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In situ fatigue of the Eurofer 97 steel

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Abstract

In order to investigate the fatigue behavior during irradiation of the EUROFER 97 steel, similar to that which would occur in a fusion reactor, a series of six *in situ* experiments was conducted using a specially designed proton irradiation system. All tests were performed at a total strain range of 0.8%, at 150 and 250 °C. In two experiments, to check the influence of the dislocation structure, the specimens were first fatigued prior to the irradiation and then only fatigued under beam. In one case, when the specimen reached the predicted number of cycles to failure, the beam was turned off and the test continued without a beam. Unexpectedly, the specimen recovered and attained almost the same number of cycles as without the beam irradiation. Secondary experiments were conducted for measuring the irradiation hardening under static stresses, as well as for measuring the flow stress under irradiation.

1. Introduction

First-wall materials used in fusion machines will be stressed and locally deformed under simultaneous irradiation with energetic neutrons. This particular condition has not been studied very often, due to the complicated and expensive experimental facilities required (see [1] for a short review). Most results available describe the evolution in terms of structure and mechanical properties of the *as irradiated* material, after it has been irradiated to the goal level. The properties of post-irradiation tested material, in some cases, may be very different from properties and structure of an *in situ* tested material, for the following reasons. Firstly, under simultaneous deformation, the irradiation takes place in a microstructure in evolution which may, depending on the mechanical conditions imposed, be very different from the *as received* condition. Secondly, enhanced diffusion of point defects and solute atoms may take place and secondary damaging mechanisms, detrimental to the mechanical properties, may occur. Finally, the moving dislocations interact with the irradiation induced defects. The irradiation substructure during *in situ* irradiations can be expected to be different. Consequently, depending on the level of plasticity in the material, irradiation hardening takes place at a smaller rate or disappears completely [2,3]. For all these reasons, it is obvious that the *in situ* condition deserves attention in future programs. The effects on the materials are not simply predictable; in some cases, as for Cu–Cr–Zr alloys [4], the *in situ* condition seems to be beneficial for the fatigue endurance. In the case of F82H, it was

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clearly detrimental since the *in situ* fatigue life was reduced compared to a post-irradiation tested specimen [1]. In this work, we describe the results obtained for Eurofer 97, a material similar to F82H but containing a lower level of W.

2. Experimental details

2.1. Material

The chemical composition in wt.% of Eurofer 97, Heat Nr 83697, was Cr:8.93, V:0.2, W:1.07, N:0.018, C:0.12 and Ta:0.14. The material was delivered in the form of 25 mm plates. The final heat treatment applied was: austenitizing at 980 °C for 31 min, followed by air cooling and tempering at 760 °C for 90 min, and subsequent air cooling.

2.2. Irradiation conditions, specimens

The irradiation was carried out with 590 MeV protons, which have been shown in the past to be, at low doses, an adequate simulation for 14 MeV neutrons. The proton beam had a Gaussian distribution, adjusted to a size of $4\sigma_x = 6$ mm and $4\sigma_y = 3$ mm, where σ_x and σ_y are the beam standard deviations in x and y directions. The beam was then wobbled with an amplitude of 2.7 mm along the gauge length, with a frequency of about 3 Hz, to achieve a constant dose distribution. The beam intensity was 12–14 μA. Correspondingly, the proton flux was 4.074–4.75×10^{14} p/cm^2 s and the mean current density 65–76 μA/cm^2. Medium energy protons produce heat. For this reason, the tubular in-beam specimen, having a cross section of 2.5×3.4 mm and a gauge length of 5 mm, was cooled by a flow of pressurized helium [2]. A classical plain cylindrical specimen having a diameter of 2.7 mm and a gauge length of 6 mm was tested in a conventional testing machine, for comparison purposes. The 590 MeV protons also generate helium and hydrogen in the alloy. The calculated values for this ferritic steels were 195 and 956 appm/dpa [5,6], respectively.

The irradiation parameters relevant to the experiments are given in Table 1. The indicated dpa dose is the result of a dosimetry analysis.

