# Mechanical properties and microstructures in F82H steel irradiated under alternating temperature 

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#### Abstract

Reduced-activation martensitic steel F 82 H was irradiated at alternating temperatures between 230 and $350^{\circ} \mathrm{C}$ to the accumulated damage level of 1.5 dpa using an irradiation capsule with temperature control independent of reactor power. Tensile tests were conducted in order to investigate the effects of the irradiation temperature variations on mechanical properties of F 82 H . Electron microscope observations were performed for the irradiated F 82 H to evaluate microstructural evolution of the specimens following varying temperature irradiation. Yield stress of the F82H irradiated at $50 \%$ alternating temperature between 230 and $350^{\circ} \mathrm{C}$ was relatively large compared with the other temperature variations in this study. Size and number densities of dislocation loops were observed to be affected by changing irradiation temperature. The distinctive hardening behavior could be interpreted by the difference in the size and density of the defect clusters in terms of the effect of varying temperature.


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## 1. Introduction

Components of fusion reactors will undergo temperature variations during operation cycles due to non-steady state plasma conditions. It has been pointed out that temperature variations during neutron irradiation may cause significant changes in microstructures due to changes in nucleation and growth of the point defect clusters that depend on irradiation temperature [1]. Larger numbers of defect clusters are introduced during neutron irradiation at lower temperatures. At higher temperatures, the growth rate of the defect clusters competes with the recombination rate of interstitial

[^0]and vacancy depending on the temperature and damage rate. The microstructural evolution may affect irradiation hardening, and result in enhanced reduction of the ductility in martensitic steels. Several reports show the effect of temperature change during irradiation on the microstructural evolution and/or the hardening behavior in materials using neutron, ion and electron irradiations [2-13]. The effect of temperature change, however, is not yet clearly understood. In order to evaluate the effects of temperature variations on microstructural changes, irradiation experiments under accurate changing temperature conditions are necessary. In this work, a reduced-activation martensitic steel F82H was irradiated under alternating temperatures using an irradiation capsule with temperature control independent from the reactor power. The effect of the temperature change on the yield stress of

F82H steel has been examined after the irradiation, and TEM observations of the microstructure were performed on the irradiated specimens. Mechanisms by which temperature variations influence the formation and growth of dislocation loops are discussed.

## 2. Experimental procedures

Specimens used in this study were F 82 H ( $\mathrm{Fe}-$ $0.1 \mathrm{C}-7.8 \mathrm{Cr}-2 \mathrm{~W}-0.2 \mathrm{~V}-0.04 \mathrm{Ta}$ ), a martensitic steel, and the austenitic steels JPCA (Fe-0.05C-14Cr$16 \mathrm{Ni}-0.25 \mathrm{Ti}-2 \mathrm{Mo}$ ) and 316 F ( $\mathrm{Fe}-0.02 \mathrm{C}-17 \mathrm{Cr}-$ $14 \mathrm{Ni}-2 \mathrm{Mo}$ ). Details about the austenitic steels were reported elsewhere [14]. The F 82 H was normalized at $1040{ }^{\circ} \mathrm{C}$ for 1.8 ks and tempered at $750^{\circ} \mathrm{C}$ for 3.6 ks. Tensile specimens and TEM disk specimens were obtained from the plate. Irradiation capsules with temperature control capability independent of the reactor power have been developed at the Japan Materials Testing Reactor (JMTR). Specimen temperature was controlled by the heat conductance of the gas gap and an electric heater located around the housings of the specimens. The capsule used was divided into six regions (with six housings). Tensile and TEM disk specimens were loaded in each housing. The temperature of each region was controlled independently. Nominal highest and lowest temperatures during irradiation were alternated between 230 and $350^{\circ} \mathrm{C}$. Temperature was controlled with six patterns, as shown in Fig. 1. Temperature is con-
stant at $230{ }^{\circ} \mathrm{C}$ through 10 cycles (1). Temperature is $50 \%$ alternated between 230 and $350{ }^{\circ} \mathrm{C}$ at the middle period of 1 cycle and repeated for 10 cycles (2). Temperature is $230^{\circ} \mathrm{C}$ for $5 \%$ and $350^{\circ} \mathrm{C}$ for $95 \%$ of 1 st cycle and constant at $350^{\circ} \mathrm{C}$ for the remaining cycles (3). Temperature is constant at $350{ }^{\circ} \mathrm{C}$ through 10 cycles (4). Temperature is $230^{\circ} \mathrm{C}$ for $50 \%$ and $350{ }^{\circ} \mathrm{C}$ for $50 \%$ of 1 st cycle and constant to be $350{ }^{\circ} \mathrm{C}$ for remaining cycles (5). Temperature is $5 \%$ alternated between 230 and $350{ }^{\circ} \mathrm{C}$ per 1 cycle and repeated for 10 cycles (6).

Irradiation was performed to a total displacement damage level about 1.5 dpa (neutron fluence of $1.48 \times 10^{25} \mathrm{n} / \mathrm{m}^{2}, E>1 \mathrm{MeV}$ ) over 10 reactor cycles. Temperature was increased to the irradiation temperature about 20 min before increasing the reactor power at each reactor cycle. The temperature of the housing was changed during operation. After reducing the reactor power to zero, the temperature of the capsule was immediately reduced and kept at about the water temperature of the reactor, which was about $30^{\circ} \mathrm{C}$. The longitudinal neutron fluence variation was below $30 \%$ along the capsule.

