学位論文の要約

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Ion-irradiation Hardening and Microstructure Evolution in Tungsten

(タングステンのイオン照射硬化および微細組織変化)

This dissertation researched the the ion-irradiation hardening and microstructure evolution in tungsten. There are 8 chapters in all, where the 8th chapter is the summary and conclusions.

In chapter 1, the previous researches on irradiation behavior of tungsten and its alloys were reviewed. Generally, the researches on tungsten were very limited, especially the materials behavior at high irradiation temperature.

In chapter 2, the materials and general experimental method were introduced. The as-received rolled tungsten was completely recrystallized at 1400°C according to the isochronal annealing. The grain size was about 1.7 x 1.0 x 1.0 μm. The texture was found (100)<011> and (111)<112> on the rolling surface. The irradiation was carried out by single-ion beam (6.4 MeV Fe^{3+}) or dual-ion beam (6.4 MeV Fe^{3+} + 1 MeV He^{+}) on both as-received and recrystallized tungsten at 300 °C, 500 °C, 700 °C and 1000 °C. The average dpa of irradiated layer was 2 dpa. For dual-ion beam irradiation, the average concentration of helium was 3000 appm in the 1.5 μm implanted range.

In chapter 3, irradiation hardening was measured by nano-indentation method. An equation to evaluate the hardness of irradiated thin area near specimen surface was proposed simply based on the assumption that geometrically necessary dislocations (GND) density at an indentation depth was same before and after irradiation. The results showed a constant hardness depth area in the range from 200 nm to 300 nm. The hardness of this area was defined as the hardness of the irradiation layer. The hardening of tungsten irradiated at 300 °C is significantly lower than those at the other higher temperatures. In the case of single-ion beam irradiation, recrystallized W exhibited a higher hardening than as-received one at all the temperatures. The effect of helium on the irradiation hardening is dependent on the material condition: as-received W showed an additional hardening by helium at all the irradiation temperatures, while in recrystallized W the hardening was not affected by helium below 700°C.

In chapter 4, the microstructures of single-ion beam irradiated as-received and recrystallized tungsten were observed by TEM. The line dislocations, dislocation loops and voids (bubbles) were observed and analyzed. Their generation processes were also discussed. The overall depth profiles of microstructures were investigated. At 300 °C, a black band, which consisted of high number density of radiation defects, appeared in the area close to the end of the damaged area. With elevating the irradiation temperature, the black band was gradually annealed out, instead, diffusion area beyond the calculated damaged area became significant. At 700 °C and 1000 °C, a low defect zone appeared between the near surface area and the damage diffusion area, which was named middle area. Both the line dislocations and dislocation loops were found in all the irradiated tungsten. The dislocation loops were found to form aggregates, especially 1D loop rafts below 700°C and 2D rafts at 500 °C. Irregular shapes of the loops as well as loop rafts were observed at lower irradiation temperatures. Besides, the interaction of line dislocations and dislocation loops was investigated. In the tungsten irradiated at 1000 °C, no loop raft but "loop lattice" was observed in the surface area. Voids were generated at 700 °C and 1000 °C. The voids were observed at the maximum depth of 1.3 μm at 700°C and 2 μm at 1000 °C. There is no significant difference in the size and density of voids between as-received tungsten and recrystallized tungsten. The void distribution was inhomogeneous in the tungsten irradiated at 1000 °C. Voids were observed mainly outside the loop lattice. The void formation became scarce in the intermediate depth area. Grain boundary effect on the formation of dislocation loops and line dislocations was discussed. The grain boundary could suppress the formation of the loops and lead to a denuded (free of loops) zone. Line dislocations are not affected by grain boundaries.

