ANL-7382

ANL-7382

Argonne National Laboratory

**REACTOR DEVELOPMENT PROGRAM
PROGRESS REPORT**

September 1967

MASTER

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II. LIQUID-METAL FAST BREEDER REACTORS

A. EBR-II

1. Operations

In preparation for the installation of the new system for determination of sodium plugging temperature and the sampling station in the Fuel Element Rupture Detector (FERD) loop, both the primary and secondary sodium systems were cooled to 350°F and the secondary sodium system was drained to the storage tank. Repacking and maintenance of the high-pressure steam valves were carried out, and the safety relief valves on the steam drum were removed, lapped, and reset. The annual leak-rate tests of the pipe penetrations for the secondary sodium into the reactor building were conducted. Refilling and heating operations began on September 10, and the primary tank temperature reached 700°F standby on September 16.

Fuel loading for Run 26A began on September 18. Monthly and biweekly interlock check sheets were completed on September 21, and the reactor was made critical on September 22. The first portion of Run 26 (designated 26A) consisted of an experimental program for gaining information relative to understanding more completely the anomalous values of the power coefficient measured in Run 25 (see Progress Report for July 1967, ANL-7357, pp. 24-25), including control-rod calibrations, power-coefficient measurements, reactor kinetics experiments, constant ΔT experiments, and measurements of banked control-rod worths. Preliminary results of these experiments are reported in Sect. II.A.5.a.(ii).

a. Fuel Handling. Fuel handling during September included loading the reactor for Run 26A. Twenty driver-fuel subassemblies and five control-rod subassemblies which were nearing the burnup limit were replaced. A burnup of 1.2 a/o had been attained in 83% of these subassemblies.

An outer blanket subassembly containing depleted uranium was removed from position 9-C-5 for surveillance examination of the blanket elements. The stainless steel subassembly U-1605, in position 13-F-6, which was used to check for alignment of the core gripper, was removed because of possible damage to the upper adapter.

A summary of the transfer operations is given in Table II.A.1.

I. PLUTONIUM UTILIZATION--EBWR

A. Operations

1. Lay-up Activities

The lay-up of the EBWR facility continued through this month. All irradiated fuel is in the Building 310 Pool for storage. The nonirradiated fuel and other miscellaneous accountable materials were placed in storage elsewhere about the Laboratory. At this date, all fissionable material has been removed from the EBWR facility.

Following the fuel movement, the Building 331 storage pit was drained and decontaminated. The pit water had to be treated as liquid active waste. The pit is now covered by the grating plus plywood flooring.

The cooling tower and all associated lines within the shell have been drained. The tower water was put to the Laboratory drain system. The draining of all other miscellaneous process lines has been completed. The only items remaining to complete the plant lay-up are the decontamination of the liquid retention system and a scrub-down of the concrete floors. A radiation survey of other areas shows that additional decontamination is not necessary.

2. Fuel-pin Shipment to Battelle-Northwest Laboratory

The last shipment of irradiated fuel pins to Battelle has been prepared and is awaiting loading into the "Yankee" transfer cask. The last shipment will include one whole plutonium fuel assembly (Pu-59) plus thirty-six pins from the various areas of the core. Those pins from the uranium core areas were removed from the fuel assemblies by cutting the upper grid plate about the desired pin. This cutting was accomplished with little difficulty using a 3/4-in.-hole saw.

3. Gamma Scan of Several Whole Elements

Several whole fuel assemblies, representing each area of the core, were gamma scanned using a NaI crystal within the fuel-storage pit. The gamma-scan data will be used as a burnup-profile indication in conjunction with individual pin gamma scan and analysis data to be conducted by Battelle.

REACTOR DEVELOPMENT PROGRAM
Highlights of Project Activities for September 1967

EBR-II

The installation of new sampling and plugging-meter systems in the Fuel Element Rupture Detector (FERD) loop was completed except for instrumentation and electrical connections.

Run 26 commenced with an experimental program (designated Run 26A) for gaining information relative to the anomalous values of the power coefficient measured in Run 25. Analyses of data are continuing, but preliminary results strongly indicate that the effect of differing control-rod worths at different rod-bank positions was largely responsible for the apparent decrease in reactivity defect during Run 25.

Neutron radiographs of capsules from experimental subassembly XO11 (the subassembly which had experienced a fission gas release) showed that fuel elements failed in two ANL capsules and one GE capsule. The remaining fuel elements are apparently intact. Examinations of failed and intact capsules are continuing in preparation for eventual continued irradiation of many of the intact capsules.

Fabrication of EBR-II driver fuel took place on a reduced schedule because of a delay in reactor startup, with consequent lack of irradiated fuel for reprocessing and filling of available storage space for reprocessed fuel; eleven subassemblies were fabricated.

ZPR-3

Work continued on Assembly 50, the third in the series of simple plutonium-fueled assemblies being studied in ZPR-3. A series of Doppler-effect measurements was made, standard fission ratios were measured, fission foils used in ZPR-3 were intercompared with those used in the ZEBRA fast critical facility, and heterogeneity effects were measured within a ZPR-3 cell.

ZPPR

Construction progress at ZPPR included completion of sand fill over the vault workroom. Two layers of gravel and one of sand were placed over the reactor cell; these layers constitute about 30 percent of the total material to be placed there. Placement of corrugated steel for the backup containment structure was completed and seal welding is in progress. Blast doors were received and mounted in their frames.

Six prospective contractors for installation of the reactor assembly machine were briefed in preparation for submittals of proposals.

AARR

Preliminary heat-transfer calculations have been made for the beryllium reflector. The third electrically heated flow-channel test section has been installed in the transient heat-transfer test apparatus; tests have started toward determining the heat-transfer and hydrodynamic behaviors of fuel plates and coolant.

Tests of metal gaskets for the beam-tube nozzles of the reactor vessel are almost complete; test data for seven types of seals are available. Fabrication of components for the beam-tube test facility has been completed.

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ANL-7382

Reactor Technology

(TID-4500)

AEC Research and
Development Report

ARGONNE NATIONAL LABORATORY
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REACTOR DEVELOPMENT PROGRAM PROGRESS REPORT

September 1967

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Issued October 31, 1967

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FOREWORD

The Reactor Development Program Progress Report, issued monthly, is intended to be a means of reporting those items of significant technical progress which have occurred in both the specific reactor projects and the general engineering research and development programs. The report is organized in a way which, it is hoped, gives the clearest, most logical overall view of progress. The budget classification is followed only in broad outline, and no attempt is made to report separately on each sub-activity number. Further, since the intent is to report only items of significant progress, not all activities are reported each month. In order to issue this report as soon as possible after the end of the month editorial work must necessarily be limited. Also, since this is an informal progress report, the results and data presented should be understood to be preliminary and subject to change unless otherwise stated.

The issuance of these reports is not intended to constitute publication in any sense of the word. Final results either will be submitted for publication in regular professional journals or will be published in the form of ANL topical reports.

The last six reports issued
in this series are:

March 1967	ANL-7317
April 1967	ANL-7329
May 1967	ANL-7342
June 1967	ANL-7349
July 1967	ANL-7357
August 1967	ANL-7371

TABLE OF CONTENTS

	<u>Page</u>
I. PLUTONIUM UTILIZATION--EBWR	1
A. Operations	1
1. Lay-up Activities	1
2. Fuel-pin Shipment to Battelle-Northwest Laboratory	1
3. Gamma Scan of Several Whole Elements	1
II. LIQUID-METAL FAST BREEDER REACTORS	2
A. EBR-II	2
1. Operations	2
2. Fuel Cycle Facility (FCF) Operations	5
3. Outside Fuel Procurement	11
4. System Design Descriptions	12
5. Reactor Analysis and Testing	12
6. Development of Methods of Nuclear Analysis	19
7. Reactor System Testing, Surveillance, and Evaluation	24
8. High Power Operation	25
9. Fuel Swelling and Driver Surveillance	26
10. Mark-II Driver Fuel Element Development	29
11. Fuel-related Equipment	33
12. Reactor and Primary Coolant System Equipment	34
13. New Subassemblies Design and Experimental Support	36
14. Instrumented Subassembly System	37
15. Packaged Loop	38
16. Process Chemistry	39
17. Experimental Irradiation and Testing	45
18. FCF Process Analysis and Testing	51
19. FCF Equipment Improvement	52
20. Hot Fuel Examination Facility	53
21. Nuclear Instrument Test Facility Study	54
B. Physics Development	55
1. ZPR-3	55
2. ZPR-6	58
3. ZPR-9	59
4. ZPPR	59
5. ZPR Fuel and Nonfissile Materials Procurement	61

TABLE OF CONTENTS

	<u>Page</u>
C. Component Development	63
1. Sodium Quality Control	63
2. Sodium Quality Measurement	63
3. Reactor Mechanism and Instrumentation	64
4. Fuel Handling, Vessels, and Internals	71
D. Fuel Development	73
1. Metallic Fuels	73
2. Oxide Fuels	75
3. Carbide Fuels	78
4. Fuel Cladding and Structure	80
E. 1000-MWe Plant	87
1. Contract Management and Liaison	87
F. Fast Flux Test Reactor	88
1. Critical Experiment Program	88
III. GENERAL REACTOR TECHNOLOGY	92
A. Applied and Reactor Physics Development	92
1. A Continuous-flow Manganese Bath System	92
2. Evaluation of Cross-section Data	95
3. Calculation of Resonance Integrals in the Unresolved Region	96
4. Burnup Analysis and Fission Yields for Fast Reactors	96
B. Reactor Fuels and Materials Development	98
1. Behavior of Reactor Materials	98
2. Chemistry of Irradiated Fuel Materials	102
3. Thermodynamics of Fuel Materials	103
4. Basic Fabricability	104
5. Engineering Properties of Reactor Materials	110
C. Engineering Development	111
1. Development of Master-Slave Manipulator Systems	111
2. Instrumentation and Control	111
3. General Heat Transfer	112
4. Engineering Mechanics	114

TABLE OF CONTENTS

	<u>Page</u>
D. Chemistry and Chemical Separations	115
1. Fluoride Volatility Processes	115
2. Compact Pyrochemical Processes	119
3. General Chemistry and Chemical Engineering	125
IV. ADVANCED SYSTEMS RESEARCH AND DEVELOPMENT	126
A. Argonne Advanced Research Reactor (AARR)	126
1. Core Research and Development	126
2. Component Development	131
3. Physics Experiments and Analyses	137
V. NUCLEAR SAFETY	142
A. Materials Behavior, Equation of State, and Energy Transfer	142
1. Vapor Pressure of UO_2	142
2. Properties of Alkali Metals	142
3. Heat Capacity of Liquid UO_2	144
4. Physical Properties of Liquid UO_2	147
B. Reactor Control and Stability	147
1. Transfer Functions (Stability)	148
C. Coolant Dynamics	148
1. Critical Flow	148
2. Coolant Dynamics	149
3. Convective Instability	150
4. Core Component Dynamics	150
D. TREAT Operations	152
E. Gaseous Effluent Studies	153
1. Plutonium Volatility Safety	153
F. Containment	153
1. One-dimensional Code for Analyzing Containment Structures	153
2. Two-dimensional Code for Analyzing Containment Structures	154
VI. PUBLICATIONS	155

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TABLE II.A.1. Transfer Operations

Transfers to and from reactor grid	57
Subassemblies transferred to Storage Basket from FCF	8
Subassemblies transferred to FCF	7
Total	72

b. Operational Support and Maintenance

(i) Spare Parts. The fabrication of the spare core gripper is being postponed pending redesign of the gripper-jaw mechanism. Delivery of the spare FERD

loop EM pump has been delayed from September to late October 1967 because of fabrication difficulties.

Purchase orders for inventory additions to spare parts have been placed for the following items:

- (a) a breaker for the instrument thimble cooling turbo compressors;
- (b) bearings for the argon-cooling-system, 100-hp turbo compressor.

(ii) Maintenance and Modifications. Sodium-vapor traps for the FUM argon cooling system and for the purification system for primary-tank argon were replaced because of excessive differential pressures across the traps.

The prefabrication of the economizer for the secondary sodium oxygen analyzers was completed and installation begun.

Installation of the FERD-loop plugging and sample loop, Plant Modification No. 122, was the primary effort during September 1967. The present status of the job is:

- (a) The prefabrication of the plugging loop piping is complete.
- (b) The sample loop is welded into the FERD-loop piping, but is isolated by valves.
- (c) The piping systems except for the final welds on the plugging loop were radiographed and helium-leak tested.
- (d) The power supply has been installed except for the final connection to the instrument control cabinet.

The small, main steam-pressure-regulating valve, VC-501-B, was replaced with a new, heavier duty valve. The welds were radiographed, and the system was hydrostatically tested with satisfactory results.

The annual leak-rate test of the secondary sodium piping through the reactor building penetrations was satisfactorily completed. The leak rate through the east penetration, R2-X-701, was 0.32 scfd. The leak rate through the west penetration, R2-X-702, was 0.06 scfd. Both penetrations were tested at 24 psig for 8 hr.

Both steam drum relief valves were disassembled, the seats and discs were lapped, and the valves were reassembled, adjusted, and tested. The present settings are as follows:

<u>Valve</u>	<u>Lift Pressure (psig)</u>	<u>Reset Pressure (psig)</u>
East P3-SV-677A	1450	1360
West P3-SV-677B	1480	1300

The main feedwater-control valve, B5-VC-610, was disassembled for inspection and repairs. Both seats, which were cut in several places, were replaced, and the valve was reassembled.

The emergency motor-operated feedwater-control valve, SF-3, was disassembled for inspection and repairs. A piece of metal was found imbedded in the stellite seat. The valve seat was lapped as much as possible to provide a seating surface. The alignment and supports for the valve operator were improved.

The startup feedwater-pump packing gland was modified in accordance with the manufacturer's recommendations by eliminating the lantern ring and providing for cooling water across the top ring of packing. Pump running time since the modification has not been sufficient to show if the change will increase the life of the packing.

The four cooling-tower riser block valves which have been very difficult to operate were disassembled and modified to accept a grease fitting in the bonnet. The modification should improve the valve performance.

In the fission-gas monitor system, a defective pump was replaced. Vapor trap "A" was removed, cleaned, and a new mesh installed. A valve which was plugged with sodium was cleaned. The wire drive was repaired, and all tubing fittings were checked for gas leakage.

Because of an intermittent ground annunciation on the 2400-V switchgear, the 2400-V bus was de-energized and meggered. A low resistance to ground was found on the ZPPR-ITF breaker. The ground was located and the condition corrected.

A rupture occurred in the primary-purification-system plugging valve bellows. A new valve was installed and the loop heater replaced.

c. Operator Training. Revision of the Coolant Systems training material continued. One requalification examination was administered and completed successfully. Two oral examinations were given for qualification and both were passed.

d. Operating Manual. Operating Manual Revision Bulletins Nos. 57 and 59 were issued. Bulletin No. 57 contained 13 revised sections for the Steam and Turbine-Generator Systems, and Bulletin No. 59 contained five revised sections for the Primary and Secondary Auxiliary Systems.

Operating Manual Revision Bulletin No. 58. "Administrative Procedures" and "Control Room and General Operating Procedures" is being reviewed by the Operations Manager.

Drafting of a new section covering operation of the Fuel Element Rupture Detector loop is in progress.

In the past month, special procedures were issued on the following subjects: Power Coefficient; Repiping of Cooling Water to Startup Feedwater Pump; Constant Power Supply; and EBR-II Reactor System Testing, Surveillance, and Evaluation.

e. Planning and Scheduling. Schedules were prepared for installation of the sample station and plugging meter on the FERD loop. Alternative schedules were prepared for installation of the rod oscillator.

2. Fuel Cycle Facility (FCF) Operations

a. Hot-line Operation. Production schedules were reduced during the past month in order to accommodate the proposed surveillance program of 16 subassemblies from Run 25. A delay in reactor startup and subsequent delay in discharge of Run 25 subassemblies has resulted in rescheduling of this surveillance program for next month.

As a result of the delay in startup of the reactor, all the available storage space for reprocessed fuel is occupied and the inventory of fuel material available for processing has been used. Because of the reduced processing activities, considerable time and effort has been spent on repair and maintenance of in-cell equipment. Table II.A.2 summarizes the production activities for the month.

TABLE II.A.2. Production Summary

	9/1/67 through 9/30/67	Total This Year
1. Subassemblies Received:		
Core, Control, Safety (for processing)	4	44
Other (X900, U1605, U1317)	3	122
2. Subassemblies Dismantled (for processing)	1	48
3. Subassemblies Dismantled (for examination, etc.)	2	35
4. Subassemblies Fabricated (includes those made with unirradiated fuel and in cold line)	11	135
5. Subassemblies Transferred to Reactor	4	75
Subassemblies Stored in L&S Vault and Interbuilding Corridor	9	24
6. Elements Decanned:		
From Irradiated Subassemblies	0	4,396
Rejects	0	772
Other	0	0
Total Decanned	0	5,168
<u>Melt Refining</u>		
	<u>Irradiated Fuel</u>	<u>Recycle Material</u> <u>New Fuel</u>
7. Number of Runs	1	5 1
8. Average Pour Yield, %	94.0	92.3 98.2
1967 to date (Total--147)	35	64 48
<u>Processing</u>		
	9/1/67 through 9/30/67	Total This Year
9. Injection-casting Runs (Total Number)	10	139
10. Elements Processed:		
Accepted	1074	12,324
Rejected	134	1,598
11. Elements Welded	1064	12,189
Rewelded	0	13
12. Elements Leak Tested:		
Accepted	871	11,501
Rejected	65	239
13. Elements Bonded (including recycle)	1265	14,206
14. Elements Bond Tested:		
Accepted	842	11,799
Rejected	58	715
15. Elements to Surveillance	0	407
Number of Subassemblies	0	40
<u>Waste Shipments</u>		
16. Cans to Burial Ground	5	85
17. Oxide and Glass Scrap to ICPP	2	50

Five special ingots were prepared and fuel pins cast from them. These castings intentionally contain higher than normal concentrations of trace elements (Si, C, Al, and Fe) for use in the program to investigate the anomalous swelling behavior of the driver fuel (see Sect. II.A.9.a).

A series of runs has been made in the melt-refining furnaces to determine the best methods of introducing trace elements into the fuel ingots. The postirradiation examination of fuel elements has shown that traces

(200-1,000 ppm) of certain elements (Si, Al, Fe, etc.) may have a desirable effect in increasing the radiation stability of the fuel alloy. These elements have been added in various chemical forms (e.g., SiO_2 and Si metal), and the liquation times for the element addition melts has been varied from 1/3 to 2 hr. Preliminary results indicate the following:

(i) Silicon Addition. The addition of silicon to the melt is time dependent and requires more than one hour for quantitative addition. Less than half of the silicon is alloyed with the matrix alloy when the liquation period is one hour or less. The method of introduction of the silica or silicon to the charge material influences the alloying. If the silica or silicon is placed below the bulk metal in the crucible, the alloying is more complete than when the additive is mixed with or placed on top of the bulk metal.

(ii) Iron or Aluminum Additions. Quantitative addition of iron or aluminum can be obtained with less than one hour of liquation.

Work is continuing to define further the effect of these parameters and to determine methods of carbon addition.

b. Cold-line Operation. Operation of equipment with depleted and enriched uranium (see Progress Report for August 1967, ANL-7371, p; 6) indicated that modifications were required in some of the equipment. Most of September was devoted to reworking and retesting the equipment and preparing final drafts of operating procedures.

Three runs were made in the alloy-preparation furnace with depleted uranium-fissium alloy. In the first run, silicon metal was added to make an ingot containing 2% silicon. This material did not pour because of a heavy oxide layer, but the material was remelted later in a second run in an oversized crucible (thoria-coated graphite) after some of the oxide was removed, and was poured successfully. Pins that were cast from this alloy can be added to regular melts to increase the silicon content of the alloy to the desired level. The third run in the furnace was a 7.5% fissium alloy. Some of the injection-cast pins from this ingot will be used for spectrographic electrode standards for the driver fuel being purchased by the Laboratory. The balance of the material will be remelted to make 5 and 2.5% fissium ingots for additional standards.

The last two runs in the injection-casting furnace caused severe warpage of the base plate. This in turn caused several of the insulating components to break. The base plate was redesigned, a new one was manufactured, and new insulators were also machined and heat treated. One set of bearings that support the upper heat shield was replaced. The furnace was run once with no metal to outgas the new components, and then the depleted uranium-7.5% fissium ingot was cast into pins.

Except for minor modifications to the instrumentation, the bonders and bond testers were operable. Six of the reject elements from Run 002-I were stripped and the pins cleaned of sodium. The pins were investigated metallographically for grain size and structure, and were found to be acceptable.

The components of the cold-line assembly machine have been received and assembled. The welder power supply that had been used by the Remote Control Division for development of the Vertical Assembler-Dismantler (VAD) was shipped also, and has been set up to weld subassemblies.

A contract with an outside vendor to supply 600 kg of fissium (95% U-52% U²³⁵) in ingot form to be used as the source material for the cold line is still being negotiated. Problems with delivery dates are being encountered. Alternate sources of this material are being investigated.

(i) Subassembly Transfer Coffin. The lead in the cask shell has been poured and the cask returned to ANL, Lemont, for final machining and painting. The cask will be shipped to Idaho in the first part of October. Installation of small parts will be completed in Idaho.

c. Maintenance and Repair. The bridge drive on the Air Cell crane failed several times in the past month. Electrical checks indicated an open circuit somewhere between the outside junction and the drive motor, and pointed to brush and bus-bar problems. A new TV system was used to watch the brush system as the crane was driven past the suspect area. The system worked very well and clear Polaroid pictures were made of the TV picture. Several areas of bus-bar pitting were noted; also some of the brushes appeared to be worn down somewhat, especially in the bottom of the groove where arcing may have occurred. The bus bars will be coated with a conductive lubricant. Further study will be required to evaluate any long-range solution to the problem.

The program for computer control of maintenance of auxiliary equipment has been put into effect.

d. Operating Manuals and Reports. One new manual section was completed and submitted for review and acceptance. One new section was issued as an accepted portion of the FCF Operating Manual. Six sections and/or portions of sections have been approved for issuance, and seven sections and/or portions of sections are in various stages of preparation.

e. Product Analysis.

(i) Chemical Analyses. The numbers of fuel-production analyses for cast-pin and ingot samples from both the "hot" and "cold-line," together with the average values and range, were as follows:

Production Fuel

<u>Analyzed for</u>	<u>Number</u>	<u>Average Value</u>	<u>Range</u>
U (total and isotopic)	21	94.95 a/o	94.19-95.54
U ²³⁵		52.20 w/o	52.12-52.27
Pu (total and isotopic)	15		
Mo	26	2.46 w/o	2.26-2.58
Ru	26	1.96 w/o	1.85-2.08
Pd	22	0.18 w/o	0.15-0.22
Rh	15	0.289 w/o	0.273-0.318
Zr	20	0.075 w/o	0.035-0.147
Nb	15	0.016 w/o	0.010-0.018
Fe	41	276 ppm	37-558
Al	13	197 ppm	71-337
Si	30	210 ppm	100-340
C	6		
Burnup (La ¹³⁹) ^a	4		
Total	254		

^aSurveillance pins.

(ii) Postirradiation Analysis of EBR-II Fuel. No spent driver-fuel subassemblies were returned from EBR-II to FCF.

Analysis of driver-fuel irradiation-swelling data as a function of silicon content in the U-5% Fs alloy has been made. Results are presented graphically in Figs. II.A.1 and II.A.2 for Mark-IA and Mark-I fuel, respectively. These data indicate a trend toward lower fuel

Fig. II.A.1. EBR-II Mark-IA Fuel Swelling at Approximately 1.1 a/o Maximum Burnup vs. Silicon Content of Fuel. (Idaho Division Silicon Analyses)

Fig. II.A.2. EBR-II Mark-I Fuel Swelling at Approximately 1.1 a/o Maximum Burnup vs. Silicon Content of Fuel. (Idaho Division Silicon Analyses)

swelling with increasing silicon content, although the amount of data is still meager and variables other than silicon content may have an influence. Such variables will be included in the analysis when further information is available. When fuel utilized in EBR-II Run 25 becomes available for postirradiation analyses, analyses will be made of 434 elements from 16 subassemblies.

Calculations of driver-fuel-element irradiation temperatures have recently become available. Using this information, a preliminary analysis intended to characterize driver-fuel irradiation swelling as a function of irradiation temperature has been made. Results for U-5% Fs alloy are presented in Fig. II.A.3. The temperature range shown for each subassembly represents the calculated temperatures across the subassembly during irradiation. Within each subassembly, all elements came from the same injection-casting batch. (From 7 to 21 elements from each subassembly were examined for this study.) Additionally, all material of each type of alloy came from the same casting batch except for the "SL" alloy, which came from two batches. It can be observed that a trend toward higher swelling rates with increasing temperatures is indicated, even for the limited temperature range available for examination. Further work is under way to determine the reason for the wide spread of data on U-Fs fuel (the program to investigate anomalous swelling behavior).

Approximately 600 Mark-IB-type fuel-element restrainers have been received. These will be used in the manufacture of the initial group of five or six Mark-IB subassemblies that will be used for evaluation of irradiation performance.

Fig. II.A.3. EBR-II Mark-IA Fuel Swelling per a/o Maximum Burnup vs. Volumetric Average Irradiation Temperature

The ICPP corrosion testing of the five irradiated EBR-II fuel elements stored in their water basin (see Progress Report for July 1967, ANL-7357, p. 57) is continuing. After two months of storage in the basin, the fuel elements show no significant evidence of fission-product leakage. Testing of the fuel elements for cladding defects after total exposure of three and six months to the basin water is planned.

The ICPP proposes to measure the fission-product leaching rate from a known area of exposed fuel in an irradiated EBR-II fuel pin. Equipment has been fabricated to perform this measurement on the small piece of de clad EBR-II fuel sent to the ICPP last month.

3. Outside Fuel Procurement

a. Procurement of Mark-IA-IB Fuel

(i) Fuel Elements. Aerojet has received approval for modifications in its special nuclear materials license and can proceed with fuel fabrication. Construction of the injection-casting furnace is under way. For nuclear safety reasons, a Freon system will be used for furnace cooling. Vycor molds have been ordered. A machine for shearing cast pins is being fabricated.

Delivery of the first lot of fuel element tubing is expected in mid-October. Arrangements were made for conducting flare, reverse flattening, tensile, and burst tests on tubing samples. An ANL engineer

will assist in setting up and initially operating eddy-current equipment for tubing tests at Aerojet. Restrainer samples submitted by Aerojet for qualification were approved, and production of restrainers is proceeding. Material for tip fabrication has been received.

The Aerojet Project Manager visited ANL-Idaho to observe cold-line operations, to discuss contract status, and to submit a process outline and list of procedures. Aerojet has determined that the standard ICC-approved shipping container planned for use for fuel elements will not hold 400 elements as expected, and that the use of a removable insert would further reduce its capacity. Information was furnished to Aerojet on another (nonstandard) container which might meet requirements. The Argonne resident inspector entered a training period, expected to last about five weeks at both Argonne, Illinois, and Idaho. Following this, he will proceed to San Ramon.

(ii) Subassembly Hardware. Additional information was obtained on request from one of the bidders on the proposal for fabrication of about 500 core, fueled inner blanket, control, and safety rod preassemblies. On the basis of this information, a recommendation for contract negotiation was made.

4. System Design Descriptions

System design descriptions are being prepared. They will make readily available to plant and equipment designers and to utility operators the design and technology information on EBR-II that is pertinent for their use as guidelines in designing and operating safe and reliable large future liquid-metal-cooled fast breeder reactor power plants. These descriptions will stress reactor-related equipment and will include features affecting safety, reliability, and performance.

Collection and organization of source data are continuing. Tests of the information-retrieval procedure have been initiated. Reviewer comments on February and June outlines are being evaluated as received.

5. Reactor Analysis and Testing

a. Operational Data. A melt-wire temperature monitor has been developed for monitoring subassembly outlet temperatures. The principle is the use of metallic pins of alloys with known melting points contained in a sealed capsule which is then mounted in the outlet of a subassembly. By means of neutron radiography the capsule may be examined after the subassembly is removed from the reactor. The maximum coolant outlet temperature would be bracketed by the melt temperature of the highest rated alloy which did melt and the melt temperature of the lowest rated alloy which did not melt.

The test capsule is a drilled stainless steel block 1 by 2 by 1/4 in. thick (designed for mounting in a subassembly upper adapter). The 1/16-in.-dia melt wires which are about 1/4 in. long, and 3/32-in.-dia stainless steel weights which are about 1/2 in. long, are mounted in 1/8-in. holes drilled into the block. A cover plate is welded over the tops of the holes to seal the melt wires and weights into the holes.

Figure II.A.4 shows X rays taken of a test capsule which was heated to temperatures indicated in an electric laboratory furnace. Two melt wires of each of three types are mounted in the block. Reading from left to right they are:

Silver-Antimony, melting point	482°C (900°F)
Zinc-Silver, melting point	434°C (813°F)
Zinc, melting point	420°C (788°F)

Fig. II.A.4. Melt-wire Coolant Temperature Monitor (X rays after heating in electric furnace)

It is expected that other alloys can be developed to narrow the difference between melt temperatures. Experiments are continuing to make and test other alloys.

b. Digital-data Collection. The magnetic tape-recording system for on-line data acquisition has been completed, installed, and satisfactorily checked out. The system now has a full six-channel capability (limited to six by the input capacity of the NAVCOR analog-to-digital converter), and has variable gain and signal-conditioning capabilities for all channels. The system is completely operational and will be used in the immediate future for high-resolution processing of rod-drop and transfer-function data, and possibly for fine control-rod calibration.

c. Nuclear Analysis

(i) Investigation of Power Coefficients as a Function of Core Size and Composition. Of particular interest in the investigation of the Run 25 power-coefficient anomaly (see Progress Report for July 1967, ANL-7357, pp. 24-25) is the effect of core size on the power-reactivity defect, defined as the reactivity necessary to bring the system from hot critical (700°F) to operating power (45 MW). Accordingly, previous power-coefficient data were studied to establish whether a correlation could be detected between core size and measured values of the reactivity defect. Although the results of the studies are not definitive, it was nevertheless noted that when core size increased, the reactivity defect tended to decrease. In an effort to establish whether the correlation is real and the effect large enough to explain the relatively small Run 25 reactivity defect, two independent mathematical studies are being conducted.

(α) One-group Calculations. By means of one-group theory and an assumption that the core is a reflected cylinder, hand-calculated values were established for the power coefficients of the following EBR-II-type cores: (1) Run 24 with a uranium blanket; (2) Run 24 with a stainless steel reflector; (3) a tenfold volumetric expansion of case (1). As a further consideration, each of the three cases was evaluated according to whether the core was "loose" or "tight," i.e., whether or not radial expansion is continuous.

The method and a summary of results will be given in a forthcoming ANL report. The results obtained thus far for the power coefficient and the isothermal temperature coefficient are given in Table II.A.3.

TABLE II.A.3. Calculated Power and Isothermal
Temperature Coefficient for EBR-II-type Cores
(One-group Calculations)

Case:	(1)		(2)		(3)	
	Uranium Blanket		SS Reflector		Tenfold Core Uranium Blanket	
	Loose Core	Tight Core	Loose Core	Tight Core	Loose Core	Tight Core
Power Coefficient, 1h/MW	-0.91	-1.41	-1.41	-1.78	-0.83	-1.29
Isothermal Temp Coefficient, 1h/°F	-0.30	-0.58	-0.71	-0.90	-0.28	-0.51

From these data the following conclusions may be drawn: (a) the substitution of stainless steel for uranium in the blankets has a strong effect on the power coefficient, (b) the effect of core tightness on the power coefficient is substantial for all assumed configurations, (c) increasing the core volume decreases the power coefficient, and (d) effects reflected in the power coefficient are also reflected in the isothermal temperature coefficient.

The one-group method is currently being programmed for the IBM-1620 computer. When the program becomes operational, additional calculations will be carried out for various Run 25 conditions.

(β) Multigroup Two-dimensional Diffusion Calculations.

In another approach, a series of two-dimensional diffusion-theory calculations (CANDID) was run to establish the relationship between power coefficient and core characteristics for Runs 16 and 24, two typical EBR-II loadings for which reliable power-coefficient data are available. Considered in the computation were the change in core height (14.22 to 13.50 in.) and the change in enrichment (48.5 to 52.4 a/o U^{235}). In both cases actual composition and enrichment were homogenized to give core, axial gap, and blanket regions, and a depleted uranium axial blanket was assumed even though Run 24 was characterized by stainless steel axial reflectors. In view of the results of the one-group calculations above, such an assumption may not be valid. The results of the calculations are summarized in Table II.A.4.

TABLE II.A.4. Effect of Core Size and Composition on Power Coefficient
(Multigroup Two-dimensional Calculation)

Principal Components	Run 16 75 Subassemblies (Ih/MW)	Run 24 81 Subassemblies (Ih/MW)
Core Sodium	-0.425	-0.425
Upper Axial Gap and Reflector Sodium	-0.310	-0.326
Radial Blanket Sodium	-0.075	-0.070
Axial Fuel Expansion	-0.510	-0.495
Radial Fuel Displacement of Sodium	-0.95	-0.95
Total	-1.415	-1.411
Measured	-1.50	-1.27

In this case only a slight decrease, from -1.415 to -1.411 lh/MW, is indicated in the power coefficient by the increase in core loading.

(ii) Experiments with a Run 25 Configuration during Run 26A.

In an effort to provide additional information on the Run 25 power coefficient anomaly, a series of experiments was conducted at the beginning of Run 26. To preserve Run 25 conditions as much as possible, the only significant loading change consisted of a replacement of 20 expended driver subassemblies and five expended control-rod subassemblies with fresh fuel. No change was made in the complement of irradiation experiments, and core size remained at 88 subassemblies.

(α) Banked Control-rod Experiment. This experiment was intended to demonstrate that the magnitude of the measured power

TABLE II.A.5. Power-reactivity Defect:
for 13.00-in. Rod Bank Position

Power Level (MWt)	Reactivity Decrement (lh)		
	9/25	9/27	9/29
0.05	0	0	0
0.50	1.0	-	-
10.0	16.0	18.4	17.3
15.0	17.6	20.4	19.4
20.0	19.3	-	20.9
25.0	21.6	24.0	22.5
30.0	23.3	-	23.9
35.0	27.0	27.3	28.0
39.0	31.5	-	32.1
45.0	37.0	39.5	38.3

coefficient depends on the banked position of control rods. The reactivity defect was studied as a function of power for the following control-rod conditions: nine rods banked at 13.00 in., one rod at 7.00 in., and the regulating rod moving between 5.61 and 8.18 in. The preliminary results corrected for temperature and burnup effects are given in Table II.A.5.

Following this series of measurements, ten control rods were rebanked at 11.00 in. at 45 MW. From the position of the regulating rod the reactivity defect was found to increase to 42.3 lh. The difference of 5.3 lh is directly attributable to the

effect of banked control rods, which has been postulated as the mechanism responsible for the apparent decrease in reactivity defect during Run 25. Although these results are preliminary, they indicate that the apparent loss in reactivity defect during Run 25 has been explained.

(β) Calibration of Control Rods. To permit the application of detailed corrections to the data, a careful calibration of control rods in the region 10-14 in. was conducted. Not only were the worths of individual rods established, but the worth of the entire bank between 11 and 14 in. was measured relative to a carefully period-calibrated rod (No. 8). The results for various rod bank positions are given in Table II.A.6.

In the course of the above measurements, rods 8 and 3 were period-calibrated over their 14-in. length. The other ten were inter-calibrated. The results indicated a

TABLE II.A.6. Reactivity Worth of Control-rod Bank

Rod Bank Position (in.) (All rods except 8 and 5)	$\Delta k/k$ (lh)
14.00	0
13.00	28.0
12.00	76.5
11.00	144.8

total control rod worth of 1565 lh, which compares favorably with a corresponding total worth of 1595 lh at the beginning of Run 25.

(γ) Calibration of Safety

Rods. The two safety rods were calibrated by subcritical counting and were found to be worth 1.07% $\Delta k/k$. Although this value is less than the 1.2% $\Delta k/k$ originally

measured, it is consistent with the increase in core size and the relative flattening of the flux in the central core region.

(δ) Other Experiments. Rod-drop tests under a variety of power and banked-control-rod conditions were conducted. Additional experiments in the form of reduced-flow/reduced-power (constant ΔT) tests were also carried out. The results of those experiments will require considerable processing.

(iii) Reactivity Code for Loading Change. A new code, which computes changes in reactivity caused by changes in loading, was used in planning the loading for Run 26A. In brief, the code uses the same input deck as the burnup code, and computes the reactivity difference between the end of one loading and the beginning of the next. As an illustration of its reliability, the calculated reactivity change of 70 lh from the end of Run 25 to the beginning of Run 26 compares satisfactorily with the measured change of 65 lh.

d. Thermal and Hydraulic Studies

(i) Temperature Calculations Necessary for Interpreting Constant- ΔT Tests. In an effort to establish the magnitude of the power-coefficient component associated with fuel expansion, reduced-flow/reduced-power tests were conducted. In these tests the flow and power were simultaneously reduced in a manner that maintained a constant axial temperature differential across the core. A measurement of the associated reactivity change may be interpreted in terms of a power-coefficient component for fuel if the temperature of the fuel is known at the various flow and power conditions. To obtain this information the HECTIC II code has been used to estimate average temperatures in the fuel and coolant for various row positions of a Run 25 configuration. Calculations have been carried out for the following conditions: 41.5 MW, 100% flow; 22.5 MW, 100, 90, 75, 60, and 54% flow. Similar calculations were carried out for 45 MW, 100% flow, for Runs 24 and 25 configurations.

(ii) Thermal and Hydraulic Considerations for the Reorificing of Stainless Steel Reflector Subassemblies. A series of calculations has been made to investigate the effect of reducing the flow of sodium through the stainless steel reflector subassemblies in Rows 7 and 8. Flow rates of 3.3 gpm in 7th-row subassemblies and 1.35 gpm in the 8th-row subassemblies were used, since these specific flow rates give bulk outlet temperatures (neglecting radial heat transfer) quite close to the calculated values for the adjacent 6th- and 9th-row subassemblies (core and blanket, respectively). In all cases calculations were made for subassemblies located on the flats of the 7th, 8th, and 9th rows. The results showed a virtually flat radial temperature profile across the subassemblies. In the above calculations, it was necessary to assume an arbitrary but reasonable temperature distribution in the 10th row. An attempt was subsequently made to calculate the 10th-row temperature distribution, but a problem developed with lack of convergence of the HECTIC II solution. The problem appears to be the result of a combination of low power production and low flow rate in the 10th row. An attempt will be made to eliminate this difficulty by subdividing the 10th row into several axial segments.

e. Safety

→(i) Rod-drop Analyses. In the absence of an oscillator rod, numerous attempts have been made in the past to obtain kinetic information from a study of the power decay during and following a steep ramp withdrawal of reactivity from the system. To provide the ramp a control rod was replaced by a stainless steel counterpart which, during scram action, gives a withdrawal rate of $0.0010 \Delta k/k/\text{sec}$ over a period of approximately 0.3 sec. Past attempts to interpret the power decay in terms of reactivity have been based on a computer program which corrects for the effects of delayed-neutron groups and gives, as output, the total reactivity in the system as a function of time during and following the ramp. Until the recent Run 25 power-coefficient difficulties, the use of this technique was limited to a qualitative comparison of a given reactivity-recovery curve with a reference curve established earlier. Attempts to place analyses on a quantitative basis were frustrated by problems which are only now being resolved.

One such problem concerns an uncertainty in the withdrawal rate which, unfortunately, is not constant with respect to time. This difficulty is being resolved in the following manner. The power trace, established during a drop at zero power (actually 500 kW), and the shape of the rod position-time trace are used to establish a calibration curve which gives reactivity as a function of rod position. A program has been written which accepts as input the power decay and the time-dependent rod position for a number of drops at zero power. The output is an average of the individual reactivity-position calibrations. The program corrects for the effects of delayed-neutron-emitting species.

