

Microstructure and tribological behavior of SPS processed Fe/Ti-15wt.%Cu-based metal matrix composites with incorporated waste Ti-chips

M. Podobová, V. Puchý*, L. Falat, R. Džunda, M. Besterci, P. Hvizdoš

Institute of Materials Research, Slovak Academy of Sciences, Watsonova 47, 04001 Košice, Slovak Republic

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Abstract

In present work, the Fe/Ti-15wt.%Cu-based metal matrix composites with three different Fe/Ti weight percent ratios (i.e., 40/25, 25/40, 5/60) and constant additions of non-metallic additives (i.e., 5 wt.% graphite, 5 wt.% SiC, and 10 wt.% ZrO₂) were investigated. An innovative and ecologically friendly approach for laboratory preparation of the experimental composite materials was based on the secondary utilization (recycling) of the waste Ti-chips (turnings) from conventional machining operations. The material mixtures for the fabrication of the studied composites were prepared by common powder metallurgy pre-operations followed by final material processing using spark plasma sintering (SPS). The microstructure of the SPS-fabricated composite materials consisted of sintered grain matrix with various amounts and distribution of Ti-chips. The friction and wear behavior of the composites was analyzed from performed tribological measurements employing “ball-on-disc” test method. The results showed that the coefficient of friction was mostly decreasing with increasing the sliding speed and the amount of Ti-chips in the composites. The lowest abrasion wear rate exhibited the composite with 40 wt.% of Ti-chips thanks to its optimal microstructure with appropriate hardness and beneficial wear mechanisms characteristics.

Key words: metal matrix composite (MMC), Ti waste reuse, microstructure, friction and wear behavior

1. Introduction

Friction composites represent the key components in automotive, railway, and aerospace brake systems [1, 2]. Reliable operation of individual components made of friction composite materials depends on a series of required properties, such as stable friction coefficient, sufficient wear and fade resistance, small wear to the counterparts, low noise and vibration, etc. [3–7]. Moreover, key issues addressing criteria for assuring low cost and environmentally friendly production and operation of considered components are still of great interest in continuing research and development. Friction composites are designed as multi-ingredient composite materials for achieving required parameters. More kinds of ingredient materials are generally used to prepare friction composites. These additive materials can be generally categorized into four classes

of matrix reinforcements, e.g., fibers, binders, property modifiers, and fillers [8–11]. A great variety of the used ingredients plays an important role in maintaining several material properties, such as thermal resistance, physical, mechanical, tribological, and other specific properties of individually designed friction composite materials [12–14]. An ideal braking pad material should be able to provide not only high friction coefficients but also good wear resistance during braking [15, 16]. The copper-based powder metallurgical materials are known to possess excellent mechanical properties and thermal conductivity [16–20]. However, due to limited raw materials resources and issues related to environmental protection, manufacturers and researchers are permanently looking for new materials and their combinations to produce composite materials with desirable functional properties and minimal effects on the environment and human health. One of

*Corresponding author; e-mail address: vpuchy@saske.sk

the possible approaches for materials design is focused on the fabrication of new composites using as additive ingredients also some waste material from other manufacturing processes, e.g. from machining operations. The use of industrial waste materials as additives in the manufacture of various products has been attracting growing interest from researchers in recent years and is becoming common practice [21]. The continued depletion of natural resources throws new light on the potential use of some industrial wastes and natural sub-products as fully-fledged alternative raw materials [22, 23].

In present work, three experimental SPS-fabricated Fe/Ti-15wt.%Cu-based metal matrix composites with different amounts of waste Ti-chips (Fe/Ti weight percent ratios) and other constant amounts of non-metallic additives (5 wt.% graphite, 5 wt.% SiC, and 10 wt.% ZrO₂) were investigated. The Ti-chips were used as additive material because of well-known beneficial properties of titanium including its excellent strength, high elastic modulus, very good wear, chemical and thermal resistance. All these material properties are very promising for many applications, e.g., in structural materials, friction materials, and wear-resistant parts working in severe operating conditions. Thus, the main aim of this work is to examine the use of Ti-chips as an alternative additive material in experimental metal matrix composites for demanding friction applications. The produced composite materials were characterized in terms of their microstructure, friction, and wear behavior. The obtained results from tribological tests are discussed concerning material microstructure and wear mechanisms characteristics.

2. Experimental materials and methods

The exact chemical composition of commercial friction materials is not commonly available in the open literature. Therefore, our material philosophy has been conceived with respect to the desired friction and wear characteristics, expected to be obtained through an appropriate combination of selected material ingredients for the preparation of environmentally friendly friction composite materials. Specifically, three different Fe/Ti-15wt.%Cu-based metal matrix composite materials were produced by spark plasma sintering (SPS). Their chemical composition in wt.% is reported in Table 1.

