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Surface Modification of Low Energy Helium Ion Implanted Austenitic Stainless Steel by Tensile Stress

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Abstract

Mechanical properties of low energy He irradiated 304SS have been investigated by means of tensile test and nano-identation hardness tests. Specimen size is 2.8×5.4×0.1mm in gauge and 12.4mm in total length. Specimens are irradiated with 8 keV helium ions at R.T., 573K and 873K up to the fluence of 3×10^{21} He/m^2. Tensile tests are performed on unirradiated and irradiated specimens at a strain rate of 3.33×10^{-3} /s. In every irradiation condition, one specimen is tested to failure and the other two specimens are strained plastically to 10% and 20%. Formation of blisters with a diameter of about 500 nm is observed on the surface of tensile-tested specimens after R.T. irradiation. Some exfoliation of the blisters is also observed. When the irradiation temperature increases, a large number of cracks with a size of about several μm are formed on the surface together with exfoliation during the tensile test. The yield stress of specimens irradiated at R.T. and 573 K increases about 10% in comparison with that of the unirradiated specimen. The result of nano-indentation tests indicate that hardness near the surface increases depending on the irradiation temperature.

Key words : *High heat load Helium, Low energy ion, Mechanical property, Tensile stress, Hardness*

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 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 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, mechanical properties of austenitic stainless steel after low energy helium irradiation have been investigated by means of tensile tests and nano-identation hardness tests to examine the influence of helium ash in the plasma.

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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 weight percent. Specimen size was 2.8×5.4×0.1mm in gauge and 12.4 mm in total length. Heat treatment for the specimens was conducted at 1323 K for 30 min. Grain size was about 20 μm. Specimens were irradiated with 8 keV helium ions at R.T., 573K and 873K up to the fluence of 3×10^{21} m^{-2} using a duo-plasma ion gun equipped with a magnet ion selector. The ion flux (He^+) was about 2.0×10^{18} m^{-2}s^{-1}.

Tensile tests were performed on unirradiated and irradiated specimens at a strain rate of 3.33×10^{-3} /s. For each irradiation, three specimens were tensile- tested. One specimen was tested to failure, and the remaining two specimens were plastically strained to 10% and 20%. After the tensile test, surface modification and microstructure change were observed with a scanning electron microscope (SEM) and a transmission electron microscope (TEM), respectively. Micro-indentation tests were also conducted on both unirradiated and irradiated specimens at room temperature with a load of 1 gf. A triangular pyramidal diamond indentor (Berkovich-type) with a semi-apex angle of 65° was used in this study. The results of Ref.[8] indicate that the indentor load (L) and the displacement (d) can be approximately related as follows:

$$L/d = Ad + B \quad (1)$$

They found good agreement between the coefficient A and Vickers hardness (Hv) for several materials according to the following empirical relation:

$$A(GPa) \sim 0.287\,Hv. \quad (2)$$

3. Results and discussion

Depth profiles of dpa and He deposition rate in 304SS irradiated with 8 keV He ions with a flux of 2×10^{18} $m^{-2}s^{-1}$ were 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.

Figure 1 shows SEM images taken from specimen surfaces before and after the irradiation. 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 was discerned on the sample surfaces irradiated at R.T. On the other hand, exfoliation of blisters was observed on the surfaces irradiated at 573 K and 873 K.

Shown in Fig. 2 is a cross sectional TEM image near the specimen surface irradiated at 873 K. Cavities with a diameter of over 100 nm, which should induce blistering, were observed in the sub-surface region of 100 nm. In addition, bubbles with a diameter of a few nm to several tens nm were also formed and distributed up to about 150 nm in depth in grain interiors. They reached up to 400 nm in depth when present at grain boundaries.

