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S. SOBULA\*<sup>#</sup>, E. OLEJNIK\*, P. KURTYKA\*\*, Ł. SZYMAŃSKI\*, T. TOKARSKI\*\*\***MICROSTRUCTURE, HARDNESS AND WEAR RESISTANCE OF LOCAL TiC-ZrO<sub>2</sub>/FeCr REINFORCEMENT FABRICATED IN SITU IN STEEL CASTINGS**

Wear resistance of TiC-ZrO<sub>2</sub>-Fe locally reinforced steel casting has been investigated. Composites were obtained by introducing to mould cavity compact with TiC reaction substrates, ZrO<sub>2</sub> particles and addition of moderator. Casting with local reinforcement – composite zone was obtained. Structure and microstructure of composite zone were investigated with use of X-ray diffraction (XRD) and scanning electron microscope (SEM) methods. The Miller slurry machine and Ball-on-Disc tester were used to determine wear resistance of the fabricated locally reinforced TiC-ZrO<sub>2</sub>-Fe composites. The worn surface of specimens after test, was studied by SEM. Due to the presence of TiC and ZrO<sub>2</sub> particles in composite zone steel castings exhibited higher abrasion resistance than chromium white cast iron with Ni addition Ni-Hard 1 type and comparable with composites with high TiC content.

*Keywords:* Wear resistance, composite casting, TiC reinforced cast steel, metal matrix composites

**1. Introduction**

Metal matrix composites belong to the group of materials which have undergone rapid development in the past thirty years period. These materials can offer very attractive properties, including high hardness combined with excellent abrasion resistance, the coefficient of thermal expansion lower than that of metals, and additionally the superior strength and stiffness [1-8]. The need to design cast construction materials resistant to abrasive wear is dictated by the growing demands for improvement of both productivity and profitability. This is particularly true in the case of industries such as mining, mineral processing, cement production and recycling of secondary raw materials. In order to meet the specified requirements, both new casting alloys and composite materials are being developed.

While increasing the wear resistance of metal alloys has reached the limit of its capabilities, metal matrix composites create special new opportunities successfully combining the potentials of the two groups of engineering materials, i.e. alloys and ceramics.

Currently, the attention is focused on functional materials, which also include castings with locally-reinforced composite zones. In this group are distinguished the local reinforcements produced by the ex situ and in situ technology. The ex situ process consists in placing in the mould cavity a previously prepared preform made from one selected phase or from a mixture of

ceramic phases, and then pouring a liquid casting alloy over this preform. In the in situ methods, the mould cavity is the place where properly prepared reactants induce the formation of one selected phase or mixture of the ceramic phases. Each of the above-mentioned processes has its advantages and disadvantages. The primary disadvantage of the ex situ process is limited infiltration due to low wettability of the preform made of fine reinforcing particles [3]. Therefore, in common use are the preforms made from relatively large particles, but this contributes to their easy spalling during abrasive wear. Additionally, between the large reinforcing particles there are areas of a similar size filled with the interpenetrating matrix, which is highly disadvantageous on account of the reduced wear resistance.

In the case of the known and currently used processes for the in situ fabrication of composites, the problem is excessive reactive infiltration accompanying usually the SHS reaction of synthesis of a specific ceramic phase. Absence of oxide contaminations at the interface of the produced particles and the possibility of controlling their size during the reaction of synthesis are the distinctive features of the in situ composites. The main limitation in this case is the price of the elements necessary to produce the reinforcing phase. Therefore, in spite of inferior quality of the ex situ composites, their fabrication is cheaper, which is reflected in the product price. Considering these pros and cons it seems necessary to search for optimal solutions that would combine the advantages of both methods.

