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**QUARTERLY PROGRESS REPORT
IRRADIATION EFFECTS
ON REACTOR STRUCTURAL MATERIALS
FEBRUARY, MARCH, APRIL, 1968**

JUNE 1968

**AEC RESEARCH &
DEVELOPMENT REPORT**

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QUARTERLY PROGRESS REPORT
IRRADIATION EFFECTS
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FEBRUARY, MARCH, APRIL, 1968

Participating Organizations:

Argonne National Laboratory
Battelle Memorial Institute
Brookhaven National Laboratory
General Atomic Division
Idaho Nuclear Corporation
Materials Research Laboratory
Naval Research Laboratory
Nuclear Materials and Propulsion Operation
Oak Ridge National Laboratory
Pacific Northwest Laboratory
Westinghouse Electric Corporation

June 1968

Edited by:

T. T. Claudson

BATTELLE MEMORIAL INSTITUTE
PACIFIC NORTHWEST LABORATORY
Richland, Washington

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SUMMARYARGONNE NATIONAL LABORATORY

Irradiation of V-20 Ti to a fluence of 3×10^{22} had no effect on the tensile ductility of the alloy at temperatures from 400 to 750 °C. The yield strength of the alloy was increased between 20 and 50% at temperatures below 650 °C as a result of irradiation; but at a test temperature of 750 °C there was no apparent increase in yield strength due to irradiation.

The minimum creep rate, creep-rupture life, and total elongation of V-20 Ti alloy at 650 °C did not appear to be affected by irradiation up to a fluence of 2×10^{22} .

Irradiation of V-15 Ti-7.5 Cr alloy to a fluence of 5×10^{21} resulted in small decreases in tensile

ductility at temperature from 550 to 750 °C. The yield strength was increased by a maximum of approximately 50% below 650 °C, but at 750 °C, irradiation had no effect on the yield strength of the alloy.

The maximum increase in tangential rupture strength of V-15 Ti-7.5 Cr alloy at 550 °C fell halfway between that observed for Type 304 SS (least increase) and Inconel 625 (highest increase) after irradiation to a nominal fluence of 5×10^{21} . The minimum circumferential ductility of irradiated V-15 Ti-7.5 Cr at 550 °C was in the same range as that observed for irradiated specimens of Hastelloy X, Inconel 625, and Type 304 SS.

BATTELLE MEMORIAL INSTITUTE

Austenitic alloys with various nickel contents were irradiated to a fast fluence of 1.5×10^{21} n/cm² and mechanically tested to determine the role of nickel on the elevated temperature irradiation induced embrittlement. It was determined that both the nickel and non-nickel austenitic alloys were embrittled by irradiation when tested at elevated temperatures. Stress rupture tests at 650 °C on Type 347 SS specimens irradiated to a fast fluence of 2.1×10^{22} n/cm² indicated that irradiation caused a significant reduction in rupture life.

Attempts were made to grow single crystals of Type 347 SS by utilizing electron beam zone melting, controlled solidification and strain annealing. Preliminary attempts at growing single crystals of stainless steel were unsuccessful.

Preliminary fracture toughness values of IX WOL specimens fabricated from the irradiated PM-2A pressure vessel were determined. The fracture toughness at -20 °F was found to be 48 to 78 ksi√in. for 7.7×10^{18} n/cm² and 4.2×10^{10} n/cm², respectively.

BROOKHAVEN NATIONAL LABORATORY

Commercially available cobalt-base alloys and a few experimental cobalt alloys were tested to determine their resistance to elevated temperature embrittlement after irradiation. Several commercial cobalt base alloys; namely Haynes 25 and UMC0 51 were found to be potential fuel cladding materials at 650 °C. These alloys are attractive over Type 304 SS because of their higher strengths.

Cobalt-base alloys are susceptible to decreasing ductility with increasing deformation temperature after irradiation. The mode of failure at 800 °C is grain boundary tearing similar to that observed in stainless steels and nickel base alloys.

Using a process of deforming Type 304 to 18% at 77 °K, annealing at 705 °C, deforming again to 18% at 77 °K and followed by annealing at 760 °C for 16 hr, it was possible to obliterate the prior austenite grain boundaries and develop a fine grain structure (ASTM 11) with a fine precipitation of carbides. After irradiation to a fluence of 1.3×10^{20} , the elevated temperature strength of material is lower but the ductility is significantly improved.

Metallographic examination indicates finer cracks than in the Type 304 SS annealed at 1050 °C but the fracture is predominantly intergranu-

lar. We believed that although the prior grain boundaries appear to be obliterated in the microstructure, fracture nevertheless follows the old boundaries. It is suggested that heavier deformation and starting out with a finer grain material might provide additional improvement.

Work has proceeded in the study of the Stage III recovery characteristics of cold-worked niobium polycrystals in an attempt to consolidate the interpretation, previously given, that the recovery involves three separate substages; i.e., the clustering of self-interstitials, their collapse to form dislocation loops and the migration of oxygen atoms to matrix traps. Experiments were performed to determine the temperature dependences of specimens strain-aged within Stage III. This data indicates that clusters, containing on the order of 5 interstitials, are formed immediately prior to collapse. Calculations on the jump frequencies and jump distances of the migrating defects are also given and discussed.

A measurement of the temperature dependence of the yield stress of irradiated tungsten crystals has been made. It is found that the temperature dependence in irradiated tungsten crystals differs from that of unirradiated crystals.

IDAHO NUCLEAR CORPORATION

In hydrogen embrittlement studies on A302B, A542 and HY-80 steels during the past quarter, 309 specimens have been encapsulated for irradiation and the behavior of hydrogen in the unirradiated steels has been investigated. During hydrogen charging the permeability of irradiated HY-80 samples is greater than for the unirradiated. Possible reasons for the phenomena are that irradiation changes the surface activity for hydrogen adsorption or disassociation, or that the activation energy for transport

of hydrogen is decreased. The stress rupture properties of unirradiated A302B steel show a slight tendency toward time dependent fracture with increased hydrogen content; the HY-80 steel exhibits a slightly greater tendency than the A302B.

In the fatigue studies of fast reactor cladding alloys and of structural alloys, 210 specimens for irradiation in two MARK B-7, EBR-II subassemblies have been prepared and delivered to Argonne National Laboratory for insertion.

MATERIALS RESEARCH LABORATORY, INC.

The fracture toughness of A302B steel at room temperature, 0, and -70 °F was evaluated with one and two-inch thick contoured double cantilever beam (DCB) specimens. To establish the effect of rate on thickness requirements, and to assess the strain rate sensitivity of K_{IC} , both initiation and arrest toughnesses were measured in tests covering a wide range of loading rates.

The effect of thickness on the measured toughness, K_C , is discussed with reference to the parameter

$$\beta_C = \frac{1}{B} \left(\frac{K_C}{\sigma_Y} \right)^2$$

where B is specimen thickness and σ_Y is the yield stress at the temperature and strain rate of the test. Measurements at each temperature defined a single level of K_C for initiation, independent of

loading rate and thickness, when the criterion $\beta_C \leq 1.0$ was satisfied. The values were 85 to 90 ksi- $\sqrt{\text{in.}}$ at room temperature and 55 to 60 ksi- $\sqrt{\text{in.}}$ at 0 and -70 °F. As an alternative to identifying these numbers as K_{IC} , an extrapolation function which associates K_{IC} with an infinite plate thickness was applied to the data. The extrapolation defined $K_{IC} = 70$ ksi- $\sqrt{\text{in.}}$ at room temperature and $K_{IC} = 55$ ksi- $\sqrt{\text{in.}}$ at the 0 and -70 °F which values are consistent with the measured lower bound of arrest toughness.

There was essentially no strain rate sensitivity of K_{IC} . The difference between initiation and arrest toughness was small, and the dependence of K_C on loading rate could be attributed to the rate dependence of σ_Y .

NAVAL RESEARCH LABORATORYCONTROLLING THE RADIATION EMBRITTLE-
MENT SENSITIVITY OF Ni-Cr-Mo WELD
DEPOSITS BY VARYING THEIR CHEMICAL
COMPOSITION

The effect of variable chemical composition on the radiation embrittlement sensitivity of weld deposits suitable for joining A543 steel is being investigated in a cooperative program between NRL and the Babcock and Wilcox Company (B&W). The first phase of this program has been completed. In this exploratory phase, Charpy-V specimens from four experimental manual metal arc weld deposits and from two submerged arc commercial weld metals were irradiated to a neutron fluence of $\sim 3.5 \times 10^{19}$ n/cm² (E > 1 MeV) at 550 °F together with specimens from an 8 in. A543 reference plate. Specimens from the two commercial weldments (Linde MI-88 and Linde 100 filler wire) exhibited Charpy-V 30 ft-lb transition temperature shifts of 415 °F and 530 °F, respectively, while specimens from the reference plate showed an increase of only 105 °F. The 30 ft-lb transition temperature shifts of the four experimental weld deposits ranged from ~ 45 °F to ~ 200 °F which indicates a significant improvement over the commercial weldments and, in two cases, an improvement over the radiation embrittlement resistance of the A543 reference plate. The improved radiation embrittlement resistance of

the experimental weld deposits is attributed largely to their low copper content (0.02 to 0.14% versus 0.24 and 0.42% for the two commercial weldments). These findings substantiate previously reported data on the effect of copper as a radiation embrittlement variable.

INFLUENCE OF PRIOR TEMPER EMBRITTLE-
MENT ON THE IRRADIATION RESPONSE OF
Ni-Cr-Mo STEEL

The influence of temper embrittlement on the subsequent irradiation response of a quenched and tempered Ni-Cr-Mo steel plate has been examined. Charpy-V specimens of temper embrittled and nonembrittled (brine quenched from tempering) plate were irradiated at 650 °F and under a 750-650-750 °F temperature cycle. Neutron fluences averaged 2.5×10^{19} n/cm² (E > 1 MeV) for both exposures.

Radiation-induced transition temperature increases for the temper embrittled condition were found to match well the increases for the corresponding nonembrittled plate condition. The results of this investigation strongly suggest that temper embrittlement introduced prior to nuclear service will not measurably alter the apparent radiation embrittlement sensitivity of Ni-Cr-Mo steel at temperatures in the range of 650 to 750 °F.

RELATIVE 550 °F IRRADIATION RESPONSE
OF BASE PLATE, WELD METAL, AND WELD
HEAT AFFECTED ZONE OF A 7 1/2 IN.
THICK A533B CLASS 1 PRODUCTION
WELDMENT

The notch ductility properties of base plate, submerged arc weld deposit, and weld heat affected zone (HAZ) of one 7 1/2 in. thick production weldment of A533B Class 1 steel have been evaluated following irradiation at 550 °F to a fluence of 1.7×10^{19} n/cm² ($E > 1$ MeV). Transition temperature increases of both base plates were very similar (70 versus 85°) but significantly less than that of the weld deposit (200°). It is suggested that the relatively high copper content of the weld metal (0.22%) was partially responsible for its high radiation embrittlement sensitivity. The HAZ properties before and after irradiation were as good as if not superior to, base plate properties for the same exposure condition.

DROP WEIGHT NDT VERSUS CHARPY-V
ENERGY ABSORPTION LEVEL IN 6 3/8 IN.
TYPE A533B CLASS I AND II STEEL PLATE

A correlation between the drop weight NDT and Charpy-V energy absorption level of 6 3/8 in. Type A533B Class I and II steel plates has been made to examine the Charpy-V energy index for this type of steel. Experimental results indicate that the Charpy-V 30 ft-lb energy absorption,

commonly used as an index of the NDT of A302B pressure vessel steel, can not be applied to the A533 Class I or II plates studied. The average Charpy-V energy at the NDT temperature was found to range between 60 and 85 ft-lb for both strength levels. Additional work on other heats of A533B steel is in progress to ascertain the most appropriate Charpy energy index for NDT assessment of this steel.

MECHANICAL PROPERTIES EVALUATION
OF PM-2A REACTOR PRESSURE VESSEL
STEEL

A previous progress report gave details of the sampling of the PM-2A reactor pressure vessel for metallurgical test specimens and also the Charpy-V notch ductility results. Evaluations are now complete on post-irradiation annealing of Charpy-V specimens for comparison with matching annealing scheduled conducted on test reactor irradiated specimens of the PM-2A sister forging. Tension test specimen evaluations have been completed at two temperatures, +72 °F, and -20 °F, representing the acid sharpening treatment and failure temperatures, respectively. A calculated neutron-spectrum analysis of the PM-2A reactor vessel wall was performed and is discussed. The mechanical properties developed have been evaluated through a failure analysis using the generalized Fracture Analysis Diagram concept.

NUCLEAR MATERIALS AND PROPULSION OPERATION

EBR-II irradiation at 540 °C to 0.27 to 2.7×10^{20} n/cm² ($E_n \geq 1$ MeV) of Incoloy 800 increased the minimum creep rate and reduced the time to rupture compared to unirradiated controls at a test temperature of 540 °C: On the other hand, at a test temperature of 705 °C the minimum creep rate was reduced and the rupture time increased compared to unirradiated control specimens. At both test temperatures, irradiation reduced the

fracture elongation by a factor of 3 to 4 compared to unirradiated controls.

Low-cycle plastic fatigue tests of 316 SS irradiated to 2.35×10^{19} (thermal) and 3.06×10^{18} ($E_n \geq 1$ MeV) at reactor ambient temperature show a radiation induced factor of approximately 3.6 loss in fatigue life (N_5) at 650 °C for a constant strain rate of 4×10^{-5} sec⁻¹ and a strain range of either 1 or 2%.

OAK RIDGE NATIONAL LABORATORY

METALS AND CERAMICS DIVISION

The Oak Ridge National Laboratory program on radiation effects in fuel cladding materials includes studies of the high-temperature irradiation embrittlement and other technologically important aspects of radiation damage at high neutron fluence. The work centers around Types 304 and 316 SS, Incoloy 800, Hastelloy N, and the effects of titanium concentration on radiation damage. Associated with the program are studies of the effects of tubing fabrication variables on the properties of Types 304 and 316 SS and related compositions.

Evaluation of the postirradiation mechanical properties of the low-carbon and high-carbon 1500 lb heats of titanium-modified Type 304 SS is continuing. Thus far, the high-carbon alloy exhibits higher strength and lower ductility than the low-carbon heat.

Tubing fabrication studies indicate that the low-carbon titanium-modified Type 304 SS can be successfully fabricated to FFTF tube size by a number of techniques. However, some of the fabrication variables differed from those used on standard Type 304 SS.

Some of the existing small heats of Type 316 SS containing various amounts of titanium have been irradiated in the Oak Ridge Research Reactor and are being tensile and creep-rupture tested. The few tests conducted thus far indicate that the titanium-modified compositions are stronger and, under creep-rupture conditions, are more ductile than the standard alloy.

The Oak Ridge National Laboratory structural materials irradiation experiments for insertion into Rows 2 and 7 of the EBR-II are at the EBR-II site awaiting installation in the reactor at the beginning of Run 28.

PACIFIC NORTHWEST LABORATORYIRRADIATION DAMAGE TO REACTOR METALSREACTOR METALS RESEARCHAlloy Selection

A 500 lb quantity of 1/2 in. diam SS rod of alloy FV-548 has been obtained from Firth Vickers Stainless Steels, Ltd., England. Procurement of tubing material from the same heat as the rod is under negotiation.

Continuing the GE-APED superheat fuel-clad materials investigations, thermally aged controls for the alloy groups irradiated in the GETR were tensile tested at 704 °C. Results are compared with the tensile properties of the irradiated material.

Irradiation Facilities Operation

A total of 182 specimens were discharged from the ETR at the conclusion of Cycles 93 and 94. The variety included tensile, creep, charpy impact, DCB (double cantilever beam), thin foils, stress-to-rupture, and corrosion specimens.

Tensile data reports have been recorded, processed, and stored in the REM (Radiation Effects on Metals) computer program for 5506 individual tests.

Irradiation Damage to Stainless Steels

Annealed and cold-worked 348 SS irradiated in the G-7 loop to a fast

fluence of 8.1×10^{21} n/cm² (E > 1 MeV) has been tested. The yield strengths are increased for irradiated specimens in the annealed condition at all test temperatures. The ductility is very low.

The tensile data on martensitic 410 SS irradiated at 60 and 290 °C are compiled.

Electron-Microscopy of ETR-Irradiated Stainless Steel

The electron microscopy examination continued on AISI type 304 SS irradiated in the ETR to 6×10^{21} n/cm² (E > 1 MeV) at 290 °C. The cavities, which are sufficient to cause the observed high temperature hardening, persist to the highest postirradiation heat treatment temperature, 1090 °C. Observations indicate that the cavities are stabilized and in equilibrium with the surface tension. However, the amount of helium gas produced by transmutation was less than one-tenth the amount required to balance the surface tension of the cavities. The same irradiated 304 SS was given a high-pressure high-temperature anneal and examined by electron microscopy. Preliminary analysis of the electron micrographs of the irradiated steel having the high-pressure high-temperature anneal showed that approximately 2×10^{13} cavities/cm³ with an average diameter of 700 Å were still present. The results definitely indicate that the

cavities do not respond like voids, but behave like gas-filled bubbles.

Irradiation Damage to Nickel-Base Alloys

Preliminary data from stress-rupture tests at 1350 °F on specimens of Hastelloy X-280, Inconel 600, and Inconel X-750 after irradiation at 1250 °F to a fast fluence of approximately 1×10^{20} n/cm² showed reductions in rupture life compared to controls ranging from a factor of 10 for Hastelloy X-280 and Inconel 600 to a factor of 300 for Inconel X-750.

Corresponding reduction after irradiations at 540 °F were only about a factor of three for all three alloys. The higher reductions after the 1250 °F irradiation are probably indicative of effects of combined thermal and irradiation-induced instabilities.

In-Reactor Measurements of Mechanical Properties

In-reactor and unirradiated control creep tests conducted on AISI Type 304 SS both showed similar discontinuous yielding upon loading. The in-reactor test fractured at 13% strain while the control test is still in progress.

A faster creep rate was measured for an annealed Zry-2 in-reactor creep test conducted at 315 °C and 10.6 kg/mm² (15,000 psi) stress than for a corresponding unirradiated control test.

In-reactor creep data for Zr-2.5 wt% Nb between 14- and 32-kg/mm²

stress show little, if any, dependence of the creep rate on stress at 300 °C.

Grain Boundary Stabilization

A study of recrystallization and grain growth in metals containing small concentrations of inert gas has recently been initiated.

An apparatus for injecting helium atoms into nickel powder by ion bombardment has been set up and operated. The amount of helium introduced is dependent upon the time of bombardment and the potential used.

Nickel powder samples, with and without helium, are being compacted under different conditions, and the recrystallization and grain growth characteristics of these materials will be investigated.

Fracture Studies

Fracture mechanics studies of A533-B quenched and tempered 12 in. plate material were initiated. The initial studies are directed at determining size effects of the Double Cantilever Beam specimen. Fracture toughness measurements at the 1/2 T position of this plate for 1/2 and 1 in. thick specimens yielded basic results whereby size criteria of the DCB specimen can be ascertained.

Heavy Section Steel

A paper entitled, "Dynamic Changes in the Internal Stress Fields of High Purity Iron," was presented at the AIME Annual Meeting in New York, February 26 to 29, 1968.

Work in progress, relating fracture toughness to work hardening behavior in the neighborhood of a crack tip, is described.

High Pressure Studies

The purpose of this phase of the program is to study the effects of ultra-high pressures (1,000 to 60,000 bars) on the production, retention, and subsequent microscopic configuration of the damage produced in irradiated solids. In addition, the high pressure synthesis of new phases and the high pressure alteration of mechanical properties will be investigated as possible methods of providing more radiation damage resistant materials for nuclear reactors.

In-Reactor Corrosion of Zirconium Alloys

Three lots of Zry-2, previously characterized in the Engineering Test Reactor G-7 loop in pH-10 LiOH, ~ 1 ppm O_2 , were exposed in the G-7 loop in pH-10 NH_4OH < 0.05 ppm O_2 . In-flux corrosion rates were only slightly accelerated on as-etched specimens. Corrosion rates were accelerated on all prefilm specimens, but the response varied with prefilm treatment. In-flux hydrogen pickups were relatively low except on a specimen have a thick (~ 250 mg/cm²) prefilm oxide.

FAST REACTOR SUPPORTING STUDIES

Fast Neutron Mechanisms

Nonisothermal gas evolution techniques have been developed and used to investigate the temperature-sensitive hydrogen effusion characteristics of the austenitic stainless steels over the 200 to 900 °C temperature range. The observed gas evolution behavior is not representative of a simple, matrix-diffusion controlled effusion process, and it is sensitive to small differences in alloy composition. In addition, cold-working significantly alters the normal effusion characteristics and appears to facilitate the gas evolution process.

EBR-II Irradiations

The effects of swelling in austenitic stainless steel induced by fast reactor exposure near one half the absolute melting point are being investigated. The greatest decrease in density measured to date is 0.34% for AISI 304L irradiated to 2.7×10^{22} n/cm² total EBR-II fluence near 800 °F.

Fuel Cladding Characterization

The purpose of this program is to determine the lot to lot variation of selected mechanical properties in two lots of AISI 304 SS tubing. The

large number of biaxial tests to be run has necessitated a further modification of the CANEL test stands.

Structural Materials and Fuel Cladding Studies

Biaxial stress-to-rupture tests have been performed on FFTF prototype fuel clad material representing the as-received (slightly cold-worked) and annealed conditions of AISI type 304 SS. The test environment was 1400 °F (760 °C) helium.

Rupture strain for the as-received material was consistently greater than for the annealed material. The highly duplex grain size of the annealed material is noteworthy in comparison to the regular and somewhat finer grain size of the as-received condition.

Test Facilities

Construction is approximately 85% complete on a facility to test irradiated- and unirradiated-candidate structural and cladding materials in sodium and controlled gas atmosphere for the FFTF program. The transformer supplying power to this building will be moved next to the building. The three tests so far completed after alignment showed that the test frames have less than 10% bending moment.

A furnace liner containing sodium has been placed in service on the air environment creep test equipment to provide a longer uniform temperature

hot zone in the test furnace. A test temperature of 1200 °F $\pm$ 1 °F and temperature gradient of $\pm$ 0.5 °F along a 2 in. sample was maintained during 500 hr of testing. The ASTM recommended specification for temperature control and temperature gradient at 1200 °F is $\pm$ 3 °F. Acceptance tests were carried out on the liquid metal loop in accordance with manufacturers recommendations. This section contains one unshielded test loop for test of prototype assemblies and control samples. An inert atmosphere glove-box for handling high purity alkali metals has passed the specified performance tests and will be shipped. A problem has been encountered in transferring and measuring sodium under reduced cover gas pressures in that dissolved helium must be outgassed from the liquid sodium before accurate measurements can be made.

RADIATION EFFECTS ON METALS - O5 PROGRAM

This program is concerned with establishing the combined effect of impurities and neutron radiation on the properties and structure of specific metals. Conditions during radiation of molybdenum, nickel, and rhenium are being varied to provide information on higher neutron flux, higher integrated exposure, and higher irradiation temperature. Post-irradiation studies include thermally activated recovery of radiation induced changes.

ATR GAS AND WATER LOOP OPERATION AND
MAINTENANCE

PNL is proceeding with R&D programs designed to assist the various GCL vendors in their fabrication problems. In addition, work is underway to assist the AE and AEC in the

acceptance standards required for the components in the vendors' plants.

Water chemistry for the ATR water loop is being completed. Test specimens and hanger rods are being prepared for startup of the ATR. A program document outlining the various tests to be completed in the water cooled loop is being published.

WESTINGHOUSE ELECTRIC CORPORATION

The irradiation of the first three capsules was completed in the Babcock and Wilcox Test Reactor (BAWTR) February 12, 1968. The three capsules had a total exposure of 132 EPFD or an estimated fluence of 1.3×10^{19} .

Machining of the specimens (WOL's, Compact K_{IC} , Charpy-V notch, tensile, and drop weight) required for the pre-irradiation testing of the 12 in. plate of ASTM-A533 Grade B Class I steel was completed this report period.

Babcock and Wilcox completed the assembly and checkout of the second three test capsules. The capsules contained specimens fabricated from the 12 in. thick plate of ASTM-A533 Grade B Class I steel. The irradiation of the second three test capsules in BAWTR was initiated March 29, 1968. The irradiation of these capsules will continue through October 1968.

THE EFFECTS OF IRRADIATION ON THE MECHANICAL PROPERTIES
OF FAST REACTOR CLADDING MATERIALS

Argonne National Laboratory

R. Carlander

The effects of fast neutron irradiation at elevated temperatures on the uniaxial and biaxial mechanical properties of vanadium alloys, AISI Type 304 SS, Hastelloy X, and Inconel 625 are being determined at temperatures from 400 to 750 °C and fluences up to 1×10^{23} . The influence of grain size, heat treatment, and alloy content on the postirradiation mechanical properties of the vanadium alloys are being investigated. The effects are compared to those observed in Type 304 SS, Hastelloy X, and Inconel 625 irradiated and tested under identical conditions.

Previous reports gave the results of tensile tests between 400 and 650 °C on specimens of irradiated V-20 Ti, V-15 Ti-7.5 Cr, Hastelloy X, and Type 304 SS and tube burst tests between 550 and 750 °C on specimens of irradiated V-15 Ti-7.5 Cr alloy. The specimens had been irradiated in EBR-II Subassembly X013 to a nominal fluence of 5×10^{21} at 600 ± 100 °C. These tests showed:

1. The yield strengths of all of the alloys were increased by approximately 50% as a result of irradiation,
2. The ductility losses of the Type 304 SS and Hastelloy X appeared to be greater than that observed for the vanadium alloys,

3. A variation in preirradiation grain size of the vanadium alloys between 10 and 35 μ did not appear to significantly influence the postirradiation tensile properties,
4. The tangential rupture strength of V-15 Ti-7.5 Cr alloy was increased by 27% at a test temperature of 550 °C, but by only 8% at a test temperature of 750 °C,
5. The circumferential ductility of the irradiated alloy was about half that of the unirradiated alloy at all test temperatures.

TUBE BURST TESTS

Tube burst tests were conducted at 550 °C on unirradiated specimens of Type 304 SS, Hastelloy X, and Inconel 625 and irradiated specimens of the same alloys from EBR-II Subassembly X013. Additional tube burst tests were conducted at 550 °C on irradiated specimens of V-15 Ti-7.5 Cr alloy from the same subassembly in an attempt to reduce the scatter observed in the strength and ductility values previously reported. The results of these tests are given in Table 3.1 and are plotted as a function of fluence in Figures 3.1 and 3.2.

The tangential rupture strengths of all of the alloys were increased as a result of irradiation (Table 3.1). The

TABLE 3.1. Tube Burst Properties at 550 °C
of Cladding Materials Specimens from EBR-II
Subassembly X013

Material	Fluence, $n/cm^2 \times 10^{21}$	Tangential Rupture Strength, kg/mm^2	% $\Delta D/D$
V-15 Ti-7.5 Cr	-	71.1	6.1
V-15 Ti-7.5 Cr	-	70.5	6.2
V-15 Ti-7.5 Cr	-	73.0	6.1
V-15 Ti-7.5 Cr	-	72.4	8.1
V-15 Ti-7.5 Cr	0.2	77.2	7.2
V-15 Ti-7.5 Cr	0.5	77.6	8.9
V-15 Ti-7.5 Cr	1.1	83.4	5.8
V-15 Ti-7.5 Cr	2.0	83.4	2.0
V-15 Ti-7.5 Cr	3.6	90.5	3.8
V-15 Ti-7.5 Cr	4.7	94.7	4.9
304 SS	-	41.2	10.9
304 SS	-	41.9	10.7
304 SS	-	40.6	12.1
304 SS	-	39.4	12.1
304 SS	-	38.7	11.0
304 SS	-	38.7	11.3
304 SS	-	39.4	12.5
304 SS	-	39.4	12.4
304 SS	0.2	38.8	14.7
304 SS	1.2	40.1	11.2
304 SS	3.6	41.7	6.3
304 SS	3.7	44.8	10.0
304 SS	3.9	45.6	3.6
304 SS	4.0	44.3	3.2
304 SS	5.3	46.2	3.0
Hastelloy X	-	56.2	13.2
Hastelloy X	-	56.2	14.0
Hastelloy X	-	56.6	14.2
Hastelloy X	-	58.1	13.5
Hastelloy X	3.5	68.8	8.1
Hastelloy X	3.7	67.6	3.7
Inconel 625	-	63.7	9.5
Inconel 625	-	65.3	12.7
Inconel 625	-	64.1	14.5
Inconel 625	-	64.1	13.0
Inconel 625	0.2	88.6	9.2
Inconel 625	1.1	90.5	15.7
Inconel 625	3.3	88.6	7.0
Inconel 625	3.3	87.9	3.3
Inconel 625	3.5	88.2	4.4
Inconel 625	3.6	89.2	10.5

FIGURE 3.1. Effect of Irradiation on the Tangential Rupture Strength of Fast Reactor Cladding Materials at 550 °C

FIGURE 3.2. Effect of Irradiation on the Circumferential Ductility of Fast Reactor Cladding Materials at 550 °C

relative increase in rupture strength was greatest for Inconel 625 (41%) and least for Type 304 SS (16%). The relative increase in rupture strengths of V-15 Ti-7.5 Cr and Hastelloy X were approximately the same and fell halfway between those observed for Type 304 SS and Inconel 625 (Figure 3.1). The rupture strengths of both V-15 Ti-7.5 Cr and Type 304 SS increased with increasing fluence; whereas, the rupture strength of Inconel 625 was increased to a maximum at low fluences ($\sim 1 \times 10^{21}$) and remained approximately the same at a fluence of 3.6×10^{21} . The effect of fluence on the rupture strength of Hastelloy X was not determined.

The circumferential ductilities of V-15 Ti-7.5 Cr, Inconel 625, and Type 304 SS appeared to have been increased as a result of irradiation to fluences of less than 1×10^{21} (Figure 3.2). At higher fluences, the circumferential ductilities of the alloys, including Hastelloy X, were decreased. The relative effect of irradiation on the ductility of V-15 Ti-7.5 Cr compared to Type 304 SS, Hastelloy X, and Inconel 625 could not be definitively established because of scatter in the circumferential ductility values (Figure 3.2). The ductility of V-15 Ti-7.5 Cr alloy, however, appeared to have been less affected by irradiation than that of the other three alloys since, although the unirradiated circumferential ductility of V-15 Ti-7.5 Cr was less (6 to 8%) than that of Type 304 SS, Hastelloy X, and Inconel 625 (10 to 14%), the minimum ductility observed for irradiated V-15 Ti-7.5 Cr (2%)

was in the same range (3 to 4%) as that observed for the other three alloys (Table 3.1).

TENSILE TESTS

Tensile tests were conducted at 750 °C and at a constant crosshead speed of 0.05 cm/min on specimens of V-20 Ti and V-15 Ti-7.5 Cr irradiated in EBR-II Subassembly X013 to a nominal fluence of 5×10^{21} . Tensile tests also were conducted at 550 and 650 °C on specimens of V-20 Ti irradiated in EBR-II Subassembly XA07 to a nominal fluence of 3×10^{22} . Both alloys had a preirradiation grain size of 10 μ . The results are given in Table 3.2. The effects of irradiation and test temperature on the yield strength and the uniform elongation of V-20 Ti and V-15 Ti-7.5 Cr are illustrated in Figures 3.3 and 3.4. The unirradiated test values are those of F. L. Yaggee.⁽¹⁾ Previous results as well as present test data are plotted in the figures.

V-20 Ti

The yield strength of irradiated V-20 Ti decreased as the test temperature was increased from 400 to 750 °C (Figure 3.3). Significant recovery of the irradiation increase in yield strength did not occur until the temperature exceeded 650 °C; at 750 °C the irradiated and unirradiated yield strengths were approximately the same. An increase in fluence from 5×10^{21} to 3×10^{22} did not appear to result in higher irradiated yield strengths; in

TABLE 3.2. Postirradiation Tensile Properties of Vanadium Alloys Irradiated in EBR-II

Alloy, wt%	Fluence, $n/cm^2 \times 10^{21}$	Test Temperature, °C	0.2% Yield Strength, kg/mm^2	Tensile Strength, kg/mm^2	Uniform Elongation, %	Total Elongation, %
V-20 Ti	33.0	550	46.0	59.6	16.5	24.1
V-20 Ti	33.0	650	39.9	48.0	13.0	27.8
V-20 Ti	5.2	750	31.2	36.0	12.0	34.1
V-20 Ti	5.2	750	31.6	36.1	9.0	39.4
V-15 Ti-7.5 Cr	5.2	750	33.8	38.7	7.5	22.6

FIGURE 3.3. Effect of Irradiation on the Yield Strength and Uniform Elongation of V-20 Ti Alloy as a Function of Test Temperature

FIGURE 3.4. Effect of Irradiation on the Yield Strength and Uniform Elongation of V-15 Ti-7.5 Cr Alloy as a Function of Test Temperature

fact, the yield strengths at 550 and 650 °C of specimens irradiated to 3×10^{22} were lower than those of specimens irradiated to 5×10^{21} and tested at the same test temperatures.

Although the uniform elongation of irradiated V-20 Ti was lower than that of the irradiated alloy at some test temperatures, it was higher than that of the unirradiated alloy at other test

temperatures (Figure 3.3). Since the irradiation temperature was determined only within 100 °C (600 ± 100 °C), the observed scatter in the ductility values was probably the result of the specimens being irradiated at either the high or the low end of the temperature range. In any case, in those instances where a decrease in uniform elongation was observed, the decrease

was small, and the ductility of V-20 Ti appeared to be unaffected by irradiation up to 3×10^{22} .

V-15 Ti-7.5 Cr

The yield strength of V-15 Ti-7.5 Cr irradiated to 5×10^{21} did not decrease with test temperature until the test temperature exceeded 650 °C; at a test temperature of 750 °C, the irradiated and unirradiated yield strengths were approximately the same (Figure 3.4).

The uniform elongation of irradiated V-15 Ti-7.5 Cr was lower than that of the unirradiated alloy at test temperatures of 550, 650, and 750 °C; at a test temperature of 400 °C, the uniform elongation of V-15 Ti-7.5 Cr was unaffected by irradiation (Figure 3.4). The difference between the irradiated and unirradiated percent uniform elongations at the higher test temperatures, however, was small ($\sim 2\%$), and, as was observed for V-20 Ti, the uniform elongation of V-15 Ti-7.5 Cr was not significantly affected by irradiation to 5×10^{21} .

CREEP TESTS

Specimens of irradiated V-20 Ti with a preirradiation grain size of 10μ were creep-tested at 650 °C in vacuum at stresses between 17.5 and 35 kg/mm². The specimens were irradiated in EBR-II Subassemblies XA07 and XA09 at 585 °C and to nominal fluences of 3×10^{22} and 2×10^{22} , respectively. The results are given in Table 3.3 and plotted in Figures 3.5 and 3.6. The unirradiated values in Figure 3.5 are those of F. L. Yaggee.¹

The minimum creep rate, the creep-rupture life, and the rupture ductility of V-20 Ti were unaffected by irradiation up to a fluence of

TABLE 3.3. Creep Properties of Irradiated V-20 Ti Alloy at 650 °C

Fluence, n/cm ² x 10 ²²	Stress, kg/mm ²	Minimum Creep Rate sec ⁻¹	Rupture Life, hr	Total Elongation, %
1.2	17.5	5.1×10^{-8}	Running	
1.0	35	2.4×10^{-6}	Running	
2.0	24.5	9.0×10^{-8}	Running	
2.1	24.5	1.7×10^{-7}	292	14.2
2.1	35	1.3×10^{-6}	172	13.3

FIGURE 3.5. Minimum Creep Rate of V-20 Ti Alloy as a Function of Applied Stress at 650 °C

FIGURE 3.6. Rupture Life Versus Applied Stress of V-20 Ti Alloy at 650 °C

2×10^{22} (Figures 3.5 and 3.6). Although the total elongation at fracture was reduced from 26.2 to 13.3% at a stress of 35 kg/mm^2 , the total elongation of the irradiated alloy was 14.2% compared to 10.4% for the unirradiated alloy at a stress of 24.5 kg/mm^2 (Table 3.3, Figure 3.6). The variation in the ductility values given above is believed to be caused by scatter from the limited number of specimens which were tested.

The indicated absence of a change in the creep properties of V-20 Ti due

to irradiation is in agreement with the absence of an effect of irradiation on the tensile ductility; however, it is not in agreement with the postirradiation increase in yield strength, which should have resulted in an increase in the creep-rupture strength. The latter indicates that the irradiation-induced strengthening was probably annealed out because of the increased duration of the creep tests compared with the tensile tests.

VOIDS IN EBR-II CLADDING AND CONTROL ROD SHROUD

S. D. Harkness

EBR-II MARK IA CONTROL ROD SHROUD

EBR-II Mark IA fuel cladding has been examined by transmission electron microscopy. The cladding sections were taken from a fuel element which exhibited high swelling (fuel $\Delta V/V > 13\%$). There was evidence that the fuel had been in contact with the cladding in this element, thereby significantly raising the clad stress level.

Foils from four different sections were examined in this study. A large number of Frank dislocation loops and polyhedral voids were found in each sample. The voids included a wide size distribution. Very few voids lay on grain boundaries, while the occurrence of depleted regions along the boundaries was common. The width of the depleted region averaged about 350 Å. Some grain boundaries contained precipitate particles that were probably carbides. Optical micrographs of this sample showed sensitization of the material. The fact that no bubbles or voids were seen at grain boundaries indicates that something other than helium bubble formation is responsible for embrittlement under fast reactor conditions.

The average void size ($\langle D \rangle$), number density (N_V) and volume fraction (V_F) calculated from a series of micrographs are listed in Table 3.4.

TABLE 3.4. Void Characteristics in EBR-II Mark IA Fuel Cladding

Sample No.	Maximum Cladding Temperature, °C	Fluence, $n/cm^2 \times 10^{-22}$	$\langle D \rangle$, Å	$N_V/cm^3 \times 10^{15}$	V_F , %
1	480	1.2	93	5.4	0.2
2	465	1.3	105	4.0	0.2
3	492	1.1	140	1.5	0.2
4	462	1.4	91	2.8	0.1

CONTROL ROD SHROUD

Sections of a Type 304 SS control rod shroud which had been irradiated in EBR-II to a peak fluence of $5 \times 10^{22} n/cm^2$ are presently being examined. This material is of great interest for two reasons. First, its fluence is higher than any 304 SS previously studied; second, it was unstressed during exposure. These points should aid in delineating the roles of fluence and stress in the void growth process.

Density measurements for three different positions have been made and are presented in Table 3.5. The experimentally determined standard deviation for these results was 0.002 g/cm^3 . The large amount of void formation indicated, implies that the presence of stress is unimportant to the phenomenon.

*TABLE 3.5. Density Results
for Control Rod Shroud*

Sample Position (in. above bottom of core)	Irradiation Temperature, °C	Fluence, n/cm ² x 10 ²²	V _F % Increase in Volume
23.5	470	1.0	0.06
17.5	470	2.0	0.28
5.5	420	4.8	1.04

Examination of the material by transmission electron microscopy (TEM) is in process. At present, one sec-

tion which had been exposed to a fluence of 4.5×10^{22} n/cm² and an irradiation temperature of 410 °C has been studied.

The general microstructure was the same as observed in the cladding. The average void diameter was 100 Å, the number density 4.5×10^{15} n/cm³ and the calculated volume fraction 0.25%.

REFERENCES

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THE EFFECT OF RADIATION ON THE MECHANICAL PROPERTIES OF CLADDING MATERIALS

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OBJECTIVE OF PROGRAM

The objective of the program is to determine the extent and nature of the radiation-induced changes in the mechanical properties of AISI Type 347 SS and other potential cladding materials for liquid-metal-cooled fast-reactor applications.

APPROACH BEING USED

Type 347 SS specimens will be irradiated in the ETR to fast fluences ranging from 3×10^{21} to 30×10^{21} n/cm² ($E > 1$ MeV). Irradiated tensile specimens will be mechanically tested at room temperature and at 315 and 750 °C. Special emphasis will be directed toward investigating the ductility loss, at elevated temperatures, found in irradiated stainless steels and nickel alloys. This ductility loss is characterized by intergranular failure. Investigation of this intergranular fracture at elevated temperatures will be carried out by use of metallography, X-ray diffraction, chemical analysis, transmission electron microscopy, fractography, and electron-beam microprobe analysis. Stress-rupture tests will be performed at 650 °C at different stress levels. Also, cyclic-strain fatigue tests will be performed on irradiated Type 347 SS specimens at room temperature and at temperatures (~650 °C)

where the irradiation-induced embrittlement has been found. In addition, the effect of fast-neutron irradiation on the impact strength of Type 347 SS will be determined by testing Charpy-V notch and Izod specimens after fast fluences of 5.75×10^{21} and 30×10^{21} n/cm² ($E > 1$ MeV).

