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Effect of irradiation temperature on microstructure, radiation hardening and embrittlement of pure copper and copper-based alloy

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Abstract

Low-temperature radiation embrittlement is one of the main negative consequences of neutron irradiation for pure copper and copper-based alloys. But currently available data on copper radiation hardening and embrittlement have been obtained in the temperature range $T_{irr} = 60–90$ °C. Systematic data on the effect of irradiation temperature in the range of radiation hardening and embrittlement (50–200 °C) are lacking. This paper presents the results of the analysis of two experiments on irradiation of pure copper and GlidCop Al25IG alloy in the RBT-6 reactor at irradiation temperatures of 80 °C and 150 °C. The irradiation dose range was 10^{-3}–10^{-1} dpa. The comparison between the dose dependencies of materials hardening and embrittlement revealed that a rise in temperature causes hardening to drop and embrittlement to decrease. The microstructure data on the density and size of complexes in irradiated materials served as the basis for calculations of the level of radiation hardening, with the Orowan–Seeger model used.

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1. Introduction

Low-temperature radiation embrittlement is one of the main negative consequences of neutron irradiation for pure copper and copper-based alloys. But much of the currently available data on copper radiation hardening and embrittlement has been obtained at temperatures in the range $T_{irr} = 60–90$ °C. Systematic data on the effect of irradiation temperature in the range of radiation hardening and embrittlement (50–200 °C) are lacking.

In 2004–2005 the authors [1,2] obtained the dose dependence of radiation hardening for pure copper and GlidCop Al25 alloy under irradiation at $T_{irr} = 80$ °C [1] and $T_{irr} = 150$ °C [2]. TEM investigations were carried out [1,2] to study the effect of irradiation dose on the size and density of defect clusters. The results obtained in [1,2] allow for systematic comparison of the irradiation temperature effect on the yield strength and strain hardening coefficient (SHC) for pure copper and GlidCop Al25. This paper presents the results of the analysis of two experiments on irradiation of pure copper and GlidCop Al25IG alloy in the RBT-6 reactor at irradiation temperatures of 80 °C and 150 °C. The irradiation dose range was 10^{-3}–10^{-1} dpa.

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2. Experimental procedure

Specimens of pure Cu (99.997%) and copper alloy GlidCop Al25 were irradiated in the RBT-6 reactor. Prior to irradiation specimens of pure Cu were annealed in vacuum at 550 °C for two hours and GlidCop Al25 alloy specimens were annealed at 1000 °C for one hour after cross rolling. Flat specimens for tension (1 mm in thickness) were irradiated in tight helium-filled capsules.

The irradiation doses in the RBT-6 reactor reached to 10^{-3}, 10^{-2} and 10^{-1} dpa. Irradiation temperature, as recorded by thermocouples, was 75 ± 5 °C for 1st irradiation and 150 ± 10 °C for 2nd irradiation. Average ratio of thermal to fast neutrons in the RBT reactor core was $\Phi t_{\mathrm{therm}}/\Phi t_{\mathrm{fast}} \sim 1.04$. The irradiation technique is described in detail in [1,2]. The irradiated and unirradiated specimens were tested in tension ($\varepsilon \sim 1.66 \times 10^{-3}\ \mathrm{s}^{-1}$) at $T_{\mathrm{test}} = T_{\mathrm{irr}}$. The TEM structure of irradiated specimens was investigated.

2.1. Results

2.1.1. Effect of irradiation temperature on mechanical properties

Fig. 1 shows the dose dependence of radiation hardening for pure copper and GlidCop Al25 at $T_{\mathrm{irr}} = 80$ °C and $T_{\mathrm{irr}} = 150$ °C. It should be noted that in unirradiated samples an increase in the testing temperatures in the temperature range of 80–150 °C does not practically affect the yield strength for pure copper and GlidCop Al25. An increase in the irradiation temperature from 80 °C to 150 °C

