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## TRIBOLOGICAL PROPERTIES OF WCCo/cBN COMPOSITES PRODUCED BY PULSE PLASMA SINTERING

In view of their advantageous properties (high hardness, good frictional wear resistance, chemical and thermal stability at elevated temperatures), cubic boron nitride (cBN) and tungsten carbide (WC) are commonly used for the fabrication of cutting tools.

The composites were consolidated at a temperature of 1100°C under a load of 100 MPa for 10 min. The density of the thus produced material was close to the theoretical value (about 99.6%), and the hardness HV30 was about 1950. The phases identified in the composite were WC, Co, and cBN. Microstructural examinations revealed that numerous trans-crystalline fractures through the cBN particles occurred in the material.

The present study is concerned with the wear of the WCCo and WCCo/cBN composites. Comparative tribological examinations were performed in a tribological tester using the ball-on-disc arrangement under the conditions of dry friction. The counter-specimens were steel and Al<sub>2</sub>O<sub>3</sub> balls. The tests were conducted under a unit load of 10 N. After the tests, the surface of the samples was examined to describe the wear mechanisms active in various composite materials.

*Keywords:* Pulse Plasma Sintering, Cubic Boron Nitride (cBN), Cemented Carbide, Composite

### 1. Introduction

Cubic boron nitride (cBN) is the material next to diamond in respect of hardness and heat conductivity, but, contrary to diamond, it shows weak reactivity with ferrous materials. In view of these properties, cubic boron nitride is an excellent material for the fabrication of cutting tools such as e.g. those made of heat-treated steels, nickel-based alloys, or cobalt [1,2].

Under elevated temperatures and under normal pressure, cubic boron nitride undergoes transformation into its stable phase hBN. Traditional cBN sinters (polycrystalline cubic boron nitride – PCBN) with a metallic matrix are usually sintered using the expensive High Pressure High Temperature (HPHT) method which requires high pressure of 4-5 GPa to avoid the transformation of hard cBN into its soft hexagonal hBN phase [3,4].

In the present study we sintered the WCCo/cBN composites using the Pulse Plasma Sintering (PPS) method which is an innovatory method that belongs to the group of electric-field activated techniques. The material to be sintered is here heated by repetitive electric impulses of several tens kA generated periodically during the discharge of a capacitor battery. This way of delivering energy creates specific heating and cooling conditions in which, during several hundred of microseconds, the energy of a dozen or so kJ is delivered to the material. During the flow of electric current, the temperature of the material increases to

a high level and after the current vanishes it rapidly decreases to the sintering temperature. Thanks to this way of delivering energy the process is very effective thermally. The powder is heated through the Joule heat and the spark discharges ignited in the pores between the individual powder particles [5-9].

The aim of the present study was to estimate the tribological properties, such as the friction coefficient and the microstructure of the wear trace of the composite materials under the conditions of dry friction. The experiments included the fabrication of the composites, preparation of the test samples, and determination of the friction coefficient in the composite-steel and composite – Al<sub>2</sub>O<sub>3</sub> pairs subjected to dry friction.

### 2. Experimental details

The WCCo/cBN composites were produced from a mixture composed of the powders of tungsten carbide (average grain size of 4 μm), ultra-fine cobalt, and cubic boron nitride (average grain size of 4.64 μm). The powders were mixed in a Turbula-type mixer using carbide balls (the ball-to-powder mass ratio = 1:1) for 5 h. The mixing process was divided into two stages: the initial stage included the preparation of the WC (94 wt. %) + Co (6 wt. %) mixture, and the next stage – the preparation of the WCCo + cBN (20 vol. %) mixture.