The ductility loss observed at elevated temperatures in irradiated materials has been found only for nickel-containing austenitic stainless steels and nickel-base alloys. Consequently, it appears that nickel bears some direct relationship to this irradiation-induced elevated-temperature embrittlement. It may be that the nickel-containing alloys are extremely susceptible to helium-induced intergranular failure or that the irradiation may produce other transmutations which, in combination with nickel, may cause elevated-temperature embrittlement. To further investigate this elevated-temperature embrittlement, it is planned to irradiate selected materials in the ETR and test them at elevated temperatures. The materials to be irradiated are pure nickel (99.999%), Nickel 200 (which contains about 1 wt% of other elements), a nickel-free manganese austenitic stainless steel (Tenelon), and two cobalt alloys. One of the cobalt alloys will contain 10 wt% nickel (Haynes Stellite 25), while the other cobalt alloy will be nickel-free (UMCo-50).

Testing of these materials at elevated temperatures, after irradiation, is expected to show the effect of nickel content on elevated-temperature embrittlement.

The elevated-temperature embrittlement in irradiated stainless steel may be caused by a diffusion-controlled process. To determine whether or not diffusion is an important factor, specimens irradiated at about 50 °C will be heated both slowly and rapidly to test temperature and then tested in tension. If the rapidly heated specimens exhibit substantially less embrittlement than the slowly heated specimens, this will indicate that a diffusion-controlled process is associated with the embrittlement behavior.

Since the elevated-temperature embrittlement in irradiated stainless steel is intergranular in nature, a better understanding of the phenomenon may be obtained by investigating the effect of grain boundaries at elevated temperature after irradiation. Single crystals of Type 347 SS (no grain boundaries) and polycrystalline specimens with elongated grains oriented parallel and perpendicular to the tensile axis will be irradiated and tested at elevated temperatures.

PROGRESS REPORTED PREVIOUSLY

The tensile properties of Type 347 SS irradiated in the ETR to fast fluences of $5.5 \times 10^{21} \text{ n/cm}^2$ ($E > 1 \text{ MeV}$) were reported in HW-74679.⁽¹⁾ In HW-83398⁽²⁾ and BNWL-CC-510⁽³⁾ the effects of radiation on the tensile properties of Type 347 SS after fast fluences of $16 \times 10^{21} \text{ n/cm}^2$ and $21 \times 10^{21} \text{ n/cm}^2$, respectively, were

reported. Tensile tests were performed on the irradiated material at room temperature and at 315 and 750 °C after the irradiated material had been annealed for 1 hr at 980 °C.

Results of the tensile tests at room temperature and 315 °C indicated that both yield strength and ultimate strength are increased by irradiation, with the yield-strength value approaching that of the ultimate strength. About a 50% reduction in total elongation at both room temperature and 315 °C occurred after a fast fluence of $5.5 \times 10^{21} \text{ n/cm}^2$. No further decrease in elongation took place with increasing fast fluence to $21 \times 10^{21} \text{ n/cm}^2$. Annealing the irradiated specimens for 1 hr at 980 °C restored the preirradiation strength properties at room temperature and 315 °C; approximately 90% of the unirradiated ductility was restored.

Tensile tests at 750 °C of specimens which had received a fast fluence of $21 \times 10^{21} \text{ n/cm}^2$ showed no change in ultimate strength as compared to unirradiated values; however, the yield strength increased to the level of the ultimate strength and the total elongation was drastically reduced from about 26% to only 0.5%. Annealing of two irradiated specimens for 1 hr at 980 and 1350 °C, respectively, was found to decrease the yield and ultimate strengths during testing at 750 °C. Elongation values remained about 0.5%. A specimen which had received a fast fluence of $0.5 \times 10^{21} \text{ n/cm}^2$ had a total elongation of 15% when tested at 750 °C. This suggests that the ductility loss at 750 °C increases with increasing fast fluence. It was further found that the drastic irradiation-induced

($21 \times 10^{21} \text{ n/cm}^2$) reduction in ductility occurred in the very narrow temperature range of 525 to 575 °C, where the total elongation was reduced from 21.2 to 1.9%.⁽⁴⁾ Annealing of a highly irradiated ($21 \times 10^{21} \text{ n/cm}^2$) stainless steel specimen at 980 °C for 1 hr resulted in almost complete restoration of preirradiation ductility when tested at 500 °C. However, testing of similarly annealed specimens at 600 °C indicated that only minimal improvement in ductility occurred (i.e., total elongation was increased from 0.7 to 3.6%, while the unirradiated value was 22.5%). It was also found that the embrittlement at 650 °C was not significantly diffusion dependent since the postirradiation ductility was not significantly affected by reducing the soak times before testing from 1 hr to 4 min. Replicas for electron fractography were taken from the fracture surfaces of both unirradiated and irradiated ($21 \times 10^{21} \text{ n/cm}^2$) specimens which had been tested at 750 °C. The unirradiated specimen exhibited a ductile transgranular fracture, while the irradiated specimen exhibited an intergranular fracture.

The cyclic-strain fatigue tests at room temperature after fast fluences of 5.5×10^{21} and $11 \times 10^{21} \text{ n/cm}^2$ indicated that for high strains the number of cycles to failure was decreased by irradiation, while for lower strains the number of cycles to failure was increased by irradiation.⁽⁵⁾ Testing of irradiated Charpy-V notch specimens indicated that irradiation to a fast fluence of $5.7 \times 10^{21} \text{ n/cm}^2$ reduced the impact strength of Type 347 SS at -196 °C and room tempera-

ture, while it had no effect on the impact strength at 315 °C.⁽²⁾

An emission spectrographic analysis which was performed on the irradiated stainless steel ($21 \times 10^{21} \text{ n/cm}^2$) indicated that some compositional changes had taken place as a result of irradiation. Of the major constituents, nickel, niobium, and silicon were decreased while the copper, zinc, and vanadium content was increased. Chemical analysis also indicated that 30 to 40% of the manganese was burned out by the long-term irradiation, while no significant changes in the sulfur content took place. Computer calculations based on thermal neutrons and known capture cross-section data predicted an increase in zinc content which was considerably smaller than the change indicated by the emission spectrographic analysis. This discrepancy could be due to the segregation of zinc or significant compositional changes produced by the epithermal and fast neutrons, which were not considered in the calculations.^(6,7)

WORK THIS PERIOD

Tensile tests were performed on irradiated specimens of Tenelon, UMoCo-50, and Haynes Stellite 25. These tests were undertaken to determine the role of nickel on radiation-induced embrittlement at elevated temperatures. Tenelon is a nickel-free austenitic stainless steel. One of the cobalt alloys does not contain nickel (UMoCo-50), while the other cobalt alloy contains 10 wt% nickel (Haynes Stellite 25). These

specimens were irradiated in the ETR at a temperature of 50 °C with the Tenelon specimens receiving a fast fluence of $1.5 \times 10^{21} \text{ n/cm}^2$ and the UMCo-50 and Haynes Stellite 25 specimens receiving a fast fluence of $1.4 \times 10^{21} \text{ n/cm}^2$. The UMCo-50 and Haynes Stellite 25 specimens were annealed for 1 hr at 800 °C before testing, and the Tenelon specimens were annealed for 1 hr at 700 °C before testing. These anneals were undertaken to remove the displacement type of radiation damage so that the radiation-induced embrittlement at elevated temperatures could be better studied. Preliminary results of tensile tests performed at elevated temperatures on irradiated and unirradiated specimens given an identical anneal are shown in Table 4.1. These tensile tests indicate that irradiation causes severe embrittlement in Tenelon, UMCo-50 and Haynes Stellite 25 at testing temperatures of 550 °C and above. The loss of uniform elongation is also accompanied by significant losses in ultimate tensile strength. At 450 °C the

tensile properties of irradiated and annealed UMCo-50 are comparable to the tensile properties of unirradiated material. However, irradiated and annealed specimens of Tenelon and Haynes Stellite 25 exhibit losses in both strength and ductility at 450 °C.

Last quarter the microstructures of irradiated ($2.1 \times 10^{22} \text{ n/cm}^2$) Type 347 SS specimens, which were annealed for 1 hr at 980 °C and tested at 500 and 600 °C were investigated.⁽⁷⁾ The investigation revealed that at 500 °C (24.2% total elongation) the fracture was transgranular, while at 600 °C (3.6% total elongation) the fracture was intergranular. Replicas taken from the fracture surfaces of these specimens were observed in an electron microscope. Figure 4.1 illustrates the fracture surfaces of these specimens. It appears that the fracture surfaces of both specimens indicate ductile fracture, although in one case the total elongation was only 3.6% and metallographic examination indicated fracture by grain boundary separation.

TABLE 4.1. Tensile Properties of Irradiated and Unirradiated Tenelon, UMCo-50, and Haynes Stellite 25

Material	Test Temperature, °C	0.2% Offset Yield Strength, 1000 psi		Ultimate Strength, 1000 psi		Uniform Elongation, %		Total Elongation, %	
		Unirradiated	Irradiated	Unirradiated	Irradiated	Unirradiated	Irradiated	Unirradiated	Irradiated
Tenelon	450	35.3	26.5	85.5	55.3	37.0	11.0	49.0	11.2
	550	30.3	26.0	77.5	48.5	32.5	6.4	33.0	6.5
	650	27.0	25.3	51.0	31.9	10.5	1.7	12.0	1.8
Haynes Stellite 25	450	36.1	29.9	96.3	70.3	40.0	22.2	42.0	22.8
	550	33.9	28.5	82.3	53.5	28.0	8.6	30.0	8.8
	650	32.3	29.8	69.3	40.5	14.0	4.5	17.0	4.5
UMCo-50	450	28.9	26.0	82.5	75.1	23.0	29.7	24.5	30.7
	550	25.0	24.6	72.0	49.1	25.9	11.5	27.0	13.9

Neg J16565

7000X

a. Tested at 500 °C
24.2% Total Elongation

Neg 16541

7000X

b. Tested at 600 °C
3.6% Total Elongation

FIGURE 4.1. Fracture Surface of Type 347 SS Tensile Specimens Irradiated to a Fast Fluence of $2.1 \times 10^{22} \text{ n/cm}^2$ and Annealed for 1 hr at 980 °C

Stress-rupture tests at 650 °C were performed on Type 347 SS specimens which were irradiated to a fast fluence of $2.2 \times 10^{22} \text{ n/cm}^2$ at 50 °C in the ETR. Results of the stress-rupture tests are shown in Figure 4.2. It can be seen that irradiation causes a significant reduction in rupture time. A stress-rupture test at 650 °C has been in progress for 330 hr at a stress of 18,500 psi.

Some specimens to be used in the study of the effect of nickel are still being irradiated in the ETR. The materials and their accumulated fast fluence are given in Table 4.2.

FIGURE 4.2. Stress-Rupture Properties of Irradiated and Unirradiated Type 347 SS at 650 °C

TABLE 4.2. Accumulation of Fast Fluence for Specimens Being Irradiated in the ETR at 50 °C

Capsule	Position	Material	Fast Fluence after ETR Cycle 92 (10/11/67), 10 ²² n/cm ² (E > 1 MeV)
BMI-24-2	Top	Type 347 SS	2.67
	Bottom	Type 347 SS	2.84
BMI-24-10	Top	Type 347 SS	2.50
	Bottom	Type 347 SS	2.36
BMI-24-16	Top	Type 347 SS	2.39
	Bottom	Type 347 SS	2.52
BMI-24-22	Top	Type 347 SS	2.36
	Bottom	Type 347 SS	2.63
BMI-24-25	Top	Haynes Stellite 25	0.210
	Bottom	UMCo-50	0.210
BMI-24-26	Top	Tenelon	0.125
	Bottom	Nickel	0.138

PLANS FOR FUTURE WORK

The testing of irradiated UMo-50, Tenelon, and Haynes Stellite 25 specimens at elevated temperatures will be continued. The testing of irradiated Nickel 200 and pure-nickel specimens will be initiated. The stress-rupture testing of irradiated Type 347 SS specimens at 650 °C will be continued.

PREPARATION OF STAINLESS STEEL SINGLE CRYSTALS

S. J. Veeck and W. H. Pfeifer

OBJECTIVE OF PROGRAM

The objective of the program is to develop a suitable technique for preparing stainless steel single crystals, of random orientation, that have a composition within the normal compositional ranges of commercial Type 347 SS.

APPROACH BEING USED

Three single-crystal growth techniques will be examined as potential

methods of producing Type 347 SS single crystals. These techniques include electron-beam zone melting, controlled solidification (Bridgman), and strain annealing. Each technique will be briefly evaluated, and the best technique will be chosen for further study to determine optimum parameters for crystal growth. Once optimum parameters are established, single crystals 2 in. in length and 1/4 in. will be grown for subsequent irradiation studies. The structure and composition of samples will be determined using metallographic, X-ray, and analytical chemistry techniques.

PROGRESS REPORTED PREVIOUSLY

Work was initiated this quarter.

WORK THIS PERIOD

Several attempts to produce Type 347 SS single crystals were made using zone-melting techniques. The equipment used was a MRC high-vacuum chamber in conjunction with an electron-beam zone refiner. The working chamber was vibrationally isolated from the pumping equipment by a metal bellows. The electron source was in a wire-ring configuration which surrounded a 1/4 in. diam Type 347 SS rod. The sample was heated by the electron beam to just above its melting temperature (~1425 °C) and slowly scanned under a vacuum of ~10⁻⁷ torr. Three different scan rates were used on three separate samples: 8.6, 5.1, and 3.1 in./hr.

Each of the three samples was examined metallographically and each was found to contain the ferrite phase, as shown in Figure 4.3. Each sample also was found to be polycrystalline. In addition to the above problems, a significant chromium loss by evaporation was apparent from each of the specimens in the form of a chromium coating on the interior of the vacuum chamber.

Because the preliminary results with zone melting did not show much promise, further work was discontinued and studies were initiated to examine controlled solidification by the Bridgman technique. The technique involves solidifying the liquid metal from one end (usually a point source) and slowly moving the solid/liquid interface over the length of the molten region until solidification is complete. The experiment is conducted in an inert atmosphere to minimize losses of volatile constituents and oxidation.

FIGURE 4.3. Microstructure of Zone-Melted Type 347 SS (Note the Ferrite Islands in the Austenite Phase.)

The system used was a platinum wound furnace with a high-purity Al_2O_3 retort which extended ~ 13 in. from both ends of the furnace. The furnace was set in a vertical position and an inert gas-feed line was attached to the retort at the base. The retort was water cooled at the top of the furnace, and a rubber stopper pierced by a 1/8 in. ID copper tube was placed in either end of the retort. Thus, the copper tube allowed both venting of the inert gas (argon) and permitted the sample to be dropped through the furnace hot zone by means of a tungsten wire. Samples were placed in closed-end high-purity Al_2O_3 tubes which were commercially available and which had inner diameters in the range of 0.1 to 0.2 in. The Al_2O_3 tubes were supported by means of a tungsten support wire which was attached to a vertically geared support arm operated by a motor. Thus, the rate at which the specimens were dropped through the furnace hot zone could be controlled by varying the motor speed. The drop speed range over which solidification studies were performed was ~ 0.5 to 1.0 in./hr. A schematic of the equipment is shown in Figure 4.4.

Two specimens were dropped through the furnace hot zone which was held at 1510 °C; one at 0.9 in./hr and the other at 0.6 in./hr. Subsequent metallographic examination of both specimens showed them to be polycrystalline, and both also contained the ferrite phase in the latter portion of the sample that was the last to be solidified. The sample dropped at

FIGURE 4.4. Schematic Representation of Test System Used in Growing Crystals by the Bridgeman Technique

0.6 in./hr did result in a single crystal having a length of $\sim 1/4$ in., as shown in Figure 4.5. The sample shown is 3 mm (0.112 in.) in diameter.

Since literature sources indicate that Type 347 SS is a metastable austenitic alloy, it is expected that slow cooling from elevated zone temperatures will result in the forma-

tion of the alpha phase. It is also possible that formation of the alpha phase may become even more pronounced when segregation occurs, and both zone melting and the Bridgman solidification techniques are prone to result in segregation when solidification occurs too rapidly to allow equilibrium conditions. Segregation

Neg 7C813

Etched

6X

- a) The structure at the left is an austenite-plus-alpha (ferrite) phase which has been overetched.
- b) Note the large-grained structure at the right where one of the grains is $2\frac{1}{4}$ in. in length.

FIGURE 4.5. Macrophotograph of a Sample Crystal Grown by the Bridgeman Technique

can be minimized by using very slow scan or solidification rates (within practical limits) and it is apparent that this procedure is needed in both the techniques already mentioned. These changes in scan rates would, however, require equipment modifications. Assuming that segregation could be minimized, it would then be necessary to anneal finished samples to dissolve the ferrite formed during slow cooling.

In comparing the two approaches examined thus far, it seems that the Bridgeman solidification technique demonstrates the best feasibility. It remains to be seen whether strain-anneal techniques will produce superior results. The molten-zone approach has the major drawback of evaporative chromium losses, which would be difficult to minimize, i.e., higher pressures will result in arc discharging of the electron beam and/or greater contamination of the sample. It should also be noted, however, that further work with controlled solidification should include special Al_2O_3 crucibles with a smooth wall (to prevent

random nucleation sites) and a pointed bottom. Also, slower drop rates are recommended for reasons already discussed, i.e., to minimize segregation.

Preliminary studies have been initiated to examine strain anneal techniques for producing single crystals. To date, approximately eight samples of Type 347 SS have been rolled to 0.050 in. in thickness (1/4 in. width) and recrystallized to an ASTM 6 grain size.

PLANS FOR FUTURE WORK

Those samples prepared for strain annealing will be strained over a range of 2.5 to 3.5% elongation. Annealing will be performed at 1350 °C for at least 100 hr. The parameters used in the strain-anneal techniques correspond well with literature sources describing growth of Fe-18 Cr-8 Ni alloy single crystals. Subsequently, the specimens will be metallographically examined to determine the potential of strain-anneal techniques.

CORRELATION OF RADIATION EFFECTS
ON MECHANICAL PROPERTIES AS CAUSED
BY A PREDOMINANTLY FAST FLUX
AND A MIXED FAST AND THERMAL FLUX

M. Kangilaski, J. S. Perrin,
and R. A. Wullaert

OBJECTIVE OF PROGRAM

The objective of the program is to compare the effects of a predominantly fast-neutron flux and a mixed fast- and thermal-neutron flux on the mechanical properties of cladding materials.

APPROACH BEING USED

Tensile and fatigue specimens of the same lot of Type 347 SS irradiated in the ETR to fast fluence of up to $3 \times 10^{22} \text{ n/cm}^2$ ($E > 1 \text{ MeV}$) will be irradiated in the EBR-II to equivalent fast fluences. Since only a limited number of the original Type 347 SS specimens were available, additional specimens of Type 348 SS will be included in the EBR-II irradiation experiment. The Type 348 SS rod, from which specimens were machined, was obtained from the stockpile of structural materials kept at Battelle-Northwest for use in experiments to study radiation effects on structural materials. The Type 347 SS can be considered as a Type 348 SS since it meets the chemical specifications for Type 348 SS (less than 0.10 wt% tantalum).

Tubular specimens of Type 347 SS and tensile specimens of Tenelon, UMo-50, and Haynes Stellite 25 will also be irradiated in the EBR-II. The tubular specimens of Type 347 SS

are being incorporated into the program to better approximate the actual behavior of cladding materials in nuclear applications. A few tensile specimens of Tenelon, UMo-50, and Haynes Stellite 25 are included to determine the effects of a predominantly fast-neutron spectrum on their mechanical properties and to relate these effects to previous results obtained in a mixed flux.

The irradiated specimens of Type 347 SS will be tested at room temperature and at 315 and 750 °C to correlate the effects of predominantly fast neutrons, and mixed thermal and fast neutrons, on the mechanical properties. Tensile tests on specimens of Type 348 SS, Tenelon, UMo-50, and Haynes Stellite 25 will be carried out at elevated temperatures to determine the susceptibility of these materials to radiation-induced embrittlement at elevated temperatures.

Since the EBR-II irradiation will take place at elevated temperatures (400 and 650 °C) and the original ETR irradiation was conducted at 50 °C, it is possible that the different irradiation temperatures will not allow a direct comparison of the postirradiation mechanical properties of Type 347 SS. Therefore, a capsule will be irradiated in the ETR with specimen temperatures of 400 and 650 °C so that a direct correlation between the effects of radiation on the mechanical properties of Type 347 SS can be made. These specimens will be tested at room temperature, and at 315 and 750 °C after irradiation. In addition, some tensile specimens

of Type 348 SS and tubular specimens of Type 347 SS will also be included in the radiation capsule.

PROGRESS REPORTED PREVIOUSLY

All test specimens for the irradiation capsules have been fabricated. The type and number of specimens and the irradiation conditions of these specimens in EBR-II and ETR were given in BNWL-678.⁽⁶⁾

WORK THIS PERIOD

The capsules for the EBR-II and ETR have been assembled and are waiting at their respective reactor sites for insertion.

PLANS FOR FUTURE WORK

The elevated-temperature ETR capsule will be inserted at the next reactor shutdown and irradiated to obtain an estimated fast fluence of $5 \times 10^{21} \text{ n/cm}^2$. The EBR-II capsule will be inserted at the next reactor shutdown and will be irradiated to a maximum fast fluence of $3 \times 10^{22} \text{ n/cm}^2$.

RADIATION EFFECTS ON THE MECHANICAL PROPERTIES OF TYPE 347 SS FERMI WRAPPER TUBE

J. A. DeMastry, J. W. Sheckherd,
and R. A. Wullaert

OBJECTIVE OF PROGRAM

The objective of the program is to determine the radiation-induced changes in the mechanical properties of Type 347 SS irradiated as a wrap-

per tube in the predominantly fast flux of the Fermi Reactor, and to compare these changes with those for Type 347 SS irradiated in a mixed fast and thermal flux.

APPROACH BEING USED

Sheet tensile specimens will be prepared from an irradiated Type 347 SS Fermi wrapper tube which has received a predominantly fast fluence of $2 \times 10^{20} \text{ n/cm}^2$ ($E > 1 \text{ MeV}$). The wrapper tube is 2.5 in. square with a weld down one side, and has a thickness of 100 mils. Specimens will be cut from both the longitudinal and transverse directions. Some of the transverse specimens will contain the weld in their gage section. The specimens will be tested in tension at room temperature and at 315, 650, and 750 °C in both the as-irradiated condition and in the irradiated-plus-annealed condition. Results from these tests will be compared with data obtained on Type 347 SS irradiated in a mixed thermal and fast flux.

PROGRESS REPORTED PREVIOUSLY

A nonstandard specimen design is being used because of the geometry of the wrapper tubes. A template for machining nonstandard tensile specimens has been prepared.

Control specimens were machined from an unirradiated Type 347 SS Fermi wrapper tube. Both longitudinal and transverse specimens and specimens containing a longitudinal weld were prepared.

Some difficulty in locating the weld in the irradiated material has been encountered since the weld is only visible after chemical etching. Transverse sections were cut from all four sides of the wrapper tube to locate the weld by chemical etching.

WORK THIS PERIOD

The weld was located in the irradiated wrapper tube, and specimen blanks were prepared. Both longitudinal and transverse specimen blanks were cut from the irradiated wrapper tube. Tensile specimens have been prepared from these blanks.

Testing of tensile specimens prepared from an unirradiated wrapper tube was initiated. Preliminary results for both longitudinal and transverse tensile specimens are shown below.

Temp.	Modulus E, 10 ⁶ psi	0.2% Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, % in 1 in.	Reduction in area, %
RT	26.5	66.5	93.7	52.4	68.5
RT	29.2	66.7	94.5	51.0	67.6

PLANS FOR FUTURE WORK

Tensile tests will be performed at room temperature and at 315, 650, and 750 °C on specimens in the unirradiated, and the irradiated-plus-annealed condition.

DETERMINATION OF THE FRACTURE TOUGHNESS OF SPECIMENS FROM THE PM-2A PRESSURE VESSEL

J. A. De Mastry, J. W. Sheckherd,
and R. A. Wullaert

OBJECTIVE OF PROGRAM

The objective of the program is to determine the fracture toughness of two types of wedge-opening-loading (WOL) specimens, designated IX WOL and IT WOL, machined from the PM-2A reactor vessel, to evaluate brittle-fracture characteristics and fracture toughness of pressure-vessel materials.

APPROACH BEING USED

Criteria for designing pressure vessels are based on tests of large unirradiated pressure vessels and small samples of irradiated and unirradiated materials. The first opportunity to test a full-size irradiated reactor vessel arose when the Army Portable Medium Power Plant (PM-2A) at Camp Century, Greenland, was retired.

The PM-2A Reactor Vessel Test to obtain brittle fracture of an irradiated reactor vessel under a set of defined conditions including an intentionally introduced material defect is described in WAPD-TM-640. The results will be used to obtain a better understanding of conditions causing brittle fracture in an operating reactor

vessel and to improve criteria for reactor-vessel design and operation.

The purpose of the present program is to make further use of materials from the PM-2A vessel to evaluate brittle-fracture characteristics and fracture toughness. Cracks will be introduced into the irradiated IX WOL and IT WOL specimens, and the cracked specimens will be tested in tension.

The fracture toughness of the irradiated specimens is unknown; therefore, approximate loads based on the results of testing unirradiated PM-2A IX WOL control specimens will be used. The control specimens fatigue cracked at a load of 2200 lb. Initially, a fatigue load of 1800 lb will be applied to the irradiated specimens with a constant-load fatigue machine. It is expected that fatigue cracking will require about 10^6 cycles.

After initial cracking has been accomplished, the crack will be grown to a length of 0.125 in. by continued cycling. It is expected that about 20,000 additional cycles will be required for this crack growth. Visual inspection of the sides of the specimen will be used to determine crack growth. In addition, a slot 0.125 in. deep by 0.003 ± 0.001 in. wide will be machined into several of the IX WOL specimens by electrodischarge machining (EDM).

After EDM slotting or fatigue cracking, the IX specimens will be pulled to failure. Testing will be conducted at temperatures of -100, -20, 72, and 150 °F. All specimens will be pulled at crosshead speeds of

between 0.02 and 0.05 in./min.

Table 4.3 shows the IX WOL specimens of orientations designated "I" and "O" to be tested, the tensile-testing temperature, and the method of slot preparation. After the specimens in Table 4.3 have been tested, the transverse IX WOL specimens shown in Table 4.4 will be tested.

PROGRESS REPORTED PREVIOUSLY

The irradiated IX and IT WOL specimens were received at the Battelle-Columbus Hot-Cell Facility.

WORK THIS PERIOD

Control work was performed on the Hot-Cell Facility push-pull fatigue machine to duplicate testing conditions for the WOL specimens. This preliminary work has been completed. The control specimens were cycled at frequency of 20 cycles/sec between 600 to 1800 lb loads. Fatigue cracks approximately 1/8 in. deep were achieved after 1×10^6 cycles. The IT WOL control specimens were cycled between 700 and 2100 lb to obtain fatigue cracks in 10^6 cycles.

Fatigue cracking of the irradiated samples has been initiated. Specimens PO-7 and PI-7 were fatigue cracked as described above and pulled at failure at -20 °F. The results for the two samples are shown in Table 4.5. Two additional samples of the I-orientation have been fatigue cracked.

TABLE 4.3. Testing Schedule for IX WOL Specimens

Temperature of Tensile Test, °F	Number of Tests to be Conducted for Indicated Orientation		Method of Preparing Defect
	I Orientation	O Orientation	
-100	1	1	Fatigue cracking
-20	2	2	Fatigue cracking
72	2	1	Fatigue cracking
150	-	1	Fatigue cracking
-20	1	1 ^(a)	EDM slot
72	1	1 ^(a)	EDM slot
-20	2	--	EDM slot and treat ^(c)
72	-	1	EDM slot and treat ^(c)
72	-	1	EDM slot and treat ^(b)

(a) These specimens will be used to repeat any test which has produced questionable data. If they are not required as replacements, they will be cycled 500 times to 1.4 x the yield load as determined in Footnote (b) and then tested as shown in Table 4.3

(b) Specimens will be loaded at 72 °F and the yield point (point at which load-deflection curve becomes nonlinear) determined. The specimen will then be loaded to 1.4 x the yield. If there is no failure, the test will be terminated and the specimen retained.

(c) Load to 1.4 x yield determined in Footnote (b), flush with 1:1 HCl for 1 hr at 72 °F, water rinse, and tensile test within 10 hr.

TABLE 4.4. Testing Schedule for IT WOL Specimens and Transverse-Orientation IX WOL Specimens

Temperature of Tensile Test, °F	Number of Tests to be Conducted for Indicated Orientation			Method of Defect Preparation
	Transverse Orientation	I Orientation	O Orientation	
	<u>IX WOL Specimens</u>			
-20	2	--	--	Fatigue cracking
72	1	--	--	Fatigue cracking
<u>IT WOL Specimens</u>				
-20	-	1	1	Fatigue cracking
72	-	1	1	Fatigue cracking

TABLE 4.5. Results of Fracture-Toughness Studies of PM-2A Materials

Specimen	Orientation	Approximate Fluence, n/cm ²	Temperature of Tensile Test, °F	Load to Failure, lb	Toughness, in.
PI-7	Inner 1/4 of vessel	7.7 x 10 ¹⁸	-20	5400	48
PO-7	Outer 3.4 of vessel	4.2 x 10 ¹⁸	-20	8100	78

PLANS FOR FUTURE WORK

Fatigue cracking of the IX WOL and IT WOL specimens from the PM-2A vessel will be completed as described above and the fracture toughness determined.

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IRRADIATION EFFECTS ON STRUCTURAL MATERIALS

Brookhaven National Laboratory

EFFECT OF IRRADIATION ON PROPERTIES
OF COBALT BASE ALLOYS -

J. G. Y. Chow

The objectives of this program are to survey the tensile properties of some commercial cobalt base alloys and some promising experimental alloys to determine if they are susceptible to elevated temperature embrittlement after irradiation.

Table 5.1 shows the nominal compositions of the alloys studied. The commercial alloys were purchased from stock and are materials from production heats. The experimental alloys were promising materials from 50 lb vacuum induction melts made by Union Carbide Corporation under contract from BNL.

All materials were solution treated at 1175 °C for 15 min prior

to test. This treatment may not be the optimum heat treatment for best properties for fuel cladding, but it is the general treatment used to attain high elevated temperature strength for these alloys.

All irradiated specimens were exposed in the HFBR for one (20 day) cycle to fluence of 1.3×10^{20} (E > 0.82 MeV). The irradiation temperature is below 100 °C.

Pre- and postirradiation tensile test data of these cobalt alloys at 25 °C and at various elevated temperatures up to 800 °C are presented in Table 5.2. All the tensile tests were made at a strain rate 0.2%/min unless otherwise indicated in the table.

At 25 °C, all alloys studied show the expected radiation hardening and exhibited appreciable increases in yield strength. Irradiated Haynes 25 and S-1 alloys show severe drops in ductility with the increases in strength. However, the other alloys, which are designed to minimize the formation of intermetallics, show only slight losses in ductility at 25 °C after irradiation.

At elevated temperatures, all alloys in the irradiated condition exhibit a drop in ductility with increasing testing temperature. In this respect, the cobalt base alloys behave similarly to other face centered cubic metals. At temperatures of 750 and 800 °C, all alloys after

TABLE 5.1. Compositions of Cobalt Base Alloys

<u>Alloy</u>	<u>Co</u>	<u>Ni</u>	<u>Cr</u>	<u>W</u>	<u>Mo</u>	<u>Fe</u>	<u>Ti</u>	<u>Others</u>
<u>Commercial Alloys</u>								
Haynes 25	Bal	10	20	15.0	-	1	-	
Haynes 188	Bal	15	20	12	-	-	-	
UMCO 51	Bal	-	28	-	-	20	-	2 Cb
S-1	Bal	10	20	-	7	2	-	<0.01 Si
Multimet	20	20	20	2	5	Bal	-	1 Cb
<u>Experimental Alloys</u>								
Ht 565	Bal	10	21	4.5	5	3	2	
Ht 567	Bal	17.5	19	12	-	-	2	
Ht 569	Bal	11	19	13.5	-	1	-	
Ht 586	Bal	18	19	12	-	2	-	1 Zr

TABLE 5.2. Tensile Properties of Cobalt Base Alloys

Alloy	Exposure Fluence, E > 0.82 MeV	Test Temp., °C	Yield Strength, psi	Tensile Strength, psi	Total Elongation, %
<u>Commercial Alloys</u>					
Haynes 25	0	25*	67,000	152,000	55
	0	800	35,900	41,500	10.5
	1.3 x 10 ²⁰	25	139,000	142,000	15
	1.3 x 10 ²⁰	650	45,600	70,600	17
	1.3 x 10 ²⁰	750	35,000	38,200	9.2
	1.3 x 10 ²⁰	800	39,200	41,500	2.3
Multimet	0	25*	71,000	121,000	43
	0	800	23,600	32,900	55
	1.3 x 10 ²⁰	25	116,000	138,000	31
	1.3 x 10 ²⁰	650	28,100	53,500	11
	1.3 x 10 ²⁰	800	30,000	31,300	6.7
Haynes 188	0	25	71,600	142,500	25
	0	800	37,500	45,500	65
	1.3 x 10 ²⁰	25	133,000	167,000	44
	1.3 x 10 ²⁰	650	46,100	60,800	10
	1.3 x 10 ²⁰	750	38,600	38,600	3
	1.3 x 10 ²⁰	800	37,400	37,400	1.5
UMCO 51	0	25	84,700	171,000	36
	0	650	37,800	72,200	14
	0	800	30,000	40,300	10
	1.3 x 10 ²⁰	25	120,000	146,000	20
	1.3 x 10 ²⁰	650	46,400	68,600	9
	1.3 x 10 ²⁰	750	38,700	46,600	7.5
	1.3 x 10 ²⁰	800	34,200	39,800	6.5
S-1	0	25	47,000	95,200	18.5
	0	800	19,500	33,800	29
	1.3 x 10 ²⁰	25	114,000	118,000	4
	1.3 x 10 ²⁰	650	37,900	47,400	5
	1.3 x 10 ²⁰	750	30,500	31,700	2
	1.3 x 10 ²⁰	800	23,600	23,600	1.5
<u>Experimental Alloys</u>					
Ht 586	0	25	45,200	129,000	60
	0	800	14,100	37,000	28
	1.3 x 10 ²⁰	25	136,000	141,000	44
	1.3 x 10 ²⁰	650	33,000	55,500	14
	1.3 x 10 ²⁰	750	29,000	26,400	4.5
	1.3 x 10 ²⁰	800	23,000	31,000	4
Ht 565	0	25	42,000	100,500	72
	0	800	23,600	37,000	5
	1.3 x 10 ²⁰	25	97,500	100,800	39.5
	1.3 x 10 ²⁰	650	25,200	47,400	11
	1.3 x 10 ²⁰	800	22,300	26,400	2.5
Ht 567	0	25	50,400	124,000	78
	0	800	32,100	34,200	5
	1.3 x 10 ²⁰	25	125,000	160,000	51
	1.3 x 10 ²⁰	650	42,200	63,500	11
	1.3 x 10 ²⁰	800	-	39,600	3
Ht 569	0	25	52,800	128,000	53
	0	800	20,900	37,400	20
	1.3 x 10 ²⁰	25	120,000	131,000	35.5
	1.3 x 10 ²⁰	650	36,600	51,800	12
	1.3 x 10 ²⁰	800	31,900	33,000	3.5

Note: Prior to test or irradiation, all specimens were solution treated at 1175 °C 15 min

All strain rate 0.2%/min except (*) 2%/min

All specimens were irradiated at less than 100 °C

irradiation have less than 10% in total elongation. The experimental alloys which are not embrittled by aging treatment were not better as far as elevated temperature embrittlement is concerned. Several of the fractured specimens were examined metallographically and the mode of failure at 800 °C was grain boundary tearing.

Some of the commercial alloys (Haynes 25 and UMC0 51) appear to be potential cladding materials at 650 °C. After irradiation, these two materials have about twice the strength as Type 304 at 650 °C. The

tensile strength to yield strength ratios of Haynes 25 and UMC0 51 are respectively 1.55 and 1.51. This indicates sound engineering materials since the alloys are capable of undergoing plastic deformation and exhibit considerable work hardening. Table 5.3 compares properties of irradiated Type 304 with those of Haynes 25 and UMC0 51. This comparison shows that these two cobalt base alloys are at least equal to Type 304 in elevated temperature ductility and should be superior material because they are twice as strong as Type 304 at 650 °C. The

TABLE 5.3. Comparison of Properties of Type 304 SS and Cobalt Base Alloys (All specimens irradiated at less than 100 °C to 1.3×10^{20} E > 0.82 MeV.)

Alloy	Test Temp., °C	Yield Strength, psi	Tensile Strength, psi	Total Elongation, %
Haynes 25	25	139,000	142,000	15
	650	45,000	70,000	17
	750	35,000	38,000	9.2
	800	39,200	41,500	2.3
UMCO 51	25	120,000	146,500	20
	650	46,400	68,600	9
	750	38,700	46,600	7.5
	800	34,200	39,600	6.5
Type 304 SS**	25*	81,500	105,000	58
	600	26,000	40,600	14
	650	19,200	29,800	7.5
	650	24,800	31,700	14
	700	23,000	23,900	10
	750	15,800	18,100	13
	800	10,500	11,700	6

* Strain rate 2%/min; all other specimens strained at 0.2%/min

** Data of material annealed from 1050 °C

1175 °C solution treatment used for the cobalt alloys enhanced the strength but it probably is not the optimum treatment for postirradiation ductility. Some compromise on strength might improve the ductility of these alloys by use of lower solution treating temperature and possibly by some thermal mechanical treatment.

Accordingly, some modifications in microstructure of Haynes 25 and UMC0 51 are being studied.

EFFECT OF MICROSTRUCTURE
ON THE ELEVATED TEMPERATURE
DUCTILITY OF STAINLESS STEELS

J. G. Y. Chow

In this program several methods of refining the grain structure of Type 304 austenitic steels are studied and the resulting fine grained structures are tested for resistance to high temperature embrittlement after irradiation.

In this report, the test results on Type 304 where low temperature deformation was used to break up the annealed structure are presented. The microstructure obtained and some of the preliminary results on material deformed at 77 °K and annealed at 760 °C have been discussed in previous reports.

Table 5.4 shows the tensile test results of the material in the annealed condition. The data generally show the severe drop in ductility when deformed at temperatures above 500 °C after irradiation. There seems to be a slight recovery in ductility from 700 to 750 °C (see also Fig-

ure 5.1) but the ductility drops again when tested at 800 °C.

The tensile test results of the materials deformed at low temperatures and followed by various annealing treatments are shown in Table 5.5. The results show that in the treatments consisting of a single deformation process at 77 °K followed by annealing, the improvement in ductility over the annealed Type 304 was nominal.

The best treatment studied appears to be the (B + 760 °C treatment) which consists of deforming 18% at 77 °K, annealing at 705 °C for 16 hr, deforming again 18% at 77 °K, followed by a final anneal at 760 °C for 16 hr. This treatment produced a material with very fine dispersion of carbides and uniform grain diameter of 0.003 to 0.005 mm (ASTM 11).

In the unirradiated condition, the (B + 760 °C) treatment produced material which is more ductile at 750 °C, but the strength is appreciably lower than the material annealed at 1050 °C.

After irradiation to a fluence of 1.3×10^{20} , the room temperature tensile elongation of the (B + 760 °C) treated material drop from 87 to 45%. The elevated temperature ductility drops to about 20% in the testing temperature range of 650 to 800 °C. The total elongation versus testing temperature is plotted in Figure 5.1 with the data of the material annealed from 1050 °C. This comparison shows that in the temperature range of interest for Type 304, the (B + 760 °C) treated materials have about twice the ductility of the material annealed from 1050 °C. Although irradiation

TABLE 5.4. *Tensile Properties of Type 304 SS - Annealed Condition*

<u>Irrad. Exposure</u>	<u>Test Temp., °C</u>	<u>Yield Strength, psi</u>	<u>Tensile Strength, psi</u>	<u>Total Elongation, %</u>
0	25**	34,900	88,500	78
0	750**	14,200	22,500	52
0	750	13,200	16,800	41
0	800	6,700	13,000	22
1.3 x 10 ^{20*}	25	81,500	105,000	58
1.3 x 10 ^{20*}	500	-	58,600	32.5
1.3 x 10 ^{20*}	600	26,000	40,600	14
1.3 x 10 ^{20*}	650	19,200	29,800	7.5
1.3 x 10 ^{20*}	650	24,800	31,700	14
1.3 x 10 ^{20*}	700	23,000	23,900	10
1.3 x 10 ^{20*}	750**	16,300	21,700	18
1.3 x 10 ^{20*}	750	15,800	18,100	13
1.3 x 10 ^{20*}	800	10,500	11,700	6

All specimens annealed at 1050 °C.

