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REPORT

**QUARTERLY PROGRESS REPORT
METALLURGY RESEARCH OPERATION
APRIL, MAY, JUNE, 1965**

JULY 15, 1965

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IN NUCLEAR SCIENCE ABSTRACTS

RICHLAND, WASHINGTON

PACIFIC NORTHWEST LABORATORY operated by BATTELLE MEMORIAL INSTITUTE

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QUARTERLY PROGRESS REPORT
METALLURGY RESEARCH OPERATION
APRIL, MAY, JUNE, 1965

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July 15, 1965

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SUMMARY

PHYSICAL METALLURGY

RADIATION EFFECTS ON METALS

A series of molybdenum foils with varying amounts of prestrain were irradiated at 60 °C to 7×10^{18} nvt ($E > 1$ MeV) and subsequently examined by electron microscopy. Because the density of defect clusters was not affected by a high dislocation density, it was concluded that dislocations do not act as sinks for the point defects which comprise the clusters. Subsequent straining of the foils causes new dislocations rather than the old ones to move from boundaries and to form channels parallel to $\{110\}$.

Dislocation channeling has also been observed in irradiated and deformed rhenium. Most channels are parallel to $\{10\bar{1}0\}$ prism planes and some are parallel to basal planes. $\{11\bar{2}1\}$ was the most common twinning plane.

Molybdenum crystals containing carbon as intentional impurity have been irradiated in the as-grown state to 1×10^{20} nvt and changes in length have been determined. Length increases ranged from 0.050 to 0.060%, and were independent of carbon content. Comparison of lattice parameter and length changes in as-grown crystals with those of annealed crystals after an irradiation to 7×10^{18} nvt leads to the conclusion that vacancies are more abundant in the annealed crystals and that dislocations in the as-grown crystals are effective sinks for the vacancies formed during irradiation.

Strain rate change tests conducted on $\langle 149 \rangle$ axially oriented tensile specimens of molybdenum, which were annealed at 1300 °C for 16 hr prior to testing, show that activation volume decreases with increasing strain. The low value of activation volume, $10 b^3$, suggests the Peierls mechanism to be rate controlling.

PLUTONIUM PHYSICAL METALLURGY

The effect of prior tensile and compressive deformation of the stable phase of plutonium on subsequent transformations has been investigated. The transformation rates of $\beta \rightarrow \gamma$, $\gamma \rightarrow \beta$, and $\alpha \rightarrow \beta$ are all increased, whereas that of $\beta \rightarrow \alpha$ is decreased. It is concluded that the $\beta \rightarrow \alpha$ transformation is martensitic and that the others are martensitic, diffusional, or both.

The effect of uniaxial tensile and compressive stresses on the temperature for the start of the $\alpha \rightarrow \beta$ transformation was determined. The data are consistent with the premise that the $\alpha \rightarrow \beta$ transformation is predominantly martensitic.

The X-ray diffraction pattern obtained from beta phase plutonium formed from alpha differs from beta phase plutonium formed from gamma. These differences, which can be eliminated by heat treatment in the beta phase temperature range, are tentatively explained on the basis of differences in structure and internal stresses.

DAMAGE MECHANISMS

Irradiation of Ferrovac E and a higher purity zone refined iron to an exposure $< 5 \times 10^{18}$ nvt produces no detectable change in microstructure, as revealed by transmission electron microscopy, but does produce a change in activation volume.

SWELLING OF IRRADIATED FISSIONABLE MATERIALS

Irradiation of capsules designed to supply data on the interrelationships between burnup, temperature, pressure, and metallurgical state of uranium base specimens has continued. A capsule controlled at 625 °C and 1000 psi has been discharged. A capsule controlled at 450 °C and 500 psi is performing satisfactorily.

Metallography and density measurements on 10 specimens irradiated at 575 °C and 1000 psi show that pressure reduces swelling in high purity uranium by factors as high as 5 to 40, depending on grain size. Pressure

also reduces swelling in alloy specimens (U + Fe, Al, Si, C) but by factors of 2 to 5. Pressure is effective in reducing intragranular pores and tears which are generally aligned crystallographically.

REACTOR METALS RESEARCH

ALLOY SELECTION

Several nickel base alloys, including Hastelloy N, Hastelloy X-280, Incoloy 800, and Inconel 600, have been irradiated at elevated temperatures and tested in tension at temperatures to 650 °C. In the case of Hastelloy N irradiated at 280 °C, both strength and ductility at 650 °C were reduced with neutron exposure. For specimens irradiated at 740 °C, the 650 °C ductility was one-third of that at room temperature after an exposure of 1.9×10^{20} nvt. The net loss in ductility due to irradiation is greater at room temperature.

Incoloy 800, irradiated at 740 °C in the solution treated, mill annealed, and 20% cold worked conditions, shows a higher retained ductility in the solution treated condition at 650 °C. A greater loss in strength was also found.

Specimens of Hastelloy X-280 were irradiated at 280 and 740 °C in the solution treated condition. Results of tests performed at 650 °C show a loss in strength and ductility for specimens irradiated at both temperatures. The percent uniform elongation for specimens irradiated at 740 °C were considerably below those irradiated at 280 °C.

A bench model of a liquid metal temperature controlled capsule was operated. Temperature control was achieved by using gaseous mixtures of helium and argon. The calculated temperature distribution closely approximated the observed distribution.

IN-REACTOR MEASUREMENTS OF MECHANICAL PROPERTIES

The objective of in-reactor measurements is to study and define the effects of neutron irradiation on the mechanical properties of structural materials. Current program efforts include the determination of the in-reactor stress and temperature dependency of creep of annealed 304 SS, annealed Zircaloy-2, and a cursory study on the in-reactor creep of Zr-2.5 wt% Nb alloy.

In-Reactor Creep of Annealed 304 SS

Minimum creep rates of the in-reactor creep of annealed 304 SS have been compared to those determined out-of-reactor as a function of temperature and stress. Within the scatter of experimental data there is no effect of neutron irradiation on the minimum creep rate. There are some differences, however, between in-reactor and out-of-reactor creep characteristics. Of greatest significance is the reduced creep to rupture life during neutron irradiation. Elongation at rupture and rupture life are reduced in-reactor to approximately 40% of that observed out-of-reactor. Secondly, the difference in the activation energies as measured by temperature change method and obtained from the slope of an Arrhenius plot of minimum creep rates suggest an irradiation modification of the primary stage of in-reactor creep. The mechanism by which this may occur is as yet undetermined but may be of the same nature which contributes to the reduction of in-reactor creep rupture properties, since it requires thermal activation and is stress dependent.

IRRADIATION FACILITIES OPERATION

With the conclusion of Engineering Test Reactor (ETR) Cycles 71 and 72, approximately 100 tensile, stress-rupture, and fracture specimens have been discharged from ETR core positions. Materials irradiated at 50 and 290 °C include nickel-base alloys, stainless steels, and Zircaloy-2. Over 65 control specimens of these same materials were discharged after exposures in the out-of-reactor loop.

All of the special quadrants containing BONUS transition joint tensile specimens have been discharged from the G-7 or out-of-reactor loops after having accumulated the scheduled exposures. All of the specimens have been examined and tested. Hydrogen analysis and metallography are being performed on specimens exposed in either the G-7 or out-of-reactor loop for greater than 2200 hr. This study is being conducted to evaluate the effects of irradiation and environment on Zircaloy-2-Inconel 600 transition tube joints. Overall responsibility for evaluation of these tube joints rests

with Combustion Engineering, Inc. on the Advanced BONUS Core Development Program. A topical report is being prepared to present the results obtained.

Data Processing

The retrieval portion of the REM Computer Program has been programmed, debugged, and utilized to generate trial reports. This program provides for editing, processing, storage, and retrieval of tensile and associated metallurgical test data. The capability for processing other types of mechanical test data which is generated in sufficient volume is also incorporated in the overall programming scheme.

Initially, very simple program requests have been generated to develop a familiarity with the system. Additional tests are being conducted to explore the retrieval capabilities and improvement of processing metallurgical test data. From a single sheet of instructions on how to specify formats, how to specify multiline reports, and other routine report features, an endless variety of reports can be generated by an engineer or technician having no programming experience. A very small number of cards are necessary for each report request. Similar reports can be obtained for any material in the file by changing one card.

The reports generated will ultimately be linked to analytical and statistical sub-routines to establish curve fits, deviations, and allowable limits of the raw data. The results from any processing step can be programmed for display on automatic plotting equipment. The file currently contains over 2000 individual tensile records on approximately 30 different materials. Tensile records are being added at the rate of about 100/mo. Continual development of this program will be emphasized to obtain data which is statistically significant in a shorter time and for a reasonable cost.

IRRADIATION DAMAGE TO STAINLESS STEELS

A series of tensile tests on 348 and 304 SS have been completed at test temperatures from 20 to 850 °C. The data includes results obtained

from materials irradiated at temperatures of 60, 290, and 750 °C. Specimens were tested in the as-fabricated condition, after exposure in a controlled environment, and after irradiation exposures greater than 1.0×10^{21} nvt (> 1 MeV).

The results of these tests identify the ranges of test temperatures at which ductility minimums exist. Although the greatest property changes occur at room temperature, the absolute ductility is much lower at elevated temperatures. The ductility minimums are produced by metallurgical instabilities that result from the combined effects of irradiation and environment or by an increase in grain boundary deformation at the higher test temperatures. Since stainless steels are utilized in numerous reactor applications over a wide range of service temperatures, it is necessary to know where these minimums exist and how they are affected by particular environments and increasing irradiation exposures.

Generally, the effect of irradiation is to increase the strength and decrease the ductility for most irradiation test temperatures of interest. At the low to intermediate temperature range between 200 and 500 °C, a ductility minimum exists. This value appears to be lower in most cases than is observed at test temperatures to 750 °C. Increasing exposures cause the temperature at which this minimum value occurs to vary, usually lowering it slightly.

The effect of irradiation is reduced considerably for test temperatures above about 500 °C. Since grain boundary deformation is the controlling factor at these higher temperatures, less change is expected for the lower exposures. Long-term exposures may result in a greater rate of decrease in the ductility if the substructure at or near the grain boundary is modified. Since both cold work and irradiation damage are annealed out at an accelerated rate with increasing test temperatures, both effects must be characterized to determine the specific contribution of each of several parameters.

An interim report is being prepared to outline the results obtained to date on this continuing program. A review of the state of knowledge on

the irradiation effects to stainless steels will insure that the specimens which are being irradiated to goal exposures of approximately 1.0×10^{22} nvt (>1 MeV) are used most judiciously.

IRRADIATION DAMAGE TO NICKEL-BASE ALLOYS

The nickel-base alloys hold much promise for high temperature nuclear application. However, they do suffer damage when irradiated at high temperature. An effort to determine the effect of modified microstructures upon the irradiation stability of nickel-base alloys is continuing.

Previous work on Hastelloy X-280 led to the conclusion that a desirable microstructure would be one in which the matrix carbon was stabilized so as to reduce the formation of embrittling grain boundary deposits. It was then shown that a combination of cold work and aging would produce a dispersion of carbide within the matrix with reduced grain boundary deposit. Tensile specimens having various combinations of cold work and aging treatments were irradiated at 540 °F to 1×10^{20} nvt and, with their control, were tensile tested at room temperature and 1350 °F in air. When compared with a standard treated irradiated material, some treatments showed improved high temperature ductility while maintaining strength, and, in general, susceptibility to radiation damage was lessened. One treatment resulted in an 84% increase in postirradiation high temperature yield strength with little ductility reduction. A maximum room temperature yield strength increase of 63% was also obtained while maintaining useful ductility of 18%.

Stress-rupture tests at 1350 °F will be initiated this next quarter.

Tensile and stress-rupture specimens of Inconel X-750, a precipitation hardenable alloy, are being irradiated in five different preirradiation treatments at 540 °F to approximately 2×10^{20} nvt, with others being prepared for 120 and 1300 °F irradiations.

FRACTURE STUDIES

Effect of Geometry Variables on DCB Specimen Performances

Unlike many specimens presently in use for determining plane strain fracture toughness data, the double cantilever beam (DCB) type of specimen

has the advantage of enabling a relatively large number of plane strain fracture toughness data to be determined from a single specimen of moderate size. This is a distinct advantage when one must conduct fracture studies on a limited volume of material, e. g., in irradiation effects programs.

Although the geometry of the specimen is simple, the selection of the appropriate dimensions was found to be complex and necessitated a trial and error basis for selection. Since recalibration of the compliance of the specimen is made necessary by change in specimen design and also since additional experiments were planned which required specimens of altogether different dimensions, it was decided to perform a number of tests to determine the effect of changing the geometry on the compliance curves. In addition, these tests provided the opportunity of observing the effects of changing the geometry on the value of the fracture toughness for the material studied.

The material selected for these tests was 7075-T6 aluminum. The variables studied were gross thickness (B), beam depth, side groove depth, and side groove root radius.

The compliance was computed from the slope of the load versus extension curve. It was found that the compliance could be accurately represented by the empirical equation $\phi = Ac^n$ where ϕ is the compliance, c is the crack length, and A and n are constant for a given set of specimen dimensions.

The compliance results indicate that the constant A is inversely proportional to the moment of inertia of the arms for a given beam depth, the proportionality constant depending on beam depth. In addition, n was found to decrease with increasing beam depth.

The fracture toughness or K_{IC} values were computed utilizing the constants from the compliance curves. A total of 700 values of fracture toughness were obtained from 20 specimens. As a result of the large amount of data acquired, a computer program was developed to relieve the computation burdens.

The data describing the effects of changing the geometry on K_{IC} indicate that the K_{IC} values for the majority of the specimens fall in the range of 26 to 29 ksi $\sqrt{\text{in.}}$. Values of 28 to 30 ksi $\sqrt{\text{in.}}$ are fairly representative of K_{IC} for 7075-T6 aluminum as obtained by other methods. There were, however, three effects observed:

- K_{IC} was found to increase when the net width to gross width ratio became very small
- For the specimens with large beam depth but narrow width, K_{IC} was found to decrease with crack length (this effect was observed in three specimens)
- It was found that increasing the side groove root radius resulted in the formation of shear lips along the side of the crack.

Concurrent with shear lip formation, the K_{IC} values tended to increase.

Delayed Failure Studies

A normally ductile pressure vessel steel such as A302B could fail by a time dependent brittle fracture process. This process, called delayed failure, is attributed to diffusion of a highly mobile interstitial (usually hydrogen) to areas of high stress in advance of a sharp notch or crack. A reactor environment could enhance the delayed failure process.

The methodology of fracture mechanics is being applied to the study of delayed failure of pressure vessel steels. Analytic techniques, using a DCB specimen, are now being developed to describe the fracture process in terms of its thermodynamic properties. The change in these properties with varying hydrogen content will contribute significantly to a basic understanding of the mechanisms of delayed failure.

In A302B pressure vessel steel a fully hardened hydrogen free structure produces a fracture toughness (K_{IC}) value of 100 ksi $\sqrt{\text{in.}}$ as compared to 80 ksi $\sqrt{\text{in.}}$ for a fully hardened hydrogenated structure. One would predict that fracture toughness should vary with hydrogen content.

Examination of a typical Instron testing machine curve for a fully hardened, hydrogenized DCB specimen tested at -40°C shows an initial

region of linear elastic deformation. After attaining a critical stress the crack moves in a nonuniform manner with crack growth appearing to be continuous (within limits of machine sensitivity) to the point of unloading. By employing a stationary crosshead technique, load decay rates are obtained from which crack velocities can be calculated. These in turn can be related to more fundamental properties.

Crack lengths can be calculated from any selected set of load and extension values by the relation

$$C = \left(\frac{Ey}{Af} \right)^{1/m}$$

where

C = crack length

E = elastic modulus at the test temperature

y = extension

f = load

A = constant = 515.3

m = constant = 2.726.

A calculation showed that the crack in a typical DCB specimen grew 0.122 in. during the period of stationary crosshead.

Load decay could be largely the result of creep in the specimen. The extent of creep can be determined through consideration of the specimen compliance $\left(\frac{y}{f} \right)$ at a point of constant extension (stationary crosshead) and during specimen unloading. If creep is occurring, the compliance and the related calculated crack length of these separate conditions will be significantly different. The computed crack lengths of the two conditions, however, differed by only 0.004 in., showing that deformation by creep during the stationary crosshead time interval is negligible.

Work planned for next quarter will include refinement of testing techniques and development of procedures for introduction and analysis of controlled quantities of hydrogen in the DCB specimen.

