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Journal of Nuclear Materials 249 (1997) 250–258

journal of nuclear materials

The effect of neutron irradiation on the mechanical properties of precipitation hardened copper alloys

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Received 15 November 1996; accepted 9 May 1997

Abstract

The effects of neutron irradiation on strength and ductility properties of precipitation hardened (PH) copper alloys are discussed. The analysis is based on the experimental study of radiation damage of PH alloys irradiated in the mixed spectrum reactor SM-2 to fluences of $3.7–5.5 \times 10^{25}$ n/m^2 ($E > 0.1$ MeV), corresponding to NRT displacement dose levels of 2.6–3.8 dpa. At irradiation temperatures of 100–285°C the processes of radiation hardening and reduction in the uniform elongation are the major effects. Irradiation at temperatures higher than 300°C causes a dramatic softening and improvement in uniform elongation of the Cu–Cr–Zr and Cu–Cr–Zr–Mg alloys. The threshold softening temperature for the PH alloys is shown to be about 300°C at a dose of 4.5×10^{25} n/m^2 ($E > 0.1$ MeV). The effect of the irradiation dose and temperature on the shift of the threshold temperature of PH copper-alloys softening is also considered.

1. Introduction

Precipitation hardened (PH) copper alloys are currently considered as one of the candidate materials for the ITER heat sink system [1]. The main advantage of PH alloys is their high strength combined with a high thermal conductivity (80–90% of thermal conductivity of pure copper). Besides, they are manufacturable and commercially available. Most of the data on the radiation resistance of these alloys were obtained mainly by irradiating specimens in fast neutron reactors at $T_{irr} > 380$°C [2–4]. In these studies the PH alloys were shown to have a high resistance to void swelling up to doses of 63 dpa at $T_{irr} = 385–450$°C, while retaining a rather high level of electrical conductivity (up to 60% of electrical conductivity of pure copper).

The assumed operation temperature of copper alloys in the ITER first wall and divertor is $\simeq 100–300$°C. Thus, the data obtained in fast neutron reactors, i.e., at $T_{irr} \geq 340$°C are not suitable to substantiate the workability of copper alloys for ITER. Few data are available on the effect of irradiation in mixed spectrum reactors at $T_{irr} \simeq 100–300$°C on the properties of high-strength copper alloys [5–7]. Irradiation of the Cu–Cr–Zr alloy in the reactor BR-II [5] at 150 and 300°C up to $\simeq 5$ dpa resulted in embrittlement and hardening of the alloy. The same effect was observed in Ref. [6] after irradiation in the HFR reactor at 150 and 250°C up to a dose of $\simeq 10$ dpa.

Our previous studies [8,9] presented the results of the irradiation effect in the reactor SM-2 up to doses of 0.5–0.7 dpa in the temperature range of 110–450°C on the properties of the alloys Cu–Cr–Zr and Cu–Cr–Zr–Mg. They revealed that irradiation at $T_{irr} \simeq 400–450$°C caused complete softening of the alloys Cu–Cr–Zr and Cu–Cr–Zr–Mg.

It is worth noting that the works [8,9] as well as most others [2–4] were devoted mainly to the problem of changes in the strength properties of the PH alloys, whereas the data on changes in the deformation behavior of these alloys (particularly at $T_{test} = T_{irr}$) are scarce in the literature.

This paper deals with the behavior of the PH alloys irradiated up to 3 dpa at $T_{irr} = 160–390$°C. The results presented here make it possible to close the existing gap in

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0022-3115/97/$17.00
PII S0022-3115(97)00145-1

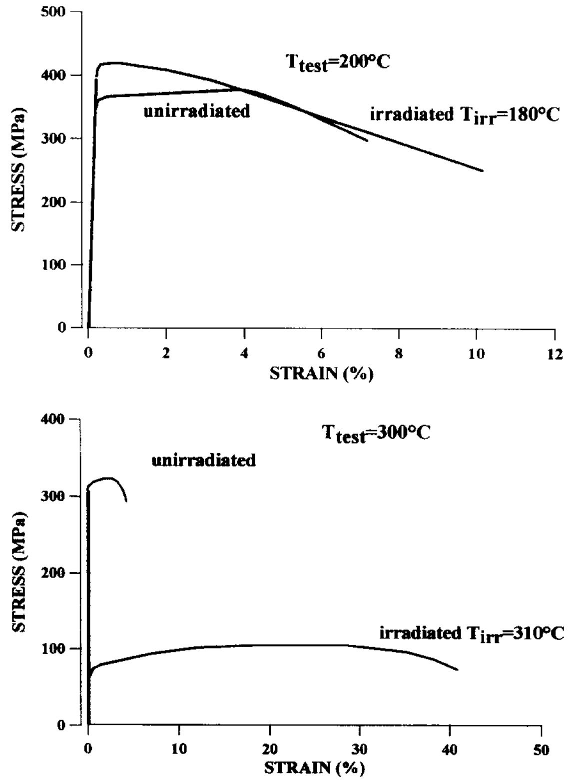

Fig. 1. Stress–strain curves for Cu–Cr–Zr alloy, unirradiated and irradiated up to 3 dpa at $T_{irr} \simeq 180$°C (a) and $T_{irr} \simeq 310$°C (b). $T_{test} = 200$°C (a) and $T_{test} = 300$°C (b);

