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Investigation of structure and properties of composite material Al_2O_3–SiC obtained by electroconsolidation process

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The results of research of influence of structure and phase composition parameters on the microstructure, physico-mechanical and cutting properties of aluminum oxide-based composites obtained by electrosparking (electroconsolidation) are described. It has been established that formation of the composite structure due to introduction of SiC nanopowders into the micropowder of aluminum oxide makes it possible to improve the physical and mechanical properties and wear resistance of Al_2O_3 ceramics.

Keywords: composite ceramics, aluminum oxide, nanopowder, silicon carbide, electrospinning, mechanical properties.

Описаны результаты исследований влияния параметров структуры и фазового состава на микроструктуру, физико-механические и режущие свойства композитов на основе оксида алюминия, полученных методом электроспекания (электроконсолидация). Установлено, что формирование композиционной структуры за счет введения в микропорошок оксида алюминия нанопорошков SiC позволяет повысить физико-механические свойства и износостойкость керамики Al_2O_3.

Формування структури і властивостей композиційного матеріалу Al_2O_3–SiC у процесі електроконсолідації. *Р.В.Вовк, Н.М.Прокопів, В.А.Чишкала, М.В.Кислиця.*

Описано результати досліджень впливу параметрів структури і фазового складу на мікроструктуру, фізико-механічні та ріжучі властивості композитів на основі оксиду алюмінію, отриманих методом електроспікання (електроконсолідація). Встановлено, що формування композиційної структури за рахунок введення у мікропорошок оксиду алюмінію нанопорошків SiC дозволяє підвищити фізико-механічні властивості і зносостійкість кераміки Al_2O_3.

1. Introduction

Today many composite materials of different types are being developed for cutting tools: oxide (Al_2O_3–ZrO_2), mixed (Al_2O_3–Ti[N,C]), nitride (based on Si_3N_4), reinforced. The materials of each of these types have a certain combination of physical and mechanical characteristics (hardness, strength, heat resistance, chemical inertness) and retain these properties at operating temperatures above 800°C [1]. However, each of these materials works most effectively in a certain narrow area. Traditionally used oxide-carbide cutting materials (Al_2O_3–TiC), for example [2], have proven themselves in the treatment of high-strength steels and cast irons. But at cutting speeds above 200 m/min, due to low thermal conductivity, the temperature in the cutting zone reaches 1100–1200°C, which leads to an increase in adhesive, chemical and diffusion wear.

Improving the heat sink in the work area is one of the main tasks when creating the new materials for the cutting tool. In Al_2O_3–SiC system, silicon carbide acts as a reinforcing element and has a thermal conductivity coefficient much higher (32–36 W/m·K) than it is for matrix of Al_2O_3 (20–24 W/m·K), which ultimately allows to increase the thermal conductivity of the composite as a whole [3].

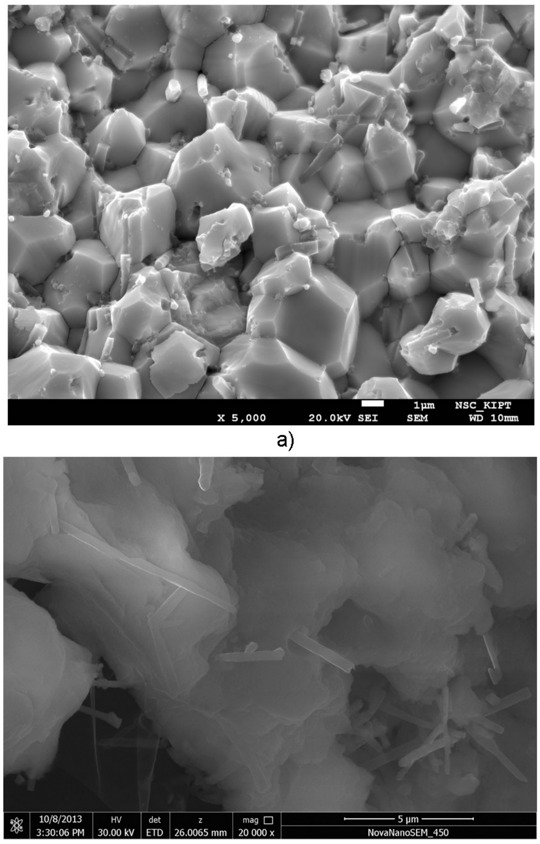
From the technological point of view, it is most expedient to take as the main phase micropowders Al_2O_3, but as a strengthening component it is better to use SiC nanopowder. The use of a combination of micro-nano has several advantages, in particular, it allows better mixing of the powders, in comparison with nano-nano, which have surplus surface energy and are easily agglomerated [4]. In addition, the cost of nanopowders is much higher than micropowders.

