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Changes of structure and properties of yttrium doped copper at deformation, annealing and irradiation

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Abstract

Results on the study of structural changes in pure and yttrium microalloyed copper (0.01–0.03 wt%) after rolling deformation to 40–90% and after isothermal and high velocity nonisothermal annealing in the wide temperature range (150–1050°C) are presented. The addition of yttrium in copper raises the recrystallization temperature, forms a fine grained homogeneous structure, changes the dislocation structure and raises the radiation resistance of copper.

1. Introduction

Pure copper and its alloys are widely utilized in electronics, electrovacuum and accelerator technology. The main attraction of copper lies in its high thermal and electrical conductivity. For the recent concept of fusion power reactor copper and its alloys may be widely utilized for the heat removal systems [1,2].

High purity copper exhibits the highest electrical and thermal conductivities, but the decrease of the total amount of impurities reduces their strength, thermal stability and structure homogeneity. A more effective method to achieve the desired strength, manufacturing and electrical characteristics of copper is alloying with some active elements, such as yttrium, scandium, hafnium and other rare elements [3–6]. Results emerging from the study of structural changes in pure and microalloyed copper on isothermal and high temperature annealing of samples deformed by rolling are presented.

2. Experimental procedure

Pure copper from vacuum electron-beam melting and copper doped with 0.01–0.03 wt% of yttrium were used [6]. The chemical compositions of investigated copper are listed in Table 1.

Samples for the study of structure and mechanical properties were cut from the rolled strip of corresponding melt, at a thickness of 0.2–0.3 mm. The total deformation of the strip changed from 40% to 90%. Metallographic examinations were made on an optical microscope MIM-8. The fine substructure was studied by electron microscope EM 100 CX. An X-ray dispersive spectrometer LINK-systems-860 was used for the analysis of second phase precipitation.

Mechanical properties of copper were defined in tension with strain rate 2×10^{-3} s^{-1}. The samples for the mechanical testing were produced by sheet forming (sheet thickness ≈0.5 mm). The sample's axis coincide with sheet rolling direction. Some samples were annealed for an hour at 520°C before testing.

3. Influence of deformation and annealing on the copper structure MVE and MMV

The structure analysis of the starting pure and microalloyed copper from electron-beam melting revealed

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Table 1
Chemical compositions of investigated copper

	Y	Zn	Bi	P	As	Si	Fe	Se	Ni	Mg	Al	Na
MVE	–	0.001	0.0008	0.001	0.0005	0.0023	0.0003	0.0008	0.0013	0.0013	0.0002	0.001
MMV	0.0090	0.0008	0.0007	0.001	0.0005	0.002	0.0003	0.0007	0.0004	0.007	0.0002	–
MMV	0.018											

that the addition of yttrium lead to a change in ingot structure, to the decrease of dendritic segregation and to the cleaning of copper from the associated impurities.

The mean size of grain in the central part of microalloyed copper ingot is three times smaller than that of cast pure copper MVE. It is well known that on all kinds of plastic deformation (drawing, rolling, extrusion) texture appears in the material – preference of certain crystallographic planes and axes in the deformation direction. Following annealing of deformed material recrystallized grains also have the preferred orientation (texture) that can be more pronounced than deformation texture. The annealed texture appears as a result of polygonization, primary and secondary recrystallization.

Metals and alloys with fee-lattice have a simple main texture after rolling: $\{1\,1\,0\}\langle 1\,1\,2\rangle$, $\{1\,1\,0\}$ plane and $\langle 1\,1\,2\rangle$ orientation are arranged parallel with the rolling plane and along the rolling direction [7]. In copper on cold rolling this texture is accompanied by a second texture that on large deformation approach to the system $\{1\,1\,2\}\langle 1\,1\,1\rangle$. After rolling a strong texture is demonstrated in samples from ingot of pure copper and microalloyed copper. The number of grains aligned with the rolling direction is less in the microdoped copper than in the pure ones.

In order to investigate the effect of annealing on the structure of rolling deformed copper the samples were annealed for an hour in vacuum in the temperature range from 150°C to 700°C with following cooling with a speed of 70°C/min.

