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Full Length Research Paper

Heat treatment of Al_2O_3-SiC-MgO nanoceramic and optimizing the fracture performance

Ali Asadi*, Ali Sadough Vanini and Amin Jabbari

Department of Mechanical Engineering, Amirkabir University of Technology (Tehran Polytechnic), 15914 Tehran, Iran.

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In this study, the procedure of sintering and heat treatment of Al_2O_3-SiC-MgO nanoceramic was considered. For improving the densification behavior and mechanical properties, experimental variables, such as sintering temperature and duration, which significantly influence the fracture performance of this nanocomposite body, were optimized. To obtain desired properties, several tests were conducted on the nanoceramic materials produced under different conditions to achieve the best physical and mechanical properties. It was observed that for specimen with 10 vol% *SiC*, sintering temperature 1650°C, and sintering duration of 90 min and sintering pressure of 30 MPa, the fracture performance of nanoceramic has been improved. Furthermore, samples produced in the mentioned conditions, were conducted under heat treatment at several temperatures from 1200°C up to 1600°C. Fracture toughness of these samples were measured and analyzed. Remarkable increase in fracture toughness was observed with increasing annealing temperature. To achieve best performance against impact, optimum heat treatment temperatures were presented.

Key words: Nanoceramic, sintering parameters, heat treatment, microcracking, fracture toughness.

INTRODUCTION

Performance of a material against impact depends on some physical and mechanical properties, such as high compressive strength, enough flexibility, lightness and resistance to penetration. Usually these properties cannot be found together in one material. However conventional ceramics are known as a good choice of these impact resistant targets, but ceramics are so brittle. To overcome the brittleness of conventional ceramics, new technique concepts for producing ceramic nanocomposites were developed (Niihara et al., 1988, 1989; Niihara, 1991). There is a way to improve the strength and toughness of ceramics, uniformly dispersing of second phase nano-particles in a pure ceramic matrix (Davidge et al., 1973). Niihara et al. (1991) proposed new material, designed significantly to improve the strength of the material by dispersing second phase nanoparticles within the alumina matrix. Unlike common composites which second phase particles were mainly located on the boundaries, SiC particles were mainly located within alumina matrix grains, because of phase transformation of alumina from the γ to α (Wang et al., 2000). Thermal expansion coefficient of alumina at temperatures ranging from 0 to 1700°C is 8.58e-6 1/°C and that of SiC in the same temperature range is 4.7e-6 1/°C. This difference between alumina and SiC nano-particles make significant improvement in fracture toughness of the body. This improvement can be resulted from nanoscale cracks made in the alumina matrix. This nanoscale cracks causing growth of the frontal process zone (FPZ) size ahead of a crack tip and therefore, the fracture toughness of the material improved (Seong et al., 2005). A theoretical model for investigating thermal expansion anisotropy of the Al_2O_3 matrix and SiC particles has been presented. High strength could be achieved, because of two residual micro stresses, despite the fact that such micro stresses are distributed in the lattice rather than being localized at the grain boundaries (Pezzoti et al., 2001). Also, small amount of MgO had a significant effect on improving the densification and mechanical properties

*Corresponding author. E-mail: Taban1293@yahoo.com or sadough@aut.ac.ir

of alumina nano-composite. This is resulted from decreasing the grain size of the alumina matrix (Wang et al., 1998; Rittidech et al., 2006; Takayasu et al., 2010). Wang et al. (1998) investigated the effects of MgO on the Al_2O_3 -5 vol% SiC ceramic. They showed that sintering density and mechanical properties of the Al_2O_3-SiC ceramic material were improved by adding a small amount of MgO. In this study several tests were conducted at different chemical composition, sintering temperature, duration, pressure and also different annealing temperatures. In fact this work is aimed to investigate the effects of experimental variables of sintering and in consequence heat treatment conditions on physical and mechanical properties of the nanoceramic body and choosing the optimum condition to improve the fracture performance.

