

## Effect of BN whisker-like additive on the flexural strength of Si<sub>3</sub>N<sub>4</sub> ceramics prepared by gel casting method

M. Vaseghi<sup>a</sup>, S. Baghshahi<sup>b,\*</sup>, M. Mashhadi<sup>c</sup> and A. Nemati<sup>d</sup>

<sup>a</sup>Department of Materials Engineering, Science and Research Branch, Islamic Azad University, Tehran, Iran

<sup>b</sup>Department of Materials Engineering, Faculty of Engineering, Imam Khomeini International University, Qazvin, Iran

<sup>c</sup>Faculty of Materials and Manufacturing Engineering, Malek Ashtar University of Technology, Tehran, Iran

<sup>d</sup>Department of Materials Science and Engineering, Sharif University of Technology, Tehran, Iran

The gel casting method was used for the production of Si<sub>3</sub>N<sub>4</sub> porous ceramics with 25 vol% solid loading, and the effect of adding 1 and 3 wt.% of BN whisker-like (synthesized in the laboratory) on the flexural strength of the composites was investigated. The gel-cast samples were prepared using an AM-MBAM monomer system and deionized water solvent. The gel cast bodies were dried at ambient temperature and humidity for 48hrs. The burnout process was carried out at 600 °C for 14hrs in the air. The samples were then subjected to heat treatment at 1650 °C for 2 hrs in a bed of 50% Si<sub>3</sub>N<sub>4</sub>-50% BN. Results showed that BN whisker-like were agglomerated in deionized water. To solve this problem and to properly disperse the whisker-like in the slurry, active surface agent groups were employed. Adding BN whisker-like to ceramic bodies, increased the porosity from 33% in samples without BN to 46% in samples containing 3 wt% BN whisker-like, resulting in a decrease in the density from 1.8 g/cm<sup>3</sup> to about 1.61 g/cm<sup>3</sup>. Since the Si<sub>3</sub>N<sub>4</sub> ceramics require aids for the sintering, SiO<sub>2</sub> and Y<sub>2</sub>O<sub>3</sub> additives were used with a ratio of 1 to 2, respectively, which led to the formation of Si<sub>2</sub>N<sub>2</sub>O phase. Si<sub>2</sub>N<sub>2</sub>O is similar to the Si<sub>3</sub>N<sub>4</sub> phase in terms of the thermal expansion coefficient and chemical properties. Because of its rod shaped morphology, it has a similar function to β-Si<sub>3</sub>N<sub>4</sub> phase in improving the mechanical properties of the ceramic. Although porosity in the ceramics containing 3 wt.% BN whisker-like was higher than those containing 1 wt.%, the porosity created vacant spaces for further growth of Si<sub>2</sub>N<sub>2</sub>O-rod shaped grains and in spite of higher porosity, better flexural strength was achieved for bodies with 3 wt.% BN whisker-like. Also, in bodies without BN whisker-like, the flexural strength was 88 MPa, due to the low porosity and the presence of stretched Si<sub>2</sub>N<sub>2</sub>O grains.

**Key words:** Si<sub>3</sub>N<sub>4</sub>-BN porous ceramic composites, Gel casting, Flexural strength, Si<sub>2</sub>N<sub>2</sub>O.

### Introduction

Silicon nitride is a unique material which has found many applications in the industry for its exceptional properties including high melting temperature, excellent mechanical properties, chemical stability against corrosive atmosphere and oxidation, low thermal expansion coefficient, excellent resistance to thermal shock, low density, and high hardness [1-4]. However, despite such desirable presence of these compounds leads to a drop in the properties of these ceramics at high temperatures and it is inevitable to use MgO, Al<sub>2</sub>O<sub>3</sub>, AlN, Yb<sub>2</sub>O<sub>3</sub>, and Y<sub>2</sub>O<sub>3</sub> individually or in various combinations for sintering at relatively low temperatures based on the ceramic forming method employed [5]. Accordingly, in the present study, SiO<sub>2</sub> and Y<sub>2</sub>O<sub>3</sub> additives were used with a 1:2 ratio as a sintering aid. Many studies have been reported on the application and effect of BN particles in Si<sub>3</sub>N<sub>4</sub> bodies. Palli et al. [6] synthesized

