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**Research Article**

# Synthesis and Characterization of Zirconia-Based Ceramics: A Comprehensive Exploration of Film Formation and Mixed Metal Oxide Nanoparticle Synthesis

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**Abstract:**

Due to its strong ionic conductivity and superior mechanical qualities, zirconia-based ceramics are frequently used in electrochemical devices. Today, zirconium oxychloride octahydrate and ethanol have been developed for the purpose of reducing zirconium thin films on glass, single-crystal silicon p-type, substrates. The phase composition and properties of the films, as well as the physicochemical processes involved in film formation, have all been investigated. Zirconia mixed metal oxide nanoparticles with various physicochemical properties can be created using a variety of synthetic processes in conjunction with additional variables like concentration, PH, type of precursor utilized, etc. In order to create zirconia mixed metal oxide nanoparticles, many synthetic techniques, including sol-gel, hydrothermal, and coprecipitation methods are discussed in this article.

## 1. Introduction

Main All Numerous biomedical uses for ceramics, metals, and polymers have emerged in recent years, particularly in dentistry. A few notable qualities of metals like Ti that make them valuable in biomedicine are good mechanical properties, strong corrosion resistance, and high biocompatibility. However, various challenges with bio-toxicity, inadequate wear resistance, and elastic modulus have recently encouraged scientists to adopt ceramics rather than conventional alloys [1]

There are three different types of crystalline zirconia that can exist: monoclinic (M), which is stable from room temperature to 1170 °C, tetragonal (T), which becomes stable from 1170 to 2370 °C, and cubic (C), which is preferable above 2370 °C. [2].

The development of zirconia-based better X-ray resistance than bones, making it easier to observe patients after surgery and having no impact on NMR imaging; decent looking. With the gingiva mucosa, it is possible to obtain the best creative synchronization while avoiding the dark grey of metal foundation piles. 141 $ZrO_2$ ceramic endosteal implants were placed by Pigot in the lower jawbones of 39 patients in 1997 to support a complete lower denture, and they had a superior clinical outcome than Ti implants throughout the same time period [1-4]; With the use of $ZrO_2$ ceramic inside-fang and inside-bone implants, Schultze created an animal model undergoing apiceotomy that allowed for the formation of excellent root canal sealing as well as osseointegration, which increased the stability of the teeth; Percy Milleding examined the traits salivary protein and plasma protein on various dental ceramics, with zirconia ceramics showing no adsorbed salivary protein but all plasma protein showing up on the bio membrane at the surface. Not only did ZrO2 ceramics experience this weak adsorption to salivary protein, but also Al2O3 ceramics. The development of the salivary pellicle and the bacterial colonization of dental surfaces are strongly connected, which accounts for the clinically observed decreased dental plaque adhesion rate on all-ceramic crowns. In contrast, ZrO2 ceramics have a strong affinity for hemocyanin (such as fibrinogen),.As a result, the strong link between ZrO2 ceramics and the tissue is favored by such high haemocyanin adsorption, which implies that ZrO2 ceramics are suited for use as an oral planting material [5, 6]. Additionally, it promotes the health of the implant and the soft tissue around it as well as the link between the soft gum tissue at the cervical margin and the implant. As a material for replacing bone, zirconia ceramics has great biocompatibility and biological activity and can develop a close chemical link with the bone tissue of an organism. It has emerged as the most promising material for replacing hard tissue ever since it was first introduced as a novel biological material in the 1970s. However, its poor strength and severe fragility limited. Successful all-ceramic series that are used in clinical practice are published in The Open Materials Science Journal, Volume 5, 2011, page 179. Zirconia Toughened Alumina (ZTA) ceramics, with a three-point flexural strength of 513MPa and a fracture toughness as high as 4.0MPa.m1/2, can be utilized to repair posterior crowns and bridges. Suarez observed the fixed bridges of the posterior teeth that had been restored with In-Ceram Zirconia for three years, and he concluded that the all-ceramic fixed bridges had shown positive clinical results throughout that time. However, due to its brief clinical application, long-term observation was necessary. When used for dental inlays, crowns, and bridges, phase-transition ZTA toughened ceramics were examined for their chemical stability and low-temperature ageing performance. The phase-transition ZrO2 toughened ceramics, in his opinion, had superior. According to research by Quinn et al. on the influence of microstructures and chemical composition on the mechanical qualities of dental ceramics, ZrO2 ceramics have superior mechanical properties. Chinese academics have also vigorously investigated the use of zirconia ceramics in dentistry. In their research on In-Ceram materials, Chai Feng, Xu Ling, and Liao Yunmao introduced nanoscale materials. They created zirconia-toughened nanometric composite ceramics by mixing nanometric zirconia powder with micro-metric Al2O3 powder, and they thoroughly investigated the correlations between the powder particle size, the content of nanometric zirconia powder, the sintering temperature, etc. [7-10]. Using cutting-edge nanomaterial technology, to produce more excellent repair-purposed all-ceramic materials (figure 1). ZTC is a brand-new fine ceramic substance that nanometric composite zirconia powder, the material was made by sintering at 1450°C under normal pressure and produced by cold isostatic pressing following dry pressing at 200Mpa. The necessary specimens were then obtained following diamond cutting and a precision grinder's ground finish. The Archimedes drainage method was used to examine the specimens' density and apparent porosity. A vernier calliperse was used to measure the length variation of the specimens before and after sintering, and the linear contraction was tested. The specimen phase analysis was performed using an X-ray diffractometer. The section micrograph of the specimens was examined using SEM. The three-point flexural strength and fracture toughness were tested using a universal mechanical testing machine. Micro hardness testing was performed.

