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STAINLESS STEEL FAILURE INVESTIGATION PROGRAM

THIRD QUARTERLY PROGRESS REPORT OCTOBER 1, 1965 - DECEMBER 31, 1965

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ATOMIC POWER EQUIPMENT DEPARTMENT

GENERAL ELECTRIC

SAN JOSE, CALIFORNIA

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Third Quarterly Progress Report
October - December, 1965

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Contract AT(04-3)-189
Project Agreement 45

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FOREWORD

The United States and the European Atomic Energy Community (EURATOM), on May 27, 1958, and June 18, 1958, signed an agreement which provides a basis for cooperation in programs for the advancement of peaceful applications of atomic energy. This agreement, in part, provides for the establishment of a Joint US-EURATOM research and development program which is aimed at reactors to be constructed in Europe under the Joint Program.

The work described in this report represents the Joint US-EURATOM effort which is in keeping with the spirit of cooperation in contributing to the common good by the sharing of scientific and technical information and minimizing the duplication of effort by the limited pool of technical talent available in western Europe and United States.

SECTION I

INTRODUCTION

A research and development program was initiated on February 15, 1965, under Project Agreement 45 of Contract AT(04-3)-189 to investigate the cause of failure of stainless steel cladding on boiling water reactor fuel. The program consists of the investigation of the effects of material composition, coolant environment, irradiation damage, and operating stresses on clad cracking.

This is the third quarterly report for this program and covers the period October 1 through December 31, 1965.

SECTION II

SUMMARY

The significant accomplishments during this reporting period are as follows:

2.1 ADMINISTRATIVE

A portable autoclave system has been assembled to corrosion test irradiated Type-304 stainless steel samples in iron chloride solutions.

2.2 TECHNICAL

Corrosion tests on Type-304 stainless steel in $\text{HNO}_3\text{-Cr}^{+6}$ to determine the effect of high gamma radiation fields ($\sim 1,000$ R) indicate that the presence of high energy gamma radiation does not increase the dissolution rate of the stainless steel.

Results of 24 hours corrosion tests obtained previously on irradiated and unirradiated Type-304 stainless steel in boiling $\text{HNO}_3 + \text{Cr}^{+6}$ are valid and indicate that irradiation exposure increases susceptibility to corrosion. The weight loss of irradiated cladding samples was consistently higher than that of unirradiated control specimens by a factor of two.

Micro-thermoelectric measurements across grain boundaries in non-irradiated and irradiated stainless steel have been completed and indicate no significant difference between the thermoelectric properties of grains and grain boundaries.

A portable autoclave system has been assembled and tested in preparation for corrosion tests on irradiated Type-304 stainless steel samples in 650°F , 0.1 g/l iron chloride solutions. The various irradiated samples for these tests have been prepared.

Electrochemical studies at elevated temperatures, up to 275°C (527°F), have been initiated. Potentiostatic and intensiostatic curves have been obtained for Type-304 stainless steel samples in deionized water and in dilute iron chloride solutions. Variations due to scan speed, temperature and different electrolytes were observed.

SECTION III

TASK I - CLADDING MATERIAL INVESTIGATION

3.1 SUBTASK A - ALLOY PREPARATION (J. S. Armijo)

3.1.1 Objective of Task

Prepare high purity Fe-Cr-Ni alloys with controlled amounts of C, N₂, Si, Mn, S, P, and O₂ for corrosion testing in solutions known to cause intergranular attack.

3.1.2 Results During Period

The initial high purity 14 percent Cr - 14 percent Ni alloy was not received from Battelle Memorial Institute and is expected early in January, 1966.

Controlled impurities addition experiments on high purity alloys have been initiated. Techniques were developed to obtain high purity alloys with known amounts of impurities by a double melting procedure. This procedure consists of initial vacuum induction melting for 30 minutes in high purity alumina or magnesia crucibles followed by button melting in an electric arc furnace under inert atmosphere. The initial melt produces a homogeneous alloy, but leaves a large shrinkage cavity. The second melt consolidates the material into a sound ingot. After melting, alloys are hot rolled to the desired thickness. Upon delivery of the 14 percent Cr - 14 percent Ni alloy controlled amounts of Si and carbon will be added. Controlled additions of N₂, O₂, Mn, S and P will follow.

3.2 SUBTASK B - CORROSION TESTS (J. S. Armijo, A. E. Pickett)

3.2.1 Objective of Task

Perform corrosion tests of high purity alloys containing various impurities to determine what solutes in stainless steel promote intergranular corrosion.

