Studying of Diffusion of the Titan in Corundum Ceramics

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ABSTRACT

For increase in hardness (durability) and fire resistance (stability to melts metals) corundum ceramics on the basis of Al₂O₃, authors of the present work have suggested to protect for the first time its surface stronger and fire-resistant coverings, for example, from TiB₂. In work results of high-temperature diffusion TiB₂ in a surface of plates from Al₂O₃ are shown. For the first time the cathodoluminescence (CL) method for identification and a condition of atom Ti after diffusion is used, in for an establishment of its quantity electron probe microanalysis (EPMA). Durability increase defined a method microindentification by means of a pyramid of Knoop and strength at a bend. Researches have shown perspectivity of hardening of a surface corundum ceramics and use of the above-stated methods for studying of results of diffusion.

Keywords: Corundum Ceramics, Covering from Titanium Diboride, Catodoluminescence

1. Introduction

Modern the materials technology is characterized by search new and optimization of known ceramic compositions (in a general sense–compounds of metals with nonmetals). The basic share thus make oxides materials, though the quantity oxygen-free (on the basis of oxygen-free refractory compounds-borides, carbides, nitrides and silicides of metals and nonmetals) ceramics recently increases. Are used thus and various protective a covering for ceramics [1-4], including oxides. So, in work [4], for example, for increase stable properties of a composite on the basis of corundum coverings from TiB₂, ZrC, B₄C, and WC have been used. As it is known, one of the strongest and cheap ceramic is the ceramics on the basis of sapphire. One of ways of its further increase durability and fire-resistant properties is introduction in its surface of strengthening impurity a method of high-temperature diffusion.

Thus, as have shown researches, such coverings co-operate at a heat with a surface of such composite, partially or completely turning to difficult compounds and solid solutions—depending on a thickness of a covering, a way of its drawing and the subsequent thermal processing. There is a formation of various zones on depth, in which diffusion covering elements, as a rule, their components-metals.

2. Materials and Methods

2.1. Materials

In the present work diffusion Ti in ceramics on the basis of pure sapphire has been investigated. Layer TiB₂ has been put on a surface of ceramic plates (5 × 5 × 1 cm), and then they have been subjected thermal processing in argon atmosphere at 1500°C - 1700°C during ~ 27 - 48 hours. As a result in sofis a layer some zones on depth to 500 micron and more, containing various quantities Ti (Figure 1) were formed. Zones consist of sapphire grains the part from which is covered by a layer containing Ti. Thus in such layer probably formation of various compounds and the solid solutions [4], which identification presents considerable difficulties.

2.2. Research Diffusion a Layer in Ceramics

Samples of such ceramics after high-temperature diffusion have been investigated by electron probe microanalysis (EPMA), local visible cathodoluminescence (CL) and microindenting by means of a pyramid of Knoop and strength at a bend.
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3. Results and Discussion

3.1. Microhardness and Strength

Cross-sections for measurement of microhardness by means of a pyramid of Knoop prepared by a usual technique. For surface polishing samples were used by diamond pastes.

Researches strength characteristics have shown that after diffusion Ti microhardness of ceramics has increased approximately twice (from ~24.5 to ~39 GPa, that is from 3% to 58% [4]). The greatest values of microhardness turned out in 1st and 3rd layers, and the least—in 2nd (see of Figure 1). Such increase is explained sometimes presence polylayers and orientations nanofragments, perpendicular by planes microindetifikation. Durability of samples with a covering on a bend (by a standard technique) has been measured. In comparison with initial, samples with coverings have shown increase in strength at a bend on from 11.5 to 45% [4].

3.2. Cathodoluminescence and EPMA

In an optical microscope it is visible that the initial ceramics consists of separate grains-crystals leicosapphire (Figure 2). Spectra CL in a range from 1.6 - 2.4 eV have been received with filter JS-17 use. Samples were investigated at energy electrons 15 KeV.

