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HYDROGEN EMBRITTLEMENT OF IRRADIATED ALLOYS

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FOREWORD

The radiation effects/hydrogen embrittlement study described in this document was performed at the Nuclear Aerospace Research Facility of the Convair Aerospace Division of General Dynamics for the Space Nuclear Systems Office, Cleveland, Ohio (SNSO-C) under Contract SNPC-73, Modification No. 3.

Test specimens and test specifications were provided by Aerojet-General Corporation (AGC), Sacramento, California, and Westinghouse Astronuclear Laboratory (WANL), Pittsburgh, Pennsylvania. The test specimens were irradiated by the National Aeronautics and Space Administration, Lewis Research Center, Plum Brook Station, Sandusky, Ohio.

This document contains a complete description of the test specimens, experimental equipment, and test procedures in addition to a presentation and discussion of the experimental results.

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ABSTRACT

The effects of gaseous hydrogen at 1500 psi and room temperature were studied on the notch tensile strengths of four types of alloys - 301 SS, A-286, Ti 5Al 2.5Sn, and Inconel 718 - which had been previously irradiated to neutron fluences of $1.3-1.6 \times 10^{20} \text{ n/cm}^2$ ($E > 1 \text{ MeV}$). The reductions in notch tensile strength of 301 SS, 81%; Ti 5Al 2.5Sn, 7.3%; and A-286, unchanged, were in good agreement with those expected for similar specimens which were unirradiated. The notch tensile strength of irradiated Inconel 718 specimens was unaffected by hydrogen, while control specimens exhibited 8-9% losses in strength.

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I. INTRODUCTION

This program was conducted to investigate the combined effects of high-pressure hydrogen gas and reactor radiation on the notch tensile strengths of four materials being considered for use in the propellant feed system of the NERVA: Inconel 718, 301 SS, A-286, and Ti 5Al 2.5Sn. Tensile tests were performed at ambient temperatures at a pressure of 1500 psi. The test specimens were irradiated in the NASA/Plum Brook reactor to a nominal fluence of 1.5×10^{20} n/cm² ($E > 1$ MeV).

No data are available in the literature on the hydrogen embrittlement of irradiated alloys of these types. The embrittlements of unirradiated specimens of Inconel 718, Ti 5Al 2.5Sn, and A-286 have been studied previously, however.

Reductions in the notch tensile strength of Inconel 718 varying from 9.1% to 54% have been reported (Refs. 1,2,3). The degree of hydrogen embrittlement of Inconel 718 was observed to increase with increasing hydrogen pressure. The data also indicate that the degree of embrittlement decreases with increasing solution annealing temperature.

The notch tensile strength of Ti 5Al 2.5Sn has been observed to undergo losses of 19% at a hydrogen pressure of 10,000 psi (Ref. 1) and 10% at 1400 psi (Ref. 3).

Specimens of A-286 have been reported to lose 3% in notch tensile strength at a hydrogen pressure of 10,000 psi (Ref. 1) and to be unaffected at 1400 psi (Ref. 3).

No data have appeared in the literature on the effects of high-pressure hydrogen on 301 SS.

II. TEST DESCRIPTION

The purpose of this test was to determine the effects of gaseous hydrogen at 1500 psi and ambient temperatures on the notch tensile strength of four irradiated alloys. The test specifications were prepared by the project sponsor, Aerojet-General Corporation, and are listed below for each material type:

<u>Material</u>	<u>Test Specification</u>
Ti 5Al 2.5Sn	37 NRO 090
Inconel 718	37 NRO 091
A-286	37 NRO 092
301 SS	37 NRO 093

The tensile tests were performed by utilizing a stainless steel pressure chamber in conjunction with an Instron test machine. High-purity hydrogen was obtained by passing bottled hydrogen through a purification system before introduction into the test chamber.

A detailed description of the specimens, apparatus, and test procedures are presented in this section.

2.1 Test Specimens

The A-286, Inconel 718, and Ti 5Al 2.5Sn specimens were supplied by AGC. These specimens were of the notched buttonhead design shown in Figure 1. The 301 SS specimens were supplied by

Dimensions in inches

Figure 1 Design of Buttonhead Specimens

WANL and were of the notched sheet design shown in Figure 2. The notch dimensions of the buttonhead specimens are given in Table I.

