Radiation-induced softening of Fe-Mo alloy under high-temperature electron irradiation

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Abstract. Effect of radiation-induced change of mechanical properties of Fe-5 wt.% Mo alloy irradiated with electrons (2 MeV) at room temperature and 400°C has been investigated. Mechanical properties were estimated by Miniaturized Disk Bend Test technique. Effect of radiation softening of the alloy is ascertained the value of which was increased with temperature rise. With the purpose of separation of thermal and radiation contributions into the effects, the tests were carried out for specimens annealed in the same thermal conditions (temperature and duration of annealing) just as during irradiation. Thermal annealing and electron irradiation at 400°C is found to bring to multidirectional effects of the alloy strengthening and softening respectively. It is concluded that irradiation suppresses the effect of thermal-induced strengthening and stimulates a softening of the alloy due to more significant changes in the structure and phase composition of it.

1. Introduction

The most important characteristics of structural materials used in nuclear power plants is their strength and plastic properties, and stability of these properties under irradiation. There are two main reasons for changes in the mechanical properties of metals and alloys under irradiation — it is the formation of the so-called radiation damages (for example, depleted zones) which are obstacles in the way of gliding dislocations, and changing of structure and phase state of materials caused by radiation enhanced diffusion processes. The first mechanism plays a leading role under cascade-forming irradiation (neutrons, swift heavy ions) and usually leads to radiation strengthening of irradiated materials.

At the same time, radiation-induced structure and phase transformations, caused by an introduction of excess concentration of radiation point defects (vacancies and interstitials) into irradiated materials and controlled by the processes of radiation-enhanced diffusion (RED) and/or radiation-induced segregation (RIS), may stimulate the transition of metastable alloys (which are the most commercial ones) in the more equilibrium state (RED effect), or, contrary, result in to local deviations from the equilibrium state of the system under the given conditions (RIS effect). Such processes can be occurred at sufficiently low temperatures when conventional thermal diffusion is almost suppressed. The upper limit of manifestation of such effects is accepted the temperature about of 0.5 \( T_{\text{melt}} \) when concentration of radiation point defects becomes comparable with the thermal-equilibrium concentration of vacancies.
It is known that radiation can be used as a tool for directional changes in the properties of materials, however for the practical realization of these opportunities it is required to understand the controlled mechanisms of radiation-stimulated processes in irradiated materials under different conditions of radiation treatment (temperature, flux and fluence of irradiation).

Most of the known data on the effect of irradiation on the mechanical properties of metals and alloys were obtained under neutron or ion irradiation, and they are evidence of radiation strengthening effect occurring due to formation of various radiation damages, resulting in displacement cascades and playing the role of obstacles for moving dislocations. At the same time, the “diffusion” effect of irradiation can lead to both strengthening and softening of irradiated material depending on what kind of structure is formed in the process of accumulation and annealing of radiation point defects. In these mechanisms of structure changing, an interaction of mobile radiation point defects with intrinsic materials “genetic” defects (dislocations, impurity atoms, phase precipitations, boundaries, and so on) plays the main role, and it is possible for any kind of irradiation when the lattice atoms are knocked out from their sites. Note that changes of materials properties due to radiation-induced structure and phase transformations may be masked by other radiation effects, especially at low doses.

In pure form, the "diffusion" effect of irradiation can be observed, for example, under MeV-energy electron irradiation when displacement cascades and defect clusters does not to be created. In this case, irradiation leads to the formation of isolated vacancies and interstitials able to freely migration before annihilation with each other or annealing at sinks (dislocations, interfaces, voids, etc.).

The most convenient way of investigation of diffusion-controlled mechanisms of radiation-induced processes is study of model binary alloys, the number of possible crystal-chemical reactions of radiation point defects with sinks in which is limited.

In this work, the experimental verification of the possibility of changing the mechanical properties of Fe-Mo alloy induced by the enhancement of the diffusion-controlled processes under electron irradiation has been carried out.

2. Experimental technique
Investigations were carried out with the use of Fe-5 wt.% Mo alloy. Accordingly to the phase diagram, the temperature of the structure transition in a two-phase state is below 500°C for this alloy, however on account of a small value of the thermal diffusion coefficient at such temperatures (~10^{-18}-10^{-19} cm^2/sec), decomposition of the solid solution does not occurred under normal conditions. The alloy was produced by vacuum casting from 99.9% Fe and 98% Mo. After graduated rolling down to a thickness ~0.2 mm, the resultant foil was annealed in vacuum at 1000°C for 4 hours. The flat specimens of 15×30 mm in size were cut from the rolled foils. Next these specimens were irradiated with 2 MeV electrons (beam current of 200 μA) in air at room temperature and 400°C for 5 hours (fluence of ~10^{18} cm^{-2}). The room temperature (20±5°C) was kept by water jet flashing of specimens, elevated temperature (400±10°C) — by compressed air blow-off of it.