3.2.2 Results During this Period

As reported previously, (1,2) irradiated Type-304 stainless steels corrode approximately twice as fast as nonirradiated control specimens in HNO₃-Cr⁺⁶ solutions. This effect was observed for five different fuel claddings and their respective controls. These data could mean that neutron damage to the metal increases its solubility in the corrosive solution. There was, however, doubt in this interpretation of the results because of the intense gamma radiation field in the hot cell where the experiment was performed. This gamma field (10-1000 Roentgens/h) could have changed the oxidizing properties of the acid and thus increased the subsequent corrosion rate.

The purpose of this investigation was to determine whether the accelerated corrosion of irradiated cladding was the result of radiation damage to the metal or of changes in the corrosive properties of the solution.

The following experiment was performed to determine the effects of gamma radiation on the intergranular corrosion of Type-304 stainless steel in $\text{HNO}_3\text{-Cr}^{+6}$ solutions. Because of size limitations of an available high intensity gamma source, it was necessary to design a special corrosion test cell. This cell, as shown in Figure 1, consisted of a 28 mm standard taper Vycor tube and stopper. Small holes were drilled through the top joint. A Pt/Pt-Rh thermocouple was fed through these holes and joined to insulated copper wires at a terminal strip. A second thermocouple (chromel-alumel) was attached at the terminal strip to measure the cold junction temperature. This was necessary because of possible gamma heating of the cold junction. After filling the Vycor capsule with 5N HNO_3 + 4 gm Cr^{+6} /liter solution, the external resistance heater was adjusted until the temperature had stabilized at $90^\circ\text{C} \pm 2^\circ\text{C}$. The specimen was then dropped into the test solution and exposed for the desired time. Solutions were changed after every 2 to 3 hours of use. Initial experiments were performed without gamma radiation until the corrosion rate had reached a rate of $\sim 1.1 \text{ mg/cm}^2/\text{h}$. The assembly (including the same specimen) was then lowered into a cobalt-60 source, and testing was continued under a gamma flux of ~ 1000 Roentgens per hour. As shown in Figure 2, the weight loss rate under gamma radiation was unchanged for the first four hours of the test. For the next 10 hours, however, the weight loss rate decreased to $\sim 0.71 \text{ mg/cm}^2/\text{h}$. The assembly was then removed from the gamma source and the corrosion test was continued for six hours. The corrosion rate during this period was $\sim 1.4 \text{ mg/cm}^2/\text{h}$.

Initial high weight loss data points observed during corrosion under gamma radiation are probably the result of grain dropping of grains which had been undermined by previous corrosion without a gamma field.

These results show that the effect of gamma radiation is to reduce the corrosive properties of $\text{HNO}_3\text{-Cr}^{+6}$ solutions. Consequently, the accelerated attack of irradiated stainless steel in $\text{HNO}_3\text{-Cr}^{+6}$ solutions is the result of radiation damage to the metal.

3.3 SUBTASK C - GRAIN BOUNDARY STUDIES (J. S. Armijo)

3.3.1 Objective of Task

Develop a correlation between susceptibility to intergranular cracking and differences in electrochemical, thermoelectric and microhardness between grains and grain boundaries.

3.3.2 Results During this Period

Micro-thermoelectric measurements across grain boundaries in nonirradiated and irradiated stainless steel have been completed. Measurements were made with the device described previously.⁽²⁾ Initial measurements were made on a specimen of annealed (1300°C - 4 h, water quenched) Type-304 stainless steel at a hot junction temperature of 83.5°C . Three different loads (2, 5, and 10 gms) were used for traverses across the same grain boundary. Results of these traverses presented in Figure 3 show that there is no significant difference between the thermoelectric properties of grains and grain boundaries within the limits of the present technique. These data also show that the measurements were independent of load.

Similar measurements were made across a grain boundary in irradiated HPD cladding material. These results were the same as those on nonirradiated material, i.e. no detectable difference in thermal emf between grain center and grain boundary. The lower absolute value of the thermal emf of the

Figure 1. Schematic of Assembly Used for Corrosion Tests in High Intensity Gamma Fields

Figure 2. Weight Loss of Annealed Type-304 Stainless Steel as a Function of Time; With and Without Gamma Radiation

Figure 3. Thermal EMF Traverses Across Grain Boundaries in Annealed Stainless Steel Fuel Cladding (EMF values measured with respect to Tungsten)

irradiated material was the result of a different method of electrical connection. The nonirradiated specimen was spot-welded to a stainless steel wire which was connected to the microvoltmeter. The irradiated specimen, however, was soldered to an aluminum block, which in turn was connected to a stainless steel wire.

In view of these negative results, no further experiments are planned with this device.

3.4 SUBTASKS D, E AND F - EXAMINATION OF IRRADIATED CLADDING

(R. N. Duncan, D. L. Zimmerman)

3.4.1 Objective of Task

Examine irradiated and unirradiated stainless steel cladding to determine changes in susceptibility to intergranular cracking as a result of irradiation.

