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Assessment of irradiation embrittlement of the Eurofer97 steel after 590 MeV proton irradiation

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ABSTRACT

The irradiation hardening of the Eurofer97 steel following 590 MeV proton irradiations was determined at three different irradiation temperatures, 50 °C, 250 °C and 350 °C, and various doses up to about 1.3 displacement per atom. The dose and temperature dependence of the irradiation hardening was characterized by a linear relationship between the irradiation hardening and the square root of the dose as: $\Delta\sigma_{0.2} = k(T)\,\mathrm{dpa}^{1/2}$. Mini pre-cracked bend bar ($1 \times 1 \times 16\ \mathrm{mm}^3$) were also tested in the lower ductile to brittle transition region before and after irradiation at 300 °C and 0.5 dpa. The effective fracture toughness-temperature curves, $K_e(T)$, were indexed on an absolute temperature scale at T_0 for $K_e = 100\ \mathrm{MPa\ m}^{1/2}$ for both the unirradiated and irradiated condition. The irradiation-induced temperature shift ΔT_0 of the $K_e(T)$ curves yielded a coefficient C_0, defined as $C_0 = \Delta T_0/\Delta\sigma_{0.2}$, of about 0.53. For these low doses, helium effects could not be identified on the fracture properties.

1. Introduction

Reduced activation tempered martensitic steels are leading candidate materials for fusion rector structural components due to their resistance to void swelling and good balance of physical and mechanical properties [1]. Since the materials surrounding the burning plasma in a fusion reactor will be highly irradiated by energetic neutrons and other types of radiation, a degradation of their overall mechanical properties induced by the radiation environment will occur. For irradiation temperatures below 400–450 °C, irradiation hardening, defined as the increase of the yield stress, occurs and is accompanied by a decrease of ductility. The degradation of the fracture toughness properties is reflected in an upward shift of the transition temperature between the brittle and ductile modes of fracture [2,3]. In a neutron fusion irradiation environment, the production of helium and hydrogen by transmutation results in additional detrimental effects on the overall mechanical properties, which mainly remain to be quantified.

The aim of this study was to characterize the effect of high-energy proton irradiation on the tensile and fracture properties of the Eurofer97 steel up to doses of about 1 dpa (displacement per atom) in the irradiation hardening temperature regime. The PIREX facility (Proton Irradiation Experiment) was used for this purpose. One of the particularities of the facility was its helium production rate, which was about 170 appm He/dpa in ferritic steels. While PIREX facility was originally designed to irradiate small flat tensile specimens, an irradiation of a series of mini pre-cracked bend bars ($1 \times 1 \times 16\ \mathrm{mm}^3$) was performed to quantify the temperature shift of the toughness-temperature curve. The mini bend bars were irradiated at 300 °C to a dose of 0.5 dpa.

2. Material and experimental procedures

The alloy investigated in the framework of this study is the reduced activation Eurofer97 steel produced by Böhler AG. The tensile specimens and bend bars used in this work were cut out of the 25 mm thick plate, heat E83697 – plate No. 2. The chemical composition is indicated in Table 1.

The final heat-treatment consisted of an austenitization at 980 °C/30.6 min followed by air quenching and by a tempering at 760 °C/90 min. The tempered martensitic microstructure resulting from this final heat-treatment is free of δ-ferrite. The prior austenite grain size, characterized by the mean intercept length l, was determined and found equal to 9.5 μm.

Tensile tests were performed at temperature corresponding to those of the irradiation temperature under an inert Ar atmosphere. Small flat tensile specimens designed for the proton irradiation facility PIREX were used (Fig. 1). The testing was performed with a screw-driven Zwick 010 machine at an imposed nominal strain-rate of $5 \times 10^{-4}\ \mathrm{s}^{-1}$. The elongation the specimens was deduced from the displacement of the machine crosshead measured with a LVDT by using a compliance correction. The stresses and strains reported hereafter are expressed in engineering units.

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Table 1
Chemical composition in wt% as measured by Böhler.

