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Microhardness of Silicon Nitride Ceramic and Its Composites

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ABSTRACT

Micro-hardness research studies on various non oxide ceramics will be carried out. These ceramics include silicon nitride and the composites of silicon nitride. Micro-hardness measurement of these ceramics i.e. vicker will be carried out using diamond indenters. Various influences of load and time of indentation on hardness values of these ceramics will be carried out in detail. Fracture toughness of these ceramics was also studied using vicker indentation. The vicker hardness of these ceramics decreases with increasing load, similar influence was observed by increasing time of indentation.

KEYWORDS: *Indentation, Vicker hardness, Composites, Ceramics*

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1. INRODUCTION

Engineering ceramics represent a wide variety of class of materials. These materials are commonly referred to as oxides, non-oxide ceramics and their composites. These materials are known for their high wear resistance, high temperature resistance, good chemical inertia, corrosion resistance, high electrical resistivity, high hardness etc. There are various classifications of ceramic materials that may depend on the application or structure. Technical ceramics are organized around three main families: oxides, non-oxides and composite materials.

1. Oxide ceramics: They are inorganic compounds of metallic (eg, Al, Zr, Ti, Mg) or metalloid (Si) elements with oxygen. For example, one can refer to aluminum oxide (Al_2O_3), zirconium oxide (ZrO_2) and aluminum titanate (Al_2TiO_5).

2. Non-oxide ceramics: They are based on carbon, nitrogen and silicon. This family includes nitrides (eg Si_3N_4) and carbides. This group of materials usually requires significantly higher temperatures.

3. Composite Ceramics: This family is rapidly evolving and is often a combination of two oxides or two non-oxides, but also a combination between members of the two families. Composite ceramics also include polymers and metals in particle and matrix form. Ceramates made of ceramic (Ser) and metallic (Mate) materials are the major elements of this class. They are mainly manufactured by casting and powder metallurgy methods [1].

It is well established that the mechanical properties of materials are related to the nature of the basic component and their relationship to the atomic scale. In the case of ceramic materials, two types of atomic linkages predominate: ionic and covalent. It is these strong ties that make up the distinguishing features of this content class. In fact, technical ceramics exhibit a remarkable combination of mechanical, electrical, thermal and biochemical properties. Some of them have good wear, oxidation and corrosive resistance in addition to an excellent thermal shock resistance [2,3]. In addition, ceramics are also lighter than most metals, which is suitable for reducing energy consumption and thus cost in many applications. In addition, ceramics are also characterized by high melting point, enabling them to maintain interesting mechanical properties at high temperatures.

These materials are used for extreme conditions of load and temperature in various engineering and other applications. For example, hybrid bearings are made with silicon nitride, IC engine components are made with silicon nitride, cutting tools are developed from nitrides, etc. Silicon nitride-based materials, which will be the subject of our study, have been widely used in various industrial domains. Since the 1950s. For example, Krstic and Krstic [4] noted applications in nuclear fusion reactors, manufacturing of thermal conductors, and gas turbines. Hampshire [3] cited structural application at high temperatures in turbocharger rotors, while Bal and Rahman [5] reported recent diagnostics of silicon nitride in spinal surgery and the current development of the femoral head of the hip joints. Reported use, today, rolling bearing construction companies are increasingly moving towards Si_3N_4 with an increase of about 40% per year. High-pressure turbo-pumps on NASA's space shuttle are a representative example [6]. In addition, porous silicon nitride is used for hot gas filtration to reduce the emission of porous and toxic particles [7].

Finally, composites based on ceramic materials are used to reduce weight and thus fuel consumption. As an application, one may refer to the cylinder sleeve in the engine, piston-recess walls, brake discs and bearings [8]. In particular, cermets are used for cutting, drilling tools and electrical components [9].

This Marshall et al. [10] has also been reported that an elastic recovery may occur after the removal of the indentation load as mismatch results between the plastic zone below the indentation and the surrounding elastic deformed material. Due to the characteristic shape of the indent, elastic retrieval during Vickers indentation is rather related to Nup indentation.

