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# A preliminary study on the physical and biocompatibility characteristics of Zirconia-Silicon Nitride bio-ceramics

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**Abstract.** Biomaterials are produced naturally or artificially using a various methods employing metals, polymers, ceramics or even some times composites. Especially for high load bearing orthopedic application like hip and knee joints the life expectancy of biomaterials is always increasing. In this work fabrication of a novel bio ceramics from orthopedic application with silicon nitride in zirconia matrix is developed. The Zirconia-Silicon Nitride composite is a novel material and it has been developed to combine the material advantages of zirconia and silicon nitride. The tradition powder forming process is used for the fabrication of the proposed Zirconia-Silicon Nitride composite in the form of pellets. Zirconia is added to the silicon nitride in the several wt% and compressed and sintered in the $1400^0$C. The Physical characteristics densities of the sintered pellets were determined by Archimedes principle and then compared with the theoretical density.  The density of the composite was studied preliminarily to assess the structural stability of the composite. The bio-inertness of the composite was evaluated by using the DISC DIFFUSION ASSAY method.

**Keywords.** Bio-ceramics, Powder forming, sintering agent, and Biocompatibility.

## 1. Introduction

In biomedical application ceramics materials taking the major role for the bio implants [1]. Basically ceramic material having high density and hardness compare than metal and polymers so ceramics suitable for the bio implants several of ceramic materials suitable for implant [2]. Silicon nitride ceramic having a great combination of chemical, mechanical and thermal properties, such as good wear resistance, high hardness, corrosion resistance, oxidation and fine thermal shock resistance [3]. Due to the high fracture toughness and good mechanical property $Si_3N_4$ has be selected or the bio implant material. In silicon nitride so many combination of composite investigation are going now. To avoid degradation of reinforcing in the silicon nitride the oxidation ceramics are best suitable for the reinforcement one of the best oxidation ceramic is zirconia [4]. By adding of zirconia to improve the fracture toughness of matrix using stress induced tetragonal phase transformation, it absorbed the energy for fracture near the crack. In the ceramic matrix $ZrO_2$ has been used as a toughing agent. But the density of the $ZrO_2$ is found to be higher than other similar orthopedic implants. The weight of the implant can

be optimized to achieve a good strength to weight ratio material [5]. So it was proposed to develop a composite with Zirconia as the matrix and silicon nitride as toughening agent.

## 2. Materials and Method

The commercially available Silicon Nitride and Zirconia powder were procured at Kovai metal mart industry in Chennai. Both fine composite powders were mixed in the various proportions (5%, 10%, 15%, 20%, 25%, 30%, 35%, 40%, 45% and 50% wt %) using the ball mill [6].



**Figure 1.** Silicon nitride Powder



**Figure 2.** Zirconia Powder

Silicon nitride and zirconia powder in the different grain structure so both materials need to mix to gather for getting the combine property so the powder should mixed in 1 hour in ball mill [7]. After milling the powder to achieving proper blending small green pellets of the length 10 mm and diameter 10 mm are made by using the hydraulic press in the room temperature . Then the small green pellets are sintered at 1400ºC in a BOX Furnace. By using the Archimedes principle the porosity, water absorption and bulk density of the sintered specimens were found[8].
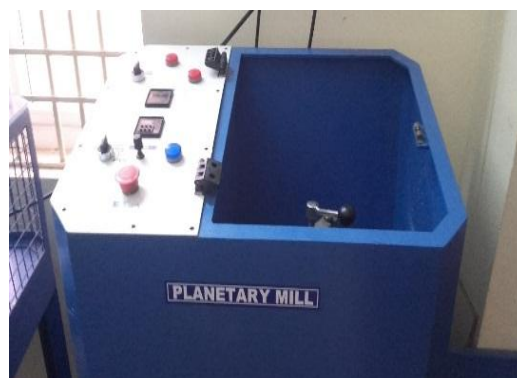


**Figure 3.** Ball milling apparatus

*2.1 Density Measurement*

The density of the composite was found using Archimedes principle as per the ASTM standards. At the outset, sintered mass of the specimen were found (Figure 4) then, the specimen was retained in hot water bath at a temperature of 70°C for 2 hours.

