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Materials Science

High Temperature Multifunctional Heteromodulus Nano Composite for Armor Plates, Turbine Disk and Fan, Refractory and Wear-Resistant Aggregates

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To obtain composite in the Al_2O_3-SiAlON system, at the first stage composite powders were prepared by metal-thermal and reactive sintering process in the nitrogen medium. Obtained powders were milled by ball milling machine and then were hot pressed at 30 MPa and 1600-1650^{0}C. Further studies were performed using micro- and macro mechanical methods as well as structural-optical, electron microscopic methods. The porous phase was studied. The percentage content and dimensions were determined, as well as the percentage content of crystalline phase – SiAlON, aluminum oxide – and grain sizes. Obtained composite is distinguished with improved mechanical properties: $\sigma_{comp.}$-1923 MPa; $\sigma_{bend.}$-470 MPa; HV-19. GPa. Micromechanical analysis showed that no cracks were formed in the SiAlON matrix during the loading process. The obtained materials may be recommended in armor engineering, when measuring temperature in metals molten as protective coatings for the thermocouple, as well as in high-temperature furnace linings, as well as in clean processing operations as a metalworking cutting material, for armor plates, turbine disk and fan. *© 2022 Bull. Georg. Natl. Acad. Sci.*

β-SiALON, reactive sintering, composite, mechanical properties

There are several types of SiAlON: α; β; X; O_1; H; R [1-3]. They can be used in oxidizing environments up to 1300^{0}C and in protective environments up to 1800^{0}C [4, 5]. Of these diverse types, SiAlON exist mainly in the form of three phases: α-β-SiAlON, and also between amorphous or partially crystallized grains. SiAlON of α and β categories are characterized by a unique combination, with higher hardness and also with high strength usually compared to silicon nitride. The α-SiAlON phase is characterized by a higher hardness than the β-SiAlON. β-SiAlON, like usual silicon nitride, is characterized by a higher impact viscosity. Ceramics are generally characterized by high hardness and abrasion resistance, but are crushable; so, our focus was more on the β-SiAlON phase to obtain a composite with relatively high impact viscosity and crack resistance [4, 5].

Table 1. Material composition of CH-8 composite

Material composition of CH-8 composite, wt.%								
Name	Geopolymer		SiAlON Phase					
	Prosyanaya kaolin (Ukraine)	Polog clay	Al	Al_2O_3	Si	Perlite (Armenia)	MgO	Y_2O_3
CH–8	13.9	4.63	23.15	27.78	25.00	2.78	0.92	1.8

Table 1 shows the material composition of the research object.

The chemical composition of Polog clay is as follows (mass %): SiO_2-47.92, Al_2O_3-35.20, Fe2O3-2.06, CaO-0.40, MgO-0.30, heat loss – 12.24, refractoriness 1710–1730^0C.

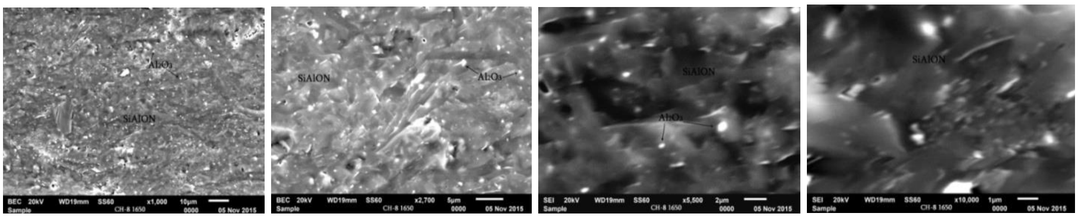
Chemical composition of kaolin (wt.%): SiO_2 – 46.45, TiO_2 – O.33, Al_2O_3 – 38.70, Fe_2O_3 – 0.46, MgO – trace, CaO – 0.36, Na_2O – 0.45, K_2O – 0.60, heat loss – 13.63. refractoriness – 17700C [5-6].

CH-8 composite X-ray showing sialon- like reflexes – dhkl:6,65; 5,45; 3,87; 3,67; 2,520;2,21; the characteristic diffraction peaks of corundum are also observed – dhkl:3,49; 2,52; 2,36; 2,09, which we introduced into burden to reinforce the SiAlON phase. The porosity and density of the samples were determined by the hydrostatic weighing method, while the compressive strength and bending strength were determined on a 2054 p 5 tensile machine.