Furthermore, it has been observed that stiffness decreases with increasing load. This phenomenon is known as indentation size effect (ISE). Swab [11] found that the stiffness of a weightless knoop can be measured at high loads (> 4 N) and they applied a load of 19.9 N to determine the stiffness of the arm's ceramic knoop recommended.

Based on the analysis, Gong [12] concludes that the difference between HV and HK at a given load for a given material may be responsible for the elastic recovery in indentation.

2. RAW MATERIALS AND MANUFACTURING PROCESS

2.1. Powder Metallurgy

Power metallurgy (PM) is a constantly and rapidly evolving technology that embraces most metal, ceramic, alloy materials and a wide variety of ceramic composite and shapes. PM is a highly developed method of manufacturing reliable machine parts.

2.2. Raw Materials

Powders that has been used for the development of sample are as:

2.2.1. Copper powder

Micro copper with 99% purity (Loba Chemie Pvt. Ltd. India), in which an average particle size of 200 lattices was used as the base matrix for the fabrication of sample composites.

2.2.2. Nickel Powder

Micro nickel with 99% purity (Loba Chemise Pvt. Ltd. India). The average particle size of 200mesh is added to the copper powder a few percent and finally we get the alloy matrix.

2.2.3. Titanium carbide

TiC nanopower is reinforced in a metal alloy matrix (Sigma Aldrich, Germany), with a particle size purity of <200nm (98%)

2.3. Batch Preparation

Each powder is weighted to an electronic analytical balance at an accuracy of 0.1mg for sample preparation

2.4. Ceramic sample

There are various ceramics fabricated for this study.

A. Si_3N_4 doped with AlN and Y_2O_3.

B. Si_3N_4 doped with TiN, AlN and Y_2O_3.

C. Si_3N_4 is doped with Y_2O_3, Al_2O_3 and BN.

2.5. Powder Blend

High energy ball milling process where a powder mixture placed in a ball mill is subjected to high energy collisions from the balls. The prepared mixed powders were ground in a high-energy ball mill for 120 min at 200 rpm in the presence of ethanol, which acted as a process control agent and restricted the generation of intermetallic compounds. Hardened stainless steel vial was used to seal the powder, and the ball-to-powder ratio was 10: 1

2.6. Dry Process of Wet Mixed Samples

The drying process is carried out with the help of rota evaporator. A rotary evaporator is a device used to evaporate solvents under vacuum with the use of rotation and heat. Vacuum is used to reduce the boiling temperature, causing damage to thermo labile materials.

2.7. Condensation of Dried Composite Powder

After separating the mixed powder from the round flask. The ground mix was compacted by uneven pressure of freezing in a rigid cylindrical die at an optimized pressure of 650MP. The amount of this pressure was applied to the dye with the help of a compression testing machine and the pressure remains at 650MP for 5 minutes. Then the sample was extracted with the help of UTM.

2.8. Sintering of green compact sample

The green pellets were sintered in an argon atmosphere at an optimized sintering temperature from 1700 °C to 1750 °C for a soaking time of 60 min.

3. EXPERIMENTAL PROCEDURE

Hardness testing was performed on the polished surface of ceramic (mirror-like finish, diamond polish) using wicker indenters. For the evaluation of hardness, the standard test procedures and equations outlined in ASTM E384 are used. Indentation load range from 0.1Kgf to 1Kgf using wicker test was performed on a universal low-load hardness tester (model UHL VMHT MoT, Walter UHI, GmbH & Co.kg, Germany).Experiments were conducted in three stages to determine the hardness of the materials. The micro hardness and fracture toughness (A to C) of the material were determined using the following experiments.

- Hardness verses load
- Hardness verses

The load range used is 100 gf, 200 gf, 300gf, 500gf, 1000 gf, 2000 gf.

4. RESULT AND DISCUSSIONS

In this research study, micro hardness and indentation based fracture toughness of various non oxide ceramics were carried out. Micro hardness of ceramic materials was studied using Vicker indentation and fracture toughness of these materials was obtained by using Vicker hardness test. All hardness values are shown in Tables 1 and 2 with different graphs.