**Table1.** Masses of composites at various stages

| S. No | Sintered Weight (g) | Soaked Weight (g) | Suspended Weight (g) |
|---|---|---|---|
| 1 | 3.3 | 3.6 | 2.8 |
| 2 | 3.2 | 3.3 | 2.6 |
| 3 | 3.2 | 3.2 | 2.5 |
| 4 | 3.2 | 3.3 | 2.7 |
| 5 | 3.0 | 3.1 | 2.5 |
| 6 | 3.1 | 3.2 | 2.6 |
| 7 | 3.1 | 3.2 | 2.5 |
| 8 | 3.2 | 3.3 | 2.4 |
| 9 | 3.4 | 3.5 | 2.6 |
| 10 | 3.3 | 3.4 | 2.7 |
| 11 | 3.0 | 3.1 | 2.3 |
| 12 | 2.9 | 3.3 | 2.3 |

Where,

$$\text{Bulk Density} = \frac{W_{sintered}}{(W_{soaked} - W_{suspended})}$$

$W_{sintered}$ - sintered weight in gram,

$W_{soaked}$ - soaked weight in gram,

$W_{suspended}$ - submerged weight in gram.

Then weighed to assess the soaked mass was measured as shown in Figure 5. Finally the suspended mass was measured using beaker filled with water in which specimen was suspended in water and the weight of the ceramic compacts is measured (Figure 6) [9]. The weight of sintered, soaked and suspended weights are given in the Table1.Here we find the weight of sintered, soaked and submerged weight of material through Archimedes principle [10].

**Figure 4.** Measurement of Sintered Weight



**Figure 5.** Measurement of suspended weight



**Figure 6.** Measurement of Soaked Weight

In the experiment method the mass of the specimen is found then the volume of the specimen is calculated by measuring it with the digital vernier caliper . Then by using the relation between the mass and density in terms of volume the density of the composite was found [11].

*2.2 Antibacterial Test (Disc Diffusion Assay)*

Each material is tested with three organismsthere are, STAPHYLOCOCCUS AUREUS, STAPHYLOCOCCUS PAUCIVORENS. An autoclave is used to improve the temperature and pressure difference in the ambient air it act as a pressure chamber, sterilization in a medical application and  cure coatings and vulcanize rubber and for hydrothermal synthesis in chemical industry [12].An autoclaves sterilizes operated by steam and attain high temperature of $115^0$C/0.689 bar., $120^0$C/1.034 bar., and $135^0$C/2.068 bar. The temperature,pressure and operation time is dependent on the desired degree of sterilization.The air is passed over the entire body with the laminar flow with uniform velocity at steady state.The cabinet works by air flow in haggard through stack of HEPA filters. This is intended to create a working atmosphere which is particle-free and ensures product protection [13].

**Figure 7.** Autoclave



**Figure 8.** Laminar Air Flow Chamber

*2.3 Antibacterial Test (Disc Diffusion Assay) Procedure*

To attain a plate culture of one of the organisms to be tested the following procedure is followed. By means of a sterile loop, emulsify a colony from the plate in the sterile saline solution.Mix thoroughly to make sure that none of the solid material of the colony is able to seen. The procedure is repeated until the turbidity of the briny solution matches the standard available.The swab is dipped in the broth culture of the organismand mildly squeeze to remove the extra fluid from the tube [14].The swab is used to streak a Mueller-Hinton agar plate for a pasture of growth. This is prepared by streaking the plate at first in one direction and then streaking at right angles to the first streaking direction, and then finally streaking crossways and finally the plates allow to dry for 5 minutes. By using a dispenser several disks at the exact distance apart the antibiotic disc can be positioned on the surface of the agar. To attain the dispenser containing the exact antibiotic disc for the organism for our requirement the following steps are followed[15]. The dispenser is positioned above the surface of the plate and with the help of on lever/plunger the disks are dispensed. Then by the sterile forceps or a loop, the disks are mildly pressed onto the surface of the agar, taking consideration of avoiding pressing them into the agar.

