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METALLURGY SECTION
ADVANCED TECHNOLOGY

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AEROJET SOLID PROPULSION COMPANY

MONTHLY PROGRESS REPORT - August & September 1971

DESIGN OF STRESS CORROSION SPECIMEN FOR DETERMINATION OF
 K_{Isc} IN PRESSURIZED GASEOUS HYDROGEN EXPOSED TO IRRADIATION

MASTER

I. INTRODUCTION

Department 4830 has been requested to determine if a synergistic effect will result when Inconel 718, A-286, SAE 9310 and Ti-5Al-2.5Sn are exposed to 1000 psi gaseous hydrogen and a nuclear reactor. The criterion used to determine any synergistic effect will be to measure the susceptibility of each test material to slow crack growth, crack growth rate and/or apparent K_{Isc} , when exposed to the nuclear environment with and without 1000 psi gaseous hydrogen. The reactor environment will include a fast neutron fluence of about 10^{20} and a specimen temperature in the range between 155 and 200°F.

The specimen development and calibration were initially to be conducted in three phases where phase 1 was to design and evaluate a test specimen for phases 2 and 3, phase 2 was to (1) test the initial specimen in the General Dynamic Reactor without gaseous hydrogen (GH_2), (2) verify instrumentation concepts and (3) confirm the specimen design through testing in the reactor environment, and phase 3 was to be in-reactor testing to determine the combined effects of 1500 psi GH_2 and the nuclear environment. In order to expedite the initiation of phase 3, a change has been made in phase 2. Phase 2 will consist solely of a reactor test to determine the noise characteristics as detected by acoustic emission. This test will be conducted to determine the applicability of using acoustic emission to detect slow crack growth in a reactor environment.

This report covers progress for a two month period since the writer was on vacation from August 27 to September 13.

II. CURRENT STATUS SUMMARY

The preliminary program outlining objectives and procedures has been submitted.

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A. SPECIMEN CONFIGURATION

1. Stress Corrosion Specimen

Sketches for modified WOL specimen, 1-1/2 and 2.0-in.-thick compact tension (CT) specimen, and encapsulated specimen design have been submitted. Minor dimensional changes to existing 1.0-in.-thick CT specimen were also made. Tentative design of stress corrosion specimen has been completed.

2. Encapsulated Specimen

The encapsulation device shown in Figure 1 was eliminated from further study due to the success obtained in fabricating specimens incorporating the design shown in Figure 2. Preliminary stress analysis has been completed for this specimen (Figure 2) and indicates that the encapsulation device had little effect on the stress at the crack tip. Consequently, the encapsulation design shown in Figure 2 will be used for this specimen.

B. INSTRUMENTATION FOR DETECTION OF SLOW CRACK GROWTH

Instrumentation is required to ascertain whether slow crack growth had initiated and arrested during the limited time in the reactor. Without such information, accurate comparisons could not be made between effects due to pressurized hydrogen and those in helium in a reactor environment. Since it will be necessary only to detect crack arrest a very high system sensitivity will not be required. Two types of instrumentation, acoustic emission and strain gages, are being investigated for this purpose.

1. Stress Wave Analysis Technique (SWAT)

a. Sensors

Preliminary information concerning stress wave emission sensors that could be used in the reactor has been obtained. Comparison test data between a possible sensor (Endevco P-15) for use in the reactor tests and the sensor (Endevco 2213E) presently being used for data collection by ASPC has been obtained. Both sensors (P-15 and 2213E) were connected to voltage amplifiers and the P-15 results compared very favorably to the results obtained using the 2213E sensor. The major problem associated with the P-15 type sensors is low capacitance (on the order of 50 pf). A second sensor fabricated from a P-14 crystal appears to be suitable because it has a higher capacitance, on the order of 300 pf. Tests have not been made on the P-14 sensor to determine its sensitivity as compared to the P-15 or the 2213, but tests at Endevco have found the P-14 to be approximately 20% as sensitive as the P-15. However, the P-14 sensor

is less sensitive to the cable capacitance than the P-15 sensor so that with 15 feet of cable, it is thought that the P-15 sensor would be twice as sensitive as the P-14 unit. Both sensors and cables have been ordered to evaluate the combined sensor-cable sensitivity.

Gulton Industries have agreed to supply ASPC with a sensor so sensitivity measurements could be compared with the P-14 and P-15. The Gulton Industries' sensor has been received and comparison tests are scheduled for next month.

