NANOPOROUS PRODUCTS FROM PURE OXIDES

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Abstract

Nanoporous ceramic products from pure aluminum oxide were fabricated by plasma spraying method. The deposition process consists of passing of sprayed material particles with high velocity through plasma flow and subsequent coating of these particles on the surface of rotating metal forming mandrel. The products manufactured by standard techniques of plasma spraying have low thermal conductivity and selectively high gas permeability. The material of these products has a heterogeneous phase composition which is formed during the high speed cycle of heating and cooling when aluminum oxide particles passing through a stream of air plasma. Aluminum oxide particles in the composition of the material of products are in the alpha state and also in the beta- and gamma states. Reduction in porosity of products to nanoscale values (100-250 nm) and translation of material of plasma sprayed oxide product into single-phase alpha-state leads to a sharp increase in its thermal conductivity. By varying the sintering modes it is possible to fabricate nanoporous products with desired values of thermal conductivity. Products from this material may be efficiently used in special electrometallurgy as containers for growing single crystals, in chemical industry, in mechanical engineering, instrumentation and special equipment, as well as in medicine and biotechnology.

Keywords: Nanoporous ceramics, aluminum oxide, plasma spraying

1. INTRODUCTION

Ceramic products, manufactured by plasma spraying from pure oxides, are used in electrometallurgy, medicine, chemical and electronic industry, special machinery, etc. Such products possess high chemical inertness, heat resistance, geometric dimensions precision and have no warping at high temperatures. Due to these properties ceramic products are widely used as containers and crucibles for growing single crystals from various metals and alloys, including chemically active [1-5]. One of the most important advantages of the plasma spray method of ceramic products manufacturing is the possibility to regulate the values of thermal conductivity in a wide range.

The present research aimed at solving the problem of thermal conductivity regulation by creating of specified porosity value in plasma sprayed products from aluminum oxide.

2. MATERIALS AND METHODS

The commercially available alumina powder (>99.5 wt. % Al_2O_3) with an average particle size of 32 μm was used as feedstock material. Optical image of raw powder is presented in **Figure 1**. Products manufactured by spraying methods in most cases represent axially symmetrical bodies (tubes, crucibles, etc.). The spraying deposition process consists of passing of sprayed material particles with high velocity through plasma flow and subsequent coating of these particles on the surface of rotating metal forming mandrel. For manufacturing of ceramic containers was used plasma spraying setup UPN-350 (Russia). Steel mandrel used as a forming surface. Design of the plasma jet implemented a scheme with a rotating anode. Compressed air used for the plasma gas. Spraying scheme is shown in **Figure 2**.

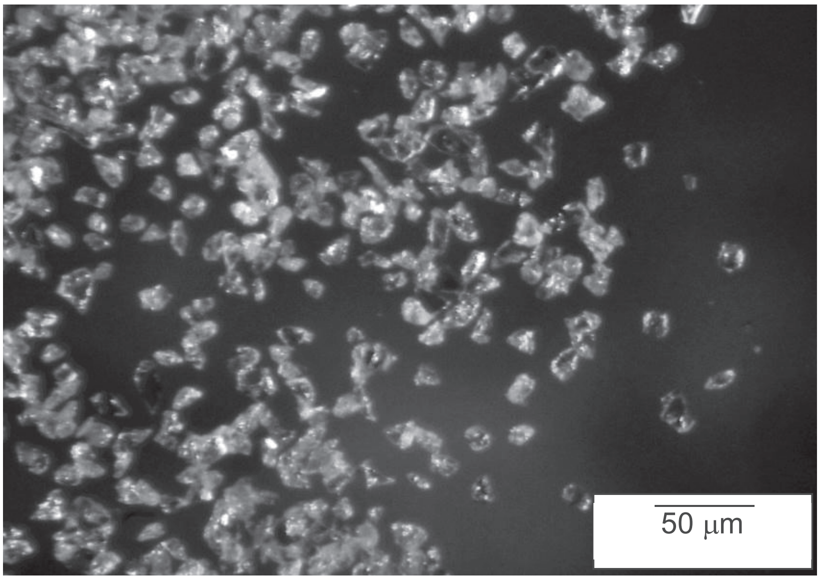

Figure 1 Optical image of alumina powder used for plasma spray deposition (×200)