3.4.2 Results During this Period

A variety of fuel rods that had been irradiated in the VBWR as part of the AEC Fuel Cycle and High Power Density Programs are being examined in the Stainless Steel Failure Investigation Program. The performance history of the fuel rods in the VBWR was described previously in Fuel Cycle⁽³⁾ and High Power Density⁽⁴⁾ Program reports. These rods had stainless steel fuel cladding with variations in level of cold work, cladding thickness, and UO₂-to-cladding gap. Average fuel rod burnups ranged from 0.8×10^{20} to 2.75×10^{20} fissions/cc (3,000 to 10,000 MWD/TU).

Fourteen fuel rods were sectioned to obtain specimens of the cladding for the various tests. A description of the rods from which specimens were obtained for the various tests are summarized in Table I. Results of various examinations and tests have been reported previously (1, 2).

3.4.3 Corrosion Tests

Corrosion tests of unirradiated and irradiated cladding in 650°F iron chloride solution will be performed during the next period. Metallographic examination of selected specimens after corrosion testing will be used to verify that the mode of attack was intergranular.

Preparation of the irradiated samples and the unirradiated controls is complete. The test samples are 1/4 inch long ring sections of stainless steel tubing from which the UO₂ fuel had been dissolved with HNO₃. Samples of irradiated Type-304 stainless steel cladding were selected from the following nonfailed fuel rods:

1 F-H	Cold worked
5L-209	Co-extruded (Rod formed by hot co-extrusion of UO ₂ in stainless steel)
5S-S	Sensitized structure - Annealed after swaging
8I-240F(16)	Cold worked
8H-423F(10)	Commercially annealed

TABLE I

IRRADIATED VBWR FUEL RODS USED
TO OBTAIN SAMPLES FOR VARIOUS TESTS

Designation	Description of Rod	O.D. of Rod, in.	Wall Thickness in.	Avg Exposure MWD/TU
Fuel Cycle 12L-C4	Cold Worked, 750 ppm borated cladding	0.485	0.011	2,972
8H-3	Failed fuel follower annealed	0.420	0.020	7,770
8H-10	Nonfailed fuel follower, annealed cladding	0.420	0.020	7,770
8H-12	Nonfailed fuel follower, annealed cladding	0.420	0.020	7,770
8I-13	Nonfailed, cold worked cladding	0.410	0.015	9,183
8I-16	Nonfailed, cold worked cladding	0.410	0.015	9,183
9I-1	Failed, cold worked cladding	0.410	0.015	8,252
16I-1	Nonfailed, cold worked cladding	0.410	0.015	8,042
5L-209	Nonfailed, coextruded UO ₂ and stainless steel	0.400	0.016	7,030
High Power Density (HPD)				
6S-F	Nonfailed, annealed thin walled cladding	0.400	0.010	5,500
6S-T	Nonfailed, annealed thin walled cladding	0.400	0.008	5,500
12S-D	Nonfailed, annealed cladding	0.363	0.014	8,850
12S-F	Failed, annealed cladding	0.363	0.014	9,120
5S-S	Nonfailed, thin walled cladding, annealed after cold swaging (sensitized)	0.400	0.010	4,600
1F-H	Nonfailed, cold worked cladding, portion of rod out of core	0.363	0.014	10,000
1F-U	Same as 1F-H	0.363	0.014	10,000

SECTION IV

TASK II - COOLANT ENVIRONMENTAL EFFECTS

4.1 SUBTASK A - ANALYSIS OF CRUD OR SURFACE SCALE FROM FAILED STAINLESS STEEL CLAD FUEL ROD (R. N. Duncan)

4.1.1 Objective of Task

Determine the composition of crud or surface scale on the surface of a stainless steel clad fuel element that has failed during service in an operating power reactor.

4.1.2 Results During this Period

Analytical methods are being reviewed for trace analysis of chlorides or fluorides in small samples of the crud and surface scale which adheres to the irradiated fuel rods.

4.2 SUBTASK B - EFFECTS OF COOLANT AND COOLANT IMPURITIES (W. L. Pearl)

4.2.1 Objective of Task

Determine the effects of coolant environment and variations in coolant impurities on the susceptibility of stainless steels to intergranular corrosion.

4.2.2 Results During this Period

4.2.2.1 Electrochemical Studies: (W. R. DeHollander)

In this task the effects of the environment on the corrosion of Type-304 stainless steel are being studied by means of potentiostatic or intensiostatic methods. In potentiostatic curves, the potential of the sample is forced linearly between certain values, while the current which flows between the sample and a working electrode fluctuates as needed. In intensiostatic curves, the current is forced linearly from one level to another and the potential fluctuates as required. Both types of curves yield comparable information and each has its merits for interpretation of the dynamic phenomena occurring at the electrode. During the period the first good potentiostatic curve was obtained. This was a total hysteresis loop from E_C , the open circuit potential anodically to incipient oxygen evolution, then back to the cathodic region (hydrogen evolution), and then back to the oxygen evolution regime.

