

Microstructure, mechanical properties and machining performance of hot-pressed Al₂O₃ - ZrO₂ - TiC composites

M Grigoriev^{1,2*}, N Kotelnikov³, S Buyakova^{1,2,3}, S Kulkov^{1,2,3}

¹ Institute of Strength Physics and Materials Science SB RAS, 12/4, pr. Akademicheskii, Tomsk, 634055, Russia

² Tomsk Polytechnic University, 30, Lenin Avenue, Tomsk, 634050, Russia

³ Tomsk State University, 36, Lenin Avenue, Tomsk, 634050, Russia.

*E-mail: MVGrigoriev@yandex.ru

Abstract. The effect of adding nanocrystalline ZrO₂ and submicron TiC to ultrafine Al₂O₃ on mechanical properties and the microstructure of the composites developed by hot pressing was investigated. It was shown that by means of hot pressing in an argon atmosphere at a sintering temperature of 1500 °C one can obtain the composites Al₂O₃ - ZrO₂ - TiC with a fine structure and minimal porosity. It has been shown that in material a multi-scale hierarchical structure is formed, which possesses high physical and mechanical properties: the hardness and fracture toughness was 21.5 GPa and 5.2 MPa·m^{1/2} respectively, the modulus of elasticity was 500 GPa and bending strength was 390 MPa. Tests on composites' cutting properties were carried out on interrupted cutting of hardened steel. All samples had wear of the cutting edge in the form of chips of the grain rear working surface of the tool, but the cutting tool which was made by Al₂O₃ - 10% ZrO₂ - 10% TiC had a minimum width of wear.

1. Introduction

The development of ceramic composites with high hardness and chemical inertness, able to withstand prolonged exposure to corrosive environments, high pressure, shock, and temperature, with high resistance to brittle fracture and fracture toughness is the main problem of materials science now [1]. This problem is very important, for example, in the cutting of high-strength tempered steel due to the non-uniform loading of the tool, resulting in the tool life being greatly reduced [2]. Conventional cutting tools such as hard-alloys cannot be used in this case due to their lack of toughness and bending strength. One of the most promising materials for the role of such cutting tools is ceramic composites [3,4].

Alumina - TiC composites are widely used as cutting tool inserts due to their ability to machine at higher speeds than cemented carbides and their superior hardness, toughness and strength compared to alumina [5]. They are commonly known as 'black ceramics', having a composition of 70% alumina and 30% TiC, has a high hardness of about 22GPa but insufficient toughness - not more than 4 MPa·m^{1/2}[6]. Alumina - zirconia composites are also used as cutting tool inserts where zirconia toughens the alumina matrix by stress induced tetragonal to monoclinic martensitic phase transformation. The hardness and fracture toughness of Al₂O₃- ZrO₂ composites is only 18 GPa and 5 MPa·m^{1/2} respectively [7]. It is well known that addition of either oxide or non-oxide additives improve mechanical properties of alumina. In this context, addition of both oxide and non-oxide



additives may be an attractive option as it may impart the beneficial effects of both the additives in the resulting composites. It becomes even more attractive if the oxides and non-oxides are nano sized as the majority of the nano particles may remain at grain boundaries and interact with cracks resulting in interesting features not observed in conventional composites.

The purpose of this research is to obtain an oxycarbide composite based on alumina and additives of zirconia and titanium carbide, in order to study its structure and physico-mechanical properties and test cutting properties.

2. Materials and experimental procedure

This study involves commercial Al_2O_3 powders (USA) with average particle size $4.7\ \mu\text{m}$ and purity 99 %; TiC powders (Russian manufacture) with particle size $5\ \mu\text{m}$ and purity 98.0% and ZrO_2 powders (Grade: TZ-3Y-E, Tosoh Corp., Japan) with nominal particle size 30 nm and purity 99.7%.

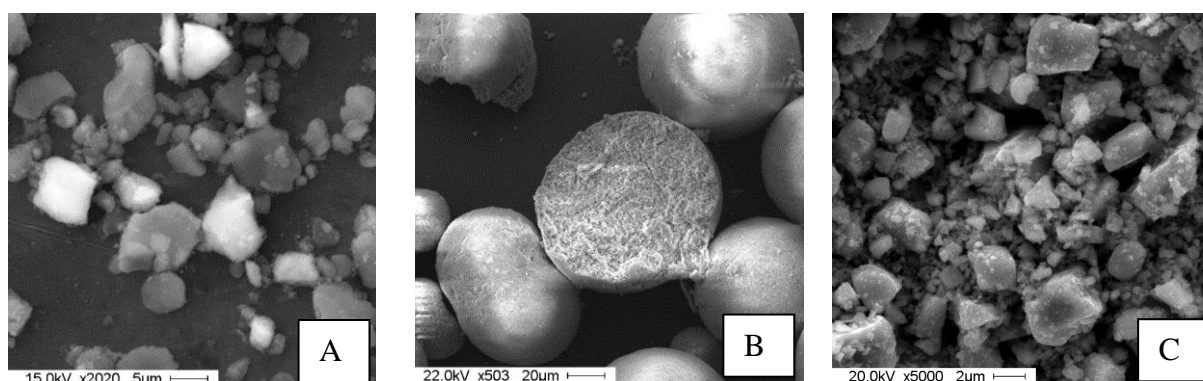


Figure 1. SEM micrograph Al_2O_3 powders (A), ZrO_2 (B), TiC (C).

