High Temp. Mater. Proc., Vol. **30** (2011), pp. 573–577 DOI 10.1515/HTMP.2011.116

Tribological Characteristics of Micro- and Nano-Composites Cu-Al_2O_3 at Room and Elevated Temperatures

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Abstract. Tribological properties by two copper based composites (Micro-Cu-Al_2O_3 with grains size 1–2 μm and Nano-Cu-Al_2O_3 with grains size 100–200 nm) were studied by pin-on-disk technique against a steel ball at a range from room temperature up to 873 K. For both systems the coefficient of friction decreased between 473 K and 673 K by about 25 %. At lower temperatures the nano-Cu was about three times more wear resistant than the micro-Cu but the wear rate of both systems decreased down to zero at 673 K due to formation of hard oxide layers.

Keywords. ECAP, system Cu-Al_2O_3, micro and nanocomposites, sliding wear, tribological characteristics.

PACS®(2010). 62.20.Qp.

1 Introduction

Copper has the leading role in industrial applications. A variety of Cu alloys has been developed but they exhibit a rather large increase of resistance in both electrical and heat conductivity and low time stability at elevated temperatures. Powder metallurgy can give a solution in dispersing particles in the prepared material appropriate characteristics [1]. Another way how such desirable properties can be achieved is creating very fine, submicron-grained microstructures [2]. Such microstructures can be prepared by inducing severe plastic deformation [3]. This can be conveniently done by technique of the Equal Channel Angular Pressing (ECAP) which by multiple pressings of material through the die achieves very fine grained microstructure (nanostructure) [4]. The materials prepared in this way are suitable for demanding parts of machines frequently exposed to intense friction and wear, such as washers, bearing liners, etc, where properties such as high strength and ductility, fatigue strength, wear resistance, etc., are required.

Material	Designation	Matrix grain size
Micro-Cu-Al_2O_3	Cu1	1–2 μm
Nano-Cu-Al_2O_3	Cu2	100–200 nm

Table 1. Experimental materials.

Aim of this work was to investigate the effect of refining of microstructure by ECAP process on tribological behavior and wear of Cu-Al_2O_3 composite at room and elevated temperatures.

2 Materials and Experimental Procedures

Reaction milling and mechanical alloying was used to prepare the samples. Cu powder with the calculated addition of Al was homogenized by attrition in oxidizing atmosphere. The distribution of the obtained CuO was uniform. A subsequent treatment at 1023 K induced the reaction of CuO with the added Al powder, and led to the formation of Al_2O_3 particles. The remaining CuO was reduced by attrition in a mixture of $H_2 + H_2O$ (rate 1 : 100). The powder was compacted by cold pressing and hot extrusion at 1023–1073 K.

Micro-grained material with 5 vol. % Al_2O_3 was transformed by the ECAP (Equal Channel Angular Pressing) method in two passes into a nanocomposite material. The experimental material was pressed through two right angled (90°) channels of a special die.

The notification of the experimental materials is explained in Table 1.

Microstructure was studied using TEM thin foils, in order to reliably identify matrix and nanosized phases.

Wear testing was performed on a High Temperature Tribometer THT, by CSM Instruments, using ball-on-disk technique. The sample was fixed on a turntable with adjustable rotational speed. The tangential force exerted on the holder was measured and from that the coefficient of friction (COF) was calculated and recorded as function of distance/time/laps. The vertical position of the holder was measured in order to monitor the displacement due to material removed by wear. As friction partners steel balls with 6 mm diameter were used. The loading of 1 N was applied using a dead weight system. The nominal wear track radius was 2 mm, the sliding speed was set to 5 cm/s and the over-

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Received: February 18, 2011. Accepted: March 28, 2011.