## 2. Properties of zirconium oxide materials:

$ZrO_2$ is stable in contact with Si and has a high dielectric constant of about 25 [5-8]. It has a high density of 5.74 gm/cm3 and a refractive index that ranges from 1.84 to 2.23, which could aid in improving impurity diffusion (table 1). The band gap of $ZrO_2$ is 5.8 eV [9], which is high enough to allow for minimal gate leakage current. It melts at a temperature of almost 2950 k. The ability of diluted hydrofluoric acid (HF), which is utilized in MOS to remove $SiO_2$ from Si substrate, to etch $ZrO_2$ was also observed [10].

## 3. ZRO2 thin films fabrication

There have been a number of attempts in recent years to create $ZrO_2$ thin films. These techniques can be broadly divided into five categories: wet

***Table1.*** *Properties of* $ZrO_2$

| S.No | Material | $ZrO_2$ |
|---|---|---|
| 1. | Band gap | 5.8 eV |
| 2. | Refractive index | 1.84-2-23 |
| 3. | Dielectric constant | 25 |
| 4. | Melting point | $2677^0$C |
| 5. | density | 5.74gm/$cm^3$ |

***Table 2.*** *Physical and Mechanical Properties of Zirconia.*

| Property | Unit | Zirconia |
|---|---|---|
| Thermal expansion | $K^{-1}$ | $11\times10^{-6}$ |
| Thermal Conductivity | W/m.K | 2.5 |
| Melting Point | $^{o}$C | 2715 |
| Density | Kg/$m^{-3}$ | 5680 |

chemical process, thermal evaporation, sputtering, and pulsed laser deposition (PLD). Wet chemical methods are typically referred to as non-vacuum methods and physical methods as vacuum methods [11] (table 2). The benefits of the wet chemical technique are

- Easily handle
- Low cost
- Less time required
- Less power consumption
- Low temperature deposition