EXPERIMENTAL PROCEDURE

Alumina powder used in this study is in γ phase and purity of 99.97% and maximum grain size of 3 µm. Also SiC powder with purity of 98% and MgO powder with maximum grain size of 80 nm were utilized. Six different mixtures of starting powders Al_2O_3 and SiC, containing 0, 2.5, 5, 7.5, 10 and 15 vol% SiC, respectively, were mixed with 500 ppm MgO. The mixtures were milled in isopropyl alcohol environment for 3 h. Milled mixtures were initially dried in magnetic mixer at 100°C followed by dry mill at 130 °C for 3 h. Produced powder is compressed and shaped for 30 s at pressure of 20 bars. The powder compacts was carried out for sintering in a high temperature graphite furnace with argon inert gas and different pressures of 30, 20 and 10 MPa at various temperatures including 1600, 1650, 1700 and 1750 °C for duration of 1 to 1.5 h. Heating rate was controlled at 10°C/min, and after sintering, specimens were slowly cooled to the room temperature. Produced samples were cut and polished for performing mechanical impact tests.

The densities of the sintered nanoceramic samples were measured according to ASTM B311 standard (2002). Flexural strength of the samples were measured using three point flexural test based on ASTM C1161 standard (2008) which was done by 5 ton Zwick machine. To perform tests sintered samples were cut into rectangular bar specimens (3 × 4 × 45 mm). Hardness of the samples was measured using Vickers method based on the ASTM C1327 (2008) standard. Hardness tests were conducted by Vickers hardness machine model UV1 made by Koopa Ltd. Surface of specimens were polished using diamond paste with grain size of 30 to 1 µm. Hardness test was done at five point of each specimen and mean of 4 coherent results reported as Vickers hardness number.

Fracture toughness of material was calculated from previous measurements using the following equation (Anatis et al., 1981):

$$K_{1c} = \alpha \frac{P}{\sqrt{C^3}} \sqrt{\frac{E}{H}} \quad (1)$$

where *a* is a constant coefficient between 0.012 to 0.02 proposed by Anstis et al. (1981), c is the penny like radial/median crack length (mm), E is the Young's modulus (MPa), H is the Vickers hardness (MPa) and P is the indentation peak load (N).

To estimate the Young's modulus of material, the slope of the stress-strain measurements from the three point test of flexural strength based on the ASTM D790 standard (2007) was used. Another method which tolerable matches with the aforementioned method is to use equation 2 for nanocomposite by substituting Young's modulus of comprised Al_2O_3 and SiC.

$$E = \sum E_i . V_i \quad (2)$$

For first step of optimization process, 24 specimens containing 500 ppm MgO and 6 different SiC mixtures at 4 different sintering temperatures were produced at sintering pressure of 30 MPa and duration of 90 min. With investigating the test results for these 24 specimens, optimum sintering temperature and SiC vol% obtained. For optimizing the sintering duration, 2 specimens were made at optimized sintering temperature and SiC vol% with duration of 1 and 1.5 h. Sintering temperature still was considered 30 MPa, respectively. The proper sintering duration was attained with studying the physical and mechanical properties of the samples at different sintering temperatures. Proper sintering pressure was attained by studying the samples at pressures 10, 20 and 30 MPa similar to sintering duration optimization. After these tests, optimum amount of sintering temperature, sintering duration, sintering pressure and SiC vol% based on proper mechanical and physical properties were attained.

A functional test was conducted on the samples which were prepared in these optimum conditions. This nanoceramic was used as front layer of a laminar ceramic/metal composite and subjected high speed impact. High velocity performance is compared with the case of using pure alumina as front layer. A steel projectile with 850 m/s velocity hits laminar composite target. Backing layer of Al2024-T6 is utilized for this experiment. Many tests were conducted on samples by different ceramic thickness and the results illustrate the practical benefits of utilizing proposed nanoceramic instead of conventional ceramic (Asadi et al., 2011).

