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Technological features of receiving and research of high density ceramic materials on the basis of silicon carbide

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Abstract. The article is devoted to the establishment of regularities of synthesis and technological aspects of the formation of composite ceramics based on silicon carbide and aluminum nitride by the method of hot pressing, as well as the study of its structural properties. The paper presents the results of the study of the microstructure and elastic properties of ceramics based on silicon carbide, obtained by hot pressing at temperatures up to 2170 K and pressures up to 35 MPa, of various compositions (0.9SiC - 0.1AlN; 0.7SiC - 0.3AlN ; 0.5SiC - 0.5AlN; 0.3SiC - 0.7AlN; 0.1SiC - 0.9AlN), the average density of which is 3.21 g/cm³. The microstructure of the obtained samples was studied with a scanning electron microscopy. Elastic moduli of SiC-AlN hot-pressed ceramic materials were determined by the method of resonant ultrasonic spectroscopy (RUS) depending on the composition. Our data on Poisson's ratio are in good agreement with the literature data and are well described by linear approximation. The values of Young's modulus are noticeably lower than the literature ones. The values of the elastic moduli lie below the additive right line of ceramic SiC and AlN with zero porosity, which is related to the influence of the porosity of our samples.

The rapid development of basic and applied researches of ceramic materials is based on the needs of all modern industries in brand new materials. It is ceramic materials based on silicon carbide and aluminum nitride that have unique functional and practical design properties (high-wearing feature, chemical resistance, heat resistance, special electrophysical properties, etc.). This determines the most promising market segments, where they gradually penetrate, displacing counterparts made of traditional materials.

The problem of the formation of new functional and structural ceramic materials based on silicon carbide and aluminum nitride with predictable properties highlights the monitoring of their properties.

The traditional method of forming products of silicon carbide powder is the method of hot pressing [1]. Upon obtaining of ceramic carbide silicon materials, the use of various technological methods, which provide for obtaining ceramics of a given composition, structure and set of useful properties, is required. The sintering process of SiC-AlN ceramics by hot pressing was carried out in N₂ medium for 1 hour, at a temperature of less than 2170 K and pressure up to 35 MPa. Another important process operation in the production of ceramics is the uniform distribution of additives (AlN) and the maximum density of the initial raw materials. Samples with the following composition were obtained: 0.9SiC – 0.1AlN; 0.7SiC – 0.3AlN; 0.5 SiC – 0.5 AlN; 0.3SiC – 0.7AlN; 0.1SiC – 0.9AlN, the average density of which was 3.21 g/cm³.

To study the microstructure of ceramics in this work, we used the method of scanning electron microscopy in the SE mode (secondary electron mode). Figures 1 and 2 show the morphology of the



ceramic sample under study at an accelerating voltage of 12.5 and 15 kV. The average grain size according to the data obtained is 2-5 μm . There are also nanograins with sizes less than 100 nm.

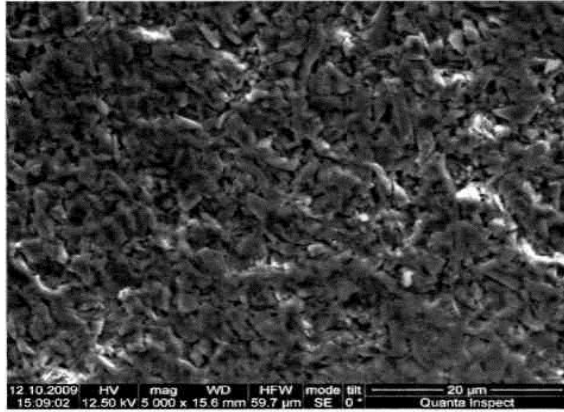


Figure 1. The morphology of the SiC-AlN ceramics sample (30% weight of AlN) at $U = 15$ kV.