After irradiation, tensile tests were performed at ambient temperature in air, with a loading rate of $8.3 \times 10^{-7} \mathrm{~m} / \mathrm{s}$. Size of sheet tensile specimens was 25.4 mm in length and 0.76 mm in thickness and the gage length and width are 7.62 and 1.52 mm , respectively. The results of tensile tests were corrected by taking into account of the longitudinal


Fig. 1. Alternating temperature patterns under irradiation. Details of changing temperature are shown in the text.
neutron fluence variation along the capsule and calculated using relations between yield stress and damage level proposed by Shiba et al. [15]. The TEM disks were electro-polished at a voltage of 40 V and nominal current of about $70-80 \mathrm{~mA}$ using an electrolyte of $5 \%$ perchloric and $95 \%$ acetic acid at $15^{\circ} \mathrm{C}$. The microstructures of the specimens were examined by a transmission electron microscope (TEM) operated at 200 kV .

## 3. Results and discussion

### 3.1. Tensile-test results of F 82 H steels irradiated at alternating temperatures

Tensile properties of F 82 H steels irradiated with the six temperature patterns showed various radiation hardening behavior. The YS of the specimens irradiated with the six temperature patterns were 862 (1), 925 (2), 800 (3), 811 (4), 845 (5) and 797 MPa (6), respectively. Each yield Stress (YS) value is the average value of two tensile tests. The change of YS after irradiation with the six temperature patterns are shown in Fig. 2. Each number corresponds to an alternating temperature pattern as shown in Fig. 1. The change of YS of the specimen irradiated at a constant $230^{\circ} \mathrm{C}(1)$ is about 270 MPa and that of specimen irradiated at a constant $350^{\circ} \mathrm{C}$ (4) is about 220 MPa . The YS change for alternating temperature irradiation using pattern (2) between 230 and $350^{\circ} \mathrm{C}$, is about 330 MPa showing excess hardening on the YS of over 100 MPa . The excess hardening observed for the F 82 H was considerably different from that found in austenitic steels (JPCA), where much less excess hardening was found [14]. Patterns for (3) and (6) showed similar hardening


Fig. 2. Change of yield stress for six irradiation temperature patterns. Each number corresponds to the alternating temperature pattern as shown in Fig. 1.
of about 200 MPa , while (5) induced a hardening of about 250 MPa . The dependence of hardening behavior on temperature pattern variation could be qualitatively explained by the microstructural evaluations described below.

### 3.2. Microstructural evaluation

Typical high magnification TEM images of specimens irradiated at a constant $230^{\circ} \mathrm{C}$, alternating between 230 and $350^{\circ} \mathrm{C}$ and a constant $350^{\circ} \mathrm{C}$ are shown in Fig. 3(1-3), respectively. Interstitial-type dislocation loops are observed for all the specimens. Mean size of the loops is 16,11 and 41 nm for (1), (2) and (3), respectively. The size of dislocation loops increased with increasing irradiation temperature from 230 to $350^{\circ} \mathrm{C}$ under constant temperature irradiation. The size of loops did not change significantly comparing cases (1) and (2), while the number density of loops increased drastically under alternating temperature irradiation between 230 and $350^{\circ} \mathrm{C}$. The size and number densities of dislocation loops were significantly changed depending on the pattern of temperature variation.

The size distribution of dislocation loops estimated from TEM images are shown in Fig. 4. The mean size of the loops is $16,11,41,30$ and 27 nm for the irradiation temperature patterns (1), (2), (4), (5) and (6), respectively. The number density of the loops is (1) $3.9 \times 10^{20}$, (2) $1.1 \times 10^{21}$, (4) $1.9 \times 10^{20}$, (5) $2.7 \times 10^{20}$ and (6) $1.8 \times 10^{20} \mathrm{~m}^{-3}$. Comparing the cases of constant irradiation temperature 230 (1) and $350^{\circ} \mathrm{C}$ (4), loop size increased


Fig. 3. Typical TEM images of three temperature patterns (1) Temperature is constant at $230^{\circ} \mathrm{C}$ through 10 cycles. (2) Temperature is $50 \%$ alternated between 230 and $350^{\circ} \mathrm{C}$ per cycle and repeated 10 times. (3) Temperature is constant at $350^{\circ} \mathrm{C}$ through 10 cycles.


Fig. 4. Size distribution of dislocation loops. Each number in bracket corresponds to the alternating temperature pattern as shown in Fig. 1.
more than twice as shown in Fig. 3 and the number density decreased by half. Therefore at the higher temperature of $350^{\circ} \mathrm{C}$, growth of the defect clusters was greater and the number density of loops was smaller. In comparison with the case of constant temperature $230^{\circ} \mathrm{C}$ (1), an alternating temperature between 230 and $350^{\circ} \mathrm{C}$ (2) resulted in a slight size decrease, but the number density increased by about three times.

Correlation between a change of YS and Nd (number density of loops multiplied by the size) as a function of time spent at the irradiation tempera-


Fig. 5. Duty time dependence on change of YS and defect accumulation rate of loops Each number in bracket corresponds to the alternating temperature pattern as shown in Fig. 1.
ture of $230^{\circ} \mathrm{C}$ is shown in Fig. 5. The change in YS initially increased with increasing fraction at $230^{\circ} \mathrm{C}$, has a maximum at $50 \%$, and decreases at higher fractions. The Nd data change similarly. The results suggest that concentration of dislocation loops depends on time spent undergoing lower temperature irradiation, so that larger numbers of defect clusters are introduced during neutron irradiation, and the radiation hardening changes accordingly.

## 4. Conclusion

Comparison between the change of yield stress and microstructure revealed a temperature-dependent irradiation hardening for F 82 H steel. Excess hardening was observed in F 82 H steel irradiated at alternating temperature between 230 and $350^{\circ} \mathrm{C}$. Microstructural evolution depends on the fraction of time at low temperature. It was evident that the distinctive hardening behavior could be interpreted on the basis of an effect of the alternating temperature on the size and density of the defect clusters.

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