the dose dependence of the PH alloys in the operation temperature range for ITER. Besides, the investigation of samples irradiated at temperatures of 300–390°C helps in investigating the softening of the PH alloys. It is worth noting that up until now there have been no data on this critical aspect of the PH alloys workability at doses ≥ 1 dpa.

2. Experimental procedure

In this work the precipitation hardened alloys Cu–Cr–Zr and Cu–Cr–Zr–Mg have been investigated. The composition and heat treatment of alloys are presented in Table 1. The grain size in the alloys was $20–30 \times 10^{-6}$ m. The structure is characterized by a high (10^{15} m^{-2}) density of unevenly distributed dislocations. Precipitates have an average size of $\simeq 5$ nm uniformly distributed in the matrix. The cylindrical tensile specimens had an overall length of 26 mm, with a gage length and diameter of 10 and 3 mm, respectively.

The specimens were irradiated in the SM-2 reactor to doses of $3.7–5.5 \times 10^{25}$ n/m^2 ($E > 0.1$ MeV), corresponding to NRT displacement dose levels of 2.6 to 3.8 dpa (neutron flux was 3×10^{18} n/m^2 s) at $T_{irr} \simeq 160$, 180, 185, 240, 285, 310, 375 and 390°C. The irradiation was performed in SM-2 in special tubular capsules. The tubes were filled with helium. The irradiation temperature regime of specimens was measured by thermocouples.

The irradiated and control samples were tensile tested in vacuum at a crosshead speed of $\simeq 1$ mm/min (corresponding to a strain rate of 1.66×10^{-3} s^{-1}) in the temperature range of 100–500°C. Two specimens were tested for each irradiation condition. The typical time needed to attain 400°C in the testing machine was as much as 1 h. Hold time during elevated temperature tensile tests was 30 min. The structure of the samples was investigated by optical microscopy.

3. Experimental results

3.1. The effect of neutron irradiation on stress strain curves of PH alloys

Fig. 1a shows typical stress strain curves for the unirradiated Cu–Cr–Zr alloy tested at 200°C and for the same alloy irradiated up to 3 dpa at 180°C and tested at 200°C. It is evident that the low-temperature irradiation results in a slight hardening of the Cu–Cr–Zr alloy and a drastic reduction in its uniform elongation. In this case the total elongation of the irradiated alloy is even somewhat higher than in an unirradiated alloy.

Fig. 1b gives typical stress strain curves for the unirradiated Cu–Cr–Zr alloy tested at 300°C and for the same alloy irradiated up to 3 dpa at 310°C and tested at 300°C. It is obvious that irradiation at an increased temperature involves in a drastic drop in the yield strength of the Cu–Cr–Zr alloy and an increase in its uniform elongation by more than a factor of 10.

Table 1
Chemical composition of PH copper alloys (wt%)

No.	Alloy	Heat treatment	Cr	Zr	Mg	C	S	P	Si	Cu
1	Cu–Cr–Zr	solution, annealed at 980°C quenched, cold worked 50%, aged at 480°C, 4 h	0.59	0.1	–	0.01	0.003	< 0.005	< 0.004	Bal
2	Cu–Cr–Zr–Mg	solution, annealed at 980°C quenched, cold worked 50%, aged at 465°C, 4 h	0.53	0.22	0.08	0.01	0.005	< 0.005	< 0.004	Bal