The experimental Fe/Ti-15wt.%Cu-based materials (Table 1) were designed with respect to their anticipated material properties, e.g., high thermal conductivity and reduced fading behavior. These properties give rise to the assumption of reliable performance of the studied materials for application in truck, aircraft, and train pads. The following raw materials

Table 1. Composition of experimental materials in wt.%

Material/sample	25Ti	40Ti	60Ti
Ti-chips	25	40	60
Fe	40	25	5
Cu	15	15	15
ZrO ₂	10	10	10
SiC	5	5	5
graphite	5	5	5



Fig. 1. “Fibers-like” Ti-chips used as a key scaffold material for the preparation of experimental friction composite materials.

were used for laboratory SPS preparation of our experimental composites: unsorted waste Ti-chips (purity: 98 %, supplier: pkchemie – kovyachemie.cz, 2 % of impurities may include: Fe, Al, V, Ni, and oil from machining), commercial powders of Fe (purity: 99.9 %, grain size: 45 μm, grade: ASC 100.29, supplier: Höganäs AB Sweden), Cu (purity: 99 %, grain size: 75 μm, supplier: Sigma Aldrich), ZrO₂ (purity: 99 %, grain size: 5 μm, supplier: Sigma Aldrich), β-SiC (purity: 99.9 %, grain size: 0.5 μm, supplier: HC Starck), graphite (synthetic, grain size: 20 μm, supplier: Sigma Aldrich). The “fibers-like” Ti-chips are expected to act like a key scaffold material enhancing friction and wear performance of the investigated composite materials. The reinforcement by “fibers-like” Ti-chips, produced by turning operations, can be achieved by variously sized and shaped chips (see Fig. 1). The anticipated function of the Ti-chips is to be the limitation of fade experience and enhancement of braking effectiveness. During the braking, there is an occurrence of friction-related heat generation, the used copper addition within the experimental composites is responsible for increasing their thermal conductivity. Thus, the more efficient cooling of the brake components,

as well as considerable improvement of their braking efficiency, can be possibly obtained.

The addition of ceramic powders, such as ZrO_2 and SiC, aims at hardness and friction behavior improvement. The desired coefficient of friction (COF) of brake materials has to be in a range of 0.3–0.7 in dry friction systems. The ceramic abrasives ZrO_2 and SiC improve the cold friction behavior, i.e., increase the friction level and also control the wear on the counter-face due to their fracturing as brittle ceramic materials. On the other hand, the graphite acts like a lubricant lowering the friction level via building-up of friction films. It also plays a role in the improvement of corrosion resistance and like an anti-noise agent on the friction surface.

For each experimental material composition, the powder mixture was dry-mixed in 3D Turbula mixer (WAB AG, Switzerland) for 30 min. Before mixing with other ingredients, the as-received Ti-chips were ultrasonically cleaned in perchloroethylene. The mixed composite powder was loaded into a graphite mold with an inside diameter of 20 mm. The SPS machine (HP D 10SD, FCT Systeme, Germany) was used to perform the sintering of prepared mixtures in a vacuum (5 Pa). A pulsed direct electric current was applied with the pulse duration of 15 ms and pause time of 3 ms throughout all sintering experiments. The temperature was measured using a top pyrometer focused inside a hole in the punch at a distance of 4 mm from the sample. The mold/punch assembly was wrapped in a graphite insulating foil and placed in the SPS. The powder was then heated up in low vacuum condition (10 Pa). The sintering temperature was 1000 °C, heating rate 100 °C min⁻¹, dwelling time 10 min, and applied pressure 50 MPa. The bulk density was measured using the Archimedes method and the overall composite hardness was determined according to Vickers hardness method at 98 N loading for 10 s per measurement. After the SPS processing, the sintered discs were ground and polished to their final thickness of 4 mm and diameter of 20 mm.

The microstructure of the sintered composites was analyzed using a scanning electron microscope (SEM) Tescan Vega-3 LMU, equipped with an energy dispersive X-ray spectrometer (EDXS) Bruker XFlash Detector 410-M for elemental chemical composition analysis. The friction and wear behavior of the composites was studied by using a tribometer HTT by CSM Instruments in the air at room temperature employing a common “ball-on-disc” technique. The tribological partner for each tested material was a polished ball with 6 mm diameter, made of conventional bearing steel, corresponding to the counter-part material in real brake systems. The applied load was 3 N, the sliding speed was in the range from 0.1 to 0.3 m s⁻¹, and the sliding distance was 500 m. The morphology of worn surfaces and wear mechanisms was analyzed