all sliding distance was 100 m. Testing was done on air (humidity 40% ± 5%), in dry conditions at temperatures 293 K, 473 K, 673 K, and 873 K. The heating was provided by an integrated furnace which reached the target temperature in the sample chamber in about 30 minutes and then during another 30 minutes it was allowed for the temperature to homogenize and stabilize. After the tests, both tribological partners (the steel ball and the sample) were observed using light microscopy. The depth and shape of the wear tracks were measured by a stylus profilometer (Mitutoyo SJ-201) on three or more places, the average trough cross section area was calculated and subsequently the volume of the removed material was estimated. The specific wear rates (W) were then expressed according to ISO 20808 [5] as the volume loss (V) per distance (L) and applied load (F_p):

$$W = \frac{V}{L \cdot F_p} \left[\frac{\mathrm{mm}^3}{\mathrm{m} \cdot \mathrm{N}}\right] \tag{1}$$

and compared for both material systems.

3 Results and Discussion

Figure 1 and Figure 2 show the TEM photographs of the microstructures of the experimental materials. Typical grain size in the material Cu1 was about 1–2 μm (Figure 1) whereas in the material Cu2 it was much smaller, typically from 100 to 200 nm. (Figure 2).

The friction behavior of both materials was in terms of coefficient of friction (COF) generally quite similar. In the beginning there was a short run-up phase (2 up to 20 meters of sliding distance) where the contact surfaces were setting up (Figure 3). The coefficient of friction exhibited either lower or higher values than expected. Then the macroscopic failure of the surface began to take place and the COF settled at 0.45–0.60, i.e. the values typical for steel-copper dry friction contact [6, 7]. This level of friction then remained

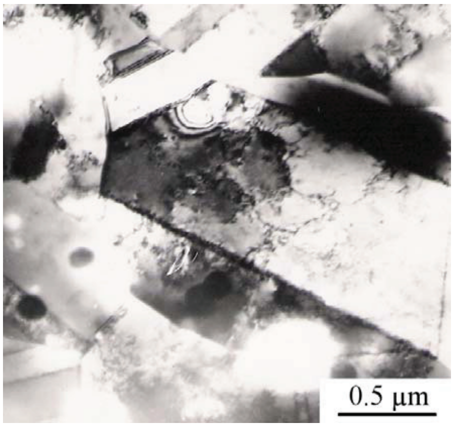


Figure 1. TEM micrograph of the micro-composite Cu1.

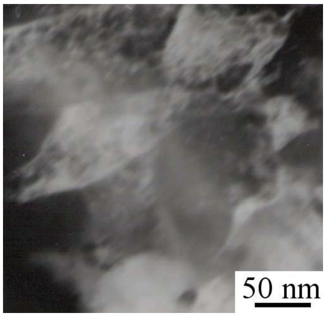
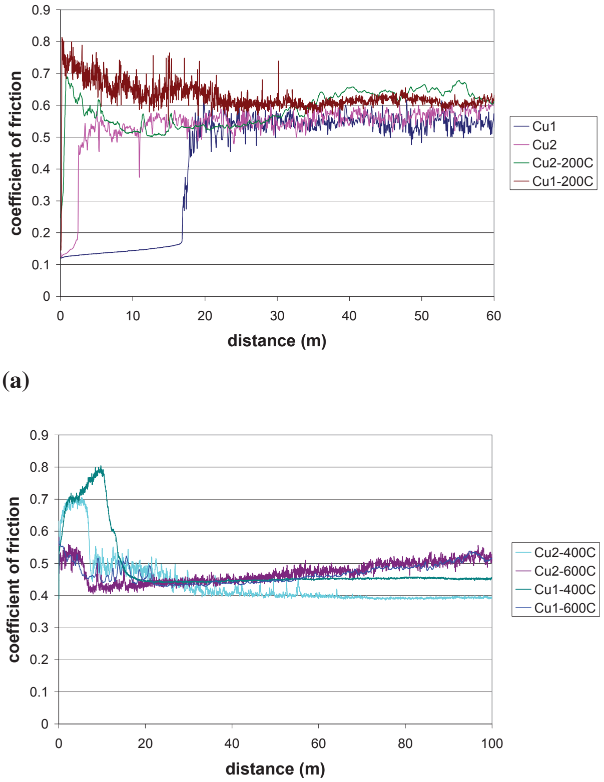


Figure 2. Experimental materials.