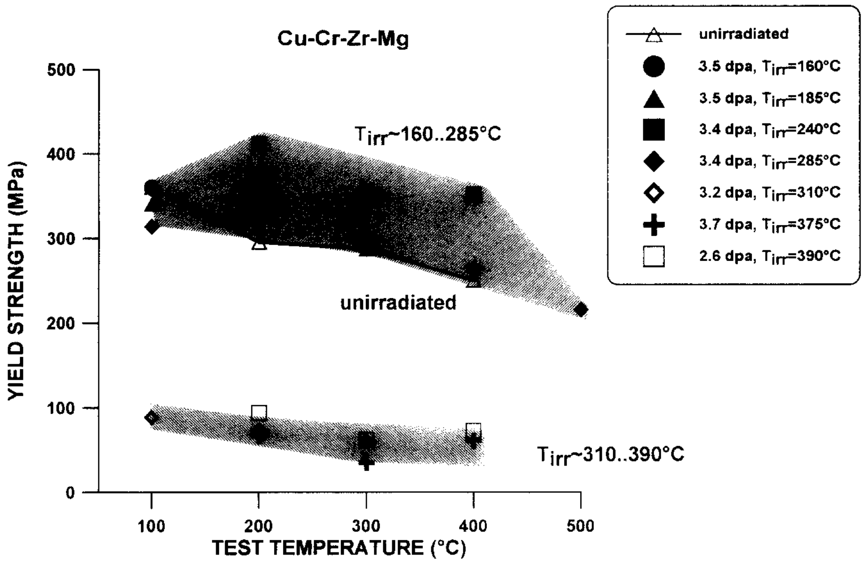

Fig. 2. Effect of neutron irradiation on the yield strength of Cu–Cr–Zr–Mg alloy. Irradiation in SM-2 up to 2.6–3.7 dpa at $T_{irr} \simeq 160–390°C$.

3.2. The effect of neutron irradiation on yield strength of PH alloys

As seen from Fig. 2, irradiation at $T_{irr} \simeq 160–285°C$ results in a weak radiation hardening of the Cu–Cr–Zr–Mg alloy throughout the testing temperature range. In going to higher irradiation temperatures the alloy softens. At T_{irr} = 310–390°C the yield strength of the alloy Cu–Cr–Zr–Mg is 80–40 MPa throughout the testing temperature range, i.e., it is close to the yield strength of pure copper. In this study the yield strength was assessed as $\sigma_y = \sigma_{0.2}$. Standing out is the critical character of this transition. At T_{irr} = 240 and 285°C the samples irradiated to 3 dpa do not demonstrate any tendency for softening. At T_{irr} = 310°C and higher the irradiated samples are completely softened.

The yield strength behavior of the Cu–Cr–Zr alloy, as follows from Fig. 3, is of the same character. A slight hardening is observed at T_{irr} = 180–280°C and total soft-

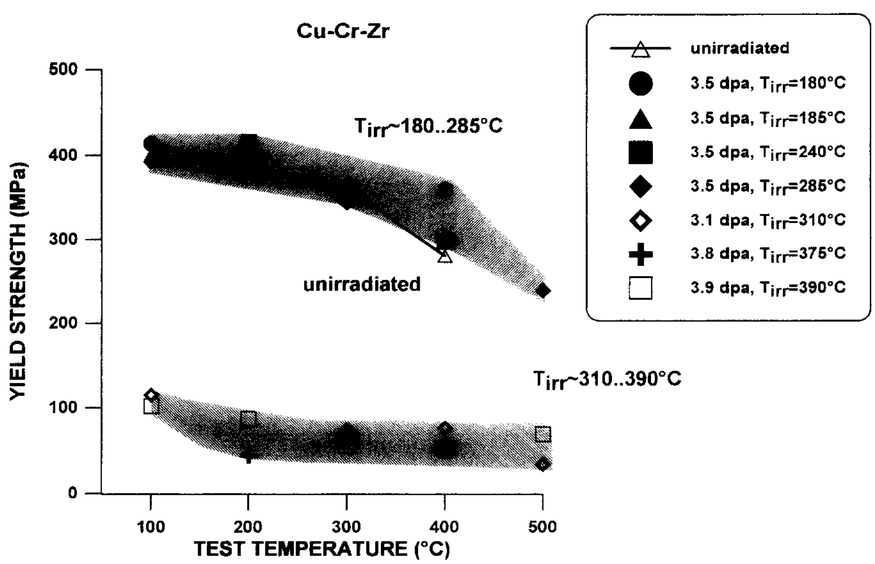

Fig. 3. Effect of neutron irradiation on the yield strength of Cu–Cr–Zr alloy. Irradiation in SM-2 up to 3.1–3.9 dpa at $T_{irr} \simeq 180–390°C$.