The paper [5] is known and devoted to the study of the composition of Al_2O_3–5 wt. % SiC after sintering at temperatures of 1380 and 1430°C. In this work, the additives of the powders are intended to improve the tribological properties of the material.

2. Experimental

As objects of research it was used ceramics produced by electropulse plasma sintering of α–Al_2O_3 micron powders with an average particle size of 1 µm micron powders α–Al_2O_3 with addition of 5 vol. % β–SiC, as well as nanopowders of alumina (average particle size 100 nm) with addition of 5 vol. % of β–F2SiC whiskers up to 50 µm in length and 1 µm in diameter. The addition of spherical particles and filaments of silicon carbide to the initial alumina powder resulted in reduction of the friction coefficient μ of the sintered ceramics by ~ 30 % (from 0.62 for Al_2O_3 to 0.46 and 0.47 for Al_2O_3 + 5 vol% SiC ceramics with particles and filamentary fibers, respectively).

Structure, physicomechanical and tribological properties of composite ceramics based on aluminum oxide obtained by the method of electropulse plasma sintering were examined. It was shown that the use of EIPS makes it possible to obtain the high-density (~ 99.7 % of the theoretical density) Al_2O_3–5 vol. % SiC ceramic with homogeneous fine-grained structure and enhanced strength properties. The authors of the article found that formation of the composite structure in ceramics based on aluminum oxide by adding a small amount of filamentary SiC-fibers leaded to increase in wear resistance both in comparison with pure alumina and in comparison with the dispersed-hardened ceramic containing submicron SiC-particles. However, no justification was given why the most optimal are 5 % by volume of SiC fibers. For example, our experience in producing composites with SiC fibers (Fig. 1) shows that it is extremely difficult to obtain a uniform distribution of the fibers. In addition, silicon carbide fibers have the high cost and it is difficult to obtain the homogeneous distribution when mixed.



a)

b)

Fig. 1. Fractogram of specimen fracture with SiC fibers in Al_2O_3 (nano)–SiC fiber composite obtained by electrosintering at temperature T = 1400°C, pressure P = 30 MPa, synthesis time τ = 3 min. a) – ×5000, b) – ×20000.

In another article [6], the material was obtained by adding the special ligature of titanium oxide and non-stoichiometric sili-

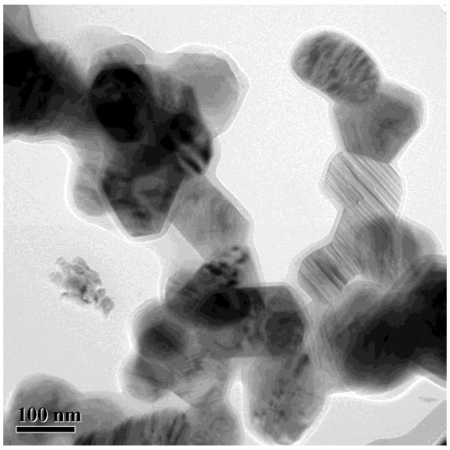


Fig. 2. Nanopowder SiC 100 nm produced by SAINT-GOBAIN (France). Brand: NANO SiC CAS No. 409-21-2.

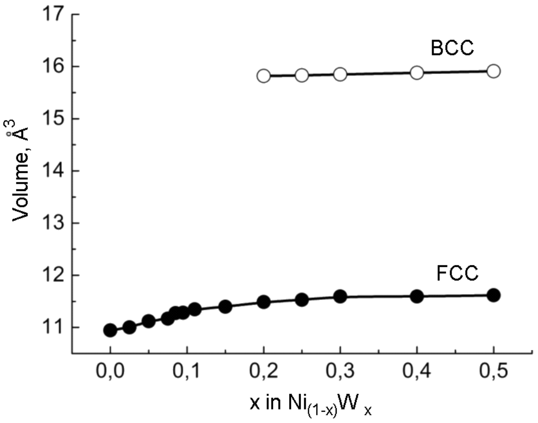


Fig. 3. X-ray phase analysis: clearly expressed phase 3C (cubic). There is a small amount of 6H–SiC phase.

