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Dry sliding wear behaviour of Cu based composite materials reinforced with alumina fibers

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ABSTRACT

Purpose: Parameters for new manufacturing route of Cu casting reinforced with alumina fibers were elaborated. There was observed improvement of hardness and wear properties of composite materials comparing to the unreinforced copper and this indicates for the proper applied process parameters.

Design/methodology/approach: Manufacturing of composite materials involves two stages, preparation of porous preforms and next their infiltration with molten Cu. Preforms exhibits semi-oriented arrangement of fibers and their open porosity makes possible the production of composite materials with 10 and 20% by volume of Al_2O_3 fibers (Saffil). Wear tests were carried out applying the pin-on-disc concept at constant sliding velocity and under two different pressures. Specimens were pressed against the cast iron counterpart prepared from standard brake disc material.

Findings: Reinforcing of pure Cu with ceramic fibers results in the significant increase of hardness both by reducing the grain size and creating high level of residual stresses due to thermal mismatch of composite components. Fibers improves effectively wear resistance and under lower pressure of 0.2 MPa, in relation to unreinforced Cu, composite with 20% of fibers exhibits 6 times lower volume lost. Under smaller pressure wear process proceeded with plastic deformation of subsurface, cracking of reinforcement and transferring such segments to friction surface. Wear products containing hard fragments of alumina fibers as well as iron and copper oxides are transferred between surfaces and abrade weared parts. Thus only after friction against composite with 10% of fiber wear of iron counterpart was relatively small.

Research limitations/implications: Reinforcing of Cu by squeeze casting method requires application of the die from high temperature resistant steel tool. Preform preheated to high temperature before infiltration, should be transferred to the mold very quickly in order to keep temperature.

Practical implications: Reinforced copper , locally reinforced, exhibit high hardness and wear resistance under applied pressures. Production of electronic devices where simultaneously the high thermal and electric conductivity and good wear resistance are required can be potential area of future applications.

Originality/value: Investigations are valuable for persons, who are interested in Cu cast composite materials reinforced locally with ceramic fiber performs.

Keywords: Squeeze casting; Cu matrix; Saffil fibers; Composite materials; Wear

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MATERIALS

1. Introduction

During the manufacturing process of composite materials preparation methods of reinforcement and creation of proper bonding at the interface matrix/strengthening materials is of large importance. In order to achieve desired properties and fulfill the demand of the customers from industry, process parameter and chemical compositions of applied materials are determined and improved. Nowadays, applied composite materials in automotive, aircraft or electronic industry usually reveals high strength at the wide temperature range, high wear resistance and low thermal conductivity. These features of materials enable to improve durability, lower the weight in the case of vehicle parts, save energy and reduce the noise level [1-3]. Generally, composite materials are designed for particular application and should reveal some selected properties.

Nowadays among various methods of manufacturing, various composite materials casting processes are intensively developed. Usually preheated molten metal alloys infiltrate porous preforms, which are characterized by the skeleton structure and open porosity. The reinforcements are typically made from ceramic, intermetallic or carbon based materials, which in separate step are processed into porous structure. Manufacturing of strengthening materials is realized by firing of components and forming channels through which, in the next stage, liquid metal can infiltrate the preform [4-6]. In the case of fiber preform skeleton structure should exhibit possibly small amount of barriers formed usually from silica or phosphor based binders [7-8].

In the most frequently applied squeeze casting method rapidly flowing metal could be hindered by reinforcement, what generates large forces effecting on the deformation of ceramic preforms. Therefore, properties of preforms and process parameters affect microstructure, level of defects, interface between the reinforcement and the matrix and finally can enhance physical and chemical properties.

In many applications composite materials should demonstrate high strength and good wear resistance in wide temperature range and at different conditions [9-12]. Dry friction under changing pressure is frequently observed in practical application, so reinforcing of ductile Cu with hard particles or fibers could be useful and beneficial. In this study pure Cu was reinforced with alumina fibers (Saffil) using squeeze casting method for which proper parameters were elaborated. Considerable attention was paid to determination of reinforcing effect of fibers and the wear mechanism.