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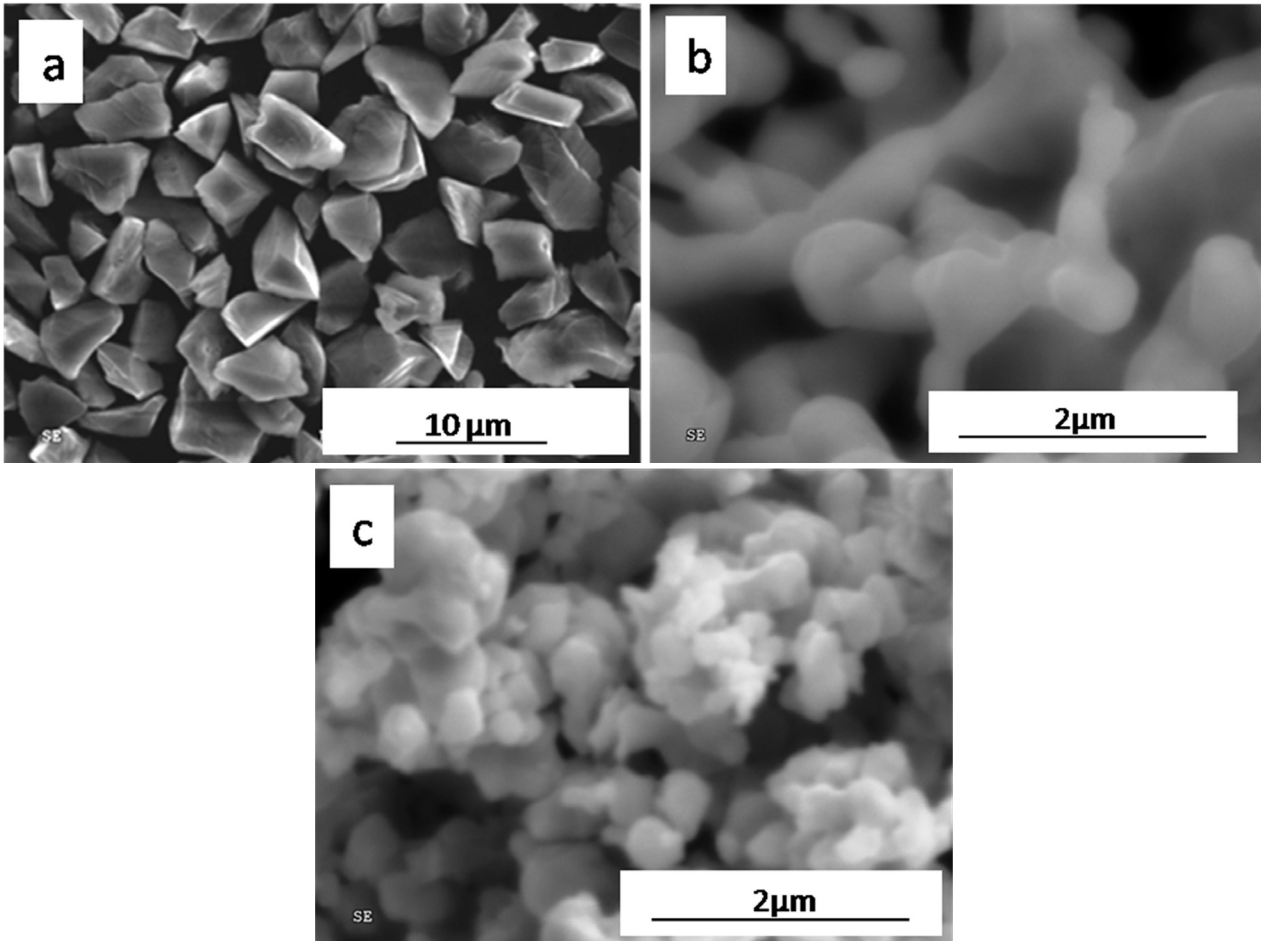


Fig. 1. SEM image of the a) cBN, b) Co, c) WC powders

The powder mixture was then subjected to preliminary consolidation which was done by hand in a graphite die under a pressure of 50 MPa. The samples thus prepared were sintered in a PPS apparatus. All the sintering processes were conducted under a reduced pressure of  $5 \times 10^{-5}$  mbar. The sintering process was also divided into two stages: the degassing stage, aimed at removing the absorbed gases, which was conducted at a temperature of 900°C under pressure of 50 MPa, and the sintering stage conducted at 1100°C under pressure of 100 MPa. The samples were then cooled to room temperature at a rate of 160°C/min in vacuum of  $5 \times 10^{-5}$  mbar under a load of 100 MPa. Fig. 2 shows schematically the variation of the temperature and load during the sintering process.

