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Radiation embrittlement of Mo–Re welds under low-temperature irradiation in the SM reactor

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Abstract

The results of irradiation influence at 120–160°C up to 3.6–6.0 × 10^{21} n/cm^2 (E > 0.1 MeV) in the SM reactor on mechanical properties and microstructure of the electron-beam welds of Mo–Re alloys with a content of 15%, 20%, 30%, and 41% Re are presented in the paper. Severe radiation embrittlement of these materials due to formation of dislocation loops is observed. The welds of Mo–Re alloys with the higher Re content are comparatively less susceptible to the radiation embrittlement. The density of dislocation loops is reduced and the fracture type is changed from intergranular to transgranular with an increase of Re content in the alloys. There is no formation of dislocation loops at all in the weld-fusion zone of Mo–41Re alloy after the initial annealing at 1400°C for 1 h. No radiation-induced formation of the second phases is detected in the investigated Mo–Re alloys.

1. Introduction

Mo–Re alloys are considered as potential structural materials of divertor and other elements of thermonuclear facing to plasma. These alloys have an advantage over other Mo alloys, e.g. low-alloyed ones, due to the better strength–plasticity combination both at increased and especially at low operating temperatures. Experimental study of radiation stability of the welds of Mo–Re alloys is necessary for the following reasons:

1. welds are an obligatory element of practically any complex construction;
2. tendency of Mo alloys to embrittle at low temperatures assumes probable degradation of the mechanical properties both during welding and under further irradiation.

The later provision requires direct experimental examination which is the purpose of this paper.

2. Experimental

All of the Mo–Re alloys, including electron-beam welds, tensile specimens and TEM disks, were manufactured by the National Research Institute for Metals, Japan. The chemical composition of Mo–Re alloys by powder metallurgy method and their heat treatment prior to irradiation (or after electron-beam welding and surface polishing) are presented in Table 1. In this work tensile specimens 25 mm long and with the gauge part of 7.6 × 1.5 × 0.8 mm^3 were used. Melt-run process of the as-rolled sample from the top surface to the bottom by one trial of electron-beam welding machine was performed in the middle of the gauge part in the crosswise direction. The welding zone was about 1 mm width. This procedure is the imitation of electron-beam welding. The welding parameters were as follows: accelerating voltage of 70 kV, beam current of 40 mA, beam movement rate of 200 cm/min. For microstructural investigations TEM disks 3 mm in diameter and 0.2 mm thick were used, made of both base metal and welding zone.

Irradiation was carried out in the SM reactor at 120–160°C up to fluences of (3.6–6.0) × 10^{21} n/cm^2 (E > 0.1

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Table 1
Chemical composition of Mo–Re alloys and its initial heat treatment

Alloy	Elements contents, wt.ppm (Re: wt%)								Initial heat treatment, °C × 1 h
	Re	Si	Cr	Ni	Fe	C	N	O	
Mo–15Re	15.9	<10	<5	<5	10	10	<1	11	900 1400
Mo–20Re	21.4	<10	<5	<5	10	10	<1	10	900 1400
Mo–30Re	31.7	<10	<5	<5	10	10	<1	8	900 1400
Mo–41Re	43.6	10	<5	<5	38	10	NA [a]	NA [a]	900 1400

[a] Contents of N and O in Mo–Re are considered as less than 5 wt.ppm and less than 15 wt.ppm, respectively, because sound welds were obtained without porosity.

Table 2
Irradiation parameters of Mo–Re alloys

Alloy	Initial heat treatment, °C × 1 h	Specimen type	T_{tot} °C	Fluence, $\times 10^{21}$ n/cm^2 (>0.1 MeV)
Mo–15Re	900	Tensile	130	4.4
		TEM disk	120	3.6
	1400	Tensile	130	5.0
		TEM disk	120	3.6
Mo–20Re	900	Tensile	160	5.5
		TEM disk	160	5.9
	1400	Tensile	160	5.5
		TEM disk	160	6.0
Mo–30Re	900	Tensile	160	6.0
		TEM disk	160	6.0
	1400	Tensile	120	5.5
		TEM disk	130	5.5
Mo–41Re	900	Tensile	120	5.0
		TEM disk	130	3.6
	1400	Tensile	120	4.4
		TEM disk	130	3.6

MeV). Table 2 presents the irradiation parameters. The specimens for irradiation were encapsuled with pure helium.