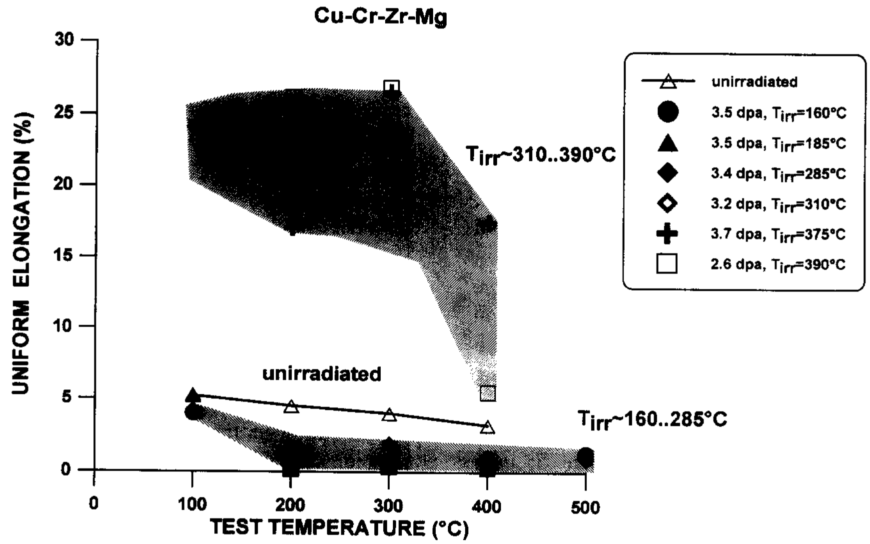

Fig. 4. Effect of neutron irradiation on the uniform elongation of Cu–Cr–Zr–Mg alloy. Irradiation in SM-2 up to 2.6–3.7 dpa at $T_{irr} \simeq$ 160–390°C.

ening at $T_{irr} \geq 310$°C. After the radiation softening the Cu–Cr–Zr–Mg alloy has, as does the Cu–Cr–Zr alloy, an extremely low yield strength up to $\simeq 40$ MPa (at the level of pure copper).

The analysis of both Figs. 2 and 3 allows for the conclusion that, prior to softening, the yield strength of the irradiated PH alloys demonstrates a typical progress of the temperature dependence, i.e., a decrease in σ_y with an increase in T_{test} (as in the case of unirradiated samples). For the softened PH copper alloys irradiated at $T_{irr} \geq 310$°C the temperature dependence of σ_y degenerates. The yield strength of irradiated samples is practically the same throughout the testing temperature range of 100–400°C.

3.3. The effect of neutron irradiation on uniform elongation of PH alloys

As follows from Fig. 4, irradiation up to 3 dpa at 160–285°C results in a reduction of the uniform elongation of Cu–Cr–Zr–Mg alloy samples. An unirradiated sample in the testing temperature range of 200–500°C has δ_{un} from 4 to 2.5%, whereas in irradiated samples δ_{un} is

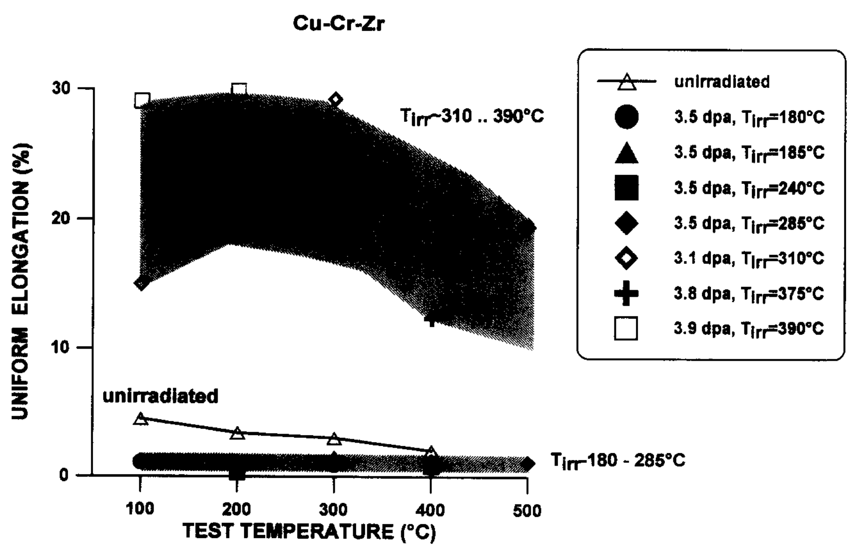

Fig. 5. Effect of neutron irradiation on the uniform elongation of Cu–Cr–Zr alloy. Irradiation in SM-2 up to 3.1–3.9 dpa at $T_{irr} \simeq$ 180–390°C.

0.3–1.5% in the same testing temperature range. Irradiation to 3 dpa at $T_{irr} \geq 310°C$ increases the uniform elongation of the alloy. The uniform elongation increases to a level of 5–25% throughout the testing temperature range $T_{test} = 100–400°C$.

Fig. 5 demonstrates that the behavior of the uniform elongation of the Cu–Cr–Zr alloy is, by and large, the same. After irradiation at $T_{irr} = 180–285°C$ embrittlement is observed, with the uniform elongation remaining extremely low, i.e., 0.5–0.9% throughout the testing temperature range of 100–500°C. Transition to high irradiation temperatures $T_{irr} = 310–390°C$ gives rise to a growth in the uniform elongation of irradiated samples at all testing temperatures $T_{test} = 100–500°C$. In this case the level of the uniform elongation is very high, i.e., $\delta_{un} \simeq 12–28\%$. This level of the uniform elongation is typical of pure copper.