A fine fibrous deformed structure is typical for the pure copper samples annealed for 1 h at 150°C. Only some fibres have the nuclei of new grains. The recrystallization process in the copper −0.01% yttrium samples is over during the annealing at 250–300°C. For the copper with 0.02% Y the primary recrystallization is over at 400°C anneal. Collecting recrystallization for the cold rolled copper doped with 0.01% Y, was observed at annealing temperature of 400–450°C and for Cu-0.02% Y at 500–600°C. Annealing at a temperature above 500°C for Cu-0.01% Y and above 600°C for Cu-0.02% Y is accompanied by secondary recrystallization. The average grain size in the samples of MMV annealing at 500–650°C was 1.5–2 times less than in pure copper (Fig. 1).

The observed increase of recrystallization temperature in yttrium doped copper agreed well with data of other authors [8–11]. The effect of small additions on the recrystallization temperature is stronger when the difference between the atomic radius of the addition and of the base is larger. Most of the rare earth elements correspond to these conditions. In our case for the copper–yttrium alloy dimensional misfit is 0.053 nm and its solubility in copper is rather limited (less than 0.05 wt% [9]).

X-ray diffraction analysis of copper ribbon had demonstrated that the crystal texture of 90% cold rolled copper MVE and MMV approach to the system $\{1\,1\,2\}\langle 1\,1\,1\rangle$ with a trace of $\{1\,0\,0\}\langle 0\,0\,1\rangle$ system. After the isothermal anneal of samples at 600°C the cube texture $\{1\,0\,0\}\langle 0\,0\,1\rangle$ is formed in plate sheets.

With the aim of reducing the anneal of deformed copper ingot we investigated the effect of rapid nonisothermal annealing on the structure. It is impossible to transfer the mechanism of structural change processes for the conditions of rising temperature. On accelerated heating more intensive nucleation and fast growth of recrystallization centers is observed. The effect of impurities decreases and the recovery process is suppressed. The high free volume metal energy accumulated on deformation is conserved up to high temperatures. The recrystallization process proceed with high speed. In Fig. 2 the microstructure of 60% deformed pure copper samples and doped copper samples are presented in the initial condition at 900°C and 1050°C. Examination of

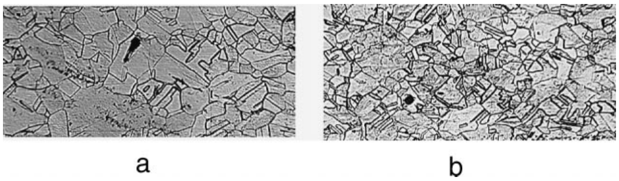

Fig. 1. Surface structure of samples MVE (a) and MMV (b) deformed to 60% and heated during 1 h to 550°C (×200).

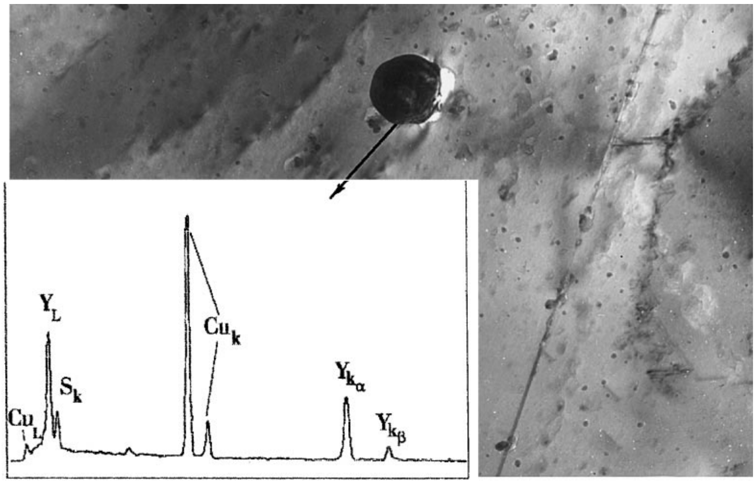

Fig. 2. Fine scale precipitates in the samples MMV (×10^5) and their X-ray spectrum.

structural changes on fast heating show the possibility for obtaining the homogeneous fine grained structure (grain size-15–30 mkm) on heating to 1050°C during 30 s for pure copper and during 60 s for microalloyed copper.