The mechanical tests were performed at the distance-type control 1236R tensile machine at the motion velocity of travelling grip of 1 mm/min (strain rate $\sim 2 \times 10^{-3}$ s^{-1}) at room temperature, 400°C and 750°C in a vacuum not worse than 1×10^{-4} Torr. Metallographic investigations were carried out by the optical microscope MIM-15D. Microhardness was measured with the microhardner PMT-4D with a load of 100 g. The fracture surface was studied using the scanning electron microscope REM-101. Microstructural investigations were performed at the transmission electron microscope EM-125.

3. Results

3.1. Mechanical properties

In the initial state the yield stress of Mo–Re alloy welds increase by about 2.5 times with increasing rhenium contents from 15 to 41 wt%. At the same time, the ductility of the material annealed at 1400°C increases at the room temperature test also. At higher test temperatures of 400°C and 750°C the ductility depended on rhenium contents to a lesser degree Fig. 1

The welds of Mo–41 Re alloy have the maximum total elongation of 13% at the room test temperature, where only this alloy showed some necking. The welds of Mo–15Re rupture absolutely brittly. The welds of

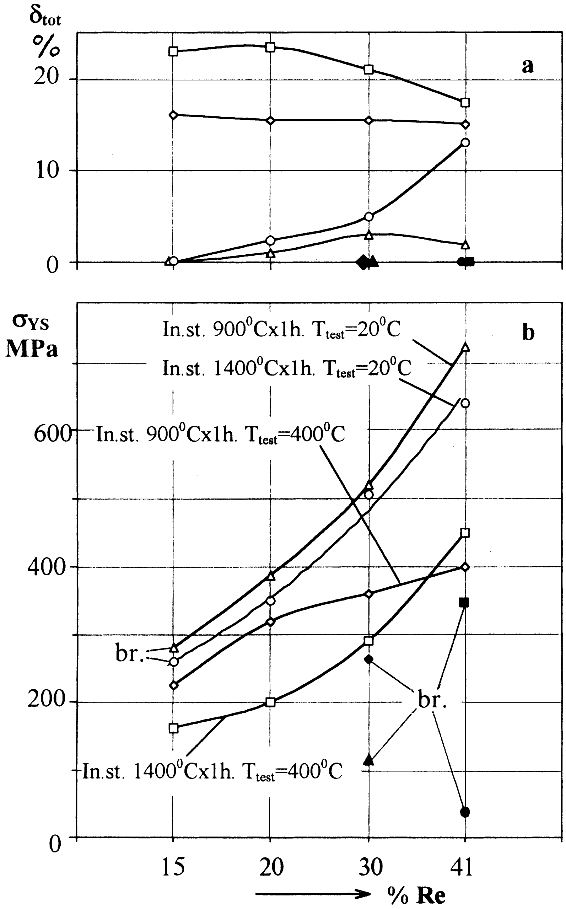

Fig. 1. Yield stress σ_{Ys} and total elongation δ_{tot} of Mo–Re alloy welding joints for test temperatures of 20°C and 400°C. △ Initial state: 900°C × 1 h, $T_{test} = 20$°C. ▲ Irradiated: 900°C × 1 h, $T_{test} = 20$°C. ○ Initial state: 1400°C × 1 h, $T_{test} = 20$°C. ● Irradiated: 1400°C × 1 h, $T_{test} = 20$°C. ◇ Initial state: 900°C × 1 h, $T_{test} = 400$°C. ♦ Irradiated: 900°C × 1 h, $T_{test} = 400$°C. □ Initial state: 1400°C × 1 h, $T_{test} = 400$°C. ■ Irradiated: 1400°C × 1 h, $T_{test} = 400$°C. br – brittle rupture.

Mo–alloys with 20 and 30 wt% rhenium failure on the uniform deforming section of load–displacement diagram at meanings of $\delta_{un} = \delta_{tot} = 1$–3.5%.

As a whole, the initial annealing at 1400°C leads to the decrease of strength and increase of ductile characteristics of Mo–Re alloy welds in comparison with 900°C annealing.

Irradiation leads to strong embrittlement of samples. From among eight tensile specimens inserted in the irradiation capsule, after capsule disassembly and samples retrieving, only four specimens of Mo–30Re and Mo–41Re retained their integrity. According to external inspection, the destruction occurred practically precisely in the middle of the gauge part, in the weld-fusion zone. At the room temperature the ultimate tensile stresses level made up as low as 35–110 MPa. Nevertheless, this fact evidences a few better resistance of Mo–alloy welds with increased rhenium contents ⩾ 30% to the radiation embrittlement.