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As part of this study, local hybrid composite reinforcements were designed and fabricated in a casting made of alloy steel, using the available potential of both in situ and ex situ methods. For this purpose, a mixture of powders containing the ready-made  $ZrO_2$  ceramic phase and TiC reactants was used. The ready-made ceramic phase was introduced in order to suppress the reactive infiltration process, resulting in fragmentation of the composite zone and reduced price of the final product. To improve the abrasive wear resistance, the dispersed TiC particles were fabricated by the in-situ method. In this way, a local composite reinforcement of the TiC/ $ZrO_2$ /Cr iron type was produced in casting made of alloy steel. The obtained material was characterized in terms of its macro- and microstructure, determining next the hardness and abrasive wear resistance by a ball-on-disc method and Miller slurry test. Obtained results were compared to popular materials used in abrasive wear conditions.

## 2. Experimental procedure

The test Y-shaped castings are shown in Figure 1. Compacts of the composition shown in Table 1 were placed in the mould cavity. For the synthesis of composite zones, the commercial powders of metals and non-metals listed in Table 1 were used.

The content of powders for the moderator synthesis was selected in such a way as to finally obtain the high-chromium cast iron of the composition given in Table 2.

Aluminium powder was added for the deoxidation of composite zones, introducing next the zirconia powder of 0.1-0.5 mm particle size fraction. The powders were mixed for 6 hours in a sealed, cylindrical vessel placed in the mixer with a horizontal axis of rotation. After mixing of the powders, compacts of  $5 \times 20 \times 50$  mm dimensions were made under a pressure of 500 MPa.

The green bodies were fixed in a mould and then the whole was poured with carbon cast steel. The temperature of pouring was  $1580^\circ\text{C}$ . From the knocked out test castings, specimens for microstructure examinations and tribological studies were machined.

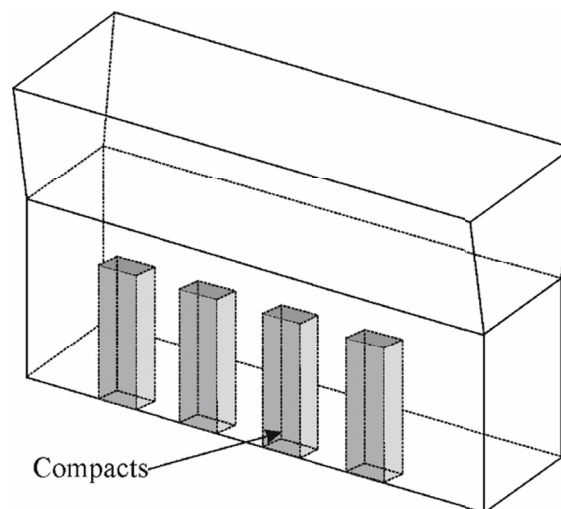


Fig. 1. Sketch of experimental casting with compacts

TABLE 1

Composition of powders used for composite zone synthesis

Substrates for synthesis reaction of:	Weight fraction, %	Composition of powders
TiC	20	Ti (99.8%, 45 $\mu\text{m}$ ) C (99.8%, 3 $\mu\text{m}$ ),
Moderator	59	Fe (99.9% 45 $\mu\text{m}$ ) Cr (99.8% 45 $\mu\text{m}$ ) Si (99.8% 45 $\mu\text{m}$ ) Mn (99.8% 45 $\mu\text{m}$ ) Ni (99.8% 45 $\mu\text{m}$ ) Mo (99.8% 45 $\mu\text{m}$ ) B (99.9% 45 $\mu\text{m}$ )
$ZrO_2$	20	$ZrO_2$ (0.1-0.5 mm)
Al	1	Al (99.9% 44 $\mu\text{m}$ )

The phase composition of the obtained alloy and of the composite zone was analyzed by Kristalloflex 4H X-ray type diffractometer made by Siemens using  $\text{CuK}\alpha$  X-rays of the wavelength of 0.15418 nm. The research was carried out at a voltage of 30 kV and a current of 20 mA. Imaging of micro-

TABLE 2

Chemical composition, wt%

	C	Si	Mn	P	S	Cr	Ni	Mo	B	Fe
Moderator-high Cr iron	3.31	0.87	0.69	—	—	26.6	0.45	1.25	0.003	Balance
Base cast steel	0.17	0.46	0.67	0.026	0.027	0.07	0.06	0.01	—	Balance