* E > 0.82 MeV

Strain rate 0.2%/min except those indicated by (**) 2%/min

TABLE 5.5. *Tensile Properties of Low Temperature Deformed Type 304*

<u>Treatment</u>	<u>Irradiation Exposure-Fluence*</u>	<u>Test Temp., °C</u>	<u>Yield Strength, psi</u>	<u>Tensile Strength, psi</u>	<u>Total Elongation, %</u>
A + 650 °C	0	25**	90,400	129,500	37
A + 650 °C	0	750**	29,600	30,600	40
A + 650 °C	1.3 x 10 ²⁰	25**	-	146,000	28
A + 650 °C	1.3 x 10 ²⁰	750 **	22,400	25,800	12
A + 760 °C	1.3 x 10 ²⁰	650	23,000	29,800	25
A + 760 °C	1.3 x 10 ²⁰	750	-	15,000	8.5
B + 650 °C	0	25**	94,300	132,000	31.5
B + 650 °C	1.3 x 10 ²⁰	750**	15,900	21,600	19
B + 705 °C	0	25**	40,600	99,000	61
B + 705 °C	0	750	11,100	16,900	45
B + 705 °C	1.3 x 10 ²⁰	750	10,800	14,500	20.5
B + 760 °C	0	25	48,800	98,000	52
B + 760 °C	0	650	-	30,500	91
B + 760 °C	0	750	15,800	21,300	87
B + 760 °C	1.3 x 10 ²⁰	25	93,500	108,000	45
B + 760 °C	1.3 x 10 ²⁰	650	17,800	29,200	21.5
B + 760 °C	1.3 x 10 ²⁰	750	12,300	14,200	28
B + 760 °C	1.3 x 10 ²⁰	750	11,400	13,800	19.5
B + 760 °C	1.3 x 10 ²⁰	800	8,150	9,750	20
B + 815 °C	0	25	50,800	97,700	46
B + 815 °C	0	650	16,000	30,500	85
B + 815 °C	0	750	15,400	20,500	85
B + 815 °C	1.3 x 10 ²⁰	650	21,600	24,800	15

Treatment:

A. Deformed 18% at 77 °K plus annealing temperature indicated for 16 hr

B. Deformed 18% at 77 °K, annealed at 705 °C for 16 hr; deformed again for 18% at 77 °K plus final annealing temperature for 16 hr

* E > 0.82 MeV

Strain rate 0.2%/min except those indicated by (**) 2%/min

FIGURE 5.1. Variation of Ductility Versus Testing Temperature of Irradiated Type 304. A Comparison of the Same Material with Different Microstructure

still lowers the elevated temperature ductility severely, a significant improvement in the ductility can be achieved in Type 304 by drastically modifying the microstructure.

Metallographic examination of the (B + 760 °C) treated material shows the fracture at 750 and 800 °C to be predominantly intergranular. The cracks seem to be finer than in annealed Type 304. From the examination of the fracture surface, it appears that the (B + 760 °C) treatment with a total of 36% deformation had not obliterated the effect of the prior austenitic grain boundaries. From the metallographic examination, we conclude that a greater amount of deformation (or perhaps starting out with a smaller grain size) might produce additional improvement in elevated temperature ductility after irradiation.

STAGE III RECOVERY IN NIOBIUM

P. Soo

In the previous Quarterly Progress Report (November, 1967 - January, 1968), it was shown that Stage III recovery in cold-worked niobium occurs in three separate substages, designated I through III, with activation energies of 0.69 ± 0.05 , 0.63 ± 0.05 and 1.16 ± 0.07 eV, respectively. Tentative explanations for the substages were given, as follows.

Substage I - Self-interstitials break free from oxygen and other impurity interstitials and segregate to form loose clusters.

Substage II - The clusters collapse and form interstitial loops. Maximum lattice hardening occurs for a critical loop size and distribution.

Substage III - Oxygen interstitials migrate to traps.

Work has continued in this field in an attempt to confirm the above interpretation. In particular, batches of niobium wires have been strain-aged to conditions I through III and the temperature-dependence of the yield stress have been ascertained for each case. The results, given in Figure 5.2, show that the temperature-dependence has been altered by annealing in the Stage III recovery range. However, the athermal stress for slip has not changed. This shows that the slip barriers which are formed during Stage III recovery are short-range or "thermal" by nature. The substages are discussed separately as follows.

FIGURE 5.2. Temperature-Dependence of the 6% Flow Stress for Mechanisms I-III and Unaged Niobium

Substages I and II

The activation energies for Substages I and II are equal, within experimental error, which indicates that the same defect is migrating. Since the activation energy for vacancy migration is about one-half the self diffusion energy ($1/2 \times 4.17$ eV),⁽¹⁾ it is clear that these defects are not responsible for Substages I or II. From the field-ion microscope studies of Attardo et al.,⁽²⁾ it seems certain that self-interstitial migration is involved. Electron microscopic studies have shown that self-interstitial migration results in the

formation of either interstitial clusters or interstitial dislocation loops.⁽³⁻⁸⁾ However, if impurity atoms are not present, the self-interstitials would annihilate almost immediately after generation and Stage III recovery would be absent. This has been confirmed by Williams et al.⁽⁹⁾ for neutron-irradiated niobium and by Kothe and Schlatt⁽¹⁰⁾ for cold-worked niobium.

Evidence to support the contention that Substage II is a loop hardening process may be obtained from the flow stress data in Figure 5.2. Interstitial loops give rise to tetragonal distortions in the lattice^(11,12) and

lead to a temperature-dependence of the thermal component of the flow stress given by

$$\tau^*{}^{1/2} = \alpha + \beta T^{1/2} \quad (5.1)$$

in which τ^* is the thermal stress, T is the absolute temperature and α and β are constants. Figure 5.3 shows that this relationship is closely obeyed. Since the measured flow stresses were accurate to within $\pm 4\%$, for the as-recrystallized specimens, the deviation from a straight line is real.

Consider the yield stress data for mechanism II in Figure 5.2. Below about 80 °K, the yield stress tends

towards a constant value of approximately 35 kg/mm². This type of behavior has also been observed, in unirradiated niobium, by Ohr et al.⁽¹³⁾ One explanation is that, below 80 °K, the dislocations no longer overcome the clusters by intersection because of the high applied stress involved. Instead, it becomes easier for the dislocations to bow between them with a stress given by:

$$\tau_B = \frac{\mu b}{\ell} \quad (5.2)$$

in which μ is the shear modulus, b the Burger's vector and ℓ the effective cluster spacing on the slip plane. An equivalent mechanism has

FIGURE 5.3. $\tau^*{}^{1/2}$ Versus $T^{1/2}$ Graphs for Mechanism II and Unaged Niobium

been postulated by Koppenaal⁽¹⁴⁾ for neutron-irradiated copper which has been annealed. It may be shown that λ is given by:

$$\lambda = \frac{b}{\sqrt{Nc}} \quad (5.3)$$

in which N is the number of adjacent slip planes over which the cluster is an effective obstacle to slip and c is the concentration of clusters. Van Bueren⁽¹⁵⁾ has shown that the number of interstitials (or vacancies) generated by a plastic strain of ϵ is equal to $10^{21} \times \epsilon^2/\text{cm}^3$, which, for a 6% elongation, gives 6.7×10^{-5} atom fraction of interstitials. If each cluster contains n interstitials, then the effective concentration becomes $6.7 \times 10^{-5}/n$ loops/matrix atom. From this value and Equations (1) and (2), one obtains:

$$\tau_B = \mu(6.7 \times 10^{-5}N/n)^{1/2} \quad (5.4)$$

By subtracting the athermal stress from the total stress, τ_B becomes 30 kg/mm^2 . Knowing that μ equals $3.8 \times 10^3 \text{ kg/mm}^2$,⁽¹⁵⁾ the theoretical values of N , which satisfy Equation (3), may be calculated for various values of n (Table 5.6). From these, the cluster volumes may be obtained from the expression $4\pi(N/2)^3/3$, assuming that the clusters are spherical. To determine the correct cluster volume obtained from this calculation, it is necessary to know the separation of interstitials within the cluster. Let us assume that this is about $2b$, so that each interstitial occupies a volume of about $8b^3$. Table 5.6 then shows the cluster volumes for various values of n . A comparison of the results in columns 3

TABLE 5.6. The Effective Cluster Diameter (N) and Cluster Volume as a Function of the Number of Interstitials (n) in the Clusters

n	N	Cluster Volume	
		$4\pi(N/2)^3/3$	$8nb^3$
2	1.86	$3.4b^3$	$16b^3$
3	2.79	$11.6b^3$	$24b^3$
4	3.74	$26.9b^3$	$32b^3$
5	4.65	$52.0b^3$	$40b^3$
6	5.58	$91.0b^3$	$48b^3$

and 4 shows that there are about 4 or 5 interstitials in the average cluster, immediately prior to collapse. However, it must be emphasized that this value is only an approximation, since several simplifying assumptions were made. For example, no allowance was made for interstitial-vacancy annihilation which might seriously alter the specific numbers of defects, although not the general scheme.

Substage III

Oxygen migration has been assigned to Substage III because of the activation energy, which equals that for oxygen migration in niobium. Williams et al.⁽⁹⁾ have already suggested that oxygen interstitials are the cause of Stage III recovery in neutron-irradiated niobium. They believe that the oxygen atoms migrate to radiation-induced defects in the lattice. Jeanotte's⁽¹⁶⁾ work indicates that in neutron-irradiated tungsten, the impurity interstitials migrate to dislocations where they are trapped.

On the basis of the above work it seems likely that in the current study, the oxygen atoms migrate to the dislocation loops which were formed in mechanism II. This new defect configuration would then be responsible for hardening in Substage III. It must be particularly stable since, from Figures 5.6 and 5.7 in the last report (BNWL-710), it is difficult to dissociate, even for anneals at long times. This would, therefore, reflect a strong binding energy between the loops and oxygen interstitials.

Jump Frequencies and Jump Distances

Useful information may be obtained from a knowledge of the number of jumps which a point defect makes before it is either annihilated or captured. Huntington⁽¹⁷⁾ has shown that the number of jumps (N) may be given by:

$$N_j = A v t \exp\left(-\frac{Q}{kT}\right) \quad (5.5)$$

where A contains an entropy term and is approximately equal to unity, v is the lattice vibrational frequency equal to 10^{13} sec^{-1} , t is the time, Q is the migrational energy, k is Boltzmann's constant and T is the absolute temperature. From this equation, values of N_j and the jump frequency have been determined for $T = 160^\circ \text{C}$ and are given in Table 5.7; along with the total jump distance $\bar{X}$ which is taken to be $\sqrt{N_j} b$. From Equation (3), the separation of clusters containing 5 interstitials is about 130b, which is much smaller than the jump distances for Substages

TABLE 5.7. *Jump Frequencies, Number of Jumps (N_j) and Total Jump Distances ($\bar{X}$) for Mechanisms I-III*

Mechanism	N_j/sec	N_j	$\bar{X}$
I	9.3×10^4	2.5×10^8	$1.6 \times 10^4 b$
II	6.3×10^5	3.3×10^9	$5.7 \times 10^4 b$
III	0.33	7.4×10^3	$8.6 \times 10 b$

I and II in Table 5.7. However, during cluster (or loop) growth, the interstitials do not migrate in a correlated fashion to their final annealed position but travel to and from many different clusters, as the latter proceed through the stages of growth and shrinkage. The total distance traveled is, therefore, much larger than the final separation between clusters.

It is reassuring to note that the defect migrating during Substage III jumps once every 0.33 sec, which is close to the value obtained in internal friction experiments for oxygen in niobium.^(18,19)

In conclusion, this work has shown:

1. Stage III recovery in cold-worked niobium, containing about 100 ppm of interstitial impurities, occurs in three distinct steps with activation energies of 0.69 ± 0.05 , 0.63 ± 0.05 and $1.16 \pm 0.07 \text{ eV}$, respectively. The first two substages are caused by the clustering of self-interstitials and their subsequent collapse to form small dislocation loops. For a tensile

elongation of 6%, the average cluster contains about 4 or 5 interstitials.

2. Annealing in the Stage III recovery range of cold-worked niobium does not cause a significant change in the athermal stress for slip. However, changes do occur in the temperature-dependence of the thermal stress.
3. Dislocation pinning is a subsidiary effect during Stage III recovery in cold-worked niobium and only occurs, to any large extent, when interstitials are freed from the matrix traps. Under suitable conditions, dislocations may be successively locked by self-interstitials, oxygen interstitials and nitrogen (or carbon) interstitials.
4. Carbon begins to precipitate at 250 °C in cold-worked niobium and gives rise to a strong lattice hardening effect.

Work has commenced on neutron-irradiated niobium, which has been irradiated to 10^{18} nvt at approximately 80 °C. The principal objective of this program is to determine whether the recovery of the damage introduced by fast neutron-bombardment shows a parallel behavior to cold-worked material, or whether the particular configuration of the defects is a major factor in the nature of recovery.

RADIATION HARDENING IN TUNGSTEN -

J. M. Galligan

In some irradiated body-centered cubic metals, measurements have shown

that the temperature dependence of the yield stress is the same as in unirradiated specimens.⁽²⁰⁾ This has been taken as evidence that the rate determining mechanism of plastic deformation is the same in irradiated and unirradiated specimens.⁽²¹⁾ In contrast to this, compared to unirradiated specimens, irradiated face-centered cubic metals exhibit a distinct change in the temperature dependence of the yield stress. We have studied this problem in relatively pure single crystals of tungsten to see if the behavior observed in other body-centered cubic metals is general.

In addition to this problem, we have also examined the effect of annealing upon the temperature dependence of the yield stress in irradiated tungsten. This is of interest because Stage III recovery in tungsten is substantially above room temperature (450 °C). In contrast to this, Stage III recovery in iron is below room temperature⁽²²⁾ and such a study is not readily accessible to examination. Also, tungsten has a low inherent interstitial impurity solubility and in the zone refined crystals used in this study, the solubility of interstitial is expected to be of the order of parts per million.

Some results from this investigation are shown in Figure 5.4. Two significant changes in the yield stress versus measuring temperature are evident.

1. The temperature dependence of the irradiated tungsten is not the same as-unirradiated tungsten.

FIGURE 5.4. The Temperature-Dependence of the Critical Resolved Shear Stress of Irradiated Tungsten Single Crystals

2. After irradiated specimens are annealed at a temperature above Stage III, the temperature dependence of the yield stress is again changed and it shows a steeper temperature dependence than either the annealed or as-irradiated crystals.

These results have a bearing on recent discussion of the temperature dependence of the yield stress in neutron irradiated body-centered cubic metals. In this discussion, it is suggested that the temperature dependence of the critical resolved shear stress (CRSS) is not altered by irradiation since the Peierls-Nabarro stress is so high. In the present

experiment, the temperature dependence of the CRSS is somewhat lower than previously reported. This difference is attributed to the avoidance of centerless grinding, which was not done by other investigators, in preparing tensile specimens. Thus, in part, previous experiments on the temperature dependence of the CRSS in tungsten have been obscured by specimen preparation.

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GENERAL ATOMIC DIVISION OF GENERAL DYNAMICS
(No contribution this quarter)

IRRADIATION EFFECTS ON REACTOR STRUCTURAL MATERIALS

Idaho Nuclear Corporation

J. M. Beeston and C. R. Brinkman

HYDROGEN EMBRITTLEMENT OF STEELS

During the past quarter, in the study of the effects of hydrogen on the mechanical properties of steels, (e.g., ductility and susceptibility to time-dependent fracture) progress has been made concerning 1) the behavior of hydrogen during electrolytic charging of irradiated samples, 2) base line data and procedures for hydrogen analysis, 3) changes in stress-rupture properties with hydrogen content, 4) plans for hydrogen permeability measurements, and 5) the encapsulation of samples for irradiation.

The Behavior of Hydrogen During Electrolytic Charging of Irradiated Samples

To determine the effects of irradiation on the rates of hydrogen occlusion during cathodic charging, specimens of HY-80 steel were irradiated and allowed to radioactively decay to 2 to 3 R/hr, and subsequently were hydrogen charged. The results of charging pre- and post-irradiated HY-80 for various times at different current densities are compared in Figures 7.1 and 7.2.

These data show that the hydrogen concentration increases with $t^{1/2}$,

FIGURE 7.1. Cathodic Hydrogenation of Pre- and Post-Irradiated HY-80 Steel at 0.01 A/in.²

FIGURE 7.2. Cathodic Hydrogenation of Pre- and Post-Irradiated HY-80 Steel at 0.1 A/in.²

initially, before leveling off at saturation. The permeability of the irradiated specimens is greater and they occlude more hydrogen than the unirradiated specimens. Although the increased slope of the curve as well as the saturation value indicate a greater permeability for the irradiated samples (slope related to diffusivity), the diffusivity cannot be unequivocally separated from the absorption and disassociation steps in the permeability process without further experimentation. Noted also is

an increase in the scatter of the data with increasing charging time. Several possibilities for this scatter exist, e.g., variations in the cell cathode contact potentials or arsenic ion depletion during charging. It has been reported⁽¹⁾ that during prolonged charging, arsenic in solution tends to form a hydride and escape from solution.

The reasons that irradiated specimens occlude more hydrogen than unirradiated specimens are presently unclear. However, several possibilities exist. Possibly, irradiation changes the surface activity in such a way that the rate of formation of molecular hydrogen

which occurs at the specimen surface is decreased; hence, the number of hydrogen ions or hydrogen atoms available at the metal-solution interface for diffusion into the metal is increased. Another possibility is that an increased surface activity favors the mechanism involved in transporting monatomic hydrogen through the metal surface by decreasing the activation energy of the process involved. Experimentally, it would be very difficult to differentiate between these two mechanisms.

Base Line Data and Procedures for Hydrogen Analysis

Additional cathodic charging experimentation has been completed in which several parameters were varied in an effort to control the hydrogen content and its distribution within a charged

Changes in Stress-Rupture Properties with Hydrogen Content

When it was felt that the conditions required to produce a given hydrogen concentration were adequately known (as established by the work presented in Figure 7.3), notched tensile specimens of A302B and HY-80 steel were hydrogen charged to several different concentrations and loaded to various stress levels below the notch tensile strength to determine to what degree these steels were susceptible to hydrogen induced time-dependent failure. Specimens that failed instantly or in a short time were analyzed for their post-fracture hydrogen contents as a final check on the reproducibility of the charging and aging technique.

Specimens of A302B were charged and aged to three different initial hydrogen concentrations, (~1, 2, and 4 to 5 ppm). Specimens of HY-80 steel were charged and aged to concentrations of 1.5 to 2.5 ppm and 4.5 to 6 ppm. For these steels, failure stresses as a function of time and hydrogen concentration are shown in Figures 7.4 and 7.5. The hydrogen concentrations given in Figures 7.4 and 7.5 are probably somewhat lower than those contained in the specimens prior to loading. The applied elastic stress gradient added to the concentration gradient would increase the flux of hydrogen atoms out of the specimen. Figure 7.4 shows that for the hydrogen concentrations studied, preirradiated A302B shows only a slight tendency towards time dependent fracture. The principal effect is a decrease in ductility with increasing hydrogen content and a resultant lowering of the notched tensile strength of this steel. The data presented in Figure 7.5 show a similar effect for HY-80 steel; however, there is a slight increase in the tendency for time dependent failure.

Plans for the Hydrogen Permeability Measurements

Current theories of hydrogen embrittlement recognize the role that the rates of hydrogen diffusion play in triggering the deleterious mechanisms involved. Many investigators⁽¹⁻⁴⁾ have pointed out an anomalous behavior of the diffusion of hydrogen below temperatures of 400 °C; i.e., the values of the diffusivity, D , are small com-

pared with the values obtained from extrapolation of high temperature data. In an effort to determine the effects of irradiation on the permeability of hydrogen through steels over the temperature range where embrittlement in reactor pressure vessel steels could occur (i.e., 25 to 300 °C), a special specimen and simple apparatus are being constructed. Several specimens of A302B steel irradiated from between 10^{19} to 10^{21} n/cm² ($E > 1$ MeV) will be used for this determination.

The Encapsulation of Samples for Irradiation

The two assemblies containing specimens for irradiation in the ETR F-6 position have been prepared and delivered to Reactor Operations for insertion. The first assembly for insertion at Cycle 95 contains nine capsules with a total of 162 specimens of A302B, A542, and HY-80 steels, thermal-bonded in helium. The second capsule assembly for insertion at Cycle 96 for three cycles contains 147 specimens, including three A302B specimens for permeability measurements.

FATIGUE OF CLADDING AND STRUCTURAL MATERIALS

The investigation to determine the elevated temperature uniaxial fatigue properties of A302B, HY-80, A542, 316 SS, 304 SS, 304L (Ti modified) SS, and Incoloy 800 is continuing. Additional program details have been given in previous reports.^(5,6) This report presents the status of preparations

FIGURE 7.4. Time Dependent Fracture Studies Compared for Preirradiated A302B Steel

FIGURE 7.5. Time Dependent Fracture Studies Compared for Preirradiated HY-80 Steel

required for irradiation and testing of the materials referred to above.

Encapsulation of the specimens into two subassemblies for irradiation in EBR II has been completed and irradiation of both subassemblies will begin with Run #28 (April, 1968). The first subassembly contains 105 specimens. Each subassembly consists of seven elements, each element containing two structural material inserts and one cladding material insert to be irradiated for 4 mo. One of the structural inserts is shown in Figure 7.6 before assembly. In the first subassembly, the structural materials will be exposed to a fluence of approximately 10^{19} to 2.7×10^{20} n/cm² ($E > 1$ MeV) and the cladding materials to approximately 2×10^{21} n/cm² ($E > 1$ MeV). The second subassembly similarly consists of seven elements, each containing two structural material inserts and one cladding material insert to be irradiated for 2 yr. The structural materials in the second subassembly will thus be exposed to a fluence of approximately 6×10^{19} to 1.6×10^{21} n/cm² ($E > 1$ MeV) and the cladding materials to approximately 1.2×10^{22} n/cm² ($E > 1$ MeV). An extensometer designed to measure the diametral changes of an hour-glass shaped specimen subjected to uniaxial push-pull fatigue has been assembled and calibrated (see Figure 7.7). An inert atmosphere shielding enclosure which will enable testing of the irradiated specimens at temperatures to 800 °C or higher is being prepared.

The cladding inserts contain specimens thermally bonded in argon with impurities less than 1 ppm. The specimen temperature will be that determined by the gamma heating and at full reactor power was designed for 700 ± 100 °C.

The structural inserts contain carbon steel specimens bonded in sodium to hold the irradiation temperature to approximately 380 °C. The sodium purified by cold trap, 10 μ filter, and zirconium getter at 650 °C is estimated to contain less than 10 ppm oxygen. The specimen loading atmosphere impurity was less than 1 ppm oxygen and 15 ppm moisture.

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FIGURE 7.6. *Photograph of Structural Insert Before Assembly*

FIGURE 7.7. *Photograph of Diametral Extensometer and Heating Coil Arrangement*

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DEVELOPMENT OF TEST PROCEDURES, FRACTURE TOUGHNESS
OF REACTOR VESSEL STEELS

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INTRODUCTION

The extension of linear elastic fracture mechanics to the testing of low strength steels presents two problems. First, to obtain plane strain crack extension and to preserve the validity of the elastic analysis, the thickness and other specimen dimensions must be considerably larger than is required in testing high strength materials. Second, the plane strain crack toughness, K_{IC} , may be strongly dependent on strain rate so that the dynamic toughness of the steel may be much lower than its static value, and crack arrest may require a reduction of the stress intensity factor to values well below K_{IC} for crack initiation.

In general, specimen dimensions must be proportioned to the crack tip plastic zone, the size of which is in turn proportional to the quantity $(K_C/\sigma_Y)^2$, where K_C is the value of the stress intensity factor at instability and σ_Y is the yield stress.* To measure a valid plane strain toughness, the specimen thickness, B , must satisfy an inequality of the type

$$B \geq \alpha (K_C/\sigma_Y)^2 \quad (8.1)$$

where α is a parameter the value of which must be established experimentally. On the basis of tests on high strength steels and aluminum, Brown and Srawley⁽¹⁾ have proposed the criterion

$$B \geq (2.5) \left(\frac{K_C}{\sigma_Y} \right)^2 \quad (8.2)$$

When this relationship is satisfied, the thickness should be sufficient to allow identification of the measured toughness, K_C , as a plane strain value, K_{IC} . However, for the low strength steels K_{IC} (psi- $\sqrt{\text{in.}}$) has values equal to or greater than σ_Y (psi) at room temperature, in which case the above criterion requires a specimen thickness of several inches.

One method of reducing the required specimen thickness is to test at high loading rates so as to elevate σ_Y . Assuming a yield stress elevation of 6 ksi per order of magnitude increase in loading rate as suggested by Irwin and Krafft,⁽²⁾ the effect of rate on the thickness required to measure a given K_{IC} value can be calculated by Equation (8.1). The results of such a calculation, plotted in Figure 8.1, show that the reduction in required thickness is substantial, particularly at lower strength levels.

* Throughout this report K_C is used to denote a general value of fracture toughness, and not specifically either a plane strain or a plane stress value.

FIGURE 8.1. Effect of Loading Rate on the Specimen Thickness Required to Measure a Given Value of K_{IC} Assuming That a Tenfold Increase in Loading Rate Elevates the Yield Stress by 6 ksi

Besides its indirect effect in controlling the plastic zone size, straining rate may have a direct influence on K_{IC} . Although experimental information on this is as yet scarce, the data which are available indicate that the effect could be quite serious. Figure 8.2 is based on data presented by Krafft and Irwin.⁽²⁾ The solid points, which are estimates of K_{IC} based on measurements of strain hardening exponent, indicate that the crack toughness at dynamic testing rates may be less than half of the static toughness. The data suggest that there is a range of strain rates (or cracking velocities) corresponding to a minimum plane strain toughness. The low K_{IC} value associated with this minimum indicates that toughness values measured in slow tests could

lead to a dangerous overestimation of the material's performance in service.

Tests at high loading rates would be expected to give more reliable values of K_{IC} , i.e., toughness values closer to, but not necessarily at the minimum. High straining rates in the crack tip plastic zone can be produced by rapid loading of a specimen containing a stationary crack; but once unstable fracture has initiated, the crack velocity determines the straining rate, and the crack should be free to accelerate until it attains the velocity corresponding to the minimum K_{IC} . Crack arrest would require that the magnitude of the stress intensity factor, K , be reduced below the minimum K_{IC} ; therefore, a measurement of K_{IC} at arrest should correspond to the minimum crack toughness.

This investigation was concerned with the fracture behavior of A302B steel. Attention was focused on the specimen thickness required to establish reliable plane strain toughness values, and in particular, on the effect of loading rate on the required thickness. Of equal concern was an evaluation of the rate sensitivity of the crack toughness. The fracture toughness measurements were made with contoured double cantilever beam (DCB) specimens, which are particularly convenient for the measurement of arrest toughness.

MATERIAL CHARACTERIZATION

Plates of A302B steel in four-inch thicknesses were used throughout the study. The composition of the plates

FIGURE 8.2. Effect of Strain Rate on K_{Ic} (-12°F , $0.20\% \text{ C Steel}$, $\sigma_y = 34 \text{ ksi}$ at room temperature)

is shown in Table 8.1 and the heat treatment was as follows:

Austenitize 7 hr at 1650°F

Water Quench

Temper 8 hr at 1200°F

Retemper 7 hr at 1250°F .

In this thickness the material is not through hardening, therefore, a survey of the tensile properties was made through the thickness. The results from specimens cut so that their tensile axes were parallel with the roll surface and in both directions with respect to that of rolling are shown in Table 8.2. Because of the large

TABLE 8.1. Composition of A302B Steel Plates

Material	wt%
C	0.22
Cu	0.07
Mn	1.33
P	0.017
S	0.013
Si	0.30
Cr	0.12
Mo	0.52
Ni	0.21

*TABLE 8.2. Static Tensile Data
(Parallel with Plate Surface)*

property variation, it was necessary that all the test material represent the same thickness position. To obtain the maximum number of specimens from the limited amount of materials, all test pieces were machined so that the center plane was evaluated in the thickness direction, as shown in Figure 8.3.

Static tensile and Charpy impact tests in this direction were used to

characterize the steel. The ambient temperature tensile yield strength was 71 ksi, which is somewhat higher than the value obtained with the specimens parallel to the rolled surface on this same plane. The Charpy impact results are shown in Figure 8.4. These data indicate a markedly higher transition temperature than would normally be expected for A302B.

TEMPERATURE AND RATE DEPENDENCE OF YIELD STRENGTH

Experimental Details

Tensile tests were made with standard 0.357 in. diam specimens located (as shown in Figure 8.3) with the tensile axis corresponding to the opening stress direction in the fracture toughness specimens. The tensile bars were instrumented with a strain gage located at the midpoint of the specimens and hence at the mid-plane of the 4 in. plate of A302B steel.

FIGURE 8.3. Location of Fracture Toughness, Charpy Impact, and Tensile Specimens in 4 in. Thick Plate of A302B Steel

FIGURE 8.4. Charpy Impact Energy on Plate Midplane

The tests were run in a closed-loop servo-hydraulic machine which could provide loading times up to the yield point as fast as 10 msec and, of course, any desired slower loading rate. Curves of load versus time and load versus strain gage output were recorded on an oscilloscope. A typical record is shown in Figure 8.5. The load-strain curve (starting from lower left) indicates a 0.2% strain offset load of 7700 lb, while the load-time curve (starting from lower right) indicates a loading time up to the yield point of $t_c = 6.5$ msec based on the linear portion of the curve.

FIGURE 8.5. Tensile Test Oscilloscope Record (Load-Strain Starts from Lower Left, Load-Time from Lower Right)

Results

Yield stress measurements obtained at room temperature (70 °F), 0, and -70 °F are summarized in Figure 8.6. The reciprocal loading time (1/t in reciprocal seconds) is used as the measure of loading rate. Figure 8.6 indicates that the yield stress at -70 °F is approximately 5 to 7 ksi greater than at 0 °F over the whole range of loading rates. At both of these temperatures, the increase in yield strength is relatively modest up to 0.1 to 1.0 sec.⁻¹, and then increases at about 6 ksi per order of magnitude increase in loading rate. The room temperature data show less rate sensitivity over the same range of loading rates. At the highest loading rates used, however, the room temperature curve also appears to approach a slope of 6 ksi per order.

FRACTURE TOUGHNESS TEST PROCEDURE

The use of contoured DCB specimens for the measurement of fracture toughness has been described previously,⁽³⁾ therefore, the description here will be limited primarily to procedures adopted since that time.

Specimen Design

The specimens used to test A302B steel are shown in Figures 8.7a and b. Two thicknesses, $b = 1.00$ in. and $b = 2.00$ in. were employed; and 45-degree, 0.010 in. radius side grooves were machined to $b_n/b = 0.75$ or $b_n/b = 0.50$. The contour for each

of the specimens shown in Figure 8.7 was saw-cut to satisfy

$$m = \left(\frac{3a^2}{h^3} \right) + \frac{1}{h} = 3.0 \text{ in.}^{-1} \quad (8.3a)$$

where "h" is the beam height at the distance "a" measured from the line of action of the load. The procedure described in Reference 3 was followed in making compliance calibrations on 1 in. and 2 in. thick specimens with $b_n/b = 0.75$. For crack lengths within the contoured region the compliance change with crack length, dC/da , was constant and given by

$$m' = \frac{Eb}{8} \left(\frac{dC}{da} \right) = 4.75 \text{ in.}^{-1} \quad (8.3b)$$

This value was used to calculate the dependence of the stress intensity factor, K, on applied load, P:

$$K = \frac{2P}{\sqrt{b} \sqrt{b_n}} \left(\frac{Eb}{8} \right) \frac{dC}{da}^{1/2} \\ = 4.36 \frac{P}{\sqrt{b} \sqrt{b_n}} \quad (8.4)$$

The value of K given by this expression is the average value across the net section.

Fatigue Precracking

Prior to testing, a fatigue crack was extended into the contoured portion of the specimen. The advance of the crack was monitored by compliance measurements, and, of course, the final position could be verified visually after the specimen was fractured.

FIGURE 8.6. Yield Stress of A302B Steel as a Function of Loading Rate

Specimens tested at room temperature were fatigue precracked at room temperature under an alternating K , i.e., $\Delta K = 5\text{--}45 \text{ ksi-}\sqrt{\text{in.}}$. This practice gave cracking rates of the order of 0.005 to 0.010 in. per thousand cycles. Specimens tested at 0 and -70°F were also fatigue cracked some distance at room temperature, after which fatigue cracking was continued with the specimens packed in dry ice. For the last stage of fatigue crack extension, the temperature was between -60 and -20°F and ΔK was 5 to 35

$\text{ksi-}\sqrt{\text{in.}}$. Cracking rates were of the order of 0.001 to 0.002 in. per thousand cycles.

The fracture surface of specimens precracked at room temperature and approximately -40°F are shown in Figure 8.8. Instead of the typical fatigue crack front which leads in the center, the use of side grooves encourages the fatigue crack to lead slightly at the edges, while the arrested fast crack continues to lead at the center. Comparison of the two specimens shows that the slower, low temperature fatigue produced a much smoother crack surface.

FIGURE 8.7a. Type A Contoured DCB Fracture Toughness Specimen ($m = 3 \text{ in.}^{-1}$)

FIGURE 8.7b. Type B Contoured DCB Fracture Toughness Specimen ($m = 3 \text{ in.}^{-1}$)

(a)

(b)

FIGURE 8.8. Fracture Surfaces;
(a) Fatigue Cracked and Tested
at Room Temperature; (b) Fatigue
Cracked at ~ -40 °F and Tested at 0 °F

Toughness Testing

Fracture toughness tests were run at three temperatures: room temperature (70 °F), 0, and -70 °F. For the low temperature tests, the specimens were chilled in dry ice then allowed to warm up in the testing machine. The temperature was monitored by two thermocouples spot-welded to the specimen, and located

3 in. apart adjacent to the crack plane. The test was run when both thermocouples gave readings within 2 °F of the desired temperature.

To obtain a wide range of loading rates, the toughness tests were run in a closed-loop servo-hydraulic testing machine which could load the specimens to fracture in times as short as 10 msec, and, of course, give any desired slower loading rate. The specimens were loaded with a programmed displacement signal of sufficient amplitude to cause complete fracture in one loading pulse. Typically, the specimens did not break in a single burst of unstable crack extension; but rather in distinct jumps readily observable on load versus time and load versus displacement traces obtained with an oscilloscope. The test record shown in Figure 8.9 illustrates the type of behavior usually observed. The load-deflection trace (starting from the lower left) was linear up to the point of fracture initiation from the fatigue crack at a load of 14,000 lb. From this point, the load dropped abruptly to 11,000 lb as the crack propagated. When the crack had arrested, the load-deflection curve again increased linearly until fast cracking was reinitiated at a load of 14,500 lb and the crack ran the length of the specimen. The load-time curve, plotted from the lower right on a more sensitive load scale, shows the same features. The test record, then, provides two load values from which to calculate K_{Ic} for crack initiation and one for arrest. The slope of the load-time curve

	<u>UTS</u>	<u>Y.S.</u>	<u>Elong.</u>	<u>R.A.</u>
	Longitudinal			
Surface	98.8	83.7	25.5	69
1/4 T	95.0	75.0	25.0	68
1/2 T	86.0	65.9	26.5	68
	Transverse			
Surface	98.5	81.2	26.5	71
1/4T	95.3	77.0	25.0	70
1/2 T	84.8	65.8	26.0	70

FIGURE 8.9. Fracture Toughness Test Record (Load-deflection curve starts from lower left, load-time curve from lower right)

measures the loading rate, which is conveniently expressed in terms of the loading time to fracture t_c . The load-deflection curve verifies substantially linear behavior up to the point of instability, and the change in slope can be used to calculate the distance over which the crack propagated between the successive initiations. Because of the linear compliance change feature of the specimen, exact knowledge of the crack position is not needed to calculate a K value for arrest; it is only necessary that the crack front be within the contoured region of the specimen.

Individual tests varied in the number of distinct initiation and arrest points which occurred during the loading pulse. In some cases no crack arrest occurred. When there were numerous arrests, it was found that successive initiation and arrest load values often increased

progressively. Sometimes the increase could be attributed to a wandering of the crack from the minimum section or "crowning" of the fracture surface; at other times, there were no obvious irregularities in the fracture appearance. Because of the anomalous behavior, only the first crack initiation from the fatigue crack and the first arrest point were used to calculate the fracture toughness values presented below.

Drop-Weight Tests

To obtain faster loading rates, the drop-weight testing arrangement shown schematically in Figure 8.10 was used. The top loading arm of the specimen is attached through a load cell to a rigid frame. A 3 ft long steel rod with a flange on the bottom is suspended from the lower loading arm of the specimen. The specimen is loaded when a 100 lb weight dropped the length of the rod is caught by the flange. To obtain loading records free of spurious vibrations, it was necessary to place a cushion between the weight and the flange so that the loading rate was limited to the natural frequency of the load train. Figure 8.11a shows the record obtained when a dummy specimen, which did not crack, was loaded with this arrangement. Load versus time is plotted from the lower left; and deflection (clip-gage output) is plotted from the upper left on the same time scale. The fact that deflection and load traces are

FIGURE 8.10. Schematic Drawing of Drop-Weight Test Arrangement

in phase is evidence that the load cell measures a meaningful load on the specimen.

The record of an actual test is shown in Figure 8.11b. The load-time trace shows the fracture load (18,000 lb) and indicates a loading time of 1 msec, which is an order of magnitude faster than the rate attainable with the hydraulic machine. Typically, as is the case in Figure 8.11b, the drop weight tests showed no crack arrest.

FRACTURE TOUGHNESS TEST RESULTS

The results of the fracture toughness tests conducted at room temperature are plotted in Figure 8.12. A limited number of specimens with deep face grooves ($b_n/b = 0.50$) were tested; however, the results of these tests were not distinctly different from results on specimens with $b_n/b = 0.75$, which comprise most of the data. Rather, the measured values of initiation toughness fell into two groups based on the gross specimen thickness, b . Tests on 2 in. thick specimens defined a toughness of 85 to 90 $\text{ksi}\sqrt{\text{in.}}$ over the entire range of loading rates from 1.0 sec^{-1} to 100 sec^{-1} , and tests on 1 in. thick specimens at 100 sec^{-1} and at 1000 sec^{-1} showed this same level of toughness. At loading rates below about 100 sec^{-1} , however, the initiation toughness values obtained from 1 in. thick specimens showed a progressive increase up to 115 $\text{ksi}\sqrt{\text{in.}}$

The measured arrest toughness values are plotted directly below the corresponding initiation values although, of course, the loading rate has no immediate significance for crack arrest. With the exception of two tests on 1 in. specimens at the slowest loading rates, the data from all of the specimens define a single arrest toughness level of 75-80 $\text{ksi}\sqrt{\text{in.}}$

The data portrayed in Figure 8.13 are for tests run at 0 °F on specimens which had been fatigue cracked at about -40 °F as described above. Although there are not enough data in

(a) Dummy Specimen

(b) Actual Test

FIGURE 8.11. Load-Time and Deflection-Time Traces Obtained in Drop-Weight Tests. The P-t Traces Start from the Lower Left Corner of the Photographs, and the δ -t from the Upper Left.

FIGURE 8.12. Room Temperature Crack Toughness of A302B Steel

FIGURE 8.13. Crack Toughness of A302B Steel at 0 °F;
Specimens Fatigue Cracked at ~-40 °F

this figure to establish a definite statistical trend, it does suggest somewhat the same behavior as observed in the room temperature test results. The two specimen thicknesses appear to give the same toughness at high loading rates. As the loading rate was decreased, there was a tendency for the toughness measured on 1 in. thick specimens to increase, while at the lowest loading rates two tests on 2 in. thick specimens gave quite low values. The 0 °F test results defined an arrest toughness of approximately 60 ksi- $\sqrt{\text{in.}}$, and, in general, these values were very close to those obtained on initiation.

The data obtained at -70 °F are plotted in Figure 8.14. Initiation toughness values measured on 1 in.

thick specimens showed a mild strain rate dependence for loading rates ranging from 0.01 to 100 sec.⁻¹. Two points at higher loading rates obtained in drop weight tests showed somewhat elevated values. One test with a 2 in. specimen at 0.001 sec.⁻¹ loading rate gave a toughness value very slightly below the trend line established by tests on 1 in. specimens. All measured arrest toughnesses were in the range 55 to 60 ksi- $\sqrt{\text{in.}}$ which is approximately the same level that was observed at 0 °F.

The tests conducted at -70 °F should represent a closer approach to plane strain fracture conditions than the tests at the higher temperatures. In addition, they extended over the greatest range of loading rates and

FIGURE 8.14. Crack Toughness of A302B Steel at -70 °F; Specimens Fatigue Cracked at ~-40 °F

showed the least scatter. The initiation toughnesses were only moderately dependent on loading rate, and the arrest toughness was only slightly below the initiation toughness. Approximately the same level of arrest toughness (60 versus to 55-60 ksi- $\sqrt{\text{in.}}$) was measured at 0 °F as at -70 °F and the results were again essentially independent of loading rate.