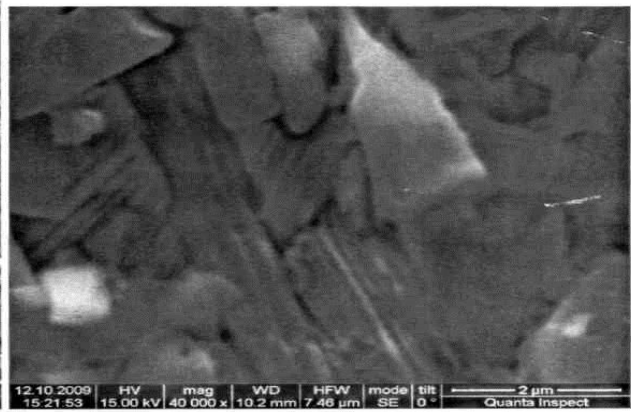


Figure 2. The morphology of the SiC-AlN ceramics sample (30% weight of AlN) at $U = 12.5$ kV.

A qualitative chemical analysis of a SiC-AlN ceramic sample for Si, Al, N, C elements was performed using scanning electron microscopy methods with a sensor resolution of 127 eV. The radiation was carried out at the brightest spectra in the $K\alpha$ mode. The largest grain was selected for analysis (Figure 3).

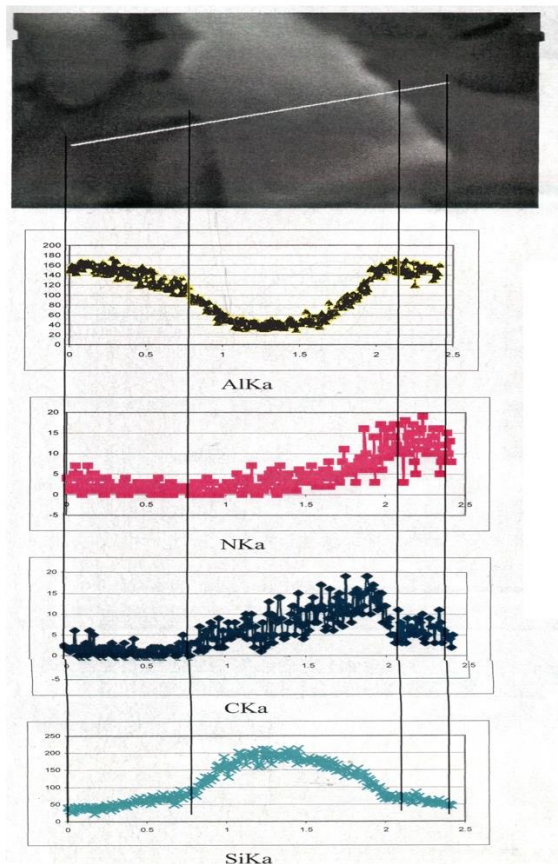


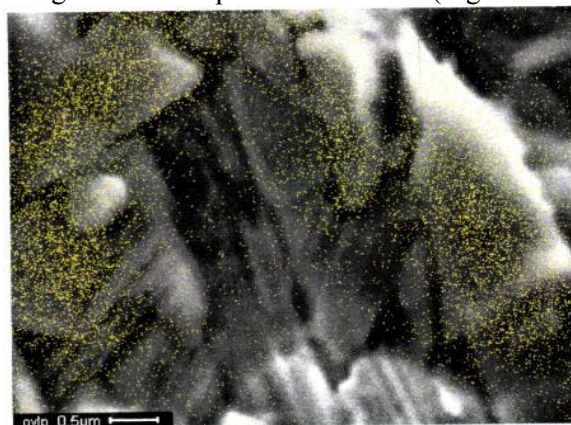
Figure 3. The results of SiC-AlN ceramics elemental analysis (30% weight of AlN).

As is clear from the image presented in Figure 3, the particle under study with a size of ~ 1 μm contains a significant amount of silicon and carbon, and a small amount of aluminum and nitrogen. Along the rim of this grain, an increase in the concentration of aluminum and nitrogen is observed,

which indicates the formation of a solid solution of silicon carbide with aluminum nitride in the boundary layer.