TABLE 3

Chemical composition, heat treatment and hardness of reference materials

Reference material	Chemical composition, wt. %								Heat treatment	Hardness HV30
	C	Si	Mn	Cr	Mo	Ni	Other	Fe		
35MnSiMo	0.35	0.7	1.3	—	0.32	—	—	Balance	As cast	—
Nihard 1	3.54	1.16	0.65	2.70	0.04	3.5	0.1Cu	Balance	As-cast	696 $\pm$ 27
Mn18 cast steel *	1.1	0.3	18.0	—	—	—	—	Balance	Solution treatment	535 $\pm$ 29
25MnSi	0.25	1.03	1.14	0.23	0.01	0.04	—	Balance	Normalized	326 $\pm$ 23
60%TiC cast steel composite	0.25	1.03	1.14	—	—	—	—	Balance	Normalized	1523 $\pm$ 290

\* specimens from surface after work

structure produced in the examined materials was done by SEM using a FEI, Versa 3D scanning electron microscope. The wear test was performed in accordance with ASTM G75 Standard Test Method for Determination of Slurry Abrasivity. Summary test period was 16 hours, after each four hours specimens were cleaned in distilled water, dried and then weighted. Load used in test was of 20 N, and slurry was consisted 50 % of SiC. Additionally the Ball-on disc method was used for characterization of wear. The ball was made of an  $\text{Al}_2\text{O}_3$  and had a diameter of 3 mm. The load of ball was 10 N. The radius of the wear track was 3 mm and the rotation speed was 192 rpm. All wear tests

were carried out in ambient temperature. As reference materials in the wear tests, alloys listed in Table 3 were used.

### 3. Results

In Fig. 2 microstructure of cross section of experimental casting is shown. Microstructure of base cast steel is consisted of ferrite and pearlite. The boundary between cast steel matrix and composite zone is sharp and regular. Traces of pearlite were found in the base alloy-composite zone interface.

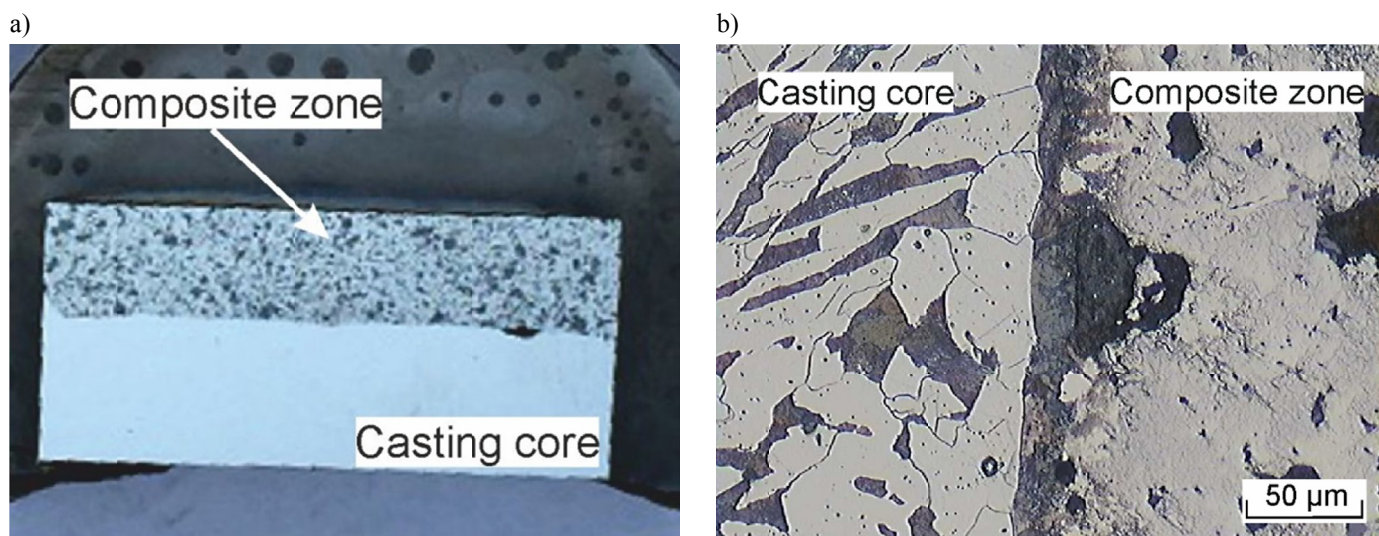


Fig. 2. Cross section of synthesized composite zones (a), microstructure of interface after etching in nital (b)