2. Experimental procedure

Composite materials were produced by pressure infiltration (squeeze casting) of porous ceramic preforms. First, from alumina Saffil fibers (see Table 1) preforms with controlled 80 and 90% porosity were produced. Saffil fibers contain ca. 3-4% of silica which constrains aluminum oxide grain growth at high temperatures as well as enhances the resistance of the fibers to chemical corrosion. Loose fibers were mixed in aqueous solution of silica binder to produce homogeneous mixture. Next by forming and dehydratation operations the rectangular 10x40x60 mm shapes were produced. Finally after drying preforms were fired at 1000°C in order to produce the strong joints between fibers and this structure can resist high loads during pressure infiltration. Preforms exhibit a skeleton structure, with disorderly arranged fibers in the horizontal plane, whereas the fibers are partially ordered in the vertical planes.

Table 1.
Chemical composition and properties of Al_2O_3 Saffil short fibers

Compounds	
Al_2O_3 - δ	96-97%
SiO_2	3-4%
shot content	negligible
Physical properties	
melting point	> 2000°C
maximum operation temp.	1600°C
tensile strength	1500 MPa
Young's modulus	300 GPa
mean diameter	3.5 μm
mean length	200 μm
density	3.3 g/cm3

Manufacturing of composite materials based on squeeeze casting method includs preheating of preform, mounting in the die, pouring and pressing of liquid copper until complete solidification. Basic parameters affecting quality of copper based composite materials are as follows:

- pressure infiltration 95-100 MPa,
- pouring temperature 1150°C,
- temperature of the die 500°C,
- preform temperature 1050°C.

Applied pressure and temperature parameters essentially affect interface quality between the matrix and the reinforcement and final composite properties. Therefore microscopic investigation of microstructure with analysis of porosity and defect quantity were performed. Microstructure and chemical compositions were examined with an optical and the scanning microscope Hitachi S-3400N.

For wear examinations cylindrical specimens were cut out and turned to diameter of 8 mm. Friction surfaces of the counterpart and composite materials were polished with abrasive paper 800 and cleaned with acetone. Composite specimens were pressed against gray cast counterpart with forces corresponding to pressures of 0.2 and 1 MPa. The tests were performed applying the device working on *pin-on-disc method* with horizontal rotating disc at constant linear velocity of 1m/s. After friction distances of 100, 500 and 1000 m the specimens were measured and weighed with accuracy of 1 mg. To evaluate the wear effect of cast iron counterpart profile of friction path was measured by means Talysurf 120L Rank Taylor Hobson profilometer.

3. Discussion of test results

3.1. Hardness HB and wear rate

Properties of composite materials strongly depends on preform structure and interface bonding created during infiltration. Therefore designing and manufacturing of preforms should aim to develop open porosity with desired volume of reinforcement and strength required for pressure infiltration. According to applied process silica phosphorus or alcohol binder can be used. In presented investigation after firing silica binder usually formed small but durable joints between the Saffil fibers. This was confirmed by appearance of fracture surfaces after bending tests. Final fracture usually preceded with crack of fiber, but not by break of joint. The wetting angle of the applied binder on alumina is small and the binder easily spreads on fibre surfaces and creates durable structure of preform. Though shape and surface of joints observed at SEM seems to be smooth and regular, the further examinations performed by means of Atomic Force Microscopy (AFM) show that their physical properties are inhomogeneous. Lateral Force Microscopy method (LFM) of surface analyzing of friction between the probe tip and the sample gives valuable information about this phenomenon. Fig.1a shows the Saffil fibre and its seemingly irregular - rough surface that reflects only friction characteristic. Similar changes of resistance against the probe tip movement can be observed on the fibre fracture, unlike in joint area, where visible distinct grains correspond with severe property micro-changes. It could be ascertained that binder joint reveals irregular properties when also considering hardness and elasticity. Presented at Fig. 1b fibre joint has the light elastic areas between dark grains of harder spots. During preform manufacturing binder spreads over entire surface of fiber forming thin layer of 80-100 nm thickness revealed by TEM examinations, but at the fibre contact created relatively thick joints reveals inhomogeneous properties what can affect the fracture progress and preform strength.