After determining optimal sintering conditions, heat treatment experiments were performed on the samples. 10 nanoceramic samples were prepared in optimal sintering conditions and 2 samples were made from pure alumina in the same sintering condition. 8 samples of nanoceramic were subjected annealing heat treatment at temperatures of 1200, 1400, 1500 and 1600°C for duration of 2 h. The temperature increasing rate was 5°C/min and after staying for 2 h at mentioned temperatures, samples were cooled very slowly to room temperature in the furnace. Samples were cut in dimensions according to ASTM C1161 which presents a standard test method for flexural strength of advanced ceramics at ambient temperature. All samples were polished by diamond plates and 30, 6 and 1 µm diamond paste. Scanning electron microscope (SEM) produced by Philips model XL30 at 20 kV was used for observing surface structure and crack formation during hardness tests.

For annealed specimens, hardness and flexural strength tests done in the same manner mentioned for raw nanoceramics. Fracture toughness for annealed specimens was calculated by Equation 1, and maximum fracture performance was detected.

RESULTS AND DISCUSSION

For Al_2O_3 with 500 ppm MgO and without SiC, alumina matrixes have abnormal grain growth. Moreover adding SiC particles to the matrix, the grain growth is decreased. Decrease of grain growth is due to placement of SiC nanoparticles on grain boundary of alumina matrix. Effects of the SiC particles on the grain growth were trivial at low SiC vol% and high sintering temperatures, which for this condition most of the *SiC* nanoparticles were placed in the alumina matrix. Measured flexural strength of Al_2O_3-SiC-MgO nanoceramic as a function of vol% SiC and sintering temperature depicted in Figure 1.

Maximum flexural strength is attributed to specimens

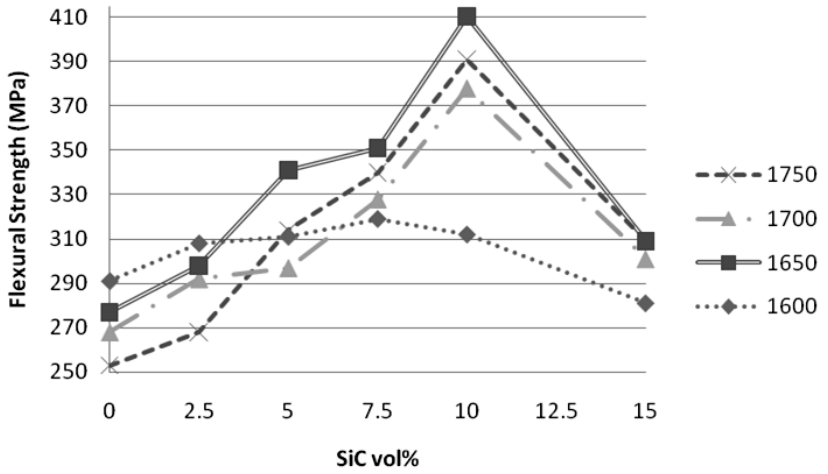

Figure 1. Flexural strength of Al_2O_3-SiC-MgO as a function of sintering temperature and vol% SiC.

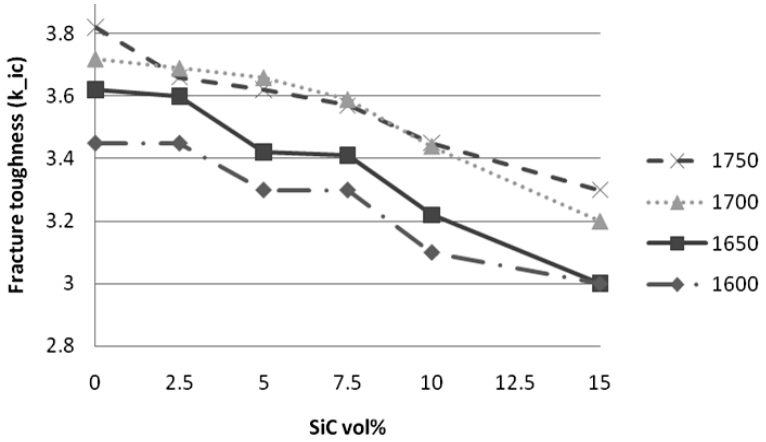

Figure 2. Fracture toughness of Al_2O_3-SiC-MgO function of sintering temperature and vol% SiC.