The mixture of powder was prepared as follows: water suspensions created individual components mixed with each other with a magnetic stirrer, followed by suspension sonicated. The deposition of the resulting composition was produced by flocculation of particles from the solution by raising the PH level, followed by vacuum drying. The resulting composite mixture is shown in table 1.

Table 1. - The ratio of components in composites

Samples	Content		
	Al_2O_3 , %	ZrO_2 , %	TiC , %
AZT-1	85	10	5
AZT-2	80	10	10
AZT-3	70	10	20
AZT-4	60	10	30
AZT-5	75	20	5
AZT-6	70	20	10
AZT-7	60	20	20
AZT-8	50	20	30

Ceramic composites were prepared by hot pressing in an argon atmosphere at a sintering temperature of 1500°C , with a pressing pressure of 50 MPa. The holding time was 10 minutes.

X-ray diffraction data were obtained using an X-ray diffractometer with CuK radiation, grain size and elemental analysis of the composites were carried out on a LEO EVO 50 (Zeiss, Germany) scanning microscope.

Densities of sintered samples were determined by Archimedes' method with distilled water. For hardness measurements sintered samples were polished by diamond paste up to $1\ \mu\text{m}$ grains and

indentations were made using a 5 kg load; an average of ten indents was tested. Vickers hardness was determined by using the formula (1).

$$H_v = 1.854 \cdot P / d^2 \quad (1)$$

where P – load, N; d – diagonal indentation, μm .

Fracture toughness was determined by the formula (2).

$$K_{Ic} = 0.035 \cdot (H \cdot a^{1/2}) \cdot (E\phi / H)^{0.4} \cdot (l / a)^{-0.5} \quad (2)$$

where H – hardness, GPa; E – Young's modulus, MPa; a – half diagonally indentation, μm ; l – the crack length from the corner indentation, μm ; ϕ – constant.

Nanoindentation was performed using a G200 nanoindenter, the load was 100 mN. On the curve load/displacement of the indenter was calculated the modulus of elasticity of the composites.

The flexural strength was measured using the testing machine GP-DLC 30 kN, and was calculated using the formula (3).

$$\sigma_{bend} = 1.5 \cdot P \cdot l / b \cdot h^2 \quad (3)$$

where: P – load, N; l – distance between supports, mm; b – the width of the sample, mm; h – sample height, mm.

Tests of composites' cutting properties were carried out on interrupted cutting of hardened steel. The material of hardened steel 40X had a diameter of 34mm. Cutting conditions: speed of rotation was 150 m/s; feed 0.1 mm; removal depth of 0.3 mm.

3. Results and discussion

Elemental analysis of the fractured surface of the Al_2O_3 – 10% ZrO_2 – 20% TiC composite (Figure. 2,a) has shown that all phases are distributed randomly (Fig. 2, b-d), but titanium carbide has a higher grain size. Measuring of the average grain size of the individual components has shown that alumina has 1.5 microns; zirconia - 0.8 microns; titanium carbide - 2.5 microns, thus the average grain size of the individual components in the structure of the composites is not significantly higher than the average particle size of the initial powders.

XRD analysis showed that the alumina in composites is in α - modification (corundum), zirconia is cubic and tetragonal modifications and titanium carbide has a NaCl-cubic lattice.

Mechanical properties of sintered composites are shown in table 2. As one can see from the table the best combination of hardness and fracture toughness was achieved in the material containing 10% zirconia, thus the optimum combination of properties was achieved in the composite AZT-3, for other contents of titanium carbide the hardness and fracture toughness are lower.

