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Strengthening mechanisms in the nanocrystalline Cu with Al_2O_3

M. Orolínová[1]*, J. Ďurišin[1], K. Ďurišinová[1], Z. Danková[2], M. Besterci[1], M. Ďurišin[1], J. Szabó[1]

[1] *Institute of Materials Research of the Slovak Academy of Sciences, Watsonova 47, 043 53 Košice, Slovak Republic*
[2] *Institute of Geotechnics of the Slovak Academy of Sciences, Watsonova 45, 043 53 Košice, Slovak Republic*

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Abstract

Nanocrystalline (nc) Cu materials with Al_2O_3 dispersoid (1 and 5 vol.% Al_2O_3) were produced using the powder metallurgy (PM) techniques. The effect of reinforcement weight fraction, deformation temperature and annealing temperature (up to 800 °C) on the strength properties and the microstructure was studied. Two strengthening mechanisms were considered at room temperature, strengthening by grain refinements and dispersion strengthening (DS). The strength properties of both nc Cu composites were strongly dependent on deformation temperature and material composition. The main contribution of nanometric Al_2O_3 particles to the strength of composites at elevated temperatures is probably the strong pinning effect on the grain/crystallite boundaries and preventing of recrystallization process. After annealing at $0.74T_m$ both composites were characterized by an excellent stability of strength characteristics.

K e y w o r d s: dispersion strengthened nanocrystalline copper, strengthening mechanisms, strength properties, microstructure

1. Introduction

The nc DS materials on the Cu base are characterized by interesting and technologically important properties. DS of the soft copper alloy matrix is achieved by introducing a small volume fraction of secondary phase (such as oxides, carbides, nitrides) to the matrix. Alumina DS nc Cu offers an unique combination of strength, electrical and thermal conductivity. These materials were developed for wide range of applications [1–4]. The method of DS has a significant reinforcing effect which can be kept at elevated temperatures. The increasing yield strength of metal matrix of nc composites could be attributed above all to the following four strengthening mechanisms: Hall-Petch (HP) strengthening, Orowan strengthening, strengthening arising from an increase in dislocation density of the matrix described in the Taylor relationship and load-bearing effect which results from the strong bonding between the dispersed particles and the matrix. In previous years, several models have been used for predicting the strength of metal matrix of nc composites, such as the summation of strengthening contributions, the Zhang and Chen model, and the Clyne model [5–7].

Orowan strengthening is carried out in consequence of dislocations interactions with stable dispersoid particles [8–11]. The degree of strengthening is a function of size, shape, spacing, hardness, and distribution of the second phase particles within the matrix. The elevation in yield strength is due to the bowing of dislocations between neighbouring undeformable particles and bypassing particles leaving a dislocation loop around them. The alumina particles are hard and thermally stable at elevated temperatures approaching the melting point of the copper matrix. Much of the research on nc metals has been focused on the HP grain size strengthening effect because grain size reduction to the nanoscale range (< 100 nm) produces very large increase in the strength [1, 9, 12]. However, the mechanisms operative in the nanoscale grain size regime differ from those for coarse-grained materials [13]. A widely accepted consensus proposes that a transition to grain boundary mediated dislocation plasticity occurs below a grain size of 100 nm, with a possible transition to grain boundary sliding/rotation

*Corresponding author: e-mail address: morolinova@imr.saske.sk

dominant processes at a very small grain size [14].

The nc DS Cu is characterized by high strength at room temperature as well as at elevated temperatures [15]. The dispersion distributed alumina particles can increase the recrystallization temperature and exhibit excellent strength at elevated temperature by pinning down the grain and subgrain boundaries of the copper matrix and impeding the movement of dislocations [5, 16, 17].

In this study, the nc Cu composites with different ratio of dispersoid (1 and 5 vol.% Al_2O_3) were synthesized using the PM method. The purpose of this study is quantitative research of strength properties (estimating the contribution of individual strengthening mechanisms using Orowan mechanism and Hall-Petch ship) and verification of an agreement with experimental data. The strength at elevated temperature, strength after thermal loading and their influence on microstructure were investigated.

