Composite materials based on fine-dispersed $\text{Al}_2\text{O}_3$ with enhanced physical and mechanical properties

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Abstract. Ceramic nanocomposites of silicon carbide reinforced $\text{Al}_2\text{O}_3$ are obtained by electroconsolidation. Mechanical properties are investigated and their dependencies on the composition and consolidation modes are shown over a wide range. The optimal composition and modes of electrical consolidation for the $\text{Al}_2\text{O}_3$-$\text{SiC}$ composite are revealed. A criterion for choosing a material for instrumental application, which takes into account the basic properties of the cutting tool, is proposed. Comparison of the obtained composites with similar materials used for the manufacture of tool products including global manufacturers has been carried out.

1. Introduction
Obtaining new competitive composite materials with improved mechanical properties requires an integrated approach in the selection of the composition and compaction parameters of powder materials. An important factor affecting the strength is the grain size. According to the theory of Griffiths [1], the strength of ceramics depends on the number of defects present, and the number of defects is proportional to the grain size for ceramic materials, so a reduction in the grains can improve the strength of polycrystalline materials. This paper presents the results of research of the mechanical and thermophysical properties of nanocomposite materials based on $\text{Al}_2\text{O}_3$ and $\text{SiC}$, as well as an analysis of the dependence of these properties on the composition and sintering conditions.

2. Experiment
The researched samples were manufactured by the method of electroconsolidation with direct current transmission on the original installation [2]. A mixture of microdispersed alumina powder with different content of nanodispersed SiC powder was used for pressing. Pressing pressure $P = 35$ MPa, holding time 2-3 min, sintering temperature from 1400 to 1700°C. The thermal conductivity coefficient was measured using the method of stationary heat flux described in [3].

Microhardness and crack resistance values were determined by measuring the diagonal of the imprint and the length of the radial cracks produced by the diamond indenter in the form of a four-sided pyramid with the apex angle $\alpha = 136^\circ$ (Vickers pyramid) using an automatic microhardnessmeter. Load $P = 2$ N, holding time 10 s. The calculation of the microhardness was carried out according to the formula

$$H_V = \frac{1.854P}{d^2}$$

where $H_V$ is the Vickers hardness, $P$ is the load (N), and $d$ is the diagonal of the imprint (mm).
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\[ H_V = \frac{kP}{(2a)^2}, \quad (1) \]

where \( P \) is the load on the indenter (kg); \( 2a \) — the average length of both diagonals of the print (\( \mu m \)); \( k \) — coefficient depending on the shape of the indenter, for the Vickers pyramid \( k = 1.854 \).

The coefficient of fracture toughness \( K_{IC} \) which characterizes the crack resistance of the sample was determined by the formula

\[ K_{IC} = 0.016 \left( \frac{l}{a} \right)^{-0.5} \left( \frac{H_V}{E\Phi} \right)^{-0.4} \frac{H_Va^{0.5}}{\Phi} \quad (2) \]

provided

\[ 0.25 \leq \frac{l}{a} \leq 2.5, \quad (3) \]

where \( E \) is Young's modulus (GPa); \( H_V \) — microhardness (GPa); \( \Phi \approx 3 \) is a constant; \( l \) — crack length from the corner of the Vickers pyramid print (m); \( a \) — semi-diagonal of the Vickers pyramid imprint (m).

3. Results

Since microhardness is determined by a number of physical characteristics of a substance (energy of interatomic bonds, covalence level, interatomic distances), the distribution of microhardness values over the sample diameter will characterize the homogeneity of the properties of the consolidated sample, the sintering quality, the uniformity of the density distribution. The latter fact determines the service life of ceramic products. In this connection, a research of the distribution of microhardness over the sample diameter was carried out, the results of which are shown in figure 1. The standard deviation of the microhardness value in diameter was 14%.

![Figure 1. Microhardness distribution \( H_V \) according to the placement of the sample Al\(_2\)O\(_3\)-15 wt.% SiC obtained at a temperature of 1600°C.](image)

Figure 2 shows the dependences of the microhardness \( H_V \) of composites based on Al\(_2\)O\(_3\) on the temperature of consolidation and the percentage of reinforcing nano-additives SiC. Microhardness increases with the addition of SiC, but when a certain value is reached, the concentration of the additive begins to decrease. The sample with the best hardness value was obtained from the composition Al\(_2\)O\(_3\) + 15 wt.% SiC sintered at a temperature \( T_{sint} = 1600^\circ C \) and a sintering time \( \tau = 3 \) min. The reason for the formation of internal defects in pure Al\(_2\)O\(_3\) composite along with the coarse-grained structure can also be the anisotropy of the thermal expansion of the matrix grain [4]. Therefore, the strength of the composite increases with decreasing grain size of the matrix due to the addition of SiC and significantly decreases with increasing sintering temperature due to strong grain growth.