(a)

(b)

Figure 3. Examples of variation of the coefficient of friction along the sliding distance.

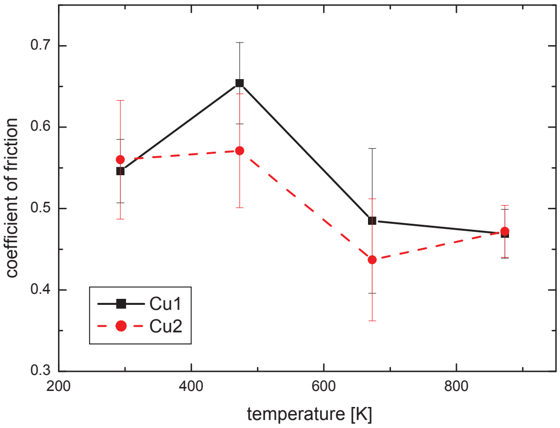


Figure 4. Coefficient of friction as function of temperature.

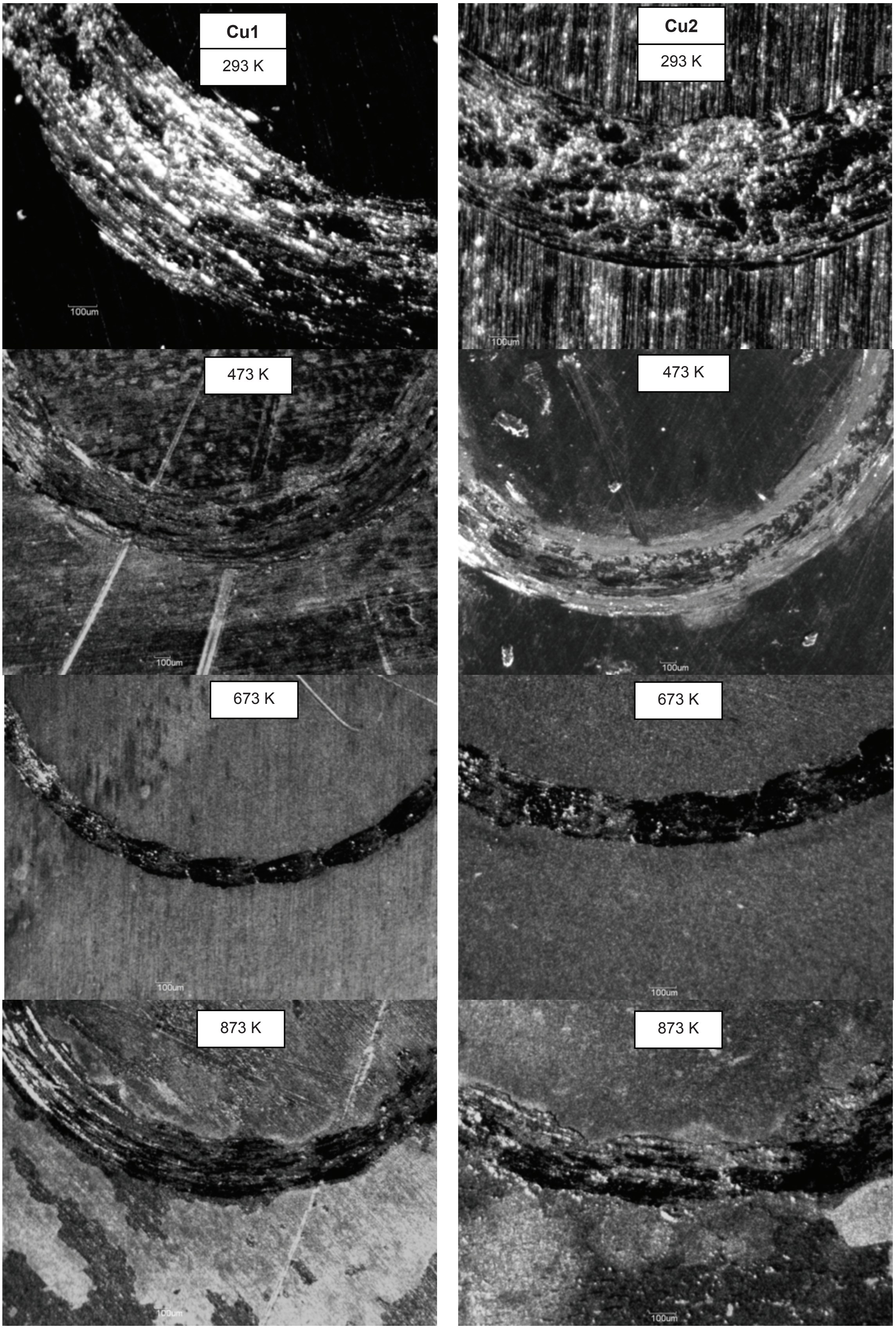


Figure 5. Wear tracks in Cu-Al_2O_3 composites made at various temperatures. Cu1 – left column, Cu2 – right column.

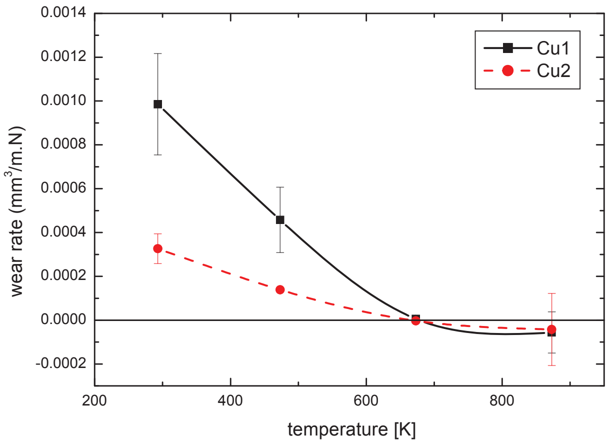


Figure 6. Temperature variance of wear rates.

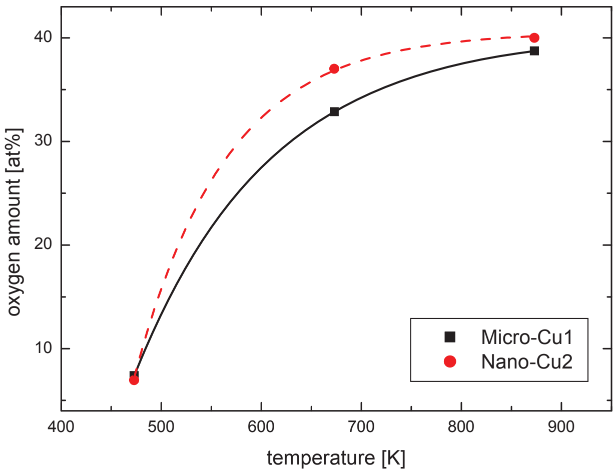


Figure 7. Concentration of oxygen (in atomic %) on the surfaces of both materials tested at various elevated temperatures.

stable at all temperatures till the end of the test, up to 100 m sliding distance (nearly 8000 laps), except 873 K, where for both materials after the initial stage the COF decreased to nearly 0.4 and then it was very slowly increasing during the whole test. The average values of COF are plotted as function of temperature in Figure 4. The tendency in both materials is similar, there is slight increase of COF at 473 K and significant decrease at higher temperatures. The Cu1 here showed higher friction. At 873 K both materials started to behave nearly identically, which can be seen also from the development of the COF along the wear distance (Figure 3).