Table 2. Physical-technical properties of CH-8 composite obtained by hot pressing at 1600^0C and 1650^0C temperatures

Composite name	Apparent porosity, w%	Total porosity, ∏, %	Density ρ, g/cm^3	Compression strength, $\sigma_{comp.}$ MPa	Bending strength, $\sigma_{bend.}$ MPa	Vickers hardness HV,GPa
CH-8 (1600^0C)	0,7	2,49	3,17	1614	456	16
CH-8 (1650^0 C)	0,01	0.13	3,21	1923	470	19

The results presented in Table 2 allow us to say that the samples of the composite of the selected composition obtained by hot pressing at 1650^0C and pressure of 30 MPa are consolidated and the phase composition corresponds to the set goal, which is confirmed by electron-microscopic examination – Fig. 1.



a) b) c) d)

Fig. 1. Electron microscope images of CH 8 composite fracture obtained at 1650^0 C.

As can be seen from Figure 1, the composite consists of two solid phases: the thinnest grains of corundum located in the SiAlON matrix, the porous phase is also represented by a small number of thin pores. From the fracture images it is clear that the material has a decomposing plastic character, a crack is formed in the SiAlON phase and its

Table 3. Content of corundum grains in the composite

Composite CH–8	Phase name	Field of vision S, μM^2	Number of the counted grains, n	The biggest grain Dmax. μM	The smallest grain Dmin. μM	Dmid. of the grains, μM	Phase content, %
1600°C	Al_2O_3	22,22	32	1,00	0,25	0,50	23,1
1650°C	Al_2O_3	30,55	39	1,50	0,20	0,49	23,9
Average		23,88	3,55	1,5	0,20	0.495	23,5

distribution is limited by both the SiAlON and the nano sized grains (400-500 nm) of corundum.

The pores are mostly rounded, the average diameter of the pores is calculated (Table 3). The total volume of the closed pores reaches 1.58% slightly different from the number of pores determined by the hydrostatic method, which should be attributed to the error of porosity measurement by each method. Pores Dmid. by 1600^0C=0.4 μM. The biggest pore Dmax.=2.0 μM The smallest pore Dmin. =0.2 μM and by 1650^0C Pores Dmid. =0.38 μM The biggest pore Dmax.=2.0 μM. The smallest pore Dmin =0.15 μM. Transverse and semi-transverse pores are not observed in the matrix. Based on the morphological images, the distribution of pores in the material is between equal and unequal.

Crystalline Phase Content and Average Sizes

We determined the dimensions and content of corundum grains in different fields of vision and calculated the phase corundum content in the composite (Table 3). The corundum grain content may actually be higher because the resolution of the finer corundum grains is limited. Since the mixture contained perlite, the composite also contained a certain amount of glassy phase. Aragats perlite is a completely glassy mass (96 mass% is glass, the rest plagioclase, pores and volatiles) that melts at 1240°C [6]. Presumably, the added 2.78% perlite in the composition produces eutectic melted with the geopolymer ingredients, especially with the alkaline oxides, and, of course, the glassy phase content (V), in the material increases. We got its content equal to 6.5%. Therefore, the number of crystalline phases must be equal to: 100-(V1+V) = 100-(0.8+6.5) = 92,7%, From here the sialon phase will be 92.7-23.5 = 69.2%. As for the dimensions of the sialon grains, its structure is presented in the form of leaf packets and in the field of visionis seen as a continuousmatrix.

[1]V – volume of the porous phase.

As can be seen from the result, that the content of the crystalline phase is high, the degree of dispersion of the crystals is also high, and the result is achieved by the fact that the phase is evenly distributed in the matrix. The dynamic hardness and elasticity modulus of the obtained composite were determined on the DUH-211S dynamic ultra-micro dynamic hardness tester in accordance with the requirements of the modern ISO-14577 international standard.

The advantage of this method over conventional static measurements or diagonal measurements of indentation is that it contains both plastic and elastic components. The measurement results do not depend on indentation size, loads, and heterogeneity of elastic recovery.

Dynamic hardness had been determined in a loading-unloading mode before elastic relaxation occurred.

Test force 200.00mn, Loading speed 1.0mn/sec, Hold time at load – 5 sec. Hold time at unload – 3 sec. Poisson's ratio – 0.250. Indenter type – Vickers. Indenter elastic – 1.140e + 006 n/mm^2. Indenter Poisson's ratio – 0.070.

Analysis of the indentation of the indenter after the hardness measurement should indentation with the load 200 mn. Penetration depth of the indenter

Table 4. Micromechanical characteristics of of CH-8 composite obtained at the temperature of 1650^0C

SEQ	F_{max}	h_{max}	hp	hr	DHV-1	DHV-2	Eit	Length	HV	Data name
	[mN]	[um]	[um]	[um]			[N/mm2]	[um]		
1	206.11	1.2419	0.4801	0.6795	666.193	4435.000	9.252e+004	4.517	1910.017	CH8-1650(1)
2	210.42	1.3860	0.6371	0.8510	546.009	2550.168	8.182e+004	4.517	1949.885	CH8-1650(2)
3	206.11	1.2701	0.6173	0.7663	636.923	2659.186	9.501e+004	4.517	1910.001	CH8-1650(3)
Average	207.55	1.2993	0.5782	0.7656	616.375	3214.785	8.979e+004	4.517	1923.301	
Std. Dev.	2.484	0.076	0.085	0.086	62.672	1058.143	7007.244	0.000	23.023	
CV	1.197	5.879	14.783	11.204	10.168	32.915	7.804	0.000	1.197	

into the material, h=1.2993 µM. Indentation diagonal length a = 4,517 µM. The crack is not fixed.

