

# Microstructural development and radiation hardening of neutron irradiated Mo–Re alloys

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## Abstract

Stress-relieved specimens and recrystallized specimens of pure Mo and Mo–Re alloys with Re contents of 2, 4, 5, 10, 13 and 41 wt% were neutron irradiated up to 20 dpa at temperatures from 681 to 1072 K. On microstructural observation, sigma phase and chi phase precipitates were found in all irradiated Mo–Re alloys. Voids were observed in all irradiated specimens, and dislocation loops and dislocations were observed in the specimens that were irradiated at lower temperatures. On Vickers hardness testing, all of the irradiated specimens showed hardening. Especially Mo–41Re were drastically embrittled after irradiation at 874 K or below. From these results, the authors discuss about the relation between microstructure development and radiation hardening and embrittlement, and propose the optimum Re content and thermal treatment for Mo–Re alloys to be used under irradiation conditions.

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## 1. Introduction

Refractory metals such as molybdenum (Mo) and tungsten (W) are considered as candidates for high heat flux materials in fusion reactor systems, because of their high melting point, high thermal conductivity and high resistance to sputtering yield, etc. [1–6]. To improve the mechanical properties at high temperatures, rhenium (Re) addition to Mo and W was proposed [7–14]. And it was shown that the Re addition increases the recrystallization temperature of these materials [7] and controls

recrystallization embrittlement. Moreover, the Re addition improve the ductility [7,9] and decreases the ductile–brittle transition temperature (DBTT) [8,10].

The main concern of using Mo under irradiation conditions is radiation embrittlement, which is caused by embrittlement of grain boundaries due to segregation of impure interstitial elements such as oxygen (O) and by hardening of crystal grains. In previous studies, to improve the resistance to radiation embrittlement, controlling the crystal grain size was found to be effective [15]. Re doping was also found effective to control radiation embrittlement because of the strengthening of grain boundaries and the increase of recrystallization temperature under irradiation conditions [16–18]. On the other hand, equilibrium Re content in Mo–Re alloy is about 41 wt% at room temperature and it is higher at higher temperatures, but radiation induced precipitates

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were observed even in the alloys containing 5 wt% Re [17,18]. However, no systematic studies about radiation induced precipitation and radiation hardening have ever been performed.

The influence of neutron irradiation on Mo–Re alloys was studied in terms of radiation induced embrittlement, hardening, microstructural development and transmutation, etc. [6,16–18]. Hasegawa et al. studied about radiation embrittlement of Mo, Mo–5Re and Mo–41Re [17,18], and reported drastic embrittlement of Mo–41Re after neutron irradiation. In Mo–5Re, embrittlement was significant in the recrystallized specimens, while some ductility was remained in the stress-relieved specimens after irradiation. In the microstructural observation, radiation induced precipitation was confirmed, and it was increased with increasing Re content.

These previous studies indicate that radiation embrittlement and hardening of Mo–Re alloys should be discussed with radiation induced precipitation. However, radiation induced precipitation has not been studied systematically from the viewpoints of Re content, thermal treatment and irradiation temperature and fluence. And there are only a few studies about the irradiation over 1 dpa, controlling irradiation temperature or other conditions properly.

The aim of this work is to define the influence of Re content, thermal treatment and irradiation temperature on radiation induced precipitation and hardening of Mo–Re alloys, and to propose the suitable Re content and thermal treatment for the Mo–Re alloys used under irradiation condition.

## 2. Experimental

### 2.1. Specimen preparation

Plates of pure Mo and Mo–Re alloys (Re content = 2, 4, 5, 10, 13, 41 wt%) made by powder metallurgy were hot worked and cold worked to 0.25 mm thickness sheets: pure Mo, Mo–5Re and Mo–41Re were supplied by Metallwerk Plansee and Mo–2Re, Mo–4Re, Mo–10Re and Mo–13Re by Allied Material Corp. Chemical compositions of the materials are listed in Table 1. These sheets were cut into 2 mm × 12 mm size, and thermally

Table 1  
Chemical composition

Alloy	Mo	Re (wt%)	C (wt ppm)	O	N
Pure Mo	Bal.	–	30	50	10
Mo–2Re	Bal.	2	12	23	4
Mo–4Re	Bal.	4	7	15	<3
Mo–5Re	Bal.	5	30	30	10
Mo–10Re	Bal.	10	4	14	<3
Mo–13Re	Bal.	13	7	20	<3
Mo–41Re	Bal.	41	30	30	10

Table 2  
Thermal treatment conditions

<i>Stress relief treatment</i>	
Mo, Mo–Re (2, 4, 5, 10, 13, 41 wt% Re)	1199 K/15 min
<i>Recrystallization treatment</i>	
Mo	1473 K/1 h
Mo–Re (2, 4, 10, 13 wt% Re)	1523 K/1 h
Mo–Re (5, 41 wt% Re)	1573 K/1 h

treated. Thermal treatment conditions are listed in Table 2. One is stress relief treatment, and the mean grain size was controlled to 2 μm. The other is recrystallization treatment, and the mean grain size was controlled to 20 μm. These thermal treatments were conducted in vacuum chamber at 10<sup>−3</sup> Pa.

