Evolution of the tensile properties of the tempered martensitic steel Eurofer97 after spallation irradiation at SINQ

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ABSTRACT
Small disk tensile specimens of the reduced activation tempered martensitic steel Eurofer97 were tested from −100 °C up to 370 °C before and after irradiation in the spallation source SINQ at Paul Scherrer Institute. The calculated irradiation dose and helium content are respectively 11 dpa and 540 appm. Even for such a large dose, the tensile curves showed a significant total elongation at all testing temperatures. Fractographic observations revealed a predominance of cleavage facets at −100 °C for unirradiated and irradiated specimens while at higher temperature the fracture surfaces were dominated by ductile dimples. True stress/strain curves were derived using a finite element based inverse method for simulating small disk tensile tests. The finite element simulations indicated that the local fracture stress in the neck is consistent with that calibrated based on fracture tests and that the micro-mechanisms of fracture were not affected by helium up to 540 appm.

1. Introduction

The reduced activation tempered martensitic steel Eurofer97 will be used in the International Thermonuclear Experimental Reactor (ITER) as a structural material for test blanket modules [1]. In order to qualify Eurofer97 for applications in the irradiation environment of a fusion reactor, it is from utmost importance to develop a solid material properties database under both unirradiated and irradiated conditions. Tempered martensitic steel irradiated in a fusion neutron irradiation environment will have to sustain irradiation damage (dpa) along with accumulation of helium (He appm) produced by transmutation reactions in a ratio of about 10 He appm/dpa. Currently, there is no intense fusion neutron irradiation facility for irradiation representative of fusion reactors. Irradiation in the Swiss spallation neutron source (SINQ) with a mixed spectrum of high energy protons and spallation neutrons has long been successfully used as a surrogate to study the evolution of material properties at large dose and high helium concentration [2], even though the ratio He appm/dpa is somewhat larger than for fusion reactors 30–85 He appm/dpa [3].

While testing of unirradiated materials can be easily performed with standard specimens, small specimens must be applied to irradiated materials [4–6] not only because limited volumes are available in most irradiation facilities, but also to reduce dose and temperature gradients through the samples. This point is critical for irradiation facility like SINQ or the planned International Fusion Materials Irradiation Facility (IFMIF) [7] where the neutron flux is strongly anisotropic and rapidly varying in the target.

In this work, we were interested in determining the plastic flow properties and fracture behavior of Eurofer97 steel after irradiation in SINQ, here to a dose of about 11 dpa and accumulated helium concentration of about 540 appm. To do so, a tensile specimen with an unusual geometry (disk tensile specimen, DTS) were used. The DTS geometry departs considerably from standard tensile specimen geometry so that non-uniform stress/strain distribution within the gage section arises even at low strain. Consequently, the determination of the constitutive behavior, in terms of true stress/plastic strain curve is not straightforward. A simple workaround to extract the material constitutive behavior is to apply an inverse finite element model (FEM) procedure, e.g. [8–13]. Contrary to standard FEM analysis, the inverse approach uses the experimental force/displacement curve as an input and outputs the material properties obtained in an iterative manner. In this paper, we present successively: tensile test results on unirradiated and irradiated Eurofer97 over a range of temperature, optical and scanning electron microscopy observations of fracture surfaces, some details of the inverse method developed in the frame of this work, and some finite elements calculations of the stress in the specimen neck at fracture.

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2. Material, experimental procedures and finite element

The reduced activation tempered martensitic steel Eurofer97 is the reference structural material for test blanket modules in ITER. The specimens used in this study were cut out the 14 mm plate (heat number E83698) produced by Böhler AG. This plate was normalized for 27 min at 980 °C, followed by 1.5 h tempering at 760 °C and air cooled. The chemical composition in wt% is: 8.93 Cr, 0.49 Mn, 1.08 W, 0.2 V, 0.15 Ta and 0.12 C. Tensile tests were conducted using non-standard disk tensile specimen. A sketch of DTS is shown in Fig. 1. Due to the small dimensions of the specimens, it is not possible to measure directly the elongation of the specimen gage length. Therefore, the strain was calculated from the displacement of the machine traverse that was corrected from the compliance of the load train.