In chapter 5, the microstructures of dual-beam irradiated tungsten were investigated by TEM observation. At 300 °C, a black band consisted of dislocation loops was observed, which is similar to the case of single-ion beam irradiation. At higher temperatures, the black band gradually disappeared and substituted by middle area where the number density of the defects is low. At 1000 °C, the microstructures were divided into three zones: front area, middle area and diffusion area. The general distribution is similar to single-ion beam irradiation except that the large size of bubbles were found in the front area. Several dislocation loops formed into a raft structure below 700°C. The orientation of 1D loop raft was determined as <110>. Helium bubbles were observed at temperatures above 500 °C. The size were stable to 700 °C but increased dramatically at 1000°C. No cavity was found in the diffusion area the grain boundaries were found to have quite different effect on loops and bubbles. The grain boundaries is considered to enhance the absorption of interstitial clusters in the matrix near the grain boundary but promote bubble growth in the grain boundary, as the bubbles were larger in size and lower in density on the grain boundaries than inside the grains. The morphology of dislocation and loops were generally similar to single-ion beam irradiated tungsten. The difference is the enhanced generation of helium bubbles. Tiny bubbles were observed at 500 °C and 700 °C, but the bubble size greatly increased to 8.4 nm at 1000 °C. In the front area, loop lattice was observed. The lattice appeared to have the same structure as single-ion beam irradiated recrystallized tungsten. The generation of lattice structure was confirmed not be affected by simultaneous helium ion beam irradiation.

In chapter 6, the self-ordering loop lattice structure in the tungsten irradiated at 1000 °C was analyzed. It has been over 40 years for the finding of "void" lattice in tungsten. It is, however, the first time the loop lattice was observed in BCC materials under irradiation. The loop lattice showed a BCC lattice structure. The lattice only formed in the front of the irradiated area. The relation between loop lattice and matrix is $[100]_{loop}//[100]_{matrix}$. The lattice constant is about 45 nm.

Through visible/invisible ($g{\cdot}b$) analysis, the loops were confirmed with ½<111> burgers vector. By inside-outside technique, the nature of the loop forming loop lattice was found to be interstitial type. The requirements of the formation of loop lattice were discussed.

In chapter 7, the effects of grain boundaries and helium on the microstructure evolution were discussed. The effect of grain boundaries as a short range or long range sinks for defects was discussed by comparing the size and density of loops and voids in the matrix and near GB. Also, the correlation of microstructure evolution and irradiation hardening was investigated by Orowan type equations. This method was applied to the hardening of dual-ion beam irradiated tungsten. The results suggested that cavities were not the main factor in irradiation hardening and that traditional size related hardening factor model overestimated the hardness values for large defects.

Chapter 8 showed the summary and conclusions.

Conclusions:

As-received and recrystallized tungsten were irradiated with single-beam of 6.4 MeV Fe^{3+} or dual-beam of 6.4 MeV Fe^{3+} + 1.0 MeV (energy degraded) He^{+} at 300, 500, 700 and 1000°C. Hardness was measured by nano-indentation method and microstructure evolution was observed by TEM.

(1) Tungsten after single-ion irradiation showed the lowest hardening at 300°C and highest at about 700°C. The hardening of recrystallized tungsten is larger than that of as-received ones. In tungsten after dual-ion beam irradiation, the hardening increased from 300°C to 700°C but no remarkable effect of recrystallization was observed. At 1000°C, the hardening is smaller than 700°C.

(2) Ion-irradiation induced both line dislocations and dislocation loops. Dislocation loops were formed into raft structures at 300°C to 700°C but lattice structure at 1000°C. There is strong interaction between line dislocations and dislocation loops at temperatures below 700°C. Helium implantation was found to enhance the formation of bubbles.

(3) The effect of grain boundaries on the defect formation is different between dislocation loops and bubbles. The grain boundaries would suppress the formation of dislocation loops but promote the bubble growth.

(4) Loop lattice structure was found in ion-irradiated tungsten. The loop lattice only appeared in the front area of tungsten irradiated at 1000°C. The lattice has a BCC structure. The orientation relationship between loop lattice and matrix is $[100]_{loop}//[100]_{matrix}$.