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Journal of Nuclear Materials 343 (2005) 191–196

journal of nuclear materials

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The high temperature three point bend testing of proton irradiated 316L stainless steel and Mod 9Cr–1Mo

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Abstract

The predicted operating conditions for a lead–bismuth eutectic target to be used in an accelerator driven system for the Advanced Fuel Cycle Initiative span a temperature range of 300–600 °C while being irradiated by a high energy (∼600 MeV) proton beam. Such spallation conditions lead to high displacement rates coupled with high accumulation rates of helium and hydrogen up to 150 appm/dpa. Some candidate materials for these applications include Mod9Cr–1Mo and 316L stainless steel. To investigate the effect of irradiation on these materials, the mechanical properties are being measured through three point bend testing on Mod 9Cr–1Mo and 316L at 25, 250, 350 and 500 °C after irradiation in a high energy proton beam (500–800 MeV) to a dose of 9.8 dpa at temperatures from 200 to 320 °C. By comparing measurements made in bending to tensile measurements measured on identically irradiated materials, a measurement of 0.2% offset yield stress was obtained from 0.05% offset yield stress measured in three point bend testing. Yield stress increased by more than a factor of two after irradiation to 9.8 dpa. Observation of the outer fiber surface of 316L showed very localized deformation when tested after irradiation at 70 °C and deformation on multiple slip systems when tested after irradiation at 250–320 °C.

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1. Introduction

The Advanced Fuel Cycle Initiative is investigating different options for transmutation of high level nuclear waste. One of these options utilizes a high energy (∼600 MeV) proton accelerator to bombard a lead bismuth eutectic target to produce a high energy neutron flux for transmuting fuel [1]. The structural materials used to contain this target will experience extensive radiation damage at operating temperatures of 400–600 °C. In addition, because the protons and neutrons will have energies greater than 20 MeV, spallation will occur causing a buildup of helium and possibly hydrogen (if the irradiation temperature is low enough) in the materials while displacement damage accumulates. To quantify the effect this environment has on the mechanical

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0022-3115/$ - see front matter Published by Elsevier B.V.
doi:10.1016/j.jnucmat.2005.03.027

properties, materials are being irradiated under these prototypic conditions and the mechanical properties are being tested after irradiation.

Because there are limited facilities available for irradiating and testing these materials and because the high dose irradiation volumes are small, it is expensive to perform these studies and because structural materials become radioactive after irradiation, it is advantageous to use small-scale specimens that can be easily aligned and tested remotely in a hot cell. One testing technique which has the previously stated benefits is through the use of three point bending.

The American Standards for Testing of Materials (ASTM) denotes three standards which are relevant to three point bend testing. ASTM E190-92 and ASTM E290-97a relate to the use of standard scale three point bend specimens tested to large strains for investigating ductility while ASTM E855-90 applies three point bending at smaller strains for spring applications. In this study, ASTM E855-90, a Standard Test Method for Bend Testing of Metallic Flat Materials for Spring Applications Involving Static Loading, was used as it was most concerned with the materials properties up to the yield stress. Our specimens had the minimal dimensions of 8 mm × 2 mm × 0.25 mm thickness using a span of 5.5 mm which was slightly below the requirements in the standard which were a minimum thickness of 0.38 mm, ratio of span to thickness greater than 15, and ratio of width to thickness of greater than 10.

Thus, this paper presents the results of three point bend testing of 316L stainless steel at 25, 250 and 350 °C and Mod 9Cr–1Mo (a ferritic-martensitic steel) at 25, 250 and 500 °C. Where available these bend results are compared with tensile test results measured on tensile specimens of the same heat of material irradiated under identical condition to obtain a correlation between bend offset yield stress and 0.2% yield stress measured in tension.

