estimated to have formed is the activity that is either lost or gained by the compact. This procedure of estimation thus resulted in an activity

Fig. 1.3. Element F05-05 Compact Inventory Ratios of $^{134}\text{Cs}/^{95}\text{Zr}$ and $^{137}\text{Cs}/^{95}\text{Zr}$ as of October 31, 1974.

balance of ^{137}Cs for the element where the amount lost by the hotter fuel was equal to the amount gained by the cooler fuel plus that in the spine and sleeve.

The results of the activity balances for the Peach Bottom elements examined are listed in Table 1.3. The amounts lost from the fuel are also tabulated as percentages; the reader will note that losses for the fuel of elements E11-07, F03-01, E01-01, and F05-05 ranged between 20 and 29%. The losses of ^{137}Cs from the fuel of elements E06-01 and E14-01 were very small, and the $^{137}\text{Cs}/^{95}\text{Zr}$ ratio was a smooth, nearly constant, function, as previously shown.^{8,10} No definite losses of ^{137}Cs from the fuel compacts were derived.

It should be noted that the assumption of a uniform distribution of the ratio of the ^{137}Cs and ^{95}Zr formed in the fuel is not strictly valid.

Table 1.3. Activity Balance for ^{137}Cs Movement in Peach Bottom HTGR Fuel Elements

Element	Total (Ci)	Lost from Fuel		Activity, Ci, Gained by:		
		(Ci)	(%)	Cold Fuel	Sleeve	Spine
E06-01	174	<1	<0.6	<1	0.03	0.01
E11-07	336	66	20	41	17	8
E14-01	439	<1	<0.2	<1	0.2	0.04
F03-01	404	101	25	55	36	10
E01-01	397	116	29	88	20	8
F05-05	392	82	21	36	37	9

Published fission yields¹⁴ show that the ratio for fission (both thermal and fast neutron) of ^{233}U is larger than for fission of ^{235}U . Since the ratio of ^{233}U to ^{235}U is larger in the central regions of the fuel elements than on the ends, the ^{137}Cs to ^{95}Zr ratio in the center should also be larger. This effect was neglected in the above calculations.

Although the activity distributions of ^{134}Cs and ^{154}Eu in the fuel elements were similar to those of ^{137}Cs , the formation of each of these nuclides is complex (depending on the square of the neutron flux, the neutron energy spectrum, and possibly other subtle factors), and no attempt was made to estimate their activity balances.

1.2.3 Fuel Particle Failure Fractions

Fuel particle failure fractions in eight compacts, taken from two irradiated Peach Bottom fuel elements, were basically determined by the hot-chlorine leaching procedure described by LaValle.¹⁵ In this method, the mass of thorium and uranium removed by chlorination is used as a measure of the degree of particle failure. However, we had to modify the procedure, since the Peach Bottom fuel compact was too large for the existing chlorination apparatus, which was designed for HTGR fuel rods. A simple method was therefore devised for cutting a sample (~ 10g) from the compact and

electrolytically cleaning the cut surfaces in a hot cell. Cleaning was necessary to remove fuel particles damaged in sample cutting to keep them from being counted as normally failed particles. A cleaned surface was obtained by a controlled, partial deconsolidation of the sample at room temperature, by use of a movable anode placed adjacent to the damaged surface of the specimen.

Before the hot-cell work, the method was tested on samples from an unirradiated archive compact, with results which are summarized in Table 1.4. Each 8- to 9-g sample contained 14,000 to 17,000 fuel particles. The two leach conditions tested showed similar failure fractions. The measured thorium-to-uranium ratio in the leachate corresponded closely to the expected ratio of 5.5, thereby lending confidence to the procedure. The last two columns in Table 1.4 indicate the calculated particle failure fraction based on a mean particle mass of 198 μg observed for the archive compact, and values of 81.3 μg of thorium and 15.0 μg of uranium per kernel, based on the observed mean kernel size and the theoretical density of $(\text{Th}, \text{U})\text{C}_2$. Both thorium and uranium leach masses yielded an average failure fraction of 0.28% for the four archive compact samples.

Table 1.4. Particle Failure Fractions for Peach Bottom Archive Compact

Sample Mass (g)	Particles		Chlorination Conditions		Metal Recovered		Weight Ratio Th/U	Failure Rate % Based on:	
	Mass (g)	Number ^a	Time (hr)	Temperature (°C)	Th (mg)	U (μ)		Th	U
8.6586	2.8585	14,400	46	1000	3.47	635	5.46	0.296	0.294
8.4610	2.8573	14,400	46	1000	3.31	581	5.70	0.283	0.269
9.9006	3.3384	16,900	6	1500	3.25	619	5.25	0.236	0.244
9.3094	3.4566	17,500	6	1500	4.36	800	5.45	0.306	0.305
Av = 0.28								0.28	

^a198 μg /particle.

^b81.3 μg Th, 15.0 μg U per particle.

We subsequently determined (by total deconsolidation and analysis of electrolyte, matrix, and residue) that about 64% of this archive failure fraction was in fact due to heavy metal contamination of the matrix. Thus, the observed archive failure rate was actually 0.10%, with the balance (0.18%) due to matrix contamination.

Particle failure fraction results are shown in Table 1.5 for eight fuel compacts contained in two fuel elements. Element E11-07 recieved 700 EFPD irradiation, while F03-01 was an EOL element, receiving 900 EFPD irradiation. Table 1.5 shows that usually the measured Th/U ratio in the leached heavy metal is less than that predicted from neutronics considerations. This was possibly caused by the greater difficulty in completely capturing ThCl_4 due to its lower vapor pressure relative to UCl_4 . Consequently, the failure fraction determinations based on leached uranium, which are on the average higher values, are probably more reliable.

Table 1.5. Particle Failure Fraction in Irradiated Peach Bottom HTGR Fuel

Element	Compact	Weight Ratio Th/U		Failed Particles, % Based on:	
		Calculated	Measured	Th	U
E11-07	2	7.11	7.64	0.68	0.64
	13	7.82	4.83	0.25	0.41
	16	7.78	9.58	0.22	0.18
	28	6.94	3.68	0.33	0.63
F03-01	2	6.51	3.54	0.17	0.31
	13	8.17	6.80	1.55	1.87
	19	7.86	3.43	0.35	0.79
	28	6.46	5.43	0.27	0.33
Archive (av of 4)		5.50	5.47	0.28	0.28 ^a

^a0.18% due to matrix contamination.

From these limited uranium-based data, we can nevertheless conclude that: (1) the percent of failed particles at 700 EFPD (element E11-07) is only slightly higher than the background value of 0.28% obtained for the archive; (2) the percent of failed particles for the 900 EFPD case (F03-01) averaged 0.55% above background, compared with 0.19% for 700 EFPD, and since the two elements were irradiated at approximately equal temperatures.

a slight irradiation service effect on failure fraction is probable; and (3) the percentage of particle failure at EOL was probably less than 1%, of which about one-fourth was background (i.e., due to manufactured failures and matrix contamination).

1.3 MIGRATION OF ^{90}Sr IN PEACH BOTTOM HTGR FUEL ELEMENT GRAPHITE¹⁶ — M. Jonathan Haire

The principal assumptions used to calculate strontium release through the sleeves of the Peach Bottom fuel elements were that: (1) transport in graphite may be characterized by an effective diffusion coefficient depending solely on graphite temperature, and (2) the transport rate across the helium boundary layer is governed by a conventional mass transfer coefficient assuming the inner film partial pressure to be equal to the vapor pressure of strontium at the predicted surface concentration. Verification of this method was sought by fitting concentration profiles calculated with the FIPER code¹⁷ with observed Peach Bottom HTGR data.^{1,7,9-11} Figure 1.4 shows typical radial profile data compared with calculated profiles. Dashed lines, A, show the profiles in the fuel element sleeve and spine calculated with effective diffusion coefficients and vapor pressures used in larger HTGR design calculations,¹⁸ assuming that 100% of the ^{90}Sr inventory in the fuel compact was available for release into the fuel-free graphite. Dashed lines, B, are drawn through the experimentally observed ^{90}Sr radial profiles. Best fit profiles (shown by solid lines, C) were obtained by adjusting the values of the source strength and the effective diffusion coefficient.

At temperatures above about 960°C, such as in the spine region shown in Fig. 1.4, this curve fitting method reproduced ^{90}Sr concentration profiles fairly well. Below this temperature, tails were observed in the concentration profile, as shown in the exterior of the sleeve in Fig. 1.4, which could not be duplicated by using the simple assumptions described above. The high strontium concentrations observed at the spine and sleeve surfaces (relative to the calculated profiles) may have been caused by a radial variation of graphite properties due to nonuniform distribution of graphite impregnant. Table 1.6 lists the diffusion coefficients determined from the best fit of the data normalized to 1000°C. For comparison, the recommended¹⁸ design value at this temperature is $1.68 \times 10^{-13} \text{ m}^2/\text{s}$.

Fig. 1.4. Radial Distribution of ^{90}Sr in E01-01 at Fuel Compact 28.

Figure 1.5 compares the fitted effective diffusion coefficients and the HTGR design¹⁸ recommendations. Above about 960°C, where the fitting procedure worked well, fairly good agreement was achieved between the design and best fit values.

The total release of ^{90}Sr from the core into the primary coolant was estimated from the average calculated release from the three fuel elements examined. The calculated whole-core strontium release-to-birth ratio was calculated to be far below the reference design value of 10^{-4} . Comparison of the calculated strontium release with that

Fig. 1.5. Strontium Effective Diffusion Coefficients Determined from Peach Bottom Core 2 Concentration Profiles. (Compact Numbers Indicated on Data Points).

observed for cold duct samples and plateout probe values obtained in the course of the Peach Bottom Surveillance Program yielded ambiguous results, primarily because of the difficulty in distinguishing between Core 1 (which experienced high releases) and Core 2 releases.

Table 1.6. Summary of Strontium Diffusion Coefficients

Fuel Element	Axial Position (Fuel Compact)	Spine Diffusion Coefficient, m^2/s	Sleeve Diffusion Coefficient, m^2/s
E11-07	12	3.5×10^{-14}	6.0×10^{-15}
	18		2.0×10^{-14}
	28		5.0×10^{-16}
E14-01	7	6.0×10^{-15} 2.0×10^{-17}	6.0×10^{-15}
	23		6.0×10^{-15}
	28		2.0×10^{-15}
E01-01	18	2.3×10^{-14} 3.0×10^{-14}	8.0×10^{-14}
	23		
	28		1.0×10^{-14}
F05-05	12	2.5×10^{-14}	2.0×10^{-12}
	18	3.0×10^{-14}	1.78×10^{-13}
	28	3.2×10^{-15}	1.0×10^{-14}

^a Assuming an activation energy of 268 kJ/mol. In the sleeve only a portion of concentration profile was fitted.

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2. FUELED GRAPHITE DEVELOPMENT

F. J. Homan

2.1 INTRODUCTION

The period covered by this report was a year of transition for the Fueled Graphite Development Program. Emphasis of the fuel development effort switched from the high-enriched uranium (HEU) cycle to the low-enriched uranium (LEU) cycle and then to the medium-enriched uranium (MEU) cycle. The current development reference is a dual-particle system (fissile and fertile), where the fissile particle contains 20%-enriched uranium and the fertile particle contains ThO_2 . A "unified" program plan was written by GA and ORNL personnel, organized around three goals:

(1) develop and qualify an LEU or MEU fuel for use in proliferation-resistant fuel cycles in HTGRs, (2) complete the fuel development work that is generic to LEU, MEU, and HEU cycles, and (3) complete the development of HEU fuel to the point of proof testing. This new program plan supercedes the plan described in the last annual report.¹ This change in emphasis has brought about a closer integration of the ORNL and GA fuel development efforts. In addition to the switch of reference fuel cycle from HEU to MEU, GA decided during this year to utilize ORNL reactors (HFIR and ORR) as their mainline vehicles for fuel testing. All HT, HRB, and ORR capsules in the future will be shared between GA and ORNL. Further, the new unified program plan does not distinguish between fresh and recycle fuel, as did the old program plan. Under the old program plan ORNL was primarily responsible for recycle fuel development, while GA had responsibility for fresh fuel development. Fresh and recycle fuel will probably be the same for the MEU fuel cycle. We also anticipate that the fuel developed for the MEU cycle will be sufficiently versatile that it will be compatible with LEU or HEU cycles as well. A close integration between GA and ORNL programs has evolved for development of this "unified" fuel.

The ORNL Program has the same mix of activities discussed in past annual reports. This year's report contains discussions of work done during the transition from the HEU to MEU fuel cycles and during the transition from emphasis on recycle fuel to emphasis on MEU cycle fuel, which will serve both as fresh fuel and recycle fuel. Continued integration of the ORNL and GA programs will occur during 1978.

2.2 FUEL PARTICLE PROCESS DEVELOPMENT AND QUALIFICATION

The work described in this section is concerned with coating studies; coating services for irradiation testing; studies of fission product attack of SiC; and studies of the influence of aging ^{233}U -containing fuel kernels on subsequent processing of these kernels. Partial reduction of ThO_2 fertile kernels (Sect. 2.2.1) is being evaluated both to reduce the thickness of the buffer layer required for the fertile particle and to increase the thermal stability of the ThO_2 kernel under irradiation. The SiC coating studies (Sect. 2.2.2) were accomplished to determine the influence of coating conditions on the SiC microstructure and density. We anticipate that these studies will be continued in 1978 to determine the influence of coating microstructure on fission product migration and attack in SiC. The coating service work was done in support of irradiation experiments (Sect. 2.2.3) and to provide SiC-coated particles containing fission product kernels for studies of corrosion kinetics under well-controlled temperatures and temperature gradients (Sect. 2.2.4). The ^{233}U -self-radiolysis work (Sect. 2.2.5) was supported by the HTGR Recycle Program in conjunction with the HTGR Base-Technology Program. This work was oriented at establishing the effect of storing Weak-Acid-Resin (WAR) derived kernels, loaded with ^{233}U , on subsequent chemical processing of the kernels (carbonization and conversion).

2.2.1 Buffer Coating Studies and Partial Conversion of ThO_2 — J M Robbins

Future high-conversion HTGRs will require fuel with increased heavy-metal loadings. Minimizing particle coating thickness together with partial carbon-thermic reduction of the ThO_2 kernel to create void volume tailored to metal content is a possible means of fabricating fuels with increased heavy-metal loadings.²

Several buffer coating runs were processed with 25-g charges of ThO_2 kernels coated at 1500°C with 25 vol % MAPP gas diluted with argon. These conditions resulted in the best compromise between coating density and standard deviation on the coating thickness. The results of the coating runs are shown in Table 2.1. These runs indicate that under a given set of conditions buffer coatings with a density of about 1.4 Mg/m^3 and a standard deviation on thickness of about $3 \text{ }\mu\text{m}$ can consistently be applied from MAPP gas. By lowering the standard deviation to $3 \text{ }\mu\text{m}$, the mean buffer thickness

requirement is lowered from 85–90 μm to 60–65 μm . Obviously, this would provide a significant improvement in heavy-metal loading and potential core conversion ratio.

Table 2.1. Buffer Coatings with Lowered Standard Deviation on Thickness Can be Applied Using MAPP Gas^a

Run	Density (Mg/m^3)	Mean Thickness (μm)	Standard Deviation	
			(μm)	(%)
OR-2689	1.14	45.9	2.02	4.4
OR-2691	1.43	49.4	2.80	5.7
OR-2692	1.41	51.1	2.93	5.7
OR-2693	1.41	47.3	2.94	6.2
OR-2694	1.41	48.8	2.94	6.0
OR-2695	1.42	48.1	2.54	5.3
OR-2703	1.34	41.5	2.05	4.9
OR-2704	1.40	42.6	1.90	4.5
OR-2705	1.38	42.4	2.11	5.0

^aCommercially available mixture of methylacetylene and propadiene with alkanes as stabilizers.

Current specifications³ call for a buffer density of 0.9–1.2 Mg/m^3 . Therefore, with the higher buffer density of 1.4 Mg/m^3 , additional void volume must be provided to at least equal that in a buffer no denser than 1.2 Mg/m^3 . This void volume is required to accommodate the fission gases generated during the fuel lifetime and could possibly be tailored to the heavy-metal content of the fuel. Early efforts to produce the additional void volume were by partial carbothermic reduction of the ThO_2 kernel.² Figure 2.1 shows the erratic behavior of the conversion process both within and between particles.

Figure 2.1(a) shows a desirable thin layer of ThC_2 extending around about three-fourths of the kernel. Figure 2.1(b) shows reaction around about one-fourth of the kernel only, and the reaction layer is much thicker than in the first photograph. A large portion of the kernel shown in Fig. 2.1(c) has reacted to ThC_2 , completely surrounding the kernel.

Fig. 2.1 Attempts to React ThO₂ Kernels with Their Buffer Coatings by Partial Carbothermic Reduction Results in Erratic Conversion. (a) Thin converted layer around three-fourths of the kernel. (b) Thick converted layer around one-fourth of the kernel. (c) Thick layer surrounding the kernel.

Interestingly, the three particles shown in Fig. 2.1 were all taken from the same furnace run, which had operated at 1975°C for 30 min. The photographs in Fig. 2.2 are shown at 750× to reveal the phases formed by heating a ThO₂ kernel with a buffer coating to 1975°C for 30 min. Figure 2.2(a) depicts the original ThO₂ kernel material as phase 4 and the buffer and outer carbon coatings as phase 1. Phase 3 is believed to be ThC₂, while phase 2 remains unidentified.

The best guess at the identity of this phase is that excess carbon has been dissolved by the carbide phase at phase 3 then redeposited from solution as a thorium-contaminated graphite at phase 2. Figure 2.2(b) shows the same phases in polarized light.

Future work will be directed toward identification of phase 2 in Fig. 2.2 as well as the control of the erratic conversion process. Better control of the process could possibly be achieved by slowing the reaction, especially in the beginning, to allow a skin of ThC₂ to form around the spherical ThO₂ kernel; then CO diffusion from the reduction should proceed more evenly. Also, CO additions could possibly be used to control the rate of reaction.

2.2.2 SiC Coating Studies — J. I. Federer

A study of the effects of coating conditions on SiC coating characteristics has been completed and the results have been published.^{4,5} The principal coating variables were temperature and reactant gas composition expressed as the volumetric ratio of hydrogen to methyltrichlorosilane (H₂/MTS). These were correlated with coating rate, morphology, composition, microstructure, and density. The results show that highly dense coatings can be deposited at higher coating rates than are usually used in HTGR fuel preparation. In fact, coating characteristics depend upon coating conditions rather than coating rate, and acceptable coatings can be produced over a wide range of coating rates if the proper coating conditions are used. A brief summary of the main findings of this investigation follows.

Coating rate is a linear function of MTS flux if temperature and H₂/MTS are within the ranges required for highly dense coatings. The maximum coating rate is obtained at the highest MTS flux and associated H₂/MTS possible without ejection of microspheres from the coating tube. The

Fig. 2.2. Carbothermic Reduction of ThO_2 at 1975°C for 30 min Results in the Expected Formation of a ThC_2 Phase (3) Plus an Unidentified Layer Shown at (2). (a) Bright field. (b) Polarized light.

minimum H_2/MTS for highly dense coatings is about 20, and H_2/MTS ratios of about 20 or more generally ensure high density ($>3.190 \text{ Mg/m}^3$) if coating temperature is within the optimum range. The optimum temperatures for highly dense coatings are within the approximate range 1475 to 1675°C. Below 1400°C the density defect is in the form of excess silicon, while above 1700°C unacceptable porosity is likely.

Coatings deposited at an H_2/MTS of about 20 or more at a temperature within the optimum range have a distinct grain structure, whereas coatings deposited at low H_2/MTS and/or low temperatures have a striated microstructure, in which grains are not readily distinguished.

Coating rate does not significantly affect density and microstructure when the coatings are deposited under optimum conditions. Coatings deposited at rates of 3.5 to 68.5 nm/s had densities in the range of 3.192 to 3.201 Mg/m^3 and similar microstructures.

2.2.3 Particle Coating in Support of Irradiation Experiments — J M Robbins

Two batches of carbon kernels were coated for irradiation in Capsule HT-34. Conditions were designed to duplicate the coatings applied to two batches of particles (OR-1975-T and OR-2013-ST) that were irradiated earlier in Capsules HT-17 through -19.⁶ The HT-17 through -19 batches were Bisco-coated ThO_2 and showed some very interesting and unexpected behavior in that experiment. The coating batches designed to duplicate the properties of OR-1975-T and OR-2013-ST were designated OR-2722-I-T and OR-2729-I-T, respectively.

Batch OR-1975-T was a Bisco-coated ThO_2 kernel of the fertile reference design. It was expected to survive all temperatures and fluences in the HT-17, -18, and -19 series. Consequently, it was irradiated in HT-19 only, at the highest temperature and fluence. Instead of complete survival as expected, the particles showed a failure rate exceeding 90%. Batch OR-2013-ST was also coated to the fertile reference design except that the carrier gas was CO_2 instead of argon. The purpose of the CO_2 was to minimize soot inclusions in the coating. This particle was irradiated in both HT-18 and -19 in the highest temperature magazines and survived completely.

Because of the unexpected behavior of these two particles in HT-17, -18, and -19, it was desirable to irradiate them again in HT-34. Also, since irradiated ThO_2 particles cannot be removed from the hot cells for

examination because of the high activity, it became desirable to duplicate the coatings from the aforementioned ThO_2 particles onto carbon kernels and irradiate them. The carbon-coated carbon particles can be removed from the hot cells after irradiation so important properties, such as permeability, can be measured. However, several problems are involved in trying to duplicate coatings on carbon by using the same fabrication conditions that were used in coating ThO_2 . The parameters to be duplicated were coating thickness and density accomplished at the same temperature and deposition rate from the same hydrocarbon and gas concentration. These problems were minimized by using a mixed bed of carbon and ThO_2 kernels for the coating charge. This kept the bed agitated and allowed it to be fluidized somewhat like a bed of ThO_2 kernels only. These particles have been irradiated and removed from the reactor and are currently awaiting postirradiation examination.

2.2.4 Triso Coating of Simulated Fission Products — J M Robbins and C. Hamby, Jr.

Several batches of crushed and sized La_2O_3 , Sm_2O_3 , Nd_2O_3 - UO_2 , and Nd_2O_3 - UO_2 - UC_2 particles were Triso coated to use in the study of chlorine movement in particles and rare-earth fission-product reactions with the SiC in Triso coatings (Sect. 2.10). Later, this work was extended to include the following fission-product mixes: (1) UO_2 - SrO - ZrO_2 , (2) UO_2 - SrO - ZrO_2 - Ce_2O_3 - Nd_2O_3 , (3) SrZrO_3 , (4) Mo - Ru - Rh - Pd , (5) Mo - Ru - Rh - Pd - UC , (6) Mo - Ru - Rh - Pd - UC_2 - UO_2 , (7) UO_2 - Ag . The small batches, irregular shapes, and high densities associated with most of these materials resulted in fluidization difficulties during conversion and coating. Some materials, like UO_2 - Ag with a low melting component, required significant deviation from normal coating procedures. Determination of a unique and appropriate set of processing variables was required for nearly every material. Despite these difficulties, acceptable process parameters were established and 33 conversion and coating runs were completed on these materials.

2.2.5 Effect of Self-Radiolysis on Subsequent Chemistry of ^{233}U Loaded Weak-Acid-Resin (WAR) Kernels — J M Robbins and W. P. Eatherly

An experiment to assess the possible effects of self-radiolysis on subsequent chemical conversion and reduction of two polyacrylic acid

resins fully loaded with ^{233}U was essentially completed. The experiment was a cooperative effort of the Chemical Technology, Analytical Chemistry, and Metals and Ceramics Divisions. The overall description and experimental rationale are reported elsewhere.⁷ Here we discuss the testing procedures and measurements to which the materials were subjected together with data evaluation and conclusions.

The as-received test materials were both Amberlite IRC-72* and Duolite C-464[†] cation exchange resins, which had been fully loaded with ^{233}U from uranyl nitrate solution. Samples of the same resins similarly loaded with ^{238}U were processed as controls. The uranium-loaded WAR samples had been dried at 100°C for 16 hr before receipt and were handled exclusively in an inert atmosphere box in subsequent operations.

The dried WAR samples were subjected to the following operations and examinations:

1. A 35-mm film was made on each sample before and after carbonization and conversion for comparison.
2. A tapped density was determined on each total sample before and after carbonization and conversion.
3. Each sample was radiographed before and after carbonization and conversion so that size and shape of the particles could be determined.
4. The total sample was carbonized at 900°C, then partially converted to UC_2 at 1500°C for 20 min. The argon flow was controlled such that the conversion target was about 39%.

In addition to the above procedures, the following operations and evaluations were done after carbonization and conversion:

1. Duplicate samples were riffled for carbon and uranium determinations so the conversion could be calculated. Oxygen content was calculated by difference.
2. Crushing strength was measured so that samples could be compared.
3. The samples were shape-separated and percentage rejects calculated so differences between samples could be detected.

*Trademark of Rohm and Haas Corporation.

[†]Trademark of Diamond-Shamrock Corporation.

Riffled archive samples and all excess material were stored in a glove box under a constantly monitored atmosphere that contained less than 2 ppm moisture and less than 6 ppm O₂.

The experimental work for this project has been completed and the final report is being drafted. Therefore, only a summary of the conclusions will be presented here, and the experimental data will be reserved for presentation in the final ORNL report.

2.2.5.1 Tapped Density

A density value was obtained both before and after conversion by measuring the volume of a weighed charge of the particles in a graduated cylindrical container. A volumetric packing fraction of 60 vol % was used to convert the observed apparent volume to a true volume.

One significant effect found in analyzing the tapped density measurements was a noticeable decrease in the ²³³U-loaded particle density, both before and after conversion, when the sample had been stored in the fully wet condition. Statistics support the conclusion that the densities of the dried resins are unaffected by storage as dried or partially dried resin, but decrease with time when the resin is stored under water. This decrease in density was not evident in the wet-stored ²³⁸U-loaded resin particles.

2.2.5.2 Diameters

Before and after conversion, the particle diameters were measured with a microscope with a calibrated filar eyepiece. Statistically we found no resolvable difference in particle diameter due to length or condition of storage. The distribution of particle diameters is too broad to reflect the observed density differences if such were totally attributable to kernel volumes.

2.2.5.3 Photographs

Each material was photographed both before and after conversion by the use of a 35-mm camera loaded with Plus Pan-X* film. No obvious

* Trademark of Eastman-Kodak Corporation.

effects of storage time and conditions were apparent on any materials until the samples stored longer than three months were examined. In these samples, the ^{238}U -loaded kernels exhibited a darkening of some of the particles relative to the usual light yellowish or brownish color. This gave to the particles a "salt and pepper" effect, which increased in intensity with increased wetness during storage. The ^{233}U -loaded kernels did not show this phenomenon. However, the ^{233}U -loaded samples stored longer than three months in the fully saturated condition did exhibit degradation of the surface of the kernels. This degradation was far more severe for the Duolite resin than for the Amberlite, and was reflected as an extreme dusting, which prevented processing of the material.

2.2.5.4 Carbonization and Conversion

These materials were carbonized and converted as a single operation in a fluidized-bed furnace. Each sample was processed as nearly identically as possible. After the sample was fluidized in the furnace tube with argon, the temperature was increased as follows:

1. ambient temperature to $250^{\circ}\text{C} - 10^{\circ}\text{C}/\text{min}$,
2. 250 to $500^{\circ}\text{C} - 2^{\circ}\text{C}/\text{min}$,
3. 500 to $900^{\circ}\text{C} - 20^{\circ}\text{C}/\text{min}$.

At this point carbonization was considered complete. To convert, the temperature was brought rapidly to 1500°C (approximately $200^{\circ}\text{C}/\text{min}$) to partially convert the uranium to UC_2 . The argon flow was adjusted to the proper flow to obtain about 39% conversion in 20 min. After conversion, the material was cooled and stored under argon.

A subject of considerable concern has been the ability to predict the conversion level of a batch of material before heat treatment. The data used to predict the conversion were those tabulated by Weber et al.⁸ Assuming that the observed conversion temperature was known perfectly, the range in the conversion level obtained with the Amberlite resin is statistically consistent with the predictions of ref. 8. However, Duolite tends to convert to higher levels than Amberlite under the same conditions. Although the conversion levels with Amberlite were statistically consistent with predictions, scatter in the results is far beyond that expected from

the accuracy and precision of the chemical analyses for carbon and uranium. Hence, the question still remains open whether the poor predictability is attributed to oxygen concentrations calculated from differences (doubtful), to lack of process control (probable), or both. No difference in conversion levels for ^{233}U - and ^{238}U -loaded resins was discernable under these conditions.

2.2.5.5 Carbon and Uranium Concentrations

These analyses showed no differences attributable to time and storage conditions. No differences were noted between the ^{233}U - and ^{238}U -loaded resins and, even in the fully saturated stored condition uranium loss in the supernatant liquid was negligible.

2.2.5.6 Shape Separation

Particles from each sample of material were shape separated on a vibrating inclined plane under presumably the same conditions for each sample. The particles were weighed and the percentages rejected were compared batch-to-batch. These data were too widely scattered to permit any conclusions whatsoever.

2.2.5.7 Crushing Strength

Crushing strength was determined by loading single particles between two platens until crushing occurred. This load was transmitted to a force gage equipped with a dial indicator. Data were widely scattered, but some differences were statistically significant. Although there were individual exceptions, there appeared to be a definite trend for the materials stored dry (5-10% moisture) and intermediately wet (10-15% moisture) to become weaker than the material that was stored under water. Some indications indicated that this was true for both ^{233}U - and ^{238}U -loaded resins.

2.2.5.8 General Conclusions

The total dosage in rads has not yet been calculated, so we will use storage time in months as an indication of self-radiolysis. It should be kept in mind that this material contained 7 ppm ^{232}U , and hence the dose

rate was small. For less isotopically pure materials, the time for equivalent dose would be much reduced. With this stipulation, the following conclusions can be drawn:

1. Contrary to previous claims, Duolite and Amberlite apparently convert with different activation energies. This experiment was not specifically designed to determine activation energies, and hence this conclusion requires verification.

2. Storage in dried, intermediate, or wet conditions up to three months leads to marginally discernable differences in the final converted kernels. However, these differences should not affect the final coated-particle behavior.

3. Storage up to six months leads to serious surface degradation, with dust being generated from the deteriorating surface. At the very least this would lead to furnace contamination and loss of uranium from the process stream.

Because of limited funding, this experiment needs to be repeated with higher statistical reliability if resin-derived kernels are to be used as a fuel kernel precursor. However, it is quite apparent that limited-time storage of the loaded resin does not lead to serious deterioration of the product kernel.

2.3 CHARACTERIZATION OF PYROCARBON COATINGS

Most of the PyC characterization work during this reporting period has been associated with determination of permeability of the coatings in Biso particles. Most of this work has been reported (refs. 9-13) and will therefore be summarized only briefly here. Extensive work was done on development and implementation of techniques for characterization of permeability. Of the four techniques now used, two are primarily used on unirradiated material (chlorine leach at 1500°C and inert gas intrusion). We plan to develop the capability of using these techniques on irradiated fuel. The other two techniques (internal gas pressure measurements and TRIGA activation) are already in use on irradiated fuel. The TRIGA activation capability does not exist at ORNL, but is available at General Atomic Company (San Diego, California).

2.3.1 Permeability Determinations Using Inert-Gas Intrusion — C. S. Morgan,
G. L. Powell,^{*} W. P. Eatherly, M. Reeves,[†] and J. S. Tolliver

The permeability of the pyrocarbon coatings of Biso fuel particles was investigated by use of neon-helium intrusion. The Ne/He ratio of gases in the void volume was determined by breaking the PyC coating in a chamber connected to the mass spectrometer after heat treatment at 1375°C for 1 hr in an atmosphere composed of half neon and half helium.¹⁴ This method affords a good comparative measure of the permeability of fuel particles in which the ratio of surface area to void volume does not vary extensively. For typical fuel particles the void space is saturated with helium after 1 hr at 1375°C. Thus, the Ne/He ratio depends primarily on the rate of neon diffusion through the PyC coating.

Significant dimensional variations can cause a misleading Ne/He ratio. For example, if the surface area is large and the buffer void volume small the permeability of the PyC coating indicated by the Ne/He ratio can appear much higher than the actual condition of the coating would support. This anomaly results from the fact that the helium content is set by the void volume and the neon content will be disproportionally increased by the large area to diffuse through and small volume to fill. To obtain true comparative values a single one-dimensional model of a coated particle was assumed. Gas moves through a barrier of admittance F , (the amount of gas flowing through a unit cube of material in unit time), Area A , and thickness L into a finite void of volume v_0 . Then the number of the moles N in the void volume at any time will be:

$$N \approx N_{\infty} [1 - \exp(-FAT/Lv_0)] ,$$

where N_{∞} is the number of moles of gas within the particle void volume at equilibrium (infinite time). Neon-helium ratios have been reported previously as a mole-fraction f :

$$\frac{\text{Ne}}{\text{He}} \equiv f = \frac{N(\text{Ne})}{N(\text{He})} .$$

^{*} Development Division, Oak Ridge Y-12 Plant

[†] Present address, Interra Environmental Consultants, Inc., Houston, Texas.

If the infinite reservoir contains equal numbers of moles (partial pressures) of neon and helium, then in equilibrium:

$$N_{\infty}(\text{Ne}) = N_{\infty}(\text{He}) ,$$

and the time-dependent ratio f becomes

$$f(t) = \frac{1 - \exp(-F_{\text{Ne}} A t / L v_0)}{1 - \exp(-F_{\text{He}} A t / L v_0)} .$$

Experimentally we can select a value for t , say τ , such that

$$(F_{\text{He}} A / L v_0) \tau \gg 1, \quad (F_{\text{Ne}} A / L v_0) \tau = 0(1) ,$$

where $0(1)$ symbolizes a number of order unity. Then

$$f(\tau) \approx 1 - \exp(-F_{\text{Ne}} A \tau / L v_0) ,$$

which permits a unique determination for the neon admittance F_{Ne} . The result is

$$F_{\text{Ne}} = \frac{-L v_0}{A \tau} \ln(1 - f) ,$$

The neon admittance represents the intrinsic diffusion rate through the pyrocarbon coating, although use of a one-dimensional model may be a small error source. The values of F_{Ne} are on the order of 10^{-13} m²/s, which is similar to that for brass.

While F_{Ne} is representative of the intrinsic properties of the PyC coating, the permeability of the fuel particle itself is best described by the term $F_{\text{Ne}} A / L$ with units of m³/s. The relative size of this term best reflects how fast fission gases would diffuse out of a fuel particle.

Most of the light inert-gas permeability measurements made during the year were on fuel particles of approximately the same size, and only the Ne/He ratios were reported. However fuel particles irradiated in HT-12

through HT-15 had various void sizes, some very small. The permeabilities, including F_{Ne} and $F_{\text{Ne}} A/L$ of archive sample of these fuel particles are reported in Table 2.2. Three batches of larger fuel particles are included for comparison. Results in the table demonstrate how the permeability measured by the Ne/He ratio can vary with geometry. The $F_{\text{Ne}} A/L$ values of the three large fuel particles are approximately proportional to and fall in the same sequence as their Ne/He ratios. However, compare the two smallest fuel particles, OR-1830-HT and OR-1837-HT, with OR-2261-HT. The Ne/He ratios show OR-2261-HT to be about one-third as permeable as OR-1830-HT or OR-1837-HT, but the $F_{\text{Ne}} A/L$ values are completely reversed, showing the small particles to be less permeable by factors of 7.4 and 4.4, respectively.

The Ne/He permeabilities were determined on 24 batches of fuel particles prepared in a study to select Biso fuel particle specimens for HT-34. These Ne/He ratios, which ranged from 0.13 to 0.7, are listed in Sect. 2.3.2 of this report along with other pyrocarbon properties. The Ne/He ratios of these coatings were redetermined after they had been heat treated at 1800°C for 30 min. There was generally a small decrease in permeability, with the Ne/He ratios now ranging from 0.04 to 0.64. The free void volume indicated by helium content also decreased slightly. For example, in the case of fuel particle batch J-592 the Ne/He ratio fell from 0.35 to 0.32, while the void volume dropped from 1.9 to $1.6 \times 10^{-11} \text{ m}^3$. The low-permeability particles showed the sharpest permeability decrease; for example, the Ne/He ratio of J-595 went from 0.13 to 0.04.

The effect of irradiation on the permeability of PyC coatings was examined with fuel particles with inert kernels. The activity associated with irradiated fuel prevented Ne/He permeability studies on irradiated fuel particles with thorium or uranium in the kernel. Inert kernel particles from three irradiation tests — HT-30, -31, and -33 — were examined. The HT-30 capsule contained neon during the irradiation, so particles irradiated at 1250°C had high Ne/He ratios due to pickup of neon in the reactor. The specimens in this test at 900°C showed a moderate decrease in permeability, with Ne/He ratios falling from 0.27 for unirradiated particles to 0.23 for particles with $3.5 \times 10^{25} \text{ n/m}^2$ and 0.20 for particles with $7.2 \times 10^{25} \text{ n/m}^2$. For inert particles in HT-31 and -33 (which had no neon in the capsule to

Table 2.2 Pyrocarbon Coating Permeabilities Adjusted for Size Factors

Batch	Average Content per Particle (Std. Deviation), ρ mol		Fuel Particle			Void Volume (μm^3)	Ne/He Normalized for Furnace Position	F_{Ne} (m^2/s)	$F_{\text{Ne}}^{A/L}$ (m^3/s)
	Helium	Neon	PyC Thickness (μm)	Diameter (μm)	Surface Area (μm^2)				
A-642-H	110.5 (28)	31 (6)	79.1	907	25.84×10^9	3.00×10^7	0.27	8.0×10^{-14}	2.61×10^{-13}
OR-1840-HT	28.0 (5)	12 (1)	50.1	552	9.57	0.76	0.49	7.4	1.41
OR-1846-HT	25.0 (5)	18 (3)	21.2	482	7.30	0.68	0.82	9.4	3.24
OR-1849-HT	97.0 (13)	31 (29)	74.9	816	20.92	2.62	0.35	11.2	3.14
OR-1850-HT	50.0 (8)	19 (1.4)	48.1	694	15.13	1.35	0.40	6.2	1.95
OR-2264-HT	118.0 (22)	16 (2)	91.5	883.3	24.51	3.20	0.13	4.6	1.23
OR-1749-HT	17.4 (3.5)	10.1 (2.0)	36.4	540	9.16	0.47	0.55	4.2	1.06
OR-1829-HT	10.7 (1.5)	7.0 (1.2)	30.6	506	8.04	0.29	0.62	3.0	0.78
OR-1830-HT	4.7 (0.6)	2.8 (0.4)	38.1	480	7.24	0.13	0.60	1.6	0.30
OR-1837-HT	6.9 (1.9)	4.1 (0.8)	24.4	452	6.42	0.19	0.62	2.0	0.53
OR-1838-HT	19.0 (4.2)	9.3 (1.8)	63.8	594	11.08	0.51	0.54	6.2	1.08
OR-2261-HT	118.0 (20)	27 (4)	88.9	791.9	19.70	3.70	0.23	10.4	2.30

interfere) the decrease in permeability with neutron fluence was dramatic. This decrease, shown graphically in Fig. 2.3, is evident at least to a fluence of $11.6 \times 10^{25} \text{ n/m}^2$ ($>0.18 \text{ MeV}$). The Ne/He ratio falls to about 30% of the preirradiation value.

This permeability decrease with fluence has been determined for inert particles only and will have to be confirmed for fuel particles that can generate fission gas pressures. In fact, this result conflicts somewhat with the data of Thiele and Bradley.¹⁵ They showed an increase in permeability for Biso-coated ThO_2 particles with irradiation in the BR2-Mol reactor. However, the permeability decrease does demonstrate that neutron damage does not disrupt the pyrocarbon structure in a manner causing greater permeability. This improves the prognosis for fuel particles that are impermeable initially.

Inert gas permeability measurements demonstrated that coatings made at GA and composed of a pyrocarbon-SiC alloy had Ne/He permeabilities in the range normally encountered in pyrocarbon coatings. For four fuel particle batches the Ne/He ratios ranged from 0.17 to 0.33 and void volumes from 3.5 to $4.1 \times 10^{-11} \text{ m}^3$.

Fig. 2.3. Permeability Decreases During Irradiation for Inert Coated Particles.

The inert gas permeability of three fuel particle batches OR-1968-T, OR-1975-T, and OR-2013-T irradiated in HT-17, -18, and -19 were determined on archive samples. The Ne/He ratios of these batches were low, 0.11 to 0.19, indicating that the pyrocarbon coatings were relatively impermeable. During irradiation each batch had 100% survival to design HTGR fluence. Gas content measurements¹⁶ showed that OR-2013-T retained fission gases after substantially exceeding the design fluence. Two fuel particle batches with inert kernels were prepared as nearly as possible to resemble batches OR-1975-HT and OR-2013-HT (which was made with CO₂ as the diluent gas). A third batch is similar to OR-1975-HT except that the seal coats were omitted. However, these inert batches had Ne/He ratios of 0.29 to 0.31, significantly higher than the original preparations, which had ratios of 0.18 and 0.13. The three batches were put in HT-34 to determine the effect of irradiation on the permeability of the PyC coatings.

The Ne/He permeabilities were determined on 33 fuel particle batches prepared in production-scale coating furnaces, partly in GA facilities. The values ranged from 0.11 to 0.69, with two batches too permeable to measure. Most of them are reported in Sect. 2.3.2 of this report.

A limited basic study of pyrocarbon permeability was carried out. Preliminary results of helium and neon isotherms for fuel particle batch OR-2261-HT yielded diffusion coefficients (D) of

$$D_{\text{He}} = 0.010 \exp(-117.0/RT) ,$$

for helium and

$$D_{\text{Ne}} = 0.013 \exp(-251.0/RT) ,$$

for neon (R in kJ/mol K). The concentration (solubility) of the diffusing gas in the pyrocarbon coating was consistent with helium or neon being stored as an ideal gas in closed pores that constitute about 3.5% of the coating volume.

The inert gas permeability of a pyrocarbon coating decreases sharply with increasing molecular weight of the gas, while the activation energy

for permeation increases. Activation energies for fuel particle batch OR-2661-HT were:

Gas	Activation Energy	
	(kJ/mol)	(kcal/mol)
Helium	117	28
Neon	166	40
Argon	251	60

The activation energies for helium permeation of four fuel particle batches with widely varying permeabilities were carefully measured. Permeation isotherms were determined at 550, 750, and 1000°C for each batch, a 450°C isotherm was determined on J-598, and a 1200°C isotherm was included for J-596. Despite the wide range of permeabilities as indicated by the Ne/He ratios, the activation energies are essentially the same, 109 to 112 kJ/mol (26.0 to 26.8 kcal/mol). The results are tabulated in Table 2.3.

Table 2.3. Activation Energy for Helium Permeation of Selected Biso Particles

Batch	Permeability Ne/He	Activation Energy	
		(kJ/mol)	(kcal/mol)
J-596	0.05	111	26.6
J-598	0.64	109	26.0
J-605	0.31	112	26.8
J-606	0.22	112	26.7

The large increase in activation energy with increasing molecular weight of the diffusing species, the nearly identical activation energies for helium permeation through pyrocarbon coatings differing widely in permeability, and the fact that the activation energies are high suggest

that the change of rate of permeation with temperature depends in large measure on structural changes in the PyC coatings. As the temperature increases the passages through which the gas atoms are diffusing increase in size, and this increase occurs similarly in each pyrocarbon coating. The wide difference in permeability from batch to batch is due to varying numbers of the passages in the pyrocarbon structure through which gas atoms move or to varying lengths of the restrictive part of the passages.

To better evaluate the diffusion of gas atoms through the pyrocarbon coatings of fuel particles the diffusion through spherical shell has been studied by a combined analytical and numerical approach. The work done is outlined below. The diffusion of gas into a spherical shell is characterized by the equation

$$\frac{dc}{dt} = \frac{D}{r} \frac{d}{dr^2}(rc) \quad , \quad (1)$$

where c is the bulk concentration in units of mass per unit volume. We let the shell have inner radius a and outer radius b and let the core be described by porosity s while the shell has porosity n . The boundary conditions require that the pressure be equal on both sides of the interface between shell and core, and we assume that s is large compared with n so that the gas is dispersed throughout the core material immediately after crossing the interface. Then, the bulk concentration inside the core is given by cs/n where c is the solution to the above equation evaluated at $r = a$. Thus, the total gas uptake by the core is

$$U = Vcs/n \quad , \quad (2)$$

where U is the uptake and V is the volume of the core.

Equation (1) is solved by a Laplace transform technique, the details of which are described by Reeves and Tolliver.¹⁷ Using this solution to Eq. (1), we obtain from Eq. (2),

$$U = \gamma \bar{c} \quad , \quad (3)$$

where

$$\gamma = \frac{4}{3}\pi\alpha^2 b c_0 s , \quad (4a)$$

$$\bar{c} = \frac{a}{b} - \sum_j g(\alpha_j, 0) \exp(-D\alpha_j^2 t) , \quad (4b)$$

$$g(\alpha, 0) = \frac{2a}{\alpha a(2q + l) \sin \alpha l + (\alpha^2 q a l - b) \cos \alpha l} , \quad (4c)$$

$$l = b - a , \quad (4d)$$

$$q = sa/3n , \quad (4e)$$

the α_j are the roots of

$$\tan \alpha l = \frac{\alpha a}{\alpha^2 q a - 1} , \quad (4f)$$

c_0 is the constant outside concentration of gas, and D is the diffusivity.

A computer program has been written to calculate the uptake as a function of time from Eqs. (3) and (4) and to vary the parameters D , γ , and q to obtain the best theoretical fit to real uptake data. The following discussion describes the steps taken and some of the results obtained from the diffusion of helium to batch OR-2261-HT fuel particles. The first step was to let the computer program vary all three parameters D , γ , and q to obtain the best fit of uptake as a function of time for data taken at each of several temperatures. Then it was assumed that the diffusivities obeyed an Arrhenius relationship

$$D = D_0 e^{-Q/RT} , \quad (5)$$

where T is the absolute temperature. A least squares fit of $\ln D$ vs $1/T$ was made, with the results shown in Fig. 2.4. At this point, the D s were forced to lie on the line in Fig. 2.4 and the computer code was allowed to search on the remaining parameters γ and q . Since γ is proportional to c_0 , which varies inversely with the absolute temperature, we next forced γ to obey the relationship

$$\gamma = v/T , \quad (6)$$

Fig. 2.4. Plot of $\log D$ vs $1/T$ Demonstrating Fit of the Arrhenius Relationship for Diffusivities (D) of Helium in Fuel Particle Batch OR-2261-Ht. Crossed points were not used in the fit.

by a least squares analysis and performed a computer search on the one remaining parameter q . From Eq. (4e) we see that q is just a collection of constants, so the values of q from the computer program were averaged to obtain our final estimate of q . Plots of the data and the fitted curves using these final values of D , γ , and q appear in Figs 2.5 through 2.12.

With the exception of the low-temperature case, Fig. 2.5, 450°C, and the high-temperature case, Fig. 2.12, 1425°C, the theoretical fits are good. From Fig. 2.4 it can be seen that the diffusivities deviate from the Arrhenius relationship at both the high and low temperatures, thus contributing to the poor fits for these temperatures. Also, the high-temperature case had only a few data points, which made determination of the parameters D , γ , and q less precise. But for the rest of the temperature range, 550°C to 1200°C, where the diffusivities fall on a line in the $\ln D$ vs $1/T$ plot, the fits are of high quality, demonstrating the correctness of our model in this region.

Fig. 2.5. Uptake of Helium by OR-2261-HT Fuel Particles at 450°C.

Fig. 2.6. Uptake of Helium by OR-2261-HT Fuel Particles at 550°C.

Fig. 2.7 Uptake of Helium by OR-2261-HT Fuel Particles at 650°C.

Fig. 2.8 Uptake of Helium by OR-2261-HT Fuel Particles at 750°C.

Fig. 2.9 Uptake of Helium by OR-2261-HT Fuel Particles at 850°C.

Fig. 2.10 Uptake of Helium by OR-2261-HT Fuel Particles at 1000°C.

Fig. 2.11 Uptake of Helium by OR-2261-HT Fuel Particles at 1200°C.

Fig. 2.12 Uptake of Helium by OR-2261-HT Fuel Particles at 1425°C.

Equipment to facilitate measurement of the permeability of the pyrocarbon coatings of irradiated fuel particles has been ordered. The basic component of this facility, which will be installed in the hot cell area, is a time-of-flight mass spectrometer and necessary peripheral equipment. A particle feed system, a particle crusher, and a furnace for heat treatment are being designed.

2.3.2 Permeability Determination Using Chlorine Leach Techniques -

B. A. Thiele, D. A. Costanzo, and D. P. Stinton

For about ten years gaseous chlorine leaching of fuel particle batches has been the standard technique to determine the fraction of defective particles. The fractional heavy metal release is measured after a 2-hr chlorination at 1500°C. Last year a long-term chlorination of Biso-coated fuel particle batches was found to give very valuable information about the gas permeability of the pyrocarbon coatings. Batches with particles having gastight coatings show no increase of heavy metal release with chlorination time (see Fig. 2.13), while particles with gas permeable coatings show a steadily increased release of the heavy metal during an 18-hr chlorination (see Fig. 2.14). Gas-permeable particle batches start with a high value for the fraction of defective ($>5 \times 10^{-5}$) and increase the fractional release by about one order of magnitude within the next 16 hr.

Long-term chlorine leach identified particles of batch OR-2275-HT gas permeable before irradiation. Particles of this batch were irradiated in HT-30. In postirradiation examination internal gas pressure measurements identified this batch to be gas permeable, too, while a short-term neutron activation test (TRIGA) could not detect this type of defect (see Sect. 2.3.4). The same technique was successful in identifying permeability in the Biso-coated driver particle batches OR-2103-HT and OR-2104-HT, causing the high fission gas release measured^{18,19} in the irradiation tests HRB-7, -8, -9, and -10.

Long-term chlorination studies on batches of two different coating studies indicated that high orientation, measured by the optical anisotropy, and high density are typical of gastight pyrocarbon coatings. These pyrocarbons show low Ne/He permeability ratio and low open porosity (see Table 2.4).

Fig. 2.13. Batches with Particles Having Gastight Coatings Show No Increase of Heavy Metal Release with Chlorination Time.

Fig. 2.14. Batches with Particles Having Permeable Coatings Show Increased Release of Heavy Metal with Chlorination Time.

Table 2.4. High Orientation (Optical Anisotropy) and Density and Low Ne/He Permeability and Porosity are Typical of Gastight Pyrocarbon Coatings

Batch	Gradient Density, Mg/m ³		Porosity (%)	Optical Anisotropy	Ne/He Permeability Ratio	Coater Size (mm)	Batch Size (kg)	Fraction of Gastight Particles after 72 hr Chlorination (%)
	Measured	Corrected						
<u>Batches with Gastight Pyrocarbon Coatings</u>								
J-636	1.94	1.89	2.70	1.059	0.29	228.6	10	100
J-637	1.92	1.84	4.21	1.033	0.23	228.6	10	
J-638	1.95	1.86	4.26	1.049	0.26	228.6	10	
J-640	1.95	1.87	4.23	1.054	0.39	228.6	12.5	
J-641	1.95	1.88	3.58	1.049	0.30	228.6	10	
J-643	1.99	1.93	3.62	1.057	0.18	228.6	10	
J-649	1.94	1.86	3.89	1.042	0.36	228.6	10	
J-650	1.99	1.93	3.39	1.045	0.55	228.6	10	
J-651	1.90	1.80	5.47	1.031	0.42	228.6	10	
J-655	1.98	1.91	3.70	1.047	0.22	228.6	10	
A-770	2.00	1.99	1.00	1.107	0.15	127.0	2	
A-803	1.80	1.73	3.92	1.037	0.43	127.0	2	
A-804	1.99	1.93	2.82	1.055	0.27	127.0	2	
A-771	1.70	1.62	3.75	1.030	0.36	127.0	2	
<u>Batches with Permeable Pyrocarbon Coatings</u>								
J-635	1.93	1.81	6.21	1.033	0.62	228.6	10	12.8
J-642	1.93	1.82	6.03	1.048	0.69	228.6	15	
J-644	1.93	1.75	9.42	1.040	>0.70	228.6	12.5	
J-646	1.94	1.75	10.18	1.048	>0.70	228.6	15	
J-648	1.95	1.82	6.62	1.054	0.68	228.6	15	10.6
A-772	1.69	1.59	5.94	1.030	0.66	127.0	2	
A-775	1.73	1.56	9.90	1.030	0.70	127.0	2	
A-783	1.73	1.53	11.70	1.023	0.60	127.0	2	

2.3.3 Permeability Determination Using TRIGA Activation — E. L. Long

Five fuel rods from the high-temperature region of HT-30 were submitted for an activation test in the TRIGA Reactor at GA. The rods selected contained particles with Ne/He values that ranged from 0.13 to 0.41. Particles from one batch (OR-2275-HT) showed thorium loss during chlorine leaching of the as-deposited coatings. The release-to-birth (R/B) ratios for ^{85}mKr obtained during the test in TRIGA at 1100°C for 0.5 hr gave no indication of defective particles²⁰ (see Table 2.5, TRIGA R/B values ranged from 2 to 12×10^{-6}). After the TRIGA test the five fuel rods were returned to ORNL for chlorine leaching and fission-gas inventory measurements. The results from short-term chlorine leaching appeared to confirm the R/B data: no indications of failed or permeable coatings in these particular rods.

After chlorine leaching, the five rods were electrolytically deconsolidated for gamma spectroscopy and internal gas inventory measurements (see Sec. 2.3.4). The gas inventory measurements showed one batch of particles to have had gas-permeable coatings during irradiation. However, this gas permeability was not detected by the TRIGA R/B measurements. This result causes the validity of the TRIGA R/B measurement to be questioned relative to identifying particles with gas permeable coatings. Also, there is the question of why chlorine leaching did not indicate that the coatings were permeable in the irradiated fuel rod. Since only a portion of the fission gas was released during irradiation, which indicates a relatively low gas-permeability value, the residence time in TRIGA and the chlorination time may have been too short to indicate permeability.

2.3.4 Permeability Determination Using Internal Gas Inventory Measurements —

B. A. Thiele and T. B. Lindemer

In Bisoparticles with gas-permeable PyC coatings the noble fission gases krypton and xenon are continuously leaking out during irradiation. Thus, fission gas inventory measurements are a sensitive check on pyrocarbon coatings becoming permeable or being permeable during irradiation. This postirradiation examination technique was applied on particles for five rods of irradiation HT-30. The TRIGA activation test showed low ^{85}mKr R/B values, indicating that the particles were intact (see Section 2.3.3).

Table 2.5. Permeability of Fuel Particles Irradiated in HT-30

Particle Batch	Design Conditions ^a		TRIGA ⁸⁵ Kr R/B	Ne/He	HT-30 Rod and Particle	Burnup Determination by Fission Product Inventory, % FIMA					Kr + Xe Released
	Burnup (% FIMA)	Fluence >29 fJ (n/m ²)				⁹⁵ Zr	¹⁰⁶ Ru	¹³⁷ Cs	¹⁴⁴ Ce	Average ^b	
J-489	7.5	7.6×10^{25}	3.9×10^6	0.28	14-1	6.82	7.27	7.97	6.91	7.00	54.8
					-2	6.64	7.55	7.87	6.91	7.03	51.1
					-3	6.89	7.29	8.03	7.14	7.11	46.7
					-4	7.17	7.53	8.12	7.03	7.24	47.3
OR-2275	8.4	8.6	2.2	0.41	25-1	5.44	5.90	6.18	5.34	5.56	21.4
					-2	6.16	6.38	6.46	5.62	6.05	3.2
					-3	5.66	5.54	6.33	5.40	5.53	7.7
					-4	5.89	6.02	6.43	5.56	5.82	17.0
					-5	5.47	5.83	6.32	5.51	5.60	17.7
OR-2262	8.2	8.4	12.0	0.20	32-1	5.99	6.33	6.84	5.93	6.08	35.7
					-2	6.28	6.50	6.96	5.99	6.26	39.8
					-3	5.71	6.70	7.06	6.31	6.24	40.2
					-4	6.20	6.68	6.99	5.99	6.29	46.0
OR-2264	7.9	8.1	2.5	0.13	36-1	6.52	6.61	7.19	6.20	6.44	34.7
					-2	6.46	7.61	7.41	6.31	6.79	46.1
					-3	6.85	7.05	7.26	6.36	6.75	42.3
					-4	6.55	7.06	7.39	6.52	6.71	38.0
J-491	7.5	7.6	4.2	0.41	39-1	7.08	7.68	7.97	6.91	7.22	33.6
					-2	7.05	7.74	8.10	7.20	7.33	45.0
					-3	7.23	7.12	7.88	7.03	7.13	36.7
					-4	6.85	7.51	7.79	6.68	7.01	43.2

^aIrradiation temperature 1250°C.^bWithout the burnup value determined by Cs inventory measurement.

However, a high preirradiation Ne/He ratio of 0.41 led to the suspicion that at least one of the batches had as-fabricated gas-permeable coatings, and in this case the activation test could not detect this special type of defect. To check the gas permeability, five rods were deconsolidated and four to five particles from each rod were analyzed for their solid fission product inventories by individual gamma spectroscopy. The particles retained Ru, Zr, Ce, and Cs. Fission gas inventories were measured by cracking individual particles at 1000°C and measuring the volume of noble gas after absorbing the active gases present. By burnup calculations the theoretical noble gas inventory for individual particles could be calculated. For these high-density ThO₂ kernels, a release of 40 to 50% of the theoretical amount at burnups of 6 to 7% and irradiation temperatures of about 1250°C is normal.

As shown in Table 2.5, four out of five batches released the expected percentage of the theoretical amounts of xenon and krypton. But batch OR-2275 had an average release of only 13.4%. It is assumed that this batch had particles with gas-permeable coatings and released xenon and krypton during the irradiation. This batch showed high heavy metal release during long-term chlorination of unirradiated particles, which the other four batches did not, as discussed in Sec. 2.3.2.

2.3.5 X-Ray Diffraction Determination of BAF — C. J. Sparks, O. B. Cavin, and C. S. Morgan

A fuel particle holder that will permit determination of x-ray diffraction from the (0002) planes of a small volume of the pyrocarbon coating has been tested.²¹ Measurements with this device, the Spark's anisotropy device (SAD), demonstrated a large difference in anisotropy for two fuel particles batches whose BAF_O differed substantially. The diffraction characteristics of the SAD have been calibrated with graphite sheets and fine crystalline silicon. Shielding was modified to reduce the hard radiation components in the x-ray beam, which added to the background radiation beneath the signal of interest. Determination of the x-ray diffraction characteristics of the pyrocarbon coatings of fuel particle batches irradiated in HT-34 has been started.

