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Neutron irradiation embrittlement of molybdenum rhenium alloys and their improvement by heat treatment

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Abstract

Irradiation-induced embrittlement is one of the major problems with molybdenum alloys, which have been considered as one of the candidates for divertor structural materials. The effects of rhenium content, heat-treatment and irradiation condition on mechanical properties and microstructural development of molybdenum rhenium alloys were studied after neutron exposure by FFTF/MOTA up to high fluence ($\sim 1 \times 10^{27}$ n/m^2, En>0.1 MeV). Appreciable plastic deformation was observed in a bending test on stress-relieved Mo–5 wt% Re irradiated at high temperatures. Fine, dense precipitates were observed in Mo–41 wt% Re, resulting in large hardening and embrittlement. © 1998 Elsevier Science B.V. All rights reserved.

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1. Introduction

The divertor structures of fusion reactors must withstand severe operating conditions such as high heat fluxes and high-flux neutron irradiation [1]. High-Z materials such as molybdenum and its alloys have been considered as one of the candidates for divertor structural materials [1–3]. Considering the application of high-Z materials under irradiation conditions, irradiation-induced embrittlement is one of the important issues for Mo alloys. Mo–Re alloys were developed to improve the low-temperature ductility [4] and weldability [5] of Mo, but the mechanical property data of neutron irradiated Mo–Re alloys including irradiation hardening and ductility are limited to low fluence levels [6–9]. We earlier reported on the tensile properties of Mo–5 wt% Re [10] after neutron bombardment (6.8–34 dpa) in the temperature range of 646–1073 K. Irradiation embrittlement was observed after lower temperature irradiation, but suppression of embrittlement at higher irradiation temperature by 5% Re addition and heat-treatment was observed.

In the Mo–Re system, Mo–Re alloys are single phase alloys up to 43% Re at 1423 K [11] and Mo–41% Re showed higher strength and relatively ductile behavior compared to that of Mo–5% Re in the unirradiated conditions [12]. On the other hand, irradiation induced precipitation was reported in higher Re containing alloys [13,18]. These precipitates are expected to influence mechanical properties of Mo–Re alloys.

In order to obtain some idea of the optimum Re concentration in Mo–Re alloys under neutron irradiation conditions, the effects of Re content (5% and 41% only), heat-treatment and irradiation condition on mechanical properties after neutron exposure up to high fluences ($\sim 1 \times 10^{27}$ n/m^2, En > 0.1 MeV) were studied in this work.

2. Experimental

Disks of 3 mm diameter with 0.25 mm thickness were prepared from powder metallurgical Mo–5 wt% Re and Mo–41 wt% Re alloy sheets, which were supplied from Metallwerk Plansee. The chemical compositions of the specimens are given in Table 1. These specimens were stress relieved at 1198 K for 15 min. Recrystallization treatment of Mo–5Re was carried out at 1473 K for 1 h in vacuum, and that of Mo–41Re at 1573 K for 1 h. The grain sizes of the stress-relieved and recrystallized materials were about 2 and 20 μm, respectively.

Neutron irradiation was performed in helium-filled stainless steel capsules in the Materials Open Test

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Table 1
Chemical analysis of Mo–Re alloys

Alloy	Mo	Re (wt%)	C (wt.ppm)	O	N
Mo–5 wt% Re	Bal.	4.9	3	7	3
Mo–41 wt% Re	Bal.	41.2	14	6	3

Table 2
Irradiation conditions [14,15]

Temperature [K]	646	679	792	873	1073
Fluence (En > 0.1 MeV) [$\times 10^{26}$ m^{-2}]	1.97	7.47	9.46	9.46	3.19
Fluence [dpa] [a]	6.8	27	34	34	11

[a] Calculated with SPECTER code

Assembly (MOTA) of the Fast Flux Test Facility (FFTF). The irradiation vehicle was MOTA-2A in FFTF cycle 11. Table 2 gives the irradiation conditions [14] and damage levels which were converted [15] from Fe–18Cr–8Ni to Mo by using the SPECTER code. The exposure fluence and irradiation temperature of each capsule depended on its position in the reactor.

Irradiation hardening was estimated by measuring Vickers microhardness at room temperature with the load of 1.96 N for 20 s. In order to estimate ductility of both unirradiated and irradiated specimens, miniature three-point bend tests were performed at room temperature using an Instron type machine with the cross head speed of 0.2 mm/min [20]. After the bend test, fracture surfaces were observed by scanning electron microscopy. Microstructures of the irradiated specimens were observed with a transmission electron microscope at 200 kV after electropolishing.

