

# MATERIALE CERAMICE COMPOZITE PE BAZĂ DE $\text{Si}_3\text{N}_4$ ȘI $\text{SiC}$ , COMPOSITE CERAMIC MATERIALS BASED ON $\text{Si}_3\text{N}_4$ AND $\text{SiC}$

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The aim of the work is to obtain through the spark plasma sintering (SPS) technique, ceramic composite materials based on  $\text{Si}_3\text{N}_4$  and  $\text{SiC}$ , with different sintering additions ( $\text{Al}_2\text{O}_3$  and  $\text{Y}_2\text{O}_3$ ) with improved mechanical properties. The spark plasma sintering process was carried out in vacuum at a pressure of 50 MPa, at temperatures between 1600-1800°C and a maximum sintering time of 5 minutes. In order to put in evidence the structural and morphological behavior of the obtained composites at different sintering temperature that lead to their improved mechanical properties, they were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques. Physical-mechanical characteristics (apparent density, apparent porosity and Vickers hardness) were determined for all sintered samples. Structural analysis by X-ray diffraction, revealed the formation of two major phases  $\alpha$  and  $\beta$ - $\text{Si}_3\text{N}_4$ , with an increase of their crystallographic transformation from one phase to another ( $\alpha \rightarrow \beta$ ) with the increasing of the sintering temperature.  $\text{Si}_3\text{N}_4$  and  $\text{SiC}$  based ceramic composites sintered at 1800°C and with a 5wt.%  $\text{Y}_2\text{O}_3$  content, showed a good correlation of physical-mechanical and structural characteristics, having a relative density of 93% of the theoretical density, Vickers hardness values of 18.39 GPa and bending strength of 336 MPa.

Scopul lucrării constă în obținerea unor materiale compozite ceramice pe bază de  $\text{Si}_3\text{N}_4$  și  $\text{SiC}$ , cu diferite adaosuri de sinterizare ( $\text{Al}_2\text{O}_3$  și  $\text{Y}_2\text{O}_3$ ), având proprietăți mecanice îmbunătățite, folosind tehnica de sinterizare în plasmă cu scânteie (SPS). Procesul de sinterizare în plasmă, s-a realizat în vid, la presiunea de 50 MPa, la temperaturi cuprinse între 1600-1800°C și o durată de sinterizare maximă de 5 minute. Pentru a pune în evidență comportamentul structural și morfologic al compozitelor obținute la diferite temperaturi de sinterizare care conduc la îmbunătățirea proprietăților mecanice, acestea au fost caracterizate prin tehnici de difracție cu raze X (DRX) și microscopie electronică de baleiaj (SEM). Au fost determinate pentru toate probele sinterizate, caracteristicile fizico-mecanice (densitate aparentă, porozitate aparentă și duritate Vickers). Din analizele structurale prin difracție de raze X, s-a pus în evidență formarea a două faze majoritare  $\alpha$ - $\text{Si}_3\text{N}_4$  și respectiv  $\beta$ - $\text{Si}_3\text{N}_4$ , observându-se că, odată cu creșterea temperaturii de sinterizare, gradul de transformare cristalografică dintr-o fază în alta ( $\alpha \rightarrow \beta$ ) crește. Materialele compozite ceramice pe bază de  $\text{Si}_3\text{N}_4$  și  $\text{SiC}$  sinterizate la 1800°C și cu un conținut de 5 % masic  $\text{Y}_2\text{O}_3$ , au prezentat o corelație bună între caracteristicile fizico-mecanice și structurale, având o densitate relativă de 93 % din densitatea teoretică, valori ale durității Vickers de 18,39 GPa și rezistența la încovoiere de aproximativ 336 MPa.

**Keywords:** Spark Plasma Sintering,  $\text{Si}_3\text{N}_4$ ,  $\text{SiC}$ , ceramic composites, X-ray diffraction, scanning electron microscopy

## 1. Introduction

Among the most attractive advanced ceramic materials, Silicon Nitride ( $\text{Si}_3\text{N}_4$ ) stands out, because the resulting microstructure after sintering was found to be similar to that of whisker-reinforced composites, which present elongated  $\beta$ - $\text{Si}_3\text{N}_4$  grains with an element-strengthening effect. In general, these materials are used in fields with structural applications at high temperatures, due to their excellent mechanical and thermo-mechanical properties. For some specific applications, such as: cutting tools, automotive components, heat exchangers, the most important mechanical characteristics are fracture resistance, hardness and mechanical wear resistance [1].