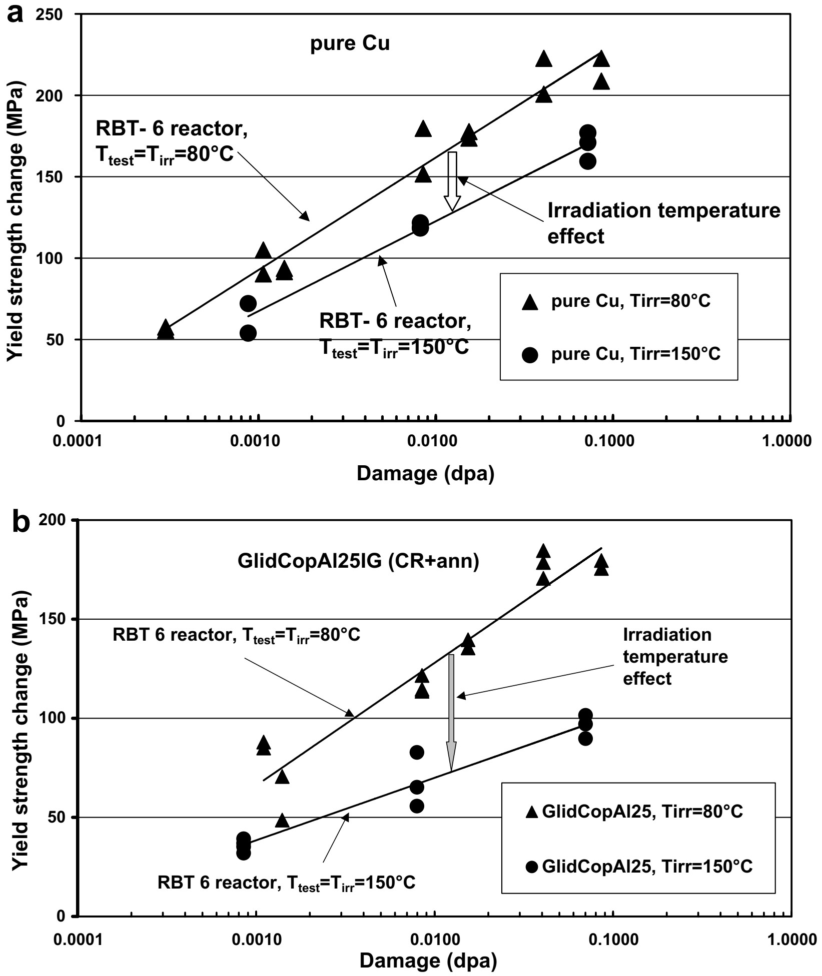

Fig. 1. Change in the yield strength plotted versus neutron damage: for pure Cu: as irradiated at 80 °C and 150 °C (a); for GlidCop Al25 alloy: as irradiated at 80 °C and 150 °C (b); RBT-6 reactor, $T_{\mathrm{test}} = T_{\mathrm{irr}}$.

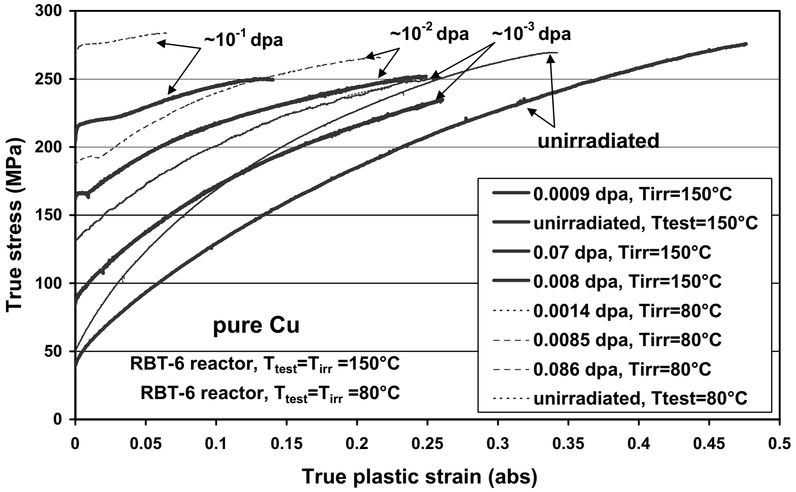

Fig. 2. Effect of irradiation temperature on the true stress–strain curves of pure Cu, RBT-6 reactor, $T_{test} = T_{irr}$.

results in a drop in radiation hardening of pure copper and GlidCop Al25 alloy (Fig. 1(a) and (b)).