Si<sub>3</sub>N<sub>4</sub>-BN ceramic composites with gel-casting and pressureless method and studied the effect of BN particles on the microstructure of the composites. The results of their research revealed a uniform microstructure in the samples, in which porosities with an almost uniform size and distribution were dispersed and the main reason was attributed to the gel casting method. Also, XRD patterns showed that with an increase in the amount of h-BN, the intensity of α-Si<sub>3</sub>N<sub>4</sub> increased and the intensity of β-Si<sub>3</sub>N<sub>4</sub> phase decreased, and the α phase to β-Si<sub>3</sub>N<sub>4</sub> transformation was delayed. Yao et al. [7] produced Si<sub>3</sub>N<sub>4</sub>-BN porous composites by nitridation of Si powder. This resulted in an improvement of the mechanical strength which was attributed to the α to β phase transformation at 1680 °C, but addition of BN particles delayed the phase transformation and BN particles prevented complete phase transformation. The addition of BN also prevented the growth of grains and reduced the aspect ratio of β grains and resulted in their interlocking structure. In their study on the structure of Si<sub>3</sub>N<sub>4</sub>-BN composites, Gao et al. [8] found that with an increase in the amount of BN particles, smaller grains and more uniform microstructures were obtained, since

\*Corresponding author:  
Tel : +98-9122164225  
Fax: +98-283-3780073  
E-mail: baghshahi@eng.ikiu.ac.ir

the particles in the second phase prevented the growth of the grain in the composite system. In addition, due to the hexagonal structure of BN, thermal coefficient was anisotropic and this resulted in the tension and lamination across (001) planes, and this very stress led to improved mechanical properties of the composites. Wang et al. [9] synthesized samples using gel casting and BN particles and observed that through increasing BN, the porosity increased and as a result of this, the density, fracture toughness, and flexural strength decreased. Long et al [10] reported that compared to monolithic porous ceramics, microstructure and mechanical properties of the Si<sub>3</sub>N<sub>4</sub>-BN-SiO<sub>2</sub> porous composites featured lower elastic modulus and higher strength, toughness, and porosity. In fact, the Si<sub>3</sub>N<sub>4</sub>-BN-SiO<sub>2</sub> composite showed high fracture strength (130MPa) and low elastic modulus (66GPa) in their study. As a result of their elongated morphology, BN whisker-like may create barriers to the growth path of the cracks. It was, therefore, hypothesized that BN whisker-like may improve the mechanical properties and particularly flexural strength of Si<sub>3</sub>N<sub>4</sub> ceramic composites. A review of the relevant literature reveals that the effect of the presence of BN whisker-like on the flexural strength of Si<sub>3</sub>N<sub>4</sub> ceramics has not been investigated yet. Thus, the present study was an attempt to investigate the effect of BN whisker-like on the bending strength of Si<sub>3</sub>N<sub>4</sub> composites. The gel casting method has recently been extensively used for the purpose of forming porous ceramics with special applications. Uniform distribution of porosity, low cost, the availability of raw materials and forming mold, the capability of forming complex shapes and low dimensional tolerance are among the most important features of this method [11].

## Experimental Procedure

### Materials

In this study,  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> powder (purity  $\geq 99.9$  wt%;  $d_{50} = 0.5 \mu\text{m}$ , China) and h-BN whisker-like (made in the laboratory based on reference [12]) were used as the matrix and reinforcement, respectively. SiO<sub>2</sub> (Quartz, cas number: 18640, Riedel, Germany) and Y<sub>2</sub>O<sub>3</sub> (purity  $\geq 99.9$  wt%;  $d_{50} = 0.5 \mu\text{m}$ , China) were used as sintering aids. Acrylamide (AM), methylenebisacrylamide (MBAM), and deionized water were used as monomer, cross linker and solvent, respectively. Tetramethylethylenediamine (TEMED), ammonium persulfate (APS) (both of Merck), and Dolapix 88 (Zschimmer & Schwarz) were used as catalyst, initiator, and dispersant, respectively. In order to adjust the slurry pH, 28% ammonia solution (Merck) was used.

