EFFECT OF CRYOGENIC IRRADIATION ON NERVA STRUCTURAL ALLOYS

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ABSTRACT*

Several alloys (Hastelloy X, AISI 347, A-286 bolts, Inconel 718, Al 7039-T63 and Ti-5Al-2.5Sn ELI) were irradiated in liquid nitrogen (140°R) to neutron fluences between 10^{17} and 10^{19} nvt (E > 1.0 Mev). After irradiation, tensile properties were obtained in liquid nitrogen without permitting any warmup except for some specimens which were annealed at 540°R. The usual trend of radiation damage typical for materials irradiated at and above room temperature was observed; e.g., increase in strength and decrease in ductility. However, the damage at 140°R was greater because this temperature prevented the annealing of radiation-induced defects which occurs above 140°R. The more significant conclusions from these tests are:

(1) The threshold for measurable mechanical property damage in these materials at 140°R is between 10^{16} and 10^{17} nvt (E > 1.0 Mev).

(2) Room temperature annealing of tensile properties was performed prior to testing some of the alloys. Al 7039-T63 recovers about half the damage incurred at the highest exposure while the other alloys recover a smaller amount. Previous similar studies of Al 2219 showed almost complete recovery after annealing at room temperature. Aluminum components in the NERVA engine will be at temperatures above 140°R during its coast periods. Additional postradiation annealing studies are required to establish design properties after thermal cycling to various temperatures.

The high reliability requirements for the NERVA engine make it imperative that candidate material properties be determined in the particular temperature, nuclear radiation or other possible detrimental environment to which the material may be subjected. This test involves the effects of fast neutron fluences up to 10^{19} nvt on the embrittlement of several alloys (Hastelloy X, AISI 347, A-286 bolts, Inconel 718, Al 7039-T63, and Ti-5Al-2.5Sn ELI). Previous investigations in this program have shown that the nuclear radiation exposure of structural materials in the NERVA engine can impose limitations on design if ductility losses are severe. Quantitative data were obtained on engineering alloys irradiated at cryogenic temperatures at higher fast neutron fluences than in previous tests. The alloys were irradiated** in liquid nitrogen (140°R) and tensile tested at 140°R without an intermediate warmup except for selected specimens which were annealed at 540°R. The test data for each alloy are presented in tables and engineering

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**This irradiation test was performed at the Ground Test Reactor of the Nuclear Aerospace Research Facility, General Dynamics, Fort Worth. These results are part of a larger test designated GTR-20C.

judgements are made of whether the design classification for these materials should be "brittle" or "ductile"* in the anticipated NERVA nuclear environment.

Hastelloy X (As-Brazed) Tensile Data (Table 1)

This material was a plate forging made from a low aluminum (0.012%) heat. It was subjected to three thermal cycles to about 1950°F and slow cooled simulating the NERVA nozzle processing heat treatment. The maximum exposure of 5 x 10^{18} nvt > 1.0 Mev is lower than the 1 x 10^{19} nvt anticipated for NERVA 10-hour service. However, the data shown in Table 1 indicate that extrapolation of properties to 10^{19} nvt can be made with confidence. The elongation decrease from 28% to 14% is significant, but the material is still considered ductile at 140°R after 10^{19} nvt > 1.0 Mev neutron fluence. The relatively large (104%) increase in tensile yield strength suggests this is a good material to use for more basic studies of radiation damage. It has been chosen for studies which will make quantitative investigations of the effects of irradiation temperature and irradiation atmosphere (GH_2 versus LN_2). The annealing studies have shown less damage recovery at 540°R than was anticipated from earlier studies made on materials irradiated to 1 x 10^{18} nvt.

AISI 347 Forging (As-Brazed) Tensile Data (Table 2)

This material was obtained from the NERVA technology nozzle S/N 033. The manufacturing processes included three thermal cycles to about 1800°F followed by a slow cool which slightly degraded the ductility at cryogenic temperatures; however, the unirradiated elongation of 52% is excellent, and it is considered a tough, ductile material. Its response to cryogenic irradiation is the most unusual in this group of alloys. After a fluence of 4.8 x 10^{18} nvt > 1.0 Mev, the tensile yield stress increased from 45 to 122 ksi (170%), but the elongation, area reduction and ultimate strength were not significantly changed. This alloy undergoes a strain-induced martensitic transformation during cryogenic tensile testing. The major part of the strain hardening is attributed to this phase transformation which appears to be insensitive to prior irradiation. AISI 347 is judged to remain ductile at cryogenic temperatures after a neutron fluence of 10^{19} nvt > 1.0 Mev.

