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MECHANICAL PROPERTIES
OF
STAINLESS STEELS IN SODIUM,
AND
APPLICATION TO LMFBR DESIGN

AEC Research and Development Report

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Printed in the United States of America
Available from
National Technical Information Service
U.S. Department of Commerce
Springfield, Virginia 22151
Price: Printed Copy \$3.00; Microfiche \$0.65

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MECHANICAL PROPERTIES
OF
STAINLESS STEELS IN SODIUM,
AND
APPLICATION TO LMFBR DESIGN

By:

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ABSTRACT

From 1965 to 1971, Atomics International conducted a base program to determine design properties of stainless steel for the Liquid Metal Fast Breeder Reactor (LMFBR). This report summarizes the program content, reviews previously reported conclusions regarding the mechanical behavior of annealed and cold-worked Types 304 and 316 stainless steel in a liquid sodium and other environments, and presents previously unpublished data. Experimental data were obtained in the temperature range of 900 to 1400°F. Specimens were tested under uniaxial, 1:1 biaxial, and 2:1 biaxial stress-state conditions, with emphasis on the 2:1 state. Except for a small number of test samples that were injected with helium on a cyclotron, all materials were tested in the unirradiated condition. Test environments included static and flowing high-purity liquid sodium, high-purity helium, sodium system conditions which induced carbon transfer both to and from the stainless steel, and copper-contaminated sodium. Statistical evaluations of the data were used to derive equations for parametric correlation of creep and stress-rupture strength, time, and temperature. A discussion of the application of our data to the design of LMFBR stainless steel components is included. An appendix contains a bibliography of reports published under the program.

I. INTRODUCTION

During the initial assessment of the state-of-the-art design application of stainless steel to the Liquid Metal Fast Breeder Reactor (LMFBR), in preparation of the development plan,⁽¹⁾ the Atomic Energy Commission (AEC) recognized that there was insufficient information regarding the behavior of stainless steels in the LMFBR environment. Consequently, a testing program was initiated to obtain data in support of alloy selection and component design for the LMFBR system. This report summarizes the results of the portion of the program which was performed by Atomics International, and which was devoted to the definition of mechanical behavior of stainless steels in a sodium environment without irradiation.

The objectives of our program required thorough characterization of test material and environments, for which we utilized the combined expertise of personnel in the areas of materials specification and quality control, sodium chemistry, physical and mechanical metallurgy, stress analysis, and development test design and procedures. At the outset, a specification for "System Quality" sodium was defined, which set the standard for all sodium used for sodium-test environments.^(2,3) The thin-walled fuel cladding of an LMFBR would be the reactor component most likely to show the effects of the sodium environment, and of variations in dimensions, metallurgical condition, and quality. Consequently, a specification was written for purchase of high-quality thin-walled tubing, to be tested under the program.⁽⁴⁾

The majority of testing, after the initial development of test techniques and the establishment of work plans, was carried out on characterized heats of Types 304 and 316 stainless steel, under closely controlled environmental conditions. Specifically, our work addressed itself to the establishment of baseline design mechanical properties data and the evaluation of high-temperature behavior of solution-treated and 10 to 15% cold-worked Types 304 and 316 stainless steel in liquid sodium systems. We obtained information regarding the effects of: (1) static and flowing high-purity liquid sodium, (2) steady-state and cyclic secondary stress (thermal gradient), (3) carbon transport, (4) degree of cold work, (5) alloy compositional variations (heat-to-heat), and (6) multiaxial stress state.

Detailed descriptions of test equipment and techniques are omitted from this report; a bibliography of published information and internal supporting documents for the program is presented in Appendix I.

Appendix II contains tabulated reference to all 2:1 biaxial stress-rupture tests conducted under this program. Appendix III contains all test data obtained since the last report (AI-AEC-12976) was written.

II. SUMMARY OF CONCLUSIONS

The major findings of the test program regarding the behavior of Types 304 and 316 stainless steels for LMFBR service are summarized in this section. Most of the testing was done with thin-walled tube samples in a biaxial stress mode, but some measurements were made on sheet samples stressed uniaxially. With the exception of a few samples that had been injected with helium on a cyclotron, tests were made on unirradiated material. Wherever possible, samples were made from one reference heat of each alloy. The body of the report contains more complete descriptions of the results, and a discussion of their application to system design.

A. ENVIRONMENTAL EFFECTS

1. Static Sodium

- 1) There was no effect on rupture life at 1200 to 1400°F of Types 304 or 316 stainless steel, annealed or cold-worked, compared with static helium.
- 2) Cold-worked Type 316 stainless steel showed greater strength at high stress levels in 1000°F helium than in static sodium, but the same strength at low stresses. Annealed Type 316 stainless steel, and Type 304 stainless steel in either the annealed or cold-worked condition, were unaffected at 1000°F.
- 3) With the exception of 1000°F test results on Type 316 stainless steel, the sodium environment reduced rupture strain in biaxial tests. Results of uniaxial tests showed the decrease in rupture strain to be caused by reduction of the amount of tertiary creep. Microstructural observations indicate that sodium affects the grain boundary composition, to promote ferrite and/or sigma phase, which influence the fracture mechanism. Additional evidence of the sodium effect on fracture behavior is that, at temperatures and stresses where explosive ruptures occurred in static sodium, pin-hole ruptures occurred in helium.

- 4) Tests of Types 304 and 316 stainless steel in zirconium-gettered static sodium showed that the presence of strong carbide-forming elements caused decarburization of the stainless steel, with a resultant decrease of stress-rupture strength at 1200, 1300, 1350, and 1400° F.
- 5) Tests, in which copper was introduced as an impurity in the sodium, showed the copper to be readily transferred by sodium and deposited on Type 304 stainless steel. The mechanical properties, however, were unaffected by the presence of copper, over the temperature range of 950 to 1300° F.

2. Flowing Sodium

- 1) Tests in flowing sodium under isothermal loop operation gave stress-rupture results identical to those obtained (at 1200° F) in the static sodium retorts, verifying the assumption that static sodium data provided appropriate controls for tests in the loop under nonisothermal conditions.
- 2) Creep-rupture tests of Types 304 and 316 stainless steel in flowing sodium near 1200° F, with a loop ΔT of 50° F, showed a weakening effect at longer times, accompanied by a decrease in rupture strain. Since the behavior was similar to that of specimens tested in zirconium-gettered sodium, it was attributed to decarburization of the stainless steel. Carbon loss was shown to have occurred, by the results of chemical analyses of specimens tested.

3. Carburizing Conditions

- 1) Case-carburized thin-wall tube specimens (1 mil depth, $\sim 2.5\%$ surface carbon content) of cold-worked Type 316 stainless steel were prepared, by controlled additions of methane, to represent a possible condition of fuel cladding material, if sudden introduction of carbon to a sodium heat transfer system occurs.
- 2) The tensile yield strength of Type 316 stainless steel at 1200° F was unaffected by the thin high-carbon case (10% of wall thickness). The ultimate tensile stress was slightly higher than that for the

uncarburized control specimen. The total elongation was 1/2 to 2/3 the total elongation of the uncarburized material.

- 3) Rotating bending fatigue tests at 1200°F showed the case-carburized Type 316 stainless steel to be essentially the same as uncarburized material at low stress amplitudes, but showed longer fatigue life at high stress amplitudes. It is believed that less plastic strain occurred in the case-carburized Type 316 stainless steel at high stress amplitudes relative to uncarburized Type 316, thereby increasing the fatigue life.
- 4) Stress-rupture life at 1200°F of the case-carburized Type 316 stainless steel was higher than that of uncarburized material, accompanied by lower creep strain at fracture. The carburized specimens exhibited burst-type ruptures, rather than pin-holes; which may be explained by the sudden imposition of high overstress on the uncarburized section, when cracking of the high-strength case occurs.
- 5) Specimens of annealed Type 304 stainless steel tubing were uniformly carburized in sodium at 1100°F, using 2-1/4 Cr - 1 Mo alloy steel as a carbon source. Specimens under stress (ASME maximum allowable) were found to contain higher carbon content than unstressed specimens, indicating an enhancement of carburization in stressed material.

B. PROCESSING EFFECTS

1. Alloy Composition

- 1) A correlation was found between creep-rupture behavior of Type 316 stainless steel and the equivalent Cr/Ni ratios in heat-to-heat stress-rupture studies. Heats with high Cr/Ni ratios exhibited higher rupture strengths, accompanied by lower rupture strain. The tendency was shown by the material in both the cold-worked and annealed conditions.
- 2) In 2:1 biaxial creep tests, the reference heat of Type 316 stainless steel showed anisotropic behavior in the cold-worked condition. The reference heat of Type 304 stainless steel displayed isotropic behavior in the annealed and cold worked conditions. The results serve as

a warning against the assumption that all stainless steels will behave isotropically.

2. Cold Work

- 1) At high stresses, the presence of cold work results in an increase of stress-rupture life of both Type 304 stainless steel and Type 316 stainless steel. At low stresses, however, the cold-worked material has lower stress-rupture strength than material in the annealed state. The stress-rupture curves of cold-worked material exhibit a sharp change in slope at a specific time to rupture. Metallographic examination of ruptured specimens revealed that the change is attributable to complicated interactions among recovery-recrystallization processes under stress, creep deformation, and fracture mechanisms. The time to rupture at which the change in slope of the stress-rupture curve occurs depends upon several factors: (1) test temperature — as test temperature increases from 1000 to 1400° F, the change occurs at shorter rupture times, (2) amount of cold work — for a given heat of material, increased cold work shortens the time to rupture at which the change occurs, (3) alloy content — under given conditions, alloying elements which tend to slow down diffusion rates in stainless steel cause the change to occur at longer times to rupture (e. g., the change occurs at longer rupture time for Type 316 stainless steel than for Type 304 stainless steel, other conditions being the same).
- 2) Over the temperature range of 900 to 1400° F, cold-worked Types 304 and 316 stainless steel rupture at lower strain than the same material in the annealed condition. The rupture strain for given rupture times is a function of test temperature, reaching a minimum at 1000 to 1100° F. The rupture strain of cold-worked Type 304 stainless steel was lower than that of cold-worked Type 316 stainless steel, under equivalent conditions of temperature and rupture time.

C. MECHANICAL METALLURGY

1. Stress-State Correlation

The correlation of uniaxial with 2:1 and 1:1 biaxial stress states for Type 304 stainless steel under stress-rupture conditions, over the temperature range of 1200 to 1400° F, follows the von Mises distortion energy yield theory.

The stress-rupture behavior of Type 316 stainless steel at 1200°F, on the other hand, shows a dependence in accordance with the maximum principal stress. It is believed that this behavior may be attributed to alloy anisotropy, previously mentioned.

2. Increasing Stress

A limited number of creep-rupture tests of Type 316 stainless steel at 1200°F under periodic uniform increments in stress indicated the validity of predictions based upon cumulative life fraction used at each stress level. The prediction of creep strain required the inclusion of primary creep at each increment of stress.

3. Secondary Stresses

a. Steady-State Thermal Gradient Across the Wall of Pressurized Tube

Stress-rupture test results on Types 304 and 316 stainless steel at 1200 to 1250°F showed a dependence of rupture life on the combined pressure and thermal stress at mid-wall, and the mid-wall temperatures. No evidence was found to indicate additional effects of the thermal gradient, such as mass transfer of interstitial elements. Thin ferrite layers were formed at the sodium-exposed external surfaces; they were too thin, however, to be expected to have a significant effect on the stress-rupture strength.

b. Cyclic Thermal Gradient

The results of tests of Type 304 stainless steel pressurized tubes, subjected to on-off cyclic internal heating in 1200°F isothermal loop sodium, showed enhancement of creep attributable to creep relaxation of the alternating stress gradients across the wall during the on and off portions of the cycle. The cyclic secondary stress caused pronounced reductions in the amount of tertiary creep, which may be explained by the greater effect of cyclic loading on intergranular crack propagation and fracture.

c. Mechanical Interaction Between Components

The results of 2000-hr creep-rupture testing of a cluster of 19 pressurized wire-wrapped Type 316 stainless steel tubes in 1200°F flowing sodium were: (1) tube shortening occurred (attributed to anisotropy) with constant loosening

of the wire wrap, (2) rupture locations were predominantly in areas where the tube wall was thinned by excessive clamping force between the wire wrap and end plug during assembly, (3) self-welding of the wires to tubing occurred under light contact loads.

III. EXPERIMENTAL TECHNIQUES

The majority of specimens tested on this program were exposed in multiple arrays of pressurized tubing, in either static sodium retorts or a flowing sodium loop. The static sodium tests used a 12-specimen retort, of the design shown in Figure 1. Details and operation of these retorts have been described previously.⁽⁵⁻⁷⁾ Flowing sodium tests utilized a hexagonal bundle of 19 pressurized tubing specimens⁽⁸⁾ in a system ΔT of $\sim 50^\circ\text{F}$ (Figure 2).

A series of single-specimen sodium tests was used to study stress-state effects⁽⁵⁾ and high-heat-flux thermal gradients across the wall of the tubular specimens.^(9,10)

A. CONTROLS AND PRECAUTIONS

1. Specimen Fabrication and Characterization

The Type 304 and 316 stainless steel specimens used in this test program were taken from two highly characterized heats of thin-wall tubing — 0.275 in. ID by 0.010 in. wall. Fabrication history and inspection-characterization data on these two heats are documented in detail.^(11,12) The tubing was received with 10 to 15% cold work, and underwent a 100% inspection for internal and surface defects, using ultrasonic and eddy-current techniques. Material which possessed defects exceeding the allowable defect size (1/4 in. long by 10% of wall) was cut from the tube lengths and discarded. Any borderline cases were also discarded. Each length of tubing was then subjected to Vidigage (eddy-current) measurements to accurately determine wall thickness. Tubing with more than a 0.0003-in. variation in wall thickness was discarded.

As required, the remaining high-quality tubing was cut into 4-in. long test specimens. End plugs and pressurization probes, made from the same alloy as the tubing, were electron-beam welded to the ends of the tube lengths. The specimen length-to-diameter ratio of $>12:1$ was more than sufficient to assure that end restraints, due to the welded plugs, would not influence the biaxial creep-rupture properties. The wall thickness of each specimen was measured to 0.0001 in. by Vidigage. On a given specimen, the variation in wall thickness was within ± 0.0003 in. The outside diameter of each tubular specimen was measured to ± 0.0001 in. with a precision profilometer. Particular attention was

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Figure 1. 12-Pin Biaxial Cladding Test Retort

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Figure 2. ΔT Leg Biaxial Stress-Rupture Test Section
(19-pin array of tubes with wire wrap)

given to attaining accurate wall and diameter measurements, so that well-defined stress levels could be established for the mechanical tests. This factor, together with the selection of high-quality material, was instrumental in reducing data scatter.

In the studies on these two stainless steels, a total of ~ 800 test specimens were fabricated, and creep-rupture tested at temperatures of 900 to 1400°F. There was no instance where specimen rupture could be associated with the end plug welds. This performance is attributed to the high quality of the electron-beam welds, purity of the environment during welding, and the narrow heat-affected zone associated with such welds.

2. Temperature Control

The 12-specimen static sodium retorts were placed in three-zone resistance-wound furnaces which provided an 8 in. isothermal region. Specimen temperatures were measured by three calibrated Chromel-Alumel thermocouples, in wells which extended into the sodium. The temperatures at the top, midplane, and bottom of the axial length of the specimen cluster were measured. Temperature variation along the length of the specimens never exceeded $\pm 2^\circ\text{F}$. Variation from the indicated nominal test temperature was normally within $\pm 3^\circ\text{F}$, although occasionally it fluctuated within $\pm 5^\circ\text{F}$ over the duration of test. On tests where helium was substituted for the sodium environment, temperature uniformity was not as well controlled. This resulted in a variation of about $\pm 5^\circ\text{F}$ along the specimen length.

Specimen temperatures, recorded continuously on strip chart recorders, were monitored daily. For long-term tests, a set of calibrated secondary thermocouples was inserted in the wells once a week, to check for thermocouple drift.

The test temperature was not as precisely defined in flowing sodium tests. Temperature measurement was complicated by the dynamic sodium conditions, heat losses through the loop walls, and difficulty in positioning thermocouples near the test specimens. The bulk inlet sodium temperature was known to $\pm 5^\circ\text{F}$; the axial thermal gradient due to heat losses through the piping walls is estimated at $\pm 10^\circ\text{F}$, over the length of the specimens.

3. Environmental Control

One of the important benefits of testing in sodium is that the test environment can be highly characterized, controlled, and reproduced. This is an important feature when studying the rather subtle parametric effects of variables, such as alloy chemistry, cold work, and stress mode.