C	Si	Mn	P	S	Cr	Mo	V	W	Ti	N	Ni
0.12	0.06	0.47	<0.005	0.004	8.93	0.0015	0.2	1.07	0.009	0.018	0.022
Cu	Co	Al	Nb	B	Ta	O	As	Sn	Zr	Sb	
0.0036	0.007	0.008	0.0022	<0.001	0.14	0.0012	<0.005	<0.005	<0.005	<0.005	

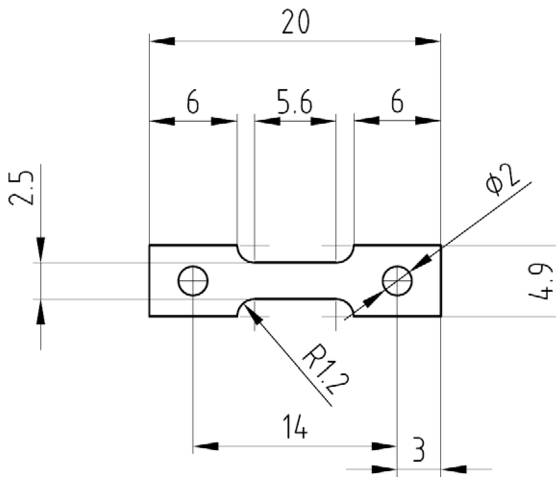

Fig. 1. 'PIREX' geometry specimen.

For fracture toughness testing, a series of mini bend bars whose dimensions are 1 × 1 × 12 mm^3 were used (Fig. 2). The specimens were extracted from larger pre-cracked bend bars using special procedures described elsewhere [4]. The final crack length ratio a/W of the mini bend bar was targeted at 0.5. However, the final a/W ratio ranges between 0.23 and 0.7. The mini bend bars were tested in the lower transition region to measure the temperature shift of the transition associated with the irradiation hardening. The bend bars were tested statically at a constant actuator velocity of 0.5 mm/min. Test procedures were based on ASTM practices to determine the critical J-integral, J_c, at the onset of cleavage [5], from which an equivalent toughness K_{J_c} can be calculated as

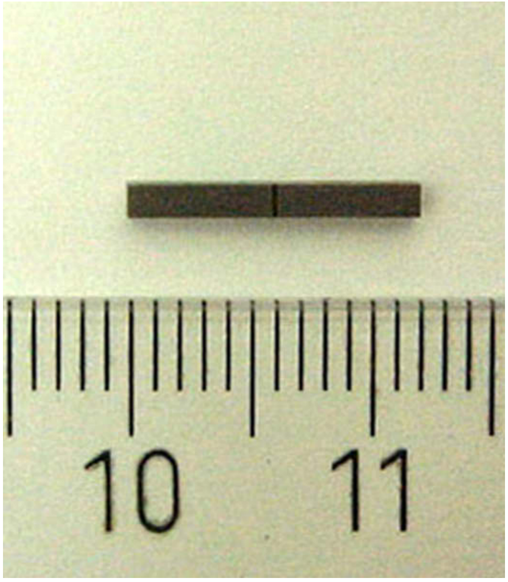

Fig. 2. Mini bend bar.

$$K_{J_c} = \sqrt{\frac{J_c E}{(1-\upsilon^2)}} = \sqrt{J_c E'} \qquad (1)$$

where E' is the plane strain Young's modulus.

3. Irradiation conditions

Tensile specimens and mini bend bars were irradiated with a 590 MeV proton beam in the PIREX facility at PSI. A detailed description of the characteristics of the facility can be found in [6]. The irradiations of the tensile specimens were carried out to a displacement range between 0.16 and 1.3 dpa at different nominal temperatures, 50 °C, 250 °C, and 350 °C. As far as the mini bend bars are concerned, they were irradiated at 300 °C up to a dose of 0.5 dpa. The actual irradiation temperatures fluctuated around these nominal values by about ±30 °C as monitored with thermocouples spot-welded on the specimens. These fluctuations were mainly attributed to the beam instabilities in shape, centering and intensity. The nominal calculated doses are reported with an uncertainty of about 20% in terms of the actual dpa level. Finally, we mention that the production of He/dpa in PIREX was previously determined to be about 170 appm He/dpa in steels [7].