The characteristic properties of Cu or Cu alloys are the relatively high ductilities compared with other cast alloys, such as cast steel or ductile cast iron. Additionally, high electric and thermal conductivity makes this material interesting for the electronic applications. On the other hand, low hardness and small wear resistance of copper need to develop the special material microstructure and reinforcing method. Cast materals produced in metal die and under pressure, usually exhibit relatively fine microstructure, low porosity and relatively good mechanical properties. Squeeze casting method, applied in this work for manufacturing of composite materials, is generally applied in the industry for manufacturing of simple or complex parts from aluminum or magnesium alloys. Infiltration of porous preforms with Cu characterized by the significantly higher melting point than mentioned light alloys, as a new idea, was connected with large effort in order to elaborate process parameters and produce sound microstructure of composite Cu casting. Pouring of molten Cu (characterized by the temperature over 1100°C) into relatively cold die (450-500°C) can result in strong preheating of the steel wall, harmful drawback treatment and fast cooling of metal limiting complete infiltration. Thus, to protect the die, new construction was elaborated and special insulating coating applied. Some process operations were shortened whereas time of holding metal in liquid state and then infiltration prolonged. The structure of metal is affected by melting process, cooling rate and applied pressure. Using induction furnace with nitrogen protecting atmosphere allowed for quick preparation of molten Cu with low content of slug. However, during operating and transfer of molten metal to the mold it was subjected to surrounding atmosphere and then oxygen was absorbed and diffused inside the copper. Therefore at the grain boundaries Cu_2O-Cu eutectic was observed, (see Fig. 2a). Such oxides could be treated as reinforcing element and in some *in-situ* processes [13] after introducing Al alloying element chemical reaction leads to formation of Al_2O_3 reinforcement. Infiltration of porous preform and solidification inside it causes reduction of Cu grain size and spreading of eutectic clusters, (see Fig. 2b).

a)

b)

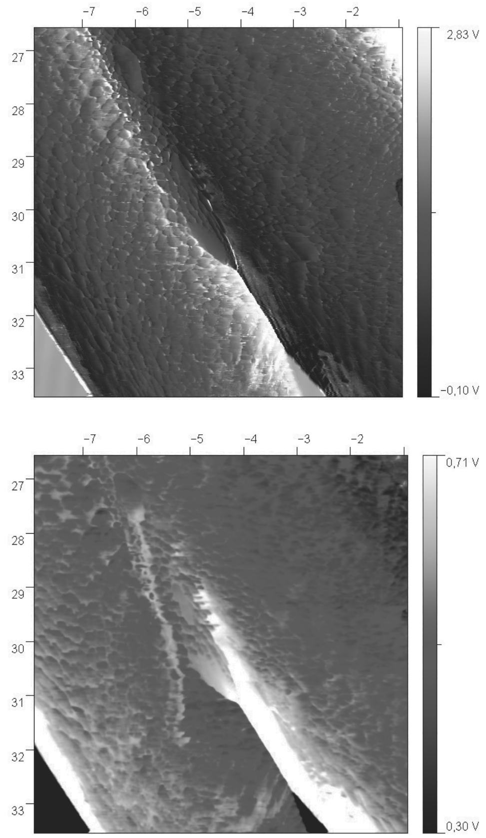


Fig. 1. Preform from Saffil fibres, a) LFM (Lateral Force Microscopy) reflecting friction forces at the joint between fibers in porous preform, b) FMM (Force Modulation Microscopy) of joints representing hardness map

Essentially, such microstructure components can improve wear resistance, though strength properties can be reduce because crack can easily develop through the intercrystalline areas. Fibres penetrate and join matrix grain so stresses are efficiently transferred. Prepared preforms were characterized by partly ordered fibre structure, therefore composite materials produced on their basis have two planes; one with semi parallel and second semi vertical oriented fibres, (see Fig. 3). Because no pull-out effect is observed, it could be ascertained that applied manufacturing method allows to produce the strong interface matrix-reinforcement. Visible fibres in the fracture are in some places covered with small particles of reaction products. Chemical analysis has shown the formation, on the base of silica binder used for manufacturing preform, complex compounds containing partly dissolved SiO_2 and Cu_2O originated from eutectic.