The specimens were irradiated in the frame of the fifth experiment of the SINQ Target Irradiation Program (STIP-V). UCSB selected unusual DTS geometry to stack the specimens in irradiation capsule tubes and to facilitate the radial heat transfer with press fit gaps so as to minimize the temperature variations and uncertainties in the specimens. The nominal calculated dose and helium concentration experienced by the DTS are 11 dpa and 540 appm He respectively. The calculated irradiation temperature based on the beam energy deposition is around 250 °C. The tensile tests were conducted with a Zwick Z010 at constant cross-head velocity corresponding to a nominal strain rate of about 5.55 × 10^{-4} s^{-1}. An environmental chamber from Instron (ECD86 D) was used to conduct tests at low and high temperatures.

An initial 3D finite element (FE) model for the ABAQUS code described in detail in [12] was slightly modified by increasing the mesh density across the thickness, as well as at the shoulders of the DTS. The FE model contains 52,892 linear hexahedral elements of type C3D8R. The elastic properties of the material are characterized by the Young's modulus and the Poisson's ratio. Isotropic hardening J2 flow theory was implemented with true stress versus true plastic strain input in tabular form. Loading of the specimen was performed in a way that simulates the experiment, where one specimen head is clamped and displaced, while the other head is clamped but remained fixed (see Fig. 1). A reference point is used to calculate the load/displacement curve in order to easily access and process the data with the inverse algorithm.

3. Experimental and simulation results

The unirradiated as well as the irradiated DTS were tested at temperatures ranging from −137 to 370 °C. Only five irradiated specimens could be tested whereas nine unirradiated tests were conducted. The resulting tensile curves in terms of engineering stress/strain are shown in Fig. 2.

The irradiation induces profound changes of the shape of the tensile curves. In the unirradiated case, a gradual transition between elastic and plastic deformation can be found with significant uniform elongation. The tensile curves of the irradiated specimens performed between −100 and 200 °C are characterized by a premature necking that occurs just beyond the yield point, followed by a continuous decrease of engineering stress (or load). At 370 °C, the tensile curve shows a low strain-hardening rate, which persists up to several percent engineering strain.

For the tests performed on unirradiated specimens from −100 °C up to 370 °C, the failure of the specimens, regarded as the total separation of the specimen in two parts, occurs over a range of strain that follows the sudden load drop characterized by an abrupt slope change of the tensile curve towards the end of the test. This behavior arises from the fact that the specimens fail only partly at this point but a remaining ligament still holds the two specimens halves together, which continue to plastically deform. At lower testing temperatures, i.e. −120 °C and −137 °C, the unirradiated DTS fail at high stress in a total macroscopic brittle manner as indicated by a vertical dashed line (see Fig. 2 - left). A similar result was found for the irradiated DTS. However, there was not such a clear trend of this phenomenon versus temperature. From five tests with irradiated specimens, only two show such a behavior.

A well-behaved temperature dependence of the yield stress at 0.2% of plastic strain below room temperature is observed for both unirradiated and irradiated Eurofer97, which is characteristic of BCC metals and alloys. The yield stresses for the irradiated and the unirradiated DTS results are compared in Fig. 3-left. When compared with the unirradiated yield stress of the same Eurofer97 batch presented in [14] for instance, our yield stress data are about 20 MPa smaller. Since the 0.2% plastic strain is deduced from the machine compliance corrected elongation and owing to the fact the DTS geometry deviates significantly from the recommended one in the ASTM standard, some deviation in σy yield with respect to standard specimens is expected. Nonetheless, the difference is about 3% only, which remains within the usual experimental scatter. Significant irradiation-hardening is observed, as can be seen in Fig. 3-right. The irradiation-hardening in Fig. 3-left shows a stronger test temperature dependence than cannot be explained by the temperature dependence of the elastic constant.

