



# Alumina-Ti(C,N) ceramics with TiB<sub>2</sub> additives

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Received 07.12.2010; published in revised form 01.01.2011

## ABSTRACT

**Purpose:** The aim of the study presented in this article is to determine the effect of TiB<sub>2</sub> addition on the selected properties of ceramic tool materials. The effect of titanium diboride additives on the density, porosity, Young's modulus and Vickers hardness of Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) matrix ceramics was determined.

**Design/methodology/approach:** Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) ceramics with TiB<sub>2</sub> addition were sintered by SPS method. Materials for SPS sintering were pressed in graphite die with pressure of 35 MPa. The max. pressure was obtained after 10 minutes. Sinter process was operated in nitrogen atmosphere. Density, Vickers hardness and Young's modulus was determined for these materials. The materials were also subjected to tribological analysis. The results were compared with the properties of Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) matrix material.

**Findings:** The use of SPS-method in the production of ceramic materials is possible reduce sintering temperature and sintering time. Depending on the TiB<sub>2</sub> additives used, the relative density values of individual materials were in the range of 94.4% to 97.7%. Young's modulus values for these materials were in the range of 392 GPa to 414 GPa, and Vickers hardness in the range of 1966 to 2225 HV1. The results of tribological analysis showed a friction coefficient value for the matrix material of 0.51. For the other materials, the friction coefficient values were in the range 0.31 to 0.49.

**Practical implications:** Ceramic materials with the addition of titanium diboride may be used in cutting tools. Study of the composition and production technology of such tools allows for the minimisation of the use of liquid cooling lubricants in the machining process and the achievement of higher cutting speeds.

**Originality/value:** Titanium diboride additives were added to the structure, resulting in a reduced coefficient of friction, which was measured at between 61% and 96% of the base material coefficient.

**Keywords:** Alumina; Titanium diboride; SPS-sintering; Mechanical properties

**Reference to this paper should be given in the following way:**

P. Putyra, M. Podsiadło, B. Smuk, Alumina-Ti(C,N) ceramics with TiB<sub>2</sub> additives, Archives of Materials Science and Engineering 47/1 (2011) 27-32.

## MATERIALS

### 1. Introduction

Ceramic Al<sub>2</sub>O<sub>3</sub>-matrix composites with the addition of reinforcement phase Ti(C,N) are characterized by high physical and mechanical properties [1]. These materials are used in the manufacture of cutting tool with the use mainly for difficult

cutting materials. Preliminary studies conducted at the Institute of Advanced Manufacturing Technology, shown that the Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) multi-point cutting tools are characterized by relatively high friction coefficient (0.51). The steel ball with a diameter of 3.2 mm in the method of "ball-on-disc" was used. For the friction coefficient reduction the PVD and CVD coatings are applied on

tool materials, quite accurately described in the literature [2-4]. Reducing the friction coefficient between the workpiece surface and the surface of the cutting tool could also be realized by introducing solid lubricants into the matrix tool [5]. Solid lubricants, also known as lubricating materials, are characterized by a layered crystal structure. It is important that in such a structure, there were weak bonds between the layers, which allow their shear [6,7].

Based on the results of mechanical and tribological properties presented in [8] assumed that the content of the lubricants in the ceramic tool should not exceed 10 vol.%. For the  $\text{Al}_2\text{O}_3\text{-Ti(C,N)}$  ceramic material with lubricant additives produced by free sintering, there is a reduction of friction coefficient (for the method of ball-on-disc with steel ball) to a value from 0.28 to 0.38 [9]. Because of high parameters for the free sintering process (high temperature, duration, high vacuum) part of the solid lubricants have been decomposed or melted. These materials were characterized by a rather low value of relative density, Young's modulus and hardness. In addition, these materials after process at high vacuum are characterized by a thick modified surface layer, which must be removed by grinding.

Compared to the conventional method of sintering substantial decreasing time and process temperature can be achieved using the SPS equipment (Spark Plasma Sintering). Ceramic materials with  $\text{Al}_2\text{O}_3\text{-Ti(C,N)}$ -matrix and lubricants additives sintered by SPS are characterized by slightly higher values of relative density, Young's modulus and hardness compared to the materials after the free sintering. For both cases (after SPS and free sintering), these values differ significantly from the hardness and Young's modulus of matrix material [10].