4. Results and discussion

We present first the fracture toughness results of both the unirradiated and irradiated mini bend bars ($T_{irr} = 300$ °C, dose = 0.5 dpa). Due to the small size of the specimens, these data do not meet the requirement of the ASTM-E1921 standard for valid K_{J_c} value [5]. Therefore, the fracture data are called hereafter, K_e, for effective fracture toughness, and were calculated from J using Eq. (1). For the unirradiated specimens, all the load–deflection curves presented some macro-plasticity even for the lowest temperature. Only one fracture data point was obtained with a fully macroscopic linear behavior, which is the point at −150 °C for an irradiated specimen. The fracture toughness temperature dependence, before and after irradiation, is plotted in Fig. 3. Two relevant observations can be made. First, the transition appears very steep due to the fact that such small specimens can maintain a high level of constraint only at very low applied K or equivalently at very low temperature. At higher temperature, constraint loss dominates resulting in very high effective toughness. Such sharp transitions were already observed by Yamamoto et al. [8] for specimens of similar size. Second, at temperature low enough all the specimens failed by cleavage below 100 MPa $m^{1/2}$. On the contrary, above about 100–120 MPa $m^{1/2}$, the specimens either break by cleavage or do not break at all. For these last cases, the load–deflection curves exhibited a maximum at which the fracture toughness was conservatively calculated. The open symbols in Fig. 3 correspond to those points where cleavage was not triggered and for which the fracture toughness was calculated at maximum load. In order to characterize the shift of the effective fracture toughness-temperature curve, $K_e(T)$, the analysis was mainly focused in the lower transition region, i.e., below 100 MPa $m^{1/2}$. The K_e data points were empirically fitted with an exponential function $K_e(T) = 30 + 70\exp(\alpha(T - T_0))$, separately on the unirradiated and irradiated data. The coefficient α was: $\alpha_{unir} = 0.0316$ and

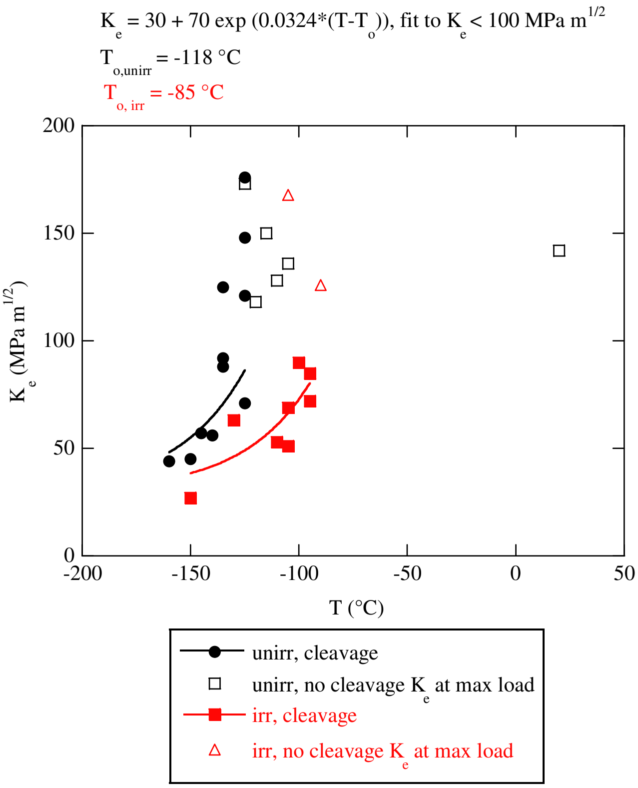

Fig. 3. Effective fracture toughness-temperature curves for unirradiated and irradiated mini-beams at $T_{irr} = 300$ °C and 0.5 dpa.

$\alpha_{ir} = 0.0332$ for the unirradiated and irradiated data respectively. Therefore, in order to determine the shift of T_0 due to irradiation, the average value of α ($\alpha = 0.0324$) was considered. Again, only the data points below 100 MPa m$^{1/2}$ were considered in the fitting. The fit to the unirradiated data yielded T_0 equal to -118 °C while that T_0 of the irradiated data, was found equal to -85 °C. Thus, a ΔT_0 shift of 33 °C was measured.

Tensile curves at different doses and at $T_{irr} = 50$ °C and 350 °C are presented along with the reference curve of the unirradiated specimen in Fig. 4(a)–(b). One salient observation is the fact that at $T_{irr} = 350$ °C, the uniform elongation increases in comparison to the unirradiated curve. This occurs typically for doses of about 0.3–0.4 dpa, beyond which the uniform elongation is strongly reduced. A careful look at the tensile curves at these low doses reveals that the initial strain hardening tends to be more linear than that of the unirradiated specimens, which presents a more pronounced curvature or equivalently a stronger strain-hardening reduction with strain. That strain-hardening behavior at small doses combined with a relative moderate increase of the overall flow stress results in an increase of the uniform elongation.