### 2.2. Thermal aging experiment

The Re content in Mo–41Re is almost boundary to precipitate sigma phase in thermal equilibrium at a higher temperature [19]. To define the difference of precipitation under irradiation and thermal equilibrium, a thermal aging experiment was conducted. Recrystallized Mo–41Re was thermally aged in an evacuated and shielded quartz tube at 673, 873, and 1073 K for one month. These aging temperatures were chosen to be close to neutron irradiation temperatures.

### 2.3. Irradiation condition

Neutron irradiation was conducted in the Material Open Test Assembly in the Fast Flux Test Facility (FFTF/MOTA-2B). The specimens were irradiated in helium filled capsules, placed near mid plain of the reactor. Irradiation conditions are listed in Table 3. The radiation damages in the table were converted to Mo from the result calculated for Iron using SPECTOR code [20]. For this conversion the threshold energy for displacement of Mo atom was assumed as 60 eV [21]. After neutron irradiation, the specimens were cut into 2 mm × 2 mm size.

### 2.4. Postirradiation experiment

(1) *Microstructural observation and chemical analysis:*  
For microstructural observation using transmission

Table 3  
Irradiation conditions [20,21]

Temperature (K)	681 ± 5	792 ± 5	874 ± 5	1072 ± 5
Fluence ( $E_n > 0.1$ MeV) [ $\times 10^{26}$ n/m <sup>2</sup> ]	5.1	5.9	5.9	5.2
Radiation damage (dpa) <sup>a</sup>	18	21	21	18

<sup>a</sup> Calculated with SPECTER code.

electron microscope (TEM), each specimen was cut into 2 mm×2 mm size, and electro-polished to a thin film with 25% sulfuric acid and 75% ethyl alcohol mixed liquid, at 55 V/0.8 A at a temperature of about 273 K.

TEM observation was carried out with JEOL/JEM2010, and chemical analysis using energy disperse X-ray spectrometer (EDS) was carried out with JEOL/2010F (Field Emission TEM) at 200 kV.

(2) *X-ray diffraction analysis*: To analyze the lattice parameter of the precipitates observed by TEM, X-ray diffraction analysis was carried out at 250 mA/30 kV using JEOL/JDX-3500.

(3) *Vickers hardness measurement*: The Vickers hardness measurement was conducted with a 1.96 N load for 20 s at ambient temperature. Five points on each specimen were indented, and the average of middle three data was adopted as Vickers hardness value.

### 3. Results

#### 3.1. Microstructural observation

Microstructural observation was conducted on the recrystallized specimens irradiated at 681, 792, 874 and 1072 K and on the stress-relieved specimens irradiated at 1072 K. Fig. 1 is the micrographs of the recrystallized Mo–Re alloys after neutron irradiation at 1072 K. Voids

were observed in all irradiated specimens. Void lattice was observed in two specimens; one was the stress-relieved pure Mo irradiated at 1072 K, and the other was the recrystallized pure Mo irradiated at 874 K. A micrograph of the void lattice in the latter specimen is presented in Fig. 2.

Platelet type precipitates and equiaxed type precipitates were observed in all Mo–Re specimens after neu-

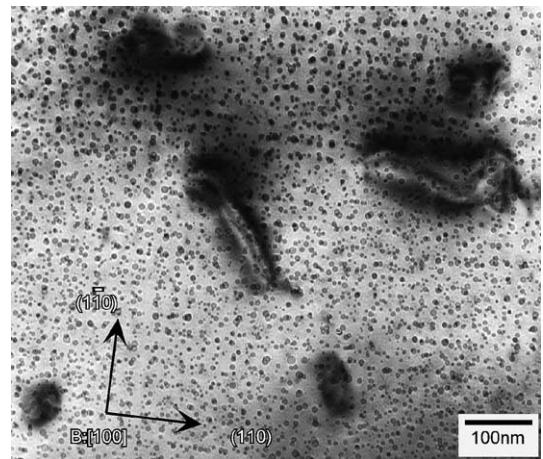


Fig. 2. Micrograph of recrystallized Mo irradiated at 874 K up to 21 dpa.