Addition of  $\text{TiB}_2$  into the  $\text{Al}_2\text{O}_3\text{-Ti(C,N)}$  matrix resulted the friction coefficient reduction and the composite is characterized high values of relative density, Young's modulus and hardness [10]. Titanium diboride with high mechanical properties, is also characterized by good wear resistance and low friction coefficient [11,12]. High physical and mechanical properties of  $\text{TiB}_2$  ceramic materials as well as oxide ceramics with the titanium diboride additives are confirmed by the results presented in papers [13-16].

## 2. Experimental

### 2.1. Materials and procedure

Investigations presented in this paper are related to the  $\text{Al}_2\text{O}_3\text{-Ti(C,N)}$  material with titanium diboride additives. The influence of  $\text{TiB}_2$  on the  $\text{Al}_2\text{O}_3\text{-Ti(C,N)}$  matrix mechanical and tribological properties was studied.

Matrix material was prepared from the following powders: 68 mass.%  $\text{Al}_2\text{O}_3$  (A16SG, ALCOA) with the addition of 2 mass.%  $\text{ZrO}_2$  and 30 mass.%  $\text{Ti(C,N)}$  (H.C.STARCK). For these powders, size of grains was less than  $1\ \mu\text{m}$ . This material application is for the manufacture of multi-point cutting tool. Its density is  $4.2\ \text{g/cm}^3$ , hardness HV0.5 of 1900 and flexural strength equal to 600 MPa. Information about the recommended cutting parameters for the multi-point cutting tools based on  $\text{Al}_2\text{O}_3\text{-Ti(C,N)}$  are shown in the Table 1.

$\text{TiB}_2$  powder (H.C. Starck;  $2.5\text{-}3.5\ \mu\text{m}$ ) was added for the matrix material in an amount of 5, 10, 30 and 50 vol.%.  $\text{Al}_2\text{O}_3\text{-Ti(C,N)}$  ceramics with additives were prepared using the SPS apparatus for sintering. Spark Plasma Sintering furnace with graphite die is shown in Fig. 1.

Materials are compacted in a graphite die using a maximum pressure of 35 MPa at vacuum. Maximum pressure was obtained after duration of 10 min. Vacuum and pressing time 10 min. was to vent the mixture. After that, protective gas was introduced to the chamber of SPS apparatus. Because of  $\text{Ti(C,N)}$  and  $\text{TiB}_2$  good electrical conductivity the passage of current through graphite elements and pressed powder grains is possible. Chamber and graphite die with ceramic powder before sintering process are shown in Fig. 2. Mechanism of SPS sintering process and pulsed current flow through powder particles are shown in Fig. 3.

Table 1.  
Recommended cutting parameters in turning for  $\text{Al}_2\text{O}_3\text{-Ti(C,N)}$  cutting tools [17]

Workpiece material	Type of machining	Cutting parameters		
		$v_c$ [m/min]	$f$ [mm/obr]	$a_p$ [mm]
Cast iron	Moderately accurate, finishing	350-600	$\leq 0.3$	$\leq 3.0$
	Moderately accurate	150-200	$\leq 0.3$	$\leq 3.0$
Normalized carbon steel, < 300 HB	Finishing	250-300	0.05-0.15	$\leq 0.3$
	Moderately accurate, finishing	80-100	$\leq 0.2$	$\leq 1.0$

$v_c$  - cutting speed,  $f$  - feed,  $a_p$  - depth of cut

In the Fig. 4 is shown the sintering parameters for  $\text{Al}_2\text{O}_3$  ceramics with  $\text{TiB}_2$  additives. The shrinkage of various materials during the sintering process is shown in Fig. 5.

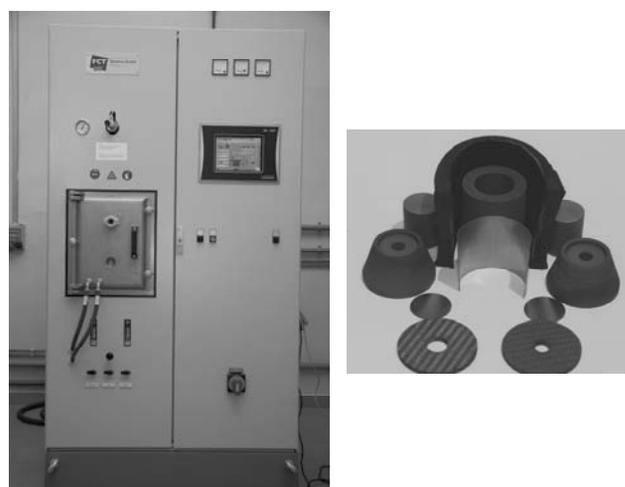


Fig. 1. SPS apparatus with graphite die

For the Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) matrix material a significant shrinkage is observed in the first stage of heating (a temperature of 1250°C). Similar shrinkage were found for materials with the 5 vol.% and 10 vol.% TiB<sub>2</sub> addition.