Wear investigations involved hardness, wear volume and counterpart friction profile measurements. Results of HB hardness, which are summarized in Table 2, confirmed high reinforcing effect of Saffil fibres. Just 10 vol.% of fibres improve hardness almost two times. It is caused by refinement of the matrix, strain hardening and a typical load transfer from the matrix to the fibres. Increased hardness should reduce wear rate according to the following formula [14]:

$$W = \frac{KdP}{3H} \qquad (1)$$

where K - wear coefficient , d - friction way, P - applied normal load and H - hardness of composite materials.

a)
b)

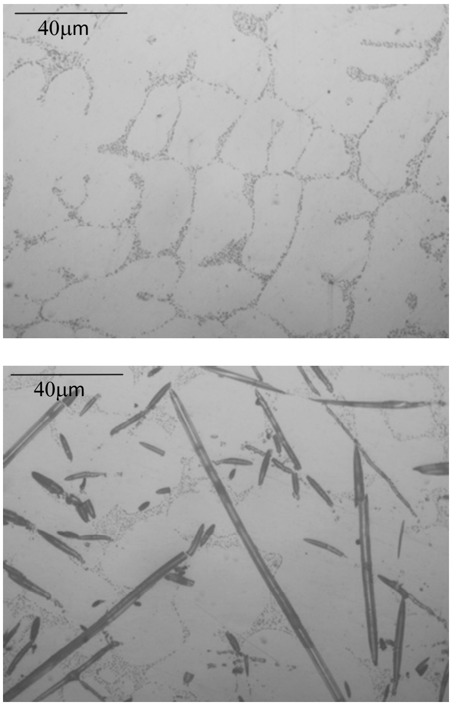


Fig. 2. Microstructure of squeeze cast copper (90MPa), a) without strengthening fibres, b) strengthened with 10 vol. % of Saffil fibrs

Therefore it could be anticipated that wear rate should be inversely proportional to the hardness and similar for composite materials with almost the same ca. 120 HB hardness. Moreover 5 times higher load (from 0.2 MPa to 1 MPa) should result in 5 times higher degree of wear.

Under larger load of 1MPa wear improvement, as a result of fiber strengthening, is still pronounced, though resistance to wear of composite materials is only 2 times better, (see Fig. 5). Unreinforced matrix is worn by 8-12 units. Probably, rubbing of debris into friction surface protects them against excessive friction. Composite materials irrespectively of fibre content (10 or 20%) shown similar resistance.

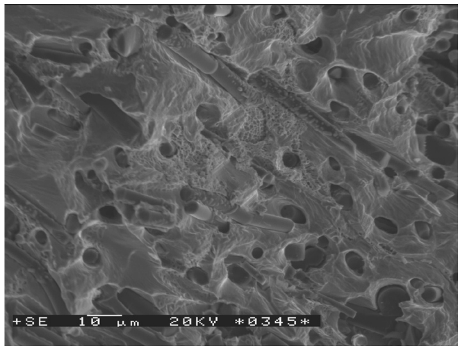


Fig. 3. Fracture of Cu based composite materials reinforced with 10 vol. % of Saffil fibres

Table 2.
HB hardness of unreinforced Cu and Cu with Saffil fibres

Materials	HB
Cu matrix	65
Composite with 10 vol. % Saffil fibres	119
Composite with 20 vol. % Saffil fibres	122

Application of force acting on the sample made of copper with 10% of Saffil ceramic fibres to the counterpart (resulting pressure of 0.2 MPa) results in significant improvement of wear resistance comparing to the unreinforced copper in the entire friction distance, (see Fig. 4). In relation to the unreinforced Cu matrix composite materials exhibit ca. 4 times smaller volume loss. Similar improvement was observed in the case of Cu-20% composite material, though difference between these types of composite was relatively small. Observed increasing of the wear rate for unreinforced Cu along friction distance (from 8 to 6 units) can result from rubbing of wear products (debris) into the surface what can protect it from adhesive tack and scuffing.

