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Fracture Toughness Characterization of 304L and 316L Austenitic Stainless Steels and Alloy 718 After Irradiation in High-Energy, Mixed Proton/Neutron Spectrum

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Abstract: This paper describes the fracture toughness characterization of annealed 304L and 316L stainless steels and precipitation hardened Alloy 718, performed at the Oak Ridge National Laboratory as a part of the experimental design and development for the Accelerator Production of Tritium (APT) target/blanket system. Materials were irradiated at 25 to 200°C by high-energy protons and neutrons from an 800-MeV, 1-mA proton beam at the Los Alamos Neutron Science Center (LANSCE). The proton flux produced in LANSCE is nearly prototypic of anticipated conditions for significant portions of the APT target/blanket system. The objective of this testing program was to determine the change in crack-extension resistance of candidate APT materials from irradiation at prototypic APT temperatures and proton and neutron fluxes. J-integral-resistance (J-R) curve toughness tests were conducted in general accordance with the American Society for Testing and Materials Standard Test Method for Measurement of Fracture Toughness, E 1820-99, with a computer-controlled test and data acquisition system. J-R curves were obtained from subsize disk-shaped compact tension specimens (12.5 mm in diameter) with thicknesses of 4 mm or 2 mm. Irradiation up to 12 dpa significantly reduced the fracture toughness of these materials. Alloy 718 had the lowest fracture toughness in both the unirradiated and irradiated conditions. All irradiated specimens of Alloy 718 failed by sudden unstable crack extension regardless of dose or test temperature. Type 304L and 316L stainless steels had a high level of fracture toughness in the unirradiated condition and exhibited reduction in fracture toughness to saturation levels of 65 to 100 MPa \sqrt{m} . The present reduction in fracture toughness is

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similar to changes reported from fission reactor studies. However, the currently observed losses in toughness appear to saturate at doses slightly lower than the dose required for saturation in reactor-irradiated steels. This difference might be attributed to the increased helium and hydrogen production associated with irradiation in the high-energy, mixed proton/neutron spectrum.

Keywords: stainless steel, spallation neutron source, fracture toughness

Introduction

This paper describes the fracture toughness characterization of 304L and 316L stainless steels, and nickel base Alloy 718 performed at the Oak Ridge National Laboratory (ORNL) as a part of the Accelerator Production of Tritium (APT) Project. The APT facility will use an accelerator to provide a spallation neutron source capable of producing tritium (^3H) through the $^3\text{He}(n,p)^3\text{H}$ reaction. Consequently, the target/blanket (T/B) components of the APT system will be exposed to unique conditions, including a high-energy mixed proton and neutron spectra. Only a limited data base is available for material applications in such an environment. Thus, a comprehensive material testing program was undertaken within the APT Project. This program includes the irradiation of candidate APT T/B system component materials at 25 to 200°C by exposure to high-energy protons and neutrons at the Los Alamos Neutron Science Center (LANSCE) at 800 MeV. The proton flux produced in LANSCE is nearly prototypic of anticipated conditions for significant portions of the APT T/B system.

Background

The APT Project will produce tritium through the $^3\text{He}(n,p)^3\text{H}$ reaction. The ^3He used for ^3H production will be contained, at approximately 120 psi, in aluminum alloy tubes that will be connected to a $^3\text{H}/^3\text{He}$ separation facility through a Type 316L stainless steel manifold/piping system. Neutrons for the $^3\text{He}(n,p)^3\text{H}$ reaction will be produced by proton induced spallation of a tungsten target. Lead blanket assemblies will moderate and multiply the spallation neutrons. The high-energy proton beam that induces spallation in the tungsten target will move from the accelerator portion of the APT system into the T/B system by passing through an Alloy 718 window. Design concepts for the target/blanket system are summarized in Ref. [1] and include cladding of the tungsten target elements and containing these elements and the target cooling water with Alloy 718 and providing containment of the lead blanket components with 6061-T6 aluminum extrusions and tubing. The containment vessel for the T/B system will be fabricated from Type 304L stainless steel, and Type 316L stainless steel will be used to fabricate much of the primary cooling water system (internals). The materials irradiation program developed to support the emerging T/B design is outlined in Ref. [2] and, for convenience, is summarized in the next few paragraphs.