The density of the sintered samples was measured by the Archimedes methods using a Gibertini E154 balance equipped with appropriate instrumentation, and their hardness was measured by the Vickers method under a load of 294 N (HV 300). The microstructure observations were performed in a HITACHI SU-70 scanning electron microscope. The phase composition was examined with a Philips PW 1140 diffractometer using  $\text{CuK}\alpha$  radiation. The tribological properties of the samples were estimated with the use of a T-21 tribological tester in the ball-on-disc arrangement under the conditions of dry friction using 10 mm diam. 100 Cr5 bearing steel balls and  $\text{Al}_2\text{O}_3$  balls as the counter-specimens. The pressure was 10 N, and the wear

radius was 7 mm. The friction coefficient was determined from the formula:

$$\mu = F_n / F_t$$

where  $F_n$  is the tangent force [N],  $F_t$  is the measured value of the normal force [N].

A schematic representation of the ball-on-disc method is shown in Fig. 3.

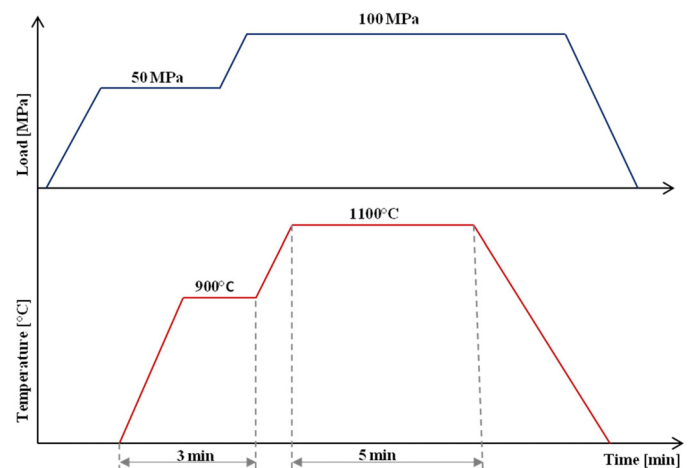


Fig. 2. Schematic representation of the sintering process used for the fabrication of the WCCo/cBN composites

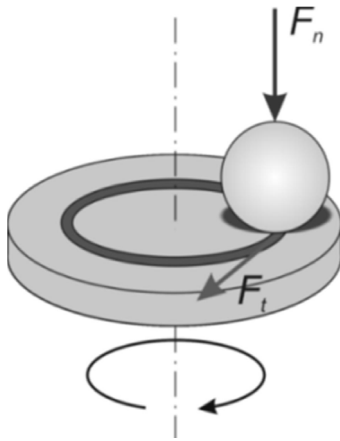


Fig. 3. Schematic representation of the ball-on-disc method

### 3. Results and discussion

Table 1 gives the measured values of the relative density and hardness of the samples used for the tests of frictional wear resistance. All the samples have high density. As to the hardness, the commercial WCCo has the lowest hardness which is due to the highest cobalt content (11 wt.%) in this material, and the composites with distributed cBN particles have the highest hardness HV 30 (1936). The spread of the results is narrow which evidences that the material is homogeneous.

The microstructure (SEM image) of a fracture of the WCCo/cBN composite is shown in Fig. 4. We can see well-shaped WC grains with sharp edges, and cobalt uniformly distributed along their boundaries which forms the so-called paths. In the entire volume, the sintered sample has a compact homogenous structure, which is reflected in the uniform distribution of hardness. There are also numerous trans-crystalline fractures through the cBN particles, which gives evidence of their strong bond with the matrix.

The occurrence of the liquid cobalt phase during pulse plasma sintering is associated with the rapid increase of the temperature up to several thousand Celsius' grades during the flow of the current pulse through the material. This observation

was also confirmed by the present experiments, since the WC grains visible in the microstructure of the cemented carbides PPS-sintered at a temperature of 1050°C (Fig. 4) are evenly surrounded by the binding cobalt phase, which is evidence that the high-current pulse sintering proceeds with the participation of this phase in the liquid state.

In the PPS method, the material to be sintered is heated by periodically repeated electric current pulses with the amplitude of about 60 kA and the pulse duration of about 0.5 ms. The essence of the PPS method is that the heating operation is realized by the application of high current pulses with the intensity of several tens of kA, obtained by discharging a capacitor battery. The use of capacitors as the source of the energy necessary for the consolidation of the powder creates specific heating and cooling conditions since the energy of several kJ is delivered to the processed powder during a time as short as several hundred microseconds. During the current flow, the powder being consolidated is heated to a high temperature and, after the current decays, the powder quickly cools down to the specified sintering temperature.

