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Characterization of Cu/SiC surface composite produced by friction stir processing

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Abstract. The main aim of this work was to obtain a copper matrix surface composite using friction stir processing (FSP). The reinforced phase was SiC particles with an average size of 5 μm. The effect of the reinforcement on the microstructure, hardness and wear behaviour were analysed. The friction treatment was carried out using a truncated cone-shaped tool with a threaded side surface. Multi-chamber technology was used to produce the composite microstructure in the copper surface layer. Changes in the material microstructure were assessed by light microscopy and scanning electron microscopy. Comparative measurement of the hardness of the initial and treated material as well as wear resistance tests were also carried out. A favourable effect of the surface treatment on the microstructure and properties of the copper was found. As a result of the friction treatment there was strong grain refinement in the copper surface layer. The average grain size in the stirring zone was about 3 µm and was over 21 times smaller than the average grain size in the initial material. Intensive dispersion of the SiC particles in the modified layer was also found, leading to the formation of a copper matrix composite. The effect of microstructural changes in the surface layer of the material and formation of the surface composite was an over two-fold increase in the hardness of the material and an increase in wear resistance.

Key words: copper, metal matrix surface composite, friction stir processing.

1. Introduction

One of the factors determining product's usable properties is its surface layer. This layer can be shaped in many ways employing various surface treatment technologies [1-3], including modern technologies using concentrated heat sources [4–6]. Friction stir processing (FSP) belongs to the solutions that are currently setting new directions in the method of constituting surface layers of engineering materials [7]. It is a solid state surface modifying technique in which the heat arising as a result of friction of a special tool on the sample surface leads to plasticization of the material. The plasticized material is displaced under the shoulder, then mixed and compacted. Changes in the microstructure of the material caused by FSP treatment are a consequence of the simultaneous interaction of heat and force and lead to the occurrence of dynamic recrystallization of the material in the stirring zone. The amount of heat generated during FSP is determined by a number of parameters, in particular the rotational speed and clamping force of the tool, as well as the shape and dimensions of the pin and the shoulder. The work tool usually has a cylindrical shape and ends with a pin whose shape determines the effectiveness of material mixing during FSP. FSP technology gives the possibility not only to make microstructural changes in the surface layer of the material, but also allows the production of a composite microstructure by introducing an additional phase in the form of particles or fibres into the modified zone [8–10]. The properties of the composite surface layer are in this case the resulting properties of individual components of the composite and their volume shares. In the case of FSP technology, a very wide range of reinforcing materials can be used, the application of which for example in liquid phase technologies could be impossible, e.g. due to the lack of wettability or adverse interactions between the components. There are currently numerous solutions that enable the introduction of reinforcing material in FSP technology. The most common is the "groove" method. It involves making a groove in the surface of the FSP treated material and placing the reinforcing material in it [8, 11]. In the first phase of processing, the operation of sealing the groove with the additional phase is performed with a tool without a pin. In the second phase of processing, proper FSP is performed with a tool equipped with a pin. The need to perform the groove closing operation results from the risk of uncontrolled movement of the additive material outside the processing zone during penetration of the rotating tool into the material. "Groove" technology therefore requires the use of two different tools, and the processing in this case is two-stage. "Multi-chamber" technology is a competitive solution [10, 12]. In this method, the additional material is placed in chambers hollowed out in the sample surface. Individual chambers are separated from each other and act as containers cyclically delivering powder to the plasticized material. In contrast to the groove method, only one tool is used in multi-chamber technology, and processing can be in one or two stages. In multi-chamber technology, individual chambers are separated from each other by a wall, thanks to which the undesirable effect of the powder being pushed by the moving pin along the direction of movement of the tool is

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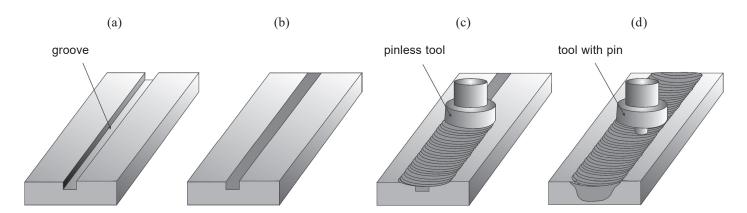


Fig. 1. FSP groove technology: a) groove design, b) groove filled with modifying material, c) pre-friction processing by tool without pin, d) proper friction processing by tool with pin [12].

significantly limited. This is achieved thanks to greater uniformity of the reinforcing phase distribution in the modified layer. Diagrams of the groove method are presented in Fig. 1, and multi-chamber in Fig. 2.