The irradiation-hardening values at 20 °C and 200 °C are compared to previously published data in Fig. 4, [15]. At Ttest = 20 °C, the increase in yield stress is about 517 MPa, which is in good agreement with the dataset obtained after irradiation at SINQ from Y. Dai. The lines in the left plot of Fig. 4 represent the neutron irradiation hardening and the saturation trend at high dose. From the single data point of this study, only a small amount of additional hardening is indicated. However, for the irradiation conditions considered here (11 dpa and 540 appm He) the hardening is essentially in line with neutron irradiation. A similar conclusion can be drawn from the DTS tested at 200 °C. The irradiation-hardening is about 453 MPa, which lies close to the dashed saturation curve (Fig. 4-left), [16]. Thus we conclude that the He contribution to irradiation-hardening is minimal at 540 appm He.
The necking behavior was characterized by optical microscopy of both the unirradiated and irradiated specimens at each testing temperature. Top view images of the gage section of the tested DTS are shown in Fig. 5. All the unirradiated DTS exhibited a diffuse necking behavior and failed at a fracture plane normal to the loading axis. From the curved shape of the fracture surfaces, it appears that fracture initiated in the middle of the specimens and propagated towards the edges of the gage section, analogous to cup-cone fracture in round tensile specimens. Qualitatively, the shape of the necked region of the irradiated specimens is similar to those in the unirradiated condition, except that the specimens tested at 20 °C and 200 °C that show a more localized necking, with fracture surfaces inclined with respect to the loading axis. At −50 °C, a slight inclination of the fracture plane is also observed.

Detailed SEM analyses were carried out on the fracture surfaces of the unirradiated and irradiated DTS tested at −100 and 200 °C in order to assess possible irradiation effects on fracture mechanisms. Fig. 6 shows the fracture surface observations of an unirradiated DTS at 200 °C. The general view (Fig. 6a) shows a typical flattened fracture surface normal to the tensile axis. The higher magnification in Fig. 6b and c show that the surface is covered with small, equiaxed shallow and dimples (Fig. 6c). Smaller areas near the center of the gauge section exhibit more complex, rougher fracture surfaces, consisting of shallow equiaxed dimples along with deeper conical dimples and micro-void coalescence (Fig. 6b). This behavior can be attributed to higher and more triaxial stresses that initiate the first damage that ultimately form a central crack that propagates to the specimen edges at the point of final fracture.

Fig. 7 shows the fracture surface of an irradiated specimen tested at 200 °C, which failed on a plane inclined by ∼70° with respect to the tensile axis. The appearance of the surface is more irregular than that of the unirradiated specimen. A major difference between the unirradiated and irradiated fracture surface is that in the latter case, quasi-cleavage facets up to ∼50 µm are observed (see Fig. 7b). The quasi-cleavage facets are distributed over the whole fracture surface, with the largest located off the loading axis as illustrated in Fig. 7a. The irradiated fracture surface is also covered with equiaxed dimples (Fig. 7c).

The fracture surfaces were also investigated at −100 °C. The unirradiated fracture surface has a normal to the tensile axis and appears quite irregular (Fig. 8). There are many cleavage facets with size and shape corresponding to laths and lath packets (Fig. 8b)). Numerous secondary out of plane cracks are visible over much of the fracture surface. The secondary cracks, indicated by the white arrows, run in the axial direction, reaching surface lengths of about 120 µm with very large surface opening widths. The largest cracks are located off the tensile axis. In addition to the small cleavage facets, some regions exhibit ductile tearing are indicated by the presence of small dimples in Fig. 8c.

The fracture surfaces of the unirradiated and irradiated specimens tested at −100 °C are similar in some respects (see Fig. 9): the surfaces...
are topologically irregular and lie normal to the tensile axis. Both contain out of plane cracks (white arrows) arranged parallel to the width of the specimen. The largest crack was around 200 µm in length and 29 µm in width. Wide areas of the fracture surface contain cleavage facets (Fig. 9b) that appear at the edges as well as in the center of the specimen. Only a few dimples are observed in the irradiated conditions,
as seen in Fig. 9c.