Figure 3 shows the data on the crack resistance \( K_{IC} \) of Al\(_2\)O\(_3\) + \( x \) wt.% SiC samples \( (x = 0, 5, 15, 25, 50) \) sintered at different temperatures. It can be seen that the crack resistance of the samples increases with increasing SiC content (to a value of 15 wt.% ) and then decreases with its content.
The larger grain size helps to improve crack resistance which is explained by the shunting effect in the polycrystalline material [5]. As a result of the addition of a small amount of SiC, the fracture toughness of the samples is improved due to the enhanced effect of SiC — crack formation or crack deflection. As seen in figure 3, the crack resistance of the composite reaches a maximum value when the SiC content is 15 wt.%. At that time, exceeding this concentration leads to a decrease in crack resistance because the grain size becomes too small.

Figure 4 shows the cracks caused by Vickers indentation on the sample surface (a) clearly shows the crack deviation and its zigzag, and with the addition of 10% SiC (b) the cracks do not change their direction and become straight.

The increase or decrease in mechanical properties may be due to residual stresses that arise due to the inconsistency of the coefficient of thermal expansion (CTE) between the Al2O3 matrix and the SiC particles. The average CTE of Al2O3 (\(\alpha \approx 8 \cdot 10^{-6} \text{ K}^{-1}\)) is approximately twice the average for SiC (\(\alpha \approx 4.5 \cdot 10^{-6} \text{ K}^{-1}\)) [6, 7], therefore the Al2O3 matrix is under voltage compression after cooling to room temperature [8]. Obviously, the residual stress increases with increasing SiC content and a higher compressive stress at the grain boundary can lead to a transcrystalline crack. Therefore, the type of destruction varies from a completely intercrystalline fracture (for pure Al2O3) to more transcrystalline destruction (for composites with a high SiC content).

With high-speed machining using tools (cutters) made of ceramic materials and their composites, the temperature in the cutting zone can reach 1100°C [9]. When choosing a material suitable for the manufacture of instrumental products, it is necessary to take into account each of the basic properties (hardness, crack resistance, thermal diffusivity) and to ensure the best combination of them. This goal is difficult to achieve and usually preference is given to one of these properties, which to a large extent affect the performance of this tool. The approach for solving such problems was proposed by Wigley [10], the essence of which is to introduce the parameter \(B\), which contains the key properties necessary for comparative evaluation of various materials.
Figure 4. The surface of Al₂O₃ ceramics samples after indentation: (a) sample of Al₂O₃, $T_{\text{int}} = 1600^\circ$C; (b) sample of Al₂O₃ + 10% SiC, $T_{\text{int}} = 1500^\circ$C; (c) sample of Al₂O₃ + 50% SiC, $T_{\text{int}} = 1600^\circ$C; (d) sample of Al₂O₃ + 80% SiC, $T_{\text{int}} = 1600^\circ$C.

The material for high-speed cutting tools must have high wear resistance and heat resistance, therefore, the thermomechanical parameter $B$ introduced by us is the product of the thermal diffusivity $a$ and the hardness $H_V$. To compare the properties of the composite material obtained in the course of the work, several existing materials used for similar tasks were taken:

- **R18** — high-speed steel is a typical representative of high-alloy steels. A large amount of tungsten contained (18%) and other carbide-forming elements in such steels ensures their high heat resistance. These steels allow you to increase the processing performance by 2-4 times, compared to conventional carbon steels, and retain a hardness of $HRC \geq 60$ to a temperature of 620°C.

- **Instrumental oxide-carbide ceramic material BOK-60.** It is made of Al₂O₃ and TiC by hot pressing. Designed for finishing and semi-turning turning of hardened structural steels with a hardness of $HRC \geq 60$, gray ductile alloyed cast irons with high cutting speeds.

- **HC1** — “White” ceramics based on aluminum oxide production NTK (Japan). Designed for semi-finishing and finishing processing of cast iron at speeds of 500 m/min.
- HC2 — “Black” ceramics based on aluminum oxide with the addition of TiC and increased hardness produced by NTK (Japan). Designed for treating hardened metals at elevated temperatures at speeds of 400 m/min.
- As15-6 — composite material obtained in the course of the work. The basis is microdispersed alumina with the addition of 15 wt.% nano-SiC sintered at $T_{\text{sint}} = 1600^\circ\text{C}$ by the method of electrical consolidation with direct current transmission. Axial pressure $P = 35 \text{ MPa}$, sintering time $\tau = 2 \text{ min}$.

Mechanical and physical properties of these materials which are necessary in a comparative analysis are placed in Table 1.