Sodium for the static retort tests was supplied from a special loop,⁽¹³⁾ operated to provide the specified⁽³⁾ system quality sodium. Oxygen and carbon impurities were controlled by cold trapping and hot trapping, respectively. Plugging meter checks, and weekly sampling for chemical analysis, assured that the specification chemistry was maintained.

Transfer of the sodium from the supply loop to the individual retorts was accomplished by a special device;⁽⁷⁾ the transfer operation introduced ~5 ppm oxygen contamination to the sodium. The all-welded retort construction, and the careful attention given to leak checking and retort cleaning, resulted in a constant sodium environment during the life of the tests. Each retort was sampled three times; and, from the data on over 50 retorts, the oxygen level was generally found in the range of 3 to 20 ppm, for exposure times of 100 to 10,000 hr. For a given retort, the oxygen usually increased slightly with time — undoubtedly due to impurities in the helium gas which was injected into the sodium each time a specimen ruptured.

For the flowing sodium tests, the oxygen and carbon impurity levels in the sodium test loop were closely monitored by weekly extractions of samples for chemical analyses. Intermediate surveillance for oxygen contamination was made, using a cross-flow plugging meter. During testing, the oxygen content of the sodium was <10 ppm, and the carbon did not exceed 15 ppm. Periodic cold trapping was employed, to maintain the test environment within the limits for system quality sodium.

Helium was used as a test environment on a limited number of tests, as a "control" in our study of the influence of sodium on stress-rupture behavior. Helium was also used for internal pressurization of the specimens, and as a cover gas above the sodium. All helium used in the program was of a special high-purity reactor grade.⁽⁵⁾

4. Loading Control

In our 2:1 biaxial stress tests, the load on the tubular specimen was applied by gas (helium) pressurization. This type of loading has several advantages which are not realized in conventional mechanical loading. For example: (1) multiple, independently loaded specimens can be tested under identical environmental conditions, (2) there are no mechanical linkages involved to introduce system compliance errors, (3) tests are not compromised by building vibrations, earthquakes, or other unpredictable shocks, and (4) the thin-wall specimen design creates a high surface-to-volume ratio, which provides a high sensitivity for environmental and structural studies. The major drawback to this type of loading is the difficulty, compounded by the sodium environment, of monitoring strain in order to establish creep curves and define the primary and secondary creep rates. However, creep curves were generated by depressurizing individual specimens in the multiple-specimen cluster at different time intervals prior to rupture. This yielded a series of individual points which defined the strain-time relationship under a given load (stress) and temperature.

The specimen loads for both stress-rupture and creep tests were applied by slowly admitting pressurized helium gas. A calibrated master gauge with a $\pm 0.5\%$ accuracy was used for initial pressurization; pressures of smaller gauges — one for each specimen — were monitored on a semi-daily basis. Specimen rupture was detected by loss of internal pressure, and a concurrent increase in cover gas pressure above the sodium. The maximum hoop stress applied to the specimen was calculated, using the formula for thin-wall cylinders.⁽¹⁴⁾ Accuracy in this value was enhanced by careful dimensional characterization of each specimen for wall thickness and diameter.

B. REPRODUCIBILITY OF TEST CONDITIONS

The simultaneous testing of a multiple array of specimens in the same environment offers a number of advantages over the conventional single-specimen testing concept. The economy in setup time, testing schedule, and equipment utilization is obvious. Furthermore, the data scatter normally associated with stress-rupture testing is greatly reduced by the identical environmental exposure of the specimens. This also permits the subtle effects of specimen variability due to dimensional and processing anomalies to be separated from environment effects.

This multiple-specimen testing concept was found to produce accurate, consistent data with a high statistical confidence. Because of the high-density testing capability, triplicate specimens were tested for each test condition. In the generation of long-term test data, more than one retort was used for a given temperature. Because of concern for a possibly inherent retort effect, stress levels were selected so that there was an overlap from one retort to the next. With this procedure, it was demonstrated that there was excellent reproducibility between retorts. This is attributed to the careful attention given to environmental control (sodium and temperature), as well as the highly characterized nature of the test specimens.

Figure 3. Comparison of Stress-Rupture Behavior of Type 304 Stainless Steel Cladding (10 to 15% Cold Work) in Static Sodium and Helium

Figure 4. Comparison of Stress-Rupture Behavior of Type 316 Stainless Steel Cladding (10 to 15% Cold Work) in Static Sodium and Helium

IV. ENVIRONMENTAL EFFECTS

In the evaluation of mechanical test results, one must be concerned about the test environment and its potential influence on the test results. The literature contains many examples where the presence of a liquid metal environment has influenced the mechanical properties of materials.⁽¹⁵⁻¹⁹⁾ High-temperature sodium, an extremely active material, has perhaps received the most attention in recent years. Its compatibility with austenitic stainless steels is good, under controlled conditions; but adverse effects, involving corrosion and mass transfer, can result, due to impurity excursions and/or system thermal gradients. Examples of some of these environmental effects are discussed in this section.

A. STATIC SODIUM

Creep and stress-rupture tests in static sodium retorts constituted a major portion of the program, and contributed to the bulk of the data generated. The static sodium provided excellent environmental control, which was necessary to define process and metallurgical effects. The rigid control on test conditions, and the large amount of data needed on such studies, made the static sodium tests both technically and economically attractive. The accurate baseline data provided by the static sodium tests permitted a direct evaluation of flowing sodium effects, and greatly reduced the number of more expensive loop tests required.

To evaluate the effect of the static sodium environment on the stress-rupture properties of Types 304 and 316 stainless steel, the sodium tests were compared with similar tests in high-purity helium under identical test conditions. The static sodium test environment was found to have no effect on the biaxial stress-rupture life of either alloy, at temperatures of 1200°F and above (Figures 3 and 4). A sodium effect was observed on cold-worked Type 316 stainless steel at 1000°F, where the material exhibited greater strength in helium at high stress levels (Figure 4). At lower stresses, the strength was identical in the two environments. Neither cold-worked Type 304 stainless steel nor the annealed Type 316 material showed any influence of test environment at this temperature.

The effect of sodium environment was found to be most pronounced on material strain. At 1000°F, where the cold-worked Type 316 stainless steel rupture life was influenced by test environment, the diametral strains were definitely lower in helium, for a given stress level. A temperature influence is apparent from cold-worked Type 316 stainless steel data at 1400°F, where the effect is reversed, with the helium data showing greater strain than the sodium tests. No comparison was made on Type 316 stainless steel at the intermediate temperature of 1200°F. The cold-worked Type 304 stainless steel was compared at 1000, 1200, and 1400°F, and no pronounced environmental effects on diametral strain were observed. There was a slight indication that the strains at 1200 and 1400°F were greater in helium, but the scatter in the data precluded any firm conclusions.

A more dramatic effect of sodium environment was encountered on a series of uniaxial tests of annealed Type 304 stainless steel. As shown in Figure 5, the static sodium environment decreased the amount of tertiary creep. A similar observation was reported by Böhm and Schneider⁽¹⁹⁾ on austenitic steel tubing filled with sodium. While comparative tests on Type 316 stainless steel tubing under uniaxial stress are limited, they do show the same environmental effect on tertiary creep as for Type 304 stainless steel.

In summary, it can be stated that the major influence of sodium environment on the stress-rupture behavior of Types 304 and 316 stainless steel is to reduce the tertiary stage of creep. This effect is not as pronounced on biaxial tests, since rupture occurs before the material develops much tertiary creep. The effects of sodium on stress-rupture properties seems to vary — even within the narrow temperature range of 1000 to 1400°F. Microstructural observations seem to suggest that the sodium depletes the grain boundaries of austenitic stabilizers, and promotes formation of ferrite and/or sigma phase to weaken the grain boundaries.⁽²⁰⁾ Further evidence that sodium influenced the fracture mechanism was the observation that, at temperatures and stress levels where explosive ruptures were encountered in a sodium test environment, pin-hole type ruptures were exclusively encountered in the helium tests.

In the operation of static sodium retorts, control of the oxygen impurity level is of major concern. The role of oxygen in promoting corrosion and mass transfer is well recognized — particularly in anisothermal systems.

Figure 5. Uniaxial Creep of Annealed Type 304 Stainless Steel in 1200°F Sodium and Helium Environments (21,000 psi stress)

One of the techniques used to reduce the oxygen level is to employ a zirconium hot trap in the system, to getter the oxygen from the sodium. This technique was applied to the three initial static sodium retorts in which specimens were tested. Zirconium foil was placed in the sodium surrounding the Type 304 stainless steel test specimens. Stress-rupture tests were conducted at 1200, 1300, and 1400°F, and sodium sampling during testing indicated that a low oxygen level was maintained. However, post-test examinations of the stainless steel revealed that the zirconium was also an extremely efficient carbon getter, and decarburized the stainless steel by a factor of 1/3 to 1/2 in <1000 hr at 1300 and 1400°F. At 1200°F, decarburization occurred only in a thin surface layer; for longer exposure times, serious decarburization would be expected. Details of these experiments are described by Lee.⁽⁷⁾ The results emphasize that caution must be exercised in the use of zirconium in a sodium circuit.

The influence of copper impurity in sodium on the stress-rupture properties and impact strength of Type 304 stainless steel (10 to 15% cold work) was

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Figure 6. High-Flux Heater With Cladding

investigated in a series of static sodium tests. It was found that the presence of copper did not influence the stress-rupture behavior or impact strength of Type 304 stainless steel at temperatures up to 1300° F.⁽²¹⁾

B. FLOWING SODIUM vs STATIC SODIUM – COLD-WORKED TYPE 304 STAINLESS STEEL

1. Isothermal Loop Conditions

To simulate conditions anticipated for fuel cladding in fast reactors, a series of creep and stress-rupture tests was conducted in a dynamic sodium loop.^(9,10) The ability to internally heat and simultaneously pressurize tubular samples permits simulation of the effect of heat transfer from the fuel to the coolant, and the effect of fission gas pressure and fuel swelling stress on the cladding. To achieve this capability, a high-heat-flux heater (10^6 Btu/ft²-hr) was designed and developed for insertion in the cladding, in such a way that the cladding could also be internally pressurized. This high-flux heater assembly, with cladding, is shown schematically in Figure 6. The developmental work and the design features of the heater assembly are described elsewhere.⁽²²⁾

Experimental work provided information on the stress-rupture behavior of the cladding under the following heat flux conditions: (1) a steady-state flux, (2) a cyclic flux involving two cycle rates, and (3) control test specimens under a zero heat flux condition. The control test assemblies were identical to the heater assemblies, in both geometric and hydraulic aspects. Without internal heating, the control test specimens were under nearly isothermal conditions, with respect to the system sodium. Mass transfer effects were minimized; and, assuming there were no internal interactions with the NaK used for pressurization, the stress-rupture behavior would be expected to be the same as in static sodium. The dynamic sodium control data agreed with the stress-rupture data obtained in static sodium retorts, within the uncertainty in temperature.

2. Nonisothermal Loop Conditions

The influence of nonisothermal loop sodium on the creep and stress-rupture behavior of cold-worked Type 316 stainless steel tubing was investigated by a series of tests in a dynamic sodium loop with a system ΔT of 50° F. These specimens were not internally heated, but were exposed in a hot leg, with the inlet sodium heated to 1200° F. The bulk sodium temperature at the inlet to the heated section was 1150° F.

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Figure 7. 19-Pin Test Assembly With Housing

AI-AEC-13000

The test assembly, which has been reported in detail by Shively,⁽⁸⁾ consisted of 19 individually pressurized tubes of cold-worked (10 to 15%) Type 316 stainless steel. The tubes, 20 in. long by 0.250 in. OD by 0.010 in. wall, were helically wrapped (12 in. pitch) with 0.070-in. wire of annealed Type 316 stainless steel. During assembly, the tubes were placed in a hexagonal array, and a shroud tube was placed around them. A strain allowance of 8% $\Delta D/D$ was built into the array. The completed assembly, shown in Figure 7, was welded into the hot leg of the test loop.

Helium pressure in the tubes was programmed to provide stress-rupture data from one group of specimens and creep data from another group. Internal pressure in the stress-rupture specimens (12 tubes at three different stress levels) was held constant until the tube failed. In the creep test, seven tubes were pressurized to the same level, held constant, and then individual tubes were depressurized at prescribed time intervals (100 to 2613 hr). After completion of the test, the diameter of each tube was measured, to obtain diametral strain as a function of time at pressure. The strains of seven tubes comprise a composite creep curve seen in Figure 8.

The rupture ductility of the wire-wrapped tubes tested in flowing sodium is less than that shown by unwrapped tubes in static sodium. This can be seen by comparison of strain at failure for the two sets of data shown in Figure 9, where the strain at rupture is plotted vs the average strain rate. The flowing sodium data are displaced from those for static sodium; and, from Figure 8, it is seen that the tertiary creep is reduced in the nonisothermal flowing sodium. This comparison suggests an effect between the two different loop sodium conditions, isothermal and nonisothermal, since the control tests in the loop under isothermal sodium conditions gave the same results as the static sodium retort tests.

A comparison of the stress-rupture results from tests in flowing (ΔT leg) sodium and those obtained in static sodium is presented in Figure 10. As can be seen, the stress-rupture data and the associated statistical band for the wire-wrapped tubes tested in flowing sodium overlaps the data and band for unwrapped tubes in static sodium. The slope of the stress-rupture curve for flowing-sodium data is steeper than that for specimens tested in static sodium. There are two possible reasons. One is that mass transfer of carbon from the

Figure 8. Biaxial Creep and Stress-Rupture Strain of Cold-Worked Type 316 Stainless Steel as a Function of Time at Pressure in 1200°F Static and Dynamic Sodium

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Figure 9. Rupture Strain of Cold-Worked Type 316 Stainless Steel
as a Function of Average Strain Rate at 1200° F

Figure 10. Stress-Rupture Behavior of Two Lots of Type 316 Stainless Steel (10 to 15% Cold Work) During Exposure to Static and Flowing Sodium at 1200°F

cladding resulted in a weakening; this effect has been observed in zirconium-gettered static sodium,⁽⁷⁾ and more recently by General Electric in flowing sodium.⁽²⁶⁾ The other reason is that wire-cladding mechanical interactions, which occurred during wire attachment and/or during the test, reduced the life under stress. The carbon migration is the more likely source of the weakening, since analyses of specimens from the test showed a 10% reduction in average carbon content during the test.

3. Carbon Transport Conditions

Carbon in austenitic stainless steels plays an important role in the strength and ductility properties of the alloy. In addition to its influence on solution and precipitation strengthening mechanisms, carbon is a stabilizer for the metastable austenitic phase. In high-temperature sodium systems, the carbon in these steels can be altered by the ability of the sodium to move carbon from one region to another, depending on the carbon activities within the sodium circuit. The effects of changing carbon content in sodium on the engineering design properties are of vital importance to the reactor designer. The problem of carbon transport and its effects on materials behavior is extremely complex, and could not be completely defined within the scope of this program. The following comments describe some observations and experiments which were performed on this program, and perhaps provide some insight on the effects of carbon transport on mechanical behavior.

a. Decarburizing Conditions

During the study of the stress-rupture properties of the two austenitic stainless steels in high-purity static sodium, zirconium foil was placed in the sodium to getter oxygen and maintain a high-purity sodium environment.⁽⁷⁾ Stress-rupture tests conducted on these specimens were compared with those obtained in nongettered static sodium. The results, shown in Figure 11, indicate that, for cold-worked Type 304 stainless steel, a significant reduction in strength occurs in zirconium-gettered sodium. This behavior was especially pronounced at 1300 and 1400°F. Further investigation revealed a depletion of carbide phases at the surfaces exposed to the zirconium-gettered sodium. The depth of the depleted zone increased with test temperature, with the entire 0.010-in. wall showing carbide depletion at 1400°F. In the carbon-depleted regions, coarse

Figure 11. Effect of Zirconium Gettering in Sodium on Mechanical Properties of As-Received Type 304 Stainless Steel (10 to 15% Cold Work)

8-F6-014-16B

sigma particles were present in the grain boundaries. Carbon analysis on the stainless steel indicated that the bulk carbon was decreased 50% (0.06 to 0.03 wt %) during the 400-hr exposure to 1400°F gettered sodium. An 800-hr exposure at 1300°F produced about a 30% bulk carbon depletion. With exposure at 1200°F for 2650 hr, carbon loss was within the experimental accuracy of the analysis, although some depletion was observed in the structure and reflected in the stress-rupture strength.

