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Microstructure of tantalum irradiated with heavy ions

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Abstract

For the purpose of investigating the general response of tantalum to irradiation, microstructures have been observed after irradiation with 3.0 or 2.4 MeV Cu^{2+} ions at 773 to 1546 K and to 20 dpa. Below 1073 K, the microstructures consisted mainly of high density of small dislocation loops and straight dislocations. The small dislocation loops observed were identified to be of vacancy-type. Voids were formed above 973 K, however, the swelling was negligibly small up to 20 dpa below ~1100 K. High density of defects observed below 1073 K may cause significant change in mechanical properties. This may be a concern in applying tantalum to fusion high heat flux components.

1. Introduction

Tantalum is one of the candidate materials for divertor structural components of fusion reactors because of its high toughness, good compatibility with liquid alkali metals and high fabricability [1]. Neutronics calculations show that tantalum has very low long-lived radioactivity after exposure to fusion neutrons and is thus regarded as a low-activation material from the waste-disposal viewpoint [2]. However, group V refractory metals have a common problem for embrittlement in the presence of hydrogen [3].

Other b.c.c. refractory metals such as Nb and Mo have been widely investigated, involving the effect of impurity atoms and alloying elements. However, studies on radiation response of tantalum is quite limited and hence substantial irradiation studies are needed for evaluation of tantalum as a candidate material for fusion reactor.

Tantalum is also a candidate target material for spallation neutron sources, which is also subject to high flux of high energy neutrons [4].

The objective of the present study is to investigate the general response of pure tantalum to irradiation by means of heavy ion irradiations. Emphasis is placed in this study on dose and temperature dependence of microstructures.

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2. Experimental

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 being cold rolled and punched into TEM disks, the specimen was annealed at 2073 K for 300 s for recrystallization. The surface layer was removed by electropolishing prior to irradiation.

Ion irradiations were carried out using 3.0 or 2.4 MeV Cu^{2+} ions at 773 to 1546 K using a Tandem type accelerator in Kyushu University. The peak damage depth calculated by the TRIM3D code were 410 and 340 nm, respectively. And the damage level was 0.03 to 20 dpa at its peak damage position. After irradiation, specimens were back-thinned by electropolishing with a solution of 2.5% HF, 5% H_2SO_4 and 92.5% methanol at 230 K to observe the microstructures by a transmission electron microscope. Microstructures were observed with JEM-2000EX II and JEM-2000FX electron microscopes of Kyushu University.

3. Results

Fig. 1 shows the temperature dependence of microstructures at 3 dpa. The line dislocations and high density of small dislocation loops are formed below 1073 K. At 973 and 1073 K, these loops were identified to be of vacancy type by the inside-outside contrast method [5].

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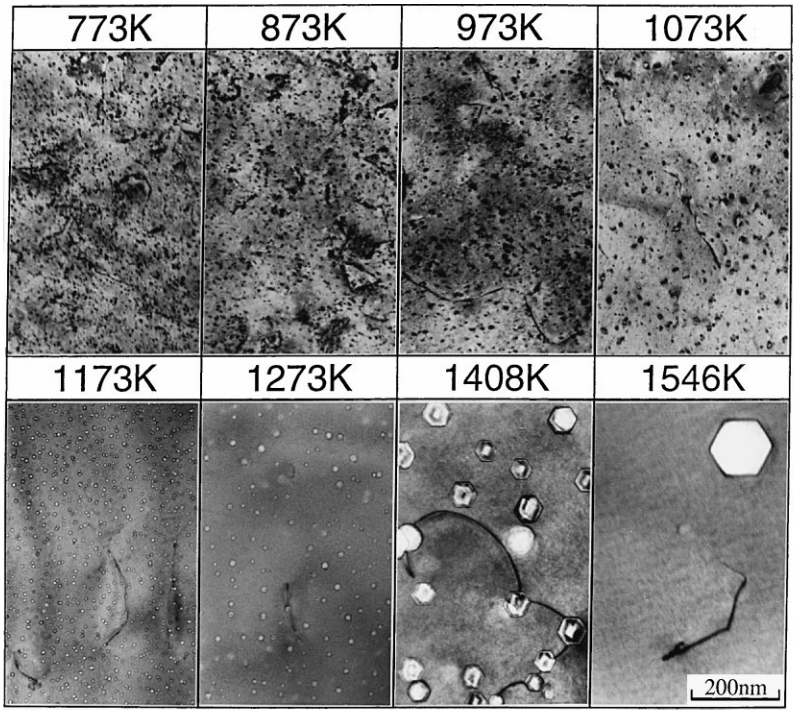

Fig. 1. Temperature dependence of microstructure in Ta at 3 dpa.