Note, that the behavior of the total elongation is, on the whole, similar to that of the uniform elongation. The samples of both alloys irradiated at $T_{irr} = 160–285°C$ have a somewhat lesser level of $\delta_{tot} = 7–10\%$, as against 13–15% in unirradiated samples. With $T_{irr} = 310–390°C$ increased, the samples have a total elongation of 25–40%, i.e., samples are fractured after a considerable local deformation.

3.4. The effect of neutron irradiation on the microstructure of PH copper alloys

The results of the investigation into the structure of irradiated samples by means of optical metallography reveal that irradiation at low irradiation temperatures of 160–285°C does not practically affect the structure of both alloys. As seen from Fig. 6a and b, the metallographic structures of the Cu–Cr–Zr alloy in the initial state and after irradiation at 285°C up to 3 dpa appears to be similar. After high-temperature irradiation the recrystallization processes develop in the PH alloys (Fig. 6c).

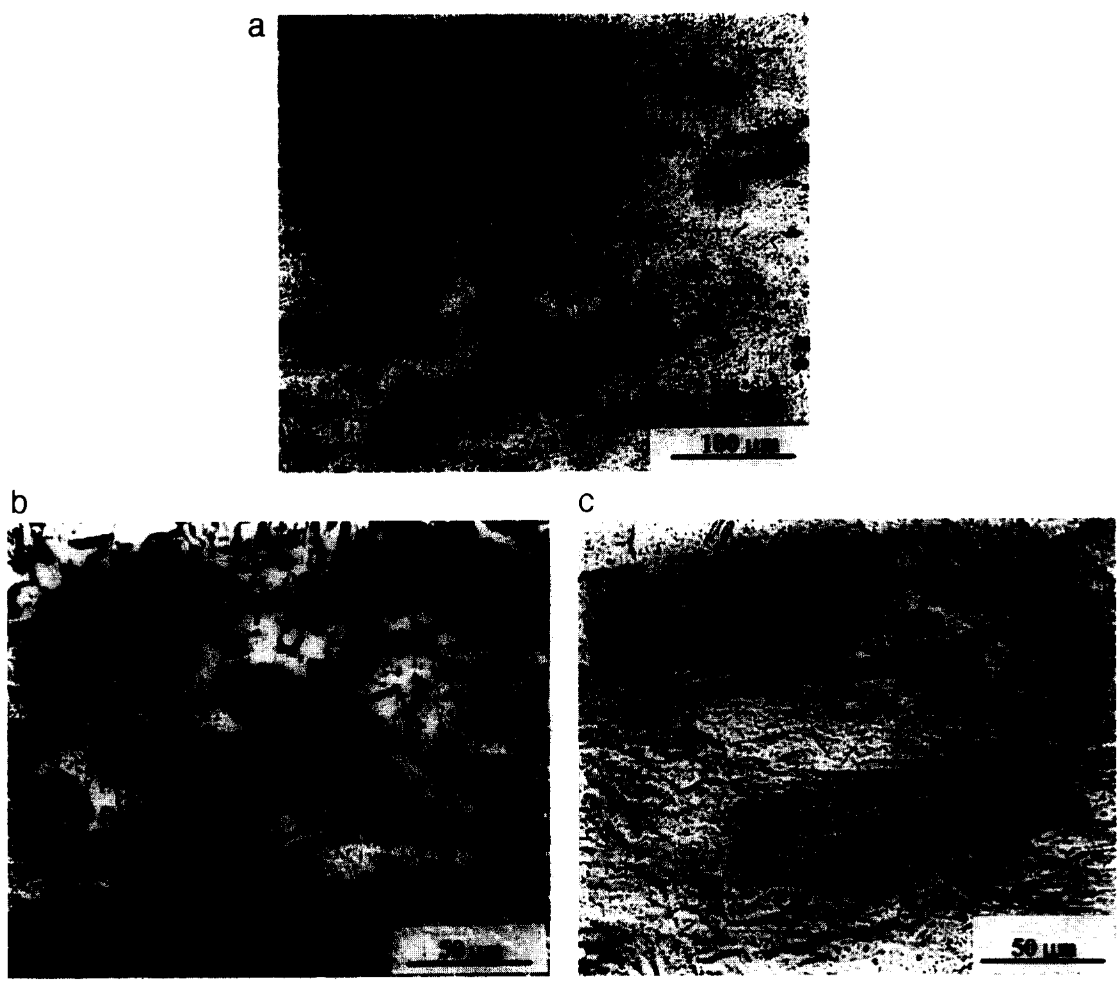

Fig. 6. Optical microstructure of the Cu–Cr–Zr–Mg alloy: (a) unirradiated, (b) 3.4 dpa, $T_{irr} = 285°C$, (c) 3.8 dpa, $T_{irr} = 375°C$.