Limited studies performed on Type 316 stainless steel show the same behavior in zirconium-gettered sodium, but the data suggest that this alloy is more resistant to decarburization than is Type 304 stainless steel. This is attributed to the presence of molybdenum, which reduces the carbon diffusion rate in the alloy.

A loss of strength due to carbon depletion would have been predicted, based on the performance of the commercial low-carbon modification of the alloys (Types 304L and 316L). However, the amount of sigma phase encountered in the carbon-depleted structures is much greater than in the low-carbon commercial alloys. While it is recognized that cold work can cause sigma formation, thermal controls on the test material revealed that cold work alone could not account for all the sigma particles observed in the 1300 and 1400°F samples. The formation of large quantities of sigma phase represents an important difference between sodium-decarburized stainless steel and the commercial low-carbon version of the alloy.

In 1200°F flowing sodium tests, decarburization of the stainless steel specimens was encountered under nonisothermal loop conditions (50°F system ΔT). Although a zirconium getter was not employed, the steel lost $\sim 10\%$ of its bulk carbon content. In these studies, both Types 304 and 316 stainless steel were in the cold-worked and annealed conditions. Differences due to alloy composition or cold work were not detectable. The microstructures showed carbon-depleted regions at the surfaces exposed to sodium, but large quantities of sigma phase were not observed. The possibility exists that the sigma particles are present in the grain boundaries, but are too small to be resolved by optical microscopy. Indications of weakened grain boundary conditions were apparent in the loss in rupture strength and ductility — the same behavior observed in the zirconium-gettered static sodium tests.

Figure 12. Rotating Sodium Retort for Case Carburization

General Electric⁽²⁶⁾ has observed significant decarburization in 1300°F sodium loops. Based on their work, and the observations in zirconium-gettered static sodium, the decarburization rate appears to be strongly dependent on temperature. At temperatures of 1000°F and lower, carbon transport within a stainless steel system may be insignificant. This feature would reduce the potential for carburizing long-term components in the LMFBR primary system from decarburized cladding.⁽²⁷⁾ While additional work is needed to determine the temperature threshold for decarburization of cladding, it would appear that only cladding at 1200°F and higher will significantly lose carbon to the primary system.

b. Carburizing Conditions

Two types of carburization conditions can exist in large complex sodium systems. A pulse-type carbon contamination in the sodium can occur during initial reactor startup (foreign material in piping, etc.), or at any time during operation by the development of a system leak (introduction of carbonaceous material due to seal failure, etc.). This type of contamination is usually short-lived, and produces a thin high-carbon layer in the structural components in contact with the sodium. The second condition which influences the carbon content of structural alloys is the gradual pickup or loss of carbon, due to mass transport in the sodium system. This transport of carbon (and possibly other interstitials) between different materials at the same temperature, or the same material at different temperatures due to the influence of thermodynamic (free energy) differences, produces a rather uniform carbon distribution across the component wall.

A retort test was completed, whereby a high-carbon case was applied to the OD surfaces of thin-walled Type 316 stainless steel tubing (10 to 15% cold work). The design of the retort is shown in Figure 12. The retort contained nine tubular specimens with outside surfaces exposed to sodium, along with control tabs of 0.001, 0.004, and 0.007 in. thickness from the same heat of material. Five calibrated amounts of carbon were introduced to the retort, in the form of methane gas, with each of the additions being allowed to react for 24 hr. In order to facilitate mixing and homogenization of the sodium, the retort was rotated at 13 rpm within the furnace. Sodium samples were extracted from the 1200°F retort after complete reaction of the first and last

Figure 13. Rotating-Beam Fatigue Tests for Type 316 Stainless Steel Tubing
(0.275 in. ID by 0.010 in. wall) at 1200° F

methane gas additions. Respective analyses for these sodium samples showed 9 and 20 ppm carbon. Based upon a diffusion equation (for carbon in Type 316 stainless steel at 1200°F),⁽²⁸⁾ the methane gas additions should have produced a carbon case of ~2.5 wt % on the specimen surfaces. Calculations based on analysis of the control tabs revealed that the carbon case was ~2.0 wt % carbon, to a depth of 0.001 in. of the 0.010-in. specimen wall.

Two of the case-carburized specimens were tensile tested at 1200°F, and the results are compared with those of the as-received material in Table 1. The data show that the carbon case increased the ultimate strength, and decreased the tensile ductility, of the thin-wall tubing. The yield strength was not changed by the carburization.

TABLE 1
1200°F TENSILE DATA FOR AS-RECEIVED AND CASE-
CARBURIZED TYPE 316 STAINLESS STEEL TUBING
(Heat 65808, 10 to 15% Cold Work)

Condition	Ultimate (ksi)	Yield (ksi)	Elongation (% in 2 in.)
As-received	67.7 - 69.9	50.5 - 55.9	17.5 - 29.0
Carburized	74.7	51.6	12
Carburized	76.3	54.4	12

Rotating-beam fatigue tests (13 rpm) at 1200°F (in air) were performed on both as-received and case-carburized tubing. The data are tabulated in Appendix III. Figure 13 shows a comparison of these data with an S-N curve for annealed material presented in the literature.⁽²⁹⁾ The displacement of the data points is due to the 10 to 15% cold work in the tubing material used in the tests. The scatter in data is considered normal for fatigue tests. The limited data points for the case-carburized tubing were found to lie within the scatter band for the noncarburized tubing at the lower strain levels. However, at 2% strain range, the case-carburized specimen seems to have greater fatigue strength. The lack of additional carburized specimens prevented further testing in this high cyclic strain region.

a. Stress-Rupture Strength

b. Strain-Rate Sensitivity to Biaxial Stress

Figure 14. Comparison of Biaxial Stress-Rupture Properties of Case-Carburized and Noncarburized Type 316 Stainless Steel Tubing (10 to 15% Cold Work) in 1200°F Helium

The biaxial stress-rupture behavior of two case-carburized specimens are compared with noncarburized material in Figure 14. As expected, the rupture strength was increased, and the strain rate decreased, by the presence of the high-carbon case. Examination of the ruptured specimens showed that explosive-type ruptures occurred. In contrast, noncarburized specimens develop pinhole-type failures at this test temperature (1200°F). The carburized specimens did not exhibit any surface cracking or crazing, as might be expected of a hard, brittle surface case. Strain measurements, taken on the diameters of these specimens, gave values of 2.57% at 40,000 psi hoop stress and 3.64% at 33,000 psi. On noncarburized material, the rupture strain was over 6% for comparable stress.

To study the behavior of stainless steels under uniform long-term carburization conditions, specimens of annealed Type 304 stainless steel tubing (0.010-in. wall) were exposed to carburizing 1100°F sodium for 3000 hr. The basic 12-specimen static sodium retort design was utilized, with the added feature of a pneumatically operated agitation device to mix the sodium. Figure 15 shows the agitator in relation to the test specimens. A timing device activated the agitator for eight cycles each hour, throughout the 3000-hr exposure. The carbon source was 2-1/4 Cr - 1 Mo alloy steel, used for the agitator and the retort shell surrounding the specimens. Three of the specimens in the retort were stressed, to evaluate the effect of stress on the rate of carburization. The remaining nine specimens were unstressed.

Carbon analyses, given in Table 2, indicate that the unstressed specimens picked up about 0.05 wt % carbon. Two of the three specimens which were under stress during the sodium exposure were analyzed, and were found to have higher carbon contents than the unstressed material. Although the number of analyses are rather limited, the extensive carbon tests performed on specimens from the ΔT loop experiments indicate that the values presented in Table 2 are significant, and represent real differences in carbon level. The tolerance due to experimental-analytical error is only about ± 0.01 wt %. It was therefore concluded that stress enhances the rate of carburization of the stainless steel.

Carbon diffusion into the steel is apparently accelerated by lattice strains in the metal due to the applied stress. However, the stress levels chosen were not of sufficient spread to establish any stress-influenced trends. Supporting

Figure 15. Specimen Complex for Bulk Carburization

TABLE 2
BULK CARBURIZATION OF ANNEALED TYPE 304 STAINLESS
STEEL IN 1100°F ISOTHERMAL SODIUM
(3000 hr, 2-1/4 Cr - 1 Mo Carbon Source)

Specimen No.	Applied Hoop Stress (psi)	Carbon Content (wt%)	
		Starting	Final
A4-85	0	0.05 - 0.06	0.11, 0.10
A4-92	27,000	0.05 - 0.06	0.14, 0.15
A4-91	30,000	0.05 - 0.06	0.13, 0.12

evidence of this phenomenon is found in a Russian study,⁽³⁰⁾ where it was observed that the rate of decarburization of 2-1/4 Cr - 1 Mo steel (ferritic) increased with stress. Based on the experimental evidence in the present work, one might speculate that cold-worked austenitic stainless steels would be more susceptible to carburization than their annealed counterparts.

Because of the phase-out of this program, the planned mechanical testing of these carburized specimens was not performed.

4. Sodium Exposure After Helium Injection

Helium generation during irradiation in a fast neutron flux can result in embrittlement of structural alloys. A limited study was performed to evaluate the effect of such embrittlement on the biaxial creep and stress-rupture behavior of Types 304 and 316 stainless steels in static sodium.

Types 304 and 316 stainless steel tubes were tested in static sodium at 1200°F in the annealed state. The stress-rupture behavior of the control specimens and the helium-injected tubes is presented in Figures 16 and 17. As can be seen in Figure 16, the presence of helium from cyclotron injection reduced the life of Type 304 stainless steel from 400 to 300 hr. In the case of Type 316 stainless steel, helium reduced the stress-rupture life by more than 50%, at each of two stress levels (Figure 17).

In conjunction with the testing of helium-injected specimens in the retort containing Type 304 stainless steel specimens, a series of creep specimens of noninjected tubes were tested, by removing the pressure stresses after various

Figure 16. Effect of Helium on Stress-Rupture Life of Annealed Type 304 Stainless Steel in Static Sodium

Figure 17. Effect of Helium on Stress-Rupture Life of Annealed Type 316 Stainless Steel in Static Sodium

Figure 18. Comparison of Biaxial Creep of Two Stainless Steels in 1200°F Static Sodium at the Same Hoop Stress

exposure times. The results of the profilometer measurements are displayed in Figure 18. Since these tubes were stressed identically to the stress-rupture tubes, with and without helium, it is possible to make a direct comparison of the strain at failure. The strain at failure of the helium-injected specimens lies on the creep curve for the noninjected specimens, within experimental error. If primary and secondary creep are unaffected by the presence of helium, then it appears that helium reduces life by reducing the amount of third-stage creep. It appears, from preliminary data, that helium has the greatest influence on nonuniform strain of Type 304 stainless steel during creep.

Comparison of the fracture strain of the helium-injected Type 316 stainless steel tubes has been superimposed on the creep curves (Figure 18), showing that helium can also influence uniform strain in the biaxial stress state. The strain at failure fell below the creep curve. The mechanism by which helium influenced the primary and secondary creep cannot be resolved from the results of these tests.

C. PROCESSING EFFECTS

1. Composition

During the early work on this program, six heats of Type 304 stainless steel tubing and two heats of Type 316 tubing were studied, to evaluate the effect of heat-to-heat variations on the biaxial stress-rupture properties of the alloys.⁽³¹⁾ The tests on mill-annealed tubing were conducted in helium, at temperatures of 1200 to 1350° F, for rupture times up to 8200 hr. Analysis of the results indicated that one might expect as much as a factor of 8 variation in stress-rupture life due to heat-to-heat effects. This variation could not be correlated with alloy composition, although the carbon-plus-nitrogen contents seemed to play the most significant role.

In a more recent study, the biaxial stress-rupture properties of three heats of Type 316 stainless steel were compared in 1200° F sodium. The tubing for all three heats was purchased from the same tube mill, to the same specification,⁽³²⁾ and contained 10 to 15% cold work. The stress-rupture properties were compared, to evaluate variations which can be expected in as-purchased tubing. To separate process variables from alloy chemistry, identical stress-rupture tests were also conducted on annealed material from the three heats.

Figure 19. Heat-to-Heat Study of Type 316 Stainless Steel Tubing (10 to 15% Cold Work) in 1200° F Sodium

Figure 20. Heat-to-Heat Study of Annealed Type 316 Stainless Steel Tubing in 1200° F Sodium

In the cold-worked condition, the biaxial stress-rupture curves are compared in Figure 19. Heat No. 20303 is the strongest of the heats at high stresses, but shows a break in the stress-rupture curve after ~ 1000 hr. Based on stress-rupture behavior of cold-worked tubing, it appears that this break is associated with recovery effects of cold work in the alloy. If this heat had a higher degree of cold work than the other two heats, the energies promoting diffusion, recrystallization, and recovery processes would be greater. The influence of these processes on stress-rupture behavior would therefore be observed sooner. Diametral strain measurements, obtained after rupture, indicated that the strongest of the three heats (Heat 20303) also exhibited considerably lower rupture strains. This behavior also indicated that the tubing from Heat 20303 contained a greater amount of cold work, and that the higher strength at high stresses might not be attributable to compositional differences. Stress-rupture tests on annealed material again showed Heat 20303 to be the strongest of the three heats (Figure 20). The rupture ductility was also lower for this heat. Since mechanical working process variations were virtually removed by annealing, it is concluded that the difference in stress-rupture properties was really due to alloy composition. The chemistry of the three heats, given in Table 3, show the elemental compositions to be quite similar. However, if the heats are evaluated on the basis of those elements favoring austenite formation vs those elements which favor ferrite and sigma, subtle differences in the heats appear. Using the formulas:⁽³³⁾

$$1) \text{ Ni equivalent (austenite formers) } = \text{Ni} + 30 \text{ C} + 0.5 \text{ Mn}$$

$$2) \text{ Cr equivalent (ferrite-sigma formers) } = \text{Cr} + 2.5 \text{ Si} + 1.8 \text{ Mo}$$

the calculated Cr/Ni ratios for the heats are listed in Table 3. Although the differences are small, the ratios indicate that Heat 03574 would have the least tendency to form sigma phase under long-term, high-temperature exposure. A comparison of the rupture strain, in Figure 21, shows a definite correlation with Cr/Ni ratio for the three heats investigated. With refinement, this technique may be a valuable tool in the heat selection for specific properties.

In conducting biaxial creep tests in flowing sodium,⁽⁸⁾ an unusual phenomenon was observed, whereby the axial length of Type 316 stainless steel specimens shortened under biaxial stress. This observation suggests that the

TABLE 3
CHEMICAL COMPOSITIONS OF TYPE 316 STAINLESS STEEL TUBING
HEATS USED IN HEAT-TO-HEAT STUDY

Heat No.	C	Mn	S	P	Si	Ni	Cr	Mo	Cu	Co	Cr/Ni
03574	0.07	1.57	0.013	0.011	0.53	13.68	17.34	2.38	0.13	0.05	1.38
65808	0.06	1.72	0.007	0.012	0.40	13.30	17.30	2.33	0.06	0.03	1.41
20303	0.06	1.53	0.013	0.010	0.58	12.95	17.30	2.39	0.05	0.05	1.48

6507 4771

Figure 21. Creep Stress as Function of Cr/Ni Ratio for Cold-Worked Type 316 Stainless Steel in 1200°F Sodium

material is anisotropic; because, for the 2:1 stress state used in these tests, the tubes should not change length during plastic strain. Since both cold-worked and annealed tubes from this heat (65808) exhibited shortening, some anisotropy is intrinsic to this heat. However, the degree of anisotropy is greater in the cold-worked steel than in the annealed steel. Annealed Type 304 stainless steel did show large creep strain, but no evidence of tube shrinkage. Hence, the Type 304 stainless steel heat is apparently isotropic.

2. Cold Work

In studies on the mechanical behavior of austenitic stainless steels in high-temperature sodium, thin-wall seamless tubing from two highly characterized heats of Types 304 and 316 stainless steel were used. The tubing was delivered with 10 to 15% cold-work — expressed as reduction in cross-sectional area in the final drawing operation at the tube mill. Batches of this tubing were solution annealed by laboratory treatment, and the creep and stress-rupture behavior were compared with the original cold-worked tubing (Type 304 — 1900°F, 1/2 hr; Type 316 — 1950°F, 1/2 hr).

Results have shown that cold work, at the level of 10 to 15%, has a significant effect on the long-term mechanical properties of the two alloys. Under certain conditions of temperature and stress, where recovery and recrystallization processes are operative, the 10 to 15% cold work reduced the stress-rupture strength of the alloy to a level below that of the annealed alloy. This is illustrated in Figures 22 and 23, which show that the strengthening effects of cold work are temporary, with the duration decreasing with increasing test temperature. The strengthening benefits of cold work are retained longer for Type 316 stainless steel than for Type 304 stainless steel, at the same temperature. These observations suggest that the recovery processes are slower, at the same temperature and stress, in Type 316 stainless steel than in Type 304 stainless steel. Other investigators^(34,35) have found improved alloy stability and higher recrystallization temperatures in austenitic stainless steels containing refractory metal additions (e. g., molybdenum in Type 316 stainless steel).