A286 Technology Bolts Tensile Data (Table 3)

These bolts designed for the NERVA technology engines were cold-reduced and aged by proprietary processes to have a room temperature minimum yield and ultimate strengths of 180 and 200 ksi, respectively. The average elongation decreased from 16 to 10 percent after a neutron fluence of 6.5 x 10^{18} nvt; however, the area reduction remained unchanged at about 35%. This excellent area reduction implies good fracture toughness. These bolts are ductile at 140°R after a neutron fluence of 1 x 10^{19} nvt > 1.0 Mev.

Inconel 718 Tensile Data (Table 4)

These specimens from a large pancake forging after irradiation at 140°R to 4.4 x 10^{18} nvt > 1.0 Mev decreased their 140°R elongation from 23 to 12%, however, the area reduction did not decrease, remaining at about 30%. Inconel 718 is ductile in this cryogenic nuclear environment. A greater fraction of the damage annealed out in 100 minutes at 540°R than the other alloys except for aluminum alloy 7039-T63.

Alloy 7039-T63 Tensile Data (Table 5)

This material was obtained from a ring forging which was processed similarly to that anticipated

*The allowable stress for a "brittle" material may be less than the tensile yield strength depending upon the maximum flaw size which could be present and is determined by linear elastic fracture mechanics data and analysis. The allowable stress for a "ductile" material can be greater than the tensile yield strength at points of high stress concentration. A more detailed specification of the "ductile" or "brittle" classification as used here may be found in SNPO-C-1, "NERVA Program Structural Design Requirements".

See Appendix for the guidelines used in this program for establishing tentative ductile-brittle classifications.

for the NERVA pressure vessel. The average elongation at 140°R decreased from 12 to 5% after a neutron fluence of 5.5×10^{18} nvt > 1.0 Mev at 140°R. This material is tentatively classified brittle uder these conditions based on low elongation; however, when fracture toughness data are completed, the judgement of "ductile or brittle" according to SNPO-C-1 can be made with more confidence. After annealing for 10 minutes and longer at 540°R, the elongation damage almost completely recovered, but 50% of the increase in yield strength remained. Previous annealing studies in AA 2219-T6 had shown almost 100% of the radiation damage in all properties annealed out after a 540°R anneal. Thus, it appears that while damage annealing behavior is similar in aluminum alloys, it is not identical and extrapolation between alloy families must be made with caution.

Ti-5Al-2.5Sn ELI Tensile Data (Table 6)

These tensile specimens were fabricated from a large forging (17 in. diameter by 10 in.). The average 140°R elongation decreased from 10 to 4% after a neutron fluence of 4.9×10^{18} nvt > 1.0 Mev. This material is judged to be brittle under these conditions based on low elongation. The NERVA application for this material involves a less severe exposure (1×10^{17} nvt > 1.0 Mev) than the maximum used in this test. For NERVA applications at 10^{17} nvt, this titanium alloy is judged to be ductile. This judgement is supported by preliminary fracture toughness tests which show no decrease in 140°R plane-strain fracture toughness, K_{Ic}, after irradiation to 3×10^{17} nvt.

CONCLUSIONS

1. A neutron fluence of 5×10^{18} nvt > 1.0 Mev at 140°R does not embrittle Hastelloy X, AISI 347 or A286 (cold-worked plus aged bolts) sufficiently to change their design classifications of ductile.

2. The same neutron fluence (5×10^{18} nvt > 1.0 Mev) at 140°R degrades the ductility of Al 7039-T63 and Ti-5Al-2.5Sn enough to require a brittle classification and different design criteria for this neutron fluence at 140°R. A lower neutron fluence of 10^{17} nvt does not degrade the ductility of these two alloys significantly.

3. Annealing experiments at 540°R following irradiation at 140°R show various amounts of improvement in these alloys of the radiation damage to ductility. Al 7039-T63 completely recovers its ductility loss after annealing at 540°R.