A hard-line triaxial cable has been selected for the tests because of a lower noise level. As the sensors will be exposed to water, it has also been decided to fabricate the cable and sensor as an integral unit in order to eliminate fittings which could be a source of leakage. A supplier for line drivers (preamplifier) that will withstand exposure to radiation and water has been found.

2. Alternate Approaches to Detect Slow Crack Growth

a. Instrumentated Bolt

An instrumentated bolt suitable for use in water at 200°F has been successfully fabricated but slow crack growth has not occurred in the tests performed to date using this instrumentation.

b. Strain Gages

Strain gages can be placed along the path of slow crack growth but difficulties are anticipated in the design of an acceptable entrance part through the encapsulation device for the lead wires. An alternate approach would be to mount the strain gages on the edge of the encapsulation device, offset from the path of slow crack growth, although the sensitivity attainable using this approach requires definition. As of yet, no tests have been run to determine the sensitivity of the strain gages as a method for detecting slow crack growth.

III. ACTIVITIES DURING THE REPORTING PERIOD

PHASE I - DESIGN AND EVALUATION

A. TESTING

Two compact tension specimens, one of Inconel 718 and one AISI 9310, were tested at 200°F; the average $K_q = 90$ and $120 \text{ ksi-in.}^{1/2}$, respectively. All of the AISI 9310 used in this investigation will be cycled through the simulated carburizing heat treat as listed below:

1. 1700°F for 4 hours, furnace cool
2. 1500°F $\pm$ 25 for 1 hour per in. of thickness (1 hour min.)
3. Quench in 100-140°F agitated oil and air cool to room temperature
4. Cool to -320°F for 1 hour, air warm to room temperature
5. Temper after step 3 within 1 hour in a recirculating air tempering furnace at 300°F minimum temperature for 3 hours, double temper.

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3)*

One A-286 modified WOL specimen was tested in water at 200°F to evaluate potential test techniques and instrumentation problems. The specimen was not fatigue cracked but the precrack was induced by overloading with the bolt. The specimen was placed in the bottom of a water filled beaker mounted on a hot plate. Bubbles forming on the bottom of the beaker and on the specimen were detected by acoustic emission and would have caused difficulties in interpreting the acoustic emission data if slow crack growth had occurred. The specimen was held for 17 days at 60 kips (approximately $280 \text{ ksi-in.}^{1/2}$) with no apparent crack growth, as indicated both by acoustic emission and COD gage.

*Test
heated?*

One AISI 9310 modified WOL specimen was tested in rapidly circulating water at 150°F. Because of the problem with bubbles observed in the previous test, the hot water system was changed to a 15 gallon plastic container and a heating unit and pump were mounted in the water reservoir. The heating unit was too small for the volume of water and the maximum temperature attainable was 150°F. The specimen was not fatigue cracked but an attempt was made to induce the precrack by overloading with the bolt. Problems were encountered during the

precracking attempt and the bolt head fractured at 40 kip. Even though the specimen had no apparent crack, it was placed in 150°F water, for the week-end, in the hope that a crack would nucleate and the subsequent slow crack growth could be detected by acoustic emission and the instrumented tup. After 66 hours exposure to the agitated water at 150°F, there still was no apparent crack formation or growth. However, the water barrier failed on the ^{cad.} strain gages resulting in the gages becoming unbonded. Alternate barrier materials and/or application techniques are being evaluated. ?

B. SLOW CRACK GROWTH DETECTION

1. Strain Gages

The weldable strain gages anticipated for use in the reactor test will be mounted on the tup as shown in Figure 2. Note, in Figure 2, that the instrumentated tup will consist of a half bridge, one gage mounted on each end. The gages and cables are an integral unit consisting of a 304 stainless steel sheath and MgO insulation. As indicated in Figure 2, the lead wire will be spot welded to the specimen in order to prevent damage to the assembly during handling. Since the gage and cable are an integral unit, it will be necessary to mount the second gage on opposite side of the tup after insertion of the tup into the specimen. The need for mounting the gage after inserting the tup into the specimen could mean that a new strain gaged tup will have to be used for each specimen. However, depending on the difficulties encountered when laying the gage on an irradiated tup, it will be feasible to salvage the tup and one gage from each test. The reference gage will be used to ascertain the changes in resistance solely as a function of radiation. good

A second possible approach when using strain gages is to mount gages in the path of anticipated slow crack growth. In order to use the gages in this manner, the encapsulation device must be greater than 3/4 in. wide vs 0.48 in. wide when not using strain gages. A second problem with this technique is the method employed to bring the lead wire out of the encapsulation device. Work is presently being conducted on this facet. A possible alternative would be to mount the strain gages outside of the encapsulation device although the sensitivity attainable with this approach has not been defined.