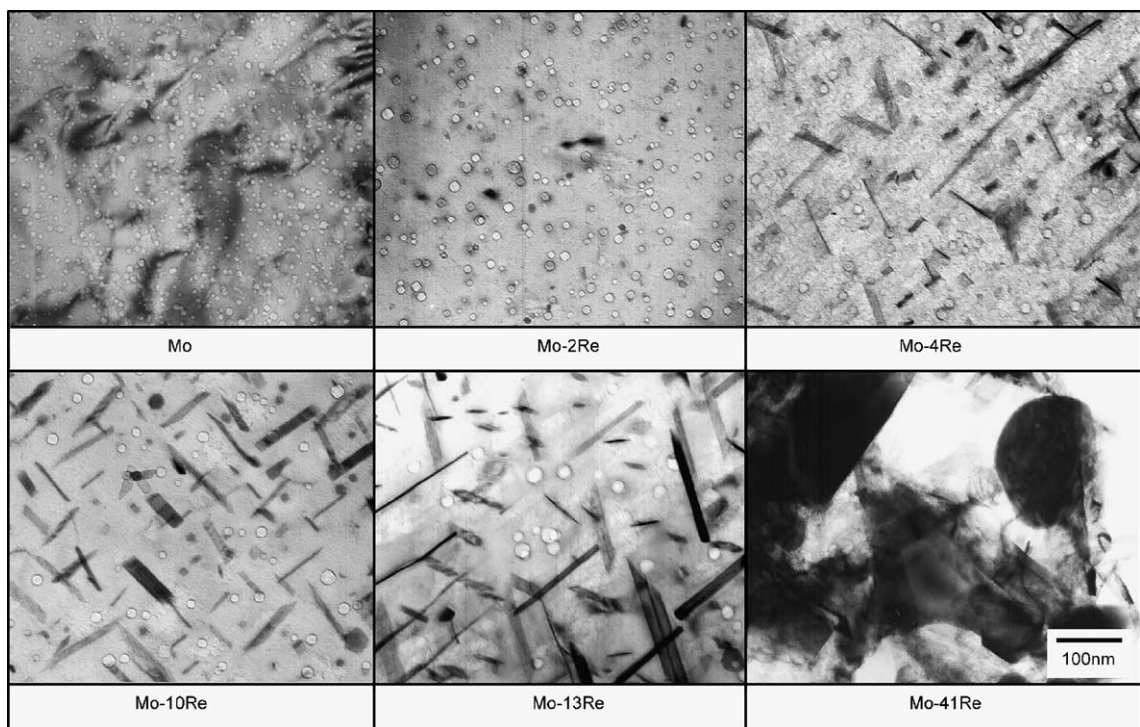


Fig. 1. Micrographs of recrystallized Mo–Re alloys irradiated at 1072 K up to 18 dpa.

tron irradiation. These precipitates were not observed on grain boundaries. On the other hand, in the Mo–41Re specimens thermally aged at the same temperature as the irradiation temperature, no evidence of the existence of precipitates was found by TEM and X-ray diffraction method. From these results, it appears that the precipitates observed in the irradiated specimens may be radiation induced precipitates.

The shapes of these precipitates were similar to those of the precipitates observed in irradiated W–Re specimens in our previous work [22]. The electron diffraction patterns from the precipitates in Mo–Re alloys were similar to those in W–Re alloys. Hence, the platelet type precipitates would be chi phase with a  $\alpha$ -Mn structure, and the equiaxed type precipitates would be sigma phase with a CrFe structure. A schematic illustration is shown in Fig. 3. The lattice constants of the sigma phase were determined to be  $a = 0.961$  nm and  $c = 0.499$  nm, and that of the chi phase was  $a = 0.960$  nm. The lattice constant of the sigma phase is similar to that of sigma phase formed in thermally aged Mo–Re alloys [19].

The results of EDS analysis of the recrystallized Mo–10Re irradiated at 874 K up to 21 dpa are listed in Table 4. Osmium (Os) transmuted from Re was detected more in the sigma phase precipitates than in the matrix. The ratio of Os to Re was 0.22 in the matrix and 0.25 in the sigma phase precipitates.

Dislocation loops were observed in the recrystallized pure Mo, Mo–4Re and Mo–10Re irradiated at 681 K up to 18 dpa and in the recrystallized pure Mo irradiated at 874 K up to 21 dpa. A micrograph of the recrystallized pure Mo irradiated at 681 K up to 18 dpa is presented in Fig. 4. These dislocation loops were all of interstitial type.

