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NONOXIDE CERAMIC COMPOSITES WITH SUPERIOR MECHANICAL PROPERTIES BASED ON SiC AND Si_3N_4

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Abstract: This paper presents comparative experimental results obtained in the development of ceramic composites based on SiC and Si_3N_4,, the classical method of sinlering and plasma sintering - Spark Plasma Sintering (SPS). Both sintering processes were carried out in a controlled atmosphere (N_2). Experiments carried out aimed to obtain ceramic samples based on SiC and Si_3N_4 with different sintering oddity (Al_2O_3, Y_2O_3) and characterization of materials obtained. Were determined physico-mechanical parameters (bulk density, apparent porosity, water absorption, compressive strength and hardness). Also were performed compositional analyses by X-ray diffraction for the two nonoxide systems developed. The results highlight the advantage of allowing obtaining superior materials characteristics by SPS sintering method, compared with classic treatment of sintering $j_{n\ normaj}$ atmosphere.
Keywords: ceramic composites, SiC, Si_3N_4, SPs sintering, physico-mechanical parameters.

1. INTRODUCTION

More and more ceramic materials become market leader because of compositional diversity, processing technology and not least the special properties. Nonoxide ceramics are materials with excellent resistance to the extremely hostile working conditions such as high temperatures, corrosive environments, mechanical stress, they are based materials with controlled microstructure and a high degree of stability of their properties. Also, in processing of nonoxide ceramics have an important role manufacturing complex conditions and low cost price. Major advantage, essentially, of the cornposite ceramics lies in the possibility of modulation properties, thus achieving a wide variety of materials, whose use is expanding in almost all fields of technical activity. Their properties are emphasized by particular constructive methods of the material, resulting materials with special properties: high specific strength and stiffness, high temperature resistance, wear resistance, increased efficiency of the total weight of machinery, vehicles, or construction of various constructions or aircraft. Silicon carbide has long been recognized as an ideal material for applications where superior mechanical properties are important such as hardness, Young modulus, flexural strength and stiffness and high temperature oxidation resistance, high thermal conductivity (120W/mXK) low thermal expansion coefficient ((4 x lO^{-6}/C), and resistance to wear and abrasion [1].
The level of mechanical properties of ceramics based on silicon nitride shows that they have a favorable combination of properties high hardness, maximum bending strength, high hardness, tensile strength and density. [2] Use of sintering additives ($A1_20_3$ Y_20_3), the nonoxide ceramics sintering can be seen not only as an aid element in the densification but also as a key element in obtaining the property, since they are directly influenced by microstructure and phase chemistry of grain. [3]
In this study we aimed to achieve nonoxide composite ceramic materials based Si_3N_4/SiC with different sintering additives with superior mechanical properties Due to outstanding performance resulting from tne research recommended that these composite materials to be used in ballistics.

2. RAW MATERIALS AND WORld^ METHOD

Nonoxide ceramic materials composite, in powder stage, have been prepared by standard ceramic materials technology, starting from ceramic materials of high puritv (Si_3N_4, SiC, Al_20_3, Y_20_3- from Alfa Aesar).

Two nonoxide ceramic materials composite recipes have been elaborated:

- *A21* recipe - **A21** - Si_3N_4-20%SiC with 2% $A1_20_3/Y_20_3$ addition
- *A22* recipe - **A21** - Si_3N_4-20%SiC with 3% $A1_20_3/Y_20_3$ addition

Composite mixtures were made according to the above compositions by wet milling (alcohol, p.a) for 4 hours, until a homogeneous material is obtain. The powder obtained was dried in the oven type Suszarka at 80°C for 12 hours to obtain a humidity of less than 5%.
Shaping was done by pressure-sintering the Spark Plasma Sintering Furnace HPD25 oven, the mold 0 = 40mm. Sintering parameters was as following: temperature 1850°C, heating rate 100°C/min, the landing of 8 minutes, 1 ms pulse, pressing force of 44 KN in N_2 atmosphere.

3. CHARACTERIZING METHODS

The apparent density of sintered samples was determinate by immersion method, using an analytical balance, with a precision of 0,1 %. The measurements were performed on at least four samples of ceramic material.
Microstructural analysis of powder particles were made by X-ray diffraction at an X-ray Difractometru Bruker-AXS D8 ADVANCE type, X-ray tube with Cu anode, 40kV/40 mA, Ni filter kb. Measurement step was: 0.04 ° one time point of measurement: Is, the field of 28= 10° - 100°.
Morpho-structural analysis by optical microscopy was performed by visualization with Carl Zeiss Jena microscope NU2 type, with DinoLite digital attached camera and image acquisition software for it. Photographs of samples were made in direct reflected light to increase the 300x.
SEM micrographics were viewed using scanning electron microscope type FESEM-FIB Auriga model produced by Carl Zeiss Germany. Bending tests of samples were performed with universal machine for mechanical testing of materials in the static model LFM 30 kN Walter & Bathrooms AG (Switzerland), according to EN 10002-1:2001 and ASTM 1820:2008. Microhardness tests were performed with micro FM700, which is determined microhardness Vickers and Rnoop loads ranging from 25 gf to 2000 gf, equipped with digital camera. Vickers microhardness (HV) is calculated by the ratio of pressure force P and lateral surface area of residual footprint produced by diamond pyramid with a square base. Trace is considered as a right pyramid with diagonal D having the same angle as the peak body penetration. The angle between two opposite sides equal to 136° was elected to establish a correlation with Knoop hardness. Vickers microhardness is calculated with:

$H_v = P/S = (2\ P \sin 6/2) / D^2 = 2P\sin 68°/d^2 = l,8544P/d^2$ $[kg/mm^2]$ where *P* is the load (in kilograms) and *d* is the average between the two diagonals (in mm) of indentation measured using a microscope.