Table I
NOTCH DIMENSIONS OF BUTTONHEAD SPECIMENS

Material	S/N	Notch Diameter (in.)	Root Radius (in.)
Inconel 718	I14	0.217	0.0014
	I15	0.215	0.0014
	I16	0.216	0.0013
	I18	0.214	0.0015
	I19	0.218	0.0014
	I21	0.217	0.0013
Ti 5Al 2.5Sn	T44	0.214	0.0013
	T45	0.215	0.0013
	T46	0.215	0.0013
	T47	0.214	0.0013
	T48	0.215	0.0014
	T49	0.215	0.0014
A-286	A1	0.216	0.0013
	A2	0.209	0.0013
	A3	0.213	0.0013
	A4	0.209	0.0013
	A5	0.215	0.0014
	A6	0.218	0.0013

The notch dimensions of the 301 SS specimens are given in Table II. The quantities r_1 and r_2 are the root radii on the two sides of the notch, t_1 and t_2 are the thicknesses at the sides of the notch, and W is the width of the notch.

Dimension in inches

Figure 2 Design of Sheet Specimens

Table II

NOTCH DIMENSIONS OF 301 SS SPECIMENS
(in.)

S/N	r ₁	r ₂	t ₁	t ₂	W
1	0.0015	0.0015	0.0348	0.0346	0.2539
2	0.0016	0.002	0.0355	0.0354	0.2528
3	0.0016	0.0015	0.0341	0.0338	0.2536
4	0.0016	0.0016	0.0353	0.0353	0.2528
5	0.0016	0.0017	0.0349	0.0349	0.2531
6	0.0017	0.0017	0.0350	0.0350	0.2544

The chemical compositions of the test specimens are given in Table III and the heat treatments in Table IV.

Table III

CHEMICAL COMPOSITIONS OF TEST SPECIMENS

Element	Inconel 718	Ti 5Al 2.5Sn	A-286	301 SS
C	0.04	0.011	0.05	0.10
Mn	0.10	<0.01	1.68	0.99
Si	0.13	-	0.65	0.45
P	0.003	-	0.021	0.031
S	0.006	-	0.009	0.010
Cr	18.21	-	14.70	17.65
Ni	52.54	-	26.43	7.1
Mo	3.06	-	1.27	0.24
Cu	0.01	-	-	0.10
Cb/Ta	5.22	-	-	-
Ti	0.91	91.82	1.99	-
Fe	19.02	0.14	52.54	73.33
Al	0.60	5.44	0.27	-
Co	0.10	0.009	-	-
B	0.0037	-	0.006	-
Sn	-	2.47	-	-
H	-	0.0094	-	-
O	-	0.083	-	-
N	-	0.009	-	-
V	-	-	0.38	-

Table IV

HEAT TREATMENTS OF TEST SPECIMENS

Material	Heat Treatment
Inconel 718	Forgings were solution annealed at 1950°F, held 1 hr and rapid air cooled per AGC Specs 90093 and 46604B. Following rough machining, the specimen blanks were aged at 1350°F for 8-10 hr, furnace cooled to 1200°F, held for a total aging time of 20 hr, air cooled (AGC 46604).
Ti 5Al 2.5Sn	Vacuum annealed at 1400°F for 1 hr, furnace cooled.
A-286	Solution heat treated at 1800°F for 1 hr, precipitation heat treated at 1300-1400°F, held minimum of 16 hrs, air cooled.
301 SS	Cold worked.

2.2 Irradiation

A total of 24 specimens, 6 of each material, was irradiated. The irradiation of the specimens, described in Reference 4, was performed during power cycles 110 and 111 of the Plum Brook Reactor (PBR) at the NASA Lewis Research Center.

The specimens were mounted in capsules which were then loaded in PBR test holes. Primary reactor water was flowed through the capsules during irradiation in order to maintain specimen temperatures of 125-150°F.

PBR power cycle 110 was initiated at 1448 on 26 June 1970 and terminated at 1040 on 9 July 1970. During the cycle, 655.6 megawatt-days were accumulated. During shutdown the capsules were rotated 180° in order to decrease the fluence gradient across the specimens.

PBR power cycle 111 was started at 1753 on 11 July 1970 and terminated at 2242 on 27 July 1970. During the cycle, 764.8 megawatt-days were accumulated.

Thermal fluences on the specimens were measured with cobalt wires and fast fluences with nickel and titanium detectors. The thermal and fast ($E > 1$ MeV) fluences received by the specimens are given in Table V. The estimated uncertainty of the fluences at the 95% confidence level is $\pm 20\%$.