At higher levels of power, for which reactivity feedback exists, the program utilizes the rod calibration data, the power versus time data, and the rod position versus time data, and, by solving the inverse kinetic equations, computes feedback reactivity as a function of time. The practice of using zero-power rod-drop data to calibrate the drop rod is preferred to a period-based calibration, since most of the error introduced by uncertainties in delayed-neutron data tends to cancel that portion of the program where the rod reactivity is subtracted from the net reactivity. This particular feature of the program is important since with a small fuel time constant, less than 0.3 sec, and a long drop time, about 0.3 sec, the feedback from the fuel may be only 20% of the rod reactivity at short decay times.

Another difficulty which has frustrated previous attempts to place a quantitative interpretation on rod-drop data concerns the existence of noise in the power trace. Attempts are being made to utilize the IBM-1620 for data smoothing prior to rod calibration and feedback calculations. If the attempted refinements prove successful, the time-dependent feedback function will be used as input for a computer program which will estimate the amplitudes and time constants of up to three feedback components.

The results of efforts to place a quantitative interpretation on rod-drop data are sufficiently encouraging to justify a considerable effort in evaluating the prompt feedback component in previously unevaluated Run 25 rod-drop tests.

6. Development of Methods of Nuclear Analysis

a. Physics Analysis of the EBR-II Power Coefficient. A preliminary evaluation of the experimental data relating to the postulated "blanket-drift model" (see Progress Report for July 1967, ANL-7357, pp. 25-26) indicates that within the experimental error there is not a measurably significant difference between the power-coefficient curves obtained before and after the attempt to randomize the attitude of the core and blanket subassemblies.

This can be seen in Fig. II.A.5 in which the data of the July 21, 22, August 17, and 18 runs have been plotted with the estimated uncertainty of ± 0.5 lh in reactivity values and ± 0.5 MWt in the power readings. Some points represent an average of the values of reactivity loss obtained at powers which differed by 0.2 MW or less. If it is assumed that this uncertainty is equivalent to a standard deviation of the measurement, statistical analysis would show that 68% of the measured values should lie within a standard deviation of the true value. The error margin used is on the low side of error range, which is usually quoted as being between

0.5 to 1.0 lh in reactivity and between 0.5 and 1.0 MWt for power. The smooth line drawn through all but one of the squares in Fig. II.A.5 appears to fall well within the statistical requirement for representing the true value of the reactivity loss as a function of the reactor power.

Fig. II.A.5. Power Coefficient Measured during Increasing Reactor Power Run

The slopes of the power coefficient curve for each run over the power range between 10 and 27 MWt were estimated, the results being given in Table II.A.7. The averaged slopes when subjected to a first-order error analysis were found to be within the margin of error.

TABLE II.A.7. Average Power Coefficient of Reactivity over the Power Range from 10 to 27 MWt

Run 25	Operating Period (MWd)	$\Delta k/\Delta P$ (Ih/MWt)
July 21, 1967	1525	0.18 ± 0.03
July 22, 1967	1540	0.19 ± 0.03
August 17-18, 1967	1560	0.26 ± 0.04

The apparent numerical differences are of no significance since the density of experimental data for each of the runs is inadequate for a proper statistical treatment of the data or for a better estimate of the averaged slopes. This is particularly so for the last two runs in the 10 to 27 MWt power range, where only 3 to 4 determinations have been made for the August run and only 2 points were included in the July 22 experiment.

It may be concluded that the reactivity feedbacks that were operable in the July 21 and 22 runs were not altered to any significant degree by the attempt to reorient the core and blanket subassemblies.

One of the more significant experiments performed in the study of the Run 25 power coefficient involved varying power level and partial flow. The series of measurements involved determining the reactivity decrement at a power level of 22.5 MWt and at 100, 90, 75, 60, and 54% of the coolant flow. This coolant flow coefficient allowed a check on the consistency of deriving the nonlinear effect, i.e., "bowing effect," from the full-flow case. In both cases one must account for the control-rod-system expansion component, the coolant temperature effect, and the fuel axial-expansion component. The two cases differ in that the relative temperature conditions of coolant, structure, and fuel are changed.

In the partial-flow experiments the fuel-temperature component of reactivity is somewhat lower than for the comparable bulk sodium environment at full flow. This increases the relative contribution of the bowing component to the total reactivity feedback. The effect of this positive component is readily observed in the partial-flow experiments whereby the coefficient displays a slightly positive slope and then becomes negative as the "bowing component" begins to saturate.

Fig. II.A.6. The Nonlinear "Bowing" Component of the Total Reactivity Feedback for Run 25 vs. Reactor Power

The consistency of the magnitude of the "bowing" reactivity effect between the full-flow and the partial-flow experiments is presented in Fig. II.A.6, where the nonlinear component of reactivity is plotted for Runs 24 and 25 as a function of reactor power. The agreement is found to be well within the experimental error.

b. Analysis. A twofold symmetric representation was adopted for the SNARG two-dimensional x,y studies of Run 25 to replace the previously used fourfold symmetric configuration. The computational representation of the Run 25 loading is included in Fig. II.A.7. Although the size limitations in the SNARG program required a loosening of the

meshing in the blanket, the meshing over the core and reflector regions, Rows 1-8, involving quartering of the subassemblies, was retained. Assuming the same z-direction buckling synthesis as in the fourfold symmetry case, the critical fuel-assembly uranium enrichment for this configuration was calculated to be 0.526, which is in agreement with the experimental value of about 0.52.

EBR-II EXPERIMENTAL LOADING RUN 25A

TWO DIMENSIONAL SNARG REPRESENTATION OF
EBR-II, RUN 25 TWO-FOLD SYMMETRY ABOUT AXIS AA

KEY:

	D	DRIVER FUEL
	SB	STAINLESS STEEL BLANKET
	C	CONTROL ROD
	S	SAFETY ROD
EXPERIMENTAL	{ D/2	1/2 FUELED DRIVER SUBASSEMBLY
LOADING ONLY	X	EXPERIMENTAL SUBASSEMBLIES
SNARG REPRESENTATION ONLY	X	{ ASSUMED 1/2 FUELED DRIVER SUBASSEMBLY COMPOSITION

NOTE: 1) CONTROL ROD #1 (C-1) CONTAINED STAINLESS STEEL PINS ONLY
2) OUTER BLANKET COMPOSED OF STAINLESS STEEL BLANKET IN ROW 8 DEPLETED URANIUM IN REMAINDER

Fig. II.A.7. EBR-II Experimental Loading Run 25 and SNARG Representation

A fuel-irradiation period simulating a core-average 0.7 heavy atom percent burnup was computed using the burnup cycle code PYRO and the two-dimensional SNARG x,y representation of Run 25D. The computed reactivity decrease of $0.005 \Delta k/k_{\text{eff}}$ agrees with the reactivity loss usually experienced in an actual EBR-II run.

A series of test- and driver-fuel irradiation and replacement steps are being planned for this configuration. The reactivity worth of subassemblies, the fission density distribution, and the control-rod worths will be compared with the experimental results obtained in Run 25. The burnup effect on the reactivity worths of subassemblies, particularly control-rod worths, will be studied by the PYRO Code.

c. Evaluation of Cross-section Sets. A value of 1.06 for the reactivity of EBR-II Run 25 was calculated by the two-dimensional transport code SNARG in an r,z geometry, using the six-energy group cross-section set 23606. The actual EBR-II loading Run 25 had a k_{eff} of 1.005.

Some of the discrepancy was thought to be due to the coarseness of the group structure and to transport effect. To investigate these two possible sources of the discrepancy, EBR-II was represented as a three-region sphere whose dimensions and compositions are given in Table II.A.8. The reactivities of the systems were computed for several orders of angular dependence with the six-group and with the 22-group sets in diffusion and S_2 approximations. The results are listed in Table II.A.9.

TABLE II.A.8. Composition of Simplified Model of EBR-II in Three-region Spherical Geometry

Material	Concentration (atom/cm ³ x 10 ⁻²⁴)		
	25.2-cm-radius Core	10.0-cm-thick Reflector	35-cm-thick Blanket
U ²³⁵	6.3 x 10 ⁻³	-	6.0 x 10 ⁻⁵
U ²³⁸	6.0 x 10 ⁻³	-	2.9 x 10 ⁻²
Pu ²³⁹	1.0 x 10 ⁻⁵	-	3.0 x 10 ⁻⁵
Fe	1.2 x 10 ⁻²	4.5 x 10 ⁻²	1.2 x 10 ⁻²
Ni	1.5 x 10 ⁻³	6.1 x 10 ⁻³	1.5 x 10 ⁻³
Cr	3.0 x 10 ⁻³	1.4 x 10 ⁻²	3.0 x 10 ⁻³
Na	1.1 x 10 ⁻²	4.0 x 10 ⁻³	4.0 x 10 ⁻³
Fission Products	1.0 x 10 ⁻⁴	-	-
Mo	7.0 x 10 ⁻⁴	-	-
Nb	3.0 x 10 ⁻⁶	-	-
Zr	6.0 x 10 ⁻⁴	-	-

TABLE II.A.9. Reactivities of Simplified Model of EBR-II
Using Different Types of Neutronic Codes
and Energy Groups

Type	Six Groups	Twenty-two Groups
Diffusion	1.00924	1.00518
S ₂	1.06287	1.058
S ₄	1.04021	
S ₈	1.03803	
S ₁₆	1.03711	

It appears that the use of the six-group set results in a 0.4% higher value of k_{eff} than that obtained from the 22-group set in both the diffusion and S₂ approximations. The difference is not large and therefore indicates that the six-group structure is a good representation of the 22-group structured set.

In part, the discrepancy is accounted for by the higher-order transport approximation as is seen in Table II.A.9. Making corrections for the coarseness of the group structure and the order of the transport approximation yields a value of 1.030 for the calculated reactivity of EBR-II.

In the attempt to improve the r,z two-dimensional representation of EBR-II loadings, a new cross-section set is being generated specifically for EBR-II core loadings more representative of Run 25 and future loadings.

Another source of discrepancy being considered in the study is the effect of the homogenization of a hexagonally geometric, nonuniform fuel distribution into a series of concentric right circular annuli.

7. Reactor System Testing, Surveillance, and Evaluation

a. Fuel-handling System. The replacement core gripper was used satisfactorily for fuel handling. Only minor adjustments were required to improve rod indication to the console operator. Some problems were encountered with the core holddown and the unit was partially disassembled for maintenance. Most of the problems were caused by a loose electrical connection on the push force calibrator system and an improperly adjusted position switch. Plans are being made for further maintenance and complete realignment of the holddown drive assembly when time permits.

b. Startup Feedwater Pump. Bids were received and evaluated for a new startup feedwater pump. An order was placed for a single-stage, high-speed centrifugal pump.

c. Repair of Mark-II Oscillator-rod Drive. The assembly of the replacement oscillator drive shaft is complete. The final shaft-seal weld has been made and stress relieved, and the final helium leak testing was satisfactory. During assembly and checkout of the oscillator gripper, the gripper jaws did not close properly on the oscillator rod upper adapter. The gripper was disassembled and one surface on each jaw was remachined. The jaws were then sent out for chrome plating. Problems were encountered with the chrome plating, and it was necessary to replate the jaws twice before satisfactory plating was achieved. The jaws were heat treated, and final gripper assembly and checkout were completed.

The Mark-II, Type-II oscillator rod with single tube of B₄C was modified to increase the flow through the annulus between the rod and control-rod thimble. The total flow has been increased from 18.6 to 24.1 gpm (calculated values) in an attempt to avoid the rubbing (due to bowing) encountered with the first oscillator rod.

A flow test of the modified rod in the water loop showed a flow of 26 gpm at 39 psid.

d. Primary-tank Deflection. Deflection readings are taken of the primary-tank cover whenever there is a cooldown and a heatup of the bulk sodium in the primary tank to determine whether the primary tank has returned to its original position after the operation. Deflection of the primary tank is caused by the vertical temperature difference across the cover. The heating and cooling of the bulk sodium also causes the primary tank to expand and contract, which results in a relative movement between the primary-tank hangers and the T-1 steel structure, i.e., the structure on which the primary tank is hung. The movement of primary tank and the tank cover affects the alignment of the transfer arm with respect to the core gripper, the storage basket, and the transfer port.

All primary-tank deflection data are being collected and organized. Analysis and comparison of these data is to begin next month.

8. High Power Operation

No progress was made on the proposal for 50-MWt operation, because personnel were employed full time on the analysis and investigation of the power-coefficient anomaly.

The proposal to install prototype reorificed subassemblies under thermocouple positions in Rows 4, 5, and 6 has been reviewed and approved by the Irradiations Review Committee. Testing of these prototype subassemblies is a prerequisite to a proposed modification of the flow orifices for Rows 4, 5, and 6.

9. Fuel Swelling and Driver Surveillance

a. Anomalous Swelling of EBR-II, Mark-IA Fuel. The total problem has been split into 11 subproblems and will be reported in this format (see Progress Report for March 1967, ANL-7317, pp. 8-12).

(i) Irradiation of MC-S-type Fuel. There was no increment in burnup this month because the reactor was not operating.

(ii) Encapsulated Irradiations of SL-type Fuel. Same comment as for Item (i) above.

(iii) Effect of Design Change. Same comment as for Item (i) above.

(iv) Effect of Enrichment. Same comment as for Item (i) above.

(v) Effect of Position and Orientation with Casting Bundle. Same comment as for Item (i) above.

(vi) Effect of Trace-element Impurities

Subassembly C-2077. Same comment as for Item (i) above.

FCF-produced Melts. The available analytical results for the five elements produced at Idaho in the FCF are given in Table II.A.10. There appears to be some difficulty in getting the silicon content high enough, and the carbon tends to exceed the specifications (see Progress Report for August 1967, ANL-7371, p. 21, Table V). The high-carbon content of the cast ingots may be due to surface contamination of the samples used for the analysis. Only iron analyses are available for EUF-63S, and no analyses are available on pins from Casting Batch 0448 cast from this ingot.

TABLE II.A.10. Alloy and Trace-element Analyses for FCF-produced Melts

Melt No.	Ingot	Alloying Elements (w/o)						Trace Elements (wt ppm)			
		Mo	Ru	Rh	Pd	Zr	Nb	C	Si	Al	Fe
I	EUF-63S										123
II	EUF-58S	2.49	1.91	0.363	0.16	0.131	0.016		140	75	189
III	EUF-42S	2.43	1.89		0.18	0.08	0.023			209	591
III	EUF-42SR	2.43	1.92	0.281	0.18	0.08	0.078	415	120	126	750
IV	EUF-46S	2.48	2.05	0.302	0.19	0.08	0.020			787	153
IV	EUF-46SR							380			
V	EUF-59S	2.34	1.92	0.289	0.19	0.059	0.025		160	262	123

Subassembly loading patterns for these melts have been established: S-1, 2, and 3 will contain pins from all five melts and will be irradiated in Row 6. S-4 will contain only pins cast from alloys with deliberate additions of presumably beneficial impurity additions. This

subassembly will be irradiated in Rows 1, 2, or 3. Since it contains none of the low-impurity Melt No. I, the possibility exists of carrying this subassembly to somewhat higher than normal burnup.

(vii) Effect of Pressure. Capsule P-1, containing the first six pressurized specimens, was removed from the CP-5 reactor and is cooling prior to decanning. Preliminary neutron radiographs of the capsule indicated that the fuel pins had swelled significantly within their jackets.

Postirradiation facilities are in readiness to determine residual gas pressures within specimen jackets, dimensional changes, and other measurements.

Status of the pressure specimens is given in Table II.A.11. In the selection of the dates for removal of the Capsules P-2 and P-3 from the reactor, cognizance was taken of the scheduled shutdown of CP-5 from September 17 to October 23, 1967, for insertion of beam-hole experiments. Capsules P-2 and P-3 will be neutron-radiographed during this period.

TABLE II.A.11. Status of Pressurized Specimens of U-5 w/o Fs Alloy, Melt No. 409, under Irradiation in CP-5

Capsule Number	Specimen Number	Cast Pin and Section No.	Date Inserted in Reactor	Date of Removal from Reactor	Nominal Temp (°C)	Nominal Pressure (psig)	Approx Burnup on 9/14/67 (a/o)
P-1	1	53-5	5/22/67	9/14/67	570	40	1.5
	2	-11	5/22/67	9/14/67	570	80	1.5
	3	-1	5/22/67	9/14/67	570	200	1.5
	4	-7	5/22/67	9/14/67	570	400	1.5
	5	-3	5/22/67	9/14/67	570	800	1.5
	6	-9	5/22/67	9/14/67	570	1600	1.5
P-2	1	53-4	7/21/67	12/67	590	2050	0.8
	2	-10	7/21/67	12/67	590	940	0.8
	3	-6	7/21/67	12/67	590	510	0.8
	4	-12 ^a	7/21/67	12/67	590	160	0.8
	5	-2	7/21/67	12/67	590	100	0.8
	6	-8	7/21/67	12/67	590	45	0.8
P-3	1	54-1	8/17/67	1/68	620	1950	0.4
	2	-2	8/17/67	1/68	620	980	0.4
	3	-7	8/17/67	1/68	620	490	0.4
	4	-4	8/17/67	1/68	620	240	0.4
	5	-5	8/17/67	1/68	620	100	0.4
	6	-6	8/17/67	1/68	620	50	0.4

^aShort specimen.

(viii) Effect of Heat Treatment. Six pins from each of the injection-casting batches made from Ingots EUF-58S and EUF-59S (Melts II and V) will be returned to Argonne, Illinois, for this study. Since the castings have not yet been made, no material for this study is available.

(ix) Hot-lab Examination of Irradiated Fuel

(α) Chemical Analysis. No analytical results are available yet on the new shipment of irradiated fuel elements (see Progress Report for August 1967, ANL-7371, p. 23). Additional silicon, iron, and aluminum

analyses are incorporated in Table II.A.12. Most of the new results for silicon were for SL-type material and were consistently in the range of 75 to 100 wt ppm.

TABLE II.A.12. Chemical Analyses (Argonne, Illinois, and Oak Ridge) of Irradiated Fuel Pins

Casting Batch and Pin No.		Melt Refining No.	Subassembly and Element No.		Chemical Analysis (wt ppm)			
					Carbon	Silicon	Iron	Aluminum
4001	1	22	C-184	87	107	88	470	295
4002	9	23	C-180	60	132	94		
4002	14	23	C-180	57	121	88		
4002	24	23	C-184	32	139	57	190	170
4002	82	23	C-184	60	146	89	155	80
4003	3	24 ^a	C-186	53	200	162	425	110
4003	55	24 ^a	C-186	52	199	165		
4003	19	24 ^a	C-186	46	198	290		
4003	48	24 ^a	C-184	57	192	217	520	195
4003	71	24 ^a	C-180	32	193	203		
4003	86	24 ^a	C-179	82	214	181	420	135
4003	90	24 ^a	C-179	81	211	216	455	140
4003	92	24 ^a	C-179	79	189	226	440	190
4004	9	25	C-184	7	108	140	435	125
4008	19	30	C-196	31	137	71	160	205
4008	30	30	C-197	20		67		
4011	8	C-29 ^b	C-196	88	250	128	130	300
4011	45	C-29 ^b	C-197	45		122		
4020	92	39	C-198	9	283	138	60	150
4020	40	39	C-199	69		127		
4022	33	C-30	C-217	3	187	180	340	160
4023	56	33	C-199	26	162	132	50	170
4024	15	28	C-218	33	207	105	180	180
4024	82	28	C-218	34		100		
4025	24	C-33	C-218	1	257	207	240	105
4025	44	C-33	C-217	78	262	213	240	215
4026	21	C-32	C-217	57	224	219	430	175
4026	54	C-32	C-217	32		231	440	220
4027	16	46	C-218	64	206	170	225	210
4027	24	46	C-217	60	229	195	135	35
SL-1	23	-	C-165	9		84		
SL-2	27	-	C-165	56		84		
SL-3	8	-	C-166	20		87		
SL-4	21	-	C-166	72		81		
SL-5	-	-	Cast Pin		146	104		
SL-6	-	-	Cast Pin		141	126		
SL-12	-	-	Cast Pin			102		
SL-14	14	-	C-175	87		64		
SL-16	26	-	C-185	87		70		
SL-17	-	-	Cast Pin			54		
SL-19	14	-	C-185	48		76		
SL-20	40	-	C-185	2	149	85	150	175
SL-20	52	-	C-184	36	143	90	145	275
SL-21	20	-	C-186	60	153	87	155	200
SL-21	-	-	Cast Pin		-	70		
SL-29	51	-	C-184	43	157	102	200	225
SL-29	68	-	C-179	52	138	116	105	95
SL-30	8	-	C-184	37	217	108	255	140
SL-32	64	-	C-197	72		74		

^aMR-24 was a C-type melt.

^bMR-C-29 was a melt of casting heels only.

(x) Examination of Unirradiated Fuel Pins. Progress in this area will be reported later.

(xi) Analysis of Fuel-pin Swelling. Computer plots of the figure of merit $R = Y/(Bu)_{\max}$ (see Progress Report for July 1967, ANL-7357, pp. 35-36) versus $Bu_{\max}$ have been completed. The preliminary results included in ANL-7371, pp. 25-26, are still valid. The complete set of plots reveals nothing new. The fuel-pin temperature is now being factored into the analyses.

The new analytical results for silicon have not altered the apparent relationship between swelling and silicon content previously reported (see ANL-7371, pp. 26-27). The additional points fall in the low silicon-high swelling region.

10. Mark-II Driver Fuel Element Development

The EBR-II Mark-II driver fuel element is an advanced design that is intended to enable EBR-II to operate with higher fuel burnups than presently achievable with the Mark-I series of fuel elements (see Progress Report for August 1967, ANL-7371, pp. 27-28).

a. Element-irradiation Tests. The principal objective of this subtask is to determine the effect of variations in design parameters on fuel-element performance. The use of a total of 62 elements, individually encapsulated for irradiation on a substitution basis in a Type B-37 special irradiation subassembly in EBR-II is anticipated. Postirradiation examinations will be made at each projected burnup level of 0.5, 1, 2, 3, 4, 5, 5.5, and 6 a/o burnup. Both 80 and 93% enriched fuel will be used so that simulation can be obtained of reactor operation at 50 and 62.5 MW. The irradiations will be used to evaluate the relative performance of the reference 24-in.-long element compared to an element of 26-in. length. Two different gas plenum volumes will thereby be tested. The irradiations will also be used to determine the relative performance of three candidate axial fuel restrainer designs.

It is often desirable, after irradiation of a capsule in EBR-II, to remove and inspect it, and then return it for further irradiation. It is essential that the integrity of the sodium bond must not be compromised by the handling and cool-down encountered. With the intention of obtaining more information as to what happens to the sodium bond in such a cycle, since no bond-inspection equipment or procedures for irradiated capsules are yet available, four encapsulated Mark-II fuel elements were given seven heating and cooling cycles while maintained in the vertical state to determine the integrity of the sodium bond between the capsule inner wall and the fuel-element outer wall. Cooling was from the top down in order to cause purposely entrapment of shrinkage voids in the sodium. Radiographs

were taken when the sodium was molten at 265°C and when solidified at room temperature. Four exposures of each condition were made at 35° of rotation intervals.

The first two cycles were carried out with the capsules being heated in the furnace for 15 min. The radiographs of the first molten sodium conditions showed a good bond free of voids. However, the radiographs of the second molten sodium condition exhibited voids in the annulus. A review of the heating procedure indicated the possibility that the time allowed for the capsule to be in the furnace, 15 min, may not have been sufficient to permit complete melting of the sodium. Therefore, the holding time was increased to 45 min. Five additional heating cycles were imposed on the four capsules; radiographs taken when the sodium was molten showed no voids to be present in the sodium bond.

Radiographs taken after the sodium had solidified by cooling from the top down after each heating cycle showed a large number of shrinkage voids. The results of this series of heating and cooling cycles indicate that once a bond is established and once the metal surfaces are wetted, the bond free from voids can readily be re-established even when the sodium, after solidifying, has a relatively large number of shrinkage voids. The results also indicate that inspection of the bond in the molten state is more appropriate since that is the condition of service.

b. Compatibility of Fuel and Jacket. The objective of this subtask is to determine the extent of fuel-jacket diffusion reactions for the times and temperatures expected in the Mark-II element.

The higher projected burnup of the Mark-II element will result in correspondingly longer times of contact between the U-5 w/o Fs fuel alloy and the Type 304 stainless steel jacket. Current compatibility data are reliable to only 1000 hr. Long-term (up to 10,000 hr) diffusion-couple compatibility experiments are being done at temperatures of 550, 600, and 650°C, which effectively bracket the maximum fuel-jacket interface temperature of 600°C calculated for EBR-II, with normal uncertainty factors.

Also being evaluated for their possible effects on solid-state reactions between fuel and jacket are intentionally added impurities of potential benefit (Al, Fe, and Si) and fission products present in irradiated fuel from EBR-II.

The temperature at which liquid-phase formation occurs between U-5 w/o Fs alloy and Type 304 stainless steel is being determined to within $\pm 5^\circ\text{C}$. A summary of the projected diffusion-couple compatibility experiments is shown in Table II.A.13.

TABLE II.A.13. Summary of U-5 w/o Fs-type 304 Stainless Steel Diffusion-couple Experiments

Specimen Number	Condition of Fuel	Added Impurities in Fuel	Condition of Jacket	Annealing		Purpose
				Temp (°C)	Time (hr)	
1	Unirradiated	None	Unirradiated	550	5,000	To determine effects of extended exposure times on U-5% Fs-304 SS compatibility.
2	Unirradiated	None	Unirradiated	550	10,000	
3	Unirradiated	None	Unirradiated	600	5,000	
4	Unirradiated	None	Unirradiated	600	10,000	
5	Unirradiated	None	Unirradiated	650	5,000	
6	Unirradiated	None	Unirradiated	650	10,000	
7	Unirradiated	Al, Fe, Si	Unirradiated	650	168	To determine effects of Al, Fe, and Si additions to U-5% Fs on compatibility with 304 SS.
8	Unirradiated	Al, Fe, Si	Unirradiated	650	1,000	
9	Unirradiated	Al, Fe, Si	Unirradiated	650	5,000	
10	Unirradiated	Al, Fe, Si	Unirradiated	650	10,000	
11	Irradiated	None	Unirradiated	650	168	To determine effects of fission products in U-5% Fs on compatibility with 304 SS.
12	Irradiated	None	Unirradiated	650	1,000	
13	Irradiated	None	Unirradiated	650	5,000	
14	Irradiated	None	Unirradiated	650	10,000	
15	Unirradiated	None	Unirradiated	710	168	To determine temperature at which liquid phases are formed in U-5% Fs-304 SS couples.
16	Unirradiated	None	Unirradiated	720	168	
17	Unirradiated	Al, Fe, Si	Unirradiated	700	168	To determine effects of Al, Si, and Fe additions to U-5% Fs on the temperature at which liquid phases form.
18	Unirradiated	Al, Fe, Si	Unirradiated	710	168	
19	Unirradiated	Al, Fe, Si	Unirradiated	720	168	
20	Unirradiated	Al, Fe, Si	Unirradiated	730	168	
21	Irradiated	None	Irradiated	700	168	To determine effects of fission products on the temperature at which liquid phases form in U-5% Fs-304 SS couples.
22	Irradiated	None	Irradiated	710	168	
23	Irradiated	None	Irradiated	720	168	
24	Irradiated	None	Irradiated	730	168	
25	Unirradiated	None	Unirradiated	550	1,000	To determine effects of moderate exposure times on 304 SS compatibility.
26	Unirradiated	None	Unirradiated	600	1,000	
27	Unirradiated	None	Unirradiated	650	1,000	

Presented in Table II.A.14 are fuel and jacket penetration depths measured for U-5 w/o Fs-type 304 stainless steel diffusion couples that were annealed for 1000 hr at 550, 600, and 650°C. Included for comparison are calculated results obtained previously from extrapolation of data for times up to 840 hr. In most cases the measured and calculated values show agreement within normal experimental error. The measured fuel penetration at 550°C is, however, significantly lower than the calculated amount. Additional annealing tests intended to clarify this finding are in progress.

TABLE II.A.14. Maximum Fuel and Jacket Penetrations (microns) for U-5 w/o Fs-type 304 Stainless Steel after 1000 hr

Specimen Number	Temp (°C)	Fuel (U-5 w/o Fs)		Jacket (Type 304 SS)	
		Measured	Calculated ^a	Measured	Calculated ^a
25	550	22	45	<3	2.3
26	600	124	120	8	5.2
27	650	340	260	12	12.5

^aCalculated from data up to 840 hr reported by C. M. Walter in ANL-6816 (March 1964).

c. Subassembly Proof Tests. The objective of this subtask is to characterize the performance of the Mark-II fuel with selected design specifications, under conditions amenable to statistical treatment, particularly in the establishment of confidence levels relating to fuel performance.

The second phase of the evaluation of the in-reactor performance of the Mark-II element consists of irradiations of unencapsulated elements in complete Mark-II 91-element subassemblies. These subassemblies will therefore function in EBR-II as driver fuel subassemblies. They will be exposed directly to sodium flow, so that any effects due to the primary coolant can be determined.

A total of five such subassemblies are scheduled for irradiation beginning in FY 1968. Their insertion will be timed so that they do not accumulate more than 0.5 a/o burnup before the encapsulated elements with 2 a/o burnup discussed in Sect. II.A.10.a above, have been removed and examined. The irradiations are scheduled to be in the range of burnup from 2 to 5 a/o.

The EBR-II Irradiation Evaluation Committee has approved in principle the irradiation of the five Mark-II subassemblies. Although final Mark-II element and subassembly drawings have been prepared under EBR-II Task 15, fabrication of components for the Mark-II elements and subassemblies is being delayed because of a request received from AEC-RDT for AEC review of the Mark-II subassembly design before fabrication of Mark-II subassemblies.

11. Fuel-related Equipment

Equipment related to fuel handling and reactor control rods is being improved to raise the fast-flux irradiation capability, reliability, and performance of EBR-II.

a. Improved Gripper and Holddown Force-limit Device. Improved force-limit devices have been designed to control remotely and monitor the forces exerted by the fuel-handling mechanisms. Such devices increase the reliability, accuracy, and sensitivity of the core gripper and holddown push and pull force-limiting devices used in fuel-handling operations. A prototype is being fabricated and will be tested.

Mechanical assembly of the test device was completed except for the drive motor, which was not suitable for vertical mounting and is being modified. The assembly of electrical controls was scheduled for mid-September, but was delayed because of manpower shortage. Components for modifying the gripper mechanism are being fabricated on schedule.

b. Control Rod of Greater Worth. An all-absorber control-rod design is being studied to replace the present fuel-type control rod so that four of the present 12 control-rod locations might be made available for the irradiation of experimental devices that require access from outside the primary tank.

Although it seems that an all-absorber-type control rod is the most feasible means of controlling the EBR-II with eight instead of twelve control rods, a modified fuel rod with an absorber follower is also being considered. Information is also being gathered on the performance of boron carbide as control-rod material in other experimental and operational reactors. When a choice is made, a sample rod will be fabricated and tested in a sodium test facility such as the Core Component Test Loop.

c. Irradiated-fuel Monitor. A scanning device employing an ionization chamber to monitor individual subassemblies is being developed and tested. It will quickly differentiate spent, refabricated, or new fuel subassemblies during fuel-handling operations within the primary tank.

The completed irradiated-fuel monitor was tested (without a radiation source) to ensure correct mechanical operation and electrical indications. The unit is being shipped to Idaho. Installation procedures have been prepared. After the plant-modification proposal has been approved, the irradiated fuel monitor will be installed in the reactor tank and tested with spent and reprocessed subassemblies.

d. Oscillator Rod Mark II. An improved rod is being developed to permit transfer-function measurements even at low rotary speeds, where

excessive thermal bowing of the rod caused the rod to rub against its thimble; other design modifications will improve handling and storage.

The detailed drawings for the Mark-IIA reduced-diameter rod, special thimble, storage basket adapter tube, and special adapter tube gripper have been checked and approved; fabrication cost is being estimated. A plant modification proposal for the new rod and associated components is being prepared. Certain long-lead-time items, such as oscillator-rod tubing, have been ordered.

Additional studies will be performed to ensure minimum bowing of the oscillator rod. Optimum distribution of the internal coolant flow will be achieved by varying the flow-hole sizes or their placement, as determined by calculations and hydraulic tests.

e. Control-rod Bushing. Examination of control rods removed from the reactor indicates corrosion or erosion of the beryllium-copper bearing surfaces. An engineering study is being conducted to determine the cause and to select a better bushing material.

Evaluations of materials and design of the control-rod lower preassembly guide bushings continues. The modified design, which simplifies the labyrinths to provide flat surfaces on the lands and grooves instead of the multiple steps of the original design, was selected for new bushing assemblies. Detailed drawings have been completed, checked, and approved.

12. Reactor and Primary Coolant System Equipment

a. Purification-cell and Sampling-cell Ventilation. The need for a permanent supply duct into the sampling cell is still under study. The existing temporary duct was set up at a time when there was insufficient cooling for instrumentation and when the exhaust system was not operating properly. New cooling ducts have been routed from the general ventilation supply to the instrument and control panels, and the exhaust system is now performing satisfactorily. To check further on cell ventilation during and following the recent startup of the purification system, the sampling-cell temperature was monitored with the doorway and temporary supply duct closed. The results of this testing during periods when a maximum heat load was being dissipated led to a conclusion that the existing temporary duct be removed and that no new permanent duct is required.

A flow of air from the sampling cell through the ceiling penetrations into the purification-system EM-pump rectifier blower has been noted. These penetrations will be plugged to prevent exhaust flow from bypassing the absolute filter in the exhaust line.

b. FERD Loop Plugging Meter and Sampler. The prefabrication of the piping for the FERD loop plugging meter and sampling system has been completed. With the exception of the instrumentation and the electrical connections, the installation of the sampler is complete. Prefabrication of the instrumentation system is complete. The instrumentation and control components for both the plugging and sampling loops have been packaged into a single control cabinet. Checkout of the cabinet has been completed.

During proof testing of the plugging-meter flow-control components in a test loop attached to the reactor secondary sodium system, a drift in flow rate was traced to the pump control (see Progress Report for July 1967, ANL-7357, p. 38). The control was improved by replacing the static power supply with a variable transformer, a stepdown transformer, and a rectifier for the FERD loop pumps. Work will continue on the static controller as time permits.

From information gained in proof testing, the heaters supplied for the test loop attached to the secondary sodium system appeared marginal. This deficiency was corrected on the FERD loop plugging meter and sampling system by using tubular or strip heaters and by installing a spare set of heaters in parallel. Both sets of heater leads have been routed to a junction box external to the shielding and connected in parallel. The heaters have been divided into groups for sequential heating of the piping sections. Sequential heating of frozen-sodium piping, which eliminates local expansion problems, should prevent damage to the valve bellows and economizer.

Because both the plugging and sample loops are covered by a lead shield in a limited access area, the temperature monitors for the piping sections are installed in pairs. Each isolation valve has been equipped with a detector for bellows leakage. Pans equipped with sodium-leak detectors will be installed under each pump and valve, for a total of eight leak detectors, for the two loops. A sodium leak or a high pipe temperature will cause a local alarm and annunciate in the main control room as an abnormal operating condition.

Heaters and thermocouples have been installed on both loops and the leak detector probes have been built but not installed.

c. New Plugging Meter for the Primary and Secondary Purification Systems. Installation drawings for the new improved plugging meters for the primary and secondary purification systems are being prepared. Some of the components for the systems have been procured.

d. Blanket-gas Monitor. An additional monitor system consisting of three single-channel gamma analyzers discriminated for the isotopes Xe^{133} , Xe^{137} , and A^{141} and a chromatograph to determine the quantities of

O₂, N₂, and H₂ in the primary-tank cover gas has been designed. The instrumentation design has been approved, and the specifications for the system are being distributed to instrument vendors with invitations to bid.

e. Surge Tank for the Primary-sodium Purification System.

A rough draft of the engineering submittal for the modification of the surge tank of the primary-sodium purification system has been completed. All engineering drawings have been finished and are being checked. The proposed change is an increase in the height of the surge tank, which should eliminate the vacuum-line sodium plugging problems encountered in the present design. Baffles will also be added to the upper part of the tank to prevent splashes of sodium from plugging outlet lines. Along with the surge-tank modification, modification of some shield blocks and construction of two new shield blocks will be required.

f. Primary Tank Pressure-Vacuum Relief. Some minor changes for this system (see Progress Report for August, ANL-7371, p. 35) have resulted from completion of the detail design. Instead of the relief device being mounted on sliding feet, it will be fixed to a support platform, and pipe expansions will be absorbed in a short length of 4-in. stainless steel flexible hose. The addition of check valves in series with the relief device has increased the differential pressure necessary for actuation of relief; thus the previously mentioned relief-device modification will not be necessary.

A rough draft of the engineering submittal for this work, which describes the modification in detail and provides installation instructions, cost estimates, etc., has been completed, and preparation of drawings for the modification is under way.

Utilization of the N-2 nozzle as a connection point for the primary tank was investigated and found to be satisfactory as the connection point for the proposed primary-tank relief system. A small argon-sample line must be relocated to make this installation.

13. New Subassemblies Design and Experimental Support

a. Mark-II Core. The design of the driver fuel is being modified to improve the attainable burnup and reliability, and to reduce costs of fabrication and assembly. Fuel-element dimensions are being changed to accommodate greater fuel growth; to keep subassembly overall dimensions the same, the upper and lower axial reflector sections must be modified. Out-of-pile and in-pile tests will verify the design.

Development of the Mark-II and Mark-IIA subassemblies continues to provide the 24-in. element length (Mark-II) and an alternate 26-in. element (Mark-IIA).

On the basis of reviewer comments on the detail drawings of the 24-in.-element subassembly, minor revisions are being made. A technical memorandum, "Design and Pressure-drop Characteristics of the EBR-II Mark-II Core Subassembly," has been prepared.

One hundred Mark-IIA (26-in.) dummy elements have been fabricated and are being used in a pressurized-water loop to determine pressure-drop and flow characteristics. Test results might lead to detail modifications on the upper and lower pieces of the Model-SCPS shield sections.

b. Irradiation Subassemblies. Prototypes of various types of special irradiation subassemblies are being designed, fabricated, and tested to provide vehicles for irradiation experiments. Each of five types is being designed in several models to accommodate experimental fuel elements or capsules of different sizes and to achieve specific experiment environments.

(i) Mark-E. The Mark-E (or "nonmelt-through") irradiation subassembly, which would be used for experiments involving risk of melting the samples, is designed to contain a partial fuel meltdown and confine all damage within the subassembly.

The Mark-E assembly drawings are being checked. Detail drawings are being modified to reflect fabrication techniques for tantalum.

14. Instrumented Subassembly System

A system is being developed for installing and removing an instrumented experiment subassembly in a control-rod location in the core by use of the normal fuel-handling equipment. The system will provide the capability for measuring operating conditions of experimental fuel and related components while they are being irradiated in the reactor core. Special tools will handle the instrument leads, which will extend out through the reactor-vessel cover and rotating shield plug to appropriate receiving instruments.

a. Design. The system and its components are being designed and evaluated, specific problems are being identified, necessary research and development is being determined, and effects of the instrumented-subassembly system on other EBR-II systems and operations are being determined.

(i) Subassembly Attachment and Lead Severance. Various concepts are being designed and evaluated for coupling and decoupling the top of the instrumented subassembly to the extension tube and for severing the instrument leads so the assembly can be removed from the core.

Lead-severance studies have continued (see Progress Report for March 1967, ANL-7317, pp. 15-19) with the purpose of simulating the lead-cutting operation which will be performed prior to removal of the extension tube from the primary tank. Cutting tests were performed on two-conductor magnesium oxide-insulated leads sheathed in Type-304 stainless steel. The leads, of 0.125-in. diameter with a 0.020-in. sheath thickness, were sheared with a tool-steel cutter (AISI-SAE Type 02) hardened to Rockwell C53. The cutting tool consists of two 180°-opposed blades on the periphery of a hollow shaft. This shaft slips over the top end fixture of the subassembly and acts as a cutting guide. The two-blade-cutter configuration was adopted to minimize the cutting forces transmitted to the subassembly through the top end fixture.