with 10 vol% SiC at sintering temperatures of above 1650°C. One of the reasons of this increase is the fact that grain size of alumina matrix is decreased by addition of the SiC particles, and also mismatch between thermal expansion of SiC and Al_2O_3 produces residual tensile stress in Al_2O_3 matrix that helps to increase the toughness of nanocomposite. Thermal expansion of Al_2O_3 is higher than the SiC particles, so during the cooling process most of the SiC particles were located in the Al_2O_3 grains and because of the mismatch between the thermal expansions, SiC particles would have less twitch when compared with alumina matrix, thus residual tensile stress is produced in Al_2O_3 grains that affect the toughness of nanocomposite (Pezzoti et al., 2001).

It was derived from Figure 1 for specimens with 15 vol% SiC besides increasing of the pores and decreasing of the density, agglomeration of SiC particles was increased and homogenous dispersion of the SiC particles in alumina matrix was reduced. Figure 1 illustrates that increase of sintering temperature for specimens with 0 and 2.5 vol% SiC, causes constantly decrease of flexural strength. Increase in grain growth with rising the sintering temperature causes the reduction in flexural strength of 0 and 2.5 vol% SiC specimens. For specimens with 5 vol% SiC and more, increase of the sintering temperature from 1600 to 1650°*C* increases flexural strength. Properly sintering of the nanoceramic due to 650°*C* difference between melting points of SiC and Al_2O_3 can be the reason.

Fracture toughness against SiC vol% for sintering temperatures from 1600 to 1750°C was shown in Figure 2. Due to proper sintering at higher temperatures, toughness increased at higher temperatures. With SiC vol% increase, toughness had extremely decreased, because

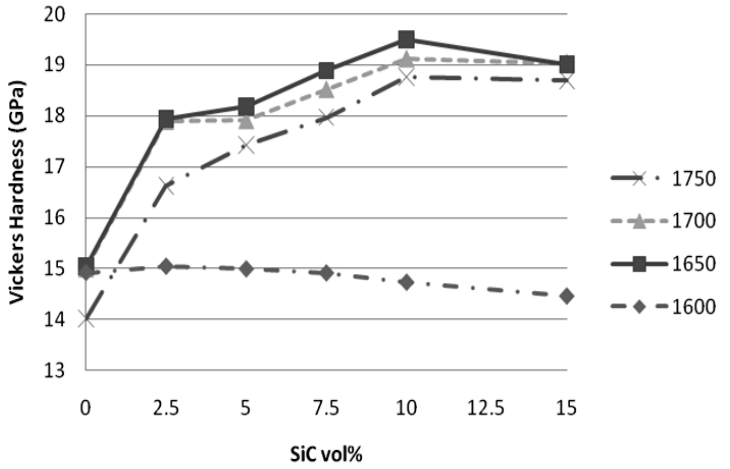

Figure 3. Vickers hardness of Al_2O_3-SiC-MgO as a function of sintering temperature and vol% SiC.

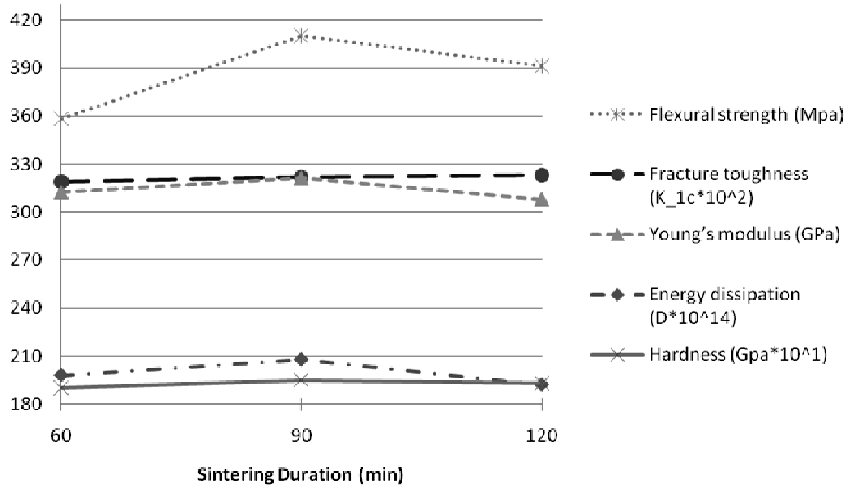

Figure 4. Mechanical properties of specimens at different sintering duration at sintering temperature of 1650°C.