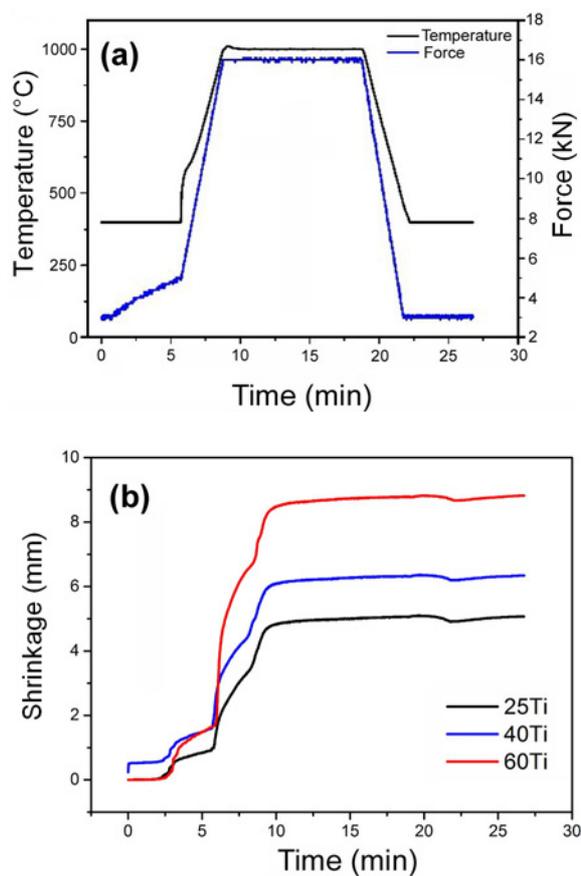


Fig. 2. Defining characteristics of the SPS process used for fabrication of studied composites: (a) temperature-force loading cycles and (b) shrinkage curves of individual composites.

using confocal 3D Optical Profiler (PLu neox, SENSOFAR) and the SEM microscope (Tescan Vega-3 LMU).

3. Results and discussion

3.1. Sintering behavior and microstructure

Figure 2 shows the defining characteristics of the sintering process used for the production of investigated composite materials. The sintering temperature and force loading cycles during the experimental SPS processing are presented in Fig. 2a. The recorded shrinkage curves of creating composites are shown in Fig. 2b.

The SPS process characteristics were confronted with the measured densities and hardness values of produced composite materials (see Table 2). It can be pointed out that the obtained densities are inversely proportional to the magnitude of observed shrinkage behavior. This observation is likely related to the increasing heterogeneity of produced composites with

Table 2. Densities and hardness of experimental composite materials

Material/sample	25Ti	40Ti	60Ti
Apparent density (g cm^{-3})	5.70	5.14	4.53
Relative density (%)	88.2	79.6	70.1
Hardness HV10	250 ± 49	465 ± 53	451 ± 78

an increasing amount of Ti-chips due to their “fibers-like” morphology and thus their imperfect mixing with other ingredients during the pre-preparation of the experimental materials within the used mixing equipment. Due to the same reason, the hardness values of prepared composites exhibit certain scattering behavior.

Figures 3 and 4 show the SEM micrographs and corresponding EDXS elemental maps, respectively, of the investigated Fe/Ti-15wt.%Cu-based metal matrix composites.

Figure 3 indicates the increasing porosity of studied composites with an increasing amount of Ti-chips. This observation correlates well with the data already presented in Table 2. The obtained elemental maps indicate the overall distribution of individual microstructural constituents (Fig. 4). It is visible that the produced experimental composites contain various amounts of Ti-chips as a major scaffold material. Because of high melting points of individual composite constituents (Table 1) and the used rapid sintering conditions (Fig. 2a), the eventual solid-state phase transitions among individual constituents are assumed to be limited to only very small interaction volumes. Nevertheless, the detailed phase analysis of individual microstructural interfaces is out of the scope of the present investigation and is the subject of our subsequent studies.

3.2. Tribological performance and wear mechanisms

Tribological characteristics (i.e., the average COF and wear rate values) of the investigated composite materials are summarized in Table 3.

The results (Table 3) clearly show that the “40Ti” composite material exhibits significantly lower wear rates compared to those of “25Ti” and “60Ti” materials. Moreover, the wear rates of all individual composites do not exhibit any significant variations depending on the used sliding speeds (Table 3). The friction characteristics represented by various dependences of the coefficient of friction (COF) obtained from tribological tests are shown in Fig. 5.

Figure 5a shows the dependences of average COF values on the sliding speed for all studied composite materials. It is observed that the highest average COF values were mostly obtained at the lowest used sliding speed of 100 mm s^{-1} . Moreover, at this slid-

