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Journal of Nuclear Materials 283–287 (2000) 418–422

journal of nuclear materials

www.elsevier.nl/locate/jnucmat

Shear punch and tensile measurements of mechanical property changes induced in various austenitic alloys by high-energy mixed proton and neutron irradiation at low temperatures

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Abstract

Tensile and shear punch tests of Inconel 718, 316L and 304L specimens irradiated with very high-energy protons and spallation neutrons showed that irradiation caused significant hardening and loss of uniform elongation. The specimens were irradiated at temperatures from room temperature to 150°C in the Los Alamos Spallation Radiation Effects Facility (LASREF) at the Los Alamos Neutron Scattering Center (LANSCE). Dose levels ranged from 0.01 to 12 dpa. Most of the observed hardening for all three alloys occurred within the first 0.1 dpa (i.e., low dose) and before significant gas accumulation, indicating that gas generation may not play a significant role in the hardening or ductility loss. The correlations between tensile and shear punch data were as expected for yield strength but were unusual for the maximum strength in comparison with previous correlations based on fission-neutron-irradiated alloys.

1. Introduction

Very high concentrations of helium and/or hydrogen can be detrimental to the performance of structural alloys. Helium is generally thought to be an embrittlement problem for higher temperature applications, while hydrogen exerts its influence on embrittlement most strongly at lower temperatures. When compounded with radiation-induced microstructural alteration, the possible combined effect of these gases becomes an issue requiring study for structural applications in fusion and especially in spallation neutron environments.

This paper focuses on the mechanical behavior of Inconel 718, and 316L and 304L stainless steels after irradiation to levels ranging from 0.01 to 12 dpa at temperatures in the range 30–150°C. The radiation damage arises from a mixed environment of high-energy spallation neutrons and a proton spectrum peaking at ~800 MeV. Under such conditions, the proton-induced generation rates of helium and hydrogen are much larger than those encountered in typical fusion spectra, in excess of 100 and 1000 appm/dpa, respectively.

Tensile data were generated primarily with conventional miniature tensile specimens, although the database was extended by shear punch tests on a number of 3 mm diameter transmission electron microscopy (TEM) disks that were dispersed among the tensile specimens during the irradiation. Previously published comparisons conducted on a wide range of materials irradiated in fission reactors at 365–600°C have shown that development of tensile–shear punch correlations is rather straightforward and successful [1–3]. However, the confident application of such fission-derived correlations to behavior prediction in irradiation environments involving low temperatures and generating very high levels of helium and, especially, hydrogen has not previously been established.

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0022-3115/00/$ - see front matter
PII: S0022-3115(00)00363-9

This paper addresses the dependence of irradiation-induced changes in mechanical properties on alloy, temperature and dpa level, and whether shear punch test data from this experimental series mirror the behavior of the tensile data.

2. Experimental procedure

Tensile specimens (type S1) and shear punch specimens (TEM disks) of Inconel 718, 304L and 316L were irradiated in both the proton beam and in neutron furnaces (away from the direct proton beam) at the LASREF facility at LANSCE at Los Alamos National Laboratory. The specimens were loaded in specially designed holders to control the specimen irradiation temperature and to isolate the specimens from the water coolant [4]. Average irradiation temperatures were between 30°C and 200°C, depending on sample thickness and sample position [5]. The irradiation temperatures were measured at three locations in each tube using thermocouples welded inside the specimen holders. TEM and tensile specimens received doses ranging from about 0.01 to about 12 dpa. The proton and neutron fluences were determined through analysis of activation foils that were irradiated in each sample group [6]. The low doses used in this paper for the neutron furnace specimens ($\leqslant$0.2 dpa) are considered preliminary but are within a factor of 2 of the correct value.

Tensile and shear punch tests were performed at 20°C, 50°C, 80°C and 164°C. The test temperatures corresponded approximately to the irradiation temperatures. The irradiation temperature decreased, as did the particle fluence, with distance from the proton beam. This interdependence resulted in those samples with the highest dpa values being tested at the highest test temperatures in order to keep the irradiation and test temperatures nearly the same. Separation of the effects of irradiation and test temperatures from the effects of irradiation per se was not attempted directly.