2. Experimental details

Specimens of 316L (annealed) and Mod 9Cr–1Mo (heat treated 1038 °C/1 h, air cooling, 760 °C/1 h, air cooling) were tested after irradiation at the Los Alamos National Laboratory (LANL) and the Paul Scherrer Institute (PSI). The elemental composition (balance Fe) of the Mod 9Cr–1Mo (heat 10148 from ORNL) was 0.089C, 0.47Mn, 0.021P, 0.28Si, 0.16Ni, 9.24Cr, 0.96Mo, 0.21V, 0.054Nb, 0.08Co, 0.035N and that of 316L irradiated at PSI was 17.17Cr, 12.24Ni, 2.31Mo, 1.75Mn, 0.077Co, 0.07Cu, 0.019C, 0.35Si, 0.02P, 0.073N while the heat of 316L irradiated at LANL was 17.26Cr, 12.16Ni, 2.57Mo, 1.75Mn, 0.26Cu, 0.019C, 0.65Si, 0.022P, 0.006S. Irradiations at LANL were performed using the Los Alamos Radiation Effects Facility (LASREF) located at the end of the 800 MeV/1 mA proton beam [2]. These specimens were irradiated in the form of 2 mm × 8 mm × 0.25 mm thick three point bend specimens to a dose of 9 dpa at an irradiation temperature of 70 °C. Measured concentration of H was 3400 ± 210 appm and of He was 1979 ± 27 appm [3]. The surfaces were ground with 800 grit paper before testing. The materials irradiated at PSI were from the SINQ Target Irradiation Program I (STIP-I) and used the target at the end of the ~570 MeV, 850 μA SINQ accelerator [4]. Those tested at LANL were rods of Mod 9Cr–1Mo and 316L irradiated to a maximum dose of 9.8 dpa at temperatures between 200 and 320 °C. Measured concentration of He was 770 appm and H was practically none as it diffused out during irradiation [5]. To obtain mechanical properties in three point bending, specimens were sliced from the rods in the hot cells to obtain rectangular specimens with the dimensions of 2 mm × 8 mm × 0.25 mm. The surfaces of these specimens were ground with 800 grit paper before testing.

The three point bend tests were conducted on a custom-designed fixture that was placed in an Instron 5567 screw driven mechanical test stand. A schematic showing a cross-section of the fixture is shown in Fig. 1. The samples are bent in three point bending on supports spaced at 5.5 mm. Circular pins of radius 1.58 mm provided the upper and lower contact points. Specimens were tested at a constant crosshead speed to produce an equivalent initial strain rate of 10^{-3}/s in outer fiber. A tungsten element furnace was added to this mechanical test machine to allow testing in argon at temperatures up to 700 °C. To avoid sticking between the specimen and the supports, a lubrication was used made by LOCTITE (1001 Trout Brook Crossing, Rocky Hill, CT 06067-3910) called Heavy Duty Anti-seize, a graphite/calcium fluoride compound good for use up to 1300 °C. The tests provided a load-deflection curve for each sample. These were converted to a

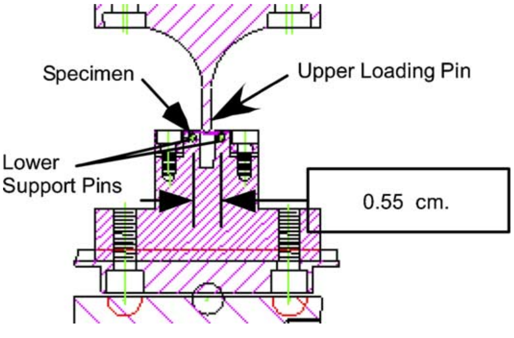

Fig. 1. Schematic showing the cross-section of the three point bend set-up used for testing materials at high temperatures.

stress–strain relationship using formulas found in ASTM E855-90.

$$\sigma = 1.5PL/bh^2 \quad \varepsilon = 6^*h^*\delta/L^2,$$

where L is the width of the supports (5.5 mm in this case), h is the thickness of the sample, b is the width, P is the applied load and δ is the measured deflection of the sample.