### Table 1. Mechanical and physical properties of different cutting tool materials

| No | Name     | Comp.                  | $\lambda$, W/(m·K) | $\rho$, g/cm$^3$ | $H_V$, GPa | $C_P$, J/(kg·K) | $\alpha \cdot 10^6$, m$^2$/s | $B \cdot 10^3$, N/s | Source |
|----|----------|------------------------|---------------------|------------------|------------|----------------|-----------------------------|----------------------|--------|
| 1  | R18      | Fe, W, Cr, Al$_2$O$_3$, TiC | 22.1               | 8.80             | 7.6       | 393            | 6.4                         | 48.6                 | [11]   |
| 2  | VOK6     | Al$_2$O$_3$, TiC        | 15.0               | 4.30             | 14.6      | 800            | 4.4                         | 64.2                 | [12]   |
| 3  | HC1      | Al$_2$O$_3$, SiC        | 17.0               | 4.00             | 18.0      | 750            | 5.7                         | 102.6                | [13]   |
| 4  | HC2      | Al$_2$O$_3$, SiC        | 21.0               | 4.30             | 21.0      | 796            | 6.1                         | 128.1                | [13]   |
| 5  | As15-6   | Al$_2$O$_3$, SiC        | 23.0               | 3.87             | 25.0      | 696            | 9.4                         | 216.2                | This work |

Due to use of SiC the thermal conductivity of which is significantly higher than the thermal conductivity of Al$_2$O$_3$ [14], it was possible to increase the thermal conductivity of the composite to $\lambda = 25-30$ W/m·K which is comparable with the thermal conductivity coefficient of the cutters made of high-speed steel R18. It is also worth noting that increasing the sintering temperature to 1700°C leads to the transformation of SiC from 3C- (cubic) to 6H-polytype (hexagonal) [15] the thermal conductivity of which is 2 times higher.

The lowest value of the thermomechanical parameter $B$ is possessed by steel P18. This is due to the low hardness ($H_V = 7.6$ GPa) compared with the hardness of ceramic materials. VOK60 oxide-carbide ceramics have a hardness of 2 times higher ($H_V = 14.6$ GPa) but relatively low thermal diffusivity which ultimately affected the parameter $B$ the value of which is only 33% higher.

Based on these data, it can be seen that ceramics from microdispersed aluminum oxide reinforced with nano-dispersed silicon carbide have a thermomechanical parameter $B = 216.2$ which is 4 times higher than the value of the corresponding parameter for high-speed steel R18 and 2 times for modern ceramics based on Al$_2$O$_3$ global manufacturers.

### 4. Conclusion

Obtaining composite materials with high mechanical properties in the consolidation of powder materials is associated with the solution of a number of difficult problems. On the one hand, an increase in sintering temperature contributes to a more active flow of intergranular diffusion and their sintering, on the other hand, it activates grain growth, which negatively affects the density and hardness of the material. It was shown that it is possible to shorten the consolidation time (and, consequently, the growth of grain) by applying the technology of electrical consolidation with direct current transmission. It was also found that it is possible to increase the temperature of consolidation, while maintaining the grain size, by using nano-dispersed additives. The use of nano-dispersed SiC powders and electro-consolidation technology made it possible to obtain a composite material with a hardness of $H_V = 25$ GPa and a crack resistance of $K_{IC} = 6.5$ MPa·m$^{1/2}$ surpassing foreign analogues in thermo-mechanical properties.
Reference

[1] Griffith A A 1920 *Phil. Trans. R. Soc. London A* **221** 163
[2] Azarenkov M O, Gevorkyan E S, Litovchenko S V, Chiskala V O, Timofeeva L A, Melnyk O M and Gutsalenko Yu G 2012 Pat. Ukraine 72841 Publ 27.08.12 *Bulletin of inventions* 16
[3] Berman R 1976 *Thermal conduction in solids* (Oxford: Clarendon Press) p 193
[4] Evans A G 1978 *Acta Metall.* **26** 1845
[5] Swanson P L, Fairbanks C J, Lawn B R, Yiu W M and Hockey B J 1987 *J. Amer. Ceram. Soc.* **70** 279
[6] Sheludyak Yu E, Kashporov L Ya et al 1992 *Thermophysical properties of components of combustible system* (Moscow: NPO Inform TEI Publ) p 184 (In Russ)
[7] Chirkin V S 1967 *Thermophysical properties of materials of nuclear technology* (Moscow: Atomizdat Publ) p 474 (In Russ)
[8] Luo J and Stevens R 1997 *J. Eur. Ceram. Soc.* **17** 1565
[9] Zhed V P, Borovsky G V, Musicant Ya A and Ippolitov G M 1987 *Cutting tools equipped with superhard ceramic materials and their application: Reference book* (Moscow: Mashinostroenie Publ) p 320 (In Russ)
[10] Wigley D 1971 *Mechanical properties of materials at low temperatures* (New York - london: Plenum Press) p 325
[11] *High speed steel bars and strips. Technical specifications* 1975 Russian State Standard GOST 19265-73 (Moscow: Izdatelstvo standartov Publ) (In Russ)
[12] *Ceramic tool materials. Grades* 1987 Russian State Standard GOST 26630-85 (Moscow: Izdatelstvo standartov Publ) (In Russ)
[13] https://www.ntk-cuttingtools.com/en/products/ceramics/overview-ceramic
[14] Okhotin A S 1979 *Thermal conductivity of solids* (Moscow: Mir Publ) p 286 (In Russ)
[15] Muranaka T, Kikuchi Y, Yoshizawa T, Shirakawa N and Akimitsu J 2008 *Sci. Technol. Adv. Mater.* **9**(4) 044204