The true stress–strain curves obtained from the testing results at $T_{test} = T_{irr} = 150$ °C and $T_{test} = T_{irr} = 80$ °C for pure Cu show that a rise in the irradiation temperature decreases both radiation hardening and radiation embrittlement and does not practically affect the curve slope for specimens irradiated to similar doses at 80 °C and 150 °C (Fig. 2). Yield drop is observed at both irradiation temperatures, at irradiation doses 10^{-1} and 10^{-2} dpa in pure copper.

2.1.2. *Effect of irradiation temperature on defects complex density and size*

The TEM investigations were carried out mainly for pure Cu [1,2]. The TEM investigations of the structure of pure copper specimens irradiated at 80 °C in RBT-6 [1] revealed a high density of defect clusters (Fig. 3(a) and (b)). The defect density is increased with the irradiation dose and is as high as $\sim 1\times 10^{23}$ 1/m^3 at a dose of 10^{-1} dpa. The analysis of the structure of defect clusters showed that they could be divided into two types, namely, stacking fault tetrahedra (SFT) and dislocation loops (DL) (Fig. 3(b)). This structure of defect clusters is typical of pure copper irradiated at 60–100 °C and was observed in many studies [3,4]. As reported in [4,5], SFT are vacancy-type clusters and DL are clusters of interstitial type. Table 1 summarize the results of measurements for the defect cluster density in irradiated pure Cu at $T_{irr} = 80$ °C [1] and 150 °C [2], obtained on the basis of processing of TEM images.

After irradiation at 150 °C in RBT-6, the TEM structure of pure copper is, on the whole, similar to the structure of pure copper irradiated at 80 °C. A high density of SFT is observed (Table 1). Assessment of the SFT density in pure copper specimens irradiated at $T_{irr} = 150$ °C revealed that an increase in the irradiation temperature increases but only slightly the size, and does not significantly affect the density of defect clusters.

3. Discussions

The experimental data obtained make it possible to analyze the mechanism responsible for the reduction of radiation hardening at higher irradiation temperatures. Irradiation can affect two key parameters determining the strength and ductility of materials. First, the defect clusters pin the dislocations and prevents the latter from acting as dislocation source causing the yield strength to increase [6]. Second, the defect clusters also suppress dislocation multiplication and dislocation network generation causing the strain hardening coefficient (SHC) to drop and, hence, the uniform elongation to reduce [7].

Let us analyze the obtained experimental results and estimate which parameter depends more on the irradiation temperature.

To assess quantitatively the effect of irradiation temperature on the increase in yield strength and on the ability of copper to harden (described by the

