FABRICATION OF THE CERAMICS BASED ON SILICON NITRIDE BY FREE SINTERING AND COLD ISOSTATIC PRESSING

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Abstract

This research paper presents the results of a study of the mechanical properties and microstructure of the ceramics based on silicon nitride. In view of its high properties, such as high hardness and strength, low coefficient of thermal expansion, resistance to abrasive wear etc. ceramics based on silicon nitride is one of the leading candidate materials for a lot of applications. Some of these applications are mechanical engineering, aerospace industry, engine-building, metallurgy, welding industry. As additives, activating sintering material were used yttrium oxide (Y2O3) with aluminum oxide (Al2O3). It was used cold isostatic pressing as a method of compacting. Sintering the compacts was carried out in a nitrogen atmosphere at a temperature of 1600 C. Thus, ceramics based on Si3N4 with high dense and strength can be produced by cold isostatic pressing and free sintering. The main results can be summarized as follows:

1. Obtained the ceramic materials of high dense and increased mechanical properties. Compressive strength is 1500 MPa. Microhardness is 1300 HV.

2. Density of the obtained product made 90% from theoretical density.

3. Obtained ceramics has an equated fine-grain structure with a grain size of about 200 nm.

Combination of properties of obtained ceramics is attractive for a wide range of practical problems. Our paper seems perspective from the point of view of further effective application in many areas of science and equipment.

Keywords: ceramics, silicon nitride, free sintering

1. INTRODUCTION

Due to the excellent combination of properties silicon nitride based ceramics has attracted the attention for the last four decades. This type of ceramics used in many areas of industry and technology. Some of them are gas turbine engines, motor engines, cutting tools etc. [1]. In particular, silicon nitride is used to produce spin roller, foils and sheets, vibrational shaker head for calibration shaker, attrition mill agitator and immersion heater for aluminum foundries, spring rings and ball-and-socket joint, nuts and bolts with thread, tubes and profiles, etc.[2]. Silicon nitride based ceramics has high mechanical electrical and chemical characteristics. In particular, it can be noted that ceramics based on silicon nitride has high fracture toughness and hardness, high resistivity, high resistance to corrosion and acids. There are some disadvantages in sintering processes of silicon nitride based ceramics which connected with badly pressed of the pure silicon nitride ceramic. So oxide additives used to form a glass phases, such as sialon and YAG. Properties and characteristics of Si3N4 based ceramics strongly depend on the oxide additives used for the densification. The ideal additives for silicon nitride based ceramics are rare earth metal oxides. In this paper, we use as an additives yttrium and aluminum oxides, which have a high melting temperature and control the $\alpha\rightarrow\beta$ phase transformation rates of the Si3N4, the fraction of the $\beta$-Si3N4 and the grain growth anisotropy [3]. The aim of this work is to develop technology for fine-grained solid composite ceramics based on silicon nitride by cold isostatic pressing.
2. EXPERIMENTAL PROCEDURE

The starting materials used in experiments were as follows: 85% wt% Si₃N₄ (Stark, Grade M11) and 15% wt%. The ratio of additives is 9 wt% Al₂O₃ (A16 SG) and 6 wt% Y₂O₃ (Stark, Grade B). The composition of the powder mixture is 85% wt% Al₂O₃ and 6% Y₂O₃. The standard ceramic technology [4] was used for the obtaining the silicon nitride ceramics, implying the preparation of the mixture, pressing, sintering and final machining. The powder mixtures were milled in a circular vibratory type mill RS200 (Retsch GMBH). The compaction of powders produced by cold isostatic pressing at the room temperature and pressure of 200 MPa. For this purpose using cold isostatic press EPSI CIP 400 - 200*1000 Y. Sintering was carried out in the nitrogen atmosphere in the high-temperature vacuum furnaces at 1650°C VHT 8/22-GR. The density of the samples was measured by the helium pycnometry using helium pycnometer Micromeritics AccuPyc 1340. Microstructure of composite ceramics based on silicon nitride samples was studied on polished cross-sections using scanning electron microscopes Quanta 200-3D and Quanta FEG (SEM) and transmission electron field emission microscope JEOL JEM 2100 (TEM). Vickers microhardness, HV, was measured on polished cross-section surfaces using a 136° Vickers diamond pyramid under a 1 kg load applied for 15 s. Mechanical tests (σB, σf, KCU) were carried out in air using an Instron 300LX testing machine and Instron impact testing machine SI-1M at the room. Investigation of the dependence of Young's modulus of the temperature was carried out using a Dynamic mechanical analysis method (DMA) using NETZSCH DMA 242.

3. RESULTS AND DISCUSSION

Fine-grained submicron microstructure of the silicon nitride based ceramics is shown in Fig. 1. According to the SEM image the microstructure consists of an equiaxial crystallites 300-800 nm in size. TEM images of the microstructure of the silicon nitride based ceramics are shown in Fig. 2. The grain size is in the range from 50 nm to 300 nm. The results of mechanical testing of ceramics based on silicon nitride are shown in Table 2 and in Fig.4-5. Changing of Young's modulus of the temperature for the obtained ceramic material shown in Fig.3. Shown in Fig.3 dependence of Young's modulus of the temperature shows that in the temperature range 250-300 °C Young's modulus is not changed. However, at temperatures above 300 °C Young's modulus stabilized, this fact shows the hardening of our ceramic material.

Fig. 1 SEM image of fracture surfaces of the Si3N4 ceramics
Fig. 2 TEM image of the Si3N4 ceramics

**Table 1** The mechanical properties of the silicon nitride ceramics

<table>
<thead>
<tr>
<th>Microhardness (HV1)</th>
<th>$\sigma_c$ (MPa)</th>
<th>KCU (kJ/m$^2$)</th>
<th>$\sigma_f$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1365</td>
<td>2025</td>
<td>3.2</td>
<td>280</td>
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Fig. 3 DMA image of the Si3N4 ceramics
Analyzing the results of the mechanical tests can be noted that the obtained ceramic composite material based on silicon nitride is characterized by high strength characteristics, namely, high compressive strength (σ_c more than 2000 MPa, flexural strength more than 280 MPa; KCU - 3.2 kJ/m² (Table 2). The obtained ceramic is characterized by a high density (3.0 g/cm³), which is about 90% of theoretical density. Also obtained ceramic material has a high KCU (3.24 kJ/m²). Due to the high mechanical properties, high density and fine grain equiaxed structure obtained ceramic material based on silicon nitride can be used in the future in many areas of industry and engineering.

CONCLUSION

We have fabricated ceramic composite material based on silicon nitride with additives of yttrium oxide and aluminum oxide by the standard ceramic technology. The composite sintered at 1650 °C showed high...
mechanical properties such as compressive strength ~2100 MPa, microhardness ~1300 HV and KCU ~3.24 kJ/m². Relative density of fabricated ceramic composite material was ~90 %.

1. Phase transition of cristobalite in received ceramic composite was found by analysis of the temperature dependence of the modulus of elasticity. Formation of cristobalite occurs in the temperature range 1400-1600 °C during sintering of the material. Transition α → β of cristobalite in received ceramics gave rise to hardening effect and accompanied by an increase of 3.7% in the inversion point, and this increase is about 5.8% from room temperature to the phase transformation. Observed effects of self-strengthening and increase in the inversion point require additional investigations.

2. Thus, dense ceramic material with high strength can be fabricated by cold isostatic pressing and the method of the free sintering from of the silicon nitride powder with the additives of the yttrium and aluminum oxides.

3. The obtained results about ceramics based on silicon nitride can be used for further investigation of effective applications of this material.

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LITERATURE