In the case of materials containing 30 vol.% and 50 vol.% TiB<sub>2</sub> shrinkage was observed also in the first and the second heating stage (at 1250°C and at 1650°C).

Irrespectively of TiB<sub>2</sub> content the shrinkage all investigated materials is from 40% to 42%.



Fig. 2. Graphite die with ceramic powder before sintering process

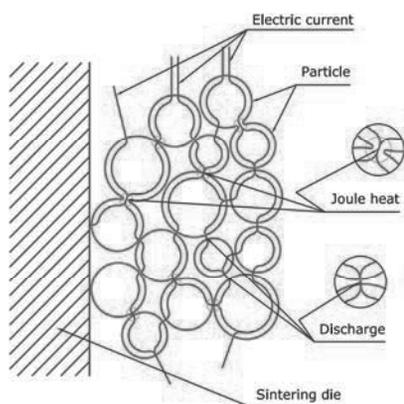


Fig. 3. Pulsed current flow through powder particles

## 2.2. Methodology

After sintering, the materials were subjected to a study of their physical and mechanical properties.

The apparent density  $\rho_p$ , was measured using the hydrostatic method. Young's modulus measurements of the sintered samples were also taken, using the ultrasonic method of measuring the transition speed of transverse and longitudinal waves, using a Panametrics Epoch III flaw detector. The hardness was determined by the Vickers method at a load of 9807 mN using a digital hardness tester (Future Tech. Corp. FM-7).

The tribological studies were performed using a UMT-T2 universal testing machine. Analysis of the tribological properties of the individual materials was performed at a temperature of 22°C and the wear track radius was in the range of 3 mm to 5 mm. The force in wear investigation on the material was 10 N, the speed of the sample was 0.100 m/s and the friction track was 100 m.

The physical, mechanical and tribological investigations were performed on the samples with a grounded and polished surface.

For materials after sintering and after tribological tests SEM observation at JEOL JSM-60LV were carried out. Analysis of chemical composition in micro-areas for individual samples were carried out using EDS microanalysis by INCA ENERGY-350 spectrometer.

## 3. Results of investigations

### 3.1. Mechanical investigations

The results of measurements of the relative density, Young's modulus and hardness of the ceramics obtained by SPS are collected in Table 2. SPS-sintered materials were characterized by the relative density values of 94.4% to 97.9%. Materials with higher content of titanium diboride, sintered at these same conditions were characterized by slightly lower values of relative densities. Material with 5 vol.% TiB<sub>2</sub> was characterized by a density equal to the relative density of the Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) matrix material. A lower value of the relative density of materials with high TiB<sub>2</sub> content confirm that these materials should be sintered at a higher temperature than 1650°C. The investigated materials were characterized by similar Young's modulus of 392 to 414 GPa. Increasing of titanium diboride participation characterized by higher hardness. The highest hardness HV1 was 2225 and was measured for the material containing 50 vol.% TiB<sub>2</sub>. In Fig. 6 is shown the microstructure of the material with the 30 vol.% TiB<sub>2</sub> addition. Analyses of chemical composition in micro-areas for this material are shown in Table 3.

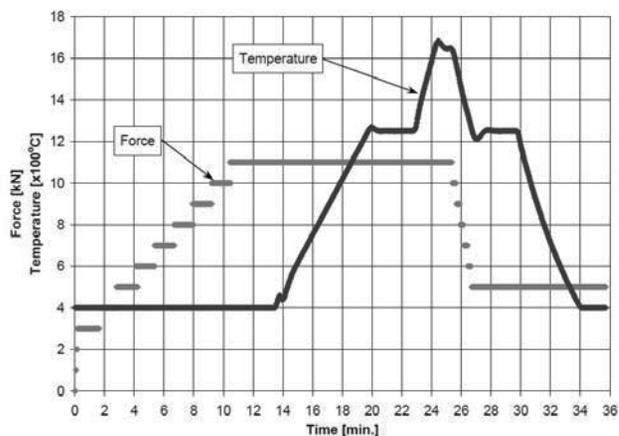


Fig. 4. Sintering parameters of Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) ceramics with TiB<sub>2</sub> additives