Figure 3 shows the diffraction patterns with marked peaks belonging to identified phases obtained in the composite zone produced in experimental steel casting. It can be seen that  $\text{Fe}\alpha$  is the main constituents of the matrix, while composite zone is

more complex and consisted of  $\text{Fe}\alpha$ ,  $\text{ZrO}_2$ ,  $\text{TiC}$ ,  $\text{Cr}_{23}\text{C}_6$ ,  $\text{Cr}_7\text{C}_3$  and a small amount of an amorphous phase.

The diffuse region of amorphous phase visible in the X-ray image is associated with the oxide precipitates present in certain regions of the composite zone (Fig. 4d).

The examined composite zone was formed as a consequence of reactive infiltration occurring during the exothermic reaction of  $\text{TiC}$  synthesis. The microstructure of the examined specimens was detected using scanning microscope. Fig. 4 displays the typical microstructure obtained from the composite zones in as-cast condition. Within the composite zone are visible large, bright, faceted phases of  $\text{ZrO}_2$  and dispersed particles of  $\text{TiC}$  (Fig. 4c). As can be seen in (Fig. 4a,b), distribution of zirconia particles in matrix is not uniform. Despite the use of pure metal powders for the synthesis, in certain areas of the composite zone, the presence of non-metallic inclusions has been traced. (Fig. 4d). As proved by SEM studies, those were the complex oxides of iron, chromium, titanium and aluminium. The next set of drawings shows the results of hardness measurements taken in the composite zone. Matrix hardness is 353 HV1, while hardness of the composite zone is 553 HV1. Noteworthy is the large scatter of hardness values, due to a non-uniform microstructure of the composite zone. The presence of oxides, including the zirconium oxide introduced to the composite zone and characterized by hardness lower than the hardness of