The miniature tensile specimens were cut from sheet stock by electro-discharge machining (EDM). These had nominal gauge dimensions of 5 mm $\times$ 1.2 mm $\times$ 0.25 mm (0.2 in. $\times$ 0.05 in. $\times$ 0.010 in.), although the specimen thickness varied by as much as a factor of 4 depending on the alloy to facilitate evaluation of the effect of specimen thickness. The 304L and 316L specimens were irradiated in the annealed condition while the Inconel 718 specimens were annealed (1065°C/air cooled) and aged (760°C/10 h, furnace cooled to 650°C and held for a total aging time of 20 h). Tensile tests were performed at a crosshead speed of 0.127 mm/min (0.005 in./min), yielding an initial strain rate of 4×10^{-4}/s. All tests were performed in air. Specimens tested at elevated temperature required about 90 min for heat-up and temperature stabilization prior to testing. Two tests were generally performed for each combination of alloy/irradiation condition. The 0.2% offset yield strength (YS), ultimate tensile strength (UTS), and uniform and total elongations (UE and TE) were obtained as per ASTM Standard E21-92.

Shear punch tests were performed [1,2] using standard 3 mm diameter TEM disks of the same thickness as the nominal S1 specimens. The TEM disks were also cut from sheet stock by the EDM method. Tests were performed at a crosshead speed of 0.127 mm/min (0.005 in./min). Specimen displacement during a test was assumed to be equal to crosshead displacement. All tests were performed in air. Specimens tested at elevated temperature required about 90 min for heat-up and stabilization prior to testing.

3. Results and discussion

3.1. Tensile data

The yield strength and uniform elongation data are shown in Fig. 1 (Inconel 718) and Fig. 2 (304L and 316L stainless steels). All three alloys exhibit an initial hardening at the very low (<0.1 dpa) damage levels attained in the neutron furnaces, where the specimens were shielded from the proton beam. At the higher dpa levels (>1 dpa) attained in the proton beam, there is a tendency for the Inconel 718 to soften somewhat from the radiation-hardened state, but the 300 series stainless steels maintain the neutron-induced hardening. The

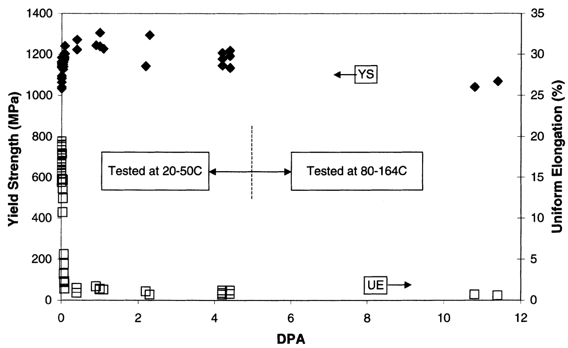

Fig. 1. Yield strength (solid symbols) and uniform elongation (open symbols) of Inconel 718 irradiated at low temperatures and tested at 20–164°C. The large number of data points that appear to exhibit a large amount of scatter on the axis at 0 dpa is an artifact of the figure scale; of the points that appear to be on the axis, only those points at very low doses (neutron furnace specimens at 0.02–0.1 dpa) exhibit UE values that are less than about 15% and YS values that are greater than about 1150 MPa. Even at the very low dose levels of the neutron furnace specimens, there is a clear dependence on dose that is consistent with the data at higher dose levels.

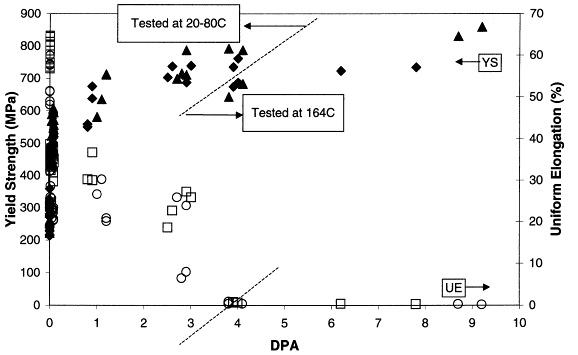

Fig. 2. Yield strength (solid symbols) and uniform elongation (open symbols) of 304L (diamond and square symbols) and 316L (triangular and circular symbols) irradiated at low temperatures and tested at 20–164°C. The large number of data points that appear to exhibit a large amount of scatter on the axis at 0 dpa is an artifact of the figure scale; of the points that appear to be on the axis, only those points at very low doses (neutron furnace specimens at 0.03–0.13 dpa) exhibit UE values that are less than about 35% and YS values that are greater than about 400 MPa. Even at the very low dose levels of the neutron furnace specimens, there is a clear dependence on dose that is consistent with the data at higher dose levels.

