Irradiation-resistant Properties of Structurally Controlled Molybdenum Alloys Through a Multi-step Internal Nitriding

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Abstract

In order to overcome the recrystallization embrittlement and irradiation embrittlement of Mo, which are major problems for its fusion applications, internally nitrided Mo alloys were prepared by a novel multi-step internal nitriding. Neutron irradiation was performed in the Japan Material Testing Reactor (JMTR). After irradiation, nitrided Mo alloys exhibited l ower ductile-brittle transition temperature than irradiated TZM. These results suggested that multi-step internal nitriding was effective to the improvement in the embrittlement by irradiation. Transmission electron microscope observation revealed that TiN particles precipitated by nitriding acted as a sink for irradiation-induced defects.

Keywords : Mo alloys, neutron irradiation, ductile-brittle transition temperature, TiN particles

1. Introduction

Recently, molybdenum and its alloys have received much attention as the most promising materials for the structural materials in severe condition such as the plasma-facing component of advanced fusion devices because of the high melting points, good thermal conductivities, and high level of strength. These materials, however, are very susceptible to intergranular failure in the recrystallized state (recrystallization embrittlement) and in the neutron irradiated state (irradiation embrittlement), which are major problems for their applications. It is therefore necessary to develop the Mo alloys that are unsusceptible to both the recrystallization embrittlement and the irradiation embrittlement.

As one of the methods for developing such alloys, we proposed a recrystallization control of dilute Mo-Ti alloys by a novel multi-step internal nitriding.[1] In this method, it is possible to disperse fine TiN particles into the rolled alloys having a deformation microstructure. These particles act effectively as a pinning point of grain boundary migration. As a result, the recrystallization temperature near the specimen surface is improved above 1873 K.

The aim of this study is to investigate the effectiveness of the multi-step internal nitriding for the improvement in the resistance to irradiation. It is expected that the multi-step internal nitriding introduces a large number of sinks for irradiation-induced defects to Mo-Ti alloys.

2. Experimental

Mo-0.5 wt%Ti alloy with a thickness of 1mm was used as the starting material. Rectangular specimens with dimensions of $2.0^{w}\times20^{l}\times1.0^{t}$ mm were cut out from the sheet in parallel to rolling direction, and mechanical and electrolytic polishing were carried out.

Nitriding was performed at 1173 K-100 h, 1273 K-25 h, 1473 K-25 h and 1773 K-25 h in flowing N_2 at 1 atm. Neutron irradiation was performed in the Japan Materials Testing Rector (JMTR). The details of irradiation conditions are shown in Table 1. Impact three-point bending tests were preformed in order to investigate the influence of irradiation embrittlement on the ductile-brittle transition temperature (DBTT) of nitrided Mo alloys. The microstructure was observed through a transmission electron microscope (TEM) in order to study the distribution of irradiation-induced defects.

Table 1. Irradiation conditions

Irradiation temperature (K)	Fluence (n/m^2)	Irradiation temperature (K)	Fluence (n/m^2)
563	1.0×10^{24}	673	9.0×10^{23}
563	1.4×10^{24}	873	7.0×10^{24}
573 ↔ 773 with 5 cycles	9.0×10^{23}	1073	7.0×10^{24}

3. Results and Discussion

Fig. 1 shows the test temperature dependence of absorbed energy before and after irradiation for nitrided Mo-0.5Ti alloy. In this study, DBTT was defined as the temperature which absorbed energy became almost zero with decreasing test temperature. Fig. 2 shows DBTT shifts (ΔDBTT) derived from Fig. 1 and Vickers micro hardness increments (ΔHv) after irradiation. Strong correlation was recognized between ΔDBTT and ΔHv. ΔDBTT increased with irradiation temperature, reaching a maximum at 873 K. Kurishita et al. showed that ΔDBTT of as-rolled TZM alloy (Mo-0.5%Ti-0.08%Zr-0.03%C) irradiated at a fluence of 8.0×10^{23} n/m^2 at cyclic temperatures between 573 and 773 K was 160 K.[3] This is 40 K higher than ΔDBTT of nitrided Mo-0.5Ti alloy irradiated under the almost same condition (8.0×10^{23} n/m^2, 573 ↔ 773 K). These results suggest that multi-step internal nitriding is effective to improve the resistance to irradiation embrittlement.

