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Evolution of the mechanical properties of the F82H ferritic/martensitic steel after 590 MeV proton irradiation

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Abstract

Tensile properties have been investigated on the low activation F82H ferritic/martensitic steels for both unirradiated and irradiated material. The irradiations have been carried out with protons of medium energy (590 MeV) at the PIREX facility and doses from 0.2 up to 1.75 dpa have been reached for temperature ranging between 310 and 673 K. The irradiation induced hardening has been systematically measured at room temperature for the different irradiation conditions (dose and irradiation temperature). The tensile tests have been supplemented with stress relaxation experiments in order to determine the activation volume of the rate controlling mechanism for dislocation motion. The effects of irradiation on the tensile properties and activation volumes are presented.

1. Introduction

Fusion reactor structural materials will be exposed to high energy neutron fluence. The accumulated displacement damage on the first wall of a commercial power fusion reactor is estimated to attain more than 100 dpa. Such a fluence is much higher than the fluence that will be attained in the experimental fusion devices, where austenitic steels are the candidate materials. For a commercial reactor, the use of austenitic steels is limited because of dimensional change caused by severe void swelling at higher doses [1]. Ferritic/martensitic steels are alternative structural materials to austenitic steels because of their good resistance to void swelling, high thermal conductivity, low thermal expansion and satisfactory mechanical properties; they are promising candidate structural materials for the blanket and first wall of commercial fusion reactors.

High recoil energies and high cross sections for nuclear transmutations characterize the interaction of energetic fusion neutrons with the nuclei of the materials surrounding the plasma. The transmutations lead in particular to hydrogen and helium production rates which can degrade the mechanical properties. Typical values of the rate productions in ferritic steels are 10 appm/dpa helium and 50 appm/dpa hydrogen. One way to investigate the fluence and helium effects on the mechanical properties in ferritic steels is, on the one hand, to irradiate specimens with protons of medium energy (several hundred MeV) which introduce helium by spallation reactions together with displacement damage produced by high energy recoiling ions after nuclear reactions. The Proton Irradiation Experiment (PIREX) at the Paul Scherrer Institute in Switzerland, which uses a proton beam of 590 MeV, has a measured typical rate of about 100 appm/dpa helium in ferritic/martensitic steels. On the other hand, another set of specimens can be irradiated with neutrons in fission reactors, where no substantial helium generation occurs, at doses and temperatures similar to those of protons irradiated in order to make a cross comparison and to separate the helium effect to those of fluence, if any. Irradiations are still in progress in PIREX and the neutron irradiations have been completed in the R2 reactor at Studsvik Material in Sweden where virgin and proton pre-irradiated specimens have been set up in the irradiation rig.

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In order to study the mechanical behavior after irradiation, tensile tests have been carried out between 300 and 673 K. These deformation tests have been supplemented with stress relaxation experiments. The stress relaxation technique has often been used to obtain information characterizing the microscopic mechanisms that control plastic deformation on a wide range of materials, e.g., [2–5]. When the plastic deformation is modeled as having an exponential stress dependence, i.e., in terms of a thermally activated process, an activation volume can be determined [6]. This parameter is of prime importance since it characterizes the stress dependence of the dislocation mobility and it has typical values and stress dependences for each atomic process. Thus, the determination of the activation volume before and after irradiation must provide useful information about the nature of the irradiation defects and about their interactions with the moving dislocations. In this paper, we report the preliminary results of the tensile tests and stress relaxation experiments performed on the specimens irradiated in PIREX as well as on unirradiated specimens.

2. Experimental procedure

The ferritic/martensitic steel F82H, an IEA standard material internationally investigated on irradiation behavior, has a composition of about 7.65 wt% Cr, 2 wt% W, and Mn, V, Ta, Ti, Si and C below 1 wt% in sum total, the balance being Fe [7].

The specimens have been irradiated with a 590 MeV proton beam in the PIREX facility [8] at PSI. The irradiations were carried out at four different temperatures (310, 523, 623 and 673 K), to a displacement range between 0.17 and 1.75 dpa. Due to the heat deposited by the protons, thin specimens have been used.