Table1: Vicker hardness values of selected ceramics.

Material	Sample denotation	Hardness value (kgf/mm²)	Hardness value (kgf/mm²)	Hardness value (kgf/mm²)	Hardness value (kgf/mm²)	Hardness value (kgf/mm²)
Load		100 gf	200 gf	300 gf	500 gf	1000 gf
Silicon Nitride (AlN, Y_2O_3)	A	1718HV	1449HV	1432HV	1362HV	1332HV
Silicon Nitride (TiN, AlN, Y_2O_3,)	B	2403 HV	2192 HV	1972 HV	1898 HV	1575 HV
Silicon nitride (Y_2O_3, Al_2O_3, BN)	C	2300 HV	2287HV	2094 HV	2002HV	1780 HV

Table2: Vicker hardness values under constant load of 500 gf.

MATERIAL	TIME(SEC) 6	TIME(SEC) 8	TIME(SEC) 10	TIME(SEC) 12
Silicon Nitride (AlN, Y_2O_3)	1362.5 HV	1361HV	1262 HV	1078 HV
Silicon Nitride (TiN, AlN, Y_2O_3,)	1898 HV	1747 HV	1676 HV	1670 HV
Silicon nitride (Y_2O_3, Al_2O_3, BN)	2002HV	1815 HV	1738 HV	1720 HV

The Vicker hardness vs load is plotted in Figures (1- 3). The Figure 1 shows that at load 0.1Kgf (100 gf,) the Vickers hardness value is 16.84 GPa (1718 Kgf/mm²). The vicker hardness values (H_v) goes on decreasing with the increase in load thus when a load of 1 Kgf (1000gf) is applied the hardness values is 13.06 GPa (1332 Kgf/mm²).

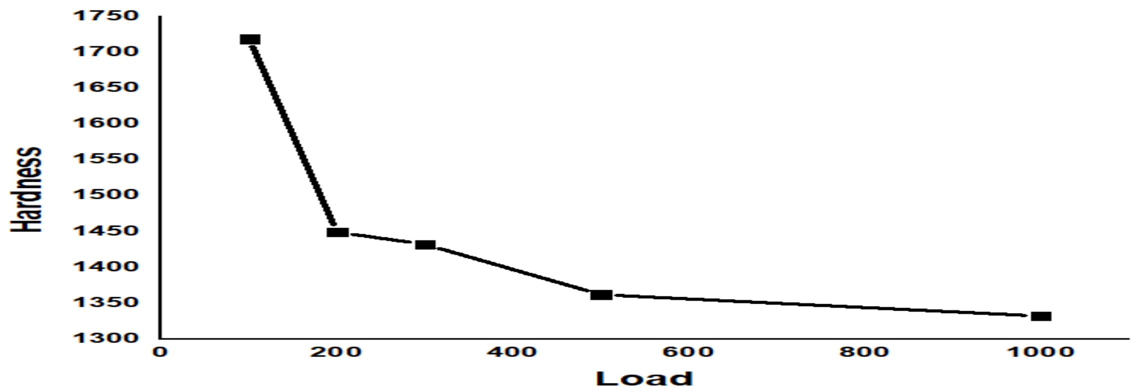

Figure1: Vicker hardness vs load for sample A

In Figure 2 the Vickers hardness value of sample B at load 0.1Kgf Vickers hardness value is 23.56 GPa (2403 Kgf/mm²). When a load of 1000 gf is applied the H_v is 15.44 GPa (1575 Kgf/mm²). For Vickers indentations difficulties arose in measuring the size of the indentations due to the small size at low loads. Thus at high loads indentation marks are clear which decreases the measurement error.

