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Plasma Processed Nanosized-Powders of Refractory Compounds for Obtaining Fine-Grained Advanced Ceramics

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Abstract. One of the ways for the production of ceramic materials with a fine-grained structure is the use of nanopowders. Different methods are used for the production of nanopowders. One of them is the method of plasmachemical synthesis. Different nanopowders of refractory materials can be obtained by this method. The preparation of nanosized powders of nitrides and oxides and their composites by the method of plasmachemical synthesis, the possibilities to receive nanopowders with different particle size and the potential advantages of nanopowders were investigated.

1. Introduction

Application of nanopowders allows producing ceramic materials with a fine structure and in many cases with properties differing from the classical materials. Many methods of nanopowders' production are known including sol-gel synthesis, spray pyrolysis, hydrothermal method, laser synthesis, mechanochemical method, gas phase condensation and the method of plasmachemical synthesis among them [1,2]. It is possible to prepare many nanopowders of refractory compounds using the plasmachemical method. One of the advantages of this method is the resource of production of different non-oxide compounds and their composites, not always possible by other methods.

Results on production of nanosized powders of nitrides, oxides, carbonitrides and their compositions by the plasmachemical method are summarized in this study.

2. Experimental

The nanosized powders of refractory compounds and their composites are prepared by evaporation of coarse commercially available powders of chemical elements and their compounds and subsequent condensation of products into a radio frequency inductively coupled nitrogen plasma (ICP). The experimental apparatus (Fig. 1) consists of a radio-frequency (5,28 MHz) oscillator with a maximum power of 100 kW, a quartz discharge tube with an induction coil, raw powder and gas supply systems, water cooled stainless steel reactor and heat exchanger, and cloth filter for collecting powder. The flow rate of the plasma-forming gas (nitrogen) is 7,2-8,0 m^3/h, the feed rate of raw powders is 0,6-1,8 kg/h.

The calculated fractions of raw powders of chemical elements, as well as some oxides are premixed and introduced into plasma tail through 4 or 8 tubes by carrier gas. Conditions of injection and particle size are determined by theoretical calculations and preliminary experiments. The complete evaporation of raw powders can be reached by varying the particle size and their injection rate, feeding rate, plasma velocity and temperature [3]. The formation of products, their particle size, chemical and phase composition are controlled by introduction of cold nitrogen, ammonia and hydrocarbon into the reaction chamber.

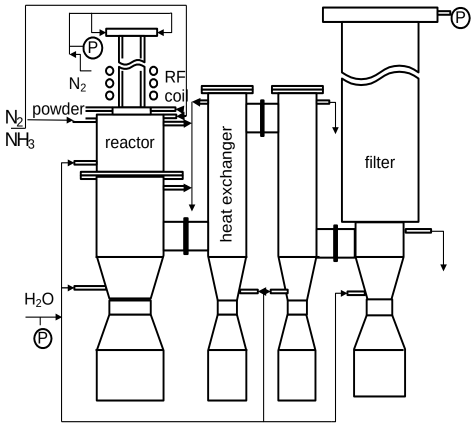

Fig. 1. Schematic view of the plasma apparatus.

3. Results and discussion

A lot of refractory compounds nanopowders and their compositions were produced by the method of plasmachemical synthesis (Table 1). This method of nanopowders production is suitable for obtaining of compounds with the wide scope of chemical composition, for example, TiN…$TiN_{0,3}C_{0,7}$, NbN…$NbN_{0,1}C_{0,9}$, Si_3N_4…10 wt. % Si_3N_4 – 90 wt. % SiC, Si_3N_4 with changeable amount and composition of oxides as sintering dopants, sialons of changeable composition, etc. as well as with the changeable grading composition.

Table 1. The list of some compounds produced by the method of plasmachemical synthesis

Compounds	Type of compounds	Examples of phase composition	SSA, m^2/g	d_{50}, nm
Powders of individual compounds	Nitrides	AlN, Si_3N_4, TiN	20-90	20-50
	Carbonitrides	TiC_xN_{1-x} $Cr_3C_{1,6}N_{0,4}$	20-50 12-25	20-40 35-60
	Oxides	Al_2O_3, ZrO_2, Y_2O_3, MgO, $MgAl_2O_4$ $NiFe_2O_4$, $CoFe_2O_4$	15-50 20-30 25-30	40-70 50-80 35-40
Composite powders	Nitride composites	Si_3N_4-TiN Si_3N_4-AlN, AlN-TiN AlN-Cr,Mo	70-100 50-80 20-40	20-30 20-40 30-50
	Nitride-carbide composites	Si_3N_4-SiC	30-60	30-60
	Nitride-oxide composites	Si_3N_4-Y_2O_3, Si_3N_4-Al_2O_3-Y_2O_3, α- and β- sialons, AlN-Y_2O_3	80-100 70-120 25-35	15-30 15-25 30-60
	Oxide composites	ZrO_2-Y_2O_3, Al_2O_3-ZrO_2, ZrO_2-Y_2O_3-NiO	20-40	25-55
	Cermets	TiN-Ni, TiC_xN_{1-x}-Fe,Ni	15-40	40-80