2.3. Mechanical tests

The fatigue test was conducted under strain control, following an $R = -1$ symmetrical signal. All experiments were conducted at a total strain amplitude of 0.8 % (half-strain amplitude 0.4%). The test frequency of the specimens tested in the *in situ* device was chosen to be 0.005 Hz ($T = 200$ s) for the in-beam test and 0.01667 Hz ($T = 60$ s) for the tests without a beam. The test frequency chosen for testing the plain specimens was 0.125 Hz ($T = 8$ s). Below 300 °C, the test frequency is not expected to influence the mechanical behavior; therefore, to save time, the test frequency was

Table 1
The mechanical tests and irradiation parameters for 590 MeV proton irradiation

	I29I02	I29I03	I29I05	I29I06	N29I04	N29I01	N29F24	N29F25
Irradiation temperature T_{irr} (°C)	150	250	250	150	–	–	–	–
Deformation temperature T_{def} (°C)	120	219	221	125	150	250	120	220
$\Delta\varepsilon_{\text{tot}}$ (%)	0.80	0.80	0.80	0.80	0.80	0.79	0.82	0.82
Cycle length T (s)	200	200	200	200	60	60	8	8
$\dot{\varepsilon}$ (s^{-1})	8×10^{-5}	8×10^{-5}	8×10^{-5}	8×10^{-5}	2.67×10^{-4}	2.67×10^{-4}	2×10^{-3}	2×10^{-3}
Accumulated current (beam current integration) D (A s)	3.742	3.042	2.667	2.809	No irradiation	No irradiation	No irradiation	No irradiation
Accumulated dose (dosimetry analysis) D (dpa)	0.21	0.17	0.15	0.16	No irradiation	No irradiation	No irradiation	No irradiation
N_a (–)	1499	1259	1505	2643	2881	2904	11614	5815

Specimen N29I04 and N29I01 have been tested without a beam, but in a helium atmosphere, in the *in situ* device. Predicted helium production ratio: 195 appm/dpa [5]. Damage ratio: $\dot{D}/\dot{\varepsilon} = 1.5 \times 10^{-2}$ dpa. Dose rate: $\dot{D} = 1.2 \times 10^{-6}$ dpa/s. I29I05 and I29I06 received a pre-deformation of 250 and 300 cycles, respectively. The unit for beam current dose is Ampère second [A s].

increased for the comparison tests. During all mechanical tests, the irradiation temperature of the specimen, was kept constant, taking into account beam heating. The temperature was monitored by two thermocouples, spot welded on the gauge length. At the center of the beam spot, the temperature corresponded to the irradiation temperature (given in Table 1) which was the sum of the cooling gas temperature and the temperature increase from beam heating. But, because of the wobbler, the temperature fluctuated locally below the irradiation temperature by approximately 30 °C. This rather high value was a result of the chosen wall thickness of 450 μm. Therefore, the real deformation temperature was close to the cooling gas temperature, T_{def}, as given in Table 1. The end-of-life criterion used to compare the fatigue life times was when the main crack penetrated the specimen wall ($N = N_{\text{a}}$). This point was clearly indicated in the in-beam experiment by a failure of the vacuum produced by released helium. In addition, this point corresponded to an inflexion point at the bottom of the strain–stress hysteresis loop, which was also clearly detectable in the plain material specimens. N_{a} is considered a better parameter for comparison since, at cycles higher than N_{a}, the mechanical conditions of the *in situ* experiments are modified. The mechanical parameters of the fatigue tests are given in Table 1.

3. Results and discussion

Fig. 1 shows the results of the first two experiments, I29I02 ($T_{\text{irr}} = 150$ °C) and I29I03 ($T_{\text{irr}} = 250$ °C). The life of both in-beam tested specimens is reduced by a factor two, compared to the specimens tested without beam. No significant temperature effect is present, whereas hydrogen effects in steels are strongly temperature dependent and effective up to 300 °C. This is an indication of the limited role of atomic hydrogen produced by the spallation reactions, since, in the presence of hydrogen, embrittlement should be more severe at these low temperatures.

The softening behavior is consistent with the behavior generally observed in ferritic–martensitic steels. The cyclic stress amplitude decreases rapidly in the first cycles and then nearly saturates at a value which decreases slowly as a function of the number of cycles. Correspondingly, the strong increase of the plastic strain observed in the first cycles is followed by a second stage in which the plastic strain increases only slowly. This behavior is found in the specimens with or without beam. I29I03 irradiated at 250 °C shows a higher level of plastic strain. This effect was also observed in the past in Cu–Cr–Zr [4,7], but was not reported for F82H and MANET II [1,8]. As explained in the previous paragraph, the temperature to be considered for