Electron microscopy examination of MVE copper annealed for 1 h at 520°C showed that the substructure was mainly composed of dislocation, twins and second phase precipitation. Dislocation density in the grain matrix was not high and constitute 5×10^6 cm^{-2}. Concentration of second phase precipitates in MVE copper was 4×10^{13} cm^{-3}. X-ray diffraction analysis revealed that the main precipitates are copper sulphide and phosphide with iron, chromium and magnesium as impurities.

Alloying of copper with yttrium influences the fine substructure of these materials, local distribution of dislocation structure components and the dislocation density changes. Density of dislocation clusters on the $\{1\,1\,1\}$ close-packed planes rise to 10^{-8}–10^{-9} cm. Besides single interstitial phase characteristics for copper (concentration 10^{14} cm^{-3}) some fine-dispersion precipitates (to 20 nm) are seen but it is very difficult to identify them. Copper with more than 0.20% yttrium has more coarse precipitates containing yttrium (Fig. 3). This confirms the refining effect of yttrium. The high density of stacking faults agrees with the fact that the implantation of yttrium in the copper solid solution leads to a decrease of stacking fault energy.

4. Mechanical properties of copper MVE and MMV

The effects of the degree of rolling predeformation (40–90%) and the annealing temperature in the range 150–600°C on the mechanical properties of pure and microalloyed copper were studied. Fig. 3 shows that microalloying by yttrium raises the resistance of deformed samples to the thermal loss of strength. The difference is more pronounced in the yield strength of samples from MVE and MMV after anneal at 150°C and 500–600°C. An essential parameter of material resistance is the so-called temperature of semi-loss of strength, i.e. temperature at which the yield stress of deformed samples decreases by half. Yttrium alloying of copper to 0.02% raises this temperature from 220°C (pure copper) to 400°C (alloyed copper) (Fig. 3).

It is seen that microalloyed copper exhibits the best combination of strength and ductility. At high temperatures ductility of MVE decreases while the microalloyed copper (0.03 wt%) above 300°C demonstrates the rise of elongation. This can be explained by the strengthening of grain boundary [12].

5. Radiation effects on structure and properties of pure copper and of microalloys

It was shown [12] that yttrium microalloying of copper decreases the radiation hardening and embrittlement on high-energy electron irradiation (225 MeV) to the fluence of 1×10^{21} e/cm^2 (0.1 dpa). Thin foils of pure and microalloyed copper were irradiated with 3 MeV Cr ions at 400°C. It was shown that following irradiation to 20 dpa the pure copper samples swell to 12%. Swelling of microalloyed copper (0.03% Y) in the same radiation conditions decrease to 3%. On lesser dose this effect is more pronounced. (Fig. 4).

It is well known that for the fusion reactor application, there is a need for materials with high radiation resistance, high thermal and electrical conductivity, and resistance to the thermal and electromagnet shock.