In room-temperature air, 84 sheathed leads were cut; the average torque required was 21.1 lb-ft/lead, the spread of values being from 18.7 to 26.5 lb-ft/lead. Another 130 sheathed leads were cut while they were submerged in 700°F sodium. The average torque required was 15.3 lb-ft/lead, the spread being from 11.0 to 17.9 lb-ft/lead. The decrease in torque required to cut the leads in 700°F sodium is attributed to the lower yield strength of the stainless steel sheath material at that temperature. Another tool, of AISI-SAE T1 steel, which exhibits better hot-hardness characteristics, has been ordered. Cutting tests with the new tool are expected to give even better results.

15. Packaged Loop

The overall objective of the proposed in-core packaged loop is to increase the effectiveness of the EBR-II reactor as an experimental facility. The task requires the conceptual design of a closed-loop facility to be installed in the EBR-II reactor.

A packaged loop in EBR-II will provide the capability of performing irradiation experiments and reactor safety experiments in the USAEC's Liquid Metal Fast Breeder Reactor Program which require isolation from the reactor primary coolant system. Included are experiments requiring different operating parameters than exist in EBR-II and experiments presenting some risk to the reactor. The packaged loop will permit irradiation experiments involving fuel elements and structural materials which require individual control of environmental parameters such as temperature, power density, flow, and coolant composition. Experiments could be conducted under instrumented conditions to provide a maximum of information which could be used to evaluate or verify the operating limits for advanced fast reactor fuels.

Included among the experiments which could be conducted in a packaged loop are fuel-irradiation experiments, vented-fuel experiments, fuel-failure experiments, irradiation of structural materials, burnup- and temperature-limit experiments, sodium-technology experiments, and in-core instrumentation testing.

The scope of this program and the criteria for the design of this facility will be based on considerations such as compatibility with the EBR-II reactor, the limits of present technology, and the USAEC's Liquid Metal Fast Breeder Reactor Program. To establish the design criteria for this facility (or facilities) further examination of the experimental requirements of such a facility will be necessary.

As a part of the overall feasibility study for the packaged loop, the maximum amount of heat that can be rejected from the loop to the EBR-II primary sodium coolant utilizing heat-exchanger configurations which conform to the necessarily rigid space requirements is being determined. This heat must be transferred to the stagnant bulk sodium above the reactor vessel cover. Heat-exchanger designs which use an outer cylinder to produce a chimney effect and those which employ fins are being studied. As a necessary part of the heat-transfer study, a preliminary flow analysis has been completed. A stress analysis is in progress to determine required pressure-tube wall thicknesses and the minimum wall thickness for optimum heat transfer.

16. Process Chemistry

a. Quality Monitoring and Control of Sodium Coolant

(i) Sampling of Primary Sodium. Samples of primary sodium were taken in a variety of containers. With the exception of the Pyrex beaker and stainless steel cup samples, all were "flow through" samples. Cesium segregation was especially studied.

The plugging valve failed in the PI-A plugging loop and the sampling station was secured for repairs.

(ii) Primary Argon. There were five periods of higher-than-normal hydrogen content in the primary cover gas. Each was in conjunction with either a cleanup of the Fuel Transfer Port or with fuel handling. Table II.A.15 summarizes the data taken from the recorder on the continuous chromatograph. Table II.A.16 shows the maximum hydrogen contents observed in the five periods.

TABLE II.A.15. Primary Cover Gas Impurities

	High	Low	Average
ppm H ₂	112	0	5
ppm O ₂	0	0	0
ppm N ₂	14,400	4,800	8,400

TABLE II.A.16. Maximum Hydrogen Content

Date	ppm H ₂	Comment
9/21	52	Transfer Port Cleanup
9/21	96	Fuel Transfer
9/25	72	Fuel Transfer
9/25	80	Fuel Transfer
9/26	112	Fuel Transfer

(iii) Analysis for Carbon in Primary Sodium. Experiments have shown that rapid or slow cooling of samples makes little difference in the

carbon values of aliquots. However, if segregation of carbon is occurring, the results may be low.

A new furnace was purchased for the reaction section of the analytical train. This furnace was modified to take 700 W instead of the designed 422 so that a speedup in carbon analyses from one or fewer samples per 8 hours to two or more may be obtained.

(iv) Analysis for Oxygen in Primary Sodium. The glovebox for the amalgamation method of determining the oxygen content of sodium is completed and ready for use.

(v) Segregation of Radiocesium in Samples of Primary Sodium. Attempts to prevent the segregation of Cs^{137} in sodium by quick chilling the molten sample were unsuccessful.

(vi) Other Analyses of Primary Sodium. A sample taken on August 23 for copper analysis was also analyzed by gamma spectrometry for radionuclides. The results are shown in Table II.A.17.

Results of the most recent analyses for copper and uranium in primary sodium are given in Table II.A.18.

TABLE II.A.17. Radioactivity
in Primary Sodium

Nuclide	$\mu\text{Ci/g}$
Na^{22}	1.5×10^{-2}
Cs^{137}	1.7×10^{-2}
Sn^{113}	trace
$\text{In}^{113\text{m}}$	trace

TABLE II.A.18. Copper and Uranium in
Primary Sodium

Date	Element	Concentration (ppm)
7/24	Cu	0.29
8/3	Cu	0.21
8/23	Cu	0.32
8/21	Uranium	<0.005

(vii) Plugging-indicator Performance. Other facilities (ORNL, GE, AI) using sodium and NaK systems have experienced double and triple breaks in plugging-indicator runs. The additional breaks, generally at temperatures above the oxide plugging temperature, have been tentatively attributed to other precipitants. No such phenomena have been seen at EBR-II. During normal plugging runs there is a slight downward drift of flow rate from beginning to end (680 to 225°F). A test has been made, however, which shows that this drift is a function of temperature. If temperature is held constant, the flow remains constant, but if the temperature is increased the flow increases. This behavior has been tentatively attributed to the fact that the flowmeter is on the plugging-indicator side of the economizer and is at the same temperature as the plugging valve.

While the primary bulk sodium temperature was at 350°F, the plugging indicator was operated at constant temperature for about 24 hr each at 335, 300, and 250°F. Also, as the primary bulk temperature was

being increased from 600 to 700°F and for about 24 hr after reaching 700°F, the plugging indicator was operated at a constant temperature of 300°F. The flow through the plugging indicator remained constant during each test. Apparently the primary sodium is not saturated with any impurity in the temperature range 300-700°F.

Further test runs of this sort are planned when the FERD loop plugging indicator is installed and operable.

(viii) Expanded Sampling Capabilities for Primary and Secondary Sodium

(α) Secondary Oxygen Meter Loop. Installation of an economizer in the oxygen meter loop was delayed because of work of higher priority in the maintenance group. It is anticipated that the economizer installation will be completed in early October and that oxygen meter testing will start by the end of October.

(β) Sodium-sampling Devices and Hardware. A sampling equipment test loop, which will consist of an inlet and outlet header, a bypass valve between headers, and taps for four side loops, is being designed for installation in the secondary system. Present plans call for testing the vacuum distillation sampler and freeze-line samplers in this loop.

The following modifications to the primary sample station are planned:

1. relocating sample taps from the plugging-indicator lines to the main purification-system lines;
2. enlarging sample lines from 1/4-in. tubing to 1/2-in. tubing or pipe;
3. providing the capability for taking multiple samples by both the "freeze-line" and "cup-and-spigot" methods;
4. eliminating the waste vessel and returning sodium to the system from all sampling devices.

The modifications will greatly improve the capability for simultaneously taking routine samples and samples for development of sampling and analysis techniques. The modifications will be carried out coincident with replacement of the primary cold trap and modification of the primary purification system.

(ix) Secondary Sodium. No secondary sodium samples were taken because the sampling system was frozen for installation of an economizer in the oxygen analyzer loop.

(x) Secondary-system Argon. The vapor trap between the secondary sodium surge tank and the analyzer section of the continuous chromatograph froze; therefore the secondary system argon sample was taken from the storage tank for a few days. Readings on both hydrogen and nitrogen were higher than usual during this period.

(xi) Effect of Recycling Type 304L Stainless Steel Tubes. The mechanical properties of Type 304L stainless steel tubular specimens that have been exposed to sodium and stored in air three times have been measured in a program to determine whether test fuel elements can be returned to the EBR-II after being nondestructively examined. For each exposure and storage cycle, the specimens were exposed in an 1100°F sodium test loop for 7 days, after which they were cleaned and then stored in either normal air or dry air for 10 days.

Consistent with the earlier resistivity measurements, the measurements show that specimens stored in dry air between exposures to sodium are affected as regards mechanical properties more than are those that were stored in normal air. After three cycles, the ductility of specimens stored in dry air decreased by 13.5%. Ultimate strength and tensile strength were affected less, showing a slight (0.5-2.5%) decrease. Correlation of microhardness and tensile-strength data with photomicrographs gives clear evidence of adverse surface effects due to the presence of sodium.

Adverse microstructures are shown in Fig. II.A.8, which shows typical tensile specimens after rupture; notches can be seen between grains at the sodium-to-metal interface. No such notches were found in the as-received specimen which was also taken to rupture in tension.

The published* maximum allowable limit for Type 304L stainless steel is Rockwell-B 90 or Knoop 183 with a 500-g load. But, as shown in Fig. II.A.9, although the average hardness values for the surfaces of all the sodium-recycle specimens fall below that limit, some of the hardness values after a second and third sodium exposure exceed that limit.

To determine the cause of the wider scatter of microhardness values after the second and third sodium recycle, detailed microhardness traverses were made at six 0.003-in. increments through the 0.019-in. tube walls (see Fig. II.A.10). It can be seen that the hardness of the inner surfaces of the tubes is the major contribution to the data scatter in Fig. II.A.9.

After hardness was measured, the specimens were tested for sensitization by means of Strauss-reagent accelerated-corrosion tests.

*Stainless Steel Handbook, Allegheny Ludlum Steel Co., Pittsburgh, Pa., 1959, p. 2.

After three exposures to Na and intermediate storages in DRY air

After three exposures to Na and intermediate storages in NORMAL air

As-received spec men (no exposure to Na)

Average mechanical properties:

Ultimate strength	92,300 psi	92,500 psi	92,900 psi
Yield strength	32,300 psi	31,600 psi	32,500 psi
Elongation	69.6 %	71.0 %	76.2 %
Reduction of area	41.5 %	45.9 %	48.0 %

Fig. II.A.8. Average Mechanical Properties and Typical Photomicrographs of Type 304L Stainless Steel, 0.019-in.-wall Sections Near, But Not At, Tensile Fracture Areas (note "necking down"). All were etched with oxalic acid before being photomicrographed.

The microstructure of the inner surface of the tubes showed more porosity than did the center or the outer surface of the tube, indicating that the grain boundaries on the inner surface were affected more by the sodium. This finding is consistent with the greater microhardness of the inner surface.

Fig. II.A.9. Average and Range of Microhardness of Type 304L Stainless Steel Tubing. Note wide ranges for specimens exposed to sodium two and three times.

It is speculated that the inner surface is affected more by sodium because of the higher residual stresses there from the tube-manufacturing process. This would be consistent with previous findings concerning the effects of increased stress on sodium-exposed Type 304 stainless steel (see Progress Report for April 1967, ANL-7329, pp. 14-16).

These microstructural and mechanical-property observations emphasize the need to determine the mechanisms for material failure in sodium, in addition to the easier-to-measure overall mechanical properties of the material.

b. Water Treatment Monitoring and Control

(i) Power Cycle Streams. The steam system was secured during this report period. The steam drum and associated piping were drained and in dry layup for several days. The system was filled with hydrazine-treated water on September 10 and remained in hot standby for the remainder of the period.

(ii) Condenser Cooling Water. Both sections of the tower basin were drained and cleaned. While the basin was empty, an attempt to blow compressed air through the acid line inside the cooling water line was unsuccessful and the line was abandoned. A temporary plastic line was run along the ground to the tower basin, but was later taken out of service for safety reasons. Equipment is being designed and built for easier handling of acid in carboys for manual pH control. Consideration is being given to moving the bulk acid storage tank and the acid feeding equipment to the point of use at the cooling tower, thus eliminating the operating and maintenance problems associated with a long underground acid injection line.

17. Experimental Irradiation and Testing

a. Experimental Irradiation

(i) Status of Experiments in EBR-II. There has been no change since that given in Table IX of the July Progress Report, ANL-7357, p. 46.

(ii) Procurement and Inventory Control of Experimental Hardware. Thirty-eight capsules containing uranium-plutonium oxide fuel elements were received from Pacific Northwest Laboratory as part of the FFTE driver-fuel development program.

Eight carbide-fuel-bearing capsules were received from United Nuclear Corporation as part of a group of 19 capsules to be irradiated at a heat rating of about 20 kW/ft. The fuel specimens incorporate several techniques of "void deployment" to accommodate fission products.

(iii) Other Work. The EBR-II oscillator rod was flow tested after modifications to increase the flow in the annulus between the oscillator rod and the control rod thimble.

Axial temperature profiles of selected surveillance driver-fuel elements were plotted for Run 17 by use of the HECTIC-II code. These data will be used in studies of fuel swelling (see Sect. II.A.9).

A proposal was prepared for monitoring subassembly outlet temperatures with melt wires mounted in the subassembly top adapters (see Sects. II.A.5.a and II.A.17.b.ii).

b. Nondestructive Testing

(i) Neutron Radiography of Capsules from Subassembly XO11.

All of the capsules from XO11 have been neutron radiographed in the TREAT facility (see Progress Report for August 1967, ANL-7371, pp. 46-48) with the exception of a PNL uranium-cermet capsule designated 5U-14.

The radiography films indicate that the fuel elements in ANL capsules HOV-4 and HOV-10, and GE capsule F4F, have failed. The remaining fuel elements are apparently intact. Sodium levels are clearly visible in the ANL capsules except for HOV-4 and HOV-10. Attempts to locate sodium levels in the GE capsules are thus far unsuccessful.

Figures II.A.10, II.A.11, and II.A.12 show the conditions of the failed fuel elements in capsules HOV-4, HOV-10, and F4F, respectively. The capsule holder contains two capsules in each case, one containing a failed element, the other an intact element. HOV-4 was radiographed with GE capsule F4K, HOV-10 with F4E, and SOV-3 with F4F.

Additional neutron radiographs were made and special photographic techniques were used in an unsuccessful attempt to show the sodium levels in the GE capsules from XO11. The meniscus of the sodium bond in the GE capsules is in the thin annulus between the fuel element and the capsule wall, making detection difficult. On the other hand, the meniscus in the ANL and PNL capsules is above the fuel element (see Fig. 7 of ANL-7371, p. 48) and extends across the entire capsule diameter, being readily seen.

(ii) Other Nondestructive Testing. X-ray examination of the ANL M-3 series of irradiation capsules continued (see ANL-7371, p. 48). One capsule with a large number of shrinkage voids was heated to approximately 450°F and X-rayed hot. Essentially all the voids disappeared. Since the sodium level was clearly evident in these X rays, the ability to see bond voids should have been good. Additional capsules were examined hot and found acceptable.

The ANL irradiation capsules containing Mark-II driver-fuel elements were X-rayed and found to be in good condition.

A neutron radiograph of a subassembly top adapter containing the melt wire holder was made. Definition of the melt wires and followers is excellent despite the varying material thicknesses in the

Fig. II.A.10. Neutron Radiographs of Damaged Capsules from Subassembly XO11. ANL--HOV-4 and GE--F4K.

Fig. II.A.11. Neutron Radiographs of Damaged Capsules from Subassembly XO11. GE--F4E and ANL--HOV-10.

Fig. II.A.12. Neutron Radiographs of Damaged Capsules from Subassembly XO11. GE--F4F and ANL--SOV-3.

structural webs of the top adapter. This technique should permit inspection of the melt wires after irradiation without removal of the holder from the subassembly top adapter.

Approximately 70 X-rays were made of prefabricated piping assemblies for the FERD loop plugging indicator and sampling system.

Test welds for welder qualification purposes were X-rayed.

Two capsules containing 5% enriched uranium oxide in Zircaloy-2 cladding were neutron radiographed for the TREAT project. These capsules are used in metal-water-reaction studies.

(iii) Antimony-Beryllium Neutron Source. One of the antimony oxide gamma sources is being irradiated in the MTR because of the delay in EBR-II operation (see ANL-7371, p. 47).

Drawings of the foil-positioning fixture for the FCF neutron radiography facility have been completed, and are undergoing final review and checking. Because of the high-priority need for neutron radiographic examination of capsules from XO11 the provision of the TREAT facility was accelerated from prior schedules. The TREAT facility is now completed and functional although some additional neutron shielding is required. The FCF neutron radiography facility utilizing the antimony-beryllium neutron source is now tentatively scheduled for completion in January 1968. This date is largely contingent upon the completion date for the in-cell handling fixture which will be fabricated outside ANL.

c. Handling and Examination

(i) Examination of Irradiated Capsules from Subassembly XO11.

All capsules from XO11 have been radiographed at the TREAT facility (see Sect. b.i above) and returned to the storage holder in the FCF Air Cell, except the cermet-fuel containing capsule PNL 5U-14 which has remained in the FCF Air Cell.

After study of the radiograph prints, GE has requested withdrawal of capsule F4D from the XO11 group as well as F4F, which contains the failed fuel element. The ANL Metallurgy Division has requested that all the ANL capsules except HOV-4 and HOV-10 be considered for reirradiation.

A general work plan has been prepared for the activities leading to a proposal for reirradiation of selected capsules from XO11. This plan includes washing with high-purity demineralized water and air-drying in the IBC all XO11 capsules except 5P9, HOV-4, HOV-10, and F4F.

A procedure for accomplishing this has been prepared, and the job is being scheduled by FCF. The washing and air-drying operation has the concurrence of the experimenters.

After washing and air-drying, the capsules will be returned to the FCF Air Cell for storage pending approval of criticality procedures for storage in the Argon Cell, where they will remain until arrangements have been completed to reassemble the capsules into new subassembly hardware.

An important element of the reirradiation plan is the metallographic examination of stainless steel capsule-wall material from capsules PNL-5P9, PNL-5P12, and GE-F4D. Capsule 5P9 is considered to be representative of the capsule handling and treatment conditions before washing and drying. Capsules 5P12 and F4D represent the conditions after washing and drying. Capsule F4D had a higher heat flux than 5P9 and 5P12. A work plan for examination of the wall material of the capsules is being prepared, and will be forwarded to GE and PNL with a request for performance of the work as soon as possible.

Arrangements are being made for a shipping cask to transport these capsules to GE and PNL. The target date for receipt of capsules by the experimenters is October 11.

Consideration is being given to further examination of capsules HOV-4, HOV-10, and F4F containing failed elements. Using optical devices in the TAN hot cells a search will be made for cracks or other capsule wall penetrations which could have released fission products to the EBR-II primary sodium.

(ii) Leak-testing of Capsules Containing Failed Fuel Elements.

A conceptual design effort has been undertaken with the objective of providing a nondestructive method for the in-cell leak testing of experimental capsules. One method being considered involves placing the capsule in a sealed furnace where the capsule would be heated to a temperature near 700°F to insure that the bond sodium is molten and to induce opening of any capsule-wall defect by thermal expansion. The exterior of the capsule would then be pressurized with high-purity argon. If the capsule wall has been penetrated, it is postulated that the pressurizing gas will penetrate the opening and mix with residual radioactive gases (xenon and krypton) and the helium in the interior of the capsule. The pressurizing gas in the furnace chamber would then be rapidly expanded out into a sample chamber at a final absolute pressure somewhat less than atmospheric. The expanded gas could be sampled and analyzed for xenon and krypton by the gamma spectrometer and for helium by a mass spectrometer.

(iii) Testing of Subassembly Sodium Removal Procedures

(α) Metallographic Examination of Capsule Wall Material (Type 304 Stainless Steel) from Capsules Irradiated in Subassembly XG06.

GE has performed some work on their examination of capsule-wall material from subassembly XG06. Samples of material from the F2U capsule wall subjected to a Strauss reagent test disintegrated in 12 hr. Other samples from the same tube, limited to one hour of Strauss reagent exposure, will be subjected to metallography to determine the nature and extent of the attack. According to current schedules, however, a delay of about two months caused by work backlog will occur before these and other GE samples can be processed. GE will attempt to expedite this work to support the proposal for further irradiation of XO11 capsules.

(β) Cyclical Testing of Types 304L and 304 Stainless Steel Tubing Exposed at 1100°F, Washed in Water, and Stored in Air. With completion of the first three phases of this program, Phase IV, involving storage periods of 120 days instead of the 10 days used for the first three phases, is in progress.

The data of the first three phases indicate that only slight changes of corrosion resistance occur in stainless steel Type 304L after the first sodium exposure, wash, and air-storage cycle as measured by resistivity and Strauss reagent tests. Effects appear to be accumulative for each cycle, however, and changes in corrosion resistance after three cycles are more pronounced. Limited data from tensile testing of samples after each phase indicate only slight changes in ultimate and yield strengths when compared to the as-received material. Samples of the sodium used for sample exposure are presently being analyzed.

(γ) Cyclical Testing of Stressed Capsules (Type 304 Stainless Steel) at EBR-II. Subassembly X900 was removed from the storage basket and washed. The first set of samples included one sample with no stress, three samples with 730 psi internal pressure, and three samples with 1500 psi internal pressure. After removal of the first seven stressed tubing samples, the subassembly was returned to the storage basket for further sodium exposure. The first sodium soaking period was extended beyond the 7 to 10 days of specified exposure because the primary-tank temperature was decreased while the subassembly was in the basket; however, soaking at 700°F was held to 10 days.

Apparatus for measuring the pressure in the sample capsule after removal from the test subassembly was built and is being prepared for use in depressurizing the capsules for metallographic examination.

(δ) EBR-II Subassembly Recycle Test. A sodium-exposure and water-wash recycle test of a subassembly containing driver fuel elements with 1.2 a/o burnup is planned after removal of a subassembly from the reactor. After five cycles of exposure to primary sodium followed by washing and drying, the cladding from several pins will be examined by metallographic techniques.

(iv) Demonstration of Integrity of Sodium Bond. The ANL Metallurgy Division has completed the requested demonstration program on capsule sodium bond integrity following heating and freezing cycles. The capsule sodium was frozen in such a manner as to cause the greatest number and size of shrinkage voids in the bond sodium. Following each of five cycles on four typical capsules, radiographs showed that shrinkage voids were present in the frozen state and that sodium bonds remained free of bubbles when returned to the molten state. There appeared to be no deterioration of the bond condition from phase changes in the sodium even under more severe freezing conditions than expected during interim inspection of experimental capsules.

18. FCF Process Analysis and Testing

a. Operational Analysis. The formats for the FCF operational data system were established, and machine programming has started. Current operational data will be fed into this program on a daily basis. In addition, pertinent data from previous runs will be extracted from run logs and fed into the program file system, consisting of a number of magnetic tapes, each of which will contain the data from a specific process operation, i.e., melt refining, leak detection, etc.

The operational data system will be used in a number of ways to increase the overall efficiency of FCF operations. For example, the required quantity of re-enriching uranium can be calculated for a melt-refining run which is to be made from fuels of a wide variety of burnup values. The composition (trace element concentration) of an injection-casting batch made with recycle material can be calculated. The effect of changes in various parameters on the efficiency of a step in the process can be investigated. The results of postirradiation examination can be correlated with preirradiation and processing variables. The use of this system will provide quick data retrieval and a simplified means of comparison of parameters.

The system will also provide summaries of all operational data, both on an overall basis and on an individual component basis.

b. Test and Analytical Methods. Calibration of the recently acquired atomic-absorption instrument and study of the conditions required for analyses of "cold" fuel constituents is under way. Calibration

curves for standard solutions of Mo, Ru, Rh, Pd, Fe, and Al have been prepared, and identification of the optimum conditions for the determination of these elements in fission samples is progressing satisfactorily. Preliminary indications are that Mo, Ru, Rh, Pd, and Fe can be determined for fission samples without prior separation. The determination of aluminum will probably require a simple extraction to concentrate the aluminum to the proper sensitivity range.

Precision and accuracy of analyses by atomic absorption should be equal to or better than present wet chemical methods and should effect a considerable savings in analysis time.

19. FCF Equipment Improvement

a. Auxiliary Equipment. A request has been submitted to purchase a prototype screening device and a gravity table for separation of uranium from Vycor glass scrap. These units will be used to separate the glass and uranium after grinding with a ball mill previously tested.

A prototype design has been completed for the in-cell filters of the argon circulation system (see Progress Report for August 1967, ANL-7371, pp. 56-58). This unit would be checked for remote operability in the mockup area and then installed on one of the Argon Cell fan systems for in-cell checking.

A request for funds to construct the interim subassembly-storage facility has been submitted to the AEC for approval.

The bottom-loading and -unloading transfer cask which is being designed will utilize an in-cask subassembly grapple system. Design drawings have been completed for one grapple system. A model of this system will be constructed and tested.

b. Modification for FCF Production Increases. Two recently purchased fuel-element bonding machines have been checked out, installed in the FCF Air Cell, and are performing satisfactorily.

Purchase orders for components of the pneumatic tube transfer system have been let. Delivery of all components is to be completed before December 31. Drawings and specifications for installation have been completed, and the installation contract is expected to be let within the next month. Project completion is scheduled for the second half of FY 1968.

Drawings for the neutron radiography components have been approved and submitted to the ANL shop for comments and estimates. After some minor revisions, the drawings will be submitted to interested

vendors for bids. The present estimate indicates six weeks to obtain bids and select a fabricator, and three to four months for fabrication time. Installation in the Argon Cell is forecast for late FY 1968.

20. Hot Fuel Examination Facility

On September 12, 1967, proposals were received from nine of the eleven architect-engineer firms to which the project prospectus was sent. After detailed review by the selection board, three of the nine firms were selected to be visited to obtain further information for qualification purposes. The architect-engineer firm is expected to be selected by October 2, 1967, and to be in active participation in the project by October 16, 1967.

The prospectus is being revised to conform with budgetary guidelines furnished by DRDT. This revision reduces the overall size of the facility. Design work is being undertaken to retain as much as possible of the proposed fuel examination capacity in the revised prospectus. Four engineers from the Chemical Engineering Division, who have had experience in the FCF design, were assigned to work with the project for the next few weeks to aid in establishment of the revised concept and prospectus. The F. C. Torkelson Co. continued with the estimation of unit costs and with the initial investigations of techniques related to particular construction efforts, such as installation and leak-checking of cell liners. Torkelson will proceed with material estimates as soon as a new conceptual plant layout has been established. (The new layout will be "frozen" during the week of October 2, 1967.)

Except for designers and draftsmen, the project is now staffed to the levels required for Title I and II efforts.

Design and operating philosophies for the in-cell gas-handling system are being evaluated based on an in-cell N_2 atmosphere with 50 ppm max of H_2O , 50 ppm max of O_2 , and an in-cell ambient temperature of $80^\circ F$. Cooling loads and circulation rates have been determined for two cell configurations, and preliminary flow diagrams have been prepared for the cooling and gas-purification systems. Oxygen purification-time curves for two different purification rates were prepared. Direct expansion Freon cooling of the cell gas and cooling with an intermediate heat-transfer fluid were also investigated. Cost estimates of commercial equipment for both the cooling and gas-purification systems was obtained.

The sealed mechanical master-slave manipulator requirements have been tentatively scoped for the HFEF. Cost estimates have been received from Central Research Laboratories, Inc.

Two concept layouts were made for shielding windows to fit into the 4.5-ft-thick and 3-ft-thick shielding walls considered for HFEF. These

windows utilize a solid glass window assembly, a seal plate, and a 6-in. (unsealed) glass slab located on the inside of the cell. This glass slab can be remotely replaced if damaged, provides flexibility in using the design in areas where less shielding is required, and provides shielding for the gaskets used on the seal plate. Corning Glass Works supplied cost estimates for these windows.

Containment boxes are being considered for use in HFEF. Concept layouts have been made of two generally different types of boxes: open-top and low-leak-rate. The open-top box, relying mainly on a downward flow of cell gas for containment, is considered for use in limiting the spread of radioactive contamination within the cell. The low-leak-rate box is considered for use in performing experiments in a high-purity atmosphere other than that of the cell atmosphere. The low-leak-rate box might also be used to prevent liquids used in polishing and etching operations from contaminating the cell atmosphere. The problems with sealing, manipulator repair, equipment repair, transfer, in-cell handling, hookup, etc., associated with these boxes are being studied.

21. Nuclear Instrument Test Facility Study

The design of a detector carrier for measuring the neutron and gamma flux distribution in the "O" thimble was completed and a prototype unit fabricated. The gamma chambers for flux mapping have been ordered, with delivery scheduled for November 1967.

The design work on a circuit to isolate the existing thimble temperature trips in the reactor shutdown circuit has been completed and issued for safety approval. A study of the effect of these changes on the reactor shutdown circuit has been completed and is ready for review.

B. Physics Development

1. ZPR-3

a. Assembly 50. Work continued with Assembly 50, the third in the series of simple plutonium-fueled assemblies being studied with ZPR-3. The core of Assembly 50 differs from that of Assembly 48 in that the sodium has been replaced by graphite, giving it the softest neutron spectrum of the three assemblies studied.

This month the major ZPR-3 work was a series of Doppler-effect measurements. In addition, standard fission ratios were measured and a short period was used to intercompare some fission foils used in ZPR-3 with foils used in the ZEBRA fast critical facility at Winfrith Heath in England. Finally, detailed foil-activation measurements of heterogeneity effects within a ZPR-3 cell were made.

(i) Doppler-effect Measurements. Doppler-effect measurements in Assembly 50 were performed for two specific purposes: (1) to compare the two types of samples used in ZPR-3, and (2) to determine the effects of different environments around the same Doppler sample.

For the Doppler experiments, a 1.25-in.-square penetration was built into the core along the axial center line. The 0.5-in.-dia Doppler samples were loaded into a Doppler "rod" which was used to oscillate the Doppler sample "in" and "out" of the core in a standard oscillation experiment. Reactivity differences were measured with a very precise auto rod. A reference or "cold sample" measurement was made at room temperature, and the sample heated in three or more steps to an upper temperature of 1100°K. The intermediate temperatures were used to provide information on the temperature law for reactivity effects. Since the ZPR-3 Doppler samples are all freely expanding, the reactivity differences measured are the sum of the Doppler effect and all reactivity effects due to physical expansion of the sample.

The ZPR-3 Doppler elements are constructed with oxide pellets of about 70% theoretical density, assembled to form individual elements which are 0.5 in. in diameter by 6 in. long. Each element contains about 130 g of oxide. The elements are used to form two types of Doppler samples. The first consists of a single 6-in.-long sample with the center of the sample located at the midplane of the core; one end is fixed and the other expands away from the midplane. The second type of sample consists of two elements assembled on the same axis with a gap of ~5 in. The center of the gap is placed at the midplane, and the samples are fixed so that one expands toward the midplane and the other expands away from the midplane.

Experiments with different environments were performed with a single-element natural uranium sample. In the "normal core" environment, the material immediately surrounding the penetration for the Doppler "rod" approximated the core composition. For the "buffered" environments, the penetration was surrounded with 2-in.-square by 3/8-in.-thick annuli of graphite, nickel, and depleted uranium in turn. The graphite and nickel provided scattering media to "smooth" the resonance structure of the flux passing from the core to the Doppler sample, thereby reducing the resonance interaction between core and sample. The depleted uranium buffer provided a flux with enhanced resonance structure at the Doppler sample. The total worth of the sample was measured in each environment by comparing the worth of the sample containing natural uranium oxide to a sample identical in all respects except that it contained no uranium.

A second set of experiments was performed to compare the worths of the double-element and single-element types of samples for both the natural uranium and pure plutonium compositions. These were performed in the "normal core" environment, and the total worth of each sample was measured as stated above. The comparison between the natural uranium sample which has small expansion effects and the plutonium sample which has large expansion effects should indicate the approximate magnitude of the axial expansion effects encountered with these samples.

In order to understand the results of these Doppler measurements, in particular the effect of the various "buffer" regions, much analysis is required, and this is being done. However, one preliminary result is of interest. The measurement of a single-element pure plutonium sample in a normal core environment gave a reactivity change of about -0.15 ± 0.01 lh when heated from 350 to 1100°K. It is interesting to note that when the plutonium Doppler-effect experiment has been performed in ZPR-3, the reactivity effect due to heating (Doppler plus expansion effect) has always been negative.

(ii) Measurements of Fission Ratio. Two fission-ratio measurements, $\text{Pu}^{239}/\text{U}^{235}$ and $\text{Pu}^{240}/\text{U}^{235}$, were made near the center of the core completing those to be made with Assembly 50. The counters were of the spherical thin-wall aluminum flow type with the two foils mounted in back-to-back geometry. The fission ratios can now be compared with those measured in Assemblies 48, 48B, and 49 to determine the effect of the different core loadings on the central reaction rates.

(iii) Heterogeneity Studies. Heterogeneity studies by means of irradiation of manganese, U^{238} , and U^{235} foils were completed. These foils were placed between the material plates at the front of the two central core drawers to determine the detailed in-cell flux distribution at this location. The capture-to-fission ratios of Mn to U^{235} and of U^{238} to U^{235} and the fission ratio of U^{238} to U^{235} will be determined at these positions. The first two of

these ratios are good indices of the effect of heterogeneity upon low-energy fluxes. The last ratio gives a good measure of the effect of heterogeneity upon the high-energy portion of the neutron flux. Hence, taken together, the effect of heterogeneity upon the entire neutron spectrum is studied.

(iv) Comparison of Idaho and ZEBRA Fission Foils. A comparison test has been made with threshold-fission foils obtained from the ZEBRA group at Winfrith, England, and similar ANL-ID foils. The foils compared are those used for determination of fission ratios. They are low-mass depositions on suitable backings and are used in parallel-plate ionization chambers.

The foils compared were placed back to back at the reactor interface in two matrix positions (O-14 and Q-18). Comparative counts were taken at a power level of 20 W, with sufficient accumulation time to achieve better than 0.5% statistics.

These measurements were made as part of a continuing effort to improve the accuracy of measurements and to relate measurements made in different reactors by intercomparison of detectors.

b. Assembly 49. Data from the central perturbation measurements with Assembly 49 have been evaluated. The materials measured were in the form of small cylindrical or annular samples. Two separate sample environments were used in the measurements. For one environment, the 1/2-in.-dia traverse tube was placed in the center of a 2- by 2- by 2-in. void in a relatively uniform neutron flux. For the other environment, core material was packed around the traverse tube so that fuel plates were in close proximity to the sample.

The results are given in Table II.B.1 together with the size and weight of the samples. The samples were nominally 2 in. long. The Mn, Cr, Al_2O_3 , Fe_2O_3 , and Na samples were sealed in stainless steel cans. The weights of the cans have been corrected for in the results. Analysis is continuing on the sample-size effect of some of the materials.

TABLE II.B.1. Central Perturbation Measurements with Assembly 49

Material	Size (in.)	Wt (g)	Ih/kg in.Void	Ih/kg in Core Material
B^{10}	0.040 ann #1	1.399		-5309 $\pm$ 30
	0.040 ann #3	1.4799	-5448 $\pm$ 9.5	-5258 $\pm$ 6
	0.020 ann #1	0.691		-5547 $\pm$ 7
	0.020 ann #3	0.8015	-5785 $\pm$ 39	-5494 $\pm$ 60
	0.010 ann #2	0.406		-5780 $\pm$ 19
	0.010 ann #3	0.4484	-5882 $\pm$ 18	-5728 $\pm$ 7
	0.200 cyl	1.011	-5290 $\pm$ 0.14	-5151 $\pm$ 5
	0.100 cyl	0.244	-5748 $\pm$ 11	-5598 $\pm$ 17
	0.060 cyl #1	0.088		-5914 $\pm$ 131
	0.060 cyl #2	0.0988	-5484 $\pm$ 29	-5807 $\pm$ 126
Ta	0.015 ann	9.6713	-113.48 $\pm$ 0.02	-109.28 $\pm$ 0.04
	0.010 ann	6.7011	-116.39 $\pm$ 0.06	-111.40 $\pm$ 0.67
	0.005 ann	3.301	-123.89 $\pm$ 0.15	-114.46 $\pm$ 1.63

TABLE II.B.1 (Contd.)

Material	Size (in.)	Wt (g)	Ih/kg in Void	Ih/kg in Core Material
Ta	0.200 cyl	17.13	-99.24 $\pm$ 0.66	-96.42 $\pm$ 0.16
	0.100 cyl	4.362	-105.58 $\pm$ 1.18	-104.48 $\pm$ 0.83
Pu (22% 240)	0.020 ann	9.090	+349.76 $\pm$ 0.33	+349.68 $\pm$ 0.27
	0.010 ann	4.673	+326.81 $\pm$ 0.36	+345.37 $\pm$ 0.58
	0.005 ann	2.400	+323.99 $\pm$ 3.26	+335.42 $\pm$ 2.21
	0.200 cyl	15.063	+347.42 $\pm$ 0.20	
	0.100 cyl	3.688	+336.43 $\pm$ 0.23	
	0.062 cyl	1.209	+353.20 $\pm$ 2.05	
Pu (4.5% 240)	0.020 ann	0.315	+389.24 $\pm$ 1.09	+405.16 $\pm$ 0.73
	0.010 ann	4.801	+383.30 $\pm$ 0.38	+400.68 $\pm$ 1.10
	0.005 ann	2.761	+385.10 $\pm$ 1.99	+391.78 $\pm$ 10.75
	0.200 cyl	14.965	+405.36 $\pm$ 0.71	
	0.100 cyl	3.574	+398.05 $\pm$ 0.88	
U ²³⁵	0.010 ann	7.128	+275.76 $\pm$ 0.98	+286.56 $\pm$ 0.61
	0.0052 ann	3.638	+278.14 $\pm$ 3.68	+284.83 $\pm$ 0.63
	0.0032 ann	2.327	+278.99 $\pm$ 1.42	+282.96 $\pm$ 1.23
	0.100 cyl	4.802	+275.12 $\pm$ 1.75	
	0.062 cyl #1	1.760	+275.07 $\pm$ 4.34	
U ²³⁸	0.022 ann	15.27	-17.10 $\pm$ 0.44	-17.12 $\pm$ 0.26
	0.42 cyl	85.76	-17.07 $\pm$ 0.10	-16.40 $\pm$ 0.08
	0.200 cyl	19.63	-17.37 $\pm$ 0.28	-15.92 $\pm$ 0.36
	0.100 cyl #1	4.908	-18.26 $\pm$ 0.99	
Polyethylene	0.42 cyl	4.072	+1479.08 $\pm$ 1.10	+1206.42 $\pm$ 0.82
SS 304	0.42 cyl	35.90	-13.27 $\pm$ 0.08	-15.10 $\pm$ 0.06
Al	0.42 cyl	12.34	-16.51 $\pm$ 0.96	-18.92 $\pm$ 0.31
Fe	0.42 cyl	35.50	-12.63 $\pm$ 0.05	-14.06 $\pm$ 0.16
C	0.42 cyl	6.910	-14.12 $\pm$ 0.71	-23.70 $\pm$ 0.56
Mo	0.42 cyl	46.25	-37.73 $\pm$ 0.22	-38.39 $\pm$ 0.07
Ni	0.42 cyl	40.30	-17.71 $\pm$ 0.09	-20.66 $\pm$ 0.09
Mn		15.70	-15.087 $\pm$ 0.442	-21.89 $\pm$ 0.24
Cr		15.36	-8.736 $\pm$ 0.368	-11.80 $\pm$ 0.13
Al ₂ O ₃		6.781	-15.232 $\pm$ 1.122	-18.92 $\pm$ 1.33
Fe ₂ O ₃		11.32	-12.99 $\pm$ 0.26	-16.64 $\pm$ 0.48
Na		3.003	-12.57 $\pm$ 3.75	-13.81 $\pm$ 4.14

2. ZPR-6

The study of the heterogeneity effects on the sodium-void coefficient in Assembly 5 of ZPR-6 has been concluded. In this study the sodium-void coefficient was measured in: (1) homogeneous samples; (2) heterogeneous samples loaded in the same manner as the rest of the core, i.e., plate-type loading; (3) heterogeneous samples which consisted of uranium carbide of the proper enrichment in pellet form. The pellets were placed in stainless steel tubes which were equally spaced in a square stainless steel can. In some of the cans the space between the pellet-filled tubes was filled with sodium. This last type of loading simulated a fuel and coolant arrangement which may be typical of power reactors. The analysis of these data is in progress.