CHEMICAL METALLURGY

ATR GAS LOOP SUPPORT

Oxidation of Hastelloy X-280 and Haynes Alloy 25 in 10^{-4} Torr dry air and oxygen atmospheres at 1120 °C gave metal penetrations of 0.26 mil for Hastelloy X-280 and 0.16 mil for Haynes Alloy 25 (45 hr exposure in dry air) and 0.18 mil for Hastelloy X-250 (71 hr exposure in oxygen). The major portion of the metal penetration was due to evaporation of the alloys rather than oxidation. Spalling of the thin oxide formed on Hastelloy X-280 in an oxygen atmosphere was evident. No evidence for spalling of Haynes Alloy 25 was noticed.

One 76 hr run with Hastelloy X in purified helium at 2050 °F and 525 ft/sec yielded an evaporation rate of about 4 mg/cm². Two attempts to duplicate this run have aborted due to equipment failure.

N-REACTOR CORROSION AND HYDRIDING

The oxidation characteristics of Zircaloy-2 have been studied in a water vapor atmosphere (25 Torr) at 900 to 1200 °C. The kinetics for oxidation fit a parabolic rate expression

$$k_p \text{ (mg}^2\text{cm}^{-4}\text{min}^{-1}\text{)} = 9.26 \times 10^8 \exp (-51,200/\text{room temp})$$

applicable over the temperature range studied. The presence of hydride in the oxidized samples was also noted. It appears that hydrogen sorption (from the corrosion reaction $\text{Zr} + 2\text{H}_2\text{O} \rightleftharpoons \text{ZrO}_2 + 2\text{H}_2$) occurs concurrent with the oxidation process at these high temperatures.

It has been confirmed that in impure helium containing H_2 , H_2O , and Co, as the film thickens the hydrogen absorption rate becomes faster and the oxidation rate slows down due to a pressure dependence on diffusion through the thick film. This reduction in oxidation rate means that Zircaloy process tubes will take considerably longer to reach weight gains where hydriding increases due to the thick film effect (100 mg/dm²) than would have been predicted from high pressure data. Also, the H_2 pressure

dependence data on hydriding rates are encouraging, because all data at 250 ppm hydrogen indicate essentially no gas phase hydriding at any film thickness up to 450 mg/dm^2 and temperatures of 250 to 400°C . Even at 2000 ppm H_2 hydriding rates at 400°C up to 100 mg/dm^2 have been only 25% of the corrosion hydrogen.

MATERIALS ENGINEERING

GAS LOOP DEVELOPMENT

Model Loop Operation

Tests that were completed on foil insulation in a flowing helium system indicate that with proper joint design the conductivity is the same in both static and dynamic systems. The conductivity varied from $0.26 \text{ Btu/}(\text{hr})(\text{ft}^2)(^\circ\text{F})$ at a mean insulation temperature of 400°F to $0.39 \text{ Btu/}(\text{hr})(\text{ft}^2)(^\circ\text{F})$ at 1000°F . A bearing failure in the eddy-current, variable speed drive in the frequency changer assembly has been the only trouble encountered in the gas bearing compressor and its associated equipment. The equipment was modified to eliminate this type of failure. Testing has been started on the prototypical Advanced Test Reactor (ATR) gas seal for the in-reactor pressure tube.

Model Gas Loop Heater and Piping Modifications

A new model gas loop heater has been designed and is being built to incorporate and investigate essential details of a compact two-stage heater presently being designed for the ATR Gas Loop.

Material and/or components for all parts of the heater electric instrumentation, power supply equipment, and loop piping modification have been ordered. Fabrication of heater components and foil insulation sections is now in progress. Work on the loop and heater should be completed in September.

Helium Purification System

The high pressure purification system purchased to functional specifications was modified to permit more thorough regeneration of the molecular

sieve beds. Used helium with approximately 3700 ppm impurities was processed twice with a resulting purity of less than 0.1 ppm H_2 , O_2 , N_2 , CH_4 , and CO, and 1.5 ppm water.

Consolidated Relief Valve 1975T, equipped with a Viton A O-ring seat, successfully seals helium at 500 psi following overpressure actuation.

PRTR PRESSURE TUBES

Properties of Irradiated PRTR Pressure Tubes

Stress-rupture tests of irradiated tube segments suggest that increasing amounts of fast neutron exposure may decrease time-to-rupture; however, some annealing of the as-irradiated strength occurs during the stress-rupture test.

Measurement of elastic constants of unirradiated specimens is expected to reveal the degree to which the material is anisotropic. The measurement is being made using sheet type specimens and a tube specimen.

Fracture Studies

A facility is now available for burst testing pressure tube specimens at sub-zero temperatures. Using this facility, the yield strength versus temperature curve for N-Reactor pressure tubing has been extended to $-33^\circ C$, showing virtually a linear relationship from that temperature to $300^\circ C$.

Irradiated Tube Testing Facility

A basin ion-exchange cleanup system has been installed to improve the visual clarity of the basin water and to remove suspended radioactive particulate matter.

An underwater tube shear has been designed to cut up ruptured test pieces into sizes that will fit into disposal casks.

USA-AECL COOPERATIVE PROGRAMZr-Nb Pressure Tubing

A series of crack propagation tests have been done at temperatures ranging from -23°C through $+265^{\circ}\text{C}$. These tests show that significant crack propagation does not occur at or above room temperatures.

PHYSICAL METALLURGY

RADIATION EFFECTS ON METALS - J. L. Brimhall, H. E. Kissinger, J. J. Laidler, B. Mastel, and K. R. Merckx

This program is directed toward establishing the combined effect of impurities and neutron irradiation on the properties and structure of specific metals, and deducing from thermally-activated recovery processes how the damage state can be altered. The major effort of present studies has been concentrated on single and polycrystalline specimens of molybdenum and the minor effort has been directed toward nickel and rhenium.

Transmission Electron Microscopy

Molybdenum foils which were prestrained varying amounts up to 20% have been irradiated to 7×10^{18} nvt ($E > 1$ MeV) as part of a study to determine the effectiveness of dislocations as sinks for point defects. General observations on all prestrained samples show no marked denuding of the cluster defects in the vicinity of the dislocations (Figure 3. 1). Actual count of the clusters indicates a decrease in the cluster density from $2 \times 10^{16}/\text{cm}^3$ to $1 \times 10^{16}/\text{cm}^3$ in the case of 20% prestrain. No significant difference in the cluster density was found between the unstrained samples and those prestrained less than 20%. Therefore, while there is evidence of some decrease in the overall density of clusters when the dislocation density is high, the dislocations do not act as significant sinks for the point defects comprising the visible clusters. Their effectiveness as a sink must be of a very short range nature.

The prestrained and irradiated samples have been further strained approximately 1 to 2% to determine how effectively the old dislocations are pinned. Cursory observation reveals that the dislocation tangles created during the prestraining do not move or act as sources during the deformation after irradiation. Channels parallel to $\{110\}$ slip planes are observed and appear to originate at grain boundaries. These observations show that new dislocation sources may be more readily operative after irradiation than the old preexisting sources.

145, 000X

FIGURE 3. 1

Microstructure of High Purity Molybdenum
Deformed in Tension 5%
and Irradiated to 7×10^{18} nvt

Neg. 30305

Analysis of dislocation channeling in irradiated and deformed rhenium shows that most of the channels are parallel to $\{10\bar{1}0\}$ prism planes and that some are parallel to basal planes. The twinning plane most frequently observed is $\{11\bar{2}1\}$, with $\{1\bar{1}02\}$ and $\{11\bar{2}2\}$ also observed. Figure 3.2 shows a twin in deformed irradiated rhenium with a dislocation channel running parallel to the twin. Such an occurrence has been observed in several instances.

X-Ray Diffraction

Molybdenum single crystal rod specimens intended for length change measurements have been irradiated to a goal exposure, 1×10^{20} nvt ($E > 1$ MeV), and have been examined. These specimens complete a series of irradiations of as-grown molybdenum containing three controlled carbon contents (15, 150, and 450 ppm). Length increases after 1×10^{20} nvt ranged from 0.050 to 0.060%, and were independent of carbon content.

Earlier irradiation experiments provided length-change data for exposures of 1×10^{18} and 1×10^{19} nvt. Lattice parameters were also measured on the length change specimens and on specimens with identical irradiation histories prepared especially for X-ray diffraction studies. A second series of irradiations of samples in the annealed condition (1300 °C for 16 hr) has been initiated.

Results from the first capsule irradiated to 7×10^{18} nvt have been given in the previous report. ⁽¹⁾ Comparison of results for this exposure shows the lattice parameter increase to be the same for crystals irradiated in the as-grown and in the annealed condition. Length changes, however, are significantly greater for the annealed crystals. Since length increases are presumably a result of vacancy defects, the vacancy concentration must be lower in the as-grown irradiated crystals. This decreased vacancy concentration may be a consequence of the increased density of sinks within the crystal and suggests that dislocations are more effective sinks for vacancies

(1) Quarterly Progress Report, Metallurgy Research Operation, January, February, March, 1965, edited by J. J. Cadwell, BNWL-79. April 15, 1965.

145,000X

FIGURE 3. 2

99. 92% Rhenium, Irradiated 7×10^{18} nvt ($E > 1$ MeV)
and Deformed Approximately 3%
(The micrograph shows a defect-free channel
running parallel to the twin boundary.)

Neg. 3189A

than for interstitials. The clusters observed in the electron microscope are thought to be interstitial clusters; so the two observations are not contradictory. Further work is in progress with emphasis on annealing behavior and the effects of higher exposures.

Mechanical Properties

The deformation behavior of molybdenum single crystals, which were prepared by triple-pass electron beam zone melting, is being studied. The crystals were seeded to yield a $\langle 149 \rangle$ axial orientation and tensile specimens were prepared by an electrolytic machining technique. After machining, the specimens were annealed for 16 hr at 1300 °C. Strain-rate change tests have been conducted at 77, 150, 196, 273, and 323 °K, and it is found that the activation volume decreases with increasing strain, in opposition to the observations made on polycrystalline molybdenum. The low values of activation volume in both single and polycrystalline molybdenum, about $10 b^3$, strongly suggest the Peierls mechanism as the rate-controlling process. The difference in the variation of activation volume with strain appears to originate in the greater rate of dislocation multiplication, presumably from grain boundary sources, in the polycrystalline material.

PLUTONIUM PHYSICAL METALLURGY - F. E. Bowman, H. E. Kissinger, B. Mastel, and R. D. Nelson

The objective of this program is to conduct basic studies on the properties of high-purity unalloyed and alloyed plutonium. Studies include investigation of phase transformation, phase stabilization, deformation, fracture, texturing, internal stresses, microcracking, grain growth, recovery and recrystallization, and measurement of mechanical and physical properties. The unique structure of several of the plutonium allotropes and their narrow temperature span of stability leads to characteristics and anomalies which, if explained, will contribute to the general area of solid state science.

The Effect of Plastic Deformation on Transformations in Plutonium

Previous experiments showed that compression of the stable beta plutonium phase inhibits the subsequent $\beta \rightarrow \alpha$ transformation rate.⁽¹⁾ This effect was observed in both beta formed from gamma, β_γ , and beta formed from alpha, β_α . Similarly, plastic deformation of the beta phase by tension decreases the $\beta \rightarrow \alpha$ transformation rate. Tensile specimens were transformed to beta from alpha at 150 °C and strained 50% in tension. A comparison of the isothermal transformation reaction curves clearly shows that plastic deformation of the beta phase by tension decreases the $\beta_\alpha \rightarrow \alpha$ transformation rate; this decrease is accompanied by an increase in the incubation time. The β_γ phase could not be deformed significantly in tension because of its very low ductility.

Plastic deformation of the beta phase increased both the $\beta_\alpha \rightarrow \gamma$ and $\beta_\gamma \rightarrow \gamma$ transformation rates. This is the opposite effect of beta plastic deformation on the $\beta_\alpha \rightarrow \alpha$ and $\beta_\gamma \rightarrow \alpha$ transformation rates. The increase in the $\beta_\alpha \rightarrow \gamma$ transformation rate was essentially the same as the $\beta_\gamma \rightarrow \gamma$ transformation rate. Again the beta phase formed from gamma was deformed only by compression since the ductility of β_γ was very low. The influence

(1) Quarterly Progress Report, Metallurgy Research Operation, October, November, December, 1963, edited by J. J. Cadwell, HW-79766. General Electric Company, Richland, Washington. January 15, 1964.

of deformation by compression on the $\beta_{\alpha} \rightarrow \gamma$ transformation rate was nearly the same as the influence of deformation by tension; both tension and compression increased the transformation rate by about the same amount for a given deformation. The effect of the deformation temperature and the effect of the amount of deformation on the transformation rate were not determined. The results clearly showed that plastic deformation of the beta phase accelerated the $\beta \rightarrow \gamma$ transformation.

Plastic deformation of the stable alpha phase accelerated the $\alpha \rightarrow \beta$ transformation at slow transformation rates. This effect is similar to the effect of beta plastic deformation on the $\beta \rightarrow \gamma$ transformation. Alpha plutonium could not be plastically deformed by tension because of its negligible ductility; it was deformed only by compression. The specimens were reduced 30 to 60% in thickness at 70 to 90 °C. The $\alpha \rightarrow \beta$ isothermal reaction curves clearly showed an accelerating effect on the $\alpha \rightarrow \beta$ transformation rate by prior plastic deformation of the parent alpha phase.

The influence of plastic deformation of the parent gamma phase on the subsequent $\gamma \rightarrow \beta$ transformation was also investigated. One specimen was compressed 80% in thickness at 220 °C after it had been transformed to gamma by holding at 250 °C for 30 min. This treatment was followed by a $\gamma \rightarrow \beta$ transformation at a temperature of 155 °C to permit transformation to occur within a reasonable time. The transformation rate of the gamma deformed metal was compared to the rate of undeformed metal. The isothermal reaction curves show that deformation accelerates the $\gamma \rightarrow \beta$ transformation. This effect is similar to the effect of alpha deformation on the $\alpha \rightarrow \beta$ transformation, is similar to the effect of beta deformation on the $\beta \rightarrow \gamma$ transformation, and is opposite to the effect of beta deformation on the $\beta \rightarrow \alpha$ transformation.

The parent phase of a martensitic transformation may be stabilized by plastic deformation. This inhibiting effect of plastic deformation on

transformation rates is referred to as mechanical stabilization. Mechanical stabilization has been observed in an 18-8 SS alloy⁽¹⁾ and other iron-nickel alloys.⁽²⁻⁵⁾ Plastic deformation of the stable parent phase may depress the M_s temperature.⁽⁶⁾

Fiedler, Averbach, and Cohen⁽¹⁾ found that plastic strains up to 2% elongation favored the austenite $\rightarrow$ martensite transformation; whereas, plastic strains above 2% elongation hindered the subsequent transformation. They suggested that mechanical stabilization of this alloy was due to the distorted structure of the deformed austenite. The high dislocation density and deformed lattice of the cold-worked austenite interferes with the propagation of martensite. Kurdjumov^(7,8) observed similar mechanical stabilization in a steel containing 0.954% C and 3.5% Mn.

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- (1) H. C. Fiedler, B. L. Averbach, and M. Cohen. "The effect of Deformation on the Martensitic Transformation in Austenitic Stainless Steel," Trans. Amer. Soc. Metals, vol. 47, p. 267. 1955.
 - (2) J. C. Shyne, V. F. Zackay, and D. J. Schmatz. "The Strength of Martensite Formed from Cold-Worked Austenite," Trans. Amer. Soc. Metals, vol. 52, p. 346. 1960.
 - (3) V. Bharuchi, G. A. Mancini, G. W. Powell, and J. W. Spretnak. "Transformation Kinetics in High Purity Iron and Some Iron Binary Alloys," Trans. Met. Soc., AIME, vol. 221, p. 498. 1961.
 - (4) B. L. Averbach. "The Effect of Plastic Deformation on Solid Reactions, Part II, The Effect of Applied Stress and Strain on the Martensite Reaction," Cold Working of Metals, Amer. Soc. for Metals, Cleveland, p. 290. 1959.
 - (5) J. F. Watson and J. L. Christian. "A Study of Austenite Decomposition at Cryogenic Temperatures," Trans. Met. Soc., AIME, vol. 224, p. 998. 1962.
 - (6) J. S. Bowles and C. S. Barrett. "Crystallography of Transformation," Progress in Metal Physics, edited by Bruce Chalmers, Pergamon Press, London, 1952. pp. 1-41.
 - (7) G. V. Kurdjumov, O. P. Maksimova, A. I. Nikonorova, Z. D. Pavlenko, and A. M. Yampol'skii. "The Effect of Prior Plastic Deformation on the Martensitic Transformation in an Fe-Cr-Ni Alloy," The Physics of Metals and Metallography, vol. 6, p. 85. 1958.
 - (8) G. V. Kurdjumov. "The Nature of Martensitic Transformations," J. Metals, vol. 11, p. 449. 1949.