When interpreting the curves presented later, the following criteria are used:

- a. E_C is the open circuit potential of the sample measured against shiny platinum.
- b. A negative potential is called cathodic

- c. A positive potential is anodic.
- d. A positive current is anodic.
- e. A negative current is cathodic.
- f. Currents are in milliamps unless stated otherwise.
- g. Potentials are in volts as referred to shiny platinum.
- h. All samples are 3-inches long $\times$ 0.424-inch $\times$ 0.019-inch open cylinder.
- i. The working electrode is a large platinum gauze electrode rolled into a tight cylinder about 1/2-inch diameter and spaced about 1-1/2-inches from the sample.
- j. A 3/8-inch thick Al_2O_3 cylinder is used to line the autoclave to help prevent shorts and leakage paths.
- k. A thermocouple well is located in the liquid phase in the autoclave.
- l. Autoclave is static, not flowing, and is blown down while coming up to temperature to remove dissolved gases.
- m. The sample is abraded with 4/0 paper and rinsed with distilled water prior to insertion.

During the reporting period experiments were run to determine the effects of temperature, scan rate, time in solution, reproducibility of scan, and the use of ferrous chloride (0.1 g per liter) as the environment. This latter environment has been shown to be capable of intergranularly cracking cold worked stainless steel.⁽²⁾

The following items have been varied:

- a. Scan speed. 5 to 100 mV/min have been tried as well as a few very fast non-linear scans.
- b. Temperature. Temperatures of 25°C, 205°C, 230°C, 250°C, 255°C, 274°C, 280°C have been used.
- c. Intensiostatic and potentiostatic curves were obtained.
- d. Distilled water and 0.1 g/liter FeCl_2 have been used.
- e. Time of immersion before first scan has varied from about 2-3 hours up to 48 hours.
- f. Time between scans has varied from minutes to approximately 4 days.

The various effects which have been observed for anodic, cathodic and anodic retraces of voltage versus current curves are shown in Figures 4-9.

Figure 4. Potentiostatic Curve: Milliamps versus Volts with Scan Speed Varied

Figure 5. Potentiostatic Curve: Milliamps versus Volts with 205°C Temperature Water

Figure 6. Potentiostatic Curve: Milliamps versus Volts 250°C Temperature Water

Figure 7. Potentiostatic Curve: Milliamps versus Volts 274°C Temperature Water

Figure 8. Potentiostatic Curve: Milliamps versus Volts 275°C Temperature 0.1 g/l FeCl_2 Solution

Figure 9. Intensiostatic Curve: Milliamps versus Volts 205°C Temperature Water

Some of the effects of scan speed are to be seen in Figure 4. Faster scans result in higher currents and move potentials up on the scale. Some phenomena are emphasized by speed change.

Temperature effects can be seen by comparing Figures 5, 6, and 7. In general, the film resistance goes down as the temperature rises similar to any semiconductor and, in a way, supporting the hypothesis that the film behavior is best interpreted in terms of semiconductor, ferro-electric type behavior. The current is lowest at the lowest temperature, highest at the highest, and midway at the intermediate temperature. Also the peak at about -0.3 volt is absent from the high temperature curve. The shape of the curve is quite similar otherwise.

By comparing Figure 7 (distilled water) with Figure 8 (0.1 g/l FeCl_2), the effects of FeCl_2 additions can be seen. The most obvious effects of FeCl_2 additions are:

- a. The E_C is raised from negative values to positive.
- b. There is a noticeably smaller loop to the cathodic return leg, i.e., Position H through O on Figure 8.
- c. Considerably less current flows at any given potential when FeCl_2 is present.
- d. There are additional peaks present in FeCl_2 solution.
- e. The M, N, O position of Figure 8 is absent in Figure 7 at comparable temperatures.

Figure 9 is the only intensiostatic curve included at this time. This curve shows the same phenomena as potentiostatic curves. Interesting areas are regions B, C, D or P, Q, D. Here it is postulated that oxygen bubble nucleation is the slow process causing the rise in potential as the current is forced to higher values. Once the bubbles have nucleated and are free to detach, the potential drops to a new level. On the return trace, only a slight bump at points H and G is seen which might indicate the final last collapse of the bubbles as they are caused to go backwards under the reduced current flow. Not until the last bubble dissolves, does the potential drop, and the current reverses direction and must work to overcome the polarization remanence of the film.