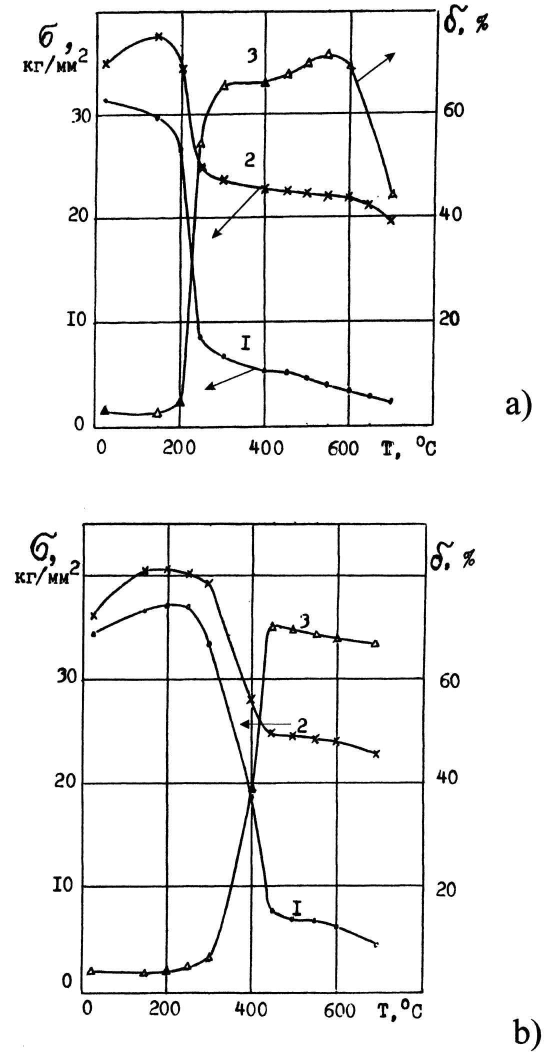

Fig. 3. Temperature dependence of yield strength (1), ultimate strength (2) and elongation (3) of the samples MVE (a) and MMV (b), preliminary deformed by rolling to 50%.

Materials most likely to meet these requirements are the pure and microalloyed copper. We investigated the effect of microalloying by yttrium, scandium, palladium, or zirconium on the properties following irradiation on pulsed plasma accelerator "Prosvet". The irradiation was done by hydrogen plasma with a period of 2 μs. The average particles energy in plasma was 2 keV and density -10^{14} cm^3. Energy spectrum of ions on plasma is similar to that of deuterium and tritium neutrals from plasma on discharge chamber wall UWMAK-11.

It is shown that after sample plasma irradiation to 10^{18} cm^{-2} the yield strength of pure copper rises more than 200% and that of microalloyed copper by 70–100%. The elongation of copper decreases from 70% to 30%, and that of microalloyed copper from 72% to 46%.

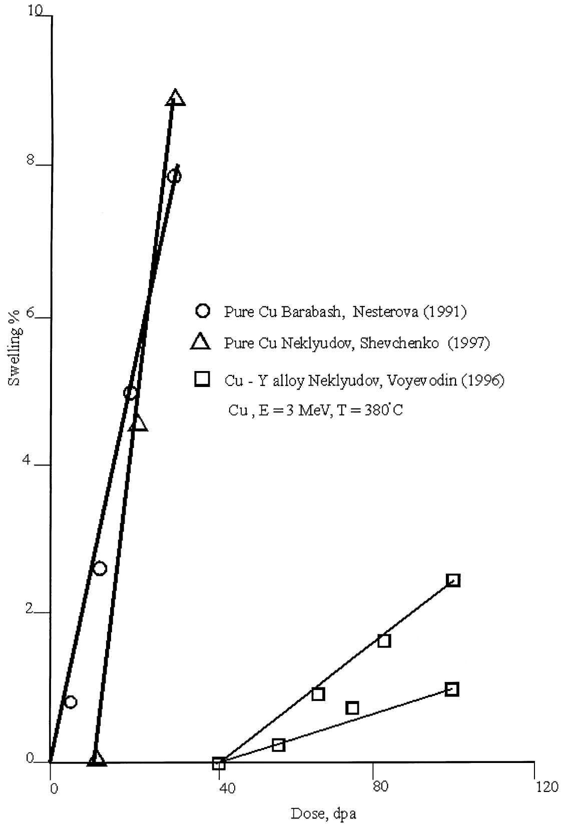

Fig. 4. Dose dependence of swelling for any copper alloys ($E = 3$ MeV, Cr^{3+}, $T_{irr} = 380°C$).

The change of sample surface microhardness is the same as the change of yield strength after plasma treatment. Microhardness of inner cross-section was not influenced by irradiation. We conclude that plasma irradiation effects are caused by surface materials modification.

6. Conclusions

Microalloying of copper by yttrium appreciably refines the structure, lowers dendritic seggregation and supresses the detrimental effects of some impurities, and refines the matrix and grain boundaries from associated elements.

Microalloying of copper increases the recrystallization temperature and promotes formation of a more homogeneous fine grained structure, and increases the thermal resistance of the microstructure. Copper alloyed by 0.01–0.02% Y possess the optimal strength properties.

The strengthening of copper–yttrium alloy is due to the interaction of dislocation with impurity atoms because of dimensional misfit and the high plasticity of copper–yttrium alloy at high temperature is due to grain boundaries strengthening. Our results demonstrate the high radiation stability of Y-alloyed copper on electron, ion and hydrogen plasmoid irradiation.

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