In the room temperature tests, a dependence of toughness on loading rate was exhibited by the tests on 1 in. thick specimens, but not on 2 in. specimens. The data, therefore, do not reflect an effect of strain rate on fracture toughness, but rather an increasing departure from plane strain cracking conditions as the loading rate for the thinner specimens was decreased. The data on 2 in. thick specimens along with the data on 1 in. specimens at the highest strain rates again indicate that the toughness is essentially independent of strain rate.

DISCUSSION

The results indicate that the direct influence of strain rate on toughness is modest, but there is an indirect effect traceable to the strain rate dependence of the yield strength. The latter effect can be viewed in the larger context of the influence of specimen size. Because the plastic zone size at a given value of K_C is proportional to σ_Y^{-2} , one can define an effective thickness B^* as:

$$B^* = B \left(\frac{\sigma_Y}{\sigma_0} \right)^2$$

where σ_0 is the room temperature static yield strength. The factor in the parenthesis can be considered a correction in "b" produced by the change in yield strength due to temperature or loading rate changes. In Figure 8.15 the toughness values given in Figures 8.12, 8.13, and 8.14 are plotted against $1/B^*$ where B has been taken as the gross thickness, b , and σ_Y was taken from the data in Figure 8.6 by matching the reciprocal loading time of the tensile test with that of the fracture toughness test. The drop-weight data were plotted at a value of σ_Y obtained by extrapolating the curves of Figure 8.6 to a reciprocal loading time of 1000 sec^{-1} . For $1/B^*$ in the range 0.4 to 0.8, most of the room temperature data give K_C in the range 85 to 90 ksi- $\sqrt{\text{in.}}$. At higher values of $1/B^*$, K_C shows a continuous increase. Most of the points, both from the 0 and -70 °F tests are in the range 60 to 65 ksi- $\sqrt{\text{in.}}$, and show no systematic dependence on $1/B^*$. Thus, the only evidence of a thickness effect is in the room temperature data on 1 in. thick specimens tested at low loading rates.

The thickness requirement for plane strain toughness can be expressed in terms of the parameter β_C defined as

$$\beta_C = \frac{1}{B} \left(\frac{K_C}{\sigma_Y} \right)^2$$

The parameter β_C is, in effect, a ratio of the plastic zone size to the specimen thickness. The Brown and Srawley criterion cited in the Introduction suggests that, if $\beta_C \leq 0.4$,

FIGURE 8.15. Dependence of Crack Toughness on Effective Thickness

then K_C may be identified as the plane strain fracture toughness K_{IC} . The lower dashed curve plotted in Figure 8.15 corresponding to $\beta_C = 0.4$ shows that this criterion rules out all of the room temperature data and most of the lower temperature data as invalid. The upper dashed curve $\beta_C = 1.0$, better represents the onset of a detectable thickness dependence in the room temperature data, and defines all of the lower temperature data as valid in agreement with the observed absence of any thickness dependence. Thus,

$$\beta_C \leq 1.0 \text{ or } B \geq \left(\frac{K_C}{\sigma_Y} \right)^2$$

seems to be a more realistic criterion for plane strain fracturing for this material.

In a more strict sense, the plane strain toughness K_{IC} might be considered to be the limiting value of K_C as β_C tends to zero. In line with this viewpoint, Irwin has suggested a relationship between K_C and K_{IC} based on experimental results from several different materials.⁽⁴⁾ The relationship can be written in the form:

$$\left(\frac{K_C}{K_{IC}} \right)^2 = 1 + \frac{1.4}{B^2} \left(\frac{K_{IC}}{\sigma_Y} \right)^4 \quad (8.5)$$

The formula associates K_{IC} with an infinite plate thickness ($B \rightarrow \infty$).

This definition of K_{IC} yields a somewhat lower value of K_{IC} than is allowable with the Brown-Srawley criterion. Indeed, if one assumes $\beta_C = 0.4$, and that this yields a toughness K_C , then the calculated K_{IC} is lower than the measured value by about 7%.

When the value of K_{IC} is calculated according to Equation (8.5) assuming that all the data of Figures 8.13, 8.14 and 8.15 are K_C values, the results plotted in Figure 8.16 are obtained. The effect of the thickness correction is shown most strikingly in the room temperature test results, particularly for the 1 in. thick specimens (see Figure 8.12). The apparent rate dependence shown by the uncorrected data has been eliminated; the corrected data for 1 in. specimens define a toughness of 70 ksi- $\sqrt{\text{in.}}$ over the whole range of loading rates. The corrected results for 2 in. thick specimens define a slightly higher toughness. Application of the thickness correction to the test results for 0 and -70 °F had only a modest effect because of the relatively lower values of $(K_C/\sigma_Y)^2$ at the lower temperatures. For both low temperatures, the results plotted in Figure 8.16 indicate a K_{IC} value of 55 to 60 ksi- $\sqrt{\text{in.}}$ in good agreement with the lower boundary of the uncorrected arrest toughness measurements.

The behavior of the thickness correction is also illustrated in Figure 8.17. Assuming K_{IC} values of 70 and 55 ksi- $\sqrt{\text{in.}}$, respectively, for the ambient and low temperature tests (from Figure 8.16), Equation (8.5) was

used to generate curves of K_C versus $1/B^*$ in Figure 8.17. The $K_{IC} = 70$ ksi- $\sqrt{\text{in.}}$ curve fits the 1 in. room temperature data very well, but underestimates the results from 2 in. specimens. Thus, calculated value, $K_{IC} = 70$ ksi- $\sqrt{\text{in.}}$, which is based primarily on data for which $\beta_C > 1.0$, is conservative with respect to the measured data obtained on the 2 in. thick specimens (i.e., $\beta_C < 1.0$). The latter suggest $K_{IC} = 85$ ksi- $\sqrt{\text{in.}}$. The K_C versus $1/B^*$ curve based on $K_{IC} = 55$ ksi- $\sqrt{\text{in.}}$ (Figure 8.17) represents the 0 and -70 °F results quite well. It is again seen that the lower boundary value of arrest toughnesses at both ambient and low temperatures defines a value consistent with the calculated K_{IC} . These values are plotted in Figure 8.17, at some arbitrary value to the left of the drop-weight tests, with the 2 in. thick data points at a $1/B^*$ position equal to roughly half that of the 1 in. thick point.

Applied to the data presented here, Equation (8.5) provides a method for converting a measured toughness, K_C , into a plane strain toughness, K_{IC} , which is conservative in comparison to the values indicated by the uncorrected data. As a value for K_{IC} which represents a lower bound for crack toughness in thick sections, a value 70 ksi- $\sqrt{\text{in.}}$ at room temperature and 55 ksi- $\sqrt{\text{in.}}$ at 0 and -70 °F is obtained. Although it is probable that the actual toughness lies between these values and the directly measured values; the difference between the corrected and uncorrected toughness numbers is not large, and the corrected ones appear to be conservative.

FIGURE 8.16. Thickness Corrected Crack Toughness of A302B Steel

FIGURE 8.17. Correlation of Thickness Correction with Initiation and Arrest Toughness Values

The most striking feature of these test results was the modest influence of loading rate on K_{IC} , and the slight differences between initiation and arrest toughness values. Where a rate dependence did occur, it could be attributed to a thickness influence.

As indicated in the Introduction, the running crack should seek out the velocity corresponding to the minimum crack toughness; hence, crack arrest should require that the stress intensity factor fall below this minimum value. In spite of this, the arrest toughness values were virtually the same as for initiation. As mentioned above, there was a tendency for successive initiation and arrest toughness values obtained on a single specimen to show a progressive increase. Such an increase must reflect a progressive change in the condition of the crack; for example, a roughening of the fracture surfaces. Just as the initiation toughness is found to be influenced by the fatigue precracking procedure, so might the arrest value depend on the amount of "roughness" developed by the running crack. Indeed, if an enhanced toughness produced by "roughening" were to develop sufficiently rapidly, the crack would be expected to arrest at a toughness level only very slightly below the initiation value. To eliminate the contribution of crack "roughness", one might compare each arrest toughness value with the immediately following initiation toughness. The tests run in this study furnished forty-eight

instances where such a comparison could be made. The difference between such an initiation and arrest toughness ranged from 9 to 36% of the initiation value, with an average difference of about 20% at each of the three testing temperatures. If a fast fracture initiated from a fatigue crack propagated without "roughening", one might guess that the arrest toughness would be of the order of 20% less than the initiation toughness. This is still a much smaller rate effect than Krafft's data would suggest; yet, unless "roughening" could always be counted on to enhance the toughness, this 20% toughness loss could be of critical practical importance.

Another factor which should be considered is variation of the condition of the crack across the specimen thickness. Normally, K_{IC} is calculated from a "pop-in" load. The "pop-in" corresponds to unstable propagation of the center portion of the crack, which (when the thickness is sufficient) represents a condition of plane strain. Arrest of the "pop-in" crack, however, is almost certainly dominated by the departure from plane strain conditions near the specimen surfaces, rather than the value of K at the center of the plate. Hence, arrest of the "pop-in" crack does not represent a meaningful arrest toughness of the material. In the tests described here, the initial instability resulted in propagation of the crack across the full specimen thickness rather than just in the center portion. This behavior may be attributed to the effect of the side

grooves in restricting plastic flow at the plate surface.

SUMMARY

For values of $\beta_c = \frac{1}{B} \left(\frac{K_c}{\sigma_Y} \right)^2$ less

than about 1.0, the measured values of initiation toughness were independent of thickness and loading rate. Thus, the criterion $\beta_c \leq 1.0$, instead of the more conservative criterion, $\beta_c \leq 0.4$, better described the thickness requirement for plane strain fracturing in this material. Using this criterion, most of the room temperature data and all of the 0 and -70 °F data represent valid toughness measurements. They define K_{IC} 85 to 90 ksi-√in. at room temperature and $K_{IC} = 55$ to 60 ksi-√in. at both 0 and -70 °F. The arrest toughness measurements were 75 to 80 ksi-√in. at room temperature and 55 to 60 ksi-√in. at the lower temperatures.

A more conservative method of defining K_{IC} was to use the $K_{IC} - K_c - \beta_c$ relationship suggested by Irwin. This gave $K_{IC} = 70$ ksi-√in. at room temperature and $K_{IC} = 55$ ksi-√in. at 0 and -70 °F. These values are also consistent with the arrest toughness measurements. While it is probable that these values underestimate the actual toughness, the difference between these numbers and the measured ones is not too large,

and the corrected values represent a reasonable lower bound for K_{IC} .

The data indicated little strain rate sensitivity of K_{IC} . The effect of loading rate on toughness was modest, and the difference between initiation and arrest values was minimal. While artificial enhancement of arrest toughness by crack "roughening" cannot be ruled out as a possibility, there was no positive evidence to support this interpretation. Despite variations in the length of unstable extension preceding arrest, arrest toughness values generally showed less scatter than the initiation.

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RESEARCH PROGRESS

Naval Research Laboratory

L. E. Steele, J. R. Hawthorne,
C. Z. Serpan, Jr., Uldis PotapovsCONTROLLING THE RADIATION EMBRITTLE-
MENT SENSITIVITY OF Ni-Cr-Mo WELD
DEPOSITS BY VARYING THEIR CHEMICAL
COMPOSITION

High radiation embrittlement sensitivity of a 3 1/2 Ni-Cr-Mo (ASME Code Case) weldment reported previously⁽¹⁾ and its implied effect on the applicability of A543 steel for reactor pressure vessel service prompted the initiation of a cooperative program between NRL and Babcock and Wilcox Company (B&W) to investigate the cause(s) of the observed high embrittlement rate and to develop a suitable, low radiation embrittlement sensitivity, weld filler composition for joining this material. A detailed analysis of the composition and metallurgical characteristics of the ASME Code Case weldment coupled with prior experimental results⁽²⁾ suggested copper content as a critical variable affecting radiation embrittlement sensitivity in the weld metal.

To assess the significance of variable copper concentration, four experimental weldments (1-in. plate) were prepared using the manual metal arc (MMA) process and different welding rod compositions. Copper and nickel were the primary composition variables. In addition, two submerged arc (S/A) weldments (2-in. plate) were prepared using commercial

weld filler (Linde MI-88 and Linde 100). Both commercial weld wires are relatively high in copper. The chemical analysis and tensile data for the individual weld deposits are given in Table 9.1: the specific welding parameters and heat treatments are summarized in Table 9.2.

Charpy-V specimens were taken from the weldments with the long axis perpendicular to the welding direction and the notch centered in the weld deposit. Selected specimens were irradiated at 550 °F together with specimens from an 8 in. A543 reference plate to a fluence of $\sim 3.5 \times 10^{19}$ n/cm² (E > 1 MeV). Postirradiation notch ductility properties of all weldments and of the 8 in. reference plate are summarized in Table 9.3 and shown graphically in Figures 9.1 and 9.2.

It can be seen from Figure 9.1 that the radiation embrittlement sensitivities of the experimental weldments, with the exception of composition 1938, were approximately equal or superior to that of the 8 in. A543 reference plate which exhibited a 105 °F Charpy-V 30 ft-lb transition temperature increase. Comparing the relative transition temperature shifts of the experimental weld compositions to their respective chemical analysis (Table 9.1), a relationship between the copper content of the weld metal

TABLE 9.1. Chemical Analysis and Approximate Tensile Strength of Experimental and Commercial Weld Deposits

Material Identification	Source [†] of Analysis	Analysis (wt%)										Ultimate* Tensile Strength, ksi	0.2% Yield* Strength, ksi
		C	Mn	P	S	Si	Cr	Ni	Mo	Cu	V		
Experimental	B&W	0.04	0.96	0.010	0.016	0.45	2.04	0.79	1.01	0.14	0.02	91.0	81.0
Composition 1934	NRL	0.04	0.87	0.008	0.010	0.40	2.08	0.77	1.01	0.07**	0.02		
Experimental	B&W	0.06	0.94	0.012	0.016	0.26	2.12	0.71	1.02	0.02	0.02	102.0	94.0
Composition 1936	NRL	0.06	0.86	0.007	0.010	0.30	2.08	0.78	1.01	0.02**	0.02		
Experimental	B&W	0.03	0.74	0.011	0.017	0.32	2.39	1.95	0.99	0.14	0.02	99.0	91.0
Composition 1938	NRL	0.03	0.72	0.009	0.010	0.34	2.39	1.62	0.92	0.08	0.03		
Experimental	B&W	0.05	0.98	0.012	0.017	0.22	2.31	1.66	0.94	0.02	0.02	98.5	90.0
Composition 1948	NRL	0.05	0.82	0.007	0.006	0.20	2.32	1.56	0.92	0.07**	0.03		
Linde M1-88	B&W	0.06	1.24	0.011	0.009	0.37	0.17	1.25	0.33	0.24	-	82.5	68.5
	NRL	0.07	1.07	0.011	0.006	0.37	0.25	1.58	0.37	0.24	-		
Linde 100	B&W	0.09	1.38	0.013	0.011	0.27	0.32	2.13	0.48	0.43	-	106.5	96.0
	NRL	0.09	1.15	0.011	0.006	0.22	0.39	2.46	0.52	0.42	-		
8 in. A543 Plate	NRL	0.17	0.34	0.010	0.015	0.25	1.93	3.45	0.51	0.06	0.02	122.0	96.0

* Strength values for the weld deposits are approximate - derived from subsize specimens

** Average of two check analyses

† The comparable analyses were made on different test plates, welded under the same conditions and using the same heat of filler material

and its irradiation response is apparent. Weld composition 1936, which proved to be least sensitive to radiation embrittlement, had the lowest copper content (0.02%) of the series. The data suggest also that the nickel contents of these weldments, in the concentrations indicated, may have some influence on their irradiation behavior. Specifically, the two weld compositions (1938 and 1948) which contained approximately 1.7 to 2% nickel showed a higher embrittlement sensitivity than the two experimental weldments which contained approximately 0.7% nickel.

The influence of copper content on the radiation embrittlement sensitivity of Ni-Cr-Mo weld metal is even more apparent in Figure 9.2 which shows the radiation response of the two submerged arc commercial weldments with relatively high copper contents (0.24 and 0.42%). The respective Charpy-V 30 ft-lb transition temperature shifts for these compositions were ~415 and 530 °F. The data are in good agreement with the earlier findings⁽¹⁾ for the ASME Code Case weldment and substantiate previously reported findings on the influence of

TABLE 9.2. Welding Parameters and Heat Treatment of the Experimental and Commercial Weldments

Experimental Weld Compositions 1934, 1936, 1938, and 1948

Welding Process:	Manual Metal Arc
Electrode:	5/32 in. diam, Type 15 coating
Current:	165 A
Voltage:	25 V
Travel:	6 in./min
Preheat Temperature:	300 °F
Interpass Temperature:	375 °F max.
Postweld Heat Treatment:	1125 to 1150 °F/30 hr, furnace cool to 600 °F

Commercial Weld Wire (Linde MI-88 and Linde 100)

Welding Process:	Submerged Arc
Electrode:	1/8 in. diam
Flux:	Type 1092, size: 20 x 200
Type of Power:	AC
Current:	450 A
Voltage:	32 V
Travel:	14 in./min
Preheat Temperature:	250 °F
Interpass Temperature:	350 °F max.
Postweld Heat Treatment:	1050 to 1075 °F/65 hr, furnace cool to 600 °F; 1075 to 1100 °F/15 hr, furnace cool to 700 °F

copper on the radiation embrittlement sensitivity of A302-B steel.⁽²⁾

Information gained from this phase of the program positively identifies copper as a major contributor to the radiation embrittlement sensitivity of Ni-Cr-Mo steel weld metals. Further, the data demonstrate that the radiation embrittlement sensitivity of these weld metals can be substantially reduced (even below the sensitivity level of the base material) by reducing the copper content of the weld deposit. In continuation of the cooperative program, submerged arc weldments of the two lowest sensitivity experimental weld compositions are being produced and will be evaluated after irradiation to insure that

TABLE 9.3. Charpy-V Notch Ductility Characteristics of the A543 Weld Deposits and Base Plate

Identification	Weldment or Plate Thickness, in.	Charpy-V 30 ft-lb Transition Temperature, °F			Approximate Full Shear Energy Absorption, ft-lb	
		Irradiated at 550 °F			Irradiated at 550 °F	
		Initial	ΔT	(E > 1 MeV)*	Initial	(E > 1 MeV)*
Experimental Composition 1934	1	-105	-10	95	120	95
Experimental Composition 1936	1	-110	-65	45	100	100
Experimental Composition 1938	1	-80	120	200	90	50
Experimental Composition 1948	1	-75	35	110	80	60
Linde MI-88 Filler	2	-125	290	415	120	57
Linde 100 Filler	2	-25	505	530	70	34
A543 Base Plate	8	-130	-25	105	70	60

NOTE: 8-in. A543 base plate heat treated as follows:

1650 °F/2 hr - water quench
1500 °F/2 hr - water quench
1185 °F/2 hr - water quench

Welding conditions and heat treatment for all weldments given in Table 9.2.

* σ 68 mb, ^{54}Mn , fission

FIGURE 9.1. Notch Ductility Behavior of Manual Metal Arch Deposited Experimental Weld Metal Compositions of Joining A543 Steel Before and After Irradiation at 550 °F

FIGURE 9.2. Notch Ductility Behavior of 2-in. HY-80 Weldments (Submerged Arc Commercial Filler) and 8-in. A543 Plate Before and After Irradiation at 550 °F

the low level of radiation embrittlement sensitivity can be reproduced in typical production welding applications.

INFLUENCE OF PRIOR TEMPER EMBRITTLEMENT ON THE IRRADIATION RESPONSE OF Ni-Cr-Mo STEEL

The fabrication of large welded structures of quenched and tempered Ni-Cr-Mo steel may entail shop heat treatments that introduce some degree of temper embrittlement in plate and weld metal. The influence of this metallurgical phenomenon on irradiation response has, therefore, been examined. Initial comparisons⁽³⁾ of temper embrittled and nonembrittled Ni-Cr-Mo plate following neutron irradiation at <280 °F suggested equivalent changes in Charpy-V notch ductility properties with low temperature nuclear service. The study has now been extended to include 650 and 750 °F neutron exposures.

The composition and heat treatment conditions of the 5/8 in. thick plates used for both the current and earlier determinations are given in Table 9.4. The two post-tempering cooling procedures produced a 130° difference in initial plate 30 ft-lb transition temperatures (-220 versus -90 °F). Specimens representing Conditions A (brine quenched) and B (furnace cooled) were irradiated as a single group at 600 to 650 °F to a neutron fluence of 2.5×10^{19} n/cm² (E > 1 MeV). A second set of specimens of Conditions A and B was irradiated for an equivalent period (12 wk), but according to a two-

temperature cycle of 750 °F (3 wk), followed by 650 °F (6 wk), followed by 750 °F (3 wk). The first set of specimens served to represent elevated temperature service below the temperature range producing temper embrittlement in the unirradiated condition. The second set of specimens represented service in the lower portion of the temperature range for temper embrittlement development.

TABLE 9.4. Quenched and Tempered Ni-Cr-Mo Plate for Evaluation of Combined Effects of Temper Embrittlement and Radiation-Induced Embrittlement

A. Chemical Composition, wt%

Carbon	0.13
Manganese	0.34
Silicon	0.26
Phosphorus	0.012
Sulfur	0.022
Nickel	4.00
Chromium	1.71
Molybdenum	0.49
Aluminum*	0.002
Nitrogen	0.008

B. Heat Treatment

- Condition A: Austenitized at 1500 °F for 1 hr, water quenched; tempered at 1200 °F for 1 hr, brine quenched.
- Condition B: Austenitized at 1500 °F for 1 hr, water quenched; tempered at 1200 °F for 1 hr, furnace cooled to 900 °F at 50 °F/hr, held 1 hr at 900 °F, furnace cooled to 600 °F at 50 °F/hr air cooled.

* Acid soluble

Experimental results for the two sets of specimens are summarized in Table 9.5. In both cases, the transition temperature increases for Condition B matched well the corresponding values for Condition A. Cyclic 750 to 650 °F-exposure temperatures, in particular, did not reveal any differences in irradiation response. The investigation strongly suggests that prior temper embrittlement has no effect on the apparent radiation embrittlement sensitivity of Ni-Cr-Mo steel at either low (<450 °F) or elevated (650 to 750 °F) temperatures.

RELATIVE 550 °F IRRADIATION RESPONSE
OF BASE PLATE, WELD METAL, AND WELD
HEAT AFFECTED ZONE OF A 7 1/2 IN.
THICK A533B CLASS 1 PRODUCTION
WELDMENT

The Charpy-V notch ductility properties of base plate, submerged arc weld deposit, and weld heat affected zone (HAZ) of one 7 1/2 in. thick production weldment of A533B Class 1 steel have been evaluated following

irradiation at 550 °F to a fluence of 1.7×10^{19} n/cm² (E > MeV). The chemical composition and heat treatment of plate and weld are given in Table 9.6. As indicated, the weldment base plates represent a single steel heat.

To assess possible plate-to-plate variability in irradiation response, the quarter thickness properties of each plate were evaluated jointly. Specimens of weld deposit were taken with their long axis perpendicular to the welding direction at the same thickness location. The HAZ specimens were taken slightly above and below the quarter thickness layers with their notch centered on a weld fusion line as revealed by conventional metallographic etching. Experimental results for base plate and weld are summarized in Figure 9.3. Data developed on the HAZ are referenced to base plate and weld metal performance in Figure 9.4.

In Figure 9.3, some difference in initial Charpy-V transition temperature behavior of base plate 1 versus

TABLE 9.5. Charpy-V Notch Ductility Behavior of Temper Embrittled and Nonembrittled 5/8 in. Ni-Cr-Mo Steel Plate Before and After Irradiation at Low and Elevated Temperatures

Condition	Initial	Charpy-V 30 ft-lb Transition Temperature, °F		
		650 °F 2.5 x 10 ¹⁹ n/cm ²	Cyclic 750/650* 2.4 x 10 ¹⁹ n/cm ²	<280 °F 1.1 x 10 ²⁰ n/cm ² **
A. Brine Quenched	-220	90	70	290
B. Furnace Cooled	-90	75	55	315

* 750 °F (3 wk) plus 650 °F (6 wk) plus 750 °F (3 wk)

** Reference 3

*TABLE 9.6. Chemical Composition and Heat Treatment
of 7 1/2 in. A533B Class I Steel Submerged-Arc Weldment*

Source	A. Chemical Composition									
	C	Mn	P	S	Si	Ni	Cr	Mo	Cu	V
Plate No. 1*- (Vendor)	0.20	1.29	0.013	0.022	0.25	0.50	-	0.49	0.14	-
(NRL)	0.27	1.20	0.008	0.015	0.24	0.50	0.08	0.48	0.12	0.02
Plate No. 2*- (Vendor)	0.21	1.24	0.011	0.020	0.24	0.53	-	0.48	0.11	-
Weld Metal (NRL)	0.17	1.25	0.015	0.011	0.32	0.11	0.16	0.53	0.22	0.03

B. Heat Treatment

Plate: Austenitized 1550 to 1650 °F, 4 hr; dip quenched in agitated water; tempered 1225 ± 25 °F, 4 hr; furnace cooled

Post Weld: Stress relief annealed 1150 ± 25 °F, 8 hr; furnace cooled; plus 1115 ± 25 °F, 16 hr; furnace cooled

* Steel Heat A

base plate 2 is noted; however, irradiation responses of plates 1 and 2 were very similar (70 versus 85° transition temperature increase). Suggestive of its higher copper content, the weld deposit illustrated a 200° transition temperature increase (30 ft-lb index) or a 140° increase (40 ft-lb index). In addition, the loss in full shear energy absorption of the weld deposit was much more severe than that of either base plate.

As illustrated in Figure 9.4, the HAZ properties before irradiation were generally superior to those of base plate (and of weld metal). After irradiation, this general relation-

ship appeared to be unchanged. Thus, the HAZ properties are at least as good as base plate properties for this neutron exposure condition.

In review, the assessment of plate, HAZ, and weld metal of one large production weldment of A533B steel revealed the weld metal to be most detrimentally affected by radiation exposure at 550 °F. In line with prior experimental observations (see pp. 9.1-9.4), it is suggested that the relatively high copper content of the weld metal (0.22%) is an important factor in its high radiation embrittlement sensitivity.

FIGURE 9.3. Notch Ductility Characteristics of a 7 1/2 in. A533B Class I Production Weldment Before and After 550 °F Irradiation, Showing the Relative Performance of Base Plate Versus Submerged Arc Weld Deposit

FIGURE 9.4. Comparison of Weld Heat Affected Zone (HAZ) Notch Ductility Properties with Those of Base Plate and Weld Metal Forming a 7 1/2 in. A533B Class I Weldment. (After irradiation at 550 °F, HAZ properties are observed to be as good as plate and superior to weld properties.)

DROP WEIGHT NDT VERSUS CHARPY-V ENERGY
ABSORPTION LEVELS IN 6 3/8 in. TYPE
A533B CLASS I AND II STEEL PLATE

A correlation between the drop weight NDT and Charpy-V energy absorption level of 6 3/8 in. Type A533B Class I and II steel plates has been made to examine the Charpy-V energy index for this type of steel. The composition and heat treatment for the plate investigated is given in Table 9.7. Standard Charpy-V (longitudinal orientation) and Type P-3 drop weight specimens were taken from the top surface, 1/4, 1/2, and 3/4 thicknesses, and bottom surface locations of the Class I plate and from 1/4 thickness location only of the re-tempered Class I plate. The test results are summarized in Table 9.8.

The most significant conclusion to be drawn from these data are that the Charpy-V 30 ft-lb energy absorption level, commonly used as an index of the NDT of A302B pressure vessel steel, can not be applied to this

TABLE 9.7. Chemical Composition and Heat Treatment of 6 3/8 in. Type A533B Class II Steel Plate

Carbon	0.24
Manganese	1.27
Phosphorus	0.008
Sulfur	0.015
Silicon	0.19
Nickel	0.53
Chromium	0.14
Molybdenum	0.48
Aluminum	0.029
Copper	0.09

Heat Treatment - Class II Plate*:

Double austenitizing, water quenching, and tempering, as follows:

1670 to 1685 °F, 6 1/2 hr,
 water dip quench;
 1650 to 1660 °F, 6 1/2 hr,
 water dip quench;
 1115 to 1125 °F, 6 1/2 hr,
 air cool.

* Class I material was obtained by subjecting a section of the Class II plate to an additional 6 1/2 hr tempering treatment at 1225 °F.

TABLE 9.8. Drop Weight and Charpy-V Notch Ductility Characteristics of a 6 3/8 in. A533B Plate

Specimen Location	Charpy-V 30 ft-lb Trans. Temp., °F	Approx. Full Shear Energy Absorption, ft-lb	Drop Weight NDT, °F	Average Charpy-V Absorption at NDT, ft-lb
<u>Class II Material</u>				
Top Surface	-120	113	-40	60
1/4 Thickness	-75	120	+20	85
1/2 Thickness	-60	102	0	60
3/4 Thickness	-75	120	+20	85
Bottom Surface	-110	113	-40	60
<u>Class I Material</u>				
1/4 Thickness	-50	138	-20	60

A533 Class I or II steel plate. Additional studies are being conducted to determine the drop weight NDT/Charpy-V energy correlation for several heats of this material in order to ascertain the most appropriate Charpy energy index for NDT assessment of A533B steel.

MECHANICAL PROPERTIES EVALUATION OF PM-2A REACTOR PRESSURE VESSEL STEEL

A previous progress report⁽⁴⁾ gave details of the sampling of the PM-2A reactor pressure vessel for metallurgical test specimens and also the Charpy-V notch ductility results. Evaluations are now complete on post-irradiation annealing of Charpy-V specimens for comparison with matching annealing schedules conducted on test reactor irradiated specimens of the PM-2A sister forging. Tension test specimen evaluations have been completed at two temperatures, +72 °F and -20 °F, representing the acid sharpening treatment and failure temperatures, respectively. A calculated neutron-spectrum analysis of the PM-2A reactor vessel wall was performed and is discussed. The mechanical properties developed have been evaluated through a failure analysis using the generalized fracture analysis diagram (FAD) concept.

Notch Ductility Characteristics

The Charpy-V specimens not broken for development of the as-irradiated condition of the PM-2A vessel steel were postirradiation heat treated at 650 °F for 168 hr. The results are

shown in Figure 9.5 and listed in Table 9.9. Since the recovery of preirradiation properties was small for this 650 °F heat treatment condition, the remaining specimens were subjected to an additional 168 hr at 750 °F. As can be seen, this combination of heat treatments resulted in very close correspondence with the unirradiated condition band for the sister forging Charpy-V data. For comparison purposes, a test reactor irradiation of sister forging specimens was conducted at 510 °F. Results showed similar recoveries of about 25 to 30% of the unirradiated-condition properties for a 168 hr, 650 °F heat treatment and, essentially, 100% for the additional 168 hr, 750 °F anneal. These recovery data also compare well with

TABLE 9.9. PM-2A Reactor Pressure Vessel Wall, 2.4 in. A350-LF3 Ring Forging - Postirradiation Annealed Condition

Longitudinal Specimens Location	Test Temperature, °F	Energy, ft-lb	Shear, %
650 °F, 168 hr			
1/4 t	50	34	30
	120	51	70
	100	45	40
3/4 t	50	50	40
	80	59	65
	120	83	85
	20	44	40
	0	22	25
Reannealed additional 750 °F, 168 hr			
1/4 t	-80	27	15
	-40	43	40
	120	131	100
	-60	34	25
	-100	16	10
	0	86	70

FIGURE 9.5. Notch Ductility Characteristics of PM-2A Reactor Ring Forging Pressure Vessel Steel After Postirradiation Heat Treatment. (Closed circles and squares represent specimens heat treated for 168 hr at 650 °F; recovery of unirradiated-condition properties relative to the sister forging are between 25 and 35%. Open circles indicate specimens re-heat treated for an additional 168 hr at 750 °F; recovery of essentially 100% of the sister forging unirradiated-condition properties is indicated.)

other postirradiation heat treatment data developed from evaluations of the same sister forging.⁽⁵⁾ Thus, in the absence of preirradiation Charpy-V values for the PM-2A Vessel steel, these results give confidence to the use of -80 °F as the initial 30 ft-lb value.

Tensile Properties

Longitudinally oriented tensile specimens, with gage diameters 0.2520 in., from both the outer and inner radius of the PM-2A vessel steel were evaluated at the -20 °F failure temperature and at the +72 °F temperature imposed during acid

sharpening of the artificial defect. The results are summarized in Table 9.10.

The general test behavior for the irradiated, PM-2A tensile specimens consisted of an upper yield point rapidly falling to the lower yield point (0.2% offset) level which was quite well defined in each case. A comparison of the reduction in area and elongation values of the unirradiated versus the irradiated condition reveals that neither were significantly affected by the irradiation. On the other hand, both the yield and tensile strengths increased markedly with neutron exposure, the increases being generally

TABLE 9.10. PM-2A Reactor Pressure Vessel Wall, 2.4 in.
A350-LF3 Ring Forging Irradiated-Condition Tensile Properties

Specimen Location in Vessel	Fluence, n/cm^2 ($E > 1$ MeV)	Temperature, $^{\circ}F$	0.2% Yield Point, psi	Tensile Strength, psi	Uniform Elongation, %	Reduction of Area, %	Elongation in 1-in., %	Hardness, R_B
Not Irradiated*	---	+75	58,600	82,200	**	68.6	33.0	**
1/4 t	7.7×10^{18}	-20	97,620	111,700	7.0	61.7	**	96
3/4 t	4.2×10^{18}	-20	90,450	106,100	10.1	67.3	29.8	94.5
1/4 t	7.7×10^{18}	+72	92,200	105,400	6.2	60.7	24.8	96
3/4 t	4.2×10^{18}	+72	84,200	99,060	7.8	65.5	28.2	94

* Average of 6 specimens from a sister ring forging
 ** Not determined

in line with irradiation data available for the sister forging.⁽⁶⁾ An overall assessment of the tensile properties, then, would suggest that the vessel steel was not significantly damaged with respect to its ability to accept and accommodate the test loading stresses and, in fact, the increased yield strength may have enhanced its ability to accommodate these stresses.

Neutron Dosimetry and Spectrum Analysis

The mechanical properties data are referenced to neutron fluences in terms of n/cm^2 ($E > 1$ MeV), assuming a fission spectrum distribution of neutrons and a fission-spectrum-average ^{54}Mn activation cross section of 68 mb. Since neutron activation detectors or wires were not available for analysis of the vessel lifetime flux and fluence, ^{54}Mn induced into the iron matrix of the A350-LF3 steel forging was employed for the fast neutron flux determinations, and ^{60}Co induced into the alloyed cobalt was

employed for 2200 m/sec thermal neutron flux. For the inner radius (1/4T) specimen location, the thermal neutron flux was about twice that of the flux >1 MeV; for the outer radius (3/4T) location, the thermal flux was about 80% of the flux >1 MeV. The sample steel used for dosimetry purposes, 95.98% Fe and 0.010% Co, was irradiated for a total of 6.3×10^7 sec at average power level of 4.77 MW, ending July 9, 1963.

The highest neutron flux and fluence value measured from drillings came from the region of the artificial defect adjacent to but not in the stainless steel cladding. Based upon the above noted fission spectrum parameters and assumptions, the flux was 1.59×10^{11} n/cm^2 /sec, and the fluence at this point was 1.0×10^{19} n/cm^2 . Drillings were taken at ten locations across and around the periphery of the slab of steel from which the four sections were taken. The material analyzed was between 1/4 to 1/2 in. from the exterior wall of the vessel. The fluences varied by $\pm 13\%$ from the average, indicating that the

total 11 in. height and 24 in. length of the stock material for the metallurgical specimens received an exposure to neutrons more uniform than that achieved in many experimental irradiations.

The distribution of neutrons as a function of energy levels has been calculated for several locations in the PM-2A reactor by Ulseth, Yoshikawa, and co-workers at Battelle-Northwest using a transport theory Sn code, Program S.⁽⁷⁾ A listing of arbitrary unit fluxes by energy and lethargy groups for the interface of the pressure vessel wall and stainless steel cladding (PVW/SSClad), the 1/4 thickness location (1/2 in. from the PVW/SSClad), and the 3/4 thickness location (1.75 in. from PVW/SSClad) are given in Table 9.11 and plotted as histograms in Figure 9.6. The 1/4 thickness spectrum has been omitted for best visibility since it lies so close to the other spectra. Also, the trend of the peak flux, occurring at progressively lower energies from the PVW/SSClad to the 3/4 thickness, is maintained by the 1/4 thickness spectrum.

Calculation of the flux spectrum of the PM-2A permits determination of spectral averaged cross section, $\bar{\sigma}$, for iron activation which will yield fluences of neutrons of energies greater than a selected lower energy limit, E_L , such as 1 or 0.5 MeV. The spectral averaged cross sections for iron activation at the three locations calculated are given in Table 9.11. It is pointed out that Helm's 82.6 mb fission spectrum cross section,⁽⁸⁾ rather than the 68 mb fission spectrum

cross section, has been used as a base for determining the calculated spectrum cross sections. Accordingly, Table 9.12 is a listing of 68 mb based fission spectrum and adjusted (82.6 mb based) calculated spectrum fluence values for neutrons >1 MeV and >0.5 MeV for the PM-2A notch ductility values.⁽⁴⁾ Included in the table are fluences corresponding to notch ductility values determined from irradiations of the PM-2A sister forging in test reactors.⁽⁹⁾

FIGURE 9.6. Graphical Representation and Relative Intensities of the Neutron Spectra in the PM-2A Reactor at the Interface of the Pressure Vessel Wall/Stainless Steel Cladding and at the 3/4 Thickness Position Located 1.75 in. into the Vessel Wall (from inside to outside)

TABLE 9.11. Calculated Neutron Flux (Arbitrary Units)
per Energy Group for Three Locations in the PM-2A Reactor*

Lethargy, u	Energy Limit (E ₁), eV	Calculated Relative Neutron Flux, n/cm ² -sec			$\bar{\sigma}$ ⁵⁴ Fe(n,p) ⁵⁴ Mn, Barns Ref. 11
		PVW/SS Clad	1/4 t	3/4 t	
		47.6 - 48.31 cm**	49.01 - 49.72 cm**	51.84 - 52.55 cm**	
0.25	7.79 x 10 ⁶	6.951 x 10 ⁻⁷	5.840 x 10 ⁻⁷	3.371 x 10 ⁻⁷	0.450
0.50	6.07 x 10 ⁶	2.236 x 10 ⁻⁶	1.871 x 10 ⁻⁶	1.071 x 10 ⁻⁶	0.495
0.75	4.72 x 10 ⁶	3.685 x 10 ⁻⁶	3.077 x 10 ⁻⁶	1.750 x 10 ⁻⁶	0.450
1.00	3.68 x 10 ⁶	4.025 x 10 ⁻⁶	3.351 x 10 ⁻⁶	1.924 x 10 ⁻⁶	0.295
1.25	2.87 x 10 ⁶	4.383 x 10 ⁻⁶	3.666 x 10 ⁻⁶	2.142 x 10 ⁻⁶	0.195
1.50	2.23 x 10 ⁶	6.450 x 10 ⁻⁶	5.320 x 10 ⁻⁶	3.067 x 10 ⁻⁶	0.110
1.75	1.74 x 10 ⁶	5.405 x 10 ⁻⁶	4.665 x 10 ⁻⁶	2.842 x 10 ⁻⁶	0.015
2.00	1.35 x 10 ⁶	5.278 x 10 ⁻⁶	4.700 x 10 ⁻⁶	3.019 x 10 ⁻⁶	
2.25	1.05 x 10 ⁶	4.649 x 10 ⁻⁶	4.322 x 10 ⁻⁶	2.910 x 10 ⁻⁶	
2.50	8.21 x 10 ⁵	4.491 x 10 ⁻⁶	4.332 x 10 ⁻⁶	3.078 x 10 ⁻⁶	
2.75	6.39 x 10 ⁵	4.739 x 10 ⁻⁶	4.465 x 10 ⁻⁶	3.039 x 10 ⁻⁶	
3.00	4.89 x 10 ⁵	4.279 x 10 ⁻⁶	4.186 x 10 ⁻⁶	3.032 x 10 ⁻⁶	
3.25	3.88 x 10 ⁵	2.442 x 10 ⁻⁶	2.162 x 10 ⁻⁶	1.419 x 10 ⁻⁶	
3.50	3.02 x 10 ⁵	2.658 x 10 ⁻⁶	2.259 x 10 ⁻⁶	1.420 x 10 ⁻⁶	
3.75	2.35 x 10 ⁵	2.333 x 10 ⁻⁶	1.990 x 10 ⁻⁶	1.249 x 10 ⁻⁶	
4.00	1.83 x 10 ⁵	1.984 x 10 ⁻⁶	1.549 x 10 ⁻⁶	8.467 x 10 ⁻⁷	
6.75	1.17 x 10 ⁴	1.355 x 10 ⁻⁵	1.130 x 10 ⁻⁵	6.708 x 10 ⁻⁶	
9.00	1.23 x 10 ³	8.162 x 10 ⁻⁶	6.587 x 10 ⁻⁶	3.724 x 10 ⁻⁶	
11.50	1.01 x 10 ²	8.974 x 10 ⁻⁶	6.629 x 10 ⁻⁶	3.122 x 10 ⁻⁶	
16.50	0.683 x 10 ²	2.032 x 10 ⁻⁵	1.510 x 10 ⁻⁵	6.774 x 10 ⁻⁶	
∞	0.0	3.040 x 10 ⁻⁴	2.148 x 10 ⁻⁴	8.685 x 10 ⁻⁵	
φ > 1 MeV		3.770 x 10 ⁻⁵	3.242 x 10 ⁻⁵	1.968 x 10 ⁻⁵	
φ > 0.5 MeV		5.032 x 10 ⁻⁵	4.454 x 10 ⁻⁵	2.821 x 10 ⁻⁵	
$\bar{\sigma}_{CS}$ > 1 MeV		159 mb	154 mb	146 mb	
$\bar{\sigma}_{CS}$ > 0.5 MeV		119 mb	112 mb	102 mb	

* Spectral calculation used 25.59 cm as effective core radius

** Distance from core center to zone represented by dimensional limits

TABLE 9.12. Notch Ductility and Neutron Fluence Data for
Irradiation of PM-2A Reactor Steel

2.4 in. Ring Forging Source	Reactor Location	Temperature, °F	30 ft-lb Transition Temperature, °F	φ _f *	$\bar{\sigma}_{CS}$ **	φ _{CS} **	$\bar{\sigma}_{CS}$	φ _{CS} **
				> 1 MeV	> 1 MeV, mb	> 1 MeV	> 0.5 MeV, mb	> 0.5 MeV
Sister	LITR C-55	490	270	1.4 x 10 ¹⁹	84.9	1.62 x 10 ¹⁹	57.3	2.40 x 10 ¹⁹
Sister	LITR C-18	510	320	2.7 x 10 ¹⁹	96.4	2.75 x 10 ¹⁹	65.2	4.06 x 10 ¹⁹
Sister	LITR C-55	500	365	2.9 x 10 ¹⁹	84.9	3.35 x 10 ¹⁹	57.3	4.96 x 10 ¹⁹
PM-2A	PM-2A 1/4t	510	195	0.77 x 10 ¹⁹	154	0.49 x 10 ¹⁹	112	0.67 x 10 ¹⁹
PM-2A	PM-2A 3/4t	510	135	0.42 x 10 ¹⁹	146	0.28 x 10 ¹⁹	102	0.40 x 10 ¹⁹

* Based upon fission-spectrum-averaged $\bar{\sigma} = 68$ mb

** Based upon fission-spectrum-averaged $\bar{\sigma} = 82.6$ mb

The data of Table 9.12 are plotted in Figure 9.7 as referenced to the NRL trend band for irradiations less than 450 °F. It is apparent and probably quite fortuitous that the open triangles, representing the fission spectrum assumption criterion for determining fluences, describe a satisfactory trend pattern for the range of fluences investigated. This would suggest that estimates of transition temperature increases can be made in terms of a fission spectrum with a reasonable degree of assurance if an acceptable dosimetry measurement can be made.