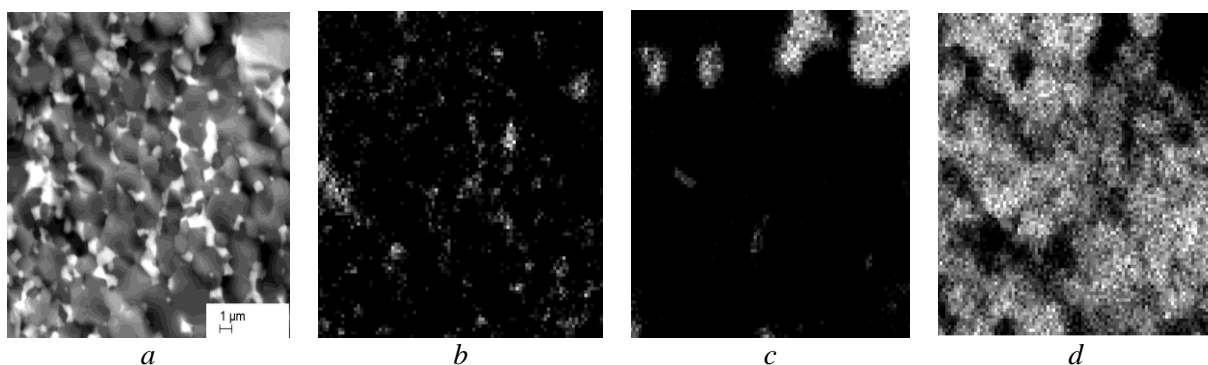


Figure 2. The fractured surface of nanocomposite Al_2O_3 - 9% ZrO_2 - 20% TiC (a); element distributions in ZrL α (b) TiK α (c) and AlK α (d) X-rays.

The results of mechanical properties of AZT 1 - 4 samples are shown in table 3. As one can see that the best combination of hardness and fracture toughness is achieved in the compositions comprising 10% zirconia. The hardness and fracture toughness in the bulk and on its surface of materials differ by

about 10%: the hardness at the surface is smaller than in the bulk, and conversely there is higher fracture toughness. Thus the optimum combination of properties is achieved in the composite AZT-3. The addition of titanium carbide than 20% lowers the fracture toughness and hardness. A typical crack indentation for sample AZT-3 is shown in Figure 3.

Such differences in the properties at the surface and in the volumes are due to contact of the mixtures with a graphite mold, whereby additional carbonization of oxides at the surface can occur.

Figure 4 shows that the optical image of the edges of cutting tools were made from AZT-2, AZT-3, AZT-4 materials and a control sample manufactured from silicon-aluminum oxide-nitride (SiAlON) with the same sizes. Average widths of wear of the cutting edge, calculated on several optical images for each sample, depending on the composition are shown in Table 3.

As one can see all the samples have wear of the cutting edge in the form of chips of grain on the rear working surface of the tool, but the cutting tool which was made from AZT-2 has a minimum width of wear. It is shown that the width of the cutting edge of the industrial tool SiAlON greater than the same values of AZT-2 and AZT-4 but chipping grains on the cutting surface were not observed.

Table 2. - Mechanical properties of the composites

Samples	Density, g/cm ³	Relative density	Hv, GPa	K _{1c} , MPa*m ^{1/2}
AZT-1	4.03	0.97	18.6	5.04
AZT-2	4.16	0.99	19.8	5.44
AZT-3	4.26	0.99	21.4	5.68
AZT-4	4.36	0.99	19.3	5.16
AZT-5	4.32	0.99	13.3	5.82
AZT-6	4.35	0.99	17.7	5.44
AZT-7	4.44	0.99	16.0	5.41
AZT-8	4.55	0.99	16.5	5.83

Table 3. Hardness and fracture toughness of the composites on the surface and in the bulk. Bending strength.

Samples	Hv, GPa Vickers		Nanohardness		K _{1c} , MPa*m ^{1/2}		σ_{bend} , MPa	Width of edge wear, μm
	Surface	Bulk	Hv, GPa	E, GPa	Surface	Bulk		
AZT-1	18.6	20.6	24.9	460	5.04	4.42	438	-
AZT-2	19.8	21.3	28.3	503	5.44	5.12	393	80
AZT-3	21.4	19.0	25.8	502	5.68	4.29	407	140
AZT-4	19.3	21.6	27.9	479	5.16	5.08	365	115
SiAlON	19.6	-	-	345	5.82	-	-	127

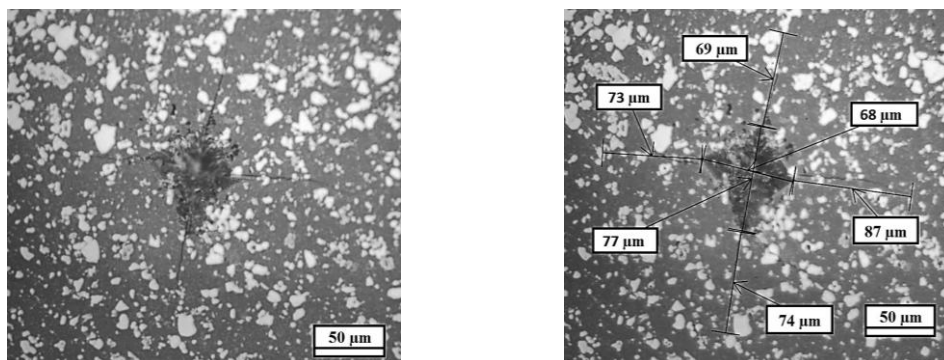


Figure 3. Optical micrograph of crack from indentation at 5 kgf for 70Al₂O₃-10ZrO₂-20TiC