### Methods

The weight ratio of the additives was Y<sub>2</sub>O<sub>3</sub>:SiO<sub>2</sub>=2:1. To investigate the effect of h-BN whisker-like on the mechanical strength of the ceramic composite, h-BN

**Table 1.** Composition of the samples.

Sample	Composition (wt%)			
	Si <sub>3</sub> N <sub>4</sub>	SiO <sub>2</sub>	Y <sub>2</sub> O <sub>3</sub>	BN
1	90	3.5	6.5	0
2	89	3.5	6.5	1
3	87	3.5	6.5	3
4	84	3.5	6.5	6

whisker-like was added at 1, 3, and 6 wt%. Si<sub>3</sub>N<sub>4</sub> powder was poured in a polyethylene jar with isopropanol for 24 hrs to dry in the ambient temperature. In order to make a slurry containing 25 vol.% solid loading, the premix solution was mixed with 1 wt% Dolapix 88 as dispersant and AM and MBAM monomers with an AM/MBAM ratio of 1.3 in distilled water. After preparing the premix solution, the ceramic powders were mixed and homogenized in an opal mortar for 10 minutes and added to the premix based on Table 1. The resulting slurry was placed in a ball mill to homogenize for 24 hrs. Then the initiator and the catalyst were added to the slurry and mixed well using a mechanical stirrer for 1 minute. The slurry was immediately cast into a polyethylene mold. After gelation, which lasted about an hour at the temperature of 60 °C, the gel samples were removed from the mold and dried at ambient temperature and humidity for 48hrs. For burnout, the heat treatment was performed at the heating rate of 1 °C/min up to 600 °C and a soaking time of 4 h. For sintering, the samples were heated at 1650 °C for 2 hrs. The heating rate was 10 °C/min. The weight composition of the samples is presented in Table 1.

### Characterization

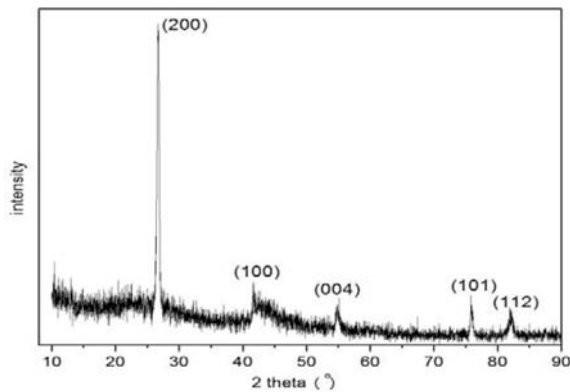
To obtain a suitable pH for slurry, Zeta potential test was used by RXE analyzer (RXE Zeta 965). The density of the samples was measured using Archimedes method (three samples per batch). The X-ray diffractometer (XRD-3003PTS, SEIFERT) was used for identification of the phases. Scanning electron microscope (SEM VEGA II) was used to study the microstructure of the samples. Three-point flexural strength test (Zwice) with loading speed of 0.2 mm / min was used on the samples of 25 mm  $\times$  3 mm  $\times$  50 mm size for 3 samples of each batch to evaluate the strength of the samples.

## Results and Discussion

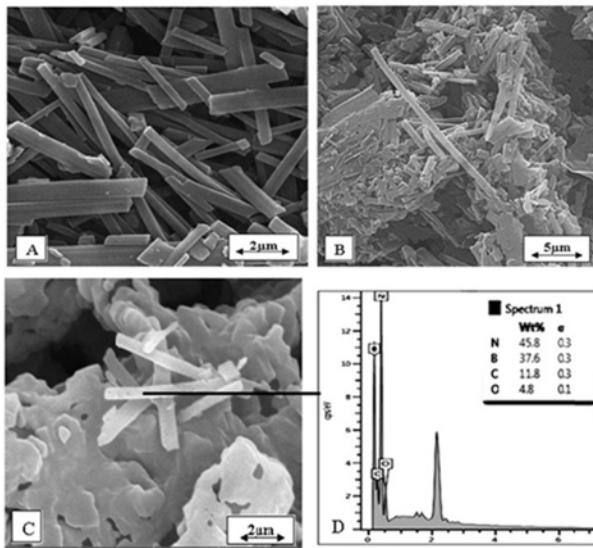
### Fabrication of BN whisker-like

Fig. 1 shows the X-ray diffraction pattern of the BN whisker-like (JCPDS90-0012), fabricated in the present research. As observed no minor phase was detected. The pattern corresponds to h-BN with the network parameters of  $a = b = 2.504 \text{ \AA}$  and  $c = 6.661 \text{ \AA}$ .