3. Results and discussion

3.1. Irradiation hardening

Fig. 1 shows the hardness increase by irradiation in the two Mo–Re alloys in various irradiation conditions. In Mo–5Re, irradiation hardening of the stress-relieved specimens is smaller than that of the recrystallized ones. The hardening decreased gradually with increasing irradiation temperature up to 873 K, and a large decrease of irradiation hardening was observed at 1073 K.

The irradiation hardening of Mo–41Re was remarkably larger than that of Mo–5Re. The hardness of recrystallized and stress-relieved specimens after irradiation at and below 873 K was in the range of 1300–1800. The temperature dependence of the irradiation hardening of Mo–41Re is not as clear as that of Mo–5Re, but hardening of Mo–41Re irradiated at 1073 K was remarkably smaller than at lower temperatures. Irradiation hardening of stress-relieved specimens was smaller than that of the recrystallized ones.

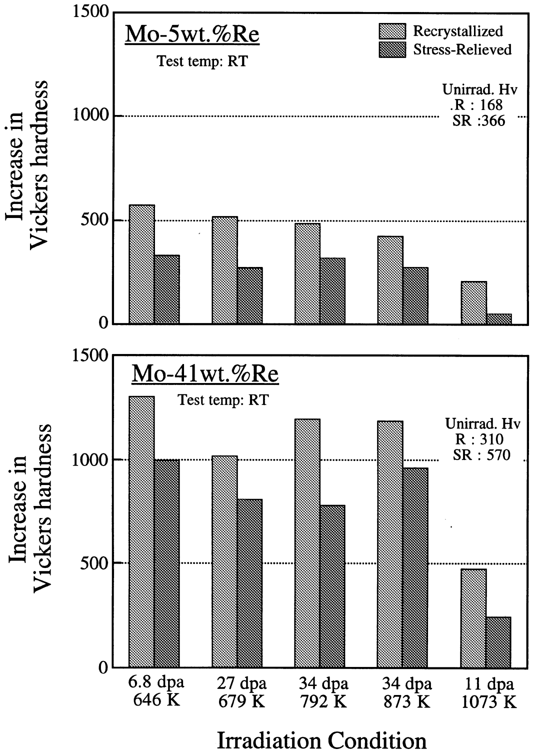

Fig. 1. Increase in Vickers hardness of Mo–5 wt% Re and Mo–41 wt% Re alloys after neutron irradiation at FFTF/MOTA-2A.

3.2. Bend deflection

Fig. 2 shows the results of plastic bend deflection in the two alloys irradiated in various conditions. In the unirradiated conditions, both stress-relived and recrystallized specimens showed large ductility, that is, plastic deflections of these specimens were above 1 mm and these specimens did not rupture during this bending test. After neutron irradiation, relatively large plastic deflections of Mo–5Re were observed in stress-relieved materials irradiated at 1073 K, and appreciable deflection was observed even in recrystallized materials irradiated at 1073 K. On the other hand, no measurable plastic deflection was observed in irradiated Mo–41Re at any irradiation condition. These specimens ruptured in the elastic regions.

3.3. SEM fractographs

Fig. 3 shows typical fractographs of the two irradiated alloys in the recrystallized and stress-relieved conditions. Cleavage fracture was observed in the recrystallized Mo–5Re and 41Re alloys, whereas, a