Due to these mechanical performances,

reinforcement, due to the hardness and resistance at high temperatures [11-14].

$\text{Si}_3\text{N}_4$ -based ceramic materials have received considerable attention from scientific communities [1-14].

In general, dense ceramic materials based on  $\text{Si}_3\text{N}_4$  successfully replace a wide range of applications where other types of materials such as metallic and polymeric materials are involved. To obtain a good densification of the ceramics based on  $\text{Si}_3\text{N}_4$ , sintering additives such as  $\text{Al}_2\text{O}_3$  [9],  $\text{Y}_2\text{O}_3$ ,  $\text{AlN}$ ,  $\text{MgO}$ ,  $\text{MgSiN}_2$  [8],  $\text{MgAl}_2\text{O}_4$  [6] or mixtures of  $\text{Al}_2\text{O}_3$  and  $\text{Y}_2\text{O}_3$  are used [1, 2, 4, 5]. The amount of sintering additives influences both the composition of the base matrix and the microstructural characteristics. It has been reported that the addition of  $\text{SiC}$  particles in  $\text{Si}_3\text{N}_4$ -based ceramic materials can substantially improve the mechanical properties, acting as a

## 2. The experimental part

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Ceramics based on Si<sub>3</sub>N<sub>4</sub> can be obtained by various techniques such as: hot pressing, gas pressure sintering, pressure less sintering and hot isostatic pressing (HIP). However, obtaining materials based on Si<sub>3</sub>N<sub>4</sub>, with a high degree of compactness, are difficult to sinter in solid state [1].

Recently, among the sintering techniques used for a series of new, difficult-to-sinter materials, spark plasma sintering (SPS) was preferred non-conventional method of consolidating powdered ceramic. Unlike conventional techniques, the spark plasma sintering method brings major advantages to the material consolidation process, through high heating and cooling speeds, reduced sintering time, precise energy management, reliability, operational safety, reproducibility, as well as reduced energy consumption. Through the SPS technique, the sintering process takes place quickly by passing a pulsed direct current, both through the electrically conductive mold in which the powder to be sintered is located, and through the sample support, so that a Joule effect is produced instantly [1,3]. Through the SPS process, the sintering time of ceramics based on Si<sub>3</sub>N<sub>4</sub> was reduced to a few minutes, causing an almost total densification of the materials, the growth of particles and the initiation of phase transformations being inhibited [4].

Ceramic composite materials based on Si<sub>3</sub>N<sub>4</sub>/SiC have been studied in numerous research works [11-15]. It was shown that the addition of 5 and 10% SiC in Si<sub>3</sub>N<sub>4</sub> has a positive effect on the fracture resistance and hardness of the Si<sub>3</sub>N<sub>4</sub>/SiC composite compared to monolithic Si<sub>3</sub>N<sub>4</sub>-based ceramics. The hardness of the ceramic materials increased from 14.48 GPa in the monolithic Si<sub>3</sub>N<sub>4</sub> ceramic to 16.99 GPa in the ceramic composite with 20 vol % SiC [12]. Yang et al. [11], also studied the influence of SiC particle dispersion on the phase transformation, microstructure and mechanical properties of Si<sub>3</sub>N<sub>4</sub> ceramics and concluded that densification was substantially delayed by SiC particle dispersions in Si<sub>3</sub>N<sub>4</sub>/SiC composites. The highest flexural strength of over 1 GPa was obtained for a SiC content of 20% vol [11].

Dense Si<sub>3</sub>N<sub>4</sub>-SiC-based micro-nano composites with added MgSiN<sub>2</sub> sintering were also studied by spark plasma sintering, and it was observed that Si<sub>3</sub>N<sub>4</sub> grain growth and  $\alpha \rightarrow \beta$  Si<sub>3</sub>N<sub>4</sub> transformation were inhibited by the addition of SiC as a secondary phase. Due to the addition of 5 vol.% SiC, the hardness of the samples increased from 17.95 GPa to 18.5 GPa [15].