The fibres on the one hand harden the friction surface and on the other hand they are transferred between rubbing pair and act as loose abrasive materials contributing to increased wear. It can be concluded that 10 vol. % of the fibers in the copper matrix is sufficient to improve the tribological properties of composite material over a wide range of loads and wear distances.

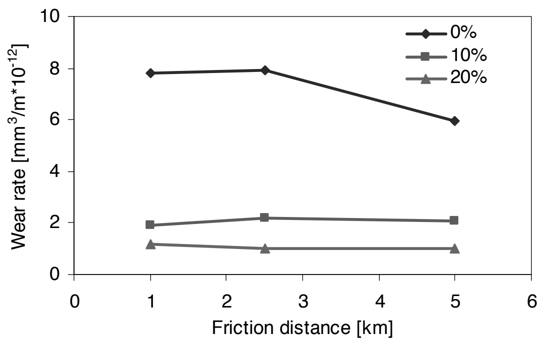


Fig. 4. Wear rate of unreinforced Cu (0%) matrix and composite materials reinforced with 10 and 20 vol. % of Saffil fibres tested under the pressure of 0.2 MPa

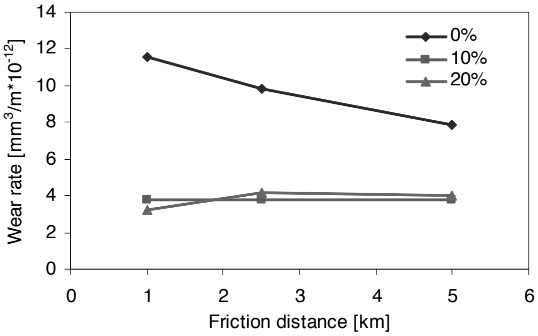


Fig. 5. Wear rate of unreinforced Cu (0%), and composite materials reinforced with 10 and 20 vol.% of the Saffil fibres tested under the pressure of 1 MPa

Measured friction coefficients stabilized after reaching of 2.5 km wear distance, (see Fig. 6). The lowest coefficient was observed when composite material reinforced with lower volume of fibers (10 vol.%) was tested. Probably smaller grooving of counterpart by composite material and smoothing them resulted in better mutual slipping. Higher fibres content means more hard Al_2O_3 particles between a rubbing pair and increase of roughness. For both materials, the friction coefficients are very similar of about 0.7. In turn, in the case of unreinforced matrix small Cu particles tack and adhere to the surface, which becomes more rough.

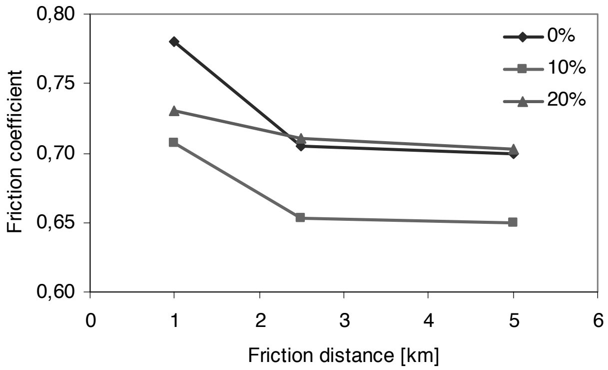


Fig. 6. Friction coefficient of unreinforced Cu and copper based composite materials with 10 and 20 vol.% of the Saffil fibres tested under pressure of 0.2 MPa

3.2. Microscopic observations of friction surface

Wear rate of unreinforced Cu matrix was similar under lower 0.2 MPa and higher 1MPa load. In both cases, the plastic flow and formation of characteristic rim around the cylindrical sample tip was observed, (see Fig. 7a). Abraded counterpart introduced iron and graphite compounds between rubbing surfaces, acting as a lubricant. Friction under lower load has more abrasive characteristic and relatively small amount of wear products are rubbed into the surface. As a result of direct contact with counterpart smearing and adhesive seizure takes place. Under higher load wear products can not be pushed out and then they are embedded directly into the friction surface. As a result of strong plastic deformation of subsurface, transport of wear products is observed, (see Fig. 7b). By this way 20-30 μm layer with changed composition and properties develops. This can be a reason of relatively high wear resistance of pure Cu in pair with cast iron counterpart.