The observation that the cold-worked alloy became weaker than the annealed alloy under long-term stressed conditions is probably attributed to a difference in fracture mode. While both cold-worked and annealed material have been

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Figure 22. Biaxial Stress-Rupture Strength of Cold-Worked (10 to 15%) and Annealed Type 304 Stainless Steel in Static Sodium

9-JY24-085-24

Figure 23. Biaxial Stress-Rupture Strength of Cold-Worked (10 to 15%) and Annealed Type 316 Stainless Steel in Static Sodium

8081-1-1

a. Annealed ($\sigma = 7200$ psi, Rupture Time = 2120 hr,
 $\epsilon = 6\%$)

8002-1-5

b. 10 to 15% Cold Work ($\sigma = 6800$ psi,
Rupture Time = 813 hr, $\epsilon = 6\%$)

Figure 24. Effect of Cold Work on Microstructure and
Void Density of Type 304 Stainless Steel
in 1400° F Sodium

found to fail intergranularly, the cold-work alloy develops numerous grain boundary cracks and voids which accelerate rupture. This behavior is illustrated in Figure 24.

As expected, a reduction in rupture strain by the presence of cold work has been observed for both alloys. Figure 25 shows the 1000-hr biaxial rupture strain of Type 304 stainless steel to be greatly reduced at all temperatures (900 to 1400°F), for the 10 to 15% cold-worked tubing. The Type 316 stainless steel tubing exhibits similar cold-worked behavior, although the magnitudes of strain in the cold-worked material are greater for Type 316 stainless steel. Under uniaxial stress, a similar strain behavior has been observed with cold work. The amount of cold work governs strain during each stage of creep, as can be seen in Figure 26. The strain for each stage of creep decreases as the cold work increases, with the greatest influence of cold work showing up in third-stage creep. While there appears to be a saturation effect on the rupture strain at higher cold-work levels, the rupture strength and strain rate have been found to be strongly influenced by high degrees of cold work. In Figure 27, the stress-rupture strength of the 38% cold-worked alloy is much less than that of the annealed material.

In the solution-annealed condition, both alloys exhibit about the same stress-rupture characteristics. As shown in Figure 28, the data could be considered to lie within the scatter band expected for heat-to-heat variation for a given alloy.⁽³¹⁾

In contrast, the stress-rupture behavior of Type 304 stainless steel and Type 316 stainless steel in the 10 to 15% cold-worked condition is presented in Figure 29. The cold-worked Type 316 stainless steel alloy is stronger in the high-temperature range (~1400°F), and weaker in the low-temperature range (~1000°F), than the Type 304 stainless steel alloy. However, the Type 304 stainless steel exhibits more rapid recovery, which makes the Type 316 stainless steel alloy more attractive for long-term, low-stress applications.

Microstructural examination of cold-worked and annealed specimens revealed that formation of sigma phase is enhanced by cold work. Cold working promotes the transformation of austenite to ferrite, which, in turn, can more readily form sigma. In Type 304 stainless steel, sigma formation was found to be

9-AU12-091-11

Figure 25. 1000-hr Rupture Strain of Type 304 Stainless Steel as a Function of Temperature

8-925-157-6B

Figure 26. Effect of Cold Work on the Strain, During Three Stages of Uniaxial Creep in Sodium at 1200°F, of Type 304 Stainless Steel

r/

Figure 27. Uniaxial Stress-Rupture Behavior of Type 304 Stainless Steel in 1200°F Sodium (0.062 in. Sheet, Reference Heat 20013)

Figure 28. Biaxial Stress-Rupture Behavior of Annealed Types 304 and 316 Stainless Steel in Static Sodium

8-025-157-2A

Figure 29. Biaxial Stress-Rupture Behavior of Cold-Worked Types 304 and 316 Stainless Steel Tubing in Static Sodium

enhanced by stress. Lula et al.⁽³⁶⁾ observed that recrystallization of other cold-worked alloys accelerates sigma formation. In Type 304 stainless steel, sigma was found concentrated at grain boundaries; but, in Type 316 stainless steel, much of the sigma was found as a fine precipitate within grains and on slip bands. In both alloys, sigma particles were observed to be more prominent at 1400 and 1200° F than at 1000 and 900° F.

While sigma-phase formation is generally regarded as undesirable, there was no evidence that it contributed to the degradation of the mechanical properties of the cold-worked alloys. It is believed that recovery and recrystallization effects contribute to the major loss in properties, and the presence of sigma-phase plays, at most, a secondary role.

D. MECHANICAL METALLURGY

1. Stress-State Correlations

During reactor operation, structural materials may be subjected to a variety of complex mechanical stresses, resulting from thermal bowing, thermal gradients, fluid pressures, and mechanical interactions among components. While the interrelationship and magnitude of these stresses cannot be accurately established, valuable insight into the influence of stress mode on stress-rupture behavior can be gained by experimental studies.

The effects of biaxial stress state on the stress-rupture properties of Types 304 and 316 stainless steel were explored under three different stress modes. The objective of this work was to attempt to correlate the properties with stress mode, so that the properties could be expressed independently from the state of stress. For the Type 304 stainless steel alloy under uniaxial, 2:1 biaxial, and 1:1 biaxial stresses, the stress-rupture curves can be brought into good agreement by applying the von Mises distortion energy yield theory⁽³⁷⁾ to the stress. This can be expressed mathematically by

$$\sigma_0^2 = 1/2 [(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2] , \quad \dots (1)$$

where:

σ_0 = effective stress

σ_1 = axial stress

Figure 30. Correlation of Stress State With Rupture Life, Using von Mises' Yield Theory, for Annealed Type 304 Stainless Steel

Figure 31. Effect of Stress Mode on Cold-Worked (10 to 15%) Type 316 Stainless Steel in 1200°F Sodium

σ_2 = tangential stress

σ_3 = radial stress.

For thin-walled tubes subjected to an axial load and internal hydrostatic pressure, radial stresses are insignificant, and Equation 1 reduces to

$$\sigma_0^2 = \sigma_1^2 + \sigma_2^2 - \sigma_1 \sigma_2 \quad \dots (2)$$

For uniaxial, 2:1 biaxial, and 1:1 biaxial stress states, the effective stresses are $\sigma_0 = \sigma_1$, $\sigma_0 = 0.866 \sigma_2$, and $\sigma_0 = \sigma_1 = \sigma_2$, respectively.

Using the effective stress, the uniaxial and biaxial stress-rupture results obtained in 1200° F sodium on annealed Type 304 stainless steel were compared. Figure 30 shows the very good correlation obtained, using von Mises' yield criterion, including the 1400° F uniaxial data, for the same heat of the alloy in vacuum.⁽³⁸⁾

For Type 316 stainless steel, the von Mises criterion did not hold — rather, the stress-rupture strength was found to correlate best with the maximum principal stress (σ_1 , σ_2 , or σ_3). The stress-rupture strengths of cold-worked Type 316 stainless steel tubing, tested under three stress modes in 1200° F sodium, are plotted in Figure 31, using the principal stress. Although only limited tests were performed on annealed Type 316 stainless steel, the same principal stress correlation seemed to hold. Norton⁽⁵¹⁾ studied Type 316 stainless steel under the same three stress states used in this investigation. He also concluded that the maximum principal stress seems to be the best criterion for rupture life of Type 316 stainless steel.

To demonstrate the effect of stress mode on the axial strain of Type 316 stainless steel tubing, two stress-rupture tests were performed at the same stress level — 27,000 psi. One test was under uniaxial stress, while the other was a 1:1 biaxial stress test. The creep curves plotted in Figure 32 show the effect of the stress modes on the axial strain. The restraint on axial strain imposed by the hoop stress on the cladding is very pronounced. The amount of tertiary creep strain is also significantly reduced, under the biaxial stress mode.

Figure 32. Effect of Stress Mode on Axial Strain of Type 316 Stainless Steel Tubing

Figure 33. Heat-Zone Temperature Profiles Along Cladding

To study the creep-rupture behavior when stress increases linearly with time, a series of pressure ramp tests was performed on thin-wall tubes. In a test to provide data for the FFTF fission gas plenum strain model, strain measurements on 20% cold-worked Type 316 stainless steel specimens subjected to uniform stress ramps were compared with constant-stressed specimens in 1200°F static sodium.⁽³⁹⁾ The results of these tests showed that the required hoop stress to produce equivalent diametral strains is ~20% greater, under a linear stress ramp, than under constant-stress conditions. In another test in flowing sodium, two cold-worked (10 to 15%) Type 316 stainless steel specimens were stress ramped to study the validity of the life fraction theory in predicting creep-rupture behavior.⁽⁴⁰⁾ The observed rupture life agreed very well with the predictions based on cumulative life fraction.

2. Creep Rupture Under Thermal-Gradient Secondary Stresses

a. Steady-State Thermal Gradient

Twelve biaxial stress-rupture tests were conducted with internally pressurized and heated cladding in flowing sodium (20 ft/sec) at 1200°F.^(9,10) Tests were performed, using two heat fluxes: 1×10^6 and 0.8×10^6 Btu/ft²-hr.

Cladding temperature profiles over the 2-in. heated zone for the higher heat flux are plotted in Figure 33. The uncertainty in temperature is $\pm 10^\circ\text{F}$. The peak mean temperature of the cladding wall is $1285 \pm 10^\circ\text{F}$, and the temperature difference between inside and outside walls varies from 40 to 60°F. The temperature gradient is assumed to be linear across the tube wall. A calculation of the thermal stresses in the specimen wall indicated $\sim 8000 \pm 1630$ psi stress due to the elastic strain gradient through the wall. This stress, when superimposed on a high-pressure stress, should diminish by creep relaxation. For steady-state heating, creep occurs under the combined action of the mechanical hoop stress generated by the internal pressurization and temperature gradient through the wall. Maximum creep and the associated failure would be expected to occur at the highest temperature and stress point. The strain profiles of tube tests with internal heating essentially followed the temperature profile along the wall of the tube. This is illustrated in Figure 34. The failures occurred in the peak strain position (which is also the peak temperature position), confirming the expected creep-rupture behavior. Therefore, when the

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Figure 34. Relationship of Strain Profiles to Temperature Profiles Along Cladding

Figure 35. Predicted Life vs Actual Life of Cold-Worked Type 304 Stainless Steel Tubes Under Constant Heating and Pressure in Flowing Sodium

tests with internal heating are compared directly to the tests in static sodium, the comparison is at the temperature and stress corresponding to the midwall in the peak temperature region.

The comparison between the stress-rupture tests in static sodium and the tests in flowing sodium with steady-state internal heating on cold-worked Type 304 stainless steel is displayed in Figure 35. The temperature band represents the uncertainty in the expected behavior, based on static sodium data. The limits on the band were obtained by interpolating for the limits of uncertainty in the peak midwall temperature of the internally heated tubes. The stress-rupture behavior of the tubes under steady internal heating and pressure fell within the predicted range for the peak conditions of temperature and stress.

It is also instructive to compare the average strain rate (strain at failure divided by the time to failure) for the tangential direction in both the static and flowing sodium tests. As before (Figure 35), it is possible to make a comparison by drawing the strain-rate vs stress limits corresponding to the range of temperatures estimated for the peak midwall temperature in the internally heated tubes. The results of the tests of tubing with steady internal heating fell within the predicted limits (Figure 36).

These test results indicate that creep-rupture behavior in the presence of a high heat flux (steady-state thermal gradient) is primarily a function of the total applied stress and temperature. If these two factors are calculated, the behavior can be accurately predicted, using isothermal test data.

The microstructures of specimens tested with internal pressure in static sodium, and the specimens tested with internal heating and pressure in flowing sodium, are essentially the same. In summary, the microstructures from both static and flowing sodium exhibited:

- 1) Intergranular failure
- 2) Void formation throughout the wall
- 3) Little or no sigma phase
- 4) Carbide precipitation at the grain boundaries.

8-MA15-084-1B

Figure 36. Effect of Stress on Average Strain Rate for Steady-State Heated Type 304 Stainless Steel in Flowing Sodium

Figure 37. Ferrite Layer on Surface Near Inlet of Internally Heated Tube Exposed to Flowing Sodium for 2600 hr

AI-AEC-13000

These observations are a further confirmation that internal heating does not radically change the behavior, mechanically or metallurgically, from that anticipated from isothermal tests. In addition, migration of interstitials under a temperature gradient is either absent, or too small to be observed, in the limited time of these tests.

One important observation concerning the microstructure of the steady heated specimens was the presence of a ferrite layer on the outside surface of the cladding material, typical of stainless steel corrosion in sodium. This layer can be seen in Figure 37. Its presence is not unexpected, since the conditions associated with an internally heated tube favor corrosion and mass transfer.⁽²³⁾ The maximum thickness of the layer occurred in the peak temperature region. The growth of the ferritic layer for all specimens appears to be parabolic with time, indicating that the growth is diffusion controlled. On the basis that the ferrite layer does not contribute directly to the load-carrying capacity of the tube, it is estimated that the ferrite layer results in a 4% reduction of stress-rupture life. This is too small to be seen, in the comparison with the static sodium tests.

b. Cyclic Thermal Gradient

Stress-rupture tests, in which cladding was pressurized and exposed to a cyclic heat flux, were performed to study the effects of cyclic thermal gradients, including reactor load variations on cladding performance. Theoretical papers^(24,25) have suggested that such variations may produce a process called "enhanced creep," as a result of repeated stress relaxation. When the thermal stress produced by the heat flux in these tests is relaxed, inhomogeneous deformation (plastic strain gradient) results across the wall. When the heat flux is removed, the plastic strain gradient establishes a residual stress of opposite sign. This stress relaxes via secondary creep, giving rise to a new thermal stress when the heat flux is reapplied. The new thermal stress is higher than that which existed when the heat flux was removed. Thus, the relaxations of the thermal stress and the associated residual stress produce an increment of primary creep strain during each "heater on" cycle. The primary creep strain increment is additive to the mechanical strain produced by the stress from internal pressure.

The results of stress-rupture tests on Types 304 and 316 stainless steel, for which the heat flux was cycled on and off, are displayed in Figure 38. The life was predicted to be reduced, by a factor of 2, with respect to tests under steady-state heater operation. One of two data points for annealed Type 304 stainless steel under cyclic heat flux was comparable to the point for the cold-worked steel. The other data point, which showed an appreciably lower life, was from a sample that was found to have a flaw on the internal surface of the tube, believed to be responsible for the reduced life. The rupture strain for the longer-lived tube was 3.4%. This value is considerably lower than that expected from stress-rupture tests on annealed Type 304 steel in static sodium (nominally, 8-1/2% $\Delta D/D$). The difference is probably due to the effects of cycling on tertiary creep. The cycling effect on rupture life for two different heat fluxes, 1.0×10^6 and 0.8×10^6 Btu/ft²-hr, is also displayed in Figure 38. Apparently, the heat flux and the associated thermal stresses are not dominating factors in determining rupture life. The strain at failure was slightly lower at the lower heat flux, but this may simply reflect a slightly lower average

9-JY24-085-20A

Figure 38. Results of Heat Flux Cycle Rate on Stress-Rupture Properties of Types 304 and 316 Stainless Steel

temperature. [The strain at failure decreased with temperature for cold-worked Type 304 stainless steel in static sodium tests.⁽²²⁾]

The rupture life of cold-worked Type 316 cladding for the 12-min cycle is about a factor of 2 longer than Type 304 at the same frequency. This parallels observations on the stress-rupture life, made from tests in static sodium in the temperature range of 1200 to 1400°F. Tests on cold-worked Type 316 stainless steel cladding (10 to 15%) were performed at two different cycle frequencies. In Type 316, the rupture life decreased as the time period increased; whereas, for Type 304 cladding, the life increased when the time period increased. For Type 316, the strain at failure decreased from 8.6 to 1.24% when the time period was increased by a factor of 6. The conclusion drawn is that thermal cycling affects the amount of third-stage creep strain.