These measurements concluded the experimental program planned for Assembly 5. The facility is being prepared for Assembly 6, a 3700-liter uranium oxide core.

3. ZPR-9

A series of measurements designed to study the "hot-cold" resonance interaction effects between the U^{238} Doppler samples and the U^{238} in the surrounding core material has been completed in ZPR-9 Assembly 18. Assembly 18 is zoned and is essentially identical to ZPR-9 Assembly 12. It has the same central core composition and plate configuration as was used for previous Doppler measurements in ZPR-6 Assemblies 4Z and 5, and in ZPR-9 Assemblies 11 and 12. In the measurements, a single 1-in.-dia UO_2 sample was used and its environment varied. With sodium cans in place, three plate orientations in the drawers adjacent to the sample were studied, as well as five different configurations with filter zones interposed between the sample and the core; specifically, 1/4-, 1/2-, and 1-in.-thick stainless steel and 1/2- and 1-in.-thick depleted uranium. With the sodium removed, three plate orientation configurations and the 1/2- and 1-in. stainless steel filter zone configurations were investigated. Reduction of the data is now underway.

Doppler measurements on a special high-temperature Doppler element are being made in Assembly 18. This element containing a natural UO_2 sample is designed to reach a maximum temperature of 2100°K.

The conceptual design of a Variable Temperature Rodded Zone Facility is essentially complete. The facility is designed to allow measurements on rodded assemblies at temperatures up to 550°C (800°K). The purpose is to permit experimental comparisons between room-temperature plate-type assemblies, of the kind normally used in the ZPR facilities, and rodded assemblies at elevated temperatures such as are used in power reactors.

The Rodded Zone Facility will consist of an electrically heated region built into the center of the ZPR-9 assembly. This central region will contain sealed cans (calandria), filled with sodium, penetrated by tubes into which oxide fuel rods can be inserted. Equipment will be provided for central reactivity measurements and distributed activation measurements to be made throughout the region at elevated temperatures.

4. ZPPR

Favorable weather has aided the contractor in placing fill material on the ZPPR vault workroom area and in the reactor cell roof. Placing of sand fill over the vault workroom area was started on August 29 and completed on September 7. The first lift of gravel, of 1- to 2-in. size, was

placed over the reactor cell September 25 and 26. This lift extended to the rim of the catenary cable. A 12-in. layer of No. 10 to 2-in. gravel was placed on the coarser gravel on September 28. The first 12-in. layer of 20-50 mesh sand was placed on September 29. Sampling of the gravel and sand as it was being placed indicated good cleanliness and gradation. Tests for gradation were also made by the contractor for the vault-workroom-roof sand and the two types gravel; results were received on September 21, and are within specifications.

Welding of the steel siding on the backup containment structure continued. All the filter holders for the backup containment structure were received. The frame for the access door to the backup containment area was returned to the fabricator for rework.

Work continued on placing floor tile, ductwork, and electrical components in the facility.

The blast doors were received and mounted on the door frames. Some alignment work by the contractor is required to eliminate mating gaps between the doors and the frames. All six seal doors have been checked for mechanical performance at the manufacturer's plant. Improper rubber molds have delayed completion of the seal gaskets for the seal doors. These seals were due by the end of September.

Six prospective contractors for installation of the reactor assembly machine were briefed in preparation for submittals of proposals. The installation packages have been completed. The proposals for installation of the reactor were due on October 2.

The month's-end status of outstanding reactor components is as follows:

Control Rod Drive Mounting Plates: One finished plate was received in Idaho in the middle of September. The rejected plate (No. 2) has been repoured and checked for cracks and defects. Preliminary results from Zyglo testings indicate a satisfactory plate. The plate is now being machined and annealed. It should be completed by the end of October.

Fuel Control Rod Drawers: The fuel control drawers were received, inspected, and accepted.

Boron Carbide Plate Fabrication: All 168 boron-enriched carbide plates were completed and accepted on September 14, 1967.

Alpha Air Monitor: The order has been placed for the alpha air monitor.

Drawer Conveyor: Drawings are complete and the bid package is now being assembled.

Fission Counter and Sample Changer: Work on the cleanup detail drawers is in progress.

5. ZPR Fuel and Nonfissile Materials Procurement

a. Properties of Uranium-Plutonium-base Alloys for Zero-power Experiments. In order to establish the phases present in two as-cast ZPPR fuel alloys prepared by the Plutonium Materials Fabrication Group, Debye-Scherrer X-ray diffraction patterns made with nickel-filtered Cu-K α radiation were obtained for needles of U-36 w/o Pu-2.5 w/o Mo (alloy designation R418) and U-36 w/o Pu-3.7 w/o Mo (alloy designation R420) alloy. The diffraction patterns showed that the predominant phase in both alloys is zeta uranium-plutonium along with a small amount of retained body-centered cubic (bcc) gamma. Alloy R420 had more retained gamma, as indicated by the greater intensity of the three lowest-angle bcc lines (110), (200), and (211). These observations are in agreement with the work of Mardon *et al.*,* who have shown that the amount of retained gamma in U-Pu-Mo alloys increases with the molybdenum content. No delta-U₂Mo was detected in either X-ray diffraction pattern.

Microhardness measurements were made at 13-mil increments across the width of cast plates of both compositions. Hardness values ranged from 380 to 400 DPH and correspond to the microhardness of the zeta U-Pu phase.** Based on the X-ray diffraction patterns and the microhardness data it is concluded that the cooling rate on casting the U-Pu-Mo plates is sufficiently slow to allow the high-temperature gamma phase to transform to zeta U-Pu. The amount of retained gamma is small as indicated by the weak gamma lines in the Debye-Scherrer photograms and by the negligible contraction observed during the initial heating cycle of the U-Pu-Mo alloys (see Progress Report for August 1967, ANL-7371, p. 66).

b. ZPPR Fuel Procurement

(i) Plutonium Blending. Atlantic Richfield Hanford Corp. has been given an additional order for 1200 kg of plutonium to be blended to 11.5% Pu²⁴⁰ during fiscal year 1968. Presently 100 kg of plutonium are approved for start of this work; approximately 150 kg more may be presently available. A meeting is scheduled between ANL, Richland Operations Office, and Atlantic Richfield Hanford Corp. on October 12, 1967 to discuss additional plutonium requirements.

*Mardon, P. G., Evans, J. P., Hodkin, D. J., North, J. M., and Pearce, J. H., "The Constitution and Fabrication of Uranium-Molybdenum-Plutonium Fuels," Plutonium 1960, A. E. Kay, W. B. H. Lord, and R. D. Fowler (eds.), 329-352, Cleaver-Hume Ltd., London (1961).

**Ellinger, F. H., Elliott, R. O., and Cramer, E. M., The Pu-U System, J. Nucl. Matl. 3, 233-243 (1959).

Production of ZPPR fuel elements was started at Dow during the first week of September. As of September 29, the Dow Chemical Co. has transferred 80 production melt batches to core fabrication, from which 560 core plates have been machined. Five hundred and twenty-two core plates were accepted by Dow inspection, 422 assigned to assembly, and 307 were assembled, welded, and accepted by Dow inspection. Dow has completed a sufficient number of analyses to establish the compositional precision levels for their process and have requested approval to use the secondary analysis sample levels provided by the specification. Machining rejections were the largest sources of scrap, and this has been traced to the alignment of fixtures on the milling machines. The machining line was closed down for two days on the last week of September in order to realign the fixtures and to replace the bearings on one milling machine. Test production on the realigned milling machines indicates that the machining precision was improved and the line was returned to production.

Nuclear Materials and Equipment Corp. (NUMEC) has completed the installation of equipment for fuel-element production and started manufacture of the preproduction fuel elements required by the specification. Fifteen castings have been made to date in 6-cavity, 15-in.-long, inclined book molds with varying degrees of success. A total of 80 core plates have been assembled into fuel elements of which 50 have been inspected by NUMEC for submission as their qualification-of-process preproduction fuel elements. These fuel elements were ready for shipment on September 30, 1967. An additional 50 dummy elements containing stainless steel core plates were completed, inspected, and transmitted to Argonne National Laboratory.

(ii) Evaluation of Fuel Elements. The ANL inspection, radiography, and gamma scan assay of the Dow preproduction fuel elements was completed. A sufficient number of chemical isotopic and spectrographic analyses were received to qualify the Dow Chemical Co. process and to approve start of production.

(iii) Oxide Fuel Elements. Because of close compositional control and dimensional requirements required of the oxide fuel elements, an experimental development program has been undertaken to determine the feasibility for a simplified method of manufacture of the (U,Pu) O_2 fuel element according to design and specification. An order has been placed for the developmental production of Pu-U oxide pellets with the Dow Chemical Co., Rocky Flats Plant. Jacket fabrication and loading methods are being developed at ANL. Of the 300,000 U_3O_8 plates being fabricated by NUMEC, the final quantities are being completed for shipment.

C. Component Development

1. Sodium Quality Control

Methods are being evaluated for removing oxygen, carbon, hydrogen, and gross fission products from sodium by means of cold traps, hot traps, and solution gettering. Purification capabilities are being improved to meet requirements as they become defined; removal methods are being simplified. To eliminate the need for piping systems to convey sodium from the primary tank to external cold traps, a diffusion-type cold trap that can be submerged in the large primary tank is being developed.

Work is continuing on the construction of a prototype submerged oxide collector (about 40% complete). The establishment of a technique to determine gross amounts of Na_2O in sodium is continuing (see Progress Report for August 1967, ANL-7371, p. 71).

2. Sodium Quality Measurement

Methods of measuring and controlling sodium purity are being established and improved with the ultimate objective of developing standardized techniques that can be applied in commercial LMFBR power stations. The program proceeds from development of sophisticated procedures useful in the laboratory to the evolution and verification of simpler industrial methods. As the program progresses, it also supports laboratory programs involving loop tests, sodium environmental tests, EBR-II operations, and FFTF development.

Both laboratory and in-line methods are being developed for determining the content and form of various impurities, particularly oxygen, carbon, and hydrogen.

a. In-line Techniques. In-line methods and devices for monitoring oxygen in sodium are being evaluated and compared on a specially controlled sodium analytical loop. The devices include plugging meters, absolute resistivity meters, electrochemical cells, in-line bypass-tube vacuum distillation samplers, and dip-type vacuum-distillation samplers.

The Sodium Analytical Loop has been operable for two months and has undergone thermal and hydraulic tests, and some instrumentation checkouts. Attempts to stabilize the system temperature at $600 \pm 0.04^\circ\text{F}$ were successful after the room air-conditioning system was improved and stray electrical currents in the loop were eliminated (see Progress Report for August 1967, ANL-7371, p. 70).

Resistance measurements of the resistivity meter were unsuccessful because of a loosened connection at the contact point. System

operating temperature has been reduced to 350°F and the analysis loop portion has been frozen so that the electrodes in the two United Nuclear oxygen-meter sensors can be installed and the resistivity-meter contact points can be adjusted.

During 600°F operation, 48 sodium plugging runs were made and eight dip-type and two line-type sodium samples were taken for analysis. Of these ten samples, two will be analyzed for metal elements by chemical and atomic absorption spectrophotometer methods; eight were vacuum distilled, of which four were successful. The residue was analyzed for oxygen. The results of these four analyses related to plugging temperature are:

Sample No. ^a	Oxygen Concentration (ppm)	Analysis Method ^b	Plugging Temp (°F)	Sample Size (g)
4D	10.0	AAS	354	4.8
6L	4.3	Ti	333	3.2
8D	6.1	Ti	333	3.7
10L	5.2	Ti	337	3.2

^a"D" (dip-type sample); "L" (line-type sample).

^bAAS (atomic absorption spectrophotometer); Ti (titration).

The solubility of oxygen in sodium, for the plugging temperatures indicated, is much lower than anticipated. Some of the more recent solubility data favors these results.

3. Reactor Mechanism and Instrumentation

The nature and effects of sodium attack on materials of interest for the LMFBR program and the wear properties of bearing materials in static and dynamic sodium systems are being investigated, particularly in the 700-1000°F range of EBR-II and in the 1000-1400°F range of FFTF.

a. Physicochemical Mechanics of Metals. Experiments are being conducted to determine and understand the nature of the grain-boundary-penetration-type attack observed on numerous stainless-steel specimens that have been exposed to sodium.

A test was performed in which a laser microspectrophotometer was used to penetrate the wall of a sodium exposed tube of Type 304L stainless steel (see Progress Report for July 1967, ANL-7357, p. 77). It was expected that incremental penetrations would show the depth of sodium penetration into the grain boundaries when the characteristic emission spectrum of each increment was examined for sodium.

The procedure involved loading a series of empty EBR-II fuel tubes (of 0.174-in. OD and 0.009-in. wall) with sodium and melting the sodium so it settled into the tubes. Two tubes were exposed to the laser as control samples. For these samples, the laser penetration was adjusted to penetrate 0.0005 in. at a time, starting at the outer wall and progressing toward the inner surface. Termination of the test coincided with the appearance of a hole in the wall of the tube as seen through the focusing microscope; at which time a very strong sodium emission was recorded on the photograph.

The remainder of the tubes were held in vacuo at 1200°F. At intervals of one week, two tubes were removed from the furnace, cooled to room temperature, and subjected to the same laser test. To date, tests have been carried out with tubes which had accumulated 1000 hr at 1200°F.

The results show that sodium, in the heated samples, is not encountered any sooner than in the unheated control specimens. Internal standard calibration of the spectrograph showed that detection of 2.5×10^{-8} g of sodium was possible. This quantity corresponds to 0.25% of the sodium which could be vaporized in a single laser discharge.

It is significant to note that sodium appears within the last 0.0005 in. prior to wall penetration in both heated and unheated samples. This would be encountered if sodium penetrated into microcracks in the interior surface. Such cracks can be found on surfaces of worked material.

Further tests using the same technique will be extended to tubes of Type 304 stainless steel to see if intergranular carbide precipitation bears any relationship to penetration by sodium.

b. Fission-gas Pressure Transducer Development. Commercially available pressure transducers for measuring the increase in fission-gas pressure in individual fuel pins during reactor operation are being reviewed and evaluated, and will be tested out-of-pile and in-pile. Three basic types of pressure transducers hold greatest promise for being useful in 0.25-in. and smaller diameters and in 1200°F sodium: a NaK-filled capillary system, a strain-gauge-type sensor, and a null-point system.

(i) NaK-filled Capillary System. Various versions of a bellows-operated NaK-filled pressure transmitter, consisting of a long capillary tube connected to a bellows and a strain gauge, are being constructed and tested to determine their suitability.

In an attempt to get a good NaK fill (see Progress Report for August 1967, ANL-7371, pp. 73-74), a second 1/8-in.-OD bellows transducer assembly was heated to 400°F and evacuated for 3 days, then NaK was injected at the transducer end of the hot system and allowed to flow out through

the bellows end of the hot system until several NaK volume changes had passed through; the assembly then was cooled to room temperature and the fill tubes were pinched off.

When the bellows was operated, no signal was detected, which indicates that the fill operation was a failure. The bellows volume would have to change by $7.85 \times 10^{-5} \text{ in.}^3$ to indicate full pressure (150 psig) on the transducer; the effective area of the bellows is 0.0078 in.^2 , so the bellows must move 0.010 in. if there are no voids in the capillary system. However, the bellows was depressed 0.125 in. or more, but did not give any output from the transducer, indicating that voids overrode the volume change available from the bellows. Apparently, filling a system which has a small bellows is a very critical operation. In the next assembly, all crevices or points which would be difficult to fill with NaK will be eliminated. Also, the system will be filled at 600°F in an attempt to wet the tube wall with NaK.

A crude test assembly with no weld joints was put together using 12 ft of 0.012-in.-ID capillary tubing and a 1/8-in. bellows. Mercury was forced through the system and the fill tubes pinched off. When pressure was applied at the bellows end, the transducer responded. From this we conclude that an assembly that is properly filled with NaK will work.

(ii) Null-point System. Null-point pressure indicators, modified for expected FFTF conditions, are being procured and tested in sodium; length of lead and insulator problems will be investigated.

The MSA Research Corp. unit is scheduled to be delivered soon (see Progress Reports for January 1967, ANL-7302, p. 38, and for February 1967, ANL-7308, pp. 22-23). A double-containment-type null-balance pressure transducer is also being procured.

c. In-core Flowmeter Development. So that sodium flow through individual FFTF subassemblies or process tubes can be measured, technology is being established to assure that small magnetic, eddy-current, or other types of flowmeters can be obtained for use in severe temperature and radiation environments. Performance and reliability will be demonstrated in out-of-pile and in-pile tests.

(i) Thermal and Mechanical Tests of Magnetic Materials. The effects of temperature on the magnetic and mechanical properties of various magnet configurations are being measured and evaluated; methods for "stabilizing" magnets for use in a flowmeter at 1200°F are being established.

The thermal testing of the Alnico alloys to determine the temperature dependence of the remanence of permanent magnets is continuing.

The material effects on the remanence caused by metallurgical changes in the basic internal structure of the magnet, frequently due to precipitation of the hardening phase, were determined in the following way. After the test sample was magnetized at room temperature, $\sim 25^{\circ}\text{C}$, its remanence, $B_d(25)$, was measured. The magnet was then exposed for 30 min to the temperature for which the material change was to be determined. After the sample was cooled in air to room temperature, it was remagnetized and its remanence, $B_d'''(25)$, measured again. The relative percentage material change is given as

$$\frac{B_d'''(25) - B_d(25)}{B_d(25)} \times 100.$$

This effect has generally been found to be zero or a negative value.

The irreversible changes of remanence, caused by the processes that are responsible for the hysteresis of ferromagnetic materials, were determined as follows. After the material effect at a given temperature was measured as above on the first heating cycle, the same test sample was again heated for 30 min to the same temperature. When the sample had cooled in air to room temperature, the remanence, $B_d''(25)$, was again measured. This procedure of remagnetizing and heating was repeated two more times (a total of three) to establish an average value for the irreversible effect. The relative percentage irreversible change is given as

$$\frac{B_d''(25) - B_d'''(25)}{B_d'''(25)} \times 100.$$

This change has generally been found to be negative. Although this effect is commonly called "irreversible," the remanence $B_d'''(25)$ is restored by remagnetizing the sample.

If the sample is not remagnetized after the final heating cycle, the test magnet usually becomes temperature-stabilized up to the maximum value to which it was exposed. Further changes of remanence due to temperature variations in this range are completely reversible.

Rod magnets (of 0.25-in. diameter) of Alnico-5, -6, and -8 were used for the determination of the material and irreversible changes. From each material, two test samples of each length-to-diameter (L/D) ratio were prepared; ratios of 8, 4, and 2 were selected for Alnico-5, -6, and -8, but an L/D of 1 was additionally tested for Alnico-8.

Remanence was measured by three different methods. The first method consisted of pulling a search coil off the neutral zone of each test magnet and reading the deflection of a fluxmeter; repeatability within 1% was easily achieved for most of the test samples measured. The second method used a Hall-effect probe to measure the flux near the pole of the

test magnet; a repeatability within 2% was achieved for most of the test samples measured. The last technique used during some of the tests was the magnetometer method previously described (see Progress Report for June 1967, ANL-7349, pp. 71-73); repeatability of better than 0.5% was achieved.

Temperatures of the test samples were measured using a special limits-of-error ($\pm 3/8\%$) Chromel-Alumel thermocouple attached to the magnet sample tray. The maximum temperature error for any measurement should be $\pm 5^\circ\text{F}$.

Results from only the first two measurement methods and from both test samples were used to give the average value for each effect. The material changes are given in Table II.C.1 and the irreversible changes are in Table II.C.2; these preliminary results may have to be modified after further tests.

TABLE II.C.1. Material Effects in Alnico Permanent Magnets

Material	L/D	Material Effect (% $\Delta B_d/B_d$)			
		1000°F	1100°F	1200°F	1300°F
Alnico-5	8	0	0	0	-44.1
	4	0	0	-15.8	-62.7
	2	0	0	-22.0	-71.3
Alnico-6	8	0	0	0	-20.3
	4	0	0	-7.5	-44.8
	2	0	0	-20.2	-63.6
Alnico-8	8	0	0	0	0
	4	0	0	0	-1.3
	2	0	0	0	-8.6
	1	0	0	0	-5.3

TABLE II.C.2. Irreversible Losses in Alnico Permanent Magnets

Material	L/D	Irreversible Loss (% $\Delta B_d/B_d$)			
		1000°F	1100°F	1200°F	1300°F
Alnico-5	8	-0.6	-1.4	-2.0	-2.4
	4	-2.2	-2.8	-3.3	-4.7
	2	-4.5	-5.5	-6.6	-7.0
Alnico-6	8	-1.4	-2.8	-3.3	-3.5
	4	-3.8	-3.8	-7.2	-6.4
	2	-6.2	-6.6	-9.2	-6.4
Alnico-8	8	-0.7	-1.6	-2.8	-3.8
	4	-3.2	-2.8	-2.6	-3.8
	2	-3.3	-5.4	-4.6	-6.2
	1	-2.1	-4.4	-5.4	-8.9

The ceramic furnace has been completed and tested for operation up to 1200°F. Minor difficulties encountered in the high-temperature operation of the turntable mechanism are being corrected. Reversible temperature effects on the stabilized samples will be determined.

Completion of the B-H tester assembly awaits delivery of a high-current, high-voltage diode for the capacitance-discharge energy source.

(ii) Magnetic-flowmeter Prototype. Prototype magnetic flowmeters are being designed, fabricated, and tested at sodium temperatures up to 1200°F to determine calibration sensitivity, impedance, and field-noise signal levels; temperature compensation schemes will be developed if needed. As a final proof test, it is planned to install a prototype magnetic flowmeter in EBR-II.

Check calibrations made with the preliminary flowmeter at 400 and 700°F gave the same flowmeter sensitivity as those found before the expansion bellows failed. Extreme care had been exercised during loop repair to prevent large welding currents from passing through the flowmeter body (see Progress Report for July 1967, ANL-7357, pp. 74-75).

A simulation of a large welding current condition was conducted. An end fitting was welded to a flowmeter assembly, thereby passing most of the welding current through the body; an 8% decrease in the flowmeter gap flux was noted after this welding. It is therefore quite possible with several such welding operations or more severe conditions to cause the approximately 30% decrease in the flowmeter sensitivity reported previously (see Progress Report for June 1967, ANL-7349, p. 73).

TABLE II.C.3. Temperature History of Flowmeter at the End of August 1967

Temperature (°F)	Time (hr)
200-300	11.7
300-400	162.0
400-500	270.5
500-600	68.7
600-700	362.8
700-800	201.7
800-900	18.0

The flowmeter has been successfully operated at temperatures slightly above 1000°F. The time at temperature of the flowmeter at the end of August 1967, is summarized in Table II.C.3. Only a small amount of operating time was accumulated in August due to loop repairs. Over 921 hr of operation above 400°F have been accumulated on this flowmeter.

Thermal shocks of 38°F/sec are commonly experienced by the preliminary prototype flowmeter during normal pump startup in the calibration loop. No deleterious effects from such thermal shocks have been detected. Attempts will be made to increase the thermal shock rate.

(iii) Magnet-irradiation Tests. Rod magnets of Alnico-5, -6, and -8 are being prepared for installation in EBR-II irradiation subassemblies so as to determine performance after irradiation in fluxes simulating the EBR-II environment.

Work is continuing on the experiment description and hazard analysis.

Magnet-handling procedures in the hot cell are being studied. Most of the equipment designed for remote operation is operating satisfactorily.

(iv) Eddy-current Flowmeter. As a backup effort to the permanent-magnet-type flowmeter, eddy-current flowmeters are being evaluated.

The prepared report on the evaluation of the eddy-current flowmeter completes the work planned.

d. Signal Lead Connectors for Sodium Service. Remote connectors for electrical and fluid-signal leads for in-core instrumentation are being developed. This involves reviewing connector lead-wire assembly designs and test procedures, making changes to meet FFTF design criteria, considering new connector concepts, establishing out-of-pile test programs, and studying and developing methods for terminating the sensor leads inside the reactor vessel, both above and within the sodium.

A detailed design of an FFTF prototype connector is being reviewed. For this connector some of the latest design criteria and goals are:

1. The coupling shall serve not only as an electrical and fluid connection, but also as a mechanical coupling between the upper and lower portion of the FFTF fuel channels.
2. The coupling shall be capable of remote operation outside the reactor vessel while in a fuel handling cask.
3. Leaktight isolation of the connector cavity from the coolant and cover-gas environment inside the reactor shall be provided.
4. The assembled fuel channel shall be mechanically stable under tensile, compressive, and torsional loads.
5. Twenty or more electric leads, one 3/8-in.-dia low-pressure sodium line, two capillary high-pressure gas lines, and one tube for insertion of flux wire are to be provided.
6. The largest outside diameter of the coupling device shall not exceed 6 in.

4. Fuel Handling, Vessels, and Internals

a. Core Component Test Loop. The Core Component Test Loop (CCTL) sodium facility is being prepared and modified for engineering tests of materials, instrumentation, controls, and core components for the FFTF and overall LMFBF program. To determine experimentally the adequacy of piping stress-analysis methods for large high-temperature sodium systems, of which the CCTL is an example, strain-gauge readings on the operating system will be evaluated.

To monitor stressed, strained, and overall safety of the Core Component Test Loop, high-temperature strain gauges were installed at critical points (see Progress Report for July 1967, ANL-7357, pp. 78-79). Early preliminary runs indicated substantial drifts in the gauges. To calibrate the gauges, and to differentiate between drifts and actual strains, a loading device has been constructed with which known deflections and strains can be compared to those recorded by the high-temperature gauges at room and at elevated temperatures.

Performance of the loading device has been checked against the action of dead weights. Maximum deflection of a 1/8-in.-thick test beam (on which strain gauges are to be mounted) was an average of 2.25% below that predicted by the derived beam equations.

A set of four special Baldwin-Lima-Hamilton Corp. full-bridge weldable gauges (with Type 304 stainless steel shim) was checked with the testing device under dead weights. Their averaged strain readings were 5.6% above those predicted by the formulas. A set of four standard BLH full-bridge weldable gauges (with Type 302 stainless steel shim) yielded an average strain 0.85% below that given by the formulas. Scatter of the test readings was noticeable. Thus, for the first set of gauges a maximum deviation of 3.1% from the average was observed. For the second set of gauges a maximum deviation of 8.1% was recorded.

Prior to taking readings on the strain gauges, cycling of the load at least once was found necessary. After that, repeatability of the strain readings was quite satisfactory.

It is difficult to predict the performance of all remaining gauges from the test results of only four samples. However, if the preceding test data were representative, then an error of strain reading of up to 10% from the calculated values should not be unusual.

Further investigations will consist of testing BLH rokode-bounded free-filament gauges. Similar testing will be performed later for all the above gauges at elevated temperatures.

In addition to the permanently affixed strain gauge on the loop, a second method for indicating possible incipient failure of the piping involving ultrasonic detection devices on the most suspect zones of the system, is being investigated.

The sodium contained in the storage tank used for the loop has been examined for total carbon concentration. These results are:

<u>Sample Number and Location in Tank</u>	<u>Total Carbon (ppm)</u>
I-3 (bottom)	9, 10, and 26
I-3 (top)	11, 7, 9, and 20
II-1 (center)	17 and 13
II-2 (bottom)	9 and 11
II-2 (top)	13 and 10

The startup schedule for the loop has been delayed by the late delivery of a throttle valve, now scheduled for October 15, 1967. This will delay startup until after November 15.

D. Fuel Development

1. Metallic Fuels

a. Performance of Uranium-Plutonium-Zirconium Alloys. Selected fuel elements from subassembly XA07 are being examined metallographically in detail. These elements have fuel of U-Pu-Zr alloys jacketed in Type 304 or Type 316 stainless steels, in Hastelloy-X, or Hastelloy-X with low cobalt. These fuel elements are identified in Table II.D.1.

TABLE II.D.1. Irradiation Conditions for U-Pu-Zr-alloy Fuel Elements
Irradiated in EBR-II in Iron- and Nickel-base Alloy Cladding

Specimen Number	Fuel Composition (w/o)	Jacket Alloy	Jacket OD (in.)	Jacket Thickness (in.)	Max (kW/ft)	Max Jacket Temp (°C)	Burnup	
							a/o	fiss/cc x 10 ⁻²⁰
ND28	U-15 Pu-9 Zr	304 SS	0.206	0.020	10.5	630	4.6	14.2
ND41	U-15 Pu-9 Zr	304 SS	0.206	0.020	10.1	625	4.4	13.9
ND32	U-15 Pu-9 Zr	316 SS	0.196	0.015	9.7	605	4.2	13.3
ND43	U-15 Pu-9 Zr	Hastelloy-X	0.196	0.015	10.1	615	4.4	13.7
ND25	U-15 Pu-12 Zr	304 SS	0.206	0.020	9.1	600	4.0	11.5
ND27	U-15 Pu-12 Zr	304 SS	0.206	0.020	9.3	605	4.1	11.7
ND26	U-15 Pu-12 Zr	316 SS	0.196	0.015	8.9	590	3.9	11.1
ND29	U-15 Pu-12 Zr	316 SS	0.196	0.015	9.1	595	4.0	11.6
ND30	U-15 Pu-12 Zr	316 SS	0.196	0.015	10.1	615	4.4	12.7
ND31	U-15 Pu-12 Zr	316 SS	0.196	0.015	9.7	610	4.2	12.3
ND33	U-15 Pu-12 Zr	Hastelloy-X	0.196	0.015	9.5	605	4.2	12.0
ND34	U-15 Pu-12 Zr	Hastelloy-X	0.196	0.015	9.7	610	4.2	12.2
ND35	U-15 Pu-12 Zr	Hastelloy-X	0.196	0.015	10.1	615	4.4	12.7
ND37	U-15 Pu-12 Zr	Hastelloy-X-280	0.206	0.015	10.1	610	4.4	12.2
ND39	U-15 Pu-12 Zr	Hastelloy-X-280	0.206	0.015	9.9	610	4.3	12.1
ND44	U-15 Pu-12 Zr	Hastelloy-X-280	0.206	0.015	9.7	600	4.2	11.7

Metallographic specimens, both transverse and longitudinal, have been taken from ND35 and polished. These specimens will be examined and compared with previous specimens from ND28, ND30, and ND43.

Specimens from ND43 are being autoradiographed for both alpha and gamma radiation. Replicas for electron microscopy have also been prepared.

A reaction zone has been noted previously on the inside of the Type 304 stainless steel jackets used to jacket several elements in subassembly XA07. Autoradiography and microprobe analyses showed no evidence of fuel penetration. To examine this phenomenon more closely, two specimens of jacket material were separated from the fuel (ND28) at approximately the center of the fuel length and prepared for examination outside of the caves. In addition, unirradiated samples of the jacket material were examined in the as-received condition and after sensitization by thermal treatment at 600°C for 96 hr.

Metallographic preparation consisted of vibratorily polishing the samples and viewing the structure in the as-polished as well as etched conditions. The etchant was 2.5 w/o oxalic acid used electrolytically.

Results of the metallographic examination indicate a definite structural change is present on the inner surface of the irradiated jacket. A discontinuous band was observed, varying from near zero to as thick as 5 mils. After etching, the band exhibits a dark, poorly resolved structure at 500X. The unaffected structure exhibits typical equiaxed austenitic grains with considerable carbide precipitation in the grain boundaries.

Comparison of these structures with the unirradiated as-received and sensitized structures indicates the possibility of a correlation between the band in the irradiated cladding and work hardening present in the original tubing. Prior to use, the tubing had been expanded by pulling an oversized mandrel through the bore. This resulted in a nonuniform degree of cold work imparted to the ID of the tube. The extent of the severe cold work varied from near zero to about the same depth as the band shown by the irradiated material. Sensitization of the original material resulted in excessive carbide precipitation throughout the cold-worked band and normal grain-boundary carbide precipitation throughout the unworked area.

These results confirm the earlier observations that the band observed in the irradiated material is not the result of interaction with the fuel material. The band appears to be a transformation structure, such as low-carbon martensite, occurring in the cold-worked band originally present on the tube ID.

Two fuel elements in subassembly XG05 (see Table II.D.2) remain in the EBR-II for further irradiation. No burnup was accumulated during the past month. About 70% of the target burnup of 7.5 a/o has been accumulated.

TABLE II.D.2. Status of Metal-fuel (Jacketed in V-20 w/o Ti)
Irradiations in Progress in EBR-II

Specimen Number	Design Parameters				Operating Conditions			
	Fuel Composition (w/o)	Effective Density (%)	Jacket OD (in.)	Jacket Thickness (in.)	Max (kW/ft)	Max Jacket Temp (°C)	Burnup to Date	
							a/o	fiss/cc x 10 ^{-20a}
ND24	U-15 Pu-10 Zr	63.6	0.208	0.015	10.0	535	5.4	12.4
NC17	U-15 Pu-10 Ti	66.7	0.204	0.016	10.0	540	5.6	12.9

^aBased on effective density.

Group M-3, consisting of fifteen U-15 w/o Pu-10 w/o Zr fuel elements (see Table II.D.3), is at the EBR-II and has been assigned to

subassembly XO28. Interim examinations by nondestructive techniques are planned at 5.0 and 7.5 a/o burnup. The target burnup is 10 a/o, and the maximum jacket temperature during irradiation has been calculated as 630°C. One (BF-10) of the four EBR-II Mark-IA encapsulated fuel rods which accompanies this group in the subassembly was damaged accidentally. It has been replaced by BF-12.

TABLE II.D.3. Group M-3 Specimen and Jacket Dimensions

Fuel Material: U-15 w/o Pu-10 w/o Zr
 Fuel Dimensions: 13.5 in. x 0.144-in. diameter

Capsule/Specimen Number	Jacket (0.166-in. ID)		Specimen Length (in.)	Plenum Length (in.)	Specimen Plenum Volume (cc) (RT)
	Material	Wall (in.)			
ANL-BA-01	304 SS	0.015	33.00	17.97	6.51
ANL-BA-02	304 SS	0.015	33.00	17.97	6.51
ANL-BA-03	304 SS	0.015	33.00	17.97	6.51
ANL-BA-04	304 SS	0.015	33.00	17.97	6.51
ANL-BA-05	304 SS	0.015	32.61	17.57	6.36
ANL-BB-01	304 SS	0.020	29.80	14.76	5.35
ANL-BC-01	316 SS	0.015	33.00	17.97	6.51
ANL-BC-02	316 SS	0.015	33.00	17.97	6.51
ANL-BC-03	316 SS	0.015	33.00	17.97	6.51
ANL-BD-03	V-20 Ti	0.015	24.68	9.65	3.49
ANL-BD-04	V-20 Ti	0.015	24.68	9.65	3.49
ANL-BD-05	V-20 Ti	0.015	24.68	9.65	3.49
ANL-BE-01	V-20 Ti	0.015	22.70	7.67	2.77
ANL-BE-02	V-20 Ti	0.015	22.70	7.67	2.77
ANL-BE-03	V-20 Ti	0.015	22.70	7.67	2.77

Injection casting of 40 fuel pins (Group M-4) of U-15 w/o Pu-12 w/o Zr with 93% enriched uranium has been started. The fuel column is 14.22 in. long and 0.163-in. diameter. Jackets for the fuel are 0.220-in. OD and 0.188-in. ID. Materials for the jackets are Types 304, 316, and 318 stainless steels, Inconel 625, Hastelloy-X, V-15 w/o Ti-7.5 w/o Cr, and V-15 w/o Cr-5 w/o Ti. Fabrication of the jacket components has been started. Burnups for the group are aimed at 10 a/o with linear heat ratings of up to 14 kW/ft.

2. Oxide Fuels

a. Performance of Mixed Oxides. The status of elements irradiated in subassembly XO11 is shown in Tables II.D.4 and II.D.5. The subassembly was removed from EBR-II when reactor operating tests indicated that fission products were escaping into the primary coolant. Neutron radiographs of the capsules, as-removed from the subassembly, subsequently disclosed the failure of two elements, Nos. HOV-4 and HOV-10. Indications of radial and axial swelling of the fuel were observed, and it appeared quite likely that the Hastelloy-X jacket had ruptured in both

elements. As shown in Table II.D.4, the failed elements operated at higher linear heat ratings than the other elements in the group.

TABLE II.D.4. Status of Mixed-oxide-fuel (Vibratorily Compacted into Cladding) Irradiations Removed from Subassembly XO11

Specimen Number	Design Parameters					Operating Conditions			
	Fuel Composition (w/o)	Effective Density (%)	Jacket Composition (w/o)	Jacket OD (in.)	Jacket Thickness (in.)	Max (kW/ft)	Max Jacket Temp (°C)	Burnup to Date	
								a/o	fiss/cc x 10 ^{-20a}
SOV-3	(U-20 Pu)O _{2.00}	83.1	304 SS	0.296	0.021	21.4	605	3.7	7.6
SOV-7	(U-20 Pu)O _{2.00}	85.1	304 SS	0.296	0.021	21.3	605	3.6	7.5
SOV-1	(U-20 Pu)O _{2.00}	79.9	304 SS	0.296	0.021	20.0	590	3.6	7.1
HOV-10	(U-20 Pu)O _{2.00}	84.8	Hastelloy-X	0.295	0.016	22.8	605	3.5	7.4
HOV-15	(U-20 Pu)O _{2.00}	79.8	Hastelloy-X	0.295	0.016	21.4	590	3.5	6.9
HOV-4	(U-20 Pu)O _{2.00}	80.0	Hastelloy-X	0.295	0.016	22.9	605	3.6	7.2
TVOV-1	(U-20 Pu)O _{2.00}	76.8	V-20 Ti	0.298	0.022	19.6	575	3.6	6.8

^aBased on effective density.

TABLE II.D.5. Status of Cermet-fuel (Core and Type 304 Stainless Steel Cladding Coextruded) Irradiations Removed from Subassembly XO11

Specimen Number	Design Parameters				Operating Conditions			
	Fuel Composition (w/o)	Effective Density (%)	Jacket OD (in.)	Jacket Thickness (in.)	Max (kW/ft)	Max Jacket Temp (°C)	Burnup to Date	
							a/o	fiss/cc x 10 ^{-20a}
5P-9	SS-40 PuO ₂	98.0	0.301	0.015	10.4	480	4.3	3.2
5P-12	SS-27 PuO ₂	98.7	0.294	0.015	6.7	440	4.3	2.1
5U-14	SS-27 UO ₂	97.9	0.298	0.013	5.8	430	3.2	1.7

^aBased on effective density.

b. Electron Microprobe Analysis of Irradiated UO₂-20 w/o PuO₂.

An unshielded electron microprobe analyzer was used to establish the composition of fission-product inclusions observed in a pneumatically impacted, vibratorily compacted hyperstoichiometric UO₂-20 w/o PuO₂ fuel element irradiated in EBR-II to 6.0 x 10²⁰ fissions/cc (2.9 a/o burnup). A complete description of the operating conditions for this mixed-oxide fuel element is given in the Progress Report for June 1967, ANL-7349, pp. 76-77. The specimen analyzed was core drilled from the midlength of an 11.5-in. fuel element and was reduced in thickness to 0.006 in. by grinding. Activity measured 12 in. from the polished surface was 140 mR/hr gamma and 4000 mR/hr beta-gamma.

Two distinct types of solid fission-product inclusions were observed in the mixed-oxide matrix (see Progress Report for May 1967, ANL-7342, p. 70): (a) spherical white metallic inclusions containing Mo, Ru, Tc, Rh, and Pd; (b) gray inclusions that occurred in the cooler equiaxed

grains, which contained barium, strontium, and cerium. Electron-probe microanalysis of several white inclusions showed that they contained essentially no U, Pu, Sr, Y, Zr, Nb, Te, Xe, Cs, Ba, La, Ce, Pr, Nd, or Pm. The gray phase located in the equiaxed region of the mixed oxide specimen contained no Tc, Cs, La, Pr, or Nd.