4. RESULTS AND DISCUTIONS

4.1 Density and apparent porosity

Results obtained from characterization of thermally treated samples are presented in Table 1. Note that samples from both recipes are apparent porosity values within the range required (<1%) for ceramic composite materials obtained, the results are presented in Table **1**.

Table 1- Physical characteristics of ceramic composite materials

Composition	Apparent porosities, [%]	Apparent density, [g/cm^3]	Water absorption, [%]
A21-2%	0.44	3.04	0.14
A22 - 3%	0.05	3.14	0.01

4.2 Mineralogical composition

Results of X-ray diffraction analysis performed on specimens of compositions **A21,** sintered at a temperature of 1850°C, presents the composition p Si_3N_4 hexagonal (crystallites size D = 143.5 nm) cubic SiC (D = 118.9 nm) $A1_20_3$ and Y_20_3 sintering additives (Figure 1). Results of X-ray diffraction analysis performed on specimens of compositions **A22,** sintered at a temperature of 1850°C, present in **p** Si_3N_4 composition hex (crystallites size D = 79.3 nm) cubic SiC (D = 34.9 nm) $A1_20_3$ and Y_20_3 sintering additives (Figure 2).

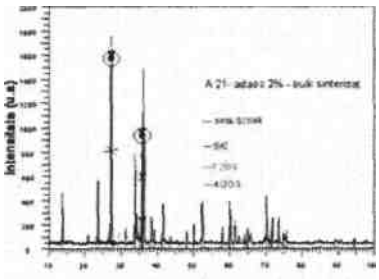

Figure 1 - A21 sample X-ray difraction pattern

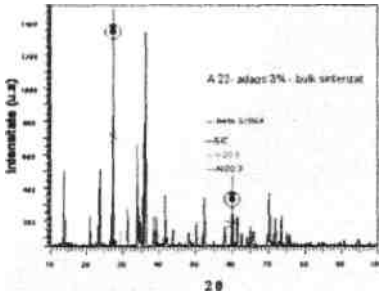

Figure 2- A22 sample X-ray difraction pattern

4.3 The samples texture

Optical and electronic microscopy (SEM) analysis, of samples of compositions **A21** (Figure 3) and **A22** (Figure 4), the evidence shows that A22 is more homogeneous particle distribution than the samples A21, which is reflected in properties obtained (density, hardness, flexural strength).

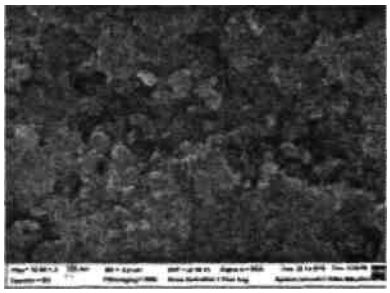

Figure 3 - A21 sample SEM micrographics

Figure 4 - A22 sample SEM micrographics

4.4 Mechanical properties

By analyzing the test results of composite ceramic materials in terms of mechanical properties is observed that the values of bending strength and modulus of elasticity in both samples have comparable values with reported values [4-6], but it is noted that A22 sample has values much better for breaking strength (abaut 600 MPa) than A21 sample (abaut 330 MPa), differece also confirmed betwen apparent porosity, apparent density, and microhardness values presented in Table 2.

Table 2- Mechanical properties of ceramic materials composite

Composition	Young Modulus, (GPa)	Bending strength, [MPa]	Vickers hardness, [GPa]
A21(2% addition)	292.39	331.52	15.7
A22(3% addition)	544.62	600.06	19.9

4. CONCLUSIONS

In the present study was aimed to obtain high performance mechanical properties for composites based on Si_3N_4 and SiC, with different $A1_20_3$ and Y_20_3 sintering additives, for use in the field of antiballistics protection.
Both (A21 and A22) obtained ceramic composites materials, has apparent porosity values within the range required (<1%), with the apparent density of 3.04 g/cm^3 for A21 sample and of 3.14 g/cm^3 for A22 sample. Electron microscopy micrographics analysis (SEM) show that in A22 samples a more homogeneous particle distribution is present than A21 samples, which is also reflected in obtained properties (density, hardness, flexural strength).

The obtained results analysis for the mechanical testing show that A22 samples properties has better values for modulus of elasticity (abaut 545 GPa) and flexural strength (abaut 600 MPa) than for A22 sample.

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