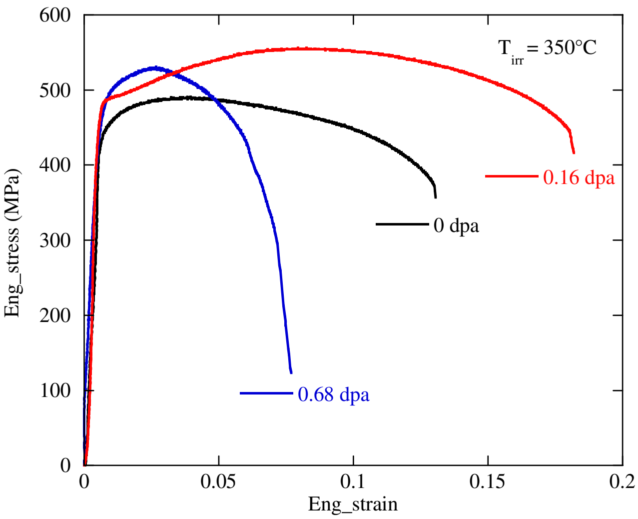

Fig. 4b. Typical tensile curves before and after irradiation at 350 °C, $T_{test} = T_{irr}$.

The irradiation hardening is presented in Fig. 5 against the square root of the dose, where a significant large dispersion of the results was measured. At these small doses, the irradiation hardening increases fast with dose so that any uncertainty in the dose determination translates into a significant variation of the yield stress. It is reminded that the doses reported here carry an uncertainty of the order ±20%. Note also that the irradiation temperature fluctuations, ±30 °C, associated with the proton beam instability, add to the overall scatter in the data. Despite these uncertainties in dose and irradiation temperature, it was possible to extract the general trend in the variation of the irradiation hardening $\Delta\sigma_{0.2}$ with dose and irradiation temperature. The initial rate of irradiation

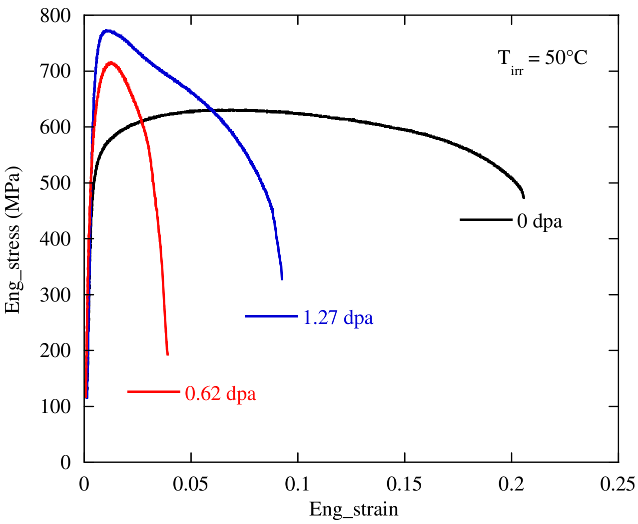

Fig. 4a. Typical tensile curves before and after irradiation at 50 °C, $T_{test} = T_{irr}$.

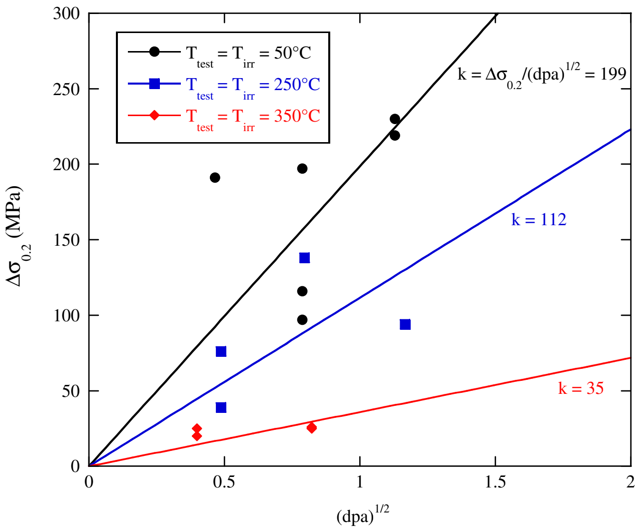

Fig. 5. Irradiation hardening versus (dose)$^{1/2}$.

