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The effect of rhenium on the radiation damage resistivity of Mo–Re alloys

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Abstract

The results of investigations of alloys in the Mo–Re system are presented with Re varying from 0.5 to 47%. Samples were irradiated in the SM-2 reactor up to doses of $1-5.5 \times 10^{25}$ n/m² (E > 0.1 MeV) at 100°C, 210°C, 260°C and 330°C and in the BOR-60 reactor to doses of $0.8-2.0 \times 10^{26}$ n/m² (E > 0.1 MeV) at 480°C, 550°C, 760°C and 800°C. Irradiation to high doses in the SM-2 and BOR-60 reactors results in pronounced embrittlement of Mo–Re alloys in the testing temperature range of 300–600°C. The study of the electrical conductivity of the alloys has shown that irradiation in SM-2 results in a linear increase of the electrical resistivity with increasing Re concentration. Based on the analysis of available data the tentative conclusion has been made, that during irradiation in a mixed-spectrum reactor the resonance reaction proceeds on Re giving rise to the accumulation of transmutants (Os) reducing the electrical conductivity of the alloys. © 1998 Elsevier Science B.V.

1. Introduction

Mo alloys are under consideration as one of the candidate materials for the heat sink system of the ITER divertor [1]. Their attractive properties are high strength and satisfactory thermal conductivity. Besides, Mo alloys are resistant to swelling. The main disadvantage of Mo alloys is their tendency to ductile-to-brittle transition-temperature (DBTT) shift to the high temperature region under irradiation [2].

To solve this problem it has been proposed recently to use Mo–Re alloys [3,4]. For the most part, the works on substantiating the applicability of Mo–Re alloys for ITER were performed by Japanese and American scientists [5,6]. It was found that alloying with 5% Re reduces DBTT of Mo to -100° C (173 K), when unirradiated. It is shown that irradiation at 590°C to a dose of 10^{24} n/m² does not cause strong embrittlement of the Mo–5Re alloy. Re addition improves essentially the strength characteristics of welds for Mo-5Re [7], this being particularly important for the production technology of the divertor.

All the above advantages have drawn attention to Mo-Re alloys and relevant works have recently appeared in Europe [8–10] and in Russia [11]. It should be noted that the real data on the effect of high-dose irradiation on the properties of the Mo-Re system have not yet been published up till now, thus making it difficult to substantiate the applicability of these materials for ITER.

This work presents the results of the investigation of various Mo–Re alloys irradiated in the temperature range of $100-800^{\circ}$ C to doses of 2×10^{26} n/m².

2. Experimental procedure

Mo–Re alloys were investigated with Re varying from 0.5 to 47%. The real composition of the alloys is presented in Table 1. Mo–Re alloys were produced by the Moscow Institute of Refractory Metals (VNIITS). The materials were supplied in the deformed and stress-relieved state in

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Table 1 Chemical composition of Mo-Re alloys (wt%)

Element	Mo pure	Mo-0.5Re	Mo-1Re	Mo-3Re	Mo-5Re	Mo-9Re	Mo-13Re	Mo-20Re	Mo-30Re	Mo-47Re
Re	no	0.51	1.08	3.17	5.7	9.2	13.2	18.7	27.6	46.5
С	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
Mg	0.05	0.04	0.04	0.05	0.05	0.04	0.04	0.05	0.06	0.06
Si	0.013	0.014	0.011	0.013	0.014	0.013	0.012	0.015	0.017	0.017
Ni	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
O ₂	0.005	0.004	0.004	0.004	0.004	0.004	0.004	0.004	0.004	0.004
N	0.002	0.002	0.002	0.002	0.002	0.002	0.002	0.002	0.002	0.002
Al	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001	0.001
Fe	0.004	0.004	0.004	0.004	0.004	0.003	0.004	0.004	0.004	0.004

the form of 8 mm diam. bars produced by hot pressing in vacuum at 1000°C, from which cylindrical tensile specimens 3 mm in gage diameter and 10 mm in length were made. To eliminate the surface cracks the gage region of all samples was mechanically polished to remove damages in the surface layer ($\approx 5 \,\mu$ m).