3.2. Metallography and microhardness

Metallography of one of Mo–15Re specimens, which broke during the capsule disassembly, is given in Fig. 2(a). We can see three representative zones: base metal (average grain size of 30 μm), heat-affected zone (grain size increases to 60 μm) and weld-fusion zone (stretched grains, up to 500 μm in length, are oriented perpendicularly to the electron beam moving during the welding). Width of fusion zone is 0.7–1.0 mm and that of heat-affected zone is 0.4–0.5 mm. The welds of other Mo–Re alloys showed similar features. There is no difference between the specimens broken during capsule disassembly (Figs. 2(a) and 2(b)) and during mechanical tests (Fig. 2(c)). The grain size of the base metal in Mo–30Re and Mo–41Re alloys decreases to 15 μm. Rupture of Mo–Re alloy specimens both prior to and after irradiation, both during capsule disassembly and during mechanical tests occurs over the melting zone only.

Microhardness of irradiated welds of Mo–Re alloys fluctuated in the wide range from 600 to 900 kg/mm^2. With increasing rhenium contents the microhardness increased. So, the values of irradiated Mo–15Re, Mo–20Re and Mo–30/41Re alloys were 580–620, 710–750, and 830–900 kg/mm^2, respectively. It should be noticed that the fusion zone microhardness was about 30–70 kg/mm^2 lesser than that of base metal.

3.3. Fractography

In initial state rupture of the weld of Mo–15Re alloy corresponds to the brittle grain-boundary one. On the fracture surface of Mo–20Re alloy apart from brittle grain-boundary surfaces, one can also see brittle transgranular rupture of separate grains. The fracture surface of Mo–30Re alloy corresponds to the mixed (intergranular and transgranular) type of brittle rupture. The fracture type of Mo–41Re alloy is practically a completely transgranular one. Only in separate sections there is brittle rupture along boundaries.

The irradiation did not change a fracture type of the welds of Mo–alloys with 15%, 20% and 30% Re. The welds of Mo–41Re in the initial state after annealing at 1400°C mainly failured by transgranular cleavage with appearance in the separate sections of the ductile fracture.

3.4. Electron microscopy

In the initial state of the welds of Mo–Re alloys only dislocations, dislocation networks and dislocation tangles

