A NEW PROCESS FOR THE PRODUCTION
OF A CHROMIUM-CONTAINING ALUMINA-BASE
MATERIAL

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The new method of reactive infiltration described in [1, 2] enables sintered materials to be produced which are characterized by a uniform distribution of finely dispersed metallic and nonmetallic components, as a result of which their physomechanical properties are superior to those of analogous materials prepared by conventional processes. The reactive infiltration technique has been used for the synthesis of sintered alumina-base cermets and aluminum-silicon alloys [1]. The main drawback of these composite materials is their low mechanical strength at temperatures exceeding \( T_m \) of their metallic phases. This prompted a search for new high-temperature materials, of other compositions. One material investigated in the course of this work was an alumina-chromium composite.

As starting materials grade "ch." ("pure") or "ch. d. a." ("pure for analysis") chromic oxide powder and A-6 technical aluminum of 99.60% purity were used. Ceramic chromic oxide blanks were prepared by cold pressing and calcining. The process employed consisted of the following operations:

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Chromic oxide powder  Plasticizer
                  |  Mixing
                  |  Predrying
                  |  Rubbing
                  |  1st pressing
                  |  Granulation
                  |  2nd pressing
                  |  Drying, burning out of plasticizer
                  |  Sintering
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The chromic oxide powder was plasticized with 2.5% aqueous polyvinyl alcohol solution. The rubbing of the plasticized powder and the granulation of compacts after the first pressing operation were performed using a sieve with 1-mm-square apertures. Primary and secondary pressing was performed under a pressure of 2.5 tons/cm², since higher pressures resulted in overpressing of compacts and disturbed their integrity. After primary pressing the compacts had an apparent density of 3.10 g/cm³ and an apparent porosity of 39%, the corresponding figures after granulation and secondary pressing being 3.30 g/cm³ and 37%.

The compacts were dried for 10 h at a temperature of 80-85°C, after which the plasticizer was burnt out of them and they were sintered for 7 h at 1000°C in a laboratory Silit-rod furnace with an air atmosphere. The resultant ceramic chromic oxide blanks had an apparent density of 3.60 g/cm³ and an apparent porosity of 33%.

The ceramic chromic oxide specimens were then subjected to reactive infiltration with aluminum at various temperatures in corundum crucibles, using a laboratory Silit-rod furnace with an air atmosphere.

Fig. 1. Variation of microstructure of alumina–chromium material with time of holding in molten Al, × 200: a) infiltration front at beginning of reaction; b) after 15 h; c) after 25 h.

An x-ray diffraction examination revealed that the reaction products contained the α modification of alumina, aluminum and chromium metals, and an intermetallic compound of composition Al₂Cr₃.

On the basis of these data it is reasonable to conclude that the aluminothermic reduction of the chromic oxide in molten aluminum occurred according to the reaction

\[ 2\text{Al} + \text{Cr}_2\text{O}_3 \rightarrow \text{Al}_2\text{O}_3 + 2\text{Cr} \]  

which, under conditions such as those prevailing in our work, commences at a temperature of about 1000°C. The incubation period of chemical transformation according to reaction (1) decreases with rise in temperature, from 8 h at 1150°C to 5 h at 1250°C. In view of the need for obtaining defect-free specimens at a sufficiently high rate of reduction, the optimum conditions for the preparation of a sintered Al₂O₃–Cr–Al material are an infiltrating metal temperature of 1250°C and a treatment time of 25 h for ceramic compacts of 10–14-mm thickness.

A material synthesized in this manner differs markedly from metal–ceramic composites (cermets) produced by powder metallurgy techniques, mainly in that its phases are finely dispersed and uniformly distributed in its structure. In micrographs of specimens (Fig. 1) can clearly be seen two phases, the light areas being the metallic component of the system and the gray areas the α-Al₂O₃ component.

The process of structure formation in the composite investigated was studied by the methods of x-ray phase, metallographic, and electron microprobe analyses.*

In Fig. 1 is shown the variation of the microstructure of the cermet with time of exposure to molten aluminum. It will be seen from the micrographs that the finely dispersed structure of the material adjacent to the reactive infiltration front (Fig. 1a) is unstable. Processes of agglomeration of fine ceramic constituents of the material and growth of larger grains at the expense of fine result in the formation of a structure (Fig. 1c) consisting of large α-Al₂O₃ grains and grain agglomerates distributed in the metallic phase. In some zones the distribution of the ceramic and metallic phases is less even, which is due to the high porosity of the starting chromic oxide ceramic and its resultant defectiveness. To obtain a more uniform structure in the material, use should be made of high-density ceramic blanks produced by the hot pressing method.

*Analyses were carried out by V. D. Borzilova and L. P. Lyashenko.