Mechanical stabilization by plastic deformation of the stable parent phase is to be differentiated from plastic deformation of the unstable parent phase. The transformation rate is accelerated by plastic deformation of the unstable phase; the extent of the effect depends upon the sluggishness of the transformation. (1-4)

The inhibiting effect of plastic deformation of the parent phase on martensitic transformations has been observed in cerium and cobalt in addition to the Fe-Ni alloys. The inhibiting effect of plastic deformation of the stable parent phase on transformation rates, namely mechanical stabilization, has not been observed in diffusional transformations. On the contrary, plastic deformation always accelerates diffusional transformations because it promotes nucleation and growth. (5) Accordingly, the $\beta \rightarrow \alpha$ transformation in plutonium, which is retarded by plastic deformation, is a martensitic transformation. On the basis of the data presented here, the other low temperature transformations, which are accelerated by plastic deformation of the parent phase, may be martensitic, diffusional, or both.

The Effect of Stress on the $\alpha - \beta$ Transformation

The effect of applied uniaxial tensile and compressive stresses on the $\alpha - \beta$ transformation in plutonium was investigated to provide indirect information on the nature of this transformation. Specifically, the $\alpha - \beta$

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- (1) A. H. Cottrell. "Tensile Properties of Unstable Austenite and Its Low-Temperature Decomposition Products," J. Iron St. Inst., vol. 151, p. 93. 1945.
 - (2) L. C. Chang and T. A. Read. "Plastic Deformation and Diffusionless Phase Changes in Metals - The Gold-Cadmium Beta Phase," J. Metals, Trans., vol. 3, p. 47. 1951.
 - (3) M. W. Burkart and T. A. Read. "Diffusionless Phase Changes in the Indium-Thallium System," J. Metals, Trans., vol. 5, p. 1516. 1953.
 - (4) A. W. McReynolds. "Effect of Stress and Deformation on the Martensite Transformation," J. Appl. Phys., vol. 20, p. 896. 1949.
 - (5) B. L. Averbach. "The Effect of Plastic Deformation on Solid Reactions, Part I, Diffusion Reactions," Cold Working of Metals, American Society for Metals, Cleveland, p. 262. 1949.

starting temperature was determined as a function of stress at a heating rate of 2°C/hr . The results are characteristic of a martensitic transformation.

Applied compressive stresses, up to 10,000 psi, raised the $\alpha \rightarrow \beta$ starting temperature about $0.05^{\circ}\text{C}/1000$ psi; whereas, applied tensile stresses lowered the $\alpha \rightarrow \beta$ starting temperature $0.6^{\circ}\text{C}/1000$ psi (Figure 3. 3). The data clearly show that applied uniaxial tensile stresses favor the plutonium $\alpha \rightarrow \beta$ transformation more than applied uniaxial compressive stresses oppose it. This is completely in keeping with the hypothesis that the transformation is martensitic. Similar effects have been observed in

FIGURE 3. 3

Effect of Applied Uniaxial Stress on the Temperature for the Start of the $\alpha \rightarrow \beta$ Transformation in Plutonium

martensitic transformations in steel, ⁽¹⁾ gold-cadmium alloys, ⁽²⁾ and indium-thallium alloys. ⁽³⁾ All shear stresses favor martensitic transformation, regardless of the volume change; but normal components of applied stresses favor or oppose martensitic transformations, depending on whether contraction or expansion accompanies the transformation. The shear stresses of both types of applied stresses and the normal component of tensile stresses favor the plutonium $\alpha \rightarrow \beta$ transformation but the normal component of compressive stresses opposes the plutonium $\alpha \rightarrow \beta$ transformation. Thus, these data are consistent with known martensite transformations.

The Properties and Structure of Beta Plutonium

A paper entitled "Transformation and Deformation in Plutonium" by R. D. Nelson, F. E. Bowman, and T. K. Bierlein has been submitted to the Institute of Metals as a contribution to the Third International Conference on Plutonium to be held in London, November 22-26, 1965. This paper is concerned with the preliminary results of a study of the beta phase of plutonium as it is affected by the phase from which it originates. The remarkable difference in strength properties, the motivation for this study, between beta formed from gamma and beta formed from alpha has previously been cited. ⁽⁴⁾ Work with both high purity electrorefined metal as well as with the less pure as-reduced material has led to the conclusion that the strength differences noted are not purity dependent. In fact, it appears from X-ray data that the differences in the two types of beta may be increased as the impurity level is lowered.

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- (1) H. C. Fiedler, B. L. Averbach, and M. Cohen. "The Effect of Deformation on the Martensitic Transformation in Austenitic Stainless Steel," Trans. Amer. Soc. Metals, vol. 47, p. 267. 1955.
 - (2) L. C. Chang and T. A. Read. "Plastic Deformation and Diffusionless Phase Changes in Metals - The Gold-Cadmium Beta Phase," J. Metals, Trans., vol. 3, p. 47. 1951.
 - (3) M. W. Burkart and T. A. Read. "Diffusionless Phase Changes in the Indium-Thallium System," J. Metals, Trans., vol. 5, p. 1516. 1953.
 - (4) Quarterly Progress Report, Metallurgy Research Operation, October, November, December, 1963, edited by J. J. Cadwell, HW-79766. General Electric Company, Richland, Washington. January 15, 1964.

Detailed creep studies of the metal in both beta forms have revealed that though the beta creep strengths vary widely, dependent upon the parent phase, the creep activation energies for both types are essentially equal, ≈ 21 kcal/g atom. The activation energy for the as-reduced metal is significantly higher than that for the electrorefined material, however. There is evidence that the stress dependencies of the creep rates for the two types of beta plutonium are not the same.

Investigations of the beta to alpha transformation characteristics of the two forms of beta under the influence of both tensile and compressive stress as well as under no restraint have been conducted. The starting temperature for the $\beta_Y \rightarrow \alpha$ transformation is consistently higher than that for the $\beta_\alpha \rightarrow \alpha$ transformation. This is true under both types of applied stress. The transformation rate of the unrestrained β_Y was significantly greater at 85 °C than for the β_α material.

It was natural to attempt to explain these differences by differences in the basic crystal structure. X-ray diffraction patterns of the two types of beta plutonium are definitely different. At first glance, it would appear that this difference could be the result of preferred orientation. However, it has been demonstrated that the abnormal pattern, that from β_Y , is not dependent upon the sample surface orientation. It also has become apparent that these two types of structure are dependent upon the temperature of the beta phase. In other words, once formed, the β_α structure can be changed to that of the β_Y by heating to about 185 °C, and conversely, β_Y can be converted to the β_α type by cooling below 110 °C.

Significant differences are noted in the alpha phase microstructure, dependent upon the type of parent beta phase. The transformation of $\beta_\alpha \rightarrow \alpha$ results in much more extensive microcracking than the transformation of $\beta_Y \rightarrow \alpha$. Differences in the two types of beta are being studied further and explanations will be advanced.

The effects of applied tensile and compressive stresses upon the starting temperature for beta to alpha transformation for both types of beta support the hypothesis that the transformations are martensitic in nature. The differences noted in the beta to alpha transformation rates of the two types of beta, if attributed to the internal stresses present in the beta phase as a result of its formation, also tend to substantiate this postulated transformation process.

The following documents have been issued and distributed: "The State Creep of High Purity Plutonium, " BNWL-32, and "The Beta-Alpha and Alpha-Beta Transformations in Plutonium Under Applied Stresses, " BNWL-33.

DAMAGE MECHANISMS - F. A. Smidt

The objective of this program is to determine how the mechanical properties of irradiated iron are modified by interaction of moving dislocations with irradiation-induced defects. The method being utilized is the application of thermally activated flow theory to the deformation process in irradiated iron.

It has been established that irradiation to an exposure of 5×10^{18} nvt ($E > 1$ MeV) produced detectable changes in the activation volume of both Ferrovac E and a higher purity zone refined iron, even though no damage is observed directly with the electron microscope. ⁽¹⁾ During this quarter, a major portion of the time was devoted to preparation of a report describing and interpreting the results obtained to date on irradiated and unirradiated iron.

Among the significant results of this analysis of thermally activated flow in iron was the discovery that the approximation $\left(\frac{\Delta \ln \dot{\gamma}}{\Delta \tau^*} \right)_T = \left(\frac{d \ln \dot{\gamma}}{d \tau^*} \right)_T$ commonly used to obtain activation volumes is not valid in regions where the $\ln \dot{\gamma} - \tau^*$ curve is changing rapidly. Although such measurements are useful for comparing two specimens deformed at the same temperature, the result measured is the secant to the curve rather than the desired tangent. In regions where the curvature is small, the secant approaches the tangent and little error results. Difficulty is encountered however when one attempts to formulate a consistent set of equations which fits the experimental results obtained from both strain rate change tests and temperature change tests. Clearly, in such cases the tangent to the curve is required. One such case is the calculation of activation enthalpies from experimental tensile data, $H = -TV^* \left(\frac{\Delta \tau^*}{\Delta T} \right)_{\dot{\gamma}}$. In the low stress region (about 200 to 300 °K), the

(1) F. A. Smidt. "Effects of Irradiation on Thermally Activated Flow in Iron," J. of Applied Physics, vol. 36, p. 2317. 1965.

values of H calculated from this equation deviate from a linear relation.

This deviation has been attributed to a change in mechanism or poor accuracy in the experimental values of $\left(\frac{\Delta\tau}{\Delta T}\right)_{\dot{\gamma}}$.

Using data from double strain-rate change tests and a standard approximation method to obtain the tangents at τ_1^* , τ_2^* , and τ_3^* (the effective stresses at $\dot{\gamma}_1$, $\dot{\gamma}_2$, and $\dot{\gamma}_3$) it was demonstrated that the H versus T relation was linear to the highest temperature where thermal activation was controlling the deformation. This confirmed the observations made exclusively from temperature change tests that no change in mechanism was apparent in this temperature region. It is concluded therefore that the presence of impurity atom clusters, small precipitates, or clusters of interstitials produced by radiation damage modifies the low temperature Peierls mechanism but does not substitute an alternate mechanism in this temperature range.

SWELLING OF IRRADIATED FISSIONABLE MATERIALS - J. L. Brimhall,
R. D. Leggett, B. Mastel, K. R. Merckx, and H. A. Taylor

When fissionable metals are irradiated, the ordered metallic structure is perturbed in a number of ways. High energy neutrons and fission fragments collide with metal atoms, knocking them out of their structural positions. The resultant defects, vacancies, interstitials, dislocation loops, etc., can adversely affect metallic properties. Similarly, the fission products generated, some of which are noble gases, may be incompatible with the structure and so precipitate or coalesce as second phase inclusions or gas pores. Furthermore, the high localized temperatures associated with the fission event can cause anisotropic fissionable metals such as uranium to drastically change shape in accordance with the crystallography of the specimens. These effects and their interactions are being studied with regard to the influence of irradiation variables such as the specimen temperature, burnup, burnup rate, and external pressure, and of metallurgical variables such as composition, structure, and geometry. The data thus far obtained on this program have established that, contrary to previous beliefs, swelling at low burnup in alpha uranium is due to mechanical processes associated with the irradiation "growth" phenomena and with fission event induced defects rather than the agglomeration and growth of fission gas pores. This is manifested as a maximum in the swelling temperature curve in the 400 to 600 °C range. Swelling continues to decrease above 600 °C up to about 700 °C. Above 700 °C, fission gas swelling begins to occur. A few tenths of a percent alloying addition or an applied external pressure have been found to be effective in overcoming this "mechanical" type of swelling. Efforts are continuing to better define the basic mechanisms involved and the interrelationships between burnup, temperature, pressure, and metallurgical state. Fundamental information developed by this program will provide a basis for engineering exploitation of the many beneficial attributes of metal fuels for use in reactor applications.

Irradiation Program

A swelling capsule controlled at 625 °C and 1000 psi reached its goal exposure and was discharged. Another capsule was charged and is operating at 450 °C and 500 psi. Two others have been assembled together in tandem for irradiation at 575 and at 625 °C, both at 500 psi. This tandem arrangement not only permits a more efficient utilization of reactor space but also provides an identical in-reactor experiment for more precise inter-comparison of the effects of temperature and pressure. Each of the above capsules contains six high purity uranium, two U + Fe-Si, and two U + Al-Fe-Si half-tubular specimens having several heat treatments.

Construction has begun on two general swelling capsules which are intended for irradiation at a controlled temperature of 700 °C at low pressure. In this type capsule the pressure is not controlled and is estimated to be about 30 psi. The intent of these capsules is to provide higher burnup data for high purity uranium irradiated in the beta and to evaluate the influence of minor alloying additions on this behavior. Each capsule contains six high purity uranium specimens, two U + Fe-Si specimens, and two U + Al-Fe-Si specimens. It is of extreme interest to establish if the excellent behavior of dilute alloys relative to high purity uranium in the range 400 to 600 °C extends up to 700 °C. It will be recalled that low burnup data (0.03 at. % burnup) on high purity uranium⁽¹⁾ revealed very little swelling at 700 °C. The limited swelling that was observed was primarily due to fission gas porosity. Thus, these irradiations will provide insight into fission gas swelling in uranium.

The controlled atmosphere welding and NaK handling box has been modified for remote manual or semiautomatic operation. With the exception of backfilling the box with high purity helium, all operations related to control of box atmosphere are regulated from a single control panel. Vacuum pumps, air-operated valves, and gages are provided with appropriate interlocks to eliminate operator errors.

(1) Quarterly Progress Report, Metallurgy Research Section, October, November, December, 1964, edited by J. J. Cadwell; HW-84573, pp. 3.39-3.50. General Electric Company, Richland, Washington. January 15, 1965.

Postirradiation Examinations.

Metallography and density measurements were completed on the specimens removed from capsule P-3 which was controlled at 575 °C and 1000 psi. The specimens were irradiated to 0.15 at. % BU of total atoms. Table 3.1 summarizes the metallurgical and irradiation histories and the observed swelling in the 10 specimens contained in this capsule. For purposes of comparison, the swelling that was observed in similar specimens irradiated in low pressure (≈ 20 psi) capsules is also included in Table 3.1. As can be readily seen, this pressure was effective in reducing the amount of swelling that occurs in high purity uranium in the as-extruded condition by a factor of from 5 to 10. Swelling was reduced by a factor of 20 to 40 in the large grained, high purity uranium specimens that were beta heat treated prior to irradiation. The difference in behavior between the as-extruded and beta heat treated specimens is attributed to the fact that the grain size is quite small in the as-extruded condition but is quite large after beta treating.

The alloy specimens also derived some benefit from the pressure. Alloy "B" was reduced about a factor of 5 while alloy "A" was reduced by perhaps a factor of 2. It should be noted, of course, that both alloys showed considerable improvement over the high purity specimens during low pressure irradiations, particularly alloy "A". The amount of swelling experienced by alloy "A" is extremely small and of about the same magnitude as the accuracy of the measurements.

Metallographic examination revealed that the influence of pressure was to severely restrict the formation of crystallographically aligned tearing that occurs during low pressure irradiation between 500 and 600 °C. It apparently had little or no effect on the grain boundary porosity that forms at 575 °C. The structure present in the as-extruded specimen, P-3 - C-1, irradiated at 575 and 1000 °C to 0.15 at. % BU is illustrated in Figure 3.4. This can be compared with Figure 3.5 which shows the structure of a similar specimen irradiated at a low pressure to only 0.10 at. % BU. The sample located at the end of the pressurized capsule (P-3) operated at only

TABLE 3. 1

SWELLING OF SPECIMENS IN CAPSULE P-3 IRRADIATED AT 575 °C AND 1000 psi TO 0. 15 at. % BU

Specimen	Type Material	Heat Treatment	Irradiation Temperature, °C		As-Irradiated, ρ_f , cm ³	% Swelling, $\frac{\rho_i - \rho_f}{\rho_f} \times 100$	"R" = % Swelling, At. % BU	"R" for Comparable Specimen Irradiated at Low Pressure
			Reactor Full	Power Zero				
A-1	T-3*	As-Extruded	540	510	17. 15 17. 33	11 10	70 65	400
A-2	T-3	As-Extruded	540	510	18. 07	5	35	400
B-1	A**	As-Extruded	565	540	18. 72	1	6	10
B-2	A	730 °C - 15 min Water Quench in the bulk prior to machining	565	540	18. 82	0. 4	3	10
C-1	T-3	As-Extruded	575	575	18. 42	3	20	330
C-2	T-3	As-Extruded	575	575	16. 88 16. 85	13 13	85 85	330
D-1	T-3	730 °C - 15 min Oil Quench	570	565	18. 81	1	7	330
D-2	T-3	730 °C - 15 min Oil Quench	570	565	18. 58	2	15	330
E-1	B†	1000 °C - 24 hr Water Quench	550	535	18. 13	5	35	120
E-2	B	1000 °C - 24 hr Water Quench + 730 °C - 15 min Oil Quench	550	535	18. 41	3	20	120

* High Purity Uranium; 65 ppm Fe, <5 ppm Al, 24 ppm Si, 6 ppm C

** U + Fe-Al-Si Alloy; 400 ppm Fe, 640 ppm Al, 85 ppm Si, 500 ppm C

† U + Fe-Si Alloy; 140 ppm Fe, ≈ 25 ppm Al, 95 ppm Si, 400 ppm C

FIGURE 3.4

3200X

Grain Boundary Tearing in As-Extruded
High Purity Uranium Specimen P-3 - C-1
Irradiated at 575 °C and 1000 psi to 0.15 at. % BU
($\Delta V/V = 3\%$)

Neg. 3148A

FIGURE 3.5

3100X

Grain Boundary and Crystallographically Aligned Tearing
in As-Extruded High Purity Uranium Specimen 17A-1
Irradiated at 575 °C at Low Pressure to 0.10 at. % BU
($\Delta V/V = 38\%$)

Neg. 2262C

about 540 °C and contained a few small patches that exhibited aligned tearing in addition to the grain boundary tearing. The tearing in this sample (P-3 - A-1) was more profuse than in the specimen (P-3 - C-1) that operated at 575 °C, consistent with the larger density change in A-1.

