Smart Materials in the SiAlON-SiC-Al_2O_3 System

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Introduction

Our work pursued to obtain SiALON-containing composites by reactive sintering, on the basis of silicium carbide and corundum. This method enables us to obtain desirable phase composition material and such materials possess properties of the hot-pressed objects. In the process of obtaining SiALONs by reaction sintering, sintering temperature is sharply decreased at the application of active materials, In our case we had to select materials which would enable us to use newly formed components obtained by the method of reactive sintering, since at the obtaining of solid solution of SiALON inculcation of a-Al_2O_3 and AlN in β-Si3N4-Si is especially simplified when its crystalline lattice is still in the process of formation. Therefore we gave preference to silica-alumina material –kaolin, aluminium powder and elemental silicium.

Aim

Purpose-in the present paper SiALON-containing nano-composite was obtained through alum-thermal and nitrogen process on the base of geopolymer (Ukraine), aluminum nano-powder, silicium, aluminum oxide, silicon carbide with little admixes of yttrium oxide, magnesium oxide and glass perlite (Aragac, Armenia).

Materials and Methods

Methods-composite was obtained in the nitrogen medium, by the reactive baking method at 1450°C. The advantage of this method is that compounds, which are newly formed thanks to interaction going on at thermal treatment: Si_3N_4, Si, AlN reactive, which contributes to SiALON formation at relatively low temperature, at 1300-1350°C. It is evident that inculcation of α-Al_2O_3 and AlN in crystal skeleton of ß-Si3N4 is easier since at this temperature interval crystal skeleton of Si_3N_4 is still in the process of formation. It should also be stated that strength and wear resistance of SiALONs increase in their presence in silicium carbamide- and corundum -containing composites. The paper offers processes of formation of SiC-SiAlON and Al_2O_3-SiAlON and ß-SiALON composites and describes their physical and technical properties.

Geopolymer, aluminium powder, silicium, silicium carbide, aluminium oxide were used as starting materials, and manganese and yttrium oxides, pearlite and refractory clay were used as additives. Blend compositions are given in Table 1.

Specimens were made of cylindrical form, size d-15 mm, by semi-dry molding method, molding pressure was 20 MPa. After drying it was sintered in a furnace, by one hour standing at the final temperature. Device for sintering for the specimens consisted of a furnace equipped with silicium carbide heaters, mark TK 30/200.

Results & Discussion

Results-open porosity of the obtained materials equaled to 15-16 %. Materials consisted of only SiALONs. To receive compact materials the composites were grinded in planetary mill for eight hours, then they was cleaned from admixtures and the obtained powder was hotly pressed at 1750°C under 25 MPa. Standing time at final temperature equaled to seven min. The results of samples testing: Density, g/cm3=3.24; Thermal expansion coefficient, 1/grad 10-6(800)=2.7-3.0; Hardness, HRA=94, HV=18 GPa; Flexural Strength, 500-550 MPa. Phase composition of the composites was studied by X-ray diffraction method, while the structure was studied by the use of optic and electron microscope. Conclusion-Obtained materials are used in protecting jackets of thermo couples used for melted metal temperature measuring (18-20 measuring) and for constructions used for placing objects in factory furnaces, and for cutting ceramics.

Key words: syalon (SiALON), hot pressing, nano-composite melted metal temperature measuring

We have studied physical-chemical properties of specimens sintered at 1500°C (Table 2). As is seen from the Table 2, C7, then C6 and C8 are distinguished by high physical-technical properties. Open porosity, correspondingly equals to 15,2, 15,0 and 15,4 % . Hardness limit at compaction is 258, 256 and 254 MPa, which refers to the fact that 1500°C is not enough for complete hardening. Despite this, chemical resistance to water and acid (H_2SO_4 ρ-1.84) is still high. Refractoriness of specimens equals to 1770°C.

To investigate physical-chemical processes taking place at SiALONs obtaining the specimens were sintered in 800-1500 °C temperature interval and the investigation was carried out by X-Ray diffraction analysis. X-Ray patterns are given on Figure1.

X-Ray diffraction analysis of sintered SN-1 specimens was carried out at 800-1500 °C by 100°C interval.
X-Ray pattern at 800 °C shows clearly cut diffraction maximums characteristic to aluminium, silicium and quartz. At 900-1000°C temperature interval peaks characteristic to aluminium and SiO_2 are sharply decreased, new phases are formed as a result of interaction of ALN and ALON nitrogen and aluminium.

At 1100-1200°C interval intensity of aluminium diminishes and intensity of ALN and ALON increases. Intensity of SiO_2. sharply decreases. At 1200°C there are no peaks of Si and SiO_2 . Peaks characteristic to mullite appear.

At 1300-1400°C (Fig. 2) the main phase is mullite. Quartz is presented in the form of ALN and ALON and peak characteristic to Si3N4 is not observed. The same picture is seen at 1500°C, which refers to the fact that at 1300°C X- SiALON of mullite structure was formed. In the X-Ray patterns of SN-2 composition (Table 1) in the specimens sintered up to 800-1500°C at 100°C interval, silicium carbide that was introduced into blend remains unchanged at all temperatures. As seen from the SN-1 specimens, here again as at 800-900°C (Fig.3) there are diffraction maximums characteristic to aluminium, silicium and SiO_2 .