Obtained products consist of particles of differing shape (Fig. 2): spherical (for example, Al_2O_3, ZrO_2, Y_2O_3, $MgAl_2O_4$), irregular (Si_3N_4) shape or small monocrystals with definite shape (for example, AlN, SiC, TiN, NbC_xN_{1-x}). Many nanocompositions are obtained as coated particles (for example, TiN coated with Si_3N_4, AlN (sample No 6, Fig. 2) or Ni) or whiskers (for example, AlN-Cr,Mo).

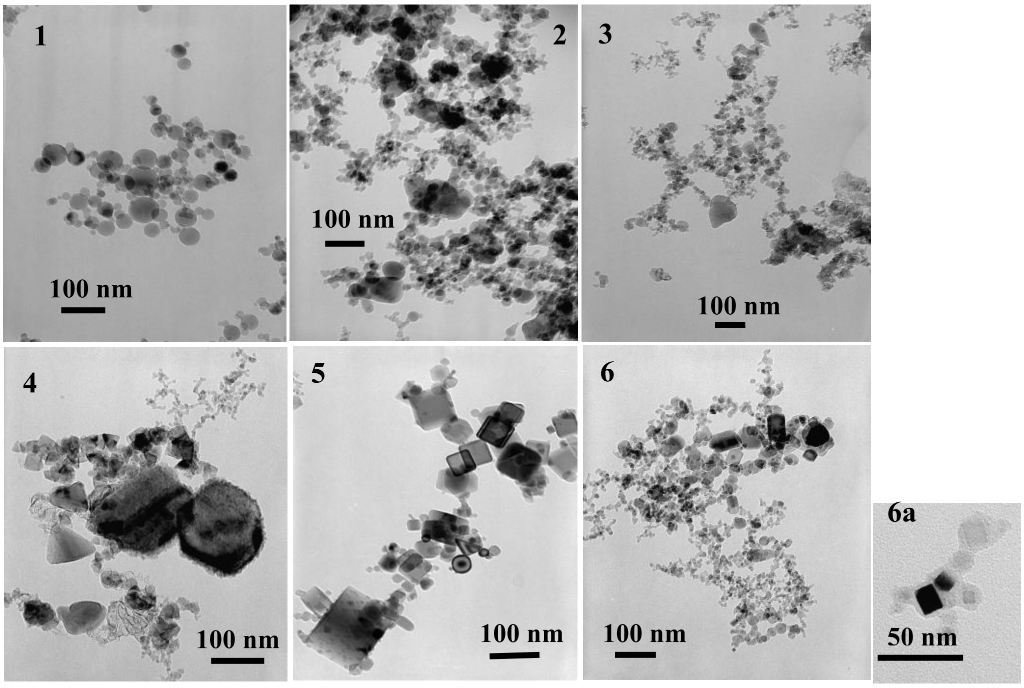

Fig. 2. Characteristic shape of particles of nanopowders: 1 – Al_2O_3; 2 – Si_3N_4-6Y_2O_3-3Al_2O_3; 3 – β-sialon; 4 – Si_3N_4-80 wt.% SiC; 5 – TiN; 6 – Si_3N_4-TiN.

The particle size distribution of plasma-produced powders is wide (Fig. 3) and this fact can have an effect on formation of homogeneous and fine grained material structure. The specific features of plasma process do not allow narrow down the particle size distribution. It was found (Table 1 and Fig. 3) that agglomerates are of medium size and can be enough easy destroyed by pressing of powder [4].

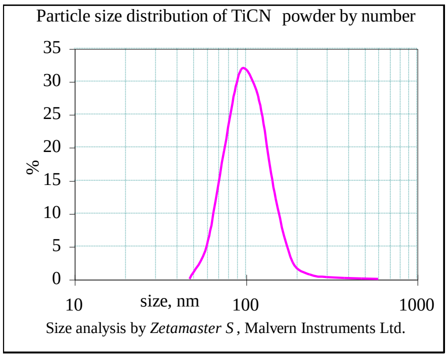

Fig. 3. Particle size distribution of Ti(C,N) nanopowder.