Table V
NEUTRON FLUENCES RECEIVED BY TEST SPECIMENS

Material	Thermal Fluence (n/cm ²)	Fast Fluence (n/cm ² E > 1 MeV)
Inconel 718	1.4×10^{21}	1.5×10^{20}
Ti 5Al 2.5Sn	1.3×10^{21}	1.3×10^{20}
A-286	1.4×10^{21}	1.5×10^{20}
301 SS	1.4×10^{21}	1.6×10^{20}

The induced radioactivity of individual Inconel 718, Ti 5Al 2.5Sn, and A-286 specimens were measured at 1 foot 4½ months after irradiation and found to be 42, 0.75, and 32 R/hr, respectively. The dose rate from the 301 SS specimens 8 months after irradiation was 15 R/hr at 1 foot.

2.3 Experimental Apparatus

The experimental apparatus used for these hydrogen embrittlement tests consisted of three systems: a tensile test machine, a high-pressure chamber, and a hydrogen-purification system.

An Instron Model TTD was used to perform the tensile tests. The loading assembly of this machine was located inside a hot cell. Remote handling and testing of the highly radioactive specimens were accomplished by means of the hot cell manipulators and remote control console of the Instron.

The test chamber was constructed of 8-in. Schedule 80 pipe of 316L stainless steel. Wall thickness was 1/2 in., and inside dimensions were approximately 7.6 in. diameter by 24 in. length. The pull rods entered the chamber through triple O-ring seals on each end. Ports were provided for hydrogen gas and instrumentation inputs.

The two sections of the chamber were joined and sealed with a remote-disconnect clamp manufactured by Gray Tool Company.

This operation was performed remotely by utilizing the Instron crosshead and a remote torque wrench. A photograph of the test chamber mounted on the Instron loading assembly appears in Figure 3.

The hydrogen used in these tests was required to meet MSFC SPEC-356A, which is defined below:

Maximum Allowed Impurities (ppm)						
O ₂ +A	N ₂	CO	CO ₂	CH ₄	Hydrocarbons as CH ₄	H ₂ O
1	2	0.5	1	2	9	9

Commercial high-purity hydrogen, 99.995%, was therefore passed through a purification system before introduction into the test chamber.

The purification system consisted of Engelhard catalytic deoxygenation units, activated charcoal and alumina adsorbents at 77°K, and a 10-μ filter. A schematic of the hydrogen purification and distribution system is given in Figure 4.

A rotary vacuum pump with Veeco foreline and liquid nitrogen traps was used to evacuate the test chamber prior to pressurization.

The purity of the hydrogen was measured with a Perkin-Elmer Model 880 gas chromatograph. Linde 5A molecular sieve and silica gel columns and a thermal conductivity detector were used in the chromatograph. Hydrogen samples could be taken directly from

Figure 3 Test Chamber Mounted on Loading Assembly

Figure 4 Schematic of Gas Distribution System

either the hydrogen purification system or the pressurized test chamber.

The chromatograph was calibrated for the gases of interest by means of gas mixtures of known composition which were prepared with a gas proportioner. The minimum sensitivity for the permanent atmospheric gases was approximately 0.3 ppm.

2.4 Test Procedures

Slotted sample grips were employed for testing of the irradiated buttonhead specimens in order to facilitate loading. Axiality was found to be very poor when the slots were parallel; maximum bending strain due to the apparatus was 31.5% (Ref. 5). It was found, however, that when the slots were oriented in opposite directions the maximum bending strain at the center of the specimen was 3.5%.

The grips were therefore mounted in the test chamber in the opposed configuration, and axiality of the entire tensile test system was measured. The maximum bending strain at the center of the specimen due to the apparatus was 7%. The maximum allowed by test specifications was 9.2%. A view of a specimen loaded in the grips is presented in Figure 5.

Analyses of the hydrogen gas were performed before initiation of tensile testing. Samples of hydrogen directly from the purification system and from the pressurized chamber were found to contain

Figure 5 Buttonhead Specimen Loaded in Test Chamber

no contaminants of O_2 , N_2 , CO, CO_2 , and CH_4 . Minimum sensitivity of the gas chromatograph was approximately 0.3 ppm. Water content of the hydrogen was found to be 0.6 ppm by means of dew point measurements.

Gas analyses were performed periodically after initiation of tensile testing. Only trace amounts of contaminants were found in any of these tests.

Both evacuation and purging were used to prepare the test chamber for final hydrogen pressurization. The chamber was first evacuated to approximately 100 microns, then pressurized to 300 psi with high-purity helium. This pressure was then bled off, and the chamber was vacuum-pumped for a minimum of 8 hours. Final pressures of a few microns were attained. The chamber was then pressurized to 100 psi with hydrogen, bled off, and evacuated for 1 hour. This pressurization-evacuation cycle was repeated, and the chamber was then pressurized to 1500 psi with hydrogen.