Tensile testing has been performed on DIN standard unirradiated specimens (3 mm diameter, 18 mm gauge length) as well as on irradiated thin PIREX specimens (5.5 mm gauge length, 0.3 mm thick). The mechanical tests have been conducted in an electromechanical computer controlled Schenck RMC100 machine at a strain rate of the order of 10^{-3}/min. The stresses and elongations reported below are the nominal stress ($\sigma = F/S_0$) and nominal elongations ($\Delta l/l_0$). The high temperature tests were done under vacuum ($<10^{-4}$ mbar).

The stress relaxation technique has been used to investigate the strain rate sensitivity of the stress. A single stress relaxation experiment consists in stopping the crosshead of the testing machine at a given level of the stress. The load applied on the specimen is allowed to relax and recorded as a function of time so that, for each time, the stress and stress rate (equivalent to plastic strain rate) are known. This yields a complete knowledge of the strain rate sensitivity of stress and an apparent activation volume is deduced [6,9,10]. The activation volumes (V_a) are expressed in terms of b^3, where b is the magnitude of the Burgers vector of a 1/2⟨1 1 1⟩ dislocation ($b = 2.68$ Å).

3. Results and discussion

The nominal 0.2% offset yield stress $\sigma_{0.2}$, ultimate tensile stress (UTS), uniform elongation (UE) and total elongation (TE), are plotted as a function of temperature for the unirradiated material in Figs. 1 and 2. $\sigma_{0.2}$ decreases from about 520 MPa at room temperature up to 450 MPa at 723 K. In this temperature range, $\sigma_{0.2}$ is distinctly smaller than UTS, indicating that plastic deformation is dominated by work-hardening. Uniform elongations of the order of 6% are obtained at room temperature, which decrease down to 2% to at 723 K. At room temperature, a comparison of the tensile properties of the DIN standard with the flat PIREX specimens has been done. The results show that a good agreement is found for $\sigma_{0.2}$, UTS and UE. On the contrary, a difference of about 4% in elongation has been found between the total elongation measured on DIN specimen and the one obtained from PIREX flat specimen.

The apparent activation volume V_a measured by stress relaxation experiments at the yield stress are also reported in Fig. 1. V_a presents an important increase between room temperature and 550 K. Like the other BCC structures, a strong lattice friction mechanism of Peierls type is expected at low temperatures. Thus, the usual decomposition of the flow stress into a thermal σ_{th} and an athermal components σ_{ath} [11] is well justified

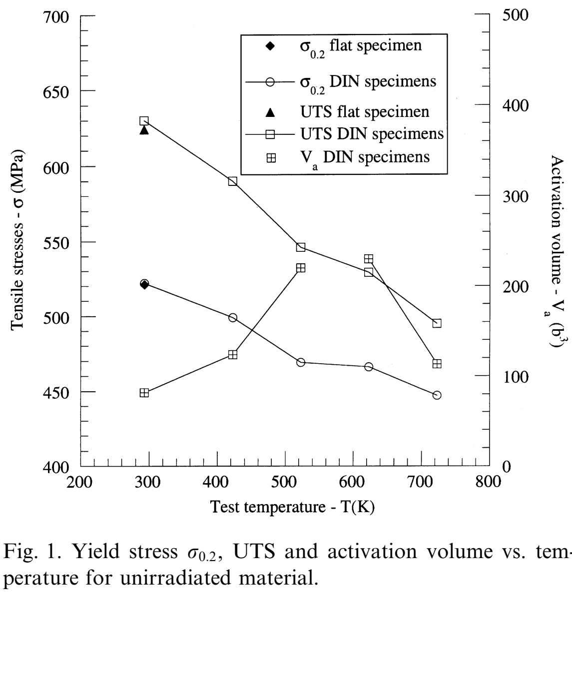

Fig. 1. Yield stress $\sigma_{0.2}$, UTS and activation volume vs. temperature for unirradiated material.

