

INFLUENCE OF HYDROGEN-THERMAL TREATMENT OF NITRIDE POWDERS ON THE PROPERTIES OF COMPOSITES ON THEIR BASE

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Introduction

Method of hydrogen-thermal treatment (HTT) of nonmetallic nitrides powders (BN, AlN, Si₃N₄) has been developed at the IPMS. HTT purifies powders from admixtures, allows both to decrease temperature, time of sintering and to obtain sintered specimens from these powders with higher density, strength and specific electric resistance [1, 2].

Results and discussion

The aim of the work is investigation of influence of HTT of non-metallic refractory compounds on the properties of composites based on these powders. Thermal treatment of powders at 1000-1200 °C in hydrogen with the use of catalyst promotes the purification from oxygen, changing of their granulometric composition due to destruction of filmlike connections among particles in aggregates and destruction of these aggregates.

Sintered powder specimens after HTT have smaller average grain size, higher content of anisometric grains and contain high-density formations from dispersed grains.

To investigate the influence of HTT on composites properties in α -Si₃N₄-AlN, AlN-BN and BN-B₄C systems green-body mixtures and mixtures of treated powders have been prepared. Above mentioned mixtures have been treated with hydrogen at 1100 °C during 30 min. Hydrogen has been obtained by decomposition of TiH₂. Obtained powder mixtures have been pressed and sintered in nitrogen at 1550-1900 °C.

Pressed α -Si₃N₄-AlN powders have been sintered at 1550 °C. Al powder as an addition to α -Si₃N₄ was expected to be nitrided during the sintering and form AlN phase. Phase composition of specimens and their strength properties are shown in table 1.

It was shown by X-Ray analysis that in specimens obtained from HTT treated powders Si₂ON admixture disappears and lines of Si₃N₄ and AlN became clearer. It evidences the ordering of crystal structures. Herein strength properties of specimens increase. Electric resistance of all the specimens exceeds 10 Ohm·cm (20 °C).

Pressed powders of aluminium and boron nitrides were sintered at 1900 °C. Properties of the specimens are shown in table 2. As follows from the data sintered specimens obtained from treated powders have higher density, compression strength, heat resistance and electric resistance at heating. Coefficient of thermal expansion (CTE) and phase composition do not change. In initial specimens at small angles besides of two main phases there is a small amount of α -Al₂O₃ admixtures which does not disappear after HTT, while the lines of main phases became clearer.

BN-B₄C specimens were sintered at 1900 °C. Sintered specimens data are shown in table 3. It is evident from table 3, that due to HTT of green body powders the electric resistances at heating and compression strength of sintered specimens are higher. Herein coefficient of thermal expansion decreases a little. There are BN, B₄C and B₂O₃ phases in initial specimens and in treated specimens only two phases left as there is no B₂O₃.

Conclusions

Preliminary data of influence of HTT of mixtures of initial powders on α -Si₃N₄-AlN, AlN-BN and BN-B₄C composites properties have been obtained.

It was determined that HTT of initial powders increases the density of sintered specimens their strength, electric resistance at heating, heat resistance, puts crystal structure in order, reduces quantity of oxygen-containing admixtures.

Table 1 – Properties of AlN-Si₃N₄ system materials

Treatment	Addition to α -Si ₃ N ₄ , mas. %, (init. mixture)	Property			
		Density, g/cm ³	Compression strength $\sigma_{\text{compr.}}$, Mpa	Thermal shocks (1800-20 °C, air)	Phase composition
Untreated	20 AlN	2,58	158	39	α -Si ₃ N ₄ , AlN, β -Si ₃ N ₄
	20 Al (powder)	2,62	167	45	α -Si ₃ N ₄ , AlN, β -Si ₃ N ₄ , Si ₂ ON
HTT treated	20 AlN	2,76	169	43	α -Si ₃ N ₄ , AlN, β -Si ₃ N ₄
	20 Al (powder)	2,80	171	49	α -Si ₃ N ₄ , AlN, β -Si ₃ N ₄

Table 2 – Properties of AlN-BN system materials (mass composition 1:1)

Treatment	Property				
	Density, g/cm ³	Compression strength $\sigma_{\text{compr.}}$, MPa	Thermal shocks (1800-20 °C, air)	Specific electric resistance, Ohm-cm (t, °C)	Phase composition
Untreated	2,17	90,1	88	10 ¹² (20); 2·10 ⁹ (500); 5·10 ⁶ (1000)	AlN, BN, α -Al ₂ O ₃ (traces)
HTT treated	2,35	102,3	105	10 ¹² (20); 1,8·10 ⁹ (500); 6,2·10 ⁷ (1000)	AlN, BN, α -Al ₂ O ₃ (traces)

Table 3 – Properties of BN-B₄C system materials (mass composition 1:1)

Treatment	Property			
	Density, g/cm ³	Compression strength $\sigma_{\text{compr.}}$, MPa	Specific electric resistance, Ohm-cm (t, °C)	Phase composition
Untreated	1,9	15,9	10 ¹³ (20); 1,5·10 ⁷ (1000); 2·10 ⁴ (1500)	BN, B ₄ C, B ₂ O ₃
HTT treated	2,1	29,8	10 ¹³ (20); 1,6·10 ⁹ (1000); 1·10 ⁶ (1500)	BN, B ₄ C

References

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