It is well known that grain boundary separation (voids) begins to occur during third-stage creep. In this case, the process is best described as localized creep at a stress concentration center. Accordingly, the creep processes result in rapid necking in the stress concentration region (notch effect), effectively concentrating the deformation in one location. This action under cyclic heat flux was observed in an annealed Type 304 stainless steel specimen in which a scratch was present on the internal surface, and in a cold-worked Type 316 specimen at the low cycle rate. Thus, once a crack is initiated by creep, it will continue to propagate under cyclic stresses acting at the crack tip. At this point, additional uniform creep in the region of the crack essentially ceases, and a mechanical instability condition prevails. In this way, the cyclic stress at the crack tip accelerates the normal crack propagation.

3. Mechanical Interaction Between Components

In a series of biaxial stress-rupture tests of cold-worked Type 316 stainless steel tubes in flowing sodium,⁽⁸⁾ the tubes were helically wrapped on a 12-in. pitch with 0.070-in. diameter annealed Type 316 stainless steel wires. These mechanical spacers were found to interact with the tubing; and, at least in some cases, they influenced the rupture life of the tubing. The wires became loose during the test, as a result of tube shortening (attributed to anisotropy), and no longer were in intimate contact with the tubes (Figure 39).

Figure 39. Loose Wire on Pin No. 21

There was indication of very localized self-welding between tubes and wires. Adhesion was observed at two specific locations along the length of a tube: (1) at the ends of the tube, where the wire remained tightly compressed against the cladding, and (2) at the center of the tube, where a loose wire touched a neighboring tube. Near the ends of the tube, the adhesion remained intact after the test. A weld of this type is shown in Figure 40. After the wire was removed from the tube, it was seen that the adhesion was essentially between high points on the surface. This suggests that the contact pressure was not very large. In contrast, the contact adhesions of loose wires to neighboring tubes were not intact after the test. There is evidence that welds were formed and broken during test. This is illustrated in Figure 41. As can be seen in the figure, small nuggets were removed from the wire and deposited on the cladding.

Besides self-welding, evidence was observed of a wire-cladding mechanical interaction, caused by the clamping force used during welding of the wires to the end caps. Seven of the twelve ruptures in these tests occurred under the wires, within 1 in. of the end caps. While the ruptures appear to be associated with the procedure used to attach the wires, self-welding was also observed at these locations. The cause-effect relation could not be resolved from the limited test data available.

Figure 40. Wire-Cladding Interaction on Tube Tested in Flowing Sodium

Figure 41. Several Self-Welds (Adhesions) of Loose Wire to Neighboring Tube

V. APPLICATION OF DATA TO FBR DESIGN

The data from the AI program have direct application in three areas: (1) high-temperature design criteria, (2) fuel rod modeling, and (3) specification preparation. The AI data apply directly to stress allowables for high-temperature design criteria. In addition, information from this program impacts on structural design rules, such as life fraction and stress-state corrections. Fuel rod performance modes make direct use of the mechanical properties information obtained on this program, in the calculations of cladding performance. Some of the required mechanical properties information provided by this program which contributes to cladding performance calculations is presented in Table 4 for two failure criteria, life fraction and ductility limit. For specification preparation, data on the effects of composition, degree of cold work, residual stresses, and scratches have provided a better understanding of how accurately these variables can be controlled and specified.

A. APPLICATION TO STRUCTURAL DESIGN CRITERIA

The present program contributes to high-temperature structural design criteria in two ways: (1) the biaxial stress-rupture data on annealed stainless steels are a significant addition to what is needed to establish better allowable stresses for stress rupture and creep, and (2) the results of the AI program have an impact on the general applicability of structural design rules (i. e., life fraction and von Mises' distortion energy applied to stress rupture). The purpose of this section is to describe the relation of the AI program to high-temperature structural design.

1. Application of Data to Stress Allowables

Section III of the ASME Boiler and Pressure Vessel Code has a new high-temperature code case (1331-5). In addition, the ASME code now extends to other components, such as piping, valves, etc., which define a pressure boundary. The data from the AI program should be considered by code committees in establishing stress allowables for future high-temperature code cases. In fact, the biaxial stress-rupture data obtained in static sodium and helium would appear to be applicable in setting stress allowables for piping. The mechanical behavior of the specimens should be comparable with that of piping, because the

TABLE 4
MECHANICAL PROPERTIES PROVIDED FOR CLADDING
FAILURE CRITERIA

Life Fraction	Ductility Limits
Effects of Stress and Temperature on Biaxial Stress-Rupture Life	Effects of Strain Rate and Temperature on Diametral Rupture Strain
Effects of Cold Work on Biaxial Stress-Rupture Life and Low-Cycle Fatigue Life	Effects of Cold Work on Diametral Rupture Strain
Effects of Decarburization on Biaxial Stress-Rupture Life	Effects of Decarburization on Biaxial Creep and Rupture Strains
Effects of Stress State on Rupture Failure Criteria	Effects of Stress State on Rupture Strain
Effects of Stress State on Time to Reach End of Secondary Creep	Effects of Stress State on the Strain at the End of Secondary Creep
Effects of Sodium on Biaxial Stress-Rupture Life	Effects of Sodium on Rupture Strain
Effects of Heat-to-Heat Variation on Biaxial Stress-Rupture Life	Effects of Heat-to-Heat Variation on Biaxial Secondary Creep and Rupture Strain
Effect of Steady-State and Cyclic Heat Flux on Biaxial Stress-Rupture Life	Effect of Steady-State and Cyclic Heat Flux on Diametral Rupture Strain
Effects of Case Carburization on Low-Cycle Fatigue and Stress-Rupture Life	Effects of Case Carburization on Diametral Rupture Strain

wall thickness exceeded the average grain size by a factor of 10 or more, and the thickness-to-diameter ratio was in the same range as that of piping.

A major contribution of the AI program to the work of code committees is the experimental data (~20,000 hr) which help establish stress allowables for long-term behavior in future high-temperature code cases. The current (1971) set of stress allowables in Code Case 1331-5, mentioned previously, for the " S_t " curves are summarized in the following listing, where " S_t " is the lowest stress value of the following time-dependent properties:

- 1) Two-thirds of the minimum stress to cause rupture at time "t".
Time "t" refers to the total duration of specified operating conditions (loading and temperature combinations) which can occur during the entire design life of the component. The stress to cause rupture is that initial stress in a conventional constant-load, uniaxial, tensile stress-to-rupture test which is associated with rupture of the specimen at "t" hours.
- 2) Eighty percent of the minimum stress to cause initiation of tertiary creep at time "t". The "initiation of tertiary creep" is defined as that point in a conventional constant-load, uniaxial, tensile creep test where the observed strain significantly departs from the secondary creep (linear) stage.
- 3) The minimum stress to produce 1.0% total strain after time "t" under a constant stress loading. The elastic, plastic, primary creep, and secondary creep strains shall be included in the total strain. These values can be obtained from isochronous stress-strain curves.

The AI data for annealed Types 304 and 316 stainless steel could improve the statistics for setting stress allowables according to the first requirement (stress rupture). The rupture data at low stresses, long times, and low temperatures are particularly meaningful. While these data were obtained in the 2:1 stress mode, the program demonstrated that (for Type 304 stainless steel, at least) the von Mises correlation can be used to convert the stress-rupture data to the uniaxial mode.⁽⁵⁾

The AI biaxial creep and stress-rupture data tended to confirm the Leyda and Rowe⁽⁴¹⁾ correlation between rupture time and the time to reach the end of secondary creep. This correlation was used to establish the allowable stresses for the onset of tertiary creep (the second code requirement).

A few AI tests have provided data on isochronous stress-strain behavior of Type 316 stainless steel. The work on cold-worked Type 316 stainless steel indicates that the rupture strain would fall below the 1% level in the code (the third requirement).

The results of efforts to correlate the stress-rupture behavior of stainless steel could influence the character of the " S_t " curves of future high-temperature

Figure 42. Larson-Miller Plot for Cold-Worked Type 316 Stainless Steel

code cases. As part of the present investigation, an evaluation was made of parameter methods of interpolating and extrapolating stress-rupture data. Results showed that an excellent fit was obtained, using a modified Larson-Miller parameter, with the hyperbolic sine of stress rather than stress to a power. The best-fit equation for cold-worked Type 316 stainless steel is

$$P = T (14.0 + \log t_f) = 16,620 - 2015 \log [\sinh (0.7 \times 10^{-4} \sigma)] , \quad \dots (3)$$

where:

P = parameter value

T = absolute temperature ($^{\circ}\text{K}$)

t_f = time to rupture (hr)

σ = stress (psi)

A parameter plot of the AI data for cold-worked Type 316 stainless steel is presented in Figure 42. The preceding correlation was developed for fuel pin modeling (see Section V-B), and applied to cold-worked Type 316 stainless steel. Also, the hyperbolic sine stress dependence has been found to improve the fit for annealed Type 304 stainless steel.

2. Application to Life Fraction and to Isotropy Assumptions

The life fraction rule is applied in designing for stress rupture and the creep-fatigue interaction. The life fraction rule, applied to stress rupture for primary loads, was partly substantiated by isothermal tests in the present program, in which the stress changed with time in predetermined steps.⁽⁴²⁾ The agreement between predicted and observed life was very close. The success of these tests in verifying the life fraction rule, compared to the results of Guarnieri⁽⁴³⁾ on Type 321 stainless steel, is attributed to the longer duration of these tests, more careful control and characterization of the material, better definition of environmental effects, and more reliable baseline data from which the predicted life was calculated. In addition, we made a correction to the stress for the creep strain which occurs during the constant-load time interval.

The program provided insight into structural design assumptions relating to mechanical isotropy. The major evaluations are summarized in Figures 30 and 31. These show, respectively, that data for annealed Type 304 correlated

with von Mises' distortion energy criteria, indicating isotropy; and data for cold-worked Type 316 correlates on the basis of maximum principal stress, indicating anisotropy. However, in 2:1 biaxial creep tests on annealed Type 316 stainless steel tubing, performed late in the program,⁽⁴²⁾ this material behaved isotropically. This was inferred from the lack of tube shrinkage. It is likely that, in the annealed condition, both Types 304 and 316 stainless steel behave isotropically; and when cold worked, both show anisotropy.

B. APPLICATION TO FUEL ROD PERFORMANCE MODELS

To judge the performance of cladding requires that a failure criterion be selected. There are two alternative approaches — one can apply a ductility limit to the thermal creep strain (damage model), or apply a life fraction rule. The advantages of each are discussed in the following sections.

1. Ductility Limit Properties

A ductility limit is frequently used as a failure criterion, or specified for the design of fuel pin cladding. To establish the design value and the associated uncertainty, a value for unirradiated stainless steel must first be obtained which includes all the effects listed in Table 4. Once the unirradiated value of the ductility limit is known, the correction for neutron irradiation can be made. The AI program provides data for determining the unirradiated value of the rupture strain (ductility limit).

In addition to the ductility limit, application of fuel pin performance codes and structural analysis codes necessitate the use of a thermal creep equation. It is generally assumed that radiation does not change the creep rate. However, other factors, such as stress state, loading history, heat-to-heat variations, and the sodium environment, may affect the rate or the time to reach mechanical instability (end of secondary creep). The AI program provides information to aid the materials engineer in establishing a thermal creep equation. The purpose of this section is to demonstrate how the AI program data can be used to help establish a ductility limit and a thermal creep equation for cold-worked Type 316 stainless steel.

The first step is to determine a steady-state thermal creep strain limit as a function of temperature, strain rate, and the other operating variables. This

by no means is an easy assignment, because the effects can interact. For example, decarburization effects on ductility are a function of alloy composition and degree of cold work.⁽⁸⁾

The rupture strain of unirradiated cold-worked Types 304 and 316 stainless steel tubing was measured as a function of temperature and strain rate under internal pressure loading (2:1 stress state) in static sodium. It was found that the rupture strain generally decreases with decreasing strain rate, and approaches 1% at low strain rates. The following equation was obtained to describe the rupture strain as a function of strain rate in the temperature range 1000 to 1200°F:

$$\log \epsilon_f = 0.6021 + 0.575 \tanh (0.2121 \log \dot{\epsilon} + 0.5615) , \quad \dots (4)$$

where:

ϵ_f = strain at rupture (%)

$\dot{\epsilon}$ = average strain rate (%/hr)

The standard deviation for calculated $\log \epsilon_f$ is ± 0.278 . At high strain rates, ϵ_f approaches 15%; while, at low strain rates, ϵ_f approaches 1%.

Now that the strain-rate dependence of rupture strain of unirradiated cold-worked Type 316 stainless steel is established, the rupture strain should be corrected for the effects of decarburization, composition and processing, and cyclic heat fluxes. At a given temperature, the effects of decarburization on rupture strain are greater at low strain rates than at high strain rates.^(40,44,45) The influence of temperature on the effects of decarburization on rupture strain of cold-worked Types 304 and 316 stainless steel is complicated by competing mechanisms. At high temperature (1400°F), the rupture ductility at a given strain rate is higher for decarburized than for nondecarburized steel. This is because the removal of carbon accelerates the recovery of cold work. However, the strain rate of decarburized Types 304 and 316 stainless steel at a given stress is greater, and must be accounted for in the calculation of creep strain. At a lower temperature (1200°F), the amount of carbon removed is less, and the cold work is not recovered. At 1200°F, the rupture strain at a given strain rate is lower for decarburized than for nondecarburized Types 304 and 316 stainless steel. At still lower temperature (1100°F), essentially no carbon

is removed, and there is no effect on rupture ductility. Thus, at high temperatures, decarburization of cold-worked Types 304 and 316 stainless steel accelerates recovery, so embrittlement effects are masked; at intermediate temperatures, the decarburization reduces rupture strain; and, at low temperatures, no effect is observed. When fuel rod cladding temperature exceeds 1100°F, some adjustment of rupture strain and creep strength must be made to account for decarburization.

An evaluation of the effects of decarburization on rupture strain of cold-worked Type 316 stainless steel at 1200°F shows that, in the intermediate strain rate range, the position of the inflection point in the plot of Equation 4 shifts to a lower strain rate at the same rupture strain. In contrast, the high and low strain rate plateaus were unaffected. The effect of decarburization on rupture strain should be negligible at 1100°F, because there will be insufficient time at temperature to remove carbon. In summary, within the temperature range of Equation 4, rupture strain at low strain rates applicable to steady-state design of fuel rods would appear to be unaffected by decarburization. Since the ductility limit at low strain rates represents the minimum value, the overall ductility limit is not reduced by bulk decarburization. (Ductility limits for reactor transients which induce higher strain rates must include a correction for decarburization associated with the strain rate.)

The effects of compositional and processing variations (heat-to-heat) on the ductility limit are difficult to assess, because heats which are more ductile generally creep faster at the same stress (are weaker). One way to evaluate these effects is by use of the Monkman-Grant correlation, which relates rupture time to secondary creep rate.⁽⁴⁶⁾ This correlation leads to the conclusion that the same factors control the rate of secondary creep and the time to failure. This assertion is further substantiated by correlations of G. V. Smith⁽⁴⁷⁾ and Leyda and Rowe⁽⁴¹⁾ between the time to failure and the time to reach the end of second-stage creep at a given test temperature. The correlation for several heats of Type 316 stainless steel, including those tested in this program, agreed with that of Leyda and Rowe. At low stresses (where primary creep is virtually absent), the Monkman-Grant correlation can be used to calculate a limiting strain, by multiplying the secondary creep rate by the rupture time. The interesting conclusion from the Monkman-Grant correlation is that the effects of

minor compositional variations on rupture strain will be compensated for by the effects of these minor compositional variations on the secondary strain rate at low strain rates. For fuel rod design, low strain rates are of primary importance. The changes of secondary creep rate with heat-to-heat variations can be taken into account by adjusting the proportionality constant and the stress dependence, in the general equation

$$\dot{\epsilon}_s = K\sigma^n \quad \dots (5)$$

where:

$\dot{\epsilon}_s$ = secondary creep rate (in. /in. /hr)

K = constant (dependent upon temperature)

σ = stress

n = constant

Work on the effects of a cyclic heat flux through the tube wall on the stress rupture of cold-worked Type 316 stainless steel demonstrated (see Section IV) an enhancement of the creep rate in biaxially stressed tubing. The results show how secondary loads (thermal gradient stresses) can interact with primary loads (membrane stresses) to produce an overall creep effect, not predicted from consideration of the membrane stress alone. In the test, the rupture strain increased by a factor of 2, while the average strain rate increased by a factor of 4, at a maximum midwall temperature of $\sim 1280^\circ\text{F}$. This is predicted by the rupture strain correlation of Equation 4. In other words, the rupture-strain - strain-rate sensitivity is apparently unaffected by a cyclic heat flux. The primary effect of a cyclic secondary load (thermal gradient) superimposed on a steady internal pressure is to increase the creep rate. The rupture ductility is increased with the increasing strain rate. While this removes the necessity for a correction for the effect of a cyclic heat flux on the strain-rate sensitivity of rupture strain, it does require the use of an appropriately modified creep rate in the overall performance estimates.