Figure 3 shows L/d-d plots for both unirradiated specimens and those irradiated at R.T., 573K and 873 K. The gradient of the plot in the loading stage corresponds to

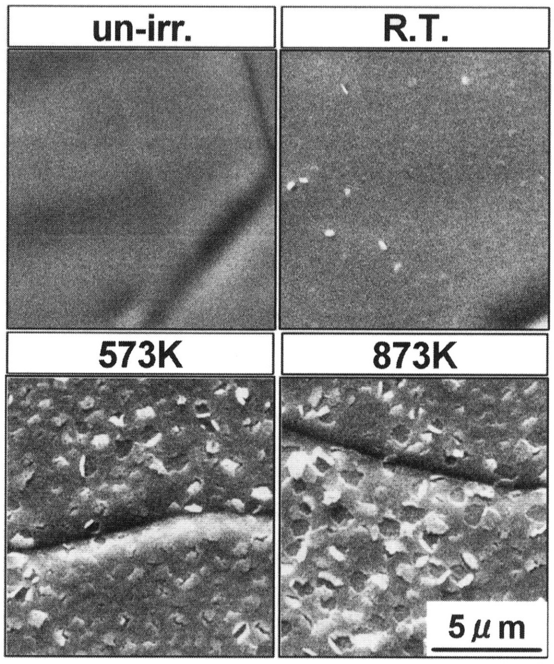

Fig.1 SEM surface images of specimens unirradiated and irradiated at R.T., 573 K and 873 K.

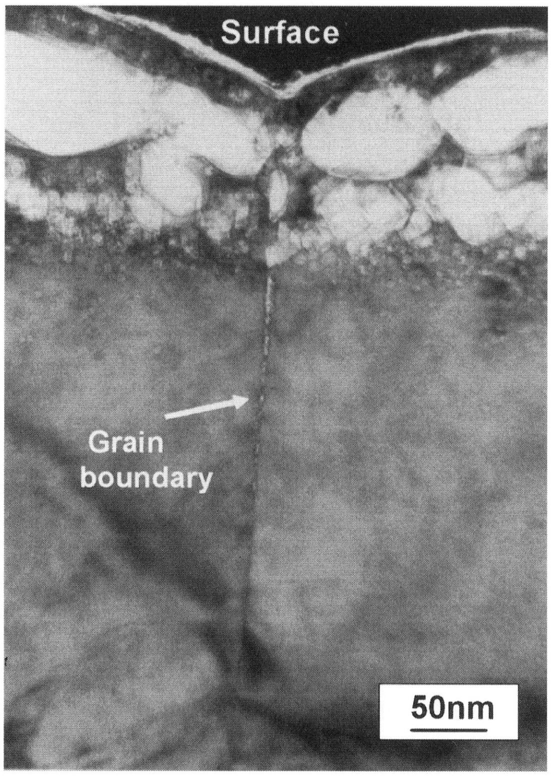

Fig.2 TEM image of cross-sectional view of specimen surface irradiated at 873 K.

the hardness according to equation (2). The plot of the unirradiated specimens show good linear correlation between L/d and d through the whole loading process. This means that hardness is constant along the depth direction. On the contrary, the gradient of the irradiated specimens changes along the depth. This change is probably attributed to hardening near the surface due to helium irradiation. The gradient also shows that the hardening near surface decreased with increasing irradiation temperature.

Figure 3 also shows that the range of hardening increased with increasing irradiation temperature. In the case of high temperature irradiation, hardening was observed up to 80 nm and 150 nm in depth at 573 K and 873 K, respectively. In the case of 873 K, the hardening spread to deeper regions where bubbles were observed in Fig. 2. It seems that formation of highly pressurized bubbles would be a cause of surface hardening noted above. Figure 4 shows the evaluated Vickers hardness of the surface based on Eq.2. The hardness decreased with increasing irradiation temperature. This trend could be, at least partially, associated with the reduction of bubble pressure, which is expected to take place through vacancy absorption and/or bubble coalescence at high temperature.