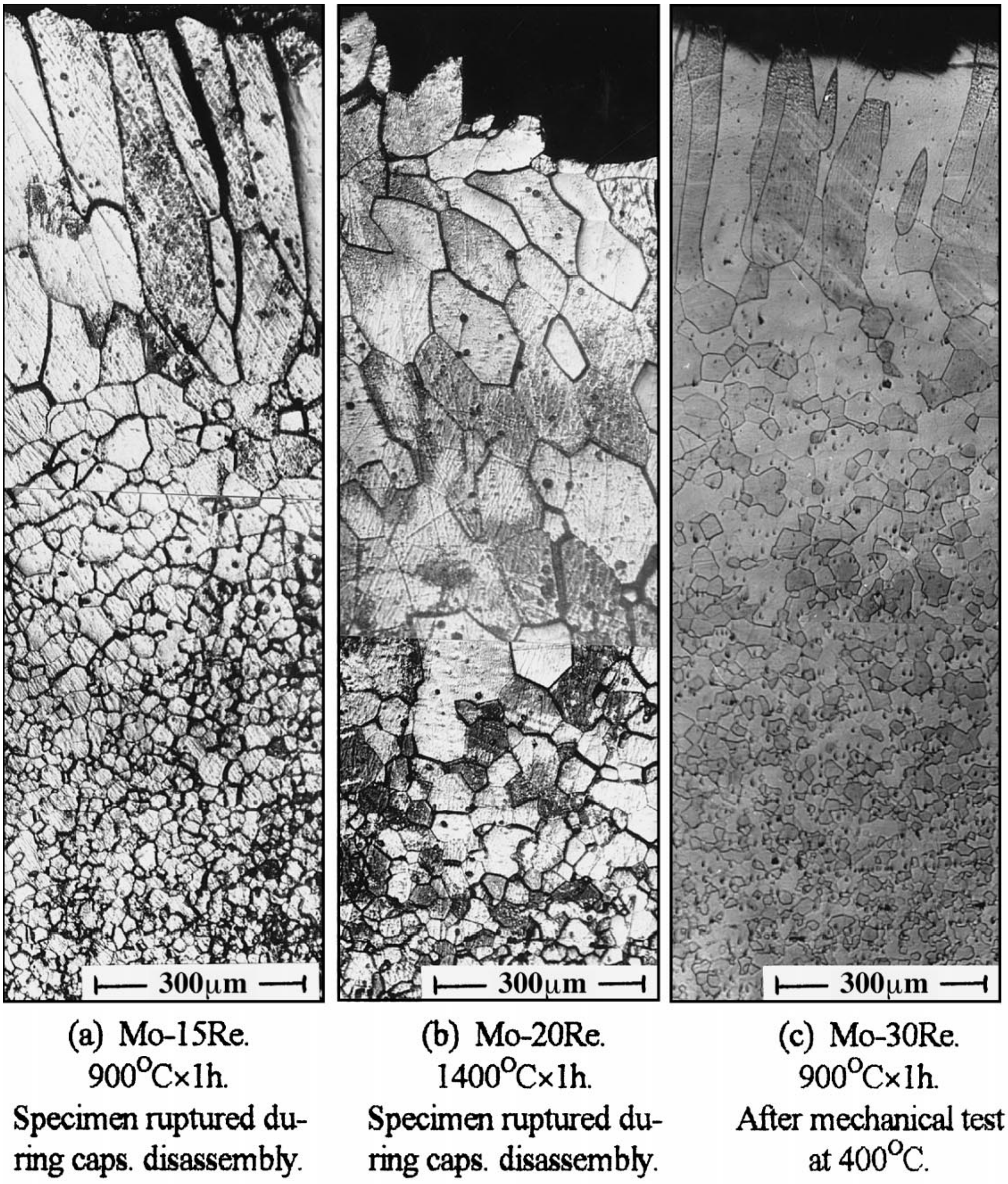

Fig. 2. Metallography of irradiated Mo–Re alloy welds.

are seen (Fig. 3a and (b)). Dislocation density in the fusion zone was less than in the base metal. So, for the Mo–15Re alloy the dislocation density in the base metal was 2.9×10^{10} cm^{-2} and that in the fusion zone of the weld was 1.9×10^{10} cm^{-2}.

The results of transmission electron microscope investigations of irradiated welds of Mo–Re alloys are presented in Fig. 3(c)–(f) and in Table 3. It shows that irradiation leads to the formation of the dislocation loops typical for such low-temperature irradiation. The average size is 7.5–10 nm.

Density of dislocation loops varied in the range from 4.5×10^{15} to 2.6×10^{16} cm^{-3} and decreased with increasing of rhenium content. Dislocation loop