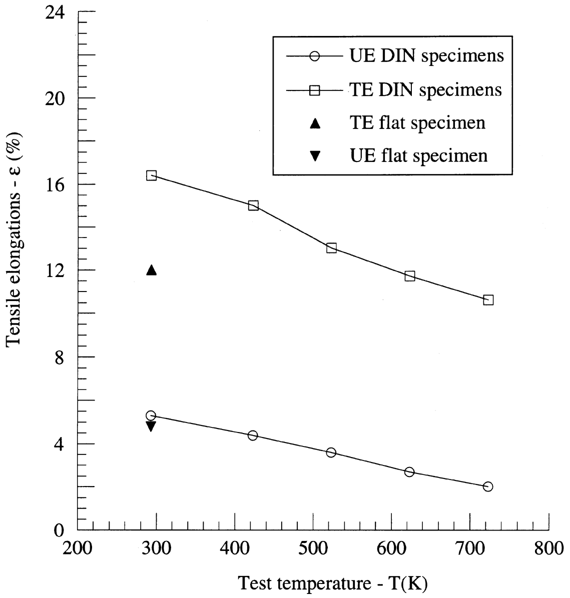

Fig. 2. Uniform and total elongation vs. temperature for unirradiated material.

$$\sigma = \sigma_{\mathrm{ath}}(\varepsilon_{\mathrm{p}}) + \sigma_{\mathrm{th}}(\dot{\varepsilon}_{\mathrm{p}}, T),$$

where σ_{ath} represents the internal stress due to the long range interaction with "large" obstacles and slowly varies with the dislocation position on its glide plane while σ_{th} characterizes the effective stress corresponding to short interactions with localized obstacles. At the athermal temperature of the rate controlling process ($\sigma_{\mathrm{th}} = 0$), the flow stress becomes independent of the plastic strain rate and the strain rate sensitivity of the flow stress $S = \mathrm{d}\sigma/\mathrm{d}\ \ln\ \dot{\varepsilon}_{\mathrm{p}}$ becomes negligible. Since the activation volume V_{a} is related to S by $V_{\mathrm{a}} = kT/S$ [9], V_{a} is expected to diverge at the athermal temperature and/or to present a singularity characterizing the change of the rate controlling mechanisms between the low and high temperature regimes. The observed activation volume behavior around 550 ± 50 K is then related to the beginning of the athermal plateau of the low temperature mechanism for which the corresponding athermal component of the stress is about 465 MPa. Finally, the experimental TEM observations showing long screw dislocations with a Burgers vector of the type ⟨1 1 1⟩ after deformation at room temperature [12] enhance the interpretation of a Peierls mechanisms governing the low temperature regime.

For the irradiated specimens, tensile tests have been performed at room temperature and $\sigma_{0.2}$ has been determined. Hereafter, the presentation and discussion of the results is confined to the irradiation hardening $\Delta\sigma$, i.e., the difference between the yield stress of the irradiated and unirradiated material measured at 293 K. $\Delta\sigma$ is the most representative parameter for characterizing the mechanical changes due to irradiation temperature and it has been used in many previous irradiation hardening studies (e.g. [13,14]). $\Delta\sigma$ will be also used to discuss the dose effect at a given irradiation temperature. Fig. 3 presents the irradiation temperature dependence of the irradiation hardening measured at room temperature; the respective doses for each point are indicated on the plot. $\Delta\sigma$ decreases monotonously with increasing temperature. The predominant irradiation hardening, which occurs only at temperature below 723 K, is characteristic of the 9–12% ferritic/martensitic steels and has already been reported for neutron irradiation; at about 723 K, the radiation induced hardening and softening are balanced [15]. Abe et al. [16] have already reported irradiation hardening on 9Cr–WVTa steels after neutron irradiation. They found, for instance at $T_{\mathrm{irr}} = 538$ K and 0.45 dpa, that $\Delta\sigma$ is about 80 and 110 MPa for a 9Cr–1WVTa and 9Cr–3WVTa steel composition, respectively. This is in rather good agreement with the $\Delta\sigma = 125$ MPa at $T_{\mathrm{irr}} = 523$ K and 0.47 dpa and 47 appm He for our 9Cr–2W steel proton irradiated. Another comparison of the irradiation hardening can also be done with the 12%Cr–steel MANET irradiated under the Dual Beam Facility of KFK [17] at 0.3 dpa and 500 appm He content. An extrapolation of these last data down to room temperature yields an irradiation hardening of about 200 MPa that must be compared to our measured strain hardening of 213 MPa at 0.26 dpa and 26 appm He. Both comparisons show that the He effect does not seem to be detectable by tensile tests up to 500 appm He content. The preliminary result at 673 K irradiation temperature (0.17 dpa) shows that the irradiation