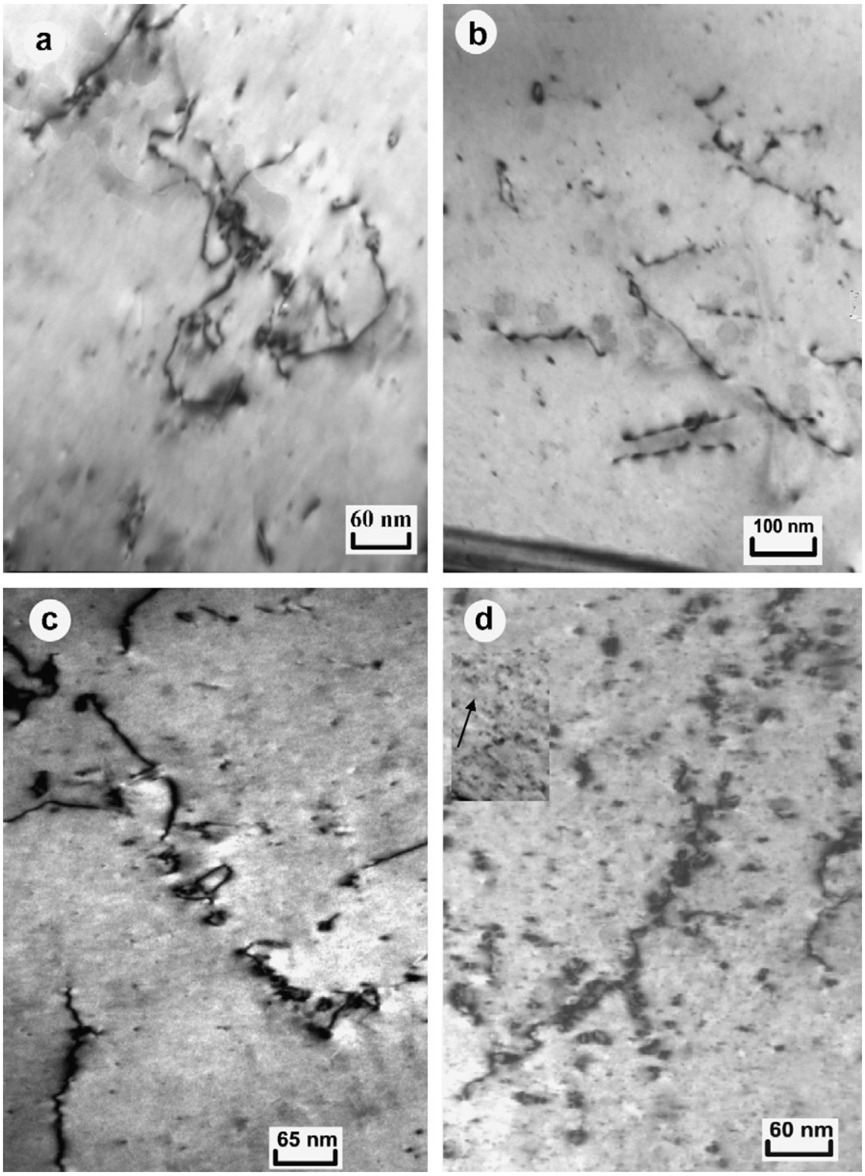

Fig. 3. Loop and SFT microstructures of pure Cu: as irradiated at $T_{irr} = 80$ °C to 0.0001 dpa (a); as irradiated at $T_{irr} = 80$ °C to 0.001 dpa (b); as irradiated at $T_{irr} = 150$ °C to 0.0009 dpa (c); as irradiated at $T_{irr} = 150$ °C to 0.008 dpa (d).

Table 1
Defects complex density and size in irradiated pure Cu

Material	Irradiation temperature, °C	Dose range, dpa	Damage (dpa)	Density SFT, 10^{22} m^{-3}	Mean diameter SFT, nm	Density dislocation loops, 10^{22} m^{-3}	Mean diameter loops, nm	References
Pure Cu	80	10^{-2}	0.01540	8.08	2.36	0.430	9.43	[1]
	80	10^{-3}	0.00107	3.93	2.67	0.360	13.22	[1]
	80	10^{-4}	0.00010	0.96	2.82	0.168	11.97	[1]
	150	10^{-1}	0.0721	28.20	3.05	0.505	14.30	[2]
	150	10^{-2}	0.00819	11.60	2.90	0.677	13.11	[2]
	150	10^{-3}	0.00088	2.55	2.60	0.212	9.80	[2]

SHC) we processed the true stress–strain curves of pure Cu using the methodology developed by the authors and reported in [7]. According to this methodology, the digitized data obtained during testing are processed by regression analysis methods to obtain the coefficients in the analytical equations describing the true stress–true plastic strain curve with the best approximation to the experimental curve. We processed the true stress–strain diagrams of pure Cu and defined the coefficients **a** and **b** describing the behavior of pure Cu using the equation:

$$\sigma_{\text{true}} = \mathbf{a} * \varepsilon_{\text{true}}^{0.5} + \mathbf{b}. \tag{1}$$

Construction of the dose dependencies of the coefficients **a** and **b** for irradiation in RBT-6 at 80 °C and at 150 °C (Fig. 4) allows for the conclusion that irradiation at a low temperature (80 °C) produces a considerable gain in the parameter **b** (corresponding to the yield strength) and little affect on the parameter **a** (corresponding to SHC) in the low dose range of $\sim 10^{-3}$–10^{-1} dpa. Thus, processing of the curves showed that the irradiation temperature affects, first of all, the yield strength of pure copper.