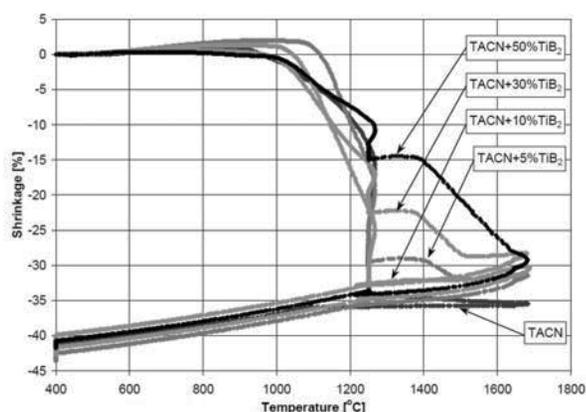


Fig. 5. Shrinkage of materials sintered by SPS-method (TACN: Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) ceramic)

Table 2.

The results of measurements of density, Young's modulus, hardness of ceramic Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) with additives after SPS-sintering

Materials	$\rho_w$ [%]	E [GPa]	HV1	$\sigma$ -HV1
Al <sub>2</sub> O <sub>3</sub> -Ti(C,N)	97.9	401	2042	56
Al <sub>2</sub> O <sub>3</sub> -Ti(C,N) +5 vol.% TiB <sub>2</sub>	97.9	407	1966	139
Al <sub>2</sub> O <sub>3</sub> -Ti(C,N) +10 vol.% TiB <sub>2</sub>	97.7	392	1996	55
Al <sub>2</sub> O <sub>3</sub> -Ti(C,N) +30 vol.% TiB <sub>2</sub>	96.6	402	2106	54
Al <sub>2</sub> O <sub>3</sub> -Ti(C,N) +50 vol.% TiB <sub>2</sub>	94.4	414	2225	247

T<sub>s</sub> - sintering temperature;  $\rho_w$  - relative density;  
E - Young modulus; HV - Vickers hardness

Table 3.

Chemical composition analysis of the Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) material with 30 vol.% TiB<sub>2</sub> additive.

Spectrum	B	C	N	O	Al	Ti	Zr
1	19.75	8.66	-	35.89	16.15	19.56	-
2	-	15.39	8.24	-	-	76.37	-
3	25.41	20.53	-	13.37	0.42	26.03	14.24
4	-	8.46	-	47.84	41.49	2.21	-

Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) matrix material after SPS-sintering achieved higher Young's modulus and hardness compared to the free sintered materials. Ceramics of Al<sub>2</sub>O<sub>3</sub> matrix with reinforcing phase Ti(C,N) after free sintering is characterized by Young's modulus of 380 GPa and hardness HV1 of 1750.

### 3.2. Tribological investigation

The friction coefficients measured for ceramics Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) with TiB<sub>2</sub> additions are presented in Table 4. Tribological test results were compared with the wear results of the matrix material.

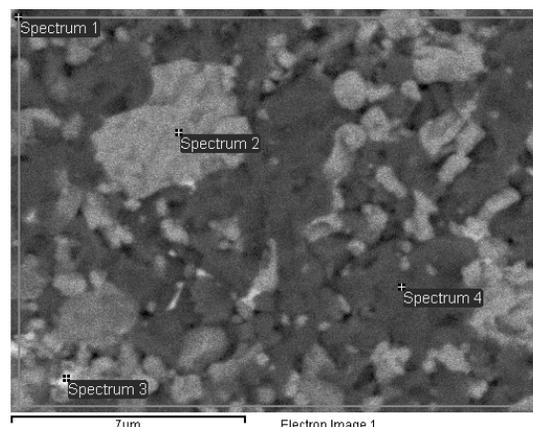


Fig. 6. Microstructure of the Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) material with 30 vol.% TiB<sub>2</sub> additive, EDS-analysis

Table 4.

The results of tribological tests on Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) ceramics with TiB<sub>2</sub> additives after SPS-sintering.