Crosshead speeds of 0.014 and 0.0093 in./min were used for the buttonhead and sheet specimens, respectively. These speeds correspond to a strain rate of approximately 0.0007 in./in./min for unnotched specimens.

Six specimens each of 301 SS, Ti 5Al 2.5Sn, and A-286 were tested. Four of each were pulled in hydrogen at 1500 psi and

two of each in high-purity helium at 1500 psi; the latter served as controls. Only four specimens of Inconel 718 were pulled, two in both hydrogen and helium at 1500 psi.

III. RESULTS AND CONCLUSIONS

The notch tensile strengths of the four types of irradiated alloys which were tested in 1500-psi helium and hydrogen are presented in this section. Unreduced areas were used to calculate notch tensile strengths from breaking loads.

3.1 Ti 5Al 2.5Sn

The notch tensile strengths obtained for irradiated Ti 5Al 2.5Sn specimens in 1500-psi helium and hydrogen are presented in Table VI.

Table VI

NOTCH TENSILE STRENGTHS OF IRRADIATED Ti 5Al 2.5Sn SPECIMENS

Spec. No.	Gas	Ultimate Load (lb)	Strength (ksi)	% Loss from He
T-48	Helium	6418	177	-
T-45	Helium	6304	174	-
T-44	Hydrogen	5988	166	5.4
T-47	Hydrogen	5930	165	6.0
T-49	Hydrogen	5949	164	6.5
T-46	Hydrogen	5668	156	11.1

The average notch tensile strength was reduced from 175 ksi in helium to 163 ksi in 1500 psi hydrogen, or an average loss of 6.9%.

Several unirradiated Ti 5Al 2.5Sn specimens were tested in air, helium, and 1500-psi hydrogen during checkout of the experimental apparatus. Average notch tensile strengths were 175 ksi in helium and air and 165 ksi in 1500-psi hydrogen for an average loss of 5.7% in hydrogen.

The notch tensile strength of unirradiated Ti 5Al 2.5Sn has been observed by other investigators to exhibit losses of 19% at a hydrogen pressure of 10,000 psi (Ref. 1) and 10% at 1400 psi (Ref. 3).

3.2 A-286

The notch tensile strengths obtained for irradiated A-286 specimens in 1500-psi helium and hydrogen are presented in Table VII.

Table VII

NOTCH TENSILE STRENGTHS OF IRRADIATED A-286 SPECIMENS

Spec. No.	Gas	Ultimate Load (lb)	Strength (ksi)	% Change from He
A-6	Helium	10,065	270	-
A-5	Helium	9,850	271	-
A-4	Hydrogen	9,565	279	+3.1
A-3	Hydrogen	9,707	272	+0.55
A-1	Hydrogen	9,945	271	+0.18
A-2	Hydrogen	9,613	280	+3.5

The average notch tensile strength of irradiated A-286 specimens increased from 270 ksi in helium to 275 ksi in 1500-psi hydrogen, or an average increase of 1.8%. This slight increase is probably the result of using the unreduced area in the calculation of notch tensile strength.

Unirradiated specimens have been reported to lose 3% in notch tensile strength at a hydrogen pressure of 10,000 psi (Ref. 1) and to be unaffected at 1400 psi (Ref. 3).

3.3 301 SS

Notch tensile strengths of irradiated 301 SS specimens in 1500-psi helium and hydrogen are given in Table VIII. Data for specimen 301 SS-1 are not reported, since it was damaged during the loading procedure.

Table VIII

NOTCH TENSILE STRENGTHS OF IRRADIATED 301 SS SPECIMENS

Spec. No.	Gas	Ultimate Load (lb)	Strength (ksi)	% Loss from He
-5	Helium	2180	247	-
-4	Helium	2198	246	-
-2	Hydrogen	427	47.6	81
-6	Hydrogen	440	49.4	80
-3	Hydrogen	404	46.9	81

The average notch tensile strength of irradiated 301 SS specimens was reduced from 246 ksi to 48 ksi, or an average loss of 81%.

There are no data in the literature on the effect of hydrogen on the notch tensile strength of 301 SS.

3.4 Inconel 718

The notch tensile strengths of irradiated Inconel 718 specimens in 1500-psi helium and hydrogen are reported in Table IX.