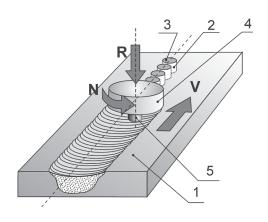


Fig. 2. FSP multi-chamber technology; 1 – modified material, 2 – chambers, 3 – SiC powder, 4 – mixing tool, 5 – pin [12]

The production of a composite surface layer is particularly justified when high surface hardness and abrasion resistance of the surface layer are required, while maintaining the ductile core. Copper is ideal for this purpose. Copper in its pure form has poor strength, wear and fatigue resistance hence, it is unsuitable for high end applications like the contact terminals of electrical switches and sliding surfaces [13]. Friction modification of copper with simultaneous fabrication of surface composites has been carried out by, among others, Mishra et al. [14]. They used friction stir processing to create metal matrix composites by consolidating graphite particles in a pure copper material matrix using a square pin tungsten carbide tool. The main emphasis in the work was placed on assessing the mechanical properties of the metal matrix composites reinforced with graphite particles. The authors found that the wear resistance of the surface composite was greatly enhanced compared to pure copper with a decrease in the coefficient of

friction. The hardness was found to decrease with an increase in graphite content at a constant traverse speed. The friction stir processing of copper with graphite powder was also the subject of research carried out by the authors of this paper [15]. In turn, in [16], friction stir processing was applied to fabricate boron carbide (B₄C) particulate reinforced copper surface composites. The authors analysed the effect of the FSP parameters such as tool rotational speed, processing speed and groove width on the microstructure and microhardness. The results indicated that higher tool rotational speed and lower processing speed produced an excellent distribution of the B₄C particles and that the higher area of surface composite due to higher frictional heat, increased stirring and material transportation. The B₄C particles bonded well with the copper matrix and refined the copper grains due to the pinning effect of the B₄C particles. In [17] the authors used ZrO₂ particles to produce a CuZrO₂ nanocomposite by friction stir processing. The groove method was used to produce the surface composite. The microstructure and mechanical properties of the composites were examined and compared with the base metal. The authors found that the use of ZrO₂ particles results in a greater reduction of the grain size in comparison with the processed samples without ZrO₂ particles. The hardness of the composite samples was about twice higher than the hardness of the base metal. The authors also showed that friction stir processing can increase the abrasion resistance by reducing the coefficient of friction.

On the other hand, Saravanakumar et al. [18] used FSP technology to create a copper matrix surface composite material reinforced with aluminium nitride particles to enrich the metallurgical and mechanical properties of copper. The authors showed that the microhardness value increased with respect to the percentage of reinforcement due to uniform distribution of the AlN particles and dynamic recrystallization. The research also showed a decrease in the trending wear rate value with respect to the increase in the percentage of reinforcement. The grain size of the FSPed copper surface composite was observed to be finer when compared to the base metal.

The fabrication of a surface composite in the surface layer of copper using FSP technology is an issue with a high cogni-

tive and application potential. However, using all the possibilities offered by FSP requires further systematic research and practical tests. As part of this work, FSP treatment of copper was carried out with simultaneous introduction of SiC particles into the copper surface layer using multi-chamber technology. Silicon carbide is a material with a number of valuable properties, which is used in surface engineering especially when there is a risk of abrasive and erosive wear. The use of SiC in the case of copper can significantly contribute to improving these parameters and increasing the application potential of copper.

2. Materials and experiment procedures

- 2.1. Materials. The research material consisted of cuboid samples measuring $100 \times 80 \times 10$ mm cut from commercially available pure copper flat bars. The experiment used SiC technical powder with an average particle size $\sim 30~\mu m$ and a polyhedral shape. The powder was ground in a FRITSCH Pulverisette 6 planetary mill for 1 hour. Grinding resulted in a powder with an average particle size of about 5 μm . This powder was used to produce a composite microstructure in the surface layer of the copper.
- **2.2. Friction stir processing.** Copper friction treatment was carried out using a vertical CNC milling machine. The FSP station is shown in Fig. 3. Multi-chamber technology was used to produce the composite microstructure. Chambers with a diameter of 2 mm and depth of 4.5 mm were hollowed out in the copper samples. Individual chambers were separated from each other by a wall whose thickness in the narrowest place was ~ 0.3 mm. The partition between the chambers could not be too thick because it would cause an above average increase in the share of matrix in the composite at this place. The main task of the partition was to reduce the risk of powder being pushed by the moving pin and thereby increasing the uniformity of SiC particle dispersion in the friction modified zone. The chambers were filled with SiC powder up to 3/4 of the chamber height and friction treatment was carried out using

