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Small punch tests on austenitic and martensitic steels irradiated in a spallation environment with 530 MeV protons

D. Finarelli [a], M. Roedig [b], F. Carsughi [c,d,*]

[a] *Dipartimento di Fisica, Politecnico di Milano and INFM Unitá di Milano Politecnico, P.za Leonardo da Vinci, I-20100 Milan, Italy*
[b] *Hot Cells, Forschungszentrum Jülich, D-52425 Jülich, Germany*
[c] *Dipartimento di Scienze Applicate ai Sistemi Complessi (SASC), Universitá Politecnica delle Marche and INFM Unitá di Ancona, Via Brecce Bianche, I-60131 Ancona, Italy*
[d] *Institut fur Festkörperforschung, Forschungszentrum Jülich, D-52425 Jülich, Germany*

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Abstract

Mechanical properties of the austenitic steel 316L (both solution annealed and cold worked) and the low activation martensitic steel Optifer have been investigated by small punch tests. The samples, in the form of TEM disks, were irradiated in the target of the spallation source SINQ to displacement doses of up to 11 dpa. Values for yield strength, ductility and fracture energy as evaluated from punch tests are given.

1. Introduction

Miniaturized specimens are becoming very interesting to assess mechanical properties of materials and are important especially when there are volume constraints limiting the dimension of test samples [1]. These limitations are related to the small volume and to the high irradiation gradient of a possible irradiation position in a spallation target. Transmission electron microscopy (TEM) discs, of 3 mm diameter, are good target specimens since they are already widely used in irradiation experiments and are usually available in numbers sufficient to obtain good statistics. Small punch (SP) test method on 3 mm discs has been applied to evaluate ductility, fracture toughness, ductile-to-brittle transition temperature (DBTT) and other mechanical properties of unirradiated and irradiated materials [1,2]. Since the deformation process experienced from samples during SP tests is much more complex than in tensile tests, it is necessary to verify the validity of the available models [3–8] to extract standard engineering data from the SP test load displacement curve. The materials here used for the SP tests are austenitic (316L) and martensitic (Optifer) stainless steels irradiated within the Spallation Target Irradiation Programme STIP-I [9,10]. During irradiation a number of different samples have been exposed to a proton beam of 530 MeV energy. The protons contributed the main part of the displacement damage, however, spallation neutrons (peak energy at 20 MeV) gave also a significant contribution. In the present work, performed in the frame of the European Spallation Source (ESS) target structural materials research and development, SP tests were carried out in the Hot Cells of Forschungszentrum Jülich (Germany) and show how yield strength, ductility and fracture energy depend on the irradiation dose.

2. Experiment

Three materials have been investigated: a solution annealed (SA) and a 20% cold worked (CW) austenitic

* Corresponding author.
E-mail address: f.carsughi@alisf1.univpm.it (F. Carsughi).

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316L stainless steel and Optifer, a low activation martensitic steel (LAMS). Their chemical composition and their thermo-mechanical treatment are reported in Table 1. The samples were prepared to final dimension, 3 mm diameter and 0.25 mm thickness before irradiation, then stacked in rods and loaded in the SINQ Spallation Target-3 at the Paul Scherrer Institute, Villigen, Switzerland. The specimens underwent irradiation with 530 MeV protons (energy of impinging protons diminished due to collisions to 330 MeV at the last rod) and high neutron flux (most flux with energy lower than 20 MeV and up to 300 MeV) producing radiation damage up to 11 displacements per atom (dpa) depending on the position within the SINQ target. The helium concentrations in irradiated samples range from 100 to 600 appm and are reported in Table 2. The components were machined in the Forschungszentrum workshop with strict tolerances. The pin tip has a hemispherical head of 1 mm diameter, the hole in the sample support is 1.5 mm diameter and the edges have a 0.2 mm radius. The sample disc is locked with an eccentric clamp. A commercial tensile test machine applied the load to the tip. The force P was measured with a 2 kN load cell, while a linear extensometer in contact with the lower surface of the sample measured the displacement d. Data from these sensors were collected and stored in a computer for subsequent processing. A typical load–displacement curve is shown in Fig. 5 of Ref. [6], where different deforming mechanisms are active as the ball punch test proceeds: first the elastic response of the whole plate, second the plastic regime mainly due to bending region, third the plastic regime mainly due to membrane stretching and fourth the instability of the whole sample, which includes the peak load and yields to the failure of the disk due to fracture. The experimental curves for all the materials are shown in Fig. 1, where, for clarity, the curves of CW 316L and Optifer have been shifted upwards by 0.25 and 0.5 kN, respectively.