of thermal expansion mismatch between Al_2O_3 and SiC particles during cooling and tensile residual stress creation. Fracture toughness of grain boundaries is usually lower than within the grains; therefore, adding SiC particles changes the fracture mode of nanoceramic from intergranular fracture to transgranular fracture. Fracture mode of the nanoceramic is transgranular. The transgranular fracture mode significantly helps the strength of grain boundaries and resisting against crack propagation. However dislocations created around SiC particles in Al_2O_3 matrix, consequently, the defect size will reduce along the grain boundaries. Dislocation mobility are difficult at room temperature, because of the SiC particles placed at grain boundaries and it cause small stress concentration in matrix, and create nanocracks around the propagating crack tip. These nanocracks slightly decrease the strength of the Al_2O_3 matrix and expand the frontal process zone size, therefore improving the fracture toughness of the materials.

Figure 3 presents result of hardness test conducted on the samples. This figure illustrates that except for sintering temperature of 1600°C, hardness increases by addition of SiC vol%. Maximum hardness was observed in samples with 10 vol% SiC at the temperature of 1650°C. Vickers hardness of 1450 MPa for pure alumina rises to value of 1950 MPa for described nanoceramic.

Figure 4 presents some of physical and mechanical properties for different sintering duration in samples with 10 vol% SiC at temperature of 1650°C. With regard of desired properties, significant duration of 90 min can be

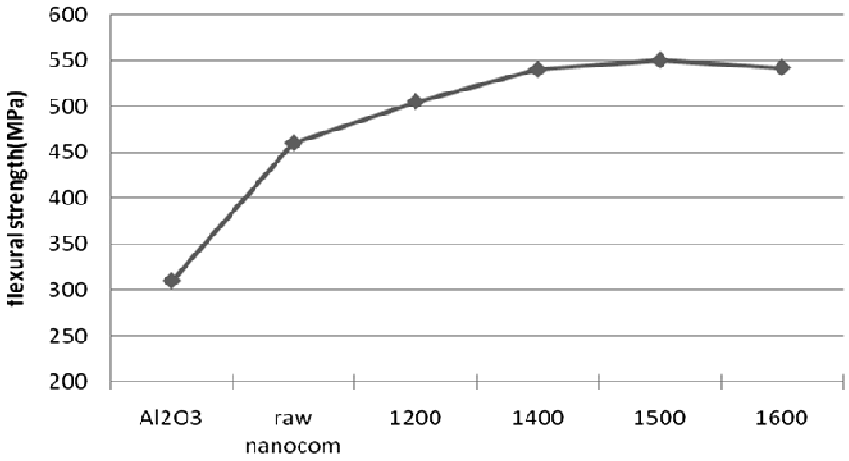

Figure 5. Flexural strength of annealed samples.

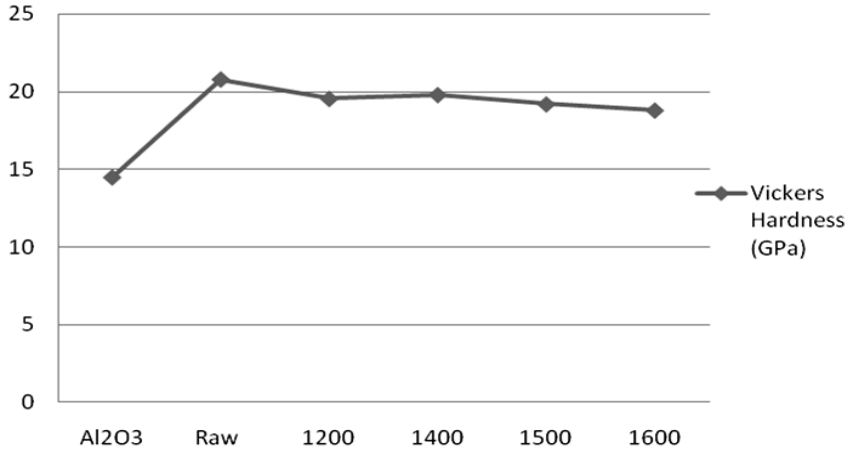

Figure 6. Hardness of the annealed samples.