TABLE 1

EFFECT OF FAST NEUTRON IRRADIATION
AT 140°R ON THE TENSILE PROPERTIES
OF LOW AL (0.012%) HASTELLOY X (AS-BRAZED)
FORGING AT 140°R

FLUENCE NVT > 1 MEV	ANNEAL TIME AT 540°R MIN.	0.2% YIELD STRENGTH, KSI		ULTIMATE STRENGTH, KSI		ELONGATION CHART, %		AREA REDUCTION, %		NO. SPECIMENS
		AVG	STD DEV	AVG	STD DEV	AVG	STD DEV	AVG	STD DEV	
Control	0	71.2	0.4	135.2	1.5	28.0	1.1	23.9	2.0	4
5.4×10^{17}	0	101.6	1.4	142.1	1.4	20.3	1.5	20.3	2.4	4
1.2×10^{18}	0	113.8	1.0	150.7	2.0	19.0	1.3	19.7	1.2	3
5.0×10^{18}	0	144.8	0.9	170.5	3.3	14.0	0.2	14.7	2.0	3
4.7×10^{18}	10	127.8	2.6	162.3	1.8	16.7	1.1	18.0	1.2	3
5.2×10^{18}	100	126.6	0.8	162.9	0.3	15.3	1.2	17.7	2.6	3
4.9×10^{18}	1000	124.4	2.8	161.7	2.8	17.8	1.2	17.5	2.1	3

TABLE 2

EFFECT OF FAST NEUTRON IRRADIATION
AT 140°R ON THE TENSILE PROPERTIES
OF AISI 347 (FORGING FROM NOZZLE S/N 033) AT 140°R

Control	0	45.4	2.0	176.8	1.5	51.6	6.0	46.0	13.9	4
6.2×10^{17}	0	68.0	0.8	177.2	1.1	50.2	5.1	43.6	11.1	4
1.2×10^{18}	0	86.0	4.4	178.6	0.8	50.0	4.7	40.6	9.9	4
5.8×10^{18}	0	121.9	2.2	179.4	1.3	52.4	3.3	49.0	11.5	3
5.2×10^{18}	10	106.9	6.2	180.2	0.0	51.0	3.1	50.2	7.3	3
4.7×10^{18}	100	109.8	1.4	183.1	4.2	51.7	2.6	43.9	10.8	3
5.0×10^{18}	1000	107.3	2.7	179.8	0.6	48.8	4.7	51.5	10.7	3

TABLE 3

EFFECT OF FAST NEUTRON IRRADIATION
AT 140°R ON THE TENSILE PROPERTIES
OF A-286 BOLTS (NERVA TECHNOLOGY) AT 140°R

Control	0	222.9	1.6	256.3	2.8	15.8	1.0	34.8	0.5	4
4.2×10^{17}	0	223.0	4.0	258.9	3.8	15.5	1.7	33.9	1.5	4
1.0×10^{18}	0	240.0	3.6	259.3	1.7	13.3	1.5	34.0	1.3	4
6.5×10^{18}	0	250.4	7.2	261.5	3.9	10.1	0.5	36.8	1.2	3
6.5×10^{18}	100	246.7	1.2	256.4	0.5	10.8	0.8	36.9	0.0	3
6.6×10^{18}	1000	243.3	1.9	254.8	0.5	11.1	0.8	36.1	2.3	3

TABLE 4

EFFECT OF FAST NEUTRON IRRADIATION AT 140°R ON THE TENSILE PROPERTIES OF INCONEL 718 (PANCAKE FORGING) AT 140°R

FLUENCE NVT > 1 MEV	ANNEAL TIME AT 540°R MIN.	0.2% YIELD STRENGTH, KSI		ULTIMATE STRENGTH, KSI		ELONGATION CHART, %		AREA REDUCTION, %		NO. SPECIMENS
		AVG	STD DEV	AVG	STD DEV	AVG	STD DEV	AVG	STD DEV	
Control	0	197.1	1.5	244.4	0.7	22.9	1.5	33.5	3.4	4
2.9×10^{17}	0	206.4	2.2	244.6	2.2	19.4	3.9	28.1	7.2	4
4.3×10^{18}	0	233.6	1.0	248.6	1.7	12.3	2.6	30.6	3.1	4
4.1×10^{18}	100	214.7	1.8	241.6	1.5	19.9	1.8	28.6	3.5	4