The specimens were irradiated in helium-filled capsules in the reactor SM-2 up to a dose of $1-5.5 \times 10^{25} \text{ n/m}^2$ (E > 0.1 MeV) at 100°C, 210°C, 230°C, 262°C and 336°C, as well as in the reactor BOR-60 up to doses of $0.8-2 \times 10^{26} \text{ n/m}^2$ (E > 0.1 MeV) at 480°C, 550°C, 585°C, 760°C and 800°C.



Fig. 1. Effect of irradiation temperature on the ultimate tensile strength of Mo–Re alloys. Irradiation in BOR-60 fast neutron reactor up to 5 dpa at $T_{irr} = 450-800^{\circ}$ C. $T_{test} = T_{irr}$. \triangle Mo pure, unirradiated; \blacktriangle Mo pure, 5 dpa; \bigtriangledown Mo–1Re, unirradiated; \checkmark Mo–1Re, 5 dpa; \diamondsuit Mo–5Re, unirradiated; \blacklozenge Mo–9Re, unirradiated; \blacksquare Mo–9Re, 5 dpa; \triangleright Mo–20Re, unirradiated; \blacklozenge Mo–20Re, 5 dpa.

The initial and irradiated specimens were tensile tested in vacuum in the testing temperature range of $100-800^{\circ}$ C. The strain rate amounted to $6 \times 10^{-3} \text{ s}^{-1}$. The electrical conductivity was measured by the standard four-point method. Some of the samples were investigated by SEM and TEM methods. The structure of the samples was investigated by optical metallography.

3. Experimental results

3.1. Unirradiated condition

Our previous study [11] demonstrates that an increase in Re concentration reduces the thermal conductivity, increases the thermal expansion coefficient and reduces the heat capacity of Mo alloys. Besides, Re increases the yield strength and ultimate strength of Mo alloys throughout the testing temperature range of 300–800°C. The study of the structure of the initial specimens revealed that Mo–Re alloys have a highly deformed structure with a high level of anisotropy. As follows from the results of the metallography, the characteristic grain sizes of the Mo–0.5Re alloy are $\approx 200 \ \mu\text{m}$ in the extrusion direction and 75 $\ \mu\text{m}$ in the transversal direction and for Mo–5Re alloy 125 $\ \mu\text{m}$ and 60 $\ \mu\text{m}$, respectively. The smallest grain size occurred in Mo–30Re and Mo–47Re alloys (120 $\ \mu\text{m}$ and 30 $\ \mu\text{m}$).

The SEM investigations of the fracture character of unirradiated specimens at 20°C demonstrate that all alloys from pure Mo to Mo–47Re are fractured by a mixed brittle–ductile manner with a brittle transgranular component. At 300–700°C all alloys fracture in a ductile manner. Unirradiated Mo–Re alloys have a relatively high ultimate strength ($\sigma_u = 400-600$ MPa) and a high elongation ($\delta_{tot} > 10\%$) throughout the testing temperature range of 300–700°C (Figs. 1 and 2).

The TEM investigations of the specimen structure revealed that all alloys have a highly deformed polygonized structure [12]. In pure Mo the average size of subgrains, d_o , is $\approx 2 \mu$ m. For Mo–9Re $d_o = 1.5 \mu$ m, for Mo–13Re $d_o = 1.3 \mu$ m, for Mo–20Re $d_o = 0.8 \mu$ m and for Mo– 47Re it again increases up to $d_o = 2.3 \mu$ m. When assessing the TEM results, it should be taken into account that the structure of subgrains was investigated only in the transverse direction (because of specifically prepared TEM specimens).



Fig. 2. Effect of irradiation temperature on the total elongation of Mo–Re alloys. Irradiation in BOR-60 fast neutron reactor up to 5 dpa at $T_{irr} = 450-800^{\circ}$ C. $T_{test} = T_{irr}$. \triangle Mo pure, unirradiated; \blacktriangle Mo pure, 5 dpa; \bigtriangledown Mo–1Re, unirradiated; \checkmark Mo–1Re, 5 dpa; \diamondsuit Mo–5Re, unirradiated; \blacklozenge Mo–5Re, 5 dpa; \Box Mo–9Re, unirradiated; \blacksquare Mo–9Re, 5 dpa; \triangleright Mo–20Re, 5 dpa.