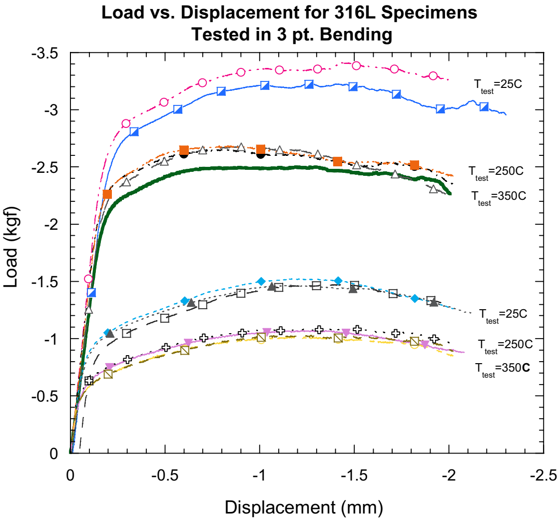

Fig. 2. Load vs. displacement obtained from three point bend testing of 0.27 mm thick annealed 316L stainless steel irradiated to 9.8 dpa at 250–320 °C.

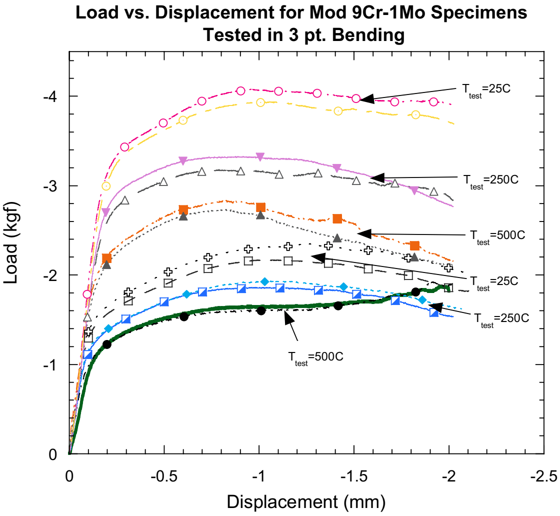

Fig. 3. Load vs. displacement obtained from three point bend testing of 0.27 mm thick Mod 9Cr–1Mo irradiated to 9.8 dpa at 200–260 °C.

3. Results

316L stainless steel was tested in three point bending at 25, 250 and 350 °C after irradiation to 9.8 dpa in the STIP irradiations at 250–320 °C. Mod 9Cr–1Mo was tested in three point bending at 25, 250 and 500 °C after irradiation to 9.8 dpa in the STIP I irradiation at 200–260 °C. Load vs. displacement curves are shown for 0.27 mm thick 316L and Mod 9Cr–1Mo in Figs. 2 and 3 respectively. Two specimens were tested under each condition showing good agreement between each test.

4. Discussion

In accordance with ASTM specification E855-90 certain elastic/plastic data may be obtained from load-displacement curves in three point bending including 0.05% offset yield, the elastic limit and the elastic modulus in bending. A typical example for calculating these values in 316L is shown in Fig. 4. In this case, for 316L tested at 25 °C, 0.05% offset yield = 360 MPa, elastic limit (bending) = 0.27% and elastic modulus (bending) = 111 GPa. For this testing, we measured 0.05% offset YS in bending to relate to 0.2% offset yield in tension.