Tearing was very limited in the beta treated high purity uranium specimen (P-3 - D-1) and was largely associated with twin bands. The alloy "B" specimen (P-3 - E-1) appeared similar except that more tearing was present. The amount of tearing in the latter was sufficient in some regions of the specimen to have an aligned appearance. As expected, very little tearing was present in the alloy "A" sample.

None of the specimens exhibited any general matrix porosity that would suggest fission gas porosity. This is wholly consistent with previous observations that fission gas porosity is not an important consideration in the swelling of uranium below 600 °C.

REACTOR METALS RESEARCH

ALLOY SELECTION - T. T. Claudson

Several nickel-base alloys are being irradiated and tested to determine the effects of irradiation and environment upon their mechanical properties. These alloys are being irradiated over a temperature range of 50 to 740 °C and tested at room temperature, 300 and 650 °C.

The results of room temperature tests upon Hastelloy N irradiated at 280 °C were reported in the last Quarterly Progress Report.⁽¹⁾ Tests performed at 300 and 650 °C have now been completed for specimens irradiated to an exposure of 1.91×10^{20} nvt at 50, 280, and 740 °C and results are shown in Figure 4.1. The strength of Hastelloy N has been found to increase with decreasing test temperature. However, the corresponding decrease in ductility was not found as might be expected. As the test temperature was increased from room temperature to 300 °C, a small increase in uniform and total elongation was found with a corresponding increase in percent reduction in areas from 12.8 to 21.5%. At a test temperature of 650 °C, a marked decrease in uniform and total elongation along with a decrease in percent reduction in area indicated an embrittling effect in the material. Control tests now being prepared will indicate the nature of this embrittling reaction in the material.

Figure 4.2 shows the effect of neutron irradiation at 50 °C upon the mechanical properties of Hastelloy N tested at 650 °C. Comparison of the data for the irradiated specimens to that of the control specimen shows a marked decrease in ultimate strength and ductility of the material. The effect of the irradiation has been to increase the yield strength approximately 38%. The benefit of this higher strength cannot be realized however because of the fourfold loss in ductility.

(1) Quarterly Progress Report, Metallurgy Research Operation, January, February, March, 1965, edited by J. J. Cadwell, BNWL-79. April 15, 1965.

FIGURE 4.1

Effect of Test Temperature Upon the Mechanical Properties
of Hastelloy N Irradiated at 740 °C to an Exposure
of 1.91×10^{20} nvt ($E > 1$ MeV)

FIGURE 4.2

Effects of Neutron Irradiation at 50 °C Upon the Mechanical Properties of Hastelloy N Tested at 650 °C in the Solution Treated Condition

A more severe loss in ductility was found in specimens of Hastelloy N irradiated at 280 °C and tested at 650 °C as shown in Figure 4.3. In this case, a slight increase in yield strength was found for specimens irradiated to 1.03×10^{20} nvt. Ductility was decreased as shown by the percent uniform elongation which was 20.22% for the unirradiated specimen to 12.71% for the irradiated specimen. For specimens irradiated to a higher exposure of 1.46×10^{21} nvt the yield strength was actually decreased as compared to the unirradiated specimen. Also, an almost complete embrittlement of the material occurred as shown by the percent uniform elongation value of 0.3%.

Similar results have been found for the Hastelloy X-280 material. Figure 4.4 shows the effect of the neutron irradiation at 280 °C upon the mechanical properties of Hastelloy X-280 after testing at 650 °C. The results of these tests show an initial decrease in strength properties with increasing exposure. At 3.2×10^{20} nvt the yield strength is about the same as the control but a marked decrease in ultimate strength is still prevalent. Decreases in ductility are also found as shown by the 14% uniform elongation for the irradiated specimen as compared to the 52% for the unirradiated specimen.

Figure 4.5 shows the effects of neutron irradiation upon Hastelloy X-280 irradiated at 740 °C and tested at 650 °C. These specimens reached an exposure of 1.9×10^{20} nvt after which a marked decrease in ductility was measured. A difference in plastic flow behavior was also found in that for the case of the control specimen the ultimate strength was achieved at about 15% elongation with fracture occurring after an elongation of 44%.

In the case of the irradiated specimen, fracture occurred shortly after reaching the ultimate load at 6.13%. The uniform elongation of this irradiated material was only 5.76% as compared to 15% for the control.

Several specimens of Incoloy 800 were irradiated to an exposure of 1.9×10^{20} nvt in the mill annealed, 20% cold worked, and solution treated conditions at 740 °C and tested at 650 °C. These data are shown in Figure 4.6

FIGURE 4.3

Effects of Neutron Irradiation at 280 °C Upon the Mechanical Properties
of Hastelloy N Tested at 650 °C in the Solution Treated Condition

FIGURE 4.4

Effects of Neutron Irradiation at 280 °C Upon the Mechanical Properties
of Hastelloy X-280 Tested at 650 °C in the Solution Treated Condition

FIGURE 4.5

Effects of Neutron Irradiation Upon the Mechanical Properties
of Hastelloy X-280 Irradiated at 740 °C
in the Solution Treated Condition and Tested at 650 °C

FIGURE 4. 6

Effects of Preirradiation Treatment Upon the Mechanical Properties
of Incoloy 800 Irradiated at 740 °C and Tested at 650 °C

and indicate substantial losses in ductility for all conditions. While all materials have approximately the same uniform elongation (16.5%) in the unirradiated case, the irradiated specimens show a uniform elongation of 3.7, 7.6, and 9.6% for the mill annealed, 20% cold worked, and solution treated conditions, respectively. A loss in yield and ultimate strength was also found for all specimens tested.

Additional tests have been completed on specimens of Inconel 600 in the mill annealed, 20% cold worked, and solution treated conditions which were irradiated at 740 °C to an exposure of 1.9×10^{20} nvt and tested at 650 °C. The results of these tests are shown in Figure 4.7. As in the case of the Incoloy 800 shown in Figure 4.6, marked decreases in ductility occurred due to the irradiation. The percent uniform elongation for the irradiated specimens in the mill annealed, cold worked, and solution treated specimens was 2.5, 2.8, and 7.6, respectively as compared to 16.7, 18.7, and 22.4 for the unirradiated controls. Most interesting were the small changes in yield strength for each specimen condition while relatively large reductions in ultimate strength were associated with the reduced ductility.

Similar specimens of Inconel 600 were irradiated at 280 °C and tested at 650 °C in the solution treated condition. The results of these tests are shown in Figure 4.8 and compared to an unirradiated control specimen tested under the same conditions. These tests indicate that a reduction in ductility occurred as a result of the irradiation as shown by the reduced percent uniform and total elongations and reduction in area. Moreover, these reductions appear to be a function of exposure shown as increased reduction in ductility with increasing exposure. Associated with the reduction in ductility is a marked increase in yield strength and decrease in ultimate strength.

To evaluate the effects of environment and neutron irradiation upon the mechanical properties of reactor structural materials, a liquid metal temperature controlled capsule has been designed. A bench model of this capsule was fabricated and tested using an electrical heater to simulate

FIGURE 4.7

Effects of Preirradiation Treatment Upon the Mechanical Properties of Inconel 600 Irradiated at 740 °C and Tested at 650 °C

FIGURE 4.8

Effects of Neutron Irradiation at 280 °C Upon the Mechanical Properties
of Inconel 600 Tested at 650 °C in the Solution Treated Condition

the gamma heat generation. Since this test simulated only the heat generation of the specimens, neglecting that of the capsule itself, heat transfer calculations were necessary to determine:

- If the maximum temperature of the capsule is below the maximum prescribed (1500 °F), and
- The temperature distribution inside the capsule itself, particularly inside the system containing the liquid sodium coolant.

A temperature profile was calculated for the capsule by superposing the effect of each heat generating constituent. As a check, that portion of the curve relative to the heat generation simulated in the bench test was compared to the actual temperature distribution recorded during the test. Correlation between the theoretical and actual experimental data was about 7%.

Next, an effort was made to determine the temperature rise across the upflow leg of the capsule, using the method proposed by D. P. Timo.⁽¹⁾

The comparison of these values to those actually obtained is shown in Figure 4.9. A digital program is now being written to determine the distribution in the capsule, using combined relaxation and Schmidt plot methods. Results of these calculations will predict the axial and radial temperature distribution through this capsule.

(1) D. P. Timo. Free Convection in Narrow Vertical Sodium Annuli, KAPL-1082, Knolls Atomic Power Laboratory, Schenectady, New York.

FIGURE 4.9

Comparison of the Theoretical and Experimental Temperature Difference as a Function of the Heat Generation for the Liquid Metal Irradiation Capsule

IN-REACTOR MEASUREMENTS OF MECHANICAL PROPERTIES -

J. A. Williams, D. H. Nyman, and J. W. Carter

The objective of in-reactor measurements is to study and define the effects of neutron irradiation on the mechanical properties of structural materials. Current program efforts include the determination of the in-reactor stress and temperature dependency of creep of annealed 304 SS, annealed Zircaloy-2 and a cursory study on the in-reactor creep of Zr-2.5 wt% Nb alloy. Preirradiation characterization of the creep-to-rupture properties of Hastelloy X is continuing. Development work on a high temperature creep capsule is also being done.

In-Reactor Creep of Annealed 304 SS

The data to date of in-reactor creep of annealed 304 SS is summarized in the Arrhenius plot of Figure 4.10. The minimum creep rates in-reactor are compared to out-of-reactor rates as a function of the reciprocal absolute temperature. Within the scatter of experimental data there is no effect, during irradiation, of the neutron irradiation on the minimum creep rate. There are some differences, however, between in-reactor and out-of-reactor creep characteristics. Of greatest significance is the reduced creep to rupture life during neutron irradiation. This is illustrated by the data in Table 4.1.

TABLE 4.1
IN-REACTOR CREEP-RUPTURE PROPERTIES
OF ANNEALED 304 SS

Temperature, °C	Stress, psi	% Creep Strain at Failure		Failure Time in Hours	
		In-Reactor	Out-of-Reactor	In-Reactor	Out-of-Reactor
650	20,000	8 to 9	25 to 26	150	570
550	30,000	3 to 4	8	770	4500

Observations of the in-reactor activation energy studies also indicate a difference in the nature of in-reactor creep process for annealed 304 SS. Activation energies determined by the temperature change method reported in a previous quarterly report⁽¹⁾ indicated that with increasing temperature

(1) Quarterly Progress Report, Metallurgy Research Operation, October, November, December, 1964, edited by J. J. Cadwell, HW-84573, January 15, 1965.

FIGURE 4.10

Arrhenius Plot of In-Reactor and Out-of-Reactor Creep Rates
of Annealed 304 SS

the activation energy increased from a value of 100 to 135 kcal/mole, the transition point of which is shifted to lower temperatures with increasing stress. However, minimum creep rates observed during these activation energy studies at 30,000 psi and plotted in Figure 4.10, do not indicate increased activation energy. If this were the case, the in-reactor creep rates should diverge upward from out-of-reactor rates at temperatures above 550 °C for the 30,000 psi data. The difference in the activation energy as measured by temperature change method and from the slope of the Arrhenius plot, suggests an irradiation modification of the primary stage of in-reactor creep.

The mechanism by which this may occur is as yet undetermined but may be of the same nature which contributes to the reduction of in-reactor creep rupture properties since it requires thermal activation and is stress dependent.

IRRADIATION FACILITIES OPERATION - J. E. Irvin

The purpose of this phase of the program is to provide for the timely accomplishment of irradiating, testing, and subsequent processing of the data obtained from a number of different structural materials. Irradiations will be performed in aqueous, gaseous, and molten metal environments. Elevated temperature irradiations and tests will be emphasized. Special tests applicable to both irradiated and unirradiated specimens will be developed.

Facilities Operation

The experiments discharged from the various Engineering Test Reactor (ETR) core positions at the conclusion of ETR Cycle 72 are given in Table 4.2 along with the estimated neutron fluxes and exposures (>1 MeV). The operating histories of the ETR and the G-7 Hot Water Loop are given in Table 4.3. No discharges were made at the end of ETR Cycle 71. The G-7 Loop was not operative because of a leak which developed in a welded joint in the sub-pile room. The leak was repaired after the loop was decontaminated during Cycle 72 shutdown. Normal operation was resumed during ETR Cycle 72. Control tests discharged from the out-of-reactor loop are given in Table 4.4. No discharges were made at the end of Cycle 68. Because of the loss of ETR Cycle 71 for irradiation purposes, it was not necessary to schedule this cycle in the out-of-reactor loop. An exposure duplicating the ETR Cycle 72 operation of the G-7 Loop is now in progress in this control loop.

All of the special quadrants containing BONUS transition joint tensile specimens have been discharged from the G-7 or out-of-reactor loops after having accumulated the scheduled exposures. All of the specimens have been examined and tested. Hydrogen analysis and metallography are being performed on specimens exposed in either the G-7 or out-of-reactor loop for greater than 2200 hr. This study is being conducted to evaluate the effects of irradiation and environment on Zircaloy-2-Inconel 600 transition tube joints. Overall responsibility for evaluation of these tube joints rests with Combustion Engineering, Inc. on the Advanced Bonus Core Development Program. A topical report is being prepared to present the results obtained.

TABLE 4.2
SUMMARY OF EXPERIMENTS DISCHARGED
FROM THE ETR FOLLOWING CYCLE 72

<u>Experiment Designation</u>	<u>Number and Type of Specimen</u>	<u>Estimated Flux, nv^(a)</u>	<u>Estimated Exposures, nvt^(a)</u>
<u>Quadrant Discharges - ETR Cycle 72</u>			
149	18 Zircaloy-2 Tensiles	9.0×10^{13}	6.4×10^{20}
150	18 Zircaloy-2 Tensiles	8.0×10^{13}	6.0×10^{20}
172	6 348 SS Tensiles	1.1×10^{14}	1.0×10^{21}
187	6 Inconel X-750 Tensiles	6.0×10^{13}	1.1×10^{20}
190(b)	2 Zircaloy-2 DCB ^(c)	4.2×10^{13}	7.3×10^{19}
<u>Capsule Discharge - ETR Cycle 72</u>			
322	8 Zircaloy-2 Tensiles	6.9×10^{13}	5.0×10^{21}
654	8 304 SS Tensiles	4.7×10^{13}	9.6×10^{20}
656	8 304 SS Tensiles	4.2×10^{13}	8.9×10^{20}
659	8 348 SS Tensiles	7.2×10^{13}	1.1×10^{21}
661	8 348 SS Tensiles	6.9×10^{13}	9.8×10^{20}
662	8 348 SS Tensiles	1.3×10^{14}	1.2×10^{21}

- (a) Neutrons with energies greater than 1 MeV.
- (b) This quadrant is twice the normal quadrant length. The average flux and exposure were estimated for the middle of the specimens.
- (c) Double cantilever beam.
- Total number of specimens discharged, Cycle 71 and 72 = 98
- Total number of specimens discharged from G-7 Hot Water Loop, Cycles 71 and 72 = 50.
- Total number of specimens discharged from G-7 Loop to date = 2416

TABLE 4.3
OPERATING HISTROY OF ETR AND G-7 HOT WATER LOOP FAC

<u>Operating History</u>	<u>ETR Cycle Number</u>	
	<u>70</u>	<u>71</u>
1. <u>Reactor History</u>		
Startup(a)	2-27-65	4-7-65
End of Cycle	3-26-65	5-10-65
Megawatt Days	2899	2982
Effective Days at 175 MW	16.6	17.0
Number of Scrams ^(b)	15	- -
Number of Shutdowns	3	5
2. <u>G-7 Hot Water Loop History</u>		
Maximum Temperature	530 °F	- - (e)
Effective Days Above 200 °F	18.2	- -
Effective Days at Operating Temperatures	14.0	- -
Operating Efficiency ^(c)	109.6%	- -
Temperature Efficiency ^(d)	76.9%	- -

- (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 with respect to effective days of reactor operation at 175 MW.
- (d) Effective days of operation between 500 and 550 °F with respect to days of operation above 200 °F.
- (e) No irradiations were conducted in the G-7 Loop during this cycle because of a leak in the subpile room piping.