The observed level of radiation hardening can be compared with the calculations, where the hardening value by the Orowan mechanism is defined by the formula:

$$\Delta\sigma = \alpha\mu Mb(Nd)^{0.5}, \tag{2}$$

where α – the constant describing the cluster strength; μ – the shear module; M – Taylor factor; b – Burgers vector; N – cluster density and d – cluster size.

The measurements of the density and sizes of cluster defects in pure copper irradiated at 80 °C and 150 °C revealed that an increase in the irradiation temperature causes a slight increase (~15% maximum) in the cluster size and does not practically affect their density (Table 1). In principle, an increase in the cluster size can, according to Eq. (2), affect the radiation hardening, but in our case a 15% increase in the cluster size yield a minor change (no more than 7%) in radiation hardening. In this case, an appreciable drop in radiation hardening with increasing irradiation temperature should be associated with a change in the parameter α in Eq. (2).

Using the values of cluster density and hardening in irradiated pure Cu obtained in [1,2,8], let us build the dependence $\Delta\sigma_y \sim \mathrm{f(Nd)}^{0.5}$ for irradiation temperatures of 80 °C and 150 °C for pure copper. Fig. 5 summarizes the results of the cluster density and size measurement in pure copper obtained during irradiation at 80 °C and 150 °C in the SM-2 and RBT-6 reactors [1,2,8].

Calculation of the α value from Eq. (2) yields $\alpha = 0.33$ for an irradiation temperature of 80 °C and $\alpha = 0.17$ for 150 °C (Fig. 5). Physically, the obtained result means that at 150 °C dislocations easily overcome obstacles (SFT). Note that the low temperature ($T_{\text{irr}} = 80$ °C) value of α obtained is somewhat higher than that obtained by Kojima et al. [9] for pure Cu irradiated with 14 MeV neutrons, $T_{\text{irr}} = 90$ °C and $T_{\text{test}} = 20$ °C ($\alpha = 0.23$).

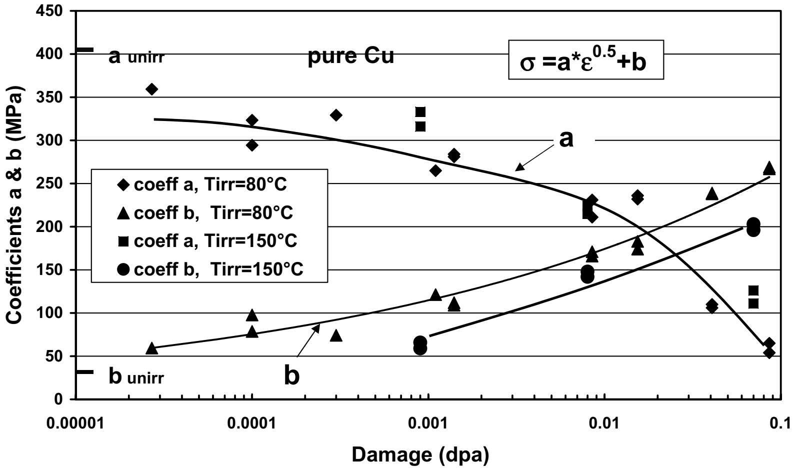

Fig. 4. Coefficients **a** and **b** taken from Eq. (1) plotted versus neutron damage, pure Cu as irradiated at $T_{\text{irr}} = 80$ °C and $T_{\text{irr}} = 150$ °C, $T_{\text{test}} = T_{\text{irr}}$.