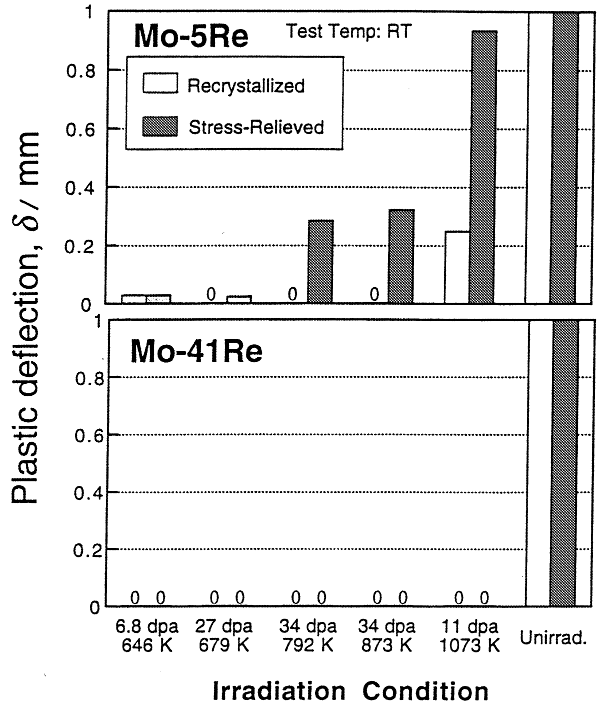

Fig. 2. Plastic deflection of bend test of Mo–5 wt% Re and Mo–41 wt% Re alloys after neutron irradiation at FFTF/MOTA-2A.

layered structure and partly cleavage fracture were observed in the relatively ductile stress-relieved Mo–5Re alloy. The layered structure, which might be induced by the rolling process before the stress-relief treatment, was also observed in stress-relieved Mo–41Re alloys. Cracks along the horizontal direction of the pictures are debonding cracks along the layers. Grain boundary fracture surface was observed in the recrystallized and irradiated specimens, but the grain boundary fracture was not the dominant fracture mode of these specimens because the fraction of grain boundary was very low and isolated on the rupture surface. In the case of stress-relieved Mo–5Re irradiated at 1073 K, the specimen was fully bent and did not rupture.

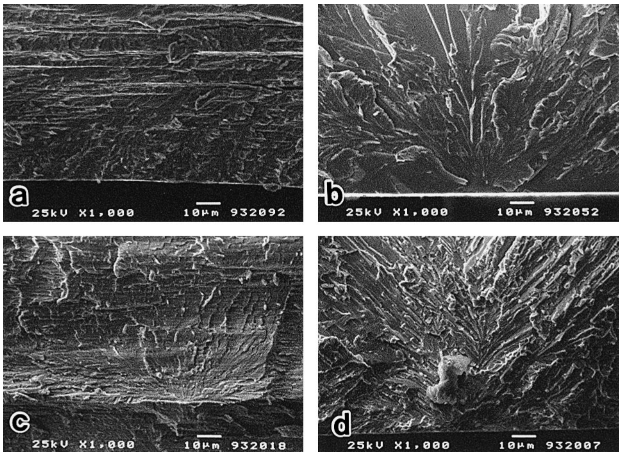

Fig. 3. SEM micrographs in recrystallized Mo–5 wt% Re and Mo–41 wt% Re alloys bend-tested at room temperature after neutron irradiation at FFTF/MOTA-2A. Irradiation conditions are: (a) Mo–5Re/Stress Relieved, 679 K/27 dpa; (b) Mo–5Re/Recrystallized, 792 K/34 dpa; (c) Mo–41Re/Stress Relieved, 873 K 34 dpa; (d) Mo–41Re/Recrystallized, 646 K/6.8 dpa.

3.4. TEM microstructures

Fig. 4 shows TEM microstructures of the two recrystallized alloys irradiated at various conditions. It is noted that various precipitates were observed after irradiation. These precipitates had not been observed before irradiation. In Mo–5Re, voids were observed in all the irradiated conditions but dislocation loops were observed at 646, 679 and 792 K. Small, fine rectangular precipitates were observed in Mo–5Re when irradiated at and above 792 K. The images of the precipitates becomes clearer with increasing irradiation temperature.

The precipitation behavior of Mo–41Re was more complicated than that of Mo–5Re. The microstructure of irradiated Mo–41Re can be divided into three categories as follows; (1) relatively large needle or thin plate precipitates at 646 K, (2) fine needle or rectangular type precipitates at 679, 792 and 873 K, (3) large block and plate type precipitates at 1073 K. Such precipitates were earlier reported in Mo–41Re [18]. A denuded zone of precipitates near grain boundary was clearly observed at 1073 K. Voids were observed at all irradiation temperatures, but their size was small and the number density was much lower than that observed in Mo–5Re.