TABLE 5

EFFECT OF FAST NEUTRON IRRADIATION AT 140°R ON THE TENSILE PROPERTIES OF AA 7039-T63 (RING FORGING) AT 140°R

FLUENCE NVT > 1 MEV	ANNEAL TIME & TEMP. MIN., °R	0.2% YIELD STRENGTH, KSI		ULTIMATE STRENGTH, KSI		ELONGATION CHART, %		AREA REDUCTION, %		NO. SPECIMENS
		AVG	STD DEV	AVG	STD DEV	AVG	STD DEV	AVG	STD DEV	
Control	0	76.5	1.8	91.2	1.3	12.4	2.0	19.6	1.2	4
4.2×10^{17}	0	85.0	1.1	92.1	1.0	11.7	0.2	20.6	2.1	4
8.6×10^{17}	0	85.8	1.1	91.5	1.0	11.0	0.8	22.2	2.7	4
8.5×10^{17}	1000, 540	79.1	1.6	92.0	1.3	13.2	1.1	20.2	2.8	3
5.5×10^{18}	0	94.9	1.6	95.0	1.5	4.9	0.4	27.5	2.9	3
5.9×10^{18}	1000, 340	89.9	0.3	89.9	0.3	9.0	1.2	26.9	1.9	3
5.9×10^{18}	10, 540	85.0	1.2	90.4	1.1	11.3	0.7	23.6	2.1	3
5.6×10^{18}	100, 540	85.5	0.8	90.6	0.8	12.2	1.6	22.3	2.7	3
6.0×10^{18}	1000, 540	83.1	0.9	92.1	0.6	11.3	1.1	19.1	4.8	3

TABLE 6

EFFECT OF FAST NEUTRON IRRADIATION AT 140°R ON THE TENSILE PROPERTIES OF A LARGE Ti-5Al-2.5Sn (ELI) FORGING AT 140°R

FLUENCE NVT > 1 MEV	ANNEAL TIME AT 540°R MIN.	0.2% YIELD STRENGTH, KSI		ULTIMATE STRENGTH, KSI		ELONGATION CHART, %		AREA REDUCTION, %		NO. SPECIMENS
		AVG	STD DEV	AVG	STD DEV	AVG	STD DEV	AVG	STD DEV	
Control	0	175.6	1.5	185.8	2.1	10.4	2.3	29.9	3.2	4
2.3×10^{17}	0	182.4	1.8	189.5	1.1	9.1	0.9	30.4	2.6	4
8.2×10^{17}	0	187.7	1.5	195.5	1.7	7.7	1.1	29.5	5.0	4
4.9×10^{18}	0	199.1	3.3	204.4	3.1	4.1	0.5	23.2	3.4	4
5.2×10^{18}	100	192.7	1.9	200.0	1.0	6.3	0.4	27.0	1.5	4

APPENDIX*

DUCTILE-BRITTLE CLASSIFICATION

Tentative ductile-brittle classifications of reactor and engine system candidate materials have been established for design utilization, applicable to the temperature regime anticipated for NERVA operation. Factors such as form, condition, and direction of the material, as well as exposure to irradiation, gaseous and liquid hydrogen, and cryogenic temperature environments, are considered for each classification.

"Ductile" classification is based upon conservative judgement where data are available from NERVA experience or literature to substantiate the decision for all anticipated NERVA environments. "Brittle" classification is also based upon conservative judgement for the established brittle materials and, in addition, includes all materials with marginal or suspect ductility that could possibly be upgraded to "ductile" by further testing and/or experience.

Judgement is exercised by perusal of all pertinent information available and includes (but is not limited to) prior NERVA experience; available tensile ductility and notch tensile data; available fracture toughness data; and professional metallurgical expertise. This expertise is especially applied for anticipated deleterious effects of irradiation damage, high-pressure gaseous hydrogen embrittlement, cryogenic temperatures, and their combined synergistic effects.

The classifications for engine materials and reactor materials apply as the required design criterion in accordance with Paragraph 3.9 of the governing Structural Specification, SNPO-C-1. However, both the classifications and the associated temperature ranges are subject to change as new test data become available. Statistical fracture toughness testing would be required for all materials utilized for design which are classified "brittle".

*ANSC Report 2275, "Materials Properties Data Book," 15 November 1970 Supplement