The overall problem with weldable strain gages is that considerable drift will occur due to irradiation and it is extremely difficult to ascertain if this change in resistance is due to a change in strain or to irradiation effects. An example of this change in resistance with time in the reactor can be seen in Figure 3* for unheat treated gages. In a discussion with Mr. C. Toy of Microdot, we were told that Microdot can reduce the resistance change with respect to time by heat treating. The resistance changes, in a negative direction due to irradiation, are analogous to heat treating the Ni-Cr alloy in the gages to its hardest condition. By heat treating the strain gages to the hardest condition, prior to irradiation, it is possible to reduce the resistance changes that occur due to irradiation. As the reference gage will be monitored during the tests, it will be possible to determine resistance changes occurring as a result of irradiation.

A second strain gage type is being investigated which will allow the mounting of a one-half bridge on each end of the tup. The gage will be mounted with M Bond 610 cement per the recommendations of Mr. C. Tallman of Los Alamos and J. Starr of Micromeasurement. The cement and gages have been successfully used during exposure to a fast fluence of 5×10^{18} but it is uncertain if they will be satisfactory at a fluence level greater than 1×10^{19} . If successful, these gages would have the following advantages over the weldable strain gages:

- a. Less problems with changes in resistance with time in the reactor because of better quality control for foil gages than weldable gages.
- b. Easier to mount than the weldable gage.
- c. Lower cost.

2. Acoustic Emission

Components have been ordered for the in-reactor evaluation. The sensors are of similar construction to those previously tested in high fluence nuclear radiation. The crystal is the Endevco Piezite P-15 and P-14 material

*M. J. Pettigrew, "An Evaluation of Weldable Strain Gages in Nuclear-Reactor Environments", Experimental Mechanics, May 1970.

*Very small
might not need
strain gage to
measure limit.*

tested by E. Sita of NERVA. The cable is a braided shield using quartz fiber insulation with a steel tubing overall. As indicated in the July Monthly Report, the sensor and cable will be fabricated as an integral unit.

The supplier for the line driver has been found and no problems in fabricating a hardened line driver are anticipated by this source. The line driver fabricator will connect the line driver and cable as an integral unit to the sensor system supplied by Endevco. The sensor, cable, line driver, and cable combination will be a total of 32 ft. long.

C. ENCAPSULATION DEVICE

The encapsulation device shown in Figure 2 has been successfully welded to a stainless steel specimen. Because of the success in welding the encapsulation device, the tests originally scheduled to be conducted in a hydrogen-charging solution will be omitted and tests conducted with pressurized hydrogen conducted immediately upon receipt of specimens.

Due to material handling problems and the desire to keep the specimen notch (.6 in.) as small as possible, the cross section shown in Figure 4 was selected for the encapsulation device. This device is electron beam welded to the specimen. The heat affected zone is on the order of .05 in. diameter at each weld and will not affect crack propagation so long as the crack does not deviate from the notch center line by more than ± 0.20 in. Deviation by more than ± 0.20 in. would be of concern from a fracture testing standpoint so that the developed fabrication method is considered satisfactory.

D. STRESS ANALYSIS OF SPECIMEN-ENCAPSULATION DEVICE

The stress analysis for the specimen and encapsulation device in Figure 5 was completed and is shown in Table I. Since the modulus of three materials (A-286, Inconel 718 and SAE 9310) to be tested is reasonably close, only one calculation was performed. Observe in Table I that the load pick-up by the encapsulation device was very small and can be neglected. The load at the pin can be assumed to have been completely transmitted through the specimen and none through the encapsulation device. The calculated values will be checked experimentally by mounting strain gages adjacent to the crack tip and recording strain gage output as a function of the COD reading.

Analysis has also shown that as the flaw grows there will be an increase in volume from 2 to 3 percent. This will have a corresponding decrease in gas pressure of the same magnitude but is not considered significant for the purpose of this program.

The specimen configuration has not been completely settled on as decisions must be made as to what position the specimen will be placed in the reactor. It is anticipated that the specimen will be placed in a 2.5 in. diameter hole which will require some specimen dimensional changes. A stress analysis has been initiated to determine if there is enough material in the lever arms so neither plastic deformation nor the arms will fail at the anticipated loads. The specimen length will not be established until tests have been completed using pressurized gaseous hydrogen.