To correlate the bend 0.05% offset yield with the tensile 0.2% offset yield stress in 316L stainless steel, three point bend specimens and tensile specimens were obtained from the same heat of material. Both of the two specimen types (tensile and bend) were irradiated at LANSCE to the same fluences (from 0 to 10 dpa) and at the same irradiation temperature. Next, the 0.2% offset yield stress that was measured in the tensile tests [6] was plotted against the 0.05% offset bend stress measured in bending (Fig. 5). By fitting this data to a

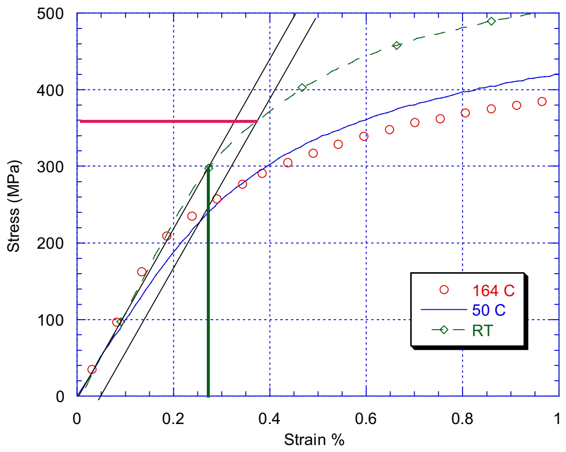

Fig. 4. Elastic stress vs. strain calculated for the outer fiber of 316L stainless steel tested at 25, 50 and 164 °C.

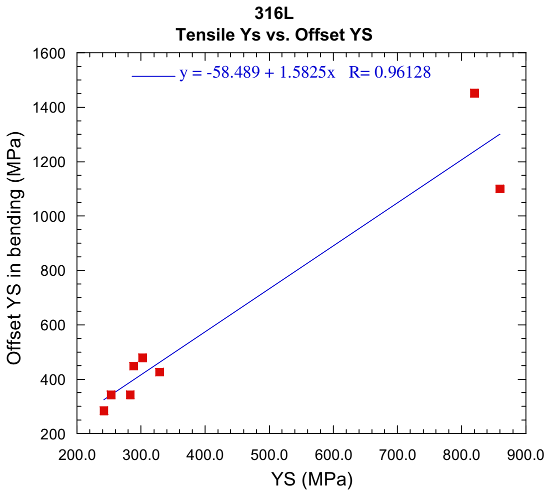

Fig. 5. 0.2% yield stress measured in tension plotted vs. 0.05% yield stress measured in bending for 316L stainless steel after irradiation in the 800 MeV proton beam at LASREF at 70 °C.

straight line, a correlation in 316L stainless steel was determined to relate the 0.2% offset yield stress measured in tension with the 0.05% yield stress measured in three point bending. The correlation obtained was

$$\begin{aligned}&0.05\%\ \text{offset yield(bend)}\\&\quad = -58.5 + 1.58(0.2\%\ \text{offset yield in tension}).\end{aligned}$$

Next, the 0.2% offset yield stress was calculated from the measurements of 0.05% offset bend stress on 316L stainless steel irradiated in STIP-1, see Table 1. To compare these predictions to data from other tensile tests, the predicted tensile 0.2% offset yield stresses obtained from the bending test was plotted along with the 0.2% offset yield stress that was measured on many tensile samples irradiated at LANSCE at 30–120 °C (Fig. 6). It is shown in this figure that the tensile yield stress predicted from the bend data follows the trends in the data measured in tensile tests providing initial justification for the use of a relatively simple and inexpensive yet quite accurate three-point bending methodology.

Table 1
The measured values of 0.05% offset yield stress used to predict 0.2% offset yield stress in 316L stainless steel after irradiation in STIP-1

Dose (dpa)	Temperature (°C)	0.05% offset yield stress in bending (MPa)	Calculated 0.2% offset tensile stress (MPa)
0	25	385, 361, 351	280, 265, 258
0	250	275	211
0	350	263, 248	203, 193
9.8	25	1286, 1274	850, 842
9.8	250	1165, 1218	773, 807
9.8	350	1025	684