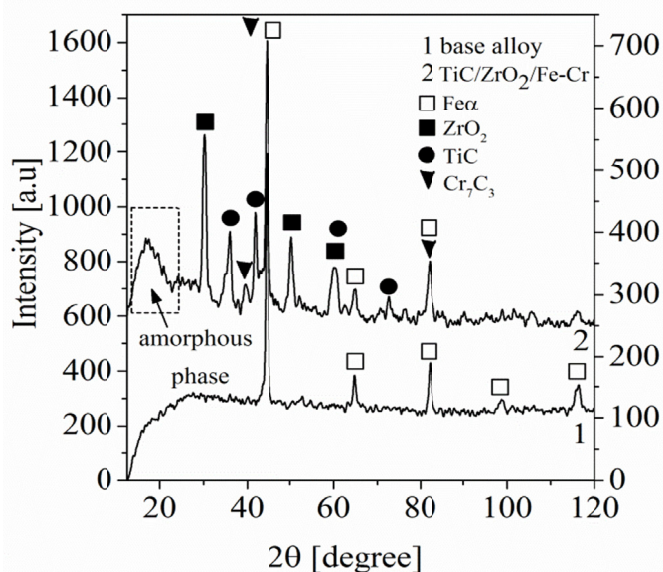


Fig. 3. X-ray diffractogram of hybrid composite zones and cast steel matrix



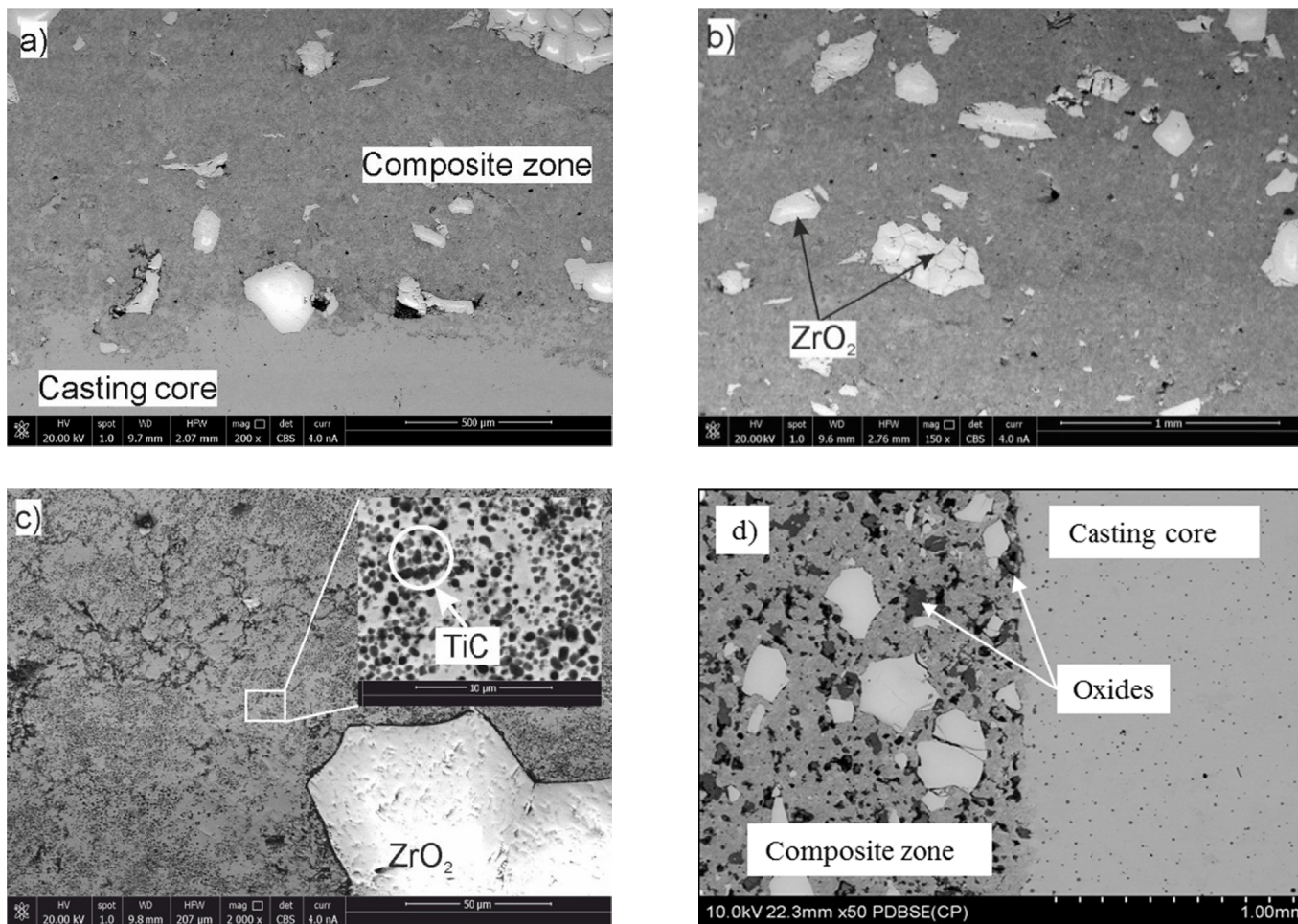


Fig. 4. SEM BSE microstructure of TiC/ZrO<sub>2</sub>/Fe-Cr composite zone fabricated in situ in steel casting