PHASE II - INITIAL SPECIMEN TESTING AT PLUMBROOK REACTOR

This test has been changed somewhat in accordance with NASA recommendations to test acoustic emission data acquisition in the reactor and evaluate effects due to inherent reactor noise problems. The test will be conducted using an ordinary accelerometer and the instrumentation of the type generally used by Aerojet Solid Propulsion Company to monitor noise. The test results will allow for a decision to be made regarding the capability of acoustic emission to operate successfully in the reactor environment.

PHASE III - TESTING THE COMBINED EFFECT OF 1500 psi H₂ AND THE NUCLEAR REACTOR

Dependent upon completion of Phase I and II.

IV. ANTICIPATED ACTIVITIES DURING THE NEXT REPORTING PERIOD

Fatigue crack the remaining modified WOL specimens and use them to test the comparative sensitivity between acoustic emission and an instrumented tup.

Completion of mathematical analysis of the specimen height to determine compatibility with reactor tube diameter.

Evaluate the encapsulation design to see if it will withstand the stress imposed by increasing the COD readout to .05 in. or 0.10 in.

Test encapsulated specimens in pressurized hydrogen to determine comparative sensitivity between acoustic emission and strain gage techniques. (After the comparison tests have been made it will be possible to determine if the strain gage sensitivity would be adequate for our purpose.)

As the D-2 tool steel loading bolt rusted when submerged into primary reactor water at Plumbrook, a substitute material and/or nickel plating will be investigated that will be compatible with the reactor water.

Prepared by:

W. G. Reuter

Approved by:

P. P. Crimmins, Manager
Metallurgy Section

Figure 1

Figure 2

Figure 3. Effect of Irradiation on Strain Gage Resistance

Figure 4. Schematic of Encapsulation Device

Figure 5

CRACK 1 TH

.4 in.

Material	A-286 Inconel 718 AISI 9310			Ti-5Al-2.5Sn E11		
	Stress @ A (ksi)	Load @ Pin (lb)	Load Across Tube (lb)	Stress @ A (ksi)	Load @ Pin (lb)	Load Across Tube (lb)
1000 psig	2	1483	1.6	2.5	1481	1.6
B = .010	20	3622	5	10.5	-	-
B = .030	64	8605	-	33	-	-
B = .050	105	13588	27	55	7567	25

1.4 in.

Material	A-286 Inconel 718 AISI 9310			Ti-5Al-2.5 Sn E11		
	Stress @ A (ksi)	Load @ Pin (lb)	Load Across Tube (lb)	Stress @ A (ksi)	Load @ Pin (lb)	Load Across Tube (lb)
1000 psig	9	2663	3	9.5	2667	9.5
B = .010	17	3349	8	10.5	-	-
B = .030	48	6006	25	24	-	-
B = .050	80	8662	51	39	5467	40

$$B = \Delta[\text{COD}] \text{ READING } ??$$

$$= \frac{\text{LOAD ACROSS TUBE}}{\text{LOAD ACROSS TUBE}}$$

ATTACHMENT

An example of the encapsulated specimen was shown to several members and guests attending the E-24 Sub 4 Committee Meeting in Los Angeles, Sept. 30, 1971. The only comment G. Kauffman of Alcoa had was that the 0.6 in. cut required a longer specimen so the specimen would conform to the 30° angle requirement shown in E-399. Our design has already taken this into consideration, so there appears to be no problem in this respect. W. Anderson of Battelle looked at the specimen and the only problem he could foresee was possible failure at the corner welds of the encapsulation device when loaded under pressure. I tried to find W. F. Brown, Jr. and J. E. Srawley to ask their opinions but Brown wasn't at the meeting and Srawley was unavailable.

Some of the comments regarding K_{Isc} testing were of considerable interest for anyone trying to determine design property K_{Isc} values. As our tests will be conducted to determine any possible synergistic effect, we will not be affected by problems associated with quantitative values.

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Dear

1. Here are four stapled and three paper clipped copies of the Aug. 1st. monthly report. I have included an attachment page at the end of the report and didn't know if you wanted it included or not.

I have given a copy each to Mel & Joe

2. One copy (original) of Test Procedure
3. One copy of ASTM info. concerning compact tension specimens.