Models of the tungsten neutron source have been irradiated with 800 MeV protons at power levels prototypic of the power levels anticipated in the APT target. The proton beam had a Gaussian-like intensity profile with a diameter of approximately

$2\sigma = 3.0$ mm, where σ is the standard deviation. The beam profile has two basic effects on the irradiation and specimen design:

1. The specimen had to be small to assure reasonable uniformity in dose within a single sample, and
2. Placement of several specimens within a sample capsule provided the opportunity to determine the effects of dose on a given material.

The specimen also had to be relatively thin, 0.25 to 2.0 mm range, to assure that the energy deposited by the 800-MeV proton beam was properly transferred to the coolant. Details of irradiation procedures and temperature and dose calculations are described in Refs. [3,4].

Materials Specifications and Heat treatment

Certified mill test reports were obtained for each alloy; the chemical composition, from these test reports, for each material is summarized in Table 1.

Alloy 718 specimens were machined from the as-received material (annealed condition), wrapped in Nb foil and encapsulated in a quartz tube (evacuated and back-filled with Ar). The encapsulated specimens were heat treated through the following steps:

1. Solution anneal at 1065°C for 30 minutes and air cool,
2. Age at 760°C for 10 hours,
3. Furnace cooled from 760 to 650°C and hold for a total aging time of 20 hours,
4. Air cool to room temperature.

The 316L and 304L stainless steel samples were electrodischarge machined (EDM) from as-received (annealed) material and irradiated in the as-machined and cleaned condition and tested after irradiation.

Testing Procedure

The J-integral-resistance (J-R) curve toughness tests were conducted in general accordance with the American Society for Testing and Materials (ASTM) E 1820-99 [5] Standard Test Method for Measurement of Fracture Toughness, with a computer-controlled test and data acquisition system [6]. In many cases, selection of a specimen design for an irradiation study is a compromise between a desire to test large size specimens to satisfy rigorous validity requirements of the ASTM standard, and necessity to use small specimens due to a variety of limitations caused by imposed irradiation conditions, such as restricted capsule space, difficulty of proper heat removal from large volumes of metal, etc. This APT irradiation project was no exception. As a result of many considerations, disk-shaped compact tension [DC(T)] specimens, 12.5 mm in diameter with thicknesses of 4 mm (0.16T) and 2 mm (0.08T), were selected for this study to develop J-R curve. This small DC(T) specimen has been previously developed at ORNL [7] for testing of irradiated materials. Available experience suggested that even such small specimens could provide valuable information on effects of irradiation on a material's ability to resist crack extension, particularly if the specimen thickness matches the component thickness. It was also anticipated that the minimum thickness requirement

would decrease for irradiated materials because the yield strength tends to increase and the fracture toughness decreases with irradiation. A thickness of 2 mm was chosen for the compact specimens for the irradiation experiments in the proton beam so that specimens could be cooled to the prototypical irradiation temperatures. Coincidentally, the 2 mm thickness was similar to the thickness for some of the materials used in the T/B system. To evaluate a potential dependence of fracture toughness on thickness, J-R curve tests in the unirradiated condition were performed with both 2- and 4-mm-thick specimens. Only a limited number of 4-mm-thick specimens were irradiated.

The specimens were fatigue precracked before irradiation to a ratio of the crack length to specimen width (a/W) of about 0.5, and then side-grooved by 20% (20% SG) of their thickness (10% from each side). The unloading compliance method used for measuring the J-integral using these specimens is outlined in Ref. [7]. Tests were conducted at room temperature, 50, 80, and 164°C. The temperatures were maintained within $\pm 2^\circ\text{C}$ during the tests. Unirradiated specimens were tested in the laboratory on a 98-kN (22-kip) capacity servohydraulic machine, and irradiated specimens were tested in a hot cell with a 490-kN (110-kip) capacity servohydraulic machine with a 22-kN (5-kip) load cell. All tests were conducted in strain control, with an outboard clip gage having a central flexural beam that was instrumented with four strain gages in a full-bridge configuration. After testing, the crack front was marked by cyclically loading the specimen at room temperature. The specimens were then broken open. The unirradiated specimens were examined with a calibrated measuring optical microscope to determine the initial and final crack lengths. The irradiated specimens were photographed, and enlarged prints of the fracture surfaces were fastened to a digitizing tablet to allow the crack length to be measured. Finally, the J-integral was determined and plotted against crack extension using the load/displacement data. From this plot, the provisional critical J-integral, J_Q , was determined as the intersection of the power law regression line with the 0.2-mm crack extension offset line. J_Q values were converted to their equivalent values in terms of stress intensity K_{JQ} :