It is not immediately obvious whether or not the presence of stress corrosion cracking or intergranular attack can be predicted by the curves shown. The work utilizing ferrous chloride is going to be repeated:

4.2.2.2 Autoclave Tests -- (A. E. Pickett, W. B. Nelson)

A portable low flow autoclave system was assembled for testing of irradiated cladding specimens described under Task I (D, E, and F) in 650°F iron chloride solution. The self-contained system, shown in Figure 10, will be moved to the Radioactive Materials Laboratory for testing of the irradiated specimens. The system consists of a high pressure autoclave protected from the test solution by an inner capsule of Inconel-600. The test solution is circulated through the test capsule at a rate of ~15 cc/min by a high pressure pump, the pressure being regulated by a back-pressure regulator pressurized by saturated steam maintained in the annulus between the autoclave vessel and the Inconel-600 test capsule, shown in Figure 11. During startup and shutdown, deionized water is maintained in the system to prevent transgranular chloride

Figure 10. Portable Low Flow Autoclave System

Figure 11. Schematic Diagram of Autoclave System

stress corrosion from occurring at lower temperatures. The irradiated cladding will be exposed as 1/4-inch wide rings stressed slightly over yield by being flattened and held in the form of an ellipse during the test.

The portable facility is initially being used to study the effects of environmental variables on mode of attack. Sensitized and unsensitized specimens of Type-304 stainless steel are being exposed in 650°F dilute solutions modified to separate the variables of pH, chloride ion, ferric-ferrous ion and oxygen concentrations.

A prototype spring-loaded specimen fixture was completed. This device, shown in Figure 12 view a, will be used to study the effects of stress and to accurately determine times to failure without discontinuing a test. A known stress can be applied to a narrow strip or wire specimen by tightening the Inconel-600 spring, precalibrated at the test temperature, to a given deflection. The specimen is electrically insulated from the fixture by ceramic inserts to prevent galvanic effects. Failure of a specimen is indicated by change in resistance of a high temperature strain gage enclosed in a force indicator to which the specimen is attached, Figure 12 view b.

The larger autoclave test facility has been designed to accommodate eight spring loaded fixtures. The electrical leads are brought through high pressure fittings in the autoclave head to a multichannel strain gage recorder for failure readout. During initial testing the device satisfactorily indicated failure of a Type-304 stainless steel specimen in the iron chloride test. Only slight modification in the method of specimen attachment will be required.

Testing of 64 steel specimens from the French steel firm Cie des Ateliers et Forges de la Loire (C.A.F.L.) was initiated in the large autoclave facility in 650°F, 0.1 g/l ferrous chloride solution. The alloys being exposed and their compositions are shown in Table II.

The alloys are exposed as 3/8-inch bent-beam specimens. Two typical specimen holders are shown in Figure 13. Stress is applied by tightening the screw to deflect one end of the specimen a calculated amount.

For each alloy, four levels of cold work are being tested; as-annealed at 1150°C (air cooled), and cold reduced 10, 20, and 40 percent by cold rolling. A machined and an electropolished specimen is being tested at each level of cold work at a calculated stress of 60,000 lb/in.². A duplicate set of the cold worked specimens is being tested at a calculated stress of 120 percent of the yield strength as estimated by Rockwell hardness. Duplicate annealed specimens are being tested at stress levels equal to the yield strength. The applied stresses during testing for these specimens is summarized in Table III.

Type-304 stainless steel bent beam control specimens are also being exposed under the same conditions of heat-treatment, cold work, and stress as are Type-304 stainless steel tubing control specimens stressed by flattening ring samples.

One three-day run was completed during the report period. No intergranular cracks were observed on the bent-beam specimens. The stressed Type-304 tubing specimen showed complete intergranular penetration of the tube wall. Previous testing has shown stressed tubing specimens to fail in several hours as compared to several days for bent beam strip specimens.

TABLE II

COMPOSITION OF CAFL ALLOYS

Alloy	Specimen Code	Composition, %						
		C	Mn	Si	Cr	Ni	Mo	Cu
Krupp VII A	VII A	0.028	0.96	0.50	17.3	11.2	---	---
Uranus-S	URS	0.014	0.98	3.56	18.5	14.2	---	---
Uranus-50	50	0.021	0.79	0.58	20.4	7.95	2.9	1.5
Uranus-50Si	50-Si	0.039	1.22	3.35	18.8	10.1	2.6	1.5

TABLE III

APPLIED STRESSES DURING TESTING OF CAFL ALLOYS IN 650°F,
0.1 g/l FERROUS CHLORIDE

Specimen Code	Rockwell Hardness	Estimated Yield Strength, lb/in ²	Applied Stress, lb/in ²
VII A-A*	R _B 65	30,000	30,000
VII A-B*	R _B 95	60,000	72,000
VII A-C*	R _C 21	70,000	84,000
VII A-D*	R _C 34	130,000	156,000
URS-A	R _B 73	30,000	30,000
URS-B	R _B 97	70,000	84,000
URS-C	R _C 26	80,000	96,000
URS-D	R _C 35	130,000	156,000
50-A	R _B 89	40,000	40,000
50-B	R _B 26	80,000	96,000
50-C	R _C 32	100,000	120,000
50-D	R _C 35	130,000	156,000
50-Si-A	R _B 97	70,000	84,000
50-Si-B	R _C 33	110,000	132,000
50-Si-C	R _C 35	130,000	156,000
50-Si-D	R _C 40	150,000	180,000