Fig. 2 shows the microscopic image of the BN whisker-like and the corresponding EDS analysis. As



**Fig. 1.** XRD pattern of the BN whisker-like synthesized in a laboratory at 1700 °C.

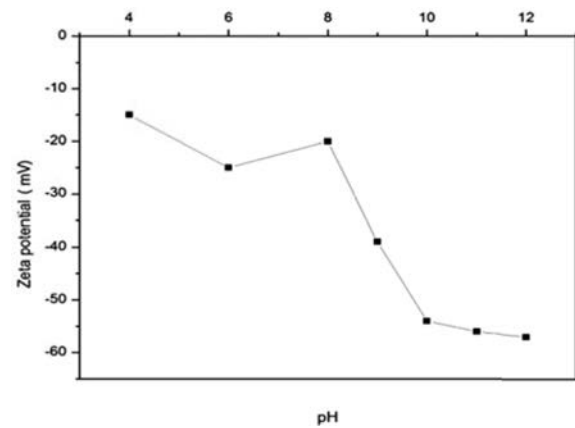


**Fig. 2.** SEM image with different magnifications of the synthesized BN whisker-like in A and B; D shows the EDS analysis of the point indicated in C.

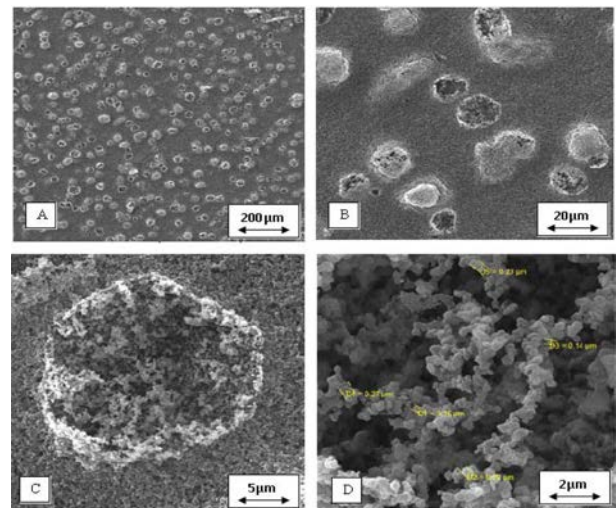
observed the whisker-like indicates that the BN is both a whisker-like and a micrometer-sized particle. The identification of carbon and oxygen in the EDS analysis can be due to the unreacted raw materials and the contamination from the graphite crucible used in heat treatment. Since the BN has a hexagonal structure, the growth of the grains occurred easier in the direction of the plate (0001) to form rod like particles.

### Zeta potential measurement

Zeta potential test results are presented in Fig. 3 to determine the range of stability of  $\text{Si}_3\text{N}_4$  suspensions. The slurry pH plays an important role in its rheology. As observed, the zeta potential increased with pH. When the slurry pH increases, its viscosity often increases rapidly. This is due to the hydroxide layer on the particle surface. Therefore, a dispersant is added to control the dispersion of powder particles in the slurry



**Fig. 3.** Zeta potential changes based on pH.



**Fig. 4.** SEM image of BN whisker-like dispersed in deionized water at various magnifications.

at high alkali pHs [13]. Adding dispersants increases the zeta potential [6]. Based on the results obtained from the zeta potential measurement, the best pH for the slurry was determined within the range of 10-12, in which the particles remained stable in the slurry. Therefore, the pH of the premix was adjusted within this range.

### Preparation of the slurry for gel casting

When preparing the slurry for gel casting, there is a problem of increasing the viscosity of the slurry and the agglomeration of BN whisker-like, when BN whisker-like is added to solid loading. Due to the low density of BN whisker-like, even addition of 1 wt.% BN, considerably increases the viscosity. Therefore, when BN whisker-like were added to the solid slurry of 6 wt%, the viscosity increased so much that it did not drain out of the jar after 24 hrs of ball milling. Thus, 6 wt.% BN whisker-like were not used in the study.