2. Experimental materials and methods

The experimental material was dispersion strengthened nc Cu-1 vol.% Al_2O_3 and nc Cu-5 vol.% Al_2O_3. The composite powder was prepared by modification of the mechano-chemical method based on the mechanical milling in an attritor and chemical reduction by hydrogen [18]. The Cu powder matrix is characterized by average crystallite/grain size 36 nm and 29 nm for 1 vol.% Al_2O_3 and 5 vol. % Al_2O_3, respectively. The compaction process consisted of powder pressing at 400 ℃ in the protective atmosphere under the pressure of 150 MPa, sintering in H_2 at 850 ℃ for 1 h, forging and extrusion at 950 ℃ with a 95 % cross section area reduction.

The micro Vickers hardness (HV10) was measured at room temperature with dwell time 10 s on Leco equipment. The Brinell hardness HB was measured under 612.9 N load with ball diameter 2.5 mm using HPO 250 machine. The values of ultimate tensile strength and yield strength at room temperature and at elevated temperatures were evaluated by static tensile test STN EN ISO 6892-2 performed by TIRATEST 2300 test machine. The specimens for tensile test were prepared according to STN EN ISO 6892-1. The specimen diameter was 3 mm and gauge length was 15 mm. The furnace temperature was varied from room temperature up to 800 ℃ with an accuracy of ± 4 ℃. The total porosity was measured using the double-weighting method.

The microstructure of the experimental materials was investigated by transmission electron microscopy (TEM) by the method of thin foils. The presence of γAl_2O_3 particles was proved by the selected area diffraction (SAED). The microstructure was consequently analyzed by X-ray diffraction (XRD) on X'Pert diffractometer with high temperature camera. The in situ measurements were realized in the range from room temperature up to 800 ℃. The XRD spectra were measured between 40 and 100° (2θ) with a step of 0.03° and a time per step of 8 s. The average grain/crystallite size D of the Cu matrix and the dislocations density ρ were determined using the Williamson-Hall equation. The Williamson-Hall equation separates the effects of size and strain in the crystals, and is convenient for the estimation of crystal size of deformed materials:

$$\frac{\beta_{\mathrm{hkl}}\cos\theta_{\mathrm{hkl}}}{k\lambda} = \frac{1}{D} + \frac{4\sigma}{E}\left(\frac{\sin\theta_{\mathrm{hkl}}}{k\lambda}\right), \qquad (1)$$

where k is the shape factor (0.9), λ is the X-ray wavelength (1.5406 Å), θ_{hkl} is the Bragg angle, D is the effective grain/crystallite size normal to the reflecting planes, σ is the lattice strain, and E is the Young's modulus. The dependence of $\beta_{\mathrm{hkl}}\cos\theta_{\mathrm{hkl}}/k\lambda$ on $\sin\theta_{\mathrm{hkl}}/k\lambda$ is linear for Cauchy distribution of diffraction peak. The simplified form of this equation has linear form:

$$y = q + kx, \qquad (2)$$

where $y = \beta_{\mathrm{hkl}}\cos\theta_{\mathrm{hkl}}/k\lambda$, $q = 1/D$, $k = 4\sigma/E$ and $x = \sin\theta_{\mathrm{hkl}}/k\lambda$. The first four most intensive reflection peaks of Cu reflecting planes (111), (200), (220) and (311) were used to construct a linear plot of y as a function of x, the grain/crystallite size might be estimated from the intersection with the vertical axis y and the lattice strain from the slope of the line [10].

The thermal stability of the microstructures was studied by the ex-situ method, after the isothermal annealing at 800 ℃ for 1 h in the air.

3. Results and discussion

3.1. Strength at room temperature

Both nc Cu composites are characterized by high hardness. The micro Vickers hardness for the 1 vol.% Al_2O_3 composite is 1400 MPa and Brinell hardness 130 HB, for the 5 vol.% Al_2O_3 composite is micro hardness 2200 MPa and Brinell hardness 170 HB. Higher volume fraction of the Al_2O_3 particles induces higher hardness of the material. As the hardness is a measure of material resistance against the plastic deformation, it is related to the stress needed to activate the dislocations motion in the material. Therefore, the hardness is structure-sensitive parameter. High hardness in both composites is a result of the presence of small Cu grain size due to the uniform distribution of very fine γAl_2O_3 particles in the Cu matrix, confirmed by TEM micrographs and the SAED patterns,