### 3.2. Dependence of microstructural development on thermal treatment, Re content and irradiation temperature

Fig. 5 presents the dependence of mean size and number density of the voids on Re content at each ir-

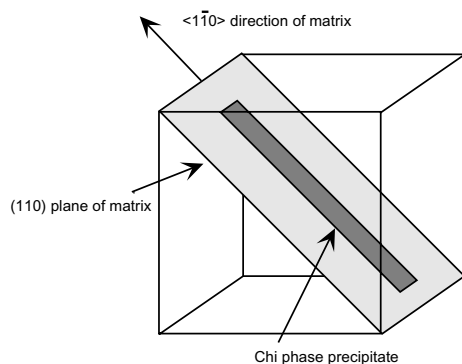


Fig. 3. Arrangement of chi phase precipitate on matrix.

Table 4  
Results of EDS analysis for recrystallized Mo–10Re irradiated at 874 K/21 dpa

	Mo	Re	Os (wt%)	Os/Re
Matrix	89	9	2	0.22
Sigma phase	27	59	15	0.25

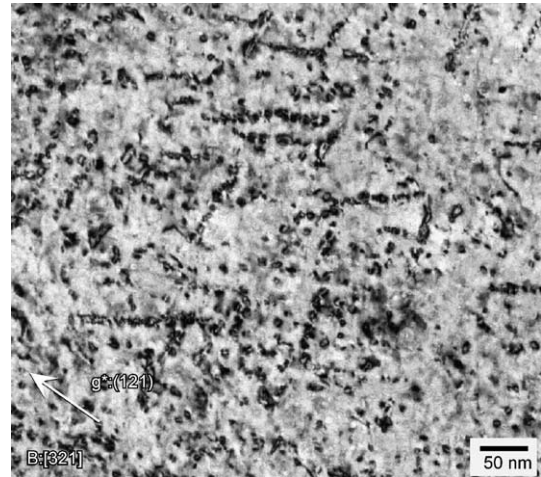


Fig. 4. Micrograph of recrystallized Mo irradiated at 681 K up to 18 dpa (dislocation image).

radiation temperature. The mean size increased and the density decreased with increasing Re content and irradiation temperature. Thermal treatment does not appear to affect the void size and density. Void swelling was about 0.5–1.5% in the alloys with the Re contents of 13 wt% or less and about 0.1% in Mo–41Re.

Fig. 6 shows the mean length of the long axis of the platelet type chi phase precipitates and the number density, which depend on Re content at each irradiation temperature. The mean length increased and the density decreased with increasing Re content, and the trends became steep with increasing irradiation temperature. The volume fraction of the chi phase precipitates was calculated on the assumption that the thickness is 1 nm for each precipitate, because the chi phase precipitates were observed to be very thin, so they may include only one or two crystal planes. The calculated volume fractions were below 0.5% in each specimen, and there were no significant differences. Thermal treatment did not show any effect on the mean length, the number density and the volume fraction of the chi phase precipitates.

Fig. 7 shows the Re content dependence of mean size and number density of the equiaxed type sigma phase precipitates at each irradiation temperature. The mean size increased and the number density decreased with increasing Re content and irradiation temperature. The

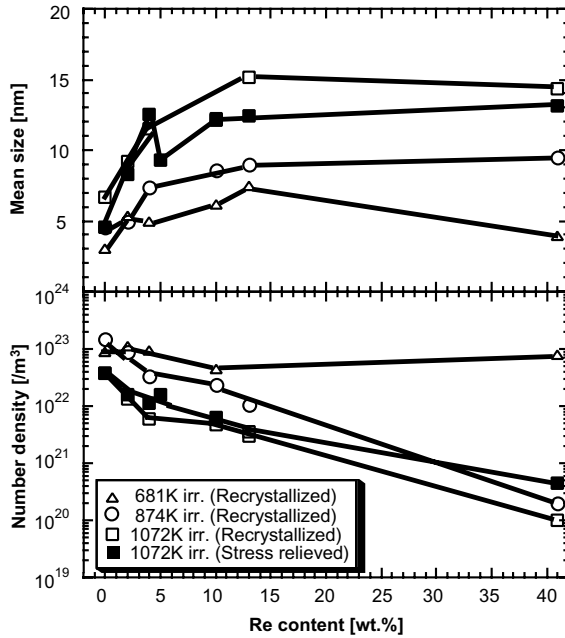


Fig. 5. Effect of Re content on the mean size and number density of voids.