The problem of increase in the viscosity of the slurry

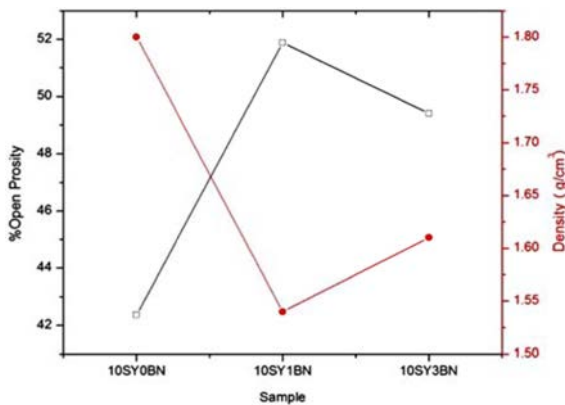


Fig. 5. The bulk density and porosity percentage of  $\text{Si}_3\text{N}_4$ -BN composites containing 0, 1 and 3 wt% BN whisker-like.

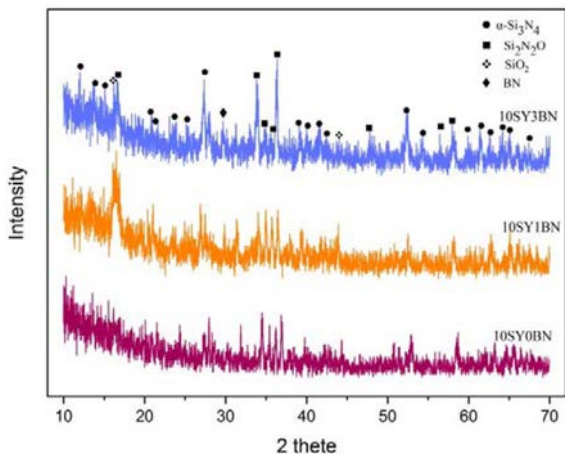


Fig. 6. XRD pattern of 10SY0BN, 10SY1BN and 10SY3BN.

after adding BN whisker-like, attracted the attention towards the microscopic dispersion of the whisker-like in deionized water. The microscopic images (Fig. 4) showed that the BN whisker-like became agglomerated in the water. In order to prevent agglomeration of the whisker-like, their surface must be coated with an agent to avoid agglomeration in contact with water. Therefore, using ammonia and oleic acid, the whisker-like surfaces were functionalized to prevent agglomeration.

#### Bulk density and porosity measurement

The bulk density ( $\rho_b$ ) of  $\text{Si}_3\text{N}_4$  porous ceramics and their open porosity were calculated using the Archimedes method. Results of density and porosity calculations are presented in Fig. 5. As the figure suggests, with an increase in BN, the porosity of the samples also increased and as a result of this, their density decreased. Due to the incompatibility of the chemical activity of the matrix and the secondary phase, no chemical reaction occurs during sintering between the matrix phase particles and the secondary phase (BN whisker-like in this case). Therefore, there

remains some space around the reinforcing phase. Particularly when the secondary phase are whisker-like, a gap is created between the matrix and the whisker-like during fabrication of ceramic composites.

Increased porosity and the subsequent decrease in the density can also be attributed to the presence of  $\text{SiO}_2$  both on the surface of  $\text{Si}_3\text{N}_4$  particles and in the added sinter aid as  $\text{SiO}_2$  prevents liquid penetration during the dissolution-sedimentation process and eventually a lot of large openings remain among the particles of the main phase of the matrix. In such a composite with a porosity percentage equal to that in a monolithic  $\text{Si}_3\text{N}_4$  sample, the strength and toughness of the fracture and the elastic modulus decrease due to the weak interface between the  $\text{Si}_3\text{N}_4$  and the secondary phase and the internal bonding of the porosity, which naturally increases the porosity by increasing the amount of BN or as a result of the presence of  $\text{SiO}_2$  [10, 14].