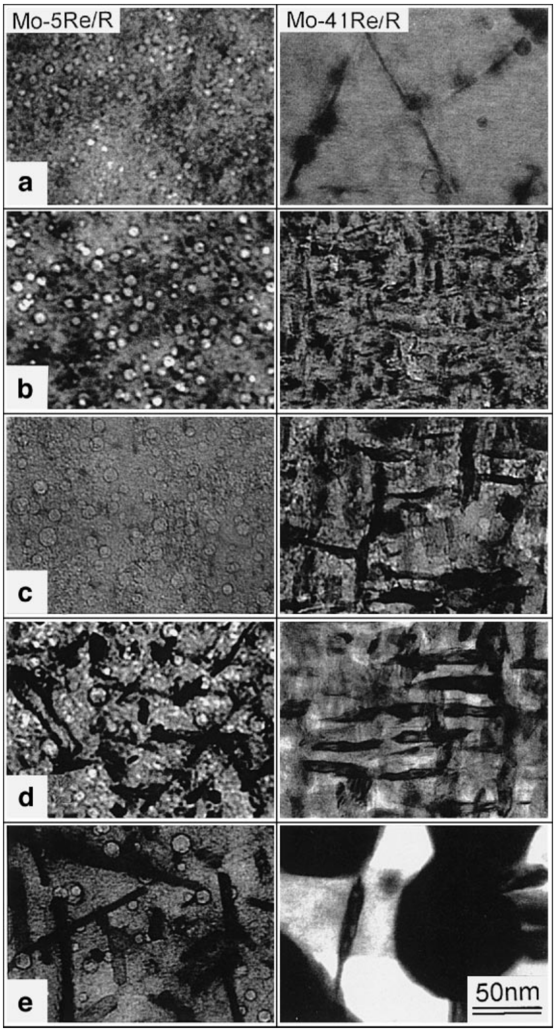

Fig. 4. TEM micrographs of recrystallized Mo–5 wt% Re and Mo–41Re alloys after neutron irradiation at FFTF/MOTA-2A. Irradiation conditions are: (a) 646 K/6.8 dpa; (b) 679 K/27 dpa; (c) 792 K/34 dpa; (d) 873 K/34 dpa; (e) 1073 K/11 dpa.

3.5. Effects of precipitation on hardening

The hardness and bending properties of irradiated Mo–Re alloys strongly depend on the Re content and the irradiation temperature. In Mo–41Re alloy, irradiation hardening in both heat-treatment conditions is remarkably higher compared to that of Mo–5Re, Mo [16] and TZM [17].

Quantitative analysis of these defect distributions was not carried out yet, but hardening mechanism can be summarized as follows: (1) Mo–5Re/646,679 K: void and dislocation loop, (2) Mo–5Re/792,873,1073 K: void and precipitate-1, (3) Mo–41Re/646 K: void and precipitate-2, (4) Mo–41Re/679,792,873: precipitate-3 and void, (5) Mo–41Re/1073 K: precipitate-4 and void. Identification of these various precipitates was not carried out in this study. However, the precipitate type can be presumed by previous works of irradiation assisted precipitation of Mo–41Re [18] and W-5,11 and 25Re [19]. Based on those earlier studies, precipitate-1 and precipitate-3 may be sigma phase (MoRe) and precipitate-4 may be chi phase ($MoRe_3$).

In the case of precipitate-2, morphology of the precipitate has not been reported previously, but it may be sigma phase. In spite of the higher hardening of Mo–41Re irradiated at 646 K, the defect density was somewhat lower than that of other irradiated specimens. Therefore, some additional invisible small defect cluster which contributed to the hardening may be postulated.

Fractographs after the bending test showed that a grain boundary fracture mode was not dominant in this work. This result means that the grain boundary strengthening effect by Re addition was effective even after irradiation-induced precipitation. From the point of view of suppression of irradiation embrittlement of Mo alloys, a 41% Re addition is too large to maintain ductility of the matrix. A lower amount of Re addition appears to adequate to propose the use of Mo–Re alloys for irradiation environments.

4. Summary

The effects of Re content and heat-treatment on hardness and bend properties of Mo–Re alloys after neutron irradiation were examined.

1. Appreciable plastic deformation was observed in a bending test in stress-relieved Mo–5 wt% Re alloy irradiated at high temperatures.
2. Fine, dense precipitates were observed in Mo–41 wt% Re, resulting in large hardening and embrittlement.
3. The irradiation embrittlement of Mo can be improved by 5 wt% Re addition on stress-relief in certain irradiation conditions, but higher Re contents may cause much higher irradiation hardening and embrittlement by precipitation.
4. Further optimizing Re content is required before this alloy series can be considered for divertor applications.

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