$$K_{J_Q} = \sqrt{EJ_Q} \quad (1)$$

where E is Young's modulus. These K_{JQ} values characterize the toughness of materials near the onset of crack extension. The ASTM standard test method E1820-99 [5] specifies numerous requirements for qualifying the J-R curve and for qualifying J_Q as the critical J-integral at the onset of stable crack extension, J_{Ic} . In general, those requirements can be separated into two categories. The first category contains the requirements that are common for all tests. It includes requirements on the test equipment, machining tolerance, fixture alignment, test rate, temperature stability, rectilinearity of the original and final crack fronts, crack extension prediction, etc. Despite some difficulties with the use of small size specimens (and especially in the irradiated condition), this category of requirements was met in all tests. The second category of requirements is related to the measuring capacity of the specimen. The standard specifies the validity box on the J-R curve, the upper boundary for which is defined by the maximum J-integral capacity for a specimen by the smaller of the following:

$$J_{\max} = \frac{b_o \sigma_Y}{20}, \quad \text{or} \quad J_{\max} = \frac{B\sigma_Y}{20} \quad (2)$$

where b_0 is the original remaining ligament and B is the specimen thickness. The boundary on the right side of the validity box is defined by the maximum crack extension capacity:

$$\Delta a_{\max} = 0.25b_0 \quad (3)$$

For the specimens in the present study, the nominal value of b_0 is about 4.6 mm. Thus, J_{\max} is limited by the specimen thickness, while the nominal value of Δa_{\max} is about 1.2 mm. Due to the small size of the specimens, this requirement category was not met in the present study.

Results

Type 304L and 316L Stainless Steels

Both alloys exhibit very high toughness in the unirradiated condition. However, the 4-mm-thick specimens of both alloys exhibit slightly higher (about 10 to 15%) toughness than the 2-mm-thick specimens (See Figs. 1 and 2). Figure 3 typifies the positioning of the J-R curve relative to the validity box for a 4-mm-thick specimen in the unirradiated condition for the stainless steels in the present study. The J-R curve for both stainless steels derived from either thickness of specimen cannot be qualified as an E1820 valid curve. Consequently, the J_Q value, denoted by a triangle on Fig. 3, cannot be qualified as J_{IC} . Such deviation of the J-R curve from the validity box, as shown in Fig. 3 is responsible (at least in part) for the observed size effects on J_Q .

Two 4-mm-thick and two 2-mm-thick specimens of each material were irradiated to a relatively low dose (~0.1 dpa). Although irradiation slightly reduced the fracture toughness of each alloy, the relationship between fracture toughness values derived from 2- and 4-mm-thick specimens remained approximately the same.

It was anticipated that any size dependence would have a propensity to diminish with dose since irradiation reduces the J-integral values of these steels and increases the yield strength resulting in an increase in the J_{\max} value, i.e., the difference between the J_Q values measured on 2 and 4 mm thick specimens after irradiation should be less than the difference measured in the unirradiated state. Figure 4 shows the J-R curve derived from testing a 2-mm-thick DC(T) specimen of 304L stainless steel irradiated up to 7.2 dpa and tested at 164°C. The J-R curve for this specimen is still outside the validity box; however, it is much closer to the box than for the unirradiated specimens.

Summarizing the size-related observations in the present study for the stainless steels, the following can be concluded. The K_{JQ} values measured from 2-mm-thick specimens are slightly less (220 to 250 MPa \sqrt{m}) than those obtained from 4-mm-thick specimens (270 to 280 MPa \sqrt{m}) for unirradiated material tested at room temperature. The observed size dependence is consistent with literature results for type 304 and 316 stainless steels in Ref. [8]. According to a statistical analysis of those fracture toughness results [8], valid K_{JIC} values derived from large specimens should be about 360 MPa \sqrt{m} on average. Thus, the K_{JQ} values obtained from small specimens in this project are conservative relative to the valid, size-independent values. Therefore, the data generated in the present study can be recommended for initial design calculations, especially for the

end-of-life conditions. This size dependence has a propensity to remain similar in nature for both stainless steels but to diminish in magnitude with irradiation dose because the J-R curves for the irradiated specimens become much closer to the validity box than the J-R curves for the unirradiated specimens.