In order to calculate the stress components of the brittle specimens at −100 °C at failure, the engineering stress/strain curves presented in Fig. 2 were converted into true stress/strain curves using an inverse method, which we previously developed for the DTS in [12]. This inverse procedure had to be modified to improve the accuracy of reconstructions for the irradiated conditions. We focused on the reconstruction of the experimental curves on the strain region up to the first major load drop in the tensile curves. The DTS FE model described above was used in combination with the experimental results to iteratively predict the experimental engineering stress/strain curve based on the true stress/strain elastic and plastic flow properties. Reaching a convergence of the predicted engineering stress/strain curve based on a self-consistent true stress/strain true constitutive law is the essence of the inverse method. A piece-wise linear reconstruction of the true stress-strain curve was chosen as the means of implementing the inverse calculations. From a practical point of view, the load-displacement curve of the DTS (see Fig. 10) can be divided into three regions:

- **Region I**: Elastic deformation.
- **Region II**: Plastic deformation up to maximum load and onset of necking.
Region III: Necking to failure.

To initiate the inverse algorithm, the following first iteration data are required:

- The experimental engineering stress/strain curve.
- A good estimate of the elastic slope and proportional limit $\sigma_0$, that is different than the yield stress $\sigma_{\text{yield}}$.
- The ABAQUS input file describing the specimen with the boundary and loading conditions.
- The initial displacement increment that was set to $d_{\text{inc}} = 0.001$ mm.

The engineering stress and strain are represented by $s$ and $e$, and the true stress, true strain and plastic true strain by $\sigma$, $\epsilon$ and $\epsilon_p$. The experimental and simulated engineering stress/engineering strain are calculated by

$$s_{\text{exp}} = \frac{P_{\text{exp}}}{A_0}, \quad e_{\text{exp}} = \frac{d_{\text{exp}}}{l_0}$$

$$s_{\text{sim}} = \frac{P_{\text{sim}}}{A_0}, \quad e_{\text{sim}} = \frac{d_{\text{sim}}}{l_0}$$

(1)

Here, $P$ is the load, $d$ the displacement, $A_0$ the initial specimen cross-section and $l_0$ the initial gage length.

Region I: The elastic Poisson’s ratio $\nu$ is taken equal to 0.3 while the measured elastic slope was fitted with the inverse method. The basic reason to fit the elastic slope is that it differs a little bit from specimen to specimen. In order to have a precise measure of the plastic strain for each specimen, a precise determination of the elastic slope is necessary. The experimental maximum elastic engineering stress $e_0^{\text{exp}}$ is naturally defined at the point on the experimental curve marked by a deviation from linearity between $s_{\text{exp}}$ and $e_{\text{exp}}$ (see Fig. 11). This point is defined by the pair ($s_0^{\text{exp}}, e_0^{\text{exp}} = d_{\text{exp}}/l_0$). In an iterative manner, the region I of the $s_{\text{sim}}(e_{\text{sim}})$ curve is generated by adjusting the value of the elastic slope until the following criterion is met:

![Fig. 9. Analysis of the tensile fracture surface of an irradiated specimen tested at $-100$ °C.](image)

![Fig. 10. Left: Engineering stress/strain curve indicating the three regions. Right: Piece-wise reconstruction of the material properties as (true stress, plastic strain) pairs.](image)
\[ \Delta s = (s_{\text{sim}}^i - s_{\text{exp}}^i) \leq \alpha \text{ at } d_i \]  

Here \( s_{\text{sim}}^i \) corresponds to the simulated engineering stress at \( d_i \) and \( \alpha \) denotes the maximum allowable error. This procedure is schematically shown in the left side of Fig. 11.