3.2. The effect of irradiation on the mechanical properties of Mo–Re alloys

3.2.1. The effect of irradiation in the fast neutron reactor BOR-60

As follows from Figs. 1 and 2, irradiation in the temperature range of 450–800°C to ≈ 5 dpa results in a catastrophic embrittlement of both pure Mo and Mo–Re alloys. All alloys from 0.5Re to 20Re show nearly a twofold reduction in the strength properties at $T_{\text{test}} = T_{\text{irr}} = 450-550$ °C and a zero plasticity. At $T_{\text{irr}} = 760-800$ °C to 5–10 dpa Mo–Re alloys demonstrate hardening and a modest total elongation of 2–3%.

The investigation of the testing temperature dependence of elongation and strength properties of Mo–Re alloys irradiated in the temperature range of 450–580°C allowed for the conclusion that the maximum embrittlement is observed at $T_{\text{test}} = 500$ °C (Figs. 3 and 4). At 300°C the specimens of Mo–1Re alloy and pure Mo have a high total elongation $\delta_{\text{tot}} > 10\%$ and high strength characteristics. Alloys with 5–20% Re are brittle. At 700°C some alloys are ductile and some alloys fracture in a brittle manner. At 800°C all irradiated materials had a satisfactory level of total elongation and hardened.

3.2.2. The effect of irradiation in the mixed spectrum reactor SM-2.

After irradiation to doses of $1-5.5 \times 10^{25}$ n/m² at 100°C, 210°C, 230°C, 260°C and 310°C most Mo alloys are absolutely brittle (Figs. 5 and 6). The results of testing of commercial alloys Mo–ZrC and Mo–Re–HfC irradiated in the same capsules demonstrate that the observed embrittlement is typical not only of Mo–Re alloys but also of other Mo alloys. At $T_{\rm irr} = 260-330$ °C Mo–13Re alloy and pure Mo demonstrate a small total elongation of $\approx 1-4\%$.

3.3. The effect of irradiation on the structure of Mo-Re alloys

3.3.1. Metallography

The optical metallography does not reveal any essential change in the grain size up to $T_{\rm irr} = 800^{\circ}$ C (as this temperature is $\approx 0.35T_{\rm melt}$, this, in principle, is insufficient for effective recrystallization of materials under irradiation).

Fig. 3. Effect of test temperature on the ultimate tensile strength of Mo–Re alloys. Irradiation in BOR-60 fast neutron reactor up to 5 dpa at $T_{irr} = 480-585^{\circ}$ C. \triangle Mo pure, unirradiated; \blacktriangle Mo pure, $T_{irr} = 480^{\circ}$ C; \Diamond Mo pure, $T_{irr} = 530^{\circ}$ C; \bigtriangledown Mo–1Re, unirradiated; \checkmark Mo–1Re, $T_{irr} = 510^{\circ}$ C; \diamondsuit Mo–1Re, $T_{irr} = 565^{\circ}$ C; \Box Mo–5Re, unirradiated; \blacksquare Mo–5Re, $T_{irr} = 480^{\circ}$ C; \triangleright Mo–20Re, unirradiated; \blacktriangleright Mo–20Re, $T_{irr} = 585^{\circ}$ C; \boxdot Mo–20Re, $T_{irr} = 525^{\circ}$ C.



3.3.2. The character of materials fracture

The specimens from pure Mo, when irradiated in the SM-2 reactor to $0.76 \times 10^{25} \text{ n/m}^2$ at 300°C and tested at 500°C ($\delta_{\text{tot}} = 0.1\%$), fracture in a brittle manner by transgranular cleavage. Recall that the fracture of this specimen, when unirradiated, was ductile.