Small voids of about 3 nm are also formed at 973 and 1073 K and above 0.3 dpa. But swelling was very low at the both temperatures even at 20 dpa. Above 1073 K, the damage structure consisted primarily of voids and low density of extended line dislocations. Voids were distributed homogeneously in the matrix. Void sizes increased and void number densities decreased with increasing irradiation temperature. Fig. 2 shows the temperature dependence of the dislocation density and void swelling. The swelling reaches its peak value of 8.2% at 1408 K and 20 dpa.

Fig. 3 shows the dislocation loop formation and evolution with dose at 973 K. Interstitial type dislocation loops of about 10 nm in diameter were formed

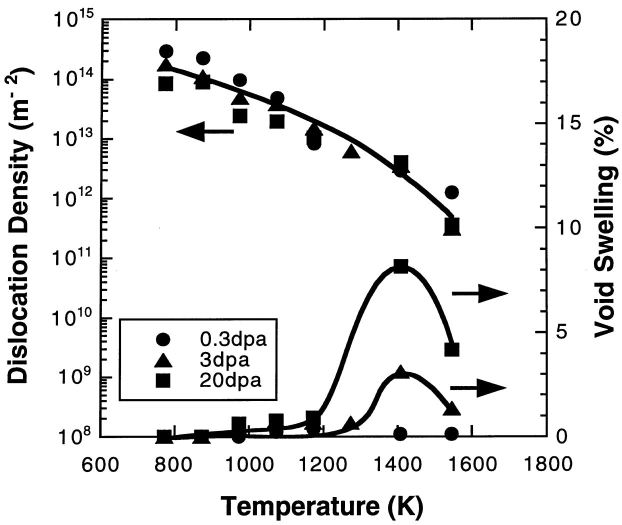

Fig. 2. Temperature dependence of dislocation density and void swelling.

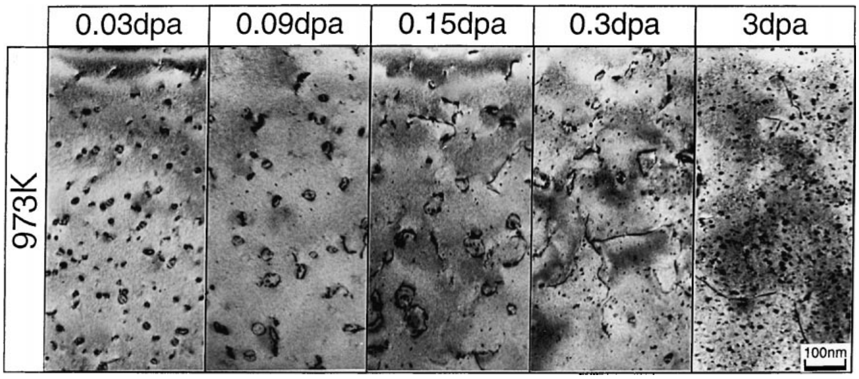

Fig. 3. Formation and evolution of dislocation loops at 973 K.

uniformly at 0.03 dpa. During the successive irradiation, these dislocation loops coalesced with each other increasing their size, and then tangled into low density of line dislocations. The formation process of the small loops was, however, quite different. They formed preferentially inside large interstitial loops. The average size and number density of the loops gradually increased with dose and reached 4.7 nm and 5.0 x $10^{22}/m^3$ at 3 dpa, respectively.

Fig. 4 shows void evolution with dose at 1173 and 1408 K. Voids were already formed at 0.3 dpa at both temperatures. Whereas the average void diameter increased with dose, the void number density was almost constant at the both temperatures. Similar saturation of void density at low dose was observed at all temperatures where voids were formed.

4. Discussion

The high density of small dislocation loops were observed in tantalum in the temperature range of 773–1073 K. According to the fact that the small loops observed at 973 and 1073 K were identified to be of vacancy type, the small loops observed at 773 and 873 K were deduced to be also of vacancy type. Moreover, enhanced formation of small loops were observed inside large interstitial loops at 973 K in the present study. The same tendency of the formation of small clusters by the irradiation with electrons was reported in gold and copper [6,7]. In the case of gold, the clusters were assigned to be aggregates of vacancies from the positional relation with the interstitial loops [6].