collected for optimal sintering duration.

Considering the results from physical and mechanical properties, such as flexural strength, hardness and toughness, specimens containing 10 vol% SiC, sintering temperature of 1650°C and sintering duration of 90 min were chosen for optimum condition.

Results of utilizing the proposed nanoceramic as front layer of laminar target in comparison with pure alumina testimonies that impact performance obviously improve. Areal-density of samples is reduced at least 30% by using nanoceramic layer suggested in this article instead of pure alumina ceramic for preparing high-speed projectile penetration resistant panels.

Results obtained from mechanical properties of the annealed specimens are shown in Figures 5 and 6. These results illustrate that flexural strength increases as the temperature of annealing increased to 1500°C and it slightly falls at 1600°C. In the case of annealing temperature of 1500°C, flexural strength is 70% greater than pure alumina. Hardness test results depicts slightly decrease in Vickers hardness number with increasing anneal temperature. But in the inferior case, annealed nanoceramic samples at least 30% harder than pure alumina. Fracture toughness of the samples which are annealed at higher temperatures is shown in Figures 7. Fracture toughness of the samples shows meaningful increase.

One of the main reasons of toughness increase is crack evolution and deviation caused by existence of tension and compression fields around the SiC particles. Due to thermal expansion difference of SiC and alumina, microcracking phenomenon occurs. Generally, crack deviation acts on crack tip. This nanoscale cracks, causes the growth of the frontal process zone size ahead of a crack tip and improves the fracture toughness. Figure 8 schematically shows this mechanism. On the other hand, growing crack deviates when faced by the second phase or boundary of grains.

Performed observations by SEM indicate this crack deviation and microcracking phenomena due to residual

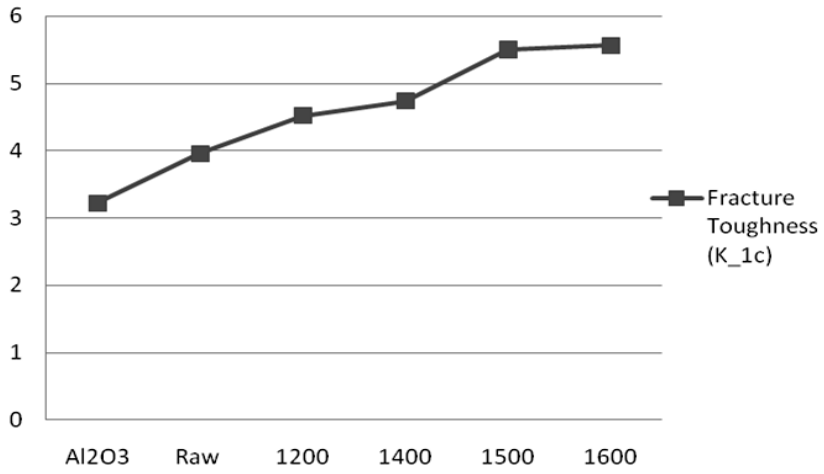

Figure 7. k_1c for annealed samples in comparison with pure alumina and raw nanoceramic.