It should also be apparent, however, that the calculated spectrum fluences for the PM-2A reactor are lower in value than the corresponding fission spectrum fluences. Conversely, the calculated spectrum fluences from the test reactor irradiations are higher than the fission spectrum fluences. Note that the comparison shown in Figure 9.7 is from the irradiation of two very similar forgings of the same steel heat at essentially the same temperature. This suggests that the difference in the neutron flux spectra of the PM-2A and the test reactor lie at the base of the disagreement.

It would appear, on the surface, that more embrittlement is seen for a given fluence of neutrons of energies greater than 1 MeV in a power reactor than in a test reactor. Since this is contradictory to nature, it follows that the concept of equal weight given to neutrons of all energies >1 MeV will have to be replaced by one which gives more or less weight to neutrons depending upon their energy.

Discussion

Figure 9.8 shows that the remaining ligament of A350-LF3 steel adjacent to the artificial defect in the PM-2A had Charpy-V 30 ft-lb transition temperatures of about +115 °F and higher. Thus, the +72 °F acid sharpening treatments and the -20 °F failure^(10,11) clearly were accomplished at temperatures below NDT, the region for determination of valid linear elastic fracture mechanics (K_{IC}) values for crack stress intensity analyses. At this time, however, K_{IC} values for the PM-2A vessel steel have not been determined. For the interim, it may be of interest to propose a simplified analysis of the PM-2A failure in terms of the mechanical properties herein developed using the fracture analysis diagram (FAD) procedures of Pellini and Puzak.^(12,13)

The temperature, stress, and flaw size are three factors upon which the FAD is based. The previous paragraph indicates both the acid sharpening treatment and failure events occurred below NDT temperature. (It is to be remembered at this point that actual NDT values have not yet been developed and that Charpy-V 30 ft-lb values are being substituted.)

The vessel hoop stress can be readily determined to a close approximation, assuming the stress in the vicinity of the flaw to be given by the hoop stress, by considering the relationship, $\sigma_h = p r/t$, where p is the applied pressure in psig, r is the vessel radius (neglecting the cladding) to the PVW/SSClad interface, and t is the vessel thickness. The specific pressures of interest are presented in Table 9.13.

FIGURE 9.7. Transition Temperature Increase (Indexed to Charpy-V 30 ft-lb level) for a Sister Ring Forging of the PM-2A Reactor Pressure Vessel Steel Versus Neutron Fluence Determined Assuming a Fission Spectrum (open points) and Calculated Spectra (partially and completely closed points). [The Low Intensity Test Reactor (LITR) data were from irradiations at the temperatures noted using the sister forging as specimen material.]

FIGURE 9.8. Schematic Representation of the Neutron Fluence Gradient and Irradiated-Condition 30 ft-lb Transition Temperatures for Locations Across the Thickness of the PM-2A Reactor Vessel Wall. (The volume of material occupied by the Charpy-V specimens is shown in relation to the artificial defect. The bands on the neutron fluence points indicate the range of material averaged by the measurement.)

TABLE 9.13. Conversion of PM-2A Test Loop Pressure into Vessel Hoop Stresses

Gage Pressure, psig	σ_h , psi	Percentage of 0.2% Yield Strength at Temperature, °F
4000	31,600	34% at 72 °F
4475*	35,400*	36% at -20 °F*
5000	39,500	43% at 72 °F

* Failure conditions; other conditions represent tests

The flaw, being the artificial defect in this case, was found to be quite sharp in the acid-treated lower half of the machined defect. The degree of sharpness approached that of a natural or fatigue crack, and there were also numerous secondary cracks visible in the same region which were also produced by the corrosive, embrittling attack of the acid. (14)

The flaw size at vessel failure can be seen to compare favorably with the 1/4 to 1/2 yield stress range which is predicted by the FAD (Table 9.13). Thus, the combination of a large sharpened flaw, a pressurizing situation, and a temperature below NDT were, as would be predicted, sufficient to trigger the running crack which fractured the vessel. Referring to the FAD, the failure occurred at a stress level wherein flaws on the order of one foot long could be expected to effect fracture. Clearly, a flaw of only several inches would not be expected to fracture the vessel based upon the material properties developed and presented in this report. In essence, the conditions at failure (although not perfectly known) were in general accordance with those projected by the FAD.

Conclusions

1. The steel in the pressure vessel wall of the PM-2A reactor was significantly embrittled by neutron irradiation, while the tensile properties showed only slight deleterious changes coupled with marked strength increases.

2. The increase in the ductile-brittle transition temperature of steel located throughout the vessel wall compares well with increases developed from test reactor irradiations when all data are plotted using an assumed fission spectrum, neutron distribution.
3. Neutron dosimetry data from locations across the thickness of the vessel wall present a linear relationship when plotted on a semi-log scale. It should, therefore, be possible to accurately estimate through-thickness embrittlement and neutron dosimetry in reactor pressure vessels provided a valid dosimetry value can be obtained from the vessel itself or from surveillance data.
4. Greater stresses were required to fracture the vessel than would be encountered during normal service. Even with this higher stress, a very large flaw was required also to effect the PM-2A fracture.
5. The conditions for failure for the PM-2A reactor vessel are in agreement with the generalized fracture analysis diagram, using the irradiated-condition yield stress as the reference rather than the unirradiated condition yield stress.
6. Documentation of the actual irradiated-condition mechanical properties of a reactor pressure vessel, which compare favorably with trends developed in experimental test reactors, affords proof that these values rather than the unirradiated condition properties should be utilized in

assessing the safety margins available for each individual reactor situation.

7. The PM-2A reactor fracture experience suggest increased confidence in the overall safety of reactors.

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RADIATION EFFECTS ON FAST REACTOR CLADDING AND STRUCTURAL MATERIALS

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The objective of the GE-NMPO radiation effects to metals program is to determine the effect of radiation on the time-, temperature-, and stress-dependent properties of selected high-temperature alloys and refractory metals, to identify the causes of any observed changes in these properties, and to develop remedial measures.

The effects of irradiation (EBR-II, 0.27 and 2.7×10^{20} n/cm², $E > 1$ MeV at about 540°C) on the postirradiation creep-rupture properties of Incoloy 800 at 540 and 704°C are presented. Multiple stress tests in which one specimen is used to generate a stress-creep rate curve is discussed.

Preliminary results on the influence of low neutron fluence (3×10^{18} n/cm², $E > 1$ MeV) irradiations at reactor ambient temperature on the 650°C plastic fatigue properties of Type 316 SS are presented.

CREEP-RUPTURE PROPERTIES OF INCOLOY 800*

J. P. Smith and R. A. Joseph

Specimens of Incoloy 800 which were irradiated at approximately

*Specimens were fabricated and irradiated by GE-APO and represent material which was used in the tensile testing program as reported by H. J. Busboom, F. A. Comprelli, K. P. Franke, and C. N. Spalaris (GEAP-4812) and by H. J. Busboom and P. W. Mathay (GEAP-4985).

540°C in EBR-II to fast neutron fluences of 0.27 and 2.7×10^{20} n/cm² ($E_n \geq 1$ MeV) were creep rupture tested at 540 and 705°C . The stress versus creep rate and stress versus rupture life data are summarized in the graphs of Figures 10.1 and 10.2, respectively. Although radiation affects the creep properties of this material at both test temperatures, the effects are somewhat different at the two temperatures. At 540°C , the minimum creep rate ($\dot{\epsilon}$) is increased and time to rupture (t_R) is reduced compared to the unirradiated material. On the other hand, at a test temperature of 705°C , $\dot{\epsilon}$ is reduced and t_R is increased (except at high stresses) compared to the corresponding unirradiated data. At both test temperatures, radiation reduced the fracture elongation by about a factor of 3 to 4, i.e., from an unirradiated elongation of approximately 55% to postirradiation elongation of 16% at 540°C ; at 705°C , from unirradiated elongation of 75 to 122% to postirradiation elongations in the 21 to 49% range.

Another significant difference in creep-rupture properties with test temperature is that for the 540°C tests the irradiation and control data tend to converge at low stresses; on the other hand, at a test temperature of 705°C there is a definite tendency

FIGURE 10.1. Minimum Creep Rate and Stress-Rupture Strength for Unirradiated and Irradiated Incoloy 800 at 540 °C

FIGURE 10.2. Minimum Creep Rate and Stress-Rupture Strength for Unirradiated and Irradiated Incoloy 800 at 705 °C

for convergence of the irradiated and control data at high stresses. This convergence at high stress is quite unique when compared to similar tests on other alloys investigated at NMPO. Generally, if there is any convergence it occurs at low stress (long time) tests, as appears to be the case at 540 °C (Figure 10.2b), which possibly indicates annealing of point defects.

Examination of the creep curves for both the 540 and 705 °C as shown in Figure 10.3 shows that, although the

second stage creep rate is accelerated in one case and reduced at the higher temperature, at both temperatures irradiation reduced third stage ductility (i.e., duration of third stage creep). The net effect is a reduction in fracture ductility at both temperatures.

Actual reactor operations do not experience the constant load condition as described by the above tests; instead, the stress will vary over a rather wide range. In order 1) to

FIGURE 10.3. Creep Curves for Irradiated and Unirradiated Incoloy 800 at 540 and 705 °C

investigate the effects of varying applied stress and 2) to determine accurately how radiation affects the low strain part of the creep curve, tests were performed where the stress on a given specimen was varied and the strain was measured accurately with a cathetometer sighting directly on the gage length of the specimen. The low strain range is of particular interest to designers since many fuel-element clad designs allow for 3% maximum strain. The procedure was to load a specimen to σ_1 then allow the test to proceed long enough to establish linear creep; the load was then increased to give σ_2 and again a

linear creep rate was established. This was repeated for 3 stress levels. Finally, the load was reduced to give σ_1 for comparison with the original creep rate at σ_1 and to determine if the substructure had been altered. This experiment was performed on irradiated and control specimens. The results are shown in Figures 10.4 and 10.5. For the unirradiated specimen (Figure 10.4), the minimum creep rate is apparently a function of prior stress or strain history, based on the fact that second-stage creep rate ($\dot{\epsilon}_s$) for σ_1 final is significantly higher than for σ_1 initial. In the case of the irradiated specimen (Figure 10.5),

FIGURE 10.4. Instantaneous Creep Rate at 704 °C of Unirradiated Incoloy 800 as a Function of Creep Time for Various Stress Levels

FIGURE 10.5. Instantaneous Creep Rate at 704 °C of Irradiated ($2.4 \times 10^{19} \text{ n/cm}^2$, $E_n \geq 1 \text{ MeV}$) of Incoloy 800 as a Function of Creep Time for Various Stress Levels

$\dot{\epsilon}_s$ is essentially identical for σ_1 initial and final. In the unirradiated results (Figure 10.4), for σ_1 initial, two separate linear portions $\dot{\epsilon}_s$ appear to be present. The exact cause of this is not known; however, it has been observed on post-test metallography of ruptured specimens that recrystallization is probably occurring during test since the grain size near the fracture is very fine and becomes progressively coarser in low-strain areas. This recrystallization could explain the two linear

portions of the initial σ_1 and the decreased creep resistance of the final σ_1 compared to σ_1 initial.

The result on the irradiated specimen indicates that irradiation "freezes in the substructure" and that stresses or strains in the region investigated do not alter this structure. Metallography and transmission electron microscopy will be performed to help evaluate these results. A similar series of tests was initiated at 540 °C. Creep rates, for the unirradiated specimens at the three

lower stress levels of Figure 10.1a were all determined from one specimen; they are in good agreement with tests performed at a constant load to failure.

To more fully evaluate all available test data; correlations are being made between creep and tensile data. The tensile data discussed here was generated at GE-APO⁽¹⁾ on samples from the same heat and irradiation as for the specimens described above.

The first correlation is shown in Figure 10.6. Note that there appears to be a good correlation of the tensile and creep data both for strength and ductility. Note in particular, 1) the effect of fluence on the ductility but not on the strength and 2) the minimum in the ductility curve for the irradiated specimens. Weir⁽²⁾ has reported such a minimum for Hastelloy N. Another approach to correlating the tensile and creep data

FIGURE 10.6. Strain-Rate and Elongation as a Function of True Stress for Incoloy 800 Tested at 705 °C. To Minimize Structure Differences Due to Work-Hardening, the True Flow Stress at About 6% True Strain Was Used to Correlate the Tensile Data with the Creep Data.

which is somewhat unique is the application of the Monkman-Grant relationship⁽³⁾ ($\log \dot{\epsilon}$ versus $\log t_R$) as shown in Figure 10.7. At both temperatures, the data fall on lines close to the predicted slope of -1. Even more interesting is that the available tensile data fits the curve quite well. The effective t_R for the tensile data was obtained by calculating $\epsilon/\dot{\epsilon}$ for each test where ϵ is the fracture strain. Refinements can probably be made (such as using uniform strain); however, the above technique appears to work quite well to a first approximation. Since the slope is unity, then $\dot{\epsilon} \times t_R = \text{constant}$. Although the units of this constant

are strain, its physical significance is not known; however, it may be used as a guide in predicting the minimum fracture strain since what it represents is the strain at failure one would obtain if no third stage creep occurred. Since most unirradiated materials do exhibit a third stage creep, the fracture ductility is generally somewhat higher than predicted by the Monkman-Grant relationship. On the other hand, many irradiated materials do exhibit little or no third stage creep and this relationship may be useful in predicting quite closely this fracture ductility.

FIGURE 10.7. Monkman-Grant Relationship for Irradiated and Unirradiated Incoloy 800 at 540 and 705 °C Using Tensile and Creep Data

LOW CYCLE FATIGUE PROPERTIES -

J. T. Berling and J. P. Smith

Preliminary tests have been performed on irradiated Type 316 SS to determine the effects of irradiation on low cycle plastic fatigue properties at 650 °C. The specimens were of the hour glass design being used in Job 81516 and were irradiated in the ORR at reactor ambient temperature to 2.35×10^{19} (thermal) and 3.06×10^{18} ($E_n \geq 1$ MeV). Two irradiated specimens have been tested to date and the results were quite consistent. Both tests were performed at a constant strain rate of $4 \times 10^{-5} \text{ sec}^{-1}$ at 650 °C. One test was performed with a strain range ($\Delta\epsilon$) of 2%; the

other was tested with a $\Delta\epsilon = 1\%$. The results of the tests performed to date are summarized in Table 10.1; the load range ($\Delta\sigma$) as a function of test time (number of cycles) for the $\Delta\epsilon = 2\%$ test is shown in Figure 10.8.

TABLE 10.1. Low-Cycle Plastic Fatigue Properties of Type 316 SS at 650 °C

Condition	$\dot{\epsilon}$, sec^{-1}	$\Delta\epsilon$, %	N_0 , Cycles	N_5 , Cycles	N_f , Cycles
Control	4×10^{-5} *	2	70	139	153
Irradiated**	4×10^{-5} *	2	27	39	44
Irradiated**	4×10^{-5} †	1	138	168	182
Control	4×10^{-5} †	1		562	611

* 3.6 cycles/hr

** ORR at reactor ambient temperature

$\phi = 2.35 \times 10^{19} \text{ n/cm}^2$ (thermal)

$\phi = 3.06 \times 10^{18} \text{ n/cm}^2$ ($E > 1 \text{ MeV}$)

† 7.2 cycles 1 hr

FIGURE 10.8. Load Range as a Function of Fatigue Life at 650 °C for Irradiated and Unirradiated 316 SS

Analysis of the data in Table 10.1 and Figure 10.8 leads to the following conclusions:

1. Stress range is unaffected by irradiation.
2. Based on a comparison of N_0 in the control and irradiated, a macro-crack is formed earlier by a factor of 2.6 in the irradiated specimen.
3. Based on a comparison of N_0/N_f control to N_0/N irradiated, the crack propagation rate is faster for the irradiated specimen.
4. Using N_5 as the criterion for fatigue life, the effect of irradiation is to reduce the fatigue resistance by a factor of approximately 3.6.

Analysis of the hysteresis loops also showed that irradiation apparently does not effect Poisson's Ratio or the elastic modulus. Further tests will continue, including constant strain rate tensile testing.

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RADIATION EFFECTS IN FUEL CLADDING MATERIALS

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DEVELOPMENT OF TITANIUM-MODIFIED TYPES 304 AND 304L SS

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Evaluation of the mechanical properties of two 1500 lb vacuum-induction melted heats of titanium-modified Types 304 and 304L SS is continuing. The composition of these two alloys is given in Table 11.1.

Table 11.2 compares the as-annealed and postirradiation creep-rupture properties of the two alloys at a test temperature of 704 °C. In each case, the material was cold-worked 50% prior to machining test specimens.

TABLE 11.1. Composition of Titanium-Modified Type 304 and 304L SS

Heat No.	3756	60508
Element, wt%		
Carbon	0.012	0.05
Chromium	19.5	19.0
Nickel	11.2	10.0
Titanium	0.19	0.21
Manganese	1.0	0.65
Silicon	0.05	0.8
Phosphorus	0.006	0.013
Sulfur	0.013	0.002
Boron	0.0003	0.0027
Nitrogen		
Total	0.006	0.009
Soluble	--	0.003

FIGURE 11.2. Creep-Rupture Properties of Titanium-Modified Type 304 SS and 304L SS at 704 °C

Alloy	Stress, psi	Unirradiated			Irradiated [†]		
		Rupture Life, hr	Creep Rate, %/hr	Total Elongation, %	Rupture Life, hr	Creep Rate, %/hr	Total Elongation, %
304L + 0.2 wt% Ti, Heat 3756*	20,000	8.2	3.6	64.0	--	--	--
	17,500	20.4	1.9	75.9	--	--	--
	15,000	106.6	0.20	56.9	42.5(59.2)	0.63(0.64)	38.7(63.0)
	12,500	372.7	0.11	53.2	--	--	--
	11,200	366.1	0.08	60.5	--	--	--
	10,000	1084	0.016	26.8	327.7(738.2)	0.083(0.044)	45.4(51.1)
304 + 0.2 wt% Ti, Heat 60508*	8,000	4110	0.0038	20.8	688.0(1460)	0.023(0.02)	53.7(66.2)
	22,500	8.1	4.7	77.2	--	--	--
	20,000	13.1	2.0	48.6	12.6	0.72	12.3
	17,500	--	--	--	23.9	0.40	12.1
	15,000	297.1	0.030	47.6	182.1	0.034	10.5
	13,500	--	--	--	220.3	0.030	9.7
	12,000	--	--	--	808.1	--	12.3
	10,000	1520.8	0.0037	20.4	--	--	--

* Irradiated for 2000 hr at 650 °C approximately 9×10^{20} n/cm² (thermal) and 7×10^{20} n/cm² ($E > 1$ MeV)

** Irradiated for 1000 hr at 650 °C approximately 2.6×10^{20} n/cm² (thermal) and 2×10^{19} n/cm² ($E > 2.9$ MeV)

† Values in parenthesis are for unirradiated thermal controls

The pretest or preirradiation anneal was 1 hr at 925 °C. Each alloy was irradiated at 650 °C, but in separate irradiation experiments; thus, the neutron fluences are different and a direct comparison of properties is difficult. In the as-annealed condition the strength and ductility properties are approximately the same for the two carbon levels. For the low-carbon alloy there is a decrease in rupture life and an increase in creep rate as a result of irradiation. Results of tests on unirradiated thermal control specimens indicate that the increased creep rate and, thus, some part of the reduced rupture life is a result of thermal aging. Ductility values for the irradiated specimens increase as the stress is reduced and for tests at 10,000 and 8,000 psi the values are significantly higher than the as-annealed specimens and slightly lower than the thermal controls.

Preliminary results on the high-carbon alloy indicate that the thermal aging effect is possibly somewhat less in that creep rates of annealed and irradiated specimens are approximately the same. It should be noted that the irradiation time for these specimens was 1000 hr as compared to 2000 hr for the low-carbon specimens. A reduction in ductility is observed as a result of irradiation. However, ductility values are essentially independent of stress (or rupture life) for the conditions investigated to date.

EFFECT OF HEAT TREATMENT
ON THE CREEP-RUPTURE PROPERTIES
OF TYPES 304 AND 0.2% Ti-304L SS

R. T. King

A number of Types 304 and 0.2% Ti-304L SS specimens have been cyclotron bombarded to a uniform concentration of 2×10^{-5} atom fractions of helium.

The compositions of the steels are given in Table 11.3. Both steels were finish cold-rolled 50% to 0.010 in. thick sheet material. Tensile and creep specimens with their gage length parallel to the rolling direction were machined having an 0.25 in. gage width and an 0.75 in. gage length. Specimens of each material were then annealed for 1 hr in argon at either 925 or 1038 °C.

*TABLE 11.3. Compositions
for Types 304 and 304L-0.2% Ti SS*

<u>Element</u>	<u>Type 304 Heat 71403</u>	<u>0.2% Ti-304L Heat 3756</u>
Carbon	0.046	0.019
Manganese	1.77	1.05
Phosphorus	0.023	0.006
Sulfur	0.010	0.017
Silicon	0.62	0.046
Chromium	18.15	19.23
Nickel	9.60	11.22
Copper	0.25	---
Titanium	--	0.22
Molybdenum	0.33	---
Cobalt	0.03	---

We are currently testing the non-bombarded control specimens at 500, 600, and 700 °C. The series of tests run at 700 °C is reported below. All creep-rupture tests were performed under an argon atmosphere of approximately 10 psig. Tables 11.4 and 11.5 list the time to rupture, minimum creep rate, and fracture elongation for the Types 304 and 0.2%-304L SS, respectively. Stresses ranged from 14,000 to 20,000 psi.

The results for the Type 304 SS indicate that over the range of stresses investigated, annealing for 1 hr at 925 °C produces shorter rupture life, higher fracture elongation, and higher minimum creep rate at any given stress than annealing for 1 hr at 1038 °C. Annealing the Type 0.2% Ti-304L SS for 1 hr at 925 °C produces higher fraction elongation than annealing for 1 hr at 1038 °C, but the rupture life and minimum creep rate parameters show no marked differences over the stresses tested.

TABLE 11.4. Creep-Rupture Properties of Type 304 SS

Heat Treatment	Stress, psi	Time to Rupture, hr	Minimum Creep Rate, %/hr	Fracture Elongation, %
1 hr at 925 °C	14,000	142.6	0.12	50.0
	16,000	72.3	0.37	72.3
	18,000	43.2	0.76	56.2
	20,000	27.6	1.22	69.3
1 hr at 1038 °C	14,000	329.0	0.042	27.1
	15,000	255.0	0.058	27.1
	15,000	262.5	0.066	23.0
	16,000	181.2	0.13	39.6
	17,000	139.3	0.074	25.0
	17,000	76.2	0.13	27.1
	18,000	59.7	0.28	31.2
	20,000	36.2	0.74	39.6

TABLE 11.5. Creep-Rupture Properties of 0.2 Ti-Modified Type 304L SS

Heat Treatment	Stress, psi	Time to Rupture, hr	Minimum Creep Rate, %/hr	Fracture Elongation, %
1 hr at 925 °C	14,000	52.9	0.59	75.0
	16,000	29.3	1.26	75.0
	18,000	13.1	2.22	79.2
	20,000	7.2	5.55	83.3
1 hr at 1038 °C	16,000	42.7	0.77	50.0
	18,000	11.35	4.5	56.2
	20,000	5.1	9.2	56.2

DRAWING OF STAINLESS STEEL TUBING

G. A. Reimann

Work has been in progress to produce tubing from the titanium-modified low-carbon grade of Type 304 SS. Ingots were obtained from Allvac Metals Company (Heat 3756), and the analysis of the heat is: 0.019% carbon, 0.017% sulfur, 1.05% manganese, 19.23% chromium, 11.22% nickel, 0.22% titanium, balance iron and 27 ppm N₂.

The ingot structure exhibits two phases (Figure 11.1) plus a distribution of cubic crystals of titanium nitride. The second phase can be dissolved in the matrix by a 12 hr, 1200 °C heat treatment but the nitride particles are essentially unaffected by this treatment (Figure 11.2). Microprobe analysis of the second phase showed concentrations of 32% chromium, 2% nickel, 0.3 to 10.0% titanium, and balance iron. The presence of this phase would make tubing fabrication needlessly difficult and it was dissolved by heat treatment.

Neg Y-86355

100X

Neg Y-86356

1000X

FIGURE 11.1. Ingot Microstructure of Type 304L + 0.2 Ti, Heat No. 3756. Interdendritic phase is 30% Chromium, up to 10% titanium, balance iron, and cubic crystals are TiN_2 .

Neg Y-86357

100X

Neg Y-86358

1000X

FIGURE 11.2. Interdendritic Phase in Ingot Structure Is Mostly Dissolved by 12 hr Treatment at 1200 °C, But Cubic Crystals Are Essentially Unaffected

To convert the Allvac ingots into a configuration suitable for tubing manufacture, an ingot section was forged at 1200 °C into a 4 in. round, machined into a billet, and extruded at 1200 °C into a tube shell. After straightening and machining the outside and inside diameter to ensure concentricity, the tube shells were fabricated into tubing by swaging and drawing. Final sizes are as follows: 0.375 in. OD x 0.345 in. ID; 0.345 in. OD x 0.315 in. ID; and 0.250 in. OD x 0.218 in. ID.

Three separate approaches were investigated to determine their suitability as a fabrication technique for this grade of stainless steel: a) plug drawing, b) mandrel swaging, and c) mandrel or rod drawing.

Plug Drawing

Plug drawing was unsatisfactory for tube shell reduction, as severe circumferential cracks developed in the internal surface after ten plug draw passes (75% total reduction). It appears that the internal flaws were related to lubrication deficiency, and it is believed that the problem could be avoided by installing a lubrication system to pump chlorinated lubricating oil into the tube. The tube interior may have been contaminated by lubricant residue during anneals and, until this possibility is eliminated, the expense of a new lubrication system may not be justified.

Mandrel Swaging

Attempts to mandrel swage the tubing to size was more successful than plug drawing but even with 15% reductions between anneals, longitudinal cracks developed in the internal tube surface after 20 passes. Flaws of this nature developed from wrinkles caused by the swaging action and these flaws were subsequently ironed out as the internal surface of the tube deformed against the hard mandrel.

Mandrel Drawing

The final tube sizes were obtained without undue difficulty by mandrel drawing; nondestructive testing did not disclose any flaws of the type described previously. Satisfactory tubing was produced in 28 to 32 passes of 15% reduction each. Tubing annealed for 20 min at 1200 °C between each pass did not appear to differ from tubing annealed after two 15% passes (~27% total reduction). The flowsheet in Figure 11.3 is based on the mandrel drawing approach and the four swaging passes were done only to reduce the tube cross section to be within the capacity of the drawbench.

The microstructure of the finished tubing is shown in Figure 11.4. The nitride precipitates can be seen at 1000X magnification and they appear to have been fractured into smaller

Neg 68-1982

FIGURE 11.3. Flowsheet Showing Mandrel Drawing Approach

Neg Y-86359

100X

Neg Y-86360

1000X

FIGURE 11.4. Microstructure of Longitudinal Section of Finished 0.250 in. OD $\times$ 0.016 in. Wall Tube. Except for crushed nitride crystals, no second phase is evident.

particles and strung out in the direction of working. The matrix must flow around these particles during deformation and internal fissures may develop when the material is cold-worked excessively.

Although nondestructive testing results are incomplete at this time, it appears that remaining problems in drawing can be solved by improvements in the drawbench technique.

EFFECTS OF NEUTRON IRRADIATION
ON THE ELEVATED-TEMPERATURE
MECHANICAL PROPERTIES OF TYPES
316 AND 316 + TITANIUM SS

E. E. Bloom and J. R. Weir, Jr.

An investigation of the effects of neutron irradiation on the mechanical properties of Types 316 and 316 + Ti SS is continuing. We have previously reported results for standard Type 316 SS (composition 0.068% carbon, 17.2% chromium, 12.5% nickel, 1.92% manganese, 0.51% silicon, 0.004% sulfur, 0.039% phosphorus, 2.06% molybdenum, and 0.0005% boron) irradiated at temperatures of 550 and 750°C to neutron fluences of approximately 9×10^{20} n/cm² (thermal) and 7×10^{20} n/cm² (E > 1 MeV). It was shown that for test temperatures of 550, 650, and 750 °C, irradiation caused a significant decrease in the tensile ductility and that this decrease was independent of irradiation temperature in the range of 550 to 750 °C. For annealing temperatures of 950, 1050, and 1150 °C and for test temperatures of 650 and 750 °C, maximum tensile ductility for both un-irradiated and irradiated specimens

(irradiated at 750 °C) was obtained by annealing 1 hr at 1050 °C.⁽¹⁾

A summary of the 650 °C creep-rupture data on standard Type 316 SS is given in Table 11.6. Although the data available at this time are limited, several trends seem evident. Neutron irradiation causes a reduction in rupture life and total elongation to fracture of the alloy. In the range of 550 to 750 °C, these changes in properties are dependent on irradiation temperature. The ductility of specimens irradiated at 550 °C is significantly lower than that of specimens irradiated at 750 °C, yet with regard to rupture life the opposite is true. Creep rates of as-annealed specimens and those specimens irradiated at 550 °C or aged for 2000 hr at 550 °C are approximately the same, while the creep rate of specimens aged for 2000 hr at 650 °C and those irradiated at 750 °C are significantly higher. These data suggest that changes in properties result both from irradiation and from thermal aging effects.

Additional tests are being conducted on specimens irradiated at 650 °C and on thermal control specimens. Optical and transmission microscopy is also underway.

Specimens from two heats of vacuum-induction melted Type 316 + Ti SS were included in this same irradiation experiment and are presently being evaluated. The composition of these two alloys are given in Table 11.7. Tensile properties of standard Type 316 SS and the two titanium-modified experimental heats after irradiation at 550 and 750 °C are listed in

TABLE 11.6. Creep-Rupture Properties of Type 316 SS at 650 °C

Heat Treatment and/or Irradiation Conditions	Stress, psi	Rupture Life, hr	Creep Rate, %/hr	Total Elongation, %	Reduction Area, %
1 hr at 1050 °C	40,000	28	0.35	36.2	39.2
	35,000	91	0.14	29.6	50.4
	32,500	117	0.029	25.1	44.9
	30,000	180	0.024	54.5	32.9
	27,500	1732	0.0046	31.4	52.6
	25,000	1311	0.0069	30.9	57.8
1 hr at 1050 °C - irradiation	30,000	15	0.085	3.2	9.0
~2000 hr at 550 °C	25,000	252	~0.005	2.7	14.3
	20,000	5127	--	8.7	13.4
1 hr at 1050 °C + 2000 hr	30,000	437	--	33.0	62.0
at 550 °C	25,000	2305	0.0017	29.2	55.2
1 hr at 1050 °C - irradiation	25,000	92	0.12	14.8	17.4
~2000 hr at 750 °C	20,000	137	0.064	9.3	6.7
	20,000	524	0.024	16.7	22.1
1 hr at 1050 °C + 2000 hr	35,000	20	1.13	52.2	71.0
at 650 °C	30,000	146	0.14	37.5	75.2

TABLE 11.7. Composition
of Type 316 + Titanium
Experimental Alloys

Heat Number	3765-3	3765-4
Element, wt%		
Carbon	0.06	0.05
Chromium	17	17
Nickel	12	12
Molybdenum	2.4	2.4
Titanium	0.22	0.33
Manganese	0.5	0.4
Silicon	0.4	0.4
Boron	0.0007	0.0007

Table 11.8. Both of the titanium-modified alloys exhibit significantly higher yield and ultimate tensile strengths than the standard Type 316 SS alloy. Elongation values for the Type 316 + 0.22 wt% titanium alloy are approximately the same as the standard alloy and those of the Type 316 + 0.33 wt% titanium are approximately the same at 550 °C, but tend to be slightly higher at test temperature of 650 and 750 °C. Results of postirradiation creep-rupture tests completed to date are listed in Table 11.9. Comparison of these tests results with those listed in Table 11.6 indicates that both titanium-bearing alloys exhibit significant

TABLE 11.8. Tensile Properties of Types 316 and 316 + Titanium SS after Irradiation at 550 and 750 °C

Alloy	Test Temp., °C	Irradiated at 550 °C*			Irradiated at 750 °C*		
		Yield Stress, psi	Ultimate Tensile Strength, psi	Elongation, %	Yield Stress, psi	Ultimate Tensile Strength, psi	Elongation, %
316 SS, Heat H10542	550	17,300	60,400	32.9	19,900	63,100	29.7
	650	14,900	43,000	19.9	22,600	45,500	19.9
	750	14,800	29,300	13.2	17,500	26,200	14.8
316 SS + 0.22 Ti, Heat 3765-3	550	22,900	119,700	31.7	28,200	74,900	22.6
	650	25,400	62,900	17.1	29,000	52,300	13.1
	750	15,100	36,700	17.9	19,700	29,200	11.7
316 SS + 0.33 Ti, Heat 3765-4	550	26,500	78,800	29.9	28,200	70,100	22.6
	650	23,200	62,000	26.6	25,900	51,400	19.1
	750	25,700	37,000	20.0	21,900	29,300	20.5

* Irradiated for ~2000 hr to 7×10^{20} n/cm² (E > 1 MeV) and 9×10^{20} n/cm² (thermal); tensile tested at 0.002 min⁻¹

TABLE 11.9. Postirradiation* Creep-Rupture Properties of Vacuum-Melted Type 316 + Titanium SS at 650 °C

Alloy	Heat Treatment and/or Irradiation Conditions	Stress, psi	Rupture Life, hr	Creep Rate, %/hr	Total Elongation, %	Reduction Area, %
316 + 0.22 wt% Ti, Heat 3765-3	1 hr at 1050 °C + irradiated at 550 °C	40,000	50.5	0.156	1.3	3.2
		30,000	1053	0.0014	3.4	5.3
		25,000	>1673	--	>1.6	--
	1 hr at 1050 °C + irradiated at 750 °C	35,000	15.6	0.58	10.6	14.9
		30,000	69.5	0.15	13.4	16.1
		25,000	250.7	0.09	30.0	13.3
316 + 0.33 wt% Ti, Heat 3765-4	1 hr at 1050 °C + irradiated at 550 °C	40,000	75.8	0.022	5.2	11.6
		35,000	138.7	0.044	7.1	12.9
		30,000	557.9	0.005	4.8	--
	1 hr at 1050 °C + irradiated at 750 °C	35,000	34.5	0.41	25.3	17.8
		30,000	132.8	0.074	17.6	19.4
		25,000	442.0	0.035	23.6	27.1

* Irradiated for ~2000 hr to 7×10^{20} n/cm² (E > 1 MeV) and 9×10^{20} n/cm² (thermal)

improvements in postirradiation stress-rupture life properties. Creep rates of specimens irradiated at 750 °C were higher than those of specimens irradiated at 550 °C which suggests that thermal aging effects are also important in these alloys. Elongation values are highest for the Type 316 + 0.33 wt% titanium SS alloy.

EBR-II STRUCTURAL MATERIALS IRRADIATION EXPERIMENTS

E. E. Bloom and J. R. Weir, Jr.

Two structural materials irradiation experiments have been completed and are to be inserted into the reactor at the beginning of Run 28. The subassemblies have been designated X034 and X035 and are to be irradiated in Rows 2 and 7 position, respectively. Oak Ridge National Laboratory materials included in the subassemblies are Types 304, 304L, titanium-modified 304 and 304L, 316, and 316L SS, Incoloy 800 (several compositions), V-20 Ti, and Nickel 270.

Specimens are contained in specimen holders as shown in Figure 11.5. The experiments were designed on the basis of nuclear heating rates as determined previously.⁽²⁾ In the design of each tube of a subassembly, a computer program was used to calculate the gas annulus between the surface of the specimen holder and the inner wall of the tube. Briefly, the technique was as follows:

1. The materials and desired irradiation temperature for each specimen holder within a given tube were determined.

2. This information, along with inlet sodium temperature, sodium flow rate, a sodium fill coefficient, the fixed (cold) geometry, the material properties such as: emissivity, density, thermal expansion coefficients and thermal conductivities as a function of temperature, and the nuclear heating rates as a function of axial position were input to the computer.
3. The computer was programmed to calculate the cold gas annulus required to attain the specified irradiation temperatures. Calculations for gas annulus were performed at three places (bottom, middle, and top) on each of the sixteen 2 1/4 in. long specimen holders.

It was assumed that at steady state all heat transfer within the capsule tube was radial, and that the total heat transferred across the gas annulus was the sum of that transferred by conduction and by radiation. Standard equations applicable to these conditions and geometry were used.