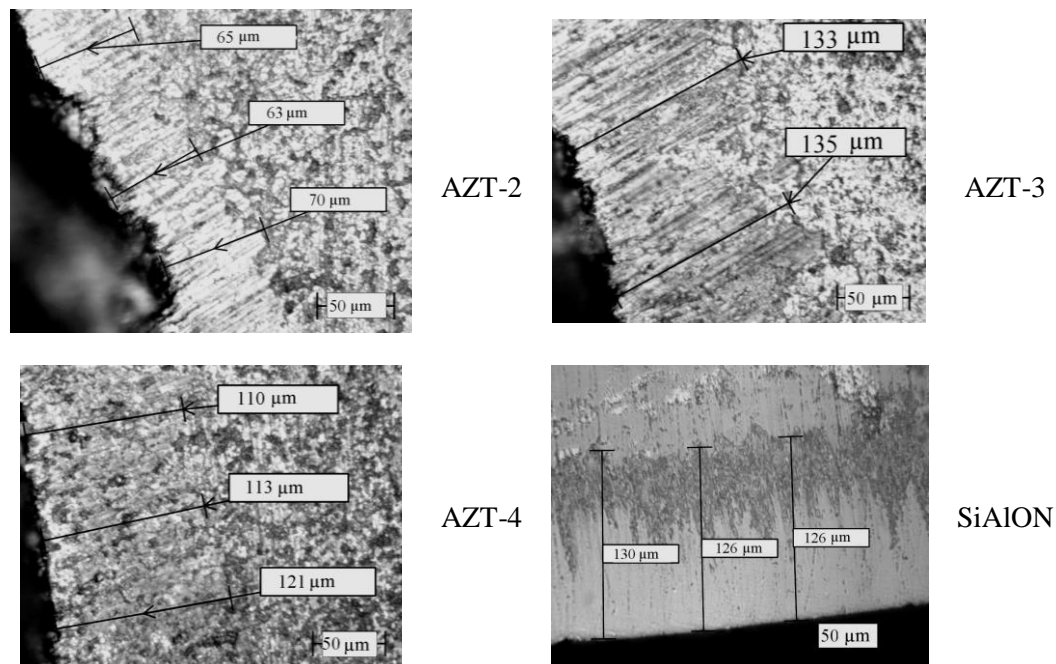


Figure 4. The optical images of the cutting edges for AZT 1 - 4 and SiAlON.

4. Conclusion

1. It was shown that by hot pressing in an argon atmosphere at a sintering temperature of 1500 °C can be obtained the composites Al_2O_3 - ZrO_2 - TiC with a fine structure and minimal porosity.
2. XRD analysis has shown that in the sintered composites phase the content is the same as in initial mixtures. Scanning electron microscopy shows a uniform distribution of the components in a matrix of alumina.
3. The best combination of mechanical properties is shown in the composite Al_2O_3 - 10% ZrO_2 - 20% TiC ; its hardness and fracture toughness was 21.3 GPa and $5.12 \text{ MPa}\cdot\text{m}^{1/2}$ respectively; the elastic modulus was 500 GPa and the bending strength was 390 MPa.

Acknowledgement

This research was conducted with partial financial support by the project #14.607.21.0056-RFMEFI60714X0056. Elemental analysis and images composite structure obtained on a LEO EVO 50 (Zeiss, Germany) in the "NANOTECH" ISPMS SB RAS.

References

- [1] Ercin Cura M, Kim S H, Muukkonen T, Varjus S, Vaajoki A, Soderberg O, Suhonen T and Hannula S P 2013 *Ceram Int.* **39** 2093-2105
- [2] Kumar A S, Durai A R, Sornakumar T 2003 Machinability of hardened steel using alumina based ceramic cutting tools *Int. J Refract Met Hard Mater* **21**109-117
- [3] Shaw M C 2005 Tool materials metal cutting principles. 2nd ed. (New Delhi: Oxford University Press) pp. 307–348
- [4] Grigoryev M V, Buyakova S P and Kul'kov S N 2013 *Refractories and Industrial Ceramics* **11/12** 20-25
- [5] Grigoryev M V, Molchunova L M, Buyakova S P and Kul'kov S N 2013 *Russian Physics Journal* **7 2** 206-210.
- [6] Zhang Y, Wang L, Jiang W, Chen L and Bai G 2006 *J. Eur. Ceram Soc.* **26** 3393-3397.
- [7] Savchenko N L, Korolev P V, Melnikov A G, Sablina T Y and Kul'kov S N 2008 *Fundamental problems of modern materials science* pp. 94-99 [in Russian]