Quantitative microanalysis was performed on about 40 white inclusions to establish the radial variation of the three most abundant fission products: Mo, Ru, and Tc. In general, the ruthenium content increases on moving up the temperature gradient in the columnar grains, whereas the technetium and the molybdenum contents both decrease slightly in this direction. Fission products detected in the mixed-oxide matrix in concentrations less than 1 w/o include zirconium, lanthanum, and the rare earth elements cerium, praseodymium and neodymium. These results indicate that solid fission-product elements that form oxides of comparable or greater thermodynamic stability than the mixed U-Pu oxide (Zr, Ce, Pr, and Nd) go into solution in the matrix while elements whose oxides are less stable than the mixed U-Pu oxide (Mo, Ru, Tc, Rh, and Pd) form insoluble metallic inclusions in the matrix.

In order to establish the effect of the radial temperature gradient on the migration of plutonium, 15 radial scans were made parallel to the columnar grain boundaries during recording of the plutonium and uranium X-ray intensity. A typical trace showing the radial variation in uranium and plutonium in the columnar grains is shown in Fig. II.D.1. In general, the plutonium concentration is low at the interface between the equiaxed and columnar grains, remains constant to the center of the columnar grains, and then increases uniformly from the center of the columnar grains to the central void. As shown in Fig. II.D.1, the uranium concentration is a maximum at the interface between the equiaxed and columnar grains and decreases uniformly between the center of the columnar grains and the central void.

Fig. II.D.1

Radial Distribution of Uranium and Plutonium in Vibratorily Compacted $\text{UO}_2\text{-20 w/o PuO}_2$ Irradiated in EBR-II to 6.0×10^{20} fissions/cc (2.9 a/o burnup)

3. Carbide Fuels

a. Mixed-carbide Fuel Performance. Elements being irradiated in EBR-II are listed in Table II.D.6.

TABLE II.D.6. Status of Mixed Carbide Fuel Irradiations in Progress in EBR-II

S/A No.	Specimen Number	Design Parameters						Operating Conditions			
		Fuel Composition (w/o)	Fuel Form ^a	Effective Density (%)	Jacket Composition (w/o)	Jacket OD (in.)	Jacket Thickness (in.)	Max (kW/ft)	Max Jacket Temp (°C)	Burnup to Date	
										a/o	fiss/cc x 10 ^{-20b}
XG05	SMV-2	UC-20 PuC	VIPAC	84.8	304 SS	0.296	0.021	24.0	675	5.9	16.5
XG05	HMV-5	UC-20 PuC	VIPAC	80.0	Hastelloy-X	0.295	0.015	26.6	685	6.2	16.2
XG05	NMV-11	(U _{0.8} Pu _{0.2})C	VIPAC	82.9	Nb-1 Zr	0.281	0.012	25.5	635	6.2	16.9
XO08	NMP-2	(U _{0.8} Pu _{0.2})C	PELLET	81.6	Nb-1 Zr	0.284	0.013	17.4	545	3.7	9.9
XO08	NMV-4	UC-20 PuC	VIPAC	80.0	Nb-1 Zr	0.283	0.013	27.0	645	5.5	14.5
XO08	NMV-7	UC-20 PuC	VIPAC	80.0	Nb-1 Zr	0.283	0.013	26.0	630	5.4	14.3
XO08	NMV-12	(U _{0.8} Pu _{0.2})C	VIPAC	85.5	Nb-1 Zr	0.284	0.014	26.7	645	5.5	15.4
XO08	HMV-1	UC-20 PuC	VIPAC	80.0	Hastelloy-X	0.295	0.015	25.2	660	5.0	13.2
XO08	HMV-4	UC-20 PuC	VIPAC	80.0	Hastelloy-X	0.295	0.016	27.2	690	5.4	14.2
XO08	HWMP-1	(U _{0.8} Pu _{0.2})C	PELLET	81.4	Hastelloy-X + W	0.296	0.020	16.9	565	3.6	9.8
XO08	HWMV-1	(U _{0.8} Pu _{0.2})C	VIPAC	82.9	Hastelloy-X + W	0.295	0.018	26.4	685	5.4	14.8
XO15	NMP-1	(U _{0.8} Pu _{0.2})C	PELLET	81.6	Nb-1 Zr	0.284	0.013	16.5	535	1.1	2.9
XO15	NMV-3	UC-20 PuC	VIPAC	80.0	Nb-1 Zr	0.284	0.013	26.0	635	1.6	4.3
XO15	HMV-2	UC-20 PuC	VIPAC	80.0	Hastelloy-X	0.295	0.015	27.0	685	1.6	4.2
XO15	TVMV-1	(U _{0.8} Pu _{0.2})C	VIPAC	83.7	V-20 Ti	0.298	0.021	23.4	640	1.5	4.0

^aVIPAC = Vibratorily compacted into cladding; PELLET = Pressed and sintered pellets.

^bBased on effective density.

Three elements are undergoing postirradiation examination (see Progress Report for August 1967, ANL-7371, Table XXII, p. 76). Element No. SMP-1 containing solid-solution pellets jacketed in Type 316 stainless steel was studied metallographically. Figure II.D.2 illustrates alpha and beta-gamma autoradiographs, with a corresponding macrograph, of a pellet transverse section near the hottest location in SMP-1. The alpha autoradiograph indicates no significant relocation of plutonium. The beta-gamma autoradiograph, however, shows significant migration of fission products. Similar results were observed in the sintered central area of Element No. VMV-1 that was vibratorily compacted with solid-solution particles of mixed carbide (see the Progress Report for May 1967, ANL-7342, pp. 71-73). The etched microstructure of the pellet illustrated in Fig. II.D.2 disclosed the presence of sesquicarbide particles plus a third unidentified phase. Sesquicarbide was present in this pellet before irradiation. Single-phase material in another lot of pellets was observed near the top of Element SMP-1.

b. Synthesis. High-purity UC and (U, 15 w/o Pu)C powder prepared by the fluidized-bed technique (see Progress Report for February 1967, ANL-7308, p. 31) is being pressed and sintered into pellets suitable for irradiation testing by the Metallurgy Division. The pressing and sintering is being carried out in a high-purity helium glovebox in the

Chemical Engineering alpha facility. To meet typical specifications, the pellets must be 0.255 ± 0.001 in. in diameter and must have a density of $85 \pm 3\%$ of theoretical. Preliminary results were reported in the Progress Report for August 1967, ANL-7371, p. 78.

10X

Macrophotograph

10X

Alpha Autoradiograph

10X

Beta-Gamma Autoradiograph

Fig. II.D.2

Autoradiographs of Center Section of Element No. SMP-1 Loaded with $(U_{0.8}Pu_{0.2})C$ Pellets after Irradiation to 1.91 a/o Burnup at 15.9 kW/ft

(i) Pellet Pressing. Since the pellets will not be ground to meet the diameter requirements, the diameter and density of the sintered pellets must be controlled by proper die selection and adjustment of the green density and the sintering temperature. In the previously reported work, stearic acid was used as a binder and as a filler to adjust the green

density. In experiments during the past month, however, it was found that pellets of adequate green strength and reproducible green density could be pressed without a binder. Also, the green density of the pellets could be varied by adjusting the pressing pressure.

The results of several UC pellet preparations are shown in Table II.D.7. The pellets were pressed without use of a binder; however, a stearic acid pellet was pressed before each UC pressing to lubricate the die. The green pellets were 1.0 to 1.5 mils larger than the die. Despite 10 to 20% variation in the amount of material fed to the press, the green density was sufficiently reproducible. Green densities of similar reproducibility have been obtained in the pressing of (U,Pu)C powder under identical conditions. This material, however, has a slightly higher green density (7.70 g/cc at 60,000 psi) than the UC pellets.

TABLE II.D.7. Pressing and Sintering of UC Pellets
Conditions: Hydraulic press, 10^{-6} Torr vacuum during 3-hr sintering at 1720°C.
Pellet Requirements: Diameter, 0.255 ± 0.001 in.; density, 11.6 ± 0.4 g/cc (85 $\pm$ 3% of theoretical)

Run No.	No. of Pellets	Pressing Pressure (psi)	Green Diameter (in.)	Green Density (g/cc)	Sintered Diameter (in.)	Sintered Density (g/cc)
P-7	4	80,000	0.299	7.45-7.60	0.254-0.248	11.6-12.2
P-8	24	80,000	0.299	7.47-7.53	0.252-0.259	11.6-12.2
P-9	20	80,000	0.299	7.48-7.52	0.257-0.261	~11.0
P-10	25	60,000	0.299	7.28-7.41	0.252-0.255	11.7-12.1

(ii.) Sintering. Sintering is carried out in a furnace heated with a graphite resistance element. This furnace is now cooled by circulating water, although tetrachloroethylene was used in the past (see ANL-7371, p. 79). Sintering at 1720 to 1730°C produced UC pellets of approximately the desired density, namely, 11.6 g/cc (see Table II.D.7). The diameters of these pellets were also near the desired diameter of 0.255 in., and thus indicated that the die selection had been properly made. About 25% of these pellets met the rather strict specifications. The remainder were rejected for being slightly undersized, oversized, "hourglassed," or conical. As further experience is gained, it is expected that the percentage of acceptable pellets will increase.

The (U,Pu)C pellets were sintered at 1550 to 1650°C. However, this temperature range was too low to attain sufficient densification. Additional sintering experiments at higher temperatures are planned.

4. Fuel Cladding and Structure

a. Irradiation of Jacket Materials. The Instron tensile testing machine was shut down for modifications and repairs. Metallographic examination of stressed and unstressed sections of irradiated Type 304 stainless steel and Hastelloy-X tested at room temperature, 400, 500, and 650°C, was initiated. Similar sections from irradiated V-20 w/o Ti and V-15 w/o Ti-7.5 w/o Cr alloys tested at room temperature, 400, and 550°C are being prepared.

Thirty-four capsules containing 103 tube-burst specimens and 420 tensile-type specimens are being irradiated in EBR-II Subassemblies XA08, XO10, XO16, XO18, and XO20 at temperatures between 500 and 650°C. These specimens have accumulated at the end of this report period maximum total neutron exposures ranging from 3.6×10^{21} to 4.1×10^{22} n/cm². The status and identification of these capsules are given in Table II.D.8.

TABLE II.D.8. Status of Jacket Materials Being Irradiated in EBR-II

S/A No.	Capsule No.	Design Parameters				Operating Conditions	
		Jacket Composition (w/o)	Type of Specimen	No. of Specimens	Specimen Environment	Max Specimen Temp (°C)	Exposure to Date (n/cm ²)
XA08	AS-1	V-20 Ti	Tube-burst	12	Argon-helium	540	3.8×10^{22}
XA08	AS-2	V-20 Ti	Tube-burst	12	Argon-helium	540	3.8×10^{22}
XA08	AS-3	Hastelloy-X	Tube-burst	12	Argon-helium	540	4.0×10^{22}
XA08	AS-4	Hastelloy-X	Tube-burst	12	Argon-helium	540	4.0×10^{22}
XA08	AS-5	304 SS	Tube-burst	12	Argon-helium	540	4.0×10^{22}
XA08	AS-6	V-20 Ti	Tensile	16	Argon-helium	580	4.1×10^{22}
XA08	AS-7	Hastelloy-X	Tensile	16	Argon-helium	580	3.8×10^{22}
XA08	AS-8	304 SS	Tensile	16	Argon-helium	580	3.8×10^{22}
XA08	AS-12	V-20 Ti	Tensile	16	Argon-helium	580	3.7×10^{22}
XO10	AS-16	V-20 Ti	Tensile	13	Argon-helium	500	1.3×10^{22}
XO10	AS-17	V-20 Ti	Tensile	13	Argon-helium	500	1.4×10^{22}
XO10	AS-18	V-20 Ti	Tensile	13	Argon-helium	500	1.3×10^{22}
XO10	AS-19	V-20 Ti	Tensile	13	Argon-helium	500	1.3×10^{22}
XO10	AS-20	V-20 Ti	Tensile	13	Argon-helium	500	1.5×10^{22}
XO10	AS-21	V-20 Ti	Tensile	13	Argon-helium	500	1.8×10^{22}
XO10	AS-22	Hastelloy-X	Tensile	13	Argon-helium	500	1.5×10^{22}
XO10	AS-23	304 SS	Tensile	13	Argon-helium	500	1.8×10^{22}
XO10	AS-24	304 SS	Tensile	13	Argon-helium	500	1.5×10^{22}
XO10	AS-25	304 SS	Tensile	13	Argon-helium	500	1.4×10^{22}
XO10	AS-26	304 SS	Tensile	13	Argon-helium	500	1.4×10^{22}
XO16	AS-29	304 SS	Tensile	9	Sodium	650	3.8×10^{21}
XO16	AS-30	V-15 Ti-7.5 Cr	Tensile	9	Sodium	650	4.2×10^{21}
XO16	AS-31	V-20 Ti	Tensile	9	Sodium	650	4.0×10^{21}
XO16	AS-32	Hastelloy-X	Tensile	9	Sodium	650	4.0×10^{21}
XO16	AS-33	V-15 Ti-7.5 Cr	Tensile	9	Sodium	650	3.9×10^{21}
XO16	AS-50	V-15 Ti-7.5 Cr	Tensile	9	Sodium	650	4.2×10^{21}
XO16	AS-51	V-20 Ti	Tensile	9	Sodium	650	4.2×10^{21}
XO16	AS-52	Hastelloy-X	Tensile	9	Sodium	650	3.8×10^{21}
XO16	AS-53	V-20 Ti	Tensile	9	Sodium	650	4.1×10^{21}
XO18	AS-56	V-20 Ti, V-15 Ti-7.5 Cr	Tensile, Tube-burst	75	NaK	650	1.0×10^{22}
XO18	AS-57	304 SS Hastelloy-X	Tensile, Tube-burst	75	NaK	650	1.0×10^{22}
XO18	AS-58	V-20 Ti, V-15 Ti-7.5 Cr	Tensile, Tube-burst	25	NaK	650	1.0×10^{22}
XO20	AS-59	V-20 Ti	Tensile	9	Sodium	650	3.8×10^{21}
XO20	AS-60	V-20 Ti	Tensile	9	Sodium	650	3.6×10^{21}

b. Effect of Annealing Temperature on the Creep Strength of V-15 w/o Ti-7.5 w/o Cr and V-15 w/o Cr-5 w/o Ti. To date vanadium-alloy compositions V-15 w/o Ti-7.5 w/o Cr and V-15 w/o Cr-5 w/o Ti have been heat treated in vacuum for one hour, some at 1250 and some at 1500°C, then creep tested at 700°C and 30 kg-mm⁻² stress (42,000 psi stress). The 1250°C annealed alloys have a grain diameter of about 0.05 to 0.06 mm (≈ 700 grains/mm²) and exhibit the highest creep strength observed for any vanadium-base alloys under these test conditions. The minimum creep rates are approximately 1.8×10^{-8} sec⁻¹ and 4.5×10^{-9} sec⁻¹ for V-15 w/o Ti-7.5 w/o Cr and V-15 w/o Cr-5 w/o Ti, respectively. In contrast, the 1500°C annealed alloys have a 0.5-mm grain diameter (≈ 5 grains/mm²) that is undesirable in thin-walled tubing, i.e., tubing with a wall thickness between 0.4 and 0.5 mm.

Since these alloys derive their creep resistance from both solid-solution strengthening and the precipitation of an impurity phase present as a small volume fraction of the total alloy matrix, there is reason to believe that their creep strength and uniform strain can be optimized by some high-temperature, short-time flash-annealing treatment. The object of the flash-annealing treatment is to manipulate the size, distribution, and perhaps the morphology of the precipitated particles while maintaining a desirable alloy grain size.

Assembly of the experimental setup for conducting the proposed flash-annealing studies has been completed and tested for maximum heating rate, maximum achievable temperature, and temperature control. The flash-annealing parameters will include heating and cooling rates in excess of 280°C.sec⁻¹ and holding times ranging from seconds to minutes. Specimens (0.5 cm in diameter by 0.95 cm long) for these studies are being machined from rod lengths of the two V-Ti-Cr ternary alloys. Specimens of V-10 w/o Ti, V-20 w/o Ti and V-40 w/o Ti are also included. The V-20 w/o Ti alloy will be used as a reference composition, and together with the other two binary alloys will provide information regarding the effect of composition on the dissolution characteristics of the precipitated phase.

c. Effects of Alloy Composition, Annealing Temperature, and Testing Temperature on the Short-term Properties of Vanadium-base Alloys. The ultimate tensile strength, yield strength, and uniform ductility are the important material properties to be considered in determining the ability of fuel cladding to survive a credible accident such as that attending a reactor excursion. Constant-strain-rate tensile tests of V-10 w/o Ti, V-20 w/o Ti, V-40 w/o Ti, V-15 w/o Ti-7.5 w/o Cr, and V-15 w/o Cr-5 w/o Ti indicate that these short-time properties can be altered by annealing temperature, testing temperature, and composition. A program has been written for the computer reduction of tensile data and the debugging of this program is essentially complete. Experimental data

for approximately 40 tensile tests on these alloys are being prepared for programming in the computer. The range of annealing temperatures covered by these data is 900 to 1250°C, and the test temperature range is 25 to 800°C.

d. Development of Refractory-metal Alloys for Service in Oxygen-contaminated Sodium. Static, oxygen-refreshed testing of vanadium and selected alloys in sodium has been started at a temperature of 600°C in two systems with different cold-trap temperatures (nominal 110 and 175°C). As usual, much larger weight losses have been recorded for the system with the higher cold-trap temperature (higher oxygen concentration) in the initial one-week exposure. A dynamic test at 450°C is nearly complete, and dynamic exposure at 600°C will begin as soon as new sample material is available.

Vanadium alloys corrode more in a static-refreshed system made of Type 304 stainless steel than in a dynamic system constructed largely of Type 318 (stabilized Type 316) stainless steel at the same nominal low cold-trap setting. Carbon transfer from the walls of the system to the vanadium alloys is suspected to be a contributing factor. Samples from the two systems are being analyzed for carbon pickup. An attempt is being made to evaluate the carburizing ability of the two systems by equilibration with pure iron foils. Carbon analysis of the sodium is not considered to be a fruitful approach since it has been shown* that the gross carbon analysis does not necessarily correlate with carburizing ability.

e. Mass Transfer of Nickel. A pumped sodium loop is being used to study the mass transfer of nickel-base alloys exposed to sodium. The loop (see Fig. II.D.3) has the unique feature that the test section for high-temperature sodium ($\geq 350^\circ\text{C}$) is constructed of essentially inert refractory material, while the components in contact with low-temperature sodium ($< 350^\circ\text{C}$) are made of Type 304 stainless steel. The test section consists of two concentric pipes fabricated of molybdenum and TZM (molybdenum-base alloy). This loop is so designed that purified (free of test-alloy constituents) sodium is continually supplied to the test section.

The nickel removed from the nickel specimens in the high-temperature sodium (avg 645°C) was partly deposited on the crystallizers in the lower temperature test section (avg 373°C). The remainder was either plated out in the reservoir at similar temperature (avg 326°C) or in the cold trap maintained at an average temperature of 122°C. The test condition and preliminary results are summarized.

*MSAR 67-103, Topical Report No. 8, Effect of High Temperature Sodium on Austenitic and Ferritic Steels (July 1967).

Fig. II.D.3. Loop to Study Nickel Mass Transfer

Test Specimen

- (a) Material--Nickel 200
- (b) Type--Strip (15.32 x 1.13 x 0.08 cm)
- (c) Quantity-- Four
- (d) Surface Area-- 37.15-cm²/Specimen; 148.6 cm² Total

Crystallizers

- (a) Material-- Nickel 200 and Commercial Titanium 75A
- (b) Type-- Strip (25.40 x 1.13 x 0.08 cm Ni)
(25.40 x 1.13 x 0.06 cm Ti)
- (c) Quantity-- Two Nickel, Two Titanium
- (d) Surface Area-- 61.48 cm² Each; 122.96 cm² Total Ni
60.27 cm² Each; 120.54 cm² Total Ti

Test Section

- (a) Specimen Temperature
 - (1) Inlet-- 640°C avg
 - (2) Middle-- 645°C avg (655°C max, 626°C min)
 - (3) Exit-- 643°C avg
- (b) Crystallizer Temperature
 - (1) Inlet-- 349°C avg
 - (2) Middle-- 323°C avg (328°C max, 312°C min)
 - (3) Exit-- 643°C avg
- (c) Flow-- 0.45 gpm avg (0.47 gpm max, 0.43 gpm min)
- (d) Velocity-- 21.2 cm/sec avg

Reservoir

Temperature-- 326°C avg (329°C max, 322°C min)

Cold Trap

- (a) Temperature-- 122°C avg (137°C max, 114°C min)
- (b) Flow-- 0.45 gpm avg (0.46 gpm max, 0.44 gpm min)

Sodium Purity

- (a) Oxygen-- 2.7 wt ppm avg by Distillation Method
(3.7 wt ppm max, 1.5 wt ppm min)
- (b) Nickel-- < 0.1 wt ppm

- (c) Iron -- < 0.1 wt ppm
- (d) Chromium -- < 0.01 wt ppm
- (e) Molybdenum -- < 0.1 wt ppm
- (f) Titanium -- < 1 wt ppm
- (g) Calcium -- < 1 wt ppm
- (h) Potassium -- < 1 wt ppm

Mass Transfer

- (a) Nickel Specimens -- 66.2-mg weight loss in 148.6 cm^2 of surface area, or 0.44 mg/cm^2
- (b) Crystallizers -- 3.4-mg weight gain in 122.96 cm^2 of nickel surface area, or 0.03 mg/cm^2
-- No significant weight gain for titanium crystallizers
- (c) Test Period -- 1152 hr

The nickel loss of 0.44 mg/cm^2 in 1152 hr is equivalent to approximately 1.8×10^{-2} mil per 1000 hr based upon linear extrapolation of the data for these test conditions with refreshed sodium. This number is given as a matter of interest only. The results of this preliminary experiment do not justify calculation of loss rates for systems of practical interest.

To increase the understanding of the mechanism for nickel mass transfer, the following two experiments will be performed next:

(a) Same test conditions as first experiment except for increase of the sodium velocity in the test section. Hopefully, the necessary changes will also result in an increased amount of nickel collected at the crystallizers (above the present level of 5%).

(b) Repeat test condition described in (a), except with cold trap valved off.

f. Nondestructive Tests of Experimental Fast Reactor Components. Thermal-pulse equipment was used to determine the thermal conductivity of two samples of V-15 w/o Cr-5 w/o Ti. The values obtained are given in Table II.D.9.

TABLE II.D.9. Thermal Conductivity of V-15 w/o Cr-5 w/o Ti

Specimen	Temperature T (°C)	Thermal Conductivity K (cal cm/cm ² -sec-°C)
1	31	0.0684
2	31	0.0712
	98	0.0749
	96	0.0754
	196	0.0779
	204	0.0815
	294	0.0822
	302	0.0825
	393	0.0865
	397	0.0881
	495	0.0895
	492	0.0891
	601	0.0959
	592	0.1000

E. 1000-MWe Plant

Argonne is managing design studies by five industrial firms to identify the research and development needed for successful application of large liquid-metal-cooled fast breeder reactors for central-station power plants in the 1980s.

1. Contract Management and Liaison

The Laboratory is coordinating the activities of the contractors to assure that all studies are being conducted in accord with the established scope and common ground rules and background information.

a. Westinghouse. Westinghouse has initiated Task-II work in the areas of reactor-vessel internals, reactivity control system, fuel management, and reactor-core system. The Task-I reactor core-concept analysis has been completed; the conceptual system-design descriptions are being prepared for the reference core. Nuclear intercomparison of the oxide and carbide cores has been completed.

Preliminary arrangement drawings of the reactor, fuel-handling, and primary- and secondary-coolant systems have been submitted by the architect-engineer. In preparation for parametric studies, a computer coding of the linearized plant-component equations, constraints, restrictions, and search logic has been completed; the code is being debugged.

b. Atomics International. Atomics International has completed the work plans for Task I and identified the subject matter for each of the technical reports to be issued during the Task-I effort. Work plans for Tasks II, III, and IV are nearing completion; the planned level of staffing for the Task-I technical effort has been attained.

c. Combustion Engineering. The system-design descriptions and the Task-I report are being prepared. The quarterly technical report for the period ending July 31, 1967 was completed and issued.

d. Dabcock & Wilcox. Task-II engineering work and tradeoff studies are continuing. Work was begun on the quarterly report for the period June 1 to August 31, 1967.

e. General Electric. Thermal and hydraulic analysis has been completed for both the advanced- and conservative-type core concepts. These cores are being optimized with the various combinations of data resulting from the thermal and hydraulic analysis. Safety work has progressed through the establishment of accident models and damage criteria. Transient analysis has been initiated to determine the validity of the safety criteria.

General arrangements for the core designs have been completed, and the refueling-systems design concept has been selected for Task I.

An initial review of several intermediate-heat-exchanger arrangements, and a series of conceptual studies for alternate primary-system arrangements, have been completed. Computer-code optimization runs were completed to determine the cost sensitivity of various equipment and parameters of the 1000-MWe LMFBR plant and systems.

F. Fast Flux Test Reactor

1. Critical Experiment Program

a. Phase-B Critical Experiment Program. In order to accommodate Doppler measurements and reaction rate traverses more readily, the proposed Assemblies II and IV were designed with the test section rotated through 90° into the vertical direction. The reasons for this are as follows: First, the Doppler measurements have been planned to be made in the plane defined by the width of the test section and the axis of the core. This plane is now horizontal and coincides with the plane in which the Doppler apparatus can be moved most easily. Secondly, this central horizontal plane or P plane already contains many traverse holes which are parallel to the dividing face and which pass through the axis of the core. Three of these

are at convenient distances from the dividing face to map adequately the small-sample reactivity worth and reaction rate of materials in the P plane. These holes are located at $1\frac{1}{2}$ and $13\frac{1}{4}$ in. from the dividing face in the fixed half of the assembly, and at $12\frac{1}{2}$ in. from the dividing face in the movable half. Traverse measurements are most easily and accurately made in holes in this direction.

Traverse measurements are planned along the central core axis, i.e., location P-16. Further mapping which is required for Assemblies II and IV can be made in an axial direction at many locations, but with less accuracy and greater difficulty. For this reason it was suggested that the horizontal traverse holes be drilled in the L plane at $13\frac{1}{4}$, $1\frac{1}{2}$, and $12\frac{1}{2}$ in. from the dividing face and in the H plane at $1\frac{1}{2}$ in. from the dividing face to provide adequate mapping of Assemblies II and IV.

The Work Plan with the above modification along with some detailed changes in the experiments suggested by Pacific Northwest Laboratory (PNL) were reviewed and incorporated into the schedule of a joint ANL-PNL meeting held on September 13, 1967 at the Idaho Falls Site.

In order to allow a timely phasing out of the present ZPR-3 experimental program with Assemblies 49 and 50 (see Sect. II.B.1), the Phase-B critical experiments are anticipated to start on or about October 23, 1967. The new schedule of experiments is included in Table II.F.1.

TABLE II.F.1. Phase-B Assembly-I Program

Modification of ZPR-3 Facility	14 days
Loading and Calibrations	22 days
Spectrum Measurements	5-6 days
Regional Worth	4-6 days
Distributed Worth	10 days
Doppler Coefficient Measurements	6 days
Reaction Rate and Reactivity Traverses	10 days
Total	71-74 days

b. Development of Cross-section Set. A 28-group cross-section set has been generated, using the MC² code, specifically for the core and reflector composition of Assembly-I Phase-B critical experiment. The atom concentration and the sources of the basic cross-section data are listed in Table II.F.2 and the energy limits of the group structures are included in Table II.F.3. The core-averaged cross sections were incorporated in a heterogeneous fundamental-mode approach and the reflector-averaged cross section in a homogeneous medium with zero buckling.

This combined set of cross sections is designated as ANL set 28001. Thermal-group cross sections being generated with the THERMOS code for the core and the reflector are to be included in the above set. These 29 groups will then be collapsed into a 6-group structured set planned to be used for the two-dimensional analysis in this series of experiments.

TABLE II.F.2. Atom Concentrations

		Cross-section Source	
	Core	Library	Reflector
Pu ²³⁹	0.001732 atom/b-cm	ENDF/B	10 ⁻⁹
Pu ²⁴⁰	0.000168	ENDF/B	10 ⁻⁹
Pu ²⁴¹	10 ⁻⁹	ENDF/B	10 ⁻⁹
U ²³⁸	0.007041	ENDF/B	10 ⁻⁹
U ²³⁵	0.000015	ENDF/B	10 ⁻⁹
O	0.012880	MC ²	10 ⁻⁹
C	0.002719	MC ²	10 ⁻⁹
Fe	0.016114	MC ²	0.007273
Cr	0.003886	MC ²	0.01406
Ni	0.001530	MC ²	0.04981
Na	0.00835	ENDF/B	0.004569
B ¹⁰	10 ⁻⁹	ENDF/B	10 ⁻⁹

TABLE II.F.3. Group Energy Limits

Group	Energy Lower Limit	Lethargy Lower Limit	Group	Energy Lower Limit	Lethargy Lower Limit
1	3.68 MeV	1.0	15	2.61 keV	8.25
2	2.23 MeV	1.5	16	2.03 keV	8.5
3	1.35 MeV	2.0	17	1.23 keV	9.0
4	0.821 MeV	2.5	18	0.961 keV	9.25
5	0.498 MeV	3.0	19	0.583 keV	9.75
6	0.302 MeV	3.5	20	0.275 keV	10.5
7	0.183 MeV	4.0	21	0.101 keV	11.5
8	0.11 MeV	4.5	22	29.0 eV	12.75
9	67.4 keV	5.0	23	17.6 eV	13.25
10	41.0 keV	5.5	24	6.48 eV	14.25
11	24.8 keV	6.0	25	3.93 eV	14.75
12	15.0 keV	6.5	26	1.44 eV	15.75
13	9.12 keV	7.0	27	0.876 eV	16.25
14	4.31 keV	7.75	28	0.532 eV	16.75

c. Preplanning Analysis of Assembly I. It is planned to mockup the 30-cm FTR nickel-sodium reflector with the present total nickel inventory. This allows a 17.4-cm reflector to be constructed adjacent to the core. In order to reproduce the neutronic environment of the 30-cm reflector in this region, it is planned to use a stainless steel composition in the region surrounding the nickel-sodium reflector. A series of studies have been completed, using a 22-group set in a one-dimensional cylindrical

geometry and in the diffusion approximation, to determine the optimum thickness of the stainless steel region. The concentration used for this computation are included in Table II.F.4. The effectiveness of the combined reflector was evaluated in terms of reproducing the total reactivity, k_{eff} , and in terms of reproducing in magnitude and energy structure, the basic 30-cm-thick nickel-sodium reflector system.

TABLE II.F.4. Atom Concentrations

	Core	Ni-Na Radial Reflector	SS Radial Reflector
Pu ²³⁹	0.001732 atom/b-cm	-	-
Pu ²⁴⁰	0.000168	-	-
U ²³⁸	0.007041	-	-
U ²³⁵	0.000015	-	-
O	0.012880	-	-
C	0.002719	-	-
Fe	0.016114	0.008817	0.055883
Cr	0.003886	0.002254	0.014288
Ni	0.001530	0.067324	0.006254
Na	0.008350	0.005016	-

The total reactivity of the various core-reflector configurations and the ratio of the peak flux in the mixed-reflector system to that of the basic configuration for the lower-energy groups (groups 13 to 22) are included in Table II.F.5. It appears that a stainless steel region of 15.4-cm thickness (3-drawers) reproduces most adequately the neutronic condition of fuel nickel-sodium reflector. The deviation of the flux ratio from unity for the neutron energy groups above 13, were proved to be negligible.

TABLE II.F.5. Reactivities

Thickness of Stainless Steel (cm):		9.9	12.6	15.4
k_{eff} :		0.9960	0.9977	0.9999
Ratio of Peak in Group with E_{lower}				
22	29.02 eV	0.8481	0.8797	0.9398
21	101.30 eV	0.9331	0.9565	0.9920
20	275.36 eV	0.9511	0.9707	1.0013
19	582.95 eV	0.9639	0.9807	1.0052
18	961.11 eV	0.9703	0.9847	1.0059
17	1.23 keV	0.9748	0.9871	1.0058
16	2.03 keV	0.9798	0.9903	1.0048
15	2.61 keV	0.9800	0.9902	1.0045
14	4.31 keV	0.9832	0.9917	1.0037
13	9.12 keV	0.9870	0.9948	1.0025

III. GENERAL REACTOR TECHNOLOGY

A. Applied and Reactor Physics Development

1. A Continuous-flow Manganese Bath System

For fast-neutron detection involving problems of high accuracy or difficult gamma-ray backgrounds, the manganese bath system has been an acknowledged success. The system consists basically of a moderator-filled tank in which dissolved manganese is activated and ultimately counted. Early techniques involved drawing a sample from the bath and then counting in a facility located away from the radiation field, or, if possible, placing counters directly into the tank after the neutron source was withdrawn.

A newer procedure* yields much higher count rates, greater stability, and improved signal-to-background conditions by pumping the liquid, in a continuous loop, from the manganese bath tank to a remotely situated counter. This permits extensive shielding of the counter in a low-background location, optimum design of the detection geometry, and maximum count rate during the buildup, saturation, and decay phases of Mn^{56} activation. Compared to the highly stable gamma-gamma coincidence system previously used,** the count rates now obtained are about one thousand times greater than the "coincidence" rate and about twenty times greater than single-channel counting of the 0.85-MeV peak. Partially offsetting this improvement is the fact that background is higher by a factor of 15; even so, a $(\text{signal})^2/\text{background}$ criterion still strongly favors the new on-line counting system.

The chief features of the continuous-flow system are given in Fig. III.A.1. Neutron sources may be placed at the center of the sphere to induce manganese activity proportional to the source strength. The active solution is continuously mixed by a circulating pump which returns the liquid to the sphere after it has passed through a dispersion unit. Simultaneously, some of the active solution is pumped into the on-line counter.

An annular detector of $\text{NaI}(\text{Tl})$, 15 x 15 cm with 2.5-cm wall was used. The gamma rays from Mn^{56} mostly escape the liquid and interact in the sodium iodide crystal with high probability. All pulses resulting in at least 50-keV transfer to light photons are accepted by the electronic level discriminator.

Shielding for the on-line counter consists of an outer shield composed of borated concrete to reduce neutron background and an inner shield composed of 10-cm-thick plates of armor plate. Between these is a cylindrical

* Axton, E. J., Cross, P., and Robertson, J. C., J. Nucl. Energy 19, 409 (1965).

** DeVolpi, A., Porges, K. G. A., and Larsen, R. N., ANL-6760 (1963).

shield made of lead. Materials in close proximity to the sodium iodide crystal, other than the manganous sulphate solution, are stainless steel, quartz, Lucite, and MgO. Access is provided through the top.

Fig. III.A.1. General Arrangement of Continuous-flow Manganese Bath System

Electronic processing of signals derived from the photomultiplier (RCA C70043G bi-alkali) is rather straightforward. No active electronic elements are located within the shield. A 50- Ω cable delivers the anode signal to a low-input-impedance preamplifier which drives a pole-zero-cancellation bipolar amplifier with a gain of 1600. The output of the amplifier is loaded with 50 Ω for an adjustable paralysis discriminator (APD)*, set at a 9.5-mA level. The combination of 1250 V applied to the phototube, the amplifier gain, preamplifier matching, and discriminator level is sufficient to count pulses in the neighborhood of 50 keV. A precise deadtime of 10 μ sec is applied by the APD. Plateaus with slopes of 0.1%/mA on the discriminator, 1%/100 V in high voltage, and comparable results in amplifier gain are obtained.

*DeVolpi, A. *et al.*, Rev. Sci. Instr. **38**, 1592 (1966).

The APD feeds either or both of two scaling systems. One contains a recycling timer which initiates, at preset intervals, the readout, resetting, and count start of the scaler. The readout is printed on continuous paper tape. This readout station is mobile. A parallel scaling system permanently located in a counting room derives a 50- Ω terminated pulse from the APD. This pulse is reshaped in one channel of a six-channel automatic readout station. The output from this system is punched on cards.

Data from either of the two readout stations may be processed by the computer program SATURATE, which determines the saturated activity for each datum point, based on the known irradiation conditions. A weighted mean is taken for all datum points. Included in the program are corrections for pumping time, mixing time, deadtime, point-by-point normalization, background, and other factors.

The system has performed satisfactorily. Corrections for finite pumping time for rates of 300 liters/hr are of the order of 1/2%. The background level is now about 33 counts/sec integral from the lower-level discriminator. Experiments indicate that by setting an upper-level discriminator at about 3 MeV an additional 4 counts/sec, caused chiefly by cosmic rays, could be excluded in anticoincidence without loss of Mn^{56} counts.

Checkouts can be made using various sources which fit a penetration in the counter. Ra^{226} in equilibrium is a useful standard because its overall spectrum and endpoint are roughly equivalent to those of Mn^{56} .

The net efficiency for counting is 0.37% per Mn^{56} disintegration in the entire bath system. This corresponds to 0.16% efficiency per source neutron (at a MnSO_4 concentration of about 400 g/liter).

Since background experience indicates a highly stable level, it is possible to consider the counting of sources with emission rates of only 10^4 /sec with a precision of 0.1% in a normal cycle of two days. The two-day cycle consists of one day to activate to saturation (ten half-lives) and one day for decay to background.

A number of sources have been calibrated and compared with the U.S. standard neutron source, NBS-II, and the neutron-emission rates for various fission sources have been determined for neutron-yield measurements.* Several precision measurements of fundamental cross sections are anticipated with the aid of this improved manganese bath system.

*DeVolpi, A., and Porges, K. G., Proc. Intern. Conf. Nuclear Data, IAEA, Paris, October 1966 (to be published).

2. Evaluation of Cross-section Data

Figure III.A.2 illustrates schematically the preparation of a library tape for the MC² code beginning with the basic data evaluation. The codes CHAD, SAD, ABACUS, OPTIC, 2-PLUS, etc., are used in the processing of experimental cross-section data and in the calculation of neutron cross sections. Evaluated cross sections are punched on cards in the ENDF/B format and are then read onto a magnetic tape. This tape is checked for errors of the "bookkeeping" type using the CHECKER code and corrected, if necessary, with the aid of the CRECT code. Then program DAMMET is used to convert from the BCD tape to a binary tape in the form required by the ETOE code. DAMMET may also be used to produce a tape containing selected data from two input tapes. Both the input and output tapes for DAMMET may be any combination of BCD tapes or either of two allowable types of binary tapes. The ETOE program produces a binary tape in the format used by the MC² code from the DAMMET binary tape and the W-table binary tape. The W-table tape contains tables of the real and imaginary parts of the complex probability integral, which are used in calculating Doppler-broadened resonance cross sections in MC².

Fig. III.A.2. Preparation of Library Tape for MC² Code

The MERMC2 code may be used to combine selected data from two MC² binary tapes or to delete materials from one MC² tape.

The system in Fig. III.A.2 is operational on the CDC-3600. A start has recently been made at conversion of the codes to the IBM system 360.

3. Calculation of Resonance Integrals in the Unresolved Region

Calculation of infinite-dilution resonance integrals in the unresolved resonance region has been performed for fissile nuclides. Both multilevel and single-level methods have been employed to generate cross sections which are used in the resonance-integral calculations. The various resonance parameters and level spacings are selected from chi-squared distributions of orders one and two.

Thirty-seven sets of random numbers have been used in calculating α , the ratio of the capture and fission resonance integrals, for Pu^{239} from 150 to 250 eV. For this isotope, the single-level result α_s is only 1.5% smaller than the multilevel ratio α_m . The $J = 0$ spin state for Pu^{239} yields large discrepancies in α_s and α_m since interference effects are more relevant. The single and multilevel α 's for the $J = 1$ spin state deviate very little since $\bar{\Gamma}/\bar{D}$ is much less than 1.0 for this state. However, in calculating a total α for Pu^{239} a summation over both spin states is required, and the contributions to α from the $J = 0$ state are small compared to the $J = 1$ results. Therefore, the total α appears to be only slightly affected by choice of single- or multilevel formalisms.