Shown in fig.5 is atypical stress-strain plot. Surface modification was observed at 10%(b), 40%(c) strain and fracture(d). Figure 6 shows SEM images of the specimen surface, which was not irradiated, before and after the tensile test. Slip lines were observed on the surface, but cracks were not formed. Shown in Figs. 7 are SEM images of the surface of irradiated specimens after the tensile test. In the case of the specimen irradiated at R.T., many blisters appeared on the surface after tensile stress application and they were exfoliated. In addition, cracks were observed in

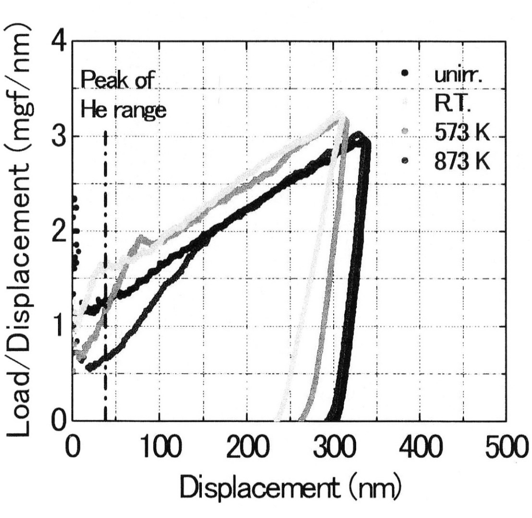

Fig. 3 L/d-d plots for specimens unirradiated and irradiated at R.T., 573 K and 873 K.

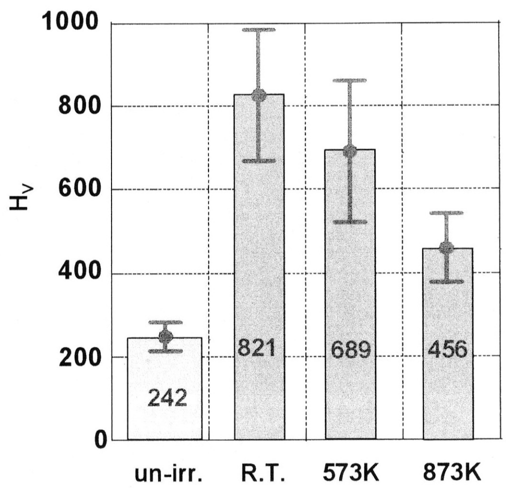

Fig.4 Hardness of surface region of specimens unirradiated and irradiated at R.T., 573 K and 873 K. Hardness was evaluated by Eq.(2).

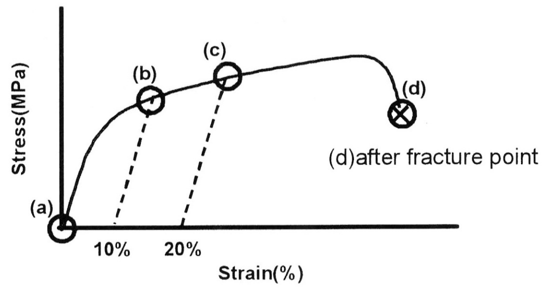

Fig. 5 Typical stress-strain plot.

the specimen after fracture. Additionally, in the case of specimens irradiated at 573 K and 873 K, exfoliation of surface layers occurred at 10% and 20% strains. Cracks were also seen after fracture. These surface modifications were considered to be caused by embrittlement of the surface layer irradiated by the helium.

The yield stress of specimens irradiated at R.T. and 573 K increased about 10% in comparison with that of the unirradiated specimen. The surface hardening layer may influence the yield stress because the hardening layer was shallow, however, hardness near surface significantly increased. On the other hand, the maximum stress was almost not changed. These results reflect the fact that since the projective range of helium implantation is shallow as compared to the thickness of the specimen, effects of low energy helium irradiation on macroscopic tensile properties are limited.

4. Conclusion

The mechanical properties 304SS irradiated with low energy helium ions have been investigated through post irradiation