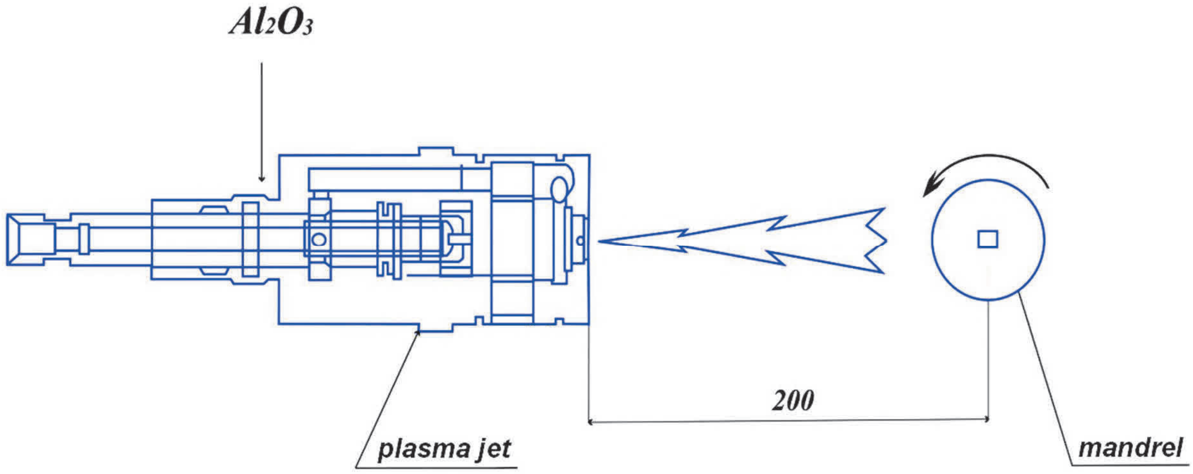

Figure 2 The scheme of spray deposition

Deposited preform was sintered in chamber resistance furnace "Nabertherm" (Germany) with changing of sintering temperature within 1200-1400 °C on the set program. Phase composition of raw material and finished product was determined using X-ray diffractometer D8 Advance (Bruker AXS) and ICDD PDF-2 crystallographic database. Porosity of deposited products was evaluated by the method of X-ray computed tomography using inspection system GE Phoenix Nanomex. The microstructure of the material was observed using ST-60 optical microscope.

Density of material was determined by hydrostatic weighing with calculation the total volume occupied by pores in the investigated sample. Thermal conductivity was evaluated at steady-state one-dimensional thermal field in the flat sample and at temperature on the hot side of the sample from 400 to 1350 °C. For determining thermal conductivity was used special experimental set-up, consisting of the working chamber, temperature and water flow control systems, measuring devices. Summary of test method is measurement of heat flow by the water flow calorimeter.

3. RESULTS AND DISCUSSION

The appearance of plasma spray deposited products is shown in **Figure 3**. Geometric dimensions of products precisely reproduced the dimensions of master-model (mandrel). Heating-cooling cycles in the temperature range from 20 to 1400 °C did not lead to warping of the products and did not change their geometric dimensions.

Figure 3 Plasma sprayed products from aluminum oxide

It was found that the entire source sprayed material consisted of the α-modification of chemical compound Al_2O_3. Passing through the plasma flow has changed the phase composition of the material. Immediately after spraying the material was represented by a set of structural modifications of aluminum oxide: α-modification - 8.0 ± 0.1 %, δ-modification - 28.7 ± 0.3 %, γ-modification - 63.3 ± 0.4 %. Obtained product was porous; the volume occupied by pores in the sprayed product is 18.9% of the total volume of the product. Pores are uniformly distributed in the volume of the ceramic product. Pore size reached values over 500 nm. Subsequent heat treatment resulted in decrease in pore sizes to nanoscale values (100 - 250 nm).

Figure 4 shows the X-ray tomographic sectional image of plasma spray deposited product from aluminum oxide after sintering. The porosity in this picture marked with small blue dots and fragments. The volume occupied by the pores in the sprayed product is 9.2 % of the total volume of the product.

It was found that the thermal conductivity of the deposited material from aluminum oxide directly depends on the volume occupied by the pores in material. The smaller the pore volume, the higher will be the value of thermal conductivity of material products. Adjusting the porosity of the material products allows widely change the thermal conductivity of the deposited material. By varying the sintering modes it is possible to fabricate nanoporous products with desired values of thermal conductivity. In this research, reduction in the porosity of alumina ceramics has resulted in an increasing of the thermal conductivity more than ten times (**Table 1**). Thermal conductivity of the deposited alumina products is further increased if all material transferred into single-phase α-state [6]. In this case range of thermal conductivity variation can reach several orders of magnitude.

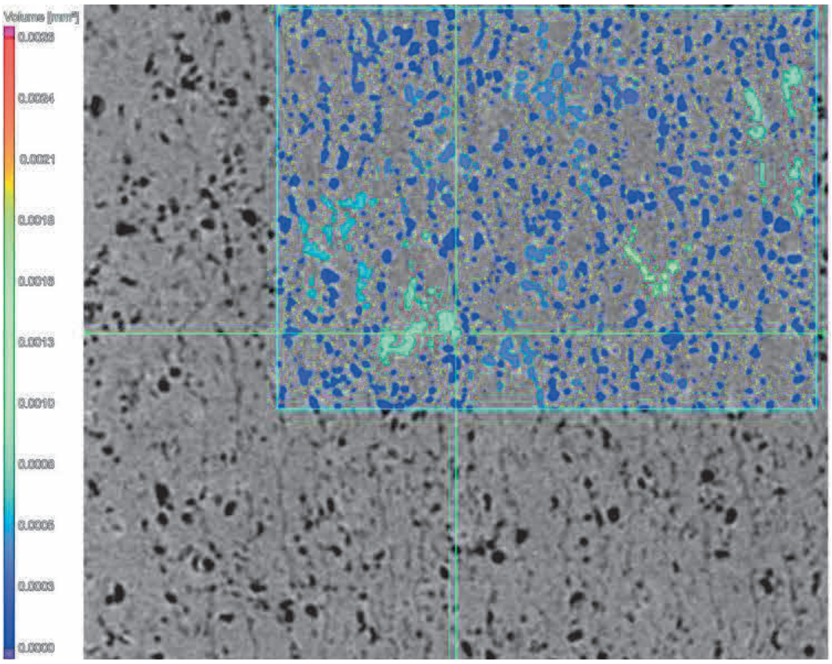

Figure 4 X-ray tomographic sectional image of plasma sprayed aluminum oxide product

Table 1 Density and thermal conductivity of alumina product after spray deposition and sintering

Specimen state	Density, g / cm^3	Thermal conductivity, W / m·K
As-deposited	3.42	0.35
Deposited and sintered	3.95	30

4. CONCLUSION

Plasma spraying of pure oxides allows obtaining nanoporous ceramic refractory products with a set value of the thermal conductivity. Reduction in porosity of products to nanoscale values and translation of material of plasma sprayed oxide product into single-phase α-state leads to a sharp increase in its thermal conductivity. Range of thermal conductivity variation can reach several orders of magnitude.

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