Thirty sets of random numbers have also been used to generate α for Pu^{239} in the interval from 900 to 1000 eV. In this range α_s is approximately 2% smaller than α_m .

Other isotopes are also being considered in this study, including U^{235} , which should exhibit greater interference effects and larger deviations in α_s and α_m . Preliminary results indicate that there are deviations of several percent in α_s and α_m for U^{235} .

4. Burnup Analysis and Fission Yields for Fast Reactors

a. Development of Analytical Procedures for Fission-product-burnup Monitors. In the determination of burnup of fast reactor fuels by fission product analysis, radiochemistry, mass spectrometry (isotope dilution), and spectrophotometry have been the analytical techniques most widely used. However, each technique (see Progress Report for July 1967, ANL-7357, p. 69) has very definite limitations. A program is, therefore, being carried out to investigate the feasibility of using other analytical techniques for the determination of burnup in fast reactor fuels.

(i) Photometric Titration of Total Rare Earths. A method being developed for determining burnup by means of a total rare earth analysis was described in ANL-7357, p. 69. Results, also given in that report, indicated complete recovery of the rare earths through the separation procedure and photometric titration.

The effectiveness of the separation and titration has since been tested with a synthetic fission solution containing a known amount of rare earths. Three aliquots of this solution, each of which contained about 0.5 mg of rare earths, 100 mg uranium, 0.4 mg zirconium, 2.3 mg molybdenum, 2.1 mg ruthenium, 0.3 mg rhodium, and 0.2 mg palladium, were put through the separation procedure and titrated. The rare earth recoveries were 99.9, 99.7, and 100.2%.

The separation and photometric titration has also been tested with a 13% enriched UO_2 fuel test specimen which had been irradiated in MTR to approximately 6% burnup. A cross-sectional cut of the fuel pin was dissolved along with its stainless steel cladding in aqua regia and a trace of hydrofluoric acid. An aliquot of this solution was then put through the separation procedure. The solution containing the separated rare earths, when examined for radiochemical purity using a Ge-Li detector system, was found to contain only rare earths. An aliquot of this solution was titrated satisfactorily; it contained 2.39×10^{-3} millimole rare earths. This amount of rare earths compares favorably with a value 1.96×10^{-3} millimole which was calculated from an approximate burnup figure obtained by heavy-element mass spectrometry. The total rare earth fission yield used was 49.5%. A more accurate burnup will be obtained by a lanthanum-139 mass-spectrometric isotope-dilution analysis.

The possibility exists that the large amounts of stainless steel cladding in the irradiated-fuel solution (approximately 20% of the sample weight) may have interfered with the rare earth determination. An X-ray analysis of the sample after it was put through the separation procedure did reveal detectable amounts of iron and chromium. Since these results were qualitative and not quantitative, it is not possible to say at this time whether or not the amounts of iron and chromium present were large enough to cause a significant error in the photometric titration for rare earths. To determine if this is a source of error, synthetic samples containing amounts of iron, chromium, nickel, and manganese comparable to the amounts present in the irradiated fuel sample will be put through the rare earth determination.

(ii) X-ray Spectrometric Determination of Rare Earth Fission Products. The possibility of using X-ray spectrometric methods for determining burnup was discussed in ANL-7357, p. 70. Since the rare earths appear to be the fission products that are least likely to migrate in oxide fuels, the application of X-ray spectrometry to burnup analysis is being directed initially toward the determination of one, two, or all of the members of this group.

Further investigation of the relative merits of the K and L spectra for the determination of the rare earth fission products has led to the choice of the L spectrum for further investigation. Despite the fact

that the L spectrum is more complex than the K spectrum, the higher sensitivity (by a factor of ~50) achievable with the L spectrum will lower the

Fig. III.A.3. X-ray Spectrogram of Simulated Rare Earth Fission Product Mixtures

amount of activity on the sample mount to a level which is acceptable from a health-physics standpoint. With 100 μg of rare earths mounted on an aluminum plate, the L spectrum shown in Fig. III.A.3 was obtained with a General Electric XRD-6 instrument under the following conditions of operation: 56 kVp, 64 mA, lithium fluoride crystal, proportional counter and helium atmosphere. The X-ray intensities shown for each of the four principal rare earths formed in fission, namely, lanthanum, cerium, praseodymium, and neodymium, are more than adequate for accurate analysis (100 μg of rare earths are produced in one gram of uranium when the burnup is 0.03%).

Since some of the rare earths are radioactive, the effect of these activities on the X-ray detectors was evaluated using a fission product mixture of the rare earths separated for the total rare earth titration method

discussed above. A measurable increase in background was observed, but it is not of such magnitude that it will hamper the application of the method. This confirms the result previously obtained (see ANL-7357, p. 71) using only cerium-144 (the principal rare earth gamma emitter).

Now that both the sensitivity and selectivity of the method for the individual rare earths have been established, efforts will be directed to the problems of making the method quantitative, i.e., establishing an accurate relationship between the intensity of a particular X ray and the amount of rare earth on the mount.

B. Reactor Fuels and Materials Development

1. Behavior of Reactor Materials

a. Irradiation of High-temperature Materials. Ceramic fuel materials of interest for FBR's and other high-temperature reactor applications are being irradiated in instrumented temperature-controlled capsules in MTR. The materials include UC-20 w/o PuC, PuC, and US, all jacketed in Nb-1 w/o Zr. A summary is shown in Table III.B.1

TABLE III.B.1. Status of Ceramic-fuel Irradiations in MTR

Capsule No.	Specimen Number	Design Parameters				Operating Conditions		
		Fuel Composition (w/o)	Effective Density (%)	Jacket OD (in.) ^a	Jacket Thickness (in.)	Max Jacket Temp (°C)	Estimated Burnup to Date	
							a/o (U + Pu)	fiss/cc x 10 ^{-20b}
56-11	MV-2	UC-20 PuC	79	0.281	0.012	470	8.4	22
56-8	MV-3	UC-20 PuC	81	0.281	0.012	715	8.9	24
56-8	MV-5	UC-20 PuC	80	0.281	0.012	705	8.6	23
56-11	MV-6	UC-20 PuC	80	0.281	0.012	480	8.9	23
56-13 ^c	Z-4	UC-20 PuC	79	0.174	0.015	665	5.0	13
56-13 ^c	Z-5	UC-20 PuC	79	0.174	0.015	585	5.0	13
56-13 ^c	Z-7	UC-20 PuC	79	0.174	0.015	570	5.0	13
56-13 ^c	C-45	PuC	84	0.174	0.009	700	6.2	16
56-8	S-7	US	80	0.281	0.012	535	7.2	14
56-8	S-8	US	89	0.281	0.012	725	9.2	18
56-8	S-9	US	76	0.281	0.012	750	9.2	17
56-8	S-10	US	91	0.281	0.012	690	9.2	20
56-11	S-15	US	82	0.281	0.012	380	6.1	12
56-11	S-16	US	90	0.281	0.012	510	8.2	18
56-11	S-17	US	88	0.281	0.012	500	6.9	15
56-11	S-18	US	77	0.281	0.012	610	8.7	16

^aAll cladding is Nb-1 w/o Zr alloy.

^bBased on effective density.

^cRemoved from MTR at end of Cycle 265.

Capsule 56-13, containing carbides, decreased uniformly in temperature during the last MTR cycle. This behavior indicates contamination of the gas annulus, used for temperature control, with either water or NaK. The capsule has been removed and is being shipped to Argonne, Illinois, for disassembly.

b. Corrosion Inhibition by Dissolved Getters in Liquid Sodium.

Loading of the dissolved getter (DG) loop with sodium, dissolution into this sodium of 1000 appm* of magnesium, and melt homogenization and sampling activities have been accomplished. Minor apparatus modifications have been made to ensure consistent operation of nickel ball check valves in the 650°C loop sodium. To initiate effort at control of hydrogen contamination of loop sodium, short lengths of nickel tubing bearing an outer jacket of platinum have been ordered.

During dissolution of magnesium in loop sodium at 650°C, a local attack of dissolution type was suffered by a nickel member supporting the magnesium. This was later attributed to a local transient occurrence of Mg-Ni mixtures in the eutectic alloy range of composition (mp 508°C). Other nickel surfaces in contact with the sodium-magnesium solution did not appear to be adversely affected, in accord with previous preliminary results.

A chemical spot test was performed to determine whether gray discoloration of V-20 w/o Ti tested in static, 650°C Na-Mg in a nickel container (see Progress Report for August 1967, ANL-7371, pp. 85-86) was due to nickel deposition. No indication of nickel on the vanadium alloy surface was observed.

*Atomic parts (of magnesium) per million (of sodium).

c. Dissolution Kinetics in Liquid-metal Systems. A proposed method for analysis of oxygen concentrations in sodium is being developed in conjunction with the study of corrosion mechanisms in refractory metal-sodium systems. The method involves the equilibration, with respect to oxygen, of body-centered-cubic refractory metal wires in liquid sodium and the subsequent measurement of the oxygen concentration in the wires by internal-friction techniques.

Calculations based on thermodynamic data indicated that tantalum was the most appropriate refractory metal for measuring oxygen concentrations in sodium from 1 to 25 wt ppm. The linear relationship between internal-friction peak height Q^{-1} and oxygen concentration $C_{O,Ta}$ in tantalum has been determined for 0.51-mm-dia tantalum specimens as

$$C_{O,Ta}(\text{wt ppm}) = 10,850 Q^{-1}.$$

The distribution coefficient for oxygen partitioning between tantalum and sodium was calculated previously from the best available solubility and free energy data as

$$K_w = 7.829 \exp\left(\frac{1098}{T}\right) = \frac{\text{wt ppm } O \text{ in Ta}}{\text{wt ppm } O \text{ in Na}}.$$

This corresponds to a value of 24 at 700°C. Since well-defined internal-friction peaks corresponding to less than 2 wt ppm oxygen in tantalum have been measured, oxygen concentrations of less than 1 wt ppm in sodium should be measurable.

An attempt has been made to verify this distribution coefficient experimentally by correlating the internal-friction peaks of tantalum wires exposed in sodium with oxygen concentrations in the sodium obtained by other methods. Significant changes, both increases and decreases, in the internal-friction peak heights have been measured with tantalum wires exposed to sodium under various conditions. The peak heights have been compared with cold-trap temperature, cold-trap solubility data for oxygen in sodium, and oxygen analyses of sodium obtained by vacuum distillation techniques. Preliminary data indicate a stronger temperature dependence of the friction peak height than is obtained from the calculated distribution coefficient. More data are required before definite conclusions can be made.

One important result obtained from these experiments is the significant increase of the nitrogen internal-friction peak measured with nearly every tantalum wire exposed to sodium. Since the diffusion rate of nitrogen in tantalum is very slow compared to that of oxygen, the nitrogen did not reach equilibrium during the exposure time. This was verified by pickling the wire to remove the outer layers and then remeasuring the friction peaks. The nitrogen peaks decreased, indicating a higher nitrogen concentration near the surface; however, the oxygen peaks were unaffected,

indicating a homogeneous oxygen concentration throughout the cross section of the wire. The nitrogen peaks also tended to increase more with longer exposure times while the oxygen peaks were relatively insensitive to time in the range of exposure times used.

Niobium and vanadium wires have also been exposed to sodium since their equilibration times for nitrogen are much shorter than for tantalum. The results of some typical exposures to 700°C sodium contained in a stainless steel system are given in Table III.B.2. Vacuum distillation analyses of the sodium indicated 1 to 2 wt ppm oxygen.

TABLE III.B.2. Internal Friction of Refractory Metal Wires Exposed to 700°C Sodium^a

Wire Material	Sample No.	Oxygen				Nitrogen			
		Before Sodium Exposure		After Sodium Exposure		Before Sodium Exposure		After Sodium Exposure	
		Q ⁻¹ x 10 ³	wt ppm	Q ⁻¹ x 10 ³	wt ppm	Q ⁻¹ x 10 ³	wt ppm	Q ⁻¹ x 10 ³	wt ppm
Ta	013	0.097	1.1	0.066	0.72	1.1		10.2	
Ta	016	4.2	45	0.24	2.6	1.2		3.6	
Nb	024	5.7	310 ^b	1.1	35	<1	220 ^c	2.7	1130 ^c
V	023	6.5	500 ^b	7.5		16	901 ^c	40	2360 ^c

^aVacuum distillation analysis of sodium indicated 1 to 2 wt ppm oxygen.

^bAnalysis by vacuum fusion.

^cAnalysis by Kjeldahl technique.

It should be noted that all of the wires picked up a considerable amount of nitrogen while the oxygen concentrations generally decreased. Since the solubility of nitrogen in sodium is generally believed to be quite low, it is possible that the sodium remains saturated with nitrogen and that the nitrogen increase in the wires results from a redistribution of nitrogen from the stainless steel container or the cover gas. If this is the case, the internal-friction peaks for nitrogen are not a measure of the nitrogen in the sodium but a measure of the nitrogen activity of the system. In either case these results indicate that extreme caution should be exercised in interpreting corrosion results of refractory metal specimens tested in stainless steel systems.

TABLE III.B.3. Thermal Diffusivity and Thermal Conductivity of Uranium Carbide

Temp (°C)	Thermal Diffusivity (cm ² sec ⁻¹)	Thermal Conductivity (cal sec ⁻¹ cm ⁻¹ °C ⁻¹)
500	0.062	0.050
600	0.058	0.048
700	0.056	0.047
800	0.055	0.047
900	0.055	0.047
1000	0.054	0.047
1100	0.053	0.047
1200	0.052	0.046
1300	0.051	0.045
1400	0.051	0.045
1500	0.051	0.045

d. Thermal-diffusivity Rig for Ceramic Materials. The thermal diffusivity of theoretically dense uranium monocarbide was measured between 500 and 1500°C. Thermal-conductivity values were calculated with the aid of published heat-capacity data.* The ANL results shown in Table III.B.3 are in general agreement with work done elsewhere.** The apparatus will now be

*Harrington, L. C., and Rowe, G. H., AEC Research and Development Report PWAC-426 (1964).

**Wheeler, M. J., in Carbides in Nuclear Energy (1963), p. 359.

used to determine diffusivities of carbides, sulfides, and phosphides of uranium and plutonium in the temperature range from 500 to 1500°C.

2. Chemistry of Irradiated Fuel Materials

a. Development of Analytical Facilities, Microsampling Techniques, and Analytical Procedures for Analysis of Irradiated Fuels. Equipment for the preparation and sampling of irradiated fuel is being designed, constructed, and tested for use in the helium-atmosphere enclosure of the shielded fuel-evaluation facility now under construction.

The preparation of appropriate samples for such analyses as alpha- and gamma-spectrometry and mass spectrometry will require the use of a variety of sampling techniques. The techniques being developed include laser-beam vaporization, microdrilling, and ultrasonic coring. Laser-beam vaporization provides samples ranging from 10^{-9} to 10^{-3} g, whereas microdrilling provides powdered samples of $\sim 10^{-4}$ g. The ultrasonic coring technique is capable of cutting out small sections of a sample matrix. This technique will be particularly useful in the preparation of high-burnup fuel for mass spectrometry or electron microprobe analysis when the fuel matrix must be left intact for further microareal sampling.

A Raytheon ultrasonic machining device (Model 2-334) is currently being tested to establish the necessary techniques for obtaining ceramic fuel core specimens. Boron carbide (B_4C) in tetradecane is being used as the abrasive slurry. Coring tools made from tubes with ID's of 0.032 and 0.045 in. produced UO_2 specimens with diameters of 0.030 and 0.040 in., respectively, at a cutting rate of >0.010 in./min. The maximum length-to-diameter ratio obtained was 4 to 1. After coring, the samples were broken loose from the matrix with a dissecting needle.

The examination of specific features of a fuel may require samples of varied shapes and sizes. Wedge-shaped samples have been cut satisfactorily from UO_2 sections with a "Y"-shaped tool. In addition, from 2 to 15 cylindrical cores have been cut simultaneously with a multitubed tool. It appears that samples of a wide variety of sizes and shapes can be produced provided that appropriate tools can be made.

Cathodic etching (ion bombardment) is being evaluated as a possible method of defining microstructure of irradiated fuels. An advantage of this etching technique is that the sample surface will not be contaminated with conventional acid etchants which may interfere with subsequent analyses.

Inactive materials are being used in preliminary tests to establish optimum conditions for etching of irradiated fuel specimens. Difficulties have been encountered in the etching of mounted samples owing to the

release of organic vapors from the plastic mounting materials. It is believed that this problem can be overcome by redesign of the equipment to avoid exposure of the mounting material during etching.

3. Thermodynamics of Fuel Materials

A major effort during the past six months has been devoted to converting the laboratory for the study of the thermodynamics of fuel materials at high temperatures to one capable of carrying out such studies on plutonium-bearing fuels. In essence, numerous experimental techniques--previously employed in mass-spectrometric, transpiration, effusion, and phase-diagram studies--have been adapted to glovebox operation by the modification of existing equipment and the design of new equipment. The current work concerning mass spectrometry is discussed below.

a. Mass-spectrometric Studies of Plutonium-bearing Systems.

Mass-spectrometric studies of the evaporative behavior of plutonium-bearing systems (e.g., Pu-O, U-Pu-O, Pu-C, and U-Pu-C) are being undertaken to determine (1) the vapor species in equilibrium with the condensed phase(s), (2) the partial pressures of the vapor species as a function of temperature, and (3) the thermodynamic parameters of the system.

For these studies, a glovebox for use with plutonium was coupled to the source end of a Bendix TOF mass spectrometer. The glovebox operates with a high-purity helium atmosphere (<5 ppm H_2O , <4 ppm O_2) at a slightly negative pressure. The helium-purification system was designed with the capacity to serve two additional gloveboxes (for transpiration and effusion studies) to be installed at a later date.

During shakedown tests of the glovebox-mass spectrometer facility, arcing between high-voltage leads (-3000 V dc) and the source-header plate (ground potential) of the mass spectrometer occurred through the helium atmosphere. Also, continuous outgassing of nitrogen from the Knudsen-cell tower assembly was observed when it was heated in vacuum.

The arcing was traced to faulty insulation. The leads had been potted to the header with RTV-102, but, presumably, voids had been left between the high-voltage leads and source-header plate. To prevent the arcing, the source header was coated with Torr Seal (an epoxy resin), and the high-voltage leads were repotted to the header with RTV-102. This arrangement has proven satisfactory.

A Knudsen-cell tower assembly, adapted from a design of Rauh et al.,* was modified to expedite glovebox operations such as filament replacement, effusion-cell alignment, and sample loading. Heat shields and filament-support rods in the hot zone are made of tantalum, the effusion-cell support rods of tungsten. The upper supports and various insulating bushings

*Rauh, E. G., Sadler, R. C., and Thorn, R. J., High-temperature Knudsen Effusion Cell Assembly, ANL-6536 (April 1962).

were made of boron nitride. Problems of outgassing of the Knudsen-cell tower assembly was traced to this material which, on reaching a temperature of $\sim 800^{\circ}\text{C}$, reacted with adjacent metals to liberate nitrogen. By replacing the insulating bushings with quartz and the upper support posts with steatite, the need for outgassing was eliminated and the assembly could be operated at effusion-cell temperatures up to 2200°C .

Final testing of the glovebox-mass spectrometer facility has been completed satisfactorily, and mass-spectrometric studies of the evaporation of plutonia are under way.

4. Basic Fabricability

a. Determination of Elastic Moduli of High-temperature Materials by Ultrasonics. Ultrasonic-velocity data were compiled for four Type 304 stainless steel specimens furnished by Reactor Engineering; the elastic moduli of the material are now being calculated. An analysis of the information will be made after all calculated results have been obtained.

A tungsten specimen is now being fabricated that will make possible the testing of tungsten up to 2500°C . This specimen will take some time to fabricate because of the material.

b. Development of a Neutron-image Intensification System. The 6-in. neutron-image intensifier tube was re-evaluated for light yield as a function of incident neutron intensity after 18 months of service. The tube still operates at the same lens setting on the vidicon camera as when first placed in service. Light measurements also confirmed that the light yield of the tube had changed only within reasonable limits for the particular photoemissive surface used in the tube. The total estimated neutron exposure for the tube is in the range of 300 hr. The conclusion is that no degradation in tube characteristics could be detected for this neutron exposure and tube life.

c. Suppression and Reduction of Noise in Electromagnetic Test Systems.

(i) The Diffusion of Pulsed Electromagnetic Fields into Good Conductors. The pulsed electromagnetic test method offers a promising solution to several important problems associated with the nondestructive testing of fast reactor components. Its application has been handicapped, however, by a lack of basic knowledge of the characteristics of pulsed current flow in good conductors. An analysis of the diffusion of a pulsed electromagnetic field into a plane conductor with the magnetic field vector arbitrarily oriented with respect to the direction of propagation has apparently never been undertaken. This means that there exists no firm theoretical basis for optimizing the transducer design and test conditions of pulsed electromagnetic test systems.

As a first approach to this problem, a plane-wave analysis was undertaken for a field having a square-wave time variation.* The pulse-current waveforms in the metal calculated from the results of this analysis agreed fairly well with experimental results, but the calculated attenuation of the field for a given thickness of metal was high by several orders of magnitude. In fact, results from the plane-wave case could have been used to show that the pulsed electromagnetic method is simply not feasible even though it has been successfully used for several years. The serious discrepancy in calculated attenuation indicated that the assumptions of the analysis had to be modified to approximate more closely the conditions existing in the actual test equipment.

Unfortunately, even modest changes of the assumed boundary conditions in the direction of realism seriously complicate the analysis. A solution was sought in a form that would allow an understanding of the wave phenomena involved. A series solution depending upon a digital computer for evaluation could be only of limited usefulness if the separate terms of the series had no clear identity. In order to identify the factors that caused the plane-wave results to depart so seriously from experimental determinations of field attenuation, the next attempt involved assuming a component of magnetic field perpendicular to the surface of a plane semi-infinite conductor. In order to achieve further simplification, a unit impulse in time was chosen for the driving function. Convolution can then be used to obtain the response for more complicated driving functions.

The analysis was two-dimensional; later it is hoped that this can be extended into three dimensions.

From Maxwell's equations it can be shown that

$$\nabla^2 \bar{\Pi} - \mu s(\sigma + \epsilon s) \bar{\Pi} = 0, \quad (1)$$

where

σ = conductivity of the conductor;

ϵ = permittivity of the conductor;

μ = permeability of the conductor;

s = complex variable as used in the Laplace transform;

$\bar{\Pi}$ = Laplace transform of the vector potential.

*Cox, C. W., and Renken, C. J., The Use of the Conducting Mask in Pulsed Electromagnetic Testing, ANL-7172 (to be published).

The y axis is taken as pointed out of the paper and it is assumed that there is no variation with y . The xy plane is the surface of the metal. The magnetic field is an odd function of x and varies as e^{-x/x_0} , where x_0 is a constant with the dimensions of length. The vector potential would then have a component only in the y direction.

Neglecting displacement current, (1) becomes

$$\frac{\partial^2 \bar{\Pi}}{\partial x^2} + \frac{\partial^2 \bar{\Pi}}{\partial z^2} = \sigma \mu s \bar{\Pi}, \quad (2)$$

which has a solution

$$\bar{\Pi} = \int_0^\infty A(\alpha) e^{-\gamma Z} (\cos \alpha x) d\alpha; \quad (3)$$

α is the eigenvalue. The coefficient $A(\alpha)$ must be determined from the boundary conditions; further,

$$\gamma = \sqrt{\sigma \mu s + \alpha^2}.$$

The Laplace transform of the magnetic field $\bar{H}$ is obtained from $\bar{H} = \sigma \nabla \times \bar{\Pi}$:

$$\bar{H}_x = -\sigma \frac{\partial \bar{\Pi}}{\partial Z} = \sigma \int_0^\infty \alpha A(\alpha) e^{-\gamma Z} \cos(\alpha x) dx; \quad (4)$$

$$\bar{H}_Z = \sigma \frac{\partial \bar{\Pi}}{\partial x} = -\sigma \int_0^\infty \alpha A(\alpha) e^{-\gamma Z} \sin(\alpha x) dx; \quad (5)$$

The Laplace transform of the electric field is obtained from $\bar{E} = \nabla(\nabla \cdot \bar{\Pi}) - \sigma \mu s \bar{\Pi}$, and

$$\bar{E}_y = -\sigma \mu s \int_0^\infty A(\alpha) e^{-\gamma Z} \cos(\alpha x) d\alpha. \quad (6)$$

The other field components are zero.

At the surface of the metal,

$$\bar{H}_Z|_{Z=0} = e^{-x/x_0} = -\sigma \int_0^\infty \alpha A(\alpha) \sin(\alpha x) dx. \quad (7)$$

If this is recognized as the Fourier transform for an odd function of α , then it can be shown that

$$\bar{\Pi} = \frac{-2}{\pi\sigma} \int_0^{\infty} \frac{e^{-Z\sqrt{\sigma\mu s + \alpha^2}}}{\alpha^2 + (1/x_0)^2} \cos \alpha x d\alpha. \quad (8)$$

Evaluation of this integral leads to the following expression for π , the vector potential expressed as a function of time:

$$\begin{aligned} \pi = & \frac{-x_0 Z \sqrt{(\mu/\sigma)}}{4\pi^{1/2} t^{3/2}} e^{-\frac{\sigma\mu Z^2}{4t}} e^{\frac{t}{\sigma\mu x_0^2}} \left[e^{-x/x_0} \operatorname{erfc} \left\{ 1/x_0 \sqrt{\frac{t}{\sigma\mu}} - x/2 \sqrt{\frac{\sigma\mu}{t}} \right\} \right. \\ & \left. + e^{x/x_0} \operatorname{erfc} \left\{ 1/x_0 \sqrt{\frac{t}{\sigma\mu}} + \frac{x}{2} \sqrt{\frac{\sigma\mu}{t}} \right\} \right]. \end{aligned} \quad (9)$$

The magnetic field in the Z direction is obtained from Eq. (5):

$$H_Z = \frac{V e^{-V^2} e^{w^2}}{2\pi^{1/2} t} \left[e^{-2uw} \operatorname{erfc}(w - u) - e^{2uw} \operatorname{erfc}(w + u) \right], \quad (10)$$

where

$$u = \frac{x}{2} \sqrt{\frac{\sigma\mu}{t}}; \quad V = \frac{Z}{2} \sqrt{\frac{\sigma\mu}{t}}; \quad w = 1/x_0 \sqrt{\frac{t}{\sigma\mu}}.$$

Equation (10) gives the vertical magnetic field in the metal for a unit time impulse of vertical magnetic field at the metal surface having an exponential decrease of the field in the x direction.

For very large values of x , it can be shown that Eq. (10) reduces to an expression identical to that obtained for the magnetic field parallel to the metal surface in the plane-wave case. This rather surprising result seems to indicate that the boundary conditions are critical in this problem. The discrepancy in the plane-wave analysis is not associated so much with the behavior of the field in the metal, but rather the extremely high air-to-metal reflection coefficient that analysis produces. Further analysis of the behavior of a vertical field at the air-to-metal boundary is planned.

(ii) Spectra of Transducers Used with Pulsed Electromagnetic Test Systems. The frequency spectra of both desirable signals and noise are dependent upon the resolution characteristics of the electromagnetic transducer used in the system. In order to calculate the frequency spectra, the time-domain response of the transducer to various conditions in the test specimen must be known.

An insight to the problem of this response can be obtained by considering the response of a transducer to a hypothetical impulse defect. If the response of an ideal transducer to such a defect is given by $B(x)$ (its transfer function), the response of the transducer to any defect that can be formed from an infinite series of impulse defects of various strengths is given by

$$g(t') = \int_{-\infty}^{t'} B(x) f(x' - x) dx,$$

where

x = distance along the path of the defect through the transducer field;

$x' = at'$;

a = scanning rate;

$f(x' - x)$ = a function expressing the variation in defect strength as a function of the distance x .

Unfortunately, a real transducer does not have a single transfer function, but rather an infinite number of different transfer functions that depend upon the shape of the transducer field and its time variation. Nevertheless, the concept of the transducer transfer function is a useful one for comparing the merits and performance of different transducers. An idea of the effective transfer function of a certain transducer along a given path through its field can be obtained by causing the transducer to scan a discontinuity in the metal that is much smaller than the effective field of the transducer. Variations in the test-system output will then trace the shape of the transducer transfer function under the chosen test conditions. The spectral content of the transfer function can then be calculated using the Fourier integral. Two transducer transfer functions and their calculated spectra are shown in Figs. III.B.1 through III.B.4. The solenoid used for the response

Fig. III.B.1. A Typical Transfer Function for a 0.058-in. Ferrite-cored Solenoid Taken along Its Axis

Fig. III.B.2. A Typical Transfer Function for a Mask Aperture Assembly with a 0.070-in. Aperture

Fig. III.B.3. Calculated Spectrum for the Function of Fig. 9

Fig. III.B.4. Calculated Spectrum for the Function of Fig. 10

curve of Fig. III.B.1 is about as small as can be made to work with most test systems. Its spectrum is shown in Fig. III.B.3. The larger transducers used with most present-day test equipment would have been poorer high-frequency response. A mask-aperture assembly with a 0.070-in. aperture produced the transfer function shown in Fig. III.B.2. Its spectral response (see Fig. III.B.4) is considerably better than that of the solenoid at the higher frequencies. How much both lack a proper high-frequency response can be judged by comparing the spectra to that of a small defect plotted to the same frequency scale but an arbitrary amplitude scale in Fig. III.B.4. The first zero of the defect is far off the high-frequency edge of the graph. This comparison graphically indicates the central problem of the application of the electromagnetic method to problems of defect detection.

Defect signals can usually be considered to be time limited, but noise is more likely to be continuous. Quite often, the noise is roughly periodic. This periodicity originates in the manufacturing process; for example, a reeling operation often used to remove a drawn tube from a hard mandrel. If the noise is roughly periodic, there is a chance that considerable improvement in signal-to-noise ratio can be attained by filtering. The noise-frequency spectrum would tend to appear as a series of discrete lines of various amplitudes at multiples of the basic rate of noise repetition whereas the defect spectrum would be continuous like those shown in Figs. III.B.3 and III.B.4.

d. Measurement of Thermal Conductivity of Irradiated Fuel as a Function of Burnup and Temperature. Thermal diffusivities and conductivities of a number of specimens from three types of EBR-II U-5 w/o Fs irradiated fuel pins have been measured. Calculations of the conductivity, burnup, and swelling for each of these specimens are being made.

e. Development of a Nonreactor Neutron Source for Radiography.

Initial results with an Am-Cm-Be neutron source, having a total yield of the order of 10^7 n/sec, have been obtained in a water moderator. An infinite water moderator thickness (equivalent to a 20-cm radius of beryllium), was used for computer calculation. Initial results indicate a peak in thermal-neutron flux in water at 1.25 cm from the center of the source; the peak value is of the order of 1.5×10^5 n/cm²-sec. Both values are in agreement with the computer prediction. A cadmium ratio, as determined with gold foils and a 0.5-mm cadmium cover, of 8.5 was found, a value which is now being checked with the computer calculation.

Some leakage intensities with the source in place are now being obtained. It is also planned to bring a neutron beam out of the water-moderator assembly in order to determine useful beam neutron intensities, and the gamma-radiation intensity.

A separate cave facility is now being arranged to begin the assembly of the BeO moderator.

5. Engineering Properties of Reactor Materials

a. Preparation of (U, Pu)O₂ Specimens. Solid-solution (U, Pu)O₂ specimens with good homogeneity and accurately known composition for use in equilibration experiments are being fabricated. The knowledge gained in making these specimens will also be applied to the fabrication of oxide specimens for future measurements of mechanical properties.

Specimens having a U/Pu ratio of 80/20 were made from powders that were physically mixed by ball milling in alcohol for $3\frac{1}{2}$ hr. The oxygen-to-metal (O/M) ratio of urania was 2.08, and that of the plutonia was 2.00. After mixing, the powder was dried and pressed at 48,000 psi into pellets 7.11 mm in diameter.

The optimum sintering temperature and atmosphere are being determined. Specimens were sintered for 4 hr at temperatures from 1510 to 1560°C in atmospheres of dry hydrogen, helium-6 v/o hydrogen, and helium-6 v/o hydrogen containing water vapor (dew point = -17°C). Final O/M ratios of 1.93, 1.97, and 2.00 were obtained for specimens sintered in H₂, He-H₂, and He-H₂-H₂O, respectively. Sintered densities ranged from 87 to 92% of the X-ray value.

The preliminary data obtained thus far are insufficient to establish the influence, if any, of composition to the homogeneity and sintering behavior of physically mixed (U, Pu) oxide specimens. Additional sintering experiments are therefore in progress.

C. Engineering Development

1. Development of Master-Slave Manipulator Systems

a. Electric Master-Slave Manipulator, Mark E4A. The heat tests of the slave arm of the Mark E4A (see Progress Report for August 1967, ANL-7371, pp. 89-90) have been continued. Some tests were made at a higher ambient temperature, 33°C, instead of 28.5°C. The plot of temperature and/or force versus time, show some irregularities. Before any conclusions are drawn, the tests will be repeated and the troubles, either in the test procedure or the Mark E4A itself, found. Further heat tests and "motion exercising" tests are to be carried out during October.

b. Low-inertia Servo Motors for Manipulators. The special 2-pole, 2-phase low-inertia motor has been tested further. The stalled torque has been measured as a function of control field voltages for a few different fixed field voltages with constant airflow being forced through the rotor and stator, as well as for different air and argon flow rates through the motor.

The tests show that the motor can supply sufficient torque for a 100-lb manipulator for continuous duty when cooled with either air or argon. For argon, the flow-rate pressure drop in the motor is considerably higher than for air. More cooling tests will be made in October, using both air and argon under different inlet and outlet temperatures.

Starting with the motor cool, a rapid drop in torque occurs as a function of time after constant voltages have been applied to both the control-field and fixed-field windings. This is undesirable because the servo stiffness would have to be set for the cool motor and would drop off as the motor heated. The trouble seems to be in the rotor. Different rotor arrangements are being studied to alleviate this problem.

Further studies on preliminary designs are being carried out on shell dc motors. The prospects for this type of motor for electric master-slave manipulators looks good.

2. Instrumentation and Control

a. Boiling Detector. A successful boiling detector will enable high-power-density fast reactors to operate without overheating the fuel and causing costly meltdowns. Acoustic frequency, neutron-flux noise, liquid thermocouple, and other techniques for detecting sodium boiling are being investigated.

Crystals of lithium niobate, tourmaline, and quartz have been selected for tests to develop the boiling-detector specifications. Significant parameters of these crystals are: (1) geometrical shape and size, which determines detector sensitivity and frequency response, and (2) crystallographic orientation, which determines the response to longitudinal and shear waves.

b. Vibration Sensor. A vibration transducer of the accelerometer or low-frequency sound type will be developed and tested for use in a fast-reactor core environment involving 1200°F sodium and high radiation intensities. Sensor outputs are to be cross-correlated with neutron-flux signals. The objective is to provide a means to diagnose fuel motion, core-structure vibration, or control-element flutter that might disturb overall reactor stability.

An exciter for calibration of accelerometers with vibration frequencies from 50 to 50,000 cps has been delivered. During acceptance tests, a defective accelerometer was discovered; its resonance frequency was 30 kc/sec instead of the specified 120 kc/sec. The exciter also will be used to evaluate mounting techniques for accelerometers for boiling detection.

3. General Heat Transfer

This program is investigating the boiling and two-phase behavior of liquid metals in forced-convection flow, improved methods for predicting temperature in reactor coolant channels and for designing liquid-metal heat exchangers and steam generators, and heat-transfer and flow characteristics of highly dispersed mixtures of liquid and vapor. The objective is to improve the fundamental techniques by which liquid-metal-cooled reactors can be designed for better performance.

a. Boiling-sodium Experiment. The heat-transfer and two-phase flow characteristics of boiling sodium are being investigated to temperatures of 2100°F and pressures of approximately 8 atm in an effort to provide the understanding of liquid-vapor behavior that is essential for analyzing the steady-state behavior and the safety of sodium-cooled fast reactors for large central-station power plants. Among the variables to be investigated are boiling heat flux and temperature difference up to the occurrence of critical heat flux, boiling and adiabatic two-phase pressure losses, vapor volume fraction, boiling stability parameters, and ultimately the transient behavior of some of the same quantities.

(i) Niobium-1% Zirconium Loop. The principal research tool consists of a Nb-1% Zr loop that is housed in a chamber capable of being evacuated to 10^{-7} - 10^{-8} Torr for protection against oxidation at high

temperature. The loop, which is constructed of 0.5-in. tubing, has a 25-ft/sec sodium flow and a 10^6 -Btu/hr-ft² heat-flux capability from a thermal-radiation heater.

Five short-term (<20 sec) boiling runs have been completed at relatively low temperatures in the following variable ranges:

Boiler outlet temperature	1600-1655°F
Subcooling	160-230°F
Heat flux	$1.5-2.3 \times 10^5$ Btu/hr-ft ²
Boiler inlet velocity	2-5 ft/sec
Boiler outlet pressure	17-21 psi

Boiling was initiated by setting the boiler power and flow rate to fixed values, allowing steady-state conditions to obtain, and then decreasing system pressure until boiling occurred. In all five runs, subcooled boiling occurred, as indicated by an excess of boiler outlet pressure over the saturation pressure corresponding to boiler outlet temperature. Stable boiling, in the sense of steady two-phase flow, was not obtained due to rapid expansion of the sodium vapor, which forced hot sodium through the fill line and dump-tank valve. This produced severe oscillations in flow rate, such that the boiler section was voided in irregular cycles. The thermal-radiation boiler can withstand the subsequent temperature excursions for a short time (a few seconds); however, since the oscillations showed no sign of damping out during runs of up to 20-sec duration, the runs were terminated. These data are being analyzed. Subsequent runs will be conducted at gradually increasing temperatures to map the regions of stable and unstable boiling.

After these initial runs, the facility was shut down to install instrumentation for continuous recording of flow, pressure, and temperature in the observed range of oscillations during the initiation of boiling.

b. Liquid-metal Heat Exchangers. Heat-transfer analyses based on the use of fundamentals are being performed and compared with results obtained from empirical heat transfer coefficients and experimental data. The objectives are to determine the accuracy of traditional engineering methods for predicting heat-transfer rates and temperature distributions in liquid-metal heat exchangers; and to find improved prediction methods.

(i) Nonsymmetrical-duct Heat Exchangers. Construction of the small sodium heat exchanger loop continues. The electrical sodium heaters have been completed, but leaktesting revealed several faulty welds. A procedure which requires a special jig has been worked out

for accurately locating and attaching thermocouples to the first test section, a double-pipe heat exchanger with continuously variable eccentric placement of the inner tube.

(ii) Liquid-metal-heated Steam Generators. Design work continues for the loop to be used for experiments on simple simulations of liquid-metal-heated steam generators. The liquid metal will be mercury; the fluid on the "steam side" most likely will be Freon-11. Loop design is based on the use of equipment from the earlier double-pipe heat-exchanger experiments and on choosing operating conditions that will resemble those in typical sodium-heated steam generators for the LMFBR program; for example, the range of operating pressure of the Freon has been chosen so that the ratio of the specific volumes of vapor and liquid will be simulated.

4. Engineering Mechanics

Bases for design methods are being developed to provide estimates of the various facets of component dynamic response to transient loads, energy-transfer mechanisms, system influence on stability and flow, and the influence of structural anisotropy on thermoelasticity. The information generated will assist in the selection of reactor core materials, component configurations, and clearance specifications, as well as have the potential of providing models for reactivity-feedback mechanisms related to hydraulic and thermally induced core displacements. This theoretical and experimental program is also expected to have application to investigations of improved design criteria for reactor vessels and containment structures.

a. Anisotropic Thermoelasticity. Refined analytical methods are being developed for determining thermally induced displacements with application to improving capabilities for the design of fast-reactor core components. For this, a study is being made of the significance of thermal distortions caused by direction-dependent effects introduced by the intrinsic anisotropic properties of fuel-element materials and by the effective anisotropic properties that describe the way regular substructure patterns influence larger structural elements. Models are being developed to describe the overall deflection of thermally loaded fuel-element assemblies and entire core structures.