Fig. 1a,b. The mean grain/crystallite size of material with 1 vol.% Al_2O_3 is 91 nm and for material with 5 vol.% Al_2O_3 is 60 nm. Accordingly, both composites are characterized by nanocrystalline Cu matrix. The Al_2O_3 particles have an extremely fine average size ~ 5 nm in radius for both materials. The results of hardness tests indicated that both composite systems had significantly high strength characteristics. The yield strength achieved 400 MPa for the 1 vol.% and 598 MPa for the 5 vol.% Al_2O_3 composite material. The ultimate tensile strength reached the value 447 and 656 MPa, respectively. The obtained mechanical properties are comparable with results published in other works [2, 12]. The Cu composite with higher volume of dispersoid is characterized by superior strength properties. However, both Cu composites with Al_2O_3 dispersoid showed markedly higher strength characteristics in comparison with pure micro grained Cu ($R_p0.2 = 156$ MPa, $R_m = 245$ MPa) [2]. There are two important strengthening mechanisms to be considered in the nc Cu with secondary phase/dispersoid: dispersion strengthening effect and grain size strengthening effect. These strengthening mechanisms are additive and the total yield strength should be considered as the sum of these mechanisms [7, 9].

The contribution of the Al_2O_3 dispersoid present in the Cu composites to the overall strength of the material should be considered. The presence of particles, acting as obstacles to the dislocation motion, is responsible for the strengthening of the material by limiting the dislocation ability to move under external stress. The Al_2O_3 particles are considered not to be shearable by the dislocations, hence the dislocations must overcome the particles in order to bypass them by moving between the particles (Orowan process). The critical resolved shear stress $\Delta\tau_0$ produced by the interaction of moving dislocation and dispersion distributed Al_2O_3 particles is as follows [9]:

$$\Delta\tau_0 = 0.84\frac{2T}{b\lambda}, \tag{3}$$

where T is the line tension of the dislocation line, b is the modulus of its Burgers vector ($b = 2.56 \times 10^{-10}$), and λ is the interparticle spacing of alumina particles. The mean interparticle spacing λ of dispersoid particles in the Cu matrix is defined by the volume fraction f and average radius r [2, 8]:

$$\lambda = r\left(\frac{2\pi}{3f}\right)^{\frac{1}{2}}. \tag{4}$$

The calculated mean interparticle spacing is 58 and 26 nm for the 1 vol.% Al_2O_3 and 5 vol.% Al_2O_3 composite, respectively. Orowan strengthening becomes more favourable by reducing the diameter and interparticle spacing of the second phase [19]. The Orowan strengthening effect of Cu was found to be 92 MPa for 1 vol.% Al_2O_3 composite material and 205 MPa for 5 vol.% Al_2O_3 composite material using $T = 1.0 \times 10^{-9}$ N. Hence the contribution of the dispersion effect is relatively important in both Cu composites. The Orowan mechanism plays more remarkable role for the strengthening of the composites, when the reinforcement size is nanometric and the interparticle spacing is shorter.

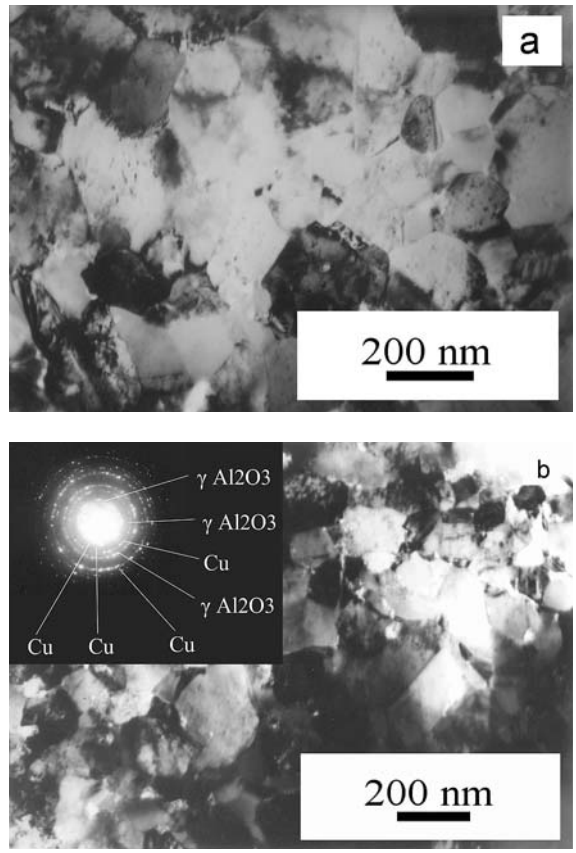

Fig. 1. TEM microstructure and SAED pattern of 1 vol.% Al_2O_3 (a) and 5 vol.% Al_2O_3 (b) Cu-Al_2O_3 composite.