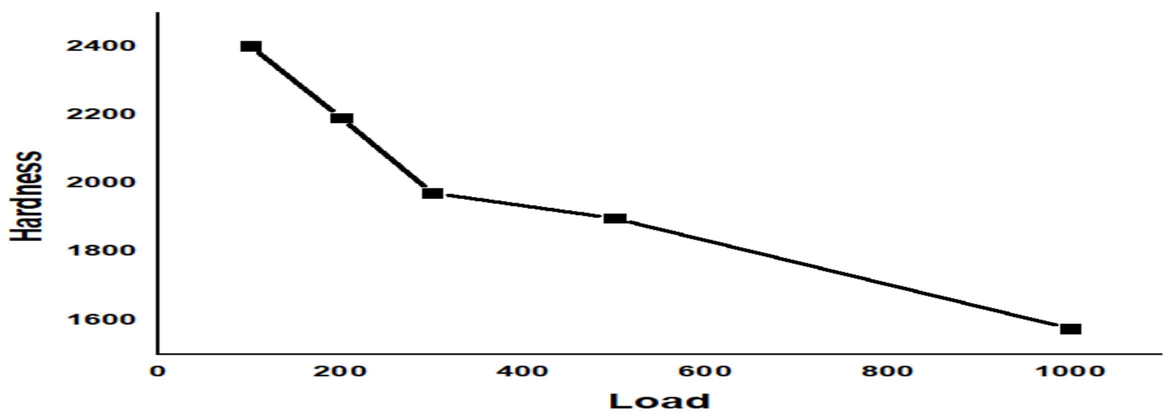

Figure2: Vicker hardness vs load for sample B

Figure 3 represent the Vickers hardness value of sample C. At a load of 100 gf the H_v is 22.55 GPa (2300 Kgf/mm²) and goes on decreasing with the increase in load thus when a load of 1 Kgf (1000gf) is applied the hardness values is 17.46 GPa (1780 Kgf/mm²).

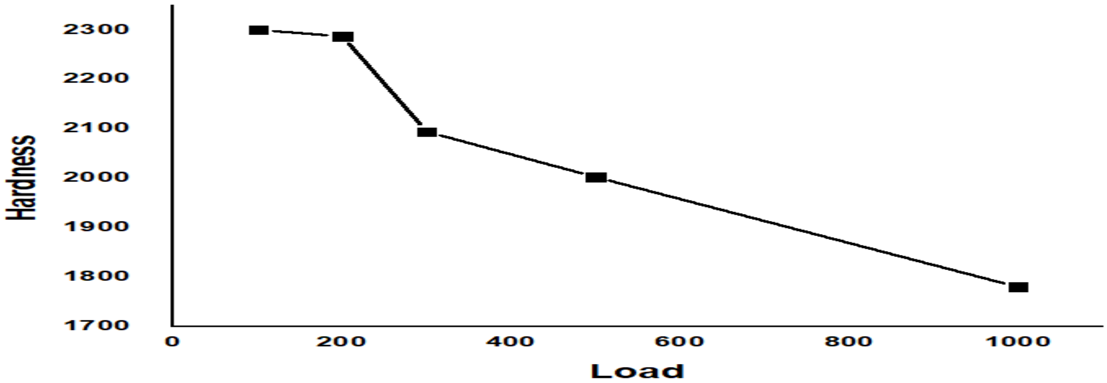

Figure3: Vicker hardness vs load for sample C

Figure 4 shows a comparison of hardness versus load for different compositions (samples A, B and C). From Figure 4 it is clear that sample C shows higher hardness, as in samples A and B. It is also noted that sample B shows higher hardness than virgin sample (sample A) as the reinforcement of TIN in the matrix increases hardness of sample B. In addition, sample C shows higher hardness values due to the addition of Al_2O_3 and BN because Al_2O_3 and BN are ceramic materials that are of higher hardness than TiN and result in higher hardness of sample C than sample A and sample B.

