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Microstructure and Properties of Ceramics Based ON Silicon Nitride Obtained by Cold Isostatic Pressing and Free Sintering

O.A. Lukianova*

Belgorod State National Research University, 85 Pobedy str., Belgorod 308015, Russian Frderation

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The paper describes the microstructure and mechanical properties of ceramic material based on silicon nitride obtained by cold isostatic pressing and sintering in a nitrogen atmosphere. Microhardness and flexural strength are examined. The work is aimed to show that obtained material has moderately high strength, good physical characteristics and submicron structure.

Keywords: Silicon nitride, particle size, structure, strength, density.

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1. INTRODUCTION

Silicon nitride based ceramics are widely used in a large range of high temperature applications due to successful combination of properties. In particular silicon nitride has high strength, hardness, thermal stability, etc. [1]. Ceramics based on silicon nitride are often used in many areas of mechanical engineering, engine building, chemical and aviation industries.

However, the most important disadvantage of the ceramics is brittleness [2]. It is well known, that due to the bad compactibility of silicon nitride oxide additives are oftenly used. Thus, such oxide additives as MgO, Al_2O_3 and Y_2O_3 are most commonly used for formation of such glass phases as sialon and yttrium aluminum garnet (YAG) [3]. The aim of this work is to develop structure and some mechanical properties of the ceramics based on silicon nitride obtained by cold isostatic pressing and free sintering in a nitrogen atmosphere.

There are many well-known commercial methods of obtaining this type of ceramics such as hot isostatic pressing (HIP), spark plasma sintering (SPS), gas pressing (GP). Nowadays, one of the most important issue is development of cheaper methods of obtaining structural ceramics silicon nitride. Cold isostatic pressing seems to be very beneficial approach to this situation.

2. EXPERIMENTAL DETAILS

2.1 Material

Ceramics based on silicon nitride was fabricated by cold isostatic pressing (CIP) and free sintering in a nitrogen atmosphere. Batch included the silicon nitride 85% wt% Si_3N_4 and aluminum 9 wt% Al_2O_3 and yttrium 6 wt% Y_2O_3 oxides additives. The powders were mixed in the attritor mill (RS200 Retsch). Then, the mixture of powders were compacted by cold isostatic pressing (EPSI CIP 400 B-9140). Sintering of the green bodies was performed in a vacuum furnace (VHT 8/22-GR) at 1650ºC temperature in a nitrogen atmosphere. For more details see Ref. [4] and RU patent № 2014127439.

2.2 Methods

The study of a microstructure of samples was carried out using scanning electron microscopy Quanta 600 FEG (SEM). Microhardness of the sample was performed on automatic microhardness tester Affri DM8 on a polished cross-section surface by Vickers method applying 0.5 kg load during 15 s. Microhardness of the sample by Knoop was performed on microhardness tester Indenter test – Device (construction and software self-made microindentation hardness tester) applying 2.5 kg load during 15 s.

The study of density of samples was carried out by means of the helium pycnometry method using helium pycnometer Micromeritics AccuPyc 1340. Bending tests were conducted in air using an Instron 300LX testing machine at the room temperature. A cross-head speed was 0.5 mm/min. The support distance for 3 point bending tests (3PB) was 30 mm. Open porosity was determined by mercury porosimetry.

To investigate the fine structure of obtained structural ceramic a JEM-2100 transmission electron microscope with accelerating voltage 200 kV, equipped with an attachment for performing local chemical analysis was used. The samples were prepared by mechanical polishing on abrasive grinding paper with a grain size reduced at the subsequent polishing using an OP-S polishing suspension. Thin «foil» of samples were prepared by polishing using ionic etching. Obtained foils were placed in an Ion Milling Model 1010 ion gun (Fischione Company), for final polishing by the ion gun (voltage 5 kV, current 5 mA) was used. The initial angle 11°of polishing was formed before opening. The sample was positioned at the angle 9° after that (for 20 min).