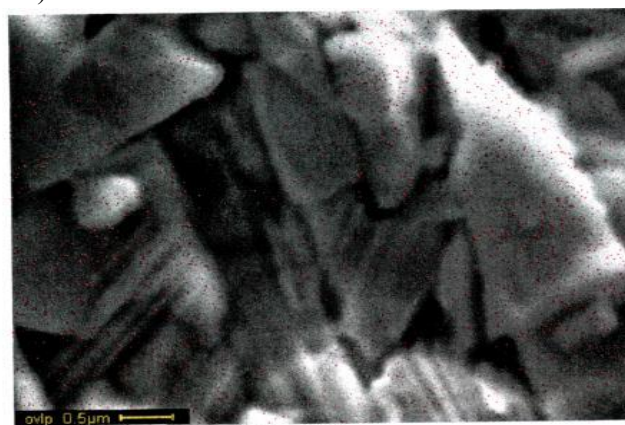
Of special interest is phase formation in hot-pressed silicon carbide. The structure and composition of SiC ceramics is affected by various media and sintering temperatures, as well as the proportion of the content of mineralizers and modifiers.

The results of phase analysis of the SiC-AlN ceramic sample (70% weight of SiC) are given below. Using the SEM compositional contrast ratio method, it is possible to identify phases, grain rims and to determine the nature of the distribution of elements over the grain section, the chemical composition of various inclusions. The observed grain contrast ratio is used to detect various dispersed phases. As a result of the studies, photographs of the morphology of the sample with the distribution of phases throughout the sample were obtained (Figures 4 and 5).



SE+SiKa

Figure 4. The results of SiC-AlN ceramics phase-shift analysis (30% weight of AlN). Phase Si distribution.



SE+CKa

Figure 5. The results of SiC-AlN ceramics phase-shift analysis (30% weight of AlN). Phase C distribution.

The figure shows that carbon is distributed evenly throughout the sample, and silicon is distributed in the form of carbide chains.

Table 1. Elastic properties of SiC-AlN ceramics.

AlN, %	C11	stdC11	C44	stdC44	std E	μ	std μ
10	439.20	6.7	175.30	4.4	409.45	10.2	0.1679
30	434.58	8.2	165.40	4.3	394.57	8.2	0.1928
50	403.18	8.1	148.38	3.1	358.73	9.3	0.2088
70	375.71	7.8	132.09	2.7	324.65	7.8	0.2289
90	334.94	8.5	114.47	2.9	283.98	9.7	0.2404

The elastic moduli of SiC-AlN hot-pressed ceramic materials are determined depending on the composition. The elastic properties of the samples at room temperature were measured by the method of resonant ultrasonic spectroscopy (RUS) on the apparatus described in Ref. 2 [2].

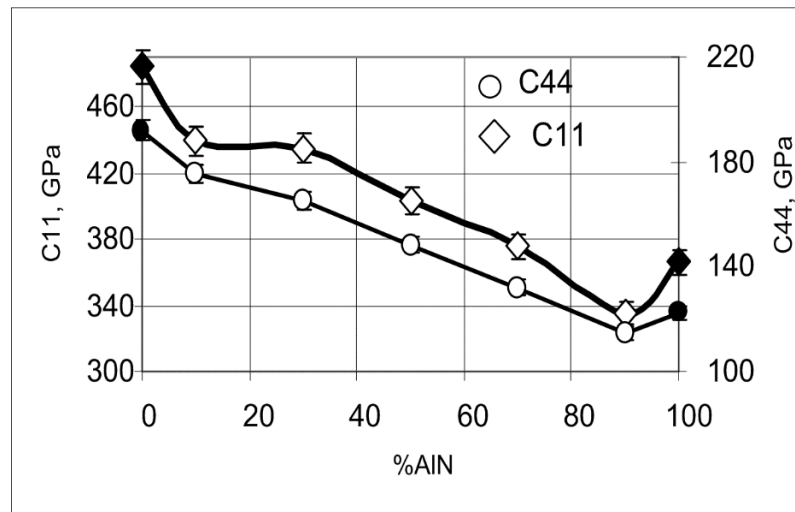


Figure 6. Concentration dependence of C11 and C44 elastic moduli

In our measurements, we used the point method of exciting and recording sample oscillations in the frequency range of 30 ... 1500 kHz. The array of digitized data “frequency-amplitude” was processed on a computer to search for the frequency and quality of the response. The Gauss-Lagrange approximation was used. The study results are presented in Table 1 and in Figures 6 and 7. The total error of measurement of elastic moduli does not exceed 2%.