Wear process for composite materials proceeds in different way under low pressure of 0.2 MPa, than under higher pressure of 1 MPa. Because abrasion of friction surface is relatively low, the microstructural transformations related to reinforcement destruction and plastic deformation of Cu are clear observed. Under wear surface fibres cracking and their fragmentation take place and moreover fibres align with the matrix according to matrix plastic deformation, (see Fig. 8). This zone of plastically deformed Cu mixed with crashed fibers extends to depth of 30-40μm. On the other hand on the surface iron oxides (visible as blue-gray fragments) from counterpart are smeared and rubbed. Usually they are treated as a lubricant medium, separating friction pair and protecting against seizure [15,16].

With the progress of the wear cracks develop in the deformed layer according to large plastic strains and fragments of layer can be peeled out. Under this layer various kinds of microstructure defects, in vicinity of porosity or broken fibres are places for additional crack development, (see Fig. 9). Cracks passing parallel to friction surface results finally in delamination of subsurface and production of flaky debris.

a)
b)

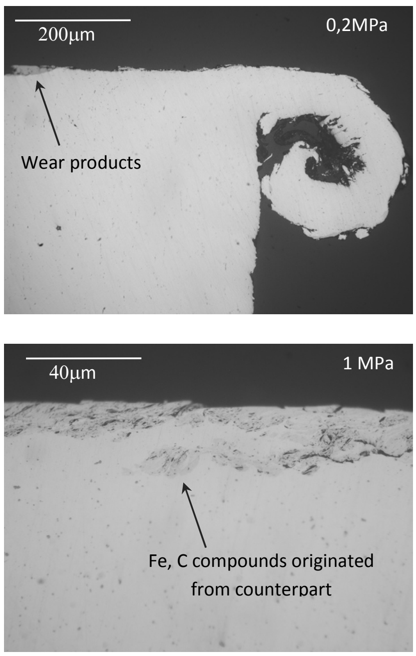


Fig. 7. Microstructure of unreinforced Cu subsurface worn under 0.2 MPa (a) and 1 MPa, visible transport of wear products inside specimen (b)

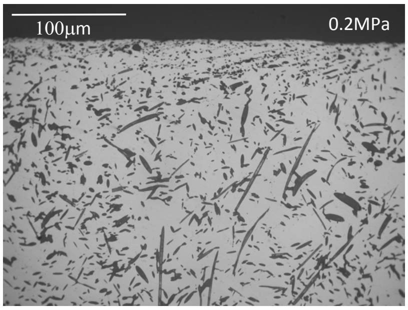
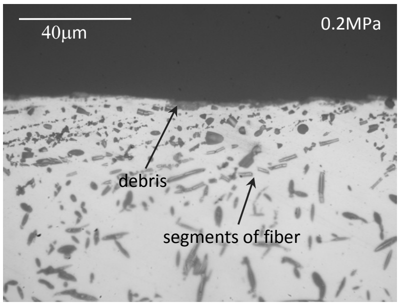


Fig. 8. Subsurface microstructure of composite materials $Cu+20\%Al_2O_3$ examined under 0.2 MPa load, vidiblefragmentation of fibers aligned according to friction direction

Friction under higher load of 1 MPa proceeds with grooving and abrasion of surface of composite materials on copper basis. Plastic deformation is observed in very limited thickness, because surface material is quickly removed by iron counterpart or hard Saffil fibre fragments, which shear and cut this layer in microscale, (see Fig.10 a,b). It could be ascertained, that process of mutual rubbing and cutting between friction surfaces, without lubricant, is relatively rapid.

a)



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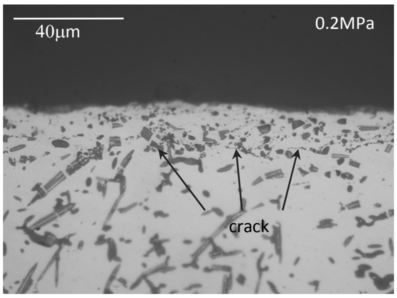
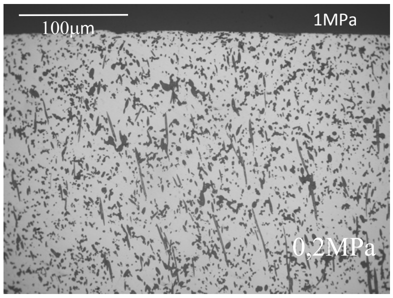