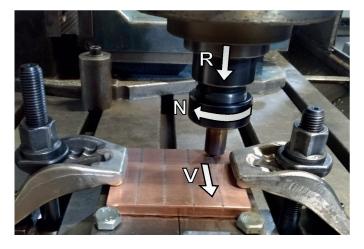


Fig. 3. Stand for the copper friction modification

a tool equipped with a pin. The pin length was 5.3 mm. The tool pin had the shape of a truncated cone with a threaded side surface. The tool was made of X37CrMoV5-1 hot work tool steel. The tool mounted on the tool holder was tilted from the straight line perpendicular to the sample surface by an angle of 2°. The tool was moved along the chamber axis after the time t = 3 seconds, measured from the moment of full penetration of the tool into the copper. This time was necessary to plasticize the copper. Single stir passes were used. The detailed parameters are summarized in Table 1. During the tests preceding the actual treatment, the rotational speed and travel speed of the tool were changed to determine the parameters at which copper plasticization and uniform distribution of the reinforcing phase would be possible, and so that the processing would not cause disqualifying changes in the geometric structure of the surface or excessive grain growth in the friction-modified zone. During the preliminary tests, treatment was carried out using N speeds equal to 800 rpm and 1000 rpm and travel speed V = 30 mm/min, in both cases obtaining very unfavourable changes in the geometric structure of the surface and the microstructure of the material.

Table 1
Main parameters of surface treatment

Process parameters	Values	
Tool rotational speed N (rpm)	1500	
Processing speed V (mm/min)	30	
Pin sinking speed R (mm/min)	6	
Tool tilt angle (°)	2	
Tool shoulder diameter (mm)	18	
Pin shape	truncated cone with threaded side surface	
Pin length (mm)	5.3	
Pin diameters (cover/bottom) (mm)	6/4	

A similar effect was obtained by increasing the tool travel speed to 60 mm/min. Based on the preliminary tests, it was found that at a tool rotation speed equal to or below 1000 rpm there is insufficient heat input and insufficient plasticization of the copper, leading to very unfavourable changes in the surface geometry and lack of the effect of mixing the SiC particles in the Cu matrix.

2.3. Macro- and microstructural analysis. The study material was subjected to macroscopic examination using an Olympus SZ61 stereoscopic microscope and microscopic examination using an Olympus GX41 light microscope as well as a Jeol JSM-6610LV scanning electron microscope. The samples for microscopic studies were ground, polished and etched. Microscopic observations were carried out on metallographic samples cut transversely to the direction of the tool movement. The place from which the metallographic sample was made

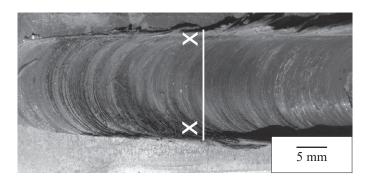


Fig. 4. Macroscopic effect of FSP

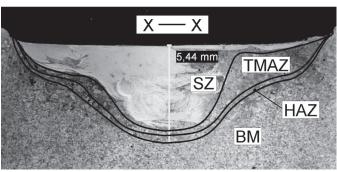


Fig. 5. Modified surface layer. Cross-section. Etched

is marked in Fig. 4 by the X-X line. This designation is also used in Fig. 5. EDS studies of micro-areas were also carried out using an EDS Oxford Instruments X-ray microanalyzer. A fracture for fractographic research was obtained by breaking a sample using Charpy hammer. The sample was previously cut on the side opposite the modified surface, and the cut depth was about 5 mm (the cut reached the bottom of the friction modified zone).

2.4. Hardness measurements and tribological tests. The material in its initial state and after friction modification was subjected to comparative hardness measurement. The hardness measurements were conducted on cross-sections of the friction stir processed zone using a Shimadzu HMV-G20 microhardness gauge with loads of 980.7 mN applied for 10 s. The tribological properties were measured using a pin-on-disc type laboratory tribometer under unlubricated sliding contact against a rotating steel ring (HRC 58–63). The samples were in the form of a pin with a diameter of 4 mm and were stationary during the tribological test. The pin was cut out of the material located in the middle of the band. In order to eliminate the influence of the running-in process of the sample, which took place in the initial phase of the test, the samples for the tribological tests were subjected to mechanical pre-treatment. This treatment consisted of removing a thin surface layer differentiating the surface roughness of individual samples by grinding it with 600 grit abrasive paper. The tests were run at the constant normal load of 40 N. The total wear distance was 1500 m. The friction force and linear material loss were recorded automatically against time by the tester software. The friction coefficient was calculated from the ratio of the friction force to the normal load.