TABLE 4.4
HISTORY AND DISCHARGE SCHEDULE FOR OUT-OF-REACTOR LOOP

Loop History	Cycle Number		
	68	69	70
<u>1. Time at Temperature (in hours)</u>			
200 °C	40.1	18.0	50.5
218 °C	13.5	2.0	15.8
246 °C	47.5	6.5	34.5
282 °C	471.9	440.0	336.2
Total time in hours	572.0	466.5	437.0
<u>2. Specimen Discharge (material, type, and number)</u>			
Nickel-Base Tensiles	--	--	34
Nickel-Base Stress Rupture	--	--	8
Zircaloy-2 Tensiles	--	18	--
BONUS Transition Tensiles	--	--	6
Total number discharged		18	48
Total number of specimens discharged to date = 1204			

Data Processing

The retrieval portion of the REM Computer Program has been programmed, debugged, and utilized to generate trial reports. This program provides for editing, processing, storage, and retrieval of tensile and associated metallurgical data. A schematic of the program functions related to processing of tensile test data is given in Figure 4.11. The capability for processing other types of mechanical test data generated in sufficient volume is also illustrated.

FIGURE 4.11

Schematic Representation of the REM Computer Program

Initially, very simple program requests have been generated to develop a familiarity with the system. Additional tests are being conducted to explore the capabilities of this program for processing metallurgical data. Figure 4.12 illustrates a request generated by an engineer with no programming experience. From a single sheet of instructions on how to specify formats; how to specify multiline reports; and similar routine report features, an endless variety of reports can be generated. Very few cards are needed for any given report. The report requested in Figure 4.12 is for one material only; however, by changing one card, the same report can be obtained for any material of interest. One page of the 30 to 40 page report generated from this request is illustrated in Figure 4.13.

Reports similar to the one illustrated in Figure 4.13 containing tables of data will be linked to analytical and statistical subroutines to establish curve fits on the raw data. These results will then become input for automatic plotting equipment. The file currently contains over 2000 tensile records on approximately 30 different materials. Tensile records are being added to this file at a rate of approximately 100/mo. As the number of records continues to increase, it becomes less and less feasible to produce the comparative plots on many materials and/or parameters in a reasonable length of time or for a price many budgets can support. Consequently, the continual development of this program will be emphasized in order to obtain data which is statistically significant in a shorter time and for a reasonable cost.

80 80

1 REQUESTED BY

IRVIN J.E.

• **DATA**

4-26-65

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TEC 721

4.21

BNWL-120

Typical REM Report Processing Request

SEQ NO	MATERIAL NAME	MAT HISTORY	TEST	INTR.	OFFSET	UT TEN	ELONGATION	RED IN	PLASTIC	
EXPERIMENT IDENTIFICATION		CW ORI WT	TEMP	FLUX	YIELD STRESS	STRESS	UNI TOTAL	AREA	WORK	
Q00196	304 STAINLESS	25 TD	70°	1.11E 20	150.7	157.6	0.8	5.2	49.8	25.5
GEH-14-461-06	HAPO RM									
Q00282	304 STAINLESS	25 TD	70°	1.72E 20	141.7	146.3	1.4	5.5	56.0	27.3
GEH-14-365-06	HAPO RM									
Q00220	304 STAINLESS	25 TD	70°	1.77E 20	142.5	157.2	1.9	7.2	66.2	37.4
GEH-20-050-15	HAPO RM									
Q00293	304 STAINLESS	25 TD	70°	1.77E 20	146.4	162.4	2.5	7.3	53.3	39.2
GEH-20-050-13	PPC									
Q00294	304 STAINLESS	25 TD	70°	1.77E 20	152.4	164.9	2.0	8.3	53.4	35.9
GEH-20-050-14	PPC									
Q00328	304 STAINLESS	25 TD	70°	3.46E 20	154.6	165.7	1.9	40.0	57.4	49.4
GEH-20-052-13	PPC									
Q00329	304 STAINLESS	25 TD	70°	3.46E 20	159.4	165.6	1.9	7.3	56.6	40.8
GEH-20-052-14	PPC									
Q00215	304 STAINLESS	25 TD	70°	7.10E 20	152.2	163.4	2.0	6.6	54.6	35.0
GEH-20-038-15	HAPO RM									
Q00378	304 STAINLESS	25 TD	70°	7.10E 20	177.0	185.6	1.3	6.0	47.0	35.7
GEH-20-038-13	PPC									
Q00379	304 STAINLESS	25 TD	70°	7.10E 20	164.2	180.4	2.0	5.9	52.4	35.4
GEH-20-038-14	PPC									
Q00187	304 STAINLESS	25 TD	70°	1.09E 21	151.0	156.7	1.2	5.6	50.0	27.9
GEH-14-463-05	HAPO RM									
Q00188	304 STAINLESS	25 TD	70°	1.09E 21	150.5	154.7	1.1	5.4	48.8	26.4
GEH-14-463-06	HAPO RM									
Q00216	304 STAINLESS	25 TD	70°	1.43E 21	162.6	174.4	2.0	6.4	48.8	36.2
GEH-20-042-15	HAPO RM									
Q00345	304 STAINLESS	25 TD	70°	1.43E 21	170.4	179.4	1.4	5.2	49.5	30.7
GEH-20-042-13	PPC									

FIGURE 4.13

Sample Page from REM Report

4.22

BNWL-120

IRRADIATION DAMAGE TO STAINLESS STEELS - J. E. Irvin

The purpose of this phase of the program is to determine the combined effects of irradiation and reactor environment on stainless steels. Changes in mechanical properties will be determined for several irradiation and test temperatures to establish under what conditions these alloys are most susceptible to failure. Particular emphasis will be placed on establishing the service conditions under which these alloys are metallurgically unstable and to determine the mechanisms by which irradiation-induced property changes occur.

Austenitic Stainless Steels

Tensile tests have been conducted on irradiated and unirradiated specimens of 348 and 304 SS in both the annealed and 25% cold-worked condition. Tests have been conducted from room temperature to 850 °C on the control specimens and from room temperature to 750 °C on the irradiated materials. The effect of test temperature on the strength of these materials in the as-fabricated conditions is illustrated in Figure 4.14. Specimens from both the transverse and longitudinal directions were tested for the 348 SS in both conditions. All of the 304 SS specimens were taken from the transverse condition.

The strength of these materials decreases gradually to temperatures between 200 to 300 °C. Between 300 and 500 °C, a plateau exists where the change in strength is affected very little by increasing test temperature. Above 450 to 500 °C, the strength is decreased fairly rapidly for increasing test temperatures. The decrease is much more pronounced in the 304 SS than in the 348 SS. Generally, the 0.2% offset yield strength will show a more pronounced change than the ultimate tensile strength. There is essentially no difference between the values of transverse and longitudinal specimens of 348 SS in the annealed condition. However, the yield and ultimate strengths are higher in the transverse specimens in the 25% cold-rolled condition than comparable specimens from the longitudinal direction.

FIGURE 4.14

Effect of Test Temperature on the Strength of 348 and 304 SS
(Specimens tested in as-fabricated condition)

Between 500 and 700 °C cold-work recovery occurs, and the amount of grain boundary deformation also increases, resulting in a drastic change in the strength and ductility of the cold-worked material. The cold-work recovery of the 25% cold-rolled 348 SS specimens from the transverse direction as a function of annealing temperature is shown in Figure 4.15. Most of the strength is recovered over a very narrow temperature range between 650 and 750 °C. The ductility recovery however occurs over a much broader range between 300 and 750 °C. All of the tests were conducted at room temperature. Specimens were annealed at temperature for 4 hr in air. The isocronal anneals were performed at 100 degree increments beginning at 100 °C. Two or more specimens were tested for every annealing temperature.

The effects of test temperature on the ductility of the 348 and 304 SS examined in the as-fabricated conditions are shown in Figures 4.16 through 4.18. Between room temperature and about 300 °C the elongation decreases steadily with increasing test temperatures. Between 300 and 750 to 800 °C two ductility minimums are present in the annealed material and an extended plateau is present in the cold-worked material. The location of the minimum and maximum ductility as a function of test temperature varies considerably between the two materials. At 850 °C the 304 SS is decreasing after having reached a maximum at about 700 to 750 °C. The 348 SS ductility is increasing at 850 °C, however, and appears to be reaching a maximum in this temperature range.

Figure 4.16 shows there is little difference between the total and uniform elongation at test temperature between room temperature and 500 °C. Beyond this, the total elongation tends to increase and the uniform elongation continues to decrease. The increase in total elongation at these elevated test temperatures occurs as a result of an increase in the necking elongation. This is illustrated for the annealed materials only in Figure 4.18.

The same trends observed for elongation are also exhibited for reduction of area. The data scatter is larger for this particular property and some irregularities are observed at test temperatures between room temperature

FIGURE 4.15

Cold-Worked Recovery of 348 SS
(All specimens 25% cold worked by rolling)

FIGURE 4.16

Effect of Test Temperature on the Ductility of 348 and 304 SS
(Specimens tested in as-fabricated condition)

FIGURE 4.17

Effect of Test Temperature on the Ductility of 348 and 304 SS
(Specimens tested in as-fabricated condition)

FIGURE 4.18

Effect of Test Temperature on the Ductility of Annealed 348 and 304 SS
(Transverse specimens)

and 300 °C as shown in Figure 4.17. Reduction of area measurements also shows a marked difference due to the orientation of the tensile axis in either the transverse or longitudinal direction. The ductility of the longitudinal specimens is greater at all temperatures because of the rolled-in impurities.

The higher ductility and toughness of the 348 SS at the very high temperatures result from grain boundary strengthening. This strengthening causes a high temperature ductility maximum between 700 and 900 °C, depending on the alloy content and fabrication method. Figure 4.19 illustrates the grain size dependence noted between a fine grain and a coarse grain austenitic stainless steel. The two alloys being used in the present study are also included for comparison. It should be noted that the ductility of the 340 SS is greater than that of the 348 SS for all test temperatures below about 800 °C. Both the materials used in this study have a fine grain size. The grain size for the 348 SS is about ASTM No. 10 to 11, and the grain size for the 304 SS is about ASTM No. 9 to 10. These materials were air cooled from the annealing temperature.

Generally, neutron irradiation increases the strength and decreases the ductility of these materials as is normally expected. However, the amount of change as a function of test temperature and irradiation temperature indicates there are a number of dynamic processes competing to produce these changes. Several test temperatures and irradiation temperatures have been used in studying these effects on stainless steels, and a portion of these results have been reported.⁽¹⁾ The data presented here are more recent tests on the same materials and are part of a continuing irradiation program.

The effects of test temperature on the ductility of 304 and 348 SS irradiated at 290 °C in the ETR G-7 Hot Water Loop are illustrated in Figures 4.20 through 4.22. Although the greatest change occurs at room temperatures as a function of increasing integrated exposures, the

(1) J. E. Irvin, A. L. Bement, and R. G. Hoagland. The Combined Effects of Temperature and Irradiation on the Mechanical Properties of Austenitic Stainless Steels, BNWL-1. January, 1965.

FIGURE 4.19

Effect of Test Temperature on the Ductility of Austenitic SS
(Total elongation)

FIGURE 4.20

Effect of Test Temperature on the Ductility of Irradiated 348 and 304 SS
in the Annealed Condition
(Irradiation temperature, ~ 290 °C)

FIGURE 4.21

Effect of Test Temperature on the Ductility of Irradiated 348 and 304 SS
in the Annealed Condition
(Irradiation temperature, $\sim 290^\circ\text{C}$)

FIGURE 4.22

Effect of Test Temperature on the Ductility of Irradiated 348 and 304 SS
in the Annealed Condition
(Irradiation temperature, ~290 °C)

temperature range between 200 and 400 °C exhibits the lowest absolute ductility. In Figure 4.20, data from only materials annealed prior to irradiation are presented. Since both cold work and neutron irradiation begin to anneal out of these materials at relatively low temperatures, it is very important to know the nature of this recovery at all test temperatures used. The effects of postirradiation annealing on both low and high temperature properties are being performed and will be reported at a later date.

The effect of increasing neutron exposures is to continually lower the absolute ductility of the materials. A minimum in uniform elongation for both materials is noted between 300 and 500 °C. This corresponds to the minimum observed in the unirradiated materials at slightly higher temperatures. Therefore, the second effect of neutron irradiation is to lower the temperatures at which this ductility minimum occurs.

Very little change occurs in the reduction of area after irradiation at test temperatures below about 500 to 600 °C. Figure 4.22 illustrates, however, the change is quite severe above these temperatures for all levels of exposure. Because of the amount of data scatter that occurs in the measurement of this property, a scatter band is given for all 348 SS data plotted. Since both longitudinal and transverse specimens are plotted, this further increases the scatter. Only data for the highest exposure tested to date are plotted for 348 SS. Again, it appears that the minimum ductility has been shifted to a lower test temperature by the neutron irradiation.

The effects of test temperature on the strength and ductility of 348 SS irradiated at about 750 °C are given in Figures 4.23 through 4.26. Both annealed and 25% cold-worked material was irradiated at this temperature. Complete recovery of the cold-worked material occurred in the irradiated material and in the unirradiated control specimens exposed for similar times at temperature. A comparison of the as-fabricated, unirradiated controls, irradiated, and specimens given a postirradiation anneal for 1 hr at 1050 °C are given in Figure 4.26. Both annealed and 25% cold-worked test data are given for all conditions. Only one level of irradiation is included for comparison.

FIGURE 4.23

Effect of Test Temperature on the Strength of Irradiated 348 SS
(Irradiation temperature, ~750 °C)

FIGURE 4.24

Effect of Test Temperature on the Ductility of Irradiated 348 SS
(Irradiation temperature $\sim 750^\circ\text{C}$)

FIGURE 4. 25

Effect of Test Temperature on the Ductility of Irradiated 348 SS
(Irradiation temperature, $\sim 750^\circ\text{C}$)

FIGURE 4.26

Effects of Postirradiation Annealing on the Strength and Ductility of 348 SS

Because of the grain growth that occurs during irradiation, the strength is slightly improved and the ductility is somewhat lower at test temperatures between room temperature and about 300 °C. As illustrated in Figure 4.23 the strength of the material is lower after irradiation at test temperatures between about 300 and 600 °C. A maximum in both uniform and total elongation occurs about the midpoint of this same temperature range as shown in Figure 4.24. The reduction of area shown in Figure 4.25 exhibits the same trend observed for the lower irradiation temperatures. Above test temperatures of about 500 to 600 °C, the tendency is for the reduction in area to drop off quite rapidly with increasing test temperature.

An interim report is being prepared which will summarize the results obtained to date on several materials. The effects of neutron irradiation and environment for long term exposures are being studied but the results of these tests will not be available for some time. The purpose of this report is to review the state of knowledge with regard to irradiation effects in stainless steels so that the limited number of test specimens which will attain goal exposures of 1.0×10^{22} nvt (>1 MeV) can be used most beneficially.

IRRADIATION DAMAGE TO NICKEL-BASE ALLOYS - I. S. Levy and
K. R. Wheeler

The combination of oxidation resistance and high temperature strength places the nickel-base alloys among the most promising materials for nuclear reactor application. These alloys, however, generally suffer damage when irradiated at high temperature which cannot be recovered by postirradiation treatments. The purpose of this program is to determine the effect of modified microstructures upon the irradiation stability of nickel-base alloys. These structural modifications are made by preirradiation thermal and/or mechanical treatments and are screened by tensile and stress-rupture tests and metallographic examination.

Solid-Solution Hardened Alloys

Results previously reported⁽¹⁾ on the room temperature tensile properties of control and irradiated specimens of Hastelloy X-280 led to the conclusion that a desirable preirradiation microstructure would be one in which the matrix carbon was stabilized in a manner which would reduce the formation of embrittling grain boundary deposits. It was later shown⁽²⁾ that an experimental treatment involving cold work and subsequent aging could produce a dispersion of carbides within the matrix with reduced grain boundary deposit.

Tensile specimens representing treatments involving 4 degrees of cold work (0, 10, 15, 20%) followed by 24 hr at either 1000 or 1500 °F were irradiated to 1×10^{20} nvt at 540 F. Examples of resulting microstructures are given in Figure 4.27. These irradiated specimens and their controls were tested at room temperature and 1350 °F in air. When compared with standard treated irradiated material, some treatments showed improved high temperature ductility while maintaining strength (Figure 4.28) and, in general, susceptibility to radiation damage was lessened (Figure 4.29).