2.4 IRRADIATION TESTING IN THE HFIR TARGET FACILITY — F. J. Homan

At the end of the last reporting period (December 1976) capsule HT-32 was still in the reactor, and irradiation of all other HT capsules (including HT-33) had been completed. HT-33 was irradiated ahead of HT-32, but all other HT capsules have been irradiated in numerical sequence. During the current reporting period capsule HT-34 was planned, constructed, and irradiated. The capsule came out of the reactor in November 1977, and postirradiation examination is scheduled for the early part of 1978.

In addition to the postirradiation examination of HT-32 and planning, fuel fabrication, capsule assembly, and irradiation of HT-34 substantial postirradiation examination was done on "old" HT capsules (pre HT-32). This work was done to resolve some unexplained results in these experiments and to assess the performance of Weak-Acid Resin (WAR) derived fissile particles containing low-enriched uranium (LEU) kernels. The performance of LEU fuel is of particular interest at present because there is serious concern over the licensability of the reference high-enriched uranium (HEU) fuel cycle for the HTGR, due to the high proliferation potential for the HEU cycle. The HT capsules have always contained Triso-coated LEU driver particles to generate heat during the early portion of the experiment. Since they did not contribute to the intended experimental objectives, they were always discarded without examination. Microprobe and metallographic examination of these particles from capsules HT-31 and -33 were conducted during the reporting period. These examinations were expected to show corrosion of the SiC layer due to the presence of the fission product palladium, which has a much higher yield from plutonium fissions than from ^{235}U fissions.

The HT-12 through -15 capsules were intended to provide data for pressure-vessel models of Biso-coated fertile particles. The results were confusing in that some particles designed to survive the irradiation failed, and others designed to fail survived. The PyC coatings of some of the particle batches were found to be permeable to fission gas, thereby invalidating the internal pressure calculation. Special postirradiation procedures have been developed (see Sect. 2.3) to quantify PyC permeability. These procedures were developed during this reporting period, and additional examination on HT-12 through -15 is in progress.

Confusing results were also obtained with the HT-17 through -19 series. The coating conditions used in two of the HT-17 through -19 batches were duplicated for both fertile particles and inert particles, both with Biso designs. These "duplicated" batches were irradiated in the HT-34 capsule (unirradiated particles from the original HT-17 through -19 batches were not available) so that the characterization techniques described in Sect. 2.3 could be applied. The results of these examinations will be included in the topical report on this capsule series.

As of the end of this reporting period no HT capsules are in the reactor, HT-34 is in the hot cells, and the next HT capsule (HT-35) is not scheduled for irradiation until the second quarter of FY 1979.

2.4.1 Capsules HT-12 Through -15 - M. J. Kania

The irradiation experiment that included capsules HT-12 through -15 was conducted in the target facility of the HFIR during the period from October 23, 1972, through May 11, 1973. An initial report describing the experiment and the fission product behavior of the fertile test particles has been published.²² Not addressed in that report were several questions regarding the integrity of the particle coatings and a correlation of the STRETCH²³ model analysis with the experimental data. The reason for not addressing these questions in the initial report was the lack of certain postirradiation data. Collection of these data required acquisition of equipment and development of procedures for new techniques associated with characterizing pyrocarbon permeability, gamma spectroscopy, and internal gas pressure measurements.

The 1000°C gaseous chlorine leach examination of particles from capsules HT-13 and -14 has been completed. The data are presented in Table 2.6. Before chlorination, all samples were optically examined at a magnification of 20× to determine the number of particles per sample and to note the general appearance of the particles before leaching. In two samples, HT-14-11 and HT-14-45, a single particle was defective with a cracked coating; otherwise all the particles appeared to be undamaged and intact. All samples were initially chlorinated for 20 hr at 1000°C, and the amount of thorium evolved from the damaged particles was measured. The samples were then chlorinated

Table 2.6. Chlorine Leach Data for HT-13 and -14 Particle Samples.

Kernel Diameter (μm)	Capsule Code	Particle Type	Number of Particles	Cumulative Chlorination Time (hr)	Cumulative Thorium Recovered (μg) ^a
508	HT-13-4	OR-1850	20	20	14 ^b
402	HT-13-5	OR-1846	23	20	5860
				40	5920
402	HT-13-11	OR-1749	20	20	1360
				40	1620
402	HT-13-42	OR-1838	20	20	<5
				40	<5
402	HT-13-45	OR-1826	21	20	239 ^b
				40	239 ^b
402	HT-13-48	OR-1830	22	20	220
				40	440
402	HT-13-51	OR-1837	20	20	1050 ^b
				40	1050 ^b
508	HT-14-4	OR-1850	20	20	8030
				40	8970
402	HT-14-5	OR-1846	19	20	4520 ^b
				40	4520 ^b
402	HT-14-11	OR-1749	20	20	4450 ^b
				40	4450 ^b
402	HT-14-42	OR-1838	20	20	4150
				40	4160
402	HT-14-45	OR-1826	18	20	4150
				26	4160
402	HT-14-48	OR-1830	20	20	4870
				40	4880
402	HT-14-51	OR-1837	20	20	4670 ^b
				40	4670 ^b

^a ≈ 300 μg of Th per particle with 402- μm kernel.

≈ 600 μg of Th per particle with 508- μm kernel.

^b Less than 5 μg Th recovered during second chlorination interval.

for a second interval, which varied from 6 to 20 hr, and the additional thorium evolved from the damaged particles was measured.

In all cases except sample HT-13-48, particle batch OR-1830, greater than 80% of the total thorium was recovered on the first chlorination period; for HT-13-48, only 50% of the total thorium was recovered in this period. In general, particles with permeable coatings do not release all their thorium at one time, but rather release it continuously over the period of chlorination. Therefore, according to the results presented in Table 2.6 all particle batches except OR-1830 in HT-13-48 seem to have gross coating failures rather than permeable coatings. This is evidenced by the large amount of thorium measured during the first chlorination period relative to the total thorium recovered.

The permeabilities based on Ne/He ratios for irradiated archive samples of particle batches from the capsule series are reported in Sec. 2.3.1 of this report. A visual examination of all irradiated particles from this series of capsules has been completed. Individual particles have been isolated for further detailed examinations to be completed during the early part of 1978. Examinations to be completed include: metallography of intact coatings and coating fragments, gamma spectroscopy on intact particles, and fission gas pressure measurements on intact particles. In situations where only a few intact particles exist, those procedures that reveal the most insight to failure mechanisms will be used. A final report dealing with the questions of coating integrity and correlation with model analysis will be prepared.

2.4.2 Capsules HT-17 Through -19 — E. L. Long, Jr.

The results of the postirradiation examination of capsules HT-17 through -19 have been reported previously.^{6,24} We are further characterizing the HT-17 through -19 particles using recently developed procedures (chlorine leach and Ne/He ratio measurements), and a final topical report is being written. This work should be completed during the first half of 1978.

2.4.3 Capsules HT-20 Through -23 — E. L. Long, Jr.

Thermal transport and expansion properties were determined on unirradiated and irradiated simulated HTGR fuel rods. The simulated rods were

irradiated in capsules HT-20 through -23;²⁵ four additional rods were irradiated in HT-31²⁶ and -32²⁷ to replace those inadvertently broken during the disassembly of the earlier capsules. The abstract of the report²⁸ on these experiments follows:

The thermal conductivity, electrical resistivity, coefficient of thermal expansion, volume, and Seebeck coefficient of simulated fuel rods for a high-temperature gas-cooled reactor (HTGR) have been measured before and after neutron irradiations to $13.5 \times 10^{25} \text{ n/m}^2$ at nominal irradiation temperatures of 1220 K. These measurements were made as functions of volume particle loading, temperature, and neutron fluence. The thermal conductivities decrease with increasing particle loading at all measurement temperatures. The large difference between the properties of specimens from two fabrication processes (extrusion and slug injection) is attributed to the fact that the densities of the continuous phases within the elements differ by a factor of 2.6. Increases in the thermal conductivity with initial neutron fluence of some of the extruded specimens are attributed to partial ordering of carbonaceous material within the composites, but an unambiguous quantitative analysis is difficult. Some electrical resistivity results obtained on slug-injected unirradiated fuel rods show that the thermal conductivity would probably be affected by graphite shim content. Previous investigators have estimated that the thermal conductivity of slug-injected fuel rods would never go below 6.9 W/m K, but measurements on a slug-injected specimen with 36.5% simulated fuel and 20.2% shim yielded a value of 5 W/m K after $8.6 \times 10^{25} \text{ n/m}^2$.

2.4.4 Capsules HT-28 Through -30 — E. L. Long, Jr.

The objectives, operating history, and results from the postirradiation examination of capsules HT-28, -29, and -30 have been discussed in previous progress reports.²⁹ A final topical report³⁰ on this experiment was completed during this reporting period. The abstract of this report follows:

Three capsules, HT-28, -29, and -30, were irradiated in the target region of the High-Flux Isotope Reactor at Oak Ridge National Laboratory to determine the relative fast-neutron stability of pyrolytic carbons that had been prepared using various deposition conditions. The pyrolytic carbon coatings of 22 batches of particles were characterized by various methods including optical anisotropy measurements, hot gaseous chlorine leaching, plasma oxidation, small-angle x-ray scattering (SAXS) measurements, mercury intrusion, immersion density, and, in a few cases, Ne/He permeability measurements. The results of the

above measurements were used to quantify microstructural differences between pyrolytic coatings derived at various conditions and to correlate the performance of the coatings with the measured properties. The most consistent results were obtained by comparing various pore-size distributions within the coatings (determined from SAXS measurements) with immersion density, mercury intrusion, chlorine leach, Ne/He permeability results, and irradiation performance of the coatings. This study also demonstrated that care must be exercised if experiments on coatings containing inert carbon kernels that were co-deposited with dense ThO_2 kernels are to yield meaningful results.

2.4.5 Capsule HT-31 - T. N. Tiegs

The design, fabrication, operation, and most of the postirradiation examination have been reported previously.²⁶ Additional work has been done in examining the low-enriched (LEU) driver particles. Because of the recent interest in LEU fuel, prompted by government policy changes, examination of these particles became important. However, these particles were irradiated without an outer LTI layer, which affected the performance of the particles.

During unloading, many of the particles were stuck in the crucibles and could not be removed. Examination with the scanning electron microscope (Fig. 2.15) showed that the particles were bonded to the graphite, and a well-defined crystal structure was observed on the graphite surface. We believe that silicon actually evaporated from the particle surfaces and condensed on the graphite with the re-formation of SiC. Thermal dissociation of SiC has been observed previously.³¹ In the low-temperature regions in an HT-capsule, where the average particle surface temperature is approximately 1200°C, a "thinning" of the SiC of about 2 μm was calculated. However, in the high-temperature regions, the average particle surface temperatures are as high as 1600°C, which would result in complete thinning of the SiC coating. This is indeed the case. Figure 2.16 shows a particle from the high-temperature region whose SiC coating is gone with the exception of one spot where this particle apparently was in contact with another.

The HT-31 driver particles were irradiated without outer LTI layers because of interest in this design relative to increasing heavy metal loadings in HTGR fuel rods. It was thought that the strength of the SiC

Fig. 2.15. Particle Adhering to Crucible 41. (a) 13, (b) 70, and (c) 340 $\times$. At 340 $\times$ crystal growth from the graphite surface is easily observed.

Fig. 2.16. Single Particle from Crucible 28. No SiC remains except at one point where probably this particle rested against another. (a) 200 $\times$. (b) 365 $\times$.

layer precluded the need for the outer LTI layer to maintain pressure vessel integrity. However, the performance of the HT-31 driver particles was poor, and the need for the outer LTI layer reaffirmed.

Because of the nonrepresentative performance of the LEU driver particles (due to absence of an outer LTI layer, as described above) we discontinued examination. Other HT capsules contained Triso-coated LEU driver fuel (Sect. 2.4.7) of a more representative particle design. Examinations of these driver particles were scheduled to add to the LEU fuel performance data base.

2.4.6 Capsule HT-32 — T. N. Tiegs, B. A. Thiele, K. R. Thoms, and M. J. Kania

The capsule design and fabrication have been reported previously.²⁷ In this reporting period the irradiation was completed and postirradiation examination was in progress.

2.4.6.1 Capsule Operation

HFIR target capsule HT-32 was removed from the HFIR on March 1, 1977, after having operated for 2308 hr at 100 MW. Table 2.7 gives the operating history of the reactor while this capsule was being irradiated. It should be noted that the first cycle of operation, cycle 140, was a slow startup cycle, where the reactor power started at 68 MW and was gradually increased to 100 MW over a 24-day period and then operated at 100 MW for 5 days. During the second cycle of operation, cycle 141, the reactor operated at 90 MW for 10 days and 95 MW for 15 days.

Table 2.7. Reactor Operating History During Operation of HT-32

Cycle	Begin		End		Irradiation Time, ^a hr	
	Time	Date	Time	Date	During Cycle	Accumulated
140	1900	11/15/76	0400	12/14/76	577	577
141	1711	12/15/76	0115	1/10/77	568	1145
142	2030	1/10/77	2000	2/3/77	576	1721
143	1830	2/5/77	2000	3/1/77	587	2308

^aIrradiation time is given in equivalent hours at 100 MW. Cycle 140 was a slow startup cycle, and during cycle 141 the reactor was operated at 90 or 95 MW.

The thermal and fast fluence for all specimens and the calculated burnups for the specimens containing fueled particles are shown in Table 2.8.

Table 2.8. Estimated Neutron Fluence and Burnup Data for Specimens in Capsule HT-32

Specimen	Distance from HMP to Specimen Center Line (mm)	Estimated Fluence, n/m ² (< 0.414 eV) (> 0.18 MeV)		Estimated Burnup, % FIMA		
		$\times 10^{26}$	$\times 10^{25}$	²³⁵ U	²³⁸ U	²³² Th
Crucible 1	246.4	1.11	4.00			
ORNL-1	196.9	1.46	6.09			
ORNL-2	139.7	1.79	7.89			
U-16	92.1	2.04	8.93			
U-17	84.9	2.08	9.10			
LASL-1	70.6	2.14	9.31	84.7	18.4	9.04
LASL-2	49.0	2.23	9.56	84.7	18.9	9.43
LASL-3	27.5	2.30	9.68	84.7	19.3	9.72
27	-20.3	2.33	9.72			9.79
28	-27.5	2.30	9.68	84.7	19.3	
29	-34.7	2.28	9.64			9.63
30	-41.9	2.26	9.60			9.54
31	-49.0	2.23	9.56	84.7	18.9	
32	-56.2	2.20	9.47			9.31
33	-63.4	2.18	9.39			9.19
34	-70.6	2.14	9.31	84.7	18.4	
35	-77.7	2.12	9.21			8.89
36	-84.9	2.08	9.10			8.71
37	-92.1	2.04	8.93	84.7	17.6	
38	-99.3	2.00	8.81			8.34
39	-106.7	1.96	8.64	84.7	17.0	
40	-147.3	1.74	7.73			6.87
41	-154.5	1.71	7.55	84.7	14.1	
42	-161.7	1.66	7.31			6.36
43	-168.9	1.61	7.06			6.10
44	-176.0	1.58	6.81	84.6	12.7	
45	-183.2	1.55	6.56			5.59
46	-190.4	1.50	6.31			5.31
47	-197.6	1.45	6.07	84.6	11.4	
48	-204.7	1.40	5.77			4.89
49	-211.9	1.35	5.53			4.68
50	-219.1	1.30	5.32	84.6	9.92	
51	-226.3	1.25	4.94			4.29
52	-233.7	1.20	4.65	84.6	8.92	

2.4.6.2 Postirradiation Examination

Disassembly of the capsule proceeded without incident. The bonded rods, ORNL-1 and -2 (containing inert particles), were transferred to the Physical Properties Group for measurement of thermal expansivities and conductivities. The loose inert particles were removed from crucible 1. Some of the particles had adhered to the rhenium foil disks that were included in the crucible for heat generation. We could see that some particles had reacted with the rhenium. The particles that had not reacted with the rhenium were transferred for use in coating studies.

The loose particles from positions U-16 and U-17 (Triso- and Biso-coated ThO_2 particles, respectively, from GA's 0.24-m-diam coating furnace) showed fair irradiation performance and faceted coatings (Fig. 2.17). Additional examination using the IMGA system will be done in the future.

The heater rods containing low-enriched fuel particles appeared to be in good condition with very little or no debonding. Failed particles were observed on the rod surface in position 27 only.

The loose Triso-coated heater particles with outer low-temperature isotropic (oLTI) coatings showed good irradiation performance with no visible failures. However, similar particles with the oLTI burned off and irradiated in the high-temperature region were bonded together in the crucibles and most would not pour out. Particles of the same type irradiated in the low-temperature region had much better irradiation performance, with only a few observed failures in crucible 44. These results are analagous to those from HT-31 (see Sec. 2.4.5), which showed that exposed SiC will dissociate, evaporate from the particle surface, and condense on the inner surface of the graphite crucible.

The Biso-coated ThO_2 particles with Al_2O_3 - SiO_2 -doped kernels showed variable irradiation performance with the different coatings. The failure fractions as determined by visual inspection for the different particle types are shown in Table 2.9. Metallography was performed on these loose particles, and observations are summarized in Table 2.9.

Fig. 2.17. First Tests with ThO_2 Particles Coated in 0.24-m-diam Coater Using a Frit Gas Distributor Show Faceted Particles with Fair Irradiation Stability. (a) Triso-coated (b) Biso-coated.

Table 2.9. Visual Failure Fractions of Loose Biso-Coated German ThO_2 Particles Doped with Al_2O_3 and SiO_2 .

Position	Coating	Heat Treatment ($^{\circ}\text{C}$)	Number of Particles	
			Examined	Broken
35	German	1800	56	5
36	German	1500	56	15
38	ORNL	1800	57	0
48	German	1800	40	1
49	German	1500	40	24
51	ORNL	1800	40	0

2.4.6.2.1 Position 35 — German Kernel, German Coating, 1800 $^{\circ}\text{C}$ Heat Treatment (Fig. 2.18). After irradiation, these particles had dense and anisotropic buffer coatings. These properties caused radial cracks through the buffer, with kernel material extruding into the cracks. Preirradiation metallography is being checked to see if any anisotropy was present in the buffer coatings of this particle batch.

The kernels developed central holes much like those observed in LMFBR pellet fuel. Columnar grains extending from the grains are produced by a vaporization-condensation process, which with UO_2 occurs at temperatures above 1750 $^{\circ}\text{C}$. The contribution to this process by the Al_2O_3 - SiO_2 additions is still unknown. Additions of SiO_2 to ThO_2 lower the temperature at which liquid is present to about 1700 $^{\circ}\text{C}$ with greater than 18 mol % SiO_2 .³² This is the first time that a central hole and columnar grains have been observed in coated particle fuel.

2.4.6.2.2 Position 36 — German Kernel, German Coating, 1500 $^{\circ}\text{C}$ Heat Treatment (Fig. 2.19). Metallographic observations indicated that these particles operated very similarly to those in position 35 (same particles but heat-treated at 1800 $^{\circ}\text{C}$). Radial cracks through the coatings due to dense and anisotropic buffers were observed. In addition, the central hole and columnar grain structure observed previously was also present.

Fig. 2.18. Typical Appearance of Loose Particles from Position 35: German Kernel, German Coating, 1800°C Heat Treatment. Irradiated at an average particle surface temperature of 1500°C. 100×

Fig. 2.19. Typical Appearance of Loose Particles from Position 36: German Kernel, German Coating, 1500°C Heat Treatment. Irradiated at an average particle surface temperature of 1500°C. 100×

2.4.6.2.3 Position 38 — German Kernel, ORNL Coating, 1800°C Heat Treatment (Fig. 2.20). The buffer coatings were porous and isotropic in this particle batch. No radial cracks or broken particles were observed.

Kernel behavior was very similar to the kernels irradiated in position 35 with the central hole and columnar grains. However, in this particle batch the buffer coating had separated from the LTI in most particles, causing a thermal gradient across the particles. Subsequently, kernel material vaporized and diffused through the separated buffer coating. The kernel material then condensed on the inner surface of the LTI forming columnar grains.

Fig. 2.20. Typical Appearance of Loose Particles from Position 38: German Kernel, ORNL Coating, 1800°C Heat Treatment. Irradiated at an average particle surface temperature of 1250°C. 100×

2.4.6.2.4 Position 48 — German Kernal, German Coating, 1800°C Heat Treatment (Fig. 2.21). Like particles irradiated in position 35, these

particles had very dense and anisotropic buffer coatings, which resulted in radial cracks through the buffer coating.

Fig. 2.21. Typical Appearance of Loose Particles from Position 48: German Kernel, German Coating, 1800°C Heat Treatment. Irradiated at an average particle surface temperature of 1250°C.

Because of the moderately low irradiation temperatures, the kernels appeared very similar to the unirradiated kernels — ThO_2 grains individually dispersed in an $\text{Al}_2\text{O}_3\text{-SiO}_2$ matrix.⁴ Cracks were observed in the periphery of the kernels and sometimes extended into the buffer coating. This was caused by the strong bond between the $\text{Al}_2\text{O}_3\text{-SiO}_2$ matrix and the buffer coating (some $\text{Al}_2\text{O}_3\text{-SiO}_2$ diffused into the buffer). No cracks were observed in the central part of the kernel because of its higher operating temperature and hence greater plasticity. This type of cracking has been observed previously (irradiation test FRJ-2-P17) without failure of the LTI.

2.4.6.2.5 Position 49 — German Kernel, German Coating, 1500°C Heat Treatment (Fig. 2.22). Like the particles irradiated in positions 35 and 48, this particle batch also had dense buffer coatings but was less anisotropic. Buffer coating failures were also similar to those observed in positions 35 and 48.

Unlike the kernels in position 48, these kernels showed no cracks. The Al_2O_3 - SiO_2 matrix and the ThO_2 grains were mixed together such that neither was distinguishable. In addition, kernel material had extruded into the buffer cracks, indicating high kernel plasticity during irradiation. Very bright inclusions appeared in particles where cracks extended through the buffer and LTI. These inclusions are thought to be Al_2O_3 formed during the migration of the SiO_2 out of the kernels with cracked coatings. Microprobe examinations will determine the exact composition of these inclusions. These observations suggest that this crucible operated at a higher temperature than design. The thermal analysis for the capsule does not substantiate this conclusion. Operating temperatures for crucible 48, 49, and 51 had peak values ranging from 1110 to 1130°C; peak particle surface operating temperatures for the crucibles ranged from 1290 to 1300°C. These differences in temperatures are insignificant.

The primary difference between the particles is that those irradiated in 48 and 51 were annealed at 1800°C whereas those irradiated in 51 were annealed at 1500°C. Examination of the radiographic plates made on the particles after annealing but before irradiation show that the 1800°C anneal forced kernel material to diffuse into the buffer in both the German and ORNL coatings. In some cases the kernel material had diffused all the way through the buffer coat to the buffer-LTI interface. Examination of the 1500°C radiographic plates shows no fuel dispersion.

This fuel dispersion decreases the center temperature of the kernel. That is, particles irradiated in crucibles 48 and 51 would have lower center temperatures than those irradiated in crucible 49. To illustrate this, a one-dimensional radial heat transfer calculation, in spherical coordinates, was made for a typical particle with and without dispersion. The power generated per particle was considered to be 0.42 W, the surface temperature was 1280°C, the thermal conductivity of the buffer was 0.1 W/m K, and nominal dimensions of the particle were considered (532-86-81). Three cases were considered: (1) the kernel material had diffused into one-half

Fig. 2.22. Typical Appearance of Loose Particles from Position 49: German Kernel, German Coating, 1500°C Heat Treatment; Irradiated at an Average Particle Surface Temperature of 1250°C.

of the buffer; (2) the kernel material had diffused into one-third of the buffer; and (3) the kernel material had not diffused. The results are shown for each case in Table 2.10.

Table 2.10 Effect of Diffusion of Kernel Material into Buffer on Kernel

Case	Penetration of Buffer	Kernel Temperature, °C	
		Center	Surface
1	1/2	1440	1415
2	1/3	1530	1503
3	none	1620	1590

This analysis considered no gas gaps between coatings and assumed that the surface temperature was uniform.

This simple analysis illustrates the temperature effect due to kernel material dispersion into the buffer coating. Particles annealed at 1500°C may have operated at temperatures up to several hundred degrees above those that were annealed at 1800°C. The differences observed in postirradiation examination are due to peak kernel center temperatures being several hundred degrees higher in particles irradiated in crucible 49 than in crucibles 48 and 51.

2.4.6.2.6 Position 51 — German Kernel, ORNL Coating, 1800°C Heat Treatment (Fig. 2.23). The buffer coatings were porous and isotropic, with no particle failures observed. The kernel appearance was very similar to that described for position 48.

Fig. 2.23. Typical Appearance of Loose Particles from Position 51: German Kernel, ORNL Coating, 1800°C Heat Treatment; Irradiated at an average particle surface temperature of 1250°C.

A review of the preirradiation metallography of the German-coated doped ThO_2 particles showed no evidence of dense anisotropic buffers. We believe that the change in buffer properties was caused by the Al_2O_3 - SiO_2 doping in the kernels.

2.4.7 Postirradiation Examination of HT-33 — T. N. Tiegs

The fabrication, operation, and most of the postirradiation examination results have been previously reported.³³ Since that time, low-enriched fuel particles have become of special interest. The driver particles were Triso-coated WAR $\text{UC}_{4.40}\text{O}_{1.43}$ with an enrichment of 7.073%.

When these particles were examined metallographically, significant corrosion of the SiC was observed (Fig. 2.24). Examination by the electron microprobe showed that corrosion was attributable to fission product palladium (Fig. 2.25).

A major concern with using low-enriched fuel particles is plutonium volatility. The low-enriched WAR particles examined showed no additional uranium or plutonium redistribution than what is ordinarily observed in high-enriched WAR particles. Normally, uranium is observed in the kernels and densified buffers of WAR particles.³⁴ However, when chlorine was detected in the particle, migration of the uranium and plutonium to the SiC coating was observed (Fig. 2.26). Because chlorine was present in such a small concentration, no discernable x-ray display could be made. Such adverse effects of chlorine have been observed previously.³⁵

2.4.8 Capsule HT-34 — M. J. Kania

The HT-34 irradiation experiment was a cooperative effort between GA and ORNL, with approximately one-half of the irradiation space allocated to each. The GA Triso-coated ThO_2 test specimens were irradiated in the upper portion of the capsule in loose-particle holders. The ORNL Triso- and Biso-coated ThO_2 test specimens were irradiated in the lower portion of the capsule in bonded bed specimens and loose-particle holders, respectively.

The detailed objectives for the ORNL test specimens were:

1. To investigate the influence of differences in pyrocarbon microstructures, resulting from variations in fabrication process variables,

50 μm

Fig. 2.24. Severe Corrosion of the SiC in HTGR Fuel Particles Made with Low-Enriched Uranium and Irradiated in HT-33-19. (a) Bright field. (b) Polarized light.

R-74848

Fig. 2.25 Electron Microprobe Identified Palladium as the Cause of SiC Corrosion in Low-Enriched Particles. (a) Backscattered electron image (b) Si $L\alpha$ x-ray display (c) Pd $L\alpha$ x-ray display.

Fig. 2.26 Electron Microprobe Showed Uranium and Plutonium Migration to the SiC in Low-Enriched Particles where Chlorine was Present. (a) Backscattered Electron Image (b) U $M\alpha$ x-ray display (c) Pd $L\alpha$ x-ray display (d) Pa $M\beta$ x-ray display

on a coating's ability to retain fission gases and remain resistant to fast neutron damage. Specific process variables that are thought to influence Biso particle irradiation performance are hydrocarbon coating gas, deposition temperature, diluent type, and diluent concentration. Process variables were systematically varied as outlined in Fig. 2.27. Coating properties of interest include pyrocarbon coating rate, buffer and pyrocarbon densities, anisotropy of pyrocarbon coating, and permeability of the pyrocarbon coating. Optimization of the process variables listed above will provide guidelines for operation of the 0.24-m coating furnace.

2. To evaluate the performance of the SiC layer of Triso-coated ThO_2 particles deposited at higher coating rates (about $0.5 \mu\text{m}/\text{min}$) than previously employed (about $0.2 \mu\text{m}/\text{min}$). The fuel product specification for the Triso-coated particle requires that 99% of the SiC coatings have a mean density exceeding $3.18 \text{ Mg}/\text{m}^3$. To meet this requirement, the SiC

ORNL - DWG 76-14084

EXPERIMENTAL PLAN

VARIABLE

Fig. 2.27 Process Variables and Respective Ranges that will be Used in Experiment HT-34 to Study Their Influence on the Overall Performance of Biso-coated ThO_2 Particles.

layer has been deposited at very low deposition rates — 0.1 to 0.2 $\mu\text{m}/\text{min}$ — to ensure integrity of the coating. From an economic standpoint, SiC coatings deposited at higher coating rates without loss of coating integrity would be more desirable. Specific performance characteristics to be investigated are the fission product retention, the coating mechanical integrity, and the influence that the SiC microstructure has on these performance aspects.

3. To correlate permeability measurements made with light inert gases and those made with fission gas release-to-birth (R/B) ratios. Present design criteria restrict fission gas R/B ratios for the isotope ^{85}mKr to less than 3×10^{-5} at 1100°C for irradiated particles. At present no quality assurance procedure is available for measuring and predicting fission gas release on unirradiated particles. However, a technique is being developed at ORNL to correlate measurements of pyrocarbon permeability to light inert gases on unirradiated particles with pyrocarbon permeability to fission gases under irradiation. Particle batches from objective 1 will also be used to meet this objective. Preirradiation permeability measurements will be made with the light inert gas technique and by GA using a photofissioning technique. Postirradiation R/B ratios will be determined at GA by the TRIGA activation technique.

4. To resolve irradiation performance anomalies between two similar particle designs, batch OR-1975-T and OR-2013-ST, which were irradiated in HFIR Target capsules HT-18 and -19. The only difference in the fabrication of these two Biso ThO_2 particles was that batch OR-2013-ST had an LTI coating deposited from a carrier gas of CO_2 instead of argon. Batch OR-1975-T, which was of the reference fertile design, was expected to survive all temperatures and fluences in the series. Consequently it was irradiated in HT-19 only, in the high-temperature, high-fluence region. Instead of complete survival, the particles showed over 90% failure. Batch OR-2013-ST, with the fabrication difference noted above, was irradiated in capsule positions in HT-18 and -19 that received the highest fluence and experienced the peak operating temperatures. Interestingly, this particle survived completely in both capsules. To resolve this anomaly both batches will be irradiated in a specially designed holder such that both particle types will receive the same neutron fluence and experience the same operating temperatures. In addition, two

batches of carbon kernels with Biso-coatings that duplicate the coating designs of the two ThO_2 particles will be irradiated in the magazine end caps.

2.4.8.1 Capsule Design

As indicated earlier, capsule HT-34 was shared between GA and ORNL. The upper portion of the capsule contained the GA test specimens and was designed similarly to the HT-33 capsule. Heavy metal loadings were identical to HT-33, and driver particles (uranium with 7%-enriched ^{235}U) were contained in warm-molded bonded bed specimens. Test particles were Triso-coated ThO_2 in loose-particle holders. Heavy metal loadings were such that operating temperatures (maximum particle surface temperature) would be about 1250°C in the low-temperature region and about 1550°C in the high-temperature region. Specimen placement and heavy metal loadings for capsule HT-34 are described in Table 2.11 and 2.12, respectively.

The lower portion of the capsule contained ORNL test specimens. This portion was redesigned to provide irradiation temperatures of 1250°C in the high-temperature and 900°C in the low-temperature region. The important design changes that this involved were new magazine dimensions and a reduced heavy metal loading for each irradiation position (specimen placement and heavy metal loadings are described in Table 2.11 and 2.12, respectively). Fuel specimens were both Biso- and Triso-coated ThO_2 particles. The Biso particles were irradiated in loose-particle holders and the Triso particles were irradiated along with the driver particles in warm-molded bonded bed specimens.

2.4.8.2 Preparation of Test Specimens

Eight batches of Biso-coated ThO_2 particles were prepared in the 0.13-m coating furnace using the frit gas distribution system to meet objectives 1 and 3. A systematic variation of the fabrication process variables, as outlined in Fig. 2.27, was employed. The specific ranges were chosen so that limits of successful performance could be obtained for each process variable. Maximum characterization of all coating properties thought to be of influence on overall performance were obtained. The eight batches were selected because they had acceptable LTI thickness and densities, the highest quality particles

Table 2.11 Loading Scheme for Irradiation Capsule HT-34

Position ^a	Temperature (°C)	Specimen Type	Fuel (Heavy Metal)	Batch ^b	Coating Type
End Plug 14-3	1250	Loose	Th	7032-149	TRISO
1	1250	Bonded	U	OR-2576-H	TRISO
2	1250	Loose	Th	-20-0161-001	TRISO
3	1250	Bonded	U	OR-2576-H	TRISO
4	1250	Loose	Th	-07-0262	TRISO
5	1250	Loose	Th	-14-0261-001	TRISO
6	1250	Bonded	U	OR-2576-H	TRISO
7	1250	Loose	Th	-14-0171-001	TRISO
8	1250	Loose	Th	-13-0161-002	TRISO
9	1250	Bonded	U	OR-2576-H	TRISO
10	1250	Loose	Th	-14-0161-001	TRISO
11	1250	Loose	Th	-15-0161-001	TRISO
12	1250	Bonded	U	OR-2576-H	TRISO
13	1250	Loose	Th	-16-0161-001	TRISO
End Plug 14-1	1250	Loose	Th, inerts	7032-149, 6351-04-0100	BISO/TRISO ^c
End Plug 15-3	1550	Loose	Th	7032-149	TRISO
14	1550	Bonded	U	OR-2576-H	TRISO
15	1550	Loose	Th	-20-0161-002	TRISO
16	1550	Bonded	U	OR-2576-H	TRISO
17	1550	Loose	Th	-07-0262-002	TRISO
18	1550	Loose	Th	-14-0161-002	TRISO
19	1550	Bonded	U	OR-2576-H	TRISO
20	1550	Loose	Th	-14-0271-001	TRISO
21	1550	Loose	Th	-14-0181-001	TRISO
22	1550	Bonded	U	OR-2576-H	TRISO
23	1550	Loose	Th	-17-0161-001	TRISO
24	1550	Loose	Th	-15-0171-001	TRISO
25	1550	Bonded	U	OR-2576-H	TRISO
26	1550	Loose	Th	-13-0171-001	TRISO
End Plug 15-1	1550	Loose	Th, inerts	7032-149, 6351-04-0100	BISO/TRISO ^c

RHMP

Table 2.11 (continued)

Position ^a	Temperature (°C)	Specimen Type	Fuel (Heavy Metal)	Batch ^b	Coating Type
End Plug 26-1	1250	Loose	inert	OR-2730-1	BISO
27	1250	Loose	Th	A-765'	BISO
28	1250	Bonded	Th, U	A-834, OR-2576-H	TRISO/TRISO
29	1250	Loose	Th	A-780	BISO
30	1250	Loose	Th	A-785	BISO
31	1250	Bonded	Th, U	A-835, OR-2576-H	TRISO/TRISO
32	1250	Loose	Th	A-806	BISO
33	1250	Loose	Th	A-762	BISO
34	1250	Bonded	Th, U	A-837, OR-2576-H	TRISO/TRISO
35	1250	Loose	Th	A-782	BISO
36	1250	Loose	Th	A-786	BISO
37	1250	Bonded	Th, U	A-836, OR-2576-H	TRISO/TRISO
38	1250	Loose	Th	A-787	BISO
39	1250	Loose	Th	OR-1975-T, OR-2013-T	BISO
End Plug 26-3	1250	Loose	inert	OR-2722-1, OR-2725-1	BISO
End Plug 28-1	900	Loose	inert	OR-2730-1	BISO
40	900	Loose	Th	A-765	BISO
41	900	Bonded	Th, U	A-834, OR-2576-H	TRISO/TRISO
42	900	Loose	Th	A-780	BISO
43	900	Loose	Th	A-785	BISO
44	900	Bonded	Th, U	A-835, OR-2576-H	TRISO/TRISO
45	900	Loose	Th	A-806	BISO
46	900	Loose	Th	A-762	BISO
47	900	Bonded	Th, U	A-837, OR-2576-H	TRISO/TRISO
48	900	Loose	Th	A-782	BISO
49	900	Loose	Th	A-786	BISO
50	900	Bonded	Th, U	A-836, OR-2576-H	TRISO/TRISO
51	900	Loose	Th	A-787	BISO
52	900	Loose	Th	OR-1975-T	BISO
End Plug 28-3	900	Loose	inert	OR-2722-1, OR-2725-1	BISO

^aFrom top of capsule.^bLoose ThO₂ particles preceded by "6252-" unless noted otherwise.^cLTI burned off.

Table 2.12 Heavy Metal Loadings for Irradiation Capsule HT-34

Position ^a	Design Temperature (°C)	Loading, g			Number of Thorium Particles
		Uranium ^b	²³⁵ U	Thorium	
End Plug 14-3	1250			≤0.0233	≤56
1	1250	0.0191	0.0014		
2	1250			0.0233	56
3	1250	0.0191	0.0014		
4	1250			0.0233	57
5	1250			0.0233	56
6	1250	0.0191	0.0014		
7	1250			0.0233	56
8	1250			0.0233	57
9	1250	0.0191	0.0014		
10	1250			0.0233	57
11	1250			0.0233	56
12	1250	0.0191	0.0014		
13	1250			0.0233	56
End Plug 14-1	1250			≤0.0233	≤56
End Plug 15-3	1550			≤0.0332	≤80
14	1550	0.0268	0.0019		
15	1550			0.0332	80
16	1550	0.0268	0.0019		
17	1550			0.0332	81
18	1550			0.0332	81
19	1550	0.0268	0.0019		
20	1550			0.0332	83
21	1550			0.0332	82
22	1550	0.0268	0.0019		
23	1550			0.0332	81
24	1550			0.0332	81
25	1550	0.0268	0.0019		
26	1550			0.0332	85
End Plug 15-1	1550			≤0.0332	≤80
RHMP ^c					

Table 2.12 (continued)

Position ^a	Design Temperature (°C)	Loading, g			Number of Thorium Particles
		Uranium ^b	²³⁵ U	Thorium	
End Plug 26-1	1250	inert			
27	1250			0.0228	42
28	1250	0.0129	0.0009	0.0145	
29	1250			0.0228	41
30	1250			0.0228	42
31	1250	0.0129	0.0009	0.0145	
32	1250			0.0228	43
33	1250			0.0228	42
34	1250	0.0129	0.0009	0.0145	
35	1250			0.0228	41
36	1250			0.0228	43
37	1250	0.0129	0.0009	0.0145	
38	1250			0.0228	42
39-1	1250			0.0114	21
39-2	1250			0.0114	21
End Plug 26-3	1250	inerts			
End Plug 28-1	900	inerts			
40	900			0.0199	35
41	900	0.0056	0.0004	0.0104	
42	900			0.0199	35
43	900			0.0199	37
44	900	0.0056	0.0004	0.0104	
45	900			0.0199	37
46	900			0.0199	36
47	900	0.0056	0.0004	0.0104	
48	900			0.0199	36
49	900			0.0199	37
50	900	0.0057	0.0004	0.0104	
51	900			0.0199	37
52	900			0.0199	37
End Plug 28-3	900				

^aFrom top of capsule.^bAll uranium is 7.07% enriched.^cReactor horizontal midplane.

in terms of BAF_0 and Ne/He ratio, and the lowest fraction defective as determined by 1000°C gaseous chlorine leach and fission gas R/B ratios. All pertinent characterization data for these eight Biso particle batches are shown in Table 2.13.

Several batches of Triso-coated ThO_2 particles were also prepared in the 0.13-m coating furnace, with the frit gas distributor, for objective 2. Silicon carbide deposition rates near 0.5 $\mu\text{m}/\text{min}$ were achieved. Of these, four were selected for irradiation in HT-34. The high deposition rates for the SiC layer were obtained by controlling the deposition temperature at 1550°C, increasing the hydrogen-to-methyltrichlorosilane ratio, and increasing the methyltrichlorosilane flux. Each particle batch selected met the density requirements and was selected for its SiC microstructure. In this manner the effect of microstructure on the irradiation performance of the coating with respect to fission product retention and mechanical integrity could be studied. The characterization data for the SiC and the oLTI coatings for the four batches selected are shown in Table 2.14.

The Triso-coated ThO_2 particles were then combined with the uranium (7% enriched) driver particles and bonded bed specimens formed. These specimens were fabricated by the warm-molded process with a total particle loading of 35 vol %. The green rods were carbonized in natural flake graphite at 1000°C and annealed at 1800°C. The annealed rods had a nominal diameter of 9.58 mm and a length of 7.0 mm.

Archive particles from batches OR-1975-T and OR-2013-ST were also selected to resolve irradiation anomalies between these two similarly designed particle batches, which had been irradiated in capsules HT-18 and -19. Both particle batches were coated in a 25.4-mm conical coating tube at 1325°C. The hydrocarbon gas was propylene (C_3H_6) in each case. The difference between the two particle designs was that for OR-1975-T an argon diluent with a concentration of 67% was used whereas OR-2013-ST used CO_2 as the diluent. The irradiation anomalies were that batch OR-1975-T, of reference design, failed miserably ($\sim 90\%$) in capsule HT-19, but OR-2013-ST showed complete survival in both HT-18 and -19. Further characterization, as indicated in Table 2.13, is continuing in order to help resolve the uncertainties.

Table 2.13 Characterization Data for ORNL Bisoparticle Batches for HT-34

Run	Hydrocarbon Gas	Diluent		Deposition Temperature (°C)	LTI Thickness (μm)	Gradient Density, Mg/m ³		Open Porosity (%)	Coating Rate (μm/min)	BAF _c	Ne/He Permeability	Fraction Defective		Crushing Strength	
		Gas	(%)			Observed	Corrected					Cl ₂ Leach	Fission Gas Release	(N)	(lb)
A-762	Mixed ^a	He	67	1225	71.4	1.962	1.904	2.96	3.25	1.043	0.36	1.3×10^{-5}	$<1 \times 10^{-5}$	26	5.9
A-765	Propylene	Ar	50	1350	78.6	1.915	1.858	2.99	3.14	1.035	0.34	1.8×10^{-5}	$<2 \times 10^{-6}$	31	7.0
A-780	Propylene	He	50	1350	78.2	1.864	1.773	4.92	4.03	1.022	0.37	4.2×10^{-6}	$<2 \times 10^{-6}$	28	6.3
A-782	Propylene	H ₂	50	1350	88.4	1.855	1.810	2.43	2.95	1.026	0.27	5.2×10^{-5}	$<1 \times 10^{-6}$	32	7.3
A-785	Mixed	Ar	67	1225	76.3	1.872	1.815	3.02	3.05	1.035	0.32	8.6×10^{-7}	$<1 \times 10^{-6}$	25	5.6
A-786	Mixed	Ar	50	1225	88.0	1.897	1.826	3.75	3.67	1.030	0.36	8.9×10^{-7}	$<2 \times 10^{-6}$	31	7.0
A-787	Propylene	H ₂	67	1350	80.5	1.738	1.691	2.71	2.68	1.031	0.35	1.5×10^{-5}	$<4 \times 10^{-6}$	28	6.3
A-806	Mixed	H ₂	67	1225	70.6	1.943	1.878	3.33	2.82	1.036	0.38	1.1×10^{-6}	$<2 \times 10^{-6}$	22	4.9
OR-1975-T	Propylene	Ar	66.7	1315	83	1.982	N ^b	N ^b	11.8	1.063	0.19	N ^b	N ^b	N ^b	N ^b
OR-2013-ST	Propylene	Co ₂	66.7	1325	84.5	2.002	N ^b	N ^b	11.5	1.028	0.14	N ^b	N ^b	N ^b	N ^b
OR-2730-1-H ^c	Propylene	Ar	40	1325	81.2	1.942	1.854	4.51	12.8		0.26			N ^b	N ^b
OR-2722-1-H ^c	Propylene	Ar	66.7	1315	81.9	1.984	1.891	4.67	12.6		0.27			N ^b	N ^b
OR-2725-1-H ^c	Propylene	Ar	66.7	1315	82.2	1.998	1.910	4.42	11.7		0.27			N ^b	N ^b

^a54% acetylene, 46% propylene.^bCharacterization is now in progress and will be reported when available.^cInert kernels; particles irradiated in end plugs of ORNL magazines in HT-34

Table 2.14 Characterization Data for ORNL Triso Particle Batches for HT-34

Run No.	SiC thickness (μm)	SiC deposition rate ($\mu\text{m}/\text{min}$)	SiC density (Mg/m^3)	SiC fraction defective Hg intrusion	OLTI thickness (μm)	OLTI observed gradient density (Mg/m^3)	OLTI corrected gradient density (Mg/m^3)	OLTI coating rate ($\mu\text{m}/\text{min}$)
A-834	32.7	0.16	3.194	1.3×10^{-4}	43.5	1.898	1.752	5.30
A-835	36.5	0.45	3.193	1.4	47.7	1.883	1.767	5.81
A-836	34.3	0.28	3.158	1.5	46.6	1.853	1.736	5.68
A-837	35.5	0.39	3.183	1.4	49.0	1.890	1.771	5.98

2.4.8.3 Operating Conditions

Irradiation capsule HT-34 was inserted into HFIR Target position C-6 on July 25, 1977, to begin a scheduled five-cycle irradiation. The capsule was removed from this facility on November 23, 1977, after completing a total of 2685 hr at full reactor power of 100 MW. The reactor power and irradiation history for the capsule are shown in Table 2.15. Total accumulated damage fluence and the calculated burnups for each ORNL test position are shown in Table 2.16.

Table 2.15 Reactor History During Operation of HT-34

HFIR Fuel Cycle	Cycle Dates		Irradiation Time, hr	
	Begin	End	In Cycle	Total Accumulated
150	7/25/77	8/17/77	549	549
151	8/19/77	9/11/77	547	1096
152	9/11/77	10/4/77	545	1641
153	10/6/77	10/29/77	537	2178
154 ^a	10/31/77	11/23/77	507	2625

^aThe reactor was shut down for 55.3 hr (from 0500 11/7/77 to 1220 11/9/77) during cycle 154.

2.4.8.4 Postirradiation Examination

The HT-34 capsule was removed from HFIR and subsequently shipped to the High Radiation Level Examination Laboratory (HRLEL) in mid-December 1977 for disassembly and examination of components. The examination is now in progress.

2.5 IRRADIATION TESTS IN HFIR REMOVABLE BERYLLIUM FACILITY — F. J. Homan

At the end of this reporting period no HRB capsules were in the reactor. One capsule (HRB-13) came out at the end of November, and two capsules (HRB-14 and -15b) were being planned for insertion in HFIR during the first half of 1978. Postirradiation examination of HRB-11 and -12 was in progress.

Table 2.16 Calculated Burnup and Damage Fluence for HT-34

Specimen	Burnup, % FIMA			Fluence, >0.18 MeV (n/m ²)
	²³³ Th	²³⁵ U	²³⁸ U	
1, 52		85.0	10.8	4.8×10^{25}
2, 51	5.6			5.1
3, 50		95.2	11.7	5.5
4, 49	6.4			5.8
5, 48	6.7			6.1
6, 47		85.5	13.2	6.4
7, 46	7.3			6.7
8, 45	7.6			7.0
9, 44		85.7	14.6	7.2
10, 43	8.3			7.5
11, 42	8.6			7.7
12, 41		85.9	15.9	8.0
13, 40	9.2			8.2
14, 39		86.3	18.3	9.1
15, 38	10.7			9.2
15, 37		86.4	18.8	9.3
17, 36	11.0			9.4
18, 35	11.2			9.5
19, 34		86.5	19.5	9.6
20, 33	11.5			9.7
21, 32	11.6			9.8
22, 31		86.6	20.1	9.9
23, 30	11.9			10.0
24, 29	12.0			10.1
25, 28		86.7	20.5	10.2
26, 27	12.2			10.2

Most of the examinations for these capsules have been completed, but some microprobe work remains along with the IMGA analysis.

2.5.1 Capsules HRB-11 and -12 — T. N. Tiegs and K. R. Thoms

The design and fabrication of capsules HRB-11 and -12 were reported previously.³⁶

2.5.1.1 Capsule Operation

The operating histories for HRB-11 and -12 are presented in Table 2.17. Fission gas release-to-birth ratios (R/B) for both capsules and calculated fuel burnups and fast fluences for HTB-12 are given in Tables 2.18 through 2.20. The calculated fuel burnups and neutron fluences for HRB-11 have been reported previously.³⁶

The measured fission gas R/B ratios were high for both capsules, especially at the end of the irradiations (see Tables 2.18 and 2.19). The HTGR fuel product specification calls for R/B values less than 3×10^{-5} for ^{85m}Kr , but the values for this experiment exceeded this value by factors of up to 15. However, since many varieties of fuel were in the capsules,³⁶ nothing definitive can be said about the R/B value for any one fuel type. The experimental objectives of these capsules were to be accomplished by comparing the performance of particles with various coating designs and deposition conditions. High failure rates in a few of the specimens would result in high R/B values, even if the majority of the specimens remained within performance specifications. A definitive measurement of the failure fraction for both fissile and fertile particles in every test specimen is possible with the IMGA System (see Section 2.9.3); IMGA analysis on the HRB-11 and -12 fuel specimens will be completed in 1978.

All reactor operations were routine during irradiation except in the case of HRB-12. It was removed from the reactor and reinserted following completion of the slow startup cycle, cycle 140. After having been out of the reactor for one month, ^{233}U and ^{239}Pu precursors had decayed so as to increase the linear heat generation rate about 25% over what it would have been if the experiment had not been removed for one cycle. When this increase in heat generation was considered along with the postirradiation dimensional

Table 2.17 Capsules HRB-11 and -12 Operating History

HFIR Fuel Cycle	Cycle Dates		Capsule HRB-11			Capsule HRB-12		
			Operating Facility	Irradiation Time, hr		Operating Facility	Irradiation Time, hr	
	Begin	End		In Cycle	Total Accumulated		In Cycle	Total Accumulated
126	12/7/75	12/30/75	PB-13	559	559			
127	12/31/75	1/23/76	PB-13	558	1117			
218	1/24/76	2/16/76	PB-13	556	1673			
219	2/19/76	3/13/76	RB-7	554	2227	PB-13	554	554
130	3/14/76	4/6/76	RB-7	558	2785	PB-13	558	1112
131	4/7/76	4/30/76	RB-7	552	3337	PB-13	552	1664
312	5/1/76	5/22/76	RB-7	490	3827	RB-5	490	2154
133	5/23/76	6/16/76	RB-7	547	4374	RB-5	547	2701
134	6/17/76	7/9/76	RB-7	534	4908	RB-5	534	3235
135	7/10/76	8/1/76	RB-7	547	5455	RB-5	547	3782
136	8/2/76	8/25/76	RB-7	540	5995	RB-5	540	4322
137	9/2/76	9/25/76	RB-7	564	6559	RB-5	564	4886
138	9/27/76	10/21/76				RB-5	574	5460
319	10/21/76	11/14/76				RB-5	574	6034
140 ^a	11/15/76	12/14/76						
141	12/15/76	1/10/77				RB-5	568 ^b	6602 ^b

^a Capsule was removed from facility during cycle 140.

^b Effective time at 100 MW; reactor operated at lower power.

Table 2.18 Capsule HRB-11 R/B Data

Sample Number ^a	Date	Accumulated Irradiation Time (hr)	Fission Gas, R/B				
			^{85m} Kr	⁸⁷ Kr	⁸⁸ Kr	¹³⁵ Xe	¹³³ Xe
			$\times 10^{-6}$	$\times 10^{-6}$	$\times 10^{-6}$	$\times 10^{-6}$	$\times 10^{-5}$
1	2/25/76	1821	7.20	4.14	4.58	3.62	1.46
2	2/27/76	1871	4.56	3.73	3.34	2.72	0.468
3	3/2/76	1965	6.62	4.49	4.20	3.29	7.79
4	3/5/76	2038	6.98	3.56	4.31	3.35	10.7
5	3/9/76	2132	5.94	3.15	4.29	3.08	2.97
6	3/12/76	2204	17.80	7.06	9.65	5.43	40.80
7	3/16/76	2273	4.55	3.15	3.11	2.69	2.53
8	3/19/76	2341	62.7	22.7	26.6	18.9	210.
9	3/23/76	2439	22.0	13.5	13.3	15.2	11.8
10	3/26/76	2509	28.0	15.7	15.8	24.0	30.8
11	3/30/76	2603	32.2	20.5	19.3	17.8	18.5
12	4/5/76	2748	27.5	20.2	16.4	19.7	23.3
13	4/13/76	2918	13.5	8.69	8.31	7.18	15.8
14	4/23/76	3158	6.46	3.39	3.41	2.37	2.80
15	4/27/76	3253	9.78	6.09	6.44	4.39	4.48
16	5/7/76	3470	7.55	5.33	5.98	4.84	6.38
17	5/14/76	3638	4.56	3.08	2.86	3.30	1.15
18	5/18/76	3737	6.31	6.65	4.10	4.95	3.91
19	6/2/76	4032	8.42	10.9	9.44	8.19	4.40
20	6/9/76	4204	6.05	7.68	7.56	5.44	3.42
21	6/11/76	4252	6.44	7.87	7.07	6.60	3.04
22	6/23/76	4529	10.5	17.4	5.86	6.22	3.54
23	6/30/76	4698	8.86	7.12	7.13	5.45	3.20
24	7/15/76	5034	145.	103.	101.	90.5	60.3
25	7/20/76	5157	126.	82.2	86.0	76.7	53.9
26	7/28/76	5345	147.	95.9	104.	104.	128.
27	8/11/76	5665	230.	171.	143.	153.	238.
28	8/18/76	5836	242.	151.	174.	168.	242.
29	9/9/76	6160	437.	299.	290.	281.	278.
30	9/14/76	6281	300.	329.	356.	277.	267.
31	9/21/76	6449	246.	269.	278.	203.	

^a Samples 1 through 7 were taken with the sweep gas at 100% He, while the remainder of the samples were taken with a helium-neon mixture and the specimens at design temperature.

Table 2.19 Capsule HRB-12 R/B Data

Sample Number ^a	Date	Accumulated Irradiation Time (hr)	Fission Gas, R/B				
			^{85m} Kr	⁸⁷ Kr	⁸⁸ Kr	¹³⁵ Xe	¹³³ Xe
			$\times 10^{-5}$	$\times 10^{-5}$	$\times 10^{-5}$	$\times 10^{-5}$	$\times 10^{-4}$
1	5/4/76	1723	9.35	5.60	5.49	5.52	3.15
2	5/7/76	1795	6.19	7.18	5.56	5.77	3.12
3	5/14/76	1964	8.15	5.19	3.68	4.60	4.58
4	5/18/76	2063	9.01	6.70	6.86	5.17	3.52
5	5/21/76	2131	10.4	7.75	6.23	6.02	5.44
6	6/2/76	2359	12.9	8.46	8.78	12.0	7.47
7	6/9/76	2531	12.0	7.44	7.90	10.8	5.97
8	6/11/76	2577	15.6	8.26	9.90	11.7	89.7
9	6/23/76	2856	7.70	6.63	5.17	9.40	6.97
10	6/30/76	3024	7.33	4.44	5.25	8.11	6.44
11	7/15/76	3361	4.88	3.09	3.33	4.50	3.01
12	7/20/76	3483	4.81	3.39	4.00	4.09	4.02
13	7/28/76	3672	6.28	3.98	4.18	6.13	6.16
14	8/11/76	3992	3.49	2.92	3.49	2.80	1.95
15	8/18/76	4163	4.11	3.59	3.64	3.33	4.05
16	9/9/76	4487	10.6	7.58	8.42	5.85	6.14
17	9/14/76	4607	7.99	8.59	9.20	6.51	9.71
18	9/21/76	4775	7.12	6.51	8.92	5.70	1.17
19	9/30/76	4964	6.13	5.36	6.22	4.89	0.668
20	10/6/76	5104	8.13	6.33	5.74	3.71	0.709
21	10/12/76	5248	8.11	6.54	6.75	4.18	8.06
22	10/19/76	5415	7.31	5.79	6.22	4.78	5.22
23	10/26/76	5570	9.30	6.70	7.54	5.22	6.67
24	11/3/76	5764	10.9	6.90	8.26	4.81	8.68
25	11/10/76	5931	11.3	7.37	7.50	5.51	7.27

^a Samples 1 through 5 were taken with the sweep gas at 100% He, while the remainder of the samples were taken with a helium-neon mixture and the specimens at design temperature.