Materials	Radius of wear track	Friction coefficient	
	[mm]	COF	$\sigma$ -COF
Al <sub>2</sub> O <sub>3</sub> -Ti(C,N)	3	0.51	0.06
Al <sub>2</sub> O <sub>3</sub> -Ti(C,N) +5 vol.% TiB <sub>2</sub>	3	0.49	0.09
Al <sub>2</sub> O <sub>3</sub> -Ti(C,N) +10 vol.% TiB <sub>2</sub>	4	0.49	0.13
Al <sub>2</sub> O <sub>3</sub> -Ti(C,N) +30 vol.% TiB <sub>2</sub>	3	0.33	0.06
Al <sub>2</sub> O <sub>3</sub> -Ti(C,N) +50 vol.% TiB <sub>2</sub>	4	0.35	0.05
Al <sub>2</sub> O <sub>3</sub> -Ti(C,N)	4	0.35	0.04
Al <sub>2</sub> O <sub>3</sub> -Ti(C,N) +30 vol.% TiB <sub>2</sub>	5	0.39	0.08
Al <sub>2</sub> O <sub>3</sub> -Ti(C,N) +50 vol.% TiB <sub>2</sub>	3	0.31	0.04
Al <sub>2</sub> O <sub>3</sub> -Ti(C,N) +50 vol.% TiB <sub>2</sub>	4	0.32	0.04

Table 5.

Chemical composition analysis of the wear layer formed on Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) ceramic +30 vol.% TiB<sub>2</sub> additive.

Spectrum	B	C	O	Al	Ti	Fe
1	22.81	5.79	41.38	27.98	2.04	-
2	17.08	7.91	24.41	0.83	3.13	46.65
3	28.72	-	16.38	1.48	53.41	-
4	21.80	6.28	33.09	11.97	3.56	23.30
5	20.37	7.42	37.14	11.68	15.20	8.19

Friction coefficient for Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) matrix material is 0.51. Materials with titanium diboride are characterized by lower friction coefficients. Wear of couples in these studies were a ceramics and hardened steel balls. With the increase of TiB<sub>2</sub> participation reduction in friction coefficient was observed. For the material with 5 vol.% TiB<sub>2</sub> additive friction coefficient was 0.49 and for the Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) with 50 vol.% TiB<sub>2</sub> material this coefficient was 0.31.

The wear tracks is mainly formed on the surface of the ceramics specimen disc. In Figures 7 to 10 are shown wear traces for various materials. With the increase of TiB<sub>2</sub> participation in the investigated materials, this layer was less uniform and more

frayed. For example, Fig. 11 presents the layer formed on ceramic containing 30 vol.% TiB<sub>2</sub>. In Table 5 were shown the results of the chemical composition analysis.

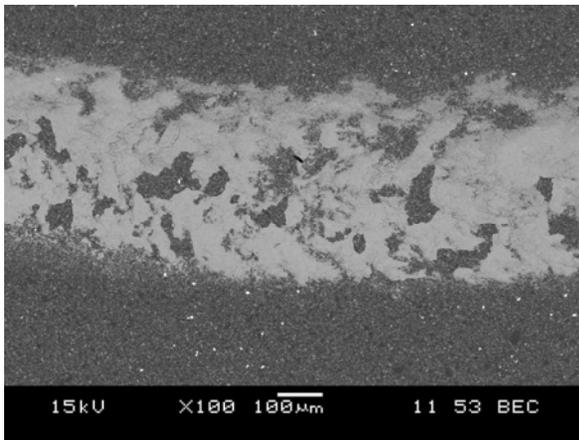


Fig. 7. Wear track of Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) ceramic with 5 vol.% TiB<sub>2</sub>

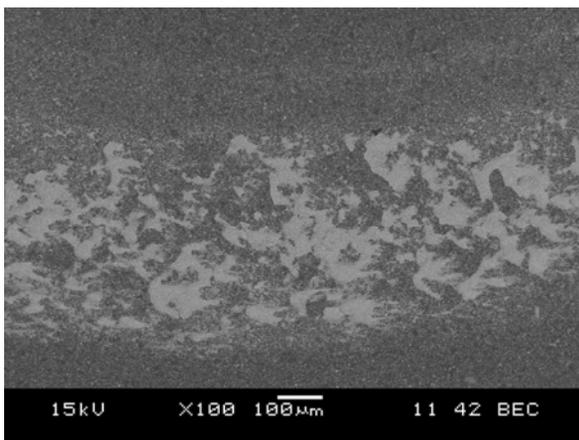


Fig. 8. Wear track of Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) ceramic with 10 vol.% TiB<sub>2</sub>

