Effect of Sintering Additives and Reinforcement on Microhardness Values of Si_3N_4 Ceramics and Composites

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Abstract—In this research study, microhardness values of silicon nitride ceramics and its composites have been obtained by using Vickers and Knoop indenters. Samples of six silicon nitride ceramics were used for this study. These are $Si_3N_4 + Y_2O_3$, Si_3N_4 + MgO, Si_3N_4 + TiC, Si_3N_4 +TiN, Si_3N_4 +BN and Nano- Si_3N_4 + Nano-BN. Samples of Silicon nitride ceramics (Y_2O_3 and MgO) and its composites of TiC, TiN and BN were prepared by hot pressing. Nano-Si_3N_4 + Nano-BN ceramic composite was developed by Spark Plasma Sintering. Vickers and Knoop hardness values were obtained under high load of 0.05 Kg to 2.0 Kg. Fracture toughness of Si_3N_4 ceramics and its composites was also obtained by Vickers micro-indentation method. Higher hardness and higher fracture toughness was observed in nano-Si_3N_4 + Nano - BN, as compared to other Si_3N_4 ceramics and its composites.

Keywords: Vickers hardness, Knoop hardness, Microhardness, Nano-Silicon nitride, Nano-Boron nitride

1. INTRODUCTION

Ceramic/silicon nitride bearing elements are an attractive design solution for high speed turbine, precision machine tools and various automotive applications. High demand of employing these elements in severe conditions of high contact stress, high temperature, high speed and restricted lubrication have put tremendous pressure on design engineers to evaluate the material and advise applicable design strategies, Khan et al.. (2005).

Silicon nitride Si_3N_4 bearing elements have shown practical advantages over traditional steel elements due to their mechanical and physical properties, Hadfield et al. (1993) and Hadfield (1998). Leading technology, demands for high efficiency and importance of sustainable development have caused loading bearing contacts in all kinds of machinery to be subjected to high speeds, high contact stresses and severe conditions of lubrication, Khan et al. (2006).

Engineering ceramics uniquely combine strength, strength retention at high temperature, hardness, dimensional stability, good corrosion resistance, low density, superior thermal shock resistance, high wear resistance, and fracture toughness, Bennewitz (2003). These excellent mechanical and tribological properties make them suitable candidate materials for various applications, ranging from cutting tools to nuclear reactors, Kanimoto et al. (2000) and Kitamura et al. (2006). Typical applications are gas turbine bearings, I. C. Engine components, turbocharger rotors, seals, rocker arms, turbine blades. Compared with, other engineering ceramics, silicon nitride has superior mechanical and tribological properties, Hyuga et al. (2005), Nakamura et al. (2001), Xu et al. (2005), Xu et al. (2007). Silicon nitride and its composites have long been used for design and development of hybrid bearings, cutting tools, values, engine parts, turbine blades etc, Andersson et al. (1991). As the use of Si_3N_4 is increasing at rapid rate, various sintering methods have been used to improve microstructure, mechanical and tribological properties of Si_3N_4 ceramics Wani et al. (1997). In addition, composites of Si_3N_4 with BN, TiN and TiC; and also composites of nano-Si_3N_4 with Nano-BN have been developed to improve microstructure, mechanical and tribological properties of monolithic Si_3N_4, Xu et al. (2006), Jones et al. (2001), Jones et al. (2001), Hyuga et al. (2003) and Ullner et al. (2001).

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Mechanical and tribological properties, such as, hardness, strength and wear resistance of Si_3N_4 ceramics depend upon (a) sintering additives (b) type of reinforcement and (c) sintering conditions (i.e. temperature, pressure etc.) used for fabrication of Si_3N_4 ceramics and its composites, Xu et al. (2006), Jones et al. (2001), Jones et al. (2001), Hyuga et al. (2003) and Ullner et al. (2001). Hardness plays a significant role, in increasing the wear resistance of Si_3N_4 ceramic, Rendtel et al. (2005) Higher hardness means higher wear resistance and vice-versa. Hardness is crucial for cutting tools, wear and abrasion-resistant parts, prosthetic hip-joint balls and sockets, optical lens glasses, ballistic armor, molds and dies, valves, and seals, Quinn (2006) and Swab (2004). Different techniques for measuring hardness of ceramics have been used, Sun et al. (2007). However, microhardness technique for measuring the hardness of ceramics has shown promising results Rendtel et al. (2005), Quinn (2006), Swab (2004) and Sun et al. (2007). In order to understand the influence of sintering additives and the composites on hardness values and wear resistance, it is essential to measure hardness of Si_3N_4 ceramics and its composites.