In spectra CL characteristic strips connected with dot defects in sapphire crystals are observed some: a strip with a maximum 3.67 eV connected with a F-centre luminescence in sapphire (vacancy of oxygen with one electron) [5]; a strip with a maximum of intensity 3.1 eV, corresponding to a luminescence R—the centers (anion-action vacancies steams) [6]; a strip with a maximum 2.4 eV connected with steams anion the vacancies which have grasped electrons (the modular centers of F-type) [7]; and a strip with a maximum 1.8 eV, corresponding to luminescence Ti^{3+}. The maintenance of impurity Ti in initial ceramics as has shown method EPMA is less than 0.00 %wt.
After carrying out of high-temperature diffusion in CL the image it is possible to allocate some the characteristic areas located on various depths from a surface. In the area located deep into on 500 micron large grains with the expressed gradient of maintenance Ti (Figure 2) were observed. Apparently, in this area Ti got into grains on depth to 20 micron. In the centre of the majority of grains there were “single crystal” part of pure sapphire with zero maintenance Ti.

Luminescent properties of the allocated areas in grains (Figure 3) have been investigated. In spectra of sapphire ceramics are allocated a strip with a maximum 3.65 eV and a strip with a maximum 1.8 eV. For a strip with a maximum 1.8 eV concentration dependence (Figure 4) has been constructed.

On depth more than 500 micron from a surface the integrated grains of ceramics also are observed. In this case Ti diffusion in grains on depth no more than 5 microns (Figure 5). On depth more than 3 mm from a surface maintenance Ti corresponded to initial ceramics, i.e., 0.00% wt.

Comparison (on the area of crimson coloring) CL pictures of the crystal sapphire initially alloyed Ti (with the maintenance nearby 1% wt., the Figure 6) with the above-stated pictures of distribution Ti in crystal grains (Figure 2 see and Figure 5) shows volume alloying Ti the grains in the crystal sample and superficial alloying on some depth of grains of ceramics from a covering.

Depth of diffusion Ti in ceramics volume (to 1 - 3 mm depending on time of heat treatment and a thickness of a covering from TiB2) on usages exceeds depth of diffusion Ti in single crystal grain of sapphire under the same conditions. This anomaly is caused by that diffusion Ti passes in ceramics on intersize to borders. Then from borders of grains there is diffusion Ti to their centre on internal dispositions and cracks (area to maintenance Ti of 0.4% wt.). Ti reaches border of single crystal grain of sapphire with the low maintenance of dispositions and diffusion on depth of an order of 1 micron (maintenance Ti in this area of 0.1% wt.). In the grain centre zero-maintenance Ti remains.

Figure 3. Spectra CL of areas in ceramics with various maintenance Ti.

Figure 4. Dependence of intensity CL of a strip with a maximum 1.8 eV from maintenance Ti in the investigated micro volume.

Figure 5. Image CL of ceramics after term process on distance more than 500 microns from a surface (in the end of 3rd layer—see Figure 1). Grains with dark-blue the middle (Ti - 0.0%) and sites with diffuse Ti - from 0.2 to 0.4% and no more than 5 micron are visible width.

Figure 6. General view CL of a picture of single crystal grains of the sapphire alloyed Ti (~1% wt.). Uniform distribution Ti is characterized by uniform coloring of grains.
4. Summary

Thus, in the present work results of high-temperature diffusion Ti from TiB₂ in a surface pure corundum ceramics for the first time are shown:

1) depending on a temperature mode it is formed diffusions a layer with various depth (to 500 µm - 3 mm) in which some zones differ;

2) at transition from a surface deep into the sample, maintenance Ti in zones decreases with ~0.4 to 0.00% wt. (It is established with the help visible cathodeluminescence);

3) also can be defined and depth of penetration Ti in a surface of grains of sapphire in the form of ion Ti³⁺ (it is maximum to 20 µm, CL);

4) Knoop microhardness after thermodiffusion increases more, than in 1.5 times.

Samples with coverings have shown increase in strength at a bend on from 11.5% to 45% [4] in comparison with initial samples.

In summary it is possible to notice that there are real prospects of improvement of physicomechanical properties of a surface of ceramics for the account of ceramic coverings with the improved properties.

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REFERENCES


