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Mechanical properties of 304L stainless steel irradiated with 800 MeV protons

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Abstract

The tensile properties of 304L stainless steel have been investigated after low temperature ($\leqslant 250°C$) irradiation with 800 MeV protons at the LANSCE accelerator to a maximum fluence of 3×10^{25} p/m^2, corresponding to a displacement dose of about 8.5 dpa. The results showed irradiation hardening and a concomitant loss of ductility with increasing proton fluence. Subsequent SEM observation revealed that the fracture mode changed from typical ductile to partial intergranular brittle after irradiation. © 1999 Elsevier Science B.V. All rights reserved.

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1. Introduction

The European Spallation Source (ESS) is a proposal for the next generation neutron source for Europe [1]. It was soon recognized that in high-power spallation targets, materials degradation by radiation damage will be the most problematic factor in determining the efficiency and the lifetime of target components [1,2]. Although there exists an extensive database on radiation effects in fisson and fusion materials, it is not possible to transfer the data directly to the spallation case where the irradiation conditions are quite different. In ESS, the neutrons will be generated by a high power (5 MW) and high energy (1.33 GeV) proton beam which induces spallation of the nuclei in a heavy metal target [1], i.e. the target materials will be exposed to mixed high-energy proton and neutron spectra. Moreover, the structural target materials of the ESS mercury target will operate at relatively low temperatures (100–250°C) [2–4]. In fact, there are very few data on the effects of radiation in a spallation source environment. Therefore extensive international irradiation programs are under way in the medium-power sources at the Los Alamos Neutron Science Center (LANSCE) and at the Paul-Scherrer-Institut, Switzerland (SINQ).

In the materials program for the European Spallation Source (ESS), martensitic stainless steels and/or austenitic stainless steels have been chosen as candidate materials for the liquid metal container, the return hull and the proton window. As part of the effort to establish a database for the ESS engineering design, the LANSCE Water Degrader made of Inconel 718 and 304L, two PSI windows made of martensitic stainless steel and a spent ISIS target made of tantalum, irradiated with 800 MeV protons were investigated at the Forschungszentrum Jülich, Germany and the Paul-Scherrer-Institut, Switzerland. First results on Inconel 718 and martensitic stainless steel have been published [5,6]. In this letter we report tensile results on 304L stainless steel specimens obtained from the liner of the LANSCE Water Degrader.

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Table 1
Chemical composition of 304L stainless steel (wt.%)

	C	Si	Mn	P	S	Cu	Cr	Ni	Mo	N	Others
Nominal	⩽0.03	⩽1.0	⩽2.0	0.045	0.03	–	17.0/20.0	10.0/12.5	–	–	–
Reference	0.012	0.46	1.75	0.027	0.023	0.22	18.95	10.37	0.24	0.058	0.191

2. Experimental

The material of the spherical liner of the LANSCE Water Degrader investigated is solution annealed AISI 304L stainless steel. Unfortunately no unirradiated reference material from the same heat as used for the Water Degrader is available. The samples for the control tests in this work were thus prepared from a piece of 304L stainless steel tube supplied by DMV Stainless Deutschland GmbH. The chemical composition of this reference 304L stainless steel is well within the nominal composition range for this type of steel as shown in Table 1. The yield strength of the as-received solution annealed material is about 210 MPa.

The LANSCE Water Degrader investigated consists of a spherical shell of 1.5 mm thickness and 190 mm in diameter. The component was irradiated with 800 MeV protons to a total charge of 5.3 Ah. The intensity profile of the proton beam at the Water Degrader has a two-dimensional Gaussian distribution with $\sigma_x = (22.6 \pm 0.4)$ mm and $\sigma_y = (28.8 \pm 1.5)$ mm and has been determined by γ-scan [5]. The maximum fluence of 2.9×10^{25} p/m^2 is calculated from this profile. Taking the displacement damage cross section of 2900 barn [7,8], the maximum irradiation dose should be about 8.5 dpa. At the position of the Water Degrader in the LANSCE beam, the flux of neutrons was very low and their contribution to radiation damage was estimated to be a few percent of the proton value at beam centre. The maximum temperature at the beam centre during the irradiation never exceeded 250°C [5].