Microhardness research studies on three advanced ceramic materials Si_3N_4, SiC Al_2O_3 have been carried out, Ullner et al. (2001). In this research study, three different indentation techniques Vickers, Knoop and Rockwell have been used, to evaluate the hardness of these three ceramics. It was observed that there is no significant difference between the abilities of these hardness techniques. Influence of BN particulate on microstructure and mechanical properties of Si_3N_4 has been studied, Kasiarova et al. (2006). In this research study, it was observed that the particulate of BN reinforced into the Si_3N_4, improves tribological properties of Si_3N_4. Silicon nitride composite of SiC has been developed to study the influence of reinforcement on the properties of monolithic Si_3N_4, Blugan et al. (2005). Researchers observed that intergranular SiC Nano- particles hinder the Si_3N_4 grain growth and thus change the chemical composition of grain boundary phase. Mechanical properties of Si_3N_4/C fibre composite have been carried to study the influence of C reinforcement on the properties of monolithic Si_3N_4, Blugan et al. (2005) Hadad et al. (2006). In this research study, it has been observed that the strength and toughness of the composite is higher comparing to monolithic Si_3N_4. Multilayered laminates of Si_3N_4 –TiN have been used to develop the Si_3N_4 / TiN composite Bai et al. (2008). Friction and wear study results have shown that wear resistance of laminates remain unchanged, however, fracture toughness of laminates is higher than that of Si_3N_4 composite. Recently, Nano-silicon nitride composites of BN have been developed for self lubrication of Si_3N_4 / Si_3N_4 tribopair, Xu et al. (2005) and Wani (2009).

In this research study, microhardness tests on Si_3N_4 ceramics and its composites have been carried out, to study influence of sintering additives and reinforcement on Vickers and Knoop hardness values of silicon nitride ceramics. In addition, fracture toughness of these ceramic materials has also been evaluated, based on Vickers indentation method.

2. EXPERIMENTAL PROCEDURE

2.1 . Materials and manufacturing process

The following powders were used as raw materials for the fabrication of Si_3N_4: Si_3N_4, 86% α- phase, with specific area of 8.78 m^2/g, containing 0.538 % calcium, 0.01 % magnesium, 0.084% sodium, 0.13 % iron, 38.6% nitrogen and 2.2% oxygen (H. C. Starck, Goslar, Germany); CeO_2 and Y_2O_3, both 99.9% (Indian Rare-Earths Ltd., Udyogmandal, India); MgO > 97% (E. Merck, Darmstadt, Germany); AlN , 65.5% aluminium and 32.4% nitrogen (H. C. Starck); and SiO_2 99.8%, Hesla Minerals, Ranchi, India). Batches of Si_3N_4 powder (100g), mixed with the requisite amounts of additives, and were attrition-milled with aluminium balls in normal hexane. The uniform powder mixture, after final sieving to remove the aluminium balls and grit, were hot pressed (GCA Vacuum industries Inc., Somerville, U.S.) as 25 mm diameter x 8 mm thick disks in a BN-Coated graphite die at 1650^o C-1700^o C under pressure of 25 MPa until shrinkage ceased. The composite of Si_3N_4 and TiC was made from nitrogen–rich, liquid- phase pressure sintered Si_3N_4 and Y_2O_3. The surfaces of samples were polished and finished with 4 μm and 2 μm diamond grit in an automatic polisher (Pedemax, Struers, and Copenhagen Denmark). The typical surface had an average surface roughness of 0.6 μm. Similarly, the composition of powders and preparation of Si_3N_4 + 30 wt % TiN composite is provided in Blugan et al. (2005) Hadad et al. (2006).

The composition of powders and preparation of Nano- Si_3N_4 +5 wt % Nano- BN and micro Si_3N_4 + 5 wt % BN ceramics samples is given elsewhere, Xu et al. (2007) and Wani (2009).

2.2. Characterization

Scanning Electron microscopy (SEM), Energy Dispersive Spectroscopy (EDS) and X-ray Diffraction (XRD) studies were carried out to study microstructure of ceramic surfaces and also to carry out elemental analysis of Si_3N_4 ceramic samples. SEM and EDS studies were carried out on Hitachi SEM S -3600, equipped with EDS. X-ray diffraction (XRD) studies were carried out on M/S Philips, The Netherlands Compact X-ray Diffraction System. Typical results of SEM with EDS and XRD are shown in Figures 1-3. Figure 1 (a - d) shows SEM and EDS of Si_3N_4. It is obvious from Fig. 1 (a) that the surface of Si_3N_4 sample contains uniform grain structure of the ceramic material. EDS analysis of the Si_3N_4 surface was carried out at three different spots (marked as 1, 2 and 3) and is shown in Figure 1 (b, c and d). Figure 1 (b) shows EDS of spot 1. Similarly, Figure 1(c and d) shows EDS of spot 2 and 3. It is evident from EDS analysis of these three spots that Si_3N_4 is evenly distributed over the surface of the Si_3N_4 ceramic.