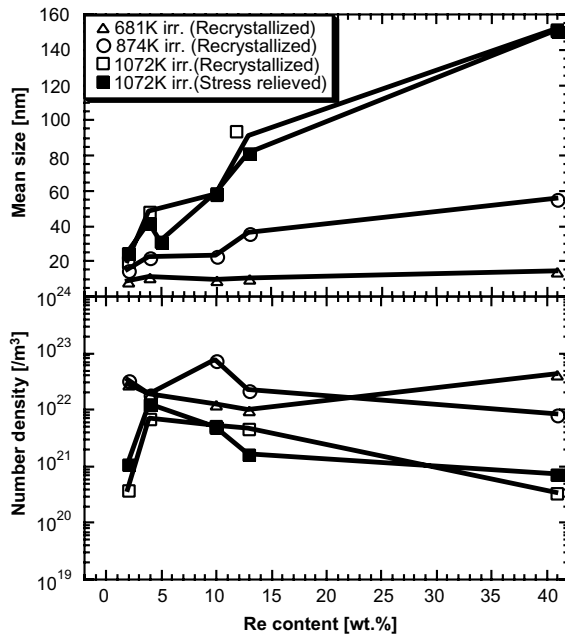


Fig. 6. Effect of Re content on the mean size and number density of platelet chi phase precipitates.

mean size of the sigma phase precipitates was larger in the recrystallized specimens than in the stress-relieved specimens after irradiation at 1072 K. Especially in Mo–

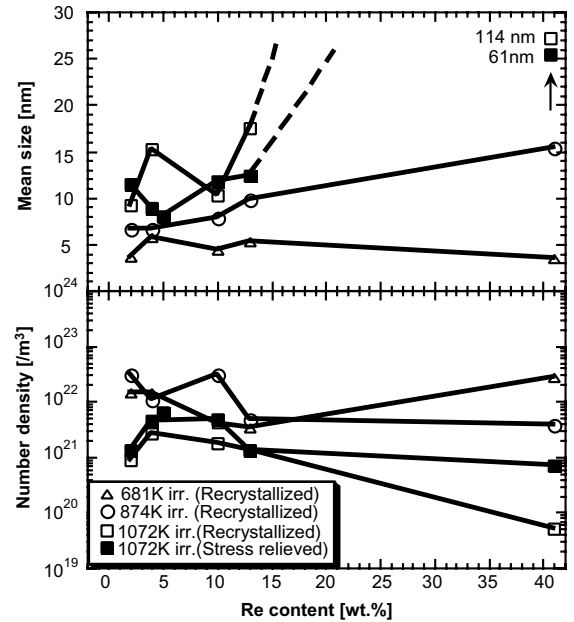


Fig. 7. Effect of Re content on the mean size and number density of equiaxed sigma phase precipitates.

41Re irradiated at 1072 K, the mean size was about 114 nm for the recrystallized specimens and about 61 nm for the stress-relieved specimens. The volume fraction of the sigma phase precipitates was almost below 1% and no significant difference was observed between specimens, when the Re content was under 13 wt%. However, in Mo–41Re irradiated at 1072 K, the sigma phase precipitates grew extremely large, and their volume fractions in the stress-relieved and recrystallized Mo–41Re were about 19% and 26% respectively.

Fig. 8 shows the effect of Re content on the mean size and number density of the dislocation loops in the recrystallized specimens. After irradiation at 681 K, the number density of dislocation loops increased with increasing Re content. In pure Mo, the mean size increased and the number density decreased with increasing irradiation temperature. The density of dislocations, observed in the recrystallized pure Mo irradiated at 874 K, was about  $7.0 \times 10^{13} \text{ m}^{-2}$ .

### 3.3. Radiation hardening

Fig. 9 shows the Vickers hardness of irradiated and unirradiated specimens as a function of Re content at each irradiation temperature. Fig. 10 shows radiation hardening, estimated from the results in Fig. 9. Every specimen was hardened by irradiation. Especially in Mo–41Re irradiated at 874 K or below, severe hardening and embrittlement occurred, and cracks were observed around the indentations after the hardness

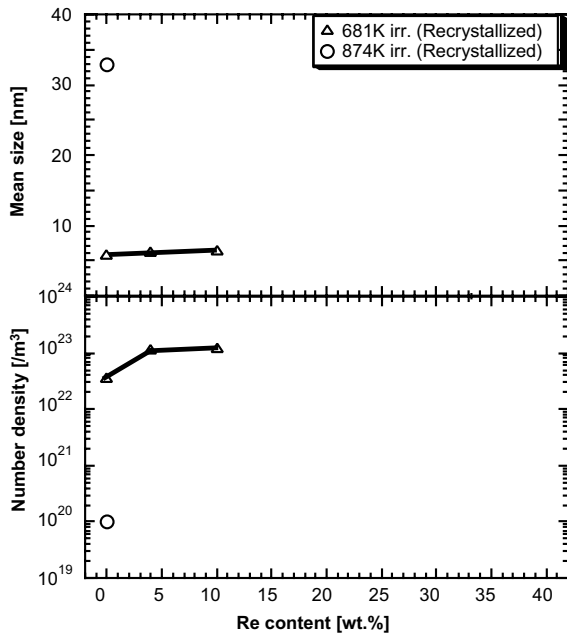


Fig. 8. Effect of Re content on the mean size and number density of dislocation loops.