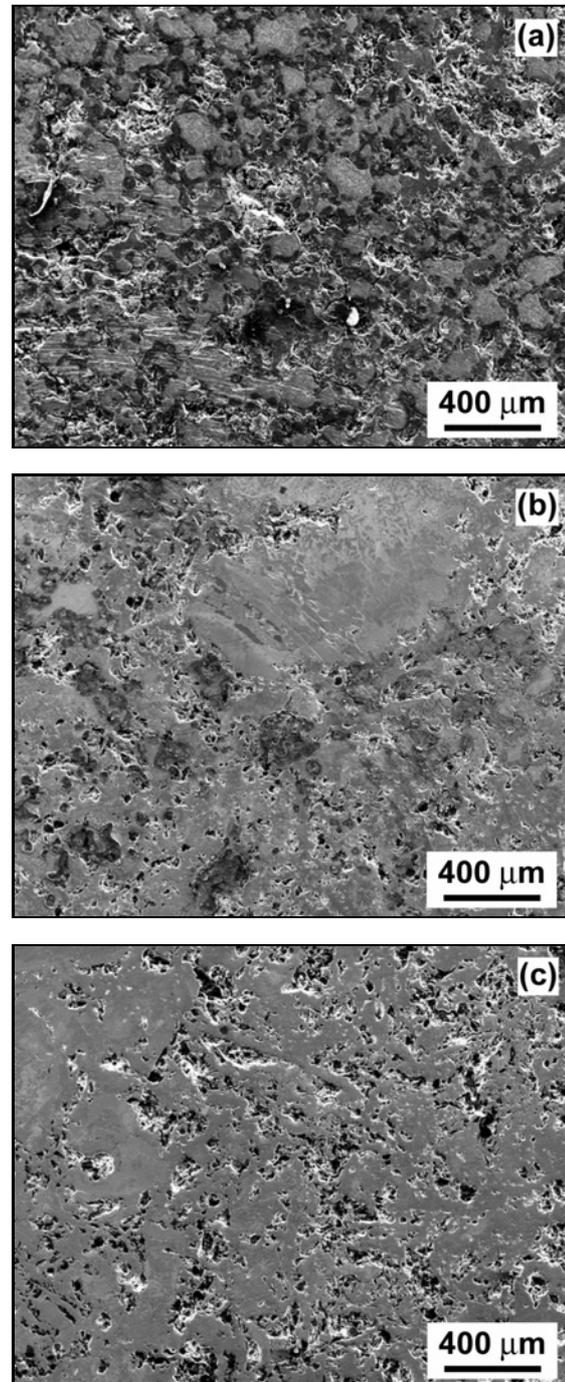


Fig. 3. SEM micrographs of studied experimental Fe/Ti-15wt.%Cu-based metal matrix composites with variable amounts of Ti-chips: (a) 25 wt.% Ti, (b) 40 wt.% Ti, and (c) 60 wt.% Ti.

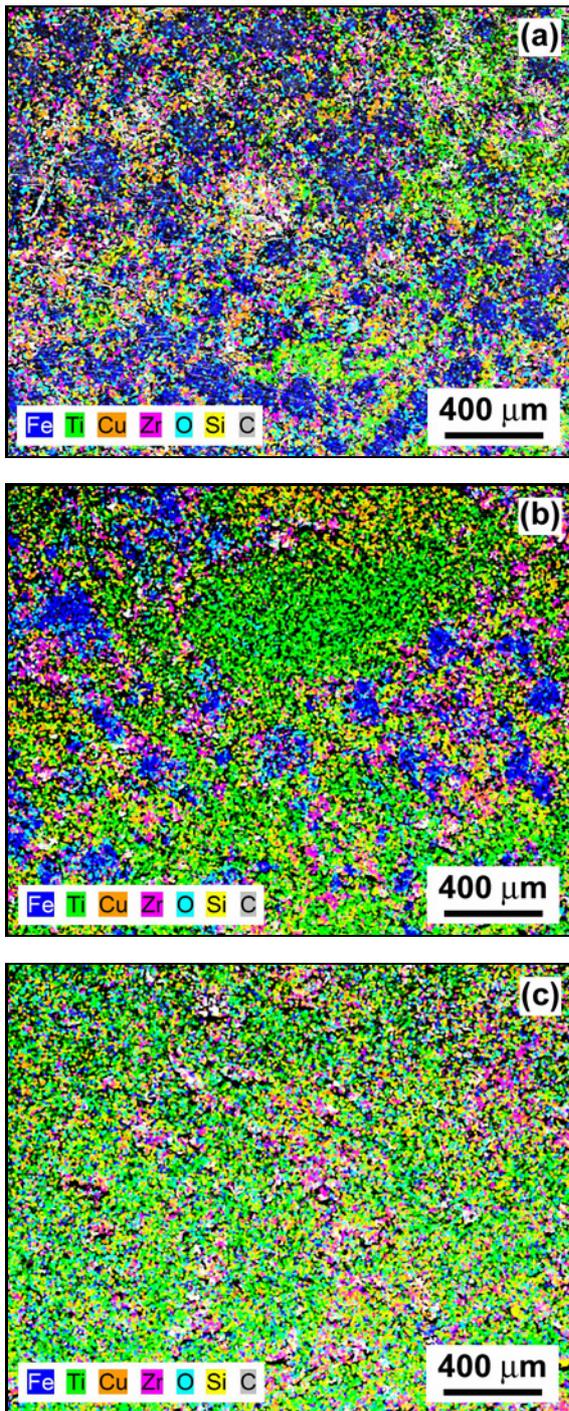


Fig. 4. EDXS elemental maps of studied experimental Fe/Ti-15wt.%Cu-based friction composites with variable amounts of Ti-chips: (a) 25 wt.% Ti, (b) 40 wt.% Ti, (c) 60 wt.% Ti.