XRD patterns and analysis of nano-Si_3N_4+ 5wt % nano- BN are shown in Figures 2 and 3 respectively. It is obvious from Figure 2 that average grain size of Si_3N_4 in nano- Si_3N_4+ 5wt % Nano-BN disc sample is 56.5 nm. X-ray diffraction pattern shown in Figure 3 indicates that major phase present in the Nano-composite is β-silicon nitride.

2.3 Microhardness tests

Hardness tests were performed on polished surfaces (mirror like finish, 1 µm diamond polish) using Vickers and Knoop diamonds. Vickers hardness (VH) and Knoop hardness (HK) tests were performed on Universal high- load hardness tester (Model UHL VMHT MoT, Walter UHi, Gmbh & Co. KG, Germany). The indentation was observed under 50 to 100 magnification. VH and HK were measured to study the influence of indentation load and time on hardness values. The indentation load was varied from 2.943 N (0.3Kg) to 19. 62 N (2.0 Kg) and indentation time was changed from 6 seconds to 12 seconds. In order to understand the influence of indentation time on hardness values (HVand HK), indentation tests were performed on selected Si_3N_4 ceramics. Each indentation test was repeated 3 to 5 times for better repeatability.

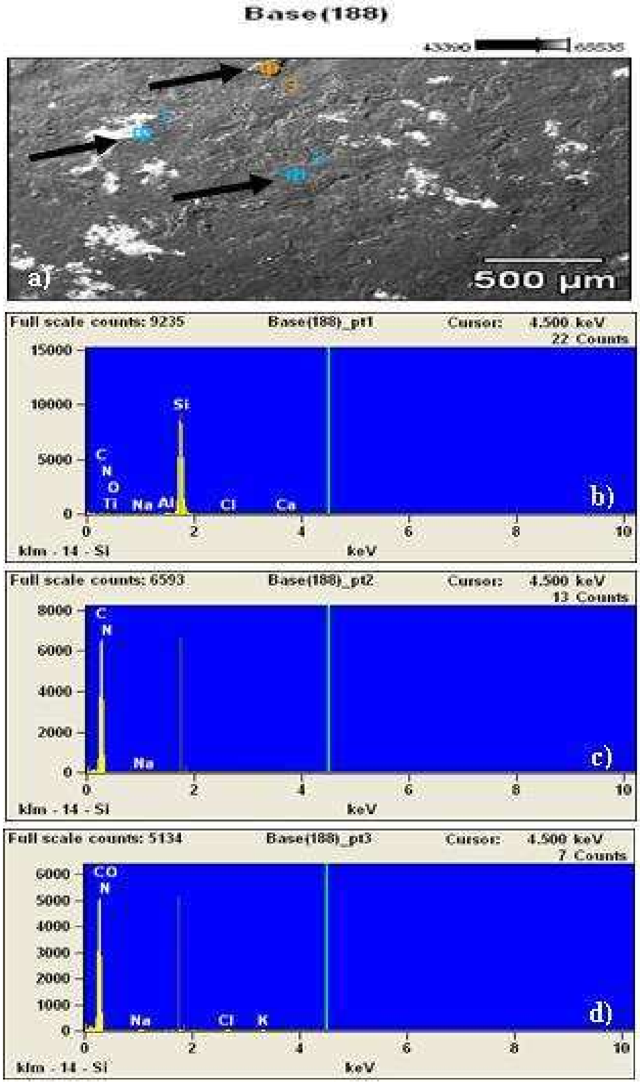


Fig. 1. Scanning electron micrograph (SEM) and Energy Dispersive Spectroscopy (EDS) of Si_3N_4 Ceramic: (a) SEM , (b, c and d) EDS of point 1, 2 and 3.

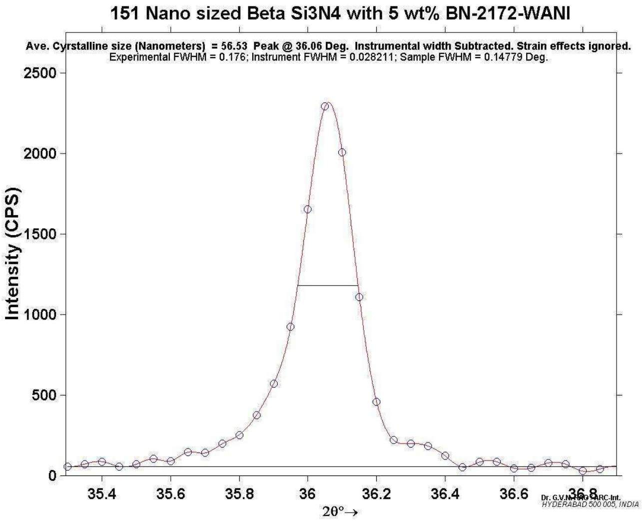


Fig. 2. XRD of Nano Si_3N_4+ 5wt % Nano BN.