Figure 5 is a diffractogram obtained for the sintered sample. The spectrum only identifies three phases namely WC, Co, and cBN. The binding cobalt phase has a regular structure characteristic of its high-temperature phase. No soft hBN phase can be identified in the diffractogram.

TABLE 1

Selected properties of the composite materials examined in the present experiments

Sample	Cobalt content, wt.%	Relative density, %TD	Vickers hardness HV30	Ra, $\mu\text{m}$
WCCo commercial	11	100.0	1527±6.7	0.012
WCCo	6	99.8	1817±11.7	0.015
WCCo/cBN	6	99.6	1936±18.2	0.025

The values of the friction coefficient measured in the composite/steel pair are shown in Fig. 6. The highest friction coefficient (0.76) was obtained in commercial WCCo. The friction coefficient of the WCCo composite produced by the PPS

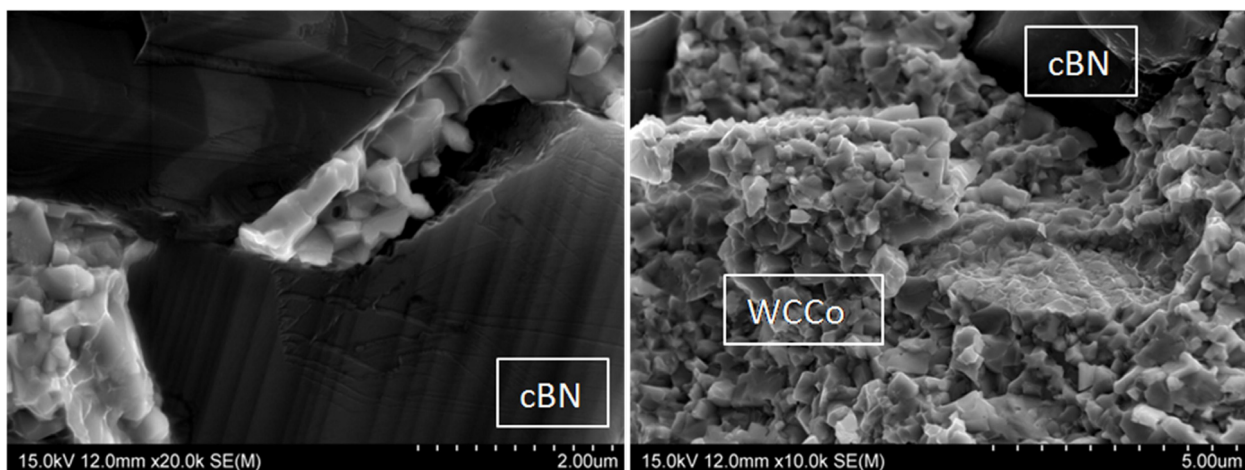


Fig. 4. Microstructure of a fracture of the WCCo/cBN composite (SEM)