3. RESULTS AND ANALYSIS

3.1 Structure and microstructure

SEM and TEM images of the structure and microstructure of the obtained silicon nitride based ceramic are presented in Fig. 1 an Fig.2, respectively. The presence of a dense submicron structure is clearly seen. The average size of the structural components varies in the range from 100 to 800 nm. It may be noted that, equi-

* Sokos100@mail.ru

axed structural components are observed. It is obvious that, the structure of ceramic always strongly affect on its mechanical and physical properties. In some cases, an elongated structure contributes to increased fracture toughness.

3.2 Physical properties

It was investigated that, the density of the obtained ceramic is 2.94 g/cm^3 and open porosity is 0.1 % (Table 1).

Table 1 – Density and porosity of the obtained ceramic material

Bulk density (g/cm^3)	Open porosity (%)
2.94	0.1

3.3 Mechanical properties

The most important mechanical properties of the ceramic are flexural strength and fracture toughness. Unfortunately, most ceramics have low fracture toughness. However, it is well known that, all ceramics have high hardness [3]. Table 2 clearly shows that the obtained ceramics have high microhardness by Knoop (975 $HK_{2.5}$) and Vickers (1380 $HV_{0.5}$). Both, the microstructure and mechanical properties of material are strongly depend on the method of producing.

Table 2 – Microhardness of produced ceramic material

$HV_{0.5}$	$HK_{2.5}$
1380	975

Results of the mechanical tests show, that the obtained ceramic composite material based on silicon nitride is characterized by moderately high strength characteristics, namely, flexural strength was 280 MPa. Similar strength demonstrates the ceramics produced by the reaction bonding.

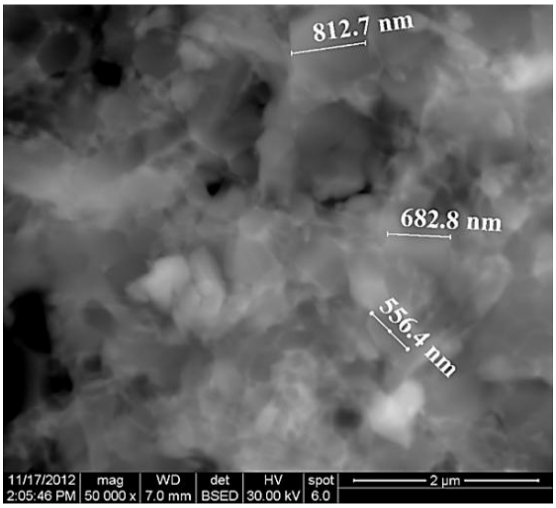

Fig. 1 – Structure of the obtained ceramics based on silicon nitride. Scanning electron microscope.

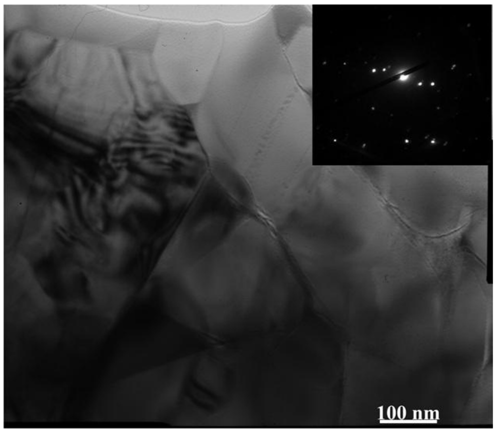

Fig. 2 – Microstructure of the obtained ceramics based on silicon nitride. Transmission electron microscope.

4. CONCLUSIONS

We measured some physical and mechanical characteristics of the cold isostatic pressed and free sintered in a nitrogen atmosphere silicon nitride ceramic. In our study we showed that the obtained material is characterized by high hardness and density, an almost complete absence of the open porosity.

- Obtained material is characterized by submicron structure with a grain size in the range from 100 to 800nm.
- Microhardness of the obtained ceramic are 1380 $HV_{0.5}$ and 970 $HK_{2.5}$, respectively. The bending strength is 280 MPa.
- Density of the produced ceramic is 2,94 g/cm^3 and open porosity is 0.1 %.

The proposed approach can be used to produce a relatively inexpensive construction material based on silicon nitride. The resulting material can be successfully used in the future in many areas of modern technology and industry in a wide range of differentof applications. The process described is a good alternative for the production of ceramic structural ceramics based on silicon nitride.

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