3. Data analysis

The load–displacement curve, P–d curve, obtained from SP tests allows to evaluate standard engineering properties, particularly yield strength, ductility and fracture energy. The yield strength can be estimated by the load P_A, being the point A defined as in Fig. 5 of Ref. [6], i.e. the intersections of the linear extrapolation of the elastic and plastic bending parts of the experimental curves. The yield strength σ_y should be then related to the load P_A by the analytical equation for the elastic response of a disc to central loading [11]

$$\sigma_y = \frac{3P_A}{2\pi t_0^2}, \tag{1}$$

Table 1
Chemical composition of investigated stainless steel in wt%

Elem.	Fe	Cr	Ni	Mo	Mn	V	Co	Cu	B	C	Si	P	S	N	Ta	W
316L[a,b,d]	Bal.	17.17	12.24	2.31	1.75	–	0.077	0.07	0.0009	0.019	0.35	0.02	0.0007	0.073	0.002	–
Optifer[c,d]	Bal.	9.48	0.06	0.002	0.55	0.245	–	–	–	0.125	0.04	0.0015	0.003	–	0.065	0.985

[a] From ISPRA, nominally solution annealed (SA).
[b] For CW samples: 20% cold working of SA 316L.
[c] From SAARSCHMIEDE GmbH, Heat No. 000735.
[d] Specimen preparation: electrical discharge machining (EDM), polishing.

Table 2
Irradiation dose (dpa) and He content (appm) in the irradiated materials

Material	Irradiation dose (dpa)	He (appm)
SA 316	2	100
SA 316	4–5	200
SA 316	8	300
CW 316	4	150
Optifer	11	600

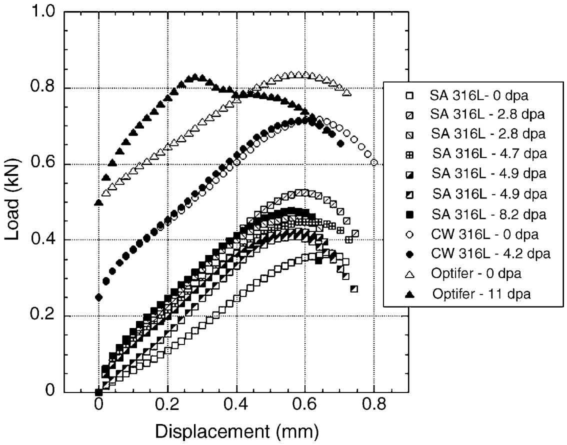

Fig. 1. Load–displacement curves of SA 316L with radiation dose from 0 to 8.2 dpa, CW 316L with irradiated dose 0 and 4.2 dpa and Optifer with irradiated dose 0 and 11 dpa.

where t_0 is the undeformed thickness. However, it has been shown that a better agreement between P_A and the yield strength σ_y from tensile tests is obtained by replacing $3/2\pi$ by an empirical factor α

$$\sigma_y = \alpha \frac{P_A}{t_0^2}. \tag{2}$$

For different steels α is found to range between 0.36 and 0.41 [6–8,15]. It is assumed that α depends only weakly on material properties or radiation damage and is mainly a function of the test geometry.

The uncertainty on the yield strength due to the thickness variation and on the P_A determination is estimated to be about 17%. Ductile samples experience, before failure, a membrane stretching behavior. Under this condition the strain field is known and fracture stretching can be calculated and described as a function of thickness of the sample at the crack edge. The fracture equivalent strain can be defined by [3,4]:

$$\epsilon_{qf} = \ln \frac{t}{t_0}, \tag{3}$$

where t is the thickness at the crack edge and t_0 the undeformed thickness.

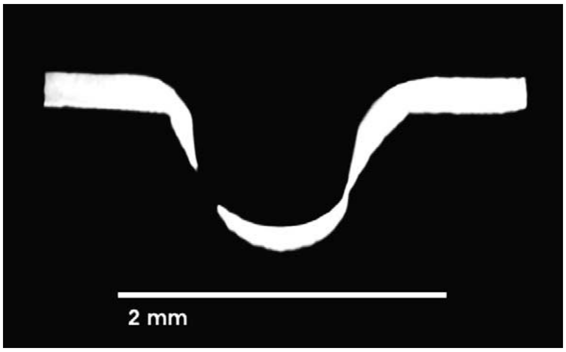

Fig. 2. Cross-section of a fractured SA 316L specimen irradiated up to 4.9 dpa. The thickness at crack edge t can be obtained from this picture.

In order to measure the thickness at the crack edge, broken samples were cut, pictures of the cross-sections were recorded and the thickness of the crack edge was directly measured. Fig. 2 shows the image of a SA 316L sample irradiated at 4.9 dpa.

