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journal of nuclear materials

Journal of Nuclear Materials 367-370 (2007) 539-543

www.elsevier.com/locate/jnucmat

# Effect of temperature change on the irradiation hardening of the structural alloys for ITER blanket and ITER TBM irradiated to 1.5 dpa in JMTR

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#### Abstract

A reduced activation ferritic steel and an austenitic stainless steel were irradiated at temperatures of 230 °C and 350 °C in an irradiation capsule with temperature control capability independent from reactor power to accumulated damage levels of about 1.5 displacement per atom (dpa). For some of the specimens temperature was changed during irradiation. The temperature change reduced the irradiation hardening of the austenitic steel. Conversely, it slightly increased the hard-ening of the reduced activation ferritic steel. The mechanism of the observed temperature change effect and the impact of the additional hardening on the residual ductility is discussed.

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#### 1. Introduction

Temperature of the in vessel components of fusion reactors strongly depends on the heat flux from the plasma and will change during operating cycles. A larger number of point defect clusters is introduced during neutron irradiation at lower temperatures. At higher temperatures, the growth rate of the clusters is increased and results in a higher dislocation density, which enhances irradiation induced hardening. At even higher temperatures, the clusters decompose and disappear, thus reducing the irradiation hardening. Hardening tends to affect the fracture mode of components. For instance, hardening reduces the margin to ductile fracture for both austenitic and ferritic alloys. It also increases the ductile to brittle transition temperature and the probability of unstable fracture for ferritic alloys [1].

The water temperature of the ITER shield blanket is designed to be about 150 °C, while the plasma facing surface temperature is thought to go beyond 450 °C during operation. ITER test blanket, which is planned to be built with reduced activation ferritic steel, is also thought to experience temperature changes during operation [2]. A number of studies have been conducted to understand the effect of temperature change on the behavior of the materials of the components [3–14]. However, the obtained results might not be easy to understand. For instance, it was pointed out that in austenitic alloys

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<sup>0022-3115/\$ -</sup> see front matter @ 2007 Published by Elsevier B.V. doi:10.1016/j.jnucmat.2007.03.202

the temperature change from 300 °C to 400 °C during irradiation caused the shrinkage of interstitial type clusters and growth of cavities. This was estimated to occur by the dissolution of vacancy type clusters, thus causing a transient high vacancy flux just after the temperature change [3,4]. The shrinkage of interstitial type clusters due to increasing temperature was also reported for reduced activation ferritic steels and vanadium alloys [7-14]. A different model was, however, employed because of the low stability of vacancy clusters in the body centered cubic alloys [15]. The model is based upon the disappearance of the highly mobile small interstitial clusters [12]. In addition, no obvious effect was reported for similar alloys for a higher damage level of 4 dpa [6,8]. The effect of temperature change was not reported to be systematic, as described above. Therefore, a temperature change irradiation experiment was conducted at the position in Japanese Materials Testing Reactor (JMTR) with the smaller neutron flux to improve the ability of the temperature control during irradiation.

#### 2. Experimental procedure

# 2.1. Materials, specimens and post irradiation examinations

An austenitic stainless steel of Fe–0.02C-17Cr-14Ni-2Mo (316F) and a reduced activation ferritic steel of Fe–0.1C-7.8Cr-2W-0.2V-0.04Ta (F82H) are used. 316F was solution annealed for 1 h at 1050 °C. F82H was normalized at 1040 °C for 0.5 h and tempered at 750 °C for 1 h. Details of the alloys are reported elsewhere [16,17].

Tensile specimens 25.4 mm long, 4.7 mm wide and 0.76 mm thick with a 7.5 mm × 1.5 mm gage section were prepared from the plates of the alloys. Also, transmission electron microscopy disk specimens (TEM disks) were obtained from the plates. Specimens were inserted in the capsule and irradiated. After irradiation, tensile tests were performed at ambient temperature with nominal strain rate of about  $1 \times 10^{-3}$ /s.

### 2.2. Neutron irradiation

Irradiation capsules with temperature control capability independent from the reactor power have been developed at JMTR. Specimen temperature was controlled by the heat conductance at the gas gap and the electric heater located around the hous-



Fig. 1. Position of the TEM and tensile specimens in the capsule.

ings of the specimens. Tensile and TEM disk specimens were loaded in each housing, as illustrated in Fig. 1. The capsule used has been divided into six levels with six housings. The temperature of each level was also controlled independently. Nominal highest and lowest temperatures during irradiation



Fig. 2. Temperature change patterns during irradiation.

were selected to be 230  $^{\circ}$ C and 350  $^{\circ}$ C, respectively. Temperature was controlled in six patterns (see Fig. 2).

Irradiation was performed for 10 reactor cycles. Temperature was raised to irradiation temperature about 20 min before raising the reactor power at each reactor cycle. The temperature of the housing was changed during operation. After reducing the reactor power to zero, temperature of the capsule was immediately reduced and kept at about the water temperature of the reactor. Neutron fluence values with a peak at  $1.48 \times 10^{25}$  N/m<sup>2</sup> varied within 30% along the capsule. Displacement damage level of the specimens ranged from 1.2 dpa to 1.5 dpa.

## 3. Experimental results

### 3.1. Austenitic stainless steels (316F)

Engineering stress–strain responses for solution annealed (SA) 316F are shown in Fig. 3. Irradiation at 230 °C and 350 °C caused hardening of 400 MPa or larger, while the hardening of the specimens irradiated at both 230 °C and 350 °C alternately (pattern 2 in Fig. 2) remains at a fairly smaller value of 200 MPa.