On the basis of initial computer calculations, the sodium flow rate to attain required outlet sodium temperature from the subassembly was determined. Subsequent calculations were then performed to determine the required gas annulus for each specimen holder within a given tube. Starting at the bottom of the tube, where the sodium inlet temperature is 370 °C, heat was added to the sodium at each calculated position. The heat added included that generated within the tube and 1/7 of the heat generated in

TABLE 11.8. Tensile Properties of Types 316 and 316 + Titanium SS after Irradiation at 550 and 750 °C

Alloy	Test Temp., °C	Irradiated at 550 °C*			Irradiated at 750 °C*		
		Yield Stress, psi	Ultimate Tensile Strength, psi	Elongation, %	Yield Stress, psi	Ultimate Tensile Strength, psi	Elongation, %
316 SS, Heat H10542	550	17,300	60,400	32.9	19,900	63,100	29.7
	650	14,900	43,000	19.9	22,600	45,500	19.9
	750	14,800	29,300	13.2	17,500	26,200	14.8
316 SS + 0.22 Ti, Heat 3765-3	550	22,900	119,700	31.7	28,200	74,900	22.6
	650	25,400	62,900	17.1	29,000	52,300	13.1
	750	15,100	36,700	17.9	19,700	29,200	11.7
316 SS + 0.33 Ti, Heat 3765-4	550	26,500	78,800	29.9	28,200	70,100	22.6
	650	23,200	62,000	26.6	25,900	51,400	19.1
	750	25,700	37,000	20.0	21,900	29,300	20.5

* Irradiated for ~2000 hr to 7×10^{20} n/cm² (E > 1 MeV) and 9×10^{20} n/cm² (thermal); tensile tested at 0.002 min⁻¹

TABLE 11.9. Postirradiation* Creep-Rupture Properties of Vacuum-Melted Type 316 + Titanium SS at 650 °C

Alloy	Heat Treatment and/or Irradiation Conditions	Stress, psi	Rupture Life, hr	Creep Rate, %/hr	Total Elongation, %	Reduction Area, %
316 + 0.22 wt% Ti, Heat 3765-3	1 hr at 1050 °C + irradiated at 550 °C	40,000	50.5	0.156	1.3	3.2
		30,000	1053	0.0014	3.4	5.3
		25,000	>1673	--	>1.6	--
	1 hr at 1050 °C + irradiated at 750 °C	35,000	15.6	0.58	10.6	14.9
		30,000	69.5	0.15	13.4	16.1
		25,000	250.7	0.09	30.0	13.3
316 + 0.33 wt% Ti, Heat 3765-4	1 hr at 1050 °C + irradiated at 550 °C	40,000	75.8	0.022	5.2	11.6
		35,000	138.7	0.044	7.1	12.9
		30,000	557.9	0.005	4.8	--
	1 hr at 1050 °C + irradiated at 750 °C	35,000	34.5	0.41	25.3	17.8
		30,000	132.8	0.074	17.6	19.4
		25,000	442.0	0.035	23.6	27.1

* Irradiated for ~2000 hr to 7×10^{20} n/cm² (E > 1 MeV) and 9×10^{20} n/cm² (thermal)

improvements in postirradiation stress-rupture life properties. Creep rates of specimens irradiated at 750 °C were higher than those of specimens irradiated at 550 °C which suggests that thermal aging effects are also important in these alloys. Elongation values are highest for the Type 316 + 0.33 wt% titanium SS alloy.

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Specimens are contained in specimen holders as shown in Figure 11.5. The experiments were designed on the basis of nuclear heating rates as determined previously.⁽²⁾ In the design of each tube of a subassembly, a computer program was used to calculate the gas annulus between the surface of the specimen holder and the inner wall of the tube. Briefly, the technique was as follows:

1. The materials and desired irradiation temperature for each specimen holder within a given tube were determined.
2. This information, along with inlet sodium temperature, sodium flow rate, a sodium fill coefficient, the fixed (cold) geometry, the material properties such as: emissivity, density, thermal expansion coefficients and thermal conductivities as a function of temperature, and the nuclear heating rates as a function of axial position were input to the computer.
3. The computer was programmed to calculate the cold gas annulus required to attain the specified irradiation temperatures. Calculations for gas annulus were performed at three places (bottom, middle, and top) on each of the sixteen 2 1/4 in. long specimen holders.

It was assumed that at steady state all heat transfer within the capsule tube was radial, and that the total heat transferred across the gas annulus was the sum of that transferred by conduction and by radiation. Standard equations applicable to these conditions and geometry were used.

On the basis of initial computer calculations, the sodium flow rate to attain required outlet sodium temperature from the subassembly was determined. Subsequent calculations were then performed to determine the required gas annulus for each specimen holder within a given tube. Starting at the bottom of the tube, where the sodium inlet temperature is 370 °C, heat was added to the sodium at each calculated position. The heat added included that generated within the tube and 1/7 of the heat generated in

the sodium coolant within the sub-assembly and 1/7 of the heat generated in the stainless steel wrapper tube. The sodium coolant temperature at each calculated position was then determined and the calculation for gas annulus was based upon the transfer of heat to sodium at this temperature.

In this experiment the primary emphasis has been placed on an investigation of the variables of irradiation temperature, neutron flux, and neutron fluence. The effects of irradiation upon the properties of the various alloys will be determined by post-irradiation tensile, stress-rupture, hot hardness, density, and resistivity measurements. These data will be correlated with changes in microstructure as determined by optical and electron microscopy.

Irradiation Temperatures will be checked by using silicon carbide monitors as described by Martin and Price.⁽³⁾

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FIGURE 11.5. Schematic of EBR-II Specimen Holder

IRRADIATION DAMAGE TO REACTOR METALSREACTOR METALS RESEARCHALLOY EVALUATION

K. R. Wheeler and J. H. Hoage

The purpose of this program is to study, on a limited basis, materials potentially useful for reactor application. From such cursory examination, the more promising alloys can be studied in depth.

A British stainless steel FV-548, which has a composition similar to 316 SS but includes a nominal 1% Nb in its chemistry, has shown smaller ductility changes than 316 when irradiated to high fast fluence levels.⁽¹⁾ Five hundred pounds of 1/2 in. diam rod of FV-548 (Heat #49625) have been procured from Firth Vickers Stainless Steels, Ltd., for inclusion in the Radiation Effects program. Negotiations are also underway to obtain 300 ft of FV-548 tubing (0.250 in. OD x 0.016 in. wall) to be fabricated from the same heat (#49625) as the rod material. Additionally, a 500 lb heat of stainless steel 3/8 in. diam rod with the nominal FV-548 chemistry was obtained on an experimental basis from the Allegheny Ludlum Research Center. Randomly selected rods from both producers are now being characterized for structure, chemistry, and mechanical properties.

As reported in previous quarterly reports, specimens of ten alloys residual from the GE-APED superheat fuel-clad materials investigations are undergoing tensile properties

evaluation at PNL. Alloys involved in that phase of the testing program irradiated in the GETR are Incoloy 825 and 801, In 102, Inconel 718, Incoloy 804, Hastelloy F, and René 62.

The tensile properties at 1300 °F (704 °C) of the thermally aged control materials are reported in Table 12.1, as a continuance of the program. For comparison, the 1300 °F (704 °C) tensile properties of the same alloys in the irradiated condition are shown in Table 12.2. As can be seen, both irradiated and control materials have the same time-temperature history. The response of these alloys to elevated temperature exposure has been well documented.⁽²⁾ In general, each of these alloys shows a degree of metastability with increased hardness and lower ductility after prolonged exposure to temperatures above 1000 °F (538 °C). Each alloy (as discussed below) varies in its chemistry and the type of precipitate structure created in the dynamics of the aging process.

Incoloy 801

This alloy is basically Incoloy 800 with a percent addition of titanium to increase strength. The alloy appears to have reached its thermal aging peak in the shorter aging at 1000 °F (538 °C), with indications of overaging at longer times and higher temperatures. The ductility is reduced drastically after irradiation.

TABLE 12.1. Effect of Pretest Thermal Aging on the 1300 °F (704 °C) Tensile Properties of Selected Nickel-Base Alloys

Spec. Number	Material	Exposure Time, hr	Aging Temp, °F	0.2% YS, ksi	UTS, ksi	% UE	% TE	% RA
D 60	Incoloy 801	620	1000	47.73	55.45	8.38	28.19	31.82
D 61	Incoloy 801	620	1000	36.44	41.95	9.01	26.90	23.73
D 70	Incoloy 801	620	1300	44.19	54.88	11.98	28.44	37.21
D 76	Incoloy 801	620	1300	42.33	52.84	10.46	32.76	27.27
D 50	Incoloy 801	5200	1000	41.28	58.72	16.40	30.93	32.56
D 51	Incoloy 801	5200	1000	41.48	59.09	15.13	32.51	37.50
D 47	Incoloy 801	5200	1300	38.07	51.14	11.11	34.30	44.32
D 48	Incoloy 801	5200	1300	36.93	50.91	11.40	36.97	42.05
E 60	Incoloy 804	620	1000	45.90	58.20	10.02	24.35	28.69
E 61	Incoloy 804	620	1000	50.00	62.29	8.32	20.36	27.12
E 70	Incoloy 804	620	1300	42.28	56.10	8.14	24.43	32.52
E 71	Incoloy 804	620	1300	42.98	57.44	8.33	25.58	35.54
B 62	Incoloy 825	620	1000	37.98	55.43	12.25	47.79	63.57
B 63	Incoloy 825	620	1000	36.82	53.88	13.56	50.77	60.47
B 70	Incoloy 825	620	1300	35.38	53.46	15.24	48.55	64.62
B 71	Incoloy 825	620	1300	37.11	54.69	15.74	49.85	64.06
B 40	Incoloy 825	5200	1300	38.46	56.15	10.94	36.46	48.46
B 42	Incoloy 825	5200	1300	40.00	56.92	9.81	36.70	45.38
H 60	Hastelloy F	620	1000	47.64	65.75	18.12	45.64	47.24
H 61	Hastelloy F	620	1000	48.85	64.00	10.51	46.58	45.38
H 70	Hastelloy F	620	1300	51.18	70.87	8.72	31.64	44.09
H 71	Hastelloy F	620	1300	49.22	68.45	9.45	29.38	41.09
A 60	Inconel 718	620	1000	127.12	138.98	4.81	19.13	23.73
A 71	Inconel 718	620	1300	114.50	125.20	2.49	18.88	31.30
J 60	Rene 62	620	1000	132.84	147.76	3.93	12.29	28.36
J 61	Rene 62	620	1000	143.41	158.91	3.30	17.30	31.78
J 70	Rene 62	620	1300	131.06	151.06	5.45	17.14	35.61
J 71	Rene 62	620	1300	128.78	149.24	5.63	16.94	33.33
M 37	In 102	5200	1000	63.01	83.74	11.10	53.85	70.73
M 38	In 102	5200	1000	60.33	80.17	11.61	50.13	73.55

Incoloy 804

Additions of chromium and nickel with decrease in iron content provide the difference in chemistry between this material and Incoloy 800. Its thermal response is essentially the same as Incoloy 801, with post-irradiation ductility again severely reduced. Precipitates in this alloy are more extensive than the 801 modification, a condition no doubt contributing to its low irradiated ductility.

Incoloy 825

This alloy with its molybdenum and increased nickel content tends to form a sigma phase type structure with a trace of matrix carbide after exposure to 1400 °F (760 °C). The evidence for this can be seen by the downward trend in ductility after thermal aging for 5200 hr at 1300 °F (704 °C).

The ductility of Inconel 825, however, after elevated temperature irradiation is still several percent

TABLE 12.2. Effect of a Mixed Spectrum Neutron Irradiation^(a) on the 1300 °F (704 °C) Tensile Properties of Selected Nickel-Base Alloys

Specimen Number	Material	Exposure Time, hr	Fast Fluence, n/cm ²	Thermal Fluence, n/cm ²	Irrad. Temp, °F	0.2% YS, psi x 1000	UTS, psi x 1000	% UE ^(b)	% TE ^(c)	% RA ^(d)
A-2	Inconel 718	630	3.6 x 10 ²⁰	2.6 x 10 ²⁰	1300	95.8	95.8	0.74	0.80	6.15
B-3	Incoloy 825	630	4.0 x 10 ²⁰	2.8 x 10 ²⁰	1300	36.9	50.6	8.75	10.43	13.08
B-4	Incoloy 825	630	4.0 x 10 ²⁰	2.8 x 10 ²⁰	1300	36.2	50.3	6.09	7.77	16.28
B-21	Incoloy 825	5200	1.3 x 10 ²¹	3.4 x 10 ²¹	1300	43.1	52.8	5.06	6.35	10.77
B-22	Incoloy 825	5200	1.6 x 10 ²¹	4.2 x 10 ²¹	1300	39.5	53.1	5.47	6.16	11.63
D-5	Incoloy 801	630	2.6 x 10 ²⁰	1.7 x 10 ²⁰	1300	45.8	49.3	1.15	1.76	9.09
D-6	Incoloy 801	630	2.9 x 10 ²⁰	2.1 x 10 ²⁰	1300	42.2	42.8	0.85	2.76	15.56
D-21	Incoloy 801	5200	0.4 x 10 ²¹	0.6 x 10 ²¹	1000	39.9	44.6	1.64	2.36	6.98
D-22	Incoloy 801	5200	0.4 x 10 ²¹	0.6 x 10 ²¹	1000	39.3	43.3	1.42	2.18	11.11
E-1	Incoloy 804	630	3.6 x 10 ²⁰	2.6 x 10 ²⁰	1300	38.7	41.7	0.79	0.94	10.48
E-2	Incoloy 804	630	3.6 x 10 ²⁰	2.6 x 10 ²⁰	1300	38.1	39.9	0.72	0.79	8.06
H-1	Hastelloy F	630	3.6 x 10 ²⁰	2.6 x 10 ²⁰	1300	53.8	65.6	7.71	14.37	17.74
H-2	Hastelloy F	630	3.6 x 10 ²⁰	2.6 x 10 ²⁰	1300	52.8	65.1	5.61	12.95	18.11
J-2	Rene 62	630	2.9 x 10 ²⁰	2.1 x 10 ²⁰	1300	106.9	133.6	8.73	9.42	12.98
J-3	Rene 62	630	2.9 x 10 ²⁰	2.1 x 10 ²⁰	1300	131.3	132.0	0.91	1.09	10.69

(a) Irradiated in GETR

(b) Uniform Elongation

(c) Total Elongation

(d) Reduction in Area

greater than that observed in Incoloy 801 and 804 with the same irradiation history.

Hastelloy F

The high (7%) molybdenum content of Hastelloy F produces an extensive precipitation of a Laves phase (Fe₂Mo). The structure appears not to be affected by radiation, since strength and uniform elongation values are comparable for both irradiated material and controls with the same time-at-temperature history. There is, however, a sharp reduction in total elongation due to irradiation.

Inconel 718 and Rene 62

Both alloys are precipitation hardenable by a gamma prime type micro-constituent, with the development of considerable strength accompanied by lower ductility values. As can be seen, these alloys, although maintaining 4 or 5% uniform elongation in the aged condition, drop to approximately 1% after irradiation.

In 102

This nickel-base material, alloyed generously with refractory (Nb, Mo, W) additions, was developed for

long time service to 1200 °F (649 °C) with a high degree of structural stability.⁽³⁾ The alloy, however, does evidence an aging reaction after exposure to elevated temperature for extended periods of time. At 1300 °F (704 °C) test temperature, the tensile ductility of In 102 is nominally 60%, as compared to 50% for aged material. Almost no irradiated data are available for In 102. Experience and results have shown the irradiated material to be extremely notch sensitive. All three tensile samples of In 102 irradiated at 1000 °F (538 °C) to an approximate fast fluence level of 1×10^{21} n/cm² fractured in the gripping tabs before attaining the 0.2% yield point of the material. Attempts will be made to verify these results with additional tests.

In general, all alloys reported here have shown a ductility loss caused by prolonged thermal exposure. When irradiation and temperature effects are combined, the resulting ductility loss, with few exceptions, is extremely large.

IRRADIATION FACILITIES OPERATION

R. W. Barker

The purpose of this phase of the program is to provide for the timely accomplishment of irradiation, testing, and subsequent processing of the data obtained from a variety of reactor structural materials. Irradiations will be performed in aqueous, gaseous, and molten metal environments. Elevated temperature irradiations and tests will be em-

phasized. Special testing techniques, applicable to either irradiated or unirradiated specimens, will be developed.

Facilities Operation

The experiments discharged from the various Engineering Test Reactor (ETR) core positions for Cycles 93 and 94 are given in Tables 12.3 and 12.4, along with the estimated neutron fluxes and exposures.

The operating history of the ETR and the G-7 hot water loop for Cycle 92 is given in Table 12.5. Loop operation was normal for this cycle. The history for the out-of-reactor loop is given in Table 12.6.

Data Processing

One hundred and thirty-four tensile data reports have been added to the Radiation Effects on Metals (REM) computer program during the quarter, which raises the accumulative total to 5506.

IRRADIATION DAMAGE TO STAINLESS STEELS

A. J. Lovell

The purpose of this program is to determine the combined effects of neutron irradiation and environment on stainless steels. Irradiation-induced mechanical property changes are determined as a function of fast- and thermal-neutron fluence, temperature and environment of irradiation, and postirradiation test temperatures and environments. Emphases are placed on tensile and

TABLE 12.3. Summary of Experiments Discharged at the Conclusion of ETR Cycle 93

Experiment Designation	No. of Specimens	Type of Specimens	Estimated Flux, $n/cm^2/sec(a)$	Estimated Exposure, $n/cm^2(a)$
GEH-20-33	18	348 SS Tensiles	10.3×10^{13}	8.1×10^{21}
266	18	Zr-2 Corrosion Tabs	10.0×10^{13}	1.1×10^{21}
269	6	304 SS Creep	10.6×10^{13}	1.3×10^{21}
271	6	304 SS Stress-Rupture	1.0×10^{13}	3.7×10^{19}
272	6	304 SS Stress-Rupture	10.4×10^{13}	1.07×10^{21}
283	3	Carbon Steel Charpy	2.5×10^{13}	9.4×10^{19}
284	3	Carbon Steel Charpy	3.7×10^{13}	1.27×10^{20}
308	6	Fe Foils	8.0×10^{13}	1.94×10^{20}
309	6	Fe Foils	7.5×10^{13}	2.75×10^{20}
GEH-14-698	6	Fe Foils	1.1×10^{13}	3.2×10^{20}
699	6	Fe Foils	1.1×10^{13}	3.2×10^{20}

(a) Neutrons with energies greater than 1 MeV.

NOTE: Total specimens discharged, Cycle 93 = 84.

Total specimens discharged, ETR G-7 Loop, Cycle 93 = 72.

TABLE 12.4. Summary of Experiments Discharged at the Conclusion of ETR Cycle 94

Experiment Designation	No. of Specimens	Type of Specimens	Estimated Flux, $n/cm^2/sec(a)$	Estimated Exposure, $n/cm^2(a)$
GEH-20-79	18	AM 350 Tensile	10.3×10^{13}	8.1×10^{21}
273	6	304 SS Stress-Rupture	10.6×10^{13}	1.1×10^{21}
277	6	19-9 DL Tensile	10.9×10^{13}	1.0×10^{21}
278	6	19-9 DL Tensile	6.0×10^{13}	5.5×10^{20}
280	6	304 SS Creep	11.1×10^{13}	1.0×10^{21}
287	22	JS 700 Tensiles	10.0×10^{13}	2.5×10^{20}
310	6	"A" Ni Foils	2.5×10^{13}	6.0×10^{19}
311	6	"A" Ni Foils	3.7×10^{13}	9.0×10^{19}
GEH-14-696	5	Mild Steel Impact Spec.	0.35×10^{13}	1.9×10^{19}
697	4	A-302 B DCB	0.3×10^{13}	1.6×10^{19}
762	6	"A" Ni Foils	$\sim 2.1 \times 10^{13}$	6.2×10^{19}
766	6	"A" Ni Foils	$\sim 3.1 \times 10^{13}$	9.1×10^{19}

(a) Neutrons with energies greater than 1 MeV.

NOTE: Total specimens discharged, Cycle 94 = 98.

Total specimens discharged, ETR G-7 loop, Cycle 94 = 76.

Total specimens discharged, ETR G-7 loop, to date = 3386.

TABLE 12.5. Summary of Operating History for ETR and G-7 Hot Water Loop, ETR Cycles 92 and 93

<u>Operating History</u>	<u>ETR Cycle Number</u>	
	<u>92</u>	<u>93</u>
1. <u>Reactor History</u>		
Startup ^(a)	10-12-67	12-7-67
End of Cycle	11-26-67	1-30-68
Megawatt Days	5949	5966
Effective Days at 175 MW	34.0	34.1
Number of Scrams ^(b)	11	19
Number of Shutdowns	3	5
2. <u>G-7 Hot Water Loop History</u>		
Maximum Temperature	525 °F	532 °F
Effective Days Above 200 °F	35.5	35.6
Effective Days at Oper. Temp.	33.0	31.2
Operating Efficiency ^(c)	104.4%	104.4%
Temperature Efficiency ^(d)	93.0%	87.7%

(a) Includes flux runs at low power.

(b) Power drops below half of full power (175 MW) with immediate recovery.

(c) Effective days of loop operation above 200 °F (95 °C) relative to effective days of reactor operation at 175 MW.

(d) Effective days of loop operation between 500 and 550 °F (260 and 290 °C) relative to days of operation above 200 °F (95 °C).

TABLE 12.6. Summary of Operating History and Discharge Schedule for Out-of-Reactor Loop, ETR Cycles 92 and 93

1. TIME-TEMPERATURE SUMMARY

<u>Temperature</u>	<u>Time, hr</u>	
	<u>Cycle 92</u>	<u>Cycle 93</u>
200 °C (392 °F)	20.4	50.6
218 °C (424 °F)	12.1	15.8
246 °C (475 °F)	26.5	42.1
282 °C (540 °F)	<u>792.0</u>	<u>746.6</u>
TOTAL TIME	851.0	855.1

2. SPECIMEN DISCHARGES: Material, Specimen Type, and Number.

Cycle 92	Type 304 SS	Creep	6
Cycle 93	Type 348 SS	Tensile	<u>18</u>
		TOTAL ^(a)	24

(a) Total specimens discharged to date = 1461.

creep investigations and on studies of the mechanisms of irradiation-induced property changes.

Annealed rolling and transverse, and 25% cold-worked transverse AISI 348 specimens have been irradiated at 290 °C, in the G-7 loop, to a fast neutron fluence of 8.1×10^{21} ($E > 1$ MeV). This fluence represents 1080 full power days in the Engineering Test Reactor (ETR) G-7

hot water loop. After being exposed to the irradiation in the pH 10 water at 2000 psia, the specimens were coated with a rather adherent corrosion layer. No evidence of localized pitting or attack was apparent.

The tensile testing is partially completed. The effects of test temperatures on the strength of the annealed specimens after irradiation are shown in Figure 12.1. The

FIGURE 12.1. The Effect of Test Temperature on the Strength of Irradiated AISI 348 Flat SS Specimens in the Annealed Condition

increase of the yield strength over the as-fabricated values was observed at all test temperatures up to and including the 750 °C test. The ductility was very low for all irradiated specimens (Figure 12.2).

The effect of test temperatures on the strength of AM-350 in the double-aged condition after an out-of-reactor age and after irradiation to 6.4×10^{21} ($E > 1$ MeV) is shown in

Figure 12.3. There was a strengthening of the yield strength at all test temperatures in the 300 to 600 °C range. Figure 12.4 shows that the uniforms and totals for all irradiated specimens were lower at all test temperatures.

The data generated on martensitic AISI 410 SS are consolidated in Table 12.7. Irradiation at 60 °C were in the ETR core where the water

Neg 0680963-1

FIGURE 12.2. The Effect of Test Temperature on the Ductility of Irradiated AISI 348 Flat SS Specimens in the Annealed Condition

Neg 0680763

FIGURE 12.3. Effect of Test Temperature on the Strength of Irradiated AM-350 in the Double Aged Condition

pH and pressure ranges are 5.5 to 6.5 and 165 to 200 psia. The 290 $^{\circ}\text{C}$ irradiations were in the G-7 hot water loop.

The limited data after irradiation at 60 $^{\circ}\text{C}$ showed increased yield strength with neutron fluence, and decreased ductility with neutron fluence for room temperature tests.

The change in strength with neutron fluence for room temperature and 600 $^{\circ}\text{C}$ test temperatures after a 290 $^{\circ}\text{C}$ irradiation is shown in Figure 12.5. The strength increased with fluence for room temperature tests. The 600 $^{\circ}\text{C}$ test first indicated a small decrease with fluence to 1×10^{20} but an increase thereafter. The effect of fluence on ductility is shown in Figure 12.6. The room-temperature test shows decreased uniform and total elongation with increasing fluence. The attempt to test at room temperature after a

Neg 0680368-4

FIGURE 12.4. The Effect of Test Temperature on the Ductility of Irradiated AM-350 SS in the Double Aged Condition

TABLE 12.7. Tensile Properties of Martensitic 410 SS

IRRADIATION DATA			TENSILE DATA					
Fluence (E > 1 MeV)	Irr. Temp, °C	Test Temp, °C	Strength KPSI			Percent Strain		Plastic Work, ft-lb
			PEL	0.2% YS	UTS	Uniform	Total	
		RT	118.7	140.4	173.5	4.48	6.75	40.74
		RT	115.0	141.0	175.0	4.84	7.27	44.86
		RT	95.0	128.0	175.5	5.53	8.79	47.47
		RT	120.0	147.5	172.7	6.02	8.71	49.06
		RT	112.5	147.0	169.5	5.53	7.85	43.65
		RT	53.0	137.9	167.5	4.14	7.04	37.66
		RT	32.2	140.1	174.5	4.80	8.62	48.46
		RT	42.1	136.1	170.3	5.81	9.59	52.95
		RT	29.6	131.8	167.5	5.04	7.97	43.28
		RT	34.3	136.0	170.3	5.77	9.81	54.99
		RT	44.6	141.1	174.5	5.24	8.69	49.13
		RT	87.1	140.5	174.6	5.48	8.02	50.09
		RT	117.5	140.7	173.7	5.17	7.81	48.24
		RT	115.0	141.0	175.0	4.84	7.27	44.86
		RT	118.8	141.1	174.0	3.87	6.30	38.69
		RT	118.7	138.9	172.7	4.65	6.99	42.00
		RT	88.4	136.9	173.0	4.98	7.63	40.95
		RT	99.8	137.9	171.2	5.17	7.87	44.41
		RT	84.2	135.5	169.7	5.17	7.84	41.90
		300	92.5	140.5	190.2	7.02	9.10	54.05
		400	64.4	125.0	182.7	9.59	12.21	72.25
		500	49.0	112.7	157.1	6.14	9.68	49.63
		600	11.2	31.3	40.7	2.99	21.79	26.01
1.72 x 10 ²⁰	60	RT	73.0	168.5	198.0	2.52	7.67	25.92
1.72 x 10 ²⁰	60	RT	82.6	163.8	185.9	2.53	2.94	16.57
6.37 x 10 ²⁰	60	RT	106.4	182.5	201.2	1.79	7.31	13.43
1.10 x 10 ²¹	60	RT	104.8	170.3	182.1	1.19	1.25	5.53
1.10 x 10 ²¹	60	RT	97.6	180.5	193.9	1.41	1.82	9.37
3.70 x 10 ¹⁹	290	RT	62.5	175.0	196.7	3.64	6.49	40.43
3.70 x 10 ¹⁹	290	RT	133.0	168.7	194.1	3.79	6.51	40.43
3.70 x 10 ¹⁹	290	RT	96.2	178.8	201.9	3.73	6.88	45.79
3.70 x 10 ¹⁹	290	RT	86.6	182.2	201.2	4.03	6.95	45.06
4.50 x 10 ¹⁹	290	RT	81.9	159.3	185.4	2.59	4.97	27.98
4.50 x 10 ¹⁹	290	RT	114.2	170.6	230.2	4.35	6.74	47.35
4.50 x 10 ¹⁹	290	RT	128.6	181.9	216.7	6.24	9.37	68.29
4.50 x 10 ¹⁹	290	450	57.9	103.3	139.7	8.95	11.68	56.49
4.50 x 10 ¹⁹	290	600	15.0	25.1	30.4	3.00	26.36	20.52
4.50 x 10 ¹⁹	290	500	13.6	22.8	30.2	3.73	19.96	17.03
1.01 x 10 ²⁰	290	RT	121.9	193.3	215.5	2.53	4.74	34.14
1.01 x 10 ²⁰	290	RT	109.1	194.8	218.2	2.76	5.17	37.55
1.01 x 10 ²⁰	290	450	41.3	85.6	95.2	1.62	6.46	19.07
1.01 x 10 ²⁰	290	450	56.1	93.5	100.5	0.93	4.75	14.36
1.01 x 10 ²⁰	290	600	7.9	14.3	18.4	4.95	38.72	20.44
1.01 x 10 ²⁰	290	600	8.9	18.0	23.4	3.15	31.38	19.50
1.01 x 10 ²⁰	290	600	12.8	20.2	25.1	4.53	25.52	19.64
1.05 x 10 ²⁰	290	RT	137.1	185.2	210.7	2.49	6.34	44.28
1.05 x 10 ²⁰	290	RT	98.2	178.6	206.0	3.91	6.68	48.42
1.05 x 10 ²⁰	290	450	60.2	118.7	155.6	6.54	9.84	50.50
1.05 x 10 ²⁰	290	450	53.9	116.7	153.7	7.09	10.33	51.45
1.05 x 10 ²⁰	290	600	11.2	20.1	25.9	3.45	33.64	22.32
1.05 x 10 ²⁰	290	600	12.9	25.2	26.6	1.42	25.85	17.29
1.19 x 10 ²⁰	290	RT	130.7	180.9	208.8	3.70	3.76	25.52
1.19 x 10 ²⁰	290	RT	125.0	184.0	212.6	4.73	7.77	55.67
1.19 x 10 ²⁰	290	450	87.1	129.2	169.4	8.53	12.05	68.86
1.19 x 10 ²⁰	290	450	48.2	107.1	145.2	8.36	11.55	58.82
1.19 x 10 ²⁰	290	600	12.7	22.4	27.7	3.68	36.24	25.71
1.19 x 10 ²⁰	290	600	10.0	21.7	26.8	3.89	34.25	24.32
2.40 x 10 ²⁰	290	RT	87.4	189.3	213.6	3.08	5.32	36.34
2.40 x 10 ²⁰	290	RT	91.3	192.3	217.5	2.92	5.29	37.27
2.40 x 10 ²⁰	290	300	101.2	166.2	195.7	3.23	5.26	31.22
2.40 x 10 ²⁰	290	300	74.6	154.7	191.3	4.44	6.57	38.96
2.50 x 10 ²⁰	290	RT	120.0	202.5	229.5	3.19	3.30	21.84
2.50 x 10 ²⁰	290	RT	139.4	204.8	215.6	2.48	4.75	32.41
2.50 x 10 ²⁰	290	300	86.8	157.6	197.8	4.08	4.53	38.78
2.50 x 10 ²⁰	290	300	72.7	155.0	194.7	3.77	5.73	34.48
2.50 x 10 ²⁰	290	450	51.9	126.2	167.5	8.19	11.35	63.86
2.50 x 10 ²⁰	290	600	13.3	26.5	31.8	3.43	26.54	22.69
6.44 x 10 ²¹	290	400	115.4	187.0	205.0	1.79	3.75	23.35
6.44 x 10 ²¹	290	450	76.2	175.0	182.5	1.81	3.99	21.71
6.44 x 10 ²¹	290	500	70.4	146.9	156.3	1.88	4.67	24.07
6.44 x 10 ²¹	290	600	19.4	45.8	50.7	1.27	12.19	16.63

Neg 0680625-2

FIGURE 12.5. The Effect of Neutron Exposure at 290 °C on the Strength of Martensitic AISI 410 SS

Neg 0680625

FIGURE 12.6. The Effect of Neutron Exposure at 290 °C on the Ductility of Martensitic AISI 410 SS

fluence of 6.4×10^{21} was not successful. The specimen became prematurely brittle at the extensometer points. In the 600 °C test, the ductility increased with increasing fluence but then decreased. It appears that the softening effect of the 290 °C age on the 600 °C tensile properties outweighed the hardening effect of the irradiation up to moderate fluence. So, as the fluence went to a higher value, the irradiation hardening out-

weighed the aging effects on the tensile properties.

The effect of test temperatures on the strength of the martensitic 410 irradiated at 290 °C to a fast fluence of 6.4×10^{21} is shown in Figure 12.7, while the ductility-versus-test temperature is presented in Figure 12.8. At all test temperatures, the yield strengths are greater and the ductilities less for irradiated specimens.

Neg 0680368-2

FIGURE 12.7. The Effect of Test Temperature on the Strength of Irradiated 410 SS in the Martensitic Condition

Neg 0680368-3

FIGURE 12.8. The Effect of Test Temperature on the Ductility of Irradiated 410 SS in the Martensitic Condition

ELECTRON MICROSCOPY OF ETR - IRRADIATED STAINLESS STEEL

H. R. Brager

The electron microscopy examination was continued on AISI type 304 SS irradiated in the ETR to 6×10^{21} n/cm² ($E > 1$ MeV) at 290 °C. This material was examined to determine the microstructure responsible for the high temperature irradiation hardening which persisted to the highest test temperature, 750 °C.

Photomicrographs presented in the last quarterly report⁽⁴⁾ were analyzed. These picture the gage section of the material as tested at 750 °C and the same material having an additional 1 hr vacuum anneal at 870 °C (Table 12.8). In addition, sections of the material tested at 750 °C were vacuum annealed 1 hr at 980 °C or 1090 °C, (Table 12.8) and examined by electron microscopy (Figures 12.9 and 12.10, respectively). The significant observations were:

TABLE 12.8. *Electron Microscopy Observations of Type 304 SS Irradiated in the ETR to 6×10^{21} n/cm² (>1.0 MeV) at 290 °C*

Material Tensile Tested at 750 °C, Plus a Vacuum Anneal 1 hr at the Indicated Temperature	Anneal Temperature, °C			
	As Tested	870	980	1090
Cavity density (cm ³) ⁻¹	2×10^{15}	3×10^{14}	2×10^{14}	1.5×10^{14}
$\Delta V/V$ (%)	0.17	0.25	0.50	1.2
Helium in equilibrium with surface tension of cavities (ppm)	300	275	400	600
Average cavity diameter (Å) (First moment/third moment)	110/115	210/250	270/350	460/575
Total cavity surface area (10 ¹⁹ Å ² /cm ³) (Calculated using first moment average cavity diameter/third moment average cavity diameter)	2.5/3.0	1.5/2.0	2.2/2.7	3.0/4.0
Total cavity volume (10 ²¹ Å ³ /cm ³) (Calculated using first moment average cavity diameter/third moment average cavity diameter)	3.0/3.5	2.5/4.5	4.5/9.2	13.5/21

Neg 5255-A

96,000X

FIGURE 12.9. *Microstructure of AISI 304 SS Irradiated in the ETR at 290 °C to a Fluence of 6×10^{21} n/cm², Tested at 750 °C, and then Annealed at 980 °C for 1 hr*

Neg 5301-B

98,000X

FIGURE 12.10. Microstructure of AISI 304 SS Irradiated in the ETR at 290 °C to a Fluence of 6×10^{21} n/cm², Tested at 750 °C, and then Annealed at 1090 °C for 1 hr

- The cavities were present after vacuum annealing at temperatures up to 1090 °C.
- The cavities were uniformly located throughout the matrix and on grain boundaries. The grain boundaries did not have a denuded zone, and the size distribution of the cavities at the grain boundaries was not significantly different than for cavities in the matrix.
- No strain fields were observed around the cavities for material vacuum annealed within the temperature range of 750 to 1090 °C and furnace cooled.
- The total surface area of the cavities was constant over the investigated temperature range of 750 to 1090 °C while the total volume of the cavities increased with increased annealing temperature. The consistency of

macroscopic cavity surface area indicates that the cavities surface tensions are balanced by an internal gas pressure.

- The amount of helium gas produced by transmutation (about 12 ppm) was less than one-tenth the amount required to balance the cavities surface tension (assuming $\gamma = 1500 \text{ ergs/cm}^2$).

The first four observations support the conclusion that the ETR irradiation produced cavities that behave like gas filled bubbles. However, the last observation suggests that the cavities are helium stabilized vacancy clusters. To provide additional information about the nature of the cavities, a sample of the ETR irradiated stainless steel was vacuum annealed 1 hr at 980 °C, annealed 1 hr at 820 °C in NaK at a pressure of 10,000 atm, and examined by transmission electron microscopy, Figure 12.11. The 980 °C vacuum anneal should have provided a high concentration ($2 \times 10^{14}/\text{cm}^3$) of large cavities (300 Å). The 1-hr, 820 °C high-pressure (10,000 atm) anneal should have promoted the removal of soluble and chemically reactant gases which could help stabilize the cavities. The preliminary examination of the electron micrographs of the high pressure annealed steel provided the following observations:

- Approximately 2×10^{13} cavities/ cm^3 with an average diameter of about 700 Å were still present after annealing the material 1 hr at 820 °C and 10,000 atm.
- There were no observable strain fields around the cavities.

- About 10^{13} precipitates/ cm^3 with an average diameter of about 400 Å were observed. The majority of the polyhedral precipitates were attached to cavities.
- Approximately 10^{14} small particles/ cm^3 with an average diameter of 100 Å were observed. These small uniformly dispersed particles are not vacancy- or interstitial-clusters.

The preliminary estimate of the amount of helium gas required to balance the 10,000 atm applied pressure plus the cavity surface tension (assuming $\gamma = 150 \text{ ergs/cm}^2$) is about 800 ppm. The concentration of the helium required to balance the surface tension of the cavities in the steel before the high pressure anneal was 400 ppm (Table 12.8). The factor of two discrepancies probably arises from the preliminary results so far obtained from analyses of the electron micrographs of samples after the high pressure anneal. Nevertheless, the recent results definitely confirm earlier results that the cavities do not respond like voids, but do behave like gas-filled bubbles.

IRRADIATION DAMAGE TO NICKEL-BASE ALLOYS

I. S. Levy

The requirements for corrosion resistance and high temperature strength place nickel-base alloys among the more promising materials for advanced nuclear reactor applications. When these alloys are irradiated at service temperatures, however, they generally suffer embrittlement and re-

Neg 5452-D

96,000X

FIGURE 12.11. Microstructure of Specimen Depicted in Figure 12.9, but After a 1 hr Anneal at 820 °C Under 10,000 atm Hydrostatic Pressure

duction in rupture life. The purpose of this program is to determine the effects of modified microstructures upon the irradiation stability of nickel-base alloys. These structural modifications are made by preirradiation thermal or thermomechanical treatments and are evaluated by tensile and stress-rupture tests and by microstructural examinations.

Recent studies on the three alloys in this program, Hastelloy X-280, Inconel 600, and Inconel X-750, have

been concerned with the effects of pretreatments and long-term (>1500 hr) irradiation at 1250 °F (fast fluence of approximately 1×10^{20} n/cm²). It has been found that the thermal exposure alone produces extensive matrix precipitation in Hastelloy X-280 and Inconel 600,⁽⁵⁾ thus causing increases in yield strength and reductions in ductility in both alloys.

Preliminary data from stress-rupture tests on the control specimens of the three alloys at 1350 °F

show little change in rupture life after this thermal exposure compared with data after exposure at 540 °F. In fact, some treatments of Inconel X-750 that resulted in poor rupture properties after 540 °F exposure (because they produced an under-aged structure) showed, after the >1500 hr exposure at 1250 °F, better properties more closely related to those of the standard treatment. Presumably, this is the result of aging reactions during exposure.

The lack of reduction in rupture life in Hastelloy X-280 and Inconel 600, after this thermal exposure, may be caused by the opposing effects upon rupture life of the lowered ductility and increased strength indicated in tensile tests.

However, after irradiation, preliminary data show that compared to control specimens, the rupture lives of all three alloys were markedly reduced. Reductions ranged from about a factor of 10 for Hastelloy X-280 (36 hr vs 320 hr) and Inconel 600 (43 hr vs 515 hr) to about a factor of 300 for Inconel X-750 (2.5 hr vs 700 hr). These reductions in rupture life are larger than those found after exposure at 540 °F; compared to control specimens, the latter were only of the order of a factor of 3 for all three alloys. Thus, irradiation at 1250 °F increased these reductions (compared to 540 °F irradiation) by a factor of 3 for the solution-hardened alloys (Hastelloy X-280 and Inconel 600) to 25 for the precipitation-hardened alloy (Inconel X-750). The higher reductions may be associated with the combined ef-

fect of thermal and irradiation-induced instabilities. The fact that Inconel X-750 suffered a greater reduction in rupture life than did the two solution-hardened alloys might be expected, since a precipitation-hardened alloy would be more susceptible to aging reactions.

IN-REACTOR MEASUREMENTS OF MECHANICAL PROPERTIES

E. R. Gilbert and N. E. Harding

The in-reactor measurements program is intended to determine the effects of irradiation on the mechanical properties of reactor structural materials, to establish property data suitable for design purposes, and to consider mechanisms through irradiation effects on macroscopic deformation. The program currently involves conducting in-reactor creep measurements on annealed 304 SS, Hastelloy X, heat treated Zr-2.5 wt % Nb, and Zircaloy-2. Capsule development is being pursued so that program capabilities may be extended.

304 Stainless Steel

Loading curves for creep tests conducted at 450 °C are shown in Figure 12.12. The curves are similar for both an in-reactor test and an unirradiated control test and show discontinuous yielding independent of time. At 450 °C, a serrated stress-strain curve, due to strain aging, is observed in tensile tests. The discontinuous curve observed in the loading of the creep tests is probably a consequence of the same

Neg 0680746

FIGURE 12.12. Loading Curves for Annealed 304 SS Tested During Neutron Irradiation and Without Irradiation at 450 °C

phenomenon that causes the serrated stress strain curve in tensile tests. However, it appears as steps under the constant load applied in the creep test in contrast to serrations

under the constant cross head speed used in the tensile test. Neutron irradiation did not influence the discontinuous yielding but it did cause decreased fracture strain.