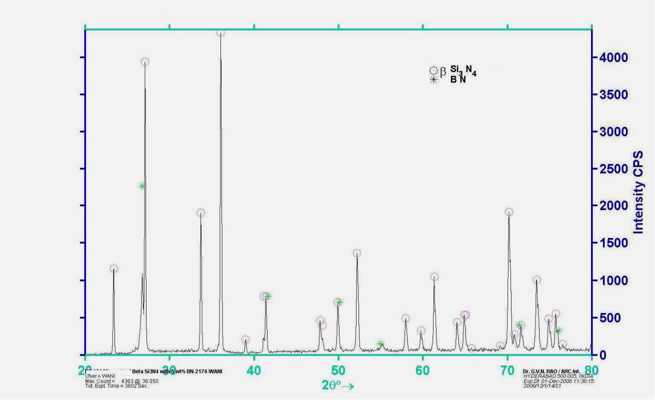


Fig. 3. XRD of Nano Si_3N_4+ 5wt % Nano BN

3. RESULTS AND DISCUSSIONS

Hardness values (HVand HK) of Si_3N_4 ceramics and its composites against indentation load and indentation time are shown in Figures 4-8. The influence of indentation load (0.05 Kg – 2.0 Kg) on HV and HK of Si_3N_4 ceramic sintered with Y_2O_3 and MgO is shown in Figure. 4. Figure 5 indicates the influence of indentation load (0.05 Kg – 2.0 Kg) on HV and HK of Si_3N_4 + TiN and Si_3N_4 + TiC ceramic composite. Whereas, the influence of indentation load (0.05 Kg – 2.0 Kg) on HV and HK of nano-Si_3N_4 + Nano BN and micro-Si_3N_4 + BN ceramics is shown in Figure 6. As can be seen in Figures 4-6, the hardness decreases with the increase in load in Si_3N_4 ceramics of Y_2O_3 and MgO; and its composites of TiN, TiC and BN. However, higher hardness values were obtained in the case of Nano- Si_3N_4 + 5 wt% Nano-BN composite, as compared to other Si_3N_4 ceramics and its composites. It is evident from Figure 4, that Si_3N_4 ceramic sintered with Y_2O_3 possesses higher hardness, as compared to Si_3N_4 sintered with MgO. This is attributed to the formation of liquid phase in presence of sintering additive Y_2O_3 in the case of Si_3N_4 +Y_2O_3. Sintering additive Y_2O_3

aids in the formation of liquid phase during sintering, which on solidification produces more intergranular refractory phase, as compared to MgO, Xu et al. (2006) and Jone et al. (2001). However, higher sintering temperature is required for Y_2O_3, as compared to MgO.

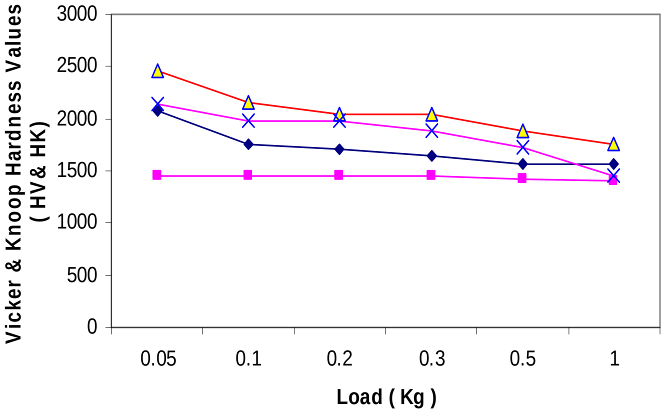


Fig. 4. Vickers hardness (HV) and Knoop hardness (HK) verses: Load (ΔHV & *HK of Si_3N_4 + Y_2O_3 & ♦HV & ■HK of Si3N4 + MgO).

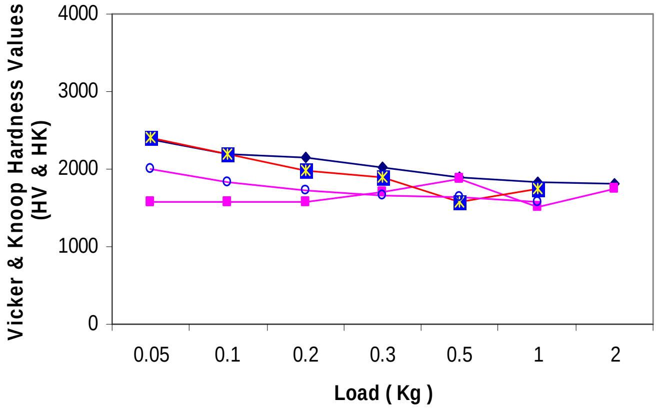


Fig. 5. Vickers hardness (HV) and Knoop hardness (HK) verses Load: (♦HV & * HK of Si3N4 + TiN and ○HV & ■ HK of Si3N4 + TiC).