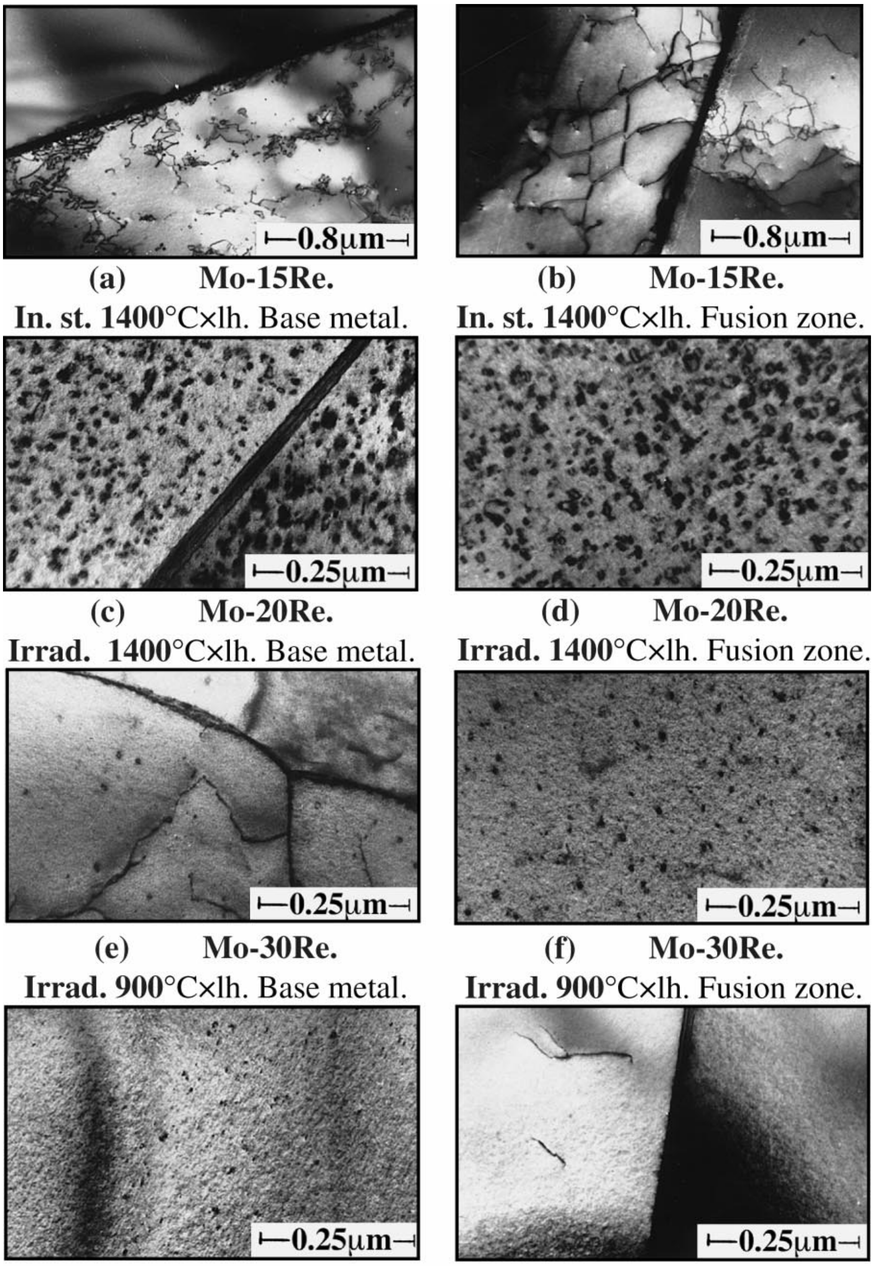

Fig. 3. TEM microstructure of Mo–Re alloy welds.

distribution in the base metal of Mo–41Re alloy was extremely irregular, but in the fusion zone the dislocation loops were absent.

The structure of all irradiated Mo–Re alloys is retained as one phase. The second phase precipitations, an appearance of which was observed in Mo–Re alloys at higher irradiation temperatures [1], were not detected.

4. Discussion

Electron-beam welding considerably deteriorates the mechanical properties of Mo–Re alloys especially because of the appearance of a weak section in the central specimen part-weld-fusion zone. With increasing rhenium contents the share of brittle intergranular fracture

Table 3
Average size and density of dislocation loops in irradiated Mo–Re alloy welds

Alloy heat-treatment	Zone	Average size (nm)	Average density (cm^{-3})
Mo–20Re	Base metal	8.5	2.6×10^{16}
1400°C × 1 h	Fusion zone	10	2.3×10^{16}
Mo–30Re	Base metal	8	4.5×10^{15}
900°C × 1 h	Fusion zone	8	9.4×10^{15}
Mo–41Re	Base metal [a]	7.5	7.0×10^{15}
1400°C × 1 h	Fusion zone	–	–

[a] Distribution of dislocation loops is extremely irregular. This is one of the clusters of the dislocation loops.

was reduced and the share of transgranular one was increased, which is in accordance with the previous result [2]. In general, alterations in the characters of the mechanical properties and the fracture and structural features under irradiation of Mo–alloys, particularly for Mo–Re alloys, corresponded to the earlier published results [3–6].

Fracture of the welded specimens both before and after irradiation happened through the weld-fusion zone only, probably, because of the following reasons:

- cross-section in the centre of tensile specimens was 1.3% less than that for the gauge part edges;
- according to the microhardness measurements the melting zone was 6–8% less strengthened than the base metal;
- probably, in the fusion zone there were some microdamages, such as microporosities, allevisting the process of crack nucleation during deforming.

5. Conclusions

1. Irradiation of the tensile specimens and TEM disks of the welds of Mo–alloys with 15%, 20%, 30% and 41% rhenium contents at 120–160°C to the neutron fluence of 6.0×10^{21} n/cm^2 ($E > 0.1$ MeV) led to the strong radiation embrittlement. The fracture took place only over the specimens centre through the weld-fusion zone. With increasing rhenium content the fracture type changed from the brittle intergranular type to the transgranular one.

2. It was shown that with increasing rhenium content the dislocation loop density with an average size of 7.5–10 nm was reduced 4–6 times and in the fusion zone of Mo–41Re welds were quite absent.

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