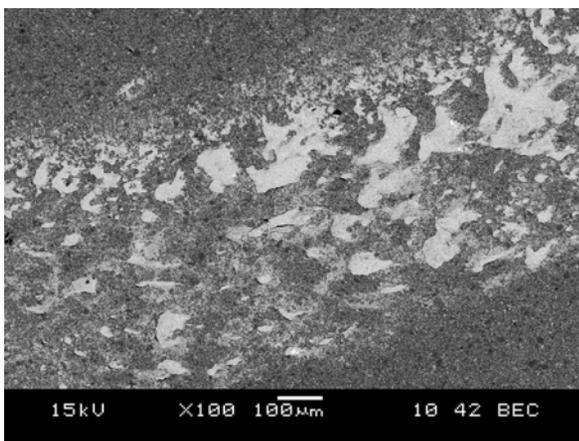


Fig. 9. Wear track of Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) ceramic with 30 vol.% TiB<sub>2</sub>

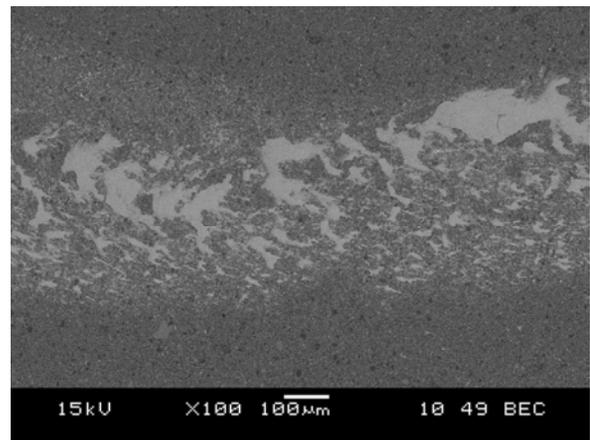


Fig. 10. Wear track of Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) ceramic with 50 vol.% TiB<sub>2</sub>.

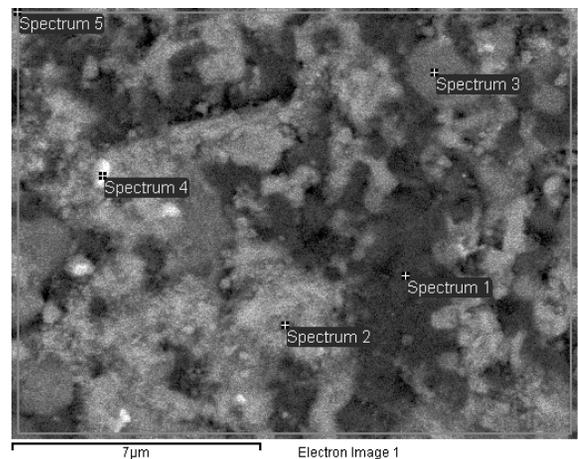


Fig. 11. Wear track of Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) ceramic with 30 vol.% TiB<sub>2</sub> additive, EDS-Analysis.

#### 4. Conclusions

The paper presents results of the mechanical properties and tribological properties of Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) ceramics with the titanium diboride addition. The materials were obtained using the SPS sintering method. This method allows to sintering process ceramics in a much shorter duration and at a lower temperature. The materials after SPS-sintering process are characterized by higher hardness and Young's modulus than the materials manufactured using the free sintering method. Materials with the TiB<sub>2</sub> addition were characterized lower value of relative density than the matrix. All materials were characterized similar values of Young's modulus. With increasing of TiB<sub>2</sub> participation the slight increase of hardness HV1 is observed. There is a bigger influence of TiB<sub>2</sub> addition on tribological properties of Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) ceramic. The value of friction coefficient for Al<sub>2</sub>O<sub>3</sub>-Ti(C,N) ceramics with titanium diboride is in range of 60.8% to 96% in relation to the friction coefficient for the matrix material. The

lowest value of friction coefficient has been measured for the material with the addition of 50 vol.% TiB<sub>2</sub>.

## Acknowledgements

This work was supported by the 2007-2013 Innovative Economy Programme under the EU's National Strategic Reference Framework, Priority Axis 1, Section 1.1.3, No. UDA-POIG.01.03.01-12-024/08-00, 26 March 2009.

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