(1) Quarterly Progress Report, Metallurgy Research Operation, January, February, March, 1964, edited by J. J. Cadwell, HW-81269, General Electric, Richland, Washington. April 15, 1964.

(2) Quarterly Progress Report, Metallurgy Research Operation, April, May, June, 1964, edited by J. J. Cadwell, HW-82651, General Electric, Richland, Washington. July 15, 1964.

FIGURE 4. 27

Example of Experimental Treatment for Hastelloy X-280
Neg. 0651831-1

FIGURE 4. 28

Improved High Temperature Postirradiation Tensile Ductility of Hastelloy X-280

FIGURE 4.29

Improvement in Radiation Damage Resistance
 of Hastelloy X-280 High Temperature Properties

Further, one treatment resulted in an 84% increase in postirradiation high temperature yield strength with little ductility reduction (Figure 4.30). A regular increase of room temperature strength and decrease in ductility with cold work were observed (Figure 4.31) with a maximum increase of 63% yield strength over standard material while maintaining useful ductility of 18%.

Metallographic studies of the tensile specimen buttonheads and fracture surface are being undertaken.

Testing of stress-rupture specimens at a stress which would cause rupture in 1000 hr for a standard-treated unirradiated specimen is to be initiated during the next quarter.

Other specimens are being readied for 120 and 1300 °F irradiation.

FIGURE 4.30

Improved High Temperature Postirradiation Strength of Hastelloy X-280

FIGURE 4.31
Room Temperature Tensile Properties
of Hastelloy X-280

Precipitation-Hardened Alloys

Irradiation of an alloy containing a precipitated phase may result in solution of the precipitates, formation of additional precipitates, or the disintegration of precipitates into small pieces by a combination of these two processes.

Tensile and stress-rupture specimens of Inconel X-750 representing five different experimental heat treatments are currently being irradiated at 540 °F to a goal exposure of $\sim 2 \times 10^{20}$ nvt. Others are being prepared for 120 and 1300 °F irradiations. The experimental treatments were chosen to give a range of γ' particle size, from underaged to overaged, and micrographs representing these microstructures were presented in the last report. ⁽¹⁾

(1) Quarterly Progress Report, Metallurgy Research Operation, January, February, March, 1965, edited by J. J. Cadwell, BNWL-79.
April 15, 1965.

FRACTURE STUDIES - R. G. Hoagland and K. R. Wheeler

Effect of Geometry Variables on DCB Specimen Performances

Introduction

Unlike many specimens presently in use for determining plane strain fracture toughness data, the double cantiliver beam (DCB) type of specimen has the advantage of enabling a relatively large number of plane strain fracture toughness data to be determined from a single specimen of moderate size. This is a distinct advantage when one must conduct fracture studies on a limited volume of material, e. g., in irradiation effects programs.

The early development of a minimal size DCB specimen proceeded on somewhat of a trial-and-error basis as far as the determination of the necessary side groove depth to maintain the crack in the mid-plane of the specimen. It was soon found that different groove depths were appropriate for different materials. Since changing this groove depth necessitates a recalibration of the specimen and also since additional experiments were planned which required specimens of altogether different dimensions, it was decided to perform a number of tests to determine the effect of changing the geometry on the compliance curves. For a particular specimen, then, the constants which define the compliance curve could be ascertained by interpolation on a master plot. In addition, these tests provided the opportunity of observing the effects of changing the geometry (such as B_N/B and side groove root radius) on the value of the fracture toughness for the material studied. The specimen design is shown in Figure 4.32.

FIGURE 4.32

DCB Specimen Design

Procedure

The material that was selected for these tests was 7075-T6 aluminum. The variables studied were gross thickness (B), beam depth, side groove depth, and side groove root radius. A total of 20 specimens were prepared and their dimensions are shown in Table 4. 5.

TABLE 4. 5
TABULATION OF SPECIMEN DIMENSIONS

Specimen Number	Beam Depth, in.	Gross Width, in.	Side Groove Depth, in.	Side Groove Root Radius, in.	Section Modulus ($\times 10^{-3}$ in. ⁴)
1	0.500	0.125	0.030	≤ 0.001	1.275
2	0.500	0.250	0.094	≤ 0.001	2.329
3	0.500	0.251	0.028	≤ 0.001	2.591
4	0.500	0.376	0.155	≤ 0.001	3.194
5	0.500	0.375	0.122	≤ 0.001	3.452
6	0.500	0.376	0.090	≤ 0.001	3.663
7	0.500	0.376	0.059	≤ 0.001	3.812
8	0.500	0.501	0.217	≤ 0.001	3.885
9	0.500	0.501	0.185	≤ 0.001	4.410
10	0.500	0.501	0.152	≤ 0.001	4.530
11	0.500	0.501	0.119	≤ 0.001	4.786
12	1.000	0.125	0.030	≤ 0.001	10.320
13	1.000	0.253	0.060	≤ 0.001	20.590
14	1.000	0.376	0.090	≤ 0.001	30.283
15	1.000	0.502	0.120	≤ 0.001	39.921
16	0.500	0.250	0.060	≤ 0.001	2.497
17	0.500	0.251	0.060	≤ 0.010	2.497
18	0.500	0.251	0.060	≤ 0.050	2.497
19	0.500	0.501	0.120	≤ 0.010	4.782
20	0.500	0.501	0.120	≤ 0.050	4.773

The compliance of a specimen for a given crack length was determined from the slope of the load-deflection curve. On most of the specimens, the crack length was readily measured by blackening the side grooves and observing the crack tip with a traveling microscope to an accuracy of ± 0.001 in. The crack length was then measured on both sides of the specimen and an average value was taken. The crack length values from one side of the specimen to the other were seen to differ by 0 to 0.030 in. The crack fronts which were observable on the completely separated specimens were seen to be essentially straight. On the specimens which had a side groove root radius of 0.010 and 0.050 in., multiple cracks were observed and the crack length measurement was subject to more error than for the other specimens.

The relationship which defined the compliance behavior of the specimens was found by

$$\phi = E \frac{y}{f} \quad (1)$$

where y is the crosshead motion, f is the applied force, and E is Young's modulus which for 7075-T6 aluminum was taken to be 1.040×10^7 psi. In all cases, it was found that the compliance could be accurately represented by the empirical equation.

$$\phi = Ac^n \quad (2)$$

where c is the crack length and A and n are constants for a given set of specimen dimensions. A computer program was used which determined A and n by an iterative process.

Using Equation (2) the strain energy release rate is given by

$$\mathcal{G} = \frac{Ey^2n}{2AB_N} \left(\frac{fA}{Ey} \right)^{\frac{n+1}{n}} \quad (3)$$

Because of the large number of fracture toughness measurements which could be made from these 20 specimens, hand calculations would be far too laborious and time-consuming. A computer program was therefore developed which was used to calculate both $\mathcal{G}_{Ic}$ and K_{Ic} at crack initiation and at crack arrest. This program also computes a number of other parameters including K and crack lengths. Approximately 700 values of fracture toughness at crack initiation were obtained from the 20 specimens.

Results of Compliance Tests

The values of A and n are given in Table 4.6 along with the 95% confidence limits on these values. Figure 4.33 shows the variation in A with section modulus. For a given beam depth the relationship was found to be

$$A = \frac{c_1}{I} \quad (4)$$

where I is the section modulus. It was found that by doubling the beam depth (0.5 to 1.0 in.) the value of c_1 is also increased by nearly a factor

TABLE 4. 6
RESULTS OF FIT TO EQUATION $\phi = Ac^n$

Specimen Number	A	n
1	1004 $\pm$ 31	2.742 $\pm$ 0.021
2	554 $\pm$ 23	2.738 $\pm$ 0.025
3	505 $\pm$ 21	2.710 $\pm$ 0.027
4	433 $\pm$ 21	2.685 $\pm$ 0.033
5	358 $\pm$ 10	2.740 $\pm$ 0.019
6	355 $\pm$ 14	2.701 $\pm$ 0.025
7	332 $\pm$ 12	2.748 $\pm$ 0.022
8	339 $\pm$ 21	2.753 $\pm$ 0.038
9	304 $\pm$ 12	2.733 $\pm$ 0.025
10	280 $\pm$ 13	2.724 $\pm$ 0.029
11	256 $\pm$ 14	2.741 $\pm$ 0.037
12	239 $\pm$ 20	2.467 $\pm$ 0.056
13	121 $\pm$ 12	2.455 $\pm$ 0.066
14	90 $\pm$ 8	2.383 $\pm$ 0.063
15	66 $\pm$ 10	2.422 $\pm$ 0.1022
16	515 $\pm$ 26	2.710 $\pm$ 0.035
17	506 $\pm$ 19	2.747 $\pm$ 0.025
18	525 $\pm$ 28	2.720 $\pm$ 0.034
19	258 $\pm$ 9	2.739 $\pm$ 0.021
20	263 $\pm$ 10	2.755 $\pm$ 0.023

of 2. However, for both beam depths, the value of c_1 is larger than would be predicted by the simple beam formula (0.667). Although the variation in c_1 with the beam depth is a result of strain in the unbroken part of the specimen, without a rigorous solution to the stress state in the beam no theoretical justification for this behavior is immediately obvious.

As Table 4.5 shows, the crack length exponential also varies with beam depth, being constant for a given beam depth but departing from the simple beam value of 3 for increasing beam depth. It would appear, intuitively, that in the limit as the beam depth approached 0 both c_1 and n would approach 0.667 and 3, respectively.

Effect of Changing Geometry on K_{Ic}

Figure 4.34 summarizes the range of K_{Ic} values determined from the various specimens. In general, the K_{Ic} values for the majority of the specimens fall in the range of 26 to 29 ksi $\sqrt{\text{in.}}$. Values of 28 to 30 ksi $\sqrt{\text{in.}}$ are fairly representative of K_{Ic} for 7075-T6 aluminum as obtained by other methods. From these specimens certain behaviors were revealed which are discussed below.

FIGURE 4.33
Variation in A with Section Modulus

FIGURE 4.34

Summary of K_{Ic} Data for the 7075-T6 DCB Specimens

Effect of B_N/B on K_{Ic}

Examination of two groups of specimens, namely the 3/8 in. width consisting of Specimens 4, 5, 6, and 7 and the 1/2 in. width consisting of Specimens 8, 9, 10, and 11, reveals a tendency for K_{Ic} to decrease with increasing B_N . This behavior is to be expected since the smaller the value of B_N/B the greater is the effect on the stress intensity for straightforward advance along the crack front. For the 3/8 in. wide specimens, K_{Ic} becomes essentially constant for B_N/B greater than 0.351, while for the 1/2 in. wide specimens K_{Ic} attains a constant value for B_N/B greater than 0.134. All other specimens which had a 1/2 in. beam depth, sharp (0.001) root radius, and B_N/B greater than 0.250 gave K_{Ic} in the range 27 to 29 ksi $\sqrt{\text{in.}}$

K_{Ic} Dependence on Crack Length

It should be noted that the specimens with the 1 in. beam depth, in particular, Specimens 12, 13, and 14, exhibited a fairly strong decrease in K_{Ic} with increasing crack length. This behavior for Specimen 12 is shown in the insert graph in Figure 4.33. In these three specimens it was found that K_{Ic} varied as $c^{-1/2}$. A dependence this strong cannot be accounted for by error in the compliance measurements. Since the other specimens showed almost no K_{Ic} dependence on crack length, with the exception of Specimens 4, 11, and 15 which exhibited a very mild dependence, it must be inferred that this effect is peculiar to the geometry of Specimens 12, 13, and 14. This effect might possibly be attributed to a difference in nominal stress biaxiality between the 1/2 in. deep beam specimens and the 1 in. deep beam specimens. Further experimentation is needed to resolve this point.

Effect of Side Groove Root Radius

The effect of increasing the side groove root radius was particularly strong in the 1/4 in. width specimens and to a lesser extent in the 1/2 in. widths as Specimens 18 and 20 reveal. After Specimen 18 (1/4 in. width, 0.050 in. side groove root radius) was separated in two halves, a sharp

flip-flop shear lip was noticeable at the base of each side groove. As no shear lip occurred for the specimens with a 0.001 in. root radius, this shearing mode along the sides of the crack in Specimen 18 accounts for the inordinately high value of this K_{Ic} scatter band. A similar shear lip also occurred at the side groove root in Specimen 20 (1/2 in. width, 0.050 in. root radius). However, the relative effect of this shear lip on K_{Ic} was smaller in Specimen 20 than in Specimen 18 because of the greater crack surface area of Specimen 20. The effect of the 0.010 in. root radius is mildly discernible for the 1/4 in. width (Specimen 17), but appears to have no effect in the 1/2 in. width (Specimen 19). It can be concluded that in this material in which the crack advances in a straightforward manner, the root radius must be selected to give a shear lip which is small relative to the net width, B_N . However, one can also conclude from the data that the required side groove root radius for the 7075-T6, where B_N is, say, 0.130 in. or larger, can be much greater than the necessary notch root radius in conventional notch tensile or bend specimens.

Delayed Failure Studies

A normally ductile pressure vessel steel such as A302B could fail by a time dependent brittle fracture process. This process, called delayed failure, is attributed to diffusion of a highly mobile interstitial (usually hydrogen) to areas of high stress in advance of a sharp notch or crack. A reactor environment could enhance the delayed failure process by lattice hardening and accelerated diffusion. Trapping of interstitials by vacancy defects could mitigate the effect.

In A302B, preliminary investigation shows a fully hardened hydrogen free structure produces a fracture toughness (K_{IC}) value of $100 \text{ ksi}\sqrt{\text{in.}}$ as compared to $80 \text{ ksi}\sqrt{\text{in.}}$ for a fully hardened hydrogenated structure. One would predict that fracture toughness should vary with hydrogen content. The mode of crack growth should likewise be sensitive to changes in hydrogen concentration in the area in advance of the crack tip. This suggests that a fracture mechanical approach would be useful in the study of delayed failure.

A DCB specimen has been adapted for use in studying brittle fracture processes and delayed failure. Analytic techniques are now being developed to describe the fracture process in terms of its thermodynamic properties. The change in these properties with varying hydrogen content will contribute significantly to a basic understanding of the mechanisms of delayed failure. The degree of susceptibility of a steel such as A302B to interstitial embrittlement might then be predicted on firmer theoretical grounds than exists to date.

The method of testing the DCB specimen has been described in a previous quarterly.⁽¹⁾ As a continuance of the program, additional DCB specimens were prepared by austenitizing at 1650°F in hydrogen and then quenching to the full hard condition. Subsequent refrigeration of the specimens minimized the loss of hydrogen before testing. The Instron curve of a typical fully hardened, hydrogenized specimen tested at -40°C is shown in Figure 4.35. As the curve shows, the initial deformation is elastic. Soon,

(1) Quarterly Progress Report, Metallurgy Research Operation, October, November, December, 1964, edited by J. J. Cadwell, HW-84573, General Electric Company, Richland, Washington. January 15, 1965.

however, the linear elastic region is exceeded in the specimen arms. This measured amount of plastic bending in the arms must be subtracted from the total indicated extension when calculating crack length. A critical stress is soon reached and the crack begins to move. Within the limits of machine sensitivity the crack growth appears to be continuous to Point B, where the specimen was unloaded. The irregular curve shape to Point A is simply non-uniform crack growth. At Point A the crosshead was stopped and the load was observed to decay in a nearly linear manner. Since the load decay rates can be measured by this technique, crack velocities can be calculated at a given temperature. These in turn can be related to more fundamental properties.

FIGURE 4. 35
DCB Loading Curve
with Extension Plus Time Axis

The crack length at any selected set of load and extension values can be calculated by the relation

$$C = \left(\frac{Ey}{Af} \right)^{\frac{1}{m}}$$

where

- C = crack length
 $E_{(T)}$ = Young's modulus at the test temperature
 y = extension
 f = load
 A = constant = 515.3
 m = constant = 2.726

The results of calculated crack lengths of Points A and B are shown in Table 4.7. Extension is corrected for plastic bending in the arms.

TABLE 4.7
CALCULATED CRACK LENGTH OBTAINED
FROM DCB LOAD VERSUS EXTENSION CURVE

Point	f , lb	y , in.	C , in.
A	480	0.1416	2.834
B	428	0.1416	2.956
Unloading Curve	Slope = 3.34×10^{-4}		2.960

A simple calculation shows that the crack has grown 0.122 in. during the period of stationary crosshead.

Load decay could be largely the result of creep in the specimen. The extent of specimen creep can be determined by a calculation of crack length from the unloading curve. The expression for crack length contains a term under the radical which is the reciprocal $\left(\frac{y}{f}\right)$ of the slope of the elastic load-extension curve. Crack length can then be obtained from the shape of the unloading curve.

$$C = \left(\frac{E}{A} \cdot \frac{1}{\text{slope}} \right)^{\frac{1}{m}}$$

where the constant have values tabulated previously. During the loading portion of the test the material work hardened to the strength level at least as great as A. Since any point of the time base curve would then be below the yield point of the material the investigation of possible creep deformation concerns a load value such as the value at B. From the expression containing

unloading curve slope data crack length C was computed to be 2.960 in. This compared favorably with the calculated C value of 2.956 at B showing that deformation by creep during the stationary crosshead time interval is negligible.