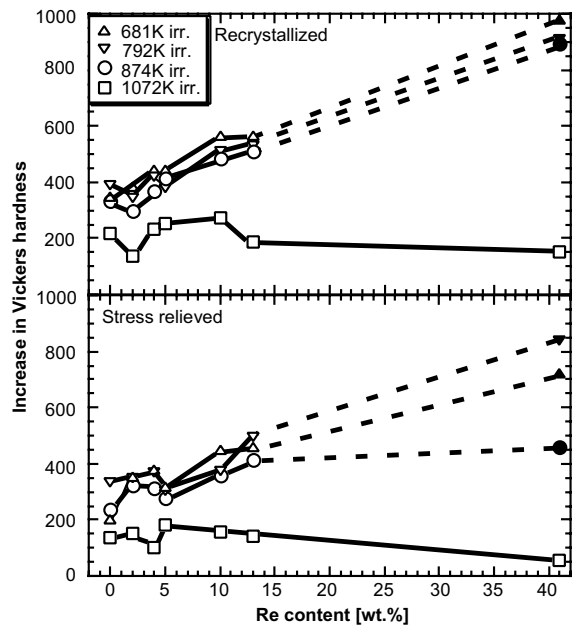


Fig. 10. Effect of Re content on radiation hardening.

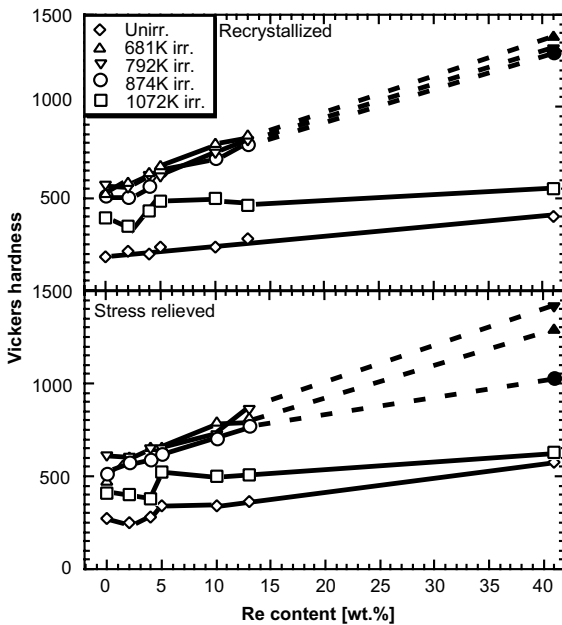


Fig. 9. Effect of Re content on Vickers hardness before and after irradiation. Around the indentation of Mo-41Re irradiated at 874 K or below, cracks were observed.

measurement. Radiation hardening was increased with Re content. Radiation hardening at 874 K or below was

not changed with the temperature obviously, and at 1072 K the hardening was decreased. The hardening of the recrystallized specimens was slightly larger than that for the stress-relieved specimens.

## 4. Discussion

### 4.1. Radiation induced precipitation

The mean size and the number density of the sigma phase precipitate observed in Mo-41Re were significantly changed with thermal treatment before irradiation. The stress-relieved specimens had smaller and denser sigma phase precipitates than the recrystallized specimens. From this result, the nucleation sites of the sigma phase precipitates are considered to be subgrain boundaries consisting of tangled dislocations, because the number density of subgrains is changed with thermal treatment. Consequently, it is considered that the number density of the sigma phase precipitates can be controlled by thermal treatment. On the other hand, the mean length and the number density of the chi phase precipitates were not changed with thermal treatment.

This result suggests that the nucleation sites of the chi phase precipitates are dislocations and dislocation loops formed at the beginning of irradiation (<1 dpa) [23], so that the density did not depend on thermal treatment condition. After nucleation of these sigma phase and chi phase precipitates, the surface of these precipitates acted

as a sink for under size elements such as Re atoms, leading to the growth of the precipitates.