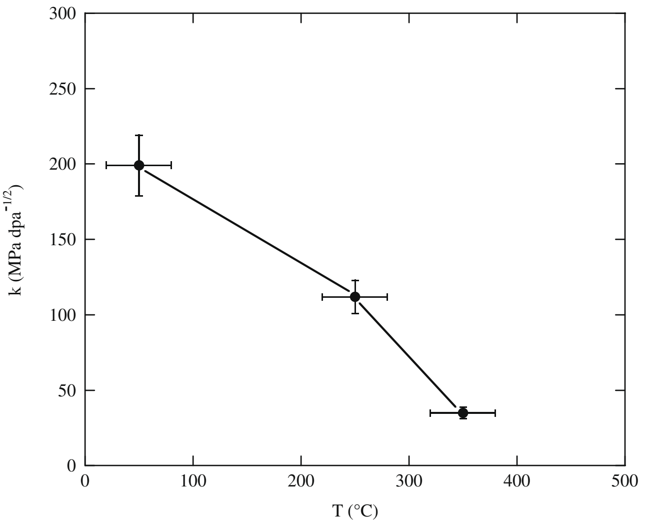

Fig. 6. Temperature dependence of the coefficient *k*.

hardening is commonly modeled by $\Delta\sigma_{0.2} = k(T_{irr})$ dpa$^{1/2}$, see for instance [9,10]. The coefficient *k* was calculated from our data and its temperature dependence is shown in Fig. 6. The indicated error bars corresponds to the fluctuations in the real irradiation temperatures and from the uncertainties in the calculated doses.

No tensile specimens were irradiated at 300 °C but the irradiation hardening at this testing temperature was estimated to be about 50 MPa. This value was deduced from the plots in Fig. 5 and in Fig. 6. In order to calculate the coefficient C_0, defined as $\Delta T_0/\Delta\sigma_{0.2\_RT}$ [8], it was necessary to convert the irradiation hardening at 300 °C (50 MPa) to the irradiation hardening at room temperature. A reasonable estimate of the irradiation hardening at $T_{test} = 20$ °C is 62 MPa. This estimate was obtained with the adjustment factor *F* derived by Rensman in [11], defined as $F = \sigma_{0.2}(T_{test})/\sigma_{0.2}(20\ °C)$, which is about 0.8 for $\sigma_{0.2}(T_{test} = 300\ °C)/\sigma_{0.2}(T_{test} = 25\ °C)$ and for the irradiation conditions and alloy corresponding to this study. Therefore, the ratio $\Delta T_0/\Delta\sigma_{0.2\_RT}$ is of the order 0.53 °C/MPa (33 °C/62 MPa). Such a value is in good agreement with previous estimates of C_0 calculated on other irradiated tempered martensitic steels [2,12,3]. We emphasize that these fracture toughness results are very encouraging and demonstrate that specimens of these dimensions can be used to quantify the embrittlement. The determined value of C_0 also indicates that the amount of helium content in our specimens remains too small to yield an additional temperature shift of the $K_e(T)$ curve that remains dominated by the irradiation hardening. This is consistent with the analysis of Yamamoto et al. [10] who estimated that that possible helium on embrittlement in the hardening regime may emerge only for concentration larger than 400–600 appm.

5. Conclusions

The tensile and fracture properties of the Eurofer97 were studied before and after 590 MeV proton irradiations. The irradiation hardening was measured at various doses, ranging from 0.16 to 1.3 dpa, and at different irradiation temperatures, namely 50 °C, 250 °C and 350 °C. The dose and temperature dependence of the irradiation hardening was characterized using a relation of the type $\Delta\sigma_{0.2} = k(T)$ dpa$^{1/2}$. At small doses(<0.3–0.4 dpa), the tensile tests indicated that the specimens kept a good ductility at the irradiation temperatures 250 °C and 350 °C, showing even an increase of uniform elongation. At higher dose, a strong decrease of the uniform elongation was observed.

The fracture properties were obtained from mini bend bars designed for PIREX irradiation facility. The temperature dependence of the static effective fracture toughness, K_e, was successfully determined in the lower transition region on both the unirradiated and irradiated condition (0.5 dpa at $T_{irr} = 350$ °C). Very steep $K_e(T)$ curves were found and the associated temperature shift ΔT_0 of the $K_e(T)$ was related to the irradiation hardening $\Delta\sigma_{0.2\_RT}$. The ratio ($\Delta T_0/\Delta\sigma_{0.2\_RT}$) was about 0.53 °C/MPa. Such a value is in very good agreement with previously published data on other irradiated reduced activation tempered martensitic steels.

Finally, it was mention that, for the dose levels and irradiation temperatures investigated, there is no indication that the helium produced by the 590 MeV protons (170 appm/dpa) influences in any way the tensile and fracture properties.

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