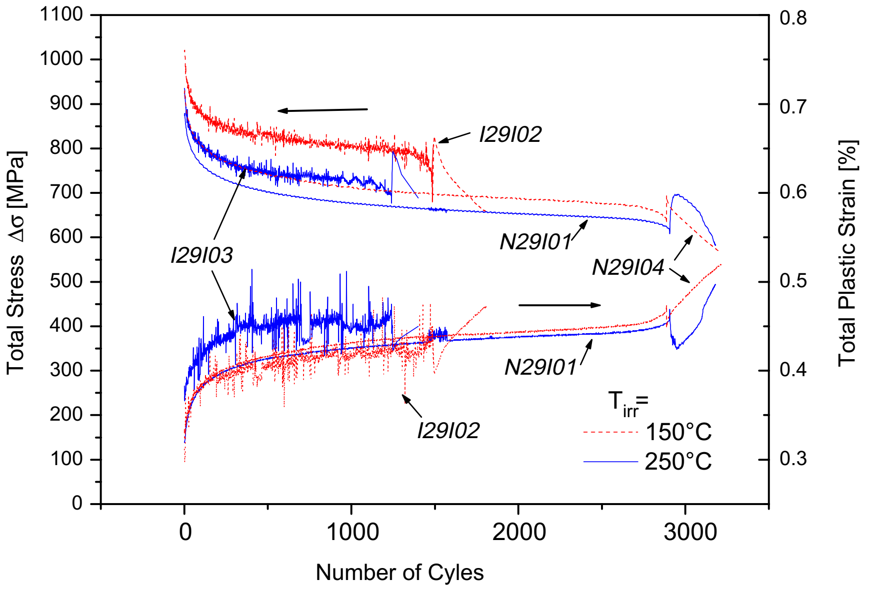

Fig. 1. Total stress and total plastic strain as a function of the number of cycles, for the in-beam experiments.

the mechanical response is near the deformation temperature, T_{def} (see Table 1). The in-beam stress curves in Fig. 1 have lower deformation temperatures than the curves without beam. Nevertheless, the increased stress level found in the in-beam specimens is not a direct indication of irradiation hardening. To assess the irradiation hardening, the curves have been compared as in a previous study [9] for Eurofer 97, on a log–log scale, to the curves of specimens tested at T_{def} (see Fig. 2).

The slope of the in-beam softening curves is clearly lower than the slope of the corresponding curves for unirradiated material. The difference can be attributed to irradiation hardening. In past studies [1,2], it has already been shown that the main parameter controlling the mechanical effects in *in situ* fatigue experiments was a so-called damage ratio $\dot{D}/\dot{\varepsilon}$, where $\dot{D}$ is the dose rate and $\dot{\varepsilon}$ is the strain rate. The damage ratio chosen in this work (1.5×10^{-2} dpa) is higher than the damage ratios of previous experiments on F82H and MANET II. Accordingly, radiation-induced hardening is clearly shown in this work and is more intense at the lower irradiation temperature, whereas it was observed to a smaller extent in the previous experiments. The elevated plastic strain in experiment I29I03 is probably a combined effect of the high damage ratio and higher temperature.

In the next two experiments, the specimen was fatigued before irradiation, in order to develop an initial microstructure containing depleted dislocation zones. Cyclic deformation transforms the lath structure in unirradiated material to an equiaxed subgrain structure with significant dislocation-free areas [10]. Segregation of impurities to boundaries and resulting embrittlement are enhanced in such a microstructure. Such an effect was probably observed previously in MANET steels [11]. Fig. 3 presents the results of I29I05 after a pre-deformation of 250 cycles and then *in situ* testing, at $T_{\mathrm{irr}} = 250$ °C. The life is decreased by a factor of about two. Compared with the experiment without pre-deformation, the number of cycles under beam is identical, thus indicating the absence of any effect in Eurofer 97. Again, the plastic deformation is increased, as was observed for I29I03.