* A - As annealed, B - 10% cold reduced, C - 20% cold reduced, D - 40% cold reduced

Figure 12. Spring Loaded Corrosion Test Specimen Fixture

Figure 13. Typical Bent-Beam Specimen Holder for CAFL Materials

4.3 SUBTASK C - HYDROGEN EMBRITTLEMENT TESTS - E. G. Brush

4.3.1 Objective of Task

Determine if normally ductile, face-centered cubic alloys can be made to fail reproducibly in a brittle manner by hydrogenation.

4.3.2 Results During this Period

The cathodic charging tests results since the last reporting period⁽²⁾ are listed in Table IV.

The results indicate that time to fracture is roughly proportional to the square root of the ratio of the charging current densities, as evident from tests No. 1-3, 2-2 and 2-3. The appearance of the fracture area in the single side charging experiments tend to support the hypothesis that internal hydrogen pressure produces failure, rather than surface energy decreases due to chemisorbed hydrogen. The evidence lies in the fact that all single sidedly charged specimens bowed in the fracture area, with the charging surface being convex, Figure 14.

The test results related to a) the rate of load application, b) the microstructure of hydrogen damage, and c) the kinetics of the charging mechanism are described below.

a. Loading Rate

The room temperature absorption of hydrogen by an unrestrained face-centered-cubic lattice is an extremely slow process⁽⁵⁾. It has been postulated that defects accompanying plastic strain,⁽⁶⁾ particularly dislocation pipes, will markedly increase the rate of hydrogen movement. This postulate is apparently supported from the comparison of failure times in tests No. 1-3, 2-1, 1-6 and 3-1, in which long initial exposure to hydrogen charging at low loads, Test 1-6 did not produce sufficient hydrogen absorption so as to cause failure within a few hours after gradual raising the load to 75,000 psi, and immediate loading to 75,000 psi, Test 2-1 takes longer to produce failure than Tests 1-3 and 3-1 which were subjected to gradual loading. This latter result may be the result of dislocation blocking by nitrogen normally contained in the austenitic stainless steels and adjacent to the dislocation fields produced by 16 percent plastic straining (caused by the 75,000 psi load).

These results suggest that the proper hydrogenation technique in delayed fracture tests of the austenitic stainless steels is to program the load application (1 to 10 psi increase per minute).

b. Microstructure

It was previously reported that the damage due to absorbed hydrogen as predominantly intergranular, and that the slip planes near the intergranular failures might be highly decorated with hydrogen.⁽²⁾ Examination of additional fractured specimens indicates that the damage is a mixture of transgranular and intergranular cracks, and that the slip planes are indeed habitats of high hydrogen content. They are often cracked, as shown in Figure 15, photo a, and etch rapidly as shown in Figure 15, photo b. It is apparent from Figure 15, photo b, that the damage produced by room temperature hydrogen absorption is not similar to high temperature stress corrosion cracks.

TABLE IV

HYDROGENATION OF ANNEALED TYPE-304
STAINLESS STEEL, CATHODICALLY CHARGED AT
ROOM TEMPERATURE IN 4% H₂SO₄

Test No.	Promoter	Current density, 2 amp/cm	Cell Voltage, Volts	Loading Cycle
1-3 (1)	As ₂ O ₃ 0.25 g/l	1.0	3.4	50,000 psi for 113 hours 65,000 psi for 101.3 hours 75,000 psi for 25.6 hours and then fracture. Total immersion time 239.9 hours
2-1 (1)	As ₂ O ₅ 0.25 g/l	5	6.1	75,000 psi for 341 hours and then fracture. Total immersion time 341 hours.
2-2 (1)	As ₂ O ₃ 2.5 g/l	5	6.4	Load increase from 30,000 psi to 75,000 in 10,000 psi increments about every 24 hours, except last increment was 5,000 psi. Took 118.1 hours to reach 75,000 psi. Fracture after 2.6 hours at 75,000 psi. Total exposure 120.7 hours
2-3 (1)	As ₂ O ₃ 2.5 g/l	5	6.5	Duplicate of 2-2. Fracture after 0.5 hours at 75,000 psi. Total exposure 119.2 hours
2-4 (2)	As ₂ O ₅ 2.5 g/l	2.5	5.7	Raise to 60,000 psi from 25,000 psi in 5000 psi increments over a 195.2 hour period. Still remaining at 60,000 psi after 1,220 hours
1-6 (2)	As ₂ O ₅ 2.5 g/l	2.5	5.2	Raise to 45,000 psi in 2,500 increments over a period of 245.7 hours. Ran at 45,000 psi for 259.4 hours. After a total exposure of 505.1 hours raised in 10,000 psi increments to 75,000 psi. 75,000 psi reached in 552.8 hours. Ran until 717.9 hours when a power failure occurred. Repaired after 7 hours of no impressed current. Ran on at 75,000 psi until failure at 829.8 hours.
3-1 (2)	As ₂ O ₅ 2.5 g/l	2.5	5.6	Raise to 75,000 psi in 10,000 psi increments from 25,000 psi. At 75,000 psi after 114.3 hours. Fractured after 139 hours at 75,000 psi. Total immersion time 253.3 hours.
3-2 (2)	As ₂ O ₅ 2.5 g/l	5	7	Raised to 40,000 psi from 25,000 psi in increments of 2,500 psi at 40,000 after 163.5 hours. Then scheduled to raise 5,000 psi every 24 hours until 75,000 psi is reached. Currently at 55,000 psi after total time of 259.3 hours - still running.
1-7 (2)	As ₂ O ₃ 2.5 g/l	5	7.8	Same as 3-2