3. Results and discussion

3.1. Macroscopic investigations The macroscopic effect of FSP treatment and the changes caused by it in the geometric structure of the surface are shown in Fig. 4. The samples experienced plastic deformation due to the frictional heat combined with the mechanical impact of the tool. As a result of friction processing, a band with a width of about 18.4 mm was obtained on the surface of the samples, corresponding approximately to the diameter of the shoulder. The depth of the zone of micro-

structural changes was about 5.5 mm and was approximately equal to the length of the pin (Fig. 5). On the edge of the band, characteristic outflows of material were found as a result of movement of a portion of the plasticized copper out of the tool working area. The resulting outflow is the result of sinking the shoulder below the surface of the processed material and indicates its plasticization. As can be seen in Fig. 4, the height of the outflow increased with the path travelled by the tool. As a result of friction processing, parallel grooves formed on the surface of the band, whose presence indicate the direction and way of plasticized material movement under the shoulder of the tool. No "tool dragging" was found in the analysed sample, the presence of which would prove insufficient plastic deformation of the copper and an insufficient amount of plasticized copper for the space behind the moving pin to be filled. This fact proves that the amount of frictional heat created during processing was sufficient in order to plasticize the material.

3.2. Microstructural investigations. The microstructure of the pure copper used in this study is shown in Figs. 6a and 6b. The material was solid and free from material defects such as microcracks or pores. This microstructure consisted of large grains with an average grain size of 65 μm. Sample grains have been dimensioned in Fig. 6b. However, very high grain refinement was found in the layer treated with FSP. The grain size in the stirring zone was in the range of 1 to 6 µm, with an average grain size of only 3 µm. Only in the narrow subsurface zone, whose thickness was about 100-120 μm, there was a lower degree of refinement, and the grain size in this zone was in the range from 3 to 20 µm, with an average grain size of about 15 μm. In Fig. 7b, examples of grains occurring in the central part of the stirring zone at a distance of about 500 µm from the surface have been dimensioned. In the subsurface zone, a significantly lower share of SiC particles was found than in the remaining part of the stirring zone, resulting in a lower hardness of the material at this point, which is discussed later in the study. Figure 7 shows the grain size in various places of the copper surface layer and at different depths from the surface of the processed material. The differences in the grain size at different places of the friction-modified zone were caused, it is supposed, by the differences in the size of the heat effect affecting the material during processing and the different time of its impact on the material. The main source of heat in FSP

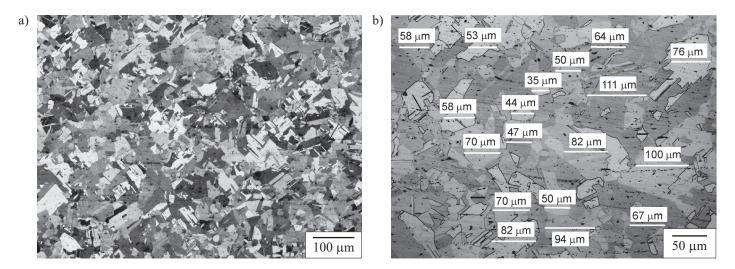


Fig. 6. Copper microstructure in its initial state

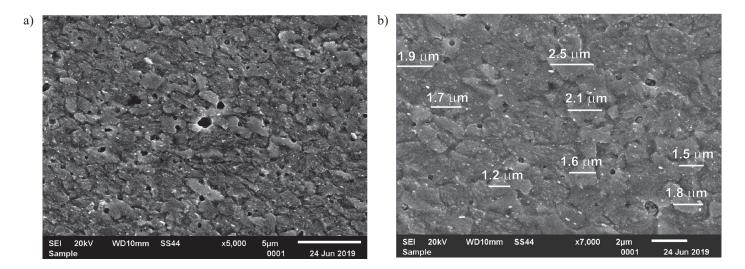


Fig. 7. Degree of refinement of copper microstructure, a) at distance of about 200 μm from surface, b) at distance of about 500 μm from surface. Etched. SEM

treatment is primarily the friction of the tool shoulder against the material surface, and to a lesser extent the friction generated by the pin. In the subsurface zone, the thermal effect generated during processing was greater, and therefore the heat of friction could have lasted longer on the material.

Thus, the cooling rate of the material located in the narrow subsurface area could be lower than in the rest of the sample, which resulted in less grain refinement at this point. Apart from the differences in the degree of grain refinement in different places of the stirring zone, it should be noted that the friction treatment caused very significant refinement of the copper microstructure in relation to the state before the treatment. The main reason for this was the dynamic recrystallization of the material caused by strong deformation of the material and the impact of high temperature. The grain refinement in the copper surface layer can also be attributed to the pinning effect of the SiC particles which impede grain growth by suppressing grain boundary sliding [16].

In the modified material, the presence of several zones characteristic for FSP treated materials was found, i.e. a clearly dominating stirring zone (SZ) with very fine and largely equiaxed grains (Figs. 7a, 7b); a thermomechanically affected zone (TMAZ) in which the grains had a more elongated shape (Figs. 8a, 8b); and a narrow heat affected zone (HAZ) bordering on the base material (BM). In the HAZ, grains larger than those observed in the SZ but smaller than in the BM were present. The transition zone between the TMAZ and the HAZ was characterized by a gradual increase in grain size. The microstructural investigations did not show the presence of "onion rings" in the friction modified zone.

