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Microstructure change and helium release due to tensile loading on austenitic stainless steel implanted with low energy helium ions

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Abstract

Microstructure change and helium release due to tensile loading on 304SS specimens implanted with low energy helium ions have been investigated. Specimens are irradiated with 8 keV helium ions at RT, 573 K and 873 K up to the fluence of 3×10^{21} He/m^2. Tensile tests are performed at RT on unirradiated and irradiated specimens at a strain rate of 3.33×10^{-3}/s. In every irradiation condition, specimens were tested to failure, and were elastically strained, yield point and plastically strained to 10%, 20% and 40% (total strain). After the tensile test, surface modification and microstructure change are observed with a scanning electron microscope (SEM) and a transmission electron microscope (TEM), respectively. It is shown that surface morphology and microstructure of irradiated specimens after the tensile test are drastically different from that of unirradiated specimens. The tensile loading also influences behavior of thermal desorption of helium and trap sites.

1. Introduction

Plasma facing materials are subjected to high-flux bombardment of low energy particles including the hydrogen fuel and helium ash. Among them, helium is known to greatly affect damage accumulation and mechanical properties due to strong interactions with defects [1]. Hence, in order to investigate its effect on surface modification of materials and plasma confinement, helium discharge experiments have been carried out by utilizing TRIAM-1M [2] and LHD [3], in which austenitic stainless steels such as 304SS and 316SS have been used for the first wall.

For mechanical properties, many experiments have been carried out on helium implanted austenitic stainless steels [4–7] and the results showed that helium induces embrittlement due to bubble formation and aggregation of bubbles on dislocation caused by strong binding with point defects and their clusters. However, these experiments have been performed under the irradiation by helium ions with energy greater than 100 keV, or with helium production due to nuclear reactions by fission neutrons, in order to simulate DT neutron

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irradiation. Therefore, further experimental investigations at the low energy relevant to fusion plasmas are required for more accurate assessments of surface irradiation. In the present work, microstructure change and helium release due to tensile stress on austenitic stainless steel implanted low energy helium ions have been investigated to examine the influence of helium ash from the plasma on material behavior.

2. Experimental

The material used in the present experiments was 304SS. The chemical composition is: C: <0.08, Si: <1.00, Mn: <2.00, P: <0.045, S: <0.030, Ni: 8.00–10.50, Cr: 18.00–20.00 in wt%. Specimen size is $2.8 \times 5.4 \times 0.1$ mm in gauge and 12.4 mm in total length. Heat treatment for the specimens is conducted at 1323 K for 30 min. Grain size is about 20 μm. Specimens are irradiated with 8 keV helium ions at RT, 573 K and 873 K up to the fluence of $3 \times 10^{21}\ m^{-2}$ using a duo-plasma ion gun equipped with a magnet ion selector. The ion flux (He^{+}) was about $2.0 \times 10^{18}\ m^{-2}\ s^{-1}$.

Tensile tests were performed at RT on unirradiated and irradiated specimens at a strain rate of 3.33×10^{-3}/s. For each irradiation condition, tensile tests were performed. Specimens were tested to failure, and were elastically strained, yield point and plastically strained to 10%, 20% and 40% (total strain). After the tensile test, surface modification before and after the tensile test plastically strained to 10%, 20% and failure was examined with a scanning electron microscope (SEM). In addition, each specimens were electropolished and thinned from the back side to observe near the irradiated surfaces. After that, microstructure change of specimens before and after the tensile test elastically strained, yield point and plastically strained to 10%, 20% and 40% were observed with a transmission electron microscope (TEM). Thermal desorption of helium was also measured with a quadrupole mass spectrometer (QMS) by heating up to 1400 K with a ramp rate of 1 K/s.

3. Results and discussion

Fig. 1 shows depth profiles of dpa and helium deposition rate in 304SS irradiated with 8 keV He ions with a flux of $2 \times 10^{18}\ m^{-2}\ s^{-1}$ calculated by the TRIM-code. The depth profiles show that helium atoms and primary damage are mostly concentrated near the surface and distributed up to 80 nm and 70 nm in depth, respectively. Their peaks are at 40 nm and 20 nm in depth, respectively.

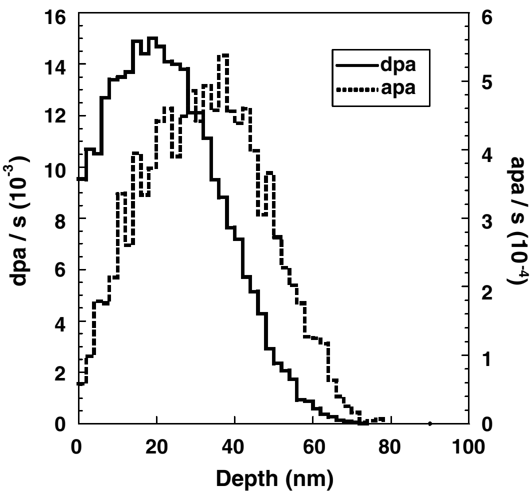

Fig. 1. Dpa and helium deposition (apa) rates in 304SS irradiated with 8 keV He ions at a flux of $2 \times 10^{18}\ He/m^2\ s$ calculated by the TRIM-code.