Zircaloy-2

An in-reactor creep test was conducted on 750 °C annealed Zircaloy-2 in the PRTR with a neutron flux of approximately 5×10^{12} nvt ($E > 1$ MeV). The strain-versus-time curve and corresponding unirradiated control creep data are shown in Figure 12.13. The in-reactor creep appears to be increased over the unirradiated control

test. The creep rates for the annealed Zircaloy-2 are plotted on a semilogarithmic plot of strain rate-versus-stress in Figure 12.14 along with data by Fidleris and Williams⁽⁶⁾ for cold worked Zircaloy-2. It appears that both cold-worked and annealed Zircaloy-2 exhibit faster creep rates during neutron irradiation.

Neg 0672975-2

FIGURE 12.13. Strain Versus Time for Creep of Zircaloy-2 Tested in the PRTR in a Neutron Flux of Approximately 5×10^{12} nv ($E > 1$ MeV). Specimens Cut in Longitudinal Direction from Plate, Annealed for 20 hr at 750 °C, and Furnace Cooled.

Neg 0673372-1

FIGURE 12.14. Strain-Rate Versus Stress on Semilogarithmic Coordinates for Zr-2.5 wt% Nb

Zr-2.5 wt% Nb

In-reactor creep rates for Zr-2.5 wt% Nb are plotted (Figure 12.14) against stress on semilogarithmic coordinates along with data for cold-worked Zircaloy-2 by Fidleris and Williams.⁽⁶⁾ The in-reactor

creep rates for Zr-2.5 wt% Nb show little if any dependence on stress between 14 and 32 kg/mm^2 and are similar in value to those for cold-worked Zircaloy-2 between 10 and 15 kg/mm^2 . Appreciably slower in-reactor creep rates are observed at stresses below 10 kg/mm^2 .

GRAIN BOUNDARY STABILIZATION

E. R. Bradley

A study was recently initiated to investigate the recrystallization and grain growth characteristics of metals containing small concentrations of inert gases.

Since impurities generally inhibit the motion of grain boundaries in metals, a large amount of work has been directed toward this problem. However, very few investigations of the interaction of the inert gases with boundaries have been conducted. Observations on uranium⁽⁷⁾ and Al-0.7 wt% Li Alloy⁽⁸⁾ after neutron irradiation indicate that grain boundaries are stabilized during irradiation. One proposed mechanism for this stabilization is a "locking" of the boundaries by inert gases produced during irradiation.

Bubbles have been observed along grain boundaries and dislocation lines in irradiated structural materials. These bubbles are proposed to contain helium which is produced by $n + \alpha$ fast reactions, or they are caused by boron impurities in the materials by the thermal reactions $^{10}\text{B} + n^1 \rightarrow \alpha$. Postirradiation annealing of these materials also indicates a stabilized grain structure.

Inert gases are presently considered important in determining the properties of iron and nickel base alloys. The extreme loss of ductility in these alloys has been attributed to the buildup of helium gas bubbles. If a small concentration of inert gas can be introduced

into a material, stabilizing the grain structure should be possible without detrimentally affecting the mechanical properties of the material. This would produce a material acceptable for prolonged service at high temperatures without catastrophic grain growth occurring.

Ion bombardment is one method of introducing small quantities of inert gas into a metal. An apparatus, shown schematically in Figure 12.15, has been constructed and shown to be effective in introducing small quantities of helium into a metal. Nickel is a convenient metal to demonstrate the practicality of the grain stabilization technique; therefore, nickel powder in the size range 3 to 7 μ was used. The bombardment apparatus consists of an evacuative chamber with a variable leak vacuum valve to maintain a dynamic helium pressure of 100 μ during bombardment. The anode is a 1/8 in. diam nickel rod; the powder sample in contact with a nickel rod is the cathode. The potential source for the glow discharge is a 5 kV power supply whose voltage and current is monitored with an x-y recorder.

The amount of helium injected into the nickel powder is determined by

FIGURE 12.15. Schematic of Glow Discharge System for Injecting Helium into Metal Powders

heating the powder in vacuum and measuring the helium liberated as a function of temperature with a mass spectrometer type leak detector. The heating rate used is 20 °C/min. A typical graph of helium liberated versus temperature is shown in Figure 12.16.

Two distinct peaks occur as the temperature is increased from 50 to 800 °C. The relative size and position of these two peaks depend on the time and potential used during bombardment. The low temperature peak occurs between 100 to 400 °C and the high temperature peak between 500 to 800 °C. This technique and the apparatus available provide information only to a temperature of 800 °C. For higher temperature data, tin is alloyed with the nickel powder to determine the amount of helium remaining after heating to 800 °C. Up to 5% of the

FIGURE 12.16. A Typical Chart Showing Helium Release from Nickel Powder Following Helium Ion Bombardment as a Function of Increasing Temperature During Heating at a Constant Rate

total helium released is found to be retained after heating in vacuum to 800 °C. The temperature at which this helium would be released from the metal powder cannot be determined by this method, but processing and compaction of the powders at temperatures up to 800 °C without complete loss of helium are feasible.

Nickel powder, with and without helium, is being hydrostatically compacted at various pressures and temperatures. Recrystallization and grain growth in these compacts will be studied by microscopy, hardness, and X-ray techniques.

FRACTURE STUDIES

J. A. Williams

Size Effects of the DCB Specimen on the Fracture Toughness of ASTM A533-B Steel

A major task of the HSST* irradiation damage program is to determine size effects of various types of fracture mechanics specimens. The objectives of this task are to determine as a function of neutron fluence:

- The smallest specimen size that will provide a valid measure of plane-strain fracture toughness at a given temperature
- The limiting temperature at which plane-strain fracture cannot be measured regardless of specimen size
- The influence on gross fracture toughness of microstructural and neutron fluence gradients in

* Heavy Section Steel Technology

heavy-section plates. Fracture mechanics specimens of the DCB, WOL, and notch bend types will be used in this task.

An initial plate, 1 in. thick x 1 in. wide x 2 in. long (RD) of ASTM A533-B Class 1 steel Lukens Heat Al008-1, was supplied by Oak Ridge National Laboratory in the heat treated condition. Heat treatment consisted of normalizing at 1600 to 1750 °F, austenitizing at 1650 °F for 4 hr, water quenching, tempering at 1225 °F for 4 hr, and furnace cooling.

Double cantilever beam specimens 0.5 and 1.0 in. thick were machined from the 1/2 T plate position, with the fracture plane normal to the plate surfaces and in the rolling direction. The results of the fracture toughness tests are tabulated in

Table 12.9. Fracture toughness tests at -160 and -140 °C were made on the 0.5 in. specimen. The second test at -140 °C resulted in fracture of both specimens arms. The 1.0 in. specimen was tested at -160, -140 and -120 °C. Specimen arm breakage occurred at -120 °C during the second test. The remainder of the 1.0 in. specimen was remachined (Figure 12.17) and tested at -160 °C to the end of the specimen. Arm breakage of both the 0.5 and the 1.0 in. specimens occurred at values less than 1.5 for $B/(K_{Ic}/\sigma_{ys})^2$.

The fracture toughness (K_{Ic}) of A533-B is plotted as a function of $B/(K_{Ic}/\sigma_{ys})^2$ in Figure 12.18d. An increase in K_{Ic} as a function of the crack length for the DCB specimen was observed (Table 12.9 and

TABLE 12.9. Results of DCB Fracture Toughness Tests of A533-B Class 1 Steel Specimens from 1/2 T Position in 12 in. Plate

Specimen Number	Thickness, (e) B, in.	Temperature, °C	Yield ^(a) Strength, ksi	Crack Length, C, in.	$\frac{B}{(K_{Ic}/\sigma_{ys})^2}$	K_{Ic} , ksi $\sqrt{\text{in.}}$	K_{Ia} , ksi $\sqrt{\text{in.}}$
01KA9	0.5	-160	95	2.23	4.3	32.1	29.9
01KA9	0.5	-160	95	2.27	4.2	32.8	30.8
01KA9	0.5	-160	95	2.43	3.0	38.6	29.8
01KA9	0.5	-160	95	2.59	3.5	35.8	32.8
01KA9	0.5	-140	86	2.78	1.1	58.5	31.3
01KA9	0.5	-140	86	3.95	0.8	68.0	(b)
01KA22	1.0	-160	95	3.06	9.7	30.4	29.9
01KA22	1.0	-160	95	3.17	7.9	33.7	31.1
01KA22	1.0	-160	95	3.55	5.5	40.5	35.7
01KA22	1.0	-160	95	4.47	5.5	40.5	37.8
01KA22	1.0	-160	95	4.75	4.9	42.8	36.3
01KA22	1.0	-140	86	5.52	3.2	48.1	37.8
01KA22	1.0	-120	79	6.92	1.7	61.1	56.0
01KA22	1.0	-120	79	7.32	1.0	77.5	(b)
01KA22 ^(c)	1.0	-160	95	2.40	5.1	42.2	33.6
01KA22	1.0	-160	95	3.07	5.4	41.0	39.0
01KA22	1.0	-160	95	3.49	4.7	43.9	(d)

(a) Yield strength data taken from Reference 10 for SA533-B 8 in. plate

(b) Both arms fractured

(c) 5 1/2 in. long specimen machined from end of 01KA22 (Figure 12.17)

(d) Fracture run to end of specimen

(e) $B_N/B = 0.33$

Neg 0680889

FIGURE 12.17. DCB (1 in. thick) Specimen of A533-B. Short Specimen Was Machined after Fracture of Both Arms

Figure 12.18d). To determine if the observed increase was a geometry effect or a material gradient, the remachined specimen from the end of 01KA22 was tested at -160°C . The fracture toughness results of all tests at -160°C are plotted as a function of crack length in Figure 12.18a and as a function of the distance from the flame cut end of the plate in Figure 12.18b. From these data, the observed change in K_{IC} does not appear to be geometry dependent, but rather a result of material gradient. Whether the material gradient is a result of flame

cutting or a gradient of the original plate is inconclusive. Metallographic examinations have been made that indicate that microstructural changes occur only to a depth of about $1/4$ in. from the flame cut surface.⁽⁹⁾ When the DCB specimen is used for plane-strain fracture toughness testing, the requirement $B/(K_{IC}/\sigma_{ys})^2$ being equal to or greater than 2.5 appears to be a valid criteria (Figure 12.18d). Whether this requirement can be decreased by using a different net thickness (B_N) is yet to be determined.

Neg 0680847-1

FIGURE 12.18. Fracture Toughness (K_{Ic}) of Q & T A533-B as a Function of (2a) Crack Length, (2b) Distance from Flame Cut Surface (2c) Temperature, and (2d) $B/(K_{Ic}/\sigma_{ys})^2$. All Measurements Made at 1/2 T Position of 12 in. Plate with the Crack Plane Normal to the Plate Surfaces and in the Rolling Direction of the Plate. (a through d progresses from top to bottom)

HEAVY SECTION STEEL

F. A. Smidt

This portion of the Heavy Section Steel Program (HSST) emphasized studies in support of the testing program on heavy sections of reactor pressure vessel steels. The objective was to gain insight into the conditions that might lead to catastrophic fractures of this material during neutron irradiation.

During this report period, some time was spent in a literature survey of fracture mechanics and processes and mechanisms of crack nucleation and propagation.

The relationship between deformation temperature and the development of dislocation networks is being studied experimentally as a function of purity and neutron exposure of iron under another program (IDRM). Having direct bearing on the HSST program, this work should lead to a better understanding of the loss in work hardenability in the neighborhood of the crack tip, which is essential in determining the fracture toughness of a material. A paper on this subject, presented at the AIME Annual Meeting in New York, February 26-29, 1968,⁽¹¹⁾ describes some of the changes in internal stress fields (which are associated with dislocation networks) that accompany a change in the temperature at which iron is deformed. It also interprets these changes in terms of work-hardening mechanisms.

Tensile testing of iron specimens irradiated to a fast fluence of approximately 2×10^{20} n/cm² ($E > 1$

MeV) has also been initiated and should be completed within the next quarter. These data should contribute to our understanding of the modification of deformation mechanisms by higher neutron exposures. These data together with previously obtained data on iron will be included in a paper* describing the effects of neutron irradiation on the deformation mechanisms in iron.

HIGH PRESSURE STUDIES

G. L. Kulcinski

High Temperature-Pressure Annealing of Irradiated 304 SS

It has been found⁽¹²⁾ that when 304 SS is irradiated to a high fluence in a thermal reactor and heated to temperatures above 750 °C, numerous cavities form in the matrix. Furthermore, these cavities enlarge as the metal is heated to higher temperatures.

The nature of these cavities has not been clear. When they are filled with gas, roughly 30 times more gas atoms are present than predicted by transmutation calculations. When they are voids, they should disappear upon heating but do not.⁽¹²⁾ To determine more about the nature of the cavities, samples in which they had already formed, due to a 980 °C vacuum anneal, were subjected to a temperature of 820 °C and a pressure of 150,000 psi (10 kbars) for 1 hr

* *The Japanese-American Information Exchange Meeting on Effects of Irradiation in Iron and Steel is to be held in San Francisco, June 23-26, 1968.*

while immersed in NaK. A post-test electron microscopy examination indicated that the high pressure treatment failed to collapse the cavities but did cause changes in the size and distribution of the cavities. Detailed results are published elsewhere in this report.⁽¹²⁾ The conclusion of the microscopy study is that the cavities are filled with gas and that they may be properly called bubbles in the solid.

Aside from the importance of the above conclusion, another interesting fact is that these samples were successfully contained in a hydrostatic medium at 820 °C under such a large pressure.

To study the effects of pressure annealing on microscopic defects produced by irradiation, it is extremely important that the sample be subjected to no shear stresses while under pressure. Defects such as vacancies, interstitials, or dislocations generated by such shear components would interfere with (and complicate) the interpretation of those defects already present due to irradiation. To insure that an external stress is transmitted hydrostatically to a sample, the sample is usually immersed in a substance that remains a liquid during the entire time of the test. At room temperature, this can be accomplished by using various organic fluids up to ~300,000 psi (~20 kbars). However, as the temperature is raised above 300 to 400 °C, the organics decompose or polymerize, thus forcing the use of other liquids. Liquid metals such as Li, Na, K, NaK, Ga, or Hg have much better

thermal stability than organics, but Ga and Hg are extremely corrosive at high temperatures and attack iron or iron alloys quite readily above 600 °C.^(13,14) NaK was picked because it is liquid at room temperature (mp = -11 °C) and is also relatively unreactive to stainless steel at temperatures to 900 °C.⁽¹⁴⁾

A schematic of the pressure cell assembly used is shown in Figure 12.19. The samples, contained in a perforated tantalum basket, were encapsulated in a NaK-filled can. This can was placed into a preassembled pressure cell (Figure 12.19), which was then placed into the tungsten-carbide die of a piston-cylinder high pressure device, described previously.⁽¹⁵⁾ As the pressure cell was crushed under an advancing tungsten-carbide piston, the can collapsed and increased the pressure in the NaK. This pressure was then transmitted hydrostatically to the sample.

The pressure was first raised to 150,000 psi (10 kbars), and the temperature then raised to 820 °C. After 1 hr at these conditions, the temperature was lowered at a rate of approximately 300 °C/min, and the pressure was released. Figure 12.20 shows the can assembly before and after the test. Although there was some shortening of the can, no NaK leakage was evident.

A measure of the hydrostaticity of the pressure transmitting media is shown in Figure 12.21, where the microstructure of the steel is shown before and after the test. The white spots are bubbles and the black spots are precipitates. The

Neg 0680861

FIGURE 12.19. Pressure Cell Assembly for Annealing Irradiated 304 SS at 820 °C and 10 kbars (150,000 psi)

relatively low concentration of dislocations in the compressed sample is proof that there are few, if any, shear stresses acting on the sample.

An increased amount of precipitate is also noted in the pressure annealed sample (Figure 12.21B); the association of bubbles with precipitates is much more prevalent in this sample than in the vacuum annealed case (Figure 12.21A).

The significance of the microstructure in the pressure annealed sample is currently under investigation.

IN-REACTOR CORROSION OF ZIRCONIUM ALLOYS

A. B. Johnson

The in-flux corrosion behavior of three lots of Zircaloy-2 was extensively characterized in pH-10 LiOH, ~1 ppm O₂.⁽¹⁶⁾ The specimens, exposed in the G-7 loop of the Engineering Test Reactor, corroded at greatly accelerated rates. After the G-7 loop coolant was converted to pH-10 NH₄OH <0.05 ppm O₂, one set of specimens exposed in the pH-10 LiOH environment was transferred to

Neg 0670478

*FIGURE 12.20. A Comparison of Collapsible Can Assemblies
Before (Left) and After (Right) a High Pressure, High
Temperature Test*

Neg 5254-C

FIGURE 12.21A. Microstructure of 304 SS Irradiated to $6 \times 10^{21} \text{ n/cm}^2$ ($>1 \text{ MeV}$) at 290°C and Subjected to a 980°C , 1 hr Vacuum Anneal

Neg 5493-D

FIGURE 12.21B. Microstructure of Sample in 12.21A After a 1 hr, 10 kbar (150,000 psi), 820°C Anneal in NaK

the ammoniated coolant. Corrosion of the transferred specimens continued at the high rates established in the high-oxygen lithiated environment. Recently, specimens of the three Zircaloy-2 lots were exposed in the ammoniated G-7 loop environment without prior exposure in the lithiated system. The samples were fabricated in the form of tensile specimens from the same stock used in the previous G-7 loop tests.⁽¹⁶⁾ However, all specimens in the previous tests were prefilmed three days in water at 300 °C. The conditions of the 6-cycle G-7 loop exposure are summarized in Table 12.10.

TABLE 12.10. G-7 Loop Exposure Conditions

Temperature, °C	270-280
Time at Temperature, days	131
ETR Cycles	87 thru 92
Flux, n/cm ² sec, >1 MeV	9.4 x 10 ¹³
Fluence, n/cm ² >1 MeV	1.1 x 10 ²¹
Fluence, n/cm ² sec, thermal	2.6 x 10 ²¹
Coolant	pH-10 NH ₄ OH
O ₂ , ppm	<0.05
H ₂ (mean), cm ³ /kg	45
C _S -138, dis/min ml	2 x 10 ⁴
NH ₃ , ppm	10

The 6-cycle Zircaloy-2 specimens were exposed in as-etched and pre-filmed surface conditions. The metallurgical and surface conditions are summarized in Table 12.11.

Control samples for certain metallurgical and surface conditions were

TABLE 12.11. Metallurgical and Surface Conditions

As-Etched Surface Conditions
As-Fabricated ⁽¹⁶⁾
Alpha Annealed (8 hr, 760 °C)
Beta Quenched (10 min, 1010 °C)
Prefilmed Surface Conditions (As-Fabricated)
300 °C, 3 days - 13 mg/dm ² (Lots AT & CT only)
400 °C, 3 days - 24 mg/dm ²
400 °C, 210 days - 192 to 258 mg/dm ²

exposed in an out-of-reactor autoclave operated at the G-7 loop temperature in pH-10 NH₄OH. Weight gains for the in-flux and out-of-reactor specimens are summarized in Figure 12.22.

Weight gains on as-etched G-7 loop specimens were only slightly higher than weight gains on the controls. In-flux weight gains were not affected by heat treatment, nor did in-flux corrosion resistance on as-etched specimens vary significantly as a function of Zircaloy-2 lot.

Corrosion rates were accelerated on all prefilmed G-7 loop specimens, but the response varied with the pre-film treatment. We are attempting to resolve uncertainties in the in-flux weight gains on 400 °C, 72 hr specimens by metallography. The least accelerated of the three prefilm conditions appears to have been the 300 °C, 73 hr surface condition. Corrosion rates were markedly accelerated on specimens with thick films formed out-of-reactor (400 °C, 210 days). The corresponding out-of-reactor,

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FIGURE 12.22. Heat Treatment and Prefilm Effects
Exposure: G-7 Loop; pH-10 NH_4OH ; 131 Days;
 $1 \times 10^{21} \text{ n/cm}^2$ ($>1 \text{ MeV}$) Est.

thick filmed specimens showed a "memory effect" in the transfer from 400 to 280 °C. A memory effect did occur for 400 °C thin film (72 hr) specimens; this agrees with the observations of Kass⁽¹⁷⁾ that the memory effect occurs only on specimens with posttransition oxides.

The high rates on the 400 °C thick-film specimens suggest that the high rates on the in-reactor specimens transferred from pH-10 LiOH to pH-10 NH_4OH were probably due to the thick films on the specimens at the time of transfer.

The adverse effect of prefilming on in-reactor corrosion in pH-10 NH_4OH agrees with previous G-7 loop results.⁽¹⁸⁾ The explanation for the accelerated corrosion on transferred and prefilmed specimens may involve the generation of local chemistry effects in the oxides.⁽¹⁹⁾ The thick, porous oxides appear to promote the generation and/or entrapment of oxidizing species.

Weight gains are compared in Table 12.12 as a function of G-7 loop environment for specimens with the same prefilm treatment (300 °C) and fluence ($\sim 1 \times 10^{21} \text{ n/cm}^2$, $>1 \text{ MeV}$).

TABLE 12.12. In-Flux Weight Gains in the G-7 Loop

Alloy Lot	pH-10 LiOH, ~ 1 ppm O_2	pH-10 NH_4OH , < 0.05 ppm O_2
AT	250	50
CT	350	62
HT	225	-

The higher corrosion rates in the lithiated system are probably not caused by the lithium content of the coolant. There is evidence⁽¹⁸⁻²²⁾ that dissolved oxygen in the coolant increases the in-flux corrosion rate. Comparative experiments at ~ 1 ppm and < 0.05 ppm O_2 are planned to test the oxygen hypothesis. The experiments will be conducted in the north loop of the Advanced Test Reactor.

Hydrogen absorption is compared in Table 12.13 as a function of Zircaloy-2 lot, surface pretreatment, heat treatment and G-7 loop environment.

In the pH-10 NH_4OH environment, lot effects are not pronounced although they were significant in the pH-10 LiOH environment. Hydrogen pickups were higher for specimens prefilmed at 400 °C than for corresponding as-etched specimens and specimens prefilmed at 300 °C. However, the thick out-of-reactor film promoted a remarkable increase in the in-reactor hydriding rate, corresponding to a pickup of nearly 100% of the hydrogen generated by in-reactor corrosion of the specimen. Transfer of specimens from the lithiated to the ammoniated G-7 loop environment also resulted in a substantial increase in hydrogen absorption for the three lots of Zircaloy-2.⁽¹⁶⁾ Entrapment of hydrogen from the hydrogen-rich ammoniated coolant in the thick, porous oxides offers the most likely explanation for the observed hydrogen absorption characteristics of the thick-film specimens.

TABLE 12.13. Zircaloy-2 Hydrogen Absorption in the ETR G-7 Loop

Lot	Specimen Condition	In-Flux Hydrogen Absorption					
		pH-10 NH_4OH			pH-10 LiOH		
		ppm	mg/dm ²	%	ppm	mg/dm ²	%
HT	As-Etched, As-Fabricated	24	1.5	51			
HT	As-Etched, β Quenched	15	0.9	31			
HT	Prefilmed, 400 °C, 72 hr	49	3.1	(a)			
HT	Prefilmed, 400 °C, 210 days	501	30.1	97			
AT	Prefilmed, 400 °C, 72 hr	28	1.7	(a)			
AT	Prefilmed, 300 °C, 73 hr	14	0.9	14	46	2.8	10
CT	Prefilmed, 300 °C, 73 hr	12	0.8	10	23	1.4	4

(a) Weight gain not available.

FAST REACTOR SUPPORTING STUDIESFAST NEUTRON MECHANISMS

R. P. Allen

The objective of this program is to determine the effects of fast neutron irradiation and in-core environmental and service conditions on fundamental aspects of hydrogen and helium behavior in fast reactor alloys. Special nonsteady-state gas permeation and evolution techniques are currently being developed and used to investigate the effects of irradiation, composition, deformation, stress gradients, phase instabilities, surface condition, and desorption environment on the normal hydrogen diffusion, occlusion, and effusion characteristics of the austenitic stainless steels.

In a previous quarterly progress report,⁽⁵⁾ a nonsteady-state gas permeation technique was described that is quite valuable for simultaneously determining equilibrium hydrogen solubilities and diffusivities at a constant temperature. From these data, inferences can be drawn regarding the trapping of hydrogen atoms at defect sites in the metal lattice. The method is such that it is readily suited to use for in-pile measurements. However, because of the special fabrication required, the thin, disc-shaped, membrane used for the measurements is not optimum for screening tests. Consequently, other techniques, which can use small pieces cut from irradiated tensile specimens or other irradiated hardware, are being de-

veloped for screening tests. These are discussed in the following sections.

Isothermal Gas Evolution Techniques

Gas evolution or "hot vacuum extraction" techniques can be used to determine hydrogen solubilities, diffusivities, and departures from normal behavior occasioned by anomalous hydrogen interactions.⁽²³⁾ For diffusivity determinations, hydrogen-containing material is heated to a suitable temperature either in a vacuum or at a constant hydrogen pressure, with the resulting hydrogen effusion rate measured as a function of extraction time. Diffusion coefficients can be calculated directly from these isothermal rate data by use of the appropriate differential solution of Fick's second law of diffusion for the particular sample geometry employed. Exceptional care must be exercised, however, to ensure that the apparent diffusivities determined actually reflect matrix diffusion and not a rate limiting surface process or other experimental artifact.⁽²⁴⁾

Isothermal evolution techniques have also been specifically adapted to study the effects of surface condition and desorption environment on the kinetics and mechanisms of the hydrogen effusion process as it relates to the removal of neutronically-generated hydrogen from fast reactor core materials.

The sample geometry used for this technique must be one for which appropriate boundary conditions to Fick's

law can be specified. Since almost any regular shape will meet these conditions, irradiated specimens can be recovered from unrelated experiments (i.e., discs cut from the grip section of irradiated tensile specimens) without need for a special irradiation experiment.

Equilibrium hydrogen solubility values are obtained by (1) equilibrating specimens with hydrogen under carefully controlled temperature and pressure conditions, followed by rapid quenching to preserve the equilibrium gas content, and (2) extracting and measuring the dissolved hydrogen. Phase boundary of other rate-affecting kinetic processes are important only insofar as they prevent the attainment of equilibrium during the hydrogen saturation period. The only requirement for the extraction step is that the total gas content be accurately determined. This method of obtaining hydrogen solubilities is capable of high sensitivity and has a number of inherent advantages over the more common Sieverts method, especially where small samples or low gas contents are involved.⁽²⁵⁾ The sample geometry employed is unimportant in this method of determining solubility. Any shape of specimen can be used.

Nonisothermal Gas Evolution Techniques

The initial emphasis has been on the development and evaluation of non-isothermal vacuum effusion techniques where the sample is heated at a constant rate and the effusion flux is

concurrently determined as a function of both temperature and extraction time. The primary advantage of this approach is that it provides a simple, rapid means of determining the qualitative effects of various material and environmental conditions and variables on the combined hydrogen diffusion, occlusion, and effusion characteristics of materials over an extended temperature interval. Therefore, the results serve to identify those particular effects and temperature ranges that merit further investigation by use of other and more quantitative-permeation and isothermal-effusion methods.

The types of effusion rate versus temperature curves generated during typical nonisothermal effusion experiments are illustrated in Figures 12.23 and 12.24 for a set of polished discoid specimens, thermally-charged with deuterium and degassed under identical conditions. The effusion flux was measured directly with a mass spectrometer type leak detector (CEC 27-120B) modified for hydrogen and deuterium service. The use of deuterium rather than hydrogen eliminated background problems at the higher extraction temperatures.

Figure 12.23 compares the non-isothermal deuterium effusion characteristics of annealed AISI types 316 and 348 SS. Neither curve is representative of a simple, matrix-diffusion controlled gas evolution process, and both show distinct, temperature-sensitive effusion stages. Although the particular mechanisms involved have not yet

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FIGURE 12.23. Characteristic Deuterium Effusion Curves for AISI Types 316 and 348 SS

been identified, the data illustrate both the significant effect that small differences in alloy composition can have on hydrogen behavior and the extreme sensitivity of the nonisothermal effusion technique to these differences.

Figure 12.24 similarly shows the effect of room temperature deformation on the nonisothermal deuterium effusion characteristics of AISI 348 SS. Hydrogen evolution appears to be enhanced rather than retarded by the deformation treatment, and the higher temperature maxima of the annealed state are eliminated or obscured. Here also, permeation and isothermal effusion studies are required to completely clarify the observed behavior, but the nonisothermal approach has

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FIGURE 12.24. Effect of Postcharging Cold Deformation on the Deuterium Effusion Characteristics of AISI Type 348 SS

served to both disclose the effect and reveal its essential characteristics.

EBR-II IRRADIATIONS

J. J. Holmes and R. E. Robbins

The objective of this program is to determine the combined effects of environment and fast-reactor irradiation on the mechanical and physical properties of candidate fast-reactor cladding and structural alloys. The current program is specifically directed at providing a basis for selection of optimum alloys for FFTF applications and at providing a description of material behavior in fast-reactor service.

Recent studies⁽²⁶⁻²⁷⁾ have shown that irradiation of austenitic stainless steel in a fast reactor at temperatures near one-half the absolute melting temperatures creates large numbers of internal voids. The density and size of these voids are sufficient to cause as much as 7% volume change. Since the measured volume changes appear to approach the order of magnitude of 10%, this swelling effect is of major engineering significance to FFTF design. Unfortunately, the current swelling data are too fragmentary to allow the relationship between fluence, temperature, and swelling to be deduced. Consequently, PNL has initiated a study to develop the swelling information required to provide a complete and detached description of swelling in the range of temperatures, times, and fluence appropriate to the FFTF.

Both, actual density measurements and void counting utilizing the electron microscope, are being used at PNL to map out the swelling characteristics of both 304 and 316 SS. Since the swelling problem was not anticipated prior to the insertion of current EBR-II irradiation experiments, no well-planned group of controlled density specimens is available for examination. Specimens for density determination must come from mechanical test specimens, capsule parts, and various EBR-II components as they become available.

Density measurements have been made on the following:

- A) One-fourth inch sections taken from an irradiation capsule irradiated to $2 \times 10^{22*}$ n/cm² peak fluence. The 1/4 in. sections were cut as various positions along the capsule to investigate temperatures and fluence variations along the capsule (700 to 800 °F).
 - B) One inch sections taken from an irradiation capsule used for a cermet fuel irradiation (Pin 5-P-9), (700 to 840 °F).
 - C) A tensile specimen irradiated at 1000 °F $\pm$ 100 °F to 2×10^{22} n/cm².
- All the above items are in the annealed condition. Items A and B are 304L while Item C is 304.

The mean density on the unirradiated tensile material was 7.927 ± 0.003 g/cm³, where the limits given refer to the 95% confidence limit. After irradiation, the density of Item A was found to be 7.913 ± 0.004

* EBR-II estimated

g/cm³. The difference between these two mean values is 0.014 ± 0.007 g/cm³. In this case, the density decrease may be caused by irradiation damage or by surface effects in the irradiated samples that are not present in the control. The irradiated specimens were exposed in flowing sodium for 156 days while the control tube was not. This sodium exposure may have changed the surface chemistry sufficiently to cause the observed density change. In addition, the control tube may have slightly different initial chemistry than the irradiated material.

Results from the 2.8×10^{22} n/cm² capsule (Item B) indicate a maximum decrease in density of 0.34%, the actual measured density being 7.898 g/cm³. In this latter capsule, the change in density corresponds to the fluence and temperature along the

capsule; for this reason it appears to be unambiguously caused by irradiation damage. Density measurements on Item C reveal a 0.24% decrease in density.

A summary of current swelling data from PNL and other laboratories is given in Table 12.14. An inspection of this table reveals the absence of data for 304 SS at high exposures at temperature above 1000 °F and the lack of data for 316 at the lower exposures and temperatures above 1040 °F. Efforts are in progress to obtain an EBR-II control rod thimble (304L) that has reached about 4.5×10^{22} total fluence. This thimble will allow swelling determination on 304 SS irradiated in the temperature range of 700 to 800 °F. In addition, GE-APO has agreed to supply PNL with 304 SS specimens irradiated in the EBR-II to fluence

TABLE 12.14. Summary of Stainless Steel Density Data

Source	Material	Irradiated Temperature	Condition	Fluence	$\frac{\Delta V}{V}$, %	Density	Method
PNL, Tensile Spec. Capsule A Capsule B (5-P-9)	304	1000 $\pm$ 100 °F	Ann	1.9×10^{22}	0.24		D
	304L	750 $\pm$ 50 °F	Ann	1.7×10^{22}	0.14	7.913 ± 0.003	D
	304L	760	Ann	2.3×10^{22}	0.13	7.916 ± 0.002	D
	304L	786	Ann	2.7×10^{22}	0.27	7.904 ± 0.002	D
	304L	819	Ann	2.7×10^{22}	0.33	7.900 ± 0.001	D
	304L	840	Ann	1.7×10^{22}	0.34	7.898 ± 0.002	D
ORNL, EBR Driver Fuel ⁽²⁸⁾	304L	844	Ann	1.2×10^{22}	0.13	7.917 ± 0.002	D
		698	Ann	0.8×10^{22}	0.07		EM
		752	Ann	1.2×10^{22}	0.15		EM
		806	Ann	1.4×10^{22}	0.17		EM
		852	Ann	1.3×10^{22}	0.16		EM
		881	Ann	0.9×10^{22}	0.08		EM
Dounreay ⁽²⁶⁾	AISI 316	842	Ann	5.2×10^{22}	0.8		EM
		950	Ann	4.5×10^{22}	1.5		EM
		1040	Ann	3.2×10^{22}	0.2		EM
		932	Ann	7.8×10^{22}	6.8		EM, D

levels of from 1×10^{22} to 2.5×10^{22} at temperatures from 700 °F to 1200 °F. It is anticipated that studies on the thimble and GE-APO specimens will allow a rudimentary description of swelling as a function of fluence and temperature to be given.

Aging furnaces have been installed and are operating to provide a facility for obtaining control (unirradiated) specimens for EBR-II irradiations. The facility will also be used to carry out a program for determining the effect of aging time and temperature on the microstructure and tensile, uniaxial stress-rupture, and biaxial stress-rupture, properties of AISI Types 304 and 316 SS. All aging will be performed in sodium at temperatures of 800, 900, 1000, 1100, 1200, and 1400 °F for times of 20, 200, 2160, 6480, and 12960 hr. The last three aging times are not firm because they will depend on the in-reactor time of the subassemblies in the EBR-II irradiation program.

Ten EBR-II fuel jackets, without the fuel elements, have been received from ANL for evaluation. The irradiation temperature profiles of the jackets range from 740 to 945 °F, and the range of estimated cladding fluences is from about 0.92 to 1.32×10^{22} n/cm². Density measurements and biaxial stress rupture

tests will be performed on selected sections from the jackets. Testing will be performed at 1100 and 1200 °F for rupture times of 10, 100, and 1000 hr.

FUEL CLADDING CHARACTERIZATION

M. M. Paxton and J. A. Yount

The purpose of this program is to determine the lot-to-lot variation in two lots of AISI 304 SS, seamless tubing 0.250 in. OD x 0.016 in. wall, that will be used in future fuel pin experiments.

The testing program as presented previously⁽⁵⁾ is progressing smoothly. The chemical analyses of tubing Lots "E" and "F" have been completed by Lukens Steel Company. The analyses (Table 12.15) agree with those provided by the manufacturer.

The metallographic examination of the as-received tubing has been completed. Tubing Lot "E" has undergone intergranular attack on the OD surface. The reason for this attack is unknown. Both "E" and "F" lots were purchased under the same specifications; however, tubing Lot "E" has a grain size approximately ASTM #6; whereas, Lot "F" has a grain size approximately ASTM #10. In addition, Lot "F" contains 5 to 7 times more nonmetallic inclusion as Lot "E". Fabrication histories are being obtained from the manufacturers to

TABLE 12.15. Chemical Composition

	<u>C</u>	<u>Mn</u>	<u>P</u>	<u>S</u>	<u>Si</u>	<u>Ni</u>	<u>Cr</u>	<u>N₂</u>
Lot "E"	0.056	1.73	0.038	0.011	0.47	9.90	18.50	0.10
Lot "F"	0.052	1.72	0.024	0.013	0.48	9.21	18.75	0.057

help establish the cause for these differences. Typical photomicrographs are shown in Figure 12.25.

Room temperature uniaxial tension

tests on full size tubular sections have been completed for Lots "E" and "F", and the results are summarized in Table 12.16.

Neg 467-1724A

80X

Neg 467-1725A

80X

Neg 467-1753A

80X

Neg 467-1755A

80X

FIGURE 12.25. A Comparison of Microstructure of Tubing Lot "E" (Top) and Tubing Lot "F" (Bottom) Type 304 SS Fuel Cladding

TABLE 12.16. Tensile Properties of Tubing Lots "E" and "F" at Room Temperature

<u>Spec. No.</u>	<u>0.2% YS, ksi</u>	<u>UTS, ksi</u>	<u>UE, %</u>	<u>Total Elongation, %</u>
1E	41.36	95.34	68.05	87.69
2E	44.49	102.54	65.00	84.46
3E	43.64	99.58	69.99	91.06
1F	49.32	104.66	65.74	82.80
2F	46.61	102.80	68.62	79.23
3F	48.73	104.66	66.27	81.03

Room temperature burst tests have been completed on "E" tubing only (Table 12.17). Burst test at 1800 °F have been completed on defective and nondefective specimens from Lots "E" and "F". Specimens are classified defective if there is a surface defect more than one mil deep, as determined by NDT methods. The data are summarized in Table 12.17.

The presence of a defect apparently has no effect on burst strength

TABLE 12.17. Burst Data for Defective and Nondefective "E" and "F" Tubing

<u>Tube Lot</u>	<u>Test Temp</u>	<u>Burst Pressure, psi</u>	<u>Hoop Stress, psi</u>	<u>Measurable Post OD</u>
E	RT	10,700	72,870	0.300
		10,700	72,870	0.305
		10,700	72,870	0.298
		10,800	73,550	0.304
		10,800	73,550	0.305
E	1800 °F	1,365	9,296	--
		1,390	9,465	0.316
		1,395	9,500	0.303
		1,410	9,620	0.305
		1,450	9,865	0.294
E (Defective)	1800 °F	1,375	9,365	0.327
		1,380	9,400	0.321
		1,385	9,435	0.325
		1,420	9,670	0.305
		1,425	9,705	0.315
		1,440	9,805	0.303
F	1800 °F	1,170	7,968	0.305
		1,180	8,036	0.292
		1,185	8,070	0.315
		1,225	8,342	0.314
		1,180	8,036	0.317
F (Defective)	1800 °F	1,275	8,660	0.295
		1,150	7,832	0.295
		1,195	8,138	0.310
		1,150	7,832	0.317
		1,200	8,172	0.292

at 1800 °F. Some defective specimens ruptured in the defect zone and others did not.

To accommodate the vast characterization program previously outlined,⁽⁵⁾ it has been necessary to further modify the CANEL test stands with a resultant increase in testing capability. The plan remains to make full utilization of the CANEL biaxial test stands. However, close inspection of those frames has shown certain parts damaged and others missing entirely. Instead, it was decided to substitute six \$167 Skutt ceramic kilns, each operating at one of six test temperatures. Each furnace is controlled by the same instrument configuration that was supplied with the CANEL test frames, but due to the increase in furnace size, eight specimen retorts may be installed per Skutt furnace instead of one per CANEL frame. The essential features are outlined as follows:

	<u>CANEL</u>	<u>Skutt</u>
Retorts	12	48
Specimens	48	384 (maximum) 192 (normal)
Furnaces Required	12 (6 available)	6
Recorder/Controllers Required	12 (6 available)	6
Temperature Profile	±5 °F/3.75 in.	±3 °F/6 + in.

The cost of purchasing all six Skutt kilns is roughly equivalent to replacing two of the original CANEL furnaces.

STRUCTURAL MATERIALS AND FUEL CLADDING STUDIES

R. W. Barker

The objective of this program is to determine the combined effects of environment and fast reactor irradiations on the mechanical properties of candidate fast-reactor cladding and structural alloys. The current program is specifically directed at providing a basis for selection of optimum alloys for FFTF applications and at providing a description of material behavior in fast-reactor service.

Pre- and postirradiation rupture properties will be determined on FFTF prototype fuel clad material (0.208 in. OD x 0.008 in. wall tubing). Types 304 and 316 SS are to be studied.

A limited quantity of unirradiated stress-to-rupture data for the Type 304 SS has been developed for 1400 °F (760 °C) test temperature in a helium atmosphere. The condition of primary interest is the as-received condition, containing a slight degree of cold work from the final fabrication step. The as-received material has been tested at stresses sufficient to cause rupture in 0.1 to 139 hr (Figure 12.26). Identical material, annealed at 1950 °F (1066 °C) for 30 min in vacuum, has been tested at stresses sufficient to cause rupture in 0.2

to 78 hr. Ten additional tests on annealed Type 304 SS, report previously,⁽²⁹⁾ were stressed at 10,000 psi at 1400 °F (760 °C), with failure occurring in all 10 tests in 88 to 120 hr. These data, however, are not recorded on the accompanying plots. The data clearly establish the tendency of the as-received and annealed curves to merge at rupture times of about 100 hr. The annealed stress-to-rupture curve indicates that the material is representative of typical Type 304 SS, with respect to rupture stress and rupture times.