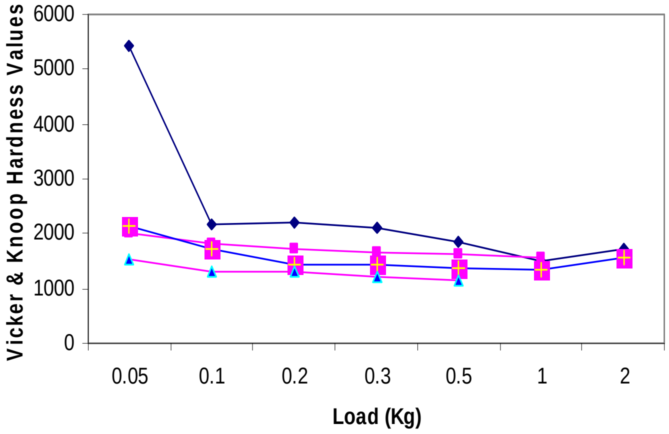


Fig. 6. Vickers hardness (HV) and Knoop hardness (HK) verses : (♦ HV, ■HK - Nao-Si3N4 +Nano- BN & +HV, ΔHK -Si3N4 + BN)

In the case, of Si_3N_4 + 30 wt % TiN and Si_3N_4 + 10 wt % TiC composites, Si_3N_4 + TiN possesses higher hardness, as compared to Si_3N_4 + TiC composite , as shown in Figure 5. This is attributed to the fact that cracks deflection, micro crack deflection and crack impedance exists in the case of Si_3N_4 + TiC. The increase in micro cracks easily leads to excessive connection of micro cracks, which reduces hardness and strength of the ceramic composite, Quinn et al. (2006). As can be seen in Figure 6, that Nano-Si_3N_4 + 5 wt % of Nano BN possesses higher hardness, as compared to composite of micro-sized Si_3N_4 + 5 wt % of BN. The higher values of hardness in the case of Nano-Si_3N_4 + 5 wt % of Nano-BN is attributed to the fact that hardness of fine-grained ceramics generally increases with decreasing grain size, e.g., due to Hall-Petch type effects on the associated plastic flow Quinn et al. (2006), Xu et al. (2006), Xu et al. (2007) and Wani (2009).

The indentation time tests were carried out to determine the influence of indentation time on hardness value (HV and HK). These indentation tests were performed on specific Si_3N_4 ceramics and its composites, viz., Nano-Si_3N_4 + Nano-BN, Si_3N_4 + TiN and Si_3N_4 + Y_2O_3 . The influence of indentation time (6 seconds-12 seconds) on HV and HK is shown in Figure 7 and 8. Figure 7, indicates the influence of indentation time (6 seconds - 12 seconds) on HV and HK at constant indentation load of 0.3 Kg, where as, the influence of indentation time (6 seconds-12 seconds) on HV and HK at constant indentation load of 0.5 Kg is shown in Figure 8. As can be seen in Figure 7 and 8, the hardness HV and HK decreases with the increase in indentation time (6 seconds - 12 seconds), however, the decrease in the values of hardness is small for indentation time of (8 seconds - 12 seconds).

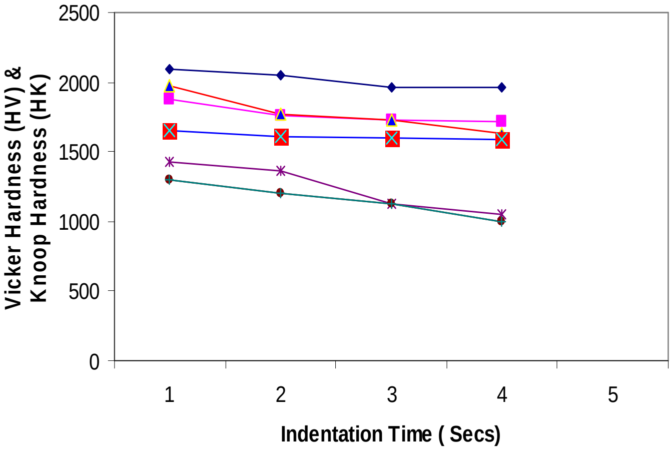


Fig. 7. Vickers hardness (HV) and Knoop hardness (HK) verses Indentation time: (♦ HV, ▲HK - Nao-Si_3N_4 + Nano-BN, ■ HV, × HK - Si_3N_4 + TiN & * HV, ○HK -Si_3N_4 Y_2O_3)