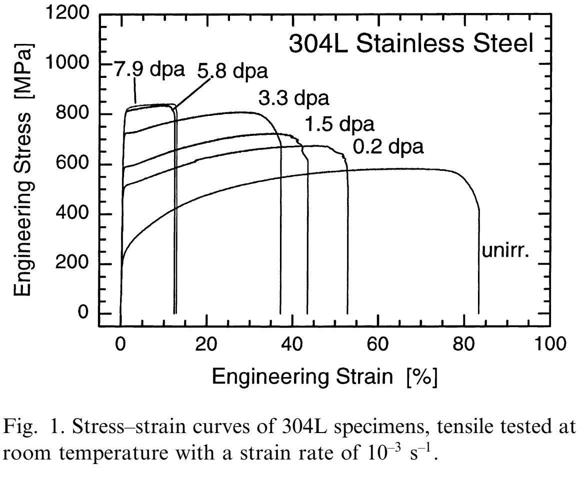

Fig. 1. Stress–strain curves of 304L specimens, tensile tested at room temperature with a strain rate of 10^{-3} s^{-1}.

To prepare tensile samples from the spherical shell of the Water Degrader, strips of 3 mm width were cut along the long axis of the beam profile, then these strips were cut into slabs of 15 mm length. Flat slices of about 0.6 mm thickness were obtained after removing the curved part of the slabs. Finally dog-bone shaped tensile samples with a gauge volume of $5 \times 1.5 \times 0.6$ mm^3 were machined from these slices. Because the strips used for the tensile test were slightly off from the beam centre, the highest dose of the tensile samples was about 8 dpa, i.e. somewhat smaller than the maximum dose of 8.5 dpa.

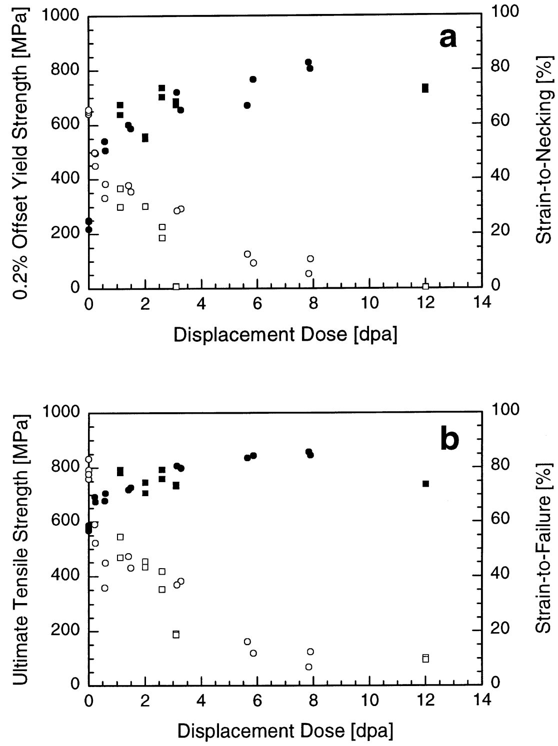

Fig. 2. (a) Yield strength (filled symbols) and strain-to-necking (empty symbols) as a function of the displacement dose for 304L stainless steel at room temperature (●, ○ this work). Also shown data from Ref. [9] (■, □). Note that here the test temperatures are 50°C for 1.12 and 2 dpa specimens, 80°C for 3.1 dpa specimens and 164°C for 3.1 and 12 dpa specimens, respectively. (b) Ultimate tensile strength (filled symbols) and strain-to-failure (empty symbols) as a function of the displacement dose. The meaning of the symbols is the same as in (a).