In the result of EDS analysis, Os transmuted from Re was detected, and it was concentrated more in the sigma phase precipitates than in the matrix. The ratio of Os to Mo in the sigma phase precipitates was about 36 wt%, which is sufficient to form Mo–Os precipitates as shown in the Mo–Os phase diagram [24]. If adequate amounts of Os exist during precipitation, Mo–Os precipitates can be formed in addition to Mo–Re precipitates. In this work, however, there was no evidence of the existence of Mo–Os precipitates verifiable by electron diffraction patterns and X-ray diffraction. The ratio of Os to Re in the sigma phase precipitates is very similar to that in the matrix. From these results, it is considered that sigma phase precipitation was saturated earlier, and that most of Os was transmuted from Re after the saturation of precipitation.

Nelson et al. [25] gave a theoretical equation of saturate radius and number density of sphere shaped radiation induced gamma prime phase precipitates in nickel–aluminum alloy, and they confirmed it by experiment. To apply the equation to the present case for sigma phase precipitates in Mo–Re alloys, the equation is simplified as

$$R = (3/4\pi C_p)(C_t/n) - B, \quad (1)$$

where  $R$  is the saturate radius of precipitates,  $C_p$  the Re content in precipitates,  $C_t$  the Re content of alloy,  $n$  the number density of precipitates,  $B$  the constant consistent with irradiation condition. This equation is one dimensional if the Re content of the precipitates ( $C_p$ ) is constant:

$$R = A(C_t/n) - B, \quad (2)$$

where  $A$  is a constant. If the precipitation is saturated, this equation is satisfied. Fig. 11 shows the dependence of  $R$  on  $C_t/n$ , calculated from the data obtained by TEM observation. The marks indicating the results of 681 and 874 K irradiations vary almost linearly and satisfy the equation, indicating that the precipitation was saturated. The marks for the results of irradiation at 1072 K are more scattered than those at 681 and 874 K. It may be because chi phase precipitation changed the Re content in the matrix, resulting in a more complicated system than the equation. The Re content calculated from the slope of the line drawn on the marks of 874 K irradiation was about 57 wt%, which is similar to the result of EDS analysis in this work, 59 wt%.

#### 4.2. Radiation hardening and embrittlement

Radiation hardening was larger in the specimens irradiated at 681 or 874 K than at 1072 K. This is considered to be because the obstacles to dislocation motion

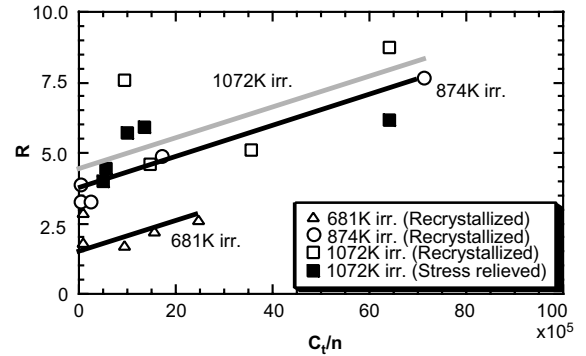


Fig. 11. The dependence of  $R$  (the mean radius of sigma phase precipitates) on  $C_t/n$  (Re content of alloy/density of sigma phase precipitates) calculated from the data of microstructural observation.

during slip deformation, such as precipitates, voids and dislocation loops were developed more densely in the specimens irradiated at lower temperature. At lower irradiation temperatures, radiation hardening increased with increasing Re content. It would be mainly because the number density of dislocation loops and the mean length of chi phase precipitates increased with Re content.

Orowan stress ( $\Delta\tau$ ) which is here used as the stress required to move dislocations between the obstacles observed in this study [26–28], is given by the following equation:

$$\Delta\tau = \sum (\alpha_n \mu b / \lambda_n). \quad (3)$$

Here  $\alpha_n$  is a proportionality constant for voids, dislocations, dislocation loops and precipitates. In this study,  $\alpha_n$  was assumed to be the same for each obstacle to make calculation easy.  $\mu$  is the modulus of rigidity and  $1.206 \times 10^5$  MPa at room temperature for Mo.  $b$  is the Burgers vector and 0.27 nm when the slip plane of Mo is (011) and the slip direction is  $a/2\langle 111 \rangle$ .  $\lambda_n$  is the mean distance of voids, dislocations, dislocation loops and precipitates on the slip plane, and was calculated from the results of microstructural observation. Hardening of materials that is evaluated from Vickers hardness ( $\Delta H_v$ ), yield stress ( $\Delta\sigma_{ys}$ ) and Orowan stress ( $\Delta\tau$ ) is related by the following equation:

$$\Delta H_v = C \Delta\sigma_{ys} = CM \Delta\tau = CM \alpha \mu b \sum (1/\lambda_n). \quad (4)$$

Here  $C$  and  $M$  are proportionality constants. The product of  $C$ ,  $M$  and  $\alpha$  was assumed to be 0.33 to fit results of the calculation and the Vickers hardness measurement.