Table 2.20 Calculated Specimen Burnup and Fast Fluence in Capsule HRB-12

Specimen	Calculated Burnup, % FIMA									Fluence, n/m ² (>0.18 MeV)		
	²³⁵ U			²³⁸ U			²³² Th			PB-13	RB-5	Total
	PB-13	RB-5	Total	PB-13	RB-5	Total	PB-13	RB-5	Total			
1	52	32	84	0.9	15.2	16.1	0.2	6.3	6.5	1.92×10^{24}	4.21×10^{25}	4.40×10^{25}
2	54	31	85	1.0	17.1	18.1	0.3	7.1	7.4	2.08	4.73	4.94
3	57	28	85	1.1	19.2	20.3	0.3	8.1	8.4	2.23	5.18	5.40
4	59	27	86	1.2	20.9	22.1	0.3	9.0	9.3	2.38	5.60	5.84
5	60	26	86	1.3	22.5	23.8	0.4	9.8	10.2	2.51	6.00	6.25
6	61	25	86	1.4	23.8	25.2	0.4	10.5	10.9	2.66	6.32	6.59
7	62	24	86	1.4	24.9	26.3	0.4	11.1	11.5	2.78	6.62	6.90
8	63	24	87	1.5	25.8	27.3	0.4	11.6	12.0	2.87	6.85	7.14
9	64	23	87	1.5	26.5	28.0	0.4	12.0	12.4	2.94	7.07	7.36
10	64	23	87	1.6	26.9	28.5	0.5	12.1	12.6	3.02	7.24	7.54
11	64	23	87	1.6	27.4	29.0	0.5	12.4	12.9	3.05	7.32	7.63
12	65	22	87	1.6	27.6	29.2	0.5	12.5	13.0	3.05	7.34	7.65
13	64	23	87	1.6	27.5	29.1	0.5	12.5	13.0	3.02	7.32	7.62
14	64	23	87	1.6	27.2	28.8	0.5	12.3	12.8	3.01	7.29	7.59
15	64	23	87	1.6	26.7	28.3	0.4	12.1	12.5	2.98	7.20	7.50
16	63	24	87	1.5	26.2	27.7	0.4	11.8	12.2	2.91	7.05	7.34
17	62	24	86	1.4	25.4	26.8	0.4	11.3	11.7	2.86	6.85	7.14
18	61	25	86	1.3	24.4	25.7	0.4	10.7	11.1	2.74	6.58	6.85
19	59	27	86	1.2	23.4	24.6	0.3	10.3	10.6	2.61	6.28	6.54
20	58	28	86	1.1	21.9	23.0	0.3	9.4	9.7	2.49	5.93	6.18
21	56	30	86	1.0	20.5	21.5	0.3	8.7	9.0	2.35	5.50	5.74
22	54	31	85	0.9	18.9	19.8	0.3	7.9	8.2	2.21	5.11	5.33
23	52	33	85	0.9	17.3	18.2	0.2	7.2	7.4	2.09	4.71	4.92
24	49	36	85	0.8	15.6	16.4	0.2	6.4	6.6	2.02	4.43	4.63
25	47	37	84	0.7	14.4	15.1	0.2	5.7	5.9	1.91	4.09	4.28

change data of HRB-11, we calculated that, even with 100% He sweep gas, the fuel centerline temperature would be 1470°C with the reactor at 100 MW. Since the design fuel centerline temperature was 1350°C, we requested that the reactor power be lowered to 90 MW. At 90 MW, the calculated fuel centerline temperature was about 1350°C. The reactor power remained at 90 MW for about 10 days and then was increased to 95 MW when enough fissile material was burned out to allow this increase. The reactor power was kept at 95 MW for the remainder of the cycle and the sweep gas maintained at 100% He for the entire cycle.

2.5.1.2 Postirradiation Examination

After disassembly, a dimensional analysis of the fuel rods and graphite sleeve and a visual inspection were performed. Results of the visual examination and dimensional analysis of HRB-11 fuel rods were reported last year.³⁶ Dimensional analysis results from HRB-12 are given in Table 2.21.

Table 2.21 HRB-12 Fuel Rod Dimensional Changes

Rod Number ^a	Postirradiation Diameter (mm)	Percent Change	Postirradiation Length (mm)	Percent Change
1	12.217	-1.90	16.312	-2.16
2	12.220	-1.86	16.528	-1.77
3	12.291	-1.51	16.744	-1.85
4	12.283	-1.33	16.477	-1.59
5	12.286	-1.25	16.629	-1.46
6	12.258	-1.63	16.891	-2.48
7	12.291	-1.45	16.467	-1.01
8	12.306	-1.30	16.665	-0.08
9	12.271	-1.25	----	--
10	12.375	-0.65	16.695	-0.86
11	12.352	-0.86	16.744	-0.99
16	12.385	-0.65	16.754	-0.75
17	12.339	-1.00	16.716	-0.90
18	12.324	-1.04	16.645	-0.92
19	13.329	-0.98	16.650	-1.50
20	12.294	-1.24	16.690	-0.93
21	12.306	-1.12	16.401	-1.43

^aRods 12-15 and 22-25 were GA rods and are not reported here.

All the HRB-12 fuel rods except in positions 9 and 19 appeared in excellent condition. Some debonding occurred with rods 9 and 19, but both were essentially intact.

Selected fuel rods were examined metallographically. Other rods were electrolytically deconsolidated in preparation for IMGA analysis.

The partially converted kernels performed better than kernels consisting entirely of UO_2 or UC_2 (Fig. 2.28). Thermodynamic considerations indicate that at least 14% conversion is necessary to prohibit amoeba migration, while a maximum of 70% conversion will stop rare earth fission product (i.e., Nd, Ce, Pr, and La) migration.³⁷ In agreement with these observations, the present results combined with previous results⁹ show the range of conversion with acceptable irradiation behavior is 15 to 60%. It should be noted, however, that all these particle types will be analyzed by the IMGA system, which will give additional information on the irradiation performance.

All previous tests used microspheres derived from resin produced by Rohm and Hass, Amberlite IRC-72, and a second source of resin was desired. Testing of kernels made from Diamond Shamrock Duolite C-464 resin showed greater irradiation shrinkage than Amberlite IRC-72 (Fig. 2.29). This difference is caused by a difference in the initial densities of the kernels: Duolite C-464 — 2.4 Mg/m^3 vs Amberlite IRC-72 — 3.2 Mg/m^3 .

WAR particles were tested with buffer coating thicknesses of $50 \mu\text{m}$ (present reference), $25 \mu\text{m}$, and 0. The particles with 50- and $25\text{-}\mu\text{m}$ buffers showed similar irradiation performance (Fig. 2.30). No detrimental effects were observed. Thus it appears the buffer thickness for WAR particles could be lowered to $25 \mu\text{m}$. On the other hand, the WAR particles irradiated with no buffer showed a "thinning" of the inner pyrocarbon coating (Fig. 2.31). Evidently additional carbon is required by the WAR kernel, and this is usually supplied by the buffer coating. The effect of the inner pyrocarbon coating thinning on particle performance and fission product retention is not known, but any degradation of the coatings is normally considered detrimental.

Silicon carbide coatings deposited at rates of 0.24, 0.52, and $0.90 \mu\text{m/min}$ were tested. (The reference silicon carbide is deposited at $0.2 \mu\text{m/min}$.) The silicon carbide deposited at rates above $0.5 \mu\text{m/min}$

Fig. 2.28 Representative Particles from HRB-11, Showing that Partially Converted Particles Performed Well. These results combined with previous tests indicate that the range of conversion with acceptable behavior is 15 to 60%. 120 $\times$. (a) 30%-Converted Particle from HRB-11-19. (b) 60%-Converted Particle from HRB-11-1. (c) 70%-Converted Particle from HRB-11-21.

Fig. 2.29 Kernels Made From C-464 Resin (Left) Show Greater Irradiation Shrinkage Than Kernels Made from Amberlite IRC-72 (Right), 150 $\times$. Particles 15% converted. This difference is caused by a difference in the initial density of the respective kernels.

(a) 50 μ m Buffer

(b) 25 μ m Buffer

Fig. 2.30 WAR Particles (50% Converted) with 50- and 25- μ m Buffers Have Similar Irradiation Behaviors, 150 $\times$. (a) 50- μ m-Buffer Particles was from HRB-11-20. (b) 25- μ m-Buffer Particle from HRB-11-3.

R-74350

Fig. 2.31 WAR Particles Irradiated with no Buffer Show Thinning of Inner LTI Where the Kernel has Adhered. This 50%-converted particle was from HRB-11-2. 150 $\times$. (a) Bright Field. (b) Polarized Light.

showed greater susceptibility to fission product corrosion (Fig. 2.32). Electron microprobe examination found the corrosion was due to fission product palladium. Since normally the silicon carbide has been deposited at low deposition rates, no palladium attack has been previously reported in high-enriched uranium particles. However, palladium becomes increasingly more important when one deals with low-enriched uranium fuel (now proposed for use in HTGR), because of the much higher fission product yield from plutonium fission than from uranium. Thus silicon carbide deposition rates above 0.5 $\mu\text{m}/\text{min}$ would not be recommended.

2.5.2 Capsule HRB-13 — E. L. Long, Jr. and K. R. Thoms

The objectives, design, details and early stage of irradiation of HRB-13 have been reported.³⁸ Capsule HRB-13 was removed from the HFIR on November 23, 1977, after having completed 14 full HFIR cycles (four cycles in the PB-facility and ten cycles in the RB facility) for a total operating time of 7721 hr at full reactor power of 100 MW (Table 2.22). The operating temperature in HRB-13 continued to increase during the last two cycles and, therefore, the sweep gas was kept at 100% He during that period. The estimated peak center-line temperature for the last cycle is 1325°C at the reactor horizontal mid-plane. A complete summary of fission gas release-to-birth rate ratios (R/B) for gas samples taken during the irradiation is presented in Table 2.23. These measurements indicate a noticeable increase in the R/B ratios for those samples taken during the last two cycles of operation. Burnup and fluence data summaries for all specimens in HRB-13 are reported in Table 2.24. Post-irradiation examination is scheduled to begin in early 1978.

2.5.3 Capsule HRB-14 — M. J. Kania and E. L. Long, Jr.

Capsule HRB-14 will be irradiated in the Removable Beryllium (RB) Facility of HFIR. The test space will be shared with GA. The primary objective of the ORNL portion of this experiment is a basic study of the irradiation-induced gas permeability of reference Biso-coated fertile particles. A secondary objective is to establish fuel product specification data on the range of BAF_0 and density values for the LTI pyrocarbon coating that will lead to successful irradiation performance.

Fig. 2.32 When Deposited at Rates Above 0.5 $\mu\text{m}/\text{min}$, SiC Shows Greater Suceptability to Fission Product Corrosion. The corrosion is easiest observed under polarized light, where it appears as bright spots. The 0.2- $\mu\text{m}/\text{min}$ particle was from HRB-12-18; 0.5- $\mu\text{m}/\text{min}$ from HRB-12-5; and the 0.9- $\mu\text{m}/\text{min}$ from HRB-12-4. 250 $\times$.

Table 2.22 Reactor History During Operation of HRB-13

HFIR Fuel Cycle	Operating Facility	Cycle Dates		Irradiation Time, ^a hr	
		Begin	End	In Cycle	Total Accumulated
140	PB-13	11/15/76	12/14/76	577	577
141	PB-13	12/15/76	01/10/77	568	1145
142	PB-13	01/10/77	02/03/77	576	1721
143 ^b		02/05/77	03/01/77		
144	PB-13	03/02/77	03/26/77	563	2284
145	RB-7	03/27/77	04/20/77	599	2843
146	RB-7	04/20/77	05/13/77	550	3393
147	RB-7	05/14/77	06/06/77	544	3937
148	RB-7	06/06/77	06/29/77	552	4489
149	RB-7	07/02/77	07/24/77	547	5036
150	RB-7	07/25/77	08/17/77	549	5585
151	RB-7	08/19/77	09/11/77	547	6132
152	RB-7	09/11/77	10/04/77	545	6677
153	RB-7	10/06/77	10/29/77	537	7214
154	RB-7	10/31/77	11/23/77	507	7721

^aIrradiation time is given in equivalent hours at 100 MW. It should be noted that cycle 140 was a slow-start-up cycle, and during cycle 141 the reactor was operated at 90 or 95 MW.

^bCapsule HRB-13 was removed from the reactor during cycle 143 to facilitate repairs to a leaking secondary gas system.

Table 2.23 Fission Gas Release-To-Birth Data For Capsule HRB-13

Sample	Date	Accumulated Irradiation Time (hr)	Fission Gas R/B				
			⁸⁵ mKr	⁶⁷ Kr	⁸⁸ Kr	¹³⁵ Xe	¹³³ Xe
			x 10 ⁻⁴	x 10 ⁻⁴	x 10 ⁻⁴	x 10 ⁻⁴	x 10 ⁻³
1	12/10/76	490	6.10	4.10	3.30	3.20	2.70
2	01/05/77	1040	1.50	1.00	0.85	0.70	0.10
3	01/27/77	1540	0.83	0.57	0.55	0.36	0.68
4	02/01/77	1660	1.10	0.64	0.70	0.48	0.97
5	03/14/77	2010	0.42	0.28	0.28	0.19	0.23
6	04/01/77	2390	6.30	4.00	4.30	6.10	2.00
7	04/05/77	2490	5.20	3.90	3.30	4.80	1.80
8	05/02/77	3120	3.70	2.40	2.60	2.60	2.10
9	05/11/77	3340	4.50	2.80	3.10	3.20	1.90
10	05/19/77	3510	2.50	1.80	1.50	1.30	0.98
11	05/26/77	3680	Sample did not analyze				
12	05/31/77	3800	3.00	2.10	2.10	1.60	1.50
13	06/10/77	4020	2.00	1.60	1.40	1.00	0.82
14	06/14/77	4120	1.60	1.20	1.20	0.67	0.51
15	06/21/77	4290	1.50	1.20	1.20	0.68	0.86
16	06/28/77	4460	2.20	1.60	1.50	1.10	0.94
17	07/07/77	4620	1.30	1.20	0.90	0.62	0.44
18	07/13/77	4760	1.40	1.10	0.97	0.67	0.45
19	07/20/77	4931	2.30	1.80	1.80	1.20	1.40
20	07/28/77	5102	1.50	1.20	1.10	0.81	0.64
21	08/03/77	5245	1.60	1.30	1.20	0.86	0.85
22	08/11/77	5438	1.80	1.50	1.40	1.00	1.20
23	08/23/77	5836	1.60	1.50	1.30	1.00	0.75
24	09/02/77	5928	2.00	1.70	1.60	1.20	1.30
25	09/08/77	6073	2.30	1.90	1.80	1.60	1.10
26	09/15/77	6224	1.90	1.80	1.60	1.40	1.20
27	09/23/77	6413	1.60	1.60	1.40	1.10	1.10
28	09/29/77	6530	2.10	1.80	1.80	1.50	1.40
29	10/24/77	7173	2.20	1.90	1.90	1.70	2.00
30	11/14/77	7584	3.40	3.00	2.80	2.60	3.00
31	11/21/77	7749	6.20	4.90	4.10	3.40	4.40

^aSamples 1 thru 7 were taken with the sweep gas being 100% Helium while the remainder were taken at design temperature. Samples 1 thru 5 were taken while the capsule was in the PB-13 facility while the remainder were taken with the capsule in the RB-7 facility.

^bTime given is equivalent 100-MW reactor power time.

Table 2.24 Calculated Burnup and Fast Fluence for Specimens in Capsule HRB-13

Specimen	Calculated Burnup, % FIMA									Fluence, n/m ² , >0.18 MeV		
	²³⁵ U			²³⁸ U			²³² Th			PB-13	RB-7	Total
	PB-13	RB-7	Total	PB-13	RB-7	Total	PB-13	RB-7	Total			
1, 2, 39, 40	60	25	85	0.93	10.5	11.4	0.35	4.2	4.6	2.6×10^{24}	4.6×10^{25}	4.9×10^{25}
3, 4, 37, 38	63	22	85	1.07	12.7	13.8	0.41	5.3	5.7	2.8	5.2	5.5
5, 6, 35, 36	66	20	86	1.26	14.6	15.9	0.49	6.2	6.7	3.1	5.8	6.1
7, 8, 33, 34	68	18	86	1.43	16.3	17.7	0.56	6.9	7.5	3.3	6.3	6.6
9, 10, 31, 32	70	16	86	1.54	17.7	19.2	0.62	7.7	8.3	3.6	6.6	7.0
11, 12, 29, 30	71	16	87	1.69	18.9	20.6	0.68	8.3	9.0	3.8	7.2	7.6
13, 14, 27, 28	72	15	87	1.76	19.4	21.2	0.72	8.6	9.3	3.9	7.5	7.9
15, 16, 25, 26	73	14	87	1.84	20.5	22.3	0.76	9.0	9.8	4.1	7.8	8.2
17, 18, 23, 24	73	14	87	1.88	21.0	22.9	0.78	9.3	10.1	4.1	7.9	8.3
19, 20, 21, 22	74	13	87	1.91	21.3	23.2	0.79	9.5	10.3	4.2	8.1	8.5

One half the experimental test volume of the irradiation capsule will contain ORNL fuel specimens. The remaining portion of the capsule will be occupied by fuel from GA. The ORNL philosophy is to irradiate a maximum number of Biso-coated fertile particle batches with variations in coating characteristics produced by a well-defined selection of coating parameters. Irradiation-induced permeability will be investigated by irradiation sets of particle batches along the length of the allotted test space.

Figure 2.33 describes the ORNL test philosophy graphically. It shows schematically how coatings described by their respective pyrocarbon crystallite disorder perform as a function of fast neutron fluence. The pyrocarbon crystallite disorder, described by BAF_0 values, and by Ne/He ratio measurements, is an indication of the permeability of pyrocarbon coatings. At one end of the spectrum are coatings with high BAF_0 and low Ne/He values. These particles can be characterized by a low crystallite disorder and are gastight as deposited. However, after a period of time in a neutron environment, the coatings develop cracks and lose their fission gases. Those particles developing macrocracks lose their fission gas early in life, while those developing microcracks lose their fission gas later. At the opposite end of the spectrum are those coatings with low BAF_0 values and high Ne/He ratios. These particles are characterized by a high crystallite disorder and are permeable as fabricated. Fission-gas loss continues from the beginning of irradiation and extends throughout the irradiation lifetime. Generally, the rate of fission gas loss decreases because of an improvement in the pyrocarbon disorder by rearrangements within the crystallites. The desired situation is between the ends of this spectrum range. Here particles have as-deposited gastight coatings and intermediate crystallite disorder. These particle coatings remain gastight through their irradiation lifetime.

By a systematic variation in process conditions, as-deposited coatings have been produced that cover the range in BAF_0 and Ne/He values discussed above. Through extensive postirradiation examination, radiation-induced permeability will be determined along with ranged in BAF_0 and Ne/He values that lead to successful irradiation performance. For those particles that develop cracked coatings, macro- or microcracks, limits in performance as a function of fast neutron fluence will be established.

Fig. 2.33 Permeability Changes of Biso Coatings May be Predictable from Knowledge of the As-Deposited Pyrocarbon Crystallite Disorder.

Experiment HRB-14 will be irradiated for ten cycles in the RB facility. The expected fluence will range from 4×10^{25} to 8.1×10^{25} n/m^2 (>0.18 MeV) at the ends of the capsule and at the reactor horizontal midplane, respectively. Both ORNL and GA specimens will have a maximum centerline operating temperature of 1250°C .

The upper portion of the capsule will contain ORNL specimens. These specimens will be in the form of planchets 12.5 mm in diameter and about 2.5 mm long. Planchets will be formed by molding the particles in graphite such that a continuous matrix surround the particles. This will produce a high-thermal-conductivity fuel body and thereby minimize the radial temperature gradient. Four sets of planchets, each set containing 18 batches (one batch per planchet) will be irradiated in tandem over the length of

the test space. Each planchet will contain Biso-coated fertile and sufficient Triso-coated uranium (20% enriched) particles to maintain the 1250°C centerline temperature at beginning of life. The current schedule calls for an insertion date in HFIR in April 1978.

2.5.4 Capsule HRB-15b — E. L. Long, Jr., and D. P. Stinton

This capsule will be shared between ORNL and GA. The primary objectives of the ORNL portion of the capsule will be to: (1) investigate the behavior of Biso-coated fertile particles irradiated to reference HTGR burnup and fast neutron fluence levels but at relatively low temperatures, and (2) assess the performance of the outer low-temperature isotropic (oLTI) coatings and SiC layers on relatively large-diameter Triso-coated particles.

During the examination of several samples of loose Biso-coated particles from an earlier series of accelerated capsule tests, apparently anomalous results were observed. Specifically, particles from the same batches survived in the high-temperature portions of the capsules but failed in the lower temperature regions.²² These failures were in the form of neutron-induced permeability, as determined by fission-gas inventory measurements and hot gaseous chlorine leaching studies.²² During the postirradiation examination of particles from HT-12 through -15 techniques other than visual examination were used to determine coating failures. Visual examination is adequate to verify pressure-vessel failure, but is not adequate to detect either the presence of microcracks or permeable coatings.

A significant number of batches of Biso-coated particles have been prepared to investigate the phenomenon of fast-neutron-induced permeability in pyrocarbon. A portion of these particles have been irradiated in capsules HT-34 (Sect. 2.4.8) and OF-2,³⁹ and the remainder are planned for inclusion in a HFIR instrumented and swept capsule, HRB-14 (Sect. 2.5.3). Capsule HT-34 is currently undergoing postirradiation examination, and HRB-14 is tentatively scheduled to be inserted in HFIR in April 1978. The surface temperature of the particles in these experiments will be relatively high compared with those planned for HRB-15b. Thus, to have a more complete understanding of neutron-induced permeability in the temperature and fluence ranges of interest in a large HTGR core, we will include particles

from batches prepared for HT-34 and HRB-14 in HRB-15. A summary of the particles scheduled for HRB-15b, along with deposition conditions and coating properties, is given in Tables 2.25 and 2.26.

The current reference recycle fissile particle is not compatible with the design requirements for an HTGR core containing MEU fuel; that is, the heavy metal loading in the WAR kernel is not sufficient to meet the mass of uranium and thorium required per unit volume of fuel rod. To meet the design requirements a Triso-coated high-density kernel with an overall nominal diameter of 800 μm is needed. To date experience in producing and evaluating the irradiation performance of large-diameter Triso-coated particles has been very limited. The oLTI of large Triso-coated particles included in two fuel rods in HRB-7 and -8 performed inadequately.⁴⁰ Since that time several batches of Triso-coated ThO_2 particles have been produced in a production-size coater at ORNL. Samples from four such batches were incorporated in small-diameter fuel rod specimens in capsule HT-34 to evaluate SiC deposition rate. Incorporation in HRB-15b of samples from these four batches plus six additional batches, which could not be included in HT-34, will result in a valuable parametric investigation into the performance of oLTI and SiC coatings on large-diameter particles. The irradiation conditions associated with HRB capsules are more representative of HTGR conditions than those achieved in HT capsules. The ten batches of Triso-coated particles proposed for Capsule HRB-15b are listed in Table 2.26 with the coating properties.

Approximately one-third of the experimental test volume of the irradiation capsule will be occupied by ORNL fuel specimens. The remaining portion of the capsule will be occupied by fuel specimens supplied by GA. The ORNL test space will contain particles from 18 batches (see Tables 2.25 and 2.26). The test specimens will be thin wafers of planchets containing loose coated particles. The ORNL portion of the capsule will contain 59 planchets, making possible the irradiation of particles from each of the 18 batches in three different locations (fluences) of the capsule.

Capsule HRB-15b is scheduled for insertion in HFIR in May 1978, and will be irradiated for ten cycles in the RB facility. The ORNL particles

Table 2.25 Positions and Properties of Biso-Coated Particles to be Tested in Capsule HRB-15b for Behavior at Low, Intermediate, and Reference Fast Fluence and Burnup at Low Irradiation Temperatures

Capsule Position	Batch	Hydrocarbon Source	Diluent	Deposition Temperature (°C)	Coater (cm)	BAF _O	Coating Rate (μm/min)	Defective Fraction Cl ₂ Leach	Ne/He Ratio	Coating Thickness (μm)	Crushing Strength		Gradient Density, Mg/m ³		Porosity (%)	Shape Ratio
											(N)	(lb)	Observed	Corrected		
95, 125, 155	A-765 ^{a,b}	Propylene	50% Ar	1350	13	1.035	3.14	1.8×10^{-5c}	0.34	78.6	31.1	6.99	1.9153	1.8580	2.994	1.110
99, 101, 129 133, 157, 159	A-786 ^{a,b}	Mixed	50% Ar	1225	13	1.030	3.67	8.9×10^{-7c}	0.36	88.0	31.2	7.03	1.8969	1.8258	3.750	1.115
109, 135, 165	J-482 ^d	Propylene	50% He	1350	13	1.026	5.9	1.4×10^{-6}	0.28	77.7	21.5	4.84	1.882	1.775	5.685	1.088
111, 113, 139 143, 169, 171	J-489 ^d	Propylene	50% He	1350	13	1.037	5.9	1.5×10^{-6}	0.28	76.9	23.2	5.26	1.904	1.793	5.830	1.114
115, 147, 173	J-491 ^d	Propylene	None	1350	13	1.039	9.3	8.5×10^{-6}		75.6	28.3	6.37	2.004	1.858	7.285	1.061
119, 149, 177	J-649 ^a	Mixed	67% Ar	1250	24	1.042	3.5	6.7×10^{-6}	0.36	74.3			1.939	1.864	3.89	1.156
123, 151, 179	J-651 ^a	Propylene	50% Ar	1425	24	1.031	4.3	4.2×10^{-6}	0.42	75.8			1.903	1.799	5.47	1.111
91, 183	OR-2262 -HT ^e	MAPP	50% Ar	1275	6.3		6.5						2.009	1.905	5.19	

^aIrradiated in HRB-14.

^bIrradiated in HT-34.

^cLess than 2×10^{-6} by R/B measurement.

^dIrradiated in OF-2.

^eReference Biso-coated particle irradiated previously; 507-μm kernel, 98-μm buffer.

Table 2.26 Positions and Properties of Triso-Coated Particles to be Tested in Capsule HRB-15b for Simulating MEU Fissile Fuel at Low, Intermediate, and Reference Fast Fluence at Low Irradiation Temperature (LTIs deposited from propylene diluted with 50% He at 1350°C in 0.13-m coater)

Capsule Position	Batch	Kernel Diameter (μm)	Thickness, μm				oLTI Coating Rate (μm/min)	Defective Fraction		oLTI Gradient Density (Mg/m ³)		oLTI Open Porosity (%)
			Buffer	iLTI	SiC	oLTI		SiC by Hg Intrusion	oLTI by Cl ₂ Leach	Observed	Corrected	
1, 29, 61	A-824	450.8	50.1	24.2	37.8	42.6	3.91	$<1.5 \times 10^{-4}$	2×10^{-4}	1.8322	1.7342	5.351
3, 35, 63	A-825	445.3	54.6	32.5	35.5	32.0	2.54	$<1.4 \times 10^{-4}$	$<2 \times 10^{-4}$	1.8432	1.7636	4.321
7, 37, 65	A-826	445.3	54.6	32.5	32.7	39.4	3.03	$<1.3 \times 10^{-4}$	$<2 \times 10^{-4}$	1.8223	1.7413	4.447
9, 39, 69	A-827	445.3	54.6	32.5	36.5	39.8	3.26	$<1.4 \times 10^{-4}$	$<2 \times 10^{-4}$	1.8179	1.7332	4.660
11, 41, 73	A-828	445.3	54.6	32.5	34.3	41.3	3.13	$<1.4 \times 10^{-4}$	$<2 \times 10^{-4}$	1.8154	1.7371	4.311
13, 45, 75	A-833	450.8	50.1	24.2	37.8	51.9	6.33	$<1.5 \times 10^{-4}$	$<2 \times 10^{-4}$	1.8804	1.7633	6.229
14, 49, 79	A-834 ^a	445.3	54.6	32.5	32.7	43.5	5.30	$<1.3 \times 10^{-4}$	4×10^{-4}	1.8984	1.7546	9.243
19, 53, 83	A-835 ^a	445.3	54.6	32.5	36.5	47.7	5.81	$<1.4 \times 10^{-4}$	8×10^{-4}	1.8832	1.7670	9.383
25, 55, 85	A-836 ^a	445.3	54.6	32.5	34.3	46.6	5.68	$<1.4 \times 10^{-4}$	$<2 \times 10^{-4}$	1.8530	1.7363	9.370
27, 59, 89	A-837 ^a	445.3	54.6	32.5	35.5	49.0	5.98	$<1.4 \times 10^{-4}$	2×10^{-4}	1.8897	1.7706	9.370

^aIrradiated in HT-34.

will be Biso- and Triso-coated fertile kernels. Layers of ORNL fertile particles will be alternated with layers of GA fissile particles. The GA particles will contain sufficient ^{235}U (20% enriched) to maintain the desired fuel centerline temperatures at beginning-of life, and the ORNL fertile particles plus the ^{238}U in the GA particles will compensate for the heat generation required as the ^{235}U burns out.

2.6 IRRADIATION TESTS IN THE ORR — T. N. Tiegs

At the end of the reporting period there were no OF-type capsules in the ORR and none are planned in 1978. A topical report on the OF-1 capsule was published during the reporting period.⁴¹ Postirradiation examination of the OF-2 capsule was completed during the reporting period, with the exception of the IMGA analysis, which will be completed during 1978. The OF-2 has been thoroughly documented in previous progress reports^{42,43} and in a preirradiation report.³⁹ A topical report⁴⁴ covering operation and postirradiation examination was completed during the reporting period. An abstract of this report is given below:

Irradiation capsule OF-2 was a test of High-Temperature Gas-Cooled Reactor fuel types under accelerated irradiation conditions in the Oak Ridge Research Reactor. The results showed good irradiation performance of Triso-coated weak-acid-resin fissile particles and Biso-coated fertile particles, which were coated in the 0.13-m-diam furnace with a fritted gas distributor. Matrix-particle interaction involving Triso particles was shown to depend on the optical anisotropy, density, and open porosity of the outer pyrocarbon coating and also on the coke yield of the matrix. An increase in these quantities outside prescribed limits showed increased matrix-particle interaction. Good irradiation performance was observed with weak-acid-resin particles converted in the range from 15 to 75%.

2.7 CAPSULE THERMOMETRY — M. J. Kania

High-temperature thermocouple systems with improved accuracy, reliability, and chemical compatibility are being developed for use in instrumented accelerated irradiation capsules. These capsules are used for HTGR fuel qualification and graphite creep tests. Fuel irradiation experiments require thermocouples for fuel centerline temperature monitoring. They must operate at temperatures near 1500°C for extended times in a high neutron flux and must be compatible with current HTGR fuel designs. The creep tests of the

OC series, conducted in the ORR, require thermocouples to operate in the temperature range from 600 to 1250°C with an accuracy of 1%. These experiments are also conducted under accelerated conditions. These thermocouples must be compatible with HTGR graphites.

During this report period continued out-of-reactor thermometry development has identified specific platinum-base alloys as the most promising candidates for thermal elements, and molybdenum with a protective ZrC coating as the most promising sheath material. Full evaluation of the sheath coating as well as evaluation of candidate insulator materials are continuing in accelerated out-of-reactor tests. Postirradiation examination of the center-line Johnson Noise-Power Thermometer of HRB-12 has been completed and revealed no evidence of incompatibility between materials in the thermocouple system, nor between assembly and fuel rods. Response times for the Chromel vs Alumel thermocouples in HRB-13 were measured. They show effects of contact between sheath and graphite, sweep-gas composition, temperature, and irradiation. The investigation of the influence of transmutation alloying in tungsten-rhenium thermocouples is continuing and data analysis is in progress.

2.7.1 Thermometry Development — R. K. Williams, E. L. Long, Jr. and D. L. McElroy

The purpose of this work is to develop for irradiation capsules improved thermometry systems that are compatible with HTGR fuels and graphites during long periods of intense neutron exposure and high temperatures. The approach taken is to evaluate thermocouple components — thermoelements, insulators, and sheaths — in systematic out-of-reactor tests to select candidate materials that are compatible with each other as well as the environment in which they must operate.

Previous thermoelement drift tests⁴⁵ at 1400°C for 1000 hr were conducted to develop improved platinum-base thermoelements and to define constraints that limit their performance. These tests revealed that the most promising thermoelement was the Pt-5% Mo vs Pt-8% Ru system. An extension of these tests to 1500°C for 100 hr in a graphite and argon environment pointed out the following constraints for low-cross-section platinum thermocouples:

- (1) compatibility of sheath-insulator system in a reducing environment
- and (2) mechanical degradation of the thermoelements due to excessive grain growth.

The high-temperature drift tests were followed by a series of compatibility tests. Small-scale thermocouples were fabricated from candidate insulators and sheath materials. These specimens were then tested in an inert gas-graphite environment for 100 and 200 hr at 1500°C. The samples and their evaluations after exposure are described in Table 2.27. In general the results were not encouraging. In this study the insulating materials were Al_2O_3 , BeO, and MgO. Sheath materials were platinum and molybdenum. Specific conclusions reached are:

1. Platinum sheath materials, containing oxide insulators, performed poorly; that is, they all swelled, and some liquated and lost weight. The swelling was presumably due to solution of the Al, Be, or Mg in the Pt and the weight loss was due to oxygen removal. Swelling of 2–17 % and weight losses of -0.1 to -2.7% were noted. Platinum tubing without insulator or thermoelement showed only slight detectable weight loss and diametrical expansion.

2. Molybdenum tubing without insulator or thermoelement swelled 2.4–3.7% and gained weight of 1.6–3.5%. However, molybdenum tubing with a reaction coating of ZrC showed only a slightly detectable weight gain of 0.2% and negligible diametral expansion after 100 hr at temperature. The latter shows that the carburization of the molybdenum can be effectively retarded by a ZrC coating.

Tests have been initiated to determine the best process for coating the sheath material with ZrC. Candidate processes are chemical vapor deposition (CVD), plasma spraying, and reaction coating. The latter two processes are the most economical and are being examined first. Here, ZrC powder, carbon powder (0.3 vol %) and silicon powder (2 wt % based on ZrC) were ball-milled, and a slurry in hexane was deposited on small-diameter molybdenum tubing, with and without a coating of ZrC applied by plasma spraying. Both the reaction coating and the plasma-sprayed coating are very porous, and it was thought that the reaction coating over the plasma-sprayed coating might reduce overall coating porosity. The tubes were then fired at 1800 to 1850°C in argon for 30 min to make a strong adhering coat.

The results of the metallographic evaluation of the ZrC-coated molybdenum tubes are shown in Fig. 2.34. The ZrC applied by the two methods described above resulted in porous coatings about 100 μm thick. The plasma-sprayed

Table 2.27 Weight Change and Dimensional Data From 1500°C Tests
In Graphite-Argon Environment

Test Specimen	Weight Change, % at		Diameter Change, % at	
	100 hr	200 hr	100 hr	200 hr
Pt tube, both ends open, no insulator or thermocouple	-0.03	-0.03	+0.1	+0.04
Pt-4% Nb wire (1.02 mm diam)	-0.04	-0.04	+0.5	+0.04
Mo tube, both ends open, no insulator or thermocouple	+1.6	+3.5	+2.4	+3.7
Mo tube coated with ZrC at ONRL	+0.22		-0.08	
Nb wire (2.29 mm diam)	+0.5	+0.6	+1.0	+1.5
Hard-fired Al_2O_3 , electron-beam (e.b.) sealed in a Pt tube	-0.9	Liquated	+1.8	Liquated
Hard-fired BeO, e.b. sealed in a Pt tube	-0.2	Liquated	+17.4	Liquated
70%-dense MgO, e.b. sealed in a Pt tube	-1.9	-2.2	+11.6	+11.6
NbC powder, e.b. sealed in a Pt tube	-0.1	-0.1	+8.3	+8.0
Hard-fired Al_2O_3 , coated with NbC by Y-12 and sealed in a Pt tube	-0.1	-0.2	+7.7	+7.4
Hard-fired Al_2O_3 , reaction coated with NbC by ORNL and e.b. sealed in a Pt tube	-0.3	-0.5	+6.7	+6.7
Hard-fired Al_2O_3 , reaction coated with ZrC by ORNL and e.b. sealed in a Pt tube	-0.6	Liquated (-2.7)	+4.1	Liquated
Hard-fired BeO, coated with NbC by Y-12 and e.b. sealed in a Pt tube	-0.6	Liquated	+8.5	Liquated
Hard-fired Al_2O_3 , e.b. sealed in a Pt tube. Tube plasma spray coated ($\sim 50 \mu\text{m}$) with NbC	-0.7	-0.5	+6.6	+5.9

Fig. 2.34 Typical Regions from Molybdenum Sheaths with ZrC Layers on the Outside Surfaces. Layers were applied by (a) reaction coating (100 $\times$) and (b) plasma spraying followed by reaction coating (200 $\times$).

plus reaction coating was less porous and more uniform in thickness than the reaction coating. The porosity of the coatings would lead one to suspect that an effective barrier that would prevent interaction with a fuel rod and carburization had not been achieved. However, the results of the compatibility tests listed in Table 2.27 indicate that the ZrC reaction coating was the best of the series and may be satisfactory to prevent reaction with pyrocarbon coatings. Similar coating evaluations using all three processes are still in progress and will be reported when completed.

These results indicate that platinum sheath and oxide insulators are not compatible. The only possible candidate system from the data in Table 2.27 is the molybdenum with a ZrC coating to prevent carburization of the molybdenum. Here data only are known at 100 hr at 1500°C, and more data must be obtained before a selection is made. Also, results from the HRB-12 capsule, Sect. 2.7.2, indicate that the ZrC-coated rhenium sheath material is compatible with a fuel-graphite-neutron environment at temperatures near 1350°C for extended periods of time.

Development has also proceeded on doped Pt-5% Mo and Pt-8% Ru thermoelement wires with the object of reducing grain growth. Two dopants, 0.5 wt % CaO and MgO, were considered and added to platinum powder by use of an alcohol solution of the nitrate. The solution was then dried and fired in air at 500°C for the MgO and 650°C for the CaO, to decompose the nitrates and form fine oxide particles. The fine oxide was then blended with the alloying element, 5 wt % Mo or 8 wt % Ru, for 2 hr and cold pressed to about 75% theoretical density. The samples then were dried at 800°C in vacuum, then at 1500°C in an inert atmosphere for 20 hr to alloy powder. Final specimens were hot swaged at 1000 to 1200°C and cold drawn to 0.38-mm-diam wires.

Four doped thermoelements — Pt-8% Ru and Pt-5% Mo with both MgO and CaO dopants — were subjected to a series of characterization tests before and after vacuum annealing in the 1100 to 1600°C range. The thermal emf homogeneity on all four thermoelements after 1 hr at 1100°C was less than $\pm 0.5^\circ\text{C}$. The thermal emf temperature dependence was consistent with undoped controls. Samples vacuum annealed for 24 hr at 1325, 1450, and 1600°C showed that the dopants effectively inhibited grain growth at 1600°C. Electrical resistance changes in the Pt-Mo alloy suggest ordering may be

occurring, and the room-temperature total elongations exceed those of the undoped controls and are near 10%, except for the doped Pt-5% Mo exposed at 1325°C. Based on these results, CaO doping was selected as slightly more favorable than MgO, and a larger stock of CaO-doped Pt-Mo and Pt-Ru wire was prepared. This will allow 1450°C stabilization to be studied as a means to avoid the low elongation noted at 1325°C.

2.7.2 Thermometry in HRB Capsules — R. L. Shepard, R. M. Carroll, T. N. Tiegs, and E. L. Long, Jr.

The irradiation performance of thermocouples used in the HRB-12 and -13 irradiation capsules is summarized below.

2.7.2.1 HRB-12

The HRB-12 irradiation capsule contained a dual-element, 18- Ω rhenium resistor Johnson noise power thermometer (JNT), insulated with BeO and enclosed in a 3.33-mm-OD ZrC-coated rhenium sheath. During operation of the capsule, both resistance elements failed open circuit after 3780 hr of irradiation. The noise temperature signals obtained before failure were plagued by vibration-induced noise, which was subsequently identified with the loosely fitting, hard-fired insulators used in the construction of the sensor.

Postirradiation examination of the capsule showed the ZrC-coated rhenium sheath of the JNT to have survived the 6672-hr, 1350°C irradiation without visible degradation. The sheath had, however, become brittle during irradiation and was recovered in three sections during capsule disassembly. One portion of the sheath about 100 mm from the closed end contained two small longitudinal cracks. Portions of the 80- μ m-diam rhenium wire resistor coils were still attached to the 0.4-mm-diam lead wires; thus, the open-circuit failures of the resistors did not occur at the welds.

Two fuel rods, 4 and 9, were stuck on the rhenium sheath. After removal of the two fuel rods, the dimensions of the sheath were measured in two places corresponding to the locations of fuel rods 4 and 9. The outer diameter of the sheath was 3.213 to 3.271 mm near the tip (fuel rod 9 location) and 3.119 mm at 100 mm from the tip (fuel rod 4 location), neither significantly different from the preirradiation measurements of 3.277 mm max OD. Fuel rods 4 and 9 inner diameters were checked with ground pins of 3.327 and 3.378 mm OD.

Fuel rod 4 allowed the 3.378-mm pin to pass through its central hole; fuel rod 9 did not but would pass a 3.327-mm pin. Preirradiation tests showed all rods passing 3.480-mm pins. Clearly, the fuel rods shrank about 3%, as shown by these tests; therefore, the inner diameter was reduced to very nearly the maximum diameter of the sheath.

A transverse section through the ZrC-coated rhenium sheath and BeO insulator was taken about 30 mm from the tip of the assembly and examined metallographically. Examination of the section showed that, although the ZrC coating remained firmly attached to the rhenium sheath, both the coating and sheath contained numerous fine cracks (Fig. 2.35). The radial cracks in the ZrC were brittle. The cracks in the rhenium sheath resulted in grain-boundary separation, which was caused by the collection of bubbles on grain interfaces. The rhenium sheath was large grained with single grains spanning the entire wall thickness. The cracks in the ZrC coating probably occurred as a result of the grain-boundary separation of the sheath. At present the source of bubbles is not known, but similar results were noted⁴⁶ during the examination of the rhenium ultrasonic thermometers in HRB-5 and -6.

No evidence of incompatibility between any of the materials in the thermometer assembly, nor between the assembly and the fuel rods, was observed.

2.7.2.2 HRB-13

The response times of eight sheathed Chromel vs Alumel thermocouples installed in the peripheral graphite sleeve of the HRB-13 irradiation capsule were measured during fabrication and then during irradiation of the capsule. A method of loop current step response (LCSR) was used; it permits in situ time response measurements. These measurements were made to show (1) the effects of contact between the thermocouple sheath and the inner surface of the graphite holes, (2) variations of capsule sweep gas composition, (3) capsule temperature, and (4) irradiation on the measured response time of the thermocouples. Differences in thermal contact between thermocouple sheath and the graphite affect the time response of the thermocouple and affect the accuracy of the thermocouple by altering the dissipation of gamma and neutron heating of the thermocouple. The LCSR method is also

Fig. 2.35. Transverse Section Through the ZrC-Coated Rhenium Thermometer Sheath from HRB-12. (a) ZrC, rhenium sheath, and BeO Insulator, 100 $\times$. (b) Intergranular separation of the rhenium sheath was the result of accumulation of bubbles along grain boundaries. Swelling of the rhenium caused cracks to develop in the ZrC. 250 $\times$.

intended to provide information on the electrical condition of the thermocouple and to identify causes of thermocouple failure.

2.7.2.2.1 Experiment Conditions. The HRB-13 capsule contained eight stainless-steel-sheathed, insulated-junction, Chromel vs Alumel thermocouples, 1.6 mm OD. These thermocouples were inserted in 2.0-mm holes drilled in a graphite sleeve surrounding the fuel specimens. The sheathed portion of the thermocouples was about 5 m long and was connected to about 6 m of fiber-glass-insulated extension wire within the capsule. A multipin connector with Chromel and Alumel pins and sockets was used to connect the capsule to the poolside junction box. About 20 m of Chromel-Alumel extension wire led from the poolside to the experiment station where the LCSR test readings were made.

In the LCSR measurement, a high-frequency (1-kHz) current of about 1 A was passed through the thermocouple circuit to heat it resistively about 2°C. The current was then cut off and the thermocouple signal was recorded as the thermocouple returned to thermal equilibrium with its surroundings. The time constant of the thermocouple was obtained from computer analysis of this exponentially decaying transient signal.⁴⁷

2.7.2.2.2 LCSR Tests Before Irradiation. The response times of the eight thermocouples were measured at each stage of capsule fabrication. Initially, with the thermocouple measuring junctions immersed in stirred water, the response times ranged from 393 to 618 ms with an average time constant of 485 ms (Fig. 2.36). Measured next, with the thermocouples inserted in the graphite sleeve, the response times ranged from 2.98 to 5.74 s with an average of 4.39 s, an increase of nearly 4 s due to the poor thermal contact between the thermocouple sheaths and the graphite sleeve.

After the capsule was completely assembled, the response times of the thermocouples were measured at poolside with neon gas in the capsule and then with helium gas (Fig. 2.36). The response times of two thermocouples — Nos. 2 and 3 — decreased by a factor of 3.2 when the gas was changed to helium. Helium has a thermal conductivity 3.2 times that of neon. The other thermocouples showed a smaller decrease, ranging from a factor of

Fig. 2.36 LCSR Time Response of Thermocouples in HRB-13 Irradiation Capsule, Showing Effects of Capsule Sweep Gas.

1.7 to 2.5, and all thermocouples showed a time constant between 0.6 and 1.2 s. These results show that thermocouples 2 and 3 are in poor physical contact with the graphite sleeve and the others — particularly 1, 4, and 6 — are in relatively good physical contact. Larger gamma heating errors should be found in thermocouples 2 and 3.

The capsule was then installed in the reactor, and the response times were measured in core with helium sweep gas before the reactor was brought to power. Slight differences in response times were observed and are attributed to movement of the thermocouples during handling and in changing from a horizontal position (in the previous measurements) to a vertical position.

2.7.2.2.3 LCSR Tests During Irradiation. The reactor was operated initially at 75 MW power with helium sweep gas in the capsule. LCSR measurements were made after about 100 hr of irradiation with the thermocouples reading temperatures of 550 to 650°C. Time constants were slightly lower than those observed at zero power and ambient temperatures because of increased helium gas conductivity and the greater contribution of radiant heat transfer between sheaths and graphite. The next LCSR tests were made six months later after 3340 hr of irradiation with the reactor at 100 MW power and with sweep gas about 25% Ne and 75% He. Thermocouple temperatures ranged from 730 to 850°C. The thermocouples had accumulated a neutron fluence of about 2.5×10^{25} n/m². Time constants of seven thermocouples were moderately higher, one thermocouple (No. 6) had increased a factor of 4, and all thermocouples apparently showed a larger increase in temperature resulting from the resistive heating. A month later, similar measurements indicated that the thermocouples were heated more than 300°C by the loop current, which is an impossible result. Measurements made at zero reactor power after 4090 hr irradiation with helium and with mixed He-Ne sweep gas showed the thermocouples to be insensitive to sweep gas composition, contrary to previous results. Time constants of six of the eight thermocouples were not significantly different from previous ambient temperature measurements, temperature indications of all thermocouples were credible, and loop resistance measurements of all thermocouples gave results near those previously measured. Measurements of time response by the LCSR method later in the course of the 7800-hr irradiation continued to show anomalous results. In some cases, the observed signal indicated a heating transient when the loop current was turned off. These anomalous results are believed to be due to the irradiation of the thermocouple insulators or to the presence of moisture in the thermocouples. Electrical insulation between the thermocouples loops and sheaths dropped from 850 kΩ initially to 3 to 80 kΩ near the end of the test. Bench tests of similar thermocouples were begun to analyze this behavior.

2.7.2.2.4 Conclusions. The LCSR method provides empirical evidence for variations in thermal contact between thermocouple sheaths and the surrounding graphite. The test results show dramatically the effects of changing sweep gas composition and increased radiant heat transfer at higher temperatures.

Test results beyond about 3000 hr irradiation time show effects of degradation of the thermocouple insulation by moisture or irradiation. No corresponding errors in the steady-state temperature indications of the thermocouples were found. Additional diagnostic tests are needed to explain the anomalies found.

2.7.3 Influence of Transmutation Alloying in W-Re Thermocouples — M. J. Kania,

The investigation of the influence of transmutation alloying in tungsten-rhenium thermocouples is continuing. Various tungsten-rhenium thermoelement wires with differing proportions of W, Re, and Os to simulate transmutation effects in a neutron environment have been fabricated. A full analysis of the temperature effect due to the alloying has not been completed and is presently under way.

2.8 NUCLEAR ANALYSIS IN SUPPORT OF IRRADIATIONS — J. F. Mincey

Irradiation support work during this reporting period was primarily concentrated in four areas: analysis of the OF-2 experiment's neutron flux dosimeters, analysis of the OC-1 experiment's neutron flux dosimeters, preparation of OC-2's dosimeters, and a design study of a proposed simulated HRB capsule for measuring nuclear heating rates in various materials in the HFIR RB and VXF facilities. A synopsis of each area follows.

2.8.1 OF-2 Neutronics Analysis

Experimentally derived one-energy group ($E \geq 5 \times 10^{-4}$ eV) total neutron flux magnitude profiles for the various locations shown in Fig. 2.37 are given by the equations below. Each equation number corresponds to the aximuthal location shown in Fig. 2.37; Z denotes the axial coordinate of the OF-2 capsule, the ORR horizontal midplane is taken to be $Z = 0$, and the positive direction is towards the top of the ORR.

$$\phi_T = 6.439 \times 10^{18} (1 - 1.256Z - 8.509Z^2 + 14.31Z^3) , \quad (1)$$

$$\phi_T = 6.739 \times 10^{18} (1 - 1.182Z - 8.493Z^2 + 12.98Z^3) , \quad (2)$$

$$\phi_T = 7.681 \times 10^{18} (1 - 1.537Z - 9.175Z^2 + 15.82Z^3) , \quad (3)$$

$$\phi_T = 6.486 \times 10^{18} (1 - 0.6062Z - 8.333Z^2) , \quad (4)$$

$$\phi_T = 6.618 \times 10^{18} (1 - 0.6065Z - 7.607Z^2) , \quad (5)$$

where Z is in meters and ϕ_T is in $n/m^2 s$. These correlations are based on $^{59}\text{Co}(n,\gamma)^{60}\text{Co}$ and $^{58}\text{Fe}(n,\gamma)^{59}\text{Fe}$ activation data and reflect 100% reactor power flux magnitudes averaged over the various core loadings used during the OF-2 experiment. To estimate the average flux magnitude (including both core loading effects and non-100% reactor power operation) multiply the equations by 0.865.

Fig. 2.37. Azimuthal Dosimeter Locations for the OF-2 and OC-1 Experiments.

Figures 2.38 through 2.42 compare plots of these five equations with data⁴⁸ based on a previous experiment in the ORR E-7 position with the following reactor core conditions: a cold, clean core with about 10% lighter core loading compared with the average OF-2 core loading and a much shorter irradiation time of about 10 hr. Hence the expected and observed result is that the average OF-2 flux magnitude profiles are somewhat lower and flatter than the ref. 48 curve. Attention should be given to the fact that these correlations are based on data largely limited to portions of

Fig. 2.38 Total Flux Magnitudes in Location 1 of OF-2.

Fig. 2.39 Total Flux Magnitudes in Location 2 of OF-2.

Fig. 2.40 Total Flux Magnitudes in Location 3 of OF-2.

Fig. 2.41 Total Flux Magnitudes in Location 4 of OF-2.

Fig. 2.42. Total Flux Magnitudes in Location 5 of OF-2.

the capsule near and above the ORR horizontal midplane, as no dosimeters were placed in the lower parts of the capsule (with the exception of location 1, see Fig. 2.38). Hence, statistical techniques were relied upon in generating the correlations. While the first and third correlations are believed to be reliable (Figs. 2.38 and 2.40), the remaining correlations are probably underestimations of the true flux magnitude profiles. These three correlations may be as much as 15% low.

Table 2.28 lists total ($\geq 5 \times 10^{-4}$ eV) cross sections and fractions of the total flux magnitude above 0.183 MeV produced by weighting and collapsing multigroup cross-section data. Two approaches were used in producing the neutron-energy flux spectra used for collapsing multigroup cross-section data into total cross-section form. The first method led to a set of spectra that reflected the increased thermalization of the energy spectrum towards the ends of the OF-2 capsule (which is a consequence of the layer of water above and below the ORR core). While this set of spectra treated gross core effects, capsule fuel loading effects were not

Table 2.28 Averaged OF-2 Neutronics Parameters

Parameter	Value for Each Specimen, 10^{-26}m^2							
	1A	2A	3A	1B	2B	1C	2C	3C
1 $^{232}\text{Th}(n,f)$	$0.018 \pm .001$	$0.018 \pm .001$	$0.017 \pm .001$	$0.017 \pm .001$	$0.017 \pm .001$	$0.017 \pm .001$	$0.017 \pm .001$	$0.018 \pm .001$
2 $^{234}\text{Pa}(n,f)$	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3 $^{233}\text{U}(n,f)$	$107. \pm 8.$	$103. \pm 2.$	$104. \pm 3.$	$105. \pm 4.$	$106. \pm 5.$	$106. \pm 5.$	$105. \pm 4.$	$109. \pm 6.$
4 $^{235}\text{U}(n,f)$	$101. \pm 6.$	$97. \pm 6.$	$98. \pm 7.$	$100. \pm 8.$	$100. \pm 9.$	$100. \pm 8.$	$100. \pm 8.$	$103. \pm 4.$
5 $^{235}\text{U}(n,f)$	$0.077 \pm .005$	$0.075 \pm .004$	$0.074 \pm .003$	$0.073 \pm .002$	$0.073 \pm .002$	$0.073 \pm .002$	$0.074 \pm .003$	$0.076 \pm .004$
6 $^{238}\text{Np}(n,f)$	387.	330.	330.	330.	330.	330.	330.	387.
7 $^{239}\text{Pu}(n,f)$	$185. \pm 14.$	$188. \pm 6.$	$190. \pm 4.$	$191. \pm 3.$	$192. \pm 2.$	$192. \pm 2.$	$192. \pm 2.$	$188. \pm 11.$
8 $^{241}\text{Pu}(n,f)$	$217. \pm 18.$	$215. \pm 2.$	$217. \pm 1.$	$219. \pm 2.$	$220. \pm 3.$	$219. \pm 2.$	$219. \pm 2.$	$220. \pm 15.$
9 $^{232}\text{Th}(n,\text{cap.})$	$2.91 \pm .27$	$2.93 \pm .01$	$2.97 \pm .03$	$3.06 \pm .12$	$3.10 \pm .16$	$3.09 \pm .15$	$3.04 \pm .10$	$2.93 \pm .25$
10 $^{233}\text{Pa}(n,\text{cap.})$	$28.7 \pm .1$	$29.0 \pm .5$	$29.1 \pm .6$	$29.1 \pm .6$	$29.2 \pm .7$	$29.2 \pm .7$	$29.1 \pm .6$	$28.8 \pm .2$
11 $^{234}\text{Pa}(n,\text{cap.})$	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
12 $^{233}\text{U}(n,\text{cap.})$	11.4 ± 1.1	$11.1 \pm .3$	$11.2 \pm .2$	$11.3 \pm .1$	$11.4 \pm .1$	$11.4 \pm .1$	$11.3 \pm .1$	11.5 ± 1.0
13 $^{234}\text{U}(n,\text{cap.})$	31.7 ± 1.2	30.7 ± 1.0	30.9 ± 1.2	31.1 ± 1.4	31.2 ± 1.5	31.2 ± 1.5	31.1 ± 1.4	$32.0 \pm .9$
14 $^{235}\text{U}(n,\text{cap.})$	19.9 ± 1.3	$19.3 \pm .8$	19.5 ± 1.0	19.7 ± 1.2	19.8 ± 1.3	19.8 ± 1.3	19.7 ± 1.2	20.2 ± 1.0
15 $^{235}\text{U}(n,\text{cap.})$	$9.29 \pm .03$	$9.13 \pm .24$	$9.14 \pm .25$	$9.16 \pm .27$	$9.17 \pm .28$	$9.17 \pm .28$	$9.16 \pm .27$	$9.31 \pm .01$
16 $^{237}\text{U}(n,\text{cap.})$	73.8	62.9	62.9	62.9	62.9	62.9	62.9	73.8
17 $^{238}\text{U}(n,\text{cap.})$	$7.63 \pm .14$	$7.27 \pm .38$	$7.29 \pm .40$	$7.32 \pm .43$	$7.34 \pm .45$	$7.34 \pm .45$	$7.32 \pm .43$	$7.66 \pm .11$
18 $^{237}\text{Np}(n,\text{cap.})$	51.7 ± 2.2	$51.0 \pm .8$	51.3 ± 1.1	51.7 ± 1.5	51.8 ± 1.6	51.9 ± 1.7	51.7 ± 1.5	52.2 ± 2.0
19 $^{238}\text{Np}(n,\text{cap.})$	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
20 $^{239}\text{Np}(n,\text{cap.})$	$21.4 \pm .6$	$21.8 \pm .2$	$21.9 \pm .1$	$22.0 \pm .1$	$22.1 \pm .1$	$22.1 \pm .1$	$22.0 \pm .1$	$21.6 \pm .4$
21 $^{238}\text{Pu}(n,\text{cap.})$	$75.5 \pm .8$	$82.0 \pm .8$	$84.1 \pm .8$	$86.1 \pm .9$	$87.2 \pm .9$	$87.2 \pm .9$	$86.1 \pm .8$	$78.6 \pm .8$
22 $^{239}\text{Pu}(n,\text{cap.})$	92.7 ± 6.7	96.2 ± 5.8	96.9 ± 5.1	97.6 ± 4.4	97.9 ± 4.1	98.0 ± 4.0	97.6 ± 4.4	93.8 ± 5.6
23 $^{240}\text{Pu}(n,\text{cap.})$	$274.9 \pm .9$	$275. \pm 5.$	275.7 ± 5.7	276.2 ± 6.2	276.5 ± 6.5	276.6 ± 6.6	276.3 ± 6.3	276.1 ± 2.1
24 $^{241}\text{Pu}(n,\text{cap.})$	73.1 ± 6.0	$72.5 \pm .6$	$73.2 \pm .2$	74.0 ± 1.0	74.3 ± 1.3	74.4 ± 1.4	74.0 ± 1.0	74.2 ± 4.9
25 $^{242}\text{Pu}(n,\text{cap.})$	$33.6 \pm .1$	$33.5 \pm .4$	$33.5 \pm .4$	$33.5 \pm .4$	$33.5 \pm .4$	$33.6 \pm .5$	$33.5 \pm .4$	$33.7 \pm .4$
26 $^{85}\text{mKr}(n,\text{cap.})$	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
27 $^{87}\text{Kr}(n,\text{cap.})$	96.5 ± 1.0	104.3 ± 1.0	106.9 ± 1.0	109.4 ± 1.0	110.6 ± 1.1	110.7 ± 1.1	109.4 ± 1.0	100.1 ± 1.0
28 $^{88}\text{Kr}(n,\text{cap.})$	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
29 $^{133}\text{Xe}(n,\text{cap.})$	$30.9 \pm .3$	$33.3 \pm .3$	$34.0 \pm .3$	$34.8 \pm .3$	$35.1 \pm .3$	$35.2 \pm .4$	$34.8 \pm .3$	$32.0 \pm .3$
30 $^{135}\text{Xe}(n,\text{cap.})$	$467,000 \pm 10\%$	$505,000 \pm 10\%$	$517,000 \pm 10\%$	$529,000 \pm 10\%$	$535,000 \pm 10\%$	$535,000 \pm 10\%$	$529,000 \pm 10\%$	$485,000 \pm 10\%$
31 Graphite damage cross sections (use with total flux)	$291. \pm 13.$	$290. \pm 4.$	$289. \pm 2.$	$287. \pm 1.$	$286. \pm 1.$	$286. \pm 1.$	$287. \pm 1.$	$289. \pm 11.$
32 Graphite damage flux fractions (fraction of total flux above 0.183 Mev)	$0.362. \pm .016$	$0.359 \pm .005$	$0.357 \pm .003$	$0.355 \pm .001$	$0.354 \pm .001$	$0.354 \pm .001$	$0.355 \pm .001$	$0.358 \pm .011$

treated, so another set of spectra was generated. This set was produced by calculating a spectrum for each different vertical fuel loading layer in the capsule. As the fuel loadings were larger on the extremities of the capsule, the spectra tend to be more thermalized towards the vertical midpoint of the capsule. Hence, the two methods used to produce neutron energy spectra tend to act in opposition to each other. Consequently an average was taken between the two sets of total cross sections produced with the two spectra sets. The tolerance associated with each value reflects the spread between these two sets of parameters, not necessarily the error.