The contribution of the grain/crystallite refinement to the strength should be considered. It is known that the grain/crystallite size has a significant effect on the mechanical behaviour of materials, in particular, on the yield stress. The relation between the yield stress and grain size is described mathematically by the Hall-Petch equation [10]:

$$\sigma_y = \sigma_0 + \frac{k_y}{\sqrt{d}}, \tag{5}$$

where σ_y is the yield stress, σ_0 is a material constant for the starting stress for dislocation movement (or the resistance of the lattice to dislocation motion, for Cu

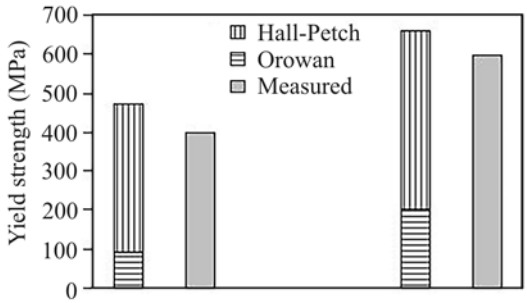

Fig. 2. Contribution of Orowan and Hall-Petch strengthening mechanisms on final strength of Cu-Al_2O_3 composites in comparison with measured yield strength.

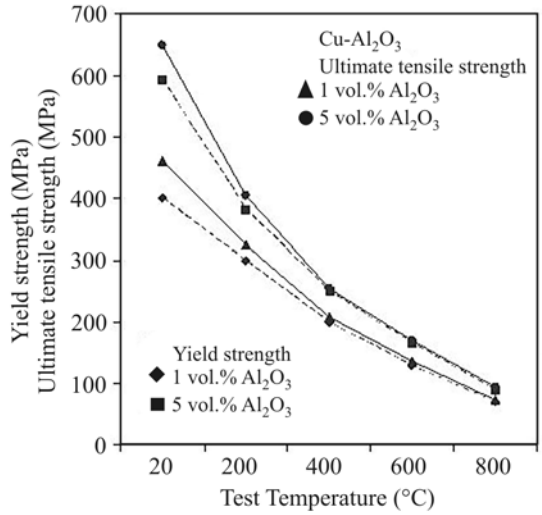

Fig. 3. Yield strength $R_p0.2$ and ultimate tensile strength R_m of Cu-Al_2O_3 composites as a function of test temperature.

25.5 MPa), k_y is the strengthening coefficient (a constant unique for each material, 3478.5 for Cu), and d is the average grain/crystallite diameter. The Hall-Petch relationship predicts that the yield stress increases with the inverse of the square root of the grain size (Eq. (5)). Theoretically, a material could be made infinitely strong if the grains are made infinitely small. According to the Hall-Petch relation an indirect proportion between the strength and grain size is valid up to the critical value of grain size (for Cu a value below $\sim$ 25 nm) because very small grains are unable to support dislocation pile-ups [6, 14]. The presence of greater volume of second phase particles leads to smaller grain/crystallite size with larger grain boundary surface area. Due to the grain/crystallite size effect it was estimated that the yield strength increment was 382 MPa and 451 MPa for 1 vol.% Al_2O_3 and 5 vol.% Al_2O_3 composite, respectively. Accordingly, the grain/crystallite size strengthening becomes the dominant mechanism. The obtained results are in agreement with other works published by Sanders, Merz and Dahlgren for nanocrystalline Cu [14].

Assuming that the total yield strength of nc Cu composites is an addition to two strengthening mechanisms [7], the calculated value of the yield strength is 474 MPa and 656 MPa for 1 vol.% Al_2O_3 and 5 vol.% Al_2O_3 material, respectively, Fig. 2. The total yield strengths of composites are approximated according to empirical Tabor's rule, hardness/3 [20]. These calculated values of the yield strength are rather higher than the measured values. The lower values of yield strength are predominantly in consequence of "flows" existing in consolidated powder materials. It is known that it is difficult to obtain high-density nc materials of low porosity and good bonding between particles by the PM method [21, 22]. The total porosity for experimental material with 1 vol.% dispersoid is about 1.5 % and for composite with 5 vol.% is higher, 1.7 %. The structure imperfections are detrimental to the properties of nc materials because with increasing porosity Young's modulus decreases. The linear decreasing of the Young's modulus as a function of porosity for nanocrystalline Cu was calculated [14].