Both the light and scanning electron microscopy studies showed that the SiC particles were intensively dispersed in the stirring zone during FSP treatment, and the average share of the reinforcing phase was $9.4\% \pm 1\%$ (Figs. 9a, 9b). Only at the border of the stirring zone, near the TMAZ, there was a higher share of the reinforcing phase recorded, which was almost twice

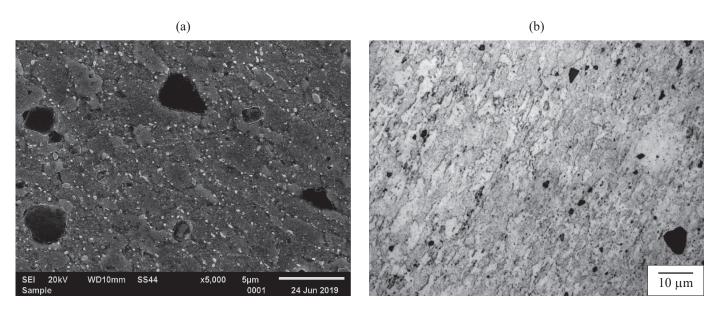


Fig. 8. Degree of refinement of copper microstructure in TMAZ. Etched. SEM (a), light microscope (b)

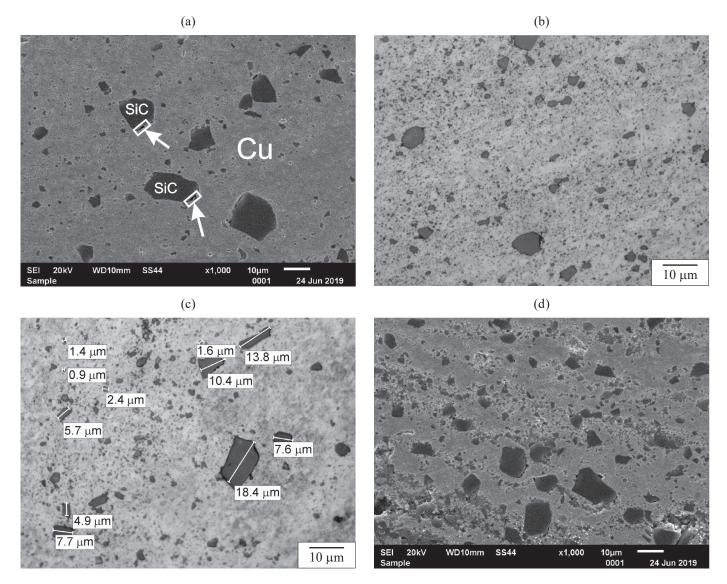


Fig. 9. Microstructure of the Cu/SiC composite in central part of SZ (a, b, c), in lower-lateral part of SZ (d). SEM (a, d), light microscopy (b, c)

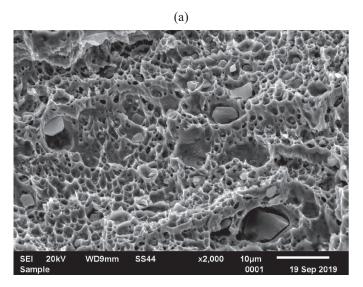
as high, i.e. $18.6\% \pm 3\%$ (Fig. 9d). A higher concentration of SiC particles, however, was recorded only in places where the material was affected by the threaded side walls of the pin. Nonetheless, there was no above average concentration of particles in the lower part of the stirring zone, i.e. in the workplace of the lower flat part of the pin. The lowest share of SiC particles was found in the narrow subsurface zone, whose thickness was about $100-120~\mu m$, this share was only $2.3\% \pm 0.5\%$. No gas bubbles were found in the analysed material. The material was solid and free of this type of material defects. During the SEM study, it was found that the SiC particles are surrounded by the metal matrix, and the matrix adheres to the particles in the vast majority on their entire surface.

This fact is important because the presence of numerous discontinuities at the Cu/SiC interface can lead to rapid particle chipping out of the matrix during the work of the element and to a decrease in its durability. There were no material discontinuities at the interface, or their presence was very limited (Fig. 9a), which did not significantly affect the properties of the composite. In the course of the microstructural tests it was also found that local discontinuities were only present for large particles of the order of 10 µm and larger (Fig. 9a). No intermediate layers were found at the Cu/SiC interface, the possible occurrence of which could indicate interaction between the metal matrix and SiC particles.