Fig. 9. Friction surface of composite material $Cu+20\%Al_2O_3$,
a) with embaged debris , b) development of crack under subsurface

The microscopic observations of the layers below friction surface allow to ascertain that the bonding between ceramic Saffil fibres and the copper matrix is relatively good. There was ascertained no pull-out of fibres from the copper matrix or generation of voids between fibres and matrix during very intensive plastic deformation of the layer below the friction surface. The heat formation and heat dissipation investigations during dry wear experiments are foreseen in the future.

a)



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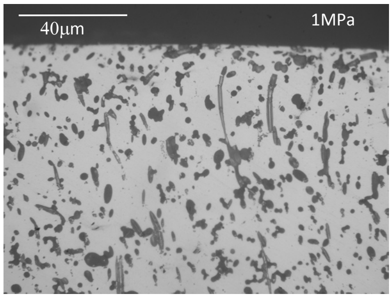

Fig. 10. Subsurface of composite material $Cu+20\%Al_2O_3$, a) worn under 1MPa load, b) small degree of plastic deformation in vicinity of friction surface at the same sample

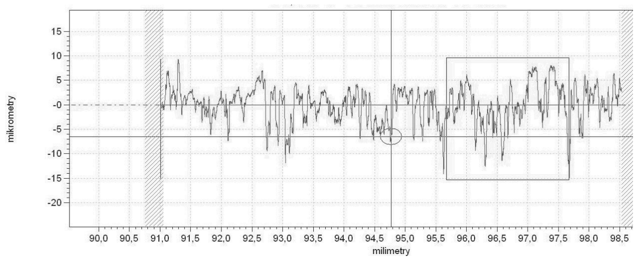

Fig. 11. Profile of cast iron disc after friction against unreinforced Cu, specimen under 0.2 MPa pressure

3.3. Investigations of wear path profile of cast iron counterpart

Wear rate of composite materials is 4-5 smaller than for unreinforced Cu. Moreover, composite materials containing 20 vol.% of fibres were worn two times slower than this one with 10 vol. % of fibres. An important consideration for the application of composite materials is the surface state and wear of counterpart. Cast iron discs used in a pin-on-disc machine were examined to determine depth of friction path profile with its roughness. Profile of path after test against unreinforced Cu pin was not clear visible. Surface was almost identical like in the other places on the disc, (see Fig. 11)

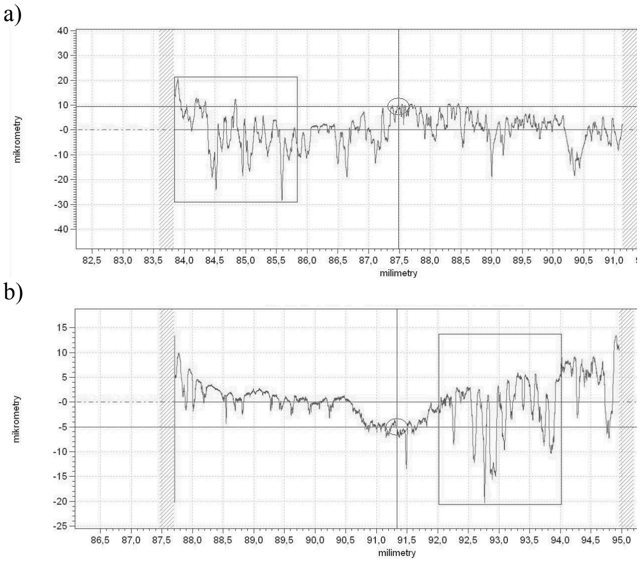

Fig. 12. Profiles of cast iron disc after friction with composite materials Cu-10 vol.% Saffil, a) under 0.2 MPa pressure, b) under 1MPa pressure