Data on biaxial rupture strain of cold-worked Type 316 stainless steel indicate that the unirradiated^(40,44,45) and irradiated⁽⁴⁸⁾ biaxial rupture strains merge at low strain rates ($\sim 10^{-5}$ in. /in. /hr). For the case of sizing a fission

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Figure 43. Strain Hardening vs Time Hardening

gas plenum, the strain rates are of the order of 10^{-5} in./in./hr (gas pressure stresses are $<10,000$ psi). Therefore, to size the fission gas plenum, the ductility criterion may be based on unirradiated properties. This approach cannot be used in applications where high stresses are present, causing plastic strain or high creep rates.

The prediction of creep behavior of fuel cladding is complicated because temperatures and stresses vary, even in the steady-state reactor operation. Most of the data available were obtained isothermally at constant load (stress increases slightly during the test). To make use of these data requires the designer to make an assumption about a creep hardening law. Presently, there are four hardening laws available (time hardening, strain hardening, theory of Rabatnov, and life fraction hardening).^(43,49,50) To illustrate the difference between strain hardening and time hardening, consider Figure 43, showing tests made at a given temperature, with periodic increase in stress. The question is, "What is the resultant creep strain if the stresses were changed from σ_1 , to σ_2 , to σ_3 , periodically through the test?"

The resultant creep strain for a periodic step increase in stress, using the strain-hardening assumption, is obtained by adding the strain increments for Δt_1 , Δt_2 , and Δt_3 , as shown in Figure 43a. The beginning of each successive time interval is determined by the amount of strain at the end of the previous interval. This is equivalent to jumping from one creep curve to the next, along an isostrain line. The resultant creep strain for a periodic step increase in stress, using the time-hardening assumption, is obtained by adding the strain increments for Δt_1 , Δt_2 , and Δt_3 , as shown in Figure 43b. In this case, the transition from a creep curve for one stress to the next higher stress is along a line corresponding to the time at the end of the interval. As can be seen in Figure 43c, the time-hardening assumption predicts less creep strain than the strain-hardening assumption, for the case where the stress is increased periodically. In most fuel cladding evaluations, the most conservative assumption is strain hardening. It should be noted that, if creep strain is a linear function of time at constant load or stress, all assumptions lead to the same result.

The next requirement for developing a thermal creep equation is to evaluate the effects of a steady temperature gradient. Work under the AI program has shown that good correlation is obtained between isothermal stress rupture and

stress rupture under internal heating, using cladding midwall temperatures and average hoop stresses. The correlation appears to depend upon relaxation of the thermal stresses, with the result that the average creep rate across the wall is given by the midwall conditions.

The following thermal creep equation is based on an evaluation of the data obtained in this program.

$$\epsilon = A_o t^{1/3} (\sinh B\sigma)^m \exp(-36,255/T) \quad , \quad \dots (6)$$

where:

ϵ = biaxial thermal creep strain (%)

$$A_o = e^{27.821}$$

t = time (hr)

σ = average true hoop stress (psi)

$$B = 3 \times 10^{-4}$$

$$m = 0.63541$$

T = absolute temperature (°K)

Plus and minus 2σ limits (statistical uncertainties) are obtained by multiplying the values of ϵ , calculated from the preceding equation, by 4.75 and 0.210, respectively.

The effects of a severe cyclic thermal gradient (heat flux change, 0 to 10^6 Btu/ft²-hr, in a few minutes) on the average creep rate of cold-worked Type 316 stainless steel were studied in this program. It is recommended that a factor be incorporated into the thermal creep equation for the amount of enhancement of creep strain per cycle. This may be accomplished by modifying the constant (A_o) in Equation 6 as follows:

$$A = A_o \left(1 + \frac{3N}{1000} \right) \quad , \quad \dots (7)$$

where:

A = constant for cyclic heat flux

N = number of cycles

In summary, to apply the ductility limit, the strain rate dependence of the rupture strain of unirradiated cladding must first be corrected for the effects of environment. The results of the AI program show that, at low strain rates, the rupture strain is not significantly affected by decarburization or cyclic heat flux. Heat-to-heat variations are accounted for by lumping their effects into the thermal creep equation. To apply ductility limit to design, a thermal creep equation is needed. To apply the creep equation to time-varying stresses and/or temperatures requires a hardening law.

2. Life Fraction Method

The life fraction method is currently used to set design limits in many components of FBR's. It is equally applicable to fuel rod design. This method sums the fractions of life expended by stress-rupture, short-term-tensile, and cycling loads. Failure is defined as that point when the sum of life fractions equals one. The principle of the method is illustrated in Figure 44 for a conventional creep-rupture curve for cold-worked Type 316 stainless steel. While the strain varies nonlinearly with time, the fraction of life increases linearly with time. This is a salient difference between the ductility limit and life fraction failure criteria. As was the case for ductility limits, estimates of the life fraction require that the unirradiated stress-rupture data on cold-worked Type 316 stainless steel be corrected for the effects of radiation, decarburization, stress state, cyclic heat fluxes, and composition. These corrections lead to a design equation for in-pile stress rupture. The design equation is then used to calculate the fraction of life expended during each period of service life; the summation must be <1 at the design life of the fuel rod, with adequate reliability margin.

The effects of radiation on stress rupture have been the subject of considerable experimental inquiry. To date, the majority of the data available are for annealed Types 304 and 316 stainless steel. Some biaxial stress-rupture data for irradiated cold-worked Type 316 stainless steel are available.⁽⁴⁸⁾ The trend of all these data indicates that, below 10,000 psi hoop stress and up to 1200°F, the unirradiated and irradiated stress-rupture curves merge. Therefore, it may be assumed that, if the cladding stresses remain below this level, unirradiated stress-rupture properties of Type 316 stainless steel may be used directly, without correction for the effects of radiation.

Figure 44. Life Fraction vs Strain Fraction for Cold-Worked
Type 316 Stainless Steel at 1200°F

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The effect of decarburization on the stress-rupture behavior of cold-worked Types 304 and 316 stainless steel was investigated in the present program. The results indicate that decarburization reduces the stress-rupture life significantly. The effect is larger at 1400°F than at 1200°F, presumably because decarburization enhances the cold-work recovery processes at the higher temperature, and the amount of carbon removed is less at 1200°F. The following equation was developed for the combined effects of temperature and stress on the rupture life, using a modified Larson-Miller correlation:

$$\log t_f = -14.00 + \frac{16,298}{T} - \frac{2111}{T} \log [\sinh (0.6 \times 10^{-4} \sigma)] , \quad \dots (8)$$

where:

t_f = time to rupture (hr)

T = temperature (°K)

σ = stress (psi)

The standard deviation for $\log t_f$ is ± 0.139 .

Equation 8 can be applied for temperatures of 1200°F and above. Below 1200°F, Equation 9, relating stress rupture of unirradiated cold-worked Type 316 stainless steel, is used, because decarburization is negligible:

$$\log t_f = -14.00 + \frac{16,842}{T} - \frac{1812}{T} \log [\sinh (0.8 \times 10^{-4} \sigma)] . \quad \dots (9)$$

The standard deviation for $\log t_f$, calculated by Equation 9, is ± 0.167 .

Equation 9 is valid at all temperatures, if no decarburization occurs. The two equations constitute a set, defining the stress-rupture properties of the cladding. The estimate of life fraction expended in a given time uses the minimum time to rupture obtained from the two equations.

The effect of composition on stress rupture was briefly studied in the course of the AI program. The stress-rupture life was correlated with the effective chromium/nickel ratio of the steel — the higher this ratio, the longer the stress-rupture life at a given stress. Since these studies on alloy composition did not provide a complete understanding of how composition influences the stress-rupture life of cladding, other guidance must be sought.

Figure 45. Heat-to-Heat Study of Stress-Rupture Behavior of Type 316 Stainless Steel

The problem of handling heat-to-heat variations can be resolved by considering where the mean of the heat of cold-worked stainless steel used for evaluation lies, relative to other heats. Stress-rupture data of several heats of cold-worked Type 316 stainless steel are presented in Figure 45, where the solid lines are plotted from Equation 9. Since the equation reasonably represents a mean for all the heats, the heat-to-heat variability may be accommodated by increasing the standard deviation on $\log t_f$ to ± 0.300 and ± 0.600 for Equations 8 and 9, respectively.

The final correction to the stress-rupture design equation is for the effects of a cyclic heat flux on stress-rupture life. Results of tests showed that cycling the heat flux on internally pressurized tubes reduced the rupture life by a factor of two. This factor must be applied to the calculated rupture life under a given stress.

In summary, the biaxial stress-rupture equation for unirradiated cold-worked Type 316 stainless steel has been modified for the effects of the environment. Decarburization, which occurs in a flowing sodium system, has a significant effect at low stresses, where the data must be applied to fuel pin design for steady state. Corrections for the effects of heat-to-heat variations and cyclic heat flux have been made. The effect of neutron irradiation may be ignored at low stresses, since the postirradiation stress-rupture life converges with the unirradiated rupture life at such stresses.

The life fraction method has at least two advantages over the ductility limit method: (1) there is no requirement for an assumption about a creep hardening law, and (2) low-cycle fatigue and creep-fatigue interactions can be included in life fraction expressions. It is not easy to see how the ductility limit fits into considerations of low-cycle fatigue or creep-fatigue interaction.

C. APPLICATION TO SPECIFICATION PREPARATION

The procurement documents prepared for this program were available for the formulation of the current RDT Standards for cladding for the FFTF Driver Fuel Pins and for other LMFBR's. Of course, additional technical requirements and quality assurance provisions have been included in these RDT Standards. The testing performed in this program has provided additional information, which can help in the preparation of future fuel cladding and structural materials specifications.

The current work has revealed the existence of anisotropy in cold-worked Type 316 stainless steel. Residual stresses introduced during cold working may account for the anisotropy observed in the cold-worked Type 316 stainless steel tubes. Up to now, tubing specifications have not called out any distribution of cold work; rather, specifications generally have required an average degree of cold work, in terms of a total reduction in cross-sectional area. Because anisotropy influences failure criteria as well as design assumptions, it may be important to include a provision for it in future specifications.

REFERENCES

1. LMFBR Program Office, "Liquid Metal Fast Breeder Reactor Program Plan, Volumes 1 through 10," WASH-1101 through 1111 (August 1968)
2. J. E. Bodine, R. L. McKisson, and E. W. Murbach, "System Quality Sodium Description and Specification," NAA-SR-TDR-11859 (April 1966)
3. R. L. McKisson and E. W. Murbach, "Specification: Sodium, Systems Quality, Fast Reactor Environment," Atomics International ST0170NB0047 (July 15, 1965)
4. W. L. Cockrell and A. Carnazzola, "Specification: Tubing, Steel and Nickel-, Iron-, Chromium Alloy, Corrosion Resistant, Seamless, Thin Wall," Atomics International ST0160NB0017 (February 16, 1967)
5. D. F. Atkins, "Stress Rupture Behavior of Types 304 and 316 Stainless Steel Cladding in High-Temperature Static Sodium," AI-AEC-12976 (September 30, 1970)
6. W. T. Lee, "Biaxial Stress-Rupture Properties of Austenitic Stainless Steels in Static Sodium," AI-AEC-12694 (June 1968)
7. W. T. Lee, "Biaxial Stress-Rupture Properties of Austenitic Stainless Steels in Zirconium-Gettered Sodium," NAA-SR-12353 (October 1, 1967)
8. J. H. Shively, "Biaxial Creep and Stress Rupture of Wire-Wrapped Cladding in 1200°F Sodium Under ΔT Conditions - Interim Report," AI-AEC-12944 (April 15, 1970)
9. J. H. Shively and M. W. Mahoney, "Thermal Gradient Effects on Stress-Rupture Behavior of Thin-Walled Tubing," AI-AEC-12896 (January 2, 1970)
10. J. H. Shively, "Thermal Gradient Effects on Stress-Rupture Behavior of Thin-Walled Tubing," AI-AEC-12695 (June 25, 1968)
11. W. L. Cockrell, "The Characterization of AISI 304 Tubing for the LMFBR Cladding Program - Task 10," NAA-SR-TDR-12514 (October 1967)
12. M. W. Mahoney and D. F. Atkins, "Characterization of AISI 316 Stainless Steel for the LMFBR Program - Task 10," AI-AEC-TDR-12845 (May 1969)
13. E. W. Murbach, J. E. Bodine, and N. W. Heath, "The Systems Quality Sodium Loop-Design, Construction and Start-up," NAA-SR-12327 (May 1967)
14. R. J. Roark, Formula for Stress and Strain (McGraw-Hill Book Co., New York, 1954) p 268
15. H. E. McCoy, Jr. and D. A. Douglas, Jr., "Effects of Environment on the Creep Properties of Type 304 SS at Elevated Temperatures," ORNL-2972 (September 5, 1962)

AI-AEC-13000

16. W. Rostoker, J. M. McGaughey, and H. Markus, Embrittlement by Liquid Metals (Reinhold Publishing Corporation, 1960)
17. N. S. Stoloff and T. L. Johnston, "Crack Propagation in a Liquid Metal Environment," Acta Met. 11 (April 1963) p 251
18. R. C. Andrews, "Topical Report No. 5, Effect of High Temperature Sodium on Austenitic Ferritic Steels," MSAR-66-78 (May 1966)
19. H. Böhm and H. Schneider, J. Nucl. Mat'ls. 24 (1967) p 188
20. R. L. McKisson and E. L. Babbe, "High Flux Heater Design and Development," AI-AEC-12681 (June 15, 1968)
21. D. F. Atkins, "Mechanical Behavior of Type 304 Stainless Steel in Copper Contaminated Sodium in the Range 950 to 1300°F," AI-AEC-12788 (April 1, 1969)
22. J. H. Shively, D. F. Atkins, and W. T. Lee, "Creep Under High Heat Flux and Stress Rupture Properties of Austenitic Stainless Steels in Elevated-Temperature Sodium," ANL-7520, Part I (November 1968) p 74
23. J. Hopenfeld and D. K. Darley, "Dynamic Mass Transfer of Stainless Steel in Sodium Under a High-Heat-Flux Condition," NAA-SR-12447 (July 15, 1967)
24. J. Bree, "Elastic-Plastic Behavior of Thin Tubes Subjected to Internal Pressure and Intermittent High-Heat Fluxes With Application to Fast-Nuclear-Reactor Fuel Elements," J. Strain Analysis 2 (1967) p 226
25. J. Bree, "Incremental Growth Due to Creep and Plastic Yielding of Thin Tubes Subjected to Internal Pressure and Cyclic Thermal Stresses," J. Strain Analysis 3 (1968) p 122
26. "Quarterly Progress Report Number 11," GEAP-13539-11 (November 1969)
27. "Quarterly Technical Progress Report - Fuels, Materials and Coolant Chemistry Programs, January-March 1971," AI-AEC-12993 (April 30, 1971)
28. W. J. Anderson and G. V. Sneesby, "Carburization of 304 Stainless Steel in Sodium," NAA-SR-5282 (September 1, 1960)
29. "A Study of Piping Materials for Sodium Piping in LMFBR Plants," CFB-4122-228 (April 1969)
30. V. N. Bykov et al., "The Problems of Corrosion Resistance of Structural Materials in Sodium," CEMA Symposium on Atomic Power Stations with Fast Reactors, Vol I, JPSR-48330 (July 1, 1969) pp 426-461
31. K. E. Horton, R. C. Andrews, R. C. Werner, and D. F. Atkins, "Effect of Sodium on the Metallurgical and Mechanical Properties of Candidate LMFBR Alloys," IAEA Symposium, Vienna, December 1966