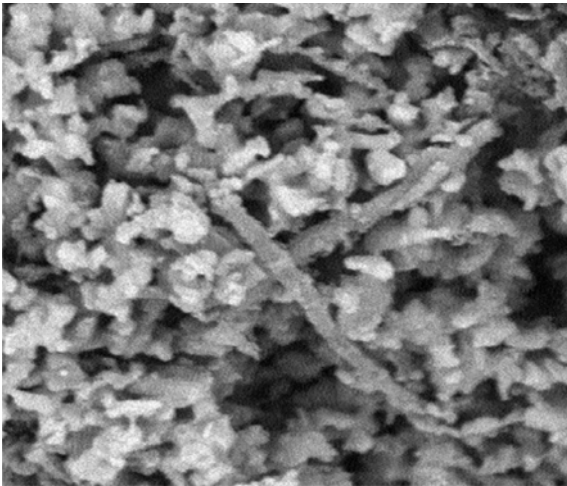
#### XRD phase analysis of the sintered samples

The XRD pattern of porous composites are shown in Fig. 6. Specific peaks for detecting the  $\text{Si}_3\text{N}_4$  phase were in accordance with (JCPDE 72-1253) and belonged to  $\alpha$ - $\text{Si}_3\text{N}_4$ , while no  $\beta$ - $\text{Si}_3\text{N}_4$  phase was observed. A close inspection of the X-ray diffraction patterns of the samples 10SY1BN and 10SY3BN indicates that when  $\text{SiO}_2$  and  $\text{Y}_2\text{O}_3$  sintering aids were used, other phases such as were also formed. This is probably due to the fact that the thermal expansion coefficient of these phases particularly those of  $\text{Si}_2\text{N}_2\text{O}$ , were closer to the  $\alpha$ - $\text{Si}_3\text{N}_4$  main phase.  $\beta$ - $\text{Si}_3\text{N}_4$  did not form, because as a result of the sintering aids a liquid phase was formed which thermodynamically favored the formation of  $\text{Si}_2\text{N}_2\text{O}$  in the expense of  $\beta$ - $\text{Si}_3\text{N}_4$  [15]. In these specimens,  $\text{SiO}_2$  (quartz) was also detected which might be due to the remaining  $\text{SiO}_2$  added as sintering aid.

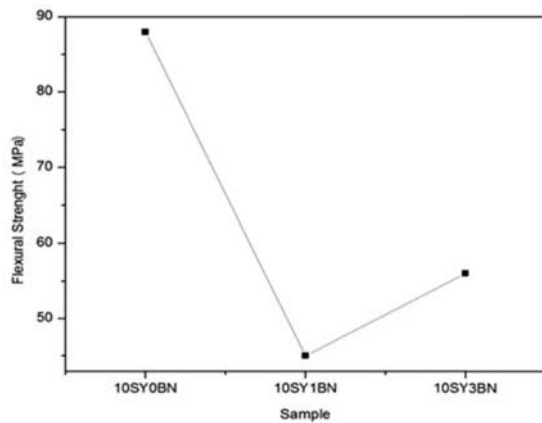
#### Scanning electron microscopy (SEM)

Fig. 7 shown the microstructure of composite ceramics prepared by gel-casting which are taken from the fracture cross-section of the 10SY1BN specimen can be attributed to  $\text{Si}_2\text{N}_2\text{O}$ . When the heat treatment is carried out for sintering,  $\text{Si}_3\text{N}_4$  and  $\text{SiO}_2$  initially react with  $\text{Y}_2\text{O}_3$  in the form of eutectics between Si-O-N-Y and Si-O-Y and form  $\text{Y}_2\text{Si}_2\text{O}_7$  and  $\text{Y}_2\text{Si}_3\text{O}_3\text{N}_4$  phases [16]. However, with increasing the temperature, the Si-O-N-Y eutectic product i.e.,  $\text{Y}_2\text{Si}_3\text{O}_3\text{N}_4$  decomposes and the relatively stable  $\text{Y}_2\text{Si}_2\text{O}_7$  phase remains. Nucleation and growth of  $\text{Si}_2\text{N}_2\text{O}$  in the  $\text{SiO}_2$ -rich liquid phase occurs when the temperature of the sinter is close to  $1700^\circ\text{C}$  and because it consumes a large amount of the liquid phase, it is believed to increase the resistance to viscous flow, especially because it has a greater specific surface area due to its elongated morphology.