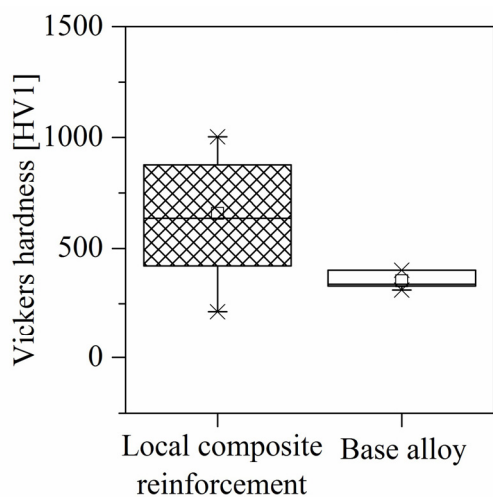


Fig. 6. Hardness of hybrid composite zones

titanium carbide (1200-1500 HV [10] vs. 2000-3200 HV [1]), has a significant impact on hardness reduction in the resulting hybrid composite zone.

Consequently, hardness of the hybrid composite zone is lower than the hardness of the composite zone containing only pure titanium carbide particles synthesized in the carbon steel matrix. Despite this fact, it is the hybrid composite zone that has

the abrasive wear resistance superior to the zone composed of pure titanium carbide (Fig. 7b).

Fig. 7a shows the results of abrasion test performed by the ball-on-disc method. The composite zones exhibit better performance than the Ni-hard 1 cast iron and are definitely more resistant to abrasive wear than the cast Hadfield steel and low-alloy steel.

Fig. 7b shows the results of Miller abrasion test carried out on the hybrid composite zone and selected reference materials [10,12]. From the comparison it follows that the zone made of a hybrid TiC-ZrO<sub>2</sub>-high-Cr iron composite is characterized by the lowest rate of wear. After the initial period of wearing-in, the wear rate was decreasing, and the 16 hour lasting abrasion test gave the total weight loss of 0.10 g. For comparison, the high-chromium white cast iron (26% Cr) showed under the same conditions the total weight loss of 0.28 g [10]. As in the ball-on-disc test, also this time, it was the cast low-alloy steel that showed the lowest wear resistance. Figure 8 presents photographs of sample surface after the 16 hour lasting Miller test. Visible are silicon carbide particles, especially in places where oxides have been present. The composite matrix made of high-chromium cast iron with evenly distributed very fine particles of titanium carbide shows only minor signs of wear. On the other hand, the zirconia particles on the wearing surface suffer visible