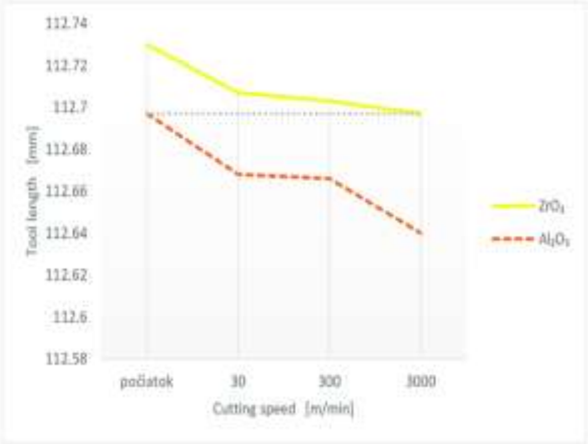

***Figure 1.*** *Cutting Speed on Dimensional tool wear*

## 4. Experimental Techniques

The substrates were submerged in a solution (a 1:1 combination of sulfuric acid and hydrogen peroxide) and then washed with deionized water. The substrate was heated at 70 °C for 5 minutes, and then rinsed with ethanol. 0.1 mol of $ZrOC_{l2}.8H_2O$ and 50 mL of ethanol were used to create the $7ZrO_2$ sol, which was then aged for three days at room temperature. Spin coating at 3000 rpm for 30 seconds was used to deposit the films on the substrates. The films were dried at 70 °C for 5 min, then annealed at 500 °C for 3 min at a rate of 10 °C/min. The Agilent E4980A precision LCR metre was used to measure the capacitance-voltage. The prepared material's structural features. The optical properties were examined with a UV-visible spectroscope. The experimental details are depicted in the flowchart in Fig. 2.

## 5. Characterization Techniques:

To investigate the materials' structural features using x-ray diffraction, morphology using a field emission electron microscope, and optical characteristics Electrical properties, Fourier transform infrared spectroscopy, and UV-visible spectroscopy We carried out capacitance-voltage measurements.

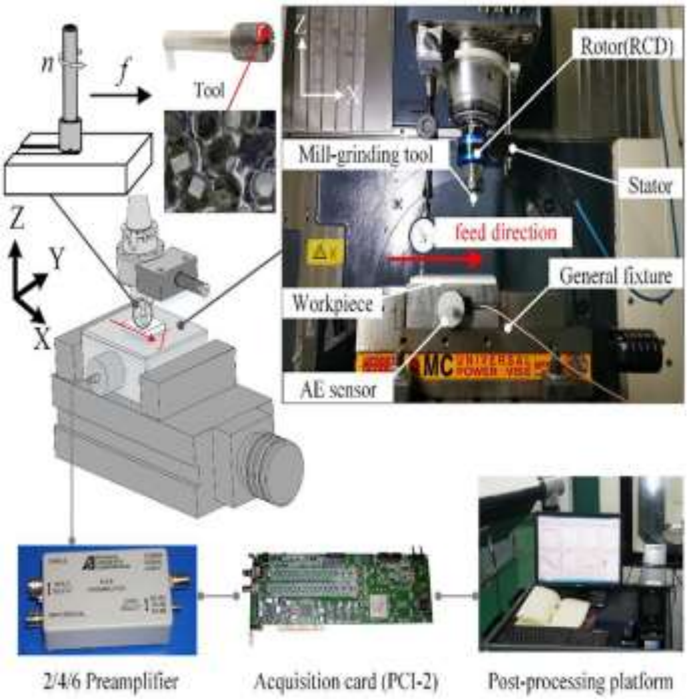

***Figure 2.*** *Schematic diagram of the Experiment*

### 5.1 X-Ray Diffraction

This method is used to examine the samples' structural specifics. By using this method, it is possible to establish the crystal's size and form, average atomic spacing [12], and orientation of the single and multilayer crystals.

The Bragg's law, which describes the interaction between an X-ray beam of a specific wavelength and an equation-defined crystalline surface,

$n\lambda = 2d \sin\theta$
$\lambda$= X-ray wavelength
d = distance between lattice planes
$\theta$= angle of incidence with lattice plane
n = integer

Different peaks are obtained when the intensity of that deflected light is plotted with respect to the angle of diffraction. The peak width of those peaks can be used to determine the material's structure as well as the size of the crystalline structure. The sample's crystalline size diminishes as the peak width widens. This suggests that the relationship between peak width and crystal size is inverse.

### 5.2 Field emission scanning electron microscope (FESEM):

In addition to offering clearer, less distorted images than SEM, FESEM also offers three to six times the resolution. By using FESEM, low voltage pictures of high quality are produced with little electrical charging of the materials. There is essentially no longer a requirement for conducting coatings on insulating materials.

### 5.3 U-V visible spectroscopy

The term "ultraviolet-visible spectroscopy" refers to spectroscopy in the ultraviolet-visible spectral region that involves absorption, reflectance, or transmittance [12]. The colour of the chemical is directly impacted by the visible range absorption or reflectance. In contrast to fluorescence spectroscopy, this method.

By using UV-Visible Spectroscopy, we can calculate the band gap of thin film using relation

$(\