Both composites are characterized by very high dislocation density. The resultant dislocations density is influenced by the method of composite preparation, in particular by the milling intensity of powders and hot extrusion. The different coefficient of thermal expansion (CTE) and the elastic modulus (EM) of the matrix and particle shared contribute to high dislocation density. Both analyzed strengthening mechanisms should contribute to the increase in the dislocation density.

The yield strength at room temperature in nc Cu composites with Al_2O_3 nanoparticles can be predicted with an accuracy of 90 % as an additive of Hall-Petch and Orowan strengthening mechanism.

3.2. Strength at elevated temperatures

The strength properties of both nc Cu composites are strongly dependent on the deformation temperature, Fig. 3. The strength decreases with the increasing test temperature. The mechanism responsible for the observed degradation in both yield strength and ultimate tensile strength, with an increase in deformation temperature is, accordingly, attributed to the several influences. The yield strength decreases in an interval between room temperature and 400 ℃ for 1 vol.% Al_2O_3 material at the rate of ca 50 MPa/100 ℃. The reduction in strength is 50 % compared to the room temperature. For the 5 vol.% Al_2O_3 composite this decrease is sharper, cca 85 MPa/100 ℃, and the reduction in strength is 58 %. This degradation in strength characteristics is high. The yield strength decreases gently from the temperature 400 ℃ up to 800 ℃ (25 MPa/100 ℃ and 42 MPa/100 ℃ for the 1 vol.%

Table 1. Dislocation density ρ of Cu-Al_2O_3 composites as a function of test temperature T

Cu-Al_2O_3	Dislocation density $\rho \times 10^{13}$								
	Test temperature (℃)								
	20	100	200	300	400	500	600	700	800
1 vol.% Al_2O_3	18.8	16.1	9.4	0.9	0.6	0.1	0.5	0.1	0.08
5 vol.% Al_2O_3	44.1	42.3	37.1	5.3	5.2	6.5	3.3	0.9	0.7

Al_2O_3 and 5 vol.% Al_2O_3 composite, respectively). The test temperature has a similar effect on the ultimate tensile strength, Fig. 3. The yield strength and ultimate tensile strength converge with the increasing test temperature.

The mechanism responsible for the observed decrease in yield strength and ultimate tensile strength with increasing test temperature is attributed to the several influences. The residual stresses, compressive in nature, induced by the material preparation, result in reducing number of the mobile dislocations available at the elevated temperature to interact with the dispersoid particles. By the increasing test temperature the Orowan strengthening effect is weak, because the thermal activation is helpful to the tangled dislocation around the nano-alumina particle climbing up. This is resulting in the stress relaxation around the alumina-Cu matrix interfaces. The dislocation movement mechanism of nc Cu composites at lower temperatures (up to $0.5\,T_m$) is sliding mechanism mainly and the strengthening effect arises from an interaction of the mobile dislocations with the Al_2O_3 particles, which restricts the mobility of the dislocations and interface strengthening [23]. At higher temperatures, the number of mobile dislocations introduced to the structure is decreased by dislocation annihilation caused by more atomic diffusion. However, the properties of the grain boundaries may be important in experimental composites because the grain boundaries occupy over 10 vol.% of the material in the case of Cu-5 vol.% Al_2O_3 composite (the grain size of 50 nm) and cca 5 vol.% of materials with the grain size 90 nm (in the case that the average width of the grain boundary is 1 nm) [24]. These values are limitations of models based on dislocation slip for metallic nc materials [25]. Consequently, in the studied nc composites the mechanical characteristics should not be controlled by dislocation movement but by diffusion controlled mechanism such as grain boundary sliding. The convergence of the yield strength and ultimate tensile strength with the increasing test temperature is probably attributed to a change in dislocation movement mechanisms or diffusion mechanism.

The microstructure changes at thermal loading were analyzed by in situ method by the X-ray diffractometer up to temperature 800 ℃ under no stress conditions (static conditions). The dislocation density of both materials decreases with the increasing test temperature, Table 1. The dislocation density decreases from the order $10^{13}\,cm^{-2}$ gradually to the order $10^{10}\,cm^{-2}$ at 800 ℃ in 1 vol.% Al_2O_3 material. In 5 vol.% Al_2O_3 material the dislocation density decreases from order 10^{14} to $10^{11}\,cm^{-2}$ at 800 ℃. However, the dislocation density at 800 ℃ in both composites is always high and corresponds to the dislocation density in heavily deformed metals.