On the other hand, transformation from  $\alpha$  to  $\beta$ - $\text{Si}_3\text{N}_4$



**Fig. 7.** SEM image of interlocking grains of  $\text{Si}_2\text{N}_2\text{O}$  in 10SY1BN specimen.



**Fig. 8.** Flexural strength of  $\text{Si}_3\text{N}_4/\text{BN}$  composite samples containing 0, 1 and 3 wt% BN whisker-like.

stops in an oxygen-rich environment and the process of transformation becomes difficult; transformation of  $\alpha$  into  $\beta$ - $\text{Si}_3\text{N}_4$  occurs through the transfer of Si and N in the liquid phase, and since the dissolution of oxygen in the  $\alpha$  network is greater than in  $\beta$  [15], the easier introduction of oxygen into the  $\alpha$  network prevents  $\alpha$ - $\beta$  transformations and the  $\beta$  seeds are not able to grow. Therefore, as evidenced in the microscopic images and XRD patterns,  $\alpha$ - $\beta$  transformation did not occur and the main phase is in situ  $\alpha$ - $\text{Si}_3\text{N}_4$  and  $\text{Si}_2\text{N}_2\text{O}$ .

### Analysis of the flexural strength

Fig. 8 shows the flexural strength of porous ceramics composites as a function BN whisker-like. The flexural strength porous ceramics strongly depends on the size and distribution of their porosity. Porosity has a significant effect on strength, so that with increasing porosity, ceramic strength decreases exponentially [14]. Therefore, high strength values are not expected in

porous ceramics especially in samples with low solid content, and in the gel cast products usually become porous after binder removal.

Another factor affecting mechanical properties of ceramics is the thermal stress in the ceramic composites. When phases with different thermal expansion coefficients are formed during the sinter or exist in the body, such as the secondary phase of BN, they cause thermal stress during cooling. If the difference between the thermal expansion coefficients of the primary phase and the secondary phases is not very high, the created thermal stress can cause deviation in the cracking path during crack growth. Crack energy may also be used to overcome the thermal stress energy and prevent its propagation.

Analysis of the results of the flexural strengths of the samples shown in Fig. 8 makes it clear that the presence of BN whisker-like reduced the flexural strength, which is in conformity with the amount of the porosity observed in the specimens because of the presence of BN whisker-like. Since the bond between BN and  $\text{Si}_3\text{N}_4$  is weaker than that of  $\text{Si}_3\text{N}_4$  grains, by increasing the amount of BN whisker-like in samples, the propagation of cracks requires less energy. Porosity can also be considered as a weak boundary phase (WBP) and therefore, the effect of porosity on flexural strength can be considered as the effect of BN. Naturally, these weak boundary phases act as sources of failure and reduce the strength [7].

On the other hand, the formation of  $\text{Si}_2\text{N}_2\text{O}$  rods in the matrix phase of  $\alpha$ - $\text{Si}_3\text{N}_4$  led to a high strength in the sample that grew randomly and with their cross-linking structure resists against crack propagation. In fact, the interlocking structure of  $\text{Si}_2\text{N}_2\text{O}$  rods is clearly observed in the microscopic images of their breakdown surface. Comparison of the strength of the 10SY1BN sample with 10SY3BN suggests that although the porosity of the two samples was approximately equal (51.88 and 50.43, respectively), the increase in the strength of the 10SY3BN sample can be attributed to the formation of more  $\text{Si}_2\text{N}_2\text{O}$  compound. Since the presence of porosity provides more space for the growth of  $\text{Si}_2\text{N}_2\text{O}$  rods, comparing the XRD patterns of the 10SY1BN and 10SY3BN samples shows that the intensity of the  $\text{Si}_2\text{N}_2\text{O}$  phase in the 10SY3BN sample increased and this increases the probability of forming more  $\text{Si}_2\text{N}_2\text{O}$  rods and this idea is supported by the results of flexural strength analysis. Therefore, there are two theories regarding the relationship between the porosity and flexural strength. The first approach, generally states that porosity reduces strength. On the other hand, when the increase in porosity leads to the increased strength, according to the second theory, the cracks that face porosity during their growth, stop or deflect in one way or the other, and therefore, an increase is observed in the samples' strength.