The problem of isotropic transient thermoelasticity that was chosen to determine the relationship between the rise times for the temperature and stress fields caused by the sudden initiation of internal heat generation in a circular cylinder has been completed (see Progress Report for July 1967, ANL-7357, pp. 118-121). Numerical results were obtained in all cases of current interest. Without exception, the maximum stress magnitudes in the transient problem were bounded from above by the steady-state solution.

D. Chemistry and Chemical Separations

1. Fluoride Volatility Processes

a. Recovery of Uranium and Plutonium from Low-enrichment Fuels: Laboratory Support Work

(i) Fission Product Fluoride Chemistry: Chemistry of Ruthenium Fluorides. As part of a study to determine ruthenium behavior under reaction conditions simulating those used in fluoride volatility processing, an experiment was performed in which a sample of RuO_2 was contacted with BrF_5 and then with F_2 . The sample, tagged with Ru^{106} , was contained in a nickel boat in a horizontal $2\frac{1}{2}$ -in.-dia, 16-in.-long reactor. The BrF_5 and F_2 steps were carried out using a gas phase containing about 20 v/o BrF_5 in nitrogen and 100% F_2 , respectively. The BrF_5 step was performed at 250°C and the F_2 step at 300 - 550°C . The off-gas (which contained volatile ruthenium species) flowed through a NaF trap and a soda lime trap in series. The contents of the NaF trap and soda lime trap were replaced after each step.

The reaction rate with BrF_5 was initially high, resulting in reaction with about 70% of the RuO_2 , after which the rate was greatly diminished; the subsequent reaction with fluorine was also slow. The solid residue after the BrF_5 step was hygroscopic and had a sharp HF -like odor suggesting the presence of RuF_4 , the complex $\text{BrF}_5 \cdot \text{RuF}_5$, or both. The reaction of RuO_2 with BrF_5 was somewhat faster than that of ruthenium metal with BrF_5 . The rate constant for the high-rate portion of the BrF_5 step of the RuO_2 experiment was 0.018 min^{-1} , whereas the corresponding rate constant for the $\text{Ru}-\text{BrF}_5$ reaction at 300°C was about 0.012 min^{-1} .

The NaF absorber trap was maintained at about 100°C instead of 400°C (used in previous experiments) to test the efficiency of ruthenium retention at the lower temperature. For the BrF_5 step, the retention of ruthenium on the NaF , as measured by the ratio of activities in the NaF and soda lime traps, was as effective at 100°C as that previously observed with the NaF trap at 400°C (activity ratio of about 2000:1); for the fluorine step, retention by the NaF trap at 100°C (activity ratio of 2:1) was not as effective as in previous work when the NaF trap was maintained at 400°C .

In other work, X-ray diffraction analyses were performed on NaF samples taken from an off-gas trap following a run in which ruthenium metal was fluorinated with BrF_5 at 250°C and then with F_2 at 300 - 550°C , with the trap at 400°C . The volatile ruthenium species was probably RuF_5 . The results indicate that the compound found on the NaF matrix is most likely to be Na_2RuF_6 .

A study of the chemistry of RuF_6 is being made to assist in the identification of the ruthenium species formed in fluid-bed fluoride volatility processes. Preliminary experiments on the reaction between RuF_6 and NaF were performed in a static system at 100°C . The RuF_6 pressure was 50 mm Hg and the reaction time was 2 hr. X-ray diffraction analysis of the solid product indicated that the ruthenium species was probably Na_2RuF_6 , the same compound as was reported formed when RuF_5 was adsorbed on NaF at 400°C (see above).

b. Recovery of Uranium and Plutonium from Low-enrichment Fuels: Engineering Work.

(i) Engineering-scale Development for UO_2 - PuO_2 Fuel: Engineering-scale Alpha Facility. The major objectives of experiments conducted in the engineering-scale alpha facility are to demonstrate handling of PuF_6 on an engineering scale and to determine the feasibility of fluoride volatility flowsheets. The facility comprises two large alpha boxes, one containing process equipment, and the other containing scrubbers and auxiliary equipment. The process equipment includes a fluid-bed fluorinator for fluorinating mixed oxide fuel to UF_6 and PuF_6 , a converter reactor for converting PuF_6 to PuF_4 by thermal decomposition or for converting mixed UF_6 and PuF_6 to mixed oxide, and a system of condensers and chemical traps for collecting hexafluoride products.

Campaign-type fluorination experiments are in progress aimed at further demonstrating that PuF_6 can be transferred from one vessel to another on a practical scale (see Progress Report for August 1967, ANL-7371, p. 94). The first of three planned campaigns has been completed successfully. It consisted of three successive fluorination experiments (Runs Pu-17, -18, and -19) and a cleanup-fluorination experiment (Run Pu-20) to recover PuF_4 deposited in the lines and equipment. A single alumina bed, consisting of 6500 g of 48-100 mesh T-61 Alcoa alumina, was used.

In each of the runs of the first campaign, approximately 200 g of plutonium as PuF_4 powder was added to the prefluorinated alumina bed and the bed was heated to 300°C . A flow of 90 v/o fluorine was then started, and the temperature was programmed to increase from 300 to 550°C over a period of 5 hr; the PuF_6 formed was collected in cold traps. The PuF_6 was transferred to NaF traps after each fluorination. Plutonium deposited on the primary filters was recovered at 300°C in the latter part of Run Pu-19. In cleanup-fluorination Run Pu-20, the secondary filter, cold traps, and connecting lines were contacted with recycling fluorine at 300°C to recover the plutonium deposited by radiation decomposition.

Analysis of grab samples of the fluidized bed taken at the end of each fluorination experiment indicated that plutonium removals had been 99.4, 99.5, and 99.7%. The final dumped bed contained 0.01 w/o plutonium, indicating that overall plutonium removal had been 99.9%.

In the next two campaigns, the starting bed fluorination temperatures will be 375 and 450°C. After completion of these campaigns and analysis of NaF samples for plutonium, fluorination efficiencies and PuF_6 production rates will be compared.

Neutron monitors (PNC-1 meters with standard BF_3 tubes, output fed to a multichannel, multivolt strip recorder) were used in this series of runs to record the movement of PuF_6 from the reactor to the cold trap during fluorination and from the cold trap to the NaF traps during PuF_6 transfer. The neutron probes have been a useful tool for qualitatively monitoring the fluorination rate during the fluorination period, and the time interval for transfer of PuF_6 to the NaF traps.

Additional information has been received on ruthenium decontamination in thermal-decomposition Runs DUP-5 and DUP-6 (see Progress Reports for November 1965, ANL-7122, pp. 63-65, through May 1966, ANL-7219, pp. 51-52, and for May 1967, ANL-7342, pp. 100-101). In these runs, plutonium was separated from a UF_6 - PuF_6 mixture by thermally decomposing the PuF_6 to solid PuF_4 , which deposited on fluidized alumina particles; the volatile UF_6 passed through the thermal decomposer and was collected separately. The PuF_4 -bearing alumina bed used for both thermal-decomposition runs has recently been analyzed for ruthenium at Rocky Flats by means of a spark-source mass spectrometer. The bed was found to contain 0.0008 g ruthenium, whereas the hexafluoride feed contained 5.2 ± 2.6 g of ruthenium. The decontamination factor for ruthenium based on these data is 10^3 - 10^4 . Further examination of thermal decomposition as a means of purifying PuF_6 streams from ruthenium is suggested.

(ii) Small-scale Studies with Irradiated Fuels. As part of an investigation of the processing of irradiated fuels by fluoride volatility methods, procedures are being developed for removing the reactive components of effluent gas streams in small experiments. Several runs were completed in which soda lime was tested for the removal of HCl and F_2 from a flowing stream of nitrogen. When the 4-in.-dia, 1-kg soda lime bed was heated to 150°C prior to introduction of the gas mixture, the soda lime was a satisfactory reagent for 25 v/o HCl in N_2 at 4 liters/min. The final bed was uncaked and free-flowing; the bed capacity was 0.20 g HCl /g soda lime. Soda lime was unsatisfactory for the disposal of 90 v/o F_2 in N_2 at 400 ml/min; the final beds were badly caked.

Established sampling techniques, such as splitting, grinding, and milling, have generally been used for sampling nonhomogeneous solids or beds of solid particles that are being studied in the development of fluoride volatility processes. Although the results obtained by use of these techniques have been acceptable, some of these techniques may lead to sampling biases. Experimental work has been undertaken to develop improved procedures. Results of the studies are as follows:

(1) As the number of splittings of a sample of -14 +20 mesh alumina was increased (2, 4, or 6 splits), sampling accuracy decreased rapidly.

(2) A 5.1-kg mixture of 0.1 w/o irradiated -100 mesh ZrO_2 , ~10 w/o -100 mesh T-61 alumina, ~60 w/o -40 +100 mesh T-61 alumina, and ~30 w/o -14 +28 mesh alumina was split to 20-g samples by means of a large splitter and a small splitter. The counting rates of the samples were obtained, and then the samples were recombined into the original mixture and resplit, for a total of four runs. Large losses of fine material from run to run--reflected by losses of activity up to 10%--were introduced in sampling the mixture of particles and powder. This loss resulted from adherence of powder to the surfaces of the splitters and from entrainment in the ventilation air flowing past the splitter mouth. This suggests a sampling bias that would tend to produce low results in material balances for those activities which accumulate as fine particulates.

(3) A tube sampler for reducing sample size from ~50 g to ~1 g was tested. It consists of an 8-oz polyethylene bottle with a lid in which a sliding copper tube is mounted. A 50-g sample is placed in the bottle and mixed; then the copper tube is pushed to the bottom of the bottle and a 1-g sample is poured out of the tube by inverting the bottle. Tests were made with a mixture of ~2 w/o -100 mesh irradiated ZrO_2 and ~98% -100 mesh alumina. For 11 tests, the coefficient of variation was 1.3%.

(iii) General Behavior of Fluid Beds. In the oxidation of uranium dioxide pellet fuel, fine particles of U_3O_8 are produced. Since the fluidization of fine particles is frequently made difficult by the tendency of the particles to aggregate or by channeling of the gas through the bed, convenient methods for determining the onset of fluidization, the quality of fluidization, and other characteristics of fluidized beds are being developed.

In fine-particle fluidization studies, measurements of pressure drop and heat transfer coefficient are commonly used to describe the quality of fluidization. The possibility of describing fluid-bed behavior by the sounds and mechanical vibrations generated is being investigated to supplement the above methods.

A 3-in.-dia column has been used for the fluidization studies. The column is made of conducting glass, which conducts away undesired static charges. Two devices have been used: a contact microphone with an amplifier circuit and a vibration detector with a separate amplifier circuit. Preliminary results indicated that the amplitude of the acoustical or vibrational wave is related linearly to the value of $v_s - v_m$ (superficial gas velocity minus minimum fluidization velocity) on a log-log plot. It also was observed that the slope of the line varies with the particle size used. Vibrational or sound amplitude values are considered preferable to pressure drop for indicating the quality of fluidization.

2. Compact Pyrochemical Processes

Studies are being made of a pyrochemical process for ceramic (e.g., oxide or carbide) fast breeder reactor fuels clad with stainless steel. The process applies salt-transport separations and liquid metal-molten salt extractions to the separation of fissile and fertile fuel constituents from the fission products. A conceptual flowsheet for the process has been described previously (see Progress Report for September 1966, ANL-7255, p. 30).

a. Process Chemistry of Molten Salt Systems. A key step in the current pyrochemical process is the separation of rare earth fission products from plutonium. Large differences in the distribution coefficients (ratio of w/o in salt phase to w/o in metal phase) of plutonium and the rare earths are obtained when Cu-Mg alloys are used as the liquid metal phase in combination with a $MgCl_2$ -based salt phase. Because the rare earths distribute strongly to the salt phase, even minor contamination of liquid metal samples by the salt can lead to erroneous distribution coefficients. This difficulty has been resolved by a new technique that permits the withdrawal of liquid metal samples without exposing them to the salt. This is accomplished by means of a tube which is mounted on the inner wall of the containment vessel and extends below the salt-metal interface. By taking liquid metal samples from within this tube, the salt phase can be excluded.

The new sampling procedure was employed to obtain distribution coefficients of cerium between a liquid Cu-33 w/o Mg alloy and a molten 50 m/o $MgCl_2$ -30 m/o NaCl-20 m/o KCl salt mixture at 600, 700, and 800°C; the resulting distribution coefficients were 580, 480, and 250, respectively, and the corresponding cerium-plutonium separation factors are 1150, 1080, and 610. These values suggest that high decontamination of plutonium should be possible.

b. Pilot-plant Operations: Small-scale (199 g Pu, 458 g UO_2) Salt-transport Experiment. Additional analytical results have been received for a small-scale experiment (Pu STP-1) in which the steps of the current pyrochemical process flowsheet were performed sequentially.

The simulated fast breeder reactor core fuel charged to the experiment consisted of 199 g of plutonium as PuO_2 (99 g of which would be expected to remain in the process as in-process inventory), 458 g of UO_2 , and 24 inactive fission product elements having a total weight of 52.4 g.

In the reduction step of the process, oxide fuel, suspended in a 47.5 m/o MgCl_2 -47.5 m/o CaCl_2 -5 m/o CaF_2 salt mixture, is reduced by a Cu-33 w/o Mg alloy. The reduction step in this experiment was carried out at 800°C for 4 hr with a mixing speed of 600 rpm. Reductions of ~99 and ~99.9% were achieved for uranium and plutonium, respectively. The magnesium oxide loading in the salt after the reduction was about 12 w/o. A 100% material balance was obtained for plutonium in the reduction step. The plutonium distribution coefficient (ratio w/o plutonium in salt to w/o plutonium in metal) was 0.33, which agrees with previously determined experimental values.

Two scrub steps were carried out in which the metal alloy from the reduction step was contacted with additional reduction salt to provide additional rare earth removal from the plutonium. Each of these two steps was conducted at 800°C for 2.5 hr with a mixing speed of 450 rpm. The observed plutonium distribution coefficient in these steps was 0.37, which also agrees with a previously determined value. Removals of 97-99% of the major rare earth elements (Pr, La, Nd, and Ce) were achieved (see Table III.D.1). It is believed that these removals could be improved to values of ~99.9% in future experiments through modification of operating procedures.

TABLE III.D.1. Fission Product Removals

(From spark-source mass-spectrometric analysis of plutonium-product solution, Zn-4 w/o Mg-2.7 w/o Pu)

Element	Removal (%)	Element	Removal (%)
Ag	>98	Pd	>99.9
Ba	>99.5	Pr	97
Cd	^a	Rb	>99
Ce	98.5	Rh	>99.9
Cs	>99.5	Ru	99.5
Eu	>98	Sb	^a
Gd	^a	Se	^a
In	^a	Sm	>99.7
La	97	Sn	^a
Mo	>99.9	Sr	^a
Nb	^a	Y	^a
Nd	97	Zr	>96

^aIndeterminant until product solution is retorted and product is sampled and analyzed.

Two plutonium strip steps were performed to remove plutonium from the waste reduction salt. Calcium fluoride salt and lithium and calcium metals were added to the reduction salt to change its composition and thereby lower the plutonium distribution coefficient. The salt was then contacted with a Cu-36 w/o Mg alloy at 700°C for 5 hr with a mixing speed of 250 rpm. Plutonium distribution coefficients measured in the first and second steps were 0.045 and 0.036, respectively. Analysis of the salt following the run revealed that 1.5 g of plutonium remained in the waste salt stream as an insoluble phase. The nature of this phase and the reason for its formation are being investigated.

In the plutonium salt-transport step, plutonium is transported at 600°C from the Cu-33 w/o Mg donor alloy to the Zn-2 w/o Mg acceptor alloy using a 50 m/o MgCl_2 -30 m/o NaCl-20 m/o KCl salt mixture as the transport salt. Fourteen cycles were conducted in this experiment and 99.3% of the plutonium was transported, with more than 90% being transported in the first five cycles. About one gram of copper (or 0.05% of the copper in the donor alloy) was found in the acceptor alloy. Operation of the salt-transport step is considered to be highly satisfactory.

In the copper-removal step, the Zn-Mg-Pu acceptor alloy from the salt-transport step is treated with ZnCl_2 to oxidize the magnesium and plutonium to MgCl_2 and PuCl_3 , which distribute to the salt phase. The plutonium is then salt-transported to a Zn-4 w/o Mg acceptor alloy, re-using the salt from the previous salt-transport step. The oxidation of the magnesium and plutonium was conducted at 600°C for 1.5 hr with a mixing speed of 570 rpm. The salt-transport portion of the copper-removal step was also performed at 600°C; six cycles were carried out. The resulting Zn-Mg-Pu acceptor alloy contained only ~0.06 g of copper, which is the level which exists in the alloy from the reagents used prior to the salt transport of the plutonium. The waste zinc stream from the original acceptor alloy of the salt-transport step contained ~0.003 g of plutonium.

The Cu-Mg supernatant from the donor vessel of the salt-transport step is poured away from the precipitated uranium, which contains residual copper and magnesium. This precipitated uranium is then exposed to a uranium wash step in which the precipitate is washed with magnesium to remove residual copper and fission products. During this experiment, the uranium precipitate was washed three times at 700-750°C with 1.0, 1.5, and 1.0 kg of magnesium. A sample of the Cu-Mg alloy prior to the wash indicated the presence of 0.67 g of plutonium. However, analysis of the wash waste streams showed that 2.2 g of plutonium was present. An attempt is being made to determine whether this plutonium loss resulted from slight coprecipitation with the uranium or from the entrapment of plutonium metal or PuO_2 , either in the precipitated uranium matrix or in a metal-salt agglomerate found on the baffles of the vessel. A summary of the overall plutonium recovery and purification achieved in the experiment is given in Table III.D.2.

TABLE III.D.2. Summary of Run PuSTP-1

Plutonium Recovery: (199.3 g Charged)	~51% (of Pu charged) in product solution ~47% (of Pu charged) in process inventory ^a ~2% (of Pu charged) in waste streams ~100% material balance
Plutonium Purity: (See Table III.D.1)	~99-99.999% removals for noble and refractory metals ~97-99% removals of Nd, La, Ce, and Pr. Other fission product contamination levels either negligible or uncertain. ≤0.1% uranium in product ~50-500 ppm copper expected in retorted product
Uranium Recovery and Purity:	To be determined after retorting operation

^aSince there was no plutonium present in reagents recycled from a previous run, the level of plutonium recycle (in-process inventory) was estimated and compensated for by having sufficient plutonium in the original charge to provide a reasonable throughput of plutonium.

Construction of equipment for retorting the uranium and plutonium product solutions has been completed and three shakedown runs have been performed. Retorting of the product solutions is expected to be completed during the next month.

c. Materials Testing and Evaluation. A niobium crucible has been exposed to a Cu-33 w/o Mg-0.1 w/o U/MgCl₂-30 m/o NaCl-20 m/o KCl system at 700°C for a total of 962 hr without failure (see Progress Report for April 1967, ANL-7329, p. 53). No deterioration of the crucible was evident by visual inspection. The crucible has been sacrificed for metallographic examination. A section of the crucible wall could be bent 30° before fracture, as compared with 180° for new material. This indicates that the ductility of the crucible was affected by exposure to the metal-salt system, but did not decrease to a dangerous level. The effects of long exposure of niobium to this system will be discussed when metallographic examinations have been completed. Although niobium is more expensive than tantalum, its extended usable life with this particular metal-salt system seems to warrant the higher cost.

d. Development of Unit Operations. A facility for high-temperature extraction has been designed and constructed to study mass-transfer rates between liquid metal and molten salt solvents in packed, countercurrent extraction columns. In addition, information on the design and operation of such equipment will be derived, limiting flow rates for columns of this type will be determined, and the entrainment of metal in the effluent salt stream will be studied.

A schematic diagram of the equipment is presented in Fig. III.D.1. The equipment consists of the column, a jack leg to control the position of the metal-salt interface in the column, supply and receiver tanks for the metal and salt solvents, a column-drain tank, and bellows-sealed valves to control the flow of the fluids. The entire facility is constructed of Type 304 stainless steel.

Fig. III.D.1. Schematic Diagram of Facility for High-temperature Extraction

The column consists of a 1-in. pipe, 3 ft long, packed with 1/4-in. Raschig rings. Liquid metal (the discontinuous phase) is sprayed into the top of the column through several small holes in a horizontal tube in the top disengaging section (see Fig. III.D.2). The disengaging section is a 3-in. pipe containing a system of weirs to maintain the salt level in the column and to separate the metal feed from the salt effluent. Liquid salt (the continuous phase) enters the bottom of the column beneath the packed section. The position of the metal-salt interface below the packed section is determined by a mutual-inductance probe which surrounds the 1/2-in. tube where the interface is maintained.

The salt and metal supply and receiver tanks are equipped with agitators, thermocouple wells, sample ports, and movable resistance probes for liquid-level detection. In addition, the metal tanks have mutual-inductance probes, and the salt tanks have time-delay reflectometry probes for liquid-level determination. Each tank has connections to the pressure-vacuum manifold to provide for movement of the solvents by pressure siphoning from tank to tank and to and from the column.

Fig. III.D.2. Top Disengagement Section of Metal-Salt Extraction Column

A single extraction experiment lasts from 20 to 120 min, depending on the flow rates, and is terminated when the supply of metal or salt is exhausted. Prior to a subsequent experiment, the solute which was extracted from the metal into the salt is removed from the salt by contacting it with a Bi-6 w/o Mg alloy in the salt-receiver tank. Additional solute is then dissolved in the alloy in the metal-receiver tank for the next run. The entire apparatus is kept at operating temperature between experiments and is only shut down completely at the end of a series of experiments.

An experiment has been performed to study the extraction of cerium from a Cd-6.9 w/o Mg-2.2 w/o Zn-0.65 w/o Ce alloy into a 52.4 m/o

MgCl₂-28.6 m/o NaCl-19 m/o KCl salt mixture at 600°C. In this experiment (Run EX-4), 40 kg of metal was contacted with about 7 kg of salt over a 32-min period. The 33 in. of packed column length provided about 2.4 theoretical extraction stages, which is in good agreement with the value predicted from preliminary studies.* An extraction rate of 1.2 g Ce/min was realized with a metal flow rate of 1080 g/min and a salt flow rate of 181 to 276 g/min. During this run only 0.1 g of cadmium from the alloy was carried into the effluent salt. In future runs the effects of flow rates, flow ratios, temperature, and packing size will be studied.

Errata

In the Progress Report for August 1967, ANL-7371, p. 98, Fig. 15, the capacity of the Constant Head Tank should read 3 gal instead of 30 gal, and the flow rate range between the Constant Head Tank and the first Metal-Salt Contactor should read 0-1 gpm instead of 0-2 gpm.

*Chemical Engineering Division Semiannual Report, July-December 1965, ANL-7125, pp.35-36.

3. General Chemistry and Chemical Engineering

a. Characterization of Carbon-bearing Species in Sodium:

Solubility of NaCN in Liquid Sodium. Cyanide has been shown to form in liquid sodium at elevated temperatures by the reaction of carbon impurities, such as sodium carbonate, with nitrogen.*,** Because cyanide is a potential carburizing agent, its behavior in liquid sodium is of interest. As a first step toward understanding this behavior, the solubility of sodium cyanide in sodium is being measured.

The solubility is being measured by a procedure that involves equilibration of carbon-14-labeled NaCN with liquid sodium, sampling the equilibrated sodium with stainless steel pipettes equipped with micrometallic filters, combusting the sample in oxygen, and assaying the resulting carbon-14-labeled CO₂ by liquid scintillation counting. The use of carbon-14 permits the determination of very low cyanide solubilities.

Preliminary experiments indicate that the solubility of sodium cyanide in sodium at 500°C is ~0.4 ppm.

*Hobart, E. W., and Bjork, R. G., Nucl. Appl. 1, 490 (1965).

**Luner, C., and Anderson, K., in Chemical Engineering Division Seminannual Report, July-December 1965, ANL-7125, p. 141 (May 1966).

IV. ADVANCED SYSTEMS RESEARCH AND DEVELOPMENT

A. Argonne Advanced Research Reactor (AARR)

1. Core Research and Development

Because the revised AARR design uses fuel elements, control elements, control-drive mechanisms, and many reactor internal structures identical to their counterparts in the High Flux Isotope Reactor (HFIR), AARR core studies involve only transient heat-transfer effects and development of structures to support the HFIR-type components in the AARR vessel. The structural design is complicated by new seismic-load criteria and by the precision needed to accommodate AARR's six blind tubes and two through-tubes that are to be used for experiments.

a. Heat-transfer Studies. Steady-state safety margins are being verified by analysis of operational malfunction and inadvertent reactivity additions so as to be assured of the adequacy of the core thermal design for the AARR conditions (4.4-MW/liter peak power density and 2×10^6 Btu/hr/ft² peak heat flux at 100 MW).

(i) Heat-transfer Analysis. The thermal performance of the core is being analyzed with respect to steady-state operation, slow and rapid transients, and shutdown-decay-heat removal and emergency cooling.

(α) Experiment Facility Analysis. Heat-transfer analyses are being made to evaluate the temperature conditions of the vertical experiment facilities (located in the internal thermal column and in the reflector) during reactor conditions of steady state, postulated accident, and shutdown.

A computer program (TRANTEMP) being developed for use with the CDC-3600 will permit calculation of the temperature behavior of the structure within the core region that contains the material to be irradiated and the flowing coolant. The code section dealing with heat transfer under conditions of low-velocity flow reversal for a stationary rabbit is about 90% complete. This section will also be developed for analyzing heat transfer for a moving rabbit and flow reversal for higher velocity.

A TRANTEMP subroutine has been developed for rapid determination of the initial steady-state temperature profile in a hydraulic-rabbit system. A numerical procedure similar to that used by the main program when it determines transient temperature profiles is used.

All TRANTEMP printed output has been labeled so that those unfamiliar with the code can easily use and interpret it.

(β) Analysis of Core Structures. The objective of the thermal analysis of core structures and reflector is to determine the adequacy of the design for steady-state and transient conditions. The status and calculational models have been described (see Progress Report for August 1967, ANL-7371, pp. 100-101). The models have been revised to approximate the actual case more closely. The former Model B has been split into two sections, Models B1 and B2. The included angle for Model B2 was decreased from 2.625 to 2.25° and divided into ten angular sections instead of the eight formerly used. There are 60 nodes in Model B2. Model C was changed only slightly by incorporating the effect of the coolant slot in the semipermanent reflector.

The analysis is about 40% complete. Calculations of the temperatures and their distribution have been made for 100 and 115 MW, the design overpower condition, but not for both power levels for all models. The boundary conditions (see Table IV.A.1) used in the calculations are conservative assumptions. Alteration of the boundary conditions and the models will be made in the course of this study.

TABLE IV.A.1. Boundary Conditions for Calculational Models

Model	Boundary Conditions	Steady-state Heat-transfer Coefficient (Btu/hr-ft ² -°F)
A	Aluminum liner insulated from inner beryllium; contact areas of beryllium insulated except at coolant holes	3750
B	Contact areas of beryllium pieces insulated except for coolant holes	3750
C	Contact area between permanent and semipermanent beryllium in near-insulated condition	$h_1 = 0.00001$ $h_2 = 3750$ $h_3 = 50$

The calculations are based on the heating rates calculated by the MAC code. Heating rates close to the core and at large radial distances are being compared with HFIR values and are being thoroughly explored in the physics analysis of AARR.

Table IV.A.2 shows the preliminary results for the removable reflector when the core power is 100 MW, coolant velocity is 13 ft/sec, and water temperature is 120°F. Table IV.A.3 shows preliminary results for 115-MW core power under identical fluid conditions in

the semipermanent and permanent reflector. The results indicate that closer examination of the inner and center removable beryllium is required because of the high temperatures (levels and gradients) and heat-flux conditions. Actual conditions are judged to be less severe. The outer removable beryllium and semipermanent beryllium have more moderate thermal conditions and are not a cause for concern.

TABLE IV.A.2. Summary of Thermal Conditions in Removable Beryllium Reflector at 100 MW

Model	Maximum Temp (°F)		Heat Flux (Btu/hr-ft ²)
	Surface	Internal	
A (Inner Removable Beryllium)	390	490	1.0×10^6
A (Center Removable Beryllium)	240	320	4.5×10^5
B1 (Outer Removable Beryllium)	200	240	3×10^5

TABLE IV.A.3. Summary of Thermal Conditions in Semipermanent and Permanent Beryllium Reflector at 115 MW

Model	Maximum Temp (°F)		Heat Flux (Btu/hr-ft ²)
	Surface	Internal	
B2 (Semipermanent Beryllium)	203	250	3.1×10^5
C (Permanent Beryllium)	327	500	1.14×10^6

The permanent beryllium, which is actually a much more complicated geometry than indicated because of the multiplicity of experimental penetrations, will also require more extensive analysis. The temperature gradient radially across the reflector is ~175°F. Between coolant holes, the temperature gradient is between 120°F (near the inside diameter) and about 80°F (near the outside diameter). The effect of these gradients on thermal stress has not been evaluated.

(ii) Transient Heat-transfer Tests. Voids and pressure pulses during transient boiling are being measured in tests using an electrically heated flow-channel section in which power is varied with time. The purpose is to provide a means by which the calculated effects of reactivity additions on core heat transfer and hydrodynamics can be compared with a physical model of fuel-plate and coolant behavior.

The third test section has been installed (see Progress Report for August 1967, ANL-7371, pp. 101-102) and pressure tested. The testing phase of the program has been initiated. Adiabatic pressure drop through the section was measured and compared with calculated pressure drop using the integrated average measured channel spacing. Maximum deviation was 4%.

Final instrumentation connections and calibrations have been completed. The noise component on the wall-thermocouple trace appears large enough to be troublesome. The noise component on the absolute inlet- and exit-pressure measurement was unexpectedly low-- only ± 2 psi at full operating power pressure.

Power has been applied to the test section. Heat balances and nonadiabatic pressure drops are being measured; pulse tests will follow.

b. Core Structure Development. Prototype core components are being fabricated, their assembly is being proof tested, and hydraulic interactions and redesign to meet seismic-load criteria are being evaluated in work aimed at verifying the fabricability of the structures needed to precisely support the core in relation to the experiment facilities.

(i) Design of Core Structure. A new pressure-vessel-and-internals assembly drawing has been made for the preliminary safety analysis report. Pressure-vessel requirements for the core-support shelf, shield-plug adapter, coolant inlet diffuser, and orientation of coolant nozzles were prepared to guide the vessel contractor. A preliminary design has been completed of outer-shroud and experimental-facilities liners. The shroud flange is being designed.

(ii) Beam-tube-liner Seal Test Fixture. The fixture and components have been designed to simulate the interface of the aluminum beam-tube liner with the permanent beryllium reflector. The prime objectives of the test are to establish the fit attainable on shrink-fitting of the liner to the simulated beryllium component and to establish anticipated rates of water leakage between the liner and reflector interface at the operating pressure differential. The latter data are desired to aid in the analysis of flow and temperature in the permanent-reflector region.

Tests will be conducted up to a pressure differential of 125 psi at room temperature. The design temperature of the fixture is 250°F and design pressure is 150 psig.

(iii) Measurement of Fuel-plate Gap. The objective of one of the experiments in the AARR hydraulic test program was to determine the extent of any fuel-plate distortion and/or movement or vibration under simulated reactor coolant flow conditions. The measurement method could not in any way interfere with the flow patterns near the test fuel assembly. Imposition of any form of physical loading on the fuel plate by contact or connection with a transducer or sensor could not be permitted. The device would have to operate in 500°F and 1250-psi water and have a sensing-gap range of 0 to 0.040 in.

A search was made for suitable commercially built linear-displacement transducers, but no available sensor met the specifications. Thus, commercial instrumentation was used for fuel-plate tests at the lower temperatures and pressures, while a linear-displacement transducer was being developed for use in the tests at high temperatures and high pressures. It was decided that the new transducer had to operate on one of three electronic principles: variable reluctance, variable impedance, or variable capacitance. All other principles, such as optics, microwave, nuclear, etc., would be too cumbersome and costly. After consideration of all factors involved in the design of pressure seals, complexity of internals, and concern for the effects of coolant temperature-density changes, the variable-capacitance transducer was selected as having the most favorable characteristics for ease of construction.

Single preliminary experiments indicated the feasibility of such an instrument. The first model was constructed of two brass sections. One section had a fixed gap to act as the "passive" arm in a capacitance bridge; the second section served as a variable capacitor that performed as the "active" arm of the bridge. In bench tests at 72°F, air was the dielectric; a bridge oscillator served as the signal source, a power amplifier provided higher input power, an electronic voltmeter supplied readout data, and an electronic twin-T diode network conditioned the signals.

Because of the good results from the simple tests, an improved configuration was assembled. The materials used for this transducer limit temperature to 150°F; sealing materials limit the maximum differential pressure across the sensor to approximately 50 psi. The capacitor plates and sensor body were made of brass, and the spacer-insulators were machined from plastic. After being polished and cleaned, the metal parts were sprayed with an electrical-insulating lacquer before being assembled. The wire and connections were potted with silicone-rubber compound to provide a watertight assembly. This transducer was

benchtested with air, water, and oil as dielectrics. Capacity readings taken with the transducer in an adjustable-gap fixture were plotted as the gap was changed in 0.005-in. increments from 0.005 to 0.030 in. The curves showed similar slopes for all three dielectrics. Some discrepancies were noted; these possibly were caused by differential temperature expansion of the brass and plastic, and by the electronic test equipment and experimental networks.

The decision to use a proven HFIR-type core in the AARR has eliminated the need for fuel-plate distortion tests, and thus for an instrument to measure the coolant gap between fuel plates. Accordingly, no further work is planned on this transducer. However, temperature and pressure ratings could be raised and dimensional stability achieved by using the following construction materials: (1) stainless steel for the housing, (2) Invar for the capacitor elements, (3) a ceramic for the dielectric spacers, (4) high-temperature ceramic spray coating for insulating the capacitor elements, and (5) high-temperature silicone-rubber compounds for potting and leakproof sealing of the terminals and cable sheathing. In addition to changes in materials for future transducers, electronic signal conditioning needs improvement; standard capacity bridges do not suffice as high-speed readout instruments.

2. Component Development

In support of the reactor design work, specialized experiment facilities are being developed.

a. Sample Irradiation Devices. Special mechanical-design concepts, fabrication methods, cooling provisions, and installation and handling techniques are being developed and prooftested with prototypes; the behavior of special materials, such as beryllium, under thermal-cycling conditions and uneven heating and cooling is being investigated. The objective is to enable the design of hydraulic- and gas-operated rabbit-type experimental facilities that will withstand reactor pressure and also allow a high neutron current to reach the experiment.

Design of the operating control centers for the rabbit system (hydraulic and gas) prototype has started. Preconstruction safety review is underway for components required for tests of the hydraulic-rabbit prototype. The gas rabbit system and blower pressure vessels have been fabricated.

b. Beam Tube Development. Extensive stress analyses, tests of rupture, bending, collapse, and radiation heating of typical beam-tube sections, as well as prototype tests under simulated operating conditions, are being made.

(i) Prototype Beam Tubes. Prototype blind and through beam tubes and their associated components have been fabricated. Except for the beam-tube adapters, all components have been received. The prototype tubes and their associated components will be prooftested with the beam-tube test facility and General-Purpose Hydraulic Loop (see next section).

(ii) Beam-tube Test Facility. A facility is being constructed to prooftest the prototype beam tubes and their associated components. The tests will include the installation and replacement of the prototype beam tubes, dimensional and clearance inspection, bending due to external loading of the beam tubes, integrity of the seals, pressure and flow tests in conjunction with the General-Purpose Hydraulic Loop, verification of the design of prototype handling tools and equipment, and the establishing of handling procedures. The facility also will be used for dimensional checkout and tests of future production-type beam tubes for the AARR.

The facility, shown in Fig. IV.A.1, is representative of a full-size section at the AARR beam-tube penetration, including the biological shield, reactor pool, reactor-vessel nozzle, nozzle Grayloc seal and connector, and the reflector sleeve. The facility is mounted on an adjustable support base to correspond to the AARR beam-tube elevations.

Detail design and fabrication have been completed, except for the Grayloc nozzle, whose sealing face and tapped holes in the flange were found damaged at delivery. The mounting face of the nozzle, the diameter of the pilot, and the bore also were not within specified tolerances. Rework of the nozzle is about 95% complete.

(iii) Metal-seal Tests. Because of concern that the Grayloc-type flange on the beam-tube nozzles of the AARR pressure vessel could not be machined without excessive cost and delay, various metal seals are being evaluated. An inverted bolted-type flange requiring a metal gasket was selected as a backup design. This same type of gasket can also be used as the high-pressure seal of the beam tubes if the prototype design with the Marmon-Conoseal gasket proves troublesome.

The 9.5-in.-ID metal gaskets selected for tests are of the low-bolt-loading and self-pressurizing variety capable of sealing against 1100 psig of water at room temperature. They have stainless steel or Inconel as base metal and are plated with soft nickel. The testing equipment (see Fig. IV.A.2) consists of two 12-in., 600-lb, Type 304 stainless steel blind flanges whose faces were machined to a 16 rms finish and a flatness of 0.001-in. total indicator reading (TIR). Spacer rings were used to achieve recommended gasket deflection. Bolts were torqued in 50-ft-lb increments. The pressurizing medium was introduced through piping interconnected with a pressure gauge at the center of one blind flange. The total pressurized volume was about 10 in.³ The gaskets (see Fig. IV.A.3) performed as follows:

Fig. IV.A.1. Beam-tube Test Facility (side-view cutaway)

Fig. IV.A.2. Metal-seal Test Facility

gasket-lip compression and (b) flange deflection the bolt circle diameter from $19\frac{1}{4}$ in. to 12 in. for ten $1\frac{1}{4}$ -in.-dia bolts. Flange flatness was checked and found acceptable. When the gasket was repressurized to 300 psig, there was no appreciable improvement in leak rate.

Metal O-Ring (Advanced Products Co., Type 321 stainless steel, pressure-vented, 0.010-in. wall). Four nickel-plated O-rings were torqued to 300 ft-lb and the system was pressurized with helium to 600 psig. Leak rates were found to be 20-50 psig/min. Such poor results indicated that there was either a deficiency in the test equipment or the nickel plating was too hard in comparison with the Type 304 stainless steel flanges for the seal to fill in the minute imperfections of the flanges.

Two similar O-rings were obtained with silver instead of nickel

Omega (Servatronics Inc. Model P-9.520-4-3-4). The twenty $1\frac{1}{4}$ -in.-dia bolts were torqued to 150 ft-lb and the system was pressurized with helium to 300 psig. The pressure dropped immediately at ~ 50 psig/min. All fittings were mass-spectrometrically leaktight. Examination with a magnifying glass showed fissures and "pimples" measuring as high as 0.001 in. on the nickel plating of the sealing surfaces on three gaskets. Of the remaining three gaskets that appeared to be free of visual defects, two would not fit into the spacer ring; the third leaked at a rate of ~ 30 psig/min under the same conditions as the first gasket checked. The free height of the gasket measured 0.111 in. and the spacer ring was 0.105 in. thick, so deflection was only 0.003 in. on each gasket lip. To produce more effective sealing: (a) the spacer ring was ground to 0.102 in. to increase the

OMEGA

LO-LOAD

K

V

COOK

Fig. IV.A.3. Gasket Cross Sections

plating. One was pressurized in 15 min at 100-psig increments up to 1100 psig. After 88 hr the pressure was 1095 psig. This same gasket was cycled three times from 0 to 1100 psig and held for a 1-hr period each time with no apparent leakage. The second silver-plated O-ring gave similar results.