Work planned for next quarter will include refinement of testing techniques and development of a method for introduction and analysis of controlled quantities of hydrogen in the DCB specimen.

CHEMICAL METALLURGY

ATR GAS LOOP SUPPORT

Corrosion Studies - L. A. Charlot and R. E. Westerman

The evaporation-oxidation behavior of Haynes Alloy 25 and Hastelloy X-280 is being studied in low oxidant pressures in an effort to predict the degree of spalling of the resultant oxides and/or presence of volatile metals in the Advanced Test Reactor (ATR) loop. The thermal microbalance system previously used to survey superalloy oxidation and evaporation was found unsatisfactory for very low oxidant pressures (i. e., replenished gas atmospheres at pressures of 0.01 to 1μ) due to in-leakage of atmospheric contamination into the reaction tube. To obtain these low oxidant pressures, a reaction tube contained in a second vacuum system has been built. The capabilities of this unit are temperatures to 1500°C and pressures to 10^{-6} Torr.

Two tests have been conducted in this double-contained vacuum system. A test consists of racking two samples of each superalloy (0.75 in. x 0.50 in. coupons) in an alumina sample holder and then placing the assembly in the reaction zone. The gas atmosphere is then established and maintained at the desired pressure, and the temperature increased to 1120°C . Table 5.1 lists the parameters and results of the tests to date.

TABLE 5.1

LOW PRESSURE OXIDATION OF HASTELLOY X-280 AND HAYNES ALLOY 25

Sample	Atmosphere	Test Duration	Weight Change, mg/cm^2	Retained Oxide	Oxide Thickness, mils	Metal Penetration, mils
Hastelloy X-280-1	Dry Air at 3×10^{-4} Torr	45 hr	-4.8	Not Determined	- -	> 0.23
Haynes Alloy 25-1			-2.8	Not Determined	- -	> 0.12
Hastelloy X-280-2			-5.1	$0.18 \text{ mg}/\text{cm}^2$	0.02	0.26
Haynes Alloy 25-2			-2.5	$0.36 \text{ mg}/\text{cm}^2$	0.05	0.16
Hastelloy X-280-3	Oxygen at 1×10^{-4} Torr	71 hr	-3.3	Negligible	0	0.16
Haynes Alloy 25-3			-0.5	Not Determined	- -	- -
Hastelloy X-280-4			-3.8	Negligible	0	0.18
Haynes Alloy 25-4			+0.3	Not Determined	- -	- -

Retained oxide was determined by descaling the samples.⁽¹⁾ The sample weight loss was assumed to be due primarily to evaporation because of the following reasons:

- The sample holder in both tests was coated nearly uniformly with a green oxide, indicating either gas phase oxidation of the evaporating metal or deposition of metal from the samples on the holder and subsequent oxidation.
- No visual evidence of spalling of the oxide film formed on each of the four samples in the dry air test or on the Haynes Alloy 25 samples in the oxygen test. The Hastelloy X-280 samples in the oxygen atmosphere showed signs of severe spalling of the oxide film.

At the end of the oxygen test, the Hastelloy samples had patches of loose oxide over the surface. The Haynes Alloy 25 samples were, however, completely coated with a uniform black-green oxide.

The results indicate that evaporation in near vacuum conditions will proceed until sufficient oxidant can diffuse through the evaporating medium and form solid oxide on the metal surface. Comparing the weight changes between the dry air and oxygen atmospheres, it is apparent that the presence of nitrogen near the samples in the dry air test allowed evaporation to proceed longer than in the oxygen test. The fact that Haynes Alloy 25 formed a uniform film is gratifying. Hastelloy X-280, on the other hand, exhibited spalling tendencies which were not expected. Future tests are planned to confirm these preliminary findings.

(1) H. P. Maffei. Descaling of Various Iron and Nickel-Base Alloys, HW-79048. General Electric Company, Richland, Washington. September, 1963.

Dynamic Materials Testing Apparatus - R. A. Thiede

Previous tests in the loop have exposed Hastelloy X, Haynes Alloy 25, and Nb-1 wt% Zr to Grade A helium at temperatures of 2050 °F and flow velocities of about 525 ft/sec. Recently, tests have been made to determine if significant evaporation of superalloys occurs in purified (nonoxidizing) gas at high temperatures. A getter bed composed of clean zirconium turnings was installed and part of the loop gas flow passed through this bed. In the first test, a sample of Hastelloy X was exposed at a temperature of 2050 °F for 76 hr. The sample was bright and shiny after exposure and apparently lost about 4 mg/cm² by evaporation. During the portion of the run in which the sample was heated, the oxygen analyzed less than 0.1 ppm, the detection limit of the chromatograph. Hydrogen reached a maximum of 17 ppm after 4 hr of the run and decreased to 6.5 ppm at the end of the 76 hr run.

A second attempt to continue the run with low enough oxygen to obtain evaporation data resulted in failure because the sample oxidized. Several leaky gaskets were replaced and the power supply to the getter bed was increased so more gas could be circulated through the getter bed. A third run was started and it also resulted in failure. Due to leakage of a flange on the bottom of the getter bed, the loop shut off when the pressure dropped to 300 psi. During the period before the loop shut off, the sample overheated and partially melted, thus blocking the gas flow. Upon further investigation, it was found that the Pt-10 wt% Rh preheater coil had failed. Further runs are awaiting a new preheater coil which probably will not arrive until late August, 1965.

N-REACTOR CORROSION AND HYDRIDING

Reaction of Zircaloy-2 with Water Vapor at 900 to 1200 °C -

L. A. Charlot and A. B. Johnson, Jr.

The oxidation characteristics of Zircaloy-2 were investigated in water vapor (25 Torr) at 900 to 1200 °C to provide data for reactor excursion analyses. Data on oxidation of Zircaloy-2 were available to 850 °C in water vapor at 25 Torr⁽¹⁾ and in steam at 15 psi from 1300 to 1860 °C.⁽²⁾ While there is evidence of pressure effects at 750 and 800 °C in water vapor,⁽¹⁾ other data at 910 °C indicate no effect of oxygen pressure over the range of 1 to 730 Torr⁽³⁾ for times to 64 hr.

Oxidation studies were conducted at 900 to 1200 °C, using a thermal semimicrobalance. Weights taken on the samples before and after each test on a second microbalance ensured the accuracy of test results; agreement with thermal balance data in every case was well within experimental error. The Zircaloy-2 samples used were of different surface areas to monitor for and eliminate gas phase diffusion control at the higher temperatures. The samples were rectangular coupons approximately 30 mils thick, except in certain 900 and 1200 °C tests, where bent 70-mil cylindrical samples were used; all samples were bright etched in an HF and NH_4OH bath prior to testing.

Figures 5.1 and 5.2 indicate the results of these tests. The slope of each of the isotherms of Figure 5.1 gives the exponential dependence of

-
- (1) R. E. Westerman. High Temperature Oxidation of Zirconium and Zircaloy-2 in Oxygen and Water Vapor, HW-73511. General Electric Company, Richland, Washington. April, 1962. Also in J. Electrochem. Soc., vol. 111, pp. 140-147. 1964.
 - (2) W. A. Bostrom. The High Temperature Oxidation of Zircaloy in Water, WAPD-104. Westinghouse Electric Corporation. March, 1954.
 - (3) J. P. Pemsler. "Studies of the Oxygen Gradients in Oxidizing Metals, III Kinetics of the Oxidation of Zirconium at High Temperatures," J. Electrochem. Soc., vol. 112, pp. 477-484. 1965.

FIGURE 5.1

Oxidation of Zircaloy-2
in Water Vapor

FIGURE 5. 2

The Effect of Surface Area
on the Oxidation of Zircaloy-2

the reaction time (or weight gain) in the empirical relation

$$(\Delta w/A) = kt^{1/n}$$

These slopes approximate a value of $1/n = 0.5$, indicating adherence to a parabolic rate law. Table 5.2 lists the calculated parabolic rate constant, k_p , for each temperature, as well as the surface areas of the samples considered and hydrogen pickup from the corrosion reaction

TABLE 5.2
OXIDATION OF ZIRCALOY-2 IN 25 TORR WATER VAPOR

Test	Sample Area, cm ²	Sample Shape	1/n	Average Rate Constant, mg ² cm ⁻⁴ min ⁻¹	Test Duration, min	Hydrogen Present, ppm
900-2	3.00	Rectangular	0.47	0.30	280	80
900-3	5.56	Rectangular	0.48	0.26	140	Not Determined
900-4	1.39	Cylindrical	0.34	0.24	350	Not Determined
950-1	7.02	Rectangular	0.51	0.55	240	553
1000-2	2.83	Rectangular	0.53	1.22	240	455
1000-4	7.02	Rectangular	0.56	1.13	130	Not Determined
1050-1	7.02	Rectangular	0.56	2.56	200	340
1100-2	3.04	Rectangular	0.52	8.38	120	72
1200-5	1.45	Cylindrical	0.54	29.05	75	Not Determined

The data yielded an activation energy of 51.2 kcal/mole (Figure 5.3) in close agreement with the value obtained by Westerman⁽¹⁾ at temperatures to 850 °C. The rate expression over the entire temperature range studied is

$$k_p (\text{mg}^2 \text{cm}^{-4} \text{min}^{-1}) = 9.26 \times 10^8 \exp(-51,200/RT)$$

where T is the absolute temperature and R is the ideal gas law constant.

At each of the temperatures studied, the oxide film was adherent, although separation of the oxide did occur at edges of samples with thick oxides (1100 and 1200 °C samples).

Oxidation of Zircaloy-2 in water vapor at temperatures to 850 °C generally approximates cubic kinetics.⁽¹⁾ Over the range of this work (900 to 1200 °C) the kinetics are parabolic. Work at Westinghouse⁽²⁾ in the range 1300 to 1850 °C resulted in kinetics approximately parabolic (1/n = 0.56 to 0.59) to 700 °C, after which 1/n increased sharply to 1.0 at 1850 °C (i. e., linear kinetics).

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- (1) R. E. Westerman. High Temperature Oxidation of Zirconium and Zircaloy-2 in Oxygen and Water Vapor, HW-73511. General Electric Company, Richland, Washington. Also in J. Electrochem. Soc., vol. 111, pp. 140-147. 1964.
- (2) W. A. Bostrom. The High Temperature Oxidation of Zircaloy in Water, WAPD-104 Westinghouse Electric Corporation. March, 1954.

FIGURE 5. 3

Rate Constant as a Function of Reciprocal Temperature

At temperatures to 800 °C in water vapor, Zircaloy-2 undergoes a marked transition (increase in rate) after 60 to 100 min. Westerman observed only a minor transition at 850 °C; transitions did not occur in this study at 900 to 1200 °C, except for a small, reproducible (Figure 5. 2) increase in rate at 1000 °C at 45 min and a small decrease in rate at 950 °C at 30 min.

Metallography (Figures 5. 4a, b, c, and d) showed the oxide films to be of relatively uniform thickness and adherent. The latter point also was checked, using a platinum basket suspended beneath the coupons to determine that oxide spalling did not occur. Oxygen-rich zones, free of

(a) 900 °C for 1300 min; B-Etched (100X)

Oxide
Oxygen-Rich Zone
Metal and Hydride

(b) 1000 °C for 60 min; As-Polished (100X)

Oxide
Oxygen-Rich Zone

(c) 1000 °C for 60 min; B-Etched (250X)

Oxygen-Rich Zone
Hydride in Metal Matrix

(d) 1100 °C for 300 min; As-Polished (100X)

FIGURE 5.4

Oxidation of Zircaloy-2 in Water Vapor

Neg. 4651062C, 4651063A, 4650163C, 5651064

hydride are evident on 900 and 1000 °C samples (Figures 5.4a and 5.4c); gold-colored hydride is evident under the microscope on samples shown in Figures 5.4a and 5.4c.

In Figure 5.4b, oxide cracking at edges and within the oxide and areas where metal had pulled out of brittle oxygen-rich zones (dark spots) was similar to results reported elsewhere⁽¹⁾ on zirconium exposed in oxygen at 850 and 950 °C.

The appearance of the sample shown in Figure 5.4d is typical of coupons exposed at 1100 and 1200 °C; the oxide cracked along two edges, but remained adherent. A row of small particles occurred in the oxide, outlining the oxide/metal interface. The oxide was thicker by nearly a factor of 2 on the ends, compared to the oxide on the flat face. Gold-colored islands occurred at the center of the sample, thought to be hydrides swept into the center of the sample by the converging oxygen-rich zones.

Hydriding of Zircaloy-2 in an oxidizing atmosphere at 900 to 1200 °C is not fully understood. Enough data have been taken, however, to indicate that hydriding does occur during the oxidation process at these high temperatures. Making liberal assumptions for our system, approximately 1 to 2% of the theoretically available hydrogen from the corrosion reaction found its way into the samples. In some cases, notably the 1100 and 1200 °C tests, the amount of hydrogen found in the coupons by hot extraction hydrogen analysis exceeded the apparent equilibrium solubility of hydrogen in the metal by a factor of 2. (The apparent equilibrium solubility was calculated from equilibrium hydrogen-zirconium isotherms⁽²⁾ and the application of Sievert's solubility law.)

Additional metallographic analysis and hydrogen analyses are underway in an effort to more fully understand hydrogen pickup under these severe oxidizing conditions.

-
- (1) G. R. Wallwork, C. J. Rosa, and W. W. Smeltzer. "Breakaway Phenomena in the Oxidation of Zirconium at 850 and 950 °C," Corrosion Sci., vol. 5, pp. 113-119. 1965.
- (2) S. Dushman and J. M. Lafferty. Scientific Foundations of Vacuum Technique, John Wiley & Company, New York, 1962. p. 537.

Effect of Thick Oxide Films on the Hydriding of Zircaloy-2 in Impure Helium - D. W. Shannon

Thick oxide films on Zircaloy-2 are being studied to determine effects on corrosion and hydriding in helium contaminated with traces of H_2O , CO , and H_2 . Experimental details and sample preparation were previously reported. ⁽¹⁾ Data are being taken at 250, 300, 350, and 400 °C. Detailed results of the 400 °C experiments are being reported this quarter. The 250 and 300 °C data show almost no corrosion or hydriding up to 10 mo. The effects at 350 °C are measurable; however, trends revealed at 400 °C are clearer.

Oxygen weight gain data for etched samples are given in Figure 5.5. These data show no measurable transition as the weight gains passed the

FIGURE 5.5

Oxidation Rate of Etched Zircaloy-2
in Impure Helium at 400 °C

(1) Quarterly Progress Report, Metallurgy Research Operation, April, May, June, 1964, edited by J. J. Cadwell, HW-82651. General Electric Company, Richland, Washington. July 15, 1964.

usual transition region of 40 mg/dm^2 . It appears the oxidation rate is increasing after 10 mo and this trend will be followed in the future.

The oxygen weight gains at 6 mo are plotted versus prefilm thickness (Figure 5.6) and are compared with 1500 psi, 400°C autoclave data.

FIGURE 5.6

Oxidation Rate of Prefilmed Zircaloy-2 in Impure Helium
(temperature, 400°C ; time, 6 mo)

It can be seen that the oxygen weight gains are considerably lower in the low parts per million water contents and, in general, increasing film thickness results in lower weight gains. The 1 mo data are plotted versus the H_2O partial pressure in Figure 5.7. Here it can be seen that down to 0.01 atm water partial pressure (10,000 ppm in He) the oxidation rate is independent of pressure. However, below 0.01 atm there is a substantial

FIGURE 5.7
Pressure Dependence of the Oxidation Rate of Zircaloy-2 at 400 °C

pressure effect. The thicker preexisting films produced a more significant pressure effect. These results are consistent with the corrosion model previously reported. ⁽¹⁾ In this model the corrosion is controlled in the thin film region by the oxygen ion diffusion, and the reaction rate is independent of water pressure. However, as thick films are formed, rate control shifts toward a process controlled by the diffusion of water through the bulk film. The fact that the corrosion rate shifts toward a linear dependence on pressure is one confirmation of bulk diffusion control. ⁽²⁾ As the water pressure is increased, the bulk diffusion rate increases until the oxygen ion diffusion process again becomes rate limiting. The fact that pretransition samples show some pressure effect at low pressures indicates the ion diffusion layer may be thinner than the total pretransition film thickness. In any actual sample, both diffusion processes are present and the exact rate curve obtained depends on the combination of temperature, prior film thickness, and pressure of water vapor.