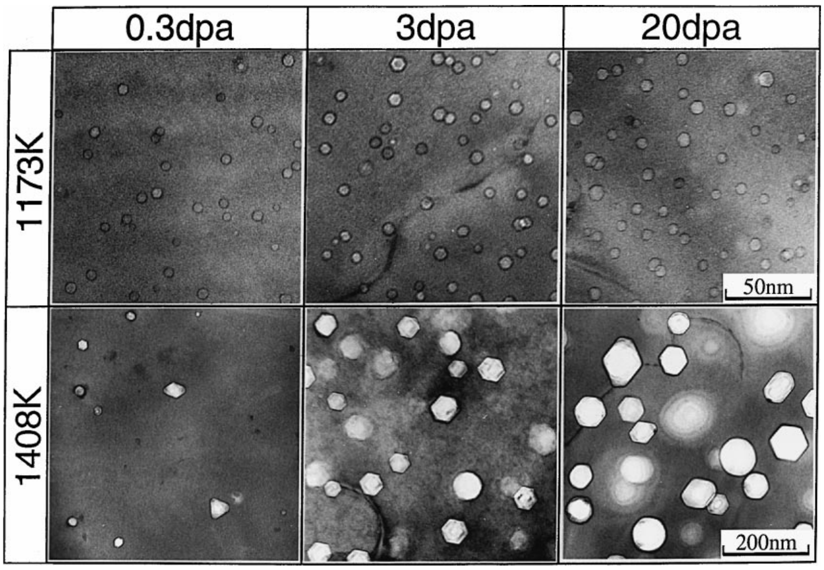

Fig. 4. Void evolution at 1173 and 1408 K.

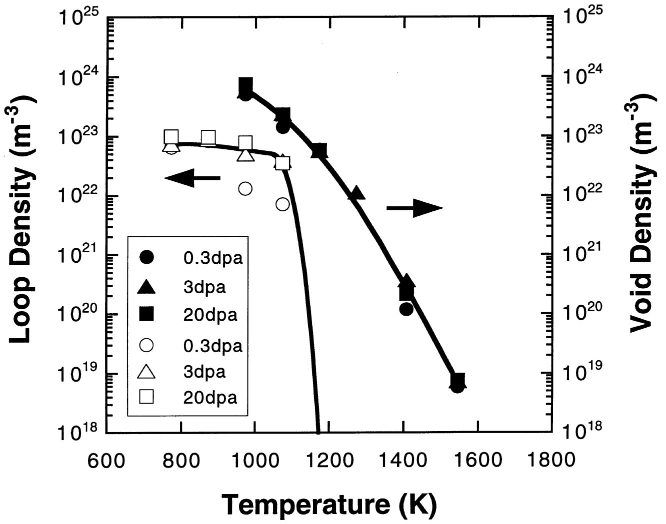

Fig. 5. Temperature dependence of dislocation loop density and void density.

At 973 and 1073 K, small voids were also observed and its number densities were about an order of magnitude higher than those of small loops at any dose as is shown in Fig. 5. These results indicate that stable morphology of vacancy cluster changes from loops to voids with the increases in the temperature. The transition temperature of the change of morphology is 973–1073 K.

The present study showed that the void swelling of tantalum is negligibly small below ~1100 K. It should be noted, however that the temperature regime of swelling by ion irradiation is higher than that of neutron irradiation mainly due to the difference in the damage rate. Typical shift of the swelling temperature was reported to be ~200 K [8]. Thus the present study may imply that the void swelling of tantalum by neutron irradiation would be negligible below ~900 K. On the other hand, change of mechanical properties is expected below ~1100 K according to the high densities of defect clusters observed. Microhardness tests of the identical irradiated specimens and the correlation with the present microstructural data are in progress.

5. Conclusions

The microstructures of tantalum induced by Cu^{2+} ion irradiations were composed mainly of high density of vacancy-type clusters and voids at low and high temperature regime, respectively. The transition temperature of vacancy-type loops to voids was 973–1073 K. Below ~1100 K, although the void swelling was negligibly low to 20 dpa, significant change of mechanical properties was expected because of the formation of high density of defect clusters. This will be a major subject of radiation effect of tantalum in the use of fusion reactor high heat flux components.

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