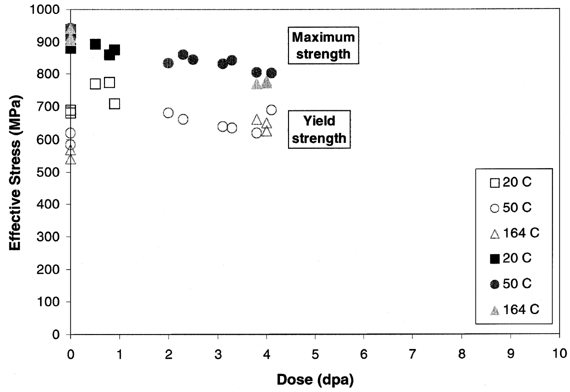

Fig. 3. Effective shear strength of Inconel 718 irradiated at low temperatures and tested at 20–164°C. Open symbols represent data for shear yield strength (SYS); solid symbols represent data for shear maximum strength (SMS).

uniform elongation of Inconel 718 falls to very low levels (<1%) at the very low neutron-induced dpa levels and remains low thereafter, but the ductility of the 300 series stainless steels falls much more slowly, requiring 3–4 dpa to reach the same values, and remains there at higher exposures.

Sencer and coworkers [7] have explained this behavior as follows: all three alloys develop high densities of small Frank loops at very low exposures that are maintained at saturation levels at higher exposure. These loops are responsible for the initial hardening. In Inconel 718, however, there is a loss of the ordered structure associated with the γ' and γ'' precipitates. This loss appears to imply that, at 0.5 dpa or less, the precipitates have been dissolved, but, in fact, they are thought to be only rendered 'invisible' in superlattice diffraction contrast. Therefore, the major precipitate contribution to hardening is still maintained initially, and it requires dose levels greater than 2 dpa to redistribute the solute concentrations associated with the precipitates. This redistribution is thought to account for the late-term softening observed in Inconel 718. In the 300 series stainless steels, there are no late-term developments in the loop microstructure, and no precipitates are involved in the evolution.

The strong drop in uniform elongation of Inconel 718 reflects only the effect of the Frank loops, which are very effective at impeding dislocation motion. In this low dose range, the formation of helium and hydrogen is judged to be too small to play a significant role in ductility loss. However, at doses above 2 dpa, the accumulating gases (~140 appm He per dpa and ~1200 appm H per dpa) may offset the dissolution of the ordered phases and keep the uniform elongation from recovering somewhat. In the 300 series stainless steels, the more gradual drop in uniform elongation may also be related to progressive gas accumulation, as no other microstructural components are observed to form, including a lack of cavity formation which indicates that all gas is retained in the matrix.

3.2. Shear punch data

Effective shear strength data determined from shear punch tests are given in Fig. 3 (Inconel 718) and Fig. 4 (304L and 316L stainless steels). Effective shear yield and maximum strengths exhibited the same trends as were observed in the tensile data, i.e., significant strengthening occurred for all three alloys. A slight softening was observed in the maximum strength with increased dose. While it is assumed that the softening in the Inconel 718 occurred for the same reason as described above for the tensile results, the reason for this is not understood in the 300 series stainless steels.

3.3. Tensile–shear punch correlations

The uniaxial yield and the effective shear yield strength data for Inconel 718 and 304L are compared in Figs. 5 and 6, respectively, for those conditions where such data are available with nearly identical irradiation conditions. There are not yet sufficient data available for 316L. While the slope of the linear regression line fit to the Inconel 718 yield data is about 1.6, it is clear from the superimposed line with a slope of 2 that the strength data can also be considered to lie on a line of slope very

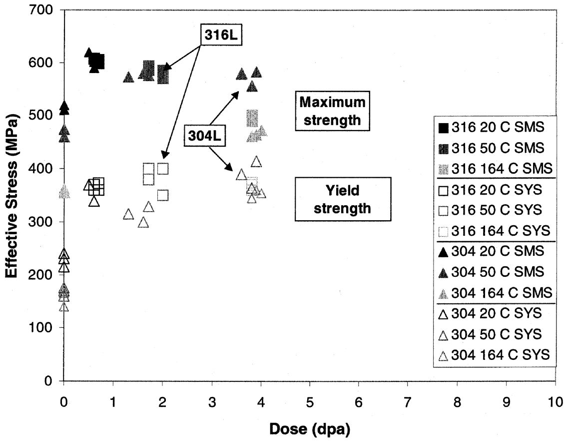

Fig. 4. Effective shear strength of 304L and 316L irradiated at low temperatures and tested at 20–164°C. Open symbols represent data for shear yield strength (SYS); solid symbols represent data for shear maximum strength (SMS).