Fig. 4 shows the effect of 300 cycles pre-deformation, then *in situ* testing, at an irradiation temperature of 150 °C. No additional embrittlement was observed since the specimen was still in good condition after a number of cycles equal to the number of cycles reached by the specimen without pre-deformation. At this point, the beam was turned off but the fatigue continued. The recovery capacity of the material was surprising, since the specimen reached almost the number of cycles to failure of the mate-

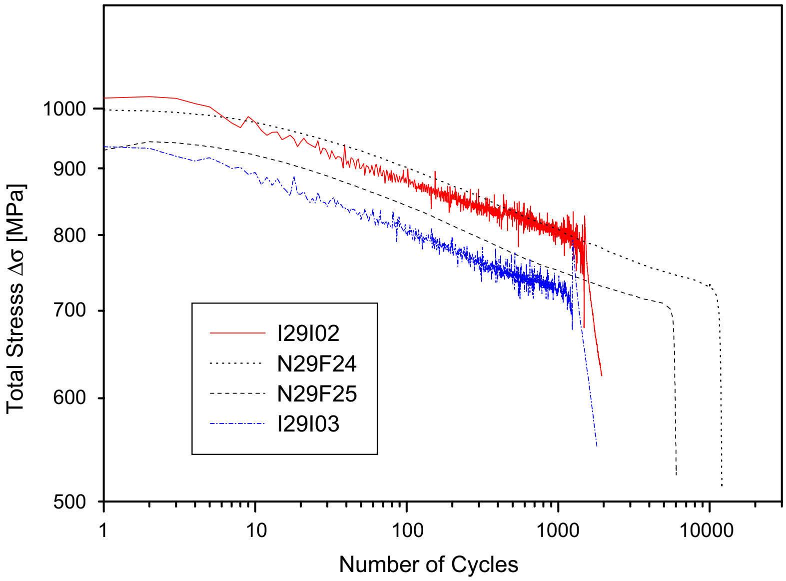

Fig. 2. Softening curves of I29I02 and I29I03 during irradiation, compared to the softening curves of unirradiated material N29F24 and N29F25 for similar test temperatures (see Table 1).

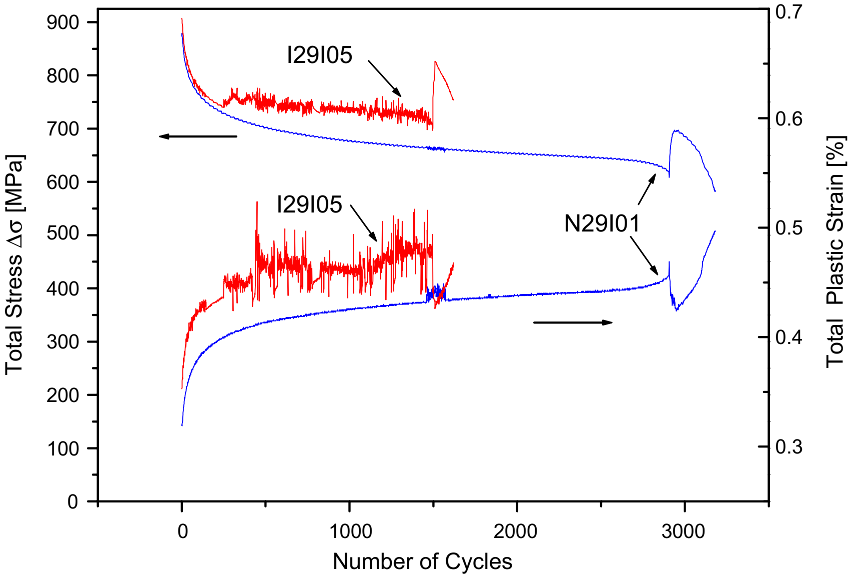

Fig. 3. Total stress and total plastic strain as a function of the cycle number for a pre-deformation of 250 cycles. $T_{irr} = 250$ °C.