ing speed, the COF did not show any significant variations depending on the amount of Ti-chips in the composites. On the other hand, at both higher sliding speeds (i.e., 200 and 300 mm s⁻¹), the COF values were gradually decreasing with an increasing amount

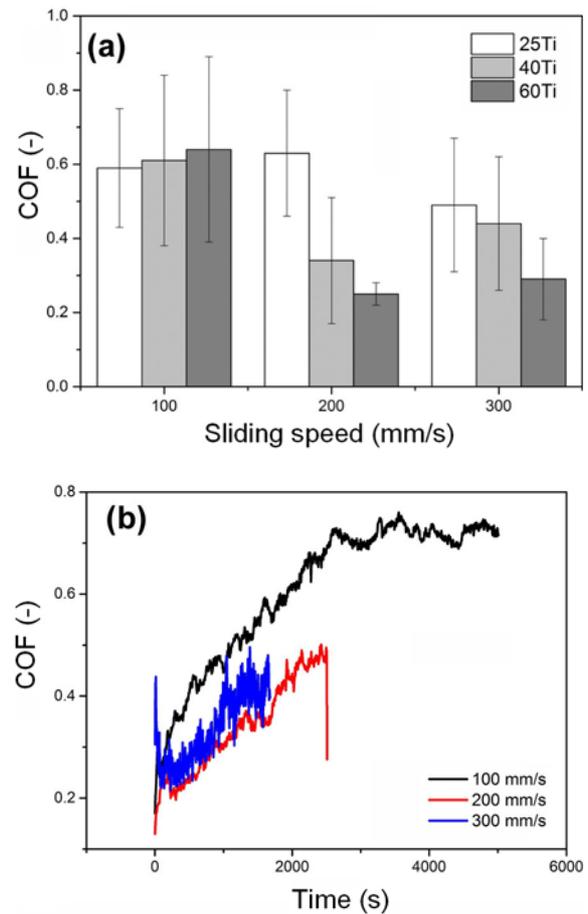


Fig. 5. Friction behavior of the investigated composites: (a) dependence of average COF values on the sliding speed and the amount of Ti-chips; (b) time-dependent behavior of COF for “40Ti” composite tested at various sliding speeds.

of Ti-chips in the composites. The “40Ti” composite, exhibiting the lowest wear rates of all tested materials (Table 3), shows at the same time highly desirable time-dependent COF behavior at the sliding speed of 100 mm s⁻¹, i.e., achieving a stabilized COF plateau region during the tribological testing (Fig. 5b). It should also be noted that for the “25Ti” composite, the COF values remain almost unchanged within the whole range of sliding speeds (Fig. 5a). This observation can likely be put into the context of variant thermal conductivity of the studied composites. It can be reasonably assumed that with an increasing amount of the composite ingredients with lower thermal conductivity (in the present case Ti-chips), the overall thermal conductivity of the composite is decreasing.

Consequently, the lowering of thermal conductivity of the composites with an increasing amount of Ti-chips might cause the observed reduction of COF values at higher sliding speeds (Fig. 5a). On the contrary, in the case of “25Ti” composite (i.e., the composite with a lower amount of

Table 3. Tribological characteristics of studied composites

Experimental materials	Sliding speed (mm s ⁻¹)	Normal load (N)	Distance (m)	Coefficient of friction (-)	Wear rate × 10 ⁻⁵ (mm ³ m ⁻¹ N ⁻¹)
25Ti	100	3	500	0.59 ± 0.16	12.92 ± 2.4
	200	3	500	0.63 ± 0.17	11.94 ± 2.2
	300	3	500	0.49 ± 0.18	12.71 ± 2.5
40Ti	100	3	500	0.61 ± 0.23	6.32 ± 1.6
	200	3	500	0.34 ± 0.17	5.21 ± 1.5
	300	3	500	0.44 ± 0.18	5.95 ± 1.8
60Ti	100	3	500	0.64 ± 0.25	25.53 ± 4.9
	200	3	500	0.25 ± 0.03	23.45 ± 3.1
	300	3	500	0.29 ± 0.11	24.86 ± 4.4