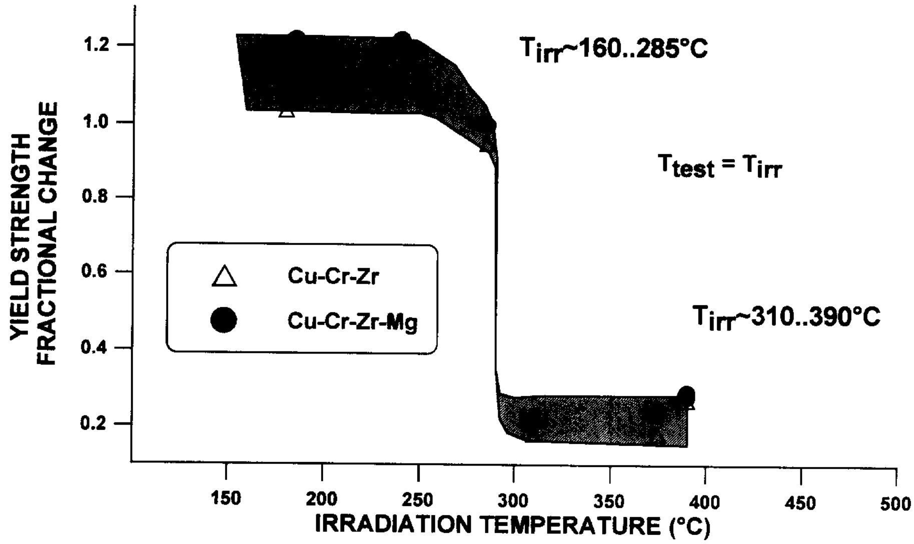

Fig. 7. Irradiation temperature effect on the yield strength fractional change $\sigma_{Y_{irr}}/\sigma_{Y_{unirr}}$ of Cu–Cr–Zr, Cu–Cr–Zr–Mg PH copper alloys, irradiated in SM-2 reactor up to 2.6–3.9 dpa and tested at $T_{test} - T_{irr}$.

The metallographic investigations of samples irradiated and tested at elevated temperatures revealed that the fracture of samples is of ductile and transcrystalline character, the samples fail after a considerable deformation with the formation of a noticeable neck.

4. Discussion

When constructing the dependence of a relative change in the yield strength and uniform elongation for the Cu–Cr–Zr and Cu–Cr–Zr–Mg alloys on the irradiation temperature (Figs. 7 and 8), it became evident that a sudden change in the irradiation effects on the mechanical properties occurs at a temperature of 300°C.

4.1. The effect of low-temperature irradiation

At relatively low irradiation temperatures, i.e., 160–285°C, the irradiation results in strengthening and reduction in uniform elongation of the PH-type copper alloys. Strengthening of the PH alloys at relatively low-temperature irradiation is associated with the formation of radia-

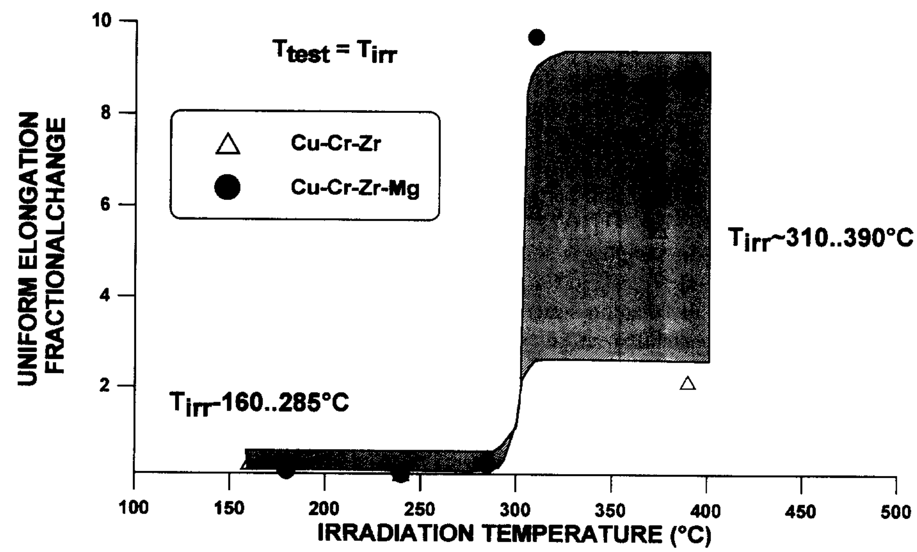

Fig. 8. Irradiation temperature effect on the uniform elongation fractional change $\delta_{uniform\ irr}/\delta_{uniform\ unirr}$ of Cu–Cr–Zr, Cu–Cr–Zr–Mg PH copper alloys, irradiated in SM-2 reactor up to 2.6–3.9 dpa and tested at $T_{test} - T_{irr}$.