Mechanical properties of the alloy were judged by the results of the Miniaturized Disk Bent Tests (MDBT) of disk specimens of 3 mm in diameter which were cut off from irradiated foils [1-3]. Tests were carried out using a special adjusting device with the help of Instron 3382 test machine by means of specimen indentation with a cylindrical punch of 1 mm in diameter with a loading rate of 0.2 mm/min. Registered “load–specimen deflection” curve allows to make a qualitative estimation of the material mechanical properties changes after irradiation because the shape of this curve is very sensitive to structure and phase state of the material [4]. Altogether in five specimens were tested for the alloy in each state (initial, annealed, and irradiated).

3. Results and discussion
Figure 1 shows the loading curves of Fe-5 wt.% Mo alloy disk microspecimens in initial state and after electron irradiation (2 MeV, ~2·10^{18} cm^{-2}) at room temperature and 400°C. For clarity, in one typical curve for each condition is shown on the figure and these curves are displaced relative to each other. As can be seen, irradiation leads to a reduction of a maximum load to specimen’s failure.
Comparison of all experimental curves for irradiated specimens (which are not shown in the figure) allows concluding about tendency towards lowering the strength characteristics of the alloy with increasing the temperature of irradiation.

![Figure 1](image-url) Loading curves of Fe-5% Mo disk microspecimens in initial state (vacuum annealing, 1000°C/4 h), after electron irradiation at room temperature and 400°C (4 h, 10^{18} cm^{-2}), and after additional vacuum annealing at 400°C/5 h.

However, enhancement of the radiation softening effect with increasing of irradiation temperature up to 400°C could be depend not only from irradiation, but also with the structural changes occurring in the alloy at higher temperatures, for example, with the collapse of the solid solution. To test this hypothesis, initial specimens were subjected to additional vacuum annealing at 400°C for 5 hours, which repeats the temperature and time conditions of irradiation. It was found that the additional annealing of the alloy after preliminary high-temperature heat treatment (1000°C/4 h) at a much lower temperature (400°C) not only leads to a softening of the alloy, as in the case of irradiation at this temperature, but conversely, causes its hardening compared with the alloy in initial state. Sensitivity of the alloy to an additional low-temperature annealing indicates clearly that the pre-annealing at 1000°C was insufficient to obtain an equilibrium structure of the alloy. Moreover, it was found multidirectional effect of irradiation and thermal annealing at the same temperature. Apparently, annealing stimulates the structure transformation causing strengthening of Fe-Mo alloy, and irradiation, on the contrary, — it’s softening.

More explicitly effect of radiation softening of Fe-5% Mo alloy was manifested during measurements of Vickers microhardness conducted on 402MVD (Woolpert Wilson Instrument) under the load on indenter (diamond pyramid with an apex angle of 136°) of 50 g (Figure 2). The depth of the analyzed surface layer was about 10 microns. Total in 5 measurements on the point was carried out and the average values of microhardness for the alloy in each state were calculated. Accuracy of determining the average value was ±5%. As can be seen, with increase of the temperature of annealing or irradiation, microhardness of the surface layer is decreased, and difference between the average values of microhardness of irradiated and annealed at 400°C specimens greater than the measurement error. At the same time, these data show that annealing at 400°C decreases the microhardness of the alloy, while, according to MDBT measurements, additional annealing at 400°C increases the strength characteristics of the alloy, i.e. strengthening effect takes place.
The observed difference in microhardness behavior and changes of the MDBT-curves for irradiated and annealed specimens may be associated with the fact that the microhardness values characterize the strength properties of sufficiently thin surface layer, whereas the MDBT-curves — that of the whole of volume. Obviously, kinetics of radiation point defects annealing near the powerful sink like the surface and in the volume of material will be different and it may have effect on the nature of radiation-stimulated processes occurring near the surface and in the volume.

Despite the low amount of radiation-induced change of mechanical properties of Fe-5 wt.% Mo alloy, the data obtained convincing enough demonstrate the radiation nature of the alloy softening effect, especially at elevated temperature. Undoubtedly, firstly, the reason of change in mechanical properties of Fe-Mo alloy under electron irradiation is radiation-induced structure and/or phase transformations and, secondly, behavior of these processes is different in the volume and near the surface. It may be related to the different kinetics of accumulation and annealing of radiation point defects as well as with different crystal-chemical reactions (e.g., oxidation) on the surface of irradiated specimen as it is cooled by water or air jet.

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4. References
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