The aim of this paper was to obtain by the Spark Plasma Sintering (SPS) technique of ceramic composite materials based on Si<sub>3</sub>N<sub>4</sub> and SiC with different sintering additives (Al<sub>2</sub>O<sub>3</sub> and Y<sub>2</sub>O<sub>3</sub>) with improved mechanical properties.

Composite non-oxide ceramic materials in powder form were synthesized from raw materials of high purity 99% (Si<sub>3</sub>N<sub>4</sub>, SiC, Al<sub>2</sub>O<sub>3</sub>, Y<sub>2</sub>O<sub>3</sub> from Alfa Aesar supplier) using the route of standard ceramic technology as shown in the previous paper [18]. The current work aims to continue previous studies on composite ceramic materials [18], in order to establish the influence of sintering parameters (temperature and atmosphere). In this work, were elaborated two types of non-oxide composite ceramic materials, named A and B, using spark plasma sintering (SPS) technology. It should also be noted that the composite ceramic material marked A has been previously studied in N<sub>2</sub> atmosphere at a sintering temperature of 1850°C [18]. The novel elements of the present work are, on the one hand, the new composition of the material marked B and, on the other hand, the samples were obtained by SPS technique in vacuum at temperatures between 1600-1800°C. Table 1 shows the chemical composition of the synthesized samples of the two composites.

**Table 1**  
Chemical compositions of the sintered samples (mass %) /  
Compoziții chimice ale probelor sinterizate (% masice)

Sample code	Si <sub>3</sub> N <sub>4</sub>	SiC	Al <sub>2</sub> O <sub>3</sub>	Y <sub>2</sub> O <sub>3</sub>
A	77	20	1.5	1.5
B	73.5	20	1.5	5

The powders were dosed to the established chemical compositions (Table 1) using a Precisa XT 220 A digital balance with digital display, accuracy class 0.1mg.

Samples were wet homogenized in a planetary mill, for 4 hours, within wolfram recipients and with SiC balls. The mixture obtained was oven-dried at 80°C for 16 hours to obtain a moisture content of less than 5%. The obtained powders were plasma sintered at different temperatures between 1600°C and 1800°C in a Spark Plasma Sintering Furnace HPD25, in vacuum, at a pressure of 50MPa, using the heating rate of 100°C/min, pulse duration of 1 ms, for a maintaining time at the maximum temperature of 5 minutes.

Disc-shaped samples with a diameter of 20±0,05 mm and a height of 2.5±0,04 mm were obtained and mechanically polished to mirror polish.

The metallographic polishing was done using the Tegramin 20-Struers semi-automatic machine and Struers products according to the method indicated by the manufacturer. For plane surface polishing, the MD-Piano-220 abrasive disc was used in a watery environment, then the polishing was done with DiaPro Plan 9µm diamond slurry on MD Plan suport and 0.25µm silicon slurry on MD Chem suport. The final polishing time was adjusted until a surface with metallographic gloss was obtained.

Samples density according to heat treatment / Densitatea probelor în funcție de tratamentul termic

Sample	Sintering temperature	Apparent density	Relative density
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**Table 2**

code	[°C]	[g/cm <sup>3</sup> ]	[%]
A	1600	2.27 ± 0.03	66.40 ± 0.2
	1650	2.47 ± 0.05	72.30 ± 0.4
	1700	2.53 ± 0.06	74.00 ± 0.5
	1750	2.56 ± 0.03	74.90 ± 0.6
	1800	2.68 ± 0.02	78.40 ± 0.3
B	1600	2.86 ± 0.04	82.50 ± 0.2
	1650	2.91 ± 0.06	83.90 ± 0.4
	1700	3.15 ± 0.03	90.80 ± 0.3
	1750	3.21 ± 0.06	92.50 ± 0.5
	1800	3.23 ± 0.05	93.10 ± 0.4