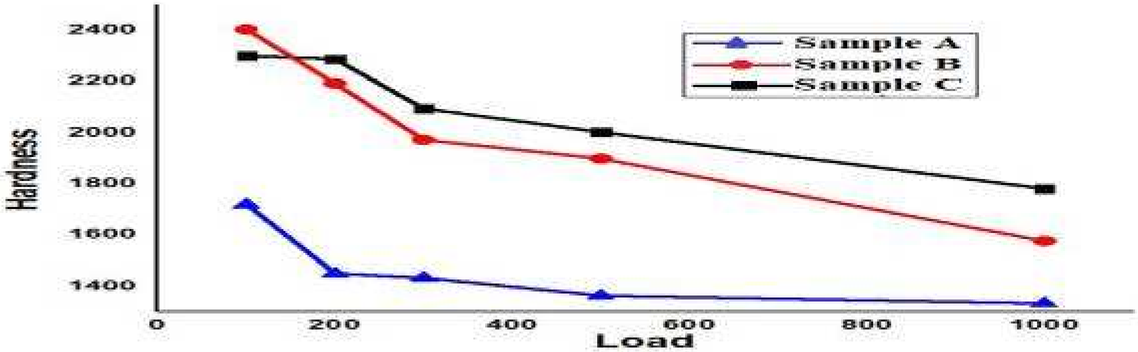

Figure4: Comparison of vicker hardness vs load for samples A, B and C

The Figures (5 -7) shows the Vickers hardness value with respect to time at a constant load of 0.5 Kgf. As the time is increased from 6s to 12s the hardness value decreases. But the variation of hardness value does not show much of change from 8s to 12s. The Figure 8 shows the comparison of hardness with dwell time. It is clear from Figure 8 that samples C shows better hardness with the increase in dwell time, as compared to, samples A and B.

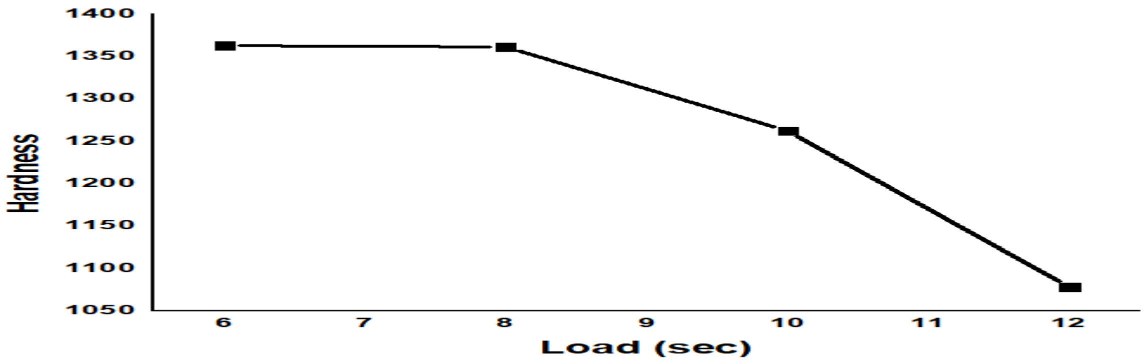

Figure5: Vicker hardness (hv) vs time at 500 gf for sample A.

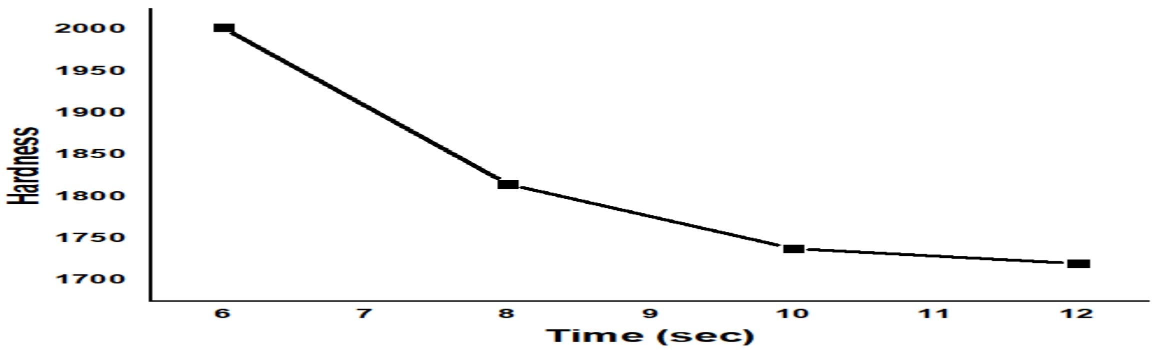

Figure6: Vicker hardness (hv) vs time at 500 gf for sample B.