2.8.2 OC-1 Neutronics Analysis

Experimentally derived neutron flux magnitude profiles for the various locations shown in Fig. 2.37 are given by the following pairs of equations. Each pair number corresponds to the azimuthal position number it matches: the vertical coordinate system duplicates that of OF-2. Each pair of equations consists of: a correlation for the total (or one) energy group ($E \geq 5 \times 10^{-4}$ eV) flux magnitude, ϕ_T ; and a correlation for the fast ($E \geq 0.183$ MeV) flux magnitude profile, ϕ_F . The ϕ_T correlations are based on the $^{58}\text{Fe}(n,\gamma)^{59}\text{Fe}$ and $^{59}\text{Co}(n,\gamma)^{60}\text{Co}$ reactions; the ϕ_F correlations are based on the $^{54}\text{Fe}(n,p)^{54}\text{Mn}$ reaction.

$$\begin{aligned}\phi_T &= 1.017 \times 10^{19} (1 - 1.860Z - 9.070Z^2 + 22.19Z^3) , \\ \phi_F &= 2.261 \times 10^{18} (1 - 2.208Z - 7.133Z^2 + 33.82Z^3) ,\end{aligned}\tag{1}$$

$$\begin{aligned}\phi_T &= 9.918 \times 10^{18} (1 - 1.605Z - 8.243Z^2 + 15.32Z^3) , \\ \phi_F &= 2.245 \times 10^{18} (1 - 1.347Z - 7.040Z^2 + 8.552Z^3) ,\end{aligned}\tag{2}$$

$$\begin{aligned}\phi_T &= 9.543 \times 10^{18} (1 - 1.671Z - 7.024Z^2 + 12.00Z^3) , \\ \phi_F &= 1.930 \times 10^{18} (1 - 3.096Z - 7.153Z^2 + 30.70Z^3) ,\end{aligned}\tag{3}$$

$$\begin{aligned}\phi_T &= 1.086 \times 10^{19} (1 - 2.215Z - 8.332Z^2 + 23.77Z^3) , \\ \phi_F &= 2.324 \times 10^{18} (1 - 1.219Z - 7.892Z^2 + 8.228Z^3) ,\end{aligned}\tag{4}$$

where Z is in meters, and ϕ_T and ϕ_F are in $\text{n/m}^2 \text{ s}$.

These correlations reflect 100% reactor power conditions averaged over the various core loading patterns used in conjunction with the OF-2 experiment. To estimate the average flux magnitudes (to include the effect of non-100% reactor power operation), multiply the correlations by 0.930. The graphite damage cross sections recommended for use with the total, ϕ_T , and fast, ϕ_F , flux magnitudes are respectively 207 and $809 \times 10^{-28} \text{ m}^2$. An error band of $\pm 15\%$ should be used with the OC-1 flux magnitude profile correlations and graphite damage cross sections. The flux magnitude correlations are compared with ref. 48 data in Figs. 2.43 through 2.46.

2.8.3 Preparation of the OC-2 Dosimeters

Dosimeters whose irradiation-induced activities will be used as a data base for the OC-2 neutronics analysis were loaded in the OC-2 capsule. Steps were taken to ensure better identification of dosimeter package locations after the disassembly of the capsule. This action resulted from difficulties encountered in identifying OF-2 and OC-1 dosimeter packages with their capsule locations after the capsules were disassembled.

2.8.4 Design Study of the MONUHRIH Experiment

The MONUHRIH (Measurement of Nuclear Heating Rates in the HFIR) design study had the following objectives:

1. establish a tentative design for a capsule housing instrumentation for determining typical nuclear heating rates in HRB capsule structural materials for both the HFIR RB and VXF facilities,
2. identify tentative problem areas arising from the proposed capsule design and explore solutions.

The tentative capsule design is presented in Figs. 2.47 through 2.49. The design consists of four calorimeters (Fig. 2.47) with type K thermocouples embedded at the three locations (shown in Fig. 2.48) to provide differential temperatures for calculating heat generation rates in the calorimeter materials. After the thermocouples are installed as shown, the capsule is assembled by welding the ascending sections together.

Several major areas were identified as potentially detrimental to the capsule design. The technique to be employed in determining nuclear heating rates relies heavily upon an accurate knowledge of a calorimeter material's

Fig. 2.43 Flux Magnitudes in the Dosimeter 1 Column of OC-1.

Fig. 2.44 Flux Magnitudes in the Dosimeter 2 Column of OC-1.

ORNL-DWG 78-6603

Fig. 2.45 Flux Magnitudes in the Dosimeter 3 Column of OC-1.

ORNL-DWG 78-6604

Fig. 2.46 Flux Magnitudes in the Dosimeter 4 Column of OC-1.

Fig. 2.47 Prototype MONUHRH Capsule Design.

ORNL-DWG 78-6583

Fig. 2.48 Prototype MONUHRIH calorimeters.

ORNL-DWG 78-6582

Material - Stainless Steel Type 347

Fig. 2.49 Prototype Calorimeter Spacers.

thermal conductivity as a function of temperature. Consequently calorimeters would be machined of very pure copper and aluminum, because these elemental conductivities are well known. However, a series of thermal conductivity measurements would have to be performed on the graphite stock to be used for calorimeter fabrication. A second area of concern was the differential thermal contraction of the graphite and copper calorimeters away from their stainless steel shells. Heat-shrink fitting construction techniques provide a possible solution. The last area identified concerns the decalibration of the type K thermocouples when operated between 200°C and 600°C — temperatures expected to characterize the MONUHRIH capsules in the HFIR RB and VXF (PB) facilities. This source of error might be reduced, but not entirely eliminated. The capsule design presented should be capable of yielding heating rates within 15% of the true value.

2.9 POSTIRRADIATION PERFORMANCE ASSESSMENT

Considerable work was done in the area of performance assessment during the reporting period. Much of this work is associated with characterization of PyC for fission gas permeability and is reported in Sect. 2.3. Work during the reporting period on thermal analysis was concentrated on analyzing specific capsules using existing computational tools. Some development work was done on the HT capsule thermal analysis techniques to deal with both bonded bed and loose particle irradiations. Also, development of the ORR capsule thermal analysis methods centered on automation of the calculational process, to remove many of the hand operations required in the old procedure.

The most significant development in postirradiation performance assessment is the IMGA system. This section describes some of the results obtained with the system, and also the deconsolidation process used to recover individual coated particles from bonded fuel rods. We anticipate that the IMGA system will be the most definitive measurement of the capability of coated particle fuels to retain their fission products available anywhere in the world.

Microprobe work continued routinely during the reporting period, with no new developments associated with either capabilities or procedures.

Some wet chemistry burnup work was done, but we anticipate that the IMGA system will provide a more accurate and less expensive measurement of burnup, by comparing fission product inventories measured in irradiated particles with well established standards. Development of this capability with IMGA is scheduled for 1978.

2.9.1 Thermal Analysis of Irradiation Capsules — M. J. Kania

During the past report period a modified version of the HTCAP computer code⁴⁹ has been completed and applied to calculating operating temperatures of both loose particles and bonded fuel rods in the HFIR target capsules. A revision was necessary because HT capsules contain both loose particle holders and bonded bed specimens. The new calculations include finding the fuel rod centerline and surface temperatures and linear heat rating for the rods. Time-temperature and time-power histories are then generated for each fuel rod. This new version of HTCAP has been successfully applied to the thermal analyses of irradiation capsules HT-29, -30, -31, -32, and -33 during the report period.

The thermal analysis of the OF-2 irradiation capsule is now in its initial stages. At this point we are attempting to use a code developed^{50,51} by students from the MIT Practice School of Chemical Engineering. This is an automated thermal analysis code that will provide a two-dimensional time-temperature history for fuel rods irradiated in a cylindrical capsule arranged in a telephone dial pattern. The code has not been fully adapted to the OF-series of irradiation capsules as yet. We are now debugging it and applying it to the OF-2 irradiation history.

2.9.2 Electron Microprobe Examination of HTGR Fuel Particles — T. N. Tiegs

A report³⁴ was written during this reporting period documenting the results of electron microprobe examinations of HTGR fuel particles made from weak-acid resins. An abstract of the report follows:

Fission product retention is of utmost importance in HTGR fuel particles. The present study concentrates on particles made from weak-acid resins, where there are possible combinations from UO_2 to UC_2 . Five compositions

were tested: $UC_{4.58}O_{2.04}$, $UC_{3.68}O_{0.01}$, $UC_{4.39}O_{1.72}$, $UC_{4.63}O_{0.97}$, and $UC_{4.14}O_{0.53}$. Particles of $UC_{4.58}O_{2.04}$ had unilateral migration of the kernel through the coatings, while $UC_{3.68}O_{0.01}$ had accumulation of rare earth fission products on the cold side of the particle and subsequent coating failures. Combinations of UO_2 and UC_2 showed improved performance and suffered from neither the kernel migration or rare earth accumulation problems.

2.9.3 Irradiated Microsphere Gamma Analyzer System — M. J. Kania and K. H. Valentine

The primary function of the Irradiated Microsphere Gamma Analyzer (IMGA) system^{52, 53} is to determine the failure fraction of irradiated fuel particles — that is, the fraction of fuel particles that have lost a significant amount of their fission products through defective or broken coatings. Basically IMGA consists of a high-resolution gamma-ray detector, a minicomputer-based pulse-height analyzer, and an automated particle handler. These three components and their communication interfacing have been integrated into a sophisticated and versatile system and demonstrated to be efficient and reliable.

2.9.3.1 Failure Fraction Measurements

Accurate measurement of the failure fraction of irradiated coated-particle fuel is vital to the HTGR fuel development program. Because of the importance of establishing accurate and reliable fuel performance data, many techniques are presently being used. Among those most widely used are:

- visual observation on loose irradiated particles and polished metallographic cross sections of irradiated fuel rods;
- ^{85}mKr fission gas release-to-birth rate (R/B) ratio measurements;
- hot gaseous chlorine leach measurements;
- gamma spectroscopy on individual or small numbers of coated particles.

Unfortunately the technique that is most widely used to determine failure fraction is also the one that is very expensive and has the least statistical significance. This technique is visual observation on loose particles or polished metallographic cross sections of irradiated fuel. Statistical

considerations⁵³ have shown that in order to satisfy fuel product specifications, requiring very low failure fractions, less than 1%, with a high degree of confidence, 95%, in the failure fraction measurement requires examination of several hundred to several thousand particles. The expense involved when trying to accomplish this task through metallographic techniques would be prohibitive. However, with the automated particle handler of the IMGA system this is a relatively simple task. Particle examination rates with the IMGA system are in the range of 1 to 10 particles per minute, which means that the failure fraction for a sample of particles can be estimated in a matter of hours.

The failure fraction determination is made possible by the different volatilities of various fission and activation products. Two isotopes of cesium, ^{134}Cs and ^{137}Cs , and ^{95}Zr are of particular interest; all three emit easily detectable gamma rays in the 600–800 keV range. In the high-temperature environment of the HTGR, cesium with a boiling point of 678°C will diffuse or escape much more readily from a defective coating than zirconium with a boiling point of 4377°C . The diffusion rate of the activation product ^{134}Cs is on the same order as the fission product ^{137}Cs . Thus, a measurement of the ratio of ^{134}Cs activity to ^{95}Zr activity provides a direct measure of a particle's ability to retain fission products. As indicated, ^{134}Cs is an activation product rather than a fission product. It is produced by the direct reaction $^{133}\text{Cs}(n,\gamma)^{134}\text{Cs}$. Cesium-133, the stable isotope of cesium, is produced in the following manner:

The direct yield of ^{133}Cs is quite small; however, its cumulative yield is about 6.6% (numbers underlined in the diagram decay chains are cumulative yields given in %). The yields above are for ^{235}U fission and do not differ significantly for ^{233}U fission. Virtually all the ^{133}Cs inventory is a result of decay from the fission gas ^{133}Xe . Thus, ^{134}Cs activity depends on the amount of fission gas ^{133}Xe retained in particle.

Previous irradiation experiments^{15,22} have shown that the permeability of the outer pyrolytic carbon coating in Biso-coated particles is a serious problem. These experiments have shown that "intact" particles examined during routine postirradiation examinations contained little or no fission gas (Kr or Xe). Also monitoring fission gas release-to-birth rate ratios (R/B) has shown that particles released their fission gases, but upon examination were "intact" with no visible defective coatings.

As indicated in the diagram, ^{134}Cs is a direct result of the amount of the fission gas ^{133}Xe that remains in a coated particle. Because ^{133}Xe has a high cumulative yield but a short half-life of about 5.3 days it is gone by the time most postirradiation examinations begin. However, ^{134}Cs has a relatively long half-life of 2.06 years and has been present in all gamma spectra examined with the IMGA system. We feel that the ratio of ^{134}Cs to a stable fission product such as ^{95}Zr in conjunction to the amount of ^{137}Cs retained will provide valuable information regarding fission gas retention characteristics of coated particle fuel. An attractive feature of the ratio determination is its insensitivity to variations in the kernel size and heavy metal loading.

2.9.3.2 IMGA Operation

The IMGA system is now fully operational and being used in the routine examination of irradiated HTGR fuel from experimental capsules. Irradiated coated particles were first introduced into the IMGA cubicle in February 1977. These were in the form of loose particles irradiated in HRB-12. The first particles from a deconsolidated irradiated fuel rod were received into the cell in March 1977. During the past year the system has been evaluated and optimized to improve handler reliability and to provide remote maintenance capabilities for the system. To date, approximately 30,000 irradiated particles have been run through the system. During these runs, each system

operation under computer control has been thoroughly checked. Initial design flaws have been corrected or redesigned.

The operational software for the Tennecomp pulse height analyzer system is written in PDP-11 machine language and was purchased with the system. The interpretive software was developed at ORNL in a language known as TIL (Tennecomp interpretive language), which is a version of FOCAL-11. This software development is complete, and the main examination program is called FAILFRAK. This program controls the entire operation of IMGA. The major functions of the program are to initialize and operate the automated particle handler, control particle spectrum acquisition, analyze spectrum and transfer data to mass storage devices, and segregate particles according to a user-supplied selection criterion. Total spectrum splitting or selected peak splitting is available upon user request. The ability to select individual peaks of interest while ignoring others greatly reduces spectrum analysis time. Two aspects of the automated examination program that further increase operating efficiency of the system are: (1) inert particle detection capabilities that decrease total operating time and (2) the ability to classify and analyze all fuel types during the same run.

The actual failure fraction determination is handled in a separate program, CRUNCH, which uses the data supplied by the examination program. This program analyzes both fissile and fertile data simultaneously while rejecting data from coating fragments of multiple loads (spectra acquired from two or more particles at the same time). Total population histograms of fission and activation product activities as well as ratio functions are created for both fertile and fissile fuel types. Graphic CRT displays and hard copies are available upon user request.

2.9.3.3 IMGA Analysis of OF-2 Fuel

Deconsolidated particles from three OF-2 fuel rods have been examined and the data analysis is nearly complete. The fuel rods examined are described in Table 2.29. These fuel rods were irradiated in Magazine C of OF-2 in the highest flux region and had a peak operating temperature of about 1350°C. The amount of time required for each of the examination runs described in Table 2.29 depended on the counting times for each

Table 2.29 Fuel Rods from OF-2 with the IMGA System.^a

Fuel Rod	Fissile Batch	Fertile Batch	Av. Fluence (n/m ²)	Number of Particles Examined		
				Fissile	Fertile	Inert
C-4-1	A-615	J-489	8.91×10^{25}	1206	2598	10,000
C-3-1	A-611	J-481	8.91	1656	3344	12,000
C-1-2	A-601	J-490	8.50	1726	3050 ^b	14,000

^aComplete description of the OF-2 experiment is in Ref. 39.

^bThe data analysis on fertile particle batch is in process; approximately 3050 particles were examined with the IMGA system.

particle. For rods C-4-1 and -1-2 the counting time (analog-to-digital converter live time) was 60 s, whereas for rod C-3-1 it was 40 s. The total operating time for the IMGA system for the examinations above ranged from 100 to 120 hr of continuous IMGA operation.

For each individual fuel particle, up to 11 gamma peaks were selected from the entire gamma spectrum. The 11 peaks recorded for each data set of rod C-4-1 are shown in Table 2.30. Peaks 1 and 2, which are the K-shell fluorescent x rays from ²³²Th, were used to separate the fissile and fertile particles. The remaining fission products and activation products were used to determine the fission product retention characteristics of the coatings.

The fissile-fertile particle split for fuel rod C-4-1 is shown in Fig. 2.50. Peaks 1 and 2 from Table 2.30 were summed for each particle analyzed, and the range of activities was divided into 50 increments or "channels" (not to be confused with the energy-increment channels of the gamma analyzer). The histogram was then formed by plotting the number of particles in each "channel" against the activity. This figure shows excellent separation between the fissile and fertile particle distributions. We chose to make the split on thorium, rather than uranium, because of the relatively low burnup obtained in fertile particles. The area of the distribution at the far left in the figure represents exactly 1206 fissile particles, and the distribution on far right represents 2598 fertile particles. The separation between the two distributions is 23 of the total 50 channels used in the histogram.

Table 2.30 Selected Peaks from Gamma Spectrum of Individual Particles from Rod C-4-1 during IMGA Examination

Peak	Isotope	Energy (keV)	Standard Deviation ^a (%)	Source
1	^{232}Th	90.0	3.67	K α X-ray excited from ^{232}Th
2	^{232}Th	93.5	2.76	K β X-ray excited from ^{232}Th
3	^{144}Ce	133.5	0.68	Fission Product
4	^{106}Ru	511.8	5.26	Fission Product
5	^{134}Cs	604.7	1.14	Activation Product from Cs(n, γ)
6	^{106}Ru	621.8	11.27	Fission Product
7	^{137}Cs	661.6	1.00	Fission Product
8	^{95}Zr	724.2	2.24	Fission Product
9	^{95}Zr	756.9	2.34	Fission Product
10	^{95}Nb	765.8	0.97	Fission Product, β decay of ^{95}Zr
11	^{134}Cs	795.8	1.20	Activation Product from ^{133}Cs (n, γ)

^aStandard deviations measured on typical fertile particle.

Figure 2.51 compares the ratio of the two fission products $^{137}\text{Cs}/^{95}\text{Zr}$ for the three fissile particle batches discussed in Table 2.29. For batch A-615 the population s (standard deviation) is 6.17% of the mean. If one considers any ratio less than $3s$ of the mean as indicative of fission product loss, then the histogram shows 14 particles with low $^{137}\text{Cs}/^{95}\text{Zr}$ ratios. Batch A-611, with an s of 9.53%, has 18 particles with low $^{137}\text{Cs}/^{95}\text{Zr}$ ratios, and batch A-601, with an s of 4.33%, has two particles with low $^{137}\text{Cs}/^{95}\text{Zr}$ ratios.

Figure 2.52 compares the ratio of the activation product ^{134}Cs to the fission product ^{137}Cs for the same fissile particle batches. Batch A-615 has three particles with low $^{134}\text{Cs}/^{137}\text{Cs}$ ratios, batch A-611 has 12, and batch A-601 has only one. All the particles noted indicate that they lost some of their ^{133}Xe fission gas. Since these are fissile particles,

ORNL-DWG 77-20758

THE HISTOGRAM IS 50 CHANNELS WIDE.

Fig. 2.50 Both Fissile and Fertile Particles Can be Measured and Split During Same Run Because of the Characteristic Fluorescent X Rays Emitted by Thorium.

Fig. 2.51 Fissile Fuel Performance in the OF-2 Experiment Based on Ratio of Fission Products ^{137}Cs to ^{95}Zr .

Fig. 2.52 Ratio of the Activation Product ^{134}Cs to the Fission Product ^{137}Cs for Fissile Particles Irradiated in the OF-2 Experiment.

the low ratios would indicate that failure occurred relatively early in life when the fission density, due to ^{235}U , was the highest.

Figure 2.53 compares the ratio of the two fission products $^{144}\text{Cs}/^{95}\text{Zr}$ for the three fissile particle batches. Batch A-615 has a total of 14 particles with low $^{144}\text{Cs}/^{95}\text{Zr}$ ratios, for batch A-611 all the particles behaved in the same manner, and batch A-601 had two particles with low ratios.

The retention characteristics of the three fissile particle batches are summarized in Table 2.31. These three batches were tested in OF-2 as a proof test before fabrication into fuel rods to be irradiated in the FSVR in the ORNL test elements. Fuel performance requirements for the FSVR were a failure fraction of less than 1% with a 95% confidence level. Figure 2.54 was derived from fuel product specifications for FSVR and represents confidence limits for fuel particle examinations with the IMGA system. The intersection with any point on the curve labeled "95% confidence that failure fraction <0.01 " yields 95% confidence that the failure fraction is less than 0.01 and therefore demonstrates that the product specification has been satisfied. Similarly, the intersection with any point on the line labeled "99.99% confidence that failure fraction >0.01 " yields 99.99% confidence that the failure fraction is greater than 0.01. Data for the three fissile particle types are shown in Fig. 2.54. Batch A-601 clearly has a failure fraction that is less than 0.01. However, for batches A-615 and A-611, the points lie within the shaded region. These points do not satisfy requirements for acceptable performance on the one hand and on the other they do not completely

Table 2.31 Fission Product Retention Characteristics of Fissile Fuel Irradiated in Magazine C of the OF-2 Experiment

Particle Type	Number of Failures Observed		
	$^{137}\text{Cs}/^{95}\text{Zr}$	$^{134}\text{Cs}/^{137}\text{Cs}$	$^{144}\text{Ce}/^{95}\text{Zr}$
Batch A-615 (75% UC_2 , Cone)	14	3	14
Batch A-611 (15% UC_2 , Frit)	18	12	0
Batch A-601 (75% UC_2 , Frit)	2	1	2

Fig. 2.53 Ratio of Fission Products ^{144}Ce to ^{95}Zr for Fissile Particles Irradiated in the OF-2 Experiment.

Fig. 2.54 Performance Assessment Based on IMGA Analysis of OF-2 Fissile Fuel.

satisfy requirements for unacceptable performance. More testing is required before a final decision can be reached.

Two of the three fissile particle types, A-611 and A-601, were used for driver fuel in the ORNL test elements for irradiation in the FSVR. Batch A-615 was rejected on the basis of results of defective fraction measurements by gaseous chlorine leach and fission gas *R/B* measurements from magazine C during the OF-2 irradiation. These decisions were reached much before the IMGA analysis on OF-2 fuel began.

Analyses by IMGA on two fertile particle batches described in Table 2.29 have also been completed. Figure 2.55 compares the ratio of the two fission products $^{137}\text{Cs}/^{95}\text{Zr}$ for the fertile particle batches J-489 and J-481. Figures 2.56 and 2.57 are histograms of the ratios $^{134}\text{Cs}/^{137}\text{Cs}$ and $^{144}\text{Ce}/^{95}\text{Zr}$, respectively, for batches J-489 and J-481. For each of the ratio distributions, the standard deviations are about 2%. An examination of these figures indicates that the majority of the fertile particles performed in the same way as to cesium and cerium retention as well as to fission gas retention.

In Figs. 2.55 through 2.57, for batch J-489 the fission product ratios for several particles are located noticeably to the far right of the major distributions. In the case of the $^{137}\text{Cs}/^{95}\text{Zr}$ ratio, there are seven and for $^{137}\text{Cs}/^{137}\text{Cs}$ and $^{144}\text{Ce}/^{95}\text{Zr}$ there is one each. The question has arisen "Are these ratios representative of particles that have retained their fission products and is the major distribution representative of those not retaining their fission products?" As a check of the measured mean values of the ratios plotted, a set of calculations using the CACA-2 code⁵⁴ were made in conjunction with OF-2 neutronics data. A comparison of the calculated and measured values are shown below:

<u>Ratio</u>	<u>Measured</u>	<u>Calculated</u>
$^{137}\text{Cs}/^{95}\text{Zr}$	0.594	0.589
$^{134}\text{Cs}/^{137}\text{Cs}$	0.883	0.884

These results show excellent agreement between measured and calculated mean ratio values.

Fig. 2.55 Ratio of Fission Products ^{137}Cs to ^{95}Zr for Fertile Particles Irradiated in the OF-2 Experiment.

Fig. 2.56 Ratio of the Activation Product ^{134}Cs to the Fission Product ^{137}Cs for Fertile Particles Irradiated in the OF-2 Experiment.

Fig. 2.57 Ratio of Fission Products ^{144}Ce to ^{95}Zr for Fertile Particles Irradiated in the OF-2 Experiment.

Possible explanations for the high ratio values are: small pieces of matrix adhering to particle surface that were not removed during deconsolidation, and that the matrix contained the volatile cesium but not the stable zirconium; or pieces of coating fragment attached to particle surface. Each of these are possible, as we see pieces of matrix and coating fragments are attached to particle surfaces when particles are examined under the stereomicroscope.

2.9.4 Deconsolidation of HTGR Fuel Rods — T. N. Tiegs

Coinciding with the development of the IMGA system, a technique was developed to obtain undamaged individual particles from HTGR fuel rods. This technique was documented during this reporting period.⁵⁵ An abstract of the report follows:

High-temperature gas-cooled reactor fuel particles are bonded into rods with a carbonaceous matrix. Because of the development of a system to determine fuel failure fractions based on gamma analysis of individual particles, a technique was developed to separate the particles from the matrix after irradiation. Electrolytic deconsolidation at low power levels (2 A at 4 V) yielded undamaged individual particles.

2.10 FISSION PRODUCT-SiC COATING INTERACTION STUDIES — R. L. Pearson and T. B. Lindemer

The main objective of this program is an understanding of the kinetic processes and thermodynamics of the system of fuel, fission product, and coating to a degree that permits identification and quantitative description of failure mechanisms and improved fuel performance by means of kernel chemical modification.

In these studies, in-reactor interactions between fission products and SiC are chemically duplicated by Triso-coated simulated fission product systems. The Triso-coated particles are heat-treated either isothermally or in a temperature gradient, and the reaction products and other phases are studied by metallographic, crystallographic, and microprobe techniques. The experimental results are used in obtaining data on the rate of attack and in the evaluation of possible failure mechanisms.

A report of the interaction of several rare earth carbides with SiC has been published;⁵⁶ an abstract of this report follows:

The reaction rate of either NdC_2 or LaC_2 kernels with SiC in coated particles similar to those used in a High-Temperature Gas-Cooled Reactor (HTGR) is essentially identical with that observed in irradiated, Triso-coated HTGR fuel particles containing UC_2 . The reaction apparently results from destructive distillation of the rare-earth dicarbide in the kernel, transport of rare-earth metal gas down the temperature gradient, formation of the dicarbide in the SiC-pyrocarbon interface, and solution of the SiC in the dicarbide along the SiC grain boundaries. The reaction rate necessary to cause SiC coating failure in four years would apparently be achieved at temperatures above ~ 1565 K. It appears that $\ll 0.003$ of the fissile fuel could fail from this reaction; this compares with a proposed large HTGR design failure fraction of ≈ 0.0039 for the fissile fuel.

Several rare earth oxides were also investigated for interaction with SiC. Neither La_2O_3 , Sm_2O_3 , $\text{Nd}_2\text{O}_3\text{-UO}_2$, nor $\text{Nd}_2\text{O}_3\text{-UO}_2\text{-UC}_2$ reacted with SiC. Triso-coated mixed kernels of $\text{La}_2\text{O}_3\text{-LaC}_2$ did interact with SiC very much like the Triso-coated LaC_2 kernels. These results indicate that rare-earth carbides interact with SiC but rare earth oxides do not.

There is evidence in the HTGR literature that the fission products Pd, Sr, and perhaps Ag interact with SiC coating under certain conditions. Thus, we have begun experimental work, similar to that previously reported for rare-earth carbides and oxides, to investigate the behavior of Pd, Sr, and Ag in Triso-coated kernels. The current interest in low-enriched and medium-enriched fuels, which lead to ^{239}Pu fissions, makes investigation of Pd and Ag behavior even more pertinent since the yield ratios of Pd and Ag for $^{239}\text{Pu}/^{235}\text{U}$ are 9.89 and 50.1, respectively.

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3. PRESTRESSED CONCRETE PRESSURE VESSEL DEVELOPMENT

J. P. Callahan

3.1 INTRODUCTION

The prestressed concrete reactor vessel (PCRV) research and development program consists of generic studies designed to contribute to the technological data base and to the development of optimized designs for the PCRV, prestressing, liner, and penetration closures. The overall program is divided into the following six technical task areas:

1. analysis methods,
2. concrete properties in nuclear environments,
3. prestressing systems,
4. liners and penetrations,
5. instrumentation, and
6. structural models.

Sections 3.2 through 3.5 describe the ongoing activities under each of these task areas. The major activities consisted of an analytical study of a multicavity PCRV designed for an HTGR power plant using an elastic finite-element code, evaluations of an improved concrete constitutive model based on the endochronic model, testing of concrete embedment strain gages, acoustic emission monitoring of a flawed steel pressure vessel and various types of concrete specimens, experimental evaluation of grouted versus nongrouted prestressing systems, and a status report on the fracture toughness testing of liner and forging metallic materials. All these activities were undertaken as a result of needs identified either through discussions with personnel directly involved in the design and development of PCRVs or from critical reviews of published information.

3.2 PCRV ANALYTICAL STUDIES — D. N. Fanning

An essential element of the PCRV program is the development and demonstration of capability for performing accurate structural analyses of PCRVs and related components such as closures. We have conducted a series of analyses both to demonstrate capability to analyze these complex structures and to provide an independent evaluation of the adequacy of a

proposed PCRV design. This analytical study was initiated under the HTGR Safety Program (189a OH019) and has been continued under the HTGR Base Program. The analyses reported herein are the last of the planned series.

The major objective of the PCRV analytical effort is to develop techniques that can be incorporated into existing modern finite-element computer programs to improve the accuracy and efficiency of these codes. The first of these techniques will employ an improved inelastic concrete constitutive model. The model has been developed and is being subjected to verification studies, the first of which is described in Sect. 3.2.2.

3.2.1 Analysis of Multicavity PCRV Elastic Response

The behavior of a PCRV representative of a current multicavity vessel was investigated with elastic analysis methods. Of primary concern was the identification of any areas where the stress in the vessel could exceed the stress allowed by the *ASME Boiler and Pressure Vessel Code*.¹ The PCRV selected for this study is based on a proposed 2000-MW(t) HTGR designed by the General Atomic Company (GA). The analysis of the HTGR was performed with the three-dimensional finite-element computer code STATIC-SAP.² A series of analyses, which were made to provide a measure of verification of the finite-element computer codes used in the study were reported previously.³

3.2.1.1 Description of the PCRV

The wall of the PCRV selected for this study contains six cavities: four for steam generators and two for auxiliary circulators, as shown in Fig. 3.1. The vessel has a height of 27.8 m (91 ft 2 in.) and a diameter of 29.3 m (96 ft). The reactor core cavity is 14.4 m (47 ft 4 in.) in height with a diameter of 10.0 m (32 ft 8 in.). The vessel is prestressed circumferentially by winding tensioned strand around the outside of the vessel. The prestressing is arranged in 24 bands with each consisting of 12 to 14 layers of seven-wire 12.7-mm-diam (0.50-in.) strand. Alternate layers within each band have 53 and 54 strands. The top head, the side wall, and the bottom head of the vessel are prestressed circumferentially by 5, 12, and 7 bands, respectively. The vertical

Fig. 3.1. Vessel Selected for Study. To convert dimensions, 1 ft = 12 in. = 0.3048 m.

prestressing is produced by 336 tendons, each of which comprises 169 6.35-mm (0.25-in.) wires. The operating pressure of the vessel is 4.90 MPa (710 psi), and the maximum cavity pressure (MCP) is 5.35 MPa (776 psi).

3.2.1.2 Description of the Analytical Model

The analytical model of the PCRV for a commercial HTGR is shown in Fig. 3.2. A 30° segment of the vessel is modeled with 213 20-node continuum elements and 1667 nodes. Because the actual vessel has two vertical planes of symmetry, a completely accurate geometric model would consist of a 90° segment of the vessel; however, the error introduced by modeling only 30° of the vessel was considered to be very small. The refueling penetrations in the top slab are not represented geometrically in the analytical model; nevertheless, their effect has been at least partially accounted for by modifying the material properties of the continuum elements in this region.

Thirty-one vertical prestressing tendons are anchored on the modeled 30° segment of the vessel. Eight of them pass through the bottom head into the support structure; the others are anchored at the lower surface of the bottom head. The bearing pads associated with the vertical tendons are too small to model without drastically increasing the number of elements and nodes of the analytical model and, therefore, increasing the cost of the analysis. A preprocessor computer program was used to convert the vertical tendon forces into equivalent nodal loads.

In the analysis the internal pressure was set equal to the maximum cavity pressure of 5.35 MPa (776 psi). This pressure is applied to the inside surfaces of the reactor cavity, the steam generator cavities, and the crossover ducts. The upward thrust resulting from pressure on the steam generator closure plug is simulated by an upward ring load on the top head around the steam generator penetration. The preprocessor that converted the circumferential prestressing into equivalent nodal loads was also used to convert the internal pressure load into equivalent nodal loads.

The dead load consists of several parts. The rebars, vertical prestressing tendons, tendon tubes, and load cells were considered to be

ORNL-DWG 77-3226

Fig. 3.2. Analytical Model for PCRV Designed for a Commercial HTGR.

uniformly distributed in the concrete, resulting in a weight density of 24.54 kN/m^3 (156.3 lb/ft^3) for the composite. The above mixture of concrete, steel, etc. includes 85% of the dead loads. The weight of the circumferential tendons, steel channel liners, and associated load cells is included as a downward surface traction on the outer wall of the vessel. The weight of the control rod drive assembly and the top deck is represented as a pressure on the top head. The steam generator weight is distributed as a downward surface traction on the walls of the steam generator cavity. The steam generator closure plug weight is applied as a ring load on the top slab around the rim of the steam generator cavity. The weight of the reactor core, the core support floor, and the segmented core barrel is applied to the bottom of the reactor cavity. The weight of the liners and cooling tubes is neglected.

Nodal temperatures are required for the effect of the thermal load to be analyzed. A thermal diffusion analysis of a PCRV reported by Saugy et al.⁴ was used to approximate the temperature distribution within the PCRV of the HTGR we are attempting to analyze. The PCRV analyzed by Saugy is for a 1000 MW(e) gas-cooled fast reactor developed by the Swiss Federal Institute for Reactor Research (EIR). The Swiss PCRV differs from the PCRV that we analyzed mainly by having nine cavities in the vessel wall rather than six. Another discrepancy is the 30°C (54°F) temperature difference between the reactor cavity walls of the Swiss vessel and the external walls compared with the 39°C (70°F) difference of the HTGR vessel. A preprocessor program was written and used to calculate the temperatures at the nodes of the HTGR vessel by use of the thermal profiles for the Swiss PCRV. The resulting isotherms for a horizontal section at midheight of the reactor cavity and for a vertical section through the steam generator cavity are shown in Fig. 3.3. Two analyses of the thermal gradient loading condition are reported by Saugy.⁴ One analysis assumed that the temperature within any one finite element was constant; it naturally resulted in a discontinuous thermal gradient within the vessel. Another analysis included the gradient within an element and thereby resulted in a more accurate continuous thermal gradient. Since the maximum stresses calculated with both assumptions were approximately

ORNL-DWG 77-7687

Fig. 3.3. Isotherms of HTGR Vessel. Contour levels (°C): A, 65; B, 60; C, 54; D, 49; E, 43; F, 38; G, 32; H, 27. (a) Horizontal section at midheight of reactor cavity. (b) Vertical section through steam generator cavity.

the same and since STATIC-SAP uses a continuous thermal gradient representation, we feel that any errors introduced by using the temperature approximated from the report by Saugy (and shown in Fig. 3.3) will be insignificant.

3.2.1.3 Analytical Results

The results of the analysis of the PCRV of an HTGR are compared with the results of an earlier analysis of the Ohbayashi-Gumi multicavity PCRV model. The results of both analyses should be similar because the vessels have similar geometries. The Ohbayashi-Gumi vessel was the subject of the verification studies mentioned previously. The modulus of elasticity for the concrete of the HTGR is 34.5 GPa (5×10^6 psi), compared with 31 GPa (4.5×10^6 psi) for the concrete of the Ohbayashi-Gumi vessel. The moduli are sufficiently close to allow a reasonable comparison of strains and, of course, do not affect the stresses.

The axial strains resulting from the vertical prestress shown in Fig. 3.4 are practically identical to those for the Ohbayashi-Gumi vessel. Both vessels exhibit a high gradient in the top slab because of the concentrated prestressing forces.

Both vessels also have tensile strains in the central portion of the top slab and high strain gradients at the haunches. The walls of the vessels have almost constant strain through the thickness. The slightly higher strains in the wall of the Ohbayashi-Gumi vessel can be explained by the lower modulus of elasticity of the Ohbayashi-Gumi vessel relative to the HTGR and the higher effective prestressing level of the Ohbayashi-Gumi vessel.

The radial strains due to circumferential prestressing presented in Fig. 3.5 are very similar to the strains in the Ohbayashi-Gumi vessel for the same loading. The inside surface of the walls of the vessels have tensile strains of approximately 50×10^{-6} . The largest compressive strains, 300×10^{-6} , occur at the haunches of the vessels. The higher relative circumferential prestressing level of the HTGR probably causes the larger compressive strains in the top and bottom slabs.

The hoop strains resulting from circumferential prestress shown in Fig. 3.6 differ from those of the Ohbayashi-Gumi vessel primarily in the

Fig. 3.4. Axial Strain Due to Vertical Prestressing for (a) HTGR Vessel and (b) Ohbayashi-Gumi Vessel.

ORNL-DWG 77-4967

(a)

ORNL-DWG 76-17633

(b)

Fig. 3.5. Radial Strain Due to Circumferential Prestressing for
(a) HTGR Vessel and (b) Ohbayashi-Gumi Vessel.

ORNL-DWG 77-4965

ORNL-DWG 77-3254

Fig. 3.6. Hoop Strain Due to Circumferential Prestressing for
(a) HTGR Vessel and (b) Ohbayashi-Gumi Vessel.

region of the vessel wall near the reactor cavity, where the strains in the HTGR are approximately 80×10^{-6} more compressive. The differing relative prestressing levels, as mentioned previously, are probably the major contributor to the larger strains in the HTGR. Throughout the remainder of the cross section the strains in the two vessels are almost the same.

The hoop strains due to internal pressure are shown in Fig. 3.7. These strains are very close (within 50×10^{-6}) to those for the Ohbayashi-Gumi vessel. For both vessels the strains are mostly tensile along the wall of the reactor cavity near midheight of the reactor cavity and compressive in the region of the vessel wall between steam generator cavities.

The analytical results for the PCRV of the HTGR appear to be reasonable according to comparisons with the earlier calculations for the Ohbayashi-Gumi model. A report being prepared at ORNL will include a comparison of the analytical results of the PCRV for an HTGR with the allowable stresses prescribed by Sect. III, Div. 2 of the ASME Code.¹

3.2.2 Analysis Methods Development

Many analytical representations that attempt to describe the response of concrete under complex loading conditions have been proposed over the past several years. To identify which of the models provides the best representation for HTGR applications, a "constitutive model driver" computer program was developed. It serves as a material test simulator; the response of various hypothetical materials defined by constitutive models can be determined for a virtually limitless combination of loading conditions.

Thus far, the endochronic constitutive model developed by Bazant⁵ and the concrete material model in the 1977 version of the ADINA finite-element computer program⁶ have been implemented in the constitutive model driver program. Test cases that have been run with available experimental data indicate that the ADINA model underestimates the predicted strain at crushing, and that repetitive loading and unloading induces an artificial increased stiffness in the concrete. For example, Fig. 3.8 compares experimental data⁷ for equal biaxial compressive loading ($\sigma_{11} = \sigma_{22}$, $\sigma_{23} = 0$)

ORNL-DWG 77-3284

ORNL-DWG 77-3253

Fig. 3.7. Hoop Strain Due to Internal Pressure for (a) HTGR Vessel and (b) Ohbayashi-Gumi Vessel.

Fig. 3.8. Biaxial Stress-Strain Curve of ADINA Concrete Model Compared with Experimental Data. 1 ksi = 6.895 MPa.

with results calculated with the ADINA concrete model. The larger symbols in the figure represent the experimental data points and the solid lines and smaller symbols represent the results obtained from the ADINA model. Up to about 50% of ultimate the ADINA model predicted the behavior of the concrete very well; however, at higher stresses the predicted strains were too small. The excessive stiffness at high stress caused the ADINA model to incorrectly predict the concrete to crush when $\epsilon_{11} = -0.0017$. A more serious flaw in the ADINA model is the assumption that the concrete carries no stress at strains larger than the strain at crushing.

The stress-strain curve calculated with the ADINA concrete model for a uniaxially loaded specimen is presented in Fig. 3.9. As shown, the first 12 load steps are increments of -0.0001 strain. In the next eight steps, the strain increment is 0.0001 (unloading). The final nine steps are increments of -0.0001 strain (reloading), which cause crushing at a strain of -0.0013 . The higher stress in step 28 compared with step 12 is

Fig. 3.9. Uniaxial Stress-Strain Curve of ADINA Concrete Model.
1 ksi = 6.895 MPa.

caused by the use in ADINA of the tangent modulus at the end of step 27 to calculate the stress increment at step 28. At the end of step 28 the ADINA model recognizes that the specimen is being unloaded and, therefore, uses the initial tangent modulus to calculate the subsequent strain increments. Since the ADINA concrete model uses a modulus of elasticity based upon the strains at the end of the previous load step, loading and unloading cause an accumulation of error, which can lead to unexpected and incorrect results such as are shown in Fig. 3.9.

Figure 3.10 shows the same experimental data as Fig. 3.8, but the theoretical values were determined with the endochronic constitutive model developed by Bazant.⁵ The superiority of Bazant's model can be clearly seen, since not only the behavior to ultimate stress but also the strain softening behavior afterward is accurately predicted. Because the endochronic model seems to be the best constitutive model currently available for modeling concrete it has been implemented into the

Fig. 3.10. Biaxial Stress-Strain Curve of Endochronic Model Compared with Experimental Data. 1 ksi = 6.895 MPa.

two-dimensional material library of the ORNL ADINA finite-element program. The subroutines for the model are written so that they can be easily modified for the three-dimensional case. Unlike the original concrete model in ADINA, the endochronic model was programmed to take advantage of equilibrium iterations during the solution of the system of equations. The use of equilibrium iterations removes any errors that are caused by using the tangent modulus of the material at the end of one load step to arrive at the displacement of the next load step; repetitive loading and unloading should not cause an artificial increase in stiffness similar to the one shown for the original ADINA concrete model (Fig. 3.8).

3.3 INSTRUMENTATION EVALUATION AND DEVELOPMENT — D. J. Naus and C. C. Hurtt

Instrumentation for prestressed concrete pressure vessels (PCPVs) is required to determine strain distributions, deflections, stresses, temperature distribution, cracking magnitudes, forces in prestressing,

and free moisture contents. These quantities are used to assess the safety and reliability of PCPVs for short- and long-term operation, to assess correctness of calculations and assumptions of analytical techniques, and to assess the complex behavior of the vessels, especially monitoring of the structural integrity for extended periods of time.

Numerous commercial concrete embedment instrumentation systems are available for indicating strains, stresses, loads, and moisture contents. Since instrumentation is so vital in providing a continuing assurance of the safe operation of PCPVs, it is imperative that these devices provide very reliable information. To provide insight into performance of these instruments, laboratory investigations are being conducted. These investigations have a two-fold purpose: (1) to evaluate current commercially available systems and (2) to provide data for modification of existing designs and/or development of new systems. The initial phase of the investigation just recently completed has included an overview of concrete embedment instrumentation and an experimental evaluation of concrete embedment strain instrumentation. Acoustic emission is also being investigated as a technique for monitoring the structural integrity of PCPVs.

3.3.1 Review of Concrete Embedment Instrumentation

Various instrumentation systems for embedment in concrete are available commercially to indicate strain, stress, and moisture. Temperature measurement capability often is included in the stress or strain instrumentation, or readings may be readily obtained with thermocouples or thermistors. PCPVs are designed so that concrete remains in compression, but if the stress becomes tensile, surface cracks that develop may be easily detected by visual, gage, or acoustic methods, and internal cracks could possibly be detected by acoustic emission⁸ or holography.⁹ Techniques for measurement of temperature and for detection of concrete surface cracks will not be discussed further since reliable methods apparently exist for each.

3.3.1.1 Strain Measurement

Strain measurements provide data used to identify stress distributions, creep, and shrinkage of concrete and their effects on prestress, and to provide input for design refinement. Problems of strain measurement in

PCPVs arise from the highly alkaline and humid conditions coupled with elevated temperatures and relatively low radiation levels. The basic techniques utilized for internal strain measurement in concrete are electrical resistance, electrical inductance, electrical capacitance, mechanical, fluidic, and acoustic. Of these, electrical resistance and acoustic techniques are used most frequently. Fluidic and mechanical embedment gages¹⁰ are under development in Germany, and an inductance gage is being developed in the United States.¹¹ Table 3.1 presents information on several of the embedment strain gages.

3.3.1.2 Stress Measurement

Measurement of stress relaxation and the identification of highly stressed regions in PCPVs are important from both structural integrity and design considerations. Strain measurements, especially in PCPVs, invariably cannot be used directly to identify stresses because the measured strains also include creep, shrinkage, thermal, and possibly chemical reaction components. Moreover, stress can exist without corresponding strain, the concrete modulus of elasticity can change, and a complex stress state may exist. Thus, when stresses are required, the best technique would be to measure them directly. The two techniques available for stress determinations in concrete involve the measurement of stress in embedded plugs or flat plates. The flat plate approach is most widely used even though it cannot measure tensile stresses. The stress plug approach generally requires access for viewing fringe development; however, one method has been developed that uses a differential transformer to assess differential length changes, thus eliminating the access requirements.¹² Table 3.2 presents three stress meters of the flat-plate type.

3.3.1.3 Moisture Measurement

Free moisture movement in concrete structures resulting from changing conditions such as pore pressure, humidity, and temperature directly affects concrete properties, particularly neutron shielding, thermal conductivity, shrinkage, and creep. In addition, prestressing losses and concrete cracking may result from loss of moisture. Measurement of

Table 3.1. Concrete Embedment Strain Gages

Type	Gage length (cm)	Strain range ($\mu\text{m}/\text{m}$)	Temperature range ($^{\circ}\text{C}$)	Sensitivity ($\mu\text{m}/\text{m}$)	Calibration factor	Readout requirements	Approximate cost/gage (U.S. \$)	Comments
Electrical resistance								
Plastic encapsulated Type A	1-13	-20,000 to +15,000	-20 to +80	10	2.11	Strain indicator	5-20	Stability in moist environment questionable, volume change of plastic
Plastic encapsulated Type B	25	50,000	-100 to +121	5-10	2	Strain indicator	50	Good resistance to grease and acids; stability in moist environment questionable
Single wire	5-15	$\pm 20,000$	-200 to +315	5-10	2	Strain indicator	55-150	High-temperature use, small cross section requires careful placement
Unbonded wire Type A	10-15	3900	65	± 5		Bridge circuit	35-75	Proven reliability
Unbonded wire Type B	10-25	+500 to -1000	-30 to +70	± 6		Bridge circuit	35-40	Proven reliability
Unbonded wire Type C	5-10	± 5000	-20 to +100	5-10	≈ 2.5	Strain indicator	50-70	Temperature compensated
Semiconductor ^a	0.16-1.3	+3000 to -10,000	-16 to +800		50-250	Bridge circuit	18-25	Very limited concrete applications, stable, high-temperature use, sensitive
Vibrating wire								
VWSG Type A	7-14	>1000	80	0.5	0.96×10^{-3}	Period or frequency meter	85-95	Used extensively, additional sealing recommended
VWSG Type B	14	>1000	80	0.5	3.00×10^{-3}	Period or frequency meter	75	Used extensively, additional sealing recommended
VWSG Type C	12.7	4000	-40 to +66	1.0	<i>b</i>	Period or frequency meter	150	Wire tension easily adjusted
VWSG Type D	13	3000		0.5	2.60×10^{-3}	Period or frequency meter	160	Temperature measurement capability
VWSG Type E	10	3000	-40 to +80	0.1	2.01×10^{-3}	Period or frequency meter	150	Temperature measurement capability, stability poor in moist environment
VWSG Type F	8.8	1000	-10 to +70	1	1.20×10^{-3}	Period or frequency meter	125	Temperature measurement capability
VWSG Type G	13.3	2000	-10 to +70 ^c	1	3.00×10^{-3}	Period or frequency meter	125-200	Temperature measurement capability
Inductance								
WES gage ^a	10.2	30,000		± 10		Carrier amplifier	200-300	Stable, low gage modulus limited number of gages fabricated

^aIncluded for comparison purposes; gages of these types were not evaluated under this study.

^bStrain determined from table supplied with gages which accounts for nonlinearities.

^cMay be specified for temperature range -30 to +200 $^{\circ}\text{C}$.

Table 3.2. Stress Meters for PCPVs

Type	Geometry (cm)	Stress range (MPa)	Temperature range (°C)	Sensitivity (kPa)	Approximate cost/gage (\$)	Comments
A	17.8 diam by 1.5	3-10	<66	21-69	250	Inability to measure tensile stresses; measures temperature
B	17.5 diam by 1.3	3-12	-26 to 71	28-97	200	Similar to Type A meter
C	7 by 14 by 0.2	0-34	-18 to 88	±7	270	Requires hydraulic lines

moisture is difficult since the water may be present in chemically combined, absorbed, and evaporable forms. Measurements of neutron or gamma ray absorption, electrical resistivity, and dielectric constant are the primary techniques employed. Accuracy of the resulting moisture measurements is generally limited to approximately $\pm 20\%$. None of the existing techniques appear to be satisfactory for long-term moisture monitoring of a PCPV. Moreover, if a satisfactory technique should be developed, it is not altogether clear how moisture measurements in a PCPV could be used to evaluate vessel performance.

3.3.2 Concrete Embedment Strain Instrumentation Evaluation

To establish performance reliability trends for available strain meters, a representative sample of strain meter types was obtained and evaluated. Because so many different types and brands of strain meters are available, we decided to restrict the study, at least initially, to meters readily available in the United States. These meters are noted in Table 3.1. Meters obtained were evaluated with respect to general gage performance, performance in a representative PCPV environment, and performance in extreme environments.

3.3.2.1 General Gage Performance

Each type of gage was evaluated with respect to sensitivity, accuracy, stability, response, data acquisition requirements, cost, adequacy of calibration, and durability (whether or not precasting the gages in

briquettes is needed for protection). With the exception of calibration and stability the evaluations were based on manufacturer's data, the results of which are summarized in Table 3.1. Studies were also conducted to determine a satisfactory calibration specimen and to determine the effect on gage performance of concrete strength and of the ratio of meter gage length to maximum concrete size.

Before the test series to evaluate the gage calibration data a limited laboratory study was conducted to select a test specimen. Three sets of specimens were cast from the same mix (gravel/cement ratio = 2.64, sand/cement ratio = 2.04) except for the water/cement ratio, which was varied (0.575, 0.500, 0.425), to produce different strengths. Each set consisted of cylinders having diameters of 0.05, 0.08, 0.10, and 0.15 m and a constant length-to-diameter ratio of 3.54. One unbonded wire Type A strain meter, which was aligned with the longitudinal axis, was embedded in each specimen. After curing in a laboratory environment, two 0.1-m surface strain gages were applied to each specimen at 180° intervals and connected in series to eliminate bending effects. The specimens were loaded in compression to failure with surface and embedded strain gage readings obtained at specific intervals. Results summarized in Table 3.3 indicate that surface strains were always less than embedded strains at the same stress level and that as the specimen diameter increased there was a trend toward better agreement between experimentally determined and manufacturer's calibration factors. For example, the differences in calibration factors for 0.05- and 0.15-m-diam cylinders were 45 and 8%, respectively. On the basis of these results a 0.15-m-diam by 0.54-m-long test specimen was used to determine calibration factors. No apparent strength effect was noted from these preliminary tests.

Three additional sets of specimens corresponding to low (20.4 MPa), medium (29.4 MPa), and high (41.8 MPa) strength concretes were cast to determine the effect of concrete strength on embedded gage performance. Vibrating wire Type G, unbonded wire Type A, and single wire gages were employed. Unfortunately, no definite data trends indicating the effect of concrete strength were observed. This might possibly be somewhat attributable to the effect of the relative moduli of the meter and the concrete tending to mask the strength influence.

Table 3.3. Calibration Specimen Size Test Series
(Unbonded Wire Type A Gages)

Mix	Cylinder diameter ^a (cm)	Calibration factor supplied	Cylinder failure stress (MPa)	Elastic modulus (GPa)	Calculated calibration factor	Calibration factor difference ^b (%)
5	5.1	532	26.4	26.7	294	+44.7
5	7.6	537	21.7	27.1	274	+49.0
5	10.2	530	29.2	33.9	407	+23.2
5	15.2	532	41.2	30.8	492	+7.5
6	5.1	538	21.8	20.1	407	+24.3
6	7.6	529	24.1	30.9	238	+55.1
6	10.2	533	29.4	31.6	374	+29.9
6	15.2	538	34.4	29.9	443	+17.7
7	5.1	534	19.6	18.4	269	+49.6
7	7.6	^c	24.3	25.2	^c	^c
7	10.2	529	26.5	23.9	444	+16.0
7	15.2	531	31.9	30.8	434	+18.2

^aCylinder length-to-diameter ratio equals 3.54.

^bPercent difference = [(gage factor supplied - gage factor calculated)/gage factor supplied] × 100%.

^cBad embedment gage.

Calibration factors were determined for the strain meters noted in Table 3.1. One embedment strain meter was cast in each cylindrical test specimen with the gages aligned with the cylinder axis. The concrete mix design consisted of Type II cement, 20-mm maximum size crushed limestone aggregate, a gravel-to-cement ratio of 1.85, a sand-to-cement ratio of 2.84, and a water-to-cement ratio of 0.56. After specimen curing, three sets of mechanical gage points with 0.20-m gage lengths were attached at 180° intervals on each specimen's circumference. The test procedure consisted of: taking zero readings; loading the specimen compressively in 22.2 kN increments to approximately 50% of ultimate load with mechanical, surface, and embedded readings taken at each increment; unloading in 22.2 kN increments with readings taken at each increment; and reloading the specimen in compression to specimen failure with surface and embedded strain readings taken at the same load increments. Figure 3.11 presents the calibration test setup. Results for the 0.152-m-diam by 0.54-m calibration test cylinders are summarized in Table 3.4.

Fig. 3.11. Calibration Test Setup.