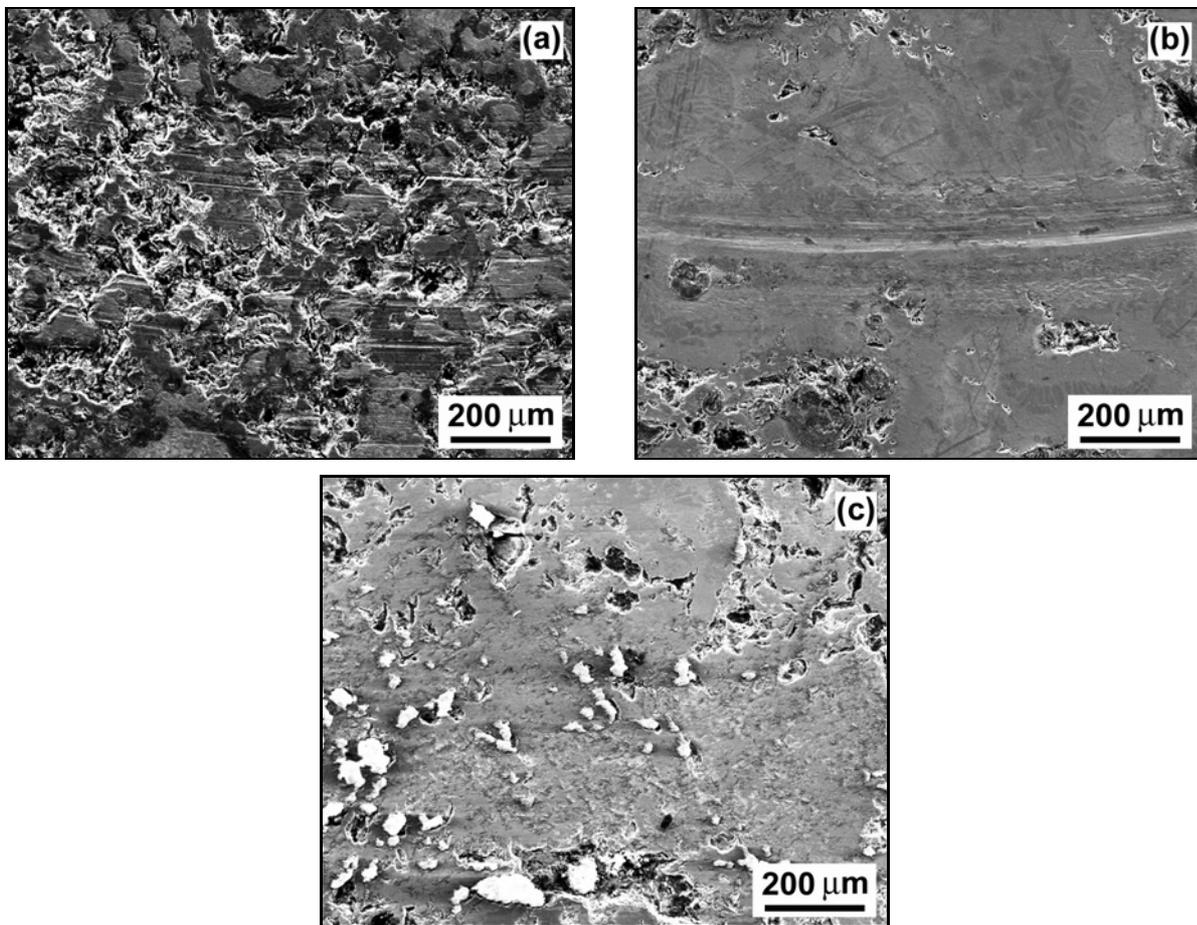


Fig. 6. SEM images from the areas including the wear tracks created after the tribological tests at 300 mm s⁻¹ sliding speed of the investigated Fe/Ti-15wt.%Cu-based friction composites with variable amounts of Ti-chips: (a) 25 wt.% Ti, (b) 40 wt.% Ti, and (c) 60 wt.% Ti.

Ti-chips and thus the higher thermal conductivity), the friction-induced heat can be easily transferred from the outer material surface into the bulk body of the composite. Therefore, the overheating of the surface of the tribological specimen is not expected to occur and the COF is much more

stable, i.e., it shows only small variations depending on the sliding speed (Fig. 5a). The results in Table 3 show that among the studied Fe/Ti-15wt.%Cu-based metal matrix composites, the optimal tribological performance (i.e., the lowest wear rates and satisfactory COF behavior) was observed for the “40Ti”