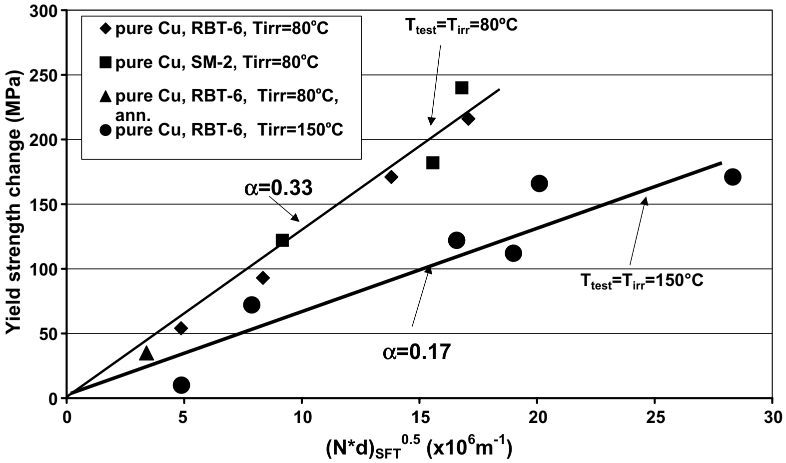

Fig. 5. Relation between the radiation hardening and square root of the product of d (defect clusters size) by N (defects clusters density). Data obtained for the yield strength and defect clusters density and size for pure Cu as irradiated at $T_{\mathrm{irr}} = 80$ °C (RBT-6 reactor and SM-2 reactor) and at $T_{\mathrm{irr}} = 150$ °C (RBT-6 reactor).

It seems that a drop in α with a rise in the temperature is determined to a greater extent by testing temperature rather than irradiation temperature. Really, the obstacle density, as follows from Table 1, does not practically change with a rise in irradiation temperature to 150 °C.

An alternative model of radiation hardening is the 'cascade-induced source hardening' (CISH) model proposed by Singh [6]. According to this model, during irradiation the grown-in dislocations are decorated by one-dimensional migrating clusters of self-interstitial atoms (SIA). In our experiments, we observed rafts of small clusters on dislocations in irradiated pure Cu (Fig. 3(d)). An increase in the irradiation and testing temperature should raise the SIA mobility.

Let us estimate how the CISH model agrees with our experiment. According to the TEM investigations, in the dose range of $\sim 10^{-3}$–10^{-4} dpa the density of rafts of small clusters on dislocation in pure Cu is low. Tensile tests showed that the yield drop is lacking and the difference between $\sigma_{\mathrm{y}80\,{}^{\circ}\mathrm{C}}$ and $\sigma_{\mathrm{y}150\,{}^{\circ}\mathrm{C}}$ for pure Cu and GlidCop Al25 alloy is small (Fig. 1).

The TEM investigations demonstrated that in the dose range of 10^{-2}–10^{-1} dpa, active decoration of dislocations by small clusters is observed in the structure of irradiated pure Cu (Fig. 3(d)). The yield drop emerges on the stress–strain curves of tested pure Cu specimens and, hence, $\sigma_{\mathrm{y}150{}^{\circ}\mathrm{C}} < \sigma_{\mathrm{y}80{}^{\circ}\mathrm{C}}$. That is, the difference in σ_{y} at $T_{\mathrm{test}} = T_{\mathrm{irr}} = 150$ °C and 80 °C is significant only when the density of small clusters on dislocations is high and the effect of clusters on deformation becomes determining. Thus, an increase in the irradiation and testing temperatures results not in a drop in the cluster density but in an increase in small cluster mobility and easier detachment of dislocations from atmospheres of such clusters. To estimate the contribution of an increase in $T_{\mathrm{test}} = T_{\mathrm{irr}}$ by 70 °C to an increase in the mobility of small CIA clusters it is necessary to perform molecular dynamic simulation. But even now, a conclusion is possible that the CISH model allows one to explain qualitatively the observed effect.

4. Conclusions

The analysis of the experimental data on the effect of neutron irradiation temperature on the mechanical properties and structure of pure copper allows the conclusion that an increase in irradiation temperature from 80 °C to 150 °C results in an appreciable drop in the yield strength and affects only slightly SHC and SFT density.

Assessment of changes in the parameter α, which describes the strength of obstacles in the Orowan equation shows that a rise in the temperature reduces by nearly one half this parameter at $T_{\mathrm{test}} = T_{\mathrm{irr}} = 150$ °C as compared with $T_{\mathrm{test}} = T_{\mathrm{irr}} = 80$ °C.

The observed drop in radiation hardening in pure copper with a rise in the testing and irradiation temperature agrees well with the 'cascade-induced source hardening' model.

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