32. Atomics International Specification ST0160NB0017, February 16, 1967
33. A. L. Schaeffer, *Welding Journal*, 26 (1947) pp 6015-6205
34. W. Siegfried, "Investigation into the Development of Intercrystalline Fracture in Various Steels Under Triaxial Stress," Creep and Fracture of Metals at High Temperature (HMSO, London, 1956) p 333
35. W. D. Roland, "Influence of Cold Work on the Creep Rupture Properties of Type 347 Stainless Steel," Masters Thesis - MIT (1959)
36. Lula et al., "The Effect of Cold Work on Elevated Temperature Properties of Type 301, 305, and 310 Stainless Steels," *ASM Transactions* (May 1957) p 921
37. G. E. Dieter, Mechanical Metallurgy (McGraw-Hill, N. Y., 1961) pp 60-64
38. D. Kramer, K. R. Garr, C. G. Rhodes, and A. G. Pard, "Helium Embrittlement of Fuel Cladding Alloys for Potential Service in Fast Breeder Reactors," Radiation Damage in Reactor Materials 1, p 303, IAEA Vienna Conference, 1969
39. "Annual Technical Progress Report, Fuels, Materials and Coolant Chemistry Programs, GFY 1970," AI-AEC-12971, pp 52-55
40. "Quarterly Technical Progress Report, Fuels Materials and Coolant Chemistry Programs, October-December 1970," AI-AEC-12987 (September 30, 1971)
41. W. E. Leyda and J. P. Rowe, "A Study of the Time for Departure from Secondary Creep for Eighteen Steels," *ASM-TR-P-9-6.1* (1969)
42. "Annual Technical Progress Report - Fuels, Materials, and Coolant Chemistry Programs," AI-AEC-13007 (August 15, 1971)
43. G. J. Guarnieri, "The Creep-Rupture Properties of Aircraft Sheet Alloys Subjected to Intermittent Load and Temperature," *ASTM STP-165* (1954) p 105
44. "Sodium Technology Development Program, Mass Transfer Investigation in Liquid Metal Systems, Quarterly Progress Report No. 15, September-November 1970," GEAP-13539-15 (December 1970)
45. W. T. Lee, "Zirconium Gettering Effect of Stainless Steel in Sodium," *Nuclear Applications and Technology*, 2 (August 1969) p 155
46. F. C. Monkman and N. J. Grant, "An Empirical Relationship Between Rupture Life and Minimum Creep Rate," Deformation and Fracture at Elevated Temperature (MIT Press, Cambridge, Mass., 1965) p 91
47. G. V. Smith et al., "Creep and Rupture of Several Chromium-Nickel Austenitic Stainless Steels," *Trans. ASM*, 42 (1950) pp 935-980

48. J. Standring, I. P. Bell, H. Tickle, and A. Glendinning, "Effects of Neutron Irradiation on the Creep-Rupture Properties of Type 316 Stainless Steel Tubes, Irradiation Effects in Structural Alloys for Thermal and Fast Reactors (ASTM-STP-457, Philadelphia, Pa., 1969) p 414
49. I. Finnie and W. Heller, Creep of Engineering Materials (John Wiley and Sons, Inc., New York, 1959)
50. Y. N. Rabotnov, "On the Equation of State of Creep," Joint International Conference of Creep, New York, August 25-29, 1963, London, September 30-October 4, 1963, p 2-117, Westminster, London, S.W.I. 1963
51. F. H. Norton, "Creep in Tubular Pressure Vessels, Trans. ASME, 61 (1939) p 239

APPENDIX I

BIBLIOGRAPHY OF PUBLISHED REPORTS AND PROGRAM SUPPORTING DOCUMENTS

A. PUBLICATIONS AND TOPICAL REPORTS

- 1) C. E. Weber, "Stability of Cladding and Structural Alloys on a High Temperature, Liquid Metal, Fast Neutron Environment," NAA-SR-Memo-11871 (March 1966)
- 2) K. E. Horton, R. C. Andrews, R. C. Werner, and D. F. Atkins, "Effects of Sodium on the Metallurgical Properties of Candidate LMFBR Alloys," SM-85/33 (December 1966)
- 3) B. R. Hayward, E. L. Babbe, and D. K. Darley, "High Heat-Flux Heater Development, Preliminary Performance Tests," NAA-SR-12242 (December 1966)
- 4) E. W. Murbach, J. E. Bodine, and N. W. Heath, "The Systems Quality Sodium Loop Design, Construction, and Startup," NAA-SR-12327 (May 1967)
- 5) J. Hopenfeld and D. K. Darley, "Dynamic Mass Transfer of Stainless Steel in Sodium under High-Heat-Flux Conditions," NAA-SR-12447 (July 1967)
- 6) W. T. Lee, "Biaxial Stress-Rupture Properties of Austenitic Stainless Steels in Zr-Gettered Sodium," NAA-SR-12353 (October 1967)
- 7) E. W. Murbach, "Sampling Blanks for the LMFBR Cladding Program," NAA-SR-12616 (January 1968)
- 8) J. H. Shively, "Thermal Gradient Effects on Stress-Rupture Behavior of Thin-Walled Tubing," AI-AEC-12695 (June 1968)
- 9) W. T. Lee, "Biaxial Stress-Rupture Properties of Austenitic Stainless Steels in Static Sodium," AI-AEC-12694 (June 1968)
- 10) R. L. McKisson and E. L. Babbe, "High Heat Flux Heater Development Status Report," AI-AEC-12681 (June 1968)
- 11) E. W. Murbach and N. W. Heath, "Evaluation of an Electrochemical Oxygen Meter," AI-AEC-12700 (June 1968)
- 12) R. L. Eichelberger, "The Solubility of Oxygen in Liquid Sodium, A Recommended Expression," AI-AEC-12685 (November 1968)
- 13) D. F. Atkins, "Mechanical Behavior of Type 304 Stainless Steel in Copper Contaminated Sodium in the Range 950 to 1300° F," AI-AEC-12788 (April 1969)

- 14) E. L. Babbe, J. H. Shively, and M. W. Mahoney, "High Heat Flux Heater Development Status Report II," AI-AEC-12813 (July 1969)
- 15) E. W. Murbach and N. W. Heath, "Operation of the Systems Quality Sodium Loop - Interim Report," AI-AEC-12801 (August 1969)
- 16) J. Hopenfeld, "Corrosion of Type 316 Stainless Steel with Surface Heat Flux at 1200°F in Flowing Sodium," AI-AEC-12898 (November 1969)
- 17) J. H. Shively and M. W. Mahoney, "Thermal Gradient Effects on Stress-Rupture Behavior of Thin-Walled Tubing," AI-AEC-12896 (January 1970)
- 18) J. H. Shively, "Biaxial Creep and Stress-Rupture of Wire-Wrapped Cladding in 1200°F Sodium Under ΔT Conditions - Interim Report," AI-AEC-12944 (April 1970)
- 19) D. F. Atkins, "Stress-Rupture Behavior of Types 304 and 316 Stainless Steel Cladding in High Temperature Static Sodium," AI-AEC-12976 (September 1970)

B. QUARTERLY AND ANNUAL PROGRESS REPORTS

<u>Report No.</u>	<u>Reporting Period</u>
NAA-SR-12175	July-Sept. 1966
NAA-SR-12296	Oct. -Dec. 1966
NAA-SR-12395	Jan. -Mar. 1967
NAA-SR-12492	July 1966-June 1967
NAA-SR-12570	July-Sept. 1967
NAA-SR-12638	Oct. -Dec. 1967
AI-AEC-12680	Jan. -Mar. 1968
AI-AEC-12721	July 1967-June 1968
AI-AEC-12744	July-Sept. 1968
AI-AEC-12777	Oct. -Dec. 1968
AI-AEC-12817	Jan. -Mar. 1969
AI-AEC-12859	July 1968-June 1969
AI-AEC-12886	July-Sept. 1969
AI-AEC-12913	Oct. -Dec. 1969
AI-AEC-12948	Jan. -Mar. 1970
AI-AEC-12971	July 1969-June 1970
AI-AEC-12980	July-Sept. 1970
AI-AEC-12987	Oct. -Dec. 1970

C. INTERNAL SUPPORTING DOCUMENTS

- 1) J. E. Bodine, R. L. McKisson, and E. W. Murbach, "System Quality Sodium Description and Specification," NAA-SR-TDR-11859 (April 1966)
- 2) W. L. Cockrell, "Fabrication of 304 Stainless Steel Tubing from Allegheny Ludlum Heat 55697," NAA-SR-TDR-12076 (July 1966)
- 3) S. K. Lee, "Post-Mortem Examination of Hi-Flux Bayonet Heaters," NAA-SR-TDR-12387 (March 1967)
- 4) N. C. Jackson, "Weld Development for High Flux Heater Sheath," NAA-SR-TDR-12461 (May 1967)
- 5) W. L. Cockrell, "The Characterization of AISI 304 Tubing for the LMFBR Cladding Program - Task 10," NAA-SR-TDR-12514 (October 1967)
- 6) T. T. Shimazaki and D. K. Darley, "Cladding Temperature Distribution - Hi-Flux Heater No. 36 Operating at 9.6×10^5 Btu/hr-ft², NAA-SR-TDR-12565 (November 1967)
- 7) J. H. Shively, "The Influence of Cold-Work on Creep-Rupture Life," AI-AEC-TDR-12657 (February 1968)
- 8) M. W. Mahoney and D. F. Atkins, "Characterization of AISI 316 Stainless Steel for the LMFBR Program - Task 10," AI-AEC-TDR-12845 (May 1969)

APPENDIX II
SUMMARY OF 2:1 BIAXIAL STRESS-RUPTURE TESTS

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TABLE II-1

2:1 BIAXIAL STRESS-RUPTURE TESTS OF TYPE 304 STAINLESS STEEL TUBING
(0.275 in. ID by 0.010 in. Wall - 12 Specimens in Each Retort)

Retort No.	Test Temperature (°F)	Cladding Condition*	Test Environment	Life Span (hr)	Raw Data Tabulation	Remarks	Date	
							Start	Completion
1	1300	CW	Na + Zr	100-700	NAA-SR-12353	Scoping Test	2-11-67	3-13-67
2	1400	CW	Na + Zr	100-400	NAA-SR-12353	Scoping Test	2-6-67	2-22-67
3	1400	CW	He	200-800	AI-AEC-12976	Effect of Sodium	3-18-67	4-21-67
4	1200	CW	Na + Zr	1000-2600	This Document	Scoping Test	3-27-67	7-14-67
5	950	CW	Na + Cu	100-3600	AI-AEC-12788	Effect of Copper	4-21-67	9-18-67
6	1400	CW	Na	200-800	AI-AEC-12976	Scoping Test	6-2-67	7-7-67
7	1100	Cw	Na	100-3300	AI-AEC-12976	Scoping Test	6-9-67	10-30-67
8	1400	Ann	Na	200-2000	AI-AEC-12976	Effect of Cold Work	6-15-67	9-12-67
9	1000	CW	Na	50-700	AI-AEC-12976	Scoping Test	8-12-67	9-11-67
10	1400	CW	Na	800-3000	AI-AEC-12976	Extension of Data	8-10-67	12-18-67
11	1200	CW	Na	300-3400	AI-AEC-12976	Extension of Data	8-12-67	1-10-68
12	1100	CW	Na + Cu	100-700	AI-AEC-12788	Effect of Copper	8-18-67	9-18-67
13	1200	CW	He	300-5000	AI-AEC-12976	Effect of Sodium	10-20-67	6-11-68
14	1200	CW + Aged	Na	100-1000	AI-AEC-12976	Effect of Thermal Aging	11-2-67	3-6-68
15	1200	CW	Na	3100-6200	AI-AEC-12976	Extension of Data	11-9-67	7-25-68
16	1000	CW	Na	100-3000	AI-AEC-12976	Extension of Data	11-22-67	3-31-68
17	1200	Ann	Na	2200-25,000	This Document	Extension of Data	2-22-68	12-21-70
18	1200	Ann	Na	100-5000	AI-AEC-12976	Effect of Cold Work	1-25-68	8-18-68
19	1000	CW	Na	3000-10,000	AI-AEC-12976	Extension of Data	4-2-68	5-12-69
20	900	CW + Ann	Na	1200-7100	This Document	Scoping Test	12-1-67	9-22-68
21	1000	Ann	Na	100-2200	AI-AEC-12976	Effect of Cold Work	2-15-68	5-20-68
22	1000	Ann	Na	3000-21,000	This Document	Extension of Data	2-29-68	7-23-70
23	1200	CW + Na Exp.	Na	200-3200	This Document	Effect of Flowing Sodium	8-22-68	1-6-69
24	1300	CW	Na + Cu	100-1200	This Document	Effect of Copper	3-26-68	5-15-68
25	900	Ann	Na	2000-16,000	This Document	Extension of Data	10-8-68	8-10-70
26	900	CW	Na	5000-12,000	This Document	Extension of Data	3-27-69	8-21-70
27	1000	CW	He	100-2800	AI-AEC-12976	Effect of Sodium	3-4-69	6-28-69

*CW = As-received tubing, containing 10 to 15% cold work
Ann = Annealed tubing

TABLE II-2

2:1 BIAXIAL STRESS-RUPTURE TESTS OF TYPE 316 STAINLESS STEEL TUBING
(0.275 in. ID by 0.010 in. Wall - 12 Specimens in Each Retort)

Retort No.	Test Temperature (°F)	Cladding Condition*	Test Environment	Life Span (hr)	Raw Data Tabulation	Remarks	Date	
							Start	Completion
101	1400	CW	Na	100-1000	AI-AEC-12976	Scoping Test	8-10-67	9-21-67
102	1200	CW	Na	100-7100	AI-AEC-12976	Extension of Data	8-12-67	6-6-68
103	1000	CW	Na	100-4900	AI-AEC-12976	Scoping Test	9-14-67	4-5-68
104	1400	CW	He	100-1600	AI-AEC-12976	Effect of Sodium	8-31-67	11-15-67
105	1400	Ann	Na	100-700	AI-AEC-12976	Effect of Cold Work	10-19-67	11-27-67
106	1200	Ann	Na	120-3000	AI-AEC-12976	Effect of Cold Work	10-24-67	1-18-68
107	1000	Ann	Na	100-600	AI-AEC-12976	Effect of Cold Work	12-6-67	1-8-68
108	1000	CW	He	600-2200	AI-AEC-12976	Effect of Sodium	1-12-68	4-9-68
109	1200	CW	Na	100-1800	AI-AEC-12976	Scoping Test	2-9-68	4-25-68
110	1000	CW	Na	2300-25,000	This Document	Extension of Data	4-23-68	
111	1000	Ann	Na	Discontinued	-	Extension of Data	5-10-68	6-4-68
112	900	Ann	Na	100-4000	This Document	Extension of Data	6-20-68	3-12-69
113	900	CW	Na	25,000-	This Document	Extension of Data	8-16-68	
114	1200	CW + Aged	Na	300-3400	AI-AEC-12976	Effect of Thermal Aging	10-29-68	6-21-69
115	1000	CW	He	800-18,000	This Document	Effect of Sodium	10-18-68	11-9-70
116	1000	CW + Aged	Na	2000-12,000	This Document	Effect of Thermal Aging	11-5-68	7-31-70
117	1200	Ann	Na	750-5600	AI-AEC-12976	Extension of Data	12-4-68	8-1-69
119	1000	Ann	Na	700-3000	AI-AEC-12976	Extension of Data	12-11-68	4-14-69
120	900	Ann	Na	10,000-	This Document	Extension of Data	12-30-68	
121	1000	Ann	He	200-4300	This Document	Effect of Sodium	6-5-69	12-5-69
122	1200	CW	Na	200-3000	This Document	Heat-to-Heat Study (Heat 03574)	8-29-69	1-6-70
123	1200	CW	Na	500-2100	This Document	Heat-to-Heat Study (Heat 20303)	8-29-69	12-1-69
126	1200	CW	Na	900-	This Document	Cleaning and Handling Study	8-20-70	10-8-70
127	1000	CW	Na	100-	This Document	Cleaning and Handling Study	10-22-70	
128	1200	CW	Na	Not Tested	-	Cleaning and Handling Study	-	
129	1000	CW	Na	Not Tested	-	Cleaning and Handling Study	-	
130	1200	CW	Na	200-3000	This Document	Lot-to-Lot Study	4-27-70	8-31-70
133	1200	Ann	Na	200-1000	This Document	Heat-to-Heat Study (Heat 03574)	9-15-70	10-27-70
134	1200	Ann	Na	300-2000	This Document	Heat-to-Heat Study (Heat 20303)	9-17-70	12-4-70

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APPENDIX III
TEST DATA GENERATED SINCE LAST REPORT

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TABLE III-1
2:1 BIAXIAL STRESS-RUPTURE DATA FOR ANNEALED TYPE 304
STAINLESS STEEL TUBING
(Heat No. 20013)