tion defect complexes (loops, black dots) in the alloy matrix [10–12]. In this case the dislocation structure and density of small strengthening particles do not practically change [10,11]. The presence of defect clusters which act as obstacles for dislocations in the matrix, defines the strengthening. In Ref. [12] the essential features of LTE (low temperature embrittlement) of the PH copper alloys are analyzed in detail and the largest strengthening and embrittlement are shown to be observed at $T_{irr} = 90–110°C$. At higher irradiation temperatures (180°C) an increase in the yield strength of irradiated samples becomes rather insignificant and the uniform elongation is also increased [12]. On the whole, this effect is a typical manifestation of the low-temperature radiation embrittlement of fcc and bcc materials and is investigated sufficiently well, particularly for austenitic steels.

4.2. The effect of high-temperature irradiation

Softening at increased irradiation temperatures is sure to be the most important irradiation effect on the PH copper alloy properties observed in this study. At T_{irr} = 310–390°C the PH copper alloys are abruptly softened (up to a level typical of pure copper $\sigma_y = 70$ MPa) and improved in uniform elongation (up to $\delta_{un} = 20\%$) also to the level typical of pure copper.

The optical microscopy of irradiated samples carried out in this study confirms that at T_{irr} = 310–390°C the PH copper alloys change their structure. After high-temperature irradiation the recrystallization processes develop in the PH alloys.

The TEM investigations of the samples from the Cu–Cr–Zr–Mg alloy irradiated in the SM-2 reactor at temperatures of 230, 300 and 375°C undertaken in Refs. [10,11] confirm that softening may be controlled by the processes of the radiation-stimulated degradation of the strengthening structure, though the long-term annealing during irradiation does play a part in the decay of the strengthening structure. The PH-type Cu–Cr–Zr and Cu–Cr–Zr–Mg alloys, when in the initial state, were characterized by a high density (10^{21} m^{-3}) of small-sized (2–5 nm) particles and a high density of dislocations (10^{15} cm^{-2}) [10,11].

As a rule, the dislocation density in samples is somewhat reduced after irradiation and at T_{irr} = 230°C, the tendency develops for the formation of subgrain structure elements. In this case the density of small-sized precipitates does not practically change [10,11].

The TEM investigations of the samples from the Cu–Cr–Zr–Mg alloy irradiated in the SM-2 reactor at temperatures 375°C undertaken in Refs. [10,11] confirm that after high-temperature irradiation a dramatic change is observed in the material structure. The PH alloys undergo complete polygonization, the strengthening particles grow and coarsen, large particles emerge on the grain boundaries and there occurs the overall drop in the density of the strengthening particles [10,11]. The decay of the strengthening structure of the material determines in the long run its softening.

The data on a slight drop in the strength characteristics of various PH copper alloys, when exposed to high-temperature irradiation in fast neutron reactors, were published in Refs. [2–4]. Thus, for example, the AmZirc (Cu–Zr) alloy was softened to a level of ≃ 70 MPa after irradiation at 385°C (in EBR-II) to 2×10^{26} n/m^2 ($E > 0.1$ MeV). But it should be noted, that the tests in these works were carried out at room temperature. Yet, the properties of materials at $T_{test} = T_{irr}$ remained unknown. In Ref. [2] the reference samples of the MZC (Cu–Cr–Zr–Mg) alloy annealed at T_{ann} = 450°C for 1000 h were thermally investigated and it was shown that annealing did not practically cause a drop in the strength properties at T_{test} = 20°C, while irradiation at 450°C in FFTF up to 2.5×10^{26} n/m^2 ($E > 0.1$ MeV) for 1000 h resulted in a considerable drop in the yield strength of the alloy at T_{test} = 20°C [2]. A similar result was obtained in Ref. [13], where the annealing of the alloys Cu–Cr–Zr and Cu–Cr–Zr–Mg at T_{ann} = 450°C for 900 h was shown to affect only slightly the strength properties at T_{test} = 20 and 300°C, though the same materials were completely softened under irradiation at 400°C in SM-2 up to 1×10^{25} n/m^2 ($E > 0.1$ MeV) within about 1000 h.

Note, that the type of strengthening particles was investigated in several studies [3,14,15]. The microdiffraction investigations and X-ray analysis allow for the conclusion that these particles are composed of pure chromium with the bcc structure. It is essential that these particles in unirradiated alloys are coherent with the matrix. In irradiated softened samples large Cr particles become incoherent with the matrix, their size increases (from 2–10 nm, when unirradiated, to 30–40 nm, when irradiated), hence, the density of precipitates is decreased [10,15].