One noteworthy characteristic of Mo–5Re alloy is its tendency to brittle intergranular fracture at $T_{\text{test}} = 300^{\circ}\text{C}$ after irradiation at 580°C to $1.55 \times 10^{26} \text{ n/m}^2$ ($\sigma_u = 382$ MPa, $\delta_{\text{tot}} = 0$). The alloy, which has a sizeable neck in the initial state, fractures after irradiation in BOR-60 absolutely without local deformation and only along the grain boundaries, with the microdeformation being observed on the grain facets [12]. Inclusions on the grain facets were also observed. The fracture of a specimen, irradiated in SM-2 at $T_{\text{irr}} = 336^{\circ}\text{C}$ to a dose of $5 \times 10^{25} \text{ n/m}^2$ and $T_{\text{test}} = 300^{\circ}\text{C}$, was also of brittle intergranular character without a noticeable neck.

The fracture of Mo–47Re alloy, irradiated in BOR-60 to 1.1×10^{26} n/m² at 480°C and tested at 300°C ($\sigma_u = 1280$ MPa, $\delta = 3\%$), is of mixed character with both brittle intergranular and transgranular brittle cleavage fracture.

3.4. The effect of irradiation on electric resistivity of Mo–Re alloys

3.4.1. The effect of irradiation in BOR-60 reactor

Irradiation in BOR-60 at 480–565°C to $0.8-1.55 \times 10^{26}$ n/m² results, as seen in Fig. 7, in a slight increase of the electrical resistivity of Mo–Re alloys, except for the Mo–13Re alloy, which shows a systematic reduction in ρ throughout the dose–temperature range of irradiation.

At increased irradiation temperatures of 770–800°C $(1.1 \times 10^{26} \text{ n/m}^2)$ a slight increase of the electrical resistivity is observed only for pure Mo and Mo–9Re and Mo–0.5Re alloys. Alloys with a higher content of Re demonstrate a reduction in ρ .

3.4.2. The effect of irradiation in the SM-2 reactor

Irradiation in the SM-2 reactor to a dose of $4.3-5.5 \times 10^{25}$ n/m² in the temperature range of 260–536°C results in an increase of the electrical resistivity of Mo–Re alloys.



Fig. 4. Effect of test temperature on the total elongation of Mo–Re alloys. Irradiation in BOR-60 fast neutron reactor up to 5 dpa at $T_{irr} = 480-585^{\circ}$ C. \triangle Mo pure, unirradiated; \blacktriangle Mo pure, $T_{irr} = 480^{\circ}$ C; \Diamond Mo pure, $T_{irr} = 530^{\circ}$ C; \bigtriangledown Mo–1Re, unirradiated; \checkmark Mo–1Re, $T_{irr} = 510^{\circ}$ C; \diamondsuit Mo–1Re, $T_{irr} = 565^{\circ}$ C; \Box Mo–5Re, unirradiated; \blacksquare Mo–5Re, $T_{irr} = 480^{\circ}$ C; \triangleright Mo–20Re, unirradiated; \blacktriangleright Mo–20Re, $T_{irr} = 555^{\circ}$ C; \boxdot Mo–20Re, $T_{irr} = 555^{\circ}$ C.





Fig. 5. Effect of irradiation temperature on the ultimate tensile strength of Mo–Re alloys. Irradiation in SM-2 mixed spectrum reactor up to 0.5–2 dpa, $T_{\text{test}} = T_{\text{irr}}$. \triangle Mo–13Re, unirradiated; \blacktriangle Mo–13Re, 0.5–2 dpa; \bigtriangledown Mo–ZrC, unirradiated; \blacktriangledown Mo–ZrC, 0.5 dpa; \diamondsuit Mo–Re–HfC, unirradiated; \blacklozenge Mo–Re–HfC, 0.5 dpa; \Box Mo pure, unirradiated; \blacksquare Mo–5Re, unirradiated; + Mo–5Re, 0.5–2 dpa; \triangleright Mo–47Re, unirradiated; \blacktriangleright Mo–47Re, 2 dpa.

In this case, with a rise in Re concentration the effect is considerably increased (Fig. 8).

