At the present time parts in constructional metallic and mineral ceramics are produced by "free" sintering of cold-pressed compacts and/or hot pressing. The long sintering times often required in order to attain maximum density in ceramic parts as a rule lead to recrystallization and grain growth, which adversely affect the operating characteristics of finished products. There is therefore considerable interest in the possibility of activating the sintering of such materials, which would enable the process to be performed for a shorter time and at a lower temperature and result in energy savings.

Application of pressure during sintering is, of course, one of the physical methods of activation of sintering. With rise in pressure, the temperature necessary for the attainment of a given density falls. In [1], e.g., a high rate of shrinkage during an initial period was observed in the sintering of titanium diboride, resulting in a density of 99.5% of theoretical being reached in 20 sec at a pressure of 2.5 GPa and a temperature of 1580°C. Similar results were recorded in a study of the sintering of titanium carbide under high pressure [2].

In all the existing methods of manufacture of cutting tool ceramics the aim is to obtain a fine-grained (1-2 μm) polycrystalline structure, which ensures high strength in sintered products [3]. It is interesting to note that in [4] pressure was found to inhibit grain growth during sintering. Thus, the rate of growth of TaC grains was 6.7 in "free" sintering, 1.2 in ordinary hot pressing, and 0.6 nm/sec in sintering under high pressure. It is therefore desirable to raise the pressure (and at the same time shorten the time of hot pressing) in order to obtain a fine-grained material.

In the present work a study was made of the shrinkage and changes in structure experienced by a Cortinite mineral ceramic material, containing 80% corundum (α-Al₂O₃) and 20% TiN, during sintering for 0.5-8 min under a pressure of 5 GPa. Sintering was performed under a 5-MN DO-137A press in anvil type high-pressure devices (HPDs) with cavities having 0.5-cm³ reaction zones. Containers for specimens converting the uniaxial load of the press into quasistatic pressure and forming shutting-off layers in the HPDs during pressing were made of lithographic stone bonded with polyvinyl alcohol. The pressure in the HPDs was measured with a gauge calibrated against changes in the electrical resistance of Bi (2.55 GPa), PbSe (4.23 GPa), and PbTe (5.1 GPa). Heating was effected by the direct passage of current through cylindrical graphite elements. Temperatures were determined, after calibration against readings of a VR 5/20 tungsten–rhenium thermocouple, from the power expended in heating.

The microstructure of sintered specimens was examined in microfractograms (Fig. 1) taken in a Camscan scanning electron microscope at an accelerating voltage of 20 kV and an electron probe current of 9·10⁻¹¹ A. To prevent the surface charge of static electricity from distorting the electron image of structure, the fracture surfaces of specimens were coated with gold films of thickness about 20 nm.

As a source of α-Al₂O₃, GLMK fine-crystalline alumina was used, having a specific surface of about 2 m²/g after 30-h vibratory milling. Sintering experiments were carried out, under identical conditions, on pure GLMK alumina and two varieties of titanium nitride (differing markedly in particle size): a grade produced by nitriding titanium by a Donetsk Chemical Reagents Factory process (TU 6-09-112-75) and having, after additional 30-h vibratory milling with hard-metal balls under ethyl alcohol, a specific surface of about 2.5 m²/g, and an ultrafine grade (Sₘ ≈ 15 m²/g).

Fig. 1. Fractograms of specimens sintered under pressure of 5 GPa: a, b) GLMK sintered for 8 and 4 min, respectively; c, d) industrial TiN, 4 and 8 min; e, f) ultrafine TiN, 4 and 8 min; g, h) Cortinite (GLMK and industrial TiN), 2 and 8 min; i) Cortinite (GLMK and ultrafine TiN), 8 min. The details in frames on the right are shown magnified five times on the left.

Fig. 2. Densification of powders during sintering under pressure of 5 GPa: 1) GLMK; 2) industrial TiN; 3) Cortinite; 4) ultrafine TiN.