"Lo-Load" (Advanced Products Co., Model AP30-9500-3-NP). Bubbles formed around the flange studs at helium pressures of 50-150 psig for each of the four gaskets checked. Three of the used nickel-plated gaskets were stripped and replated, one with silver, one with indium-lead, and one with a 0.003-in. thickness of nickel rather than the standard 0.001-in. thickness. When tested, the silver-plated gasket leaked at a rate of ~20 psig/min from the initial helium pressure of 1300 psig and settled down to about a 1-psig/min loss at pressures below 600 psig. To preclude causing waviness of the flange lips, the vendor had performed no lapping or finishing operations after plating the gasket; the result was rather lumpy plating. Inspection of the gasket after pressurization showed radial scratches on the silver plating due to the sliding action of the gasket lip against the flange as compression took place. (This type of scratch, which should be normal, was not visible on the nickel-plated gaskets.) However, the fact that scratching or marring did not occur all the way around on the gasket seating surface caused doubt that the gasket lips bore against the flanges evenly. The gasket whose nickel plating was 0.003 in. thick performed as poorly as the 0.001-in.-nickel-plated seals. The lead-indium-plated gasket sealed against 700 psig helium for 4 hr, whereupon further pressurization to 1100 psig caused leakage of ~0.2 psig/min down to 730 psig, where the pressure was relieved; the gasket was repressurized with water to 2000 psig and after 24 hr the reading was 1900 psig.

Haskel "K" (Haskel Engineering and Supply Co., Model 18968). Variation of the bolt torque in the range 250-500 ft-lb had no effect on the sealing qualities of the Haskel K seal. Regardless of bolt torque, helium leakage was ~20 psig/min at a pressure of 500 psig and 5 psig/min at pressures below 225 psig. All three gaskets checked behaved similarly. Scratches and mars were noted on the flanges where the gaskets had seated. After the flanges were reworked to an 8 rms finish, further tests with both helium and argon showed no significant changes in leak rate. Two of these gaskets are being plated with silver and gold for further tests.

Parker "V" (Parker Seal Co.). The V seal differed from the previous gaskets in the greater amount of lip deflection required to seal, i.e., 0.019-in. total compression as compared to about 0.006 in. However, this feature precludes reusability because a permanent set occurs in the gasket. With this gasket, as with other gaskets, the sealing surfaces felt rough to the touch. The leak rate for the gasket as supplied was ~20 psig/min in the 600-psig pressure range. Two gaskets were carefully hand lapped with crocus cloth, which improved the leak rate to ~1 psig/min in the 600-psig range; they still held a pressure of 50 psig after 16 hr.

Cook (Cook Airtomic Div. of Dover Corp.). This nickel-plated gasket performed poorly under test conditions. Regardless of how carefully the gasket was loaded, one gasket lip would be compressed toward the reinforcing web while the opposite lip would remain open. This pattern alternated around the gasket.

In addition to the commercially available gaskets listed above, several 2S-aluminum O-rings were made from 1/8-in.-dia wire. These gaskets held helium leaktight at 1100 psig for several days if the gaskets were firmly backed by a 0.105-in.-thick spacer ring. However, the gaskets could not be resealed if any cycling was attempted; additional bolt torquing would not aid resealing.

A standard 6-in. set of Grayloc F-316 blind flanges, complete with clamp and 17-4PH seal ring, was borrowed from Gray Tool Co. for tests (see Fig. IV.A.4). The four clamp bolts were torqued to 120 ft-lb and the assembly was pressurized with helium to 850 psig. System pressure remained constant for 4 days. The pressure was increased to 1200 psig; after 6 days the pressure was 1100 psig. The apparatus was taken apart, and the seal ring turned upside down and rotated 90° from its original position. The reassembled system held helium at 1100 psig for 3 days with no discernible leak. This seal also remained tight against 2000-psig water for 3 days.

Fig. IV.A.4. Test Assembly for Grayloc Flange

To simulate flange misalignment, such as could occur on the AARR pressure vessel, a 0.015-in.-thick 180° shim was placed between the seal ring and flange flat faces to create misalignment in the tapered surfaces. In 5 days the helium pressure dropped from 1100 psig to 655 psig. With a 0.010-in. shim, the pressure remained a constant 1080 psig for 2 days. Upon disassembly, scoring was observed on the tapered surface of the flanges, although the seal ring remained unmarked. An effective seal could not be maintained with the same seal ring after the scoring occurred.

The Grayloc seal proved to be a rugged and dependable joint that is excellent for high-pressure service. It requires no particular care in

assembly, and the seal ring is self-aligning. Although the tested ring was completely satisfactory in an unplated condition, plating normally is used to ensure sealing integrity. These seal rings are also reusable under normal use; their design allows them to be secured to one of the mating flanges for retention.

On the other hand, the gaskets in Fig. IV.A.3 are more delicate; they require careful handling and a sealing surface of 8 to 32 rms finish with accurately controlled flatness and parallelism. Whether gasket diameter is 1 in. or 9.5 in., the same amount of lip deflection is required for sealing. But when this lip deflection is only 0.006 in. and the gasket circumference is 30 in., quality control of both the flange and gasket becomes critical. Since the two gasket lips act independently, waviness of 0.0015 in. on the gasket sealing surface opposed by a similar condition on the flange seat would prohibit sealing. Even with acceptable tolerances, the gasket plating material must be softer than the flange, i.e., a nickel-plated gasket does not work with a Type 304 stainless steel flange, as is evidenced by the performance of the nickel-plated standard O-ring. The gaskets with the softer plating offer promise, and they will be tested further. Also, to determine how nickel-plated gaskets seat against a hardened surface, tests are planned with stainless-steel flanges that have been chrome-plated or nitrided.

b. Internal Thermal Column Development. Devices are being developed to insert and remove irradiation samples into and from the internal thermal column (ITC) while the reactor is operating.

Prototype designs of the ITC irradiation facility valve head assembly, core assembly, and the required handling tools have been completed. Detailed shop drawings are 90% complete for the valve head assembly. Detail work on the core assembly is about to begin.

Preliminary design of the movable section of the ITC rabbit system conduit (tube section connecting the valve head assembly to sample transfer stations) has been completed; the prototype is being designed.

3. Physics Experiments and Analyses

a. Heating Rates and Dose Rates in the Biological Shield. The MAC* computer program has been used to evaluate the effectiveness of the bulk-biological shield axially below the reactor core. This axial-downward shield includes, within the reactor pressure vessel, 7.5 ft of water, then 1.5 ft of steel (shield plug), 5 ft of water, and, finally, the bottom head of the pressure vessel (~1 ft thick). The calculated gamma-ray dose rate at the bottom surface of the bottom head of the reactor pressure vessel is 0.004 mR/hr at full power (100 MW). This dose rate is for an unpenetrated

*Peterson, E. G., MAC--A Bulk Shielding Code, HW-73381 (April 1962).

bottom shield and it does not include effects of radiation streaming in vertical tubes containing thermocouples and N^{16} probes. An estimate of the magnitude of this streaming is discussed below.

An axial distribution of heating rates has been inferred from the above-mentioned MAC calculation. Immediately below the active fuel plate is a 2-in.-long aluminum end piece. The heating rate, due to gamma-ray heating alone, is calculated to be ~ 20 W per gram of aluminum at the bottom of the aluminum end piece. Most of this heating is due to low-energy gammas escaping the reactor core. The gamma-ray flux spectrum hardens with increasing distance below the reactor core and therefore the heating rate drops off less rapidly with increasing distance. Table IV.A.4 lists heating rates at various distances below the active fuel plate, in units of watts per gram of iron since most of the metallic pieces directly below the core are steel, e.g., components of the control-rod drives.

TABLE IV.A.4. Heating Rates Axially Downward from Reactor Core in Bulk Biological Shielding (100 MW)

Distance below Bottom of Active Reactor Core (ft)	Approximate Heating Rate (W/g Fe)	Distance below Bottom of Active Reactor Core (ft)	Approximate Heating Rate (W/g Fe)
0.17 (2 in.)	$\sim 20^a$	3.0	0.06
0.5 (6 in.)	8	4.0	0.015
1.0	2.3	5.0	0.004
2.0	0.3	7.5 (top of shield plug)	0.00025

^aThis is due to gamma-ray heating only. Neutron slowing down causes some additional heating close to the reactor core, but this contribution drops off rapidly with increasing distance from the core.

Gamma-ray streaming in the N^{16} -probe assemblies has been evaluated. Four assemblies are planned for the AARR to house thermocouple tubes and possibly also N^{16} probes. In each such assembly there is a straight vertical run, corresponding to a 1/4-in.-dia pipe, from the bottom surface of the bottom head of the reactor pressure vessel upward to within inches of the bottom of the reactor core. Over most of the distance there is water in this vertical run; the bottom 6 in. consists of filler material for thermocouple tubes. The reason for particular interest in the gamma-ray streaming in the N^{16} -probe assemblies is that the bottom head of the reactor vessel forms part of the "ceiling" of the rod-drive access room, to which personnel will have (limited) access during reactor operation at full power.

The analysis of this problem of radiation streaming begins with a MAC calculation to obtain ϕ_{γ_j} (entrance), the group-j gamma-ray energy flux at the top surface of the bottom shield plug. If the angular distribution into the bottom half-space is assumed to be cosine, the group-j energy flux at the point where the equivalent 1/4-in.-dia pipe exists from the bottom of the bottom head of the reactor vessel is calculable from the formula

$$\phi_{\gamma_j}(\text{exit}) = \left(\frac{r}{L}\right)^2 \phi_{\gamma_j}(\text{entrance}) \exp(-\bar{\mu}_j L),$$

where

r = radius of equivalent penetrating pipe ($r = 0.32$ cm);

L = total distance from the top surface of the shield plug to the bottom surface of the bottom head ($L = 230$ cm);

$\bar{\mu}_j L$ = the total number of attenuation mean free paths for a beam of gamma rays of energy-group j in this vertical run.

The factor $\exp(-\bar{\mu}_j L)$ has been included to account for the removal of gamma rays from the beam by the material in the pipe. Otherwise, this formula is a standard result* for streaming in a long thin pipe.

The total local gamma-ray dose rate at the exit of the bottom head is ~ 2 mR/hr, corresponding to

$$\sum_{j=1}^7 \phi_{\gamma_j}(\text{exit}).$$

The calculation described involves net attenuation factors $(r/L)^2 \sim 2 \times 10^{-6}$, and $\exp(-\bar{\mu}_j L) < 5 \times 10^{-3}$, and such calculations remain outstanding problems of shielding analysis. The calculation, in this case, should not be considered to be any better than order of magnitude. Nevertheless, radiation streaming at these penetrations would result only in highly localized "hot" spots in the rod-drive access room. This is confirmed by measurements (made in the subpile room of the HFIR.** If N^{16} probes are incorporated in the system, local radiation shielding will be provided to minimize dose rates from these sources to personnel in the rod-drive access room.

*Rockwell, Theodore, III, Ed., Reactor Shielding Design Manual, First Edition, Naval Reactors Branch, Division of Reactor Development USAEC, TID-7004, pp. 288-291.

**Private communication from T. E. Cole (ORNL).

b. Radial Distribution of Heating Rate in the Radial Beryllium Reflector. One source of uncertainty in the calculation of heating rates in the beryllium reflector is the restriction in the MAC program that the gamma-ray buildup factors in the various spatial regions are calculated with a single assignment of an "equivalent" buildup material. The calculations summarized earlier (see Progress Report for May 1967, ANL-7342, pp. 105-106) were obtained using buildup factors for water. Additional MAC calculations have been performed with buildup factors corresponding to aluminum, or to iron, to evaluate uncertainties in the computations of heating rates in the beryllium.

The largest effect on heating rate arising from the choice of buildup factor occurred at the core surface. At this point, the use of water buildup factors yielded a heating rate approximately 40% higher than the heating rate determined with iron buildup factors, and 20% higher than with aluminum buildup factors. At a radial distance of 30 cm from the core surface, use of water buildup factors resulted in a heating rate one-fourth larger than that for iron buildup factors, and ~13% larger than for aluminum buildup factors. It is concluded that the uncertainty in the calculated heating rate due to the approximate model of a single set of buildup factors is no more than ~10% in the radial beryllium reflector. The heating rates summarized in ANL-7342 are conservative in this regard.

Preliminary shielding analysis of the unpenetrated lateral biological shield indicates that, at least in the azimuthal directions, where no aluminum "neutron windows" are present, the shield design should be conservatively acceptable. With beam tubes and through tubes represented as locally homogenized voids in the beryllium reflector and in the water between the beryllium and the reactor pressure vessel, a MAC computation for the unpenetrated shield implied a gamma-ray dose rate of ~0.05 mR/hr at the outside surface of the lateral concrete shield. MAC computes gamma-ray dose rates only in slab geometry. The primary source of these gamma rays is the reactor core, which acts more nearly like a small spherical source. If corrected for geometry, it is estimated that the MAC computation would imply a dose rate of less than 0.01 mR/hr at this surface. Of course, there will be penetrations of the bulk concrete shield which will raise the dose rate from this source. Analysis of the more detailed shield configurations is continuing.

c. Reactor Physics Analysis. Multigroup diffusion-theory calculations have been performed in one-dimensional cylindrical geometry, with 6 groups and 16 groups of neutrons, and in two-dimensional (r-z) geometry, with 6 groups of neutrons. The calculations were intended for comparisons of such important data as the gross reactivity of the system and the reactivity effect of voiding of some of the water in the ITC.

The 6-group and 16-group one-dimensional calculations agreed to within 0.3% in gross reactivity, with the larger value of available excess

reactivity predicted by the 16-group calculation. The two-dimensional calculation with 6 groups of neutrons predicted an available excess reactivity 1.2% higher than that indicated by the one-dimensional 16-group calculation. Probably, a two-dimensional 16-group calculation would predict a somewhat higher value for the available excess reactivity. There is some uncertainty due to the coarseness of the mesh in the spatial partitionings of the various reactor regions, although finer meshes were used in the vicinity of interfaces between spatial regions.

A reactivity difference of +1.2% corresponds to a change of +3.0 cm in the total equivalent bare height in the one-dimensional 16-group calculations. In large part, the higher average reflector savings is due to the fact that the radial beryllium reflector extends above and below the active portion of the reactor core.

If the reactivity effect of the boron burnable neutron poison in the reactor core is included, but fission-product poisoning by xenon, samarium, etc., is excluded, the net available excess reactivity in the operating fresh reactor is predicted to be 8.3% by the two-dimensional analysis. Of this, +0.48% is due to the replacement of some of the water in the 20-in.-long active portion of the ITC by a full loading of target tubes and targets, with a total additional neutron poisoning equivalent to 0.5 g natural boron in the ITC, due to neutron absorption by target material. The corresponding one-dimensional result is +0.42%. ("ITC water" is defined to mean water within the radial boundary of the ITC insert, namely, the ITC shroud.)

One-dimensional, 16-group diffusion theory calculations for cases of partial voidings of water in the ITC yielded a peak reactivity gain of ~1.4%, with ~50% of all the water in the ITC voided.

With 70% of the water in the ITC removed, the one-dimensional prediction is a net reactivity gain of 1.0%. For this void fraction in the 20-in.-high active portion of the ITC, the two-dimensional computation predicted 2.0% net reactivity gain. A more realistic appraisal of an accidental voiding in the ITC must consider voiding in axial regions below and/or above the active zone, in which case the maximum net reactivity gain would be much smaller than 2%, because an important factor in the larger net positive reactivity gain, due to partial voiding of ITC water, predicted by two-dimensional calculations is the increased importance of the axial reflectors; this effect is not included in the one-dimensional analysis. The comparison of one- and two-dimensional calculations is given in Fig. IV.A.5.

Fig. IV.A.5. Calculated Reactivity Effects of Partial Voidings of the Bulk Water in the Active Portion of the AARR Internal Thermal Column

V. NUCLEAR SAFETY

A. Materials Behavior, Equation of State, and Energy Transfer

1. Vapor Pressure of UO_2

The experimental apparatus for determination of the vapor pressure of UO_2 has been assembled. Difficulty with welding a circular diaphragm of thin tungsten sheet (0.005 in.) delayed fabrication until a technique was developed to machine electrochemically a circular area of 0.005-in. thickness in a more easily welded piece of 0.030-in. tungsten.

The apparatus will be mounted in the high-temperature furnace and the sensitivity of the diaphragm determined as a function of temperature. At room temperature a simple electrical contact method, using an ohmmeter, can determine the diaphragm position to 0.00025 in.

Initial experiments will use depleted uranium dioxide of unspecified stoichiometry to test the operation of the equipment before using a very well-characterized single-crystal sample provided by Battelle-Northwest.

2. Properties of Alkali Metals

The alkali metals are of interest as potential fast reactor coolants, and sodium is of special interest. Analysis of operational and safety characteristics requires knowledge of the density, thermal-expansion coefficient, compressibility, and heat of vaporization over a wide temperature range.

Since density data are available for cesium closer to its thermodynamic critical point than for the other alkali metals, theoretical and empirical relations were used to estimate its critical temperature, density, and pressure. This technique was first tested with the known characteristics of CO_2 and mercury. The estimated critical temperature and experimental heat of vaporization data for cesium were used to construct the relation between the entropy of vaporization and reduced temperature, i.e., the absolute temperature divided by the critical temperature. Because of the lack of sufficient high-temperature data for the other alkali metals, cesium was used as the "corresponding states" model for the other metals by a method similar to the estimation technique of Grosse,* who used mercury. Since mercury has two valence electrons and the alkali metals have only one, it is believed that the procedure used here represents an improvement over the estimates of Grosse. Estimates of the critical constants of the alkali metals were then used with experimental data to extrapolate properties to the critical point. These estimates are given in Table V.A.1.

*Grosse, A. V., J. Inorg. Nucl. Chem. 22, 23 (1961).

TABLE V.A.1. Estimated Critical Constants of the Alkali Metals

Material	Critical Temperature T_c (°K)	Critical Density ρ_c (g/cc)	Critical Pressure P_c (atm)	Critical Compressibility Z_c
Li	4310 ± 100	0.0637	985	0.3032
Na	2733 ± 40	0.1818	408.2	0.2302
K	2287 ± 40	0.1805	168.6	0.1946
Rb	2164 ± 40	0.3314	178.6	0.2594
Cs	2040 ± 40	0.4043	113.7	0.2233

The asymptotic relations that were used for behavior in the critical region were:

vapor density,* ρ_V

$$(1 - \rho_{VR}) = A_V (1 - T_R)^{B_V}, \quad (1)$$

where

$$\rho_{VR} = \rho_V / \rho_c; \quad T_R = T / T_c;$$

liquid density,* ρ_L

$$(\rho_{LR} - 1) = A_L (1 - T_R)^{B_L}, \quad (2)$$

where

$$\rho_{LR} = \rho_L / \rho_c;$$

heat of vaporization,**,† ΔH_v

$$\frac{\Delta H_v}{RT_c} = A_H (1 - T_R)^{B_H}, \quad (3)$$

where R is the gas constant. Also used was the "law of the rectilinear diameter"

*Miller, D., Reactor Physics Division Annual Report July 1, 1964 to June 30, 1965, ANL-7110, p. 398.

**Martin, J. J., and Edwards, J. B., A. I. Chem. Eng. J., 11(2), 331 (1965).

†Viswanath, D. S., and Kuloor, N. R., Can. J. Chem. Eng. 45, 29 (1967).

$$\frac{\rho_L + \rho_V}{2} = C - DT = \rho_C - D(T_C - T), \quad (4)$$

or

$$\frac{\rho_{LR} + \rho_{VR}}{2} = 1 + \frac{DT_C}{\rho_C} (1 - T_R), \quad (5)$$

and the relation*,**

$$\frac{\rho_{LR} - \rho_{VR}}{2} = A(1 - T_R)^B, \quad (6)$$

which comes from the lattice-gas model of the critical point or is an empirical generalization of the work of Guggenheim.[†] Constants fitted to these equations for the alkali metals are given in Table V.A.2 and can be used to estimate the properties where no experimental data exist.

TABLE V.A.2. Coefficients and Exponents for Eqs. (1), (2), (3), and (4)

Material	$\rho_{LR} - 1$		$1 - \rho_{VR}$		$\Delta H_V/RT_C$		Rectilinear Diameter Constants	
	A_L	B_L	A_V	B_V	A_H	B_H	C (g/cc)	D (g/cc/°K) $\times 10^4$
Li	8.785	0.792	1.143	0.227	4.78	0.296 ^a	0.2858	0.532
Na	4.097	0.682	1.422	0.389	4.83	0.377	0.5082	1.194
K	3.702	0.513	1.329	0.293	4.55	0.282	0.4554	1.202
Rb	3.722	0.625	1.582	0.424	4.61	0.301	0.8168	2.232
Cs	3.636	0.559	1.158	0.187	4.75	0.327	1.0150	2.939

^aBased on values from Clapeyron equation.

Details of the estimation technique and other results for sodium have been accepted for the Proceedings of the International Conference on the Safety of Fast Breeder Reactors, Aix-en-Provence (France), September 19-22, 1967.

3. Heat Capacity of Liquid UO_2

Work is continuing on the program to measure the heat capacity of liquid uranium dioxide, an important quantity in fast reactor safety calculations. The experimental method to be used involves induction heating of encapsulated UO_2 and measurement of the heat content by drop calorimetry. Initial experiments were designed to test the suitability of tungsten as a container for the UO_2 at temperatures above 3000°C. A series of tests (see Progress Report for September 1966, ANL-7255, p. 86) has shown that tungsten will indeed behave satisfactorily. Several designs of tungsten

*Ibid., page 143.

**Martin, J. J., and Edwards, J. B., A. I. Chem. Eng. J., 11(2), 331 (1965).

[†]Viswanath, D. S., and Kuloor, N. R., Can. J. Chem. Eng. 45, 29 (1967).

Fig. V.A.1. Tungsten Capsule for Measurements of UO_2 Heat Capacity

capsules for holding the liquid oxide have been tested. As a result of these tests, a double-walled capsule (see Fig. V.A.1) was chosen for initial measurements. This capsule style will minimize heat losses during the drop. A similar capsule without the outer wall will also be used in order to help evaluate problems of heat loss during the time the sample falls from the furnace to the calorimeter.

While experiments were in progress to test materials, fabrication methods, and design for sample capsules, the design of the entire calorimeter system was also proceeding. The final design is shown schematically in Fig. V.A.2. The

encapsulated oxide will be heated inductively and temperatures measured through a sight port and prism by means of a Pyrometer Instrument Company automatic optical pyrometer. Black-body holes will be provided in the capsule for temperature measurements (see Fig. V.A.1).

The capsule containing the UO_2 will be suspended by a tungsten wire which in turn is to be connected, via appropriate electrodes, across a high-voltage condenser bank. When dropping of the capsule into the calorimeter is desired, the condenser discharge will vaporize the tungsten suspension wire extremely rapidly and allow the capsule to fall. This simple scheme insures a vertical drop.

Considerable effort has been expended in various calibrations. A Pyrometer Instrument Company automatic optical pyrometer has been calibrated against secondary standard lamps using conventional procedures.* The pyrometer was found to be accurate to within the precision of the calibration when used to measure the freezing point of copper in a standard copper-point cell.

Measurement of the temperature rise in the calorimeter will be made using a Dymec quartz thermometer. The accuracy of this instrument has been checked by comparison with a platinum resistance thermometer, and also by measuring the triple point of water. Performance of the thermometer has been satisfactory. An electrical calibration system, needed to measure the energy equivalent of the calorimeter, has also been constructed.

* Kostkowski, H. J., and Lee, R. D., Temperature, Vol. III, Pt. 1, Reinhold Publishing Co., New York (1962).

Fig. V.A.2. High-temperature Calorimetric System for Measurement of the Heat Capacity of Liquid Uranium Dioxide

Initial experimental measurements of enthalpy will be carried out with tungsten. Measurements have been reported in the literature for tungsten up to 3100°K;* our results will be compared with these literature values before proceeding to uranium dioxide.

*Kirillin, V. A., Sheindlin, A. E., Chekhovski, V. Ya., and Petrov, V. A., Russian J. Phys. Chem. 37, 1212 (1963).

4. Physical Properties of Liquid UO_2

The lack of reliable physical property data for liquid uranium dioxide constitutes one of the major problems in the analysis of reactor hazards, both fast and thermal. As was pointed out by Bright,* information on the properties of liquid uranium dioxide is either nonexistent or sketchy. Data on such fundamental properties as viscosity, surface tension, compressibility, expansion coefficients, heat of fusion, thermal conductivity, and many others are necessary for reliable loss-of-coolant or reactivity-insertion safety analyses. The extreme temperatures and severe compatibility problems encountered in measuring these properties make the selection of appropriate techniques difficult. Fortunately, extreme accuracy is not essential for such data to be of value in reactor hazards analyses. However, present theories do not allow prediction of these quantities to even modest accuracy.

Several techniques are being considered which appear to be suitable for the determination of the viscosity of liquid uranium dioxide. These include the counterbalanced sphere method used by Bacon *et al.*** for determining the viscosity of fused silica, and the capillary flow method used by Ermolaeva *et al.*† for determining the viscosity of spinel melts. Techniques under consideration for the determination of surface tension are the dipping cylinder method of Shartsis and Smock,†† the analysis of frozen menisci as developed by Biery,‡ and the more classical techniques of pendent and sessile drops. Methods of measuring thermal conductivity or diffusivity, utilizing liquid films, are also under examination.

Vastly more difficult than the determinations discussed above is the experimental exploration of the equation of state of uranium dioxide. Several possible techniques are under consideration for measurement of pressure-energy relationships at extremely high temperatures. These include measurements of the speed of sound, determination of compressibility, and utilization of condenser-discharge methods.

B. Reactor Control and Stability

Methods are being developed for foreseeing potential hazards, to provide information for designing systems as free of hazards as possible, and for measuring dynamic performance, so that large liquid-metal fast breeder reactors can be designed to operate safely over their intended life spans.

*Bright, G. O., Nucl. Safety **8**, 116 (1967).

Bacon, J. F., Hasapis, A. A., and Wholley, J. W., Jr., Phys. Chem. Glasses **1, 90 (1960).

†Ermolaeva, E. V., Gyzenko, G. F., and Mirak'yan, M. M., Zhur. Priklad. Khimii **38**, 1725 (1965).

††Shartsis, L., and Smock, A. W., J. Res. Nat. Bur. Stand. **38**, 241 (1947).

‡Biery, J. C., J. Electrochem. Soc. **114**, 225 (1967).

1. Transfer Functions (Stability)

The definition of spatially dependent transfer functions and the interpretation of the measurements will be studied for various models of large fast reactors so as to determine how methods that have been used to study system stability from transfer-function measurements of small reactors, such as EBR-II, need to be modified for the various geometries of large fast reactors.

To gain an understanding of the various concepts of spatially dependent transfer functions, a generalized mathematical formulation has been introduced. From this formulation, various forms of spatial transfer function of a reactor system can be derived (e.g., point model, modal, nodal transfer functions, etc.). Since the generalized formulation includes the system characteristics as well as the input-output characteristics of instruments, the spatial effects due to the presence of these instruments can be accounted for in the derivation of the system transfer functions. This approach enables the study and comparison of the various forms of spatially dependent transfer functions from the same formulation.

C. Coolant Dynamics

Analyses and experiments are being used to develop analytical models that realistically describe both the coolant behavior and its mechanical interaction with the surrounding fuel structure during abnormal reactor conditions. The objective is to determine the extent to which rapid changes of coolant density or flow patterns adversely influence heat transfer and reactivity, and thus the safety, of sodium-cooled fast reactors.

1. Critical Flow

Because two-phase critical flow could occur during an accidental power surge in a sodium-cooled reactor and could lead progressively to detrimental voidage of coolant channels, shock phenomena, and pressure buildup, measurements are being made of critical flow rates, pressure profiles, qualities, void distributions, and sonic velocities in simulated fuel channels so that methods can be developed for predicting critical flow velocities, metastability, superheat, velocity ratios, and flow structure in high-velocity two-phase flow.

A program has been initiated to study the propagation of pressure pulses in two-phase flow. The prime objective is a basic understanding of the mechanisms involved so that the magnitude of these effects during an accident can be assessed. Steam-water and air-water mixtures will be used as the working media. The propagation speeds of finite pulses as well as the sonic velocity will be measured.

Construction of the test facility has begun and the test section is being fabricated. The instrumentation is being checked out.

2. Coolant Dynamics

Sodium superheat and expulsion are being investigated so that analytical models can be developed for predicting coolant behavior during various stages of reactor accidents.

a. Superheat. The independent and combined influences of pressure, heat flux, dissolved gas, oxide content, surface characteristics, system pressure-temperature history, and ionizing radiation are being measured to determine the mechanism and amount of liquid superheat that can build up before nucleate boiling begins in sodium under various simulated reactor conditions.

Data obtained from test runs show that system pressure, heat flux, and the pressure-temperature history significantly affect the incipient boiling superheats in sodium.

Fig. V.C.1. Effects of Heat Flux and Pressure on Superheat

Figure V.C.1 summarizes the effects of pressure for five different heat fluxes. The incipient boiling superheats decrease as the system pressure is increased; however, it is somewhat surprising that the superheats increase markedly as the heat flux increases. It is anticipated that this latter effect will diminish in importance at larger heat fluxes.

b. Expulsion. Liquid superheats, vapor formation and distribution, and pressure propagation during coolant expulsion from a simulated

reactor channel are being measured to enable formulation of an analytical model that describes how reactor coolant behaves during transient heating in a power surge or loss of coolant flow. Nonflowing tests to determine the effects of heating rates, subcooling, inertial head, dissolved gas, channel geometry, and surface finish are providing the foundation for experiments with rapidly heated flowing sodium.

The programmed electron-beam heater is now operating. However, one of the function generators which controls the horizontal sweep of the electron "gun" was defective; the vendor is making repairs, after which final assembly of the test system will commence.

Assembly of the eddy-current interface detectors is being completed.

3. Convective Instability

Experiments will yield fundamental data on transient free-convection heat transfer in narrow-channel liquid-metal systems so that heat-transfer effects in fast-reactor startup or loss-of-cooling accidents can be analyzed. Effects of Prandtl and Rayleigh parameters on system heat transfer are being determined analytically so that a readily usable nondimensional correlation can be derived.

Sonic booms from supersonic aircraft have shattered the quartz test sections. The overflights are scheduled to cease by the end of September, so this problem should end. The system has been filled. Work on all other minor details has been completed so that data can be obtained soon after a new test section is installed.

4. Core Component Dynamics

Analytical models are being developed to explain and predict the results of out-of-pile experiments so as to determine the manner in which coolant expulsion and core structural deformation interact so that the variation of pressure with time during a power transient can be interpreted. The investigations of coolant-structure coupling will proceed from simple configurations to the more complex situations inside a reactor and to studies of wave propagation, impulse loading of shells, and the energy-absorbing capabilities of core components.

The problem concerning the effect that coolant-structure coupling during coolant expulsion has on the pressure-time function of a flat-walled rectangular channel (see Progress Report for February 1967, ANL-7308, pp. 84-85) has been extended toward defining a coupling model for a cylindrical channel. Previous reports discussed some of the particulars of the elastic analyses under way, such as the existence of a "characteristics deformation length," its independence of load magnitude, and its dependence on load configuration (see Progress Reports for July 1967, ANL-7357, pp. 143-144, and August 1967, ANL-7371, pp. 120-122). These subjects are now being extended into the plastic shell response for loads below rupture. Models are being examined for time-dependent loadings. The inertial characteristics of the shell also will be added so that the shell response can be fed back into the loading function.

Because of the interesting features of the elastic case for the general loading configurations used so far, a preliminary coupling model is being studied in terms of an assumption of a one-dimensional shock wave with an exponentially decaying spatial and temporal intensity, e.g.,

$$p(x, t) = P_0 e^{-\left(\frac{t}{\theta} + b \frac{|x|}{r}\right)}, \quad (1)$$

where p is pressure, x an axially oriented shell coordinate, t time, P_0 a maximum pressure, θ a time decay constant, b a spatial decay constant, and r a shell radius.

The governing differential equation for the elastic response of the shell (approximated as a beam of finite length ℓ_0 on an elastic foundation; ℓ_0 is the characteristic deformation length defined by the load distribution) can be written as

$$m \frac{\partial^2 y}{\partial t^2} + EI \frac{\partial^4 y}{\partial x^4} + ky = 2P_0 e^{-\left(\frac{t}{\theta} + b \frac{|x|}{r}\right)} - \rho c \frac{\partial y}{\partial t}, \quad (2)$$

where y is radial shell displacement, m the mass per unit area including virtual mass of fluid, EI the flexural rigidity, k an elastic constant for the "foundation," ρ fluid density, and c the speed of sound in the fluid. The first term on the right in Eq. (2) is the blocked pressure and the second the radiation pressure. The deflection can be represented by the series

$$y(x, t) = \sum_n \phi_n(x) q_n(t). \quad (3)$$

If we assume effective pinned end conditions, the shape function

$$\phi_n(x) = \cos \frac{(2n-1)\pi x}{\ell_0} \quad (4)$$

satisfies the implied boundary conditions.

A Laplace transform solution gives, as the equation for the generalized coordinates,

$$q_n(t) = \frac{8P_0 \left[(-1)^n \frac{(2n-1)\pi}{\ell_0} e^{-\frac{b}{r} \frac{\ell_0}{2}} - \frac{b}{r} \right]}{m\ell_0 \left[\frac{(2n-1)^2 \pi^2}{\ell_0^2} + \left(\frac{b}{r} \right)^2 \right] \left[\frac{A}{\theta} - \frac{A^2}{4} - \frac{1}{\theta^2} - \omega_n^2 \right]} \left\{ e^{-\frac{t}{\theta}} - \left[\cos \omega_n t + \left(\frac{\frac{A}{2} - \frac{1}{\theta}}{\omega_n} \right) \sin \omega_n t \right] e^{-\frac{A}{2} t} \right\}, \quad (5)$$

where

$$A = \frac{\rho c}{m};$$

$$\omega_n^2 = \left\{ \frac{Eh}{mr^2} \left[1 + \frac{(2n-1)^4 \pi^4}{4\beta^4 \ell_0^4} \right] - \frac{A^2}{4} \right\};$$

$$\beta = \frac{1.28}{\sqrt{rh}}.$$

The deflection due to the applied time- and spatial-dependent pressure loading can now be written from Eq. (3) as

$$y(x, t) = P_0 \sum_n \bar{q}_n(t) \cos \frac{(2n-1)\pi}{\ell_0} x, \quad (6)$$

where $\bar{q}_n$ is q_n/P_0 , and q_n is given by Eq. (5). The amplitude of the radial shell deflection is proportional to the maximum pressure P_0 .

D. TREAT Operations

Neutron radiography of the capsules from EBR-II subassembly XO11 was completed. High-quality radiographs were obtained.

To evaluate further the potential of the neutron radiography facility the following items were radiographed:

1. two PBF fuel rods which had been subjected to TREAT transients;
2. a GE capsule containing vibratory-compacted, mixed-oxide fuel which had been subjected to a TREAT transient;
3. two vibratory-compacted UO_2 fuel rods which had been subjected to TREAT transients in the metal-water transparent capsule series;
4. one EBR-II assembly top adapter containing a melt-wire assembly.

Two metal-water transparent capsules without fuel were subjected to transients in an effort to determine the source of in-capsule clouding seen in the latest experiments. The films taken of these capsules are being developed.

E. Gaseous Effluent Studies

1. Plutonium Volatility Safety

a. Effluent Gas Treatment: Chemistry of Tellurium Fluorides.

The radiochemically toxic tellurium fission product compounds are troublesome components of the process streams of the fluoride volatility process. The required decontamination factor (DF) for tellurium has been calculated to be ~ 1300 . Gas-flow sorption studies (see Progress Report for July 1967, ANL-7357, pp. 144-145) have shown that activated alumina is an excellent sorbent for TeF_6 in fluorine-containing gas systems.

A two-part experiment was performed to determine the TeF_6 surface loading on activated alumina and to determine the extent of TeF_6 removal by fluorine. In the first part of the experiment, air containing 500 ppm TeF_6 and 500 ppm F_2 was passed through two 6-in. packed beds of activated alumina in series at $\sim 25^\circ\text{C}$ and at a gas flow rate of 20 ft/min. The results showed that 25% TeF_6 surface loading can be achieved across the lower 2 in. of the first bed, and that a DF of 2×10^4 is maintained across the entire first bed.

Extrapolation of a log-log plot of DF versus percent TeF_6 loading on a 2-in. bed of activated alumina obtained in earlier work indicated that it should be possible to obtain about 28% TeF_6 surface loading. The 25% TeF_6 surface loading obtained in current work is approximately the value predicted from results of the earlier work.

In the second part of the experiment, an air stream containing 12,200 ppm fluorine was passed through the two beds at 20 ft/min. About 5% of the sorbed TeF_6 in the first bed was displaced by fluorine. During the period that TeF_6 was displaced, the bed temperature increased from 25 to 50°C and then dropped to 25°C , suggesting that reaction occurred only at the active surface sites of the alumina.

F. Containment

1. One-dimensional Code for Analyzing Containment Structures

One-dimensional computer codes are being developed to predict the response of the LMFBF containment structures to core excursions ranging from a mild one to one involving hundreds of megawatt-seconds. This work will provide design guidance and a realistic basis for analyzing the safety of LMFBFs.

Dynamic elastic response of a finite circular cylinder to axisymmetric internal pressure pulses is being calculated (see Progress Report for August 1967, ANL-7371, p. 123). As the first step, the natural frequencies

of the cylinder for various boundary conditions are being determined with an analog computer for a wide range of Poisson's ratio, so that these results can be applied to a range of structural materials used in primary containment structures. Once the natural frequencies have been determined, the dynamic response of the cylinder to an internal pressure pulse will be determined by the finite integral transform technique. From this formal solution the dynamic stresses and displacements will be calculated for various types of pressure pulses.

The scope is being extended to include the effects of concurrent core phenomena. This is necessary for the lower-energy releases because the neutronics is affected by the structural deformation of the core. Hence, coupled neutronics-thermoelastic equations will be derived for this aspect of the work to obtain insight into the problem. Computer-code modules will be developed so that the work can be incorporated into postburst phenomena studies.

2. Two-dimensional Code for Analyzing Containment Structures

The objective and scope of this task are the same as for the one-dimensional case (see Sect. V.E.1 above), but the problem is considerably more complex.

The differential equations of shock hydrodynamics in two spatial dimensions have been formulated in Lagrangian coordinates (see Progress Report for August 1967, ANL-7371, pp. 123-124). The rectangular Lagrangian meshes will be used in the evaluation of difference equations. In one dimension, the spatial derivative in the difference equation can be obtained easily from Taylor's expansion. However, in two dimensions, the use of Taylor's expansion to spatial derivatives will result in a set of overdetermined equations. The overdeterminacy of the difference equations is inherent in the system of rectangular meshes. Attempts have been made to devise schemes that avoid this overdeterminacy. The use of triangular meshes or least-squares calculation in rectangular meshes have been considered. Although both methods eliminate overdeterminacy, the triangular-mesh method has the disadvantage of not fitting together well among meshes and on boundaries; the least-square method is very time-consuming and is susceptible to instability. A number of alternative difference schemes have been studied: (1) the midpoint method, (2) Green's transformation, and (3) the virtual work method. They are all based on rectangular meshes. It is very difficult at this stage to predict which form of the difference equations is the best for the purpose; all seem to yield satisfactory results if the mesh size is sufficiently small and if the meshes are not severely distorted. The least number of arithmetic steps for evaluation of spatial derivatives will probably be the decisive factor in the choice of difference form for use in the two-dimensional computer code. Further studies are under way.

VI. PUBLICATIONS

Papers

Purification of Inert Atmospheres

C. E. Johnson, M. S. Foster, and M. L. Kyle

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L. Leibowitz and L. Mishler

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R. J. Beals and R. S. Kern

Bull. Am. Cer. Soc. 46(9), 901 (September 1967) AbstractANL Reports

ANL-7208

ZPR-9 ASSEMBLIES NO. 6-9 CRITICAL EXPERIMENTS

R. C. Doerner, W. G. Knapp, R. A. Karam, and
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ANL-7248

CRITICAL STUDIES OF THE SOUTHWEST EXPERIMENT
FAST OXIDE REACTOR (SEFOR) IN ZPR-3 (Assembly 47)R. L. McVean, A. Weitzberg, J. A. Kremser,
A. M. Leridon, and J. K. Long

ANL-7303

THE EFFECTIVENESS OF CONTROL-ROD MATERIALS

H. P. Iskenderian

ANL-7325

CHEMICAL ENGINEERING DIVISION SEMIANNUAL
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