4. Discussion

4.1. The effects observed after irradiation in fast neutron reactors

4.1.1. Analysis of causes for embrittlement of Mo–Re alloys in the range of $T_{irr} = T_{test} = 400-600^{\circ}C$ (irradiated in BOR-60)

The investigations undertaken testify clearly, that at $T_{\rm irr} = T_{\rm test} = 480-585^{\circ}$ C at a dose of ≈ 5 dpa Mo-Re alloys demonstrate a catastrophic embrittlement with the fracture mechanism changed. At $T_{\rm test} = 300-700^{\circ}$ C all irradiated alloys fracture in a ductile manner. After irradiation at $T_{\rm irr} = T_{\rm test} = 480-585^{\circ}$ C at a dose of ≈ 5 dpa the total elongation of specimens is zero, and their strength drops by a factor of 2–3. A simultaneous decrease in the strength and elongation are typical for brittle intergranular fracture. Indeed, the optical microscopy shows that the

Mo–5Re alloy, for example, fractures without linear necking only along the grain boundaries.

The tendency of Mo alloys for brittle intergranular fracture was observed previously [2,13] but at small irradiation doses for recrystallized alloys. In this work the material was not recrystallized. In the studies by Wiffen et al. [6,12], embrittlement of pure Mo was also observed at T_{test} up to 550°C after irradiation in EBR-II to doses of 2.5×10^{26} n/m². Let us consider the reasons that might be responsible for such a behaviour.

4.1.2. Impact of swelling on embrittlement

In the embrittlement temperature range Mo–Re alloys swell but weakly. The Mo–5Re structure contains, for example, small voids with a high density. Grains have a void-free zone along their boundaries, where the deformation might localize.

4.1.3. Radiation induced segregation (RIS)

As the investigated alloys contain moderate amounts of O and N of about 70 appm, the possibility of the grainboundary segregation is highly probable at 500°C (i.e.





Fig. 6. Effect of irradiation temperature on the total elongation of Mo–Re alloys. Irradiation in SM-2 mixed spectrum reactor up to 0.5-2 dpa, $T_{\text{test}} = T_{\text{irr}}$. \triangle Mo–13Re, unirradiated; \blacktriangle Mo–13Re, 0.5-2 dpa; \bigtriangledown Mo–ZrC, unirradiated; \bigstar Mo–ZrC, 0.5 dpa; \diamondsuit Mo–Re–HfC, unirradiated; \bigstar Mo–Re–HfC, 0.5 dpa; \Box Mo pure, unirradiated; \blacksquare Mo pure, 2 dpa; + Mo–5Re, unirradiated; + Mo–5Re, 0.5-2 dpa; \triangleright Mo–47Re, unirradiated; \blacktriangleright Mo–47Re, 2 dpa.

 $0.27T_{melt}$). Morito [14], when investigating the surfaces of the grain boundary facets of brittle Mo–13Re, found thereon oxygen segregation. The effect of Tc, Ru and Os transmutants on RIS also must not be ruled out.

Thus, the observed catastrophic embrittlement can be explained by two reasons. On the one hand, the high density of radiation defect complexes (loops and pores) harden the grain body (matrix). On the other hand, the radiation-induced segregation (RIS) of O and N impurities and conceivably transmutants (Tc, Ru accumulated on Mo, as well as Os accumulated on Re) causes the boundaries to weaken. Both these factors, when combined, result in the localisation of microdeformations along the grain boundaries and the fracture at zero elongation and a low level of fracture stress. Such an approach correlates well with the experimentally observed changes in the ductility and strength of Mo–Re alloys irradiated at $T_{\rm irr} = 480-585^{\circ}C$ and tested in the temperature range of $300-800^{\circ}C$ (Figs. 3 and 4).

Really, at $T_{\text{test}} = 300^{\circ}$ C strengthening of alloys is rather appreciable, but $T_{\text{test}} = 0.2T_{\text{melt}}$ is not enough for the realisation of the grain-boundary sliding. As a consequence, material is fractured mainly in a brittle transgranular manner.