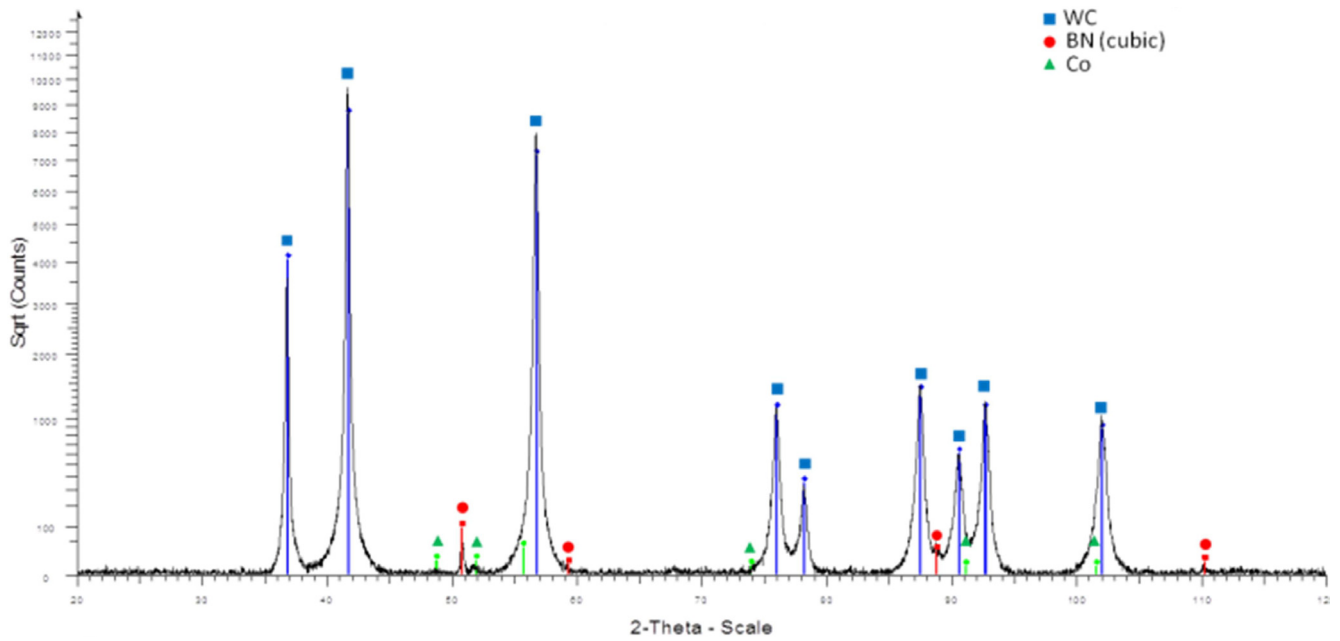


Fig. 5. X-ray diffraction spectrum obtained for the WCCo/cBN composite

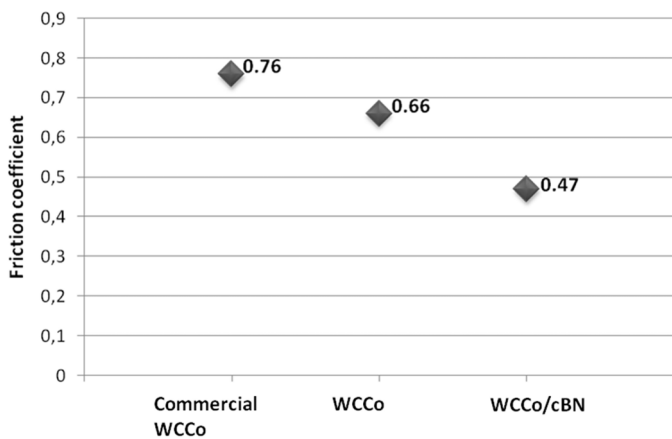


Fig. 6. Average friction coefficient measured in the composite/steel pair

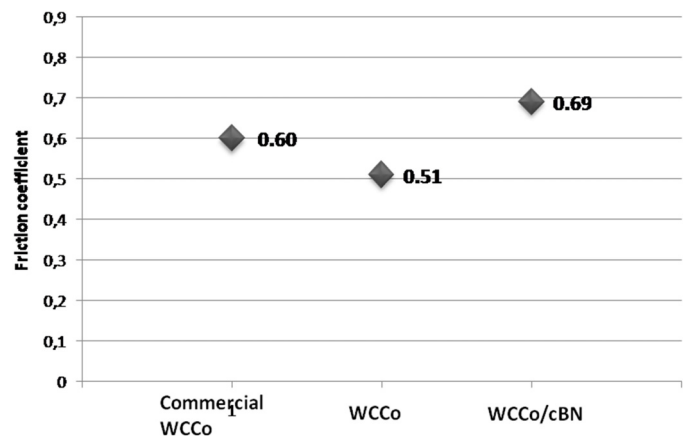


Fig. 7. Average friction coefficient measured in the composite/ $Al_2O_3$  pair

method was lower by 10% than that of the commercial WCCo. This difference should be attributed to the difference in the cobalt content, and, in consequence, the difference in the hardness of these materials. The lowest friction coefficient was obtained in the WCCo composite added with 20 vol. % of cBN particles. It is lower by about 30% than that of the WCCo composite produced the PPS. Comparing the measured values of the friction coefficient with the hardness (Table 1) we can see that it decreases in proportion to the hardness of the sample.