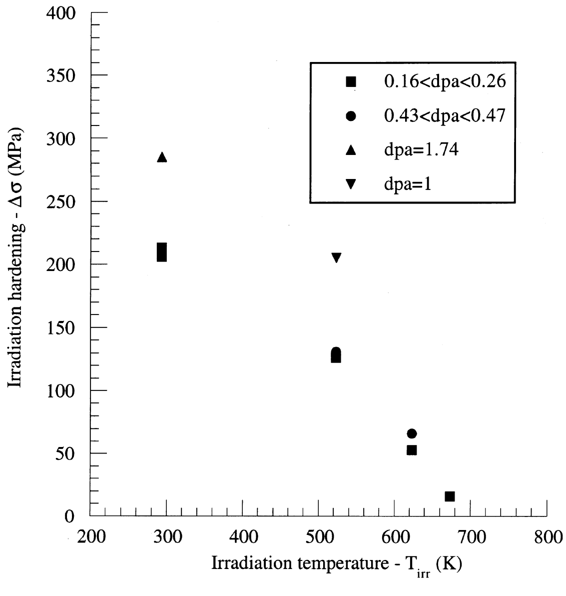

Fig. 3. Irradiation induced hardening measured at room temperature vs. irradiation temperature.

hardening by protons becomes very small at this latter temperature. This also indicates that the 17 appm He content, corresponding to 0.17 dpa of the tested specimen, is not high enough to stabilize the irradiation defects and does not play a significant role on the strength of this kind of steel. Irradiation at 673 K and to higher proton fluence (0.5 dpa) and neutron fluence up to (0.7 dpa) have been recently carried out. The residual radioactivity of these specimens is still too high at present for mechanical testing and another cooling period of six months is needed before the irradiated specimens can be tested. The expected weak or absent effects of He for concentration of the order of 100 appm He on the irradiation hardening at temperatures below 673 K has still to be confirmed by tensile testing of the latter specimens.

Fig. 4 shows $\Delta\sigma$ as a function of the proton fluence in dpa for the different irradiation temperatures. In the range of fluences available at present for testing, $\Delta\sigma$ exhibits a linear dependence when plotted against the 1/4 power of the fluence. Such a dependence has already been observed for neutron fluence on iron [13], pressure vessel ferritic steel [14] and 316 austenitic steel [18] at doses lower than 0.1 dpa and for temperature below 573 K. Our results indicate that the fourth root of the fluence provides a good description for the sets of data at the different irradiation temperatures and seems to describe properly the irradiation hardening up to 1.75 dpa. It must be noted that substantial decrease of the slope of the fit takes place with increasing irradiation temperature. The 1/4 power dependence of the increase in the yield strength was predicted by calculations that include the size and number density of defect clusters [19], both of them increase as the square root of the neutron fluence. Let us emphasize that the defect clusters or helium bubbles produced by irradiation could not be directly observed by TEM, indicating that, if present, they are smaller than the resolution of conventional TEM observations, that is to say below 1 nm.

The apparent activation volumes measured at room temperature on all the irradiated specimens are presented in Fig. 5 as a function of the yield stress. As expected, the activation volume decreases with increasing stress [20]. It must be noted that no clear influence of the irradiation conditions (doses and irradiation temperatures) has been found on the behavior of the activation volume as a function of stress. The activation volume measured for the highest dose available (1.75 dpa, 175 appm He) is comparable to the one measured in the unirradiated providing an indication that the irradiation hardening and even this high amount of He does not influence the rate controlling process. However, Abe et al. [21] have shown that, for tests performed at low temperatures of neutron irradiated low activation 9Cr–1V ferritic steel, the irradiation hardening is the sum of two components; one being athermal $\Delta\sigma_{ath}$ while the other is due to thermally activated obstacles $\Delta\sigma_{th}$. On the one hand, small point defects or point defect-solute atom pairs or complexes are supposed to give rise to $\Delta\sigma_{th}$, while, on the other hand, invisible small dislocation loops could cause the measured athermal component of the strain-hardening. In other words, Abe et al. have evidenced a change in the temperature dependence of the yield stress eventually reflecting changes in the rate controlling mecha-