An important technological problem occurring especially in liquid matrix technologies is the possibility of the formation of unfavourable local aggregates of particles, i.e. agglomerates. The presence of agglomerates can cause, among others, a higher variation of hardness across the surface composite [16]. In agglomerates, the particles are not usually separated from each other by the matrix, hence such material usually has a lower resistance to cracking. Agglomerates are often accompanied by material discontinuities. These discontinuities are most often located in the middle of the agglomerate due to the impossibility or limited possibility of liquid matrix entering the agglomerate.

The agglomerate particles also show an above average tendency to chip from the metal matrix during work of the element, which reduces the operational value of the product. Therefore, in this study special attention was paid to the presence of this type of technological defects. An increased concentration of particles was found at the boundary of the stirring zone but these particles were separated from each other by the metal matrix, thanks to which they could effectively strengthen the matrix. The absence of agglomerates in the microstructure of the tested material indicates that the processing parameters, chamber geometry and the strengthening phase share were correctly selected for the type of material being modified.

As can be seen in Fig. 9c, the particle size in the composite surface layer varied widely, ranging from tenths of a micrometre to even over a dozen micrometres, with the dominating fraction being particles with an average size of 1–2 µm, and thus more than twice smaller than the average SiC particle size used in FSP treatment. This fact proves that during FSP treatment the powder underwent fragmentation. However, there were no differences in the shape of the powder before and after friction treatment. In both cases, equiaxed polyhedral shapes were dominant. The next stage of research included fractographic research. The nature of material fracture constitutes one of the criteria of the initial quality assessment of the material, its homogeneity and purity, susceptibility to cracking/breaking, mechanical properties, etc. The fractographic studies showed the plastic nature of the fracture and very good connection of the particles with the matrix. The number of places showing that the particles were chipping out of the matrix during fracture was negligible. This fact proves that the copper was sufficiently plasticized so that the SiC particles were thoroughly surrounded by the matrix and effectively "trapped" in it. During the fractographic examination, no microcracks or places that could indicate an increased cracking tendency were found. The material fracture was homogeneous throughout the entire mixing zone. Exemplary morphologies of the obtained fractures are presented in Fig. 10.



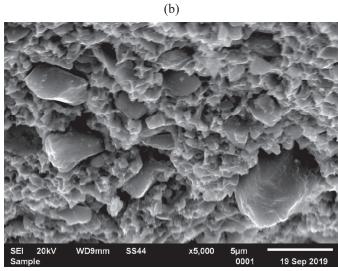


Fig. 10. Fracture of FSP treated sample

3.3. EDS results. EDS tests were performed to determine possible differences in the chemical composition of the material at different places in the stirring zone. Three representative micro-areas were analysed: the first of them was located in the upper part of the surface zone, i.e. at a distance of about 0.1 mm from the surface, the second was located in the central part of the stirring zone, i.e. at a distance of about 2.5 mm from the surface, and the third was in the bottom of the stirring zone, i.e. about 5 mm from the surface. The EDS tests showed high homogeneity of the chemical composition of the material in the surface layer, with the exception of the narrow subsurface zone, whose difference was a consequence of the smaller share of SiC phase in relation to the remaining part of the stirring zone.

During the tests, the presence of iron was also found (in an amount of 0.5 to 5% by mass), whose source was the tool material which wore off as a result of its contact with the hard SiC particles. In addition, a small amount of oxygen (from 0.75 to 1.13 mass%) was found, whose presence in turn should be explained by copper oxidation at the temperature conditions of the process.

An example of EDS analysis of the material located in the central part of the stirring zone is shown in Fig. 11a and Table 2, and the material located in the lower-lateral part of the stirring zone in Fig. 11b and Table 2.

Table 2 Results of EDS analysis

Element	Central part of SZ		Lower side part of SZ	
	Weight %	Atomic %	Weight %	Atomic %
Cu	83.68	60.81	65.10	34.98
Si	9.16	15.05	19.78	24.04
C	5.51	21.20	13.46	38.25
Fe	0.89	0.74	0.53	0.32
О	0.76	2.20	1.13	2.41

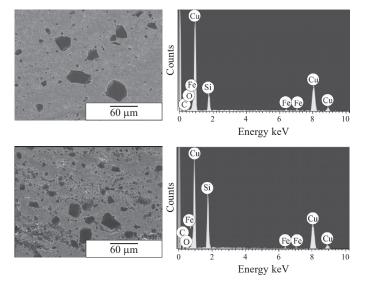


Fig. 11. Results of EDS analysis

3.4. Hardness results. Measurement of the hardness was for the authors of the work an important test of the effectiveness of the conducted treatment and correct selection of the process parameters. The hardness measurement was made on the transverse specimen as a function of distance from the friction-modified surface. In the surface zone, the measurement was made with a measuring step of about $100 \, \mu m$, while at a distance of about $1000 \, \mu m$ from the surface the measuring step was increased to $200-250 \, \mu m$. Three independent measurements were made, and the results of these measurements were used to gain knowledge about changes in material hardness as a function of distance from the surface.