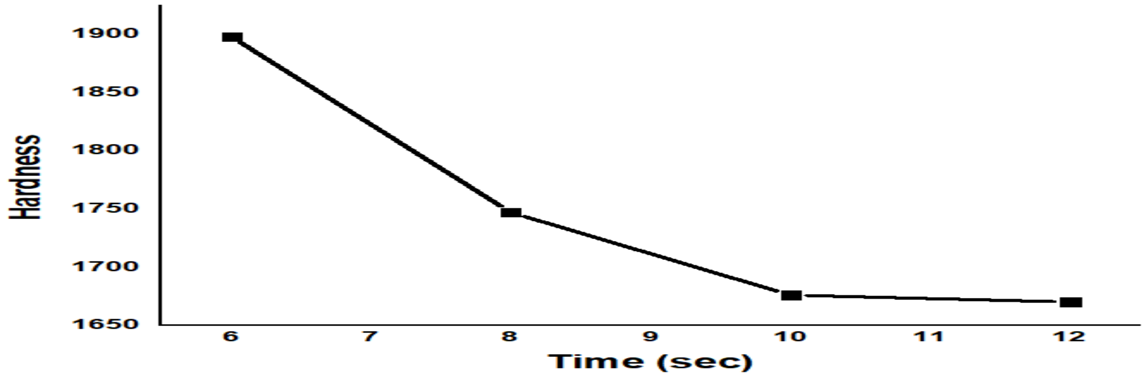

Figure7: Vicker hardness (hv) vs time at 500 gf for sample C.

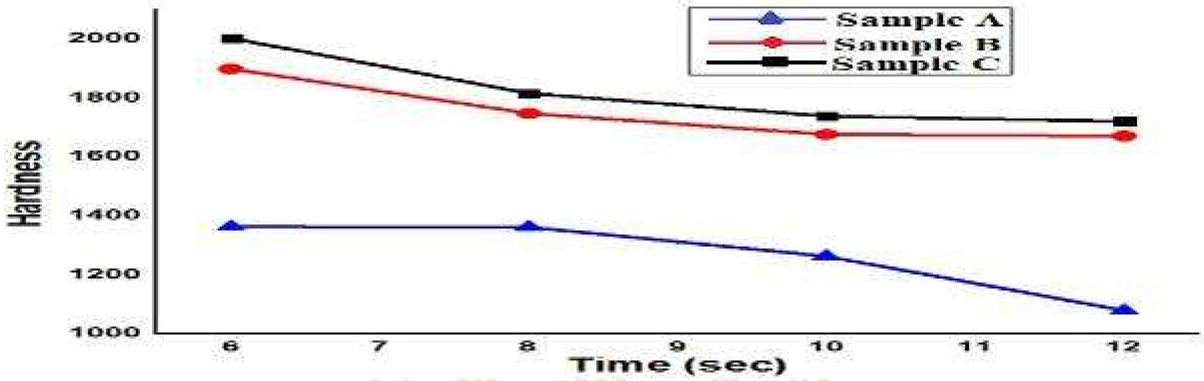

Figure8: Vicker hardness (hv) vs time at 500 gf for samples A, B and C.

5. CONCLUSION

In the present research work, 3 different types of materials were selected with different composition. The following conclusions have been made from all of the results which were obtained from experiments

- Micro hardness (H_v) of Si_3N_4 doped with titanium Nitride fabricated through sintering process is lower than Si_3N_4 doped with AlN and Y_2O_3 material.
- Micro hardness (H_v) of Si_3N_4 doped with BN and fabricated through sintering process shows higher hardness as compared to other prepared ceramics composites.
- Micro hardness (H_v) also decreases with increase in load due to indentation size effect.

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