#### 3.2. Reduced activation ferritic steel (F82H)

F82H hardened by irradiation at 230  $^{\circ}$ C and 350  $^{\circ}$ C by 250 MPa and 150 MPa, respectively. Moreover, the largest hardening of 300 MPa or even higher was exhibited for the irradiation at tem-



Fig. 3. Engineering stress–strain curves for 316F before and after irradiation.



Fig. 4. Engineering stress-strain curves for F82H before and after irradiation.

peratures of 230 °C and 350 °C alternately (pattern 2 in Fig. 2). Engineering stress–strain responses for F82H specimens are seen in Fig. 4.

### 4. Discussion

# 4.1. Effect of temperature change on irradiation hardening

As summarized in Fig. 5, the temperature change (pattern 2) affected the irradiation hardening behavior of both 316F and F82H. Although the effect on F82H was smaller, it seems that additional hardening was introduced when compared to those irradiated at 230 °C and 350 °C; deviation of yield stress at temperatures between 250 °C and 350 °C was smaller than 70 MPa even after irradiation to 5 dpa, as seen the reports [2,18,19]. Damage level depended on the position in the capsule. Hardening values were corrected by normalizing to a damage level of 1.5 dpa by using the 'vield stress-damage level' relations compiled by Shiba and Rensman for the results of reduced activation ferritic steels [2,20]. Irradiation hardening mostly depends on the size and the number density of the radiation produced interstitial (I) clusters at temperatures below 400 °C for ferritic and austenitic alloys [21]. Microstructural examination of TEM specimens of F82H has also been conducted. Results suggest that I-loops mainly caused the hardening of F82H [22]. Temperature change introduced larger number of



Fig. 5. Effect of temperature change on the yield stress levels.

I-loops with larger average diameter, but almost no vacancy clusters was observed.

More than 10 papers have been published on the subject of the effect of temperature change on microstructure and hardening [3–14]. In the austenitic stainless steels, a number of tiny V-clusters (e.g. di-vacancies) would be formed during irradiation at lower temperatures. By raising the temperature, the tiny V-clusters would decompose to cause a high transient V flux that caused annihilation of I-clusters. This model has been proposed by Yoshida et al. [3]. This qualitatively agrees with the present results obtained for austenitic stainless steels. However, the diffusion of the accumulated vacancies during the lower temperature irradiation was suggested

to play a dominant role for the disappearance of Iclusters at the higher temperature. As seen in the figure, 316F hardened even at a higher temperature of 350 °C. This indicates that the diffusivity of vacancies was limited at 350 °C, suggesting they might not be effective in annihilating I-clusters. This does not fully agree with the conditions of the model.

For the bcc alloys of the reduced activation ferritic steel and the vanadium alloy, shrinkage of I-clusters during higher temperature irradiation was also reported by Kasada and Kimura [12] and Ochiai et al. [11]. However, those results do not agree with the present results.

One of the authors has conducted an in situ electron irradiation experiments in a 400 kV electron microscope to understand the effect of temperature and flux change on the irradiation produced microstructure in aluminum [23]. The results indicated that the smaller dislocation bias for I of the smaller I-clusters caused their shrinkage during irradiation at higher temperatures, while larger I-loops with higher dislocation bias continue to grow. This also suggests that the smaller dislocation bias in bcc alloys may not lead to the shrinkage of I-clusters during irradiation at higher temperatures. This may result in additional hardening. These are consistent with the present results, although additional experiments will be needed for verification.

# 4.2. Additional hardening and ductile unstable fracture

The temperature change introduced additional hardening for the reduced activation ferritic steels, although the additional level is rather small. Comparing with the results at a constant temperature of 230 °C and those at 350 °C, the hardening increased by 60–80 MPa and 120–150 MPa, respectively. Comparing with the damage level dependence of the irradiation hardening reported by Shiba and Rensman, this increment corresponds to 2–5 times, and 4–13 times higher damage levels at 230 °C and 350 °C, respectively [19,20]. For the ITER test blanket, irradiation experiments to a higher damage level of 3 dpa seem to be required.

As seen in Fig. 4, reduced activation ferritic steel tends to exhibit diffuse necking just after yielding. The alloys, however, maintain marginal ductility until the ductile fracture, corresponding to the localized necking strain. This is schematically shown in Fig. 6.



Fig. 6. Effect of irradiation hardening on the residual ductility to ductile fracture.

Irradiation hardening tends to saturate with dose [17]. Also, it is expected that the saturation level remains lower than the stress level of the localized necking (ductile fracture). This difference acts as a safety margin to fracture. In case the temperature change results in an increase of this saturation level, it should be examined because of the reduction of the safety margin to ductile fracture.

#### 5. Conclusions

- (1) The temperature change decreased irradiation hardening for solution annealed 316F austenitic stainless steel.
- (2) Diffusion of vacancies at 350 °C is limited. Therefore, the annihilation of defects necessary to reduce irradiation hardening might not be caused by annealing, but rather occurred during irradiation at higher temperatures. The smaller dislocation bias of the smaller I-clusters might cause their shrinkage during irradiation.
- (3) The temperature change caused an increase of the irradiation hardening for F82H reduced activation ferritic steel. The amount of additional hardening corresponds to several times higher dose levels.

(4) If the temperature change causes an increase of the saturation level of the yield stress, it will decrease the safety margin to fracture.

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