

# Processing Of Zirconia Ceramic Nanocomposites for Dental Applications

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**ABSTRACT-** The 3% yttria-stabilized zirconia matrix ceramics doped with 10% (volume percent) nano- $Al_2O_3$  ( $10Al_2O_3-90ZrO_2$ ) were fabricated by microwave sintering. The relative density of  $10Al_2O_3-90ZrO_2$  composite ceramics reached 99.18% by microwave sintering. Its hardness, wear rate and friction coefficient were 10.42 GPa, 0.35  $\mu m$ , and 0.45, respectively. Adding nano- $Al_2O_3$  could improve microstructure of the ceramics and have an effect of dispersion strengthening, which enhanced the properties of the materials. Compared with the other ceramics, the properties of  $ZrO_2$  composites with aluminium addition were greatly improved.

**Key Words:**  $Al_2O_3$ ,  $ZrO_2$ , Nanocomposite, Dental Ceramic.

## 1. INTRODUCTION

Nanocomposites have allowed significant breakthroughs in bio-engineering and medical fields. Nanocomposites is a multiphase solid materials that incorporate Nano sized (<100nm) particles into a matrix of standard material, which lead to drastic improvement in mechanical, physical and other functional properties. The unique attributes of nanocomposites are their reactivity, greater surface area and robustness compared to their bulk counterparts. Nanocomposites are widely used in biomedical, microelectronics, and aerospace applications.

### 1.1 Nanocomposites for Dental Implantations

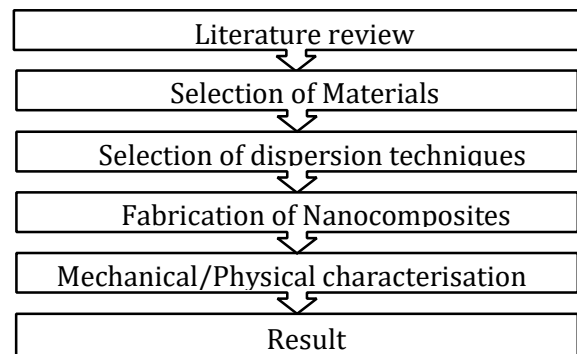
The metal based dental and orthopedic dental implant including stainless steel, titanium and cobalt-chromium are widely used because of their suitable properties like high mechanical strength and light weight chemical stability. Recently the zirconia is the best option for metal free implants and alternative to titanium implants. Zirconia implants are hypoallergenic, inert and non-corrosive.

## 2. OBJECTIVES

Develop alumina reinforced yttria-stabilized zirconia ceramic composites by microwave sintering technique. Develop alumina-silicate reinforced zirconia composite by microwave sintering technique. Evaluation of the mechanical and physical properties and of the prepared zirconia ceramic composites.

## 3. METHODOLOGY

Materials selection that is, zirconium oxide, yttrium oxide, aluminum oxide. Mechano-Chemical Processing of composite in different ratio of reinforcing materials by planetary ball milling. Fabrication of the composites by uniaxial compression. Determination of physical properties like green density, volume and area. High temperature sintering of the compressed samples. Evaluation of mechanical and physical properties.



**Fig3.1.** Methodology Diagram

## 4. EXPERIMENTAL PROCEDURE

Zirconium/aluminium nanocomposites were prepared. The concentration of Zirconium/aluminium as mentioned in the table-4.1. Appropriate amount of 3% Yttrium stabilized zirconium and aluminum oxide were accurately weighed in electronic weighing balance. The materials were homogenized using planetary ball milling. The powders were taken in a metal container having toughened zirconia balls of 1mm size and sufficient amount of water/isopropanol (1:1 ratio) is added and milled for 30min at 250rpm. The amount of balls and powders were maintained in 1:3 ratio for efficient homogenization. After milling the balls and slurry was separated using mesh. The slurry was dried at 100°C for 12hours and sintered at 1500°C.

#### 4.1 Materials composition for making composites

Materials	Sample1	Sample2	Sample3
Zirconia	90%	80%	70%
Alumina	10%	20%	30%

The dried powder was then grinded with agate and dry milled to get fine fractions of uniform size. Then the powder was compacted by adding 2% CMC polymer binders using uniaxial hydraulic press under the load of 3 ton. The composites were compacted into 11mm (Dia) cylindrical pellet and 40mm (Dia) circular disks. Then the compacted pellets were preheated to reduce the moisture content. Since the moisture more than 1% will propagate thermal crack. Hence the pellet were kept in hot air oven maintained at 110°C for 10hours. After cooling to room temperature the weight of the pellets were taken and density of the green samples were measured using the formula-1 and then sintered at 1500°C.

$$\text{Density (g/cm}^3\text{)} = \text{Mass/Volume} \text{----- (1)}$$



Fig 4.1. Sintered Al<sub>2</sub>O<sub>3</sub>/ZrO<sub>2</sub> Composite

## 5. RESULTS AND DISCUSSION

### 5.1 Wear and friction Test

The zirconia composites fabricated in the form of cylindrical shaped pellet with 1mm diameter and 4mm thickness were subjected to wear and friction resistance analysis. The wear and friction behavior of the zirconia/alumina composites were presented in the Chart 5.1a and 5.1b respectively. The wear resistance properties of the composites increases with increase in the concentration of reinforcing materials. In zirconia/alumina composites the reinforcing of alumina imparts wear resistance to the zirconia. The composite made with 30% reinforcement of alumina performs well and have a friction coefficient of 0.45 over a time of 500 seconds. The improved wear behavior is due to the mixed phase formation of zirconia grains in which the

alumina grains stacked randomly in between the zirconia grains. (1A,2A,3A are symbolic representation of three sintered component respectively).