Shown in Fig. 2 is a cross-sectional TEM image near the specimen surface irradiated at 873 K.

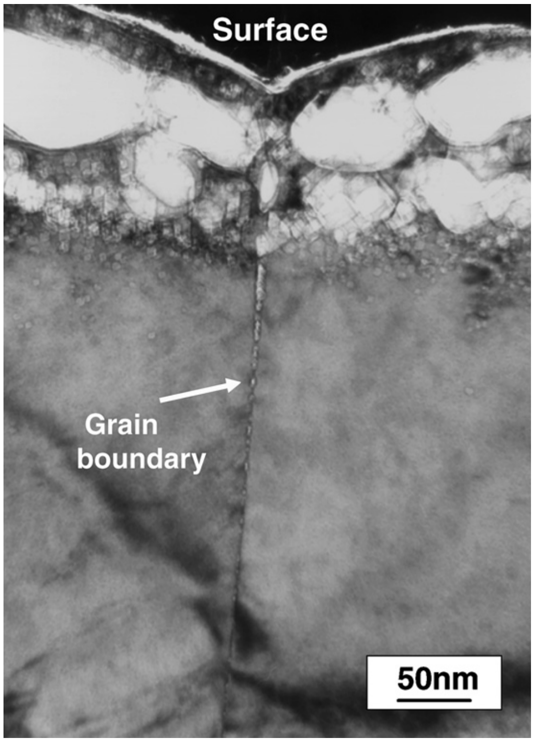

Fig. 2. TEM image of cross-sectional view of region near specimen surface irradiated at 873 K.

Cavities with a size of over 100 nm, which should induce blistering, are observed in the sub-surface region at a depth up to 100 nm. In addition, bubbles with a diameter of a few nm to several tens nm are also formed and distributed up to about 150 nm in depth in grain interiors. They reach up to 400 nm in depth when present at grain boundaries.

Fig. 3 shows SEM images of the surfaces of irradiated specimens before and after the tensile loading. Grain boundaries were easily detected due to thermal etching during annealing before the helium irradiation. No significant modification except for slight dispersion of small blisters is discerned on the surface surfaces irradiated at RT before the tensile test. On the other hand, exfoliation of blisters is observed on the surfaces irradiated at 573 K and 873 K before the tensile test. In the case of the specimen surface which was not irradiated, slip lines were observed on the surface, but cracks were not formed after the tensile test. In the case of the specimen irradiated at RT, many blisters appear on the surface after tensile stress application and they are exfoliated. In addition, cracks are observed in the specimen after fracture. Additionally, in the case of specimens irradiated at 573 K and 873 K, exfoliation of surface layers occurs at 10% and 20% strains. Cracks are also seen after fracture. These surface modifications are considered to be caused by embrittlement of the surface layer irradiated by the helium.

Fig. 4 contains TEM images showing the damage structure of the specimens irradiated at RT and 873 K before and after the tensile test. White contrast indicates helium bubbles. A high density of fine bubbles of a few nanometer in diameter are formed at room temperature. After the tensile tests, bubble densities for the elastically strained specimen, 10% strain, 20% strain and 40% strain decrease as shown in Fig. 4. Fig. 5 shows a TEM image of the damage structure of the specimen irradiated at RT after the tensile test to yield point. It can be seen that a hole and cracks along the grain boundary are formed by