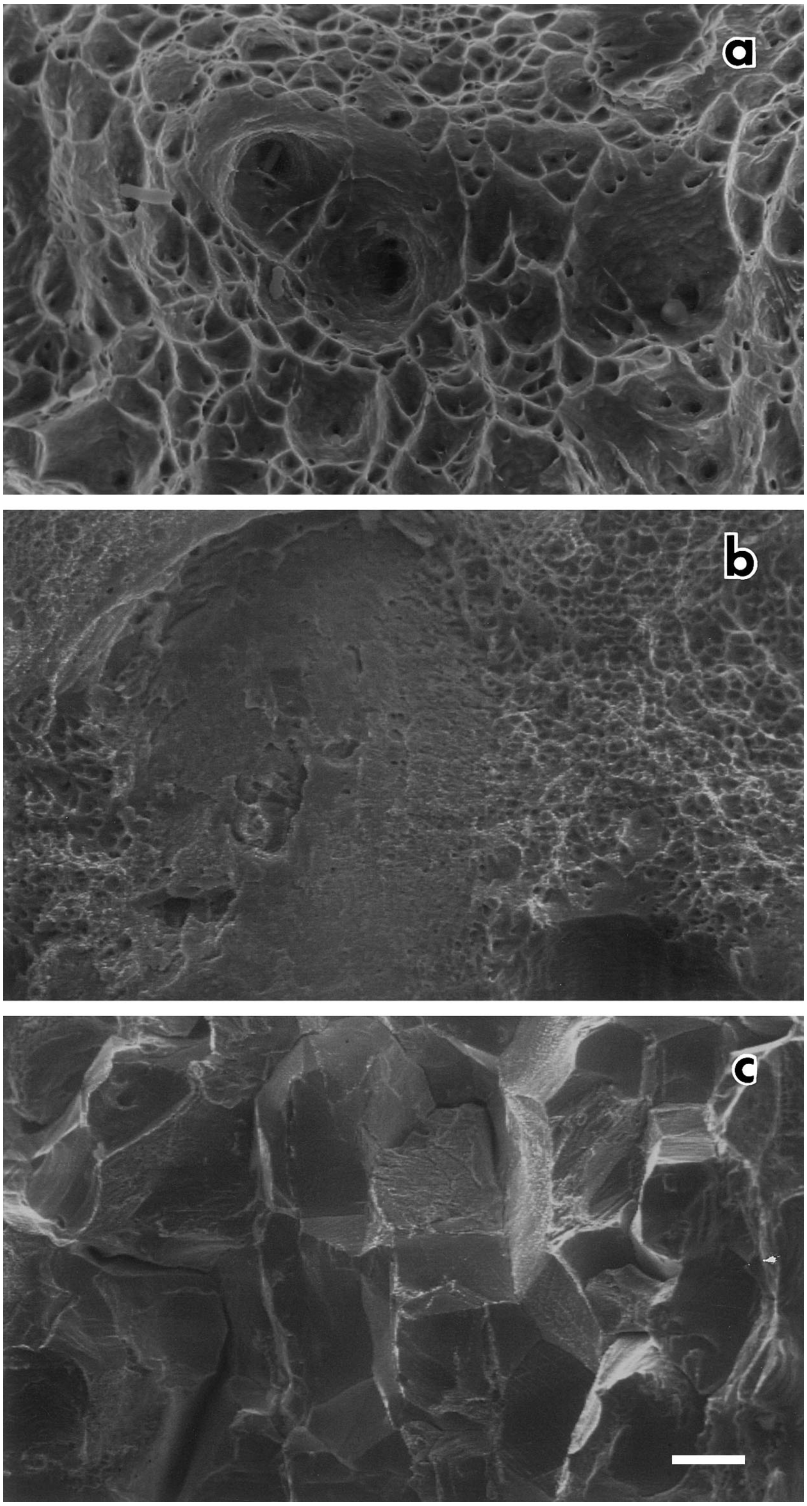

Fig. 3. Scanning electron micrographs of fracture surfaces of tensile tested 304L stainless steel specimens, (a) unirradiated sample. (b) sample with 3.3 dpa and (c) sample with 7.8 dpa. The scale in the photo indicates 5, 20 and 50 μm for (a), (b) and (c), respectively.

Tensile tests were performed at room temperature using a 2kN MTS tensile machine equipped with a video-extensometer so that the elongation could be measured directly in the gauge area. After tensile testing, the fracture surfaces were observed by SEM to identify the fracture mode.