The 4-mm-thick DC(T) specimens of both stainless steels were tested at room temperature, 50 and 164°C. The toughness decreases slightly from ~275 MPa√m to ~210 MPa√m as the test temperature was increased from 25 to 164°C (Fig. 5), which is a common temperature dependence for the critical J-integral for these type of stainless steels.

Specimens were irradiated to a wide range of doses from 0.1 dpa to approximately 7 to 9 dpa. This irradiation resulted in significant decreases in the toughness of both alloys, although appreciable levels of toughness (~65 to 100 MPa√m) were still retained (Figs. 6 and 7). Even after a dose of only 0.1 dpa, fracture toughness decreased more than 25% (see Figs. 6 and 7). The current data indicate that the decrease in the fracture toughness of 304L stainless steel reaches a lower plateau after dose of 3 to 4 dpa at a level of about 100 MPa√m. On the other hand, the fracture toughness of 316L stainless steel continues to decrease until it saturates at a level of approximately 60 to 70 MPa√m. However, another important observation is that at doses above 4 dpa 316L stainless steel has a tendency to intermittently lose resistance to stable crack growth. Specimens tested after doses of 4.6, 9.3, and 9.4 dpa exhibited local ductile instability (fast crack propagation in a ductile mode for a constant loading rate) after achieving the maximum load. Figure 8 illustrates the load-displacement traces of specimens J4 and J20. Specimen J4 was irradiated up to 9.3 dpa and exhibited ductile instability, while specimen J20 was irradiated up to 3.11 dpa and demonstrated a typical load-displacement trace for a specimen exhibiting stable crack growth. Examination of the fracture surfaces of some of the irradiated 316L specimens using a scanning electron microscope (SEM) Philips XL30 did not reveal anything other than ductile tearing modes of crack extension. Figure 9 is a SEM image of the fracture surface of highly irradiated (9.3 dpa) specimen J4 and it shows only ductile dimples. The fact that the crack would propagate for a short distance in an unstable fashion and then arrest, resume propagation in a stable fashion and then repeat the process, may indicate that after irradiation at low temperature in the high energy, mixed proton/neutron spectrum to doses above 4.5 dpa, 316L steel has a propensity to form local zones with very low resistance to stable ductile crack growth.

There is a small amount of fracture toughness data available regarding the behavior of similar austenitic steels after fission neutron irradiation at such low irradiation temperatures. The present data showed that fracture toughness saturates at about the same toughness level as that for material after irradiation in a fission reactor. It appears, however, that the fracture toughness values at low doses for similar, fission neutron-irradiated austenitic stainless steels [8] decrease at a slower rate with dose than that observed in the present study. Taking into account that the helium and hydrogen production rates are extremely high under spallation conditions, this difference is consistent with the observation that both hydrogen and hydrogen/helium accumulation lower the room temperature fracture toughness of austenitic stainless steels [9]. However due to very limited data available for low temperature fission reactor irradiation of these steels, additional microstructural studies, as well as direct experiments with fission

reactor irradiation of the same materials at low temperatures are needed to confirm this statement.

Alloy 718

The crack-extension resistance of the precipitation-hardened Alloy 718 is much different from that of the stainless steels. All of the 4-mm-thick specimens failed by sudden, unstable crack extension. The unirradiated 2-mm-thick specimens have an initiation toughness similar to those of the 4-mm-thick specimens, but show more stable crack extension (with some elements of short term instabilities) than the 4-mm-thick specimens (See Fig. 10). After irradiation, only one 2-mm-thick specimen, E14, irradiated to the lowest dose of ~0.1 dpa showed some stable crack extension. All other irradiated specimens failed by sudden unstable crack extension regardless of the dose. For material exhibiting such crack-extension resistance, the J-integral value which corresponds to the point of failure by sudden unstable crack extension, J_c , is reported as the critical J-integral value. For a limited number of 2-mm-thick specimens that demonstrated a mixture of stable and unstable crack propagation, the J-integral at the point of maximum load, J_{pmax} , is reported as a conservative estimate of the onset of stable crack extension.