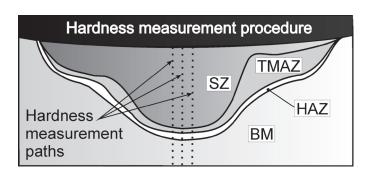


Fig. 12. Hardness measurement procedure

The applied procedure for measuring hardness is shown in Fig. 12. The tests showed a very significant increase in the hardness of the material subjected to friction modification in relation to the hardness of the starting material. The mean hardness of the starting material was 55 HV0.1 (the standard deviation value was 3.4), while the mean hardness of the material after friction modification was 118 HV0.1 (the standard deviation value was 16.7), thus it was more than twice as high as the hardness of the material before processing. Such a significant increase in hardness should be explained by very strong grain refinement in the friction-modified zone, and by the presence of SiC hard particles in the soft copper matrix. Nevertheless, the hardness distribution within the stirring zone was not uniform. The hardness recorded in the stirring zone ranged from about 90 HV0.1 in the narrow subsurface zone and about 75 HV0.1 in the lower part of the zone of microstructural changes to about 136 HV0.1 in the central part of the stirring zone. The lower hardness in the subsurface zone should be explained by the significantly lower share of SiC phase in this zone, and the increase in hardness observed in this case compared to the hardness of the starting material was mainly caused by grain refinement. In the central part of the stirring zone the material hardness was the highest and was ranged from 109 to 136 HV0.1 (the mean value was 125 HV0.1).

Such a significant increase in hardness was a consequence of both very strong refinement of the microstructure and the presence of SiC particles in the matrix. In the lower part of the zone of microstructural changes, the hardness was lower and ranged from 75 HV0.1 to 116 HV0.1 (the mean value was

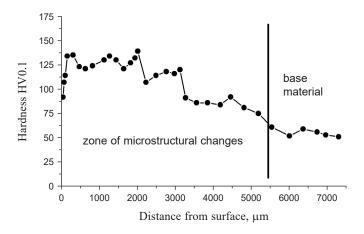


Fig. 13. Hardness measurement results

89 HV0.1). An exemplary distribution of material hardness as a function of distance from the surface is shown in Fig. 13. The significant increase in material hardness after friction modification indicates the desirability of using this technology for copper.

3.5. Wear results. During the tribological tests, the friction coefficient and the linear loss of material were recorded as a function of test duration. The tribological studies were comparative in nature as both FSP treated material and untreated material were tested. Fig. 14 shows the linear loss of material recorded as a function of friction time. As can be seen, in the initial phase of the test a larger linear loss of material was recorded for both samples.

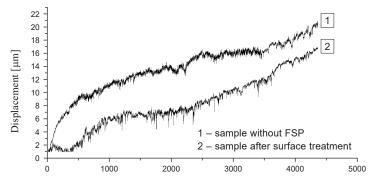


Fig. 14. Results of tribological tests

This was due to rubbing of the surfaces "sample-counter-sample" tribological pair. This effect occurred despite the prior sanding of a thin surface layer with abrasive paper, which was performed in order to obtain uniform initial conditions for samples subjected to friction modification and samples without treatment. The analysis of the obtained wear characteristics shows, however, that as the abrasion time passes, the linear loss of material was smaller in the case of the FSP treated samples compared to the linear loss of material recorded in the case of pure copper. The higher abrasion resistance is a consequence

of introducing the hard and abrasion-resistant SiC particles as well as favourable changes in the material microstructure, and above all, strong grain refinement. Notwithstanding, the material subjected to friction modification had a higher coefficient of friction by about 7%. The higher coefficient of friction is a consequence of introducing SiC particles into the microstructure.

4. Conclusions

The effect of friction stir processing on the microstructure and properties of copper was studied. As a result of friction treatment a copper matrix composite (CMC) was obtained. Based on the results and discussion above, the following conclusions can be made:

- 1. During FSP the copper undergoes plastic deformation due to the frictional heat combined with the mechanical impact of the tool.
- 2. Friction stir processing leads to the formation of a copper matrix composite (CMC) in the surface layer
- 3. As a result of FSP processing, there is strong grain refinement which is a consequence of dynamic recrystallization of the material.
- 4. The consequence of microstructural changes in the material caused by FSP treatment is the increase in hardness and wear resistance of the copper.
- 5. The material subjected to friction modification is characterized by a lower linear loss of material as a function of time than the material without processing but a higher coefficient of friction.
- Due to favourable microstructural changes caused by friction treatment, material with better properties and greater application potential is obtained.
- 7. The research and results indicate that FSP technology is an effective and promising solution allowing one to shape the microstructure and properties of the material and is an alternative to other methods and technologies used in surface engineering.