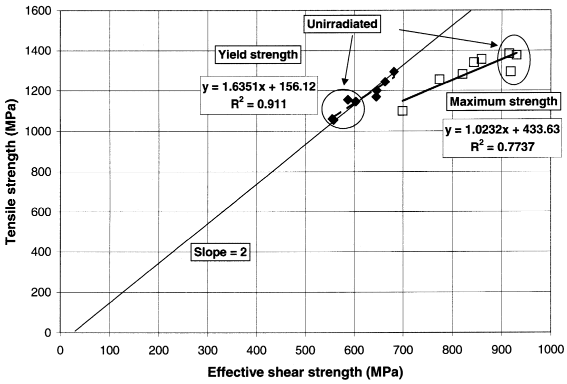

Fig. 5. Tensile–shear punch correlations for Inconel 718.

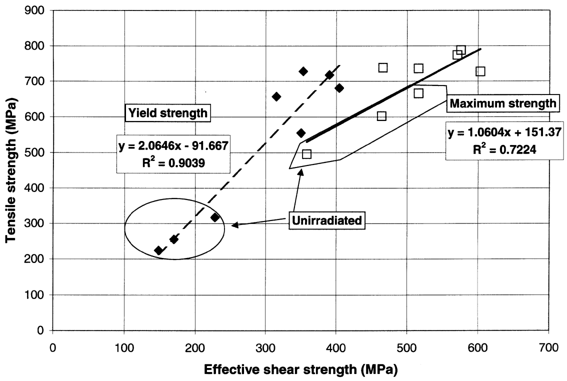

Fig. 6. Tensile–shear punch correlations for 304L.

near 2, as do the 304L data. These results are consistent with earlier work on this type of correlation [1–3]. The ultimate tensile strength and effective shear maximum strength data for both of these alloys do not lie on lines with a slope near 2, however, and appear to lie on lines with a slope near 1, instead. This is not consistent with earlier work on fission-neutron-irradiated alloys, where the slope of the maximum strength correlation was generally slightly higher than 2. The absence of agreement in this area between the current and earlier work is not understood.

4. Conclusions

Displacement damage induced by spallation neutrons and high energy protons at relatively low temperatures causes significant changes in mechanical properties in both Inconel 718 and in 304L and 316L stainless steels over the range 0.01–12 dpa. These changes exhibit similar trends with displacement level when measured by either tensile tests or shear punch tests. The strongest change is manifested as a near-total loss of uniform elongation, which occurs rather abruptly at <0.1 dpa in Inconel 718, but occurs more gradually in the 300 series stainless steels, requiring ~4 dpa.

There are some differences in alloy behavior, however, especially between Inconel 718 and the two 300 series stainless steels, which exhibit very similar behavior. Both types of alloys harden initially at <0.1 dpa as indicated by both the tensile and shear strengths. Whereas the radiation-induced strength levels are maintained to higher exposures in the 300 series stainless steels, there is some partial recovery or softening in Inconel 718 beyond ~2 dpa. While changes in the tensile yield strength correlate as expected with the changes in the effective shear punch yield strength, changes in the corresponding maximum strength quantities appear to be related differently than expected on the basis of previously reported fission neutron irradiation data. The reason for this is not yet known.

Sencer and coworkers [7] propose that the differences in the behavior of the two alloys can be explained in terms of their differing microstructural evolution. The changes in mechanical properties at very low doses are attributed primarily to the formation of high densities of Frank loops, with spallation-induced gas production playing a progressively stronger role as exposure increases. Whereas the microstructural evolution of the two 300 series stainless steels does not involve any precipitate phases, there is a loss of precipitate order of the γ' and γ'' precipitates in Inconel 718 and a gradual redistribution of the solutes contained in these precipitates at higher exposures.

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