Table 3

Elemental cell parameters, mean crystallite size and crystallographic phase content of the samples obtained by SPS  
*Parametrii celulei elementare, dimensiunea medie de cristali $\tilde{t}$  și conținutul fazelor cristaline pentru probele obținute prin SPS*

Sample code	Sintering temperature (°C)	Crystallographic phase	Rietveld semiquantitative analysis [%masice]	Elemental cell parameters		Mean crystallite size D (nm)
				a [Å]	c [Å]	
A	1600	α-Si <sub>3</sub> N <sub>4</sub> (hexagonal)	50.8	7.75	5.62	91
		β-Si <sub>3</sub> N <sub>4</sub>	35	7.60	2.9	81
		SiC	14	3.08	15.12	70
A	1700	α-Si <sub>3</sub> N <sub>4</sub>	39.1	7.74	5.619	87
		β-Si <sub>3</sub> N <sub>4</sub>	48.2	7.60	2.90	83
		SiC	12.5	3.08	15.11	64
A	1800	α-Si <sub>3</sub> N <sub>4</sub>	9	7.74	5.61	74
		β-Si <sub>3</sub> N <sub>4</sub>	77.7	7.60	2.90	89
		SiC	13.1	3.08	15.11	72
B	1600	α-Si <sub>3</sub> N <sub>4</sub>	57.3	7.75	5.62	75
		β-Si <sub>3</sub> N <sub>4</sub>	30	7.60	2.91	62
		SiC	12.5	3.08	15.12	78
B	1700	α-Si <sub>3</sub> N <sub>4</sub>	31.9	7.74	5.61	76
		β-Si <sub>3</sub> N <sub>4</sub>	57.1	7.60	2.90	72
		SiC	10.8	3.08	15.11	88
B	1800	α-Si <sub>3</sub> N <sub>4</sub>	16.4	7.74	5.61	71
		β-Si <sub>3</sub> N <sub>4</sub>	72.6	7.60	2.90	78
		SiC	10.8	3.08	15.11	86

On the obtained samples, the apparent density was determined by the Archimedes method according to the SR EN ISO 18754:2022. The measurements were performed on three samples of each ceramic material, prepared in the same way as presented in reference [19]. The mineralogical composition formed during the sintering heat treatment were examined by X-ray diffraction with the D8Discover diffractometer (Bruker AXS) configured in Braag-Brentano geometry, equipped with X-ray tube with Cu anode, U=40kV and I=40 mA, respectively 1D LynxEye detector. Diffractograms were recorded in the angular range  $2\theta = 10^\circ - 100^\circ$ , with an increment of  $0.04^\circ$ . The identification of diffraction maxima was performed using the ICDD PDF2 database. The crystal structure parameters were determined by Rietveld analysis using the TOPAS analysis program and the FindIt database and the microstructure of the samples was examined using the FESEM-FIB type scanning electron microscope model Auriga (Carl Zeiss Germany). Hardness tests were performed with the universal hardness tester model VRSD (AFFRI) and Vickers indentation using the 136<sup>0</sup>

diamond pyramid indenter according to SR EN ISO 6507:2018. The indentation load used was 294.198 N (30Kgf) for a time of 15s. The mechanical three-point bending tests were performed on the composite ceramic materials, using the universal machine for the mechanical testing of materials in a static regime (model LFM 30kN, Walter&Bai AG from Switzerland) according to SR EN ISO 17138:2022.

### 3.Results and discussions

#### 3.1.Sintering behaviour of SPS sintered samples

Samples were sintered at temperatures between 1600°C and 1800°C in vacuum by the SPS technique. The theoretical density was calculated by the additive method [18], using the gravimetric percentages of the compounds entering the composites.