At $T_{\text{test}} = 800^{\circ}\text{C}$ the segregation on the grain boundaries disappears, defects in the matrix are partially annealed while under testing. Mo–Re alloys have a satisfactory level of strength and ductility properties. At $T_{\text{test}} =$ 500°C radiation defect complexes in the grain body are stable during testing. The segregation on the grain boundaries weakens cohesion. The testing temperature T = $0.27T_{\text{melt}}$ is sufficient for the grain-boundary sliding to show up. As a consequence, the samples fracture in a brittle intergranular way at a low level of fracture stresses.

4.1.4. The effect of high-temperature irradiation and transmutation on phase precipitation in Mo–Re alloys

The analysis of the behaviour of Mo–Re alloys irradiated at increased temperatures (700–800°C) reveals that in alloys with 47% Re the Re-enriched phases precipitate intensively. This process is attested, first of all, by a change in the strength properties. As follows from Fig. 9 (where the ultimate strength gain $\Delta \sigma_u$ of Mo–Re alloys is shown as the function of the irradiation temperature), the



Fig. 7. Measured change in electrical resistivity ($\Delta \rho_{irr} = \rho_{irr} - \rho_{unirr}$), $T_{test} = 20^{\circ}$ C vs. irradiation temperature for Mo–Re alloys irradiated in BOR-60 fast neutron reactor up to 5–10 dpa. \triangle Mo pure, \blacktriangle Mo–0.5Re, \bigtriangledown Mo–1Re, \blacklozenge Mo–5Re, \diamondsuit Mo–9Re, + Mo–13Re, \blacklozenge Mo–20Re, \blacksquare Mo–47Re.

level of $\Delta \sigma_{\rm u\,irr}$ is increased with a growth in Re concentration in samples from 100 MPa for Mo-1Re to 450 MPa for Mo-47Re at $T_{\rm irr} = 500^{\circ}$ C. As seen in the phase diagram, the χ -phase precipitates in the Mo–47Re alloy at temperatures of 700-800°C. Hence, during irradiation at 700–800°C the χ -phase should precipitate as the result of long-term high-temperature exposure [15]. On the other hand, in the irradiated Mo-47Re alloy transmutants (Os) will also contribute to the precipitation. Besides, the acceleration of irradiation-stimulated diffusion can add to the precipitation of both the χ -phase and other phases from a solution. At $T_{\rm irr} = 800^{\circ}$ C the effect of Re concentration on radiation strengthening $\Delta \sigma_{irr}$ is essentially higher also for Mo–47Re, $\Delta \sigma_{\rm irr} = 1000$ MPa, i.e. twice as high as when irradiated at 500°C, due to intensification of the χ -phase precipitation at $T_{\rm irr} = 800^{\circ}$ C.

The analysis of the effect that Re has on the electrical resistivity of Mo–Re alloys shows that in going from $T_{\rm irr} = 500^{\circ}$ C to $T_{\rm irr} = 800^{\circ}$ C the electrical resistivity gain $\Delta \rho_{\rm irr}$ caused by irradiation is sharply decreased for Mo–47Re alloys attesting unambiguously to that Re precipitates from solid solution (Re in solid solution increases nearly linearly with the electrical resistivity of Mo–Re, i.e. $\Delta \rho = kC_{\rm Re}$).

These results allow the conclusion to be made, that at $T_{\rm irr} \approx 700 = 800^{\circ}$ C in BOR-60 in Mo–Re alloys an intensive precipitation of secondary Re-enriched phases occurs (χ -phase according to existing diagrams). This results in an extreme strengthening of alloys with a high concentration of rhenium (47% Re) and a drop in the electrical resistivity $\Delta \rho$ as a result of Re precipitation from a solid solution.

It was demonstrated in [16] that after irradiation in FFTF up to 60 dpa at 645°C and 730°C voids in the Mo–41Re alloy are lacking. But on the other hand, an intensive precipitation of needle-shaped phases \approx 100 nm in length and 20 nm in width is observed in the alloy, with the density of the phases being very high. The average distance between the phases is $\lambda = 80$ nm.