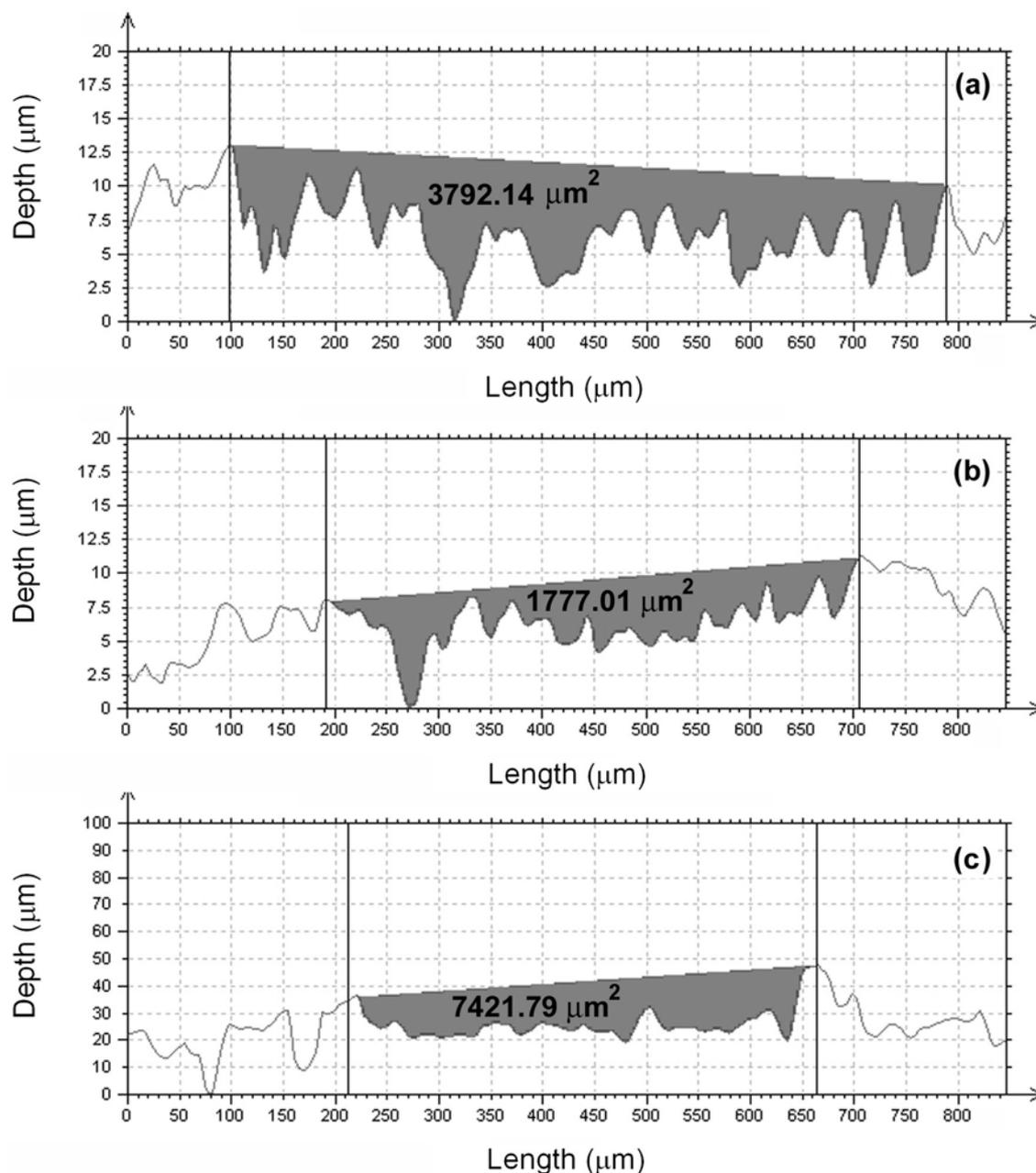


Fig. 7. Representative tribo-track profiles created after the tribological tests at 300 mm s^{-1} sliding speed of investigated Fe/Ti-15wt.%Cu-based friction composites with variable amounts of Ti-chips: (a) 25 wt.% Ti, (b) 40 wt.% Ti, and (c) 60 wt.% Ti.

composite. It can be concluded that the microstructure and chemical composition of the experimental Fe/Ti-15wt.%Cu-based friction composites containing Ti-chips have to be properly balanced concerning both mechanical/tribological and thermal properties for considered brake systems applications.

The representative SEM images of individual wear tracks created after performed tribological tests of the investigated friction composites at a sliding speed of 300 mm s^{-1} are shown in Fig. 6.

From Fig. 6, it is obvious that all investigated composites show the dominant wear mechanism to be

the abrasion. The most severe abrasive wear is generally related to a so-called “micro-cutting” wear micromechanism since it is well-known to be responsible for the most significant removal of the abraded material from the worn material surface. From the individual images of Fig. 6 it is clear that the “40Ti” composite shows the most beneficial abrasive wear characteristics, i.e., the lowest occurrence of the “micro-cutting” micromechanism on the surface of the tribo-track (Fig. 6b). In contrast, the other studied composites (Figs. 6a and 6c) show the more abraded wear tracks that indicate the greater wear rates, following

Table 3. The morphology of the individual wear tracks was studied by confocal profilometry. The representative 2D visualization of the wear track profiles is documented in Fig. 7.

The size of tribo-track profile areas in Fig. 7 can be directly correlated with corresponding wear rates (Table 3) of individual Fe/Ti-15wt.%Cu-based composite materials. The individual EDXS elemental maps from the areas depicted in Fig. 6 involving the wear tracks obtained at a sliding speed of 300 mm s^{-1} are shown in Fig. 8.