In the unirradiated condition, the Alloy 718 exhibited moderate toughness (~120 to 160 MPa \sqrt{m}) at test temperatures from 25 to 164°C (See Fig. 11). Despite the scatter in the data at 50°C, there is a slight decrease in toughness as the test temperature increases (See Fig. 11).

The fracture toughness of Alloy 718 decreases steadily after irradiation up to ~ 4 to 5 dpa. After that, the fracture toughness saturates and the dose dependence diminishes (See Fig. 12). Specimens tested after irradiation up to ~ 12 dpa showed fracture toughness values of about 50 MPa \sqrt{m} . This reduction in fracture toughness is similar to that reported from fission neutron reactor studies [10,11]. However, the neutron irradiation-induced decreases in toughness of heat-treated Alloy 718 after irradiation in fission reactors appeared to saturate after about 8-10 dpa [10,11]. The present data reveal that saturation after mixed proton/neutron beam irradiation occurs at smaller doses than that observed after fission neutron-irradiated Alloy 718. This trend is also similar to that observed for stainless steels in the present study.

SEM images (Fig. 13a,b) of specimen E9 did not reveal any stable ductile crack extension prior to brittle instability. This specimen was irradiated to the highest dose, 12.35 dpa. A higher magnification image (Fig. 13b) suggests intergranular fracture as the main mode of crack propagation for this specimen.

Summary

The data presented in this paper summarize the fracture toughness results obtained for type 304L and 316L stainless steels and Alloy 718 after exposure in an environment typical of that for the spallation neutron source proposed for the Accelerator Production of Tritium (APT) Project. These materials were tested in the unirradiated condition and after irradiation at 25 to 200°C by high-energy protons and neutrons at the Los Alamos

Neutron Science Center (LANSCE) to doses up to ~12 dpa. Subsize disk-shaped compact tension specimens, 12.5 mm in diameter with thicknesses of 4 mm or 2 mm, were used for fracture toughness characterization of these materials. It is shown that the smallest specimen geometry provides a conservative estimate of J-integral values at the onset of stable crack extension.

Both 304L and 316L stainless steels exhibited very high toughness in the unirradiated condition. Specimens were irradiated in a wide range of doses from 0.1 up to approximately 7 to 9 dpa. This irradiation resulted in significant decreases in the toughness of both alloys, although appreciable levels of toughness (~65 to 100 MPa√m) were retained. It was also observed that at doses above 4 dpa 316L stainless steel has a tendency to lose resistance to stable crack growth.

Precipitation-hardened Alloy 718 exhibited intermediate toughness (~120 to 160 MPa√m) in the unirradiated condition. This material exhibits a propensity toward unstable crack propagation once tearing is initiated. This phenomenon becomes more pronounced after irradiation. The fracture toughness of Alloy 718 decreases steadily with dose up to ~4 to 5 dpa. After that, the fracture toughness has a tendency to saturate at levels of about 50 MPa√m. The SEM examination of a fracture surface of one of the highly irradiated specimens revealed a brittle intergranular mode of crack propagation without any evidence of stable crack growth.

The data presented in this paper demonstrate that the available data base of radiation effects on the fracture toughness of structural metals and alloys exposed to fission reactor environments provides useful indicators of the anticipated trends for property/dose predictions in spallation neutron environments. However, the enhanced gas production associated with the high-energy proton/neutron spectra in spallation neutron sources may cause enhanced degradation of fracture toughness at low doses. Hydrogen production, and a susceptibility to hydrogen embrittlement, may play key roles in the low temperature, low dose degradation. Clearly, some additional microstructural studies and direct experiments with low temperature fission reactor irradiation of these materials are essential to clarify this observation.

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Table 1. Chemical composition (wt %) of materials tested

Material	Al	C	Cr	Cu	Fe	Mn	Mo	Ni	P	S	Si	Ti	Others
Alloy 718	0.47	0.04	17.71	0.15	BAL	0.12	3.00	54.79	0.013	0.001	0.13	0.98	Nb and Ta-4.98; Co-0.19; B-0.002
316L	n/a*	0.010	17.33	0.18	BAL	1.61	2.09	10.62	0.024	0.019	0.43	n/a	Co-0.21; N-0.060
304L	n/a	0.013	18.15	0.23	BAL	1.80	0.18	8.35	0.025	0.010	0.43	n/a	Co-0.17, N-0.085
n/a* = non available.													