It is generally recommended that the gage length of embedded gages be at least 3 to 5 times the largest maximum aggregate size. These conclusions are derived largely from the work of Cooke and Seddon,¹³ in which bonded wire resistance gages with gage lengths of either 12.7 or 25 mm were attached to the surfaces of specimens fabricated from concretes with maximum aggregate sizes ranging from 1.3 to 9.5 mm. To substantiate that this is also true for strain meters embedded in concrete, a limited test series was conducted. Specimens with the same geometry as the standard calibration test specimens were used in the study. Either unbonded wire Type A or vibrating wire strain gage (VWSG) Type G strain meters were cast in concretes having either 9.5, 19.1, or 38.1 mm maximum aggregate sizes. Ratios of gage length to maximum aggregate size thus varied from 2.7 to 10.6 for the specimens containing the unbonded wire Type A gages and from 3.5 to 14.0 for those containing the VWSG Type G gages. Casting, curing, and testing the specimens followed the same

Table 3.4. Summary of Test Results for 0.152-m-diam by 0.54-m Calibration Test Cylinders

Gage type	Gage	Mix	Calibration factor supplied	Initial load cycle				Failure load cycle			
				Maximum stress level (MPa)	Elastic modulus (GPa)	Calculated calibration factor	Calibration factor difference ^a (%)	Cylinder failure stress (MPa)	Elastic modulus (GPa)	Calculated calibration factor	Calibration factor difference ^a (%)
Electrical resistance											
Plastic encapsulated Type A	1	1	2.11	14.6	32.1	2.27	-7.6	33.9	31.7	2.36	-11.8
	2	3	2.11	17.1	24.4	1.56	+26.1	23.4	20.5	1.55	+26.5
	3	3	2.11	17.1	28.2	2.18	-3.3	23.4	23.9	2.14	-1.4
	4	19	2.11	14.6	30.6	1.95	+7.6	32.1	27.9	1.88	+10.9
	5	21	2.11	14.6	29.0	2.09	+1.0	32.8	29.5	2.11	0
Plastic encapsulated Type B	1	18	2.00	14.6	29.8	1.92	+4.0	27.3	28.0	1.83	+8.5
	2	19	2.00	14.6	26.5	1.96	+2.0	28.7	27.4	1.84	+8.0
	3	18	2.00	14.6	27.5	1.79	+10.5	23.9	26.5	1.78	+11.0
	4	19	2.00	14.6	28.1	2.07	-3.5	31.4	28.5	2.02	-1.0
Single wire	1	3	2.00	17.1	24.8	1.63	+18.6	24.1	20.2	1.74	+13.0
	2	4	2.00	17.1	30.5	1.65	+17.5	35.6	29.2	1.71	+14.5
	3	4	2.00	17.1	32.3	1.94	+3.0	36.1	31.6	2.02	-1.0
	4	21	2.00	14.6	30.3	1.54	+23.0	30.6	31.9	1.63	+18.5
	5	21	2.00	14.6	31.8	1.98	+1.0	29.2	31.7	2.20	-10.0
	6	16	2.00	9.8	22.2	1.54	+23.0	22.3	21.6	1.51	+24.5
	7	17	2.00	14.6	28.9	1.82	+9.0	30.3	27.4	1.75	+12.5
	8	15	2.00	17.1	35.1	1.38	+31.0	43.8	34.1	1.52	+24.0
	9	15	2.00	17.1	35.1	1.41	+29.5	41.4	35.0	1.50	+25.0
	10	12 ^b	2.00	17.1	28.7	1.63	+18.5	29.9	25.0	1.73	+13.5
	11	13 ^c	2.00	12.2	20.8	1.31	+34.5	22.4	22.0	1.45	+27.5
Unbonded wire Type A	1	2	528	17.1	25.8	422	+20.1	24.9	20.8	429	+18.8
	2	2	532	17.1	26.3	451	+15.2	25.2	21.8	469	+11.8
	3	4	536	17.1	29.7	449	+16.2	33.0	26.3	433	+19.2
	4	20	527	14.6	24.9	526	0	38.0	27.0	520	+1.3
	5	16	527	17.1	23.4	444	+15.7	24.1	21.1	440	+16.5
	6	16	529	9.8	24.4	487	+7.9	19.0	25.5	474	+10.4
	7	17	536	14.6	28.8	433	+19.2	27.6	26.7	431	+19.6
	8	17	536	14.6	28.8	468	+12.7	30.3	27.5	463	+13.6
	9	15	541	17.1	34.4	503	+7.0	41.4	35.8	515	+4.8
	10	15	535	17.1	36.2	520	+2.8	41.0	35.5	519	+3.0
	11	5	532					41.2	31.8	492	+7.5
	12	6	538					34.4	30.6	443	+17.7
	13	7	531					31.9	30.0	434	+18.2

Table 3.4. (Continued)

Gage type	Gage	Mix	Calibration factor supplied	Initial load cycle				Failure load cycle			
				Maximum stress level (MPa)	Elastic modulus (GPa)	Calculated calibration factor	Calibration factor difference ^a (%)	Cylinder failure stress (MPa)	Elastic modulus (GPa)	Calculated calibration factor	Calibration factor difference ^a (%)
Unbonded wire Type A (contd.)	14	12 ^b	539	17.1	25.0	507	+5.9	27.9	23.8	504	+6.5
	15	12 ^b	533	17.1	27.4	474	+11.1	28.2	26.1	471	+11.6
	16	13 ^c	544	12.2	28.7	453	+16.7	29.2	30.3	454	+16.5
	17	13 ^c	532	12.2	28.3	361	+32.1	25.6	28.8	356	+33.1
Unbonded wire Type B	1	3	636	17.1	26.8	504	+20.8	26.0	23.9	515	+19.0
	2	17	655	14.6	24.4	551	+15.9	25.9	24.4	566	+13.6
	3	20	617	14.6	30.5	573	+7.1	37.0	30.5	545	+11.7
	4	20	638	14.6	31.2	579	+9.2	36.6	30.3	581	+8.9
Unbonded wire Type C	1	20	2.43	14.6	30.9	2.33	+4.1	41.1	31.5	2.60	-7.0
	2	20	2.34	14.6	29.2	2.46	-5.1	38.3	31.4	2.55	-9.0
	3	20	2.44	14.6	33.1	2.85	-16.8	38.7	33.5	3.05	-25.0
	4	14	2.52	17.1	32.3	2.84	-12.7	31.4	30.9	2.86	-13.5
Vibrating wire											
VWSG Type A	1	1	0.96×10^{-3}	17.1	36.0	0.60×10^{-3}	+37.5	34.1	34.8	0.64×10^{-3}	+33.3
	2	3	0.96×10^{-3}	17.1	25.7	0.75×10^{-3}	+21.9	25.9	27.6	0.78×10^{-3}	+18.8
	3	3	0.96×10^{-3}	17.1	27.7	0.77×10^{-3}	+19.8	26.8	23.5	0.72×10^{-3}	+25.0
VWSG Type B	1	1	3.00×10^{-3}	17.1	34.8	2.75×10^{-3}	+8.3	34.1	32.9	2.58×10^{-3}	+14.0
	2	2	3.00×10^{-3}	17.1	28.0	2.44×10^{-3}	+18.7	23.9	25.1	2.71×10^{-3}	+9.7
VWSG Type C	1	14	NA	17.1	30.6	3.17×10^{-3}	+41.0 ^d	30.3	28.7	3.37×10^{-3}	+27.6 ^d
	2	14	NA	17.1	28.3	3.64×10^{-3}	+19.7 ^d	31.9	26.4	3.76×10^{-3}	+17.4 ^d
	3	18	NA	14.6	27.3	3.35×10^{-3}	+27.5 ^d	29.9	26.7	3.59×10^{-3}	+29.4 ^d
	4	20	NA	14.6	30.5	3.68×10^{-3}	+19.7 ^d	39.2	31.9	3.68×10^{-3}	+25.4 ^d
	5	20	NA	14.6	32.8	3.35×10^{-3}	+28.9 ^d	37.8	33.4	3.49×10^{-3}	+30.5 ^d
VWSG Type D	1	1	2.60×10^{-3}	17.1	32.9	3.00×10^{-3}	-15.4	34.0	29.9	3.03×10^{-3}	-16.5
	2	17	2.60×10^{-3}	14.6	27.4	2.82×10^{-3}	-8.5	26.0	27.7	2.89×10^{-3}	-11.2
VWSG Type E	1	1	2.01×10^{-3}	17.1	31.9	1.75×10^{-3}	+12.9	33.2	30.1	1.68×10^{-3}	+16.4
	2	2	2.01×10^{-3}	17.1	26.1	1.83×10^{-3}	+9.0	24.4	21.2	1.90×10^{-3}	+5.5
VWSG Type F	1	8	1.20×10^{-3}	17.1	30.5	1.15×10^{-3}	+4.2	38.5	29.9	1.10×10^{-3}	+8.3
	2	18	1.20×10^{-3}	14.6	26.1	1.00×10^{-3}	+16.7	26.7	24.4	0.95×10^{-3}	+20.8
	3	19	1.20×10^{-3}	14.6	27.7	1.07×10^{-3}	+10.8	33.2	25.6	1.07×10^{-3}	+10.8
	4	1	1.24×10^{-3}	17.1	30.9	0.98×10^{-3}	+21.0	34.5	28.8	1.00×10^{-3}	+19.4
	5	2	1.24×10^{-3}	17.1	27.4	1.08×10^{-3}	+12.9	25.9	23.5	1.09×10^{-3}	+12.1

Table 3.4. (Continued)

Gage type	Gage	Mix	Calibration factor supplied	Initial load cycle				Failure load cycle			
				Maximum stress level (MPa)	Elastic modulus (GPa)	Calculated calibration factor	Calibration factor difference ^a (%)	Cylinder failure stress (MPa)	Elastic modulus (GPa)	Calculated calibration factor	Calibration factor difference ^a (%)
VWSG Type G	1	4	3.00×10^{-3}	17.1	30.2	2.37×10^{-3}	+21.0	33.0	27.8	2.38×10^{-3}	+20.7
	2	4	3.00×10^{-3}	17.1	28.7	2.26×10^{-3}	+24.7	28.1	24.8	2.14×10^{-3}	+28.7
	3	4	3.00×10^{-3}	17.1	30.9	2.25×10^{-3}	+25.0	34.3	29.2	2.24×10^{-3}	+25.3
	4	20	3.00×10^{-3}	14.6	29.3	2.65×10^{-3}	+11.7	38.3	30.3	2.50×10^{-3}	+16.7
	5	16	3.00×10^{-3}	9.8	23.5	2.63×10^{-3}	+12.3	19.0	21.4	2.68×10^{-3}	+10.7
	6	16	3.00×10^{-3}	9.8	25.2	2.16×10^{-3}	+28.0	16.6	24.9	1.82×10^{-3}	+39.3
	7	15	3.00×10^{-3}	17.1	39.9	2.19×10^{-3}	+27.0	41.5	39.2	2.14×10^{-3}	+28.7
	8	15	3.00×10^{-3}	17.1	37.0	2.22×10^{-3}	+26.0	41.6	37.0	2.23×10^{-3}	+25.7
	9	12 ^b	3.00×10^{-3}	17.1	28.8	2.22×10^{-3}	+26.0	32.5	27.1	2.23×10^{-3}	+25.7
	10	12 ^b	3.00×10^{-3}	17.1	29.8	2.27×10^{-3}	+24.3	31.7	27.3	2.31×10^{-3}	+23.0
	11	13 ^c	3.00×10^{-3}	12.2	21.8	2.25×10^{-3}	+25.0	24.2	23.4	2.52×10^{-3}	+16.0
	12	13 ^c	3.00×10^{-3}	12.2	23.2	2.36×10^{-3}	+21.3	23.3	24.4	2.24×10^{-3}	+25.3

^aPercent difference = [(gage factor supplied - gage factor calculated)/gage factor supplied] × 100%.

^b0.95 cm maximum aggregate size.

^c3.81 cm maximum aggregate size.

^dGage factor not supplied so difference represents difference in moduli obtained from surface gages and from manufacturer's calibration table.

procedure as for the calibration specimens. Results summarized in Table 3.5 indicate that as the gage length-to-maximum-aggregate size ratio for the unbonded Type A meter changed from 10.6 to 5.3 to 2.7 the

Table 3.5. Summary of Test Results for Effect on Meter Performance on Ratio of Gage Length to Maximum Aggregate Size

Mix	Gage length-to-diameter ratio	Calibration factor supplied	Initial load cycle				Failure load cycle			
			Maximum stress level (MPa)	Elastic modulus (GPa)	Calculated calibration factor	Calibration factor difference ^a (%)	Cylinder failure stress (MPa)	Elastic modulus (GPa)	Calculated calibration factor	Calibration factor difference ^a (%)
Unbonded wire Type A										
12	10.6	539	17.1	25.0	507	+5.9	27.9	23.8	504	+6.5
12	10.6	533	17.1	27.4	474	+11.1	28.2	26.1	471	+11.6
2	5.3	528	17.1	25.8	422	+20.1	24.9	20.8	429	+18.8
2	5.3	532	17.1	26.3	451	+15.2	25.2	21.8	469	+11.8
4	5.3	536	17.1	29.7	449	+16.2	33.0	26.3	433	+19.2
13	2.7	544	12.2	28.7	453	+16.7	29.2	30.3	454	+16.5
13	2.7	532	12.2	28.3	361	+32.1	25.6	28.8	356	+33.1
VWSG Type G										
12	14.0	3.00 × 10 ⁻³	17.1	28.8	2.22 × 10 ⁻³	+26.0	32.5	27.1	2.23 × 10 ⁻³	+25.7
12	14.0	3.00 × 10 ⁻³	17.1	29.8	2.27 × 10 ⁻³	+24.3	31.7	27.3	2.31 × 10 ⁻³	+23.0
4	7.0	3.00 × 10 ⁻³	17.1	30.2	2.37 × 10 ⁻³	+21.0	33.0	27.8	2.38 × 10 ⁻³	+20.7
4	7.0	3.00 × 10 ⁻³	17.1	28.7	2.26 × 10 ⁻³	+24.7	28.1	24.8	2.14 × 10 ⁻³	+28.7
4	7.0	3.00 × 10 ⁻³	17.1	30.9	2.25 × 10 ⁻³	+25.0	34.3	29.2	2.24 × 10 ⁻³	+25.3
13	3.5	3.00 × 10 ⁻³	12.2	21.8	2.25 × 10 ⁻³	+25.0	24.2	23.4	2.52 × 10 ⁻³	+16.0
13	3.5	3.00 × 10 ⁻³	12.2	23.2	2.36 × 10 ⁻³	+21.3	23.3	24.4	2.24 × 10 ⁻³	+25.3

^aPercent difference = [(gage factor supplied - gage factor calculated)/gage factor supplied] × 100%.

average difference between manufacturer's supplied calibration factors and experimentally determined calibration factors increased from 9.1 to 16.6 to 24.8%; however, for the VWSG Type G meter, where the ratio changed from 14.0 to 7.0 to 3.5, the calibration factor average differences remained relatively constant at 24.4, 24.9, and 20.6%, respectively. Relative results obtained from the embedded gages and from the surface wire resistance strain gages used by Cooke and Seddon,¹³ Binns and Mygind,¹⁴ and Peattie¹⁵ are presented in Fig. 3.12. Similar trends are apparent for both the wire resistance and embedded gages. Offset of embedded gage data from surface strain gage data depends on both the type of embedded gage (modular ratio) and the fact that the gages are measuring internal strains, which may differ slightly from surface strains. The data thus verifies that the ratio of meter gage length to maximum aggregate size should be at least 3.5.

Fig. 3.12. Effect of Ratio of Gage Length to Maximum Aggregate Size on Strain Meter Performance.

The stability of embedded strain gages was determined with the copper-sealed cylindrical concrete test specimen 0.41 m diam by 1.02 m long shown in Fig. 3.13. The specimen was cast from a mix with a 20-mm maximum aggregate size, a gravel-to-cement ratio of 2.60, a sand-to-cement ratio of 2.02, and a water-to-cement ratio of 0.48. Contained within the cylinder were 18 embedment strain gages consisting of three unbonded wire Type A, one unbonded wire Type B, two unbonded wire Type C, two polyester encapsulated, three single wire, two vibrating wire Type A, one vibrating wire Type B, two vibrating wire Type F, and two vibrating wire Type G. Mechanical gage points were attached to the cylinder at 120° intervals, and three weldable strain gages were attached at 120° intervals to a steel reinforcement cage. The cage maintained gage alignment during fabrication. Strain and temperature were monitored periodically while the cylinder was maintained in a laboratory environment. Results of the stability test are presented in Fig. 3.14 through 3.16 for the bonded wire, unbonded wire, and vibrating wire gages, respectively.

Photo 6044-77

Fig. 3.13. Gage Stability Test Specimen.

ORNL DWG 78-3873

Fig. 3.14. Strain Meter Stability Test Results for Bonded Wire Resistance Meters.

ORNL DWG 78-3872

Fig. 3.15. Strain Meter Stability Test Results for Unbonded Wire Resistance Meters.

ORNL DWG 78-3871

Fig. 3.16. Strain Meter Stability Test Results for Vibrating Wire Meters.

3.3.2.2 Gage Performance in Representative PCPV Environments

Figure 3.17 presents the setup for testing concrete embedment strain instrumentation under simulated PCPV operating conditions. The test specimen fabricated from the same mix design as the gage stability test specimen was a copper-sealed cylindrical concrete test specimen 0.41 m diam by 1.02 m long.

Fig. 3.17. Test Setup: Representative PCPV Environment.

Embedded in the cylinder were 12 embedment strain gages consisting of one unbonded wire Type A, one unbonded wire Type B, one unbonded wire Type C, one polyester encapsulated, one single wire, one vibrating wire Type A, one vibrating wire Type B, one vibrating wire Type D, one vibrating wire Type E, two vibrating wire Type F, and one vibrating wire Type G. Also included were three weldable strain gages attached to the steel reinforcement cage at 120° intervals and three thermocouples. After curing for three months, the cylinder was heated with an insulated heating blanket. The temperature was maintained at $65 \pm 5^\circ\text{C}$ for approximately six months. Then an axial compressive loading was applied to the heated cylinder. The specimen was loaded in increments of approximately 2.4 MPa. Temperature and strain readings were obtained just before, 1 hr after, and during each loading increment. Results obtained to date are presented in Figs. 3.18 and 3.19 for the wire resistance and vibrating wire strain gages, respectively.

3.3.2.3 Gage Performance in Extreme Environments

Performance of concrete embedment strain instrumentation under non-typical operating conditions was examined. Samples of each of the gages noted in Table 3.1 were tested by immersion in alkaline ($\text{pH} > 9$) water at room and elevated temperature. Effects of a combination of elevated temperature and pressure on performance of gages embedded in concrete were also investigated with the same specimen that was used for testing gage performance in a representative PCPV environment.

The test setup for the room-temperature (21°C) and elevated-temperature ($\sim 65^\circ\text{C}$) alkaline water baths are shown in Figs. 3.20 and 3.21, respectively. A small amount of cement was added to the water solution to increase the pH to approximately 9, samples of each of the gages were placed in the appropriate water baths, and indicated strain was monitored periodically as a function of time. Table 3.6 summarizes results.

Effects of a combination of elevated temperature and pressure on gage performance were investigated with the same concrete cylinder used to determine gage performance in a representative PCPV environment. The test procedure consisted incrementally increasing the nominal concrete cylinder temperature from 66°C to 70°C , to 80°C , and to 90°C . Temperatures were maintained at these values $\pm 5^\circ\text{C}$ for 10, 21, and 74 days, respectively.

Fig. 3.18. Test Results for Wire Resistance Strain Meters in Representative and Extreme PCPV Environments.

Fig. 3.19. Test Results for Vibrating Wire Strain Meters in Representative and Extreme PCPV Environments.

Fig. 3.20. Room-Temperature Alkaline Water Bath.

After exposure at 90°C, the concrete cylinder was permitted to cool to room temperature. On conclusion of the elevated-temperature test phase all embedded gages had failed except for one single wire, one unbonded wire Type A, one unbonded wire Type C, and one vibrating wire Type G.

3.3.2.4 Preliminary Conclusions from Strain Gage Study

1. Gage calibration factors should always be determined on representative samples from the particular batch of gages to be used.
2. Calibration test specimens should be big enough to provide reliable results and be fabricated from a representative concrete mix.
3. Gage selection should match the application; that is, a gage suitable for use in a model structure may be too short to be suitable for a prototype structure.
4. Research should be conducted on other measurement techniques based on inductance, capacitance, semiconductors, and fluidic principles in an effort to improve meter accuracy and reliability.

Photo 6040-77

Fig. 3.21. Elevated-Temperature Alkaline Water Bath.

3.3.3 Acoustic Emission Study

The ability of acoustic emission to monitor structural test members, specifically PCPVs, is also being examined under this task. During this reporting period the multichannel acoustic emission source location system being tested was used to monitor two test series: (1) a flawed thick-walled steel pressure vessel tested under the Heavy Section Steel Technology (HSST) program, and (2) grouted and nongrouted posttensioned concrete beams investigated under the structural model testing task area. Results for the cylinder tests will be reported in this section, and results for the beam tests will be reported in Sect. 3.4.4.

Table 3.6. Alkaline-Water Immersion Test Results

Gage type	Room-temperature water bath			Elevated-temperature water bath		
	Number tested	Number and type failure ^a		Number tested	Number and type failure ^a	
		a	b		a	b
Unbonded wire						
Type A	4	0	0	8	2	1
Type B	2	0	0	2	0	0
Type C	1	0	0	1	0	0
Polyester encapsulated						
Type A	3	3	0	6	6	0
Type B	6	6	0	0	0	0
Single wire	9	4	0	16	7	0
Vibrating wire						
Type A ^b	3	0	1	3	0	1
Type B ^b	1	0	1	0	0	0
Type C	2	0	0	2	0	0
Type D	2	0	2	1	0	1
Type E	1	1	0	1	0	1
Type F	3	0	0	4	0	1
Type G	2	0	0	3	0	2

^aType failure: a = excessive strain; b = no reading obtainable.

Excessive strain failure by definition is a strain in excess of a 3 $\mu\epsilon$ /day drift rate. This value was arbitrarily selected and in some cases may be too lax and others too stringent.

^bTested as received; manufacturer recommends additional sealing.

^cErratic readings at temperatures greater than manufacturer's stated upper limit.

Intermediate test vessel V-7B of the HSST program was fabricated from ASTM A 533, grade B, class 1 steel plate. The test vessel had an outside diameter of 0.99 m (39 in.) and an inside diameter of 0.69 m (27 in.). A flaw formed by hydrogen charging of an electron-beam weld in the heat-affected zone of a previous weld repair was the region of interest. Details of the test vessel and flaw are presented in Figs. 3.22 and 3.23, respectively. Results were obtained during the two pressurization cycles of the flawed vessel.

Fig. 3.22. Intermediate Test Vessel V-7B. To convert dimensions to meters, multiply by 0.0254.

Fig. 3.23. Flaw Design for V-7, V-7A, and V-7B. To convert dimensions to millimeters, multiply by 25.4.

3.3.3.1 Instrumentation

The multisensor comprehensive data (MSCD) system shown in Fig. 3.24 was used for acoustic emission monitoring of intermediate test vessel V-7B. Each input channel of the system consisted of a sensor, pre-amplifier, and amplifier. The amplified and filtered signals were fed into a processor, which derived the individual acoustic emission parameters from the detected signal, stored the data sets and their transfer to the computer, generated the control parameters for the entire MSCD system, and performed discrimination functions. Discrimination functions consisted of sensor activity detection (establishes reset time to clear system for the next data set), exclusion signals (accepts only valid sensor arrival sequences), and range gate (assures signals are grouped in complete sets within the instantaneous array). The MSCD measured the magnitude of acoustic emission events as a function of their location, time of occurrence, and parametric value. All derived acoustic emission parameters were processed, stored for off-line analysis, and displayed on the CRT screen in real time as either raw data, activity distribution, or magnitude distribution. Permanent records of the CRT screen contents were obtained during the test as desired.

Fig. 3.24. Acoustic Emission Instrumentation.

3.3.3.2 Experimental Procedure

The cylindrical test section of vessel V-7B was instrumented with eight transducers. Six were used as active sensors and two were used as slave sensors to lock the system out to events occurring outside the flawed region of interest. The entire cylindrical section of the vessel could not be monitored during this test because the required heating elements covered most of the surface. Transducers were attached to the vessel by epoxy shoes that had been glued to the vessel surface with an elevated-temperature epoxy (Barco Bond MB-100X epoxy adhesive from the Astro Chemical Company). Locations of active transducers (1-6) and slave transducers (S1,S2) are shown in Fig. 3.25. The epoxy shoe and the

Fig. 3.25. Transducer Positioning on Test Vessel.

transducer were coupled by a thin layer of Dow Corning 340 silicone heat sink compound. The MSCD system specifications during the test are given in Table 3.7. Pressure was monitored throughout the test by use of a parametric input to the MSCD from a pressure transducer.

Vessel temperature during the test was maintained at approximately 87°C. Vessel V-7B was tested hydraulically in two pressurization cycles. The first cycle was to 72.4 MPa, and the second was to failure at 151 MPa. Acoustic emission data were obtained during both cycles.

3.3.3.3 Cycle 1 Test Results: Pressurization to 72.4 MPa

The initial loading cycle consisted of pressurization to 72.4 MPa with intermediate pressure holds at 27.6 and 55.2 MPa for instrument scans.

Table 3.7. Specifications for Multisensor Comprehensive Data System During Test

Transducers	250-kHz resonant frequency, single ended
Filter frequencies, kHz	100-400
Preamplifier gain, dB	40
Signal conditioner gain, ^a dB	
Channel 1	44 (84 total)
Channel 2	47 (87 total)
Channel 3	47 (87 total)
Channel 4	55 (95 total)
Channel 5	43 (83 total)
Channel 6	44 (84 total)
Channel 7	43 (83 total)
Channel 8	56 (96 total)
Arrival time resolution, μ s	1
Sensor activity constant, ms	1
Threshold, mV	400
ΔT range selector, μ s	300
Wave propagation velocity, ^b km/s	2.5

^aSignal conditioner gain was established by balancing channels for equivalent background noise intensity.

^bEstablished by use of software program.

Acoustic emission (AE) data were obtained throughout the cycle. We detected 448 valid events during this cycle. Figure 3.26 presents a histogram of event occurrence for each 3.4 MPa loading interval. Figure 3.27 presents a summation of event magnitude voltages as a function of elapsed time and pressure. A two-dimensional grid displaying sensor and flaw locations, and events and event magnitudes at conclusion of the initial pressurization cycle is presented in Fig. 3.28. In the figure, the 0.203-m base of the flawed region is noted as a solid line, and the flawed region extending from the base to the vessel outer surface is noted by the dashed line. Each square of the grid where events have been noted contains two numbers: the upper number corresponds to the total number of accepted events and the lower number, the summation of the magnitudes of the voltages for the events. Regions with the most intense activity have been crosshatched.

Fig. 3.26. Event Occurrence Histogram: Cycle 1.

Fig. 3.27. Event Magnitude as a Function of Elapsed Time and Pressure: Cycle 1.

Fig. 3.28. Grid Display for Initial Pressurization Cycle, 448 Events. In each square of the grid, the upper number represents the number of events and the lower the summation of magnitudes.

All these regions with one exception were adjacent to the flaw, and the activity for the upper part of the flaw appeared greater than that for the lower. The one region not adjacent to the flaw but showing intense activity corresponds to the position where the stainless steel patch was fillet welded to the inner surface of the vessel, and this activity may be associated with a weld defect.

3.3.3.4 Cycle 2 Test Results: Pressurization to Failure

During the second pressurization cycle the vessel was loaded incrementally until a leak developed through the vessel wall at the flaw. The pressure was held constant for short intervals intermittently during the

test while instrument scans were taken. As for the first cycle, AE data were obtained throughout the loading cycle. During this cycle 931 valid events were detected. Figure 3.29 presents a histogram of event occurrence for each 3.4 MPa of load. Figure 3.30 presents a summation of event magnitude voltages as a function of elapsed time and pressure. Figure 3.31 presents a display of events and voltage magnitudes as a function of location at conclusion of the test. Notations in this figure are the same as for Fig. 3.28. As noted in the figure regions of primary activity were adjacent to the flaw.

Fig. 3.29. Event Occurrence Histogram: Cycle 2.

Fig. 3.30. Event Magnitude as a Function of Elapsed Time and Pressure: Cycle 2.

3.3.3.5 Conclusions

The following conclusions may be drawn from acoustic emission monitoring of HSST vessel V-7B.

1. Primary AE activity was adjacent to the flaw. Some activity was noted at a location corresponding approximately to the region where the stainless steel patch was fillet welded to the inner surface of the vessel. Data scatter may be attributable to both specimen geometry and the relatively close proximity of the transducers. (Spacing of transducers was less than 5 times the vessel thickness.¹⁶)

2. Plots of COD gages and summation of events as a function of pressure were geometrically similar.

3. At impending failure the slope of the summation of events as a function of pressure changed drastically. This thus provides a possible technique for monitoring the vessel so that pressurization may be halted before failure. Also, as failure was approached the magnitude of the emissions increased.

Fig. 3.31. Grid Display Pressure Cycle 2: Test Conclusion; 931 Events. In each square of the grid, the upper number represents the number of events and the lower the summation of magnitudes.

4. Results of this test tend to indicate that AE was able to identify regions where significant activity was occurring.

The AE monitoring of this test of a flawed steel vessel provided the opportunity to gain valuable experience in monitoring a complex structure to failure, and to obtain data on the types of acoustical signatures associated with overpressurization of a flawed steel structure. We plan in the future to monitor relatively simple prestressed concrete structures (see Sect. 3.4.4) as well as more complex PCPV models to provide the data required to adequately evaluate potential capability for AE monitoring of PCPVs in nuclear power plants.

3.4 CONCRETE STRUCTURAL MODEL TESTS — D. J. Naus and C. C. Hurtt

Concrete exhibits time-dependent strains, which influence stress redistribution. Analytical techniques for long-term time- and temperature-dependent behavior, especially for complex structures such as PCPVs and where there are departures from elastic behavior, require development and refinement. The series of model studies to be conducted under this task will be designed to provide input for development of new and/or refinement of existing analytical techniques for defining PCPV long-term time-, temperature-, and load-dependent behavior. Initially these models will involve relatively simple structural members. The models will then increase in complexity until they represent a PCPV or major sections of a PCPV.

The initial structural model test series, which is nearing completion, was selected to be a study of the relative behavior of grouted vs nongrouted tendons for application to PCPVs. The experimental investigation has been divided into four phases: (1) structural behavior, (2) new materials, (3) corrosion behavior, and (4) tendon monitoring techniques (acoustic emission).

3.4.1 Structural Behavior

The relative structural performance (static, dynamic, and cyclic) of grouted and nongrouted post-tensioned structures is being evaluated by testing in flexure simple beam members 0.15-m wide by 0.30-m deep by 3.1-m long such as shown in Fig. 3.32. The beams were cast in a steel mold from the concrete mix design presented in Table 3.8. Specimen fabrication included: placement of shear reinforcement and tendon (also conduit for grouted beams) in the molds, application of sufficient tension to the tendon so that it remained straight during the casting process, concrete placement in two layers with compaction by vibration after each layer, leveling of the exposed surface, casting of control specimens according to appropriate ASTM standards, and covering specimens with wet absorbent paper and plastic after the concrete had attained initial set. The specimens were removed from their molds 48 to 72 hr after casting and stored in the laboratory environment. After curing for approximately three months the beams were post-tensioned to the desired level of either 0.5, 0.6, or 0.7 times steel strand ultimate strength. Beams to be grouted were then grouted. Grouted and nongrouted tendon beam structural behavior test variables are summarized in Table 3.9. Plastic concrete properties, hardened concrete

DIMENSIONS IN INCHES

Fig. 3.32. Beam Structural Test Member Geometry. To convert dimensions to meters, multiply by 0.0254.

Table 3.8. Concrete Mix Design for Grouted and Nongrouted Tendon Study

Material	Size Range	Mix Proportion (wt %)
Cement		16.37
Sand	< No. 4	33.55
Gravel	No. 4 to 20 mm	43.12
Water		6.96

properties, post-tensioning levels, and test results for the grouted and nongrouted beams used to evaluate relative performance are summarized in Table 3.10.

Static flexure testing of the beam structural elements used the test fixture presented in Fig. 3.33. The test procedure consisted of: calibrating and zeroing load and displacement transducers, applying load at a rate of 74 N/s until first cracking occurred, holding load constant while the extent of crack propagation was marked, and continuing loading in 74-N increments until failure occurred, with crack extent marked at each load increment. Centerline deflection was monitored throughout testing with a direct current differential transformer (DCDT). Results for the nongrouted and grouted tendon flexure tests are presented in Fig. 3.34. Results indicate that as the post-tensioning level was increased the load capacity of the beams increased. For the same level of post-tensioning, grouted tendon beams were stronger than nongrouted beams, and the grouted tendon beams developed more flexure cracks, but the cracks widths were much smaller.

Table 3.9. Grouted and Nongrouted Tendon Test Variables

Variable	Test parameter	Relevant test specimen	
		Grouted	Nongrouted
Load rate	74 N/s	G4	NG15
	0.74 kN/s	G10	
	7.4 kN/s	G5	NG12
	74 kN/s	G8	NG11
Prestressing level ^a	0.5 f'_s	G1	NG14
	0.6 f'_s	G4, G15, G24, G25, G26	NG15
	0.7 f'_s	G2	NG16
Fatigue ^b	0.10 to 0.50 P_u	G6	NG9
	0.10 to 0.70 P_u	G7	NG13

^a f'_s = ultimate tensile strength of the tendon.

^b P_u = static failure load.

Table 3.10. Grouted and Nongrouted Tendon Beam Mix Properties^a
and Test Results for Structural Behavior Test Series

Beam designation	Concrete properties					Posttensioning level (% of $f_s^{\wedge}$) ^b	Beam test results		
	Plastic			Hardened			Maximum load (kN)	Maximum centerline deflection (cm)	Type of failure
	Air content (vol %)	Slump (mm)	Unit weight (kg/m ³)	Ultimate strength (MPa)	Elastic modulus (GPa)				
Grouted tendon beams									
G1	2.30	64	2425	36.7	35.2	50	107.3	>2.5 ^c	Shear
G2	2.20	70	2425	35.6	31.9	69	110.6	>1.5 ^c	Steel strand
G3	2.40	48	2438	38.1	33.9	59	108.4	>1.9 ^c	Concrete crushing
G4	2.20	83	2435	38.9	32.0	65	108.4	>1.9 ^c	Concrete crushing
G5	2.30	70	2428	39.8	37.5	59	105.9	2.5	Concrete crushing
G6	2.00	89	2428	37.4	30.6	59	103.4	2.3	Concrete crushing
G7	2.40	64	2428	38.2	34.0	59	d	d	Steel strand
G8	2.00	95	2428	39.9	37.0	54	109.4	3.7	Concrete crushing
G10	2.40	76	2409	38.5	35.4	62	113.0	3.4	Concrete crushing
G15	2.75	44	2422	37.9	33.0	58	105.6	2.3	Concrete crushing
G24	2.60	25	2435	43.6	36.7	58	102.3	1.8	Concrete crushing
G25	1.60	38	2460	45.5	35.8	60	101.2	2.8	Concrete crushing
G26	2.50	25	2444	42.6	36.1	62	103.4	3.7	Concrete crushing
Nongrouted tendon beams									
NG9	2.40	38	2435	37.7	32.5	50	84.5	2.3	Concrete crushing
NG10	2.30	38	2416	38.2	35.5	62	82.8	2.7	e
NG11	2.55	38	2438	39.0	34.1	62	104.5	3.2	Concrete crushing
NG12	2.25	32	2435	39.7	33.9	61	96.7	4.6	Concrete crushing
NG13	2.30	32	2428	38.9	36.4	59	99.0	3.1	Concrete crushing
NG14	2.55	32	2435	38.3	35.8	49	89.4	>3.0 ^c	Concrete crushing
NG15	2.55	64	2393	31.0	28.8	61	97.0	>3.2 ^c	Concrete crushing
NG16	2.65	38	2419	34.3	30.7	66	95.2	>2.4 ^c	Concrete crushing

^aThe concrete batch weights for cement, sand (SSD), gravel (SSD) and water were 79.4, 162.7, 209.1, and 33.8 kg, respectively.

^b f'_s = ultimate tensile strength of the tendon.

^cDeflection exceeds range of displacement gage.

^dNot applicable; fatigue test.

^eDropped while placing in test fixture.

Fig. 3.33. Flexure Test Setup.

Dynamic flexure tests were conducted with the same test setup as the static flexure tests. Both grouted and nongrouted beams with prestressing steel post-tensioned to 0.6 times ultimate were tested. The test procedure was identical to the static flexure tests except loading rates of 0.74, 7.4, and 74 kN/s were used and cracks were not marked. Figure 3.35 presents results of the dynamic flexure tests for the nongrouted and grouted tendon beams. Results indicated no significant difference (<8%) in ultimate moment capacities for either grouted or nongrouted tendon beams for the range of loading rates investigated. We anticipated that the moment capacity would increase as the load rate increased, but this trend was not apparent because the stiffness and capacity of the test system were apparently not sufficient to apply a truly dynamic loading effect, and results were thus still within the static domain. However, the beam centerline deflection at ultimate load tended to increase as the loading rate increased for both types of beams.

ORNL-DWG 77-3423

(a)

ORNL-DWG 77-3426

(b)

Fig. 3.34. Static Flexural Test Results on (a) Nongrouted and (b) Grouted Tendons.

Fig. 3.35. Dynamic Flexure Test Results. (a) Nongrouted and (b) grouted tendon beams.

Flexure fatigue loading of grouted and nongrouted tendon beams post-tensioned to 0.6 times steel ultimate strength was also tested with the static flexure test setup. The beams were loaded sinusoidally at a rate of 1 Hz either between 10 and 50% or 10 and 70% of the ultimate load capacity of the appropriate companion static flexure specimen. Each cyclic test was terminated when either the beam failed or 10^6 cycles had been applied. Neither the grouted nor the nongrouted tendon beams fatigued between 10 and 50% their static flexure capacity failed within 10^6 cycles, so the test was terminated and the beams were statically loaded (74 N/s) to failure. Failures occurred at 95 and 87% of the static flexure values for the grouted and nongrouted specimens, respectively. The grouted tendon beam fatigued between 10 and 70% of its static flexure value failed after 625,400 cycles by fracture of the prestressing strand. The corresponding nongrouted tendon beam did not fail within 10^6 cycles, so the test was terminated and the beam statically loaded to failure, which occurred at 102% of the static flexure value.

3.4.2 New Materials

An overview is being conducted of three material systems that are relatively new to PCRV applications. Steel-fiber-reinforced concrete is being evaluated as a potential structural material because of its increased flexure strength, ductility (energy absorption), and resistance to penetration relative to conventional concrete. An inorganic polymer corrosion-resistant cement,* which has desirable properties of rapid strength development, good bond to minerals, nonshrinking, and high temperature capability ($>1090^{\circ}\text{C}$), is being evaluated for application as a grout for prestressing steel and for elevated-temperature uses. Expansive cement, which is designed to compensate for tensile stresses produced during drying shrinkage of concrete, is being investigated as a potential grout material. A summary of the test variables for the study of these new materials is presented in Table 3.11.

*Developed at Southwest Research Institute.

Table 3.11. Test Variables for Study of New Materials

Variable	Test parameter ^a	Relevant test specimens			
		Grouted beam	Nongrouted beam	Bond pull-out	5.1 cm cubes
Posttensioning level (grout material)	0.5 f'_s PS	G19, ^b G21, ^c G27 ^d			
	0.5 f'_s SC	G16			
	0.5 f'_s CG	G1			
	0.5 f'_s CNG		NG14		
	0.6 f'_s PS	G11, ^b G14, ^b G22, ^c G28 ^d			
	0.6 f'_s SC	G18			
	0.6 f'_s CG	G15, G24-G26			
	0.6 f'_s CNG		NG15		
	0.7 f'_s PS	G12, ^b G23, ^c G29 ^d			
	0.7 f'_s SC	G13			
	0.7 f'_s CG	G2			
	0.7 f'_s CNG		NG16		
Bond development length — failed anchorage	PS	G20			
	SC	G17			
	GC	G9			
Tendon bond strength	0.5 f'_s PS			3 specimens	
	0.5 f'_s SC			3 specimens	
	0.5 f'_s GC			3 specimens	

Table 3.11. (Continued)

Variable	Test parameter ^a	Relevant test specimens			
		Grouted beam	Nongrouted beam	Bond pull-out	5.1 cm cubes
Tendon bond strength (contd.)	0.6 f'_s PS			3 specimens	
	0.6 f'_s SC			3 specimens	
	0.6 f'_s CG			3 specimens	
	0.7 f'_s PS			3 specimens	
	0.7 f'_s SC			3 specimens	
	0.7 f'_s CG			3 specimens	
Temperature	Room temp.				2 specimens Mixes 1 & 2
	260°C				5 specimens Mix 2
	540°C				5 specimens Mix 1
	816°C				5 specimens Mix 2
	1093°C				5 specimens Mix 1
Structural material	0.5 f'_s FC	FC9	FC5		
	0.5 f'_s CG	G1			
	0.5 f'_s CNG		NG14		

Table 3.11. (Continued)

Variable	Test parameter ^a	Relevant test specimens			
		Grouted beams	Nongrouted beam	Bond pull-out	5.1 cm cubes
Structural material (contd.)	0.6 f'_s FC	FC3, FC10	FC1, FC6, FC8		
	0.6 f'_s CG	G4			
	0.6 f'_s CNG		NG15		
	0.7 f'_s FC	FC4, FC11	FC2, FC7		
	0.7 f'_s CG	G2			
	0.7 f'_s CNG		NG16		

^aPS = polymer silica cements, SC - shrinkage compensating cement;
CG = control, grouted; CNG = control, nongrouted; FC = fibrous concrete.
 f'_s = ultimate tensile strength of tendon.

^bPolymer silica material A: material not up to specifications.

^cPolymer silica material B: material used in laboratory but too viscous for field grouting using required water content.

^dPolymer silica material C: material formulated for field grouting.

3.4.2.1 Fiber Reinforced Concrete

Fiber-reinforced concrete consists of hydraulic cements containing fine aggregate, or fine and coarse aggregates, and discontinuous, discrete fibers, which are blended into the concrete during the mix cycle. The addition of the fibers increases the concrete's strength, ductility, and resistance to dynamic loadings. Fibrous concrete is being evaluated as a structural material by casting, testing, and comparing flexure test results obtained from 0.15-m wide by 0.30-m deep by 3.1-m long post-tensioned grouted and nongrouted tendon beams with those obtained for the conventional concrete beams. Test results are summarized in Table 3.12 for the fibrous concrete beams. Ultimate loads were similar for the fibrous concrete beams and the conventional beams. However, beam ductility approximately doubled with the addition of the steel fibers. Also the fiber addition changed the mode of failure from concrete crushing to steel strand failure.

3.4.2.2 Polymer Silica Cements

The polymer silica corrosion-resistant cements are inorganic polymers that chemically set upon the addition of water to form materials with purported advantages relative to conventional material systems based on portland cement. Among their advantages are rapid strength gain, acid resistance, strength retention at elevated temperature, resistance to thermal shock, nonshrinking, and excellent bond to steels. These materials are being investigated for application as both grout and liner materials.

Performance of these materials as a potential grout for prestressing strand is being evaluated by conducting tendon bond strength and flexure tests with the polymer silica material used as the grout. The tendon bond strength tests will be conducted with test specimens such as shown in Fig. 3.36. These specimens were fabricated by placing three steel-encased annular concrete specimens having sections 737 mm long, 152 mm OD, and 32 mm ID in the reaction fixture shown in Fig. 3.37, placing a prestressing strand through the longitudinal holes in the specimens, attaching grouting fixtures to the specimen ends, tensioning the strand to the desired prestressing level of either 0.5, 0.6, or 0.7 f'_g , grouting the strand in place, curing the grout for 7 days, detensioning, and removing

Table 3.12. Grouted and Nongrouted Tendon Beam Mix Properties^a
and Test Results for New Materials Test Series

Beam Designation	Plastic			Hardened		Posttensioning level (% f_s^c) ^b	Beam test results		
	Air content (vol %)	Slump (mm)	Unit weight (kg/m ³)	Ultimate strength (MPa)	Elastic modulus (GPa)		Maximum load (kN)	Maximum centerline deflection (cm)	Type failure
Grouted tendon beams									
G9	2.00	76	2444	40.3	36.5	63	55.6	>5.1 ^c	Steel strand pullout
G11	2.75	76	2403	36.8	32.0	61	102.9	2.7	Concrete crushing
G12	2.70	51	2406	31.3	31.6	69	105.9	3.0	Concrete crushing
G13	2.90	44	2416	34.5	32.2	69	118.4	2.4	Concrete crushing
G14	2.90	32	2412	35.0	32.9	60	99.5	2.6	Concrete crushing
G16	2.50	38	2438	37.1	29.6	49	111.8	3.1	Shear
G17	2.65	25	2435	39.8	35.7	58	63.4	>2.0 ^c	Steel strand pullout
G18	2.35	32	2419	38.0	33.9	58	118.4	3.2	Concrete crushing
G19	1.85	89	2428	37.8	35.0	50	96.7	3.6	Concrete crushing
G20	1.75	127	2435	39.3	38.2	62	100.6	2.7	Concrete crushing
G21	2.70	38	2422	45.4	35.7	53	101.2	2.4	Concrete crushing ^d
G22	2.25	64	2454	45.6	36.0	61	104.0	3.8	Concrete crushing
G23	1.90	57	2448	42.7	34.1	70	94.5	1.8	Concrete crushing
G27	2.40	25	2448			<i>e</i>	106.8	3.2	Concrete crushing
G28	2.45	32	2435			<i>e</i>	100.6	2.3	Concrete crushing
G29	2.70	19	2441			<i>e</i>	105.6	3.2	Concrete crushing
FC3	3.60	57	2153	17.9	12.3	59	97.9	>2.0 ^c	Concrete crushing
FC4	4.10	13	2191	20.2	13.5	69	106.2	>1.9 ^c	Concrete crushing
FC9	2.50	51	2387	38.4	28.7	51	103.4	>5.1 ^c	Steel strand fracture
FC10	3.00	51	2390	33.5	26.2	60	107.3	>5.1 ^c	Steel strand fracture
FC11	3.05	48	2390	47.0	32.3	73	105.1	3.8	Steel strand fracture
Nongrouted tendon beams									
FC1	3.40	38	2163	16.2	10.5	61	82.3	>5.0 ^c	Concrete crushing
FC2	3.50	29	2179	21.9	15.3	69	102.8	>2.3 ^c	Concrete crushing
FC5	2.65	108	2319	31.2	22.8	50	92.3	>5.1 ^c	Concrete crushing
FC6	2.30	89	2371	30.9	21.9	61	92.3	>5.1 ^c	Concrete crushing
FC7	2.70	89	2348	29.1	24.3	71	81.2	1.4	Concrete crushing
FC8	1.95	64	2422	37.7	30.6	59	96.7	>5.1 ^c	Concrete crushing

^aThe plain concrete and fibrous concrete batch weights for cement, sand (SSD), gravel (SSD), and water were 79.4, 162.7, 209.1, and 33.8 kg, and 83.9, 251.7, 83.9, and 50.3 kg, respectively. The fibrous concrete beams also contained 1.5% by volume of 2.54-cm-long steel fibers.

^b f_s^* = ultimate tensile strength of the tendon.

^cDeflection exceeds range of displacement gage.

^dTendon not straight in beam.

^eBeam cast but not tested.

Photo 6048-78

Fig. 3.36. Tendon Bond Strength Test Specimen.

Fig. 3.37. Tendon Bond Strength Reaction Test Fixture.

specimens. When these specimens are tested, bond strength results will be compared with those of a companion series fabricated with conventional grout. The polymer silica material for grout applicability is also being evaluated by comparing results obtained from 0.15-m-wide by 0.30-m-deep by 3.1-m-long beam structural elements in which the tendons were grouted with polymer silica with results obtained from beams grouted with the conventional grout. The first series of specimens produced flexure ultimate load capacities of -9, -5, and -4% relative to standard grouted beams with prestressing levels of 0.5, 0.6, and $0.7 f'_g$, respectively. These reduced strengths were probably the result of low grout strength obtained when the water content of the polymer silica grout had to be increased above that recommended by the manufacturer so that the grout was pumpable. A more flowable grout formulation has been obtained from the manufacturer, and new beams have been fabricated for testing.

Prestressing bond transfer lengths for the polymer silica cements have also been determined for comparison with conventional grout. The test procedure consisted of fabricating a standard 0.15-m-wide by 0.30-m-deep by 3.1-m-long beam structural element, permitting the beam to cure, post-tensioning the beam tendon to $0.6 f'_s$ by the same technique as for previous beams except that a bracket was included between the end of the beam and the anchorage providing access to the strand, grouting the beam, permitting the grout to cure, epoxying mechanical gage points along the bottom surface of the beam at 51-mm increments, slowly cutting the steel tendon, and determining the length of beam required for strains (length changes) to become uniform. This length is the prestressing transfer length. The prestressing transfer length obtained for the polymer silica grouted beam was approximately 0.15 m, as compared with 0.76 m obtained for the conventionally grouted beam. The beam was then tested in flexure, and the load capacity improved 81% relative to the conventionally grouted beam with an anchorage removed. In addition, the mode of beam failure was changed from tendon pullout, which occurred in the conventionally grouted beam, to concrete compression failure.

A second formulation of the polymer silica material for elevated-temperature applications has also been obtained for evaluation. Two mixes of twelve 51-mm cubes each were fabricated from the material supplied. Five cubes from mix 1 have been placed in one oven, where the temperature is maintained at 260°C, and an additional set of 5 cube specimens has been placed in a second oven at 816°C. Similarly five-cube sets of mix 2 specimens have been placed in ovens maintained at 540 and 1093°C. Periodically specimens will be removed from each of the ovens, permitted to cool to room temperature, and tested to failure in compression. Results will be compared with the strengths of the remaining two specimens of each mix, which are maintained at room temperature.

3.4.2.3 Shrinkage-Compensating Cements

Shrinkage-compensating cements are designed to compensate for the tensile stresses produced during drying shrinkage of concrete. If proper design, construction, and quality control procedures are exercised these materials can eliminate or reduce drying shrinkage cracks. One particular

brand of shrinkage-compensating cement has been obtained for evaluation as a grout for prestressing. Three beam structural models 0.15 m wide by 0.30 m deep by 3.1 m long were post-tensioned to 0.5, 0.6, and 0.7 f'_s and grouted with the shrinkage compensating cement. Ultimate load capacity improved by 4, 9, and 7%, respectively, relative to the conventionally grouted beams post-tensioned to the same levels. One additional beam specimen was fabricated to indicate prestressing transfer length for the cement. Test procedures were the same as for the polymer-silica-grouted specimen. The prestressing transfer length obtained was 0.51 m vs 0.76 m for the conventional grout thus indicating improved bond strength. The flexure specimen was then loaded to failure, and although both the shrinkage-compensated cement and conventionally grouted beams failed by tendon pullout, the shrinkage-compensating cement grouted beam exhibited a 15% improvement in ultimate load capacity relative to the conventionally grouted beam. Specimens have also been fabricated to evaluate tendon bond strengths when the tendons are grouted with shrinkage-compensating cement.

3.4.3 Corrosion Behavior

A bench-scale study is currently being conducted on the corrosion protection provided tendons by grouting. This study has been broken down into two phases: (1) stressed tendons and (2) nonstressed tendons. Investigations in each of these areas are designed to complement a corrosion study currently being conducted in the ORNL Metals and Ceramics Division.

3.4.3.1 Stressed Tendons

Test specimens are the central straight wire of a seven-wire strand for nongrouted tendons. The specimens are prepared by degreasing the wire with acetone, inspecting for flaws, applying (if required for the particular test) the corrosion-inhibiting compound* to the wire surface within the gage length, placing the wire through a polyethylene bottle, filling the

*These commercially available corrosion-inhibiting compounds are of the type that would be used for protection of circumferential prestressing of an HTGR and for protection of secondary containments in PWRs. Grout and grout containing known flaws were also investigated.

bottle with demineralized water, slowly bubbling hydrogen sulfide gas* through the water for approximately 15 min, sealing the bottle with a rubber stopper, placing the specimen in the testing machine, loading the specimen to the desired load level (general range of interest was $0.5-0.7 f_g$), and maintaining the load at this level. If the specimen did not fail within 24 hr the specimen was unloaded, the water resaturated with hydrogen sulfide, and the specimen reloaded to the previous level. This procedure was repeated until either the specimen failed or six days lapsed. If failure did not occur due to corrosion within this time interval the specimen was removed from its corrosive environment and loaded to failure. Figure 3.38 presents the stressed tendon corrosion test setup. Results obtained to date are summarized in Table 3.13.

3.4.3.2 Nonstressed Tendons

A companion test series of specimens, also using the central straight strand of a seven-wire strand for nongROUTED tendons, has also been conducted. In this test series the tendons were not stressed while subjected to the corrosive environment. Four corrosive environments were used: water saturated with hydrogen sulfide, $0.2 M NO_3^-$ at $66^\circ C$, $0.1 M Cl^-$, and the outdoor environment of Oak Ridge.[†] Variables in the test series were type of corrosion inhibiting agent (grout with or without flaws and commercial products) and length of exposure. The test procedure consisted of removal of specimen sets from the appropriate corrosive environment after exposure times of 30 to 132 days and testing the specimens to failure in tension. Effectiveness of the various corrosion inhibiting compounds was judged on ultimate loads and times to failure for the protected wires relative to nonprotected companion wire sections. Results obtained for wires subjected to the four media are summarized in Tables 3.14 through 3.17.

* Hydrogen sulfide saturated water was the only corrosive medium investigated because the NO_3^- and Cl^- solutions would not produce corrosion-induced failures within a reasonable time frame.

[†] Only unprotected wire was considered.

Fig. 3.38. Stressed Tendon Corrosion Test Setup.

Table 3.13. Stressed Tendon Corrosion Test Result Summary

Specimen	Load (% of ultimate)	Corrosion inhibiting compound	Corrosive solution	Failure time (hr)	Failure load (kN)
1	<i>a</i>	None	None		28.11
2	<i>a</i>	None	None		28.29
3	<i>a</i>	None	None		28.25
4	<i>a</i>	None	None		28.47
5	<i>a</i>	None	None		28.65
6	<i>a</i>	None	None		28.65
7	<i>a</i>	None	None		28.65
8	<i>a</i>	None	None		28.91
9	<i>a</i>	None	None		28.58
10	<i>a</i>	None	None		28.80
11	<i>a</i>	None	None		28.80
12	<i>a</i>	None	None		28.87
13	<i>a</i>	None	None		29.00
31	80	None	H ₂ S + H ₂ O	1.94	22.89
32	80	None	H ₂ S + H ₂ O	2.53	22.89
33	80	None	H ₂ S + H ₂ O	1.79	22.89
14	75	None	H ₂ S + H ₂ O	2.38	21.46
15	75	None	H ₂ S + H ₂ O	0.72	21.46
17	75	None	H ₂ S + H ₂ O	0.78	21.46
18	75	None	H ₂ S + H ₂ O	0.67	21.46
19	70	None	H ₂ S + H ₂ O	2.38	20.03
20	70	None	H ₂ S + H ₂ O	1.70	20.03
29	70	None	H ₂ S + H ₂ O	1.20	20.03
36	60	None	H ₂ S + H ₂ O	2.07	17.17
37	60	None	H ₂ S + H ₂ O	1.18	17.17
38	60	None	H ₂ S + H ₂ O	1.30	17.17
39	60	None	H ₂ S + H ₂ O	2.10	17.17
26	70	2889 ^b	H ₂ S + H ₂ O	144.00 ^c	27.36
28	70	2090-P4 ^b	H ₂ S + H ₂ O	161.00 ^c	27.58
34	80	1601 Amber ^b	H ₂ S + H ₂ O	143.00 ^c	29.36
35	80	2090-P4 ^b	H ₂ S + H ₂ O	137.00 ^c	29.67
41	60	2889 ^b	H ₂ S + H ₂ O	160.00 ^c	17.17
43	60	Grout	H ₂ S + H ₂ O	152.00 ^c	17.17
44	60	None	0.2 M NO ₃ ⁻	174.00 ^c	30.92
50	60	Grout	H ₂ S + H ₂ O	169.00 ^c	
52	70	Grout	H ₂ S + H ₂ O	167.00 ^c	28.36
61	60	Grout with 3.2-mm flaw	H ₂ S + H ₂ O	35.3	17.17
62	60	Grout with 3.2-mm flaw	H ₂ S + H ₂ O	33.4	17.17
68	60	Grout with 3.2-mm flaw	H ₂ S + H ₂ O	58.3	17.17
75	60	Grout with 3.2-mm flaw	H ₂ S + H ₂ O	38.5	17.17

Table 3.13. (Continued)

Specimen	Load (% of ultimate)	Corrosion inhibiting compound	Corrosive solution	Failure time (hr)	Failure load (kN)
78	60	Grout with 3.2-mm flaw	H ₂ S + H ₂ O	7.51	17.17
63	60	Grout with 1.6-mm flaw	H ₂ S + H ₂ O	73.30	17.17
71	60	Grout with 1.6-mm flaw	H ₂ S + H ₂ O	54.70	17.17
73	60	Grout with 1.6-mm flaw	H ₂ S + H ₂ O	49.50	17.17
76	60	Grout with 1.6-mm flaw	H ₂ S + H ₂ O	27.50	17.17
77	60	Grout with 1.6-mm flaw	H ₂ S + H ₂ O	24.30	17.17
66	60	Grout with 0.8-mm flaw	H ₂ S + H ₂ O	172.00 ^c	17.17
67	60	Grout with 0.8-mm flaw	H ₂ S + H ₂ O	120.00	17.17
70	60	Grout with 0.8-mm flaw	H ₂ S + H ₂ O	232.00 ^c	27.91
72	60	Grout with 0.8-mm flaw	H ₂ S + H ₂ O	231.00 ^c	26.13
74	60	Grout with 0.8-mm flaw	H ₂ S + H ₂ O	212.00 ^c	27.25
69	60	Grout with 0.3-mm flaw	H ₂ S + H ₂ O	197.00 ^c	26.69

^aNot applicable, control specimen.

^bViscosity Oil Company.

^cNo failure in less than six days, specimen then loaded to failure.

Table 3.14. Nonstressed Tendon Corrosion Test
Result Summary ($H_2S + H_2O$)

Specimen	Corrosion inhibiting compound	Length of exposure (days)	Failure location ^a	Failure load (kN)	Failure time (min)
1	None	33	GL	26.80	17.8
2	None	33	GL	26.24	11.8
3	None	33	GL	25.80	10.0
4	2889 ^b	33	G	28.02	35.0
5	2889 ^b	33	G	27.69	32.3
6	2889 ^b	33	G	27.80	35.3
7	2090-P4 ^b	33	G	28.02	31.8
8	2090-P4 ^b	33	G	28.02	33.5
9	2090-P4 ^b	33	G	27.91	32.5
10	1601 Amber ^b	33	G	27.80	33.3
11	1601 Amber ^b	33	G	27.91	33.3
12	1601 Amber ^b	33	G	27.80	34.5
13	Grout (t $\approx$ 15.9 mm)	33	G	28.02	34.3
14	Grout (t $\approx$ 15.9 mm)	33	GL	28.02	31.8
15	Grout (t $\approx$ 15.9 mm)	33	G	27.80	31.3
16	None	77	GL	8.67	2.5
17	None	77	GL	24.90	9.5
18	None	77	GL	27.80	18.0
19	2889 ^b	77	GL	27.02	28.8
20	2889 ^b	77	GL	27.47	32.0
21	2889 ^b	77	GL	28.47	34.8
22	2090-P4 ^b	77	GL	26.58	35.5
23	2090-P4 ^b	77	G	28.80	36.0
24	2090-P4 ^b	77	G	28.80	36.3
25	1601 Amber ^b	77	GL	27.80	34.0
26	1601 Amber ^b	77	G	27.47	37.5
27	1601 Amber ^b	77	GL	28.91	31.3
28	Grout (t $\approx$ 15.9 mm)	77	GL	27.36	31.0
29	Grout (t $\approx$ 15.9 mm)	77	GL	27.91	32.9
30	Grout (t $\approx$ 15.9 mm)	77	G	28.80	34.8
	Control	c	G	28.80	34.5
31	None	120	GL	24.69	9.0
32	None	120	GL	25.47	11.5
33	None	120	GL	25.91	10.8

Table 3.14. (Continued)

Specimen	Corrosion inhibiting compound	Length of exposure (days)	Failure location ^a	Failure load (kN)	Failure time (min)
36	2889 ^b	120	CE		
37	2090-P4 ^b	120	GL	28.58	31.5
38	2090-P4 ^b	120	GL	28.47	32.5
39	2090-P4 ^b	120	GL	28.91	25.5
40	1601 Amber ^b	120	GL	22.91	8.3
41	1601 Amber ^b	120	GL	24.47	11.3
42	1601 Amber ^b	120	GL	26.24	10.0
43	Grout (t ≈ 15.9 mm)	120	GL	28.36	30.8
44	Grout (t ≈ 15.9 mm)	120	GL	28.36	30.0
45	Grout (t ≈ 15.9 mm)	120	GL	28.58	31.0
46	Polymer silica (t ≈ 7.9 mm)	30	GL	28.47	32.8
47	Polymer silica (t ≈ 7.9 mm)	30	GL	28.47	27.5
48	Polymer silica (t ≈ 15.9 mm)	30	GL	28.25	32.5
49	Polymer silica (t ≈ 15.9 mm)	30	GL	28.25	27.0
50	Polymer silica (t ≈ 7.9 mm)	74	GL	28.14	20.8
51	Polymer silica (t ≈ 7.9 mm)	74	GL	27.91	17.5
52	Polymer silica (t ≈ 15.9 mm)	74	GL	28.47	27.0
53	Polymer silica (t ≈ 15.9 mm)	74	GL	28.36	20.0
54	Polymer silica (t ≈ 7.9 mm)	127	GL	26.69	21.3
55	Polymer silica (t ≈ 7.9 mm)	127	GL	26.24	25.5
56	Polymer silica (t ≈ 15.9 mm)	127	GL	25.58	24.3
57	Polymer silica (t ≈ 15.9 mm)	127	GL	25.69	24.3
58	Grout with 0.01-mm flaw	35	GL	28.47	37.8
59	Grout with 0.03-mm flaw	35	G	28.36	33.5

Table 3.14. (Continued)

Specimen	Corrosion inhibiting compound	Length of exposure (days)	Failure location ^a	Failure load (kN)	Failure time (min)
60	Grout with 0.05-mm flaw	35	G	28.80	33.8
61	Grout with 0.10-mm flaw	35	G	28.47	36.8
62	Grout with 0.25-mm flaw	35	G	28.58	36.5
63	Grout with 0.51-mm flaw	35	G	28.80	32.3
64	Grout with 0.76-mm flaw	35	G	28.47	34.3
65	2090-P4 ^b (scraped)	35	GL	27.62	18.5
66	2889 ^b (scraped)	35	GL	27.13	12.0
67	None	35	GL	19.79	5.8
68	Grout with 0.01-mm flaw	119	GL	26.71	33.3
69	Grout with 0.03-mm flaw	119	GL	27.13	33.5
70	Grout with 0.05-mm flaw	119	GL	26.82	34.8
71	Grout with 0.10-mm flaw	119	GL	26.60	34.3
72	Grout with 0.25-mm flaw	119	GL	26.60	32.5
73	Grout with 0.51-mm flaw	119	GL	26.13	32.8
74	Grout with 0.76-mm flaw	119	GL	25.58	32.0
75	2889 ^b (scraped)	119	GL	23.91	10.5
76	2090-P4 ^b (scraped)	119	GL	24.24	12.5
77	None	119	GL	20.57	7.0
	Control	^c	G	27.36	35.3

^aG = grips; GL = gage length.