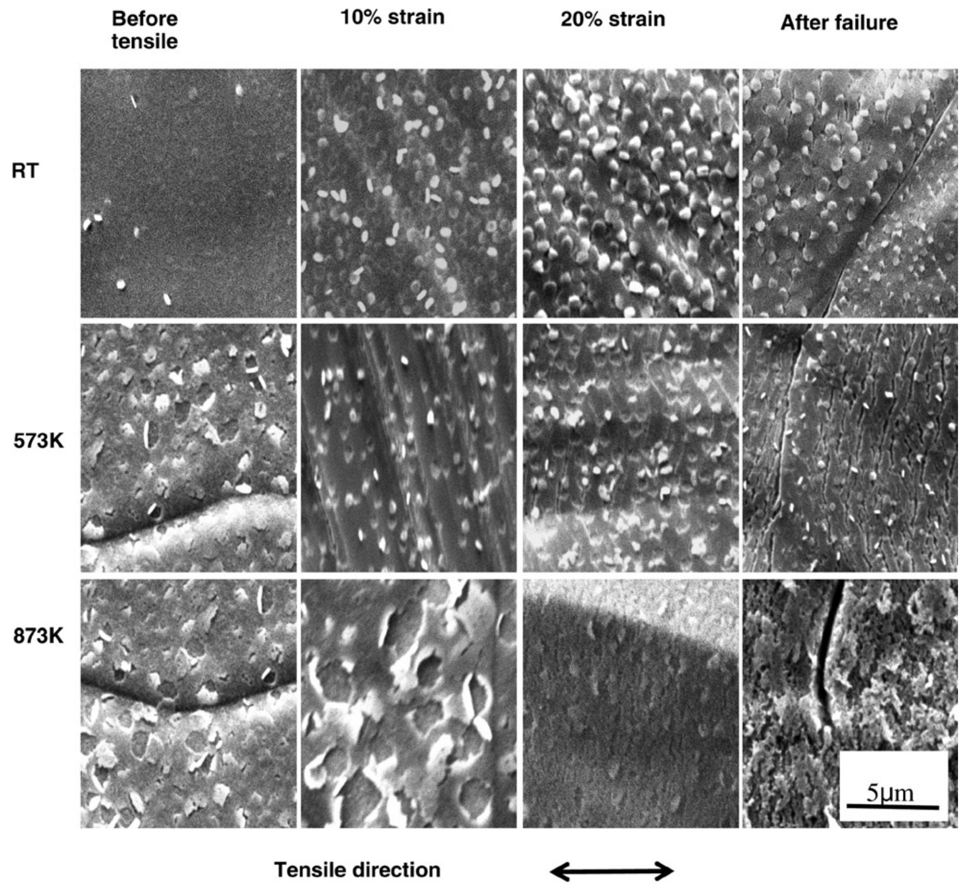

Fig. 3. SEM images of specimen surfaces irradiated at RT, 573 K and 873 K before and after tensile test.

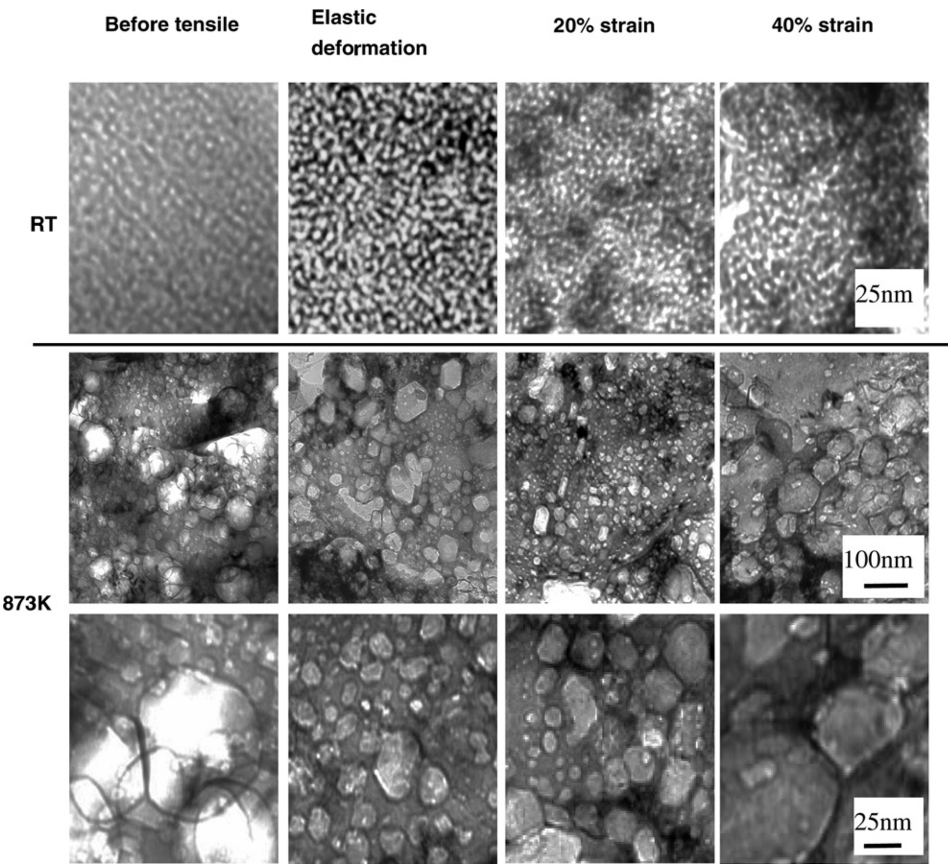

Fig. 4. TEM images of region near specimen surfaces irradiated at RT and 873 K before and after tensile test.

tensile stress. Our data from the SEM observation on surface modification as well as the TEM observation indicates that the decrease of helium bubbles may be caused by helium release through cracks on the surface and inter-bubble fracture. In contrast, the number of bubbles decreases but the individual bubbles grow larger by aggregating with each other at a temperature of 873 K where sufficient thermal migration of vacancies is expected. After the tensile test, the spherical shape of bubbles becomes irregular. One of the reasons may be that coalescence and growth of bubbles through a migration process that is enhanced by interaction with moving dislocations causes the formation of the bubble clusters like this.