## 3. Results and Discussion

The following table 2 shows the density of Theoretical, Archimedes and Normal experimental method. The theoretical density (TD) given by the "RED-BARS", give us the idea of the sintered density of the zirconia-Silicon nitride composite system by varying the percentage of silicon nitride. With pure zirconia without the addition of silicon nitride a TD of 6.04 $g/cm^3$was calculated. But, this higher density can be achieved only at $1600^0$C[9]. Similarly pure silicon nitride without addition of zirconia has found to have 3.31 $g/cm^3$ calculated TD. The experimental and the closeness in the prediction of by Archimedes principle and theoretical method is observed in the whole range. The density of the composite was found to be maximum and close acceptance with the TD when 15 to 25 wt% of silicon nitride is added to the zirconia matrix (Figure 9).

**Table 2.** Calculation of Density Measurement

| Percentage of Si$_3$N$_4$ | Weight g | Height Cm | Diameter cm | Bulk Density g/cm$^3$ | Theoretical Density g/cm$^3$ | Density g/cm$^3$ |
|---|---|---|---|---|---|---|
| 0 | 3.328 | 1.175 | 0.956 | 3.94 | 6.04 | 4.13 |
| 5 | 3.194 | 1.162 | 0.912 | 4.21 | 5.90 | 4.57 |
| 10 | 3.249 | 1.126 | 0.919 | 4.35 | 5.77 | 4.57 |
| 15 | 3.201 | 1.005 | 0.897 | 5.04 | 5.63 | 5.33 |
| 20 | 3.044 | 1.044 | 0.91 | 4.48 | 5.49 | 5.00 |
| 25 | 3.133 | 1.014 | 0.917 | 4.68 | 5.36 | 5.17 |
| 30 | 3.156 | 1.182 | 0.952 | 3.75 | 5.22 | 4.43 |
| 35 | 3.247 | 1.352 | 0.974 | 3.22 | 5.08 | 3.56 |
| 40 | 3.396 | 1.457 | 0.926 | 3.46 | 4.95 | 3.78 |
| 45 | 3.4 | 1.156 | 0.886 | 3.32 | 4.81 | 4.71 |
| 50 | 3.062 | 1.152 | 0.932 | 3.15 | 4.68 | 3.75 |
| 100 | 3.014 | 1.32 | 1.051 | 2.63 | 3.31 | 2.90 |
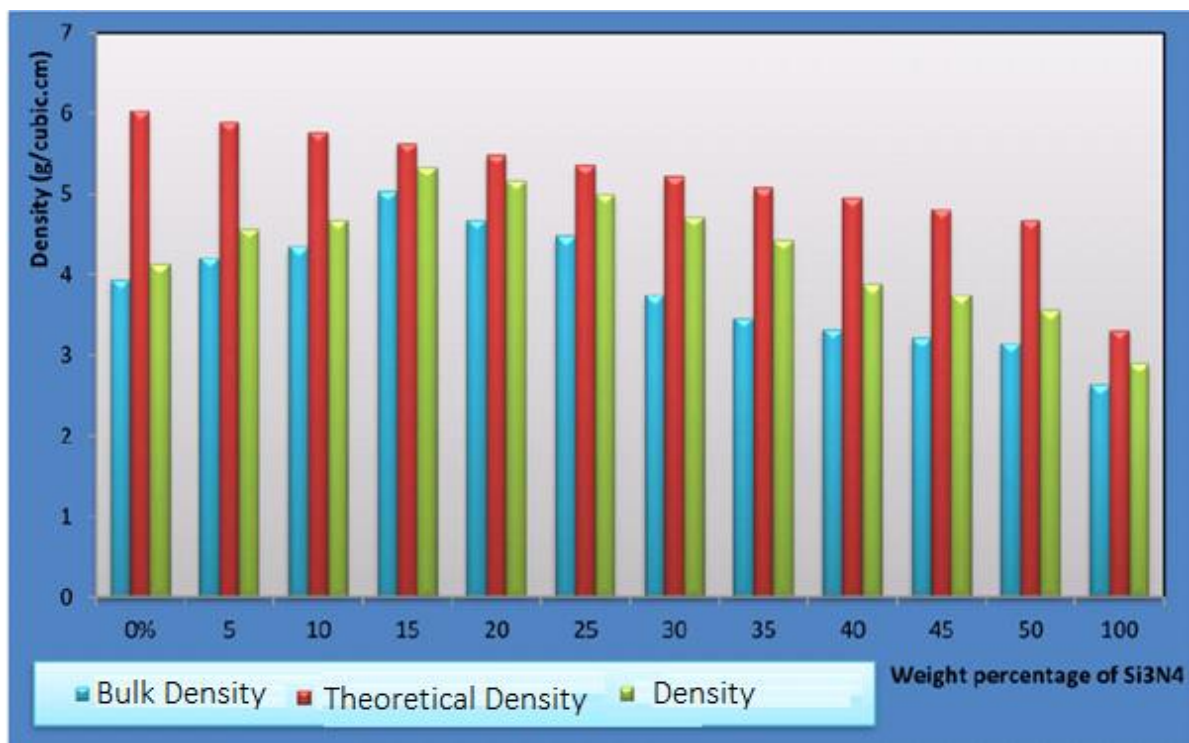