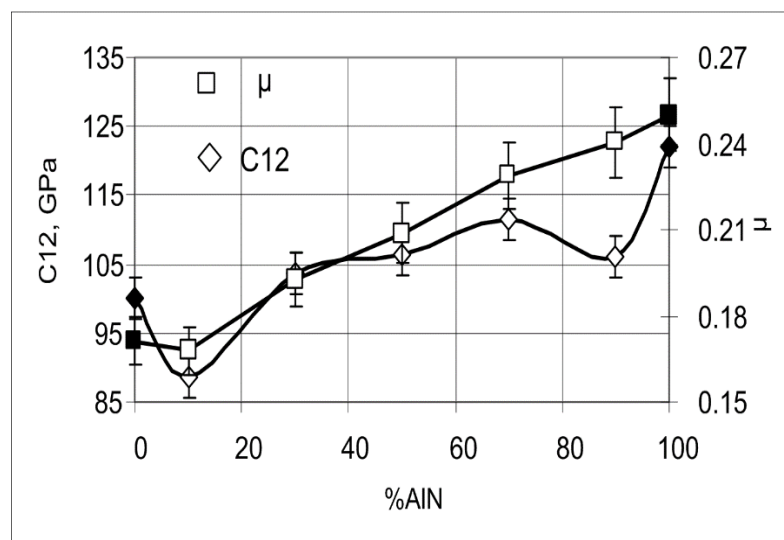


Figure 7. Concentration dependence of C12 elastic modulus and Poisson's ratio

The propagation speeds of Cl and Ct ultrasonic waves are calculated from the C11 and C44 elastic moduli, respectively. Dark icons (Figures 6-8) show literature data on pure ceramic SiC and AlN.

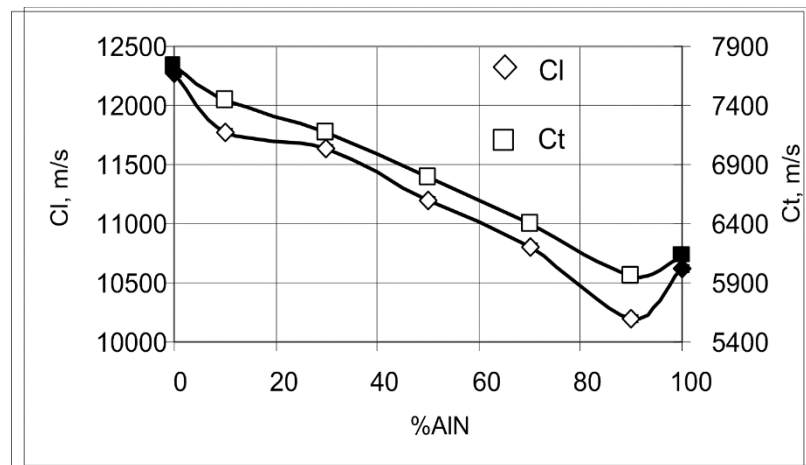


Figure 8. Concentration dependence of the propagation velocity of longitudinal Cl and transverse Ct waves.

Our data on Poisson's ratio are in good agreement with the literature data and are well described by linear approximation. The values of Young's modulus are noticeably lower than the literature ones. The values of the elastic moduli lie below the additive right line of ceramic SiC and AlN with zero porosity, which is related to the influence of the porosity of our samples.

The results obtained make it possible to establish regularities of composite ceramics synthesis based on silicon carbide and aluminum nitride by the method of hot pressing, as well as to optimize the technology of its production.

References

- [1] Kardashova G D 2004 Sintering processes and electrophysical properties of ceramics based on silicon carbide with activating additives (Makhachkala: PhD dissertation)
- [2] Pashuk E G and Khalilov Sh A 2006 *Proc. All-Russian Conf. on Physical Electronics (Makhachkala)* (Makhachkala: Dagestan State University) p 225-228