Specimen No.	Hoop Stress (psi)	Rupture Life (hr)	Maximum Diametral Strain (%)	Average Diametral Strain Rate (%/hr)
Retort No. 25 - 900° F Sodium				
A4-47	48,000	16,007	8.14	5.08×10^{-4}
-71	48,000	14,605	9.38	6.42×10^{-4}
-61	48,000	15,933	9.48	5.95×10^{-4}
-62	52,000	8,350	13.49	1.61×10^{-3}
-63	52,000	6,922	12.34	1.78×10^{-3}
-64	52,000	6,622	11.64	1.76×10^{-3}
-65	54,000	5,173	†	-
-66	54,000	4,817	13.18	2.74×10^{-3}
-67	54,000	4,842	14.26	2.94×10^{-3}
-69	56,000	2,819	16.38	5.81×10^{-3}
-70	56,000	2,238	†	-
Retort No. 22 - 1000° F Sodium				
A4-43	37,000	16,400	4.93	3.01×10^{-4}
-44	37,000	20,942	5.06	2.41×10^{-4}
-45	37,000	10,462	4.43	4.23×10^{-4}
-48	40,000	4,620	5.05*	1.09×10^{-3}
-49	40,000	7,360	7.03*	9.55×10^{-4}
-51	40,000	7,179	5.52*	7.69×10^{-4}
-53	42,000	5,944	6.28	1.05×10^{-3}
-56	42,000	8,856	6.53	7.37×10^{-4}
-57	42,000	7,066	8.72	1.23×10^{-3}
-58	45,000	2,895	7.03*	2.43×10^{-3}
-59	45,000	2,922	7.96	2.72×10^{-3}
-60	45,000	2,709	6.53*	2.41×10^{-3}
Retort No. 17 - 1200° F Sodium				
A4-1	13,000	24,752	3.70	1.49×10^{-4}
-2	13,000	21,608	3.30	1.53×10^{-4}
-3	13,000	21,896	3.53	1.61×10^{-4}
-4	15,000	16,449	5.55	3.37×10^{-4}
-5	15,000	15,432	5.12	3.32×10^{-4}
-6	15,000	14,296	4.27	2.98×10^{-4}
-7	17,000	9,728	5.28	5.43×10^{-4}
-8	17,000	11,460	7.98	6.96×10^{-4}
-9	17,000	9,768	6.89	7.05×10^{-4}
-10	22,000	3,106	7.90	2.54×10^{-3}
-11	22,000	2,234	4.17	1.87×10^{-3}
-12	22,000	2,984	7.37	2.47×10^{-3}

*Explosive ruptures - strain measured adjacent to ruptures

†Specimens collapsed, due to explosive ruptures

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TABLE III-2
2:1 BIAXIAL STRESS-RUPTURE DATA FOR 10 TO 15% COLD-
WORKED TYPE 304 STAINLESS STEEL TUBING
(Heat No. 20013)
(Sheet 1 of 2)

Specimen No.	Hoop Stress (psi)	Rupture Life (hr)	Maximum Diametral Strain (%)	Average Diametral Strain Rate (%/hr)
Retort No. 20 - 900° F Sodium				
4-196	65,000	5,395	1.82*	3.37×10^{-4}
-197	65,000	5,462	1.75*	3.20×10^{-4}
-198	68,000	3,096	2.22*	7.17×10^{-4}
-199	68,000	3,336	2.02*	6.05×10^{-4}
-200	72,000	1,827	4.03*	2.20×10^{-3}
-201	72,000	2,220	2.93*	1.32×10^{-3}
Retort No. 26 - 900° F Sodium				
4-224	59,000	12,320	1.00*	8.12×10^{-5}
-225	59,000	11,313	0.72	6.36×10^{-5}
-226	59,000	10,856	0.44*	4.05×10^{-5}
-227	62,000	5,985	0.17	2.84×10^{-5}
-228	62,000	8,999	0.92	1.02×10^{-4}
-229	62,000	9,825	1.00*	1.02×10^{-4}
-230	66,000	7,192	1.00*	1.39×10^{-4}
-231	66,000	5,064	0.50	9.87×10^{-5}
-232	66,000	6,743	1.24*	1.84×10^{-4}
-233	70,000	4,740	1.48*	3.12×10^{-4}
-235	70,000	4,916	0.84*	1.71×10^{-4}
Retort No. 23 - 1200° F Sodium (After Exposure 1000 hr in MSAR Loop)				
4X-1	16,000	3,229	3.46	1.07×10^{-3}
-2	16,000	3,159	3.87	1.22×10^{-3}
-3	16,000	2,572	1.65	6.41×10^{-4}
-4	20,000	1,674	2.83	1.69×10^{-3}
-5	20,000	1,809	2.99	1.65×10^{-3}
-6	20,000	1,676	2.76	1.65×10^{-3}
-7	27,000	609	3.10	5.09×10^{-3}
4-220	27,000	1,185	2.35	1.98×10^{-3}
4X-9	27,000	385	1.58	4.10×10^{-3}
-10	31,000	177	†	-
-11	31,000	251	2.52*	1.00×10^{-2}
-12	31,000	201	†	-

TABLE III-2
2:1 BIAXIAL STRESS-RUPTURE DATA FOR 10 to 15% COLD-
WORKED TYPE 304 STAINLESS STEEL TUBING
(Heat No. 20013)
(Sheet 2 of 2)

Specimen No.	Hoop Stress (psi)	Rupture Life (hr)	Maximum Diametral Strain (%)	Average Diametral Strain Rate (%/hr)
Retort No. 4 - 1200° F Zirconium-Gettered Sodium				
4-37	19,000	2,601	1.03	3.96×10^{-4}
-38	19,000	2,593	1.98	7.64×10^{-4}
-39	19,000	2,517	2.18	8.66×10^{-4}
-40	21,000	2,048	1.91	9.33×10^{-4}
-41	21,000	2,024	1.58	7.81×10^{-4}
-42	21,000	2,000	1.34	6.70×10^{-4}
-43	23,000	1,677	1.91	1.14×10^{-3}
-44	23,000	1,200	0.90	7.50×10^{-4}
-45	23,000	1,550	1.84	1.19×10^{-3}
-46	25,000	1,256	-	-
-47	25,000	1,104	2.41	2.18×10^{-3}
-48	25,000	1,030	1.24	1.20×10^{-3}

*Explosive ruptures - strain measured adjacent to rupture

†Specimens collapsed, due to explosive rupture

TABLE III-3
2:1 BIAXIAL STRESS-RUPTURE DATA FOR ANNEALED
TYPE 316 STAINLESS STEEL TUBING

Specimen No.	Hoop Stress (psi)	Rupture Life (hr)	Maximum Diametral Strain (%)	Average Diametral Strain Rate (%/hr)
Retort No. 120 - 900° F Sodium - Heat 65808				
A6-114	44,000	No Rupture	12.0	-
-115	44,000	No Rupture	11.1	-
-116	44,000	No Rupture	11.6	-
-117	48,000	16,668	14.5	8.71×10^{-4}
-118	48,000	14,617	14.8	1.01×10^{-3}
-119	48,000	14,883	15.3	1.03×10^{-3}
-120	50,000	13,586	-	-
-121	50,000	14,268	-	-
-122	50,000	13,442	16.7	1.24×10^{-3}
-123	52,000	10,443	20.7	1.98×10^{-3}
-124	52,000	10,535	-	-
-125	52,000	10,954	19.0	1.74×10^{-3}
Retort No. 133 - 1200° F Sodium - Heat 03574				
AK6-1	27,000	275	18.7	6.80×10^{-2}
-2	27,000	246	20.3	8.25×10^{-2}
-3	27,000	275	20.4	7.42×10^{-2}
-4	25,000	435	22.5	5.17×10^{-2}
-5	25,000	435	23.9	5.49×10^{-2}
-6	25,000	435	22.0	5.06×10^{-2}
-7	23,000	999	21.8	2.18×10^{-2}
-8	23,000	903	23.5	2.60×10^{-2}
-9	23,000	903	24.4	2.70×10^{-2}
-10	22,000	200-hr Creep	2.4	-
-11	22,000	500-hr Creep	4.1	-
-12	22,000	1000-hr Creep	9.3	-
Retort No. 134 - 1200° F Sodium - Heat 20303				
AT6-1	28,000	333	7.21	2.16×10^{-2}
-2	28,000	362	8.04	2.22×10^{-2}
-3	28,000	321	7.47	2.33×10^{-2}
-4	26,000	801	5.87	7.33×10^{-3}
-5	26,000	825	5.97	7.24×10^{-3}
-6	26,000	959	6.74	7.03×10^{-3}
-7	24,000 25,000*	1,859	6.71	3.61×10^{-3}
-8	24,000	No rupture - 1,946 hr	4.61	2.37×10^{-3}
-9	24,000	2,074	6.97	3.36×10^{-3}
-10	21,257	200-hr Creep	1.37	-
-11	21,257	500-hr Creep	1.57	-
-12	21,257	1000-hr Creep	1.83	-

*Stress increased to 25,000 psi after 1,458 hr

TABLE III-4

2:1 BIAXIAL STRESS-RUPTURE DATA FOR 10 TO 15% COLD-
WORKED TYPE 316 STAINLESS STEEL TUBING
(Sheet 1 of 3)

Specimen No.	Hoop Stress (psi)	Rupture Life (hr)	Maximum Diametral Strain (%)	Average Diametral Strain Rate (%/hr)
Retort No. 113 - 900°F Sodium - Heat 65808				
6-95	60,000	23,178	NA	-
-96	60,000	22,546	NA	-
Retort No. 115 - 1000°F Helium - Heat 65808				
6-115	40,000	17,978*	0.44*	2.45×10^{-5} *
-116	40,000	10,281	0.67	6.52×10^{-5}
-117	40,000	15,200	0.60	3.95×10^{-5}
-119	46,000	2,052	0.27	1.31×10^{-4}
-120	46,000	2,517	0.33	1.31×10^{-4}
-121	50,000	1,425	0.33	2.31×10^{-4}
-122	50,000	1,485	0.30	2.02×10^{-4}
-123	50,000	777	0.17	2.19×10^{-4}
-124	56,000	767	0.67	8.73×10^{-4}
-125	56,000	825	0.64	7.76×10^{-4}
-126	56,000	811	0.64	7.89×10^{-4}
Retort No. 116 (Sodium aged for 3000 hr at 1000°F) - 1000°F Sodium - Heat 65808				
6-127	40,000	12,181	0.74	6.07×10^{-5}
-128	40,000	7,005	0.40	5.71×10^{-5}
-129	40,000	10,618	0.70	6.59×10^{-5}
-130	42,000	4,786	0.47	9.82×10^{-5}
-131	42,000	4,340	0.40	9.22×10^{-5}
-132	42,000	3,970	0.34	8.56×10^{-5}
-133	44,000	3,908	0.64	1.64×10^{-4}
-134	44,000	3,876	0.57	1.47×10^{-4}
-135	44,000	3,297	0.40	1.02×10^{-4}
-136	48,000	1,930	0.54	2.80×10^{-4}
-137	48,000	2,476	0.40	1.61×10^{-4}
-138	48,000	2,578	0.40	1.55×10^{-4}
Retort No. 127 - Cleaning Studies (Aged 2000 hr in 1000°F Sodium) - 1000°F Sodium				
6-165	46,000	2,795		
-166	46,000	817		
-167	46,000	3,202		
-168	46,000	2,360		
-169	46,000	275		
-170	46,000	1,994		
-171	46,000	1,896		
-172	46,000	2,453		
-173	46,000	1,281		
-174	46,000	151		
-175	46,000	2,604		
-176	46,000	1,151		

TABLE III-4
2:1 BIAXIAL STRESS-RUPTURE DATA FOR 10 TO 15% COLD-
WORKED TYPE 316 STAINLESS STEEL TUBING
(Sheet 2 of 3)

Specimen No.	Hoop Stress (psi)	Rupture Life (hr)	Maximum Diametral Strain (%)	Average Diametral Strain Rate (%/hr)
Retort No. 110 - 1000° F Sodium - Heat 65808				
6-80	38,000	15,561	0.50	3.21×10^{-5}
-81	38,000	No Rupture	0.84	-
-82	38,000	No Rupture	0.84	-
-83	40,000	19,424	0.84	4.32×10^{-5}
-84	40,000	12,648	0.67	5.30×10^{-5}
-85	40,000	10,745	0.60	5.58×10^{-5}
-63	41,000	6,289	0.44	6.99×10^{-5}
-64	41,000	9,022	0.64	7.09×10^{-5}
-65	41,000	5,049	0.44	8.71×10^{-5}
-66	46,000	2,585	0.40	1.55×10^{-4}
-67	46,000	2,270	0.39	1.63×10^{-4}
-68	46,000	3,177	0.50	1.57×10^{-4}
Retort No. 130 - 1200° F Sodium - Heat 65808 - Lot 2				
62-1	25,000	2,216	2.61	1.18×10^{-3}
-2	25,000	3,024	3.71	1.22×10^{-3}
-3	25,000	2,648	6.21	2.34×10^{-3}
-4	28,000	919	7.13	7.76×10^{-3}
-5	28,000	907	10.73	1.18×10^{-2}
-6	28,000	796	4.38	5.50×10^{-3}
-7	30,000	445	5.79	1.30×10^{-2}
-8	30,000	445	6.21	1.39×10^{-2}
-9	30,000	445	5.88	1.32×10^{-2}
-10	32,000	258	4.88	1.89×10^{-2}
-11	32,000	248	4.46	1.80×10^{-2}
-12	32,000	225	3.24	1.44×10^{-2}
Retort No. 126 - Cleaning Studies - (Aged 2000 hr in 1200° F Sodium) - 1200° F Sodium				
6-153	28,000	895	NA	-
-154	28,000	731	NA	-
-155	28,000	895	NA	-
-156	28,000	849	NA	-
-157	28,000	895	NA	-
-158	28,000	897	NA	-
-159	28,000	936	NA	-
-160	28,000	899	NA	-
-161	28,000	899	NA	-
-162	28,000	897	NA	-
-163	28,000	899	NA	-
-164	28,000	873	NA	-

TABLE III-4
2:1 BIAXIAL STRESS-RUPTURE DATA FOR 10 TO 15% COLD-
WORKED TYPE 316 STAINLESS STEEL TUBING
(Sheet 3 of 3)

Specimen No.	Hoop Stress (psi)	Rupture Life (hr)	Maximum Diametral Strain (%)	Average Diametral Strain Rate (%/hr)
Retort No. 122 - 1200° F Sodium - Heat 03574				
K6-1	27,000	3,080	5.47	1.77×10^{-3}
-2	27,000	2,997	3.39	1.13×10^{-3}
-3	27,000	3,069	4.73	1.54×10^{-3}
-4	30,000	1,187	7.42	6.25×10^{-3}
-5	30,000	1,051	6.58	6.26×10^{-3}
-6	30,000	1,329	6.98	5.25×10^{-3}
-7	32,000	559	11.45	2.05×10^{-2}
-8	32,000	601	9.77	1.62×10^{-2}
-9	32,000	635	7.32	1.15×10^{-2}
-10	35,000	215	6.58 [†]	3.06×10^{-2}
-11	35,000	242	7.08 [†]	2.92×10^{-2}
-12	35,000	233	9.16	3.93×10^{-2}
Retort No. 123 - 1200° F Sodium - Heat 20303				
T6-1	27,000	2,158	1.64	7.60×10^{-4}
-2	27,000	2,119	1.43	6.75×10^{-4}
-3	27,000	2,119	1.47	6.94×10^{-4}
-4	30,000	1,531	1.90	1.24×10^{-3}
-5	30,000	1,484	1.57	1.06×10^{-3}
-6	30,000	1,484	1.74	1.17×10^{-3}
-7	32,000	1,248	1.84	1.47×10^{-3}
-8	32,000	1,230	1.94	1.58×10^{-3}
-9	32,000	1,202	2.24	1.86×10^{-3}
-10	35,000	720	2.07	2.87×10^{-3}
-11	35,000	549	1.37	2.49×10^{-3}
-12	35,000	776	2.24	2.86×10^{-3}

NA: Data not available - test still in progress

*Test terminated before rupture

†Explosive Ruptures - strain measured adjacent to ruptures

TABLE III-5
 ROTATING-BEAM FATIGUE DATA IN 1200°F AIR ON 10 to 15%
 COLD-WORKED TYPE 316 STAINLESS STEEL TUBING
 (0.010 in. Wall, Heat No. 65808)

Strain (%)	Stress Maximum Fiber (ksi)	Cycles to Failure (N _F)
1.00	56.13	581
1.00	56.13	676
1.00	56.13	598
1.00*	59.14*	1,763*
0.80	55.27	917
0.80	55.27	1,173
0.60	53.22	1,975
0.585	53.01	996
0.58*	56.45*	2,386*
0.50	51.61	4,317
0.50	51.61	2,876
0.35	47.31	3,845
0.35	47.31	6,279
0.35	47.31	5,404
0.32*	48.92*	12,211
0.27	43.00	35,698
0.27	43.00	27,204
0.27	43.00	12,384
0.21	37.10	203,792

*Case-carburized specimens