The results of the relative densities obtained after sintering by SPS are presented in Table 2. It can be seen that the ceramic material sintered at 1800°C (sample A) shows poor sintering

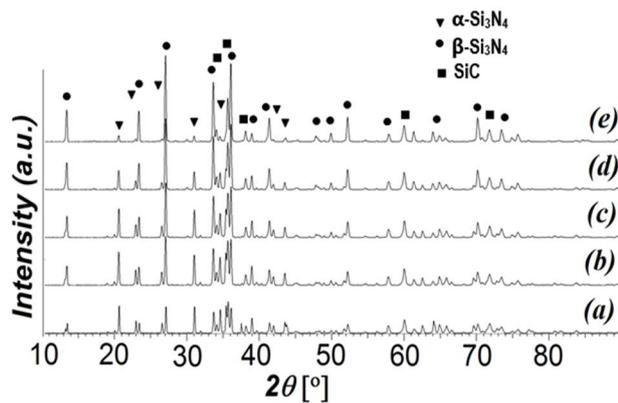


Fig. 1 - X-ray diffraction spectra of samples from composite material A sintered at / Spectrele de difracție de raze X a probelor din materialul compozit A sinterizate la: (a) 1600°C, (b) 1650°C, (c) 1700°C, (d) 1750°C, (e) 1800°C

behavior, obtaining a relative density of only 83.90% of the theoretical density. In the case of the material sintered at the same temperature, 1800°C, (sample B), the higher amount of  $\text{Y}_2\text{O}_3$  addition (5 wt.%) influences the degree of densification of the material, obtaining a density of 3.23 g/cm<sup>3</sup> (relative density higher than 93% from theoretical density). The sintering time was the same (5 min.) for both types of materials.

### 3.2. Mineralogical composition

The mineralogical composition of the two materials was determined by X-ray diffraction and the spectra obtained are shown in Figures 1 and 2. Elemental cell parameters, average crystallite size and crystallographic phases for the two composite materials A and B sintered by SPS are shown in Table 3.

Taslicukur et al. [10], made  $\text{Si}_3\text{N}_4$  composites with SiC with additions of AlN and  $\text{Y}_2\text{O}_3$  (with different wt.%) by sintering in SPS in nitrogen atmosphere, at 40 MPa pressure for 5 min and showed that transformation of  $\alpha\text{-Si}_3\text{N}_4$  phase to  $\beta\text{-Si}_3\text{N}_4$  is initiated as early as 1400-1500°C. Khajelakzay et al. [17] made  $\text{Si}_3\text{N}_4\text{-SiC}$  micro-nano composites with  $\text{MgSiN}_2$  additions, also by sintering in SPS and in nitrogen atmosphere, at a pressure of 40MPa, for 400s and showed that the transformation of  $\alpha\text{-Si}_3\text{N}_4$  phase to  $\beta\text{-Si}_3\text{N}_4$  is initiated at 1400°C and increases at 1600°C and reaches 98 vol.% at 1800°C [17].

From the semi-quantitative Rietveld analysis (Table 3) and the X-ray diffractograms shown in Figures 1 and 2, it can be seen that for both sample A and sample B, at the sintering temperature of 1600°C, the transformation of the  $\alpha\text{-Si}_3\text{N}_4$  phase into  $\beta\text{-Si}_3\text{N}_4$  is more than 50 wt% and reaches more than 77 wt% (sample B) at 1800°C, while SiC remains constant in the matrix as a secondary phase.

### 3.3. Morphological characterization

Electron microscopy images of the two samples A and B sintered by SPS in vacuum are

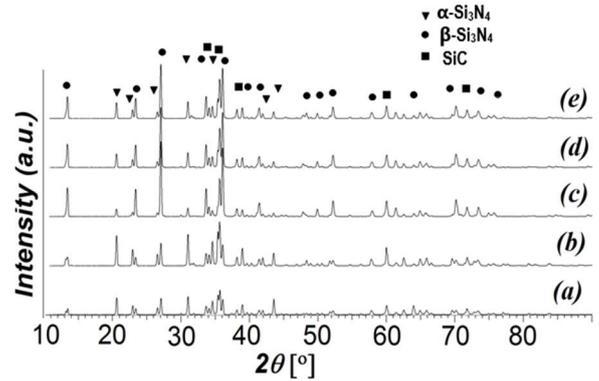


Fig. 2 - X-ray diffraction spectra of samples from composite material B sintered at / Spectrele de difracție de raze X a probelor din materialul compozit B sinterizate la: (a) 1600°C, (b) 1650°C, (c) 1700°C, (d) 1750°C, (e) 1800°C

shown in Figures 3 and 4. SEM analysis of composition A samples (Figure 3) sintered at 1600°C shows a bimodal pore microstructure consisting of elongated prismatic  $\beta\text{-Si}_3\text{N}_4$  grains with sizes between 1.5 - 2  $\mu\text{m}$ , surrounded by small rounded  $\alpha\text{-Si}_3\text{N}_4$  grains. With increasing sintering temperature up to 1800°C