The EDS analysis of the phases revealed that they are 8% Mo, 75% Re and 17% Os [16]. In our case, the Os concentration should be considerably lower, i.e. $\approx 2-4\%$. Therefore, the phases should also contain Re and Mo. Somewhat unexpected is the fact that despite an extremely high strengthening of ≈ 1000 MPa these precipitates do not cause embrittlement of the Mo–47Re alloy. On the contrary, this alloy, aged while under high-temperature irradiation, has a high ductility of $\approx 10\%$, when irradiated.





Fig. 8. Measured electrical resistivity ρ_{irr} vs. irradiation temperature for Mo–Re alloys irradiated in SM-2 mixed spectrum reactor up to 0.5–2 dpa. \triangle Mo pure, 0.5 dpa, \blacktriangle Mo–0.5Re, 2 dpa, \bigtriangledown Mo–9Re, 2 dpa, \spadesuit Mo–30Re, 2 dpa, \diamondsuit Mo–47Re, 2 dpa.

This fact testifies that the formation of phases in the grain body weakens the grain-boundary segregation (since the same elements, Re and Os, are involved). It is worth noting that the precipitation of Re-enriched phases, while under irradiation, increases both the strength and ductility of the alloy and decreases its electrical resistivity, hence increases its thermal conductivity.

4.2. Effects observed in the mixed-spectrum reactor SM-2

Irradiation in the SM-2 reactor results in embrittlement of the alloys, which is particularly intensive at low irradiation temperatures. It may be concluded that the samples irradiated at $T_{\rm irr} = 100^{\circ}$ C in SM-2 fracture in a brittle transgranular way at zero deformation.

At $T_{\rm irr} = 260-360^{\circ}$ C, a difference is observed in the behaviour of Mo–Re alloys and pure Mo or alloys of Mo–Zr–C type. The latter is not practically hardened at $T_{\rm irr} = 260-360^{\circ}$ C. The alloys with 13–47% Re are hardened, with hardening being increased with increasing Re concentration.

 $T_{\text{test}} = T_{\text{irr}} = 300^{\circ}\text{C}$ is not high enough to form Re phases in the alloys. So, rhenium impact is attributable to another reason.

In our opinion the main effect of irradiation in SM-2 is a high accumulation rate of heavy elements of transmutants produced by the resonance reactions on Re with an extremely high cross section of $= 100 \times 10^{-24}$ cm⁻².

These are

$$\operatorname{Re}^{185}(n, \gamma) \to \operatorname{Re}^{186}(\beta^{-}) \to \operatorname{Os}^{186}$$
$$\operatorname{Re}^{187}(n, \gamma) \to \operatorname{Re}^{188}(\beta^{-}) \to \operatorname{Os}^{188}.$$

Garner and Greenwood [16] were the first who suggested that effective accumulation of Os and other heavy isotopes on Re is possible.

The results obtained in our study on the gain in the electrical resistivity of Mo–Re alloys (irradiated in the SM-2 reactor) are only attributable to transmutation on Re and on thermal neutrons.

In our studies [17] we developed the methodology for the assessment of transmutant contribution to the gain in the electrical resistivity of pure copper. With this methodology used and the dependence of the gain $\Delta \rho_{\text{trans}} = \Delta \rho_{\Sigma}$ $-\Delta \rho_{\text{rad,def.}}$ built up for Mo–Re alloys, it becomes clear that there is a linear dependence of the gain $\Delta \rho_{\Sigma}$ (Fig. 10) on Re concentration. To build up the dependence in Fig. 10 we used the results of measurements of $\Delta \rho_{\Sigma}$ for the



Fig. 9. Effect of irradiation temperature and rhenium concentration on the ultimate tensile strength change ($\Delta \sigma_{irr} = \sigma_{unirr} - \sigma_{irr}$) of Mo–Re alloys. Irradiation in BOR-60 fast neutron reactor up to 5–10 dpa. \triangle Mo–47Re, 5–10 dpa, \blacktriangle Mo–13Re, 5–10 dpa, \blacksquare Mo–1Re, 5–10 dpa.

specimens irradiated in one ampoule and on the same floor, i.e. irradiated by the same fluence of thermal neutrons. In this case a linear gain in $\Delta \rho$ is observed therein with a growth in rhenium concentration. When using the data obtained at the same dose of fast neutrons, no linear correlation of $\Delta \rho$ with Re concentration was observed.