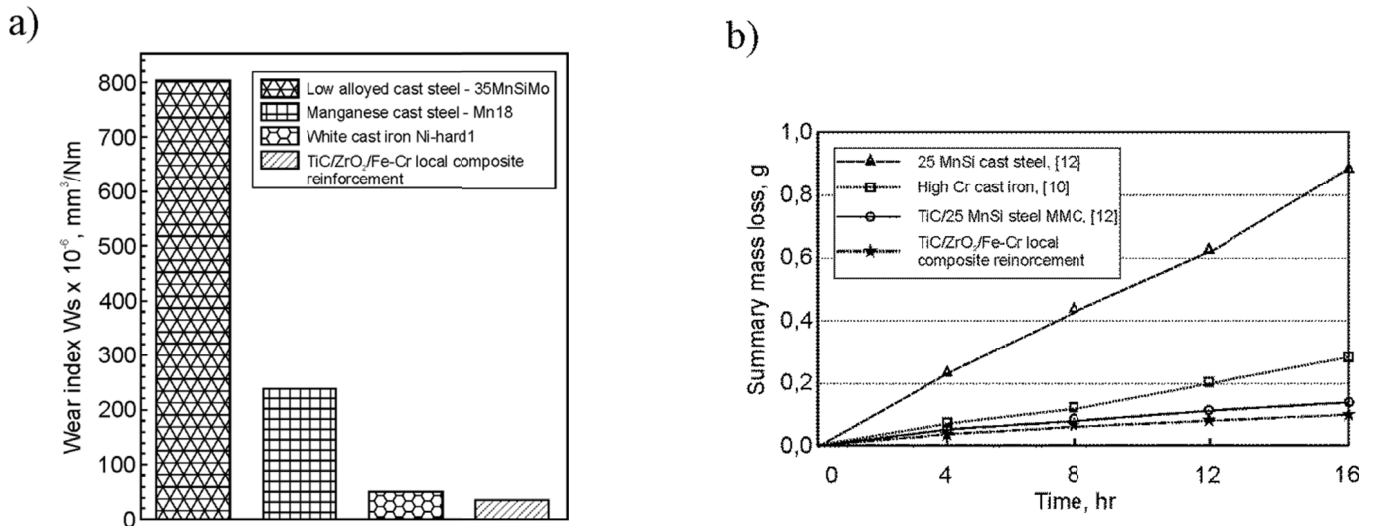


Fig. 7. Wear index in Ball-on-disc test (a) and summary mass loss in Miller slurry machine test (b) of tested and reference materials

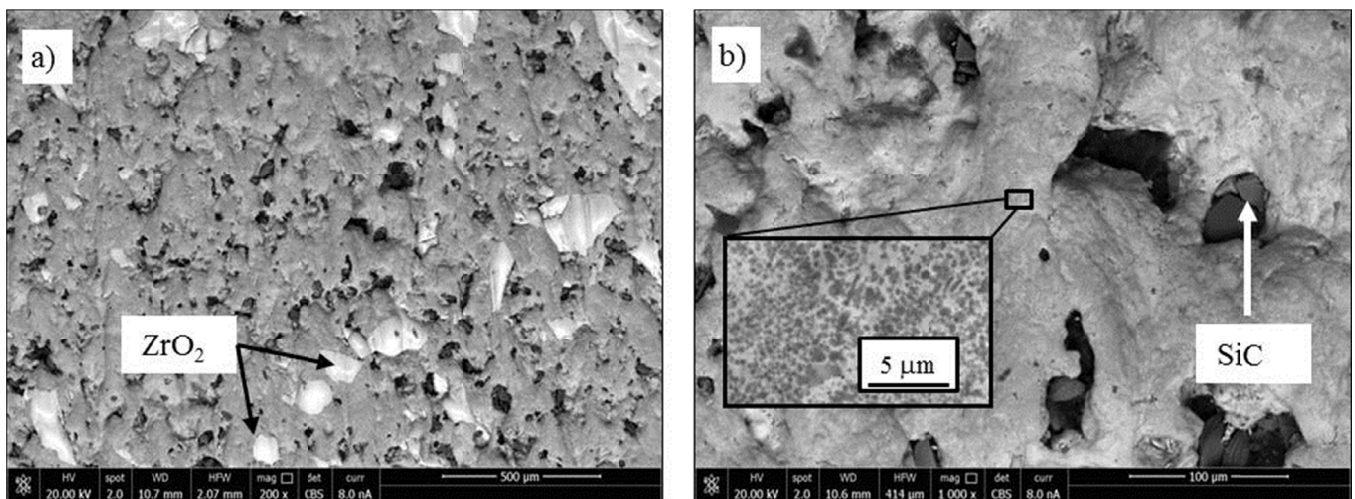


Fig. 8. Surface of investigated hybrid composites after wear test in slurry

signs of wear in the form of oblong scratches consistent with the direction of abrasion (Fig. 8a).

The morphology of titanium carbide particles has a very beneficial effect on the wear rate of samples. In practice, these particles are very small and their size does not exceed 1 μm (Fig. 8b). Consequently, the predominant mechanism of the TiC-ZrO<sub>2</sub> composite wear in Miller test is that of polishing.

#### 4. Conclusions

1. The resulting TiC-ZrO<sub>2</sub>-High-Cr iron composite zones are characterized by the hardness values comprised in a range of  $553 \pm 320$  HV1.
2. Despite this hardness, the total weight loss is only 0.10 g after the 16 hour lasting abrasion test performed on a Miller machine.
3. The wear index obtained in the ball-on-disc test for the examined zones containing ZrO<sub>2</sub> is similar to the wear index of Nihard 1 cast iron and its value amounts to  $32,4 \text{mm}^3/\text{Nm}$

4. The produced zones contain in a large amount of oxide inclusions derived from the presence of zirconium oxide.

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