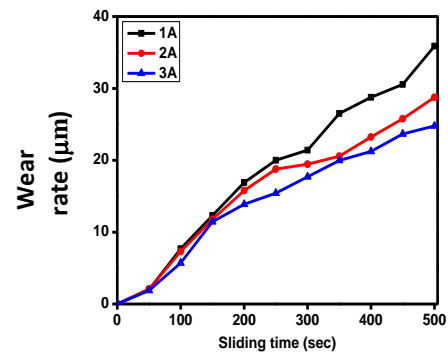


Chart 5.1(a) Wear behavior of Zr/Al

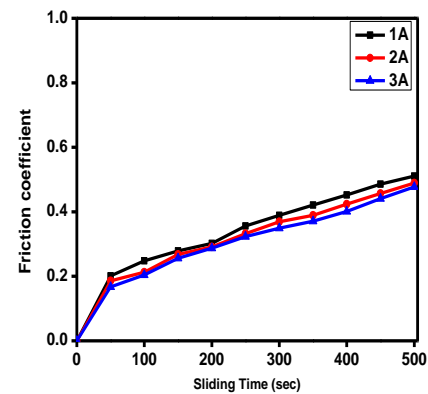


Chart 5.1(b) Friction coefficient behavior of Zr/Al

### 5.2 Colour Analysis

Colour analysis is an excellent tool to measure the whiteness, brightness and yellowness of the dental samples. The brightness of the materials were used theoretically calculated as per IS standards, the yellowness is the difference between the reflectance at the wavelength of 457 and 570nm. In case of zirconia-alumina composite the brightness improves and yellowness decreases with increase in concentration of alumina, hence these composite is fit dental implantations which resembles the colour of the natural teeth.

Parameters	Zirconia-alumina composite		
	1A	2A	3A
L*	93.82	98.62	100.15
a*	-0.67	-0.29	-0.12
b*	9.15	3.14	1.74
Whiteness	88.93	96.54	98.34
Brightness	86.04	96.99	100.87
Yellowness	6.98	4.96	3.27

Table 5.1: colour values of the zirconia composites

### 5.3 Phase Analysis

The formation of multiple phases with respect to sintering temperature were identified from the corresponding powder X-ray diffraction pattern of zirconia/alumina composites. The crystalline size of the composites were calculated based on Scherrer formula given as follows,

$$\text{Scherrer equation, } D = \frac{k\lambda}{\beta \cos\theta}$$

Where, D = crystal size

k = shape factor

$\lambda$  = wavelength of the x-ray used

$\beta$  = full width half maximum of the peak (FWHM)

$\theta$  = diffraction angle

The XRD pattern of the zirconia/alumina composite showing characteristic peak for tetragonal and monoclinic phases of zirconia along with alpha-alumina phases. The monoclinic phases of zirconia is dominated, since above 1200°C monoclinic phase will be developed and above that temperature tetragonal grains are developed which imparts toughness to the composite. The formation of alpha-alumina will enhance the toughening mechanism of zirconia composite. The crystal grain size is found to be 75nm.

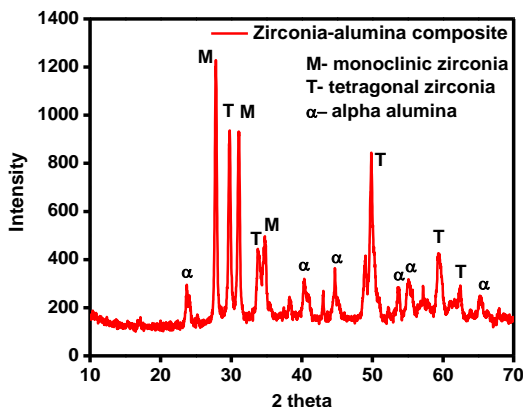


Fig5.2 XRD of Zr/Al composite

### 5.4 Hardness Test

From Table 5.2 it was observed that the maximum Vickers hardness is 13.4502 GPa for the composition containing 12.3 wt. % Al<sub>2</sub>O<sub>3</sub> and minimum Vickers hardness is 10.3758 GPa for the composition containing 0.75 wt. % Al<sub>2</sub>O<sub>3</sub>.

Table 5.2: Vickers Hardness test results

Sample	Vickers Hardness Hv (GPa)
1	10.3758
2	11.7637
3	13.4502

### 5.5 Micro structural analysis

The microstructural features of the selected sample of Zr/Al were analyzed with SEM and given in the fig 6.5. From the SEM image of the Zr/Al the grains are found to be diffused with each other having sharp defined edges of overlap disordered networks. Because the formation of grains with mixed phases of zirconia (tetragonal and monoclinic) fused with alumina phase at higher temperature. The alumina act a nucleating agent to control the grain size and atomic diffusion between grains.

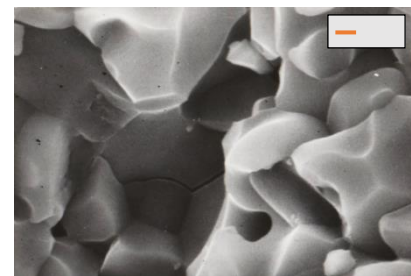


Fig 5.3 SEM of Single Zr/Al Component

### 6. CONCLUSION

From the critical review of the materials for the dental implantations, zirconia composites are identified as an excellent candidate. Hence in this project work the development and mechanical properties of zirconia/alumina composites were explored. The zirconia/alumina composites were successfully developed by microwave sintering technique at 1500°C with varied composition of reinforcing material and matrix material. The systematic characterization were also carried out using relevant techniques. The dependency of the bulk density and zirconia phase formation with respect to the concentration of reinforcing material was validated in this study. The influence of aluminium phase transformation on the zirconia phase and grain property noticed from the XRD analysis.

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