The Cu matrix grain/crystallite size for the 1 vol.% Al_2O_3 material remains unchanged up to 400 ℃ (91 nm) and then slowly increases, at 800 ℃ reaches 110 nm, Fig. 4. For the 5 vol.% Al_2O_3 composite it is unchanged up to 500 ℃ (60 nm) and then increases, however, at temperature 800 ℃ remains always in the nanometric scale (75 nm). The grain/crystallite growth in these materials is inhibited by the presence of Al_2O_3 nanoparticles in Cu matrix. The slightly more pinning effect on the grain/crystallite boundary migration is owing to the small inter particle spacing and larger effective area for the fixed volume fraction of dispersoid. The higher volume of fine Al_2O_3 particles provides the formation of the finer Cu grain/crystallite size during the composite preparation and their conservation in nanometric dimension at thermal loading.

The analyzed nc Cu composites present higher strength properties at elevated temperatures in comparison with micro grained unreinforced Cu [3]. The high level of strength in these nc Cu alloys resulted in the fact that copper matrix maintained the ultra-fine grain/crystallite size at high temperature, too. The main contribution of thermal stable nanometric Al_2O_3 particles to the composite strength at elevated temperatures is probably the strong pinning effect on the grain/crystallite boundaries and preventing the recrystallization process.

3.3. Strength after annealing

The hardness of both nc Cu composites after annealing at 800 ℃ during 1 h remained practically unchanged in comparison with the hardness at room temperature, Table 2. Accordingly, the softening did not occur in both materials up to $0.74\,T_m$ because the

Table 2. Micro Vickers hardness HVM, Brinell hardness HB, yield strength $R_p0.2$, ultimate tensile strength R_m of composites Cu-Al_2O_3 after annealing at 800 °C/1 h in comparison with initial state

Cu-Al_2O_3	Initial state				After annealing 800 °C			
	HVM (MPa)	HB	$R_p0.2$ (MPa)	R_m (MPa)	HVM (GPa)	HB	$R_p0.2$ (MPa)	R_m (MPa)
1 vol.% Al_2O_3	1400 ± 15	130	400	447	1380 ± 13	124	394	435
5 vol.% Al_2O_3	2300 ± 18	165	598	656	2290 ± 17	165	598	643

Table 3. Grain/crystallite size D and dislocation density ρ of composites Cu-Al_2O_3 after annealing at 800 °C/1 h in comparison with initial state

Cu-Al_2O_3	Initial state		After annealing 800 °C	
	D (nm)	ρ (m^{-2})	D (nm)	ρ (m^{-2})
1 vol.% Al_2O_3	91	18.8×10^{13}	100	8.9×10^{13}
5 vol.% Al_2O_3	60	44.1×10^{13}	65	21.3×10^{13}

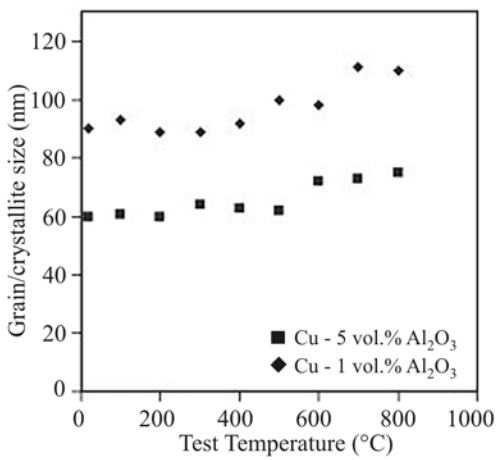

Fig. 4. Grain/crystallite size D of Cu-Al_2O_3 composites as a function of test temperature.