**Figure 9.** Density plot of the Zirconia-Silicon nitride composite

The composite is found to have a maximum density of 5.17 g/cm$^3$ at the sintering temperature of 1400$^0$C. The density of the composite was found to be reduced as well as sintering temperature of the pure zirconia system is reduced to 1600$^0$C to 1400$^0$C. The silicon nitride has acted aiding agent and also help in reducing the final composite density which can certainly reduce the weight of the products produced by the composite.
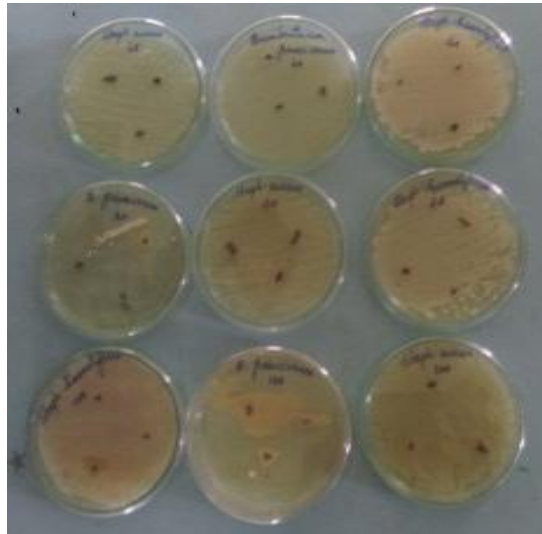
**Figure 10.** Bacterial Culture

The 15,20 and 25 wt% of the composite with the maximum density were taken and the DISC DIFFUSION ASSAY is done and is presented in Figure 10. The test result suggested that there is no growth of the three spices of microbe's colony in the fabricated composite. This is an excellent confirmation that the fabricated composite of bio-inertness of the fabricated composite.

## 4. Conclusion

The addition of silicon nitride in zirconia at the ratio of 15% to 25% of silicon nitride has found to reduce the density of the composite which will be helpful to reduce the final product weight. By addition of silicon nitride has proven to improve the sintering property of zirconia. The DISC DIFFUSION ASSAY for each material is tested with three organisms namely Staphylococcus Aureus, Staphylococcus Haemolyticus and Staphylococcus Paucivorens and thee bio-inertness of the composite were validated.

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