(1) Single side charging into 0.001 inch notch.

(2) Double side charging with no notch.

Figure 14. Vertical View of Hydrogenated Fracture Surface Test 2-2.
Specimen cross section: 0.010×0.125 inch

photo a

500×

photo b

Etched

500×

Figure 15. Cracks Produced by Hydrogen Absorption (60% HNO_3 Electrolytic)

All of the failures produced to date have occurred at a load of 75,000 psi. Test 2-4 is an attempt to establish a time to fracture versus load response for a charging density of 2.5 A/cm^2 , and to produce a fracture in which the contribution of hydrogen damage is greater than it was at the higher load of 75,000 psi. In so doing, it is hoped to obtain hydrogen damage cracks that are more than the one grain deep cracks observed in all specimens that fractured under a load of 75,000 psi.

c. Absorption Kinetics

It has been mentioned previously that room temperature hydrogen absorption is a slow process. It can be improved by increasing the current density and by the use of promoters, such as As_2O_3 or As_2O_5 that produce AsH_3 at the charging surface. The effect of adequate AsH_3 production is to the effect an apparent increase of about 15 fold of the applied current density. That is for an applied current density of 5 A/cm^2 good promotion makes it appear as though it were 75 A/cm^2 .⁽⁷⁾ It was previously argued that As_2O_5 had the thermochemical promise of being a better promoter than As_2O_3 .⁽²⁾ However qualitative evidence indicates that As_2O_3 may actually be the better promoter.

The concluding tests in this phase of the investigation will be conducted during the next period and will be oriented toward producing longer cracks and establishing the effects of gradually applied versus rapidly applied loads.

SECTION V

TASK III - CLADDING SURFACE FILM STUDIES

(R. N. Duncan, G. F. Rieger, D. L. Zimmerman)

5.1 OBJECTIVE OF TASK

Investigate the relationship between the cladding surface film and susceptibility to intergranular corrosion.

5.2 RESULTS DURING THIS PERIOD

Longitudinal sections from irradiated cladding are being prepared by metallographic techniques to preserve the thin surface films. Four areas from fuel rod 6-S-F were selected for detailed examination by using diameter profile traces of this fuel rod and photomicrographs of the longitudinal sections. Preparation of these four samples for examination at high magnifications is in progress.

SECTION VI

TASK IV - EFFECTS OF APPLIED STRESS (G. F. Rieger)

6.1 OBJECTIVE OF TASK

Investigate the effects of operating stress levels, stress-strain relationships, and power cycling in causing intergranular cracking.

6.2 RESULTS DURING THIS PERIOD

As discussed in previous progress reports,^(1,2) the investigation of the effects of applied stress includes utilization of diameter profile measurements of the outer surface of the fuel rods to complement the analytical work.

The post-irradiation fuel rod profiles are used to evaluate residual curvatures in the cladding. These may be either circumferential ridges usually associated with locations adjacent to pellet interfaces or longitudinal ridges which are formed when the clad deforms to an oval shape, or combinations of both. Since pre-irradiation measurements of fuel and cladding diameters of accuracies comparable to those of the post-irradiation profile measurements are not available, the nominal design dimensions were used to estimate the differences and changes of the mean cladding diameters. The nominal dimensions, the available operating histories, and the residual curvature profile measurements were combined to estimate the stresses and strains which existed in the cladding during operation.

To date, only the circumferential ridge deformations at regions near the axial peak power regions have been evaluated. A first order approximation of the longitudinal surface strain in the cladding may be obtained by considering only the local circumferential ridge with respect to the adjacent base diameter region located over the midportion of the fuel pellets. The differential between the ridge and adjacent base diameters is a measure of the elongation strains at the ridge. This does not allow a direct evaluation of the base diameter expansion.