After finishing the testing the wear tracks were observed and measured by optical microscopy and profilometry, in order to quantify the wear resistance. Figure 5 shows optical micrographs of the wear tracks. The development of wear damage with temperature can be immediately appreciated as the wear tracks become narrower with increasing test temperatures. Figure 6 compares the wear rates of the two materials at the testing temperatures. It shows that the material Cu2 was at lower temperatures about 3 times more wear resistant than the Cu1. This finding is analogous to literature data [8] for pure copper with submicro- and nanocrystalline microstructures. At higher temperatures both materials behave almost identically. They do suffer the wear damage, but the wear tracks are very thin and hardly any penetration into the material is found. At 873 K even deposition of ferrous oxides could be observed on some parts (negative values of depth with high scatter). This significant drop of wear damage between 473 K and 673 K is partly related to the recrystallization process which for Cu-5Al_2O_3 occurs at about 673 K [1]. The material becomes susceptible to strong deformation and creates a superplastic tribological film in the contact zone. Furthermore, at testing in air at elevated temperatures, i.e. in an oxidizing environment, the wear behavior of copper-based materials with high level of oxidation depends on the rate of oxidation and on thickness, morphology, adherence, and toughness of the oxide layer that forms on the surface of the material during the oxidation process [9–11]. In our case, with increase of temperature oxidation intensifies according to the parabolic law of oxidation (Figure 7), as it was confirmed by EDX. Very similar concentrations in both materials suggest that the oxidation is governed by volume processes rather than by the grain boundary diffusion. The formation of the hard oxide layer (Cu_2O) affects the wear in such a way that the mechanisms dominant at room temperature (extensive yielding and plastic shearing and/or material transferring to the pin) are less important and oxidized surface layer mechanisms based on adhesion and brittle fracture become more significant.

Wear of the spherical steel pin was also observed and evaluated but here only little changes were seen.

4 Conclusions

The results of the experiments allow to conclude the following.

For both materials coefficient of friction decreased at 673 K. The friction of Micro-Cu-Al_2O_3 tended to be slightly higher than that of Nano-Cu-Al_2O_3.

At lower temperatures the nano-grained material was about three times more wear resistant than the micro-grained one. With increasing temperature the wear rate decreased and at and above 673 K it reached zero. This is caused mostly by formation of hard oxide layer on the surfaces of both experimental materials.

Oxidation of both materials follows the parabolic law and has very similar profile for both materials which suggests that the oxidation is dominated by volume diffusion.

Acknowledgments

This work was realized within the frame of the project "Centre of Excellence of Advanced Materials with Nano- and Submicron- Structure", which is supported by the Operational Program "Research and Development" financed through European Regional Development Fund. The experiments could be carried out thanks to the projects VEGA 2/0120/10, 2/0025/11 and COST Action MP0701.

References

[1] M. Besterci, L. Kováč., *Int. J. of Materials and Product Technology*, **18** (2003), 26.

[2] H. Gleiter, *Nanostructured Materials*, **1** (1992), 1.

[3] R. Z. Valiev, N. A. Krasilnikov, N. K. Tsenev, *Mater. Science Eng. A*, **137** (1991), 35.

[4] L. Kunz, P. Lukáš, M. Svoboda, *Mater. Science Eng. A*, **424** (2006), 97.

[5] ISO 20808:2004(E), "Fine ceramics – Determination of friction and wear characteristics of monolithic ceramics by ball-on-disc method", (2004).

[6] `http://www.engineeringtoolbox.com/friction-coefficients-d_778.html`

[7] E. Marui, H. Endo, *Wear*, **249** (2001), 582.

[8] F. A. Sadykov, N. P. Barykin, I. R. Aslanyan, *Wear*, **225–229** (1999), 649.

[9] R. G. Bayer, "*Wear analysis for Engineers"*, HNB Publishing, New York, 2002.

[10] B. Bhushan, "*Principles and applications of tribology*", J. Wiley & Sons, New York, 1999.

[11] N. Krishnamurthy, M. S. Murali, P. G. Mukunda, *High Temperature Materials and Processes*, **29** (2010), 111.