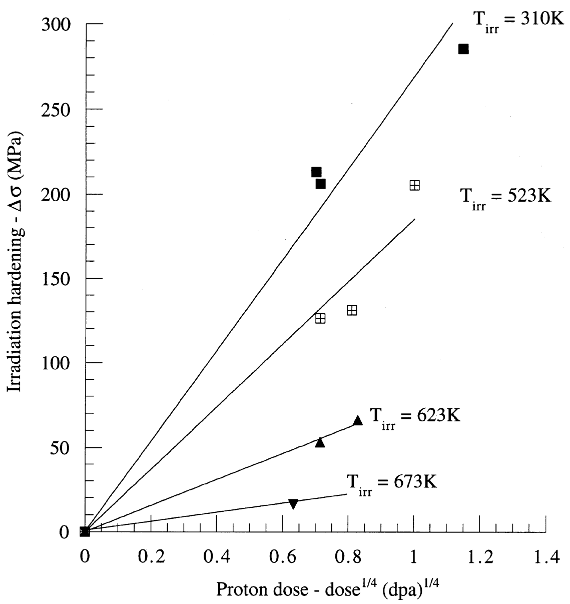

Fig. 4. Dose and temperature dependence of irradiation induced hardening.

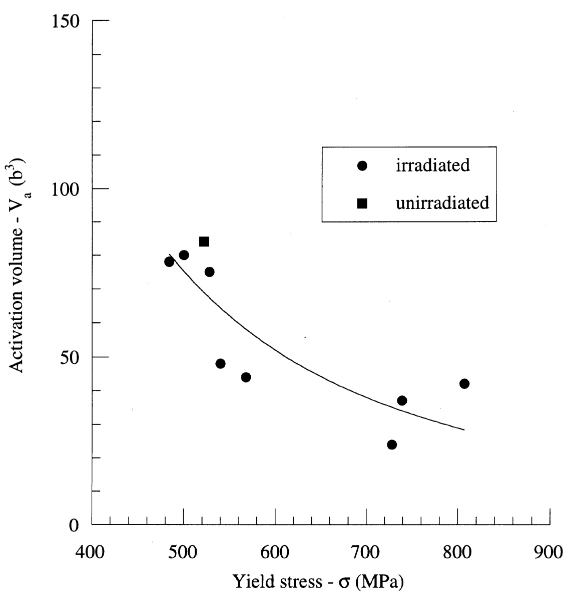

Fig. 5. Activation volume measured at $\sigma_{0.2}$.

nisms. At this point, our data are too scarce to clearly confirm that the irradiation induced defects do not change the deformation rate controlling process and must be completed for both the unirradiated and irradiated material over a larger range of temperatures including temperatures below room temperature. It is also worth mentioning that the strain-hardening taking place during the relaxation yields only to an apparent activation which should be corrected by a strain hardening coefficient in order to obtain the effective activation volume [4,9]. A quite self-consistent analysis would require to plot the effective activation volume as a function of the effective stress acting on the mobile dislocations. This necessitates to determine, after each irradiation conditions, the actual internal stress that can be modified by the dose and irradiation conditions. Basically, we should have a set of measurement of $\sigma_{0.2}$ and activation volume on a large range of temperatures for a given dose.

4. Conclusions

Tensile properties of the F82H ferritic/martensitic steel have been determined on both unirradiated and proton irradiated specimens. Four irradiation temperatures have been investigated, namely 310, 523, 623 and 673 K with proton fluences ranging between 0.3 and 1.75 dpa.

1. For measurements at room temperature, the induced radiation hardening is shown to decrease rapidly with increasing irradiation temperature and practically disappears at $T_{irr} = 673$ K
2. For low He contents, no effects of He has been clearly evidenced on the flow stress. Furthermore, the small activation volume found in specimens irradiated to higher fluences (and therefore higher He content) seems to rule out any changes in the rate controlling dislocation motion due to He bubbles that might be eventually present in the irradiated microstructure.

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