(Figure 3 (c)), a significant increase in  $\beta\text{-Si}_3\text{N}_4$  [2] grain size up to 3 - 3.5  $\mu\text{m}$ , surrounded by small SiC grains can be observed. The presence of irregularly shaped pores larger than 1.5  $\mu\text{m}$  is also observed.

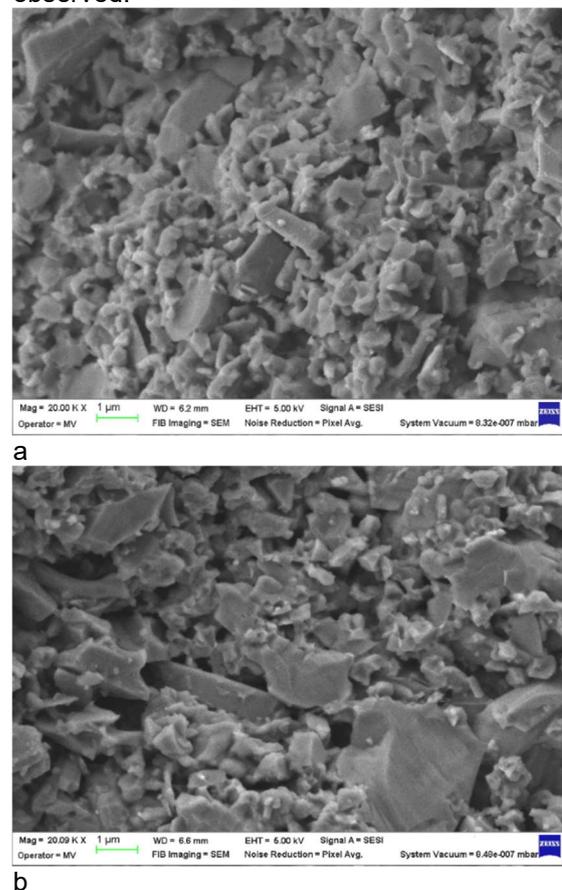
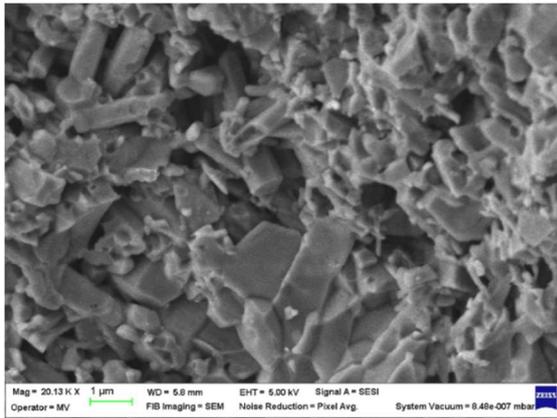


Fig. 3 continues on next page



C

Fig. 3 - SEM micrographs of sample A sintered by SPS in vacuum / *Micrografii SEM ale probei A sinterizată prin SPS, în vid: (a) 1600°C; (b) 1700°C; (c) 1800°C*

SEM microscopy images of sample B (Figure 4) show that the particle distribution is more homogeneous and dense, with small pores ( $<1.5 \mu\text{m}$ ) compared to sample A. The microstructure of sample B (Figure 4 (a)) is composed of prismatic  $\beta$ -  $\text{Si}_3\text{N}_4$  grains around  $1\text{-}1.2 \mu\text{m}$  and distributed among rounded  $\alpha$ -  $\text{Si}_3\text{N}_4$  grains of  $0.5 \mu\text{m}$ . Irregularly distributed  $\text{SiC}$  grains are also observed in the matrix of composite B material, which may lead to the limitation of  $\text{Si}_3\text{N}_4$  grain growth [12, 20].