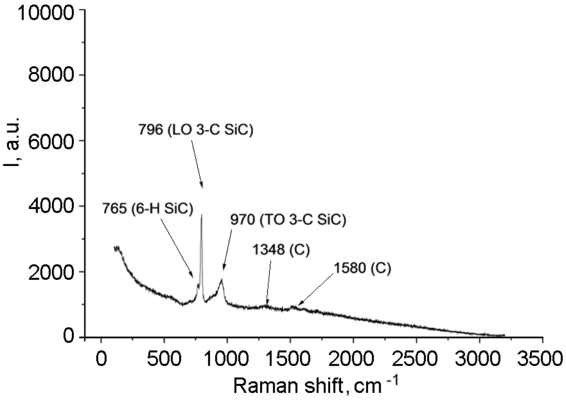


Fig. 4. Raman spectroscopy: clearly expressed pennant and longitudinal modes of cubic SiC.

con carbide, which, in the authors opinion, activated the process of electropulse hot pressing, thereby contributed to obtaining the high physical and mechanical properties. However, this article does not specify the degree of non-stoichiometry of silicon carbide, and it is not entirely clear why the activation of the sintering process takes place.

In our researches various compositions were pressed, both in proportion and in the fraction of the components from the micropowders of aluminum oxide and nanopowders of silicon carbide, in order to obtain the best characteristics. The composition with micro-Al_2O_3 (produced by Zaporozhye Abrasive Plant) and 15 wt. % nano-SiC (Saint-Gobain, France) was optimal.

3. Results and discussion

Results of the powder analysis of the NANO type SiC show that the average SiC crystallite size is about 100 nm. The predominant phase of SiC is cubic 3C. Analysis of X-ray patterns and Raman spectra indicates the presence of minor amounts of the hexagonal phase of 6H–SiC and carbon.

Mixing of the initial powders was carried out in a planetary mill in alcohol solution [7]. Pressing was carried out on the original plant [8], modernized to more accurately determine the shrinkage during the hot pressing. The sintering temperature varied from 1370°C to 1800°C, which allowed achieving the best mechanical properties.

It is known that obtaining the bulk nanomaterials is a challenge to modern nanoparticle consolidation technologies that can be collectively called sintering. In these processes, compaction and grain growth are competing, which makes the problem difficult. The key task of the nanomaterials sintering is to achieve a high material density, provided that nanosized grains are preserved in the range where the size effect is observed [9]. As our experience shows, the choice of the consolidation process depends on the structure of grain boundaries, or interphase boundaries. Residual porosity and defective boundaries substantially impair the nanostructured materials properties. This problem can be solved through intensification of the sintering process and, accordingly, decrease in the time of the high-temperature stage for high-intensity processes with the high rates of heating of the rate of consolidation can grow by several orders of magnitude [10]. This leads to complete compaction of the powders in a very short time with the preservation of the nanoscale internal structure. Electrosintering allows to obtain the consolidated ceramic materials, such as Al_2O_3, ZrO_2, TiC,

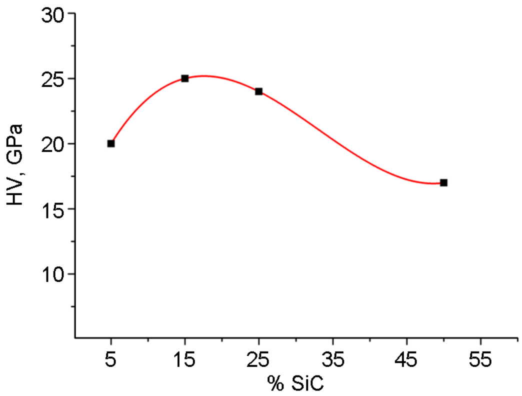


Fig. 5. Dependence of hardness of Al_2O_3–SiC composite from content of mass. % of SiC nanopowders in hot pressing method of electrosparking at T = 1400°C, P = 30 MPa, sintering time τ = 2 min.

WC without impurities and with minimal grain growth over time of the order of 10 min, whereas the traditional sintering requires several hours and special additives that degrade the material properties [11]. At the same time, electric sintering is used with the equally high result for both electrically conductive and non-conductive powders (by using electrically conductive graphite molds). The choice of nanocomposites as an object of research is explained not only by their large practical significance, but also by the fact that during the sintering the recrystallization of different phases of the composite blocks each other. This facilitates the achievement of the consolidated nanostructure.

Preliminary researches of the mechanical properties of Al_2O_3–SiC composites obtained by electrosparking at different SiC contents showed that the highest hardness was possessed by the composite with content of 15 % by weight (Fig. 5).The distribution of matrix elements and filler can be seen in the electronic pictogram in Fig. 6.

Considering the distribution of chemical elements at individual points of the composite material, it can be seen that the gray phase is the grains of aluminum oxide, and the light phase is the silicon carbide grain, which basically retain their nanoscale, with some exceptions (Fig. 7, Tabl.).