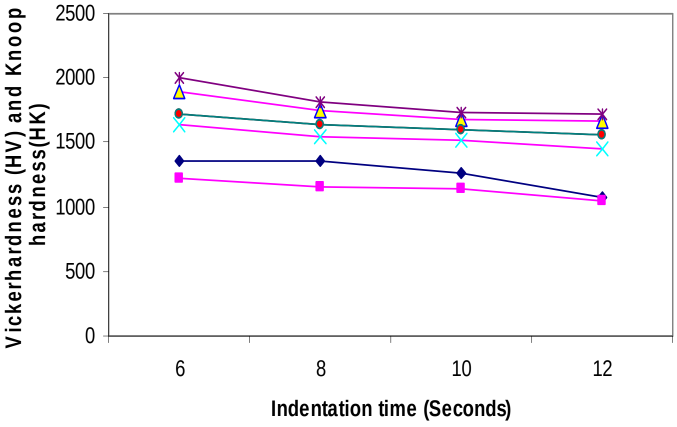


Fig. 8. Vickers hardness (HV) and Knoop hardness (HK) Verses indentation time (*HV, ΔHK- Nao-Si_3N_4 + Nano-BN, ●HV, × HK Si_3N_4 +TiN &♦HV ■ HK Si_3N_4 +Y_2O_3)

As mentioned in previous section that indentation (Vickers and Knoop) were observed under optical microscope at 50 and 100 magnification. Indentation images on the surfaces of Si_3N_4 ceramics and its composites are shown in Figures 9-11. Indentation images on the surface of Si_3N_4 ceramic sintered with Y_2O_3 are shown in Figure 9. Figure 9 (a, b and c), indicates the indentation image of Vickers indenter at 0.5 Kg, 1.0 Kg and 2.0 Kg, respectively. Where as, Figure 9 (d and e), indicates the images of Knoop indenter at 0.5 Kg and 1.0 Kg, respectively. Figure 10, shows indentation images on the surface of Si_3N_4 + 30 wt % TiN. Indentation images on the surface of Si_3N_4 + TiN at 1.0 Kg and 2.0 Kg are shown in Figure 10 (a and b **)**, respectively. Where as, Figure 10 (c and d) shows Knoop indentation images on the surface of Si_3N_4 + TiN at 0.5 Kg and 1.0 Kg, respectively. Indentation images on the surface of Nano- Si_3N_4 + 5 wt % of Nano BN are shown in Figure 11. Indentation image on Nano composite at 0.5 Kg, 1.0 Kg and 2.0 Kg are shown in Figure 11 (a, b and c), respectively. Knoop indentation image of Nano composite is shown in Figure 11 (d and e) at 0.5 Kg and 1.0 Kg. As can be seen in Figure 10 (c) and Figure 11 (c), the cracks are developed at the edges of Vickers indenter at indentation load of 2 kg in the case of Si_3N_4 + 30 wt %TiN and Nano-Si_3N_4 + Nano-BN, respectively. This is marked by an arrow in Figure 10(c) and Figure 11 (c). The hardness values obtained are given in table 1.

Fracture toughness of ceramic composites was obtained by measuring crack length at the edges. Indentation fracture toughness is calculated, in terms, of toughness parameter (TP) relationship given in the reference, Jones et al. (2001).

TP = 10.282 $E^{0.4} P^{0.6} a^{-0.7} (c'/a)^{-1.5}$ MPa $m^{1/2}$, where E = Elastic Modulus (GPa), P = indentation load (N), c' = ½ total crack length (µm) and a = diagonal length (µm).