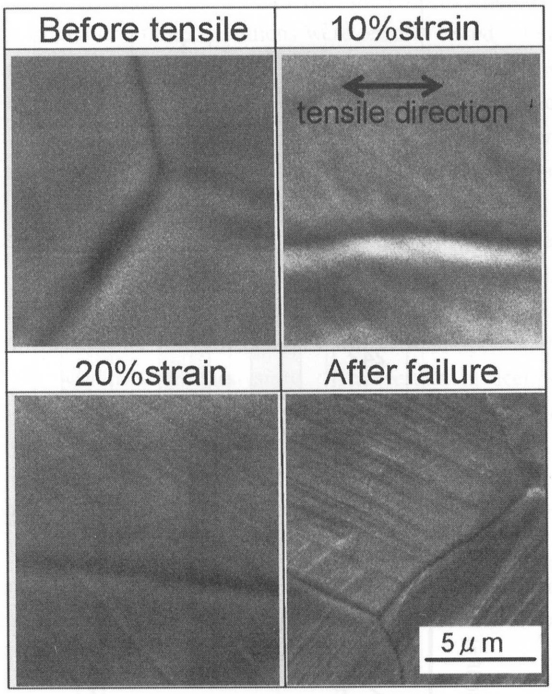

Fig.6 SEM surface images of unirradiated specimen before and after tensile test.

tensile and nano-identation hardness tests to examine the effect of helium ash in fusion plasma. It is shown that surface morphology of irradiated specimens after the tensile test is drastically different from that of unirradiated specimens. Nano-indentation tests indicate that hardness near the specimen surface increases depending on the irradiation temperature. It is hence suggested that helium irradiation would bring about embrittlement of the surface layer of 304 stainless steel. 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 fusion environment.

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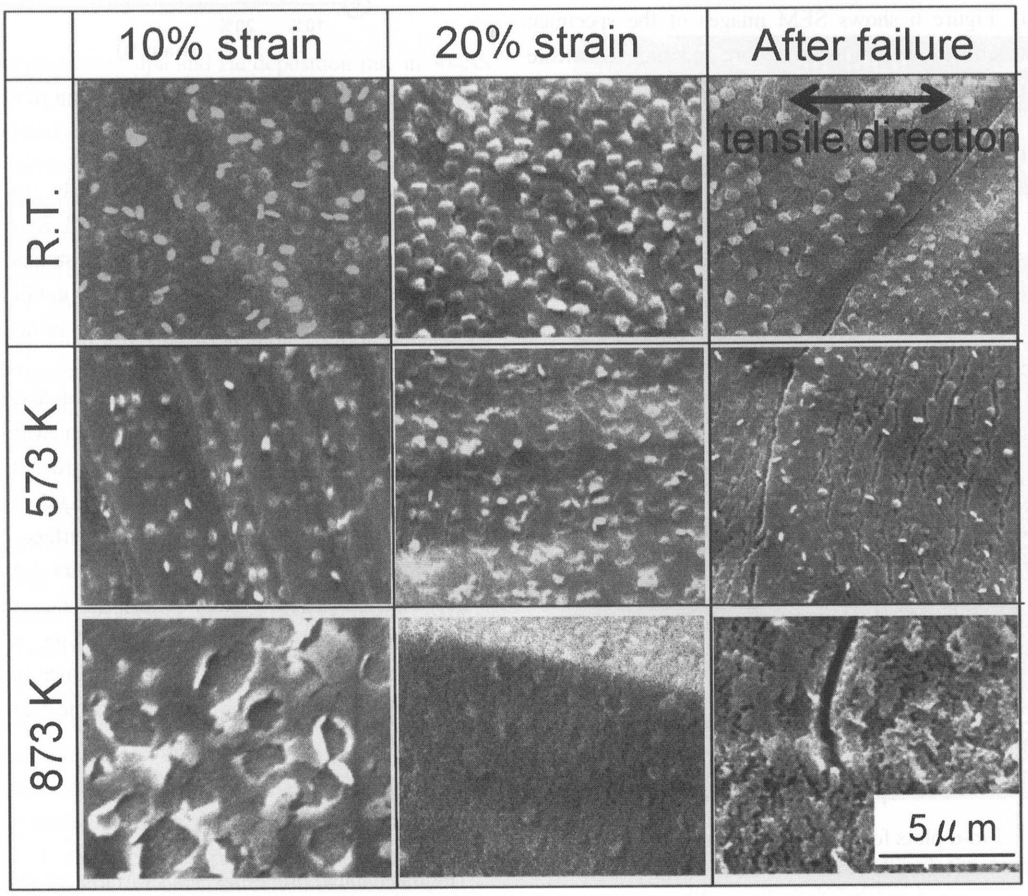

Figure 7 SEM images of specimen surfaces irradiated at R.T., 573 K and 873 K after tensile test.

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