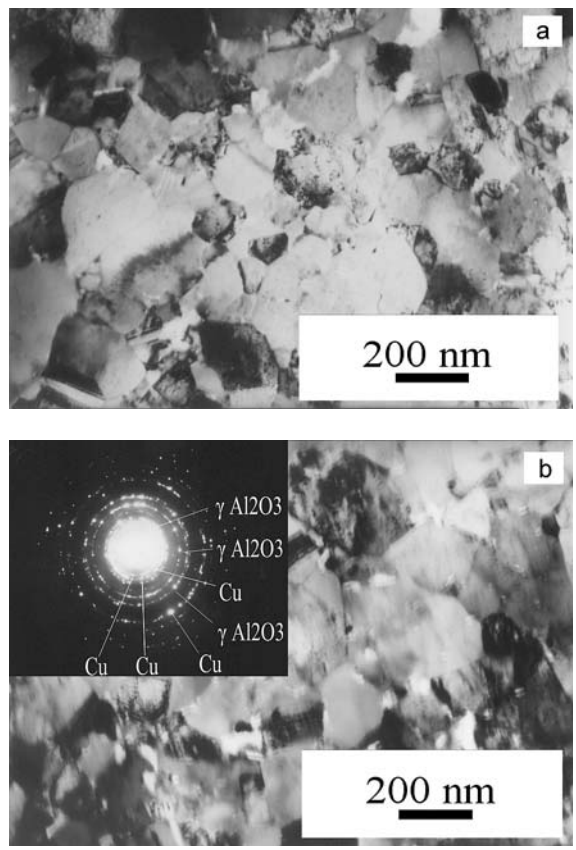

Fig. 5. TEM microstructure and SAED pattern of 1 vol.% Al_2O_3 (a) and 5 vol.% Al_2O_3 (b) Cu-Al_2O_3 composite after annealing at 800 °C/1 h.

softening temperature is defined as the annealing temperature in relation to the sharp decrease of the composite hardness.

The strength properties remained unchanged after the annealing at 800 °C for 1 h in comparison with strength measured prior to the annealing in both materials, Table 2. This high thermal stability of material properties is dependent on the high thermal stability of the structure. The analyzed TEM (the size and distribution of the γAl_2O_3 particles), Fig. 5, and X-ray microstructure parameters (grain/crystallite size and dislocation density), Table 3, are comparable with the initial parameters. It was documented that at thermal loading the dislocation density decreased. Its increase on the value prior to the annealing, Table 2, is inducted by thermal stresses during the cooling on the phase interface following the expressive different CTE and the elastic modulus EM of the matrix and particle. The Cu has higher CTE ($\alpha = 16.5 \times 10^{-6}\,K^{-1}$) than

Al_2O_3 ($\alpha = 8 \times 10^{-6}\,K^{-1}$) and higher EM (Cu – 150 GPa, Al_2O_3 – 300 GPa).

The excellent softening resistance and thermal strength stability of nc Cu composites is due to the sufficient volume of Al_2O_3 nanometric particles, which are effective to counteract the movement of grain/crystallite boundaries and so strongly impede the recrystallization process.

4. Conclusions

The following conclusions can be drawn:

It was affirmed that the yield strength at room temperature for nc Cu composites with Al_2O_3 nanoparticles is possible to be predicted with closeness on 90 % as an additive of two strengthening mechanisms. The major contribution to the total yield strength is grain/crystallite size effect (Hall-Petch strengthening mechanism) and the rest is attributed to the presence of Al_2O_3 particles (Orowan mechanism). A relatively good agreement was found between the measured values of Vickers micro hardness and the estimated yield strength, too. The real measured strength appeared to be lower than calculated in consequence of porosity existence which reduced yield strength of the compacted powder materials.

The composite with higher reinforcement weight fraction is characterized with higher strength properties at room temperature. The Al_2O_3 particles are extremely fine with the mean size $\sim$ 5 nm in radius in both materials. The presence of higher volume of second phase particles leads to shorter interparticle spacing and simultaneously to finer Cu grain/crystallite size with larger grain boundary surface area which leads to higher contribution of both described strengthening mechanisms.

The strength parameters of both nc Cu composites are strongly dependent on the deformation temperatures. An increase of the test temperature resulted in the decrease of strength of the materials. The effect of deformation temperature is more significant for composite with higher volume fraction of dispersoid. With the increasing test temperature the yield strength and ultimate tensile strength converge, this is probably attributed to a change in dislocation movement mechanisms. The main contribution of nanometric Al_2O_3 particles to the composite strength at elevated temperatures is the strong pinning effect on the grain/crystallite boundaries.

The nc Cu composites after annealing are characterized with the excellent softening resistance and thermal stability of strength properties, up to 0.74 T_m. This is attributed to sufficient volume fraction of nanoalumina particles that are effective to counteract the movement of grain/crystallite boundaries, preventing the recrystallization process. The effect of CTE and EM mismatch is very important mechanism from the point of view of ensuring the excellent thermal stability of structure as well as strength properties of nc Cu composite.

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