^bViscosity Oil Company.

^cControl specimen not subjected to corrosive environment.

Table 3.15. Nonstressed Tendon Corrosion Test Result Summary
(0.2 M NO_3^- at 66°C)

Specimen	Corrosion inhibiting compound	Length of exposure (days)	Failure location ^a	Failure load (kN)	Failure time (min)
1	None	42	GL	25.47	13.8
2	None	42	GL	23.91	10.3
3	None	42	GL	24.35	10.5
4	2889 ^b	42	G	27.02	39.0
5	2889 ^b	42	G	27.69	36.0
6	2889 ^b	42	G	27.25	33.5
7	2090-P4 ^b	42	G	27.02	34.3
8	2090-P4 ^b	42	G	27.58	36.5
9	2090-P4 ^b	42	G	27.13	34.5
10	1601 Amber ^b	42	G	26.58	36.3
11	1601 Amber ^b	42	G	27.58	36.8
12	1601 Amber ^b	42	G	28.02	35.3
13	Grout (t $\approx$ 7.9 mm) ^c	42	GL	26.91	24.0
14	Grout (t $\approx$ 7.9 mm) ^c	42	GL	28.91	23.3
15	Grout (t $\approx$ 9.5 mm) ^c	42	GL	26.69	24.3
16	Grout (t $\approx$ 9.5 mm) ^c	42	GL	28.91	22.5
17	Grout (t $\approx$ 15.9 mm) ^c	42	GL	26.80	24.8
18	Grout (t $\approx$ 15.9 mm) ^c	42	GL	27.69	19.8
19	Grout (t $\approx$ 15.9 mm) ^c	42	GL	26.24	21.8
	Control	d	G	28.80	34.5
20	None	78	GL	26.58	13.8
21	None	78	GL	26.47	13.3
22	None	78	GL	27.25	17.0
23	2889 ^b	78	G	28.36	34.5
24	2889 ^b	78	G	28.36	35.8
25	2889 ^b	78	G	28.36	32.0
26	2090-P4 ^b	78	G	28.36	37.3
27	2090-P4 ^b	78	G	28.36	37.3
28	2090-P4 ^b	78	G	28.47	36.8
29	1601 Amber ^b	78	G	28.47	37.8
30	1601 Amber ^b	78	G	28.47	34.0
31	1601 Amber ^b	78	G	28.47	32.8
32	Grout (t $\approx$ 7.9 mm) ^c	78	GL	25.47	9.5
33	Grout (t $\approx$ 7.9 mm) ^c	78	GL	27.02	19.5

Table 3.15. (Continued)

Specimen	Corrosion inhibiting compound	Length of exposure (days)	Failure location ^a	Failure load (kN)	Failure time (min)
34	Grout (t $\approx$ 7.9 mm) ^c	78	GL	28.47	20.5
35	Grout (t $\approx$ 9.5 mm) ^c	78	GL	25.91	10.3
36	Grout (t $\approx$ 9.5 mm) ^c	78	GL	27.57	20.5
37	Grout (t $\approx$ 9.5 mm) ^c	78	GL	27.13	15.8
38	Grout (t $\approx$ 15.9 mm) ^c	78	GL	27.91	23.0
39	Grout (t $\approx$ 15.9 mm) ^c	78	GL	27.80	21.5
40	None	132	GL	25.58	10.3
41	None	132	GL	27.13	15.0
42	None	132	GL	25.02	9.8
43	2889 ^b	132	G	28.69	38.5
44	2889 ^b	132	G	28.47	35.0
45	2889 ^b	132	G	28.80	33.3
45	2090-P4 ^b	132	G	28.69	32.8
47	2090-P4 ^b	132	G	28.36	36.0
48	2090-P4 ^b	132	G	28.36	34.0
49	1601 Amber ^b	132	GL	27.69	25.3
50	1601 Amber ^b	132	G	28.58	33.5
51	1601 Amber ^b	132	G	28.47	35.3
52	Grout (t $\approx$ 7.9 mm) ^c	132	GL	28.14	25.3
53	Grout (t $\approx$ 7.9 mm) ^c	132	GL	26.24	11.0
54	Grout (t $\approx$ 9.5 mm) ^c	132	GL	27.91	23.5
55	Grout (t $\approx$ 9.5 mm) ^c	132	GL	27.13	16.8
56	Grout (t $\approx$ 15.9 mm) ^c	132	GL	26.69	14.3
57	Grout (t $\approx$ 15.9 mm) ^c	132	GL	26.69	15.0
58	Grout (t $\approx$ 15.9 mm) ^c	132	GL	26.47	14.3
59	Polymer silica (t $\approx$ 9.5 mm)	34	GL	28.58	28.8
60	Polymer silica (t $\approx$ 9.5 mm)	34	GL	28.69	29.5

Table 3.15. (Continued)

Specimen	Corrosion inhibiting compound	Length of exposure (days)	Failure location ^a	Failure load (kN)	Failure time (min)
61	Polymer silica (t ≈ 15.9 mm)	34	GL	28.91	34.0
62	Polymer silica (t ≈ 15.9 mm)	34	GL	28.69	26.3
63	Grout (t ≈ 7.9 mm)	38	GL	28.69	24.5
64	Grout (t ≈ 7.9 mm)	38	GL	28.58	26.0
65	Grout (t ≈ 7.9 mm)	38	GL	28.58	26.8
66	Grout (t ≈ 9.5 mm)	38	GL	28.80	28.0
67	Grout (t ≈ 9.5 mm)	38	GL	28.69	30.8
68	Grout (t ≈ 9.5 mm)	38	GL	28.80	29.5
69	Grout (t ≈ 15.9 mm)	38	G	28.69	35.3
70	Grout (t ≈ 15.9 mm)	38	G	28.58	35.5
71	Grout (t ≈ 15.9 mm)	38	G	28.58	33.3
72	Grout (t ≈ 7.9 mm)	87	GL	28.47	21.3
73	Grout (t ≈ 7.9 mm)	87	GL	28.14	23.3
74	Grout (t ≈ 7.9 mm)	87	GL	27.69	22.8
75	Grout (t ≈ 9.5 mm)	87	GL	28.14	22.5
76	Grout (t ≈ 9.5 mm)	87	GL	27.69	23.0
77	Grout (t ≈ 9.5 mm)	87	GL	27.91	22.3
78	Grout (t ≈ 15.9 mm)	87	GL	28.14	28.3
79	Grout (t ≈ 15.9 mm)	87	GL	27.69	26.0
80	Grout (t ≈ 15.9 mm)	87	GL	28.58	27.3
81	Grout (t ≈ 7.9 mm)	122	GL	26.24	18.8

Table 3.15. (Continued)

Specimen	Corrosion inhibiting compound	Length of exposure (days)	Failure location ^a	Failure load (kN)	Failure time (min)
82	Grout (t $\approx$ 7.9 mm)	122	GL	25.80	19.5
83	Grout (t $\approx$ 7.9 mm)	122	GL	26.36	22.0
84	Grout (t $\approx$ 9.5 mm)	122	GL	26.24	20.0
85	Grout (t $\approx$ 9.5 mm)	122	GL	26.13	21.0
86	Grout (t $\approx$ 9.5 mm)	122	GL	26.24	22.8
87	Grout (t $\approx$ 15.9 mm)	122	GL	26.24	24.3
88	Grout (t $\approx$ 15.9 mm)	122	GL	26.47	26.0
89	Grout (t $\approx$ 15.9 mm)	122	GL	26.70	25.3
	Control	<i>d</i>	G	26.47	33.3
	Control	<i>d</i>	G	27.91	35.0
90	None	33	GL	27.25	18.3
91	None	33	GL	27.25	19.8
92	None	33	GL	27.25	20.5
93	Polymer silica (t $\approx$ 15.9 mm)	33	GL	28.47	33.5
94	Polymer silica (t $\approx$ 15.9 mm)	33	GL	28.36	31.0
95	Polymer silica (t $\approx$ 9.5 mm)	33	GL	28.58	28.8
96	Polymer silica (t $\approx$ 9.5 mm)	33	GL	28.47	29.5
97	2889 ^b (scraped)	33	G	28.47	35.0
98	2889 ^b (scraped)	33	GL	28.36	35.8
99	2090-P4 ^b (scraped)	33	G	28.47	34.0
100	2090-P4 ^b (scraped)	33	G	28.47	32.8
101	Grout with 0.03-mm flaw	33	G	28.47	33.0
102	Grout with 0.03-mm flaw	33	G	28.36	36.0
103	Grout with 0.13-mm flaw	33	G	28.47	35.3
104	Grout with 0.13-mm flaw	33	G	28.47	32.8

Table 3.15. (Continued)

Specimen	Corrosion inhibiting compound	Length of exposure (days)	Failure location ^a	Failure load (kN)	Failure time (min)
105	Grout with 0.25-mm flaw	33	G	28.47	35.0
106	Grout with 0.25-mm flaw	33	G	28.47	32.3
107	Grout with 0.76-mm flaw	33	G	28.47	34.5
108	Grout with 0.76-mm flaw	33	G	28.47	33.0
109	Grout with 3.18-mm flaw	33	GL	28.47	33.3
110	Grout with 3.18-mm flaw	33	G	28.36	32.3
111	None	84	GL	25.02	12.8
112	2889 ^b (scraped)	84	G	26.36	36.8
113	2090-P4 ^b (scraped)	84	G	26.69	32.8
114	Polymer silica (t $\approx$ 15.9 mm)	84	GL	27.13	27.5
115	Polymer silica (t $\approx$ 9.5 mm)	84	GL	27.13	26.5
116	Grout with 0.03-mm flaw	84	GL	27.13	27.5
117	Grout with 0.13-mm flaw	84	GL	27.25	27.3
118	Grout with 0.25-mm flaw	84	GL	27.58	26.3
119	Grout with 0.76-mm flaw	84	GL	27.36	24.5
120	Grout with 3.18-mm flaw	84	GL	25.58	19.5
	Control	d	G	28.02	31.5
121	Grout with 0.03-mm flaw	130	GL	26.36	25.0
122	Grout with 0.13-mm flaw	130	GL	26.47	21.8
123	Grout with 0.25-mm flaw	130	GL	25.80	23.0
124	Grout with 0.76-mm flaw	130	GL	25.69	22.3
125	Grout with 3.18-mm flaw	130	GL	25.91	19.3
126	2889 ^b (scraped)	130	G	26.47	36.0

Table 3.15. (Continued)

Specimen	Corrosion inhibiting compound	Length of exposure (days)	Failure location ^a	Failure load (kN)	Failure time (min)
127	2090-P4 ^b (scraped)	130	G	26.80	33.3
128	Polymer silica (t $\approx$ 15.9 mm)	130	GL	26.13	25.3
129	Polymer silica (t $\approx$ 9.5 mm)	130	GL	26.24	25.0
130	None	130	GL	24.27	11.6

^aG = grips, GL = gage length.

^bViscosity Oil Company.

^cGrout cracked and spalled due to heating-cooling and wetting-drying.

^dControl specimen not subjected to corrosive environment.

Table 3.16. Nonstressed Tendon Corrosion Test Result Summary
(0.1 M Cl⁻ at Room Temperature)

Specimen	Corrosion inhibiting compound	Length of exposure (days)	Failure location ^a	Failure load (kN)	Failure time (min)
1	None	30	GL	28.47	27.3
2	None	71	GL	27.58	17.8
3	None	71	GL	27.47	17.8
4	2889 ^b	71	G	28.69	36.0
5	2889 ^b	71	G	28.80	34.5
6	2889 ^b	71	GL	28.36	37.8
7	2090-P4 ^b	71	GL	28.58	35.3
8	2090-P4 ^b	71	GL	28.36	36.3
9	2090-P4 ^b	71	G	28.47	37.5
10	1601 Amber ^b	71	GL	28.69	39.0
11	1601 Amber ^b	71	G	28.47	34.5
12	1601 Amber ^b	71	G	28.47	34.0
13	Grout (t ≈ 7.9 mm)	71	G	28.58	37.0
14	Grout (t ≈ 7.9 mm)	71	G	28.47	35.5
15	Grout (t ≈ 7.9 mm)	71	GL	28.69	31.8
16	Grout (t ≈ 9.5 mm)	71	GL	28.58	38.0
17	Grout (t ≈ 9.5 mm)	71	G	28.36	38.0
18	Grout (t ≈ 9.5 mm)	71	G	28.58	37.5
19	Grout (t ≈ 15.9 mm)	71	GL	28.47	29.0
20	Grout (t ≈ 15.9 mm)	71	GL	28.36	30.5
21	Grout (t ≈ 15.9 mm)	71	GL	28.58	29.3
22	Polymer silica (t ≈ 15.9 mm)	71	GL	28.58	35.8
23	Polymer silica (t ≈ 15.9 mm)	71	GL	28.58	33.5
24	Polymer silica (t ≈ 15.9 mm)	71	GL	28.47	32.3
25	None	107	GL	26.02	12.3
26	None	107	GL	25.13	11.3
27	None	107	GL	26.02	18.0
28	2889 ^b	107	G	27.13	30.3
29	2889 ^b	107	GL	27.25	30.4

Table 3.16. (Continued)

Specimen	Corrosion inhibiting compound	Length of exposure (days)	Failure location ^a	Failure load (kN)	Failure time (min)
30	2889 ^b	107	GL	27.02	30.5
31	2090-P4 ^b	107	G	27.58	30.5
32	2090-P4 ^b	107	GL	26.47	26.0
33	2090-P4 ^b	107	G	26.91	32.0
34	1601 Amber ^b	107	GL	26.13	24.0
35	1601 Amber ^b	107	GL	26.70	26.8
36	1601 Amber ^b	107	GL	26.13	27.0
37	Grout (t $\approx$ 7.9 mm)	107	G	27.58	32.3
38	Grout (t $\approx$ 7.9 mm)	107	GL	26.69	35.0
39	Grout (t $\approx$ 7.9 mm)	107	G	26.02	25.3
40	Grout (t $\approx$ 9.5 mm)	107	G	27.69	31.8
41	Grout (t $\approx$ 9.5 mm)	107	GL	27.02	32.0
42	Grout (t $\approx$ 9.5 mm)	107	G	26.91	32.0
43	Grout (t $\approx$ 15.9 mm)	107	G	27.36	35.0
44	Grout (t $\approx$ 15.9 mm)	107	G	26.80	32.0
45	Grout (t $\approx$ 15.9 mm)	107	G	26.91	31.8
46	Polymer silica (t $\approx$ 15.9 mm)	107	GL	27.69	23.5
47	Polymer silica (t $\approx$ 15.9 mm)	107	G	26.47	24.8
48	Polymer silica (t $\approx$ 15.9 mm)	107	GL	26.47	24.0
	Control	c	G	27.91	34.3
49	None	164	GL	24.69	11.8
50	None	164	GL	24.69	11.8
51	None	164	GL	24.91	14.3
52	2889 ^b	164	G	26.47	34.0
53	2889 ^b	164	G	26.69	31.8
54	2889 ^b	164	G	26.69	33.5
55	2090-P4 ^b	164	G	26.58	32.5
56	2090-P4 ^b	164	G	26.24	33.0
57	2090-P4 ^b	164	G	26.47	33.0
58	1601 Amber ^b	164	GL	26.58	25.3
59	1601 Amber ^b	164	GL	25.47	29.8

Table 3.16. (Continued)

Specimen	Corrosion inhibiting compound	Length of exposure (days)	Failure location ^a	Failure load (kN)	Failure time (min)
60	1601 Amber ^b	164	GL	25.91	19.0
61	Grout (t ≈ 7.9 mm)	164	GL	26.69	34.8
62	Grout (t ≈ 7.9 mm)	164	GL	25.24	12.5
63	Grout (t ≈ 7.9 mm)	164	G	26.58	30.8
64	Grout (t ≈ 9.5 mm)	164	G	26.36	33.5
65	Grout (t ≈ 9.5 mm)	164	GL	26.69	33.0
66	Grout (t ≈ 9.5 mm)	164	GL	26.47	30.8
67	Grout (t ≈ 15.9 mm)	164	G	26.80	35.0
68	Grout (t ≈ 15.9 mm)	164	G	26.69	27.5
69	Grout (t ≈ 15.9 mm)	164	GL	26.91	34.8
70	Polymer silica (t ≈ 15.9 mm)	164	GL	25.91	20.3
71	Polymer silica (t ≈ 15.9 mm)	164	GL	26.02	23.0
72	Polymer silica (t ≈ 15.9 mm)	164	GL	25.91	23.8
73	Grout with 0.03-mm flaw	64	G	28.58	35.8
74	Grout with 0.03-mm flaw	64	G	28.80	35.0
75	Grout with 0.13-mm flaw	64	G	28.80	33.3
76	Grout with 0.13-mm flaw	64	GL	28.80	32.3
77	Grout with 0.25-mm flaw	64	GL	28.69	30.5
78	Grout with 0.25-mm flaw	64	GL	28.91	32.8
79	Grout with 0.76-mm flaw	64	GL	28.69	32.3
80	Grout with 0.76-mm flaw	64	GL	28.80	33.5
81	Grout with 3.18-mm flaw	64	GL	28.14	22.0

Table 3.16. (Continued)

Specimen	Corrosion inhibiting compound	Length of exposure (days)	Failure location ^a	Failure load (kN)	Failure time (min)
82	Grout with 3.18-mm flaw	64	GL	28.58	23.8
83	2889 ^b (scraped)	64	G	28.58	34.0
84	2889 ^b (scraped)	64	GL	29.02	39.8
85	2090-P4 ^b (scraped)	64	G	28.58	34.5
86	2090-P4 ^b (scraped)	64	G	28.47	30.5
87	Polymer silica (t $\approx$ 9.5 mm)	64	GL	28.14	29.0
88	Polymer silica (t $\approx$ 9.5 mm)	64	GL	28.80	28.3
89	Polymer silica (t $\approx$ 9.5 mm)	64	GL	28.47	29.3
90	None	64	GL	26.58	23.7
91	Polymer silica (t $\approx$ 9.5 mm)	110	GL	26.80	27.0
92	Polymer silica (t $\approx$ 9.5 mm)	110	GL	26.13	27.3
93	Polymer silica (t $\approx$ 9.5 mm)	110	GL	26.91	27.8
94	2889 ^b (scraped)	110	GL	26.80	32.8
95	2090-P4 ^b (scraped)	110	GL	26.36	25.8
96	Grout with 0.03-mm flaw	110	GL	27.02	31.3
97	Grout with 0.13-mm flaw	110	GL	26.91	31.5
98	Grout with 0.25-mm flaw	110	G	26.69	30.8
99	Grout with 0.76-mm flaw	110	GL	26.02	22.8
100	Grout with 3.18-mm flaw	110	GL	25.24	11.5
101	None	110	GL	26.02	22.1
	Control	c	G	27.36	34.8
102	Grout with 0.03-mm flaw	153	G	26.58	31.5
103	Grout with 0.13-mm flaw	153	G	26.24	33.5
104	Grout with 0.25-mm flaw	153	G	26.91	36.5

Table 3.16. (Continued)

Specimen	Corrosion inhibiting compound	Length of exposure (days)	Failure location ^a	Failure load (kN)	Failure time (min)
105	Grout with 0.76-mm flaw	153	G	26.24	37.3
106	Grout with 3.18-mm flaw	153	GL	23.58	8.8
107	2889 ^b (scraped)	153	G	25.58	34.8
108	2090-P4 ^b (scraped)	153	GL	25.13	30.3
109	Polymer silica (t $\approx$ 9.5 mm)	153	GL	24.91	24.0
110	Polymer silica (t $\approx$ 9.5 mm)	153	GL	24.58	22.8
111	Polymer silica (t $\approx$ 9.5 mm)	153	GL	24.69	22.0

^aG = grips, GL = gage length.

^bViscosity Oil Company.

^cControl specimen not subjected to corrosive environment.

Table 3.17. Nonstressed Tendon Corrosion Test
Result Summary (Outside Environment)

Specimen	Corrosion inhibiting compound	Length of exposure (days)	Failure location ^a	Failure load (kN)	Failure time (min)
1	None	38	G	27.69	33.3
2	None	38	G	27.91	32.5
3	None	38	G	28.02	34.0
4	None	79	G	26.58	37.5
5	None	79	GL	26.36	36.0
6	None	79	G	26.02	32.3
	Control	<i>b</i>	G	28.58	35.3
7	None	114	G	25.58	32.8
8	None	114	G	25.35	36.3
9	None	114	GL	25.35	34.8

^aG = grips, GL = gage length.

^bControl specimen not subjected to corrosive environment.

3.4.4 Tendon Monitoring Techniques

Associated with the instrumentation evaluation and development task area described in Sect. 3.3, a phase of this investigation is looking at potential techniques for monitoring the structural integrity of grouted tendons. Acoustic emission (AE) has been selected as the initial technique for examination. Basic tests are being conducted under this task to develop AE data for the composite grout-tendon system. These tests are related to plain concrete microcracking and fracture and behavior of the composite concrete-prestressing tendon system.

Behavior of concrete under both compression and tensile loadings is being investigated. Concrete cylinders 0.15 m diam by 0.54 m long will be used to obtain AE data for plain concrete under compression loads. Concrete flexure specimens 0.125 by 0.125 by 0.91 m will be used for tensile loads. Three series of four compression specimens and one flexure specimen each will be cast from low-, medium-, and high-strength concretes to determine whether strength and emissions correlate. Before loading, each cylinder will be strain gaged so that AE data may be obtained as a function

of both load and strain. For three of the four cylinders for each set, the test procedure will include: attachment of four AE transducers* to the specimen surface, calibrations and zeroing load and strain outputs, and loading the specimens slowly to failure, with strain, load, and AE data being obtained throughout the loading. The fourth compression specimen of each set will be instrumented the same as for the previous specimens, but they will be slowly fatigued by loading initially to approximately half their capacity, unloading, reloading with a 10% load increment, unloading, and repeating this procedure with incremental loadings until the specimen fails. For each cycle, AE data will be obtained. This test series is designed to provide an indication if the Kaiser effect† applies to concretes. Testing the flexure specimens in four-point bending will provide AE during tensile loadings. The test procedure will include: attachment of strain gages and AE transducers to the tensile surfaces of the specimens, calibrating and zeroing load and strain outputs, and slowly loading the specimens in flexure to failure, with AE data obtained throughout the load cycle.

The composite behavior of concrete-prestressing strand is being investigated with AE by monitoring selected grouted tendon beams tested as described in Sects. 3.4.1 and 3.4.2. Before testing, six 50-kHz AE transducers are attached to the bottom surface of the beam to be monitored. Two are attached near the reaction points for the major span and are used as slaves to lock out localized reaction noises. The remaining four transducers are spaced at 0.60-m increments to monitor the central 1.80 m of the beam to provide data on flexural crack locations. The beams are then tested to failure by the same test procedure as defined in Sect. 3.4.1. Figure 3.39 presents the test setup for the AE tests. Data obtained to date indicate good correlation between AE-predicted crack locations and actual crack locations as the cracks first appear, but as the cracks propagate toward the upper beam surface under increasing loads, data

*Two transducers will monitor the central 0.25 m of the specimen, and two transducers, which will be located near the upper and lower loading platens, will be used as slave transducers to lock out platen noise.

†Certain materials upon reloading are relatively quiet until the previous load cycle has been exceeded.

ORNL Photo 1216-78

Fig. 3.39. Acoustic Emission Beam Test Setup.

scatter increases because the emissions have to go around the cracks to reach transducers. This is demonstrated in Fig. 3.40, in which typical AE outputs of activity distribution (number and location of events) and magnitude distribution (intensity of events by location) are presented in conjunction with actual cracking locations for a grouted tendon beam monitored during testing.

3.5 LINER AND PENETRATION STUDIES — R. K. Nanstad and D. A. Canonico

The prestressed concrete reactor vessel (PCRv) for the HTGR contains several steel penetrations and closures, which are part of the liner system. In the previous annual report,¹⁷ the requirements of Appendix G to Div. I, Sect. III of the *ASME Boiler and Pressure Vessel Code*¹⁸ were discussed in some detail, especially regarding the K_{IR} (reference fracture toughness) curve and RT_{NDT} (reference nil-ductility temperature) requirement. That report also discusses the materials of interest to the liner and penetration steels testing program as well as specimens and test procedures being employed.

Initial results from drop-weight NDT tests, tensile tests, Charpy impact tests and pre-cracked Charpy fracture toughness tests were discussed previously¹⁷ for the A 508, class 1 and A 537, classes 1 and 2 materials. Further results from those tests, as well as results from fracture toughness tests with compact specimens (CS), are presented in this report.

The K_{IR} curve is shown in Fig. 3.41. The curve is normalized and plotted relative to the RT_{NDT} , which depends on the material being tested. Thus, the curve can be overlaid on a graph of fracture toughness versus actual test temperature provided that it is plotted with respect to the RT_{NDT} of the particular material.

In all cases reported herein, specimens were transverse and removed from the $(1/4)t \times t$ location, with the notch oriented in the major working direction. Figure 3.42 provides Charpy V-notch impact results for the forging F1 and plates P1, P2, and P4. The two quenched-and-tempered materials (F1 and P4) show similar Charpy toughness behavior, with very high upper-shelf values in the range 275 to 310 J. The curves for the two normalized plates (P1 and P2) lie at higher temperatures and have

ORNL-DWG 78-6508

Fig. 3.40. Cracking Pattern and Acoustic Emission Data for a Grouted Tendon Beam.

ORNL-DWG 77-9639

Fig. 3.41. Reference Fracture Toughness Curve from ASME Code.

Fig. 3.42. Charpy Impact Energy vs Temperature for Various Liner Steels.

lower upper shelf values in the range 180 to 240 J. The RT_{NDT} values reflect similar comparisons. The RT_{NDT} for F1 and P4 are -57 and -51°C , respectively; for P1 and P2 they are -40 and -46°C , respectively.

Initial results of fracture toughness tests with pre-cracked Charpy specimens of the forging and plate materials were presented previously.¹⁷ We have tested further the same materials with CS as well as pre-cracked Charpy specimens (PCC_V). All CS tests were at slow loading rates (static tests), while PCC_V tests were under slow bend (static) and impact (dynamic) loading conditions.

In Fig. 3.43 are shown curves of fracture toughness versus temperature for forging F1. The RT_{NDT} is denoted at -57°C and the K_{IR} curve is shown to allow comparison with the data. The fracture toughnesses are denoted K_{Icd} (static) and K_{Idd} (dynamic), indicating that the equivalent-energy method of analysis was used. The J-integral method was used to calculate fracture toughness also and, in all cases, provided values within a few percent of those calculated by the equivalent-energy method. With both methods, the area under the load-COD (crack opening displacement) curve

Fig. 3.43. Static and Dynamic Fracture Toughness of A 508, Cl.1. Steel (Forging F1) Measured with Pre-Cracked Charpy (PCC_V) and Compact Specimens (CS).

was measured to maximum load. Figure 3.43 shows the effect of loading rate with pre-cracked Charpy specimens. When tested under dynamic conditions, the fracture toughness curve (at the 200 MPa $\sqrt{m}$ level) is translated to a temperature about 60°C higher than that for slow loading. Thus, at -75°C, the specimen experienced fully plastic behavior with high fracture toughness under slow loading, and brittle behavior with low fracture toughness under dynamic loading. The 0.394TCS [compact specimen with thickness of a Charpy specimen, 10 mm (0.394 in.)] tests gave results similar to those from static PCC_V tests except at the "upper shelf" energy levels, where the C_VCS specimens exhibit somewhat higher values of fracture toughness. The results from tests with ITCS [25 mm (1 in.) thick] specimens were similar to those with the smaller specimens in the transition region but exhibited considerably higher upper shelf values. The comparison of behavior among the various tests and specimens noted are similar to those observed for other carbon and low-alloy steels. All data fell well to the left of the K_{IR} curve except for those on the lower end of the dynamic PCC_V curve, which were very close to the K_{IR} curve.

Figure 3.44 reflects the results from testing A 537, class 1, plates P1 and P2. In general, the results reflect similar behavior to that discussed for forging F1. Although all data fall to the left of the K_{IR} curve, it is interesting to note that the dynamic PCC_V curve for P1 lies very close to the K_{IR} curve as high as 0°C and does not exhibit a change from brittle to ductile behavior as sharply as does P2. Additionally, the upper shelf fracture toughness levels with PCC_V specimens, although quite high, are higher for P2 than for P1.

Figure 3.45 gives the results of fracture toughness tests with specimens from the quenched-and-tempered plate P4 (A 537, class 2). Again, the behavior is similar to those discussed previously and the actual results are very similar to those for the normalized plate P2, with the curves of P4 falling at slightly lower temperatures.

Figure 3.46 summarizes the distribution of static and dynamic fracture toughness data for both the A 508, class 1 forging and A 537 plate materials relative to the K_{IR} curve.

All steels tested exhibited a high degree of fracture toughness. Regarding the relationship of the data to the K_{IR} curve now existing in

(a)

(b)

Fig. 3.44. Static and Dynamic Fracture Toughness of A 537, Cl. 1 Steel Measured with Pre-Cracked Charpy (PCC_V) and Compact Specimens (CS). (a) Plate 1. (b) plate 2.

Fig. 3.45. Static and Dynamic Fracture Toughness of A 537, Cl. 2 Steel (Plate P4) Measured with Pre-Cracked Charpy (PCC_V) and Compact Specimens (CS).

Fig. 3.46. General Range of Static and Dynamic Fracture Toughness for PCRVLiner Steels.

the ASME Code, only the dynamic PCC_V results fall close to the curve at the lower values of fracture toughness. Dynamic tests with compact specimens will be tested to verify the PCC_V behavior for the base materials.

Other testing currently under way includes characterization and fracture toughness tests of various weldment samples, including weld metal and HAZ studies.

Additionally, an SA-182, F11 forging (1 1/4 Cr-1/2 Mo) has been postweld heat-treated and is currently undergoing a 10,000-hr aging at 343°C.

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4. STRUCTURAL MATERIALS

J. R. DiStefano

4.1 INTRODUCTION

A data base on the properties of materials is required for the safe design and high reliability of HTGR structural components. Many of these components must be designed to operate for very long times at temperatures where properties are time dependent. In addition, studies to date have indicated that the effect of the HTGR environment on properties must be determined.

In meeting our primary objective of providing a reliable data base, experiments on mechanical properties, welding development, and steam corrosion are being carried out. Equipment has been developed that allows measurement of these properties in simulated HTGR helium. Although primary emphasis has been placed on the development of a data base as required by ASME Code rules, tasks are also included, where appropriate, to understand the basic behavior of current and near-term materials.

4.2 MECHANICAL PROPERTIES — C. R. Brinkman

4.2.1 Tensile Testing — H. E. McCoy

Tensile tests were run previously^{1,2} on Hastelloy X, 2 1/4 Cr-1 Mo steel, 9 Cr-1 Mo steel, Hastelloy S, and Inconel 617. During the last reporting period this testing program was expanded and included aged samples of some of these same alloys and weldments. These alloys are described in Table 4.1, and their chemical compositions are given in Table 4.2.

Blocks of Hastelloy X were aged in argon at 538, 704, and 871°C for 2500 and 10,000 hr. Specimens were then machined and tested to determine the changes in tensile properties. The results of these tests are given in Table 4.3. The main effect of aging Hastelloy X is to reduce the fracture strain at ambient temperatures. Of the aging and test conditions examined thus far, the lowest fracture strain resulted from aging at 704°C for 2500 hr and testing at room temperature.

Table 4.1. Alloy Characterizations

Material	Heat	Source	Product Form	Grain Size (μm)	Heat Treatment
Inconel 617	XX01A3US	INCO	12.7-mm (1/2-in.) plate		Solution annealed by vendor
Hastelloy X	2600-3-4936	Cabot	12.7-mm (1/2-in.) plate	80	Solution annealed by vendor
	2600-3-2792	Cabot	31.8-mm (1 1/4-in.) bar	55	Solution annealed by vendor
	2600-4-4284	Cabot	12.7-mm (1/2-in.) plate		
	2600-4-4345	Cabot	4-mm (5/32-in.) wire		
Alloy 800H	HH6993	INCO	19-mm (3/4-in.) bar		
	HH7728A	INCO	12.7-mm (1/2-in.) plate		
2 1/4 Cr-1 Mo	X-6216	B and W	51-mm-OD (2.0-in.) tube	27	Isothermally annealed ^a
	36202	B and W	51-mm-OD (2.0-in.) tube	20-27	Isothermally annealed ^a
	72768	B and W	51-mm-OD (2.0-in.) tube	27	Isothermally annealed ^a
9 Cr-1 Mo	316381				
Hastelloy S	8635-3-7189	Cabot	1.6-mm (1/16-in.) wire		
	-8365T	Cabot	12.7-mm (1/2-in.) plate		
Inco A	D-28159		4.8-mm (3/16-in.) wire		
ERNiCr-3	A3143T382V	Arcos	1.6-mm (1/16-in.) wire		

^a927 $\pm$ 14°C for 30 min, cooled to 704 $\pm$ 14°C at a maximum rate of 83°C/hr, held at 704 $\pm$ 14°C for 2 hr, cooled to room temperature at a maximum rate of 6°C/min.

Table 4.2. Alloy Compositions

Material	Content, %													
	Ni	Cr	Co	Mo	Fe	Al	Si	C	Mn	S	W	Ti	Cu	Others
Inconel 617	57.35	20.30	11.72	8.58	1.01	0.76	0.16	0.07	0.05	0.004				
Hastelloy X 4936	bal	21.82	1.68	9.42	19.09		0.44	0.07	0.58	<0.005	0.63			
2792	bal	21.25	1.94	8.99	18.96		0.41	0.10	0.57	<0.005	0.56			
4284	bal	21.79	2.40	8.82	19.06		0.35	0.06	0.59	<0.005	0.63			
4345	bal	21.92	2.09	8.85	18.81		0.40	0.08	0.70	<0.005	0.42			
Alloy 800H 6993 ^a	33.43	20.36			43.60	0.43	0.39	0.07	0.77	0.004		0.46	0.49	
7728A	31.85	19.46			46.08	0.43	0.24	0.08	0.90	0.003		0.42	0.54	
2 1/4 Cr-1 Mo X-6216		2.19		1.00	bal		0.26	0.12	0.44	0.016				
36202		2.24		0.96	bal		0.34	0.12	0.43	0.023				
72768		2.16		0.94	bal		0.38	0.10	0.41	0.012				
9 Cr-1 Mo	0.4	8.83		1.0	bal		0.6	0.0803	0.5	0.0208				0.02 V
Hastelloy S 7180	bal	15.15	0.06	14.46	0.44		0.38	0.006	0.47	0.006	<0.10		0.01	0.019 La
8365T	bal	15.3	0.15	15.3	0.5	0.40	0.40	0.0129	0.5			0.01	0.04	0.02 La
Inco A	68.70	15.04	<0.01	0.89	9.05		0.41	0.030	1.52	<0.001		0.06	0.30	2.29 Nb 0.037 N
ERNiCr-3	72.15	20.90			0.11		0.03	0.022	3.34	0.005		0.42	0.03	2.88 Nb + Ta

^aChemical composition not available for this heat. Analysis shown is for HH13-6979.

Table 4.3. Tensile Properties of Hastelloy X (Heat 2600-3-4936)
As Received and Aged in Argon

Test	Aging Conditions			Test Temperature		0.2% Yield Strength		Ultimate Tensile Strength		Elongation in		Reduction of Area (%)	Notes
	31.8 mm (1.25 in.)												
	Temperature		Time (hr)	(°C)	(°F)	(MPa)	(ksi)	(MPa)	(ksi)	Uniform (%)	Total (%)		
(°C)	(°F)												
14892			0	22	72	359	52.0	762	110.5	43.8	50.5	59.8	
14893			0	22	72	349	50.6	764	110.8	43.9	51.5	59.2	
14894			0	22	72	350	50.8	764	110.8	44.3	51.9	61.4	
17885	538	1000	2,500	22	72	333	48.3	764	110.8	49.5	55.5	54.4	
17886	538	1000	2,500	22	72	340	49.3	766	111.2	46.8	54.4	50.0	
17889	704	1300	2,500	22	72	482	69.9	916	132.9	7.7	7.7	8.7	a
17890	704	1300	2,500	22	72	476	69.0	939	136.1	9.8	9.8	8.1	a
17893	871	1600	2,500	22	72	367	53.3	835	121.1	15.8	15.8	14.7	
17894	871	1600	2,500	22	72	368	53.4	818	118.7	13.4	13.4	12.3	
XC7	538	1000	10,000	22	72	385	55.9	777	112.7	43.1	43.2	39.7	
XC25	704	1300	10,000	22	72	357	51.8	854	123.8	18.2	18.2	16.1	
XC43	871	1600	10,000	22	72	399	57.9	732	106.2	15.0	15.1	10.5	
14807			0	538	1000	239	34.6	592	85.8	46.5	49.9	40.9	
14808			0	538	1000	221	32.1	596	86.5	43.9	48.8	31.4	
17887	538	1000	2,500	538	1000	222	32.2	603	87.5	54.0	58.6	48.0	b
17888	538	1000	2,500	538	1000	227	32.9	601	87.2	49.3	52.7	35.5	a,b
XC8	538	1000	10,000	538	1000	236	34.2	600	87.0	48.6	48.7	42.9	b
14799			0	704	1300	223	32.4	425	61.6	23.0	48.5	41.7	
14800			0	704	1300	215	31.2	452	65.5	29.9	37.3	33.6	
14801			0	704	1300	212	30.8	460	66.7	30.8	37.3	35.9	
17891	704	1300	2,500	704	1300	294	42.6	443	64.3	6.3	58.1	55.0	
17892	704	1300	2,500	710	1310	284	41.2	429	62.2	5.9	74.2	58.2	c
XC26	704	1300	10,000	704	1300	230	33.3	439	63.7	11.1	436.3	37.5	
14815			0	871	1600	161	23.4	165	24.0	2.1	79.8	88.9	
14898			0	871	1600	163	23.6	168	24.3	2.3	84.4	88.2	
17895	871	1600	2,500	871	1600	146	21.2	161	23.4	2.5	96.4	82.4	
17896	871	1600	2,500	871	1600	141	20.4	159	23.0	2.5	101.0	86.3	
XC43	871	1600	10,000	871	1600	151	21.9	162	23.5	5.8	78.5	94.5	

^aBroke at gage mark.

^bSerration occurred before 0.2% yield.

^cDouble necking.

Blocks of Inconel 617 were aged for 2500 and 10,000 hr at 538, 704, and 871°C in argon. Specimens were machined from these blocks, and they were tested in tension. The results of the tests are given in Table 4.4. The most deleterious property change was the reduction in room temperature fracture strain; however, the loss was not as large as noted for Hastelloy X discussed above. The effect became larger as the aging temperature was increased.

Impact and notched-tensile specimens were made of some of the Hastelloy X and Inconel 617 blocks aged 10,000 hr. The impact sample used was the Charpy V-notch type with a cross section 10.16 by 10.16 mm (0.400 by 0.400 in.). The notched tensile specimen had a uniform gage diameter of 6.35 mm (0.250 in.) and a diameter at the notch of 4.78 mm (0.188 in.). Both types of tests were carried out at room temperature, and the results are given in Table 4.5. Specimens of both materials were notch-strengthened by aging at 538°C; they were almost unaffected by aging at 704°C, and notch-weakened by aging at 871°C. The impact energy was reduced by aging, and the magnitude of the reduction increased with increasing aging temperature. The impact energy of Inconel 617 was at least double that of Hastelloy X under all aging conditions.

Tensile tests were conducted on samples of various materials that had previously been creep tested in a simulated HTGR-helium environment as part of the Dragon-Oslo Phase II studies.³ Samples of four different materials involving 15 different heats were sent to ORNL by the High-Temperature Materials Program (HTMP) with the desired tensile test conditions already specified. Additional samples were sent to other organizations by HTMP, who will collate and disseminate the information from the test program.

The specimens had a gage section 4.5 mm diam by 25.4 mm long (0.18 by 1.0 in.) and an overall length of 44.2 mm (1.74 in.). The ends were threaded for gripping. Some specimens were in the unexposed condition (controls), and others had been exposed to simulated HTGR-helium containing partial pressures of 50–100 μatm H_2 , 25–50 μatm CO , 3–8 μatm CH_4 , and 0.5–3.0 μatm H_2O (1 μatm = 0.101 Pa). The specimens had been exposed at various temperatures, stresses, and times. The specimens were then tensile tested at ORNL in air in an Instron machine at a crosshead speed of

Table 4.4. Tensile Properties of Inconel 617 (Heat OXX1A3US)
As Received and Aged in Argon

Test	Aging Conditions			Test Temperature		0.2% Yield Strength		Ultimate Tensile Strength		Elongation in 31.8 mm (1.25 in.)		Reduction of Area (%)	Notes
	Temperature		Time (hr)	(°C)	(°F)	(MPa)	(ksi)	(MPa)	(ksi)	Uniform (%)	Total (%)		
	(°C)	(°F)											
16545			0	22	72	303	43.9	747	108.3	54.1	54.3	36.2	
16546			0	22	72	301	43.7	757	109.8	59.6	60.9	40.7	
16547			0	22	72	305	44.3	754	109.3	57.4	57.9	32.4	
17897	538	1000	2,500	22	72	358	51.9	794	115.2	66.0	68.1	49.3	
17898	538	1000	2,500	22	72	360	52.2	796	115.5	67.6	69.8	48.6	
17901	704	1300	2,500	22	72	371	53.8	847	122.9	39.2	39.2	32.0	
17902	704	1300	2,500	22	72	372	54.7	848	123.1	44.1	44.1	35.0	
17905	871	1600	2,500	22	72	331	48.0	797	115.6	29.9	29.9	25.0	a
17906	871	1600	2,500	22	72	334	48.5	822	119.2	34.6	34.6	29.3	
IC43	538	1000	10,000	22	72	392	56.9	805	116.8	53.3	53.4	43.8	
IC25	704	1300	10,000	22	72	369	53.5	798	115.7	29.6	29.6	23.3	
IC7	871	1600	10,000	22	72	290	42.0	695	100.8	28.3	28.3	21.0	
16657			0	538	1000	216	31.3	610	88.5	64.7	67.3	49.8	
16658			0	538	1000	207	30.0	606	87.9	61.1	64.4	46.1	
17899	538	1000	2,500	538	1000	260	37.7	638	92.5	63.4	68.5	51.7	b
17900	538	1000	2,500	538	1000	259	37.6	638	92.5	66.3	69.2	48.9	b
IC44	538	1000	10,000	538	1000	222	32.2	669	97.0	36.4	36.5	30.5	b
IC26	704	1300	10,000	538	1000	274	39.8	629	91.3	58.9	58.9	46.3	b
16664			0	704	1300	199	28.8	466	67.6	29.3	68.0	49.3	
16665			0	704	1300	196	28.4	443	64.2	25.9	69.9	53.1	
16666			0	704	1300	196	28.4	454	65.9	29.9	74.7	52.5	
17903	704	1300	2,500	704	1300	257	37.3	502	72.8	16.2	75.3	57.6	b
17904	704	1300	2,500	704	1300	256	37.1	475	69.0	14.4			b,c
IC27	704	1300	10,000	704	1300	211	30.6	461	66.9	16.9	68.8	56.3	
16672			0	871	1600	181	26.3	194	28.1	3.2	88.0	87.8	b
16673			0	871	1600	189	27.4	194	28.1	3.2	91.6	86.0	
17907	871	1600	2,500	871	1600	154	22.3	168	24.3	5.1			c
17908	871	1600	2,500	871	1600	170	24.6	179	25.9	4.3	98.2	91.0	
IC8	871	1600	10,000	871	1600	170	24.7	181	26.2	8.0	90.9	79.2	

^aBroke at gage mark

^bSerration occurred before 0.2% yield.

^cPull rod broke before specimen did.

Table 4.5. Results of Notched Tensile and Impact Tests on Inconel 617 and Hastelloy X Aged 10,000 hr at the Indicated Temperature (Tested at 25°C)

Material	Aging Temperature (°C)	Ultimate Tensile Strength, Ratio Notched/Unnotched	Impact Energy	
			(J)	(ft-lb)
Hastelloy X	538	1.12	39	29
Hastelloy X	704	1.01	15	11
Hastelloy X	871	0.92	7.5	5.5
Inconel 617	538	1.15	80.5	59.5
Inconel 617	704	1.03	31	23
Inconel 617	871	0.94	25	18.5

6.35 $\mu\text{m/s}$ (0.015 in./min). Strain measurements were taken from the crosshead motion, except for the fracture and reduction of area measurements, which were taken from the reassembled fractured specimen.

Information supplied by HTMP on composition, product form, and heat treatment is summarized in Table 4.6. The results of tensile tests and the pretest exposure conditions are given in Table 4.7

Interpretation of these data is hampered by such factors as fragmentary data, lack of purely thermal controls, and failure at gage marks. However, the results generally are consistent with the picture that the impure helium environment is either neutral or decarburizing to 2 1/4 Cr-1 Mo steel and carburizing to all other materials. The properties of the 2 1/4 Cr-1 Mo steel were not changed greatly. In general, the other alloys experienced an increase in yield stress and a decrease in total elongation. These changes are consistent with the hypothesis that all these alloys were carburized. Other properties — such as ultimate tensile stress, uniform elongation, and reduction of area — are very dependent upon the influence of the dissolved carbon or precipitated carbides, and upon the work hardening and fracture characteristics of the alloy. Further analysis of all the data by HTMP should be useful in pinpointing the effects of alloy composition and pretest exposure on the tensile properties.

Two weld joints of interest for HTGRs are being investigated. One involves joining Hastelloy X to Hastelloy X with filler metals Hastelloy X

Table 4.6. Composition, Product Form, and Heat Treatment of Alloys
Used in the Dragon-Oslo Phase II Work

Alloy	Content, wt %													Product Form	Heat Treatment
	C	Si	Mn	Cr	Ni	Mo	Nb	Fe	Ti	N	S	P	Others		
B2 2 1/4 Cr-1 Mo	0.11	0.33	0.50	2.31		0.93		Bal			0.021	0.013		19 mm bar	Normalized and Tempered
B3 2 1/4 Cr-Mo-Nb	0.08	0.27	0.51	2.34		1.01	0.86	Bal			0.034	0.020		Unknown	1050°C, 1 hr, AC ^a + 750°C, 2 hr, AC
D3a AISI 316	0.04	0.34	1.7	17.05	12.04	2.81		Bal			0.019	0.028		12 mm plate	1050°C, 30 min, AC
D3b AISI 316	0.053	0.61	1.66	16.5	13.2	2.22		Bal		0.031	0.01	0.02		16 mm bar	1070°C, 15 min, WQ ^b
D3c AISI 316 Modified ^c	<0.03	0.50	1.7	18.0	12.0	2.7		Bal		0.10		0.003	0.4 V 0.004 B	Unknown	Unknown
D3d AISI 316	0.065	0.55	1.47	16.8	15.6	2.33	0.16	Bal					0.003 B	13 mm bar	1150°C, 30 min
H1b Alloy 800 Gd 2	0.06	0.53	0.57	20.9	31.1			Bal	0.41	0.011	0.009	0.010	0.37 Al	Unknown	1150°C, 20 min, WQ ASTM grain size 3.5
H1e Alloy 800 Gd 2	0.06	0.35	0.61	21.0	30.5	0.07		Bal	0.54	0.011	0.012	0.017	0.52 Al	Unknown	1150°C, 30 min, AC ASTM grain size 2-3
H2a Alloy 800	0.06	0.53	0.57	20.9	31.1			Bal	0.41	0.011	0.009	0.01	0.37 Al	16 mm bar	1000°C, 20 min, WQ
H2b Alloy 800	0.017	0.50	0.63	20.8	33.8			Bal	0.59	0.022			0.26 Al	16 mm bar	1000°C, 20 min, WQ
H5 Alloy 800 Modified	0.07	0.4	0.8	21.0	32.0		1.1	Bal					0.4 Al	23 × 15 mm bar	1150°C, 30 min, WQ
H8Eb Alloy 800 Experimental	0.03	0.44	0.61	19.8	35.5	1.01	0.48	Bal		0.012			2.93 W	12 mm bar	1150°C, 30 min, AC
H9Eb Alloy 800 Experimental	0.044	0.25	0.68	20.3	35.3	1.10	0.27	Bal		0.32			2.66 W 0.10 Hf	12 mm bar	1150°C, 30 min, AC
H01-L Alloy 800 Ni-Plated								Unknown						Unknown	Unknown
J2a Hastelloy X Cabot	0.098	0.38	0.68	21.83	47.4	9.0	0.01	17.8	0.05		0.005	0.021	1.79 Co 0.001 B 0.003 Ta	Unknown	Unknown

^aAC = air cooled.

^bWQ = water quenched.

^cUddeholm 724-LN.

Table 4.7. Tensile Properties of 2 1/4 Cr-1 Mo Steel, Type 316 Stainless Steel, Alloy 800, and Hastelloy X After Prior Creep in Simulated HTGR Helium Environment in the Dragon-Oslo Phase II Work.