Produced nanopowders possess high chemical activity. Decreasing the particle size reduces their oxidation temperature and stability in air, acids and alkalis, but high dispersity, activity and homogeneity of the composite powders promote their sintering at lower temperature (Fig. 4).

The second and maybe the main advantage of nanopowders application is the possibility to obtain material with more fine-grained microstructure. For example, when using directly in plasma synthesized composite of Si_3N_4 with sintering additives (nanocomposite Si_3N_4-Al_2O_3-Y_2O_3), it is possible to obtain material with a homogeneous fine structure (Fig. 5) and good mechanic and anticorrosion properties with a low friction coefficient and wear rate [5]. Ceramic ball-bearings for acting in extreme conditions without lubricant have been produced from such a material.

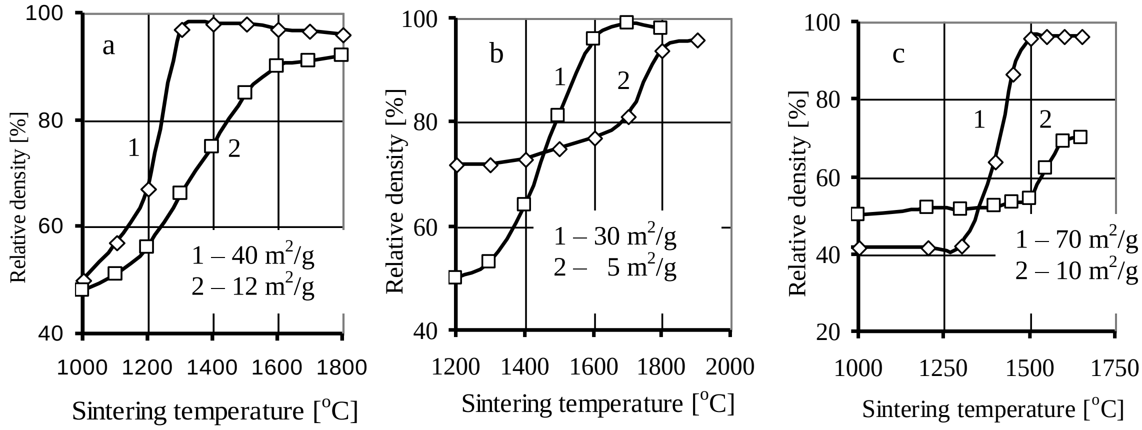

Fig. 4. Effect of sintering temperature and dispersity of powders on relative density of TiN (a), AlN (b) and α- SiAlON (c). 1 – nanopowders, 2 – coarse powders.

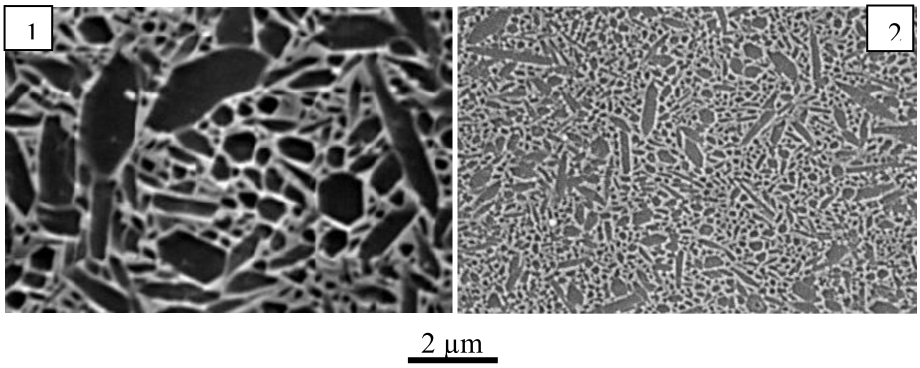

Fig. 5. Typical microstructure of Si_3N_4-6wt.%Y_2O_3-3wt.%Al_2O_3 ceramics obtained from commercial Si_3N_4 (1) and nanosized (2) Si_3N_4-Y_2O_3-Al_2O_3 composition.

Summary

The plasma technique allows producing of several nanosize compounds (oxides, nitrides, carbides and carbonitrides) and their composite powders important for development of new materials and obtaining of homogeneous mixtures of the refractory compounds with sintering aids. The shape and morphology of nanosize particles depends on the thermodynamic characteristics of the components and their ratio. The main advantage of nanopowders application is the possibility to decrease the sintering temperature of ceramic materials and to obtain structure with more fine grains. Use of nanocomposites with Si_3N_4 results in a material with high strength and low friction coefficient, for application in ceramic bearings acting in extreme conditions without lubricants.

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