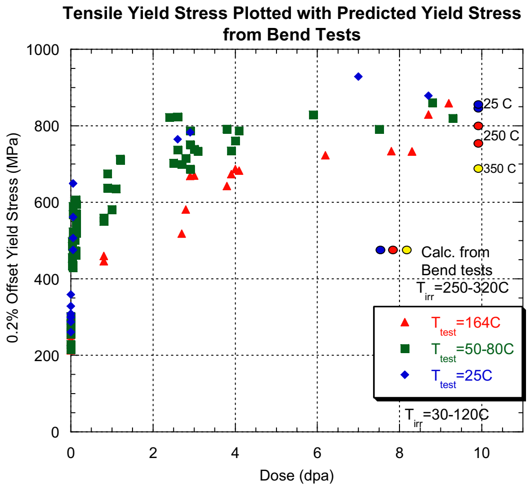

Fig. 6. Calculated tensile yield from bend tests plotted with measured tensile yield stress on proton irradiated 316L stainless steel.

Three point bend tests were performed on 316L stainless steel after irradiation at the 800 MeV proton beam at LANSCE at a temperature of 70 °C and after irradiation at the 570 MeV proton beam at PSI at a temperature of 250–320 °C. A comparison between three point bend results between the two different irradiation conditions is shown in Fig. 7. Very similar load/displacement

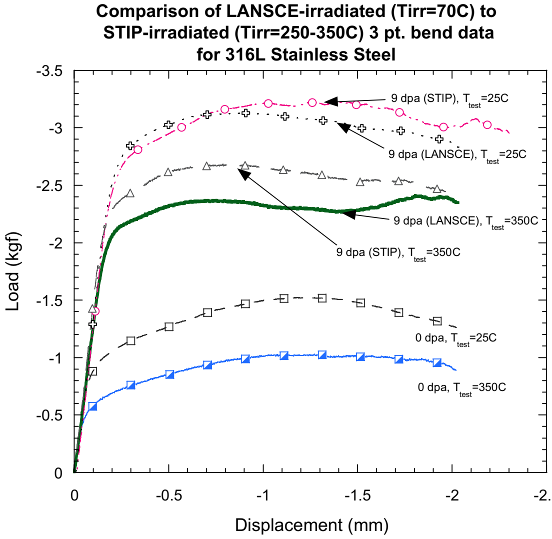

Fig. 7. Comparison of the load/displacement measured on (0.27–0.25 mm thick) 316L stainless steel in three point bending on specimens after irradiation in STIP (T_{irr} = 250–350 °C) and LANSCE (T_{irr} = 70 °C).

curves are observed for the LANSCE irradiated material when testing at 25 °C and 350 °C. To investigate this comparison in more detail, the outer surfaces of the three point bend specimens were investigated in the SEM after testing at 350 °C (Fig. 8(A) and (B)). These micrographs show clearly different deformation mechanisms for the different irradiation conditions. The specimen irradiated at LANSCE at an irradiation temperature of 70 °C shows a few slip lines and cracks on the surface, but the dominating feature is a large (~10 μm wide) slip band (upper right corner of Fig. 8(A)) starting in the center of the bend bar and continuing at an angle across the surface of the specimen in the deformed region. On the other hand, the specimen irradiated at PSI at an irradiation temperature of 250–320 °C shows a quite different deformation response. Numerous slip bands (spaced ~5 μ apart) on multiple slip systems are observed in all grains in the deformed region. This difference in deformation mechanism may be evidence of the effect of the underlying defect microstructure on deformation in these materials. The specimen irradiated at low temperatures is postulated to have a uniform array of small He clusters and vacancies (<1 nm in diameter, not visible with TEM) and larger interstitial clusters and Frank loops (visible by TEM) throughout the specimens [7] as the low temperature does not allow the vacancies and He clusters to diffuse to form larger clusters. On the other hand, the specimens irradiated at higher temperatures has larger helium clusters (~1–1.5 nm) distributed throughout [8]. The fine array of obstacles in the specimens irradiated at lower temperature leads to very localized slip in one large band often called channeling while the more widely spaced larger clusters in the higher temperature irradiated specimen allow for slip on multiple slip systems throughout the deformed region.