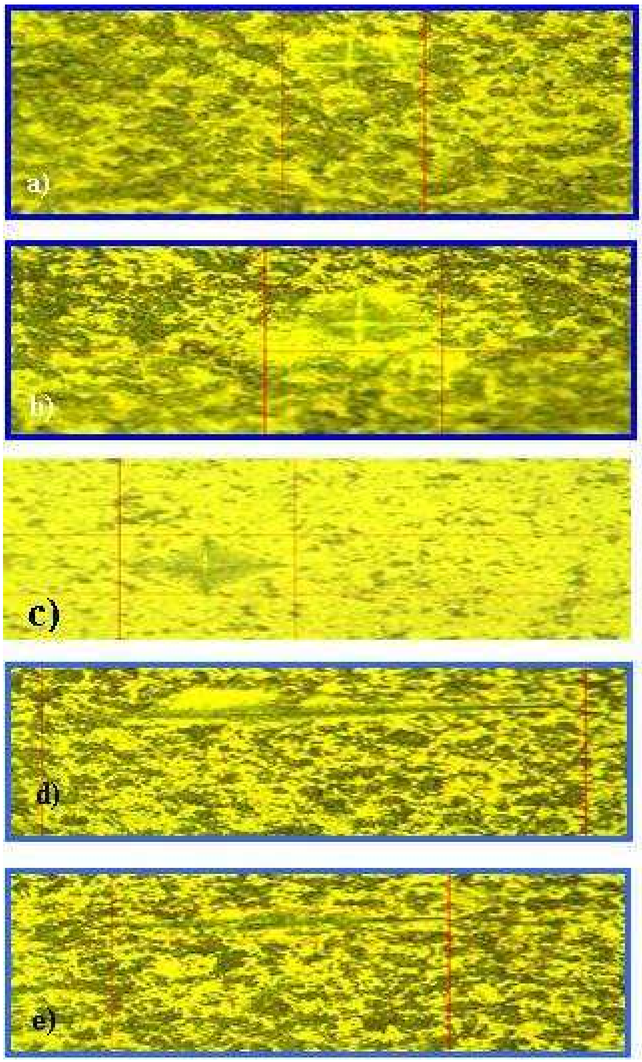


Fig. 9. Indentation images of Si_3N_4 Ceramic sintered with Y_2O_3. : a – HV (0.5Kg), b– HV (1.0Kg), c – HV (2.0Kg), d-HK (0.5Kg) e-HK (1.0 kg)

Fracture toughness value of 8.0 MPa $m^{1/2}$ and 7.0 MPa $m^{1/2}$ were obtained for Si_3N_4 + Y_2O_3 and Si_3N_4 + MgO, respectively. Where as, fracture toughness value of 9.00 MPa $m^{1/2}$ and 8.0 MPa $m^{1/2}$ were obtained for Si_3N_4 + 30 wt %TiN and Si_3N_4 + 10 wt % TiC, respectively. In the case, Si_3N_4 + 5 wt % BN and micro-sized Si_3N_4 + 5 wt % of BN, fracture toughness value of 11.0 MPa $m^{1/2}$ and 8.17 MPa $m^{1/2}$were obtained for Nano sized Si_3N_4 + 5 wt % BN and micro-sized Si_3N_4 + 5 wt % of BN, respectively. Nano-sized Si_3N_4 /BN composite possess highest fracture toughness value, as compared to other Si_3N_4 ceramics and its composites.

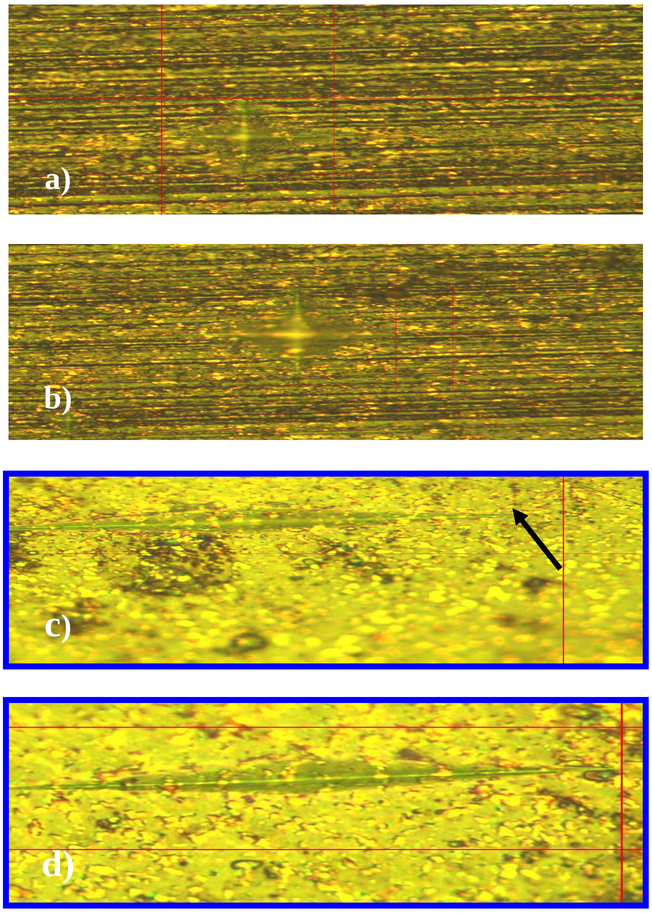
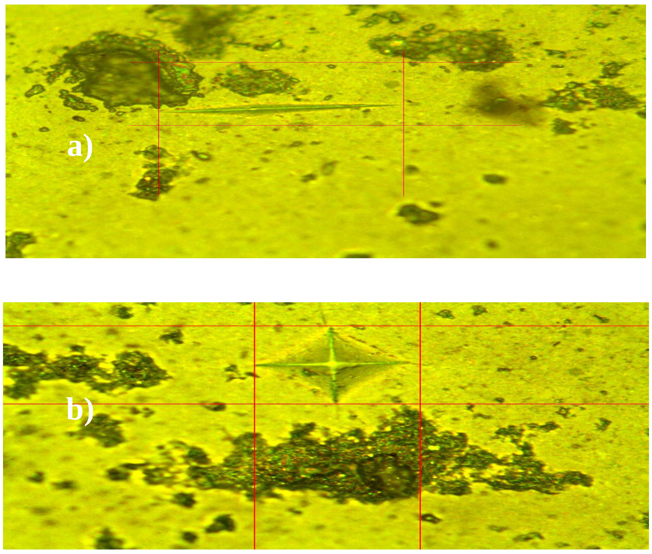