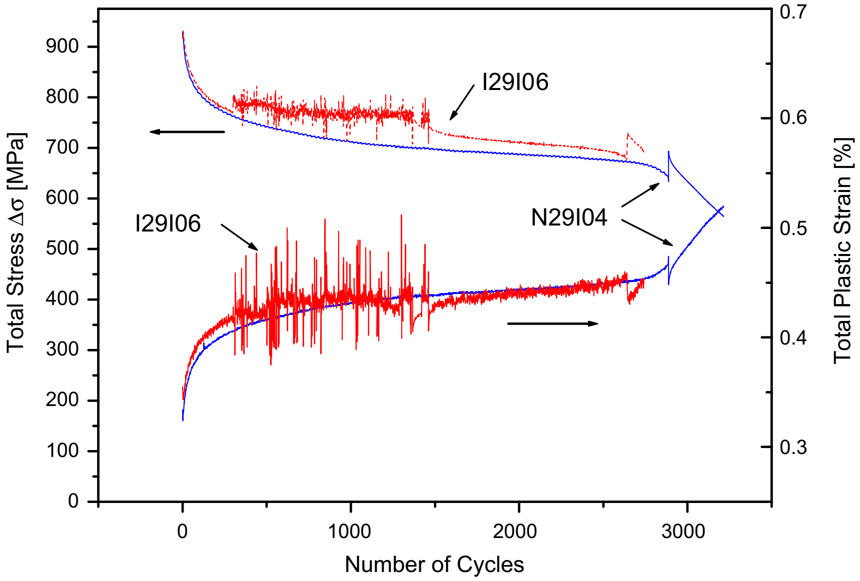

Fig. 4. Total stress and total plastic strain as a function of the cycle number for a pre-deformation of 300 cycles. $T_{irr} = 150$ °C.

rial tested without a beam. Radiation-induced hardening is clearly visible in both experiments, especially in experiment I29I06, where the beam has been stopped and the test continued at the same temperature.

During the first two experiments, sub experiments were carried out in order to study the irradiation hardening under static stresses. The beam was cut for 10–12 fatigue cycles in order to stabilize the structure. The specimen was then irradiated statically to 0.02 A s (0.0019 dpa). The results are shown for I29I03 in Fig. 5, for a static stress of 246 MPa and $T_{irr} = 250$ °C. The same type of experiment was carried out for I29I02, but at a static stress of 274 MPa and $T_{irr} = 150$ °C. Under positive stress, the change of the total stress was +33.4 MPa but −14.2 MPa under negative stress and +22.7 MPa at zero stress. Apparently, greater irradiation hardening was induced under positive stresses.

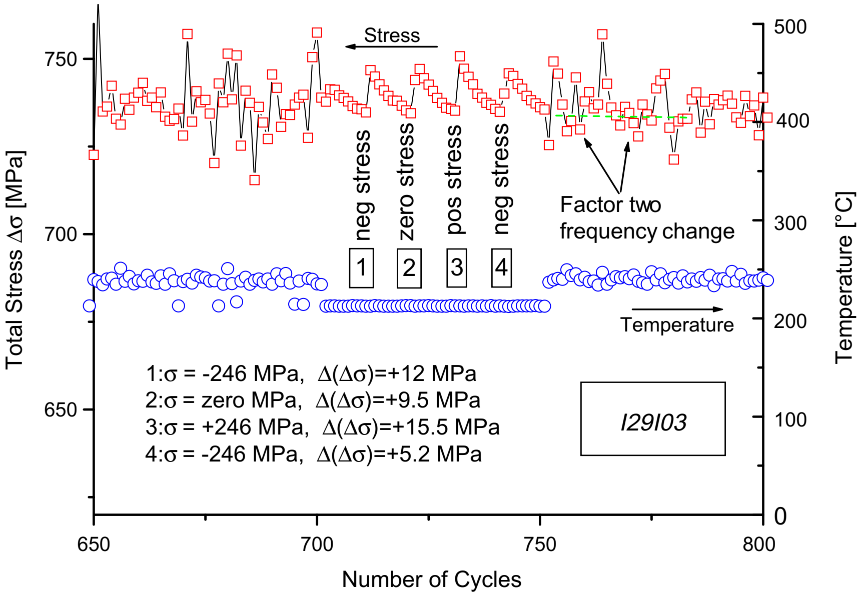

Fig. 5. Influence of static irradiation under positive, negative or zero stress on the total stress, and influence of a change of cycling frequency on the level of the total stress during in-beam fatigue.

In both experiments, the influence of the test frequency on the flow stress was measured, as shown in Fig. 5, for I29I03. As can be seen in the cycle plot, there was no effect on the response of the total stress through the transient. The same behavior was observed in I29I03. These results are consistent with previous results from stress relaxation during *in situ* fatigue experiments [8].

4. Conclusions

Fatigue tests have been carried out on Eurofer 97 at 150 and 250 °C under simultaneous irradiation with 590 MeV protons. At end of life, the dose reached was about 0.17 dpa and the He concentration 33 appm. The results indicate that:

- The cyclic life is strongly reduced under in-beam fatigue conditions.
- At $\dot{D}/\dot{\varepsilon} = 1.5 \times 10^{-2}$ dpa, radiation-induced hardening is clearly observed.
- The in-beam damage can be readily recovered by cyclic deformation.
- Irradiation-induced hardening is greater under positive static stresses.

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