When assessing changes in the composition of the Mo–30Re alloy (using the cross sections of thermal neutron accumulation of Os on Re from Ref. [15]), it can be seen that the alloy is transferred to Mo–13Re–17Os at a thermal neutron fluence of $\approx 7 \times 10^{25}$ n/m². In this case Re burning will result in an increase of the electrical resistivity. Then, Os increases ρ of Mo more sharply than Re does.

In samples with 13 and 47% Re irradiated up to 2 dpa in SM-2, osmium is accumulated in the amount of 7 and 25%, respectively. This transformation of alloys is consistent (as follows from Fig. 10) with a practically linear gain in the electrical resistivity $\Delta \rho_{\rm irr}$ depending on the initial Re concentration in the alloys. The same samples also demonstrate considerable strengthening of $\Delta \sigma_{\rm irr} = 250-$ 300 MPa.

For samples from pure Mo and a Mo–ZrC alloy (not containing rhenium) and irradiated under the same condi-

tions $(T_{irr} = T_{test} = 260-330^{\circ}C$ and a dose of 2 dpa) the strengthening is practically lacking. As the irradiation temperature $T_{irr} = 0.2T_{melt}$ is obviously insufficient for the formation of Re- and Os-enriched phases, the observed effect of the ultimate strength gain by 250 MPa in Mo-13Re and Mo-47Re alloys is likely to be attributable to the effect of solid transmutants (Os first of all) on the ultimate strength gain $\Delta \sigma_{\rm irr}$. The linear gain in the electrical resistivity $\Delta \rho_{irr}$ with an increase in rhenium concentration for samples irradiated in SM-2 (Fig. 10) is also likely to be associated with the effect of osmium (in solid solution) on the electrical resistivity. In this case, the results obtained testify that osmium increases the electrical resistivity more significantly than rhenium does. As osmium accumulation is accompanied with rhenium burn-out, the gain in the electrical resistivity of irradiated samples may be explained only if we assume that osmium produced by the resonance reactions affects the gain in the electrical resistivity of Mo-Re-Os alloys more significantly than Re does.

To understand the problem in detail the Mo–Os and Mo–Re–Os alloys are to be analyzed for phase stability and electrical conductivity in the future. On the other hand, the EDS investigations should be undertaken to study



Fig. 10. Effect of rhenium concentration on electrical resistivity change ($\Delta \rho_{irr} = \rho_{irr} - \rho_{unirr}$) of Mo–Re alloys irradiated in SM-2 reactor at 260°C up to 2 dpa.

specimens of Mo–Re alloys irradiated to large doses in mixed-spectrum reactors. It would be desirable to assess the gain in ρ on specimens with varying thermal fluence, i.e. with the Cd shielding used, as was the case in [17]. But even now it is obvious that the transmutation will play a significant part in substantiating the applicability of Mo–Re alloys for fusion reactors.

5. Conclusions

As follows from the results of the investigations, the embrittlement of Mo–Re alloys under irradiation in the BOR-60 and SM-2 reactors is controlled by different factors.

In BOR-60 swelling and radiation damage of specimens result in hardening of the grain matrix and in intergranular embrittlement. This effect is likely to be stimulated in the same manner as RIS. At increased irradiation temperatures (700–800°C) RIS disappears and Mo–Re alloys have a sufficiently high level of strength and a satisfactory level of total elongation.

In the SM-2 reactor the resonance reactions on Re result in the accumulation of considerable amounts of Os transmutants, which effectively reduce the electrical conductivity of Mo–Re alloys and harden them. The embrittlement in this case is determined by matrix hardening, fracture at $T_{\rm irr} = T_{\rm test} = 100-200^{\circ}$ C is of transgranular and at $T_{\rm irr} = 300^{\circ}$ C of intergranular character. This is likely to be attributable to the segregation of transmutants along the grain boundaries.

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