It can be observed in Figure 12.27 that the rupture strain for the annealed material is significantly less than for the as-received material of those specimens exhibiting intergranular failure. The same phenomena is observed in the room temperature tensile values (Table 12.18) where the as-received material (slightly cold worked) possesses 50% greater ductility than the annealed material.

The decreased ductility of the annealed material has been attributed to the selective grain growth occurring during annealing of the only slightly cold-worked material. A comparison of the grain size of the two conditions is shown in Figure 12.28. (Note, in the annealed material, bands of fine grains are adjacent to grains whose diameters are approximately equal to the wall thickness.)

Post-test comparisons of grain size and microstructural characteristics with untested material can be made in Figure 12.29 (as-received condition) and in Figure 12.30 (annealed

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FIGURE 12.26. Stress to Rupture Properties of As-Received (slightly cold worked) and Annealed Type 304 SS Prototype FFTF Fuel Cladding

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FIGURE 12.27. Rupture Strain of As-Received (slightly cold worked) and Annealed Type 304 SS Prototype FFTF Fuel Cladding

TABLE 12.18. Tensile Properties of As-Received and Annealed Type 304 SS Tubular Material, 0.208 in. OD x 0.008 in. Wall

<u>Material</u>	<u>0.2% Yield Stress, psi</u>	<u>Ultimate Stress, psi</u>	<u>Uniform Elongation, %</u>	<u>Total Elongation, %</u>
304 As Received	40,000	100,000	65	76
304 As Received	41,000	99,400	66	73
304 Annealed	35,800	89,000	43	52
304 Annealed	34,800	88,400	37	45

Neg 0680842-5

FIGURE 12.28. A Comparison of the Grain Size of the As-Received (top) and Annealed (bottom) Type 304 SS Fuel Cladding

Neg 0680842-4

FIGURE 12.29. Grain Size and Microstructure of As-Received Type 304 SS Prototype Fuel Cladding in Untested Condition and After Rupture at 1400 °F (760 °C).

condition). The following general observations can be made for both material conditions.

- The growth of carbides increases with time-at-temperature. Cold work present in the as-received test material has had no observ-

able effect on the size or location of the carbide precipitate.

- For the test times available, the nominal grain size remains unchanged throughout the test.

Further examination will proceed on these materials and on additional

Neg 0680842-3

FIGURE 12.30. Grain Size and Microstructure of Annealed Type 304 SS Prototype Fuel Cladding in Untested Condition and After Rupture at 1400 °F (760 °C)

specimens as ruptured specimens become available.

Ultimately, stress-to-rupture testing will be performed to establish base curves for Types 304 and 316 SS at temperatures of 1400, 1200, and 900 °F (760, 649, and 482 °C).

TEST FACILITIES

R. H. Todd

To comprehensively investigate the effects of irradiation and environment on candidate, structural and cladding materials for the FFTF, a

facility was needed that could provide the necessary test environments. Such facility for testing irradiated specimens in sodium and controlled gas atmospheres is now under construction, and as of March 1, 1968, was 85% complete. With construction 3% ahead of schedule, most of the remaining work involves installation of the heating and air conditioning equipment (due for delivery in March). The piping trenches will be covered with concrete which, after curing, will be covered with floor tile. Present plans call for the main transformer for this facility to be moved to a new location next to the building. Full power cannot be supplied to this facility until this move is made, so it is expected in the very near future.

Tests performed by the test stands, temporarily located in the mezzanine work shop, have given the following results in air environment (Table 12.19).

The complete analysis of these tests will be made by A. J. Lovell. The first of two test stands for

static sodium environment has been aligned to provide uniaxial stress on the specimen with less than 10% bending moment. All parts of the machine had to be cleaned and realigned; also some parts of the sodium containment vessel had to be redesigned to meet the above specification. Although more stringent than the 15% limit prescribed by the ASTM specification for conducting creep and time-for-rupture tension tests of materials (ASTM E 139-66T), the 10% limit eliminates a great deal of scatter in the test results. A pair of short term tests will be run with Type 304 SS to determine the proper stress level for a test of about 1000 hr duration at 650 °F (1202 °C). Emphasis will be placed on the long term tests, since the effect of sodium-versus-inert gas environment is nearly negligible in short term tests. As soon as the second static sodium test stand can meet testing standards, it also will be placed in service on this program.

Generally, the ASTM recommended practice for creep tests is a set of

TABLE 12.19. Creep Tests of Type 304 SS at 1200 °F in Air

<u>Material</u>	<u>Prior History</u>	<u>Diameter, in.</u>	<u>Stress</u>	<u>Test Temp, °F</u>	<u>Hr to Rupture</u>	<u>Total Elongation, %</u>
304 SS	20% Cold Worked Sol Treated 1 hr 1950 °F	0.160	25,000 psi	1202	113.9	54
304 SS	As-Received; Sol Treated 1 hr 1950 °F	0.125	25,000 psi	1202	60.3	45
304 SS	As-Received; Sol Treated 1 hr 1950 °F	0.125	22,000 psi	1202	625	41

operating rules that actual tests attempt to meet. Much useful data can be obtained without meeting the requirements of the ASTM specification, but such data have an inherent scatter difficult to deal with. Two of the most important parameters are the axiality of the applied stress and temperature control. The requirement for axiality of the applied stress (bending moment) is being exceeded in this laboratory as described above. The problem of temperature control has been solved with the invention of a furnace liner filled with sodium. The ASTM specification calls for a temperature uniformity of ± 3 °F along the length of the specimen. The temperature profile of most furnaces make this condition very difficult to meet, and even more difficult to maintain for the duration of a test. The liner, placed in service here, is a tubular container made of stainless steel and filled with sodium. The liner fits inside the standard furnace and admits the specimen, grips, and pullrods through the ID of the liner. The liner and the specimen train do not touch one another. With suitable baffling and use of loose packing materials to restrict the flow of air through the channel, this new liner has reduced the temperature variation across a 2 in. specimen to less than 1 °F total variation. Operator time required to achieve temperature balance (in the region of 1200 °F) has been reduced to essentially zero. To date, the data from the two tests run with this liner show a definite reduction in the amount of scatter in the plot of creep data. Efforts are being made to provide each test fur-

nace with a liner of this type as soon as possible.

The support section of the liquid metal loop has been assembled in working position with controllers and recorders attached. This section contains one unshielded test leg, along with the electromagnetic pumps and cold trap for circulating and purifying the sodium. Work on the supporting utilities progressed far enough to allow start-up of the sodium loop for operational acceptance tests and operator check-out. Some deficiencies were found, and these are now being corrected. Design work for the shielded test loops was completed, while design work on the radiation shield was begun in February. A date for start of construction on this portion of the sodium loop has not yet been set.

An inert atmosphere glove-box for handling high purity alkali metals has been built by Vacuum Atmospheres Corporation and will be shipped in March. This glove-box will provide a high-purity inert atmosphere with evacuable transfer port and a vacuum oven large enough to handle any of the alkali metal capsules or containers now in use on testing programs.

Recent experiments with the sodium filling apparatus with receiving vessels closed to air atmosphere have shown that helium does dissolve in liquid sodium. The solubility of helium is sufficient to cause bubbles to form when the sodium is exposed to vacuum. The possibility of the bubbles being formed by sodium vapor was considered, but sodium vapor pressure at the highest temperatures involved

(600 °F) would support a column of sodium less than 1 mm high; whereas, the observed effects require pressure to support a column of sodium several centimeters high. Special procedures for outgassing the helium from liquid sodium and for the removal of bubbles entrained in liquid sodium have been developed. Since the problem is of sufficient magnitude, if the special procedures are not followed, accurate measurements of sodium within closed vessels cannot be done reliably.

The vacuum manifold for the interior of the prototype cell has been completed and installed, but work in and around the prototype cell is still being held up by building construction. A prototypical testing unit, ready for installation in the cell and for attachment to the vacuum manifold, will be installed as soon as the building construction items are cleared up.

RADIATION EFFECTS ON METALS — Q5 PROGRAM

T. K. Bierlein, E. R. Bradley,
J. L. Brimhall, H. E. Kissinger,
G. L. Kulcinski, T. J. Larson,
B. Mastel, and F. A. Smidt, Jr.

ELECTRON MICROSCOPY

In progress are annealing studies of molybdenum that contains various amounts of carbon and which had been irradiated to a fluence of 4×10^{19} n/cm² at 400 °C. The grouping of the small clusters of loops into aggregates or "rafts" was previously reported. Figure 12.31 illustrates the transformation from the "rafts" to large interstitial loops. A combina-

tion of prismatic glide and conservative climb are necessary for two prismatic loops to come together and coalesce or annihilate. Since dislocation climb, a diffusion controlled process, must occur in order for the loops to coalesce, there is little indication of coalescence before 700 °C. Significant coalescence of the small loops within the "rafts" is occurring at 700 and at 800 °C; there is no evidence of any "rafts" remaining. The large loops observed after the 800 °C anneal are interstitial in nature and correspond approximately to the size of the original "rafts". This is strong evidence that the small clusters in the "rafts" are in fact small interstitial loops. Since the total line length, and hence the energy, of a large dislocation loop is much less than the combined length of a "raft" of many small loops, there is a considerable driving force for this mechanism.

Nickel foils of 99.997% purity were irradiated in the same capsule as the molybdenum described above. In addition, nickel samples were also irradiated in a helium filled capsule at a calculated temperature of 400 to 450 °C, and to a fluence of 4×10^{19} n/cm².

Figure 12.32 shows the presence of voids or pores in the nickel after irradiation in the NaK filled capsule. Pores, similar both in size and shape, were seen in the nickel irradiated in the helium filled capsule. Stereo-electron microscopy shows that the pores are not associated with either surface, but lie within the foil. There are several additional reasons

Neg 5349C

5481A

5291B

96,000X

(a) 600 °C

(b) 700 °C

(c) 800 °C

FIGURE 12.31. Molybdenum Containing 100 to 100 ppm Carbon Irradiated to a Fluence of 4×10^{19} n/cm² at a Temperature of 400 °C and Given Postirradiation Anneals at the Indicated Temperatures. (These micrographs illustrate the development of large loops from "rafts" of small loops with increasing annealing temperature.)

why these pores are interpreted to be voids within the foil and not surface pits:

1. The change in contrast at thickness contours is consistent with a pore decreasing the effective thickness
2. An increase in pore density with increase in foil thickness is observed
3. No such pores are present in unirradiated control specimens.

The pores present in the foils irradiated in NaK to a fluence of 5×10^{19} n/cm² varied in size from 50 to 150 Å and had a density of 4×10^{15} /cm³. Based on average size

of 100 Å, the pore volume fraction is 2×10^{-3} . The pores that formed in samples irradiated to a fluence of 4×10^{19} n/cm² at a slightly higher temperature in the helium environment had a similar size, but a factor of 2 lower density.

The pores have a definite geometric shape. Careful measurements show that they are not cubic. In a grain where the foil normal was near [110], the edges of the pores lie parallel to projections of the <110> directions. These measurements suggest that the true shape is an octahedron formed by {111} planes.

Neg 5504A

100,000X

FIGURE 12.32. Voids Produced in High Purity Nickel by Neutron Irradiation at 400 °C to a Fluence of 5×10^{19} n/cm² ($E > 1$ MeV)

Small zones $\approx 1000 \text{ \AA}$ wide adjacent to the grain boundaries and to the incoherent twin boundaries were denuded of voids. There appeared to be no denuding or interaction of the voids with the radiation produced dislocation structures.

There are two possible explanations for the development of pores: 1) they are gas bubbles resulting from agglomeration of gases produced by neutronic reactions, or 2) they are voids resulting from condensation of vacancies produced by displacement collisions. A calculation of the amount of helium produced by fast (n, α) reactions in pure nickel shows that approximately 0.1 ppm He should have been produced

during the irradiation. If the pores are all helium filled and if the gas pressure is balanced by the surface tension, then the gas in the pores would amount to the equivalent of approximately 700 ppm helium atoms. Accordingly, it is concluded that the pores are essentially empty, and therefore are voids.

The voids must develop from the coalescence of vacancies produced by the displaced atoms. For 500 vacancies produced per primary knock-on, the number of vacancies produced after $5 \times 10^{19} \text{ n/cm}^2$ is $5 \times 10^{21} \text{ vac/cm}^3$. A void density of $4 \times 10^{15} \text{ /cm}^3$ and average void size of $100 \text{ \AA}$

represents 1×10^{20} vac/cm³. Apparently there are sufficient vacancies produced to account for the pores. Isothermal annealing studies have been initiated to determine the annealing kinetics of the voids.

The most unambiguous method for determining the nature of the small defect clusters in irradiated metals is stereo-electron microscopy. This method has recently been used in the analysis of the defect clusters in neutron irradiated rhenium.

Rhenium, 99.992% pure, was irradiated to a fluence of 1.5×10^{19} n/cm² ($E > 1$ MeV) at a temperature of 550 °C. The samples in the form of foils, 10 mm diam by 0.075 mm thick, were positioned in a capsule filled with molten NaK and were maintained at a constant temperature during the course of their irradiation.

The defects produced by neutron radiation were 25 to 120 Å in diameter (Figure 12.33) and had a density of 0.5 to 1.0×10^{16} /cm³. By letting $S = 0$, the anomalous black-white images were observed, (Figure 12.34). Measurements of the images were made using a [20.0] and [11.0] reflection Gold, evaporated on the bottom surface, served as a reference point for the stereo-analysis. The samples were tilted into the kinematical condition for photographing stereo pairs. The tilt angles giving good kinematical imaging were 16° for [20.0] and 18° for [11.0], $\pm 0.5^\circ$.

The chief contribution to error was the lack of reproducibility in measurements on the parallax bar. For the location of one cluster, three different measurements varied

Neg 4891B

96,000X

FIGURE 12.33. Defect Clusters in Rhenium Produced by Neutron Irradiation at 550 °C to 1.5×10^{19} n/cm² ($E > 1$ MeV). (Kinematical conditions)

Neg 4890C

96,000X

FIGURE 12.34. Same Area as Shown in Figure 12.33 except Under Dynamical Conditions ($S = 0$).

by an amount equivalent to ± 20 Å. Errors in magnification and tilt angle are only relative and do not affect the observation of the distribution of the defects in the foil. The resultant measurements are plotted in Figures 12.35a and b for the two different reflections. The sign of

Neg 80490

FIGURE 12.35. The Sign of the Product $(\bar{g} \cdot \bar{l})$ for Bright Field as a Function of the Distance of the Defect Clusters Measured Experimentally from the Bottom Surface of the Foil Pictured in Figure 12.33 and 12.34; Each Circle Is One Defect Cluster

$\bar{g} \cdot \bar{l}$ ($\bar{l}$ = vector drawn from black to white part of image) is plotted as a function of distance from the bottom surface; the grouping of the clusters into layers is plainly evident. The analyses of the bright field and dark

field images for $\bar{g} = [20 \cdot 0]$ were consistent with each other. From the rules given by Rühle,⁽³⁰⁾ these clusters are interpreted to be predominantly interstitial in nature.

The Burger's vector of the loops was determined from the direction of $\bar{b}$. In all cases, this was $\langle 11\bar{2}0 \rangle$. Since the $\bar{b}$ cannot lie in the plane of the loop, the loops must be lying on one of the prism planes.

Previous observations of radiation effects in rhenium have shown small clusters after irradiation at 60 °C,⁽³¹⁾ but these were not identified. Large interstitial loops have been observed in irradiated zirconium after postirradiation annealing at elevated temperatures.⁽³²⁾ The present results on the nature of the defects in rhenium irradiated at 550 °C (0.25 Tm) compare favorably with results of McIntyre on copper irradiated at 65 °C (0.25 Tm).⁽³³⁾ The interstitials are evidently mobile enough to migrate and cluster together at this temperature. The vacancies are still relatively immobile and remain either isolated or as very small clusters. A few of the images indicated vacancy clusters.

The measurements made here are probably biased to a certain size range (40 to 100 Å). It is difficult to accurately locate the large clusters in the foil and the very small ones are in many cases too faint for good measurement. In this sense, it is not comparable to results of Ruhle in which measurements down to 20 Å were made.⁽³⁰⁾ It is concluded, however, that the predominant defect above 40 Å in diameter in rhenium irradiated at 550 °C (0.24 Tm) is an interstitial dislocation loop with Burger's vector $a/3 \langle 11\bar{2}0 \rangle$.

ANNEALING STUDIES ON MOLYBDENUM

Past and current results on the behavior of single-crystal versus polycrystalline molybdenum rods have been re-evaluated and integrated. The following material describes the present status of this study.

Annealing work has continued on irradiated polycrystalline rods and single-crystal samples of high purity molybdenum. Length changes and lattice parameter changes have been measured during two-hour isochronal anneals at 100 °C intervals from 100 to 900 °C. Stored energy release measurements have also been completed.

Samples were high purity molybdenum single-crystals prepared by three-pass zone-refining followed by extended annealing; a technique which has been shown to produce nearly-perfect crystals.⁽³⁴⁾ Samples of the parent rod were prepared also, with the same dimensions. Both sample types were irradiated in the identical manner to the same fluence levels, 5×10^{18} and 1×10^{19} n/cm². Post-irradiated annealing was carried out on both sample types simultaneously. Stored energy measurements were performed in a differential calorimeter with a six-junction thermopile as the sensing element.

Significant differences were observed in the length and lattice parameter changes resulting from annealing the single-crystal and polycrystalline rods. The polycrystalline rods exhibited a simultaneous length and lattice parameter recovery at

150 to 200 °C ($\sim 0.15 T_m$) amounting to about 60% recovery. Single-crystals showed no lattice parameter recovery in this temperature range, but the length change recovered about 40% between 200 and 300 °C. Polycrystalline samples released about 0.3 and 0.6 cal/g for the 5×10^{18} and 1×10^{19} n/cm² exposures, respectively. Single-crystals irradiated to 5×10^{18} n/cm² showed no perceptible stored energy release, while those irradiated to 1×10^{19} n/cm² showed two small peaks, about 0.08 cal/g at 150 °C and 0.2 cal/g at 210 to 220 °C.

Since the large stored energy release in polycrystalline samples is associated with a simultaneous recovery of length and lattice parameter changes, it is concluded that a recombination of vacancies and interstitials is taking place in these samples. Apparently, interstitials are tightly bound in the single-crystals, so that no recombination and mutual annihilation can occur. This conclusion is supported by electron microscope observations which have shown the clusters, presumably interstitial in origin, to be much larger in the irradiated single-crystals than in polycrystalline foils. The length change and corresponding small energy release in single-crystals above 200 °C must be a result of formation of vacancy clusters.

This difference in annealing behavior of single-crystals and polycrystalline samples will be the subject of continuing investigation. Since impurity content may be altered by the zone-refining treatment, single-crystals doped with specific

impurities will be examined. Also, a single-crystal has been converted into a polycrystal by cold working and pulse annealing. The results of these experiments, when completed, should demonstrate whether the observed results are a consequence of impurity content or crystal perfection.

X-RAY SCATTERING FROM VOIDS IN NICKEL

Small-Angle X-Ray Scattering from Voids Produced in High-Purity Nickel During Neutron Irradiation*

Cavities or voids 50 to 150 Å in diameter have been reported by B. Mastel and J. L. Brimhall⁽³⁵⁾ in high-purity nickel after neutron irradiation at elevated temperature. That voids in nickel should be stable to very large sizes has been shown by Sigler and Kuhlman-Wilsdorf⁽³⁶⁾ and Johnson⁽³⁷⁾ from potential energy calculations.

Inhomogeneities in electron density introduced by voids should produce X-ray diffraction effects at very small angles. Voids in quenched and aged copper have been observed by this method.⁽³⁸⁾ Similar results on quenched and aged nickel have not been obtained.⁽³⁹⁾ The existence of voids in the neutron irradiated samples examined by Mastel and Brimhall provided an opportunity to test the applicability and sensitivity of the small-angle-scattering method to the study of voids in nickel.

A portion of the irradiated foil was used for the X-ray experiments.

* H. E. Kissinger, author (submitted for publication to J. Nucl. Mater.)

The sample was a section approximately 1 by 3 mm. The effective thickness, determined by attenuation of the transmitted X-ray beam, was about 0.01 mm.

Measurements were made with a Rigaku small-angle goniometer mounted on a Jarrell-Ash microfocus X-ray generator. Crystal-monochromated copper K α radiation ($\lambda = 1.54$ Å) was employed. The X-ray beam was defined by slits, 0.3 by 1.0 mm, separated by 270 mm. A proportional counter tube having a background counting rate of less than 0.1 count per second was employed as the detector. Measurements were made at 2 min angle intervals between 10 min and 1 degree. Counts were recorded for 100 sec at each position. Sixteen independent determinations were made to average any random errors due to source or counter instability.

The observed small-angle-scattering, corrected for background, is shown in Figure 12.36. In addition to a scattering which decreases with increasing angle, three conspicuous inflections appear in the scattering curve at approximately 25, 37, and 53 ft. The spacing of these inflections suggests they may be the second, third, and fourth maxima of an interparticle interference function having a first maximum masked by the edge of the direct beam, at about 13 min of angle.

Analysis of the scattering was performed by use of Guinier's formula,⁽⁴⁰⁾

$$I(h) = I(0) \exp(-h^2 R_o^2/3)$$

where

$$h = 4\pi \frac{\sin \theta}{\lambda}$$

In this formula, $I(h)$ is the intensity scattered at angle h , and R_o is the "radius of gyration", an electron-density analog to the mechanical term.⁽⁴⁰⁾ R_o may be determined by plotting $\ln I(h)$ against h^2 and measuring the slope of the resulting line as shown in Figure 12.37.

From such an analysis an electronic radius of gyration of 49 Å is obtained. For a sphere of radius R_s ,

$$R_o^2 = 3/5 R_s^2,$$

so the observed X-ray scattering is equivalent to that from spherical particles 63 Å in radius or 126 Å in diameter. This is in excellent agreement with the diameters of the voids revealed by electron microscopy, and is convincing evidence that the voids are the source of the scattering.

Analysis of the interparticle interference is not so simple. Of the several interpretations possible^(40, pp. 140-149) the "para-crystal" model is the most direct. If an average interparticle separation and a pseudo-close-packed configuration of scattering centers exists, then the first maximum in the scattering curve becomes the Bragg peak for the lowest-order pseudo-planes in the para-crystal. For the first maximum at about 13 min of angle, an interparticle distance of about 500 Å is indicated. This leads to a particle density of $10^{16}/\text{cm}^3$. This is higher than the value of $4 \times 10^{15}/\text{cm}^3$ found by Mastel and Brimhall, but the discrepancy is not surprising in view of the simple model utilized to evaluate the interparticle interference. Failure to observe small-angle scattering

Neg 681863-1

FIGURE 12.36. Experimental Small-Angle Scattering from Irradiated Nickel Containing Voids, Average of 16 Determinations. Sample Absorption Was 0.4 of Primary Beam. Sample Had Been Irradiated with Neutrons at 380 °C to a Fluence of 5×10^{19} n/cm² ($E > 1$ MeV).

Neg 682863-2

FIGURE 12.37. Guinier Plot of Data of Figure 12.36

from quenched and aged nickel⁽³⁹⁾ then indicates either an incorrect quenching rate or an unsuitable aging treatment, as calculations show voids to be an energetically favorable configuration for vacancies.

In summary, voids in irradiated nickel are shown to serve as a source of small-angle X-ray scattering. Analysis of the scattering yields an estimate of void diameter and concentration in agreement with electron microscope measurements.

ISOBARIC-ISOCHRONAL ANNEALING EXPERIMENTS

The objects of this program are to

- Measure activation volumes of migration for Stage III - IV annealing in molybdenum
- Determine the effect of postirradiation compression on clustering of point defects
- Determine the effect of simultaneous irradiation and compression on point defects in irradiated molybdenum.

Isobaric, isochronal annealing studies on irradiated high purity molybdenum have progressed into the high pressure (20 kbar) range. Samples identical to those described previously^(41,42) have been annealed at temperatures of 25, 50, 80, 90, 110, 175, 205, and 300 °C for 2 hr under 20 kbars pressure in a 50-50 mixture of isopentane and n-pentane. NaK was used as the hydrostatic media for anneals at 350, 400, 490, and 580 °C. The resistivity of the samples was measured in liquid He after each test, and unirradiated control specimens showed no change, which indicates that the pressure was truly hydrostatic at all times.

Figure 12.38 shows the results of the isochronal tests for both the vacuum and high pressure anneals. It can be seen that the damage anneals out at a much lower temperature under pressure than in a vacuum. The displacement of the Stage III peak is ~-60 °C, and Stage V is lowered by about 225 °C.

It can be shown that, if Stage III annealing is an activated process,

the associated activation volume, ΔV , is related to the activation energy, E_m , and the pressure P in the following fashion:

$$\Delta V = \frac{E_m}{P} \left(\frac{T_2 - T_1}{T_1} \right)$$

where T_2 is the Stage III annealing temperature under pressure and T_1 is that found at low pressure. Since $E_m = 1.25$ eV,⁽⁴²⁾ then $\Delta V \approx -0.9 V^*$ where V is the molar volume. This value is almost unreasonably large for the simple process of single interstitial or vacancy migration and additional study of this effect is required before any definitive statements can be made.

ATR GAS AND WATER LOOP OPERATION AND MAINTENANCE

J. E. Minor

ATR GAS LOOP SUPPORT

Gas-Cooled-Loop Design Review

The design of the lower specimen train portion of the Gas Cooled Loop (GCL) inpile tube has been under review. The design of the specimen containers required that the original hanger-rod design be changed to a plate-type hanger. Vendor response to the incorporation of these changes into the existing inpile tube order indicated that additional design effort should be made. Such an effort would simplify the configuration and thus obtain a workable assembly at a more reasonable cost. The design change was modified as necessary to include only those portions of the

Neg 06813181

FIGURE 12.38. Effect of Hydrostatic Pressure on the Isochronal Recovery of the Electrical Resistance of Neutron Irradiated High Purity Molybdenum

lower specimen train present in the original order. The portions requiring redesign will be procured separately by BNW on a procurement directive to AEC-RL0. An estimate to perform this work is currently being prepared for submittal to AEC-ID.

All cognizant agencies have agreed to the final draft of the HSW Clean Room and Quality Control Procedures.

Because of the change in GCL funding, the clean rooms that have been constructed will probably be used for component storage.

Analyses of the GCL Loop primary and regeneration heat exchangers were performed and reported by Idaho Nuclear Corporation. Their results indicate that flow-induced vibrations in these units are of such limited

amplitude and frequency that no vibration difficulty is expected.

Vendor Data Review

Vendor data reviewed include submittals from manufacturers of the primary circulators, purification train, heat exchanger and miscellaneous smaller equipment. The data have been generally acceptable. The heat exchanger manufacturer has submitted revisions to his welding procedures to permit use of recently procured automatic welding equipment. Presently, the only area in question is his proposal to make all the tube-to-tube sheet welds without filler metal. He was requested to submit chemical analyses for the actual components to be welded for evaluation so that the probability of obtaining acceptable welds by this means could be determined. The results of this evaluation should be available within a few weeks. One item, concerning welding in the analytical panel, requires some R&D effort prior to preparation of a recommendation. Sample components are enroute to PNL for development of an acceptable method for joining 303 SS to 304.

Meetings

On January 10, A.M., a meeting was held with INC, AEC-ID, and BNW at the ATR Building. The purpose was to establish how the GCL materials will be stored to comply with the AEC directive indefinitely postponing the GCL construction.

It was agreed that instrument and electrical panels will be mounted in

their planned installation location for storage, and the remaining equipment will be stored in the GCL cubicle and sample area. Critical materials will be in a segregated area. INC will have custody of the equipment until such time as installation work resumes; it will also maintain inventory and location records.

On January 10, P.M., AEC-ID, BNW, and INC Procurement and Engineering personnel met to resolve additional procurement problems on the GCL in-pile tube. Change orders were recently issued to the vendor covering necessary modification to the test train to accommodate thermocouple connectors and sample holders. Vendor response was that the complexity and specified tolerances would require him to submit an excessively high bid. Various possibilities to resolve the problem were discussed, and BNW was requested to submit a recommendation. This was done by letter on January 18, 1968.

On January 12, BNW was requested to assist INC in reviewing the status of GCL orders to determine whether any were less than 50% complete. Based on criteria that the project would be changed in excess of 50% of the contract price in the event of termination, all major GCL orders were estimated to be in excess of 50% complete.

On January 17, the third General Procurement meeting was held at the AEC-ID offices with all agencies in attendance. Procurement activities relative to the recent directive to terminate construction were discussed.

It was determined that factory inspection and acceptance should be incorporated into all orders, while receiving inspection would be only for shipping damage. Each vendor would be asked for recommendations to prepare equipment for long-term storage and for possible extension of warranties. Discussion on the status of the orders revealed additional slippage in projected delivery dates on some orders. Procurement activity and expediting will continue on a nonpremium basis.

On January 18, BNW personnel visited the plants of the transmitter panel subvendor to survey the equipment and plans for welding the interconnecting tubing. Weld qualification samples had been prepared, tested, and approved. The manufacturer is considered capable and well equipped to do the work.

On January 19, BNW personnel visited the primary heat exchanger vendor to discuss welding and other topics. The vendor had recently acquired and assembled new, automatic equipment for this job, but had not yet performed any welds. Since all his presently approved procedures are for a manual weld process, he will also have to submit new procedures and qualify the process and operators. Another visit is planned soon to determine whether any fabrication problems exist and to offer such technical assistance as might be required.

A meeting was held February 27, 1967 with representatives of INC, Lockheed-Georgia, B&W, and AEC to review the status of the transfer

cask design effort. Lockheed-Georgia reviewed modifications to the scope design, which they had incorporated at the suggestion of B&W, and requested approval. BNW pointed out that the previous design had been acceptable for function, but that the design alterations also appeared to be acceptable if none of the required functions were compromised. A request was made for an opportunity to review those drawings involving changes that affected function.

A TRA user's meeting was held in Idaho Falls on March 18 and 19, but no topics pertinent to the GCL program were discussed.

Loop Surveillance

No further effort is anticipated in this category until reactivation of the loop project, although procurement of the necessary representative materials will continue.

Transfer Facility

Completion of the construction of the transfer facility and preparation for long-term storage will be the only immediate action until the GCL is reactivated.

Thermocouples

Since the specifications for thermocouples and connectors were recently issued and procurement was less than 50% complete, procurement will probably be terminated.

Liaison for GCL Material

The GCL hold-up tank has been re-cleaned, purged, and stored for future installation. Also, a quantity of stainless pipe has been cleaned and repackaged. The recleaning of these items was caused by the loss of packaging integrity and subsequent contamination. At a recent General Procurement meeting, approval was given, to perform the final GCL equipment inspection and acceptance at the vendor's plant. This arrangement should insure compliance with packaging specifications and, therefore, lessen the amount of recleaning required on site.

ATR WATER LOOP SUPPORT

A third water chemistry test was run in the ATR North pressurized water loop. The test conditions included filling the loop with raw LDW (low-pressure demineralized water, 90 psig), pressurizing the loop to 2000 psig, heating to 540 °F, and circulating at 80 gpm. The pH of the water was adjusted to 10.5 with lithium hydroxide. The loop water was circulated in a closed system and was sampled every 2 hr for oxygen and pH. The following results were recorded:

<u>Time</u>	<u>pH</u>	<u>Oxygen (ppm)</u>
13:40	10.50	10.90
15:50	10.35	8.70
17:57	10.27	8.00
19:54	10.16	8.75
22:08	--	Yellow Water --

The loop was cooled down and the test was terminated. The yellow water

sample, indicating chromate, was qualitatively analyzed, and chromium was verified and estimated at about 10 ppm. A trace of iron was found and estimated at 1 ppm. Nickel was not found and sodium showed about 0.1 ppm. At first it was believed that sodium dichromate inhibited HDW (high-pressure demineralized water, 240 psig) leaked into the loop because HDW is used to cool the shell side of the three heat exchangers and the three primary circulation pumps. However, because the loop was at 2000 psig, no makeup water had been added, and the leak rate was small, the possibility of HDW leakage was discounted. Investigation revealed that similar yellow water incidents have been seen in other loops when excess oxygen was in the water. It is now believed that chromate was formed from the stainless steel piping in the loop, the chemical reactions being in two steps:

Chromic acid was formed which ionized to give free chromate ion and the yellow colored water.

Later, a fourth water chemistry test was run with the loop filled with LDW, pressurized to 2000 psig, heated to 540 °F, and circulated at 60 gpm. Before the loop was heated, the oxygen was reduced from 8 ppm to 4.25 ppm by degassing for a few hours. A side flow of 1.5 gpm was flowing through the purification ion exchangers. The following results were recorded:

Time	pH	Oxygen (ppm)	Date
10:30	10.05	4.25	2-12-68
11:22	--	4.35	2-12-68
13:30	10.03	4.20	2-12-68
09:00	10.08	4.20	2-13-68
10:09	10.06	1.85	2-13-68

The system was degassed after 09:00 and the oxygen content was reduced as shown. Prior to that time, little change in pH or oxygen concentration was noted.

Another similar test is planned with the reactor at about 100 MW to see what effect neutrons have on the oxygen level.

A tilt table and work tray were shipped from Richland to the ATR. The 3 by 5 ft tray and 24 ft long tilt table will soon be installed in the ATR canal and will be used to assemble and disassemble loop sample trains.

Spare parts that have been ordered are being delivered to the NRTS site. Such parts, which will help insure continuous operation of the ATR pressurized water loop, include spare thermocouples, gaskets, valves, packing, diaphragms and related items.

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FRACTURE MECHANICS EVALUATION OF REACTOR VESSEL STEELS

Westinghouse Electric Corporation

T. R. Mager, W. S. Hazelton, and F. Thomas

An experimental program directed towards radiation effects on pressure vessel materials is being performed. Experimental testing of a group of reactor vessel steels is being performed to investigate the application of the fracture mechanics technique to these materials in both the un-irradiated and irradiated conditions.

The 12 in. thick plate of A533 Grade B Class I (MnMo with Ni) steel was sectioned into specimen blanks.

The layout drawing for sectioning the 12 in. thick plate is given in Figure 13.1. Specimens were machined from the top surface, 1/4 thickness, 3/4 thickness, and bottom surface of the 12 in. thick plate. The type and number of specimens being utilized as well as specimen location as to the position through the thickness of the plate are given in Table 13.1.

FIGURE 13.1. Specimen Layout for 12 in. Thick Plate SA-533 Grade B Class I Steel - Irradiation Effects Program

TABLE 13.1. Type, Quantity and Location
of Test Specimens from 12 in. Thick Plate

Friction Through Thickness	Type of Specimens					Drop Weight
	1X-WOL	1T (Compact)	2T (K _{IC})	Charpy's	Tensiles	
Top Surface	10 (6)	9 (3)	9 -	26 (12)	11 (4)	6 -
1/4 Thickness	10 (6)	9 (3)	9 -	26 (12)	11 (4)	6 -
1/2 Thickness	10 (6)	9 (3)	9 -	26 (12)	11 (4)	6 -
3/4 Thickness	10	9	9	26	12	6
Bottom Surface	10	9	9	26	12	6

() - For Irradiation Experiment

The preirradiation testing of the tensiles, Charpy-V notch and drop weight specimens fabricated from the 12 in. thick plate of A533 Grade B Class I (MnMo with Ni) steel will be initiated in April 1968. The tensile properties will be evaluated from -250 to + 600 °F. Fracture toughness testing of the 12 in. thick plate utilizing 1X WOL, 1T and 2T Compact K_{IC} specimens will also be initiated next report period.

Preirradiation fracture toughness testing of three of the four AEC approved materials (A533 Grade B Class I steel from the 8 in. thick plate, the European Forging Grade Steel, and a weldment from a heat of A533 Grade B Class I steel) was completed. As discussed in Reference 1, the requirements of the type of fracture toughness testing employed are unusual in that it is necessary to

compare post-test data to specimen pre-test dimensions to determine if the fracture toughness measured (K_Q) is the critical value (K_{IC}) for the material. In other words, validity of the test can be determined only after testing. Two criteria for validity have been proposed.

1. Reference 1 proposes use of the secant offset method to determine that the majority of strain is due to crack elongation.
2. Reference 2 proposes (specifically for bend specimens, of which WOL and Compact K_{IC} specimens are modifications) that both the crack length and specimen thickness exceed 2.5X (Fracture Toughness/Yield Strength).⁽²⁾

Fracture toughness measured for specimens meeting these criteria is considered to be the critical value

(K_{IC}) for the material. If the criteria are not met, the measured fracture toughness (K_Q) is considered to have been biased by specimen size. The stress intensity factors (fracture toughness) for the AEC approved materials are presented in Figures 13.2 through 13.9. All of the data shown in Figures 13.2, 13.4, 13.6, and 13.8 are valid K_{IC} measurements according to the criteria discussed in References 1 and 2. The data which were obtained, but which failed to meet either the "a" (crack length) or "B" (thickness) size requirements are shown in Figures 13.3, 13.5, 13.7, and 13.9. Inspection of Figures 13.2 through 13.9 reveals some discrepancy as to valid or invalid results. For example, in Figure 13.3, we have a 1X WOL specimen at -200 °F that not only lies in the valid data band but gives a K_{IC} value which is essentially the same as the valid 1T WOL specimen shown in Figure 2. This type of specimen behavior is repeated in Figure 5. Here we have a 1X WOL specimen that fails to meet the required criteria but gives the most conservative K_{IC} measurement for this temperature. In both examples, Figures 13.3 and 13.5, the specimens discussed met the "B" (thickness) requirement but failed the "a" (crack length) requirement. Probably the most anomalous behavior appears in the testing of the weld material. All the 2T WOL specimen K_{IC} valid points lie above the valid points for the 1X and 1T specimens at a given temperature. Valid plane strain data will give approximately

the same value for all valid thicknesses. Figure 13.6 suggests that although the 1X and 1T WOL specimens meet the proposed criteria, the specimens are not valid at -200 and -250 °F. Additional testing is being planned to verify the curve.

The irradiation of the first three test capsules was completed in the Babcock and Wilcox Test Reactor (BAWTR) February 12, 1968. The three capsules had a total exposure of 132 EFPD or an estimated fluence of 1.3×10^{19} . The capsules were removed from the reactor and will be shipped to Westinghouse's Waltz Mill postirradiation laboratory the first week of April 1968.

During this quarter, the test specimens (WOL's, Compact K_{IC} 's, Charpy-V notch, and tensiles) required for the second three test capsules were fabricated and delivered to Babcock & Wilcox (B&W). The specimens were fabricated from the 12 in. thick plate of A533 Grade B steel. B&W assembled the irradiation capsules and checked out all the heater wires and thermocouples. Irradiation of the test capsules was initiated March 29, 1968. The test capsules contained the following neutron monitors.

1. Iron (drilling from fractured Charpy-V notch impact specimens)
2. Neptunium oxide
3. Cobalt (aluminum wire, 0.15% cobalt)
4. Cadmium covered cobalt (aluminum wire, 0.15% cobalt)
5. Copper wire.

FIGURE 13.2. Results of WOL Fracture Toughness Tests (SA533 Grade B Class I - from 8 in. Plate)

FIGURE 13.3. Results of WOL Fracture Toughness Tests (SA533 Grade B Class I - from 8 in. Plate)

FIGURE 13.4. Results of WOL Fracture Toughness Tests (European Forging Grade Steel)

FIGURE 13.5. Results of WOL Fracture Toughness Tests (European Forging Grade Steel)

FIGURE 13.6. Results of WOL Fracture Toughness Tests (SA533 Grade B Class I - Weld)

FIGURE 13.7. Results of WOL Fracture Toughness Tests (SA533 Grade B Class I - Weld)

FIGURE 13.8. Results of WOL Fracture Toughness Tests (SA533 Grade B Class I - HAZ)

FIGURE 13.9. Results of WOL Fracture Toughness Tests (SA533 Grade B Class I - HAZ)

Neutron flux dosages will be determined by the following reactions:

1. $^{54}\text{Fe} \rightarrow ^{54}\text{Mn}$
2. $^{237}\text{N} \rightarrow ^{137}\text{Cr}$
3. $^{59}\text{Co} \rightarrow ^{60}\text{Co}$ (with and without Cd cover)
4. $\text{Cu} \rightarrow ^{60}\text{Co}$.

This system of monitoring the neutron flux is identical with the monitoring of the first three irradiation capsules. Measurement of fast neutron fluxes using the above reactions will be conducted according to ASTM standard procedures developed by ASTM Committee E-10 on Radioisotopes and Radiation Effects.

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