REFERENCES

- [1] T. Chmielewski, D. Golański, M. Hudycz, T. Sałacinski, and R. Świercz, "Surface and structural properties of titanium coating deposited onto AlN ceramics substrate by friction surfacing process", *Przem. Chem.* 98(3), 208–213 (2019) [in Polish].
- [2] J. Winczek, M. Gucwa, M. Mičian, R. Koňár, and S. Parzych, "The evaluation of the wear mechanism of high-carbon hardfacing layers", Arch. Metall. Mater. 64(3), 1111–1115 (2019).
- [3] M. Gwoździk and Z. Nitkiewicz, "Topography of X39Cr13 steel surface after heat and surface treatment", Opt. Appl. 39(4), 853–857 (2009).
- [4] Y. Li, S. Arthanari, and Y. Guan, "Influence of laser surface melting on the properties of MB26 and AZ80 magnesium alloys", Surf. Coat. Technol. 378, 124964, (2019).
- [5] M. Szafarska, J. Iwaszko, K. Kudła, and I. Łęgowik, "Utilisation of high-energy heat sources in magnesium alloy surface layer treatment", Arch. Metall. Mater. 58(2), 619–624 (2013).



- [6] J. Kusinski, S. Kac, A. Kopia, A. Radziszewska, M. Rozmus-Górnikowska, B. Major, L. Major, J. Marczak, and A. Lisiecki, "Laser modification of the materials surface layer a review paper", *Bull. Pol. Ac.: Tech.* 60(4), 711–728 (2012).
- [7] R.S. Mishra, Z.Y. Ma, and I. Charit, "Friction stir processing: a novel technique for fabrication of surface composite", *Mater. Sci. Eng.* A 341, 307–310 (2003).
- [8] N.E. Mahallawy, S. Zoalfakar, and A.A. Ghaffar, "Microstructure investigation, mechanical properties and wear behavior of Al 1050/SiC composites fabricated by friction stir processing (FSP)", Mater. Res. Express 6(9), 096522 (2019).
- [9] R. Vaira Vignesh, R. Padmanaban, and M. Govindaraju, "Synthesis and characterization of magnesium alloy surface composite (AZ91D SiO₂) by friction stir processing for bioimplants", Silicon (2019).
- [10] J. Iwaszko, K. Kudła, and K. Fila, "Friction stir processing of the AZ91 magnesium alloy with SiC particles", Arch. Mater. Sci. Eng. 77(2), 85–92 (2016).
- [11] R. Sathiskumar, N. Murugan, I. Dinaharan, and S.J. Vijay, "Fabrication and characterization of Cu/B4C surface dispersion strengthened composite using friction stir processing", *Arch. Metall. Mater.* 59(1), 83–87 (2014).
- [12] J. Iwaszko, K. Kudła, and K. Fila, "Technological aspects of friction stir processing of AlZn5.5MgCu aluminum alloy", *Bull. Pol. Ac.: Tech.* 66(5), 713–719 (2018).

- [13] S. Cartigueyen and K. Mahadevan, "Role of friction stir processing on copper and copper based particle reinforced composites a review", J. Mater. Sci. Surf. Eng. 2(2), 133–145 (2015).
- [14] R.S. Mishra, J. Anshul, V. Jegenathan, and M.S. Ranganath, "Synthesis of copper graphite composite using friction stir processing and evaluating parameters effecting hardness and wear", *Int. J. Res. Eng. Innov.* 1(3), 199–208 (2017).
- [15] Md. Noorul Hoda, R. Muttanna Singari, and V. Jeganathan Arulmoni, "Friction stir processing (FSP) of copper and enhancement of its mechanical properties using graphite powder (C)", *Int. J. Res. Sci. Innov.* III(IX), 58–65 (2016).
- [16] R. Sathiskumar, N. Murugan, I. Dinaharan, and S.J. Vijay, "Role of friction stir processing parameters on microstructure and microhardness of boron carbide particulate reinforced copper surface composites", *Sadhana* 38(6), 1433–1450 (2013).
- [17] B. Beglarzadeh and B. Davoodi, "Study the microstructures of nano-composite copper/zirconium dioxide under the process of FSW (Friction Stir Welding)", *Indian J. Sci. Technol.* 9(7), 1–11 (2016).
- [18] S. Saravanakumar, S. Gopalakrishnan, K. Kalaiselvan, and R. Sathiskumar, "Experimental analysis of copper matrix surface composite fabricated by friction stir processing", *Taga J.* 14, 298–305 (2018).