Increasing the amount of dopant in the case of sample B, i.e. 5wt.%  $\text{Y}_2\text{O}_3$ , may influence the microstructure by reducing the grain size while increasing the sintering temperature. At  $1700^\circ\text{C}$  sintering temperature (Figure 4(c)), the grain sizes of  $\beta$ -  $\text{Si}_3\text{N}_4$  grains decrease to about  $1\mu\text{m}$ , being placed among small round  $\alpha$ -  $\text{Si}_3\text{N}_4$  grains with a diameter smaller than  $0.5 \mu\text{m}$ .

The addition of 1.5%  $\text{Al}_2\text{O}_3$  and 5%  $\text{Y}_2\text{O}_3$  to the composition of sample B contributed to the formation of a dense structure with fewer pores and a relative density of 93.10%.

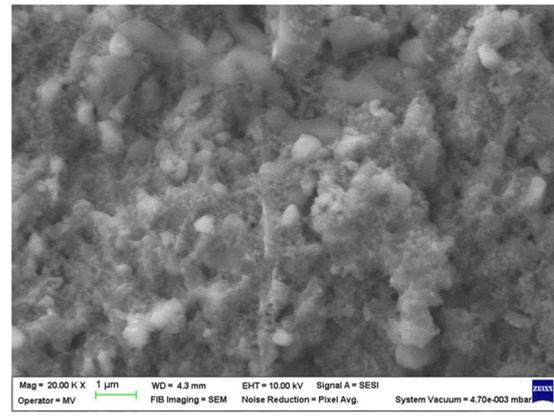
Although the transformation of  $\alpha$ - $\text{Si}_3\text{N}_4$  to  $\beta$ - $\text{Si}_3\text{N}_4$  appears to be more pronounced for type A composites, the physical and mechanical property results for type B composites are better and are comparable to those reported in the literature [3, 8].

As can be seen from the SEM images shown in Figures 3 and 4, sample A sintered in gaseous atmosphere ( $\text{N}_2$ ) [18], showed a higher degree of sintering than that obtained by sintering in vacuum.

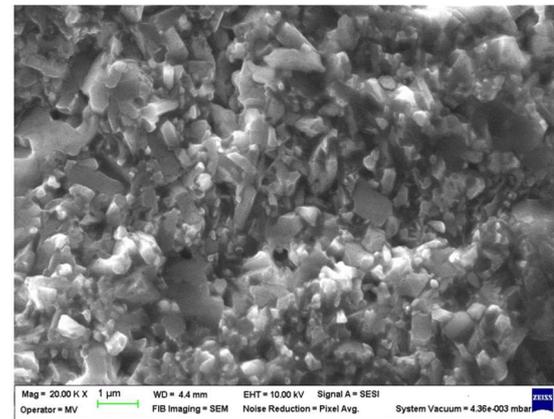
### 3.4. Mechanical Properties

Analyzing the results obtained from the mechanical testing of composite samples A and B, large differences are observed between the two compositions, depending on the sintering temperature. These results are in agreement with the results of the microstructural evaluation.

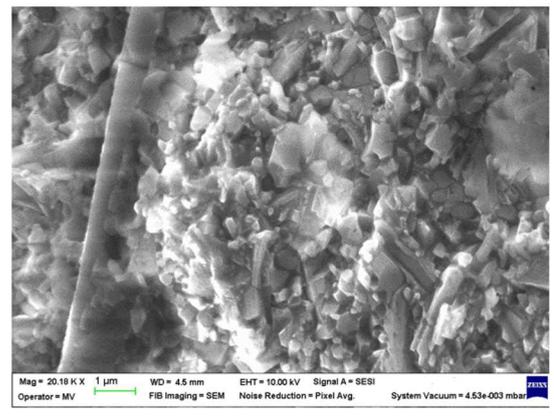
Type A composite samples showed Vickers hardness values between 3-7 GPa, while Type B composites showed Vickers hardness values



a



b



C

Fig. 4 - SEM micrographs of sample B sintered by SPS in vacuum / *Micrografii SEM ale proba B sinterizată prin SPS, în vid: (a) 1600°C; (b) 1700°C; (c) 1800°C*

between 17-19 GPa, values comparable to those reported in the literature [2, 8].