3. Results

The engineering tensile curves of some samples from 800 MeV proton irradiated material and from the reference material are given in Fig. 1. From the stress–strain curves the yield stress $\sigma_{0.2}$, the ultimate tensile strength σ_{UTS}, the strain-to-necking $\varepsilon_{\mathrm{STN}}$ and the total elongation ε_{t} are obtained and plotted as a function of displacement dose in Fig. 2. The tensile curves show that the irradiation increases the strength and decreases the ductility of 304L stainless steel. The yield stress increases rapidly at low displacement doses, i.e. from 210 MPa of the unirradiated samples to about 550 MPa at 1 dpa and then increases relatively slowly at higher displacement doses with a tendency to saturation at around 800 MPa. The irradiation induced strength increase was accompanied by a loss in work hardening ability (see Fig. 1). The lack of work hardening in 304L stainless steel causes a significant reduction of the strain-to-necking, i.e. from 65% of the reference material down to 8% of the samples with the maximum dose (8 dpa). Low temperature tensile results from recent LANSCE irradiations [9] are in good agreement with our data. However, at elevated test temperature they indicate an enhanced degradation of the ductility (Fig. 2(a)).

SEM pictures of the fracture surfaces are given in Fig. 3 for specimens of different doses as well as the reference material. The fracture surface of the reference material is shown in Fig. 3(a) which illustrates a typical ductile fracture mode. For the irradiated samples, the fracture mode changes gradually from almost pure ductile at 0.6 dpa to partial cleavage at 3.3 dpa (Fig. 3(b)) and then to partial intergranular brittle fracture mode at high doses, $\approx$8 dpa. Fig. 3(c) demonstrates the situation at a dose of 7.8 dpa. It can been seen that the intergranular fracture covers a portion of about 60% of the total area on the fracture surface.

Unfortunately no reports of tensile tests of reactor neutron-irradiated 304L stainless steel at low temperatures (less than 300°C) could be found. At present it is therefore not possible to answer the question about a possible influence of the high energy recoil spectrum and/or the high helium concentrations produced by the 800 MeV protons, or in other words, whether the dpa-number is an appropriate parameter for describing the observed hardening.

4. Conclusion

With increasing proton fluence, the yield strength of 304L stainless steel increases and is accompanied by a loss of ductility (Figs. 1 and 2). The initial ductile fracture mode is changed to a partially intergranular brittle failure after irradiation to 8 dpa. This general trend is similar to radiation effects on the mechanical properties of structural materials exposed to fission and fusion environments. Preliminary results at Los Alamos [9] indicate that the strain-to-necking of 304L is very sensitive to the test temperature. The much higher production of helium and hydrogen in a spallation environment is possibly responsible for this enhanced loss of ductility at test temperatures of 164°C (see Fig. 2(a)) and for the appearance of intergranular fracture at high dose (Fig. 3(c)). Further studies, including an extension of the temperature and dose regions are required to clarify the hardening mechanism in 304L stainless steel caused by protons and neutrons in the 1 GeV energy range. Furthermore, detailed microstructural investigations must accompany the mechanical tests. Transmission electron microscopy observation are under way and will be published elsewhere.

Acknowledgements

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References

[1] ESS – A Next Generation Neutron Source for Europe, vol. 1–3, ESS Council (I. Kjems, chairman) 1997, ISBN 090 237 6 500.

[2] H. Ullmaier, F. Carsughi, Nucl. Instrum. Meth. B 101 (1995) 406.

[3] L.K. Mansur, H. Ullmaier (Eds.), Proceedings of the International Workshops on Spallation Materials Technology, Oak Ridge, Tenn., 23–25 April 1996, ORNL Report CONF-9604151.

[4] F. Carsughi, L.K. Mansur, W.F. Sommer, H. Ullmaier (Eds.), FZJ Report JÜL-3450, ISSN 0944-2952, Ancona, Italy, 19–22 September 1997.

[5] F. Carsughi, H. Derz, P. Ferguson, G. Pott, W.F. Sommer, H. Ullmaier, J. Nucl. Mater. 265 (1999) 78.

[6] Yong Dai, G. Bauer, F. Carsughi, H. Ullmaier, S.A. Maloy, W.F. Sommer, J. Nucl. Mater. 265 (1999) 203.

[7] S.L. Green, J. Nucl. Mater. 126 (1984) 30.

[8] S.L. Green, W.V. Green, F.H. Hegedus, M. Victoria, W.F. Sommer, B.M. Oliver, J. Nucl. Mater. 155–157 (1988) 1350.

[9] M.R. James, M. Louthan, S.A. Maloy, W.F. Sommer, A preliminary report: the change in the mechanical properties of Inconel 718, 304L and 316L stainless steel, and Al6061 after irradiation at the LANSCE accelerator, 1999, private communication.