The recorded EDXS elemental maps (Fig. 8) show the increased occurrence of oxygen within the wear tracks which indicates some additional oxidation during the tribological testing of the studied composites. Thus it has been shown that the tribological behavior of the studied Fe/Ti-15wt.%Cu-based metal matrix composites is controlled by both abrasive wear and surface oxidation processes.

4. Conclusions

The experimental feasibility of using waste Ti-chips for the preparation of alternative Fe/Ti-15wt.%Cu-based friction composites via SPS technique has been investigated. The produced metal matrix composites were subjected to microstructural and tribological investigations. The obtained results can be summarized in the following conclusions:

- With an increasing amount of Ti-chips, the overall microstructural heterogeneity of the investigated composites increases and their apparent density decreases. This behavior can likely be related to imperfect mixing of the used “fibers-like” Ti-chips with other composite ingredients during the pre-preparation of the experimental materials along with observed shrinkage phenomena during the SPS process. Therefore, our future research needs to include a focused investigation aiming at the homogeneity improvement of the experimental composites under consideration.

- Friction behavior of the investigated composites was characterized by the coefficient of friction (COF). The average COF values were mostly decreasing with increasing the sliding speed and the amount of Ti-chips in the composites. This behavior can likely be put into the context of decreasing thermal conductivity of studied composites with an increasing amount of Ti-chips.

- In conditions of the present study, the optimal tribological performance has been observed for the “40Ti” (i.e., 25Fe-40Ti-15Cu-10ZrO₂-5SiC-5 graphite, in wt.%) composite which showed satisfactorily high average COF values and the lowest wear rates among all currently studied metal matrix composites. However, further studies on mechanical properties (e.g.,

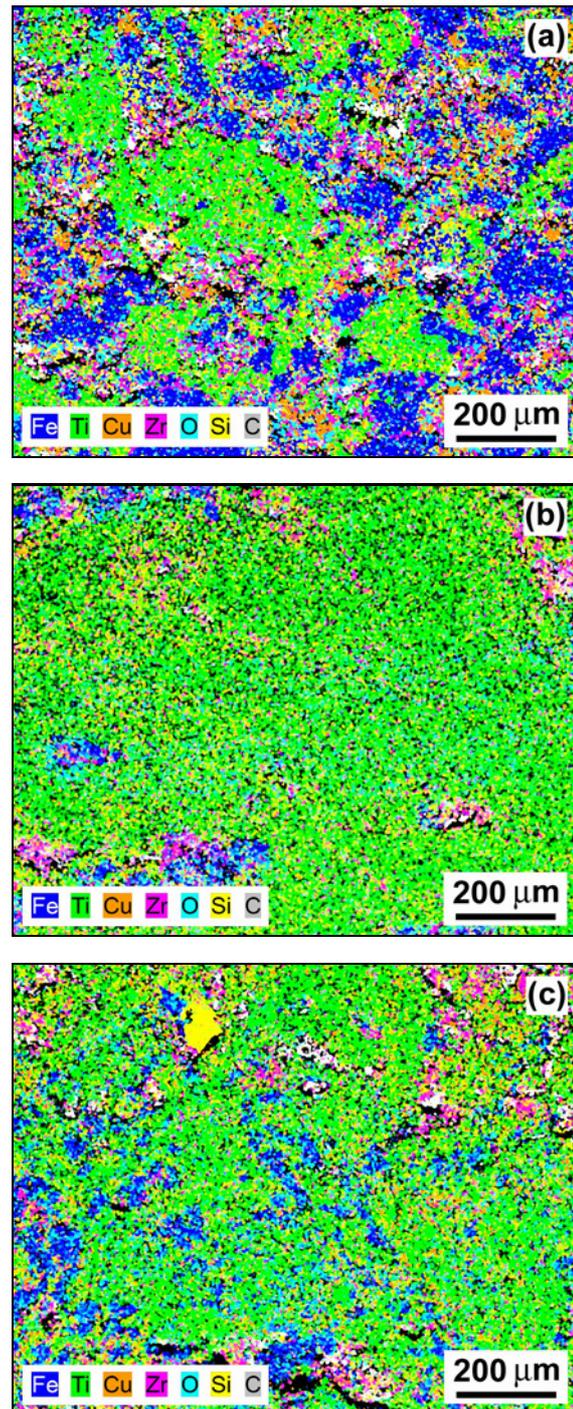


Fig. 8. EDXS elemental maps from the areas including the wear tracks created after the tribological tests at 300 mm s^{-1} sliding speed of the investigated Fe/Ti-15wt.%Cu-based friction composites with variable amounts of Ti-chips: (a) 25 wt.% Ti, (b) 40 wt.% Ti, and (c) 60 wt.% Ti.

from compression and fracture toughness tests) and tribological behavior of considered friction composites at more severe testing conditions (i.e., higher testing loads at shorter sliding distances) are needed to judge

on their suitability for demanding brake systems applications.

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