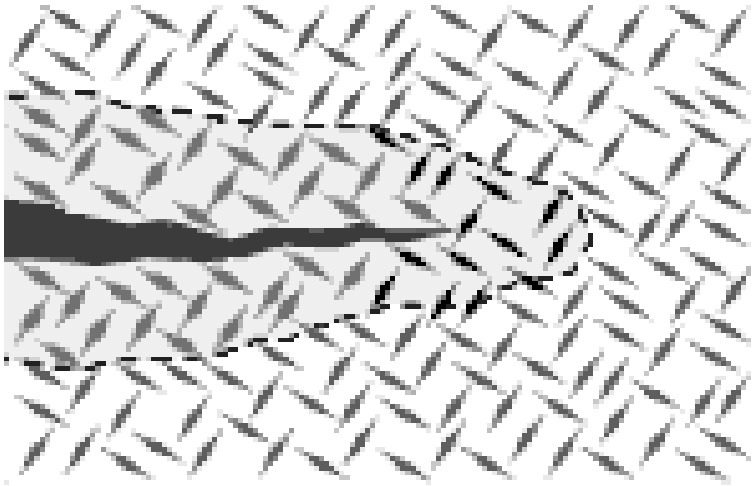

Figure 8. Phase transformation at the crack tip.

stresses in cooling specimen. Figure 9 indicates the cracks along rectangular trace diameters which are created by Vickers pyramid on raw specimen. Size of these cracks illustrates brittleness of material.

Tests which were conducted on annealed samples indicate reduction in hardness and rise of toughness in the samples with higher annealing temperature. Figure 10 shows the surface of an annealed specimen and supports this claim that fracture toughness impressively increased by heat treatment procedure. So, the specimen has no cracks after hardness test. A larger magnification of pyramid edge clearly demonstrates lack of any cracks.

Conclusions

Based on the performed tests that were presented, experimental variables in sintering procedure of Al_2O_3-SiC-MgO nanoceramic, such as sintering temperature, pressure, duration and SiC vol% was investigated. Optimum values of these parameters achieved for best mechanical properties particularly fracture performance. By addition of the SiC particles, fracture mode changed from intergranular mode to transgranular and this change, improved the strength and fracture toughness of the material. All physical and mechanical properties of the nanoceramic had ascending relation with increase of sintering pressure. Also addition of 10 vol% of SiC increased the flexural strength of the pure alumina ceramic nearby 60%. At sufficient sintering temperatures, hardness increases by addition of SiC vol%. Heat treatment procedure which was performed on samples with optimum sintering conditions, demonstrate that much better fracture performance could be achieved by