Control tests of unstressed-unexposed specimens are listed for comparison

Heat	Creep Test Conditions ^a						Tensile Test Temperature		0.2% Yield Strength		Ultimate Tensile Strength		Elongation in 25.4 mm (1.0 in.)		Reduction of Area (%)	Remarks ^b	
	Temperature		Time (hr)	Stress		Strain (%)	Temperature		Strength		Tensile Strength		25.4 mm (1.0 in.)				
	(°C)	(°F)		(MPa)	(ksi)		(°C)	(°F)	(MPa)	(ksi)	(MPa)	(ksi)	Uniform (%)	Total (%)			
<u>2 1/4 Cr-1 Mo Steel</u>																	
B2	550	1022	9,000	85	12.3	1.60	room	room	336	48.7	547	79.3	10.3	24.0	68.0		
	550	1022	3,000	100	14.5	1.55	room	room	356	51.7	563	81.6	10.6	23.0	71.4		
	550	1022	3,000	120	17.4	1.82	550	1022	250	36.2	288	41.7	4.7	52.5	77.5		
			control				room	room	460	66.7	593	86.0	7.6	19.6	74.1		
			control				room	room	505	73.2	589	85.4	6.4	22.1	73.2		
			control				room	room	443	64.3	586	85.0	10.6	26.6	72.4		
			control				550	1022	339	49.2	367	53.2	2.2	29.7	82.6		
<u>2 1/4 Cr-1 Mo-Nb Steel</u>																	
B3	550	1022	3,000	120	17.4	0.02	room	room	337	48.9	490	71.1	13.7	28.4	75.8		
	550	1022	3,000	85	12.3	0.62	room	room	314	45.5	479	69.5	14.5	26.4	73.8	c	
	550	1022	3,000	100	14.5	0.09	550	1022	254	36.8	281	40.8	2.6	20.2	80.4		
			control				room	room	359	52.0	498	72.3	11.5	27.1	79.1		
			control				room	room	352	51.0	496	71.9	11.8	27.4	78.4		
			control				550	1022	274	39.7	316	45.8	4.4	22.1	80.0		
			control														
<u>Type 316 Stainless Steel</u>																	
D3a	650	1202	10,000	79	11.5	0.44	room	room	299	43.4	701	101.7	34.8	35.8	35.6	BGM	
	650	1202	11,000	94	13.6	3.27	room	room	305	44.3	694	100.6	32.4	33.6	21.9	BGM	
	750	1382	14,000	33	4.8	5.18	room	room	288	41.7	635	92.1	13.1	13.2	11.1	BGM, d	
	700	1292	12,000	48	7.0	0.78	room	room	282	40.9	703	102.0	19.9	20.7	28.7	BGM	
	700	1292	12,000	57	8.3	3.55	room	room	301	43.6	696	101.0	17.5	17.8	10.6	BGM	
	750	1382	15,000	35	5.1	4.91	room	room	325	47.1	583	84.5	7.2	7.4	6.9	BGM	
	700	1292	16,000	48	7.0	1.73	700	1292	152	22.0	324	47.0	12.9	51.2	61.2		
	700	1292	12,000	48	7.0	0.98	700	1292	152	22.1	323	46.9	15.4	57.6	64.3		
			control				room	room	285	41.4	651	94.4	49.5	68.1	76.6		
			control				room	room	283	41.0	645	93.6	48.0	69.7	75.0		
			control				700	1292	144	20.9	299	43.4	19.5	61.0	55.2		
			control				700	1292	137	19.8	298	43.2	20.3	57.3	54.9		
	D3b	750	1382	7,000	42	6.1	3.34	room	room	257	37.3	661	95.9	28.4	29.2	19.9	BGM
		750	1382	7,000	37	5.4	0.49	room	room	254	36.9	648	94.0	27.3	28.3	25.3	BGM
		800	1472	6,000	27	3.9	1.83	room	room	227	32.9	612	88.7	39.0	40.4	37.0	BGM
		800	1472	3,000	21	3.0	0.85	room	room	257	37.3	639	92.7	42.6	54.9	48.7	
		650	1202	3,000	96	13.9	0.67	room	room	299	43.3	678	98.4	40.4	51.4	62.0	
		650	1202	3,000	83	12.0	0.13	room	room	280	40.6	659	95.6	43.4	55.9	63.8	
		650	1202	3,000	87	12.6	0.33	650	1202	168	24.4	343	49.8	18.4	64.6	73.1	
700		1292	16,000	59	8.6	2.38	700	1292	148	21.5	283	41.1	11.9	70.3	66.0		
800		1472	6,000	21	3.0	1.96	800	1472	121	17.5	170	24.7	7.7	76.3	58.2		
			control				room	room	262	38.0	656	95.2	60.0	71.9	80.1		
			control				room	room	255	37.1	604	87.6	78.0	72.8	78.3		
			control				650	1202	151	21.9	352	51.0	29.3	49.1	49.1		
			control				650	1202	142	20.6	348	50.4	30.0	53.3	52.2		
			control				700	1292	136	19.7	274	39.8	21.0	56.1	43.5		
			control				700	1292	141	20.4	279	40.4	20.3	65.2	52.7		
			control				800	1472	126	18.2	176	25.5	9.0	76.0	79.1		
D3c		700	1292	8,000	69	10.0	0.18	room	room	361	52.3	686	99.5	8.9	8.9	8.2	BGM
		650	1202	8,000	145	21.0	0.85	room	room	425	61.6	787	114.2	10.0	10.2	11.2	
		650	1202	8,500	146	21.2	2.40	room	room	447	64.9	866	125.6	22.5	23.0	18.0	BGM
	650	1202	9,500	142	20.6	0.06	room	room	599	86.9	925	134.1	19.4	24.2	31.3		
	700	1292	8,000	89	12.9	0.49	room	room	359	52.0	689	100.0	8.6	8.7	9.4	BGM	
	650	1202	8,000	137	19.9	0.76	650	1202	261	37.9	451	65.4	11.0	44.7	62.6		
			control				room	room	299	43.4	646	93.7	51.6	74.6	78.4		
			control				room	room	303	43.9	652	94.5	50.2	69.7	76.9		
			control				650	1202	151	21.9	451	65.4	34.4	44.8	71.6		
			control				650	1202	148	21.4	439	63.6	33.4	39.1	39.8	BGM	
	D3d	650	1202	3,000	95	13.8	0.35	room	room	283	41.0	581	84.3	43.8	45.5	43.4	BGM
		800	1472	2,000	48	7.0	0	room	room	228	33.1	556	80.6	33.8	34.8	30.4	BGM
		800	1472	2,000	47	6.8	0	800	1472	121	17.5	175	25.4	9.9	76.1	83.4	DN
				control				800	1472	140	20.3	201	29.2	13.9	52.9	81.8	
				control				800	1472	139	20.1	238	34.5	12.5	45.6	49.0	
				control				room	room	254	36.9	579	84.0	58.3	75.5	78.6	
				control				room	room	245	35.6	583	84.5	58.3	76.5	78.3	
	<u>Alloy 800</u>																
	H1b	750	1382	7,000	35	5.1	0.18	room	room	219	31.7	587	85.1	36.2	42.9	50.1	
750		1382	7,000	41	5.9	0.98	room	room	208	30.2	547	79.4	24.4	25.8	33.5		
750		1382	3,000	42	6.1	0.18	room	room	219	31.8	583	84.5	34.6	43.6	56.4		
800		1472	5,000	32	4.6	0.89	room	room	205	29.8	547	79.3	33.0	38.6	49.7		
800		1472	6,000	31	4.5	1.15	room	room	198	28.7	432	62.6	11.9	12.1	13.1	d	
700		1292	12,000	57	8.3	2.53	room	room	250	36.3	598	86.7	27.0	28.7	32.4	Cracks ^f	
700		1292	12,000	48	7.0	0.39	room	room	243	35.3	616	89.4	31.0	39.2	52.9		
700		1292	12,000	57	8.3	0.99	700	1292	177	25.7	286	41.5	8.1	13.3	15.7		
750		1382	3,000	41	5.9	0.22	750	1382	137	19.9	216	31.1	5.5	71.7	56.1		
			control				room	room	219	31.7	543	78.8	40.2	51.2	76.0		
			control				room	room	216	31.3	559	81.1	43.5	53.5	75.5		
			control				700	1292	122	17.7	316	45.8	21.8	27.0	22.3		
			control				700	1292	125	18.1	314	45.6	21.0	25.7	21.5		
			control				750	1382	119	17.2	238	34.5	12.8	33.0	29.1		
			control				750	1382	119	17.2	238	34.5	12.8	35.5	30.5		

Table 4.7. (Continued)

Heat	Creep Test Conditions ^a						Tensile Test Temperature		0.2% Yield Strength		Ultimate Tensile Strength		Elongation in 25.4 mm (1.0 in.)		Reduction of Area (%)	Remarks ^b
	Temperature		Time (hr)	Stress		Strain (%)							Uniform (%)	Total (%)		
	(°C)	(°F)		(MPa)	(ksi)		(°C)	(°F)	(MPa)	(ksi)	(MPa)	(ksi)				
H1e	750	1382	7,000	49	7.1	4.13	room	room	227	32.9	543	75.7	15.0	16.0	11.4	BGM
	750	1382	7,000	39	5.7	0.40	room	room	239	34.7	562	81.5	17.4	19.4	38.0	BGM
	800	1472	6,000	20	2.9	2.80	room	room	210	30.5	581	84.3	32.6	36.9	39.7	
	800	1472	3,000	31	4.5	2.31	room	room	195	28.3	572	82.9	34.8	41.7	56.4	
	750	1382	7,000	32	4.6	0.09	750	1382	150	21.7	240	34.8	5.8	43.7	38.0	BGM
H2a	650	1202	3,000	85	12.3	0	room	room	432	62.6	709	102.9	14.9	15.5	25.4	
	650	1202	8,000	75	10.9	0.22	room	room	316	45.8	665	96.5	18.5	23.9	53.9	d
	650	1202	3,000	73	10.6	0	650	1202	332	48.2	410	59.5	5.2	7.0	11.6	
			control				room	room	390	56.6	581	84.3	15.3	17.1	38.4	
			control				room	room	389	56.4	403	58.4	0.6	0.9	7.6	Course-grain ^g
			control				650	1202	155	22.5	381	55.2	25.5	40.0	40.7	BGM
			control				650	1202	239	34.7	394	57.1	14.3	17.4	20.7	
H2b	650	1202	3,000	75	10.9	1.11	room	room	276	40.0	617	89.5	31.6	42.7	68.3	
	650	1202	3,000	74	10.7	0.89	650	1202	201	29.2	370	53.6	14.1	31.1	38.9	BGM
			control				room	room	232	33.7	552	80.0	43.3	58.8	77.6	
			control				room	room	219	31.8	549	79.6	38.6	52.6	78.1	
			control				650	1202	147	21.3	335	48.6	21.3	48.6	59.6	
H5			control				650	1202	143	20.7	330	47.8	20.2	49.7	55.8	
	650	1202	11,000	110	16.0	0.13	room	room	321	46.6	595	86.3	15.5	16.0	21.6	BGM
	750	1382	11,000	49	7.1	0.49	room	room	241	35.0	575	83.4	15.0	17.8	20.6	BGM
	750	1382	11,000	55	8.0	1.46	room	room	234	33.9	518	75.1	12.2	12.5	18.7	BGM
	750	1382	14,000	44	6.4	0.28	room	room	270	39.1	658	95.5	25.0	33.5	49.9	
	800	1472	10,000	40	5.8	1.46	room	room	228	33.1	555	80.5	21.1	22.2	33.5	
	800	1472	13,000	33	4.8	3.92	room	room	232	33.6	532	77.2	16.2	16.4	24.9	
	800	1472	13,000	28	4.1	4.65	800	1472	133	19.3	170	24.7	4.8	75.0	58.8	BGM
			control				room	room	250	36.3	602	87.3	36.8	50.4	69.7	
			control				room	room	243	37.3	593	86.0	37.4	48.5	70.4	Bent ^h
H8Eb ⁱ			control				800	1472	146	21.2	190	27.6	5.9	76.2	78.8	
			control				800	1472	154	22.3	202	29.3	9.2	75.8	78.4	
	750	1382	2,000	46	6.7	0.04	room	room	268	38.8	653	94.7	32.4	35.7	33.8	
	750	1382	2,000	46	6.7	1.02	room	room	241	35.0	652	94.5	30.2	35.8	41.8	
	800	1472	3,000	38	5.5	0.09	room	room	228	33.1	629	91.3	30.0	30.0	26.0	BGM
	800	1472	3,000	38	5.5	1.07	room	room	229	33.2	630	91.4	31.0	31.9	24.8	BGM
	750	1382	2,000	52	7.5	0.44	room	room	251	36.4	653	94.7	30.7	31.5	26.8	BGM
H9Eb ⁱ	750	1382	2,000	52	7.5	0.13	750	1382	156	22.6	268	38.8	9.4	58.9	56.9	
			control				room	room	227	32.9	601	87.2	40.8	55.0	67.4	
			control				room	room	224	32.5	599	86.9	41.3	52.3	69.0	
			control				750	1382	119	17.3	276	40.1	16.7	24.1	30.0	
			control				750	1382		16.7		40.4	17.3	21.1	19.6	
	750	1382	2,000	51	7.4	0.84	room	room	239	34.7	646	93.7	33.0	40.0	50.2	
			control				room	room	225	32.7	612	88.7	40.1	50.1	69.1	
H01-L ^k			control				room	room	237	34.4	510	74.0	20.5	28.0	25.1	Split ^j
	800	1472	2,000	23	3.3	0.10	room	room	230	33.4	551	79.9	20.0	22.0	28.7	BGM
	900	1652	2,000	9	1.3	0.62	room	room	262	39.4	510	74.0	6.3	6.3	0	BGM
Hastelloy X																
J2a	750	1382	15,000	62	9.0	2.16	room	room	396	57.4	616	89.4	2.4	2.4	3.6	BGM
	750	1382	5,000	54	7.8	0.32	room	room	538	78.0	833	120.8	4.4	4.4	3.8	BGM
	750	1382	15,000	55	8.0	0.46	room	room	430	62.4	692	100.4	2.9	2.9	3.6	BGM
	750	1382	3,000	60	8.7	0.53	room	room	394	57.2	725	105.2	5.6	5.6	7.2	BGM
	750	1382	15,000	62	9.0	2.27	750	1382	252	36.5	399	57.8	7.2	33.4	25.9	BGM
			control				room	room	334	48.4	775	112.4	45.5	51.2	45.0	BGM
			control				room	room	413	59.9	789	114.4	43.4	46.7	43.2	BGM
		control				750	1382	227	32.9	420	60.9	14.0	34.3	38.3	BGM	
		control				750	1382	217	31.4	404	58.6	22.5	50.7	40.4		

^a Except where indicated, tested in simulated HTGR helium with partial pressures in μatm as follows: 50-100 H_2 , 25-50 CO , 3-8 CH_4 , 0.5-3 H_2O . 1 μatm = 0.101 Pa.

^b BGM = Broke at gage mark. DM = double necking.

^c The strain rate during the tensile test of this specimen was $3.3 \times 10^{-4}/\text{s}$. All the other tests were conducted at $t = 2.5 \times 10^{-4}/\text{s}$.

^d Creep test in air.

^e Modified alloy.

^f Some cracks resulted from the creep test were visible on the fracture surface.

^g Brittle fracture and very coarse-grained structure.

^h Specimen was bent a little.

ⁱ Experimental alloy.

^j Specimen split longitudinally.

^k Nickel plated.

and Hastelloy S. Welding processes of potential interest are gas tungsten-arc (GTA) and shielded metal-arc (SMAW). Three weldment types were made in duplicate for evaluation (Table 4.8). Another joint is Hastelloy X joined to alloy 800H. Potential welding processes and filler metals are GTA with ERNiCr-3 and SMAW with Inco weld A. Two weldments of each type were made for evaluation, and these are being evaluated by creep and tensile tests in the as-welded and aged condition. The test samples were taken transverse to the welding direction so that the gage length includes both base metals and the weld deposit.

The tensile properties of the five weldments in the as-welded condition are given in Table 4.9. The three Hastelloy X weldments have very similar tensile properties. The location of the fracture varied between the base metal and the weld metal. For example, at 25°C two weldments failed in the base metal and one failed in the weld metal. The weldments of Hastelloy X to alloy 800H failed in the alloy 800H base metal at all test conditions. Specimens from the GTA weld had slightly higher yield stress than those from the SMAW weld.

The tensile properties of the weldments after aging 2000 hr are shown in Table 4.10. Aging produced significant changes in the properties of both materials. As shown in Fig. 4.1, aging Hastelloy X at all temperatures

Table 4.8. Definition of Weld Constituents^a

Welds	Base Metal	Filler Metal	Base Metal	Welding Process
121, 122	Hastelloy X heat 2600-4-4284	Hastelloy X heat 2600-4-4345	Hastelloy X heat 2600-4-4284	GTA
123, 124	Hastelloy X heat 2600-4-4284	Hastelloy S heat 8635-3-7180	Hastelloy X heat 2600-4-4284	GTA
119B, 120	Hastelloy X heat 2600-4-4284	Hastelloy X heat 2600-4-4345	Hastelloy X heat 2600-4-4284	SMAW
125, 126	Hastelloy X heat 2600-4-4284	ERNiCr-3 ^b heat A3143T382V	Alloy 800H heat HH7728A	GTA
117, 118	Hastelloy X heat 2600-4-4284	Inco Weld A heat D-28159	Alloy 800H heat HH7728A	SMAW

^aSee Tables 4.1 and 4.2 for detailed information on each constituent.

^bOften known by trade name Inconel 82.

Table 4.9. Tensile Data for Weldments in the As-Welded Condition

Weld	Specimen	Weld Description ^a	Test Temperature (°C)	Stress, MPa (ksi)		Elongation, %		Reduction of Area, %		Fracture Location	Serrated
				Yield	Ultimate	Uniform	Total	Sample	Weld		
125	16721	X-82-800H GTA	25	339 (49.2)	568 (82.4)	19.0	20.0	63.8	16.3	Base	No
	16722		482	269 (39.0)	485 (70.3)	24.0	24.2	50.4	24.9	Base	Yes
	16723		538	260 (37.7)	468 (67.9)	24.0	24.5	54.0	12.5	Base	Yes
	16724		649	248 (35.9)	425 (61.7)	18.0	19.0	14.1	14.0	Base	Yes
121	16703	X-X-X GTA	25	456 (66.2)	725 (105.2)	38.0	40.0	44.1	44.1	Weld	No
	16703		593	304 (44.1)	548 (79.5)	33.0	33.5	52.2	52.2	Weld	Yes
	16705		649	308 (44.6)	535 (77.6)	36.0	36.8	43.8	43.8	Weld	Yes
	16706		760	283 (41.0)	443 (64.2)	15.0	30.9	36.4	33.0	Base	No
	16707		871	248 (35.9)	262 (38.0)	5.0	26.0	79.7	79.7	Weld	No
123	16712	X-X-X GTA	25	460 (66.7)	815 (118.2)	46.0	46.8	69.9	53.4	Base	No
	16713		593	309 (44.8)	554 (80.3)	39.0	39.5	63.7	63.7	Weld	Yes
	16714		649	308 (44.6)	537 (77.9)	40.0	40.9	56.3	56.3	Weld	Only at end
	16715		760	306 (44.4)	452 (65.6)	18.0	26.3	65.5	65.5	Weld	Yes
	16716		871	256 (37.1)	269 (39.0)	7.0	48.6	77.4	21.2	Base	No
117	16730	X-A-800H SMAW	25	329 (47.7)	567 (82.3)	21.0	21.7	55.8	11.3	Base	No
	16731		482	235 (34.1)	480 (69.6)	23.0	25.2	52.5	20.2	Base	Yes
	16732		538	235 (34.1)	474 (68.7)	25.0	25.5	47.0	18.0	Base	Yes
	16733		649	220 (31.9)	415 (60.2)	20.0	21.0	40.4	9.4	Base	No
119B	16694	X-X-X SMAW	25	454 (65.9)	784 (113.7)	37.0	38.3	60.8	21.2	Base	No
	16695		593	251 (36.4)	580 (84.1)	38.0	38.3	43.8	30.0	Base	Yes
	16696		649	290 (42.0)	547 (79.3)	37.0	38.1	36.3	20.7	Base	No
	16697		760	308 (44.6)	450 (65.2)	14.0	19.0	30.0	30.0	Weld	No
	16698		871	253 (36.7)	267 (38.7)	18.1	18.1	36.2	36.2	Weld	No

^aSee Table 4.8 for detailed description.

Table 4.10. Tensile Data for Weldments Aged 2000 hr (Strain Rate 0.05/min)

Weld	Specimen	Weld Description ^a	Temperature, °C		Stress, MPa (ksi)		Total Elongation (%)	Reduction of Area, %		Fracture Location	Serrated
			Aging	Test	Yield	Ultimate		Sample	Weld		
121	16776	X-X-X GTA	593	25	492 (71.3)	855 (124.0)	28.8	35.4	30.8	Base	Yes
121	16777	X-X-X GTA	593	538	334 (48.4)	628 (91.1)	25.2	44.7	23.3	Base	No
122	16778	X-X-X GTA	649	25	720 (104.4)	1112 (161.3)	5.07	13.5	13.5	Weld	No
	16779	X-X-X GTA	649	649	524 (76.0)	695 (100.8)	9.84	30.5	30.5	Weld	No
	16780	X-X-X GTA	760	25	557 (80.8)	996 (144.4)	8.59	11.1	11.1	Weld	No
	16781	X-X-X GTA	760	760	351 (50.9)	437 (63.4)	21.3	56.2	56.2	Weld	No
	16782	X-X-X GTA	871	25	376 (54.6)	784 (113.7)	12.8	22.8	22.8	Weld	No
	16783	X-X-X GTA	871	871	198 (28.7)	244 (35.4)	49.8	75.4	75.4	Weld	No
125	16792	X-82-800H GTA	482	25	346 (50.2)	589 (85.4)	21.2	37.6	17.0	Base	No
126	16793	X-82-800H GTA	482	482	279 (40.4)	506 (73.4)	26.5	54.6	7.10	Base	Yes
	16794	X-82-800H GTA	538	25	410 (59.4)	641 (93.0)	22.9	45.8	4.16	Base	No
	16795	X-82-800H GTA	538	538	276 (40.0)	520 (75.4)	23.8	42.6	7.19	Base	Yes
	16796	X-82-800H GTA	649	25	392 (56.8)	647 (93.8)	14.1	29.5	10.8	Base	No
	16797	X-82-800H GTA	649	649	308 (44.6)	437 (63.4)	8.57	33.0	1.59	Base	Yes
123	16784	X-S-X GTA	593	25	478 (69.3)	834 (121.0)	29.6	35.4	16.3	Base	No
124	16785	X-S-X GTA	593	538	336 (48.8)	634 (92.0)	31.1	38.7	20.8	Base	Yes
	16786	X-S-X GTA	649	25	495 (71.8)	836 (121.3)	12.1	33.1	33.1	Weld	No
	16787	X-S-X GTA	649	649	346 (50.2)	616 (89.3)	16.3	39.1	39.1	Weld	Yes
	16788	X-S-X GTA	760	25	490 (71.0)	897 (130.1)	11.4	24.8	24.8	Weld	No
	16789	X-S-X GTA	760	760	323 (46.8)	467 (67.8)	31.2	63.7	11.1	Base	No
	16790	X-S-X GTA	871	25	393 (57.0)	800 (116.0)	19.7	23.0	23.0	Weld	No
	16791	X-S-X GTA	871	871	222 (32.2)	256 (37.2)	44.5	73.8	20.0	Base	No
117	16782	X-A-800H SMAW	482	25	315 (45.7)	584 (84.7)	24.7	63.8	6.89	Base	No
118	16763	X-A-800H SMAW	482	482	226 (32.8)	492 (71.3)	27.2	58.9	10.7	Base	Yes
	16765	X-A-800H SMAW	538	538	248 (36.0)	505 (73.2)	26.6	42.0	7.00	Base	Yes
	16766	X-A-800H SMAW	649	25	402 (58.3)	654 (94.8)	16.9	52.4	14.2	Base	No
	16767	X-A-800H SMAW	649	649	301 (43.7)	432 (62.6)	10.4	35.3	5.01	Base	No
119B	16768	X-X-X SMAW	593	25	466 (67.6)	811 (117.6)	23.7	36.0	3.46	Base	No
120	16769	X-X-X SMAW	593	593	301 (43.6)	603 (87.4)	22.7	34.0	3.33	Base	Yes
	16770	X-X-X SMAW	649	25	679 (98.5)	1023 (148.4)	6.34	17.3	2.22	Base	No
	16771	X-X-X SMAW	649	649	481 (69.8)	673 (97.6)	9.23	26.2	26.2	Weld	No
	16772	X-X-X SMAW	760	25	564 (81.8)	799 (115.9)	2.50	2.39	2.39	Weld	No
	16773	X-X-X SMAW	760	760	359 (52.1)	424 (61.5)	14.5	47.4	47.4	Weld	No
	16774	X-X-X SMAW	871	25	386 (56.0)	684 (99.2)	5.28	5.78	5.78	Weld	No
	16775	X-X-X SMAW	871	871	212 (30.7)	238 (34.5)	22.8	64.1	64.1	Weld	No

^aSee Table 4.8 for detailed description.

Fig. 4.1. Strength of Hastelloy X Weldments at 25°C After Aging for 2000 hr.

except 871°C caused the strength at 25°C to increase. Aging at 650°C caused the largest increase in strength, while aging at 871°C actually caused a slight decrease in yield stress. The weldment made with Hastelloy S filler metal displayed smaller strength changes than those made with Hastelloy X filler metal. The influence of aging on the reduction of area at room temperature is shown in Fig. 4.2. Ductility effects were greatest for all three weldments after aging at 760°C. The weldment made with Hastelloy S filler exhibited the least aging effect and the SMAW weld (Hastelloy X filler) exhibited the greatest effect of aging. The changes in the weldments of Hastelloy X to alloy 800H were relatively small. As shown in Fig. 4.3, the strength increased about 10% from aging at 538 and 649°C. The reduction of area at 25°C (Fig. 4.4) was decreased slightly for the GTA weldment but was unchanged for the SMAW weldment.

An aging experiment to be run in simulated HTGR helium will be started early in 1978. It will contain 144 creep and tensile, 48 fatigue, and 18 crack growth specimens. The materials being studied are Hastelloy X and Inconel 617 base metal and GTA weldments. The aging temperatures will

Fig. 4.2. Reduction of Area of Hastelloy X Weldments at 25°C After Aging for 2000 hr.

Fig. 4.3. Strength of Weldments of Hastelloy X to Alloy 800H at 25°C After Aging for 2000 hr.

ORNL-DWG 78-4436

Fig. 4.4. Reduction of Area of Weldments of Hastelloy X to Alloy 800H at 25°C After Aging for 2000 hr.

be 593, 704, and 871°C and the aging times will be 10,000, 20,000 and 50,000 hr. Postexposure evaluation will involve weight changes and tensile, creep, fatigue, and crack growth studies.

4.2.2 Creep Testing of Base Materials — H. E. McCoy and B. McNabb

4.2.2.1 Environmental Test Facilities

Ten environmental and six air creep frames operated during the year, and a new test facility having 22 environmental and 12 air creep frames was brought on line. A typical single environmental test machine is shown in Fig. 4.5. The environmental chamber consists of a 0.30-m-long (24-in.) section of 51-mm (2-in.) sched 40 type 304L stainless steel pipe. Water jackets on each end provide cooling for the seals. The seal between the pull rods and the environment is made with neoprene U-cups. U-cups are also used to seal the four extensometer rods, which exit from the bottom of the test chamber. The bellows on the top and bottom are for alignment, and the gas pressure on those bellows is not transmitted to

Fig. 4.5. Typical Environmental Creep Test Unit. Top half of the instrumentation rack contains control instruments for this machine and bottom half services machine to right (not shown).

the pull rods. The specimen is heated by a tube furnace capable of operation at 1200°C . The test gas enters the chamber at the top and exits from the bottom. Four Chromel vs Alumel thermocouples are used to monitor temperature. Three thermocouples go through Teflon seals at the top of the chamber and attach directly to the test specimen. One

thermocouple is just outside the furnace winding and operates in air. The temperature is initially controlled with the internal thermocouples; later control is switched to the external thermocouple to prevent thermocouple drift due to interaction with the test gas.

Strain is measured by extensometer rods, which extend directly from the top and bottom shoulders of the specimen. These rods are shown more clearly in Fig. 4.6. **The specimen extension can be read directly from a dial indicator or from a digital indicator.** The movement is converted to an electrical signal by the transducer attached to the dial gage and the electronic package in the bottom of the control cabinet (Fig. 4.5). The digital system automatically logs readings at 1-hr intervals.

Fig. 4.6. Partially Disassembled Environmental Test Unit Showing Top Bellows (Left), Top Pull Rod, Test Specimen, Bottom Pull Rod (Right), and Extensometer Rods.

The specimen used in these tests is shown in Fig. 4.7. A split grip fits around each end. This type of unthreaded connection gives excellent alignment, so that the bending stress in the specimen should be very small.

NOTE: ALL DIMENSIONS IN INCHES

$D_1 = 0.125 \pm 0.001$ DIAM

$D_2 = \text{FROM } 0.0010 \text{ TO } 0.0015$
GREATER THAN D_1

Fig. 4.7. Details of Creep Specimens for Environmental Tests. To convert dimensions to millimeters, multiply by 25.4.

The instrument package includes an L&N proportioning recorder and controller, an alarm to indicate off-temperature conditions, a unit to shut off power should the temperature rise above a preset value, and a solid-state power supply. The controls for two creep frames are located in one cabinet.

More general views of the new creep facility are shown in Figs. 4.8 and 4.9. Figure 4.8 shows six of the environmental creep units in service. Figure 4.9 shows 12 dead-load creep units for testing in either controlled or air environments.

The test gas is obtained from cylinders of helium containing 175 ppm H_2 , 17.5 ppm CH_4 , and 10.5 ppm CO . The test chambers are pressurized at 83 kPa gage (12 psig), so the test environment contains 318 $\mu\text{atm } H_2$, 32 $\mu\text{atm } CH_4$, and 19 $\mu\text{atm } CO$ (1 $\mu\text{atm} = 0.101 \text{ Pa}$). The gas is distributed to the individual test machines through 3.2-mm-diam (1/8-in.) stainless steel lines. The piping permits inlet and exit gas samples to be taken through the gas chromatograph. The flow rate to each machine is 0.25 ml/s except during start-up, when the flow rate is about 0.7 ml/s. The gas analyses of the

Photo 0574-78

Fig. 4.8. Row of Lever-Arm Environmental Test Units.

Fig. 4.9. Row of Dead-Load Creep Units for Tests in Controlled and Air Environments.

inlet and exit streams are identical except during the first 24 hr of each test, when all impurities run higher. The front and back of the gas distribution panel at the new facility are shown in Figs. 4.10 and 4.11, respectively.

Fig. 4.10. Front of Gas Distribution Panel. Chromatograph and moisture analyzer are below the panel. Gas flows from the panel to each machine and returns to exit.

Fig. 4.11. Back of Gas Distribution Panel Shows the Use of 3.2-mm-diam (1/8-in.) Stainless Steel Tubing and Small Bellows Valves for Flow Control.

The model 2110 Bendix gas chromatographs and model 340 Beckman moisture analyzers have been used during the year. Repairs to both instruments have been minimal. Impurity levels have generally remained within the limited ranges of the premixed gases. Leaks are usually detected by increased flow rates (since the system is pressurized),

rather than by changes in impurity levels. Since the gas contains hydrogen, it is passed through a furnace at 500°C as it leaves the cylinder to react the oxygen down to a very low level. The chromatograph readings do not detect oxygen in any samples unless there is an air leak (evidenced by presence of oxygen and nitrogen). All machines are read once per week, and the compositional ranges noted during the year are given in Table 4.11.

Table 4.11. Compositional Limits of Test Gases^a

Impurity	Level Noted (ppm)
H ₂	150-184
O ₂	10 ⁻²² atm ^b
N ₂	~1
CH ₄	12-16
H ₂ O	~5

^aTest chambers operate at 184 kPa (12 psig, 26.7 psia). Multiply ppm × 1.82 to obtain μ atm; by 0.184 to obtain Pa.

^bGas passed through furnace at 500°C to react O₂ with H₂. Estimate based on equilibrium.

4.2.2.2 Hastelloy X

The results of creep tests on Hastelloy X are summarized in Table 4.12. Many of these tests are in progress, and the information in Table 4.12 is incomplete. Tests were run in air and helium and involved two heats of material. The test specimens are mostly of the type shown in Fig. 4.7, but two other geometries were also used (Fig. 4.12). One specimen had a rectangular gage section 1.27 by 6.35 mm (0.050 by 0.250 in.) and was used to provide greater sensitivity to diffusion-controlled environmental influences. A double specimen was used for some air tests. The two test sections had slightly different diameters, which resulted in different stresses. Separate extensometers extended from the top and bottom of the

Table 4.12. Results of Creep Tests on Hastelloy X

Test	Temperature		Stress		Environment	Time, hr					Steady-State Creep Rate (hr ⁻¹) × 10 ⁻⁵	Elongation, %		Reduction of Area (%)
	(°C)	(°F)	(MPa)	(ksi)		to 1% Creep	to 2% Creep	to 5% Creep	to Tertiary	Rupture or Test		Loading	Creep	
Heat 4936														
19306	1150	2102	5.2	0.75	He	2	4	10	48	61	460	0.01	39.5	28.3
19230	1093	2000	14	2.0	He	3.5	5	9	6.5	28.0	450	0.00	37.1	27.9
19281	1093	2000	6.9	1.0	He	18	26	48	22	110	52	0.01	25.1	11.7
19181	1036	1897	17	2.5	He	30	61	95	68	154	31	0.15	23.1	23.0
19104	981	1798	28	4.0	He	32	49	86	39	165	23	0.21	21.7	20.4
18996	927	1700	41	6.0	He	16	38	90	65	198	47	0.005	30.3	36.0
18973	871	1600	69	10.0	He	11	24	63	65	157	78			60.4
18592	871	1600	48	7.0	He	210	430	770	400	1332	3.0	0.07	28.8	45.0
17850	871	1600	34	5.0	Air	1050	3000			5771 ^a	0.62	0.09	3.7	
16126	871	1600	34	5.0	He	3414	4000	5400	2350	6527	0.10	0.01	14.2	12.8
17375	816	1500	69	10.0	Air	119	400	970	690	1905	3.4		43.7	52.5
17332	816	1500	69	10.0	He	253	887	1745	900	2790	1.4	0.15	25.7	
17330	760	1400	138	20.0	Air	18	35	95	113	297	46		52.4	71.5
16125	760	1400	138	20.0	He	16	30	103	142	305	40		38.9	75.3
18451	760	1400	103	15.0	Air	65	240	830	753	2269	4.8	0.14	41.7	70.6
16490	760	1400	103	15.0	He	377	1110	2180	1480	2690	1.2	0.24	18.5	29.8
18450	760	1400	69	10.0	He					4148 ^{b,c}	0.71		1.4	
18449	760	1400	69	10.0	Air	875				4655 ^{b,c}	0.37		1.9	
17875	760	1400	69	10.0	He	2500				10199 ^b	0.11		1.8	
18861B	760	1400	69	10.0	Air					2537 ^b			1.4	
18861A	760	1400	52	7.5	Air					2537 ^b			0.2	
18879	704	1300	207	30.0	He					116 ^b				
15771	704	1300	172	25.0	He	49	104	321	510	1007	14	0.14	35.2	63.3
15058	704	1300	138	20.0	He	113	344	1920	1950	4045	1.7	0.10	21.4	5.8
15792	704	1300	138	20.0	He	148	500	2590	2000	4588	1.1	0.02	22.9	45.7
16268	704	1300	138	20.0	Air	98	297	2139	1950	4504 ^b	1.2	0.05	18.2	23.7
17376	704	1300	103	15.0	Air	150	550			13221 ^b	0.15	4.6		
17328	704	1300	103	15.0	He	350	5600			13494 ^b	0.11		4.8	
18878	649	1200	345	50.0	He					95.2				
17863	649	1200	207	30.0	He	375	560	1800	2600	4188	2.1	0.05	22.1	53.9
16267	649	1200	172	25.0	Air	250	700	3900	8750	16528	0.48	0.36	34.7	56.0
15772	649	1200	172	25.0	He	471	850	2170	3950	6466	1.7	0.15	34.7	52.8
18150	649	1200	138	20.0	He	1260				7550 ^b	0.34			
17329	649	1200	138	20.0	He	805	3163			4793 ^a		0.07	2.3	1.3
18435	649	1200	138	20.0	Air	1300				5923 ^{b,c}	0.60		2.5	
18434	649	1200	138	20.0	He					5923 ^{b,c}	0.39		2.2	
18436B	649	1200	138	20.0	Air	1200				5898 ^b	0.68		2.2	
18436A	649	1200	103	15.0	Air					5898 ^b	0.35		1.1	
Heat 2792														
16054	871	1600	62	9.0	He	93	197	327	205	554 ^b	9.5	0.07	34.7	68.5
18454	871	1600	34	5.0	He					3718 ^b			10.1	
16031	760	1400	152	22.0	He	7	15	38	44	159 ^b	120	0.09	56.6	80.5
18447	760	1400	69	10.0	He					4147 ^b			-0.07	
18448	760	1400	69	10.0	Air					4655 ^b			1.5	

^aDiscontinued before fracture.^bTest in progress.^cFlat specimen.

Y-147872

Fig. 4.12. Test Specimens. Middle specimen used in most tests, lower specimen used to accent environmental effects, and upper double specimen used only in air to better utilize test equipment. Scale is 25 mm.

furnace to give strain measurements for each segment. Small auxiliary heaters were used on the top and bottom pull rods to help offset heat losses out the ends of the furnace. These double specimens give better equipment utilization.

The rupture data are shown in Fig. 4.13, and the data for time to 1% strain are shown in Fig. 4.14. Although the data are fragmentary at this point and conclusions are tentative, test environment (air or He) or which heat of material is involved appears to have very little influence. Two sets of lines are shown in Fig. 4.13. The solid lines were obtained by fitting the results of 268 tests with a model of the form^{*}

$$\log t = b_0 + b_1/T + b_2 T \log \sigma + (b_3/T) \log \sigma ,$$

^{*}Models fit by T. L. Hebble, Computer Sciences Division, ORNL.

Fig. 4.13. Stress-Rupture Data for Hastelloy X.

Fig. 4.14. Time to 1% Strain for Hastelloy X.

where

t = time,

T = temperature,

σ = stress, and

b_0, b_1, b_2, b_3 = constants.

The dashed line was derived by fitting most of the data in Table 4.12 to a model of the form^{*}

$$\log \dot{\epsilon} = a_0 + a_1/T + (a_2/T) \log \sigma .$$

The data for the two heats of material involved in this study show significantly greater strengths than most of the test data available for Hastelloy X.

The minimum creep rate is shown as a function of stress in Fig. 4.15. The time to 1% strain ($t_{1\%}$) is shown in Fig. 4.16 as a function of the time to rupture (t_r). The data show a lot of scatter but correlate reasonably well with the equation

$$t_{1\%} = 0.072 t_r^{0.982} .$$

A similar correlation for the time to tertiary creep (t_t) is shown in Fig. 4.17. The data correlate very well with the equation:

$$t_t = 0.274 t_r^{1.065} .$$

The rupture ductilities (Table 4.12) generally show that the fracture strain decreases with increasing rupture life and temperature.

4.2.2.3 2 1/4 Cr-1 Mo Steel

The test results for 2 1/4 Cr-1 Mo steel are given in Table 4.13. Three heats of material were included in this study (see Table 4.2 for

^{*}Models fit by T. L. Hebble, Computer Sciences Division, ORNL.

Fig. 4.15. Minimum Creep Rate of Hastelloy X As a Function of Stress.

Fig. 4.16. Time to 1% Strain as a Function of Rupture Life for Hastelloy X.

Fig. 4.17. Correlation of Time to Tertiary Creep as a Function of Rupture Life for Hastelloy X.

detailed chemical analyses). The time to 1% strain is shown in Fig. 4.18 as a function of stress. Cross plots of pairs of tests show some support for the premise that this steel is stronger in air than in helium. However, the data shown in Fig. 4.18 do not totally support this conclusion, and further testing is needed. The time to rupture is shown in Fig. 4.19 as a function of stress. The lines in this figure were derived from a model by Booker et al.,⁴ which applies to 563 tests run at temperatures ranging from 454 to 600°C. The data from the present investigation agree quite well with the predictions of the model. Figure 4.20 makes use of another model by Booker et al., in which the time to tertiary creep is related to the time to rupture. The data fit the model very well, but at least four points for tests at 482°C fall to the right of the predicted line. Since Booker's model was not determined from tests run at 482°C, this deviation is not unreasonable.

Thus, the test results for 2 1/4 Cr-1 Mo steel indicate that the material is weaker in helium than in air but fail to demonstrate this

Table 4.13. Results of Creep Tests On 2 1/4 Cr-1 Mo Steel

Test	Temperature		Stress		Environment	Time, hr					Steady-State Creep Rate (hr ⁻¹) × 10 ⁻⁵	Elongation, %		Reduction of Area (%)
	(°C)	(°F)	(MPa)	(ksi)		to 1% Creep	to 2% Creep	to 5% Creep	to Tertiary	Rupture or Test		Loading	Creep	
Heat X-6216														
17864	649	1200	68.9	10.0	He	167	248	370	140	513	3.9		29.1	86.6
17333	593	1100	138	20.0	He	5	11	34	42	89.5	130		42.1	86.6
17334	593	1100	103	15.0	He	39	105	306	365	793	14	0.02	44.5	80.6
15047	593	1100	68.9	10.0	He	2990	4553	6510	3150	7784	0.25	0.10	26.6	63.3
17876	593	1100	68.9	10.0	Air	3700				10327 ^a			4.4	
15023	593	1100	68.9	10.0	He	3000	4950	7350	3750	9660 ^b	0.24	0.08	38.9	77.0
15045	593	1100	68.9	10.0	He	3900				3186 ^b	0.22	0.15	2.8	
17918	538	1000	138	20.0	He	95	225	674	1050	2655	6.7	0.085	38.9	75.5
15046	538	1000	103	15.0	He	1086	2777	7640	8000	14957	0.61	0.05	38.2	75.8
17494	482	900	207	30.0	He	300	750	2000	2300	4895	2.3	0.17	30.3	77.5
Heat 36202														
17948	649	1200	68.9	10.0	He	47	97	204	115	361	18		34.6	84.9
16883	593	1100	103	15.0	He	17	48	144	188	436	32		46.8	83.0
18149	538	1000	138	20.0	He	65	170	475	820	1717	9.8	0.15	40.5	75.2
16264	538	1000	103	15.0	Air	1150	2800	11050		17918 ^a	0.19		8.1	
15369	538	1000	103	15.0	He	842	2047	6144	6800	15232	0.75	0.07	41.6	62.3
18222	482	900	241	35.0	He	50	200	670	675	1796	6.0	0.20	32.3	74.7
16263	482	900	207	30.0	Air	204	620	1800	2030	6611	2.5	0.25	45.1	71.1
15373	482	900	207	30.0	He	349	787	1660	500	3494	1.1	0.95	34.8	73.7
15374	482	900	172	25.0	He	1500	2705	5200	1800	13509	0.39	0.40	39.7	70.9
17849	482	900	172	25.0	Air	1250	2900			10656 ^a	0.58		6.4	
Heat 72768														
17495	649	1200	68.9	10.0	He	28	59	137	100	288	31		47.2	87.0
18075	649			5.0	He	2025	2400	2950	1800	4044	0.11		48.0	71.4
18446	649	1200	20.7	3.0	He					5065 ^{a,c}			0.45	
18862	649		20.7	3.0	He					2372 ^a			0.23	
17377	593	1100	103	15.0	He	13	30	92	130	328	49	0.03	50.8	82.9
16882	593	1100	103	15.0	He	0.8	3	81	600	1337	4.4	0.40	50.9	81.6
16266	593	1100	103	15.0	Air	10	26	90	175	388	48	0.07	52.4	69.5
16360	593	1100	68.9	10.0	Air	971	2115	4435	1625	6698	0.54		27.3	81.3
18859	538	1000	103	15.0	Air					2634 ^a			2.6	
18860	538		68.9	10.0	He					2517 ^a			-0.06	
18508	538	1000	48.3	6.0	He					6019 ^{a,c}			-0.24	
18252	538	1000	48.3	6.0	He					7139 ^a			0.15	
17947	482	900	207	30.0	He	186	402	860	600	2471	4.4	0.72	41.3	78.9
18438	482	900	103	15.0	He					5659 ^a			0.08	
18374	482	900	103	15.0	He					6404 ^a			0.26	
18439	482	900	103	15.0	Air					5853 ^{a,c}			0.13	
18437B	482	900	103	15.0	Air					5877 ^a			0.18	
18437A	482	900	88.5	11.0	Air					5877 ^a			0.11	

^aTest in progress.^bDiscontinued.^cFlat specimen.

ORNL-DWG 78-4422

Fig. 4.18. Creep Results for 2 1/4 Cr-1 Mo Steel: Time to 1% strain.

ORNL-DWG 78-4423

Fig. 4.19. Stress-Rupture Results for 2 1/4 Cr-1 Mo Steel.

Fig. 4.20. Time to Tertiary Creep as a Function of Time to Rupture for 2 1/4 Cr-1 Mo Steel.

effect unequivocally. Carbon contents (see Sect. 4.2.2.5) show that the material is decarburized at 593 and 649°C, and this would account for the observed environmental effect.

4.2.2.4 Miscellaneous Alloys

The test results for Inconel 617, Hastelloy S, alloy 800H, and 9 Cr-1 Mo steel are summarized in Table 4.14. The efforts on these materials are quite small compared with those on Hastelloy X and 2 1/4 Cr-1 Mo steel. The times to 1% strain for Inconel 617 and Hastelloy S are compared in Fig. 4.21 with those for Hastelloy X. Both Hastelloy S and Inconel 617 are stronger than Hastelloy X. A similar comparison of the rupture strengths of the three materials is shown in Fig. 4.22, and the results again show that Hastelloy S and Inconel 617 are stronger than Hastelloy X. Data are insufficient to make a valid comparison between Hastelloy S and Inconel 617.

Table 4.14. Results of Creep Tests On Miscellaneous Alloys

Test	Temperature		Stress		Environment	Time, hr					Steady-State Creep Rate (hr ⁻¹) × 10 ⁻⁵	Elongation, %		Reduction of Area (%)
	(°C)	(°F)	(MPa)	(ksi)		to 1% Creep	to 2% Creep	to 5% Creep	to Tertiary	Rupture or Test		Loading	Creep	
Inconel 617														
18873	871		34.5	5.0	Air					658 ^a			0.8	
18868	850		103	15.0	Air	6	10	25	65	107	200	0.01	66.2	71.2
18866	850		68.9	10.0	Air	36	150	400	145	1879	12	0.055	32.7	34.6
18869	760		68.9	10.0	Air					1342 ^a			0.6	
17563	760		172	25.0	He	9	20	57	105	280	89	0.23	53.6	74.1
19106	650		414	60.0	He	1	55	157	168	186	16	15.3	7.5	16.1
18483	650		276	40.0	He					6013 ^a	0.070		0.92	
19182	650		207	30.0	He					1605 ^a			-0.06	
18440	650		276	40.0	Air					5829 ^a	0.11		0.93	
Hastelloy X														
18874	871		34.5	5.0						644 ^a			0.70	
18441	650		207	30.0	He	260	465			5587 ^a	0.48		5.8	
Alloy 800H														
18453	760		68.9	10.0	He	210	355	625	275	1163 _b	3.7	0.37	17.2	21.7
18783	649		172	25.0	He	25	59	97	50	121 _b	18	5.2	9.1	12.6
18926	649		172	25.0	He	60	130	410	95	449	8.9	2.1	10.4	8.84
19309	649		103	15.0	He					523 ^a			0.06	
19226	649		103	15.0	Air					1363 ^a			0.43	
9 Cr-1 Mo Steel														
18676	538		138	20.0	Air	300	900	2100	1800	4475	1.63	0.00	44.1	83.3
15363	538		103	15.0	He	3000	4500	7600	3200	13584 ^a	0.24		19.5	

^aTest in progress.^bTest discontinued prior to fracture.

Fig. 4.21. Time to 1% Strain for Inconel 617 and Hastelloy S Compared With that for Hastelloy X (Solid Lines).

Fig. 4.22. Time to Rupture for Inconel 617 and Hastelloy S Compared With that for Hastelloy X (Solid Lines).

The ORNL and GA results for alloy 800H are shown in Fig. 4.23. The lines are copied from ref. 5. The purpose of these tests is to compare results between ORNL and GA, and the results thus far indicate acceptable agreement.

Fig. 4.23. Creep Properties of Alloy 800H.

4.2.2.5 Changes in Carbon Content

The HTGR helium significantly affects the carbon content of specimens exposed to the gas. Post-test carbon determinations on creep specimens and small metal tabs have indicated the changes in carbon content that occur during exposure. This program has involved various specimen geometries having cross sections of 1.6 mm (1/16 in.) diameter, 3.2 mm (1/8 in.) diameter, and 1.6 by 13 mm (1/16 by 1/2 in.). In most cases the samples were taken so that the resultant analytical values are averages of the entire cross section.

The results for 2 1/4 Cr-1 Mo steel are shown in Fig. 4.24. There is a slight indication that carburization occurred at 482°C, that no detectable change occurred at 538°C, and that decarburization occurred at 593 and 649°C. The results for Hastelloy X are shown in Fig. 4.25. Carburization occurred at 871 and 816°C, but no detectable changes occurred at 760, 704, and 649°C.

Fig. 4.24. Carbon Contents of 2 1/4 Cr-1 Mo Steel Exposed to HTGR Helium.

Fig. 4.25. Carbon Content of Hastelloy X Exposed to HTGR Helium.

Small coupons of Hastelloy N modified with 2% Ti (Ni-12% Mo-7% Cr-2% Ti-0.05% C) were exposed together with Hastelloy X coupons. The times were relatively short; the most severe exposure was 198 hr at 871°C. Under these conditions the titanium-modified Hastelloy N did not show detectable carburization, whereas Hastelloy X was carburized. Additional tests will be run to further evaluate the resistance of titanium-modified Hastelloy N to carburization.

4.2.3 Creep Properties of Weldments — H. E. McCoy, B. McNabb, and J. F. King

One set of GTA weldments tested was Hastelloy X joined to Hastelloy X with Hastelloy X filler metal and Hastelloy X joined to alloy 800H with ERNiCr-3 filler metal. These weldments were aged for 2000 hr at several temperatures and then subjected to post-aging creep tests in an air environment. The results of these tests are given in Table 4.15.

Most of the Hastelloy X to Hastelloy X specimens failed in the weld metal (Table 4.15). The creep strains were lowest for the specimens aged at 649°C and varied from 5.8 to 7.5%. The specimens aged at 760°C had creep strains at fracture of 10 to 20%, and those aged at 871°C had fracture strains from 18 to 41%. As shown in Fig. 4.26, the rupture time was a strong function of the pretest aging treatment. Generally, the rupture time was slightly greater than that for Hastelloy X base metal (shown by lines in Fig. 4.26) except for those specimens aged at 871°C.

The failures in the weldments of Hastelloy X to alloy 800H consistently occurred in the alloy 800H base metal, and very little deformation occurred in the weld deposit. Aging at 538°C produced the lowest fracture strains and longest rupture times (Fig. 4.27). The shortest rupture times resulted from aging at 649°C, but the rupture time was equal to that of the base metal. The time to rupture varied by a factor of 10 depending upon the pretest aging treatment.

The weldments described in Table 4.8 were creep tested in HTGR helium. These welds involved Hastelloy X base metal, two filler metals, and two welding processes. The results of these tests are given in Table 4.16, and the stress rupture properties are shown in Fig. 4.28. The time to rupture at a given stress level was equal to or greater than that of the base metal for all filler metals and welding processes. Generally, the

Table 4.15. Creep Tests on Hastelloy X and Incoloy 800 Weldments

Test	Temperature, °C		Stress		Strain, %		Minimum Creep Rate (%/hr)	Time, hr, to				Failure Location	Reduction of Area, %	
	Aging ^a	Test	(MPa)	(ksi)	Loading	Creep		1% Strain	5% Strain	Tertiary	Rupture		Sample	Weld ^b
Air Hastelloy X														
17536	unaged	760	103	15.0	1.56	7.00	0.00079	10	2250	1800	2371	Weld	31.0	2.2
17554	659	760	103	15.0	0.46	6.9	0.000080	3450	3730	3300	3734	Weld	24.6	
17539	760	760	103	15.0	0.32	15.2	0.0011	400	1950	1250	2660	Base	45.2	
17558	871	760	103	15.0		29.3	0.022	30	215	270	672	Weld	75.2	
17537	unaged	760	138	20.0	0.043	22.5	0.024	10	150	160	273	Weld	60.0	
17553	649	760	138	20.0	0.024	0.024	0.0018	325	495	350	500	Weld	47.6	
17540	760	760	138	20.0	0.37	7.54	0.027	15	114	73	193	Weld	62.8	
17555	871	760	138	20.0	0.046	40.5	0.15	3	30	45	138	Weld	78.0	
17538	unaged	871	48	7.0	0.18	17.8	0.00011	250	1350	750	2099	Weld	48.3	3.5
17557	649	871	48	7.0		5.8	0.0017	410		2100	2341	Weld	13.0	
17541	760	871	48	7.0	0.11	10.9	0.0010	625	1950	1000	2313	Base	34.7	
17559	871	871	48	7.0	0.16	17.7	0.0057	160	550	400	1184	Base	53.1	
Hastelloy X to Alloy 800H														
17544	unaged	538	345	50.0	4.72	6.0	0.000075	4700	8225	7600	8227	800H	12.4	-0.5
17547	427	538	345	50.0	5.60	2.76	0.000004	1000		5650	6985	800H	13.7	-1.1
17550	538	538	345	50.0	2.51	2.26	0.000060	6500		5350	6797	800H	11.1	-0.9
17543	649	538	345	50.0	1.50	12.8	0.013	60	365	370	577	800H	49.2	0.6
17549	unaged	649	172	25.0	0.15	5.64	0.00048	1600	2700	2250	2720	800H	27.2	-0.6
17556	427	649	172	25.0	0.21	3.95	0.00062	620		1800	2009	800H	14.71	0.5
17551	538	649	172	25.0	0.23	4.44	0.00023	2300		5670	5758	800H	3.6	1.6
17542	649	649	172	25.0	0.10	8.91	0.0023	170	940	500	1029	800H	48.0	0.6
17548	unaged	649	241	35.0	0.81	9.81	0.0098	68	168	94	183	800H	44.2	0.3
17546	427	649	241	35.0	0.63	8.26	0.014	57	158	72	167	800H	37.4	11.3
17552	538	649	241	35.0	0.30	6.07	0.0068	90	590	575	593	Weld	15.3	1.9
17545	649	649	241	35.0	0.22	12.4	0.086	11	45	22	60.9	800H	48.5	

^a Aged for 2000 hr.^b Applies to weld when fracture was in base metal.

Fig. 4.26. Stress-Rupture Properties of Hastelloy X Weldments in Air. Lines represent the average properties of Hastelloy X sheet.

Fig. 4.27. Stress-Rupture Properties of Weldments of Hastelloy X to Alloy 800H in Air. Lines represent the average properties of annealed alloy 800H.

Table 4.16. Results of Creep Tests in HTGR Helium on Weldments of
Hastelloy X to Itself and to Alloy 800H

Test	Temperature		Stress		Time, hr					Steady-State Creep-Rate (hr ⁻¹)	Elongation, %		Reduction of Area (%)	Failure Location
	(°C)	(°F)	(MPa)	(ksi)	to 1% Creep	to 2% Creep	to 5% Creep	Tertiary Creep	Test or Rupture		Loading	Creep		
Welds 119B, 120, all Hastelloy X, SMAW														
17564	593	1100	345	50.0	2300				>6690 ^a	0.30 × 10 ⁻⁵		2.1		
18422	649	1200	207	30.0	375	2200		1600	2185	0.35	0.03	3.91	11.0	Weld
18424	760	1400	138	20.0	155	208		201	209	1.3	0.06	4.64	16.1	Weld
18425	871	1600	69	10.0	155			147	157	0.20	0.03	4.0	14.2	Weld
Welds 121, 122 All Hastelloy X, GTA														
18426	593	1100	241	35.0	2100				>6352 ^a	0.50				
18864	649	1200	241	35.0	125	235	880	1080	1353	4.2		3.4		
18426	649	1200	207	30.0	190	470	4250	4025	4404	0.53	0.30	10.2	38.5	Weld
18428	760	1400	138	20.0	3	225			477		0.003	6.5	19.4	Weld
18429	871	1600	69	10.0	140	220	283	180	330	5.0	0.40	7.1	44.1	Weld
											0.06	16.1	30.1	Weld
Welds 123, 124, Hastelloy X with Hastelloy S Filler, GTA														
18430	593	1100	241	35.0	1900				>6188 ^a	0.28		2.2		
18431	649	1200	207	30.0	375	770	2300	4300	5455	2.1	0.13	21.6	78.7	Weld
18432	760	1400	138	20.0	21	49	120	110	326	38.0	0.18	26.3	71.6	Weld
18433	871	1600	69	10.0	51	80	158	67	198	16.0	0.20	9.8	31.7	Weld
Welds 117, 118, Alloy 800H to Hastelloy X with Inco A, SMAW														
18442	482	900	414	60.0					>5565 ^a			3.2		
18863	538	1000	414	60.0					164 ^a			0.8		
18443	538	1000	345	50.0	3266	3266			3266	0.13	4.6	2.0	15.9	Base
18870 ^b	538	1000	345	50.0					>956 ^a			0.2		
18444	649	1200	172	25.0	650	1800	2318	1665	2318	0.65	0.16	15.2	20.4	Base
Welds 125, 126 Alloy 800H to Hastelloy X with ERNiCr-3, GTA														
18445	482	900	414	60.0					>4821 ^a	0.16		1.5		
18867	538	1000	414	60.0	1	155		340	340	1.4	9.2	4.5	21.8	Base
18452	538	1000	345	50.0					>4199 ^a	0.42		0.28		
18703	649	1200	172	25.0	938	1930	2180	1880	2189	0.60	-0.18	5.1	15.7	Base

^aTest in progress.

^bAged 2000 hr.

Fig. 4.28. Stress-Rupture Lives of Hastelloy X Weldments in HTGR Helium.

rupture time was least for the Hastelloy X filler metal deposited by the SMAW process and greatest for the Hastelloy X filler deposited by the GTA process. As indicated in Table 4.16, fracture occurred in the weld metal in all test specimens. The fracture strain was least for the weld metal deposited by the SMAW process (~4%) and greatest for Hastelloy S deposited by the GTA process (~20%).

The results of creep tests on two weldments of Hastelloy X to alloy 800H are summarized in Table 4.16 and Fig. 4.29. The rupture times for both weldments were quite comparable with those for base metal and were similar to each other. The fracture occurred in the alloy 800H base metal, and creep fracture strains as low as 2% were noted. The data are insufficient to draw a conclusion concerning the superiority of one weld metal over the other.

4.2.4 Low Cycle Fatigue and Creep-Fatigue — J. P. Strizak

Studies are being conducted to characterize the fatigue and creep-fatigue behavior of structural alloys proposed for use in HTGR systems.

Fig. 4.29. Stress-Rupture Lives of Weldments of Hastelloy X to Alloy 800H in HTGR Helium.

The principal HTGR high-temperature alloys are Hastelloy X, 2 1/4 Cr-1 Mo steel, and alloy 800H. Emphasis thus far has been placed on providing a significant understanding of the fatigue behavior of 2 1/4 Cr-1 Mo steel and Hastelloy X.

Current elevated-temperature ASME code design allowable stresses are based on properties measured in air, but the impact of HTGR environments on these properties must be determined for design and safety. During the past year an environmental fatigue testing facility was put into operation, and 2 1/4 Cr-1 Mo steel is being tested in a prototypical helium environment. A number of continuous cycling strain-controlled fatigue tests were conducted at 538°C on 2 1/4 Cr-1 Mo steel in a helium environment containing about 300 μatm^* H_2 , 30 μatm CH_4 , 18 μatm CO , 5 μatm of H_2O , and trace quantities of N_2 . Fatigue life in the helium environment is compared with that in air in Fig. 4.30. The data indicate that fatigue life of 2 1/4 Cr-1 Mo steel may be slightly greater in helium. However, only a limited number of short-term (<110 hr) helium tests were conducted. Additional experimental details are given in other reports.^{6,7}

* 1 μatm = 0.101 Pa.

Fig. 4.30. Comparison of Results of Continuous Cycling Strain-Controlled Fatigue Tests Conducted on 2 1/4 Cr-1 Mo Steel in Air and HTGR Helium Environments at 538°C.

The effect of the helium environment on longer term behavior of the material was more pronounced. Creep-fatigue tests were conducted in the helium environment at 538°C under the following cyclic strain conditions: (1) 0.5% total strain range, (2) $4 \times 10^{-3}/\text{s}$ strain rate, with (3) 0.1-hr hold periods each cycle at peak tensile and/or compressive strain amplitudes. Results of these tests are compared in Table 4.17 with similar tests conducted in air.

Contrary to the general behavior of 2 1/4 Cr-1 Mo steel in air, tensile hold periods in helium reduced the continuous cycling fatigue life to a larger extent, and were more damaging than compressive hold periods. In terms of cyclic life a combined tensile and compressive hold mode was the most damaging. The lower cyclic life of specimen ITL-27 suggested an interaction of the tensile and compressive hold periods. Accordingly, an attempt was made to estimate the fatigue life of specimen ITL-27 from other specimen life data in helium and the following interaction formulation:

Table 4.17. Comparison of Results of Continuous Cycling and Creep-Fatigue Tests^a Conducted in Air and a Simulated HTGR Helium Environment at 538°C

Specimen	Environment	Hold Mode ^b	Hold Period (hr)	Values at $N_f/2$		Cycles to Failure N_f	Time to Failure t_f (hr)
				Plastic Strain Range $\Delta \epsilon_p$ (%)	Stress Range $\Delta \sigma$ (MPa)		
MIL-10	Air		0	0.280	391.0	16,036	11.1
ITL-111	HTGR		0	0.257	510.2	21,645	15.0
MIL-12	Air	C	0.1	0.330	367.6	4,496	453.0
ITL-109	HTGR	C	0.1	0.314	417	11,188	1126.3
ITL-106	Air	T	0.1	0.252	436	15,450	1555.6
ITL-101	HTGR	T	0.1	0.293	437	5,860	589.9
c	Air	T+C	0.1				
ITT-27	HTGR	T+C	0.1	0.318	413	2,953	592.4

^aAll tests were conducted in completely reversed strain control with a total strain range $\Delta \epsilon_t = 0.5\%$ at a strain rate, $\dot{\epsilon} = 4 \times 10^{-3}/s$.

^bT implies a hold period at peak tensile strain amplitude; C, compression.

^cTest to be scheduled.

$$N_f^{tc} = N_f^t N_f^c / N_f^o ,$$

where

N_f^t = cyclic life of a specimen at a given strain range, temperature, and tensile hold time,

N_f^c = cyclic life of a specimen at a given strain range, temperature, and compressive hold time,

N_f^o = cyclic life of a specimen at a given strain range and temperature without a hold period, and

N_f^{tc} = cyclic life of a specimen subjected to both tensile and compressive hold times.

The predicted number of cycles to failure was 3029 in comparison with an actually observed life of 2953 cycles. Another set of data will be obtained at 482°C under the same cyclic strain conditions to support the above initial observations.

In addition to the environmental testing program discussed above, experience was gained throughout the year in the use of strain-range partitioning

concepts for predicting the time-dependent strain controlled cyclic lifetimes of uniaxial specimens of 2 1/4 Cr-1 Mo steel and Hastelloy X. Interim results of analyses of available creep-fatigue data obtained in air have been presented in several reports.^{8,9} Generally experimental and predicted lifetimes agreed well. Application of strain-range partitioning concepts to environmental effects will be investigated as sufficient data are available.

4.2.5 Fatigue Crack Growth — J. P. Strizak

Interim observations of the effects of a simulated HTGR helium environment on the fatigue subcritical crack growth behavior of Hastelloy X and 2 1/4 Cr-1 Mo have been discussed in several recent reports.

A preliminary investigation of the effects of temperature, frequency, and environment on the fatigue crack growth rate (FCGR) of Hastelloy X in the solution annealed condition has been reported.¹⁰ Of the three variables, temperature had the greatest effect. The FCGR at 871°C was roughly an order of magnitude greater than that at 538°C in air and HTGR helium. The effect of test frequency, however, was small within the range (1.0 and 0.1 Hz) investigated. A tenfold decrease in frequency increased the FCGR at 538 and 871°C by a factor of approximately 1.5. Finally, the FCGR was noticeably higher in air than in HTGR helium, as shown in Fig. 4.31. The FCGR of isothermally annealed 2 1/4 Cr-1 Mo steel has been reported¹¹ as a function of stress intensity, temperature, mean stress, frequency, and environment. The growth rate in air increased markedly with stress intensity, temperature, and inverse frequency. Small variations in the growth rate in air were due to heat-to-heat differences, aging, carbon content, and welding effects. Growth rates in vacuum and helium with controlled impurities were substantially less than those measured in air, as shown in Fig. 4.32. Growth rates in steam were only slightly less than in air. An analytical model of the results in air was prepared as a multiple function of stress intensity, temperature, and frequency. The model is useful to predict optimal material behavior and upper limit growth rates for use in safety analysis.