Table IX

NOTCH TENSILE STRENGTHS OF IRRADIATED INCONEL 718 SPECIMENS

Spec. No.	Gas	Ultimate Load (lb)	Strength (ksi)	% Change from He
I-16	Helium	10,925	298	-
I-19	Helium	10,720	287	-
I-15	Hydrogen	10,490	289	-1.2
I-21	Hydrogen	10,963	296	+1.2

The notch tensile strength of irradiated Inconel 718 specimens had an average value of 292 ksi in helium and exhibited no reduction in 1500-psi hydrogen.

Hydrogen embrittlement of Inconel 718 has been investigated in several studies (Refs. 1,2,3). Reductions in notch tensile

strength varying from 0 to 54% were observed over hydrogen pressures from 100 to 10,000 psi. The degree of embrittlement increased with increasing hydrogen pressure, although it was not a strong function at pressures greater than a few hundred psi. Walter and Chandler (Ref. 2) tentatively concluded that the degree of embrittlement was also related to the heat treatment of the Inconel 718 specimens.

Seven unirradiated Inconel 718 specimens of the same material heat and with the same heat treatment as the irradiated specimens were tested in hydrogen during the present study. Average reductions in notch tensile strength of 6.9% and 8.3% were observed at hydrogen pressures of 500 and 1500 psi, respectively. Since no specimens were tested in an inert atmosphere, a tensile strength of 289 ksi, which had been previously obtained for similar specimens (Ref. 3), was used as a reference value.

A summary of the available hydrogen-embrittlement data on unirradiated Inconel 718 specimens with different heat treatments appears in Table X. Solution-annealing temperatures varied from 1750°F to 1950°F; while the subsequent aging procedures for the different studies were quite similar. Although comparisons are complicated by the different hydrogen pressures of the several

Table X

SUMMARY OF HYDROGEN-EMBRITTLEMENT DATA ON UNIRRADIATED INCONEL 718

Ref.	Hydrogen Pressure (psi)	% Reduction of Notch Tensile Strength	Heat Treatment					
			Solution Annealing		First Aging		Second Aging	
			°F	Min	°F	Hr	°F	Hr
1	10,000	54	1750	60	1325	8	1150	10
3	500	34	1800	60	1325	8	1150	12
1	1,000	40*	1850	60	1360	10	1175	10
2	1,000	9.1	1900	20	1400	11	1200	9
	1,500	10.2						
	2,000	15.3						
3	500	50**	1800	60	1325	8	1150	12
			1950	60	1350	8-10	1200	10-12
Present Study	500	6.9	1950	60	1350	8-10	1200	10-12
	1,500	8.3						

* Material did not meet AMS composition specifications for Inconel 718.

**Material received double heat treatment with 1950°F anneal and aging following 1800°F anneal and aging.

studies, the data, with one exception, demonstrate a trend to lower degrees of embrittlement at higher solution annealing temperatures.

Hydrogen-embrittlement data on the doubly heat-treated specimens are anomalous. They appeared to be relatively unaffected by the second heat treatment, since the 50% effect observed was approximately that expected for specimens receiving the 1800°F heat treatment only. The 40% effect demonstrated by the 1850°F specimens was somewhat higher than that expected, if a correlation between embrittlement and annealing temperature is assumed. It should be noted, however, that the material composition of these specimens did not meet AMS specifications for Inconel 718.

The hydrogen embrittlement exhibited by the unirradiated Inconel 718 specimens in the present study was thus approximately that expected for specimens with a 1950°F annealing temperature. However, the irradiated specimens, with identical material composition and heat treatment, were unaffected by 1500-psi hydrogen. The two possible explanations for this phenomenon are corrosion and radiation damage.

The irradiated Inconel 718 specimens exhibited severe corrosion in localized areas. Since this material is corrosion-resistant, it has been postulated that the corrosion was due to

surface contamination of the specimens. However, since most theories of mechanisms of hydrogen embrittlement espouse hydrogen interactions at fresh metallic surfaces formed during stress, inhibition of embrittlement by outer surface contamination is difficult to understand.

Neutron irradiation is known to produce effects in alloys which are similar to metallurgical techniques for manipulation of their properties, i.e., cold-working, solution-hardening, quenching, and heat treatment. In particular, similarities have been observed in precipitation-hardened alloys between radiation effects and heat treating at temperatures below normal heat-treating temperatures.

Since the degree of embrittlement of Inconel 718 appears to be related to solution-annealing temperature, neutron irradiation may produce similar effects in the material which inhibit hydrogen embrittlement. However, an acceptable explanation for the lack of embrittlement in these specimens can only be furnished by further testing.

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