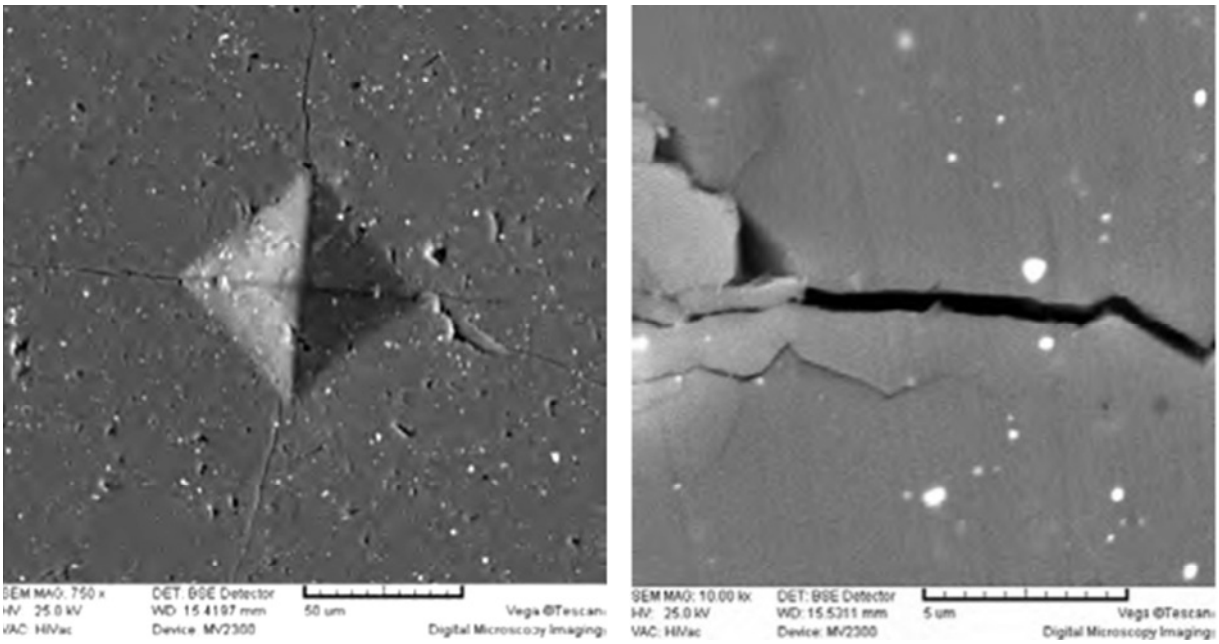

Figure 9. SEM photograph of the cracks created by indentation of Vickers pyramid on the raw specimen.

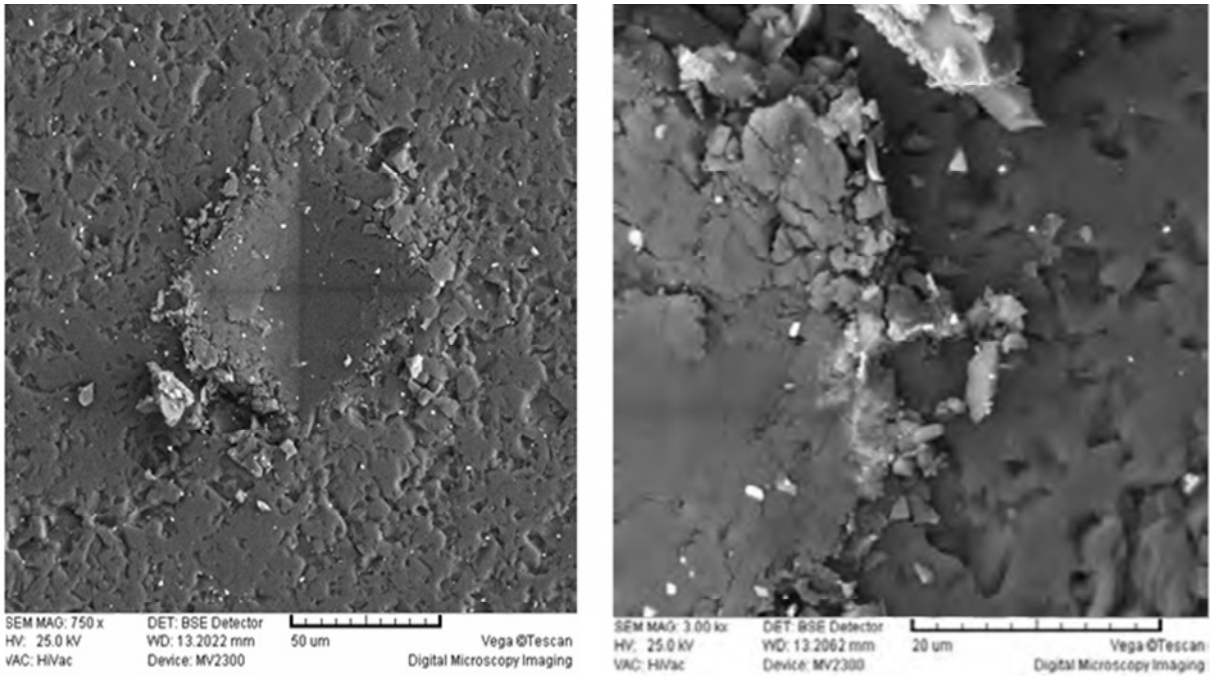

Figure 10. SEM photograph of indentation of Vickers pyramid on the 2 h annealed specimen at 1500 ℃.

annealing. Fracture toughness and flexural strength of the samples which are annealed at higher temperatures, show meaningful increase. Optimum fracture properties of annealed samples were obtained at annealing temperature of 1500℃ for staying duration of 2 h. In this case flexural strength increases by 20% and fracture toughness shows 40% rise when comparison with not annealed samples.

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