Figure 7 shows the average friction coefficient determined for the composite/ $Al_2O_3$  pair. We can see that the differences in this coefficient between the commercial WCCo, PPS-treated WCCo, and WCCo/cBN composites are identical to those observed in the tests conducted with the steel balls. The friction coefficient of the WCCo carbide produced under laboratory conditions by the PPS method is lower by about 10% than that of the commercial WCCo carbide, whereas the WCCo/cBN composite has the highest friction coefficient (0.69).

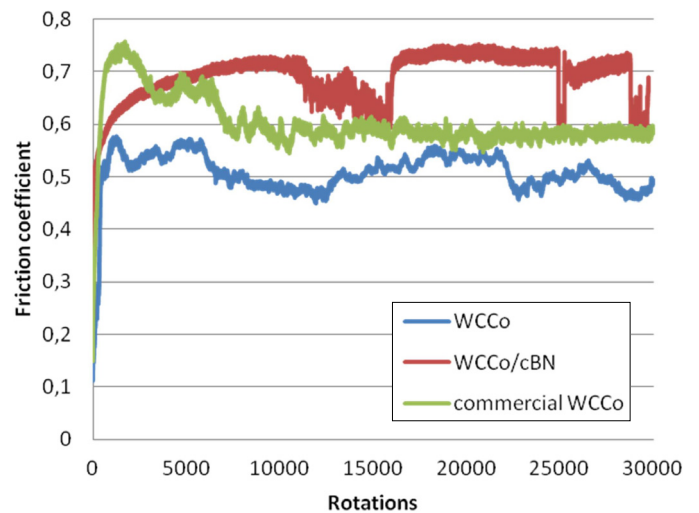


Fig. 8. Variation of the friction coefficient tested in the composite/ $Al_2O_3$  pair as a function of the friction path

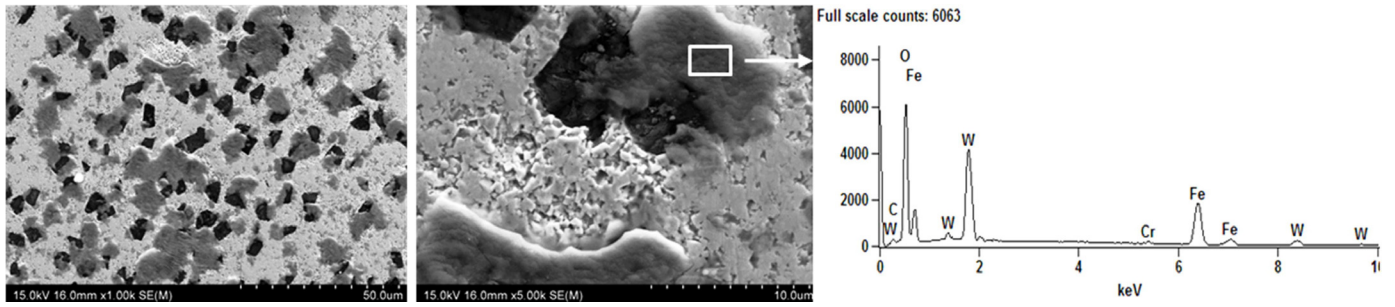


Fig. 9. SEM images of the surface layer of the composite after the friction test conducted against the steel counter-specimen and result of the point analysis of its chemical composition

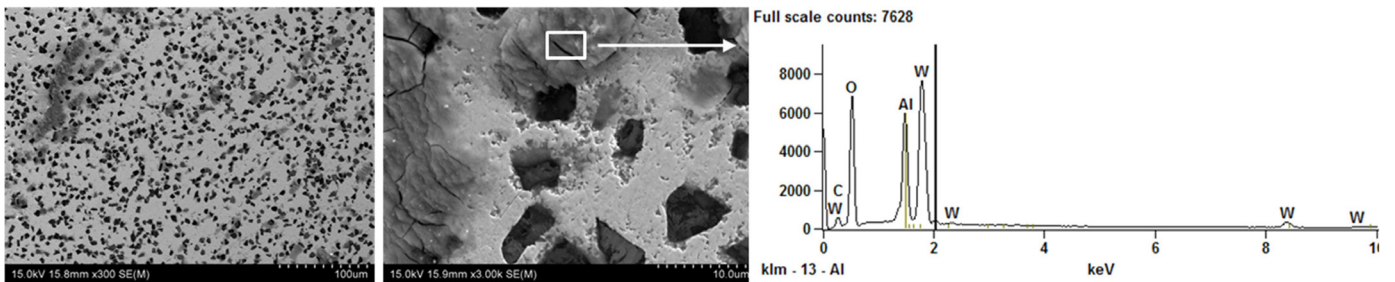


Fig. 10. SEM images of the surface layer of the composite after the friction test conducted against the  $\text{Al}_2\text{O}_3$  counter-specimen and result of the point analysis of its chemical composition