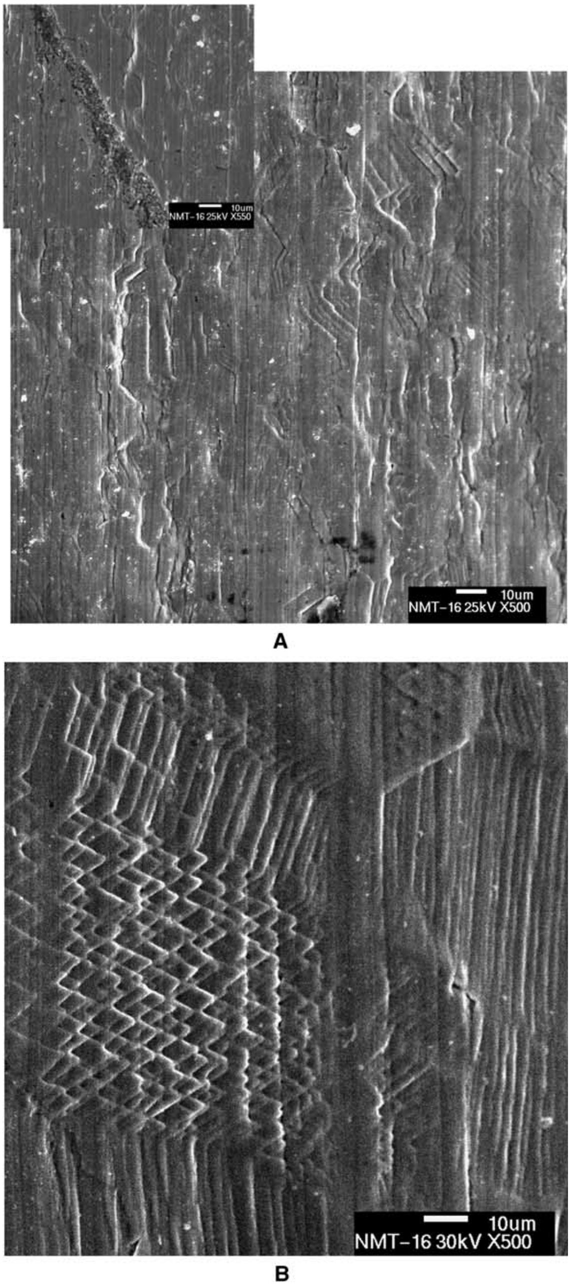

Fig. 8. SEM micrographs of the outer surface of 316L stainless steel after three point bend testing at 350 °C. Specimen in A was irradiated at LANSCE at 70 °C and B at PSI at 250–350 °C to a total dose of 9.8 dpa.

5. Conclusions

Three point bend testing was performed on 316L and Mod 9Cr–1Mo at 25, 250, 350 and 500 °C after irradiation in a high energy proton beam to a dose of 9.8 dpa at 250–320 °C. The following were concluded:

1. Using the experimental data obtained for 316L stainless steel subjected to uniaxial tensile testing, it was shown that the 0.02% tensile yield stress can be calculated from 0.05% offset yield stress measured from three point bend load/displacement curves.
2. Results from three point bend testing show over a factor of 2 increase in yield stress after irradiation to 9.8 dpa for both 316L and Mod 9Cr–1Mo.
3. Observation of the outer fiber of 316L bend specimens after testing shows localized flow for low temperature irradiation and slip bands activated on multiple slip systems in each grain after high temperature irradiation.

Although the three point bend data shows a good correlation with tensile yield stress, four point bending testing may yield more information on work hardening rate and ductility which are both very important in predicting the long term performance of materials.

Acknowledgements

The authors would like to thank Dan Schwartz of LANL for performing the SEM analysis on these specimens. This work was performed under the auspices of the Advanced Fuel Cycle Initiative program for the Department of Energy.

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