An empirical relation between the equivalent fracture strain ϵ_{qf} and the displacement at fracture d_F is available; however, access to d_F is not feasible with the present design of the test machine. A similar equation for displacement at maximal load d^* is evaluated and compared with the direct measurement of the thickness at the crack edge [6]

$$\epsilon_{qf} = \beta \left(\frac{d^*}{t_0} \right)^2, \tag{4}$$

where β is a fitting parameter.

The fracture energy of the samples is derived by Eq. (5) from the data stored during the SP test.

$$J = \int_{\delta=0}^{\delta=d^*} P \, d\delta. \tag{5}$$

4. Yield strength

P–d curves of unirradiated samples are used to estimate the parameter α in Eq. (2) by comparison to yield strength of unirradiated materials available in the literature for 316L both SA [12] and CW [13], and for Optifer [14]. Scattering of results and uncertainties on reference yield strength in the range of 17% yields $\alpha = 0.38 \pm 0.06$, consistent with the values found in the literature [6,15]. Yield strength values are shown in Fig. 3.

5. Equivalent fracture strain

In order to evaluate the equivalent fracture strain, ϵ_{qf}, Eq. (3) has been applied using the pictures of cut

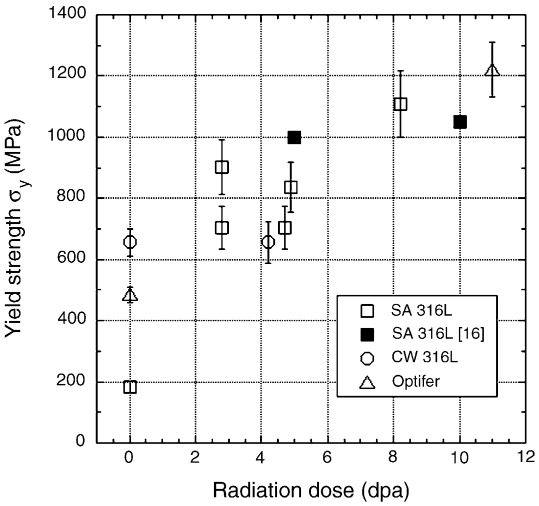

Fig. 3. Yield strength derived for SA 316L, CW 316L and Optifer as a function of radiation dose (dpa), using $\alpha = 0.38 \pm 0.06$ in Eq. (2). Some tensile test data on irradiated SA 316L are given for comparison [16].

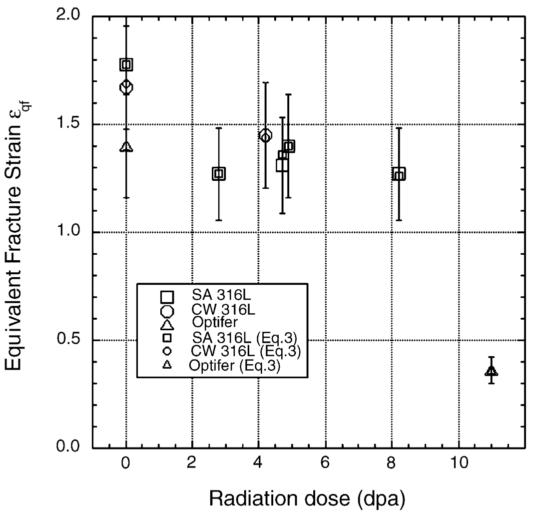

Fig. 4. Equivalent fracture strain of SA 316L, CW 316L and Optifer as evaluated from the thickness measurement and calculated from max displacement (Eq. (4)) with $\beta = 0.254$. For comparison, the data obtained from Eq. (3) are also shown. Note that for Optifer the measured and calculated values coincide for 11 dpa, since only this point is available for t measurement.

samples. An unirradiated Optifer sample picture is useless since the sample crack edges are not distinguishable from the thinning shape of the plate. To assess the validity of the empirical relation (Eq. (4)), the parameter β has been optimized by fitting the values calculated from Eq. (3). Good results are obtained for 316L steel, both SA and CW, with

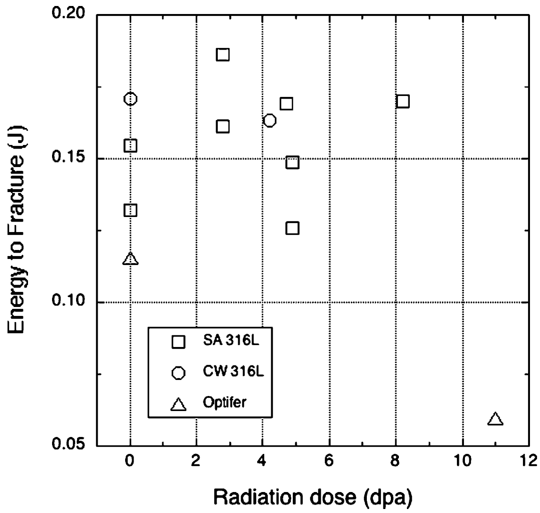

Fig. 5. Fracture energy of SA 316L, CW 316L and Optifer as a function of the equivalent fracture strain.