Fig. 4.31. The Fatigue Crack Growth Rate of Hastelloy X is Greater in Air than in Simulated HTGR Helium at (a) 538°C and (b) 871°C.

Fig. 4.32. Fatigue Crack Growth Rate of 2 1/4 Cr-1 Mo Steel in Air at 593°C (1100°F) and 0.67 Hz with $R = 0.05$ Compared with that in (a) Steam and (b) HTGR Helium and Vacuum.

4.3 WELDING DEVELOPMENT — J. F. King

Several components in various gas-cooled reactor system designs call for Hastelloy X to be used as a structural material. During this reporting period the welding program investigated the weldability of Hastelloy X and alloy 800 and the metallurgical stability of welds joining Hastelloy X to Hastelloy X and to alloy 800H using the shielded metal-arc welding (SMAW) process.

4.3.1 Weldability Testing — Hastelloy X

Weldability testing, using the Tigamajig hot-cracking test, was conducted on several commercial heats of Hastelloy X. The objective was to determine how heat-to-heat variations affect this alloy's susceptibility to hot-cracking. The basics of the Tigamajig test equipment have been previously reported¹² and will not be discussed here since usual testing procedures were followed. Cracking data from this test were obtained from the total crack length measured as a function of percent augmented strain used on each specimen.

Nine different heats of Hastelloy X plate material were obtained for this comparison. Six heats were in the form of 9.5-mm-thick plate and three were 12.7-mm-thick. The chemical composition of each plate conforms to ASTM specification B 435. No peculiarities were observed during testing of any material heat.

Specimens were prepared by making weld deposits by the shielded metal-arc (SMA) and gas tungsten-arc (GTA) welding processes and machining to the same coupon geometry used for base metal testing. Hot-cracking tests were conducted by remelting the deposited weld metal according to the same test procedure employed on base metal specimens. The weld metal deposits of the two processes were made with the same heat of Hastelloy X filler metal. The SMA electrodes had a flux coating added to a core wire of the same heat as the bare wire used for GTA welding.

Total crack length measurements were obtained from each deposit and compared with the base material data shown in Fig. 4.33. Crack lengths for the GTA deposited weld metal fell along the lower portion of the scatter band for the base material. The SMA deposits exhibited slightly less cracking, with all data points falling below the scatter band.

Fig. 4.33. Tigamajig Hot-Cracking Data for Nine Commercial Heats of Hastelloy X Shows Relatively Large Variation in Cracking Tendency.

This testing program has established a base for future comparison of Hastelloy X heats requiring weldability evaluation. Relatively large variations in cracking tendency were observed among these nine heats. However, no correlation of hot-cracking with chemical composition was evident. In addition, none of the heats tested would be expected to cause problems during welding. The heat of Hastelloy X exhibiting the most cracking has been extensively used in laboratory welding experiments with no problems. The weld metal test results showed little difference between the deposits made by the two processes and that both are generally less susceptible to cracking than the base material.

4.3.2 Weldability Testing — Alloy 800

Seven commercial heats of alloy 800 were selected for weldability testing using the Tigamajig device. The heats were selected mainly because

of availability and not for any specific characteristic they possessed. They included sheet material, plates of various thicknesses, pipe, and both alloy 800 and 800H. The one exception to random selection of material was a heat that had exhibited heat-affected zone fissuring during welding. Examination of the chemical analysis of this heat revealed it to be in accordance with ASTM specification B 409, and no reason was apparent for it being crack sensitive.

Each heat was tested at five strain levels ranging from 1.4 to 4.9%. The sum of all crack lengths for each specimen was obtained and plotted as a function of augmented strain (Fig. 4.34). Cracking data for six heats of alloy 800 are shown in the crosshatched scatter band. The crack-sensitive heat, shown by the dashed line, clearly exhibited a higher cracking tendency in this test than did the other heats. Thus, the Tigamajig test procedure appears to be useful for determining the relative weldability of alloy 800.

After establishing the ability of the Tigamajig test to detect crack-sensitive heats of alloy 800, we tried to correlate cracking data with variations in chemical analyses. No good correlation was found. No single element or combination of elements produced reproducible correlations. The ratio $(Al + Ti)/(C + Si)$ was found, however, to be a reasonable predictor of cracking behavior compared with the other variations evaluated. This ratio was previously suggested by York and Flury,¹³ who indicated that heats with this ratio less than 2 had lower resistance to hot cracking.

More detail on this work is reported elsewhere.¹⁴

4.3.3 Shielded Metal-Arc Weldments of Hastelloy X

The objective of this study was to determine the tensile properties and aging characteristics of Hastelloy X weldments made with Hastelloy X SMA electrodes. The welds were made in 12.7-mm-thick plate by the manual SMA process. After radiographic inspection, specimens were thermally aged at 649, 760, and 871°C for periods of time up to 7.2 Ms (2000 hr).

Room-temperature tensile tests were performed on specimens taken transverse to the weld in these aged Hastelloy X weldments. The tensile specimens contained weld metal, heat-affected zone, and base metal in the gage length. The results of these tests are presented in Table 4.18. All

Fig. 4.34. Tigamajig Test Data for Alloy 800 Shows that Crack Sensitive Material (Heat A) can be Identified by this Test.

ruptures occurred in the weld metal except for specimens aged at 649°C for periods of 0.36 and 3.6 Ms (100 and 1000 hr). Aging at 649°C for 7.2 Ms raised the yield and ultimate tensile strengths of the Hastelloy X weldments to the highest level. However, the loss of ductility was significant, with the reduction of area decreasing from nearly 40% in the as-welded coupons to 9% after aging. These data for the SMA weldments are very similar to those on GTA weldments of Hastelloy X previously reported.¹² Significant differences are not apparent in the aging response of these two types of weldments.

Microhardness measurements across these weldments also reflected age hardening of the material. Generally weld metal and base metal exhibited

Table 4.18. Room-Temperature Tensile Data for Hastelloy X Shielded Metal-Arc Weldments^a

Aging Conditions		Strength, MPa		Reduction of Area (%)	Fracture Location
Temperature (°C)	Time (Ms)	Tensile	Yield		
As-Welded		742.8	425.9	37.3	Weld metal
649	0.36	804.4	452.8	30.5	Base metal
649	3.6	981.4	583.0	21.5	Base metal
649	7.2	1054.0	675.3	8.83	Weld metal
760	0.36	931.5	565.4	8.7	Weld metal
760	3.6	872.5	532.7	10.3	Weld metal
760	7.2	908.3	532.3	10.8	Weld metal
871	0.36	847.6	460.0	15.5	Weld metal
871	3.6	778.2	401.0	14.5	Weld metal

^aValues are average of three tests. Tensile specimens taken transverse to welding direction.

the same hardnesses for the 760 and 871°C aging treatments. The weldments aged at 649°C showed similar hardnesses for the two metals, but the heat-affected zone was harder after aging for 7.2 Ms. The peak hardness of the HAZ near the fusion line was 400 DPH, with the base metal and weld metal averaging nearer to 350 DPH. This was not observed previously in the GTA weldments of the same materials.

A typical microstructure of a Hastelloy X weldment is shown in Fig. 4.35. This shows the shielded metal-arc weld deposit, fusion line, and solution-annealed base metal of a specimen in the as-welded condition. After aging for 7.2 Ms at 760°C, the Hastelloy X weldment microstructure (Fig. 4.36) contains a fine carbide precipitate randomly distributed within the grains. This same structure was present after 3.6 Ms. Thermal aging at 871°C for 7.2 Ms produced a microstructure (Fig. 4.37) with relatively larger precipitate particles, which are randomly distributed within the grains as well as the grain boundaries. These same microstructures were produced in the previously studied gas tungsten-arc weldments.

Fig. 4.35. As-Welded Microstructure of Hastelloy X Weldment Showing Weld Metal, Fusion Line, and Solution-Annealed Base Material, 100 $\times$.

4.3.4 Shielded Metal-Arc Welding of Hastelloy X to Alloy 800H

Three shielded metal-arc electrodes were used to join Hastelloy X to alloy 800H. Welds were made in 12.7-mm-thick plate of the two materials using Hastelloy X, ENiCrFe-3, and ENiCrFe-2 electrodes. Defect-free welds, as determined by radiographic and metallographic inspection, were produced with all three electrodes. Specimens from these weldments were thermally aged at 537 and 649°C for periods up to 7.2 Ms. Tensile specimens from the aged weldments were machined to contain both base metals, HAZ, and weld metal in the gage length. All room-temperature tensile ruptures occurred in the alloy 800H base metal outside the HAZ. This indicates only that alloy 800H is the weak link (in tensile) of this joint. The tensile testing and metallographic examination of these dissimilar-metal weldments revealed no degradation of the joints during aging. Creep testing is being conducted on this dissimilar metal combination and may aid in determining an optimum welding electrode or filler metal.

Fig. 4.36. Microstructure of Hastelloy X Weldment After Aging for 7.2 Ms at 760°C. 100×.

4.4 STEAM CORROSION — J. C. Griess, J. H. DeVan, and W. A. Maxwell*

The purpose of this program is to determine the corrosion behavior of selected materials that have possible application in steam supply systems under both normal and faulted conditions. Testing is being performed by the NUS Corporation, under subcontract to ORNL, at the Bartow Plant of the Florida Power Corporation in St. Petersburg, Florida. The steam comes directly from the superheater circuit of that fossil-fired electrical generating plant. Since the results from this program are useful to high-temperature reactors in general, funding was provided by both HTGR and LMFBR projects during FY 1977 and preceding years; during FY 1978, however, the sole source of funding is the HTGR project.

* NUS Corp., Clearwater, Florida.

Fig. 4.37. Microstructure of Hastelloy X Weldment After Aging for 7.2 Ms at 871°C. 100×.

4.4.1 Inconel 617 Loop

The Inconel 617 loop has two test chambers or autoclaves operating at 482 and 538°C (900 and 1000°F). In the present test, only pure superheated steam passes through the autoclaves at a rate of 45 kg/hr, producing a steam velocity of 1.5 m/s (5 fps) past the test specimens. The steam pressure is 10.5 MPa (1525 psi). Duplicate sets of specimens — coupons and U-bends, both welded and unwelded — are in the autoclaves. Periodically, all the specimens are examined and all except the U-bends are weighed at that time. Occasionally, specimens are permanently removed for destructive examination and are replaced with new ones of the same material.

At the end of calendar year 1977, the loop had been in operation for 13,500 hr and will continue to operate to 30,000 hr. The specimens were last examined after approximately 12,000 hr, and the weight change data and other pertinent observations are summarized below.

The weight gain data obtained from the specimens exposed for the entire test are summarized in Table 4.19. Initially seven specimens of each type were installed in the loop, but some of these were removed after certain times and new ones were added. The data shown in Table 4.19 are average values for those specimens installed in the loop at the beginning of the test. In all cases, the agreement among replicate specimens has been very good.

Except for on 2 1/4 Cr-1 Mo steel, all the corrosion product has adhered to the specimen. A very few flecks of oxide were missing from both the U-bend and flat coupons of 2 1/4 Cr-1 Mo steel exposed at 538°C after 11,731 hr. No oxide was missing after the 8005-hr exposure. Based on weight gain data and a knowledge of the composition of the corrosion product, the metal penetrations after 11,731 hr were calculated, and these values are also shown in Table 4.19. In no case did the penetration exceed 34 μm (1.3 mils) during the 11,731-hr period.

For about the first 5000 hr, logarithmic plots of specimen weight gain versus time produced straight lines with slopes of 0.4 to 0.5. However, after longer exposure times, the data appear to indicate a low constant rate for all materials except alloy 800 (ground and welded), Inconel 617, and type 304 stainless steel. The former two alloys corrode at such low rates that the kinetics cannot be established from weight change and the latter alloy did not oxidize uniformly. Longer exposure times will be required to confirm the constancy of the corrosion rate for the other alloys. If the corrosion product begins to spall from the steel specimens (2 1/4 Cr-1 Mo, 9 Cr-1 Mo, HT-9) weight change data will be worthless, and it will be necessary to descale the specimens to determine the extent of corrosion.

The corrosion of alloy 800 specimens produced an interesting result. As indicated by Table 4.19, the "ground" surfaces of alloy 800 were much more resistant than the "pickled" surfaces. The beneficial effect of cold work on the corrosion resistance of this alloy is well established,¹⁵ but in this particular case there is an additional reason for the apparent high rate of attack of the pickled specimens. After grinding, the latter specimens were solution annealed in argon (1177°C for 30 min), air cooled, and then pickled in HNO_3 -HF to remove the thin surface oxide. Apparently,

Table 4.19. Corrosion of Materials in the Inconel 617 Test Loop
by Superheated Steam at 10.5 MPa

Alloy	Surface ^a Condition	Average Weight Gain, mg/cm ²						Av Metal ^b Penetration (μm)
		504 hr	1621 hr	3051 hr	5073 hr	8005 hr	11,731 hr	
<u>Exposed at 482°C</u>								
2-1/4 Cr-1 Mo Steel	Ground	1.92	3.24	4.21	5.13	5.57	5.95	19.1
	Annealed	1.58	2.74	3.68	4.62	5.27	6.09	20.2
	Pickled	1.97	3.27	4.36	5.39	5.97	6.47	21.5
Alloy 800	Ground	0.03	0.03	0.03	0.04	0.04	0.04	0.14
	Pickled	0.83	1.33	1.86	2.35	2.74	3.13	10.0
	Welded ^c	0.07	0.10	0.12	0.13	0.14	0.16	0.56
Inconel 617	Ground	0.01	0.02	0.02	0.02	0.02	0.03	0.12
304 Stainless Steel	Ground	0.01	0.04	0.10	0.28	0.43	0.63	2.1
9 Cr-1 Mo Steel	Ground	1.38	2.29	3.12	3.92	4.41	5.05	16.5
Sandvik HT-9	Ground	1.39	2.21	2.85	3.55	3.88	4.41	15.0
<u>Exposed at 538°C</u>								
2-1/4 Cr-1 Mo Steel	Ground	2.98	4.69	6.32	7.90	9.01	9.64	32.0
	Annealed	2.92	4.82	6.13	7.99	9.12	10.21	33.8
	Pickled	3.12	5.15	6.65	8.64	9.64	10.05	33.3
Alloy 800	Ground	0.05	0.06	0.06	0.07	0.08	0.08	0.28
	Pickled	1.59	2.78	3.68	4.79	5.36	6.14	21.6
	Welded ^c	0.12	0.15	0.17	0.21	0.21	0.21	0.74
Inconel 617	Ground	0.04	0.04	0.04	0.05	0.05	0.07	0.27
304 Stainless Steel	Ground	0.60	1.29	2.23	3.10	3.39	4.10	13.4
9 Cr-1 Mo Steel	Ground	2.47	4.23	5.61	7.20	8.04	8.70	28.4
Sandvik HT-9	Ground	2.20	3.52	4.22	4.85	5.28	5.61	19.1

^aGround on 100-mesh belt grinder; annealed in argon after grinding; pickled to remove light surface film.

^bCalculated from weight gain after 11,731 hr assuming corrosion products were Fe₃O₄, Cr₂O₃, NiO, CoO.

^cMill annealed and pickled before autogenous welding.

the rate of cooling was slow enough to allow carbides to precipitate at grain boundaries, and subsequent pickling produced intergranular attack to a depth of about 0.2 mm (8 mils). Figure 4.38 shows the attack on a

Fig. 4.38. Intergranular Attack on an "Annealed and Pickled" Alloy 800 Specimen Exposed to Superheated Steam at 538°C for 5073 hr. 100×

pickled specimen exposed for 5073 hr at 538°C. Examination of a specimen given the same heat treatment and pickling, but not exposed to steam, showed intergranular attack to the same depth, indicating that corrosion did not continue during steam exposure. The "welded" alloy 800 specimens (Table 4.19) were made from mill annealed and pickled sheet and contained an autogeneous weld across the narrow dimension of the coupon. As indicated, these corroded at a greater rate than the "ground" surfaces but at a much lower rate than the "pickled" specimens. The surface films were so thin on both the "welded" and the "ground" specimens that they were not detectable on the metallographically polished cross sections.

In contrast to alloy 800, the surface condition of 2 1/4 Cr-1 Mo steel had no significant effect on corrosion. Figure 4.39 shows the oxide formed on 2 1/4 Cr-1 Mo steel during 8005-hr exposures at 482 and 538°C (900 and

Fig. 4.39. Cross Section Through 2 1/4 Cr-1 Mo Steel Specimens Tested in Superheated Steam for 8005 hr. 500 $\times$. Exposed at (a) 482°C and (b) 538°C.

1000°F. In both cases, the oxide was composed of two layers, the inner layer containing chromium and molybdenum in about the same volume concentration as the alloy and the outer layer containing essentially pure Fe_3O_4 . In both cases, the oxide was adherent and compact.

Figure 4.40 shows cross-sectional views of the oxide formed on Sandvik Alloy HT-9 (11.5% Cr, 1% Mo + Ni, W, V at <1%) at 482 and 538°C (900 and 1000°F) during an 8005-hr exposure. The oxide formed at 482°C is composed of three distinct layers; at 538°C the oxide also contains three layers, but the innermost layer is very thin and barely visible. The same observation was made on specimens exposed for 3000 and 5000 hr. Ion-microprobe analyses showed that at both temperatures the outer layer was essentially iron oxide, but the middle and inner layers contained both chromium and molybdenum. Why three oxide layers are visible in this case and why the inner layer is more prominent at 482 than 538°C is not known.

Figure 4.41 shows the oxide formed on 9 Cr-1 Mo steel specimens that were in the loop for 8005 hr at 482 and 538°C. Each case shows three distinct oxide layers, but in contrast to the oxide formed on HT-9, the innermost layer is more distinct on the specimen exposed at 538°C than on the one at 482°C. The total thickness on the oxides is not greatly different from that formed on 2 1/4 Cr-1 Mo steel and HT-9 at the same temperature, but 9 Cr-1 Mo steel had places where the metal corroded very little [Fig. 4.41(b)]. Ion-microprobe analyses of the metal in the region where attack was slight showed no detectable difference in composition from that close to regions that corroded normally. Also, analyses showed that the outer oxide contained only iron, whereas the two inner layers contained chromium and molybdenum in approximately the same volume concentration as the alloy.

The Inconel 617 specimens corroded at such low rates that the corrosion product on the surface was not detectable on metallographically polished cross sections of the specimens. The weight gains on type 304 stainless steels have been erratic, and further exposure is required before a definite trend can be established. Additionally, the oxide formed on the specimens has not been uniform as with the other alloys. This is illustrated in

Fig. 4.40. Cross Section Through Sandvik HT-9 Specimens Tested in Superheated Steam for 8005 hr. 500 $\times$. Exposed at (a) 482°C and (b) 538°C.

Fig. 4.41. Cross Section Through 9 Cr-1 Mo Steel Specimen Tested in Superheated Steam for 8005 hr. 500 $\times$. Exposed at (a) 482°C and (b) 538°C.

Fig. 4.42, which shows a cross section through one of the specimens that was exposed at 538°C for 8005 hr. The reason for this type of attack is not apparent at this time.

Fig. 4.42. Cross Section Through Type 304 Stainless Steel Specimen Exposed for 8005 hr to Superheated Steam at 538°C. 500×.

None of the U-bend or welded specimens showed any unusual effects. Exposure of these and most of the coupon specimens will continue during FY 1978.

4.4.2 Inconel 625 Loop

The Inconel 625 loop, which was used previously in chloride-contaminated steam for several years, has been reinstalled at the Bartow Plant. During this fiscal year, this loop will be used primarily to examine the effects of very low levels of sodium hydroxide in superheated steam and stress on the corrosion of several different heats of alloy 800H covering a range of carbon contents. This test is being planned in cooperation with General Atomic personnel, who will supply the specimens for the loop.

4.4.3 Heat Transfer Corrosion Tests

Over the past several years a program has been conducted to determine the effect of high heat fluxes on the corrosion of 2 1/4 Cr-1 Mo steel in superheated steam. In the first series of tests standard air-melted steel was used. At a heat flux of 126 kW/m^2 ($40,000 \text{ Btu/hr ft}^2$) and at surface temperatures of 510 to 540°C (950 – 1005°F) the oxide that formed was porous, was poorly adherent to the substrate, and began to spall from the surface after about 2000 hr. The details of this test have been reported.¹⁶

A second test series was conducted at the same heat flux, but in this case, the test material was vacuum-arc-remelted 2 1/4 Cr-1 Mo steel and the surface temperature was lower [502 – 522°C (935 – 972°F)]. Under these conditions the oxide was also porous, but longer times were required to produce oxide exfoliation than at the higher temperatures.

One valid criticism of the results from both these series of tests is that the tubes were heated with electrical heaters from the inside and cooled by steam flowing in an annulus around the outside of the tube, whereas in actual superheaters the tubes are heated from the outside and cooled by steam flowing through the tubes; that is, in our tests oxide formed on convex surfaces rather than on concave surfaces as it does in superheaters. Subsequently, we modified our test equipment so that specimens could be heated from the outside and cooled by steam flowing through the tubes. A recently completed test in the modified facility demonstrated that under the same conditions as used in the first series of tests (air-melted 2 1/4 Cr-1 Mo steel, 126 kW/m^2 , and 510 – 540°C) oxide exfoliation occurred as readily, if not more so, on the inner surfaces as on the outer surfaces. The complete experimental details and results from this test as well as that from the heat flux tests with vacuum-arc-remelted 2 1/4 Cr-1 Mo steel have been reported.¹⁷

At present, the heat-flux corrosion testing has been discontinued, although we believe that this is an area that badly needs study if low-alloy ferritic steels are to be used successfully in superheaters of HTGRs and LMFBRs, where temperatures will be lower but heat fluxes can be much higher than those used in this test program. Exfoliation of the steam-formed corrosion product increases the corrosion rate of the steel, but probably more significant is the erosive effect of the spalled oxide on downstream components, particularly turbine blades.

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5. CHARACTERIZATION AND STANDARDIZATION OF GRAPHITE

W. P. Eatherly and J. A. Conlin

5.1 INTRODUCTION

The graphite program at ORNL now involves two major subprograms; (1) the continuing set of irradiation experiments on compressive creep being conducted in the ORR, and (2) a new program to irradiate in HFIR moderator graphites produced in the Federal Republic of Germany.

During calendar year 1977, the creep experiments went through a hiatus with the temporary reduction in ORR operations. This gave us, however, time to deliberately redesign those aspects of capsule OC-1 that had given problems in the reactor. Design changes have been made in centering devices for the load train, the load sensors, and the movable thermocouple contacts. We plan to insert capsule OC-2 in the ORR in April 1978, with OC-3 following immediately afterward.

Irradiation of the German graphites, being conducted under the joint US/FRG Thermal Gas Reactor Program, is aimed at qualifying a graphite for the top reflector of a pebble bed reactor. Total fluences in this region are extremely high. Seven graphites have been furnished by KFA for irradiation in HFIR, with the intent that rapid accumulation of fluence in this reactor will permit some of the candidate graphites to be removed early from the much more extensive but lower flux irradiations being conducted in Europe. The first of four capsules under this program will be inserted in HFIR in March 1978.

5.2 CREEP EXPERIMENTS OC-2 AND OC-3 — R. L. Senn

Several modifications were incorporated in the design for capsule OC-2 and subsequent capsules of this series as a result of experience gained during the irradiation of capsule OC-1. Although the LVDT load cells used in OC-1 at the bottom of each stressed column were unsatisfactory, they did give some indication that one of the stressed columns may have been wedged because of misalignment of the bellows during irradiation. This has been corrected by adding alignment pins to the bottom flange of the bellows and to the top of the load cells, such that the entire bellows,

push rod, specimen, load cell train is securely guided to prevent misalignment. Details of these changes are shown in Fig. 5.1, a view of the in-core portion of capsule OC-2. Note the guide pins extending below the bellows and above the load cells. The LVDT load cells have been replaced with a new device, and the movable thermocouple design has been improved, as discussed in the following paragraphs.

Fig. 5.1. In-Core Portion of Typical 600 to 900°C Creep Capsule.

5.2.1 Modification to Pressure Sensor System

A pneumatic electromechanical device, designated a PEM cell, has been designed to replace the LVDT load cells used in capsule OC-1. The new load cell, shown in Fig. 5.2, uses metal bellows similar to those used to load the specimen columns. When the load is applied to the

specimens, an insulated contact button located inside the PEM cell bellows is in electrical contact with the top flange of the bellows. The PEM cell bellows is then pressurized with helium to a pressure high enough to overcome the load on the specimens and open the electrical contact. By measuring the pressure required and detecting the resistance between the contact button and the grounded bellows (infinite when open), the load being applied to the specimen column can be determined, thus assuring that the load is indeed being applied to the column. By virtue of its all-metal construction, except for the alumina insulator required, we expect the PEM cell to be relatively insensitive to damage from high temperature and radiation.

Fig. 5.2. Pneumatic Electromechanical Load Cell as Installed in Lower End of OC Capsule. Multiply dimensions by 25.4 to convert to millimeters. Loads are 2.515 kN. Movement of PEM cell is about 1.3 mm.

Bench testing for the PEM load cell was satisfactorily completed. The device was tested at room temperature and at 260°C by applying a load against the bellows of the cell and pressurizing the PEM cell bellows until the electric contact was opened, signifying that the PEM cell was just overcoming the applied load. Various loads were applied to the PEM cell by pressurizing a bellows positioned above the PEM cell at pressures ranging from 0.69 to 5.52 MPa gage (100–800 psi). The differential pressure required to open the PEM cell contact ranged from 69 kPa (10 psi) to 207 kPa (30 psi), reflecting a hysteresis effect caused by the bellows geometry and friction within the bench test setup. The results were essentially the same at room temperature and 260°C.

We found less hysteresis if the device was operated at a pressure slightly positive with respect to the applied load, such that the contacts would remain open until the PEM cell pressure was relieved. This required a differential pressure ranging from 0 to 124 kPa (18 psi). The irradiation experiment will probably be operated in this mode.

The PEM cell was operated at 0.69 MPa (100 psi) with 5.52 MPa (800 psi) pressure applied, resulting in the contacts being tightly closed, for two overnight periods in an effort to determine if there was any tendency for the Inconel contact button to self-weld to the Inconel end cap of the bellows. In each instance the contacts opened at about 200 kPa (~30 psi) differential pressure, indicating that self-welding did not occur over these periods.

5.2.2 Modifications to Movable Thermocouple System

The movable thermocouple subassemblies for OC-2 were completed. These incorporate some improvements from the design used in capsule OC-1. The sliding contacts, which transmit the thermocouple signal from the moving part of the thermocouple to the lead wire, were redesigned to provide a more rugged reliable contact. The contacts were designed with spring-loaded graphite brushes, which bear against the gold-plated strip that serves as the electrical pickup. This will prevent the loss of contact that we ultimately observed during the operation of OC-1. A bench test was satisfactorily concluded with one of these new units having been cycled up and down throughout its 0.41-m travel 360 times into a furnace operating at 216°C. None of the parts showed signs of undue wear and no detectable

difference occurred in the thermocouple readout compared with a similar thermocouple attached to the same location as the sensing end of the movable thermocouple.

5.2.3 Status of Construction and Irradiation Schedule

Assembly of capsules OC-2 and OC-3 has continued during this reporting period as manpower was available. Capsule OC-2, which is designed to operate at 600°C, is scheduled for installation in the ORR during the currently scheduled shutdown April 4, 1978. It will begin April 7 and is scheduled to end May 28, 1978. Capsule OC-2 will be irradiated during reactor cycle 144.

Capsule OC-3, the second irradiation of the 900°C specimens, is presently scheduled for a two-cycle irradiation during reactor cycles 145 and 146, ending about Sept. 17, 1978.

5.3 KFA GRAPHITE IRRADIATIONS

5.3.1 Capsule Design and Construction — R. L. Senn

A set of four HFIR target facility capsules, designated HTK-1, -2, -3, and -4, are required for this program. An existing target capsule design will be used, modified to contain 64 small cylindrical graphite specimens to be irradiated at $500 \pm 50^\circ\text{C}$. The internal parts for the KFA target capsules will require a new design for the graphite specimens, their diameters, spacers, and SiC monitors, based on the desired 500°C specimen temperature, the resultant gas gaps, and proper fill-gas to obtain the necessary thermal resistance.

Other than these parameters, however, the design for target capsules is well established. The experiment assembly consists of a singly contained uninstrumented capsule, having basically the exterior shape and dimensions of a typical HFIR target rod assembly. Graphite specimens are loaded into the central 0.508 m length of the capsule, corresponding to the HFIR active core length. The specimens are centered in the bore of the capsule in a helium atmosphere and are dimensioned such that the gas gap inside diameter provides the required thermal conductance (varying with the corresponding gamma heating rate of ~35 to ~19 W/g) to maintain the desired specimen temperatures.

Silicon carbide temperature sensors will be used to corroborate the thermal design (following termination of the irradiation). Methods of determining temperatures from these sensors are explained in the literature.

The outer shape and dimensions of the capsule are identical to a conventional target rod assembly for proper fit in the HFIR target region. The capsule is formed by a 6061-T6 aluminum tube 16.6-mm-OD and 13.46-mm-ID with a 1.58-mm wall. The upper closure is formed by a cap with a small vent hole. This vent is used for filling the capsule with helium after pressure and leak tests are completed. The vent hole is then sealed by pressing a soft aluminum plug in place. This plug is then covered by a 6061-T6 aluminum screw, which is seal welded in place. The external shape of the aluminum top cap matches the external configuration of a target rod upper assembly. The bottom is sealed by the bottom cap assembly, which serves as both a seal and as a pressure relief in the event of high internal pressure as a consequence of water logging.

This pressure relief device is a 0.254-mm-thick pure aluminum rupture sleeve. This sleeve will protect the reactor from the consequences of a sudden failure in case of excessive internal pressure buildup. The sleeve is designed to fail at 4.1 to 5.5 MPa at an operating temperature of 93°C before neutron irradiation. The effect of neutron irradiation will be to increase the effective bursting pressure to about 10 MPa.

For the capsule tube the ASME maximum allowable internal working pressure (at 93°C) is 12.7 MPa. If the code is assumed to be conservative by a factor of 4, the estimated bursting pressure of the containment would be about 58 MPa.

The capsule will be filled with helium (moisture free) at slightly above 0.1 MPa (1 atm) pressure before the final closure weld.

Poco graphite splines will contain SiC temperature monitors. The graphite specimens are supported on these graphite splines. These splines are in turn supported by a spider consisting of a graphite disk and four small metal pins. The pins serve as a centering device. A typical specimen configuration showing these features is shown in Fig. 5.3.

The lower end of the specimen stack is supported on a graphite spacer pedestal with a pyrolytic carbon foil to insulate the specimen from the pedestal. The pedestal is supported on an aluminum spacer. At the upper

end of the specimen stack a special support spider of stainless steel is used with an Inconel X spring. A space at the top of the capsule is provided for axial expansion. The spring is used to prevent the specimens from becoming disengaged from the support splines.

Fig. 5.3. Typical Specimen Configuration for HTK Capsules: Sixty-Four Specimens in Four Groups of Eight.

Final heat transfer calculations have been completed to determine the required specimen diameters for each specimen location. The specimens are 6.35-mm-thick (0.250-in.) cylinders with 3.20 to 3.23 mm-diam. (0.125-0.126 in.) central hole (to fit on graphite splines required to position and center the specimens). Their outside diameters vary with the specimen elevation in the capsule according to the incident gamma heating at that elevation in the reactor. The outside diameter also depends on the properties of the particular type of graphite used for each specimen.

Upon receipt of the information providing the graphite properties, a one-dimensional heat transfer calculation was made for each specimen using the known properties and the known elevation. The diameter of

the specimen was varied until the outside surface temperature of the specimen reached the design point of 500°C. The calculations assumed helium in the gas gap between the specimen and the containment tube. The calculations were done on the PDP-10 computer using GENMD2 and GENMD3, special codes devised for these experiments based on GENGTC,¹ a heat transfer code used successfully on many similar experiments. The modifications were principally concerned with installation of the properties for the seven types of graphite required and rearrangement of some data input and output to permit varying the specimen diameter and gamma heat values in a more straightforward manner than was the case with the original program. The results from GENMD2 and GENMD3 were verified against results from the complete parent program. The resulting calculated diameters vary from 10.77 mm (0.424 in.) at the extreme ends of the experiment to 12.29 mm (0.484 in.) near the midplane. These are consistent with preliminary results obtained during the conceptual design.

5.3.2 Material Evaluation of KFA Graphites — C. R. Kennedy

One of the major problems in evaluating graphite is the variability of properties, particularly in large blocks as used in moderator structures. These graphites generally have density gradients and variations in the preferred orientation of both the porosity and crystallite structure. These characteristics have pronounced effects on the density and orientation-dependent properties such as the mechanical properties, flow characteristics, thermal properties, and irradiation stability. Also, the properties of sampled blocks may, in some cases, yield only indirect evidence of the actual integrated responses of a whole block under service conditions. Therefore, in the evaluation of large graphite blocks, it is critical to clearly define the variability in structure and associated properties across the sampled section. Only in this way will the evaluation be relevant in understanding the expected behavior of the full block and potential variations within a block and from block to block. For this purpose the KFA graphites were evaluated before specimens were selected for irradiation in the HFIR experiments.

Seven grades of graphite were supplied to ORNL from KFA in the form of representative slabs taken from large blocks. The blocks sampled are

the base-line grades for a much larger physical property and irradiation evaluation program in Germany. The HFIR irradiation program, because of our ability to accumulate large damage fluences rapidly, is designed to lead the German program. The results will permit the reduction of candidate materials and minimize dilution of effort. Each graphite slab was carefully examined to ensure representative sampling. Also, adequate physical property testing was done to ensure the compatibility of ORNL test results with KFA results.

Each block was surveyed by eddy currents, qualitative and quantitative radiography, and longitudinal and shear sonic velocities. The results of these surveys permitted the blocks to be sampled for irradiation, bend, and brittle ring specimens. Sonic velocity measurements, coefficient of thermal expansion, and brittle ring strengths were obtained from the irradiation specimens for comparison with values after irradiation. The bend samples were also used to obtain the electrical conductivity for direct comparison with eddy-current values. The brittle ring specimens of a standard ORNL size were used to ensure a valid comparison of the subsize HFIR brittle ring test specimens.

Each block was surveyed by eddy-current measurements every 25 mm on each face of the block. The relationship of eddy-current response to electrical resistivity formed for the KFA blocks is given in Fig. 5.4. The electrical resistivity measured at ORNL on the bend specimens is compared with the KFA reported values in Fig. 5.5. The results of a typical scan by eddy currents is shown by the contour map in Fig. 5.6. In general, the results of the electrical resistivity testing indicate agreement of results at ORNL and KFA. The eddy-current scans do show the general variation one would expect with density across the block, with some anomalies close to the exterior surfaces. These blocks were all fairly isotropic, and variations in the preferred orientation were masked by the density variations. The small UKAEA block 11 supplied as a comparison grade gave evidence of the highest variability, with a high-resistance region of over one-fourth of the volume.

Both longitudinal and shear sonic velocities were measured every 50 mm on each surface of the blocks. The elastic constants were calculated by the method of Kennedy² and are given in Table 5.1. Young's modulus as

Table 5.1. Elastic Constants from Sonic Measurements

Elastic Constants	Value for Each Block						
	V483	V356	ATR-2R	ATR-2E	ASR-1R	ASR-2R	UKAEA-11
C_{11} , GPa	10.49	10.69	10.90	9.08	11.01	9.36	8.69
C_{53} , GPa	10.24	9.61	9.33	10.66	9.41	7.70	9.42
C_{44} , GPa	4.33	4.56	3.87	3.98	4.13	3.48	3.64
C_{66} , GPa	4.21	4.78	4.07	3.73	4.39	3.82	3.54
C_{12} , GPa	2.07	1.13	2.76	1.62	2.23	1.71	1.61
C_{13} , GPa	1.70	1.01	2.33	1.82	1.90	1.50	1.76
$-S_{12}/S_{11}$	0.18	0.10	0.21	0.15	0.17	0.16	0.15
$-S_{13}/S_{11}$	0.14	0.11	0.20	0.14	0.17	0.16	0.09
$-S_{13}/S_{33}$	0.14	0.09	0.17	0.17	0.14	0.14	0.17
$1/S_{11}$, GPA	9.90	10.47	9.86	8.59	10.31	8.85	8.29
$1/S_{33}$, GPA	9.68	9.44	8.54	10.04	8.88	7.30	8.82

reported by KFA is compared with these results in Fig. 5.7, showing very good agreement. A typical contour map of the dynamic Young's Modulus is shown in Fig. 5.8. The modulus values, in agreement with the eddy-current results, follow the density gradients in the block.

Qualitative low-voltage radiographs were made of each block to examine the structure for localized abnormalities in density. The resolution is about 0.5% of the density, or a void about 2 mm in size can be detected. Most blocks appeared fairly uniform with the exception of block ATR-2E and UKAEA-11, where flow lines were clearly observed. Grades ATR-2E and UKAEA were the only extruded grades. The quantitative radiographs were made by scanning along the length every 25 mm. The x-ray source is a collimated beam 2 mm in diameter detected by a Geiger tube. The results of two scans are given in Fig. 5.9 and 5.10 comparing the block ATR-2E with the more uniform block V356. Again the UKAEA block had the largest variability, with the low-density region conforming with the high resistivity measurements.

After examination of the variability in each block, sampling plans were devised to obtain HFIR irradiation specimens from two locations. The first from a location most "representative" of the block in general and the second from the location having the maximum deviation from the mean. Most of the blocks were very uniform and the "representative" specimens were selected arbitrarily from the central part of the block. The "nonrepresentative" specimens were taken from the edge or close to the surface of the block. Bend specimens (44.45 by 6.4 mm by 3.2 mm) and brittle ring specimens (17.8 mm OD, 10.2 mm ID, and 6.4 mm thick) were also taken from the same location as the representative HFIR specimens. Bend specimens were taken both with the grain and across the grain except for blocks ATR-2R, ASR-1R, and ASR-2R. These blocks had the small dimension (≈ 40 mm) in the across-grain direction, and the specimens were too long. The exception was in sampling UKAEA to exclude the low-density region. Even so, the bulk density of the specimens was much lower than reported by KFA. Thus, results cannot be directly compared with KFA results for this grade.

We have already demonstrated³ that the standard HFIR specimen could be used to determine brittle-ring bend strength of irradiated specimens. These values, of course, can be internally compared to directly demonstrate

Fig. 5.7. Agreement Between ORNL and KFA Dynamic Modulus Values.

Fig. 5.8. Dynamic Modulus by Sonic Velocity Measurements in Block ATR-2E.

Fig. 5.9. Bulk Density Scans by Quantitative Radiography of Block ATR-2E.

Fig. 5.10. Bulk Density Scans by Quantitative Radiography of Block V356.

the effect of irradiation damage on strength. However, all mechanical tests are relative and dependent upon the statistical characteristics of the defect structure in the material. While Wiebull statistics have been used rather successfully for ceramic materials in describing the effects of stress volume and stress gradients, their application to graphite has had limited success.⁴ The uniform fine-grained graphites used in aerospace applications have generally been successfully described by Weibull statistics over a wide range of conditions. It is the nonuniform coarse-grained reactor-grade graphites that have yielded inconsistent results. This study is to clarify the general area of uncertainty in evaluating the small HFIR specimens.

Bend specimens, 17.8-mm-OD brittle ring specimens, and HFIR-size specimens were all destructively tested, and the results are given in Table 5.2. To make relevant comparisons, HFIR specimens from our archives and specimens made from two blocks of grade H451 were also tested. The results of these tests are also given in Table 5.2. The bend test results are compared with the reported KFA values in Fig. 5.11. The agreement is very good suggesting that stress volume of the KFA specimens is slightly larger than that of the ORNL specimen. The 17.8-mm-OD brittle ring strength is compared with bend strength in Fig. 5.12(a). A similar comparison is given in Fig. 5.12(b) for the HFIR specimens brittle ring tested. These data clearly indicate linear strength ratios for graphites fabricated by similar procedures. The strength ratio for the molded grades is a constant over a very wide range of strengths from 11.5 to 79 MPa. The extruded grades have the highest strength ratio and the vibratorily compacted grades just slightly lower. Why the vibratorily compacted grades do not respond more like the molded materials is unclear at this time. It is also of interest to compare the results of bend testing with tensile testing of some molded experimental materials in Fig. 5.13. Again the strength ratio appears to be constant.

In comparison of the two brittle ring specimen data sets with the bend test data, we are comparing the results of bend testing specimens with essentially the same width and thickness. Therefore, the stress gradient in these specimens is similar but the stress volume varies over almost three orders in magnitude. This geometry then allows the exclusion of the

Table 5.2. Fracture Strength Testing on Graphite

Grade	Density	Orientation Relative to Grain	Fracture Strength, MPa			
			KFA Bend	ORNL Bend	17.8 mm Brittle Ring	HFIR Brittle Ring
<u>KFA Grades</u>						
V483	1.75	With	31.4	33.8	40.1	46.5
		Across	30.5	32.4		
V356	1.76	With	32.2	37.4	39.7	48.3
		Across	29.4	31.8		
ATR-2R	1.74	With	24.4	25.1	32.5	43.7
		Across	22.4			
ATR-2E	1.82	With	23.5	22.6		
		Across	18.5	19.9	31.8	41.4
ASR-1R	1.82	With	26.0	26.6	35.4	45.4
		Across	23.0			
ASR-2R	1.72	With	19.4	20.2	26.4	39.1
		Across	15.8			
UKAEA-11		With		20.3		
		Across		19.9	27.4	
<u>HTGR Grades</u>						
H451	1.74	With		30.7		
		Across			32.8	41.1
H451	1.70	With		24.9		
		Across		18.2	27.7	36.8
<u>Miscellaneous Grades</u>						
A681	1.64	Isotropic		37.2		48.3
AXM-5Q	1.68	Isotropic		60.7		77.1
AXF-5Q	1.80	Isotropic		79.2		102.5
AXJ-5	1.85	With		47.0		56.1
H337	1.99	Isotropic		42.7		56.5
AGOT	1.74	Across		9.6		20.8
SA45	1.55	Isotropic		11.6		13.6

Fig. 5.11. Correlation of KFA and ORNL Bend Test Results.

consideration of fracture governed by surface flaw distributions; only the stress volume must be considered. From Weibull statistics, the effect of volume is:

$$\sigma_1 / \sigma_2 = (V_2 / V_1)^{1/m} . \quad (1)$$

Comparing the strength ratios with stress volume in Fig. 5.14 confirms the validity of the stress volume effect in Eq. (1). The tensile data strength ratio was adjusted for the nonlinear stress-strain behavior in calculating the bend strength.⁵

(a)

(b)

Fig. 5.12. Comparison of (a) 17.8-mm-OD and (b) HFIR Specimen Brittle Ring Strengths with Bend Strength.

Fig. 5.13. The Stress Ratio is Constant for Bend and Tensile Tests Over a Wide Range in Strength.

Fig. 5.14. The Relationship Between Strength and Stress Volume Varies with Method of Fabrication.

A most significant result is the existence of stress volume strength ratios for each method of manufacture. The implication of the smaller value of m for the extruded blocks is that the coefficients of variability would be larger for these grades. This is, in general, what would be expected. It strongly implies that the coefficient of variation is fixed by the method of processing. Studies that yield coefficients much different than those determined in this study are a result of either variability within a block or specimens strongly affected by surface flaw distributions.

5.3.3 Summary and Conclusions

Slabs from seven experimental German graphites have been surveyed and characterized. All of the material property measurements made at ORNL agree very well with the KFA results except the grade UKAEA-11. The sample piece we have has a lower density and associated physical properties than reported by KFA. "Representative" and "nonrepresentative" specimens have been machined from the graphites for irradiation testing in HFIR.

The ORNL determination of mechanical properties of the KFA grades was in good agreement with KFA results. Studies of the relevance of brittle ring testing HFIR specimens were successful. The specimen stress volume effect was clearly demonstrated to be the main factor in relating the results of the subsized specimens. The effect of fabrication was clearly noted in the strength ratios between specimens of different stress volume.

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6. EVALUATION OF PEBBLE-BED TYPE HTR

D. E. Bartine, D. R. Vondy, B. A. Worley, and E. T. Tomlinson

6.1 INTRODUCTION

The purpose of this effort is to examine the design and mode of operation of the pebble bed HTR concept and project its performance. Ore utilization and fuel cost are of primary interest, with design constraints satisfied. This work is under a technical information exchange umbrella agreement with FRG. Applications for power and process heat are of interest.

6.2 PBR CORE PERFORMANCE AND FUEL CYCLE CALCULATIONS — D. R. Vondy and B. A. Worley

Calculations were made at ORNL to assess the performance of the gas-cooled pebble-bed reactor concept.¹ The concept is illustrated in Fig. 6.1, which shows the once-through, continuous fueling model adopted for the analysis. The illustration is one of possible designs studied for high fuel production: two streams of different pebble types, one fed through the core and one fed to the inlet axial blanket and to a fixed radial blanket. Complexities include variable residence times (especially when pebbles of two different sizes are considered), variation in pebble packing fractions, and the presence of both moving and fixed material.

A design for breeding with ^{233}U fuel was proposed by the General Electric Company.² (This design was a modification of a design proposed in Germany as an advance over its AVR reactor and involves small fertile pebbles trickling between larger fueled primary pebbles.) Core physics and fuel cycle analyses were performed for this concept and also for a reactor design with two pebble types of the same size, with primary emphasis on ^{233}U feed. Results of this analysis were reported.³

A low carbon-to-thorium ratio and a high thorium loading are attractive to maximize the conversion ratio. However, a high fissile inventory is required, which causes the fissile inventory doubling time to be long. A high breeding ratio (1.1) was found with ^{233}U fuel for a very short residence time. With recycle and a longer residence time forced by economic considerations, and after consideration of processing losses, a breakeven fissile balance is more likely than significant production of fuel. Economic

justification was not found for for holdup of the pebbles outside the reactor after short exposure (for ^{233}Pa decay) and subsequent recycle; high indirect fuel cost is associated with such holdup.

ORNL-DWG 77-13629

NOTE:

THIS IS AN ILLUSTRATION OF ONE OF THE MANY POSSIBILITIES.

THREE FLOW PATHS THROUGH AXIAL BLANKET AND CORE ARE SHOWN.

Fig. 6.1. The Once-Through, Continuous Fueling Model of a Pebble-Bed Reactor.

The reliability of the local calculations has been improved in the continuing effort by adding sophistication to the modeling. Special attention has been paid to the resonance shielding, the fission product representation, and the reactor model in one and two dimensions.

In the continuing study we are considering low-enriched uranium feed of nonproliferation quality, a pre-breeder to generate ^{233}U fuel, and a ^{233}U -fueled design that would have a high conversion ratio. One-dimensional parameter studies have been made for fully enriched ^{235}U feed and for ^{233}U feed. Two-dimensional calculations are made for selected reference conditions. Calculations have been made for a single pebble size with two feed streams, one containing ^{235}U -fueled pebbles and the other fertile pebbles. Selected results from parameter studies of a blanketed core with fully enriched ^{235}U fuel at \$45/g and two sets of unit processing costs are shown in Table 6.1.

Table 6.1. Characteristics with Fully Enriched Uranium Feed

Carbon-to-heavy-metal ratio	250	325	325	400
Residence time, years	4	3	4	3
Exposure, MWd(t)/kg HM				
Primary	212	200	249	240
Fertile	51	43	57	48
Axial peak to average power density	3.3	3.4	4.1	4.3
Conversion ratio	0.636	0.617	0.581	0.554
Fissile U rate, kg/day				
Feed	2.127	2.208	2.158	2.325
Discharge	0.706	0.721	0.534	0.578
Throwaway fuel cycle at 0.75				
Load factor (commitment)				
Ore, kg U_3O_8 /MWyr(e)	167	171	170	178
Fuel cost, mills/kWhr(e)				
high	6.10	6.08	6.17	6.29
low	5.75	5.73	5.78	5.91

The results for a carbon-to-heavy-metal ratio of 325 show the dependence of exposure on residence time. The throwaway cycle fuel cost has a small variation in this range. As the C/HM is reduced, longer residence time is required to effect the same exposure. Fair agreement is found between our results and the reported results obtained in FRG for a C/HM of 325 and an average exposure of 100 MWd(t)/kg (about 3.5 years residence). The indirect fuel costs contribute substantially to our assessment, pushing the choice toward a relatively low C/HM and a lower exposure than expected. Recent results show a slightly decreased fuel cost in the ore requirements of nearly 10% with once-recycle of the fertile particles compared with the throwaway cycles. For throwaway cycles, ore requirements for medium enriched uranium feed (denatured) are about 20% higher than for fully enriched fuel.

The methods of calculation have been upgraded in this effort. A point, two-group model is used for parameter studies considering a 30-year reactor history with selective recycle.⁴ A once-through, continuous fueling model was implemented to apply the VENTURE diffusion theory neutronics code for one- and two-dimensional calculations.⁵ A direct iteration procedure is used to establish the fuel enrichment of the feed required to effect a steady-state critical reactor. This scheme, which ignores the early operating history, has now been used to treat the case of once recycle of fertile pebbles, establishing the steady-state condition for a specified set of parameters. (This is not the method developed in Germany and used by the LASL staff in its assessment.) The amount of information produced in a calculation has been gradually increased. The direct iteration procedure in use often converges at a reasonable rate, but some situations have required special action. This crucial area is under continuing study, and the procedures are being modified as found to be desirable from application experience.

6.3 PBR MECHANICAL DESIGN — E. T. Tomlinson

The high-temperature helium-cooled graphite-moderated pebble-bed reactor design concept has recently received a degree of interest from the Department of Energy and General Electric Corporation. The objective of this study was to provide, mostly in general terms, a cursory review

of the pertinent mechanical design features of such a reactor system. It is not the interest of this discussion to evaluate any specific design, but to examine some generic design constraints and/or advantages. The specific areas that will be addressed are: limits of practical heavy metal loadings, single versus multiple ball pressure drops and their effects on plant efficiency, and the effect of additional axial blankets.

6.3.1 Heavy Metal Loadings

The heavy metal loading (i.e., mass of heavy metal per unit volume of core) is a crucial quantity in determining the size as well as the neutron performance of the reactor system. This particular quantity depends on a number of design specifications as well as natural limitations.

In the single-ball system the maximum packing fraction (solid fraction) for the graphite pebbles is 61%. If a smaller second pebble is added to the system with a ratio of diameters of 6, the average maximum packing fraction can be raised to approximately 75%. This, of course, is an average pebble packing fraction and ignores the variation in packing near the edges of the reactor.

The second independent variable that directly affects the heavy metal loadings is the thickness of the pebble's graphite shell. The thickness of the shell can be limited either by fabrication constraints for the small pebbles or by structural constraints imposed by their movement through the core for the larger balls.

The third variable is the size of the coated fuel particle that is suspended in the pebble's inner graphite matrix. The fourth independent variable is the packing fraction of the coated fuel particles in the inner graphite matrix. A typical fuel pebble is illustrated in Fig. 6.2 for clarification of each of these terms.

The mass of heavy metal in a given core can be related to the aforementioned quantities by Eq. (1) for a single-ball system:

$$M(\text{mass}) = F_1 F_2 F_3 F_4 \rho_{\text{fuel}} V_{\text{core}} \frac{M_{\text{HM}}}{M_{\text{fuel}}} , \quad (1)$$

where

- F_1 is the packing fraction of pebbles,
- F_2 is the volume fraction of the pebble that is the inner matrix,
- F_3 is the particle packing fraction,
- F_4 is the volume fraction of the particle that is fuel,
- ρ_{fuel} is the density of the fuel (ThC_2),
- V_{core} is the volume of the reactor core,
- M_{HM} is the molecular weight of the heavy metal,
- M_{fuel} is the molecular weight of the fuel.

On a multisized pebble system Eq. (1) must be applied to each size ball separately, and the sum of the results is the heavy metal loading of the core.

ORNL-DWG 78-4713

Fig. 6.2. A Typical Pebble Bed Reactor Fuel Element.

A quantity of more interest is the heavy metal loading of each pebble, which is given by Eq.(2):

$$M(\text{mass}) = F_2 F_3 F_4 \rho_{\text{fuel}} V_p M_{\text{HM}} M_{\text{fuel}}, \quad (2)$$

where each term retains the aforementioned definitions and V_p is the volume of the pebble. Two examples are included for illustration. These represent the range of ball sizes recommended by General Electric in their conceptual designs.^{2,6} Specifications for these balls are given in Table 6.2. It should be noted that the fueled zone in the large balls consist of a fuel shell rather than an internal fuel plug in the pebble core. It should also be noted that a fractional fuel loading of 67% by weight (49% by volume) is a rather optimistic number. The maximum fractional loadings with present technology are in the 40% by volume range.⁷ This reduces the loadings in the small (breeder) pebbles from the range considered by General Electric to 1.171 and 9.372 for each case, respectively, thus having a detrimental effect on the two-size pebble breeding

Table 6.2. Core Loading and Fuel Element Characteristics for Thermal Breeder Fuel Cycle in Pebble-Bed Breeder Reactor

	Breeder Pebbles	Zoned Seed Pebbles
Fuel	Th	Th + U
Fuel particle diam, μm	400	400
Particle fuel fraction, vol %	0.225	0.225
Buffer fraction, vol %	0.320	0.320
Coating fraction, vol %	0.455	0.455
Fractional fuel loadings, wt %	67	33
Heavy metal loading (g/ball)	1.583-12.665	50-400
Moderation ratio (active core) C/HM	110	110
Fuel element diam, mm	15.35-30.70	100-200
Shell ID, mm	12.80-25.60	83.4-166.8
Fuel matrix ID, mm	0	72-144
Density of pebble shell, Mg/m^3	1.9	1.9
Fuel matrix, Mg/m^3	1.7	1.7
Core, Mg/m^3	0.5	0.5
Fill fractions	0.14	0.61
Height/diameter ratio	0.90-0.625	0.90-0.625

performance. It should be noted, however, that the heavy metal loadings presented by General Electric for the small pebbles can be obtained if uncoated fuel particles are used rather than silicon-coated particles. Loadings of 5.20 to 41.65 g/ball are possible in this configuration.

6.3.2 Pressure Drop

The calculation of the pressure drop through the core of a pebble bed reactor is perhaps the most critical factor influencing the design of the system. It may also be the most uncertain factor, particularly for systems in which two sizes of pebbles are employed.

The pressure drop through the pebble bed is a function of a number of reactor design parameters. Of particular interest in this study is the dependence of the pressure drop on the size of the pebble or pebbles used for fuel. Five variations on a base design were studied to assess this dependence.

The specifications for the base case are given in Table 6.3. The pebble size for this case is the same as that used in the AVR. The

Table 6.3. Specifications for the Pebble-Bed Case

Core diameter, m	11.78
Core height, m	5.50
Thermal output, MW	3000
Operating pressure, MPa (atm)	4.1(40)
Outlet temperature, °C	985
Inlet temperature, °C	550
Thermal efficiency	0.45
Pebble diameter, mm	60
Helium flow rate, Mg/s	1.326
Superficial velocity, m/s	6.7525
Average coolant density kg/m ³	1.802
Superficial area, m ²	108.9
Superficial Re	4199
f	0.15
ΔP core, kpa (atm)	69.5(0.686)
ΔP total, kpa (atm) ^a	125(1.23)
MW_p Pump power, MW (e)	81.77
Circulator efficiency	0.6
MW_{pt} Pump power, MW (th)	136.78
ϵ	0.429

^a Assumes a 55.0 KPa (0.543 atm) pressure drop through balance of the primary system.

remaining specifications are typical of the GE pebble bed design.² The first variation consists of adding a 10-mm pebble to the base case system. This effectively decreases the core coolant volume fractions to 0.25 from 0.39. The second variation is once again a single pebble system using a 100-mm pebble. The third is the same as the second with a 15-mm pebble added to the 100-mm pebble. This system represents the lower end of the range of pebble sizes proposed by General Electric. The fourth variation consists of a single pebble system with a 200-mm pebble. The last variation is the same as the fourth, with a 30-mm pebble added to the 200-mm pebble system. This is representative of the upper end of the range of pebble sizes proposed by General Electric.

The core pressure drop was calculated with Eq. (3):⁸

$$\Delta P = 6fH(1 - F)V^2\rho/D_p F^3, \quad (3)$$

where

- ΔP is the core pressure drop in Pa,
- f is the friction factor (0.15) for these cases,
- H is the core height in m,
- F is the void fraction,
- V is the superficial velocity in m/s,
- ρ is the average coolant density in kg/m³,
- D_p is the pebble diameter in m.

The net pressure drop through the remainder of the system (i.e., steam generators, circulators, etc.) is assumed to be 55 kPa (0.54 atm). A helium circulator efficiency of 0.6 was also assumed. The overall system efficiency was then calculated with Eq. (4):

$$e = \frac{\epsilon_{th}(3000 - MW_{pt})}{3000}, \quad (4)$$

where

MW_{pt} is the thermal pump power in megawatts.

The same procedure was followed for the remaining variations. A volume weighted "equivalent" diameter was used in the two-sized pebble system calculations. The results of these calculations are presented in Table 6.4.

Table 6.4. Calculated Pressure Drops and Overall Plant Thermal Efficiencies

Pebble Sizes (mm)	ΔP Core		ΔP_T		MW_{pt}	ϵ
	(kPa)	(atm)	(kPa)	(atm)		
60	72	0.71	125	1.23	136.8	0.429
60 + 10	347	3.42	402	3.97	439.8	0.384
100	42	0.41	97	0.96	105.9	0.434
100 + 15	208	2.05	262	2.59	287.6	0.407
200	20	0.20	76	0.75	83.1	0.438
200 + 30	104	1.03	159	1.57	174.1	0.424

An examination of this table indicates that the use of a two-sized pebble system has an increasing cost to overall plant efficiency as the size of the pebbles decreases. A 4.5% reduction in overall plant efficiency is calculated for the 60 + 10 mm system while only a 1.4% efficiency loss is noted for the 200 + 30 mm ball system. This behavior is a result of the increase in the "equivalent" diameter with increasing pebble size.

It is evident from Eq. (3) that the pressure drop through the core is directly proportional to the core height and proportioned to the square of the power density (through the velocity term). Since the pressure drop is sensitive to these factors, considerable attention should be paid to the pressure drop calculations before increasing the core power density and/or increasing the height-to-diameter ratio by addition of axial blanket to the system.

It should also be kept in mind that these results are preliminary scoping studies and should be used as such. Experimental verification of the pressure drop is recommended, particularly for the two-pebble system.

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