Fig. 12 shows the Re content dependence of radiation hardening calculated from results of the microstructural observation. The calculated results agreed with the

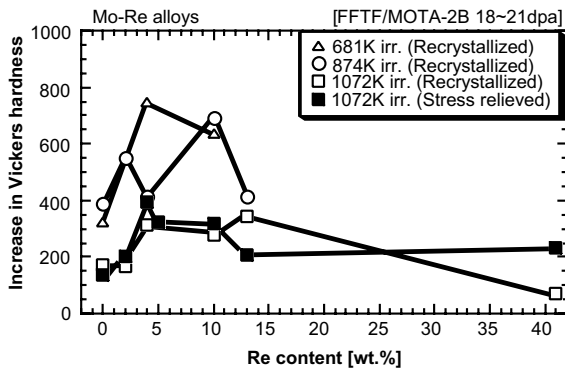


Fig. 12. Effect of Re content on the radiation hardening calculated from the data obtained by microstructural observation.

results of Vickers hardness measurement in the specimens irradiated at 1072 K. In the specimens irradiated at 681 or 874 K, the scatter of the calculated results was larger. It is considered because there would be invisible small defects in the specimen irradiated at lower temperatures. However, the hardening level and tendency were similar to the results of Vickers hardness test.

For Mo–41Re irradiated at 874 K or below, there were cracks observed around the indentation after Vickers hardness measurements. This embrittlement is thought to be caused by large and hard sigma phase precipitates. Because Vickers hardness of the sigma phase precipitates is about 1500 [12], which is much larger than that of the matrix, and hence the surface or inside of such hard precipitates would be the initiation sites of cracks. Thus, it is considered that the formation of large sigma phase precipitates led to drastic embrittlement in the irradiated Mo–41Re.

In our previous works [17,18], Vickers hardness and tensile tests were conducted for the irradiated specimens of Mo–Re alloys. In the specimens ruptured in the elastic region in tensile tests, Vickers hardness tests did not give any evidence of embrittlement. However, in the most specimens that exhibited appreciable ductility, radiation hardenings estimated by the two methods agreed in their tendency. Hence, evaluation of radiation hardening by Vickers hardness test would be useful, but the results should be carefully evaluated when specimens are expected to be embrittled.

#### 4.3. Optimization of Re content and thermal treatment for Mo–Re alloy as fusion reactor application

In the irradiation conditions of this work, higher Re contents led to larger radiation induced precipitation, radiation hardening and embrittlement. The main purpose of Re addition to Mo is to control the recrystallization embrittlement and to improve the ductility in the unirradiated conditions. In previous works, recrystalli-

zation temperature and ductility increased with increasing Re content, however, the increase rate was significant at and below 5 wt% Re addition [7,9]. Hence, we can say that 2–5 wt% Re addition would be the best content to suppress both of the recrystallization embrittlement and the radiation embrittlement.

Hasegawa et al. [18] reported that the stress-relieved Mo–5Re retained some ductility after irradiation even when the recrystallized Mo–5Re was embrittled. They discussed that smaller grain size in the stress-relieved specimens would suppress crack propagation. In this work, the growth of sigma phase precipitates was suppressed in the stress-relieved specimens. Smaller mean sizes of sigma phase precipitates in the stress-relieved specimens would affect to suppress embrittlement as well. Therefore, it is considered that the stress relief thermal treatment would be more effective in suppressing radiation embrittlement than the recrystallization thermal treatment.

## 5. Conclusion

Mo–Re alloys with several Re contents (0–41 wt%) were heat treated to be stress-relieved or recrystallized, and were neutron irradiated at about 681–1072 K up to about 20 dpa in FFTF/MOTA-2B. After the irradiation, microstructural observation and Vickers hardness testing were conducted. The following results were obtained.

- (1) In all irradiated Mo–Re alloys, equiaxed sigma phase and platelet chi phase precipitates were observed. Growth of sigma phase precipitates was suppressed in the stress-relieved specimens. Os transmuted from Re by neutron irradiation was detected more in the sigma phase precipitates than in the matrix. In all irradiated specimens, voids were observed. Dislocation loops were observed in the specimens irradiated at 681 and 874 K.
- (2) All of the irradiated specimens showed radiation hardening. In the specimens irradiated at 874 K or below, radiation hardening was increased with increasing Re content. In Mo–41Re irradiated at 874 K or below, there were cracks observed around the indentation after Vickers hardness testing.
- (3) The best Re content of Mo–Re alloys to be used under irradiation, was proposed as 2–5 wt% in the irradiation conditions of this work.

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