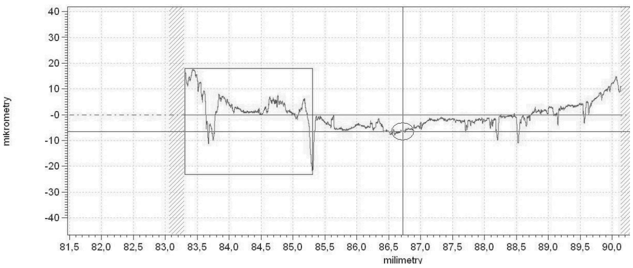

Fig. 13. Profiles of cast iron disc after friction with composite materials Cu-20 vol. % Saffil under 0.2 MPa pressure

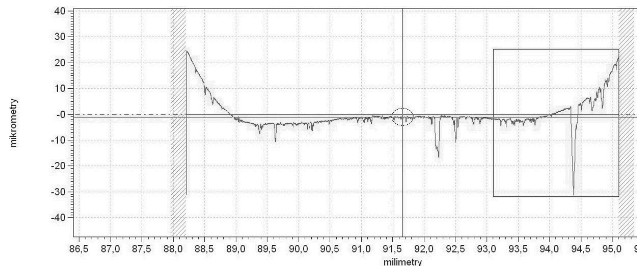

Fig. 14. Profiles of cast iron disc after friction with composite materials Cu-20 vol. % Saffil under 1.0 MPa pressure

Friction of composite material Cu-10 vol.% Saffil fibres under the pressure of 0.2 MPa also results in slight wear of cast iron disc, (see Fig. 12a). No clear groove on friction path was observed, only roughness determined with Ra factor increased from 2.9 for pair with unreinforced Cu to 5.2 with Cu-10 vol.% Saffil fibres. Only application of higher load 1 MPa caused grooving of disc to the depth of ca. 20 μm, (see Fig. 12b). Fragmented fibres, as a loose material, were transferred between friction pair and then micro cut and partly smoothed surface of disc. Factor Ra decreased to 3.4. Higher 20 vol. % content of fibres resulted in faster wear of counterpart even under lower load of 0.2 MPa. Depth of groove ranges between 15-20 μm, (see Fig. 13), whereas factor was Ra = 2.2. Under higher load depth of groove, in shape of letter u, increased to 25-30 μm, (see Fig. 14). Smoothing of surface proceeded, Ra=1.9, whereas loose broken fibers polished and simultaneously as a hard particles grooved it. It is evident that the new friction pair should be chosen for the further investigations, because of large wear of cast iron counterparts. The new counterpart should be from the group of metal alloy based composite materials strengthened with ceramic particles preferably Al_2O_3 or SiC.

4. Conclusions

Parameters of squeeze casting method for manufacturing Cu based composite materials reinforced with Al_2O_3 Saffil fibres were elaborated. Produced materials were subjected to dry wear examinations. Analyzing wear test results under different pressures and performing the microscopic observations of surface layer the following conclusion can be drawn:

1. Application of the squeeze casting method allows for reinforcing of Cu castings with Saffil ceramic fibres and interfaces fiber-matrix are characterized by good bonding.
2. Hardness of composite materials increased about 2 times in relation to unreinforced Cu.
3. Reinforcing of pure Cu with Saffil fibres improved wear resistance under low pressure of 0.2 MPa and under high pressure of 1.0 MPa between composite samples and cast iron discs.
4. The significant decrease of wear rate, about 10 times comparing to unreinforced copper, was observed under low pressure of 0.2 MPa for composite materials reinforced with 20 vol.% of Saffil fibres.
5. For composite materials reinforced with 10% of fibres the friction coefficient slightly decreased from 0.7 (at the wear distance of 1 km) to 0.65 (at the wear distance of 2.5-5.0 km).
6. Friction of unreinforced Cu proceeded with plastic deformation of surface grains and rubbing of wear products.
7. Subsurface of composite materials under lower pressure of 0.2 MPa was plastically deformed with flowing of broken fibers. Increase of pressure to 1.0 MPa caused higher rate of surface abrasion.
8. Surface of counterpart was intensively worn under high pressure of 1.0 MPa by composite material reinforced with 20 vol. % of Saffil fibres.

Acknowledgements

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