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ABOUT MAKING OF CORUNDUM MICROPOWDERS, ALLOYED BY CHROME, AT CONDITIONS OF HYDROTHERMAL SYNTHESIS

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There is studied the influence of high pressure of water vapor on the processes of aluminum oxide hydrate dehydration for conditions of nonisothermal treatment. There are defined border conditions, providing structural recrystallization for aluminium hydroxides and implantation of chrome ions in the crystalline grate of corundum crystals.

Keywords: corundum, micropowder, high pressure, hydrothermal synthesis, alloying by chrome

Creation of progressive energy-saving technologies and new materials is one of major directions of scientific and technical development. To such tasks it is possible to take the making of metallurgical argil with the set phase composition, and also corundum (α-Al_2O_3) alloyed by chrome, which find more wide application during making of the special ceramics.

A main factor which limits their application complication serves making of the alloyed powders for monocorundum as with the size of crystals less than $5 \cdot 10^{-6}$ m. The alloyed powders with near descriptions get by melting in electric furnaces, and also by the method of hydrothermal synthesis [1,2], however need implementation of operation of the mechanical growing shallow, which negatively influences on a chemical purity and abrasive possibility of the got products.

In this connection the special attention is deserved by the method of making of different modifications for aluminium oxide by thermocouple and hydrothermal recrystallization of its hydroxides.

The analysis of experimental data about polymorphic transformations of aluminium hydroxides in the process of their heat treatment testifies to difficult character of such processes [3,4]. The limits of thermal firmness of hydroxides and oxides of aluminium are largely determined by pressure of water vapor. It is set that to the temperature 130 °C in all range of the studied pressures of water vapor a stable phase is gibbsite. A boehmite appears in the interval of temperatures 130-385 °C. Diasporas exist in the narrow interval of temperatures 275-415 °C and pressure of water vapor of higher, than 14.0 MPa. At a higher temperature the stable phase of connections of aluminium is corundum.

It is known that transformation γ-Al_2O_3 on α-Al_2O_3 also takes place through intermediate metastable aluminium oxide modifications, such as (χ-Al_2O_3, δ-Al_2O_3, (θ-Al_2O_3 and γ-Al_2O_3, which are characterized by the increased catalytic and adsorption activity, that conditioned by their structure [5]. Except for that, the presence of water vapor at heating allows to regulate phase composition of aluminium oxide for temperatures below, than 1000 °C [6].

The purpose of this research are determinations of phase composition and boundary conditions, which provide structural recrystallization of aluminium

hydroxides penetration of chrome ions to the crystalline grate of corundum crystals, which appear, after pressure of vapor-phase less than 20.0 MPa and temperature higher, than 100 °C. The researches are executed on the laboratory apparatus of hydrothermal synthesis, which consists of warming and reactionary autoclave and also mine electric furnace.

Control of temperature in the swept volume of furnaces was carried out by means of thermocouples, fixed on the external walls of warming and reactionary autoclaves. To the worker space of reactionary autoclave three are entered a control thermocouple and with the help flanged breech-block joined an exemplary manometer with the range of measuring 0-0.25 MPa.

A warming steam autoclave was filled by water and after placing of its in a mine electric furnace by means of steam line hermetically connected to the reactionary autoclave. Samples of initial material which contains aluminium hydroxide and calculation quantity of chrome connection, placed in a alumina boat and loaded to the reactionary autoclave.

After pressurizing of the noted autoclave it is included to mine electric furnace. Heating of reactionary autoclave executed at speed 5-8 °C/min to the temperature of synthesis duration of which at a stationary temperature in all experiments folded 2.0 hours. Pressure in a reactionary autoclave was created by heating of warming steam autoclave to the temperature 150-350 °C, that provided possibility of adjusting of pressure value in the interval of 0.5-16.5 MPa. At completion of experience reactionary and warming autoclaves cooled, pressure in a reactionary autoclave was reduced to the atmospheric value and a boat with products of synthesis drew out.

Products of synthesis were got as powder of pink color which contains 41.6-100 % α-Al_2O_3, other are a boehmite (γ-*AlOOH*). Chrome was looked in the crystalline grate of corundum as a sosoloid by a quantity from 0.05 to 4.8 %.

The analysis of the got results shows that at temperatures 500 °C and pressure of steam phase less than 10,0 MPa phase composition of products of thermocouple recrystallization becomes by polymineral. Thus complete recrystallization of гідроксиду of aluminium on a corundum (α-Al_2O_3) does not take place and there is in products of synthesis is a boehmite (γ-*AlOOH*). With the increase of pressure of steam phase higher than 10.0 MPa look the considerable increases of sizes of corundum crystals. Increase of temperature of process to 550 °C and creation of maximal pressure (10.0 MPa) provides the stable passing of recrystallization process for aluminium hydroxide on corundum after the simultaneous braking of process of increase for crystals. At the process of recrystallization active penetration of ions of chrome in the crystalline grate of corundum is provided.

With the increase of temperature of synthesis process to 1000 °C size of pressure of steam phase, sufficient for passing of recrystallization for aluminium hydroxide on corundum, can be monastic to the value of 0.5 MPa. At the simultaneous increase of temperature for synthesis process to 1000 °C and pressure to 10.0 MPa takes place considerable increase of sizes of corundum parts.

Characteristically, that in samples, got at the conditions of thermocouple recrystallization, a phase γ-Al_2O_3 is absented, which is characteristic for the conditions of burning of aluminium hydroxide at atmospheric pressure.

It is experimentally set that content connections of chrome, which add to the charge, coming from its rational use, must fold 0.05-5.0 % in a count on clean chrome. At content chrome less than 0.05 % does not look the improvement of physical and mechanical descriptions of micropowder by comparison to samples which alloying additions did not enter to. At content of connection chrome more than a 5.0 % measure of mastering of this metal, which is included in the crystalline grate of corundum, goes down to 80 %.

On the basis of the executed experimental researches evidently, that the process of hydrothermal treatment of charge it follows to execute in the interval of temperature 550-1000 °C and pressure of steam phase 0.5-10.0 MPa. So, the minimum level of temperature of synthesis process is answered by the maximal or near to its value of pressure for steam phase and, opposite, the maximal level of temperature of synthesis process is answered by minimum pressure of steam phase or other value of pressure in the interval of 0.5-10.0 MPa. Duration of treatment, at which provide passing of processes of phase transformations to the charge, in this case grows short and folds from 0.3 to 0.5 hours.

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