The total hydrogen absorption for 6 mo are plotted versus film thickness in Figure 5. 8. These data show the extent of hydriding becomes more severe as film thickness increases. In Figure 5. 9 the data are plotted versus the partial pressure of hydrogen and it can also be seen that thick film samples hydride more readily, and that there is a significant pressure dependence to the hydriding reaction. It should be emphasized that some inhibiting water vapor is present in all these experiments. Without the inhibitor, catastrophic hydriding would have occurred.

In Figure 5. 10 the hydriding rate and oxidation rate of etched samples are compared. These samples are important because all the oxide film grew during the 10 mo exposure in the experiment. It can be seen that at all three hydrogen pressures an increase in hydriding rate occurred at the normal transition weight gain of about 40 to 50 mg/dm² even though no

-
- (1) Quarterly Progress Report, Metallurgy Research Operation, April, May, June, 1964, edited by J. J. Cadwell, HW-82651. General Electric Company, Richland, Washington. July 15, 1964.
 - (2) R. E. Westerman. Unpublished Data, Pacific Northwest Laboratory. (Personal Communication)

FIGURE 5. 8

Hydrogen Absorption of Prefilmed Zircaloy-2 in Impure Helium
(temperature 400 °C; time, 6 mo; 900 ppm H_2O)

FIGURE 5.9

Hydrogen Absorption of Prefilmed Zircaloy-2 versus H₂ Content of Helium
(temperature, 400 °C; time, 6 mo; CO content = H₂ ppm; H₂O constant = 900 ppm)

FIGURE 5.10

Hydriding Rate of Etched Zircaloy-2 in Impure Helium at 400 °C

inflection was detected on the weight gain curve. This is further evidence for the existence of film cracking at about 40 mg/dm^2 . The hydriding rate after this inflection is more sharply dependent on hydrogen pressure. However, it should be noted that even at 2000 ppm hydrogen in the gas the hydrogen absorbed is only about 25% of that released by the oxidation reaction (etched sampled only).

In summary, it has been confirmed that in impure helium containing H_2 , H_2O , and CO the hydrogen absorption rate becomes faster and the oxidation rate slows down as the film thickens, due to a pressure dependence on diffusion through the thick film. This reduction in oxidation rate means that Zircaloy process tubes will take considerably longer to reach weight gains where hydriding increases due to the thick film effect (100 mg/dm^2) than would have been predicted from high pressure data. Also, the H_2 pressure dependence data on hydriding rates are encouraging, because all data at 250 ppm H_2 indicate essentially no gas phase hydriding at any film thickness up to 450 mg/dm^2 and temperatures of 250 to 400°C . Even with a 2000 ppm H_2 environment and 100 mg/dm^2 oxide film, hydrogen pickup at 400°C up has been only 25% of the corrosion hydrogen generated.

MATERIALS ENGINEERING

GAS LOOP DEVELOPMENT

Model Loop Operation - L. J. Defferding

The thermal conductivity tests on the foil insulation in a flowing helium system were completed. The results indicate that with proper design of the joints between sections of insulation the conductivity is the same in both static and dynamic systems.

The dimensions of the foil insulation tested in the model gas loop were 4.56 in. OD by 2 in. ID, and there were 30 to 35 layers of 0.002 in. thick Inconel 600 foil per inch of insulation. The helium flow was varied from 100 to 440 lb/hr; the linear velocity at the higher flow rate is equivalent to about 4000 lb/hr in a 6 in. pipe. The gas temperature was varied from 500 to 1800 °F. The conductivity increased (see Figure 6.1) slightly with increased gas flow. Flow dams over the ends of the foil effectively prevented flow longitudinally through the insulation, but did not prevent a small flow between the pressure bearing pipe and the outer

FIGURE 6.1

Thermal Conductivity Foil Type Insulation in Flowing Helium

shell of the insulation. Conductivity varies from 0.26 ± 0.03 BTU/(hr)(ft²)(°F) to 3.9 ± 0.03 Btu/(hr)(ft²)(°F) at a mean temperature of 400 to 1000 °F, respectively. During April a bearing failure occurred in the eddy-current-variable speed drive for the frequency changer assembly. The bearing apparently had been run with some of the cooling water in the housing. Three bearings, which were available in the United States, were replaced. The fourth bearing in the assembly, which was in good condition, is available only in France where the machine was manufactured. The bearing housings on the variable speed drive were modified by drilling a hole in each housing. The holes are located so that any water seeping by the slinger-type seal will drain out the holes before it can enter the bearings. This should prevent additional failures. Since the repairs were completed the equipment has operated satisfactorily. The total operating time on the gas bearing compressor is 481 hr with 115 starts. The compressor has operated with no required maintenance to date.

Columbium Sleeve Leak Test

The specimen train and 2000 °F helium in the test section will be separated from the in-reactor tube of the Advanced Test Reactor (ATR) Gas Loop by a Columbium Sleeve. If helium leakage through the seal at the top of the sleeve is excessive, there will be too much heat loss through the pressure tube wall. The installation of the test fixture to measure this leakage was completed and room temperature runs were started on a prototypical gas seal. The nominal diametral clearance between the Columbium Sleeve and the Invar seal is 0.010 in. as compared to a design clearance of 0.020 ± 0.010 in. If the present clearance gives satisfactory leakage rates, the fixture may be modified to increase the clearance to the maximum and another test will be run.

Model Gas Loop Heater and Piping Modifications - R. J. Evans

A new model gas loop heater has been designed and is being built to incorporate and investigate essential details of a compact two-stage heater presently being designed for the ATR Gas Loop. Material and/or components for all parts of the heater, electric instrumentation, power supply equipment, and loop piping modifications have been ordered.

Detail drawings of electrical modifications and additions are 95% complete. Detail drawings of piping modifications and metallic foil insulation for the piping are 75% complete.

Machining of all components for the heater, except the molybdenum parts has been completed. Fabrication of foil insulation sections for the heater is 60% complete. Final assembly of the heater will begin after receipt of the balance of ceramic parts and molybdenum heating tubes now on order.

Heating element tubes for each stage of the heater will form a series electrical circuit. Each tube will be connected to its neighbor and to power supply electrodes by a "dog bone" connector. Since it is desirable to minimize connector heat losses, good contact between the tube and the connector must be made. Roll-fitting, shrink-fitting and welding are being investigated for these connections. The nichrome tubes will be roll-fitted into the nickel connectors and the ends TIG welded with a filler rod of Inconel 82. The molybdenum-molybdenum connections are still being investigated.

A computer program was written to determine optimum insulation thickness, piping sizes and cooling water flow for the piping from the regenerative heat exchanger to the new heater and from the heater to the test section of the model loop. The program also investigated the effects of cooling water flowing in the jacket around the piping either concurrent or countercurrent with the gas flow. Six in. pipe with 2 in. thick foil insulation will be used between the regenerative heater and the concentric heater. The first straight run of pipe immediately following the heater will be used as an additional high temperature test section and will be 10 in. pipe with 3 in.

of thick foil insulation. The heater and all new piping will be cooled by water flowing concurrent with the gas flow. All elbows and tees will be air cooled so that hot spots due to possible joint separation of the foil insulation in the curved pipes can be detected.

First attempts at making sections of foil insulation indicated that structures of 30 layers per inch of 2 mil Inconel foil between 34 mil inner and outer liners had no strength. Increasing the density of foil to 60 layers per inch helped support the inner liner, but was not desirable from a conductivity standpoint. A satisfactory design was made with 30 layers per inch of foil by inserting pins of 1/8 in. Haynes-25 welding rod through the outer liner and foil and either butted against or welded to the inner liner. The pins were then welded to the outer liner.

Although 95% of the electric equipment ordered has arrived, work will not be started on the electrical system until the heater is nearly completed so as to shorten loop downtime.

Pipe and fittings for loop modifications are due to be shipped during August and work on the loop should be completed during September.

Helium Purification System - J. H. Hoage

A high pressure helium purification system that operates independent of the model loop is used to study cleanup system components, to supply high purity helium for other systems, and to upgrade discharged helium. This system was placed in service following repairs to the compressor. Helium was recovered from the 2500 ft³ balloon with the following analysis:

ppm	H ₂	O ₂	N ₂	CH ₄	CO	H ₂ O
Purity From Balloon	*	640	3000	33	*	8
Purity After First Cleanup	3.5	1.3	1.9	*	*	7.5
Purity After Second Cleanup	*	*	*	*	*	6.0

*No detectable amount from chromatograph, sensitivity is about 0.1 ppm.

Moisture Removal

The heaters for regeneration of the room temperature molecular sieves (these sieves remove water and CO₂) were enlarged and placed directly on the molecular sieve vessel replacing those that heated the regenerating air supply. The beds are now regenerated by passing air through the bed until a temperature of 500 °F is reached for 1 hr followed by heating to 600 °F under vacuum. Cooling to room temperature is also under vacuum. As a result of this ability to regenerate more completely, the moisture analysis of helium recovered from the plastic storage balloon has been approximately 1.5 ppm maintaining less than 0.1 ppm of H₂, O₂, N₂, CO and CH₄.

Helium Relief Valve Heat Leakage Evaluation

A Consolidated Relief Valve 1975T, equipped with a Viton A O-ring seat was tested for its ability to seal helium following operation of the valve. The valve was set to relieve at 500 psi. No leakage was detected before exceeding the set pressure. Following operation of the valve, either manually or over pressure, the valve reseated at 480 psi and no leakage was detected when the pressure returned to 500 psi. The test was conducted at both 75 °F and 150 °F. The leak detector was a Consolidated Electrodynamics Corporation Helium Leak Detector 2U-120A, operating on the most sensitive scale.

PRTR PRESSURE TUBES

Properties of Irradiated PRTR Pressure Tubes - M. C. Fraser

Some stress-rupture tests of tube specimens have been performed with test conditions of 550 °F and hoop stress of 64,000 psi and 66,000 psi. The hoop stress was about 90% of ultimate. The results suggest that increasing amounts of fast neutron exposure may be accompanied by a decrease in time-to-rupture.

Some annealing of irradiation damage may have occurred during these stress rupture tests.⁽¹⁻⁴⁾ Tensile tests of ring specimens after about 40 hours and 1000 hours at a temperature of 550 °F showed the 1000 hr specimens had 4% lower ultimate strength. The ring specimens were taken from an irradiated tube segment whose exposure was 5×10^{20} fast nvt.

-
- (1) L. M. Howe. The Annealing of Irradiation Damage in Zircaloy-2 and the Effect of High Temperature Irradiation Upon the Tensile Properties of Zircaloy-2 CRMET 922 (AECL-1024). Atomic Energy of Canada Ltd. April, 1960.
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 - (3) A. L. Bement and J. E. Irvin. The Effect of Hot Water Thermal Treatments on the Cold Work Recovery of Zircaloy-2, HW-80309. General Electric Company, Richland, October, 1964.
 - (4) R. S. Kemper and D. L. Zimmerman. Neutron Irradiation Effects on the Tensile Properties of Zircaloy-2, HW-52323. General Electric Company, Richland, August, 1957.

Properties of Unirradiated PRTR Pressure Tubes - M. C. Fraser and
F. E. Bard

Measurement of the elastic constants will reveal the degree to which these tubes are anisotropic. If the difference between the longitudinal and the hoop elastic constants is less than 10%, then from a practical view, these tubes may be considered isotropic and application of Lamé elastic constant formulas is satisfactory. However, if the difference between the longitudinal and the hoop elastic constants is greater than 10% the use of Lamé equations is not satisfactory.

Sheet type tensile specimens have been fabricated from unirradiated tube segments. Some are of small size - 1/4-in. gage length and 25/1000 in. thick - to include specimens from the hoop axis. Others are of large size to provide for effects of specimen size upon tensile test behavior.

The variation of elastic strain in the hoop and longitudinal directions has been measured as a function of distance from a burst test specimen end fitting. The specimen was a 3 1/2 ft long piece of tube. Strain was measured with resistance type gages attached at 2, 5, 8 and 11 in. from the end fitting. The specimen has been pressurized under closed end conditions which correspond to a load ratio of 1/2. The data show that the ratio hoop to longitudinal elastic strain was 5. The Lamé equations gave values for Poisson's ratio of 1/3 and Young's modulus of 14×10^6 . There was no significant change in strain with distance from the end fitting.

Additional values of the elastic constants need to be obtained under different load ratios before making conclusions in this regard. The absence of a significant strain variation between the points measured indicates that burst test specimens significantly shorter than 20 in. may be confidently used.

Fracture Studies - R. C. Aungst and P. M. Jackson

A facility for studying the bursting characteristics of pressure tubing at subzero temperatures has been fabricated. It consists of an insulated polyethylene vessel in which a methanol bath can be cooled with dry ice. Temperatures slightly below -100°F are attainable and are measured by reversing the leads on an ordinary iron constantan thermocouple affixed to the test specimen. The tube undergoing test is pressurized with a glycol mixture that will resist freezing at the test temperature. The immediate area of the low temperature bath is blanketed with CO_2 as a precaution against ignition of the methanol by a chance spark that might be generated by the bursting specimen.

A stress-strain curve (Figure 6.2) has been plotted from strain gage readings taken on a specimen of N-reactor pressure tube pressurized to failure at -33°C . The 0.2% yield strength and the ultimate strength were virtually coincident at 115,000 psi. This compares with 105,000 psi at room temperature, 80,000 at 150°C , and 60,000 at 300°C . These four values form a nearly linear relationship (Figure 6.3) between yield strength and temperature within this range.

Extension of the yield strength curve to the subzero region is expected to be helpful in forthcoming efforts to determine the nil ductility transition temperatures of the various types of pressure tubing, and to construct fracture analysis diagrams of the type developed by Pellini, et al., at the Naval Research Laboratory. Measurements of this type will be attempted in an effort to describe the fracture characteristics of reactor pressure tube materials in terms commonly used for ferrous alloy pressure vessels.

FIGURE 6.2

Load-Elongation Curves for Zircaloy-2
N-Reactor Pressure Tubes

FIGURE 6.3

Effect of Temperature on Yield Strength
of Zircaloy-2 N-Reactor Pressure Tubing

Irradiated Tube Testing Facility - P. M. Jackson

A basin Ion exchange unit is being installed in the recirculating irradiated tube testing basin to remove suspended radioactive particles and keep the water clean for improved operational visibility.

An underwater tube shear was designed and is currently being fabricated. This shear will reduce the volume to weight ratio of burst tube sections. It will now be possible to simultaneously dispose of several spent tube sections by the routinely used disposal equipment.

A device to measure the inside diameters of pressure tubes has been fabricated and is currently being evaluated. This device, when connected to a recording instrument, will measure six consecutive diameters while the probe is moved through the tube even while submerged in water. The goal for this device is to simultaneously measure six tube diameters to an accuracy of ± 0.0003 in.

USA-AECL COOPERATIVE PROGRAM

Zr-Nb Pressure Tubing - P. J. Pankaskie

Tubular specimens 2 in. ID by 0.180 in. wall, were tested under biaxial stress conditions to determine the crack propagation characteristics at temperatures ranging from -23 through 265 °C. In these tests the ratio of hoop to axial stress was two to one. All Test specimens were prepared with a 1/16 in. wide milled slot. The slot depth is 80% of the tube wall thickness. The "web", which amounts to 20% of the original wall thickness, is first failed by internal pressurization. The "cracked slot" is sealed with a rubber plug and the test piece is repressurized until failure. Slot lengths ranging from 1/2 in. to 2 1/2 in. were used in these tests. The test results are shown in the following table.

CRACK PROPAGATION DATA FOR Zr-2.5 wt% Nb ALLOY

<u>Specimen Identification</u>	<u>Specimen Condition, % Cold-Worked</u>	<u>Test Temperature, °C</u>	<u>Slot Length, in.</u>	<u>Hoop Stress at Failure, psi</u>	<u>Crack Length After Failure</u>
30-6	30	25	1/2	106,500	2 3/4 in. from each end of slot
30-3	30	25	1 1/2	47,000	1 1/2 in. from each end of slot
60-3	60	25	1 1/2	45,500	1 1/2 in. from each end of slot
30-4	30	25	2 1/2	24,300	very short cracks transverse to slot
30-5	30	250	2 1/2	10,500	no propagation
60-4	60	25	2 1/2	23,300	3/4 in. from one end of slot only
60-5	60	265	2 1/2	10,600	only very short crack at each end of slot
60-6 ⁽¹⁾	60	-23	1/2	73,500	Brittle behavior, crack propagated full length of test piece.

- (1) An attempt was made to fail this test piece at 300 °C without success. The seal plug failed at about 7100 psi internal pressure ($\sigma_\theta = 51,500$ psi) and there was little or no plastic deformation about the "cracked slot." This piece was then retested at -23 °C.

The results of tests now completed indicate that the Zr-2.5 wt% Nb alloy is not susceptible to brittle failure at room temperature or above. A series of similar tests on hydrided test pieces will be done to evaluate the effect of hydride on brittle behavior.

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