At 1000-1100°C new phases were formed: ALN and ALON, while at 1100°C - mullite. At all other temperatures phase formation proceeded by the scheme similar to that of SN-1, but with less intensity, depending on the composition. Thus silicium carbide composite with X-SiAlON binder is obtained (Fig.4). Similar to SN-1 and SN-2 chemical processes in SN-3 composition specimens (Table 2) proceeds by the same scheme (Fig.5-6). a- AL_2O_3 remains unchanged to the end and the composite corundum with X-SiALON binder is obtained.

On the grounds of the obtained results, to obtain SiC and a-AL_2O_3-containing SiALON composites, we introduced into charge composition, metallic silicium, silicium carbide in smaller amount and a-AL_2O_3 (Table 1: SN-6, SN07, SN-8). X-Ray patterns of three composites (SN-6, SN-7, SN-8) sintered at1500°C- are offered on Figures 7,8, 9 . SN-6 composite mainly consists of ß-SiALON . It showed diffraction maximums characteristic to carbide and corundum.

The main phase at the SN-7 X-ray pattern is ß-SiALON. The composite alongside with ß-SiALON consists of silicium karbode; it contains X-SiALON in trace quantity.

SN-8 composite, similar to the above referred composites consists of ß –SiAlON and the introduced a-AL_2O_3.
Results of microstructure analysis (Fig.10) confirm the data of X-Ray structural studies. Microstructure of SN-1 composite is presented mainly by X-SiALON phase, in which particles of Si_3N_4 are inserted as inclusions. Lattice of SN-6 composite is ß –SiALON with silicium carbide and corundum grains spread in it. SN-7 composite lattice is analogous to that of SN-6. in which silicium carbide grains of the size exceeding that of new-formed silicium nitride are clearly visualized. Lattice of SN-8 composite contains of the very ß –SiALON with a-AL_2O_3 crystals. This Figure shows pores which are presented in quantity in this composite.

Conclusion:

At sintering of kaolin and aluminium powder blend in 800-1500°C interval temperatures of formation of aluminium and silicium nitrides and on their base mullite structure X-SiALONs was fixed and proved. While at sintering of SiC-aluminium powder, silicium and a-AL_2O_3 - aluminium powder – silicium blend the SiC-SiAlON and AL_2O_3-SiAlON composites on ß-SiALON lattice were obtained. The obtained results are confirmed by X-Ray diffraction and microscopic analyses.

Table 1.

Composite index.	Initial compound composition , mass.%								
	kaolin	Al	Al_2O_3	SiC	Si	Perlite, Armenia	Y_2O_3	MgO	Refractory clay, Ukraine
SN-1	80.00	20.00							
SN-2	20.00	10.00		70.00					
SN-3	20.00	10.00	70.00						
SN-6	18.52	18.52	18.52	18.52	20.37	2.78	1.85	0.92	
SN-7	13.89	23.15		27.78	25.00	2.78	1.85	0.92	4.63
SN-8	13.89	23.15	27.78		25.00	2.78	1.85	0.92	4.63

Table 2.

Composite index	Open porosity π,%	Hardness limit at compaction, MPa	Density, ρ, g/cm³	Chemical resistance, %	
SN-1	16.2	230	2.26	Water	Sulfuric acid, ρ 1.84
SN-2	15.0	245	2.8	99.41	99.16
SN-3	16.0	240	3.2	99.36	99.15
SN-6	15.0	256	2.25	99.82	99.20
SN-7	15.2	258	2.31	99.79	99.25
SN-8	15.4	254	2.78	99.80	99.30

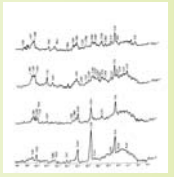


Fig.1. X-Ray patterns of SN-1 composite (800-1100°C)


Fig. 2. SN-1 composite X-RAY (1200-1500°C)


Fig. 3. SN-2 composite X-RAY (800-1100°C)

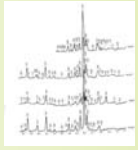


Fig. 4. SN-2 composite X-RAY (1200-1500°C)


Fig.5. SN-3 composite X-Ray (800-1100°C)

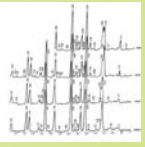


Fig.6. SN-3 composite X-Ray (1200-1500°C)

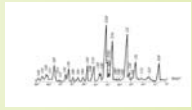


Fig. 7. SN-6 composite X-RAY (1500°C)

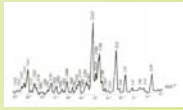


Fig.8. SN-7 composite X-Ray (1500°C)

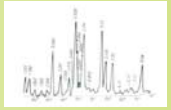
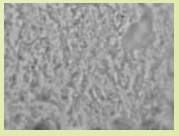
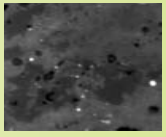
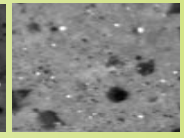


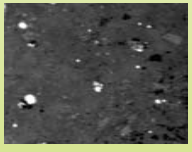
Fig.9. SN-8 composite X-Ray (1500°C)

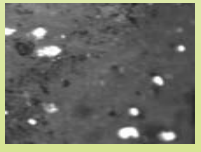

SN-1 X 200

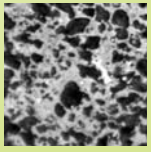

SN-1 X200


SN-6 X200


SN-6 X500


SN-7 X 200


SN-7 X 500


SN-8 X200

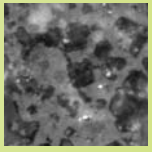

SN-8 X500

Fig. 10. Microstructures of composites