Also, the values of bending strength ( $R_m$ ) for sample A were about 240 MPa and 336 MPa for sample B and Young's modulus ( $E$ ) for sample A were about 214 GPa and 353 GPa for sample B, respectively, values obtained on samples sintered at  $1800^\circ\text{C}$ .

The presence of  $\text{SiC}$  in the two samples, due to the different expansion coefficient compared to the base matrix, can lead to the appearance of microcracks. Thus, the composite B samples sintered at the lowest temperatures (1600 and  $1650^\circ\text{C}$ ) showed a high degree of brittleness, most.

Table 4

Mechanical properties of studied composite ceramic materials  
Proprietățile mecanice ale materialelor ceramice compozite studiate

Sample code	Temperature	Vickers hardness, [GPa]
A	1600°C	3.77 ± 0.1
	1650°C	5.07 ± 0.14
	1700°C	5.43 ± 0.13
	1750°C	6.46 ± 0.16
	1800°C	6.92 ± 0.17
B	1600°C	-
	1650°C	-
	1700°C	17.55 ± 0.41
	1750°C	17.93 ± 0.44
	1800°C	18.39 ± 0.37

probably induced also by the presence of SiC particles, and also a low degree of phase transformation  $\alpha \rightarrow \beta$ , and could not be mechanically tested.

The results obtained from the mechanical testing of type B composites are comparable to those obtained previously [18] in terms of Vickers hardness, but the values of fracture toughness and Young's modulus are lower, which is possible due to the lack of N<sub>2</sub> atmosphere during sintering, the fact that  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> was not completely transformed, and the presence of SiC grains.

### 3. Conclusions

The aim of this work was to obtain, by the spark plasma sintering technique, two types of Si<sub>3</sub>N<sub>4</sub> and SiC based composite materials with different sintering additions Al<sub>2</sub>O<sub>3</sub> and Y<sub>2</sub>O<sub>3</sub> and with improved mechanical properties. The sintering process took place in vacuum at different temperatures in the range 1600°C-1800°C, at a pressure of 50 MPa, a heating rate of 100°C/min and a holding time of 5 min.

The processing conditions through the SPS technique in vacuum allowed to obtain Si<sub>3</sub>N<sub>4</sub> and SiC based composite materials with sintering additions of Al<sub>2</sub>O<sub>3</sub> and Y<sub>2</sub>O<sub>3</sub>, with improved physical-mechanical properties. Thus, for composite material B sintered in vacuum at 1800°C for 5 minutes at a pressure of 50 MPa, the following physical-mechanical properties were obtained: a high relative density (93.1%), a high Vickers value (18.39 GPa) and a bending strength of 336 MPa due to both microstructural homogeneity and a high degree of compaction. Both ceramic composite materials showed microstructures consisting of  $\beta$ -Si<sub>3</sub>N<sub>4</sub> grains with a prismatic shape,  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> grains and also SiC grains in a small amount.

The novelty of the work is that Si<sub>3</sub>N<sub>4</sub>/SiC-based ceramic composites were obtained using the spark plasma sintering (SPS) technique in vacuum. Although for sample A, obtained by both vacuum and N<sub>2</sub> sintering, the mechanical properties values were lower after vacuum sintering compared to those

obtained after N<sub>2</sub> sintering (e.g., HV in vacuum was ~ 6.92 GPa vs. ~ 19.9 GPa in gas [18]), it was shown that by increasing the percentage of sintering additions (in sample B), the results obtained in terms of mechanical properties for sample B sintered in vacuum (Vickers hardness ~ 18.39 GPa), are close to those previously obtained on type A samples in N<sub>2</sub>. The advantages of this vacuum sintering method is that Si<sub>3</sub>N<sub>4</sub>/SiC-based composite ceramic materials can be obtained with mechanical properties similar to those obtained in N<sub>2</sub> atmosphere.

The results obtained by the vacuum SPS technique suggest that composite ceramic materials based on Si<sub>3</sub>N<sub>4</sub> with high mechanical properties can be obtained.

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