**Region II & III:** The experimental engineering stress/strain curve is now divided into \( n \) small segments. Each of them corresponds to a certain displacement increment \( d_{\text{sim}} \) in the load/displacement curve, which causes an increment of plastic deformation. For each increment, a corresponding pair of true stress/true plastic strain values can be determined. For the sake of clarity, the details of the algorithm for the first increment are as follows. The FE model is updated by imposing a total displacement such as \( d = d_{\text{et}} + d_{\text{inc}} \), and assuming a constitutive law with a proportional limit equal to \( \sigma_0 \text{sim} \) followed by plastic behavior characterized by a linear strain hardening rate, \( K_i \) (see Fig. 10 right). As in Region I the slope of \( K_i \) is adjusted until the criterion that

\[ \Delta s = (s_{\text{sim}}^i - s_{\text{exp}}^i) \leq \alpha \text{ at } d_i \]  

is fulfilled (see Fig. 11 right). Knowing \( K_i \) is not enough at that point because we are essentially interested in the pair \( (\sigma_{\text{p}}, \epsilon_{\text{p}}) \). The true stress/true plastic strain is extracted from the FE model by calculating the average von Mises stress \( \sigma_{\text{p}}^\text{sim} \) and true plastic equivalent strain \( \epsilon_{\text{p}}^\text{sim} \) from the stress and strain tensor elements that contribute to deformation. At the onset of plastic deformation, these are simply the elements with \( \epsilon_{\text{p}} > 0 \). However, at intermediate displacement of the DTS FE model, the elements have to be selected differently. If Eq. (3) is fulfilled a true stress - plastic strain pair for the current incremental deformation can be extracted by considering only the elements whose deformation increment at \( i \) exceeds that of the previous increment \( i-1 \). Those elements are then used to derive the average plastic equivalent strain \( \epsilon_{\text{p}}^\text{sim} \) and true stress \( \sigma_{\text{p}}^\text{sim} \) as

\[ \epsilon_{\text{p}}_{\text{sim}} = \frac{\sum_{j=1}^{N} \epsilon_{j}^{\text{p}} \cdot V_{j} \cdot (\epsilon_{j}^{\text{p}} > \epsilon_{j,i-1}) \cdot V_{j} \cdot (\epsilon_{j}^{\text{p}} > \epsilon_{j,i-1})}{\sum_{j=1}^{N} V_{j}^{\text{p}} \cdot (\epsilon_{j}^{\text{p}} > \epsilon_{j,i-1}) \cdot V_{j} \cdot (\epsilon_{j}^{\text{p}} > \epsilon_{j,i-1})} = \epsilon_{\text{p},i} \]

\[ \sigma_{\text{p}}_{\text{sim}} = \frac{\sum_{j=1}^{N} \sigma_{j}^{\text{p}} \cdot V_{j} \cdot (\epsilon_{j}^{\text{p}} > \epsilon_{j,i-1}) \cdot V_{j} \cdot (\epsilon_{j}^{\text{p}} > \epsilon_{j,i-1})}{\sum_{j=1}^{N} V_{j}^{\text{p}} \cdot (\epsilon_{j}^{\text{p}} > \epsilon_{j,i-1}) \cdot V_{j} \cdot (\epsilon_{j}^{\text{p}} > \epsilon_{j,i-1})} = \sigma_{i} \]

where \( V_{j} \) is the volume of element \( j \) and \( \sigma_{j} \) is the von Mises stress of the element. \( N \) denotes the number of nodes within the extended gage length \( L_{\text{ext}} \).

In Fig. 12, the reconstructed true stress/strain curves of unirradiated and irradiated DTS using the inverse method described above are shown.

The unirradiated flow stress curves show a gradual transition from the onset of plastic deformation. As a matter of fact, the true stress-strain curves were recalculated with the inverse method up to very large strains (>100%). From an engineering point of view, only the first part of the curves, up to around 10–15%, is relevant. Indeed, 10–15% of strain represent a large structural limit. In addition, for finite element simulations of cracked components or structures with stress concentrators it is necessary to know the plastic flow properties up to the typical levels attained in the process zone around cracks or stress concentrators, which typically do not exceed 10–15%. It is here assumed and expected that up to 10–15% of plastic strain no internal damage occurs in the materials. The softening at very large plastic strains is the consequence of the reconstruction of the load-displacement curves in Fig. 2 up to the final drop-off. This softening can certainly be attributed to damage development in the form of micro-voids and/or micro-cracks that must occur at larger strains but which is not explicitly modeled in our approach.

