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Annealing effects on mechanical properties and microstructure of F82H irradiated at ⩽60 °C with 800 MeV protons

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Abstract

Martensitic steel F82H was irradiated with 800 MeV protons up to 7.8 dpa at low temperatures ⩽60 °C. Tensile tests have been performed at 22, 160, 250, 350 and 400 °C. The microstructure of specimens with doses of 6–7.8 dpa after annealing 20 min and 2 h at 160, 250, 350 and 400 °C has been studied. The tensile results show that the yield of irradiated specimens decreases gradually with increasing test temperature. The ductility starts to recover at about 300 °C and is substantially recovered at 400 °C. The recovery depends on irradiation dose. The TEM results indicate that the number density of dislocation loops induced by irradiation decreases with annealing temperature, while the mean size of loops increases only slightly at 400 °C. A part of dislocation loops transferred into network dislocations and resulted in increase of network dislocation density. However, the total dislocation density (including loops) decreases gradually with annealing temperature.

1. Introduction

The irradiation induced changes in microstructure and mechanical properties of materials irradiated at low temperatures can be partly or even completely recovered when they are annealed at higher temperatures. Although a lot of annealing experiments have been done on materials irradiated with neutrons in fission reactors, similar investigations on materials irradiated with high-energy protons and neutrons in spallation targets are still very limited, especially for studies on microstructure.

In the case of the irradiation in SINQ targets (STIP), the irradiation temperature and dose depend strongly on the position in the target due to relatively small size of the proton beam [1]. The number of specimens with exactly same irradiation condition is very limited. The strong position dependence of irradiation condition makes the comparison between specimens and materials more difficult. Considering the operation temperatures at the beam windows of liquid metal target containers in different spallation sources are mostly close to 200 and 250 °C, a common test temperature of 250 °C was selected by the STIP partners [2–4]. However, for the sake of developing the liquid lead–bismuth target for the megawatt pilot target experiment (MEGAPIE) [5],

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it is also interesting to investigate the behaviour of irradiated materials at around 350 °C which is higher than the irradiation temperature of most STIP-I specimens. It is of essential interest to study annealing effects on the microstructure and mechanical properties of materials irradiated at low temperatures. The F82H specimens irradiated in the acceleration production of tritium (APT) program [6] are ideal for the purpose since they were irradiated at 60 °C and below. In the present work, a series of tensile tests were performed at 22, 250, 350 and 400 °C for investigating the changes in tensile properties, and meanwhile, transmission electron microscopy (TEM) observations were performed for studying the microstructural changes after corresponding tensile tests and additional annealing.

2. Experimental

Material of F82H was obtained from the fusion materials program [7]. The specimens were EDM cut from a 15 mm thick plate (IEA Heat 9741). The nominal composition is: Fe+ 7.7Cr, 0.16Mn, 0.16V, 1.95W, 0.02Ta, 0.11Si and 0.09C in wt%. The plate was normalized at 1040 °C for 38 min and tempered at 750 °C for 1 h.

Miniature type tensile specimens of 0.25 mm thick, 5 mm long and 1.2 mm wide in gauge area were irradiated with other specimens of the APT materials irradiation program at the Los Alamos Neutron Science Center (LANSCE) [5] to doses up to 7.8 dpa at 60 °C and below. More detailed information about the irradiation of these tensile specimens can be found in Ref. [6].

Tensile tests were performed on a 2 kN MTS mechanical test machine equipped with a video-extensometer so that the displacement was measured directly from the gauge area. The tests were performed at 22, 160, 250, 350 and 400 °C with a strain rate of about $10^{-3}\ s^{-1}$. Each tensile test was run until the specimen failed. Tests at temperatures above room temperature (22 °C) were performed 15–20 min after the temperature stabilized. At each temperature, three specimens of doses about 0.8, 1.7 and 6–7.8 dpa were tested.

Four discs of 1.0 mm diameter were punched from the grip sections of each tensile specimen for transmission electron microscopy (TEM) observation. To investigate annealing time effects, two of the four discs were again heated up to the tensile test temperature and aged for 2 h. The TEM investigation on the microstructure was performed with a JEOL 2010 type microscope equipped with an EDX analysis system. The most often used image conditions were bright field (BF) and weak beam dark field (WBDF) at $(\mathbf{g}, 4\mathbf{g})$ or $(\mathbf{g}, 6\mathbf{g})$, $\mathbf{g} = \mathbf{110}$. For all specimens, only the micrographs of $\mathbf{g}(5\mathbf{g})$ $\mathbf{g} = \mathbf{110}$, $\mathbf{z} = \mathbf{111}$ were used for quantifying the size and number density of defect clusters. The thickness of a thin foil was deduced from the number of fringes, which had an uncertainty of about ±15%.

3. Results

3.1. Tensile tests

Fig. 1 presents the engineering tensile stress–strain curves of the specimens tested at 22, 160, 250, 350 and 400 °C. It can be seen that the yield stress shows a general trend of gradually decreasing with increasing test temperature. As illustrated in Fig. 2, the yield stress shows a linear dependence of test temperature except for that of the specimen of 1.8 dpa and tested at 400 °C. At 250 °C and below, the tensile curves of the specimens with similar doses resemble each other. The

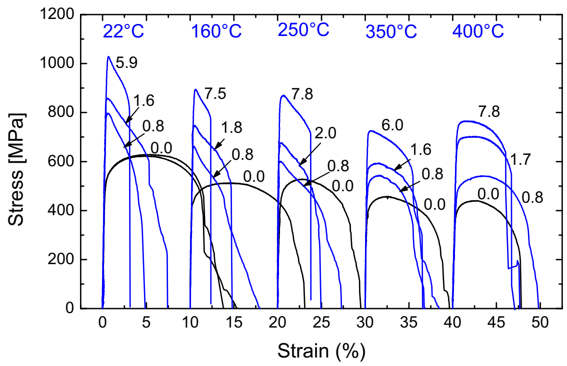

Fig. 1. The engineering tensile stress–strain curves of the irradiated and unirradiated F82H specimens tested at 22, 160, 250, 350 and 400 °C. The shift in x-axis between curves at different temperatures is 10%.

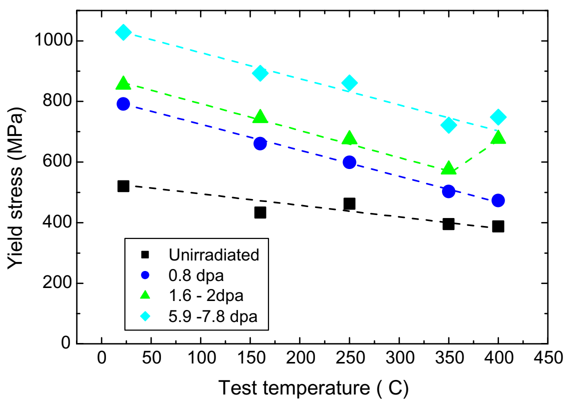

Fig. 2. The test temperature dependence of the yield stress of the irradiated and unirradiated F82H specimens.