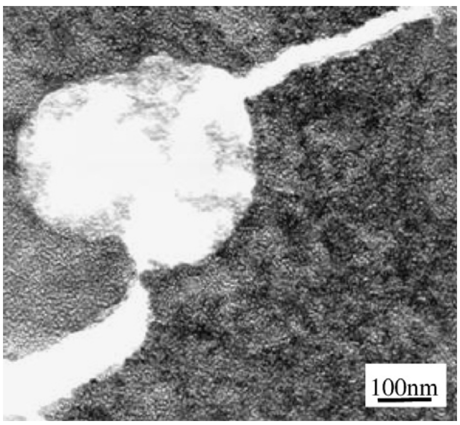

Fig. 5. TEM image of region near specimen surfaces irradiated at RT after tensile test to yield point.

Fig. 6 contains TDS spectra from the specimens irradiated at RT and 873 K before and after the tensile test. It seems that there are two stages of helium release at low (300–900 K) and high (900–1400 K) temperatures before the tensile stress. The release at high temperature is considered to be the bubble migration to the surface [8]. On the other hand, helium is also weakly trapped in the distorted lattice around high pressure bubbles and other types of lattice defects at dose above 1×10^{21} He/m^2. Helium trapped in this way is considered to be released at

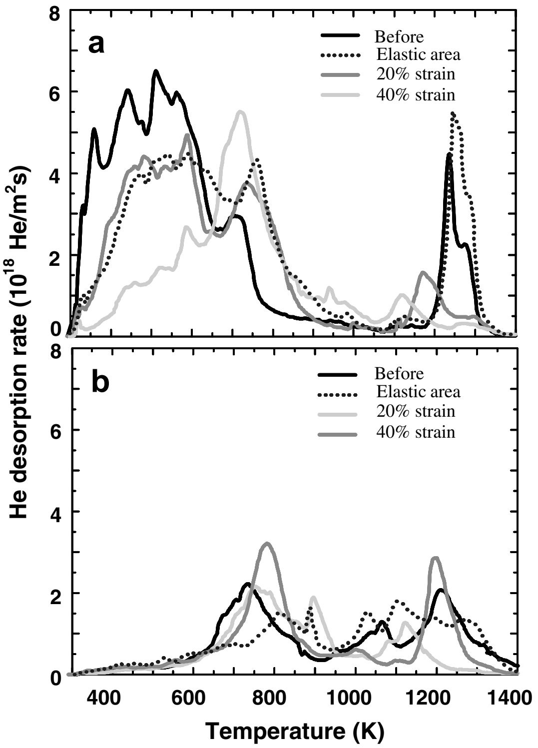

Fig. 6. TDS spectrum of specimens irradiated at (a) RT and (b) 873 K before and after the tensile stress.

the low temperature [8]. As shown in Fig. 6, amount of helium released in the temperature range from 300 K to 650 K decreases and amount released from 650 K to 900 K increases as strain increases. In the case of high temperature, the amount of the helium released decreases as strain increases and the peaks shift to at lower temperature except for the elastically strained specimen. The total amount of helium released decreases as strain increases. On the other hand, TDS spectra from the specimens irradiated at 873 K before and after the tensile test shows helium releases in a wide temperature range compared with that from the specimens irradiated at room temperature. Changes of behavior of the helium release by tensile stress is considered to be due to the formation and breaking of blisters, surface exfoliation, crack formation and movement of dislocations. Detailed work will be required to investigate the mechanisms. In addition, it is more correct to perform tensile tests at temperature close to operation temperature of the fist wall.

4. Conclusion

Microstructure change and helium release due to tensile stress on austenitic stainless steel implanted with low energy helium ions have been investigated. It is shown that surface morphology and microstructure of irradiated specimens after the tensile test are drastically different from that of unirradiated specimens. It is hence suggested that helium irradiation would bring about embrittlement of the surface layer of 304 stainless steel. The tensile stress also influences behavior of thermal desorption of helium and trap sites. These results indicate the importance of synergistic effects of helium irradiation from the plasma and applied tensile stress on surface modification and erosion of the first wall under a fusion environment.

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