## Conclusions

Gel casting method was used to make porous composites of  $\text{Si}_3\text{N}_4$ -BN in order to obtain a uniform distribution of porosity in the body. Because BN whisker-like were of nano-scale, they increased the slurry viscosity. Also, whisker-like were agglomerated in an aqueous environment and their dispersion was very difficult in the slurry. Therefore, the whisker-like surface was functionalized to facilitate their dispersion in the aqueous environment. Dry-gel bodies were burned-out at 600 °C for the organic material to exit and sintered at 1650 °C for 2hrs. Result of the density analysis showed that the increase in the amount of BN whisker-like led to an increase in porosity, which in addition to reducing the density, led to a decrease in flexural strength. This phenomenon was attributed to the poor interfacial bounds between  $\text{Si}_3\text{N}_4$  and BN. The presence of BN prevented the grain growth and the transformation of  $\alpha$ - $\text{Si}_3\text{N}_4$  to  $\beta$ - $\text{Si}_3\text{N}_4$ . Porosity, on one hand, reduced the flexural strength, but on the other hand, provided a good space for the growth of the  $\text{Si}_2\text{N}_2\text{O}$  phase, which had a rod morphology and helped to improve the flexural strength.

## References

1. B.J. McEntire, R. Lakshminarayanan, P. Thirugnanasambadam, J. Seitz-Sampson, R. Bock, D. O'Brien, *Bioceram Dev Appl.* 6 [1] (2016) 1-9.
2. J. Z. Jiang, F. Kragh, D.J. Frost, K. Stahl, H. Lindelov, *J. Phy.: Con. Matter.* 13 [22] (2001) 515.
3. X. Zhu, Y. Sakka, *Sci. Technol. Adv. Mater.* 9: 033001 (2008) 1-47.
4. M.K. Park, H.N. Kim, K.S. Lee, S.S. Baek, E.S. Kang, Y.K. Baek, D.K. Kim, *Key Eng. Mat.* 287 (2005) 247-252.
5. D. Tuyen, Y. Park, H. Kim, B. Lee, *Ceram. Int.* 35 (2009) 2305-2310.
6. N. ThallaPalli, K.K. Kumar, C.S.P. RAO, *Mat. Sci. Res. India* 13[1] (2016) 28-33.
7. D. Yao, Y.P. Zeng, K.H. Zuo, D. Jiang, *J. Am. Ceram. Soc.* 94 [3] (2011) 666-670.
8. L. Gao, X. Jin, J. Li, Y. Li, J. Sun, *Mat. Sci. & Eng. A* 415 (2006) 145-148.
9. S. Wang, D. Jia, Z. Yang, X. Duan, Y. Zhou, *Cer. Int.* 3 (2013) 4231-4237.
10. N. Long, J. Bi, W. Wang, M. Du, Y. Bai, *Cer. Int.* 38 (2012) 2381-2387.
11. R. Gilissen, J.P. Erauw, A. Smolders, E. Vanswijgenhoven, J. Luyten, *Mat. & Desi.* 21 (2000) 251-257.
12. Z. Lin, W. Hongkang, X. Zhipeng, W. Changan, *J. of The Chin. Cer. Soc.* 42[7] (2014) 831-835.
13. K.K. Kandil, S.K. Pal, C.S.P. Rao, *IOP Conf. Series: Mat. Sci. & Eng.* 149 (2016) 1-8.
14. X. Yang, B. Li, C. Zhang, S. Wang, K. Liu, C. Zou, *Mat. Sci. & Eng. A* 663 (2016) 174-180.
15. S. Wang, Z. Yang, X. Duan, D. Jia, F. Ma, B. Sun, Y. Zhou, *Int. J. Appl. Ceram. Technol.* 11[5] (2014) 832-838.
16. H. Yue, Z. Wang, J. Tian, *Ceram. Int.* 40 (2014) 8525-8532.