$\beta = 0.254 \pm 0.004$. For Optifer, the only direct evaluation of ϵ_{qf} is available for the 11 dpa sample and again $\beta = 0.254$ is found to be a good value by the fit procedure. Under this assumption, the equivalent fracture strain ϵ_{qf} for unirradiated Optifer is given through regression from the 11 dpa sample and application of Eq. (4). The values of the equivalent fracture strain ϵ_{qf} are shown in Fig. 4, where a comparison of the data obtained by using Eqs. (3) and (4) shows consistency of the two calculations.

6. Fracture energy

The energy to break the sample is calculated using Eq. (5) and is shown as a function of dpa in Fig. 5. For SA 316L the data show a rather large experimental uncertainty. It is reasonable to state that, within the experimental uncertainty of about 30%, the data do not show a significant variation around the mean value of 0.15 J. On the other hand, CW 316L does not show a significant change of the fracture energy, although we have only one irradiated sample. Concerning Optifer, the single irradiated sample at 11 dpa shows a large reduction of about 50% of the fracture energy.

7. Conclusions

The SA 316L and Optifer steels show a large dependence of the behavior on the irradiation dose in the SP test, while CW 316L steel seems to be less sensitive to irradiation.

Irradiation produces hardening: for 316L steel the yield strength quickly rises at low doses until a first plateau is reached. The 8 dpa irradiation shows that a further increase is possible and caused by a different hardening mechanism. For Optifer only two points are available, at 0 and at 11 dpa. Therefore it is not possible to give more than a general trend for increasing yield strength.

Fracture strain decreases for 316L steel with irradiation, ϵ_{qf} values for SA specimens range from 1.8 to 1.2 while CW specimens from 1.6 to 1.4.

It is interesting to notice that the values of the fracture energies for irradiated 316L SA and CW samples do not vary significantly with the radiation dose within the experimental uncertainty. Concerning the Optifer steel, although we have measured only the 0 and 11 dpa samples, a large reduction of the fracture energy was found, showing the weakness of this material under irradiation. Moreover, Optifer data at 11 dpa are at the limit of ductile fracture at ambient temperature. The embrittlement of Optifer is evident at least at such a level of irradiation damage.

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References

[1] G.E. Lucas, Metall. Trans. A 21 (1990) 1105.
[2] M.P. Manahan, A.E. Browing, A.S. Argon, O.K. Harling, Miniaturized Disk Bend Technique, ASTM STP 888, Philadelphia, 1986, p. 17.
[3] M.R. Bayoumi, M.N. Bassim, Int. J. Fract. 23 (1983) 71.
[4] J. Chakrabarty, Int. J. Mech. Sci. 12 (1970) 315.
[5] A.K. Ghosh, S.S. Hecker, Metall. Trans. A 6 (1975) 1065.
[6] X. Mao, H. Takahashi, J. Nucl. Mater. 150 (1987) 42.
[7] X. Mao, T. Shoji, H. Takahashi, J. Test. Eval. 15 (1987) 30.
[8] V. Vorlivek, L.F. Exworthy, P.E. Flewitt, J. Mater. Sci. 30 (1995) 2936.
[9] Y. Dai, G.S. Bauer, J. Nucl. Mater. 296 (2001) 43.
[10] Y. Dai, Y. Foucher, M.R. James, B.M. Oliver, J. Nucl. Mater. 318 (2003) 167.
[11] R.J. Roarke, W.C. Young, Formula for Stress and Strain, 6th Ed., McGraw-Hill, 1980.
[12] P. Marshall, Austenitic Stainless Steels Microstructure and Mechanical Properties, Elsevier Applied Science, 1984.
[13] G.J. Kraaij, KFA, Report Jül-94, 1980.
[14] L. Schäfer, in: E. Daum, K. Ehrlich, M. Schirra (Eds.), Proceedings of the Second Milestone Meeting of European Laboratories on The Development of Ferritic/Martensitic Steels for Fusion Technology, Karlsruhe, 1996, p. 29.
[15] Y. Ruan, P. Spätig, M. Victoria, J. Nucl. Mater. 307–311 (2002) 236.
[16] M.G. Horsten, M.I. de Vries, D.S. Gelles et al. (Eds.), 17th International Symposium on Effects of Radiation on Materials, ASTM STP 1270, American Society for Testing and Materials, West Conshohocken, PA, 1996, p. 919.