A simplified example may be used to illustrate the basic features of the evaluation technique. The residual curvature measurements obtained from the profiles are used to estimate the relative interaction expansion between cladding and fuel. The relative expansion is also calculated using the nominal design dimensions and the operating histories of that particular fuel rod. The two expansions so determined are then compared to estimate the probable as-fabricated gap. This value is incorporated into a calculation to estimate the overall stress and strain distributions. For brevity, only a simplified version of the former calculation will be discussed here, and a one dimensional model will be used to illustrate the multiaxial case.

For a tubular shell or a beam, a moment-curvature diagram, similar to a stress-strain diagram, may be developed which incorporates the tube geometry and the material properties. An example of a moment-curvature plot is shown in Figure 16.

Figure 16. Moment-Curvature Plot

As the fuel-clad interaction expansion increases, the cladding curvature k is increased and the associated bending moment M is increased along path O-B through the elastic limit M_{el} to the plastic load moment M_L . At M_L the curvature is k_L . If the bending were fully elastic, at k_L the moment would have the value of the equivalent elastic load moment M_{EL} . When the load is removed, the curvature is reduced according to path B-C to the value k_c , which is the residual curvature measured by the profilometer (after suitable corrections for temperature and pressure). The path B-C is parallel to the original elastic slope O-A. The actual cladding is subjected to a further reduction in curvature because of the pressure, and for the thinner wall rods, actually produces a multiaxial plastic hinge which will not be discussed here.

The residual longitudinal bending strain may be evaluated from the residual curvature from

$$\Delta\epsilon_{B_{Resid}} = \frac{h}{2} \Delta k_{Resid} \quad (1)$$

where

- $\Delta\epsilon_{B_{Resid}}$ = Residual longitudinal bending strain
- h = Tube wall thickness
- Δk_{Resid} = Residual longitudinal curvature

To evaluate the conditions during operation, the load point B must be established. This can be accomplished by developing both the load moment and equivalent elastic load moment relations. The load moment diagram is used to establish the path from O through B, and the equivalent moment the slope of the line from C to the intersection at B.

The stress-strain curve of the Type-304 stainless steel may be approximated by a power law function of the form

$$\sigma = A \epsilon^n$$

where

σ = Stress

ϵ = Strain

A, n = Material property constants

Approximate values for the Type-304 stainless steel clad material used are:

Material Condition	Temperature	A, ksi	n
Annealed	Room Temperature	124	0.125
Annealed	650°F	68	0.125
Work hardened (or irradiation hardened)	Room Temperature	131	0.065
Irradiation hardened	650°F	102	0.065

The power law stress-strain relation may be developed into a moment-curvature relation

$$M_L = 1.20 \left(\frac{A}{n+2} \right) \left(\frac{h}{2} \right)^{n+2} \left(k_L^n \right) \quad (3)$$

Similarly, the equivalent elastic load moment-curvature relation may be written as

$$M_{EL} = D k_{EL} \quad (4)$$

where

$$D = \text{Flexural rigidity, } D = \frac{Eh^3}{12(1-\nu^2)}$$

E = Young's Modulus

ν = Poisson's ratio

Also, M_{EL} may be written in terms of k_L and the differential diameter between the ridge and adjacent base diameters as

$$k_A = \frac{2}{h} \sqrt{3(1-\nu^2)} \left(\frac{\Delta D_R}{D_{cl}} \right) \quad (5)$$

where

ΔD_R = Differential diameter between ridge and adjacent base

D_{cl} = Clad adjacent base diameter

The load point B may be established by simultaneous solution of the moment and the curvature relations using the elastic recovery of the curvature to establish M_L .

$$M_L \text{ (eqn 3)} = M_{EL} \text{ (eqn 4)}$$

$$1.20 \left(\frac{A}{n+2} \right) \left(\frac{h}{2} \right)^{n+2} \left(k_L^n \right) = \Delta k_{B-C} \quad (6)$$

and

$$k_L = \Delta k_{Resid} + \Delta k_{el \text{ rec}} \quad (7)$$

$$= \Delta k_{Resid} + \Delta k_{B-C}$$

Thus both k_L and M_L are determined. The bending strain during operation may then be found from

$$\Delta \epsilon_{Bop} = \left(\frac{h}{2} \right) \left(k_L \right) \quad (8)$$

In a similar manner equation (5) can be used to obtain the ridge displacement. Additional strains associated with circumferential elongation of the ridges, as well as those caused by the mean fuel expansions and ovality bending, are added to the strain values obtained from (1) and (8) to assess the strain state. A multiaxial equation of the general form of (2) can be used to assess the stress state.

An extension of this work is in progress.

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