Fig. 3 shows TEM images before and after irradiation. Irradiation-induced defects were clearly observed after irradiation, while only TiN particles were observed before irradiation. Types of defects induced at 563 and 873 K were black dot (dislocation loop) and void, respectively. Both defects were recognized in the specimen irradiated at cyclic temperature between 573 and 773 K. The size of void in the specimen irradiated at 873 K was larger than that in the specimen irradiated cyclically. It is notable that most defects exist around TiN particles in the case of the specimen irradiated at 563 K. This result suggests that the interface between TiN particle and molybdenum matrix acts as a sink for irradiation-induced defects.

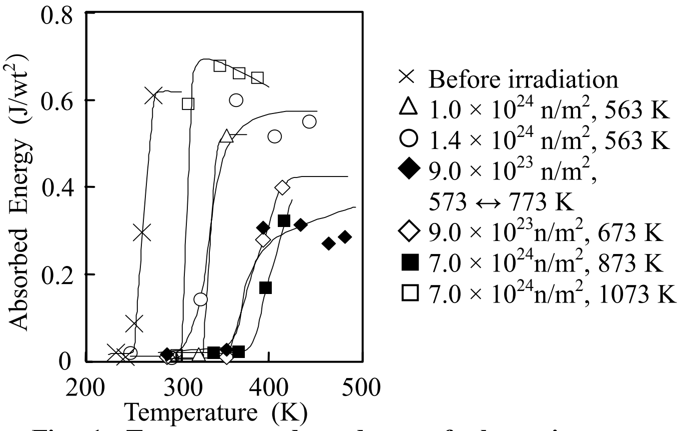

Fig. 1. Temperature dependence of absorption energy before and after irradiation for nitrided Mo- 0.5Ti alloys.

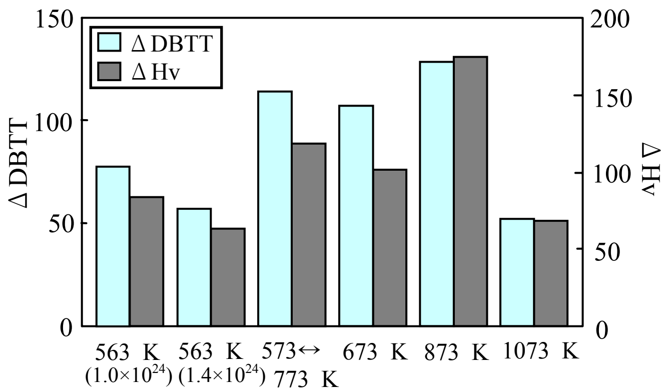

Fig. 2 The DBTT shifts and Vickers microhardness increments after irradiation for nitrided Mo-0.5Ti alloys.

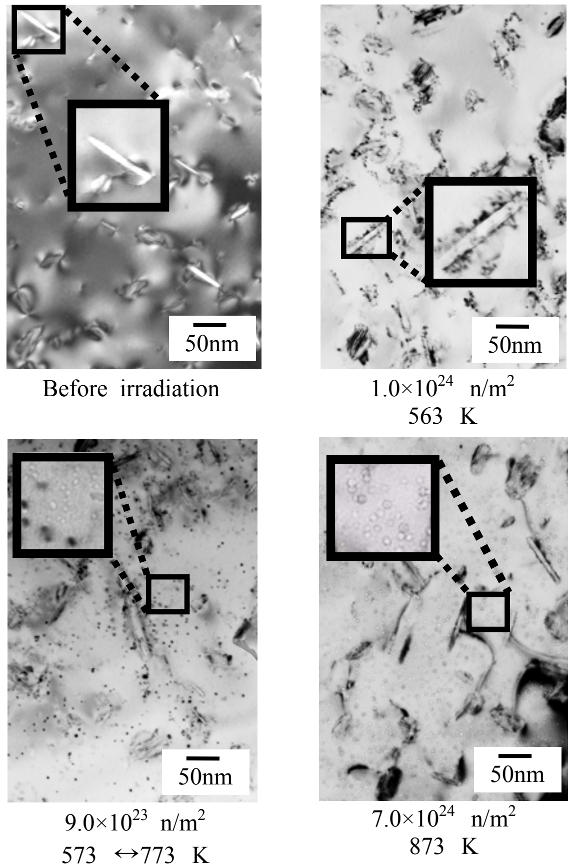

Fig. 3. TEM images before and after irradiation for nitrided Mo-0.5Ti alloys.

3. Summary

ΔDBTT after irradiation for nitrided Mo-0.5%Ti alloy was smaller than that of irradiated TZM alloy. Possibility that the interface between TiN particle dispersed by multi-step internal nitriding and molybdenum matrix acts as a sink for irradiation-induced defects was found out. Thus, it is possible to bring the resistance to irradiation embrittlement to as-rolled Mo-Ti alloy by multi-step internal nitriding technique.

4. References

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