Mechanical modulus of materials. For calculating the mechanical modulus of the material, the formula of Kovziridze module was used [6]:

$$M = \frac{Kvol.E.Kic.Pd}{Km.Gvol..Pvol..Pm} MPa / \mu M^2,$$

where Kvol is the volume of the crystalline phase in the material in%; E – elasticity modulus MPa; Kic – critical stress intensity factor; Pd – the pores distribution factor in the matrix, which is taken equal to 1 in the case of even distribution, equal to 0.9 1 in the case of uneven distribution and 0.8 in the case of coalescence of the pores. Km – the middle size of the crystals in the matrix, µM; Gvol – the content of the glassy phase in the matrix, %; Pvol. – pores volume in the matrix, %; Pm – the middle size of the pores in the matrix. Modular dimension is MPa/µM^2. The formula cannot take into account Grifiths cracks [7], dislocations in crystals, nano-defects in glass, but the formula gives us a complete picture of the resistance to external loading of the material, which is close to the calculated values of the bond strength between atoms. This is exactly why the elasticity modulus is included in the formula:

M=92.7x8,987x40.75x0,9/2,5x6.5x0.855x0.4=30184.6/7.6=5.45 GPa/µM^2.

Conclusion

The composite was synthesized in the Al_2O_3-SiAlON system via the method of metal-thermal and reactive sintering in the nitrogen medium. To obtain a dense material, the porous (13–15%) composite was hot pressed at 1650^0C after being grounded in an attritor, and further studies were performed using both micro- and macro mechanical as well as structural-optical, electron microscopic methods. The porous phase was studied, the percentage content and dimensions were determined, as well as the percentage content of crystalline constituents – sialon, aluminum oxide – and grain sizes.

The results of the study showed that ß-SiAlON was obtained with a silicon nitride structure. The obtained material is characterized by high-performance properties. The mechanics are 470 MPa for bending and 1923 MPa for compression. Micromechanical analysis showed that no cracks were formed in the sialon matrix during the loading process.

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მასალათმცოდნეობა

მაღალტემპერატურული მრავალფუნქციური ჰეტერომოდულური ნანო კომპოზიტი ჯავშან ფილებისთვის, ტურბინების დისკებისა და ფრთებისთვის, ცეცხლგამძლე და ცვეთამედეგი აგრეგატებისთვის

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**საქართველოს ტექნიკური უნივერსიტეტი, ბიონანოკერამიკისა და ნანოკომპოზიტების ტექნოლოგიის ინსტიტუტი, თბილისი, საქართველო*

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Al_2O_3-SiAlON კომპოზიტის მისაღებად, პირველ ეტაპზე, მომზადდა კომპოზიციური ფხვნილები მეტალო-თერმული და რეაქციული შეცხობით, აზოტის ატმოსფეროში. მიღებული ფხვნილები დაიფქვა ბურთულებიან წისქვილში და შემდეგ დაიწნეხა ცხლად 30 მპა წნევასა და 1600-1650^{0}C ტემპერატურაზე. განისაზღვრა მიკრო- და მაკრომექანიკური თვისებები, ჩატარდა სტრუქტურულ-ოპტიკური და ელექტრონულ-მიკროსკოპიული კვლევები. შესწავლილია ფორიანი ფაზა. განისაზღვრა ფორების ზომები და პროცენტული შემცველობა, განისაზღვრა ასევე კრისტალური ფაზების – SiAlON და Al_2O_3 პროცენტული შემცველობა და ზომები. მიღებული კომპოზიტი გამოირჩევა გაუმჯობესებული მექანიკური თვისებებით: $\sigma_{\mathrm{Comp.}}$ – 1923 მპა; $\sigma_{\mathrm{bend.}}$ – 470 მპა; HV-19. GPa. მიკრომექანიკურმა ანალიზმა აჩვენა, რომ დატვირთვისას SiAlON მატრიცაში ბზარები არ წარმოიქმნება. რეკომენდებულია მიღებული მასალების გამოყენება საჯავშნე მასალებად, ნალღობებში ტემპერატურის გასაზომად (თერმოწყვილის პერანგი), ასევე მაღალტემპერატურული ღუმელის ამონაგისთვის, ლითონების საჭრელ და დასამუშავებელ მასალებში, ტურბინების დისკებისა და ფრთების მასალებში.

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