§5. Correlation between Defect Structures and Hardness in Tantalum Irradiated by Heavy Ions

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Tantalum is one of the candidate materials for structural components of divertors because of its high toughness, high sputtering threshold energy, high fabricability and low-activation properties. However, little is known about radiation effects at present. In the present study, detailed TEM observation and hardness estimation and post-irradiation annealing of irradiated specimens were carried out.

The material used in this study was 99.95% pure tantalum with impurities of ~30 wt. ppm O, ~20 wt. ppm N and ~30 wt. ppm C. After cold rolling and punching into TEM disks, the specimens were annealed at 2073 K for 300 sec in a vacuum of <3x10^{-5}Pa for recrystallization. Irradiation was performed with 2.4 MeV Cu^{2+} ions at several temperatures between R.T. and 1073K using a Tandem type accelerator in Kyushu University. The damage level of the irradiation varied from 0.03 to 3dpa at its peak position of damage. The peak damage depth calculated by the TRIM3D code was 340nm. After the irradiation, specimens were back-thinned by electropolishing with a solution of 2.5% HF, 5% H_2SO_4 and 92.5% methanol at about 230K. Microstructures at the depth of 250nm were observed with a JEM-2000E X I I transmission electron microscope at Kyushu University. The micro-indentation tests were also conducted on both unirradiated and irradiated specimens at room temperature using an Elionix ENT-1100 with a load of 1gf. A triangular pyramidal diamond indentor (Berkovich-type) with a semi-apex angle of 65 degrees was used in this study.

Hardening below 573K saturates at a low level. On the other hand, at temperatures between 673 and 873K, the hardness increases with dose and becomes about three times higher than that of unirradiated value at 3dpa. Above 873K, hardening decreases with increasing temperature. It must be emphasized that the dose dependence of hardening is quite different below 573K and above 673K. To examine the difference in defect structure, the microstructure of specimen irradiated at 573K and 673K up to 3dpa was observed. Vacancy loops of about 4nm were observed in dark field images at both temperatures. A high density of voids of <2nm diameter were formed at 673K but not at 573K. Small white spots, which are presumably due to strain field around voids, were also observed in the dark field image at 673K. The density of small voids is about two orders of magnitude higher than that of vacancy loops. Therefore, the difference of hardness at 573K and 673K is due to the existence of a high density of voids.

In order to obtain the annealing induced change in the effects of irradiation on the hardness, the hardness change as a function of isochronal annealing temperature of the specimen irradiated at R.T. and 0.3dpa was estimated. For irradiated specimens, there is a prominent hardness increase at 573-673K. The high hardening is maintained up to 873K, and then decreases with increasing the annealing temperature. On the other hand, hardness change is not observed in unirradiated specimens at any temperature.

Dark field TEM microstructure image shows that small loops and a high density of small dot defects of ~1nm, which are probably vacancy clusters, appeared at 723K. At 1073K, dot defects are not observed in dark field images. Instead, a low density of voids of about 2nm were formed. These results suggest that the hardness increase above 573K is attributed to small vacancy clusters as observed 723K.

In conclusion, correlation of microstructure and hardness in heavy ion-irradiated pure tantalum has been studied. Significant hardening occurred under irradiation or post-irradiation annealing between 573K-673K. TEM observation revealed that the formation of small voids is mainly responsible for radiation hardening in this temperature regime.