The composite material had hardness of H_V = 25 GPa, fracture toughness K_{IC} = 5.5–6 MPa·m$^{1/2}$, flexure toughness σ_{fl} = 50 MPa. Comparative tests of the cutting properties of the composite material with the widespread oxide-carbide instrumental ceramic material BOK-71 were performed.

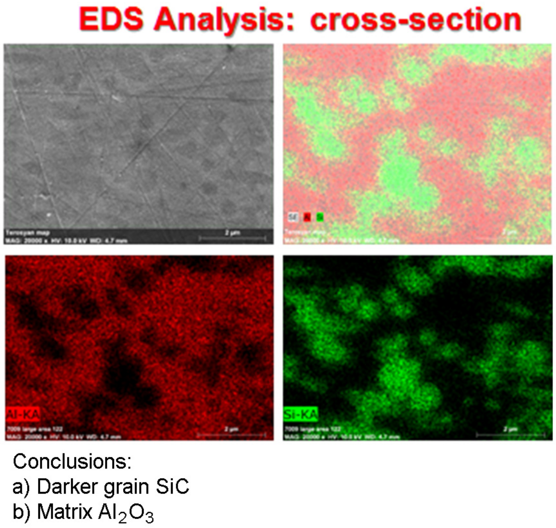


Fig. 6. Phase distribution of component composites of composite material Al_2O_3-15 wt.% SiC, obtained by electrosintering at T = 1400°C, P = 30 MPa, sintering time τ = 2 min.

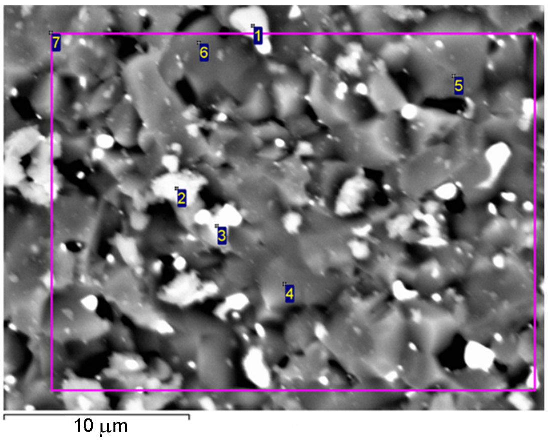


Fig. 7. Distribution of chemical elements in Al_2O_3–15 wt.% SiC, obtained by electrosintering at T = 1400°C, P = 30 MPa, sintering time τ = 2 min.

When rotating the calender shafts of stainless steel (with hardness HRC = 56.58) under cutting conditions v = 150 m/min, s = 0.01 mm/rev, t = 0.3 mm and wear criteria for the back surface 0.4 mm, the strength of the BOK-71 was 30 min, and for the obtained material it was 48 min.

4. Conclusions

Thus, as a result of the researches of composite materials of different composition, obtained under different electrosintering modes, it was established that the composition of Al_2O_3–15 wt. %. SiC with combination of grain sizes of micro-nano

Table 1. Distribution of chemical elements at the individual points of the composite material Al2O3-15 mas.% SiC

Range	C	N	O	Al	Si	Fe	Nb	W	??
1	6.51	3.00	51.68	31.87	2.02	1.11	0.00	3.82	100.00
2	6.09	5.52	39.54	43.14	4.31	0.62	0.03	0.75	100.00
3	5.35	12.12	41.88	30.94	7.53	1.21	0.00	0.96	100.00
4	6.96	2.74	46.14	42.67	0.95	0.22	0.00	0.32	100.00
5	3.25	17.09	44.95	27.72	6.92	0.07	0.00	0.00	100.00
6	2.10	3.76	47.89	42.98	2.10	0.17	0.36	0.64	100.00
7	4.08	5.86	48.42	34.78	4.50	0.73	0.14	1.48	100.00
Average	4.91	7.15	45.79	36.30	4.05	0.59	0.08	1.14	100.00
Stand. error	1.81	5.41	4.11	6.54	2.52	0.46	0.14	1.27	
Max.	6.96	17.09	51.68	43.14	7.53	1.21	0.36	3.82	

at the sintering temperature $T = 1400°C$, sintering time $\tau = 2$ min, pressure P = 30 MPa had the best characteristics. The obtained samples have hardness $H_V = 25$ GPa at $K_{IC} = 5.5–6$ MPa·m$^{1/2}$. The best cutting ability is explained not only by high mechanical characteristics, but also, apparently, by the higher thermal conductivity. The obtained material is promising for the processing of calenders made of alloyed high-hardness steel.

Proceeding from the fact that Al_2O_3 powders are available raw materials of domestic production. Cutting ceramic materials based on it remain promising and require subsequent improvement, search for new additives and improvement of characteristics of already known compositions.

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