Fig. 10. Indentation images of Si_3N_4 +30 wt % TiN. a – HV (1.0Kg), b – HV (2.0Kg), c-HK (0.5Kg) &d-HK (1.0kg)



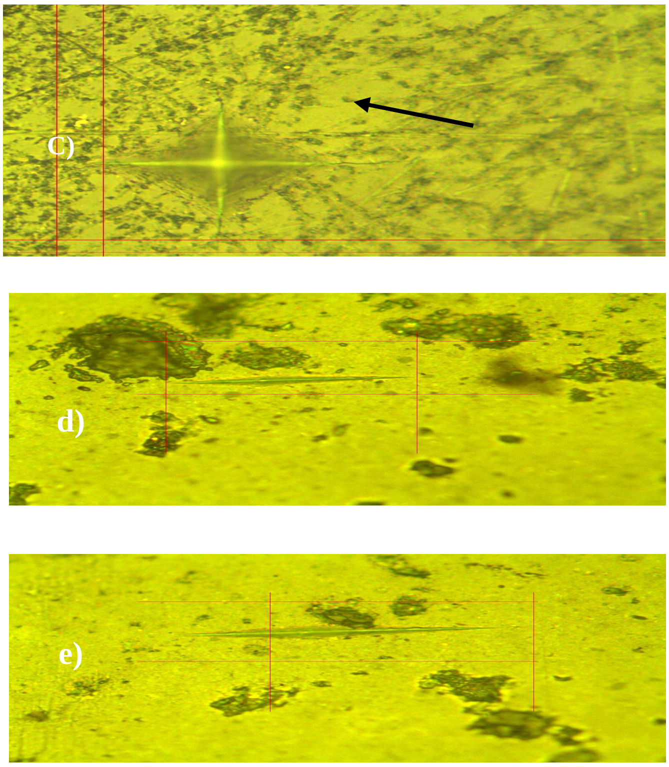


Fig. 11. Indentation images of Nano-Si_3N_4 +5.0 wt % Nano-BN: a – HV (0.5Kg),b– HV (1.0Kg), c – HV (2.0Kg), d-HK(0.5Kg) &e-HK (1.0 kg)

Table 1. Vickers and Knoop hardness values of Si3N4 ceramics and Composites

Ceramic Material	**Density (g/cm3)**	**Load (Kg)**	**0.05**	**0.1**	**0.2**	**0.3**
Si_3N_4 (MgO)	3.22	HV	x	2068	1763	1432
		HK	x	1450	1450	1450
Si_3N_4 (Y_2O_3)	3.22	HV	2450	2150	2035	2050
		HK	x	2140	1981	1871
Si_3N_4 + TiC	3.22	HV	2383	2193	2159	2018
		KH	1575	1584	1584	1700
Si_3N_4 +TiN	3.22	HV	x	2403	2192	1972
		KH		2005	1820	1733
Nano-Si_3N_4 + 5wt% Nano BN	3.22	HV	5432	2169	2192	2506
		KH	x	2005	1820	1733
Si_3N_4 + 5 wt % BN	3.20	HV	x	1718	1450	1432
		HK	x	1536	1307	1296

* Cracks observed at the edges

4. CONCLUSION

Silicon nitride ceramics and its composites were developed using hot pressing and Spark Plasma Sintering methods. Microhardness values (HV and HK) of Si_3N_4 and its composites were studied, using Vickers and Knoop indentation methods. Fracture toughness values of Si_3N_4 and its composites were also determined on the basis of Vickers indentation methodology. Microhardness values of Si_3N_4 ceramic is highly influenced by the presence of sintering additives. Si_3N_4 ceramic sintered with Y_2O_3 possesses higher microhardness values (HV and HK), as compared to Si_3N_4 ceramic sintered with MgO. Microhardness values (HV and HK) of Si_3N_4 ceramic composite of TiN, as compared to Si_3N_4 ceramic composite of TiC. Nano-Si_3N_4 / Nano-BN composite possesses higher values of Vickers and Knoop hardness, as compared to micro-sized Si_3N_4 / BN composite. Nano-Si_3N_4 / Nano-BN composite possesses microhardness values (HV and HK), as compared to Si_3N_4 +Y_2O_3, Si_3N_4 +MgO, Si_3N_4 +TiC, Si_3N_4 +TiN and micro-sized Si_3N_4 + BN ceramics. Nano-Si_3N_4 / Nano-BN composite possesses highest fracture toughness (11.0 MPa $m^{1/2}$), as compared to Si_3N_4 ceramics of (Y_2O_3 and MgO) and Si_3N_4 ceramic composites of BN, TiC and TiN.

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