The irradiated flow curves show a gradual transition from the elastic to plastic regime only at the highest temperature of 370 °C. At lower temperatures, the elastic-plastic transition is quite abrupt. At 200 °C and 20 °C, a small strain range of perfectly plastic Lüders-type behavior occurs. At the lowest two temperatures of −50 and −100 °C show a feature similar to a yield drop followed by strain-hardening. The softening at higher plastic strains close to failure is also observed in the irradiated case. This again can be attributed to damage accumulation in the neck. Interestingly, the tensile true stress-strain after irradiation clearly show that, even after a dose of 11 dpa, the tempered martensitic steel keep a significant strain-hardening capacity as can be seen in the initial part of the curve (<10%) where no internal damage is expected.

The true stress-strain curves derived with the inverse method at −100 °C were used as input in FE models to calculate the stress state at failure of the DTS. Since at this temperature, the fractographic observations shows a dominance of small cleavage facets, the calculated fracture stress defined as the maximum principal stress, was compared to the calibrated fracture stress on Eurofer97 for cleavage fracture deduced from fracture toughness test experiments. The nominal fracture stress values of the unirradiated and irradiated DTS was respectively found equal to 1700 and 2000 MPa (see Fig. 13), which are in very good agreement with the value of local fracture stress determined with fracture toughness tests [17,18].

This result indicates that for the irradiation conditions of this study, 11 dpa and 540 appm He, the micro-mechanisms of fracture are not significantly affected by helium. This is in general consistent with the embrittlement analysis done by Yamamoto et al. on tempered martensitic steels that indicate that the so-called He non-hardening embrittlement effect emerges for concentration higher than 400–600 appm [19]. Thus, the He concentration attained in our specimen is likely to be...
too low to induce a change in the local micro-mechanisms of fracture. Our tensile test data are also in good agreement with the conclusion drawn by Wang et al. [20] who showed for F82H steel that only specimens with a dose and He content greater than 15 dpa and 1370 appm respectively present intergranular fracture mode along with pure elastic loading up to fracture.

4. Conclusions

Eurofer97 disk tensile specimens were tested from −100 °C to 370 °C before and after irradiation in the spallation neutron source SINQ at Paul Scherrer Institute. The calculated nominal irradiation dose was 11 dpa and 540 appm He and the irradiation temperature was about 250 °C. The irradiation hardening was measured over the selected temperature range. At 20 °C and 250 °C, the irradiation hardening of 450 MPa and 520 MPa respectively was found in very good agreement with previous spallation irradiation data as well as an empirical neutron irradiation-hardening model. There is little or no additional contribution of helium to hardening compared to neutron irradiation that produces only a small amount to helium. All irradiated tensile curves had a very low uniform elongation except at 370 °C.

The true stress-strain curves were determined with an inverse method. The derived curves show that irradiated Eurofer97 maintains a significant strain-hardening capacity. The maximum principal stress on the crack plane, calculated by finite elements simulations using the reconstructed true stress/strain curves at −100 °C, was found to be about 1700 MPa and 2000 MPa for the unirradiated and irradiated specimens respectively. These values of fracture stress are quite similar to the local fracture stress determined by pre-cracked toughness tests on various reduced activation tempered martensitic steels. This also shows that the micro-mechanisms of fracture are not significantly affected by the helium concentration of ≈500–600 appm. This is further corroborated by the fractographic observations, which showed that the fracture mode remained transgranular cleavage rather than intergranular fracture that is observed at higher helium levels.

Conflict of interest

No conflict of interest have been identified.

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Fig. 12. True stress/strain curves of the unirradiated and irradiated DTS at different testing temperatures determined by the inverse method.

Fig. 13. Calculated maximum principal stress on the fracture at −100 °C of unirradiated and irradiated specimens.
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