Thus, the analysis of the available microstructure data [3,10,11,15] on the softening of the PH copper suggests, that this effect may be controlled by the processes of the radiation-stimulated dissolution of small Cr-precipitates and thermal annealing (precipitate overaging) effects. Undoubtedly, the main factor determining the softening is a drop in the density of strengthening Cr particles during radiation-stimulated overaging of precipitation-hardened copper alloys.

This naturally raises the question of whether the critical temperature of softening depends on the irradiation dose. As follows from the results of our previous study [8], the Cu–Cr–Zr–Mg alloy, when irradiated to a dose of 0.7 dpa at T_{irr} = 310°C, was not yet softened and had at T_{test} = 300°C a yield strength of 270 MPa. At the same time this alloy, when irradiated at T_{irr} = 300°C to 3.5 dpa, softened to a level of 70 MPa. Thus, the critical temperature of softening under irradiation is likely to depend on the irradiation dose and goes into the area of lower temperatures with the dose increased.

Of interest is the comparison of our results with those obtained in other two studies, where the effect of irradiation to large doses, i.e., 5–10 dpa, was investigated in mixed-spectrum reactors [5,6]. It is worth noting, that the behavior of the Cu–Cr–Zr alloy irradiated to 5 dpa at 150 and 300°C [4] and to 10 dpa at 150 and 250°C [5] correlates well with the results of this study. At relatively low irradiation temperatures, i.e., 150°C, the Cu–Cr–Zr alloy strengthens and loses ductility. At higher irradiation temperatures, i.e., 250–300°C, the Cu–Cr–Zr alloy strengthens only slightly and its uniform elongation increases, and in some cases, reaches the initial level.

On the basis of the results of this study and the analysis of the available data on the behavior of the PH alloys under irradiation [5–9] it may be concluded that in the dose range of 0.2–10 dpa at T_{irr} = 150–285°C the PH copper alloys like Cu–Cr–Zr, Cu–Cr–Zr–Mg retain their high strength properties at a level of the initial materials and effectively resist to softening.

The analysis of the temperature dependence of the mechanical properties of the PH alloys obtained in this study makes possible the conclusion that the structure produced under irradiation to 3 dpa at T_{irr} = 160–285°C is very stable and quite resistant. Thus, the high-temperature deformation (T_{test} = 500°C) of samples irradiated at 160–285°C does not result in their softening, as well as no recovery of plasticity is observed. This circumstance is of great practical importance. Short-term overheating, local high-temperature deformation of a copper tile, when operated, for example, in the ITER divertor, will not cause its instant softening. Of particular importance is that this stability is observed after considerable irradiation doses, when up to 50 appm helium is accumulated in copper alloys because of the hard ITER spectrum. If such a helium-content material were instantly softened after a short-term heating, helium accumulation on the boundaries of growing grains would give rise to intergranular cracks immediately at the overheating moment. But the results obtained in this study provide strong evidence, that the structure produced at a relatively low-temperature irradiation of 160–285°C is highly resistant to the recovery processes up to 500°C. We think this fact to be critical for practice, which means the tendency. The real effect of short-term overheating on Cu–Cr–Zr strength (plasma disruptions, etc.) under operation conditions calls for a separate study.

5. Conclusions

It follows from the results of the study that in the operation temperature range of the ITER heat sink system (100–350°C) two essentially different processes will control a change in the mechanical properties of the PH copper alloys. At irradiation temperatures of 100–285°C these are the processes of radiation hardening and the reduction of the uniform elongation. It should be noted that at temperatures of 160–285°C the embrittlement manifests itself weaker than at lower irradiation temperatures. Irradiation at temperatures higher than 300°C causes a dramatic softening and improvement in uniform elongation of the Cu–Cr–Zr and Cu–Cr–Zr–Mg alloys.

The comparison of the present results on changes in the mechanical properties of the PH alloys irradiated up to 3.5 dpa with the results of our previous investigations [8,9], where samples of the same alloys were irradiated to a dose of 0.7 dpa, indicates that the critical temperature of softening may decrease with an increase in the dose.

It has been shown that the microstructure produced under irradiation to 3.5 dpa at T_{irr} = 160–285°C is stable and resistant to high-temperature recovery. The deformation of such samples in the absence of irradiation at 500°C does not give rise to softening.

On the whole, of major importance is the conclusion following from the results of this work. In the temperature range typical of the operation temperatures of the ITER heat sink system, i.e., 160–285°C, the Cu–Cr–Zr alloy has highly stable and slightly varying mechanical properties (a sufficiently high yield strength and a satisfactory level of the uniform elongation), when irradiated in the dose range of 0.5–3.5 dpa.

Acknowledgements

The authors thank V.P. Chakin for providing help in tensile tests and V.R. Barabash and S.J. Zinkle for fruitful discussions.

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