The variation of the friction coefficient in the composite/ $\text{Al}_2\text{O}_3$  pair in dependence on the number of test cycles (up to 30000 revolutions) is shown in Fig. 8. The curves obtained for the composite made of commercial WCCo and those obtained for the composite produced by the PPS method have a similar character, but with the former composite, the spread of the results is the narrowest. In the WCCo/cBN composite, on the other hand, the variation of the friction coefficient with the number of cycles is the least stable. The stepwise changes of the value of this coefficient can probably be explained in terms of chipping of the  $\text{Al}_2\text{O}_3$  counter-specimen associated with the presence of hard cBN particles in the WCCo matrix.

Examinations of the steel and  $\text{Al}_2\text{O}_3$  counter-specimens have shown that the wear mechanisms active in them are different. The steel ball is more plastic and harder than the  $\text{Al}_2\text{O}_3$  ball and also than the composite material (Figs. 9,10). The soft material of the steel ball fills the voids present on the surface of the composite and may function as a lubricant during the subsequent test cycles. The worn material of the  $\text{Al}_2\text{O}_3$  ball, on the other hand, is deposited in the form of build-ups on the sample surface forming a sort of loose abrasive which is easily removed from the wear trace.

The composites produced by the PPS method are substantially harder than the test balls and, thus, the balls undergo intensive wear. The loss of the counter-specimen volumes is shown in Fig. 11. With the WCCo/cBN composite, the wear of the counter-specimens is stronger than that observed with the WCCo composite, and the volumes of the worn material are similar in the steel and  $\text{Al}_2\text{O}_3$  balls. With the WCCo composite, the volume of the worn material of the steel ball is several times smaller than that of the  $\text{Al}_2\text{O}_3$  ball. This is so since, in the absence

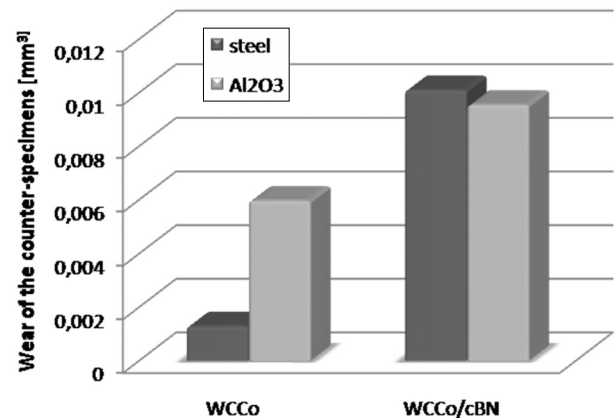


Fig. 11. Wear of the counter-specimens

of the reinforcing cBN particles, the amount of iron gathered in the wear trace is greater and, after a certain number of cycles, it becomes so large that the surface area of the contact between the composite and steel diminishes whereas the steel-to-steel contact surface area increases. In effect, the wear rate of the steel ball decreases. On the other hand, the greater loss of volume observed in the  $\text{Al}_2\text{O}_3$  ball can be associated with the brittleness of its material. It is probable that a sort of 'micro-cutting' takes place which leads to the surface area of the composite-to- $\text{Al}_2\text{O}_3$  contact being increased.

#### 4. Conclusions

The Pulse Plasma Sintering method permits producing a bulk WCCo composite with cBN particles distributed within

it, which has a density close to theoretical and is characterized by high hardness.

The cBN particles (20 vol. %) present in the carbide matrix decreased the friction coefficient of the composite but only when it was in friction with the steel ball. In view, however, of the difference between the wear mechanisms active in the Steel and Al<sub>2</sub>O<sub>3</sub> balls, we cannot unequivocally say whether the addition of cBN particles improve the resistance to frictional wear of the composite. It seems, therefore, that the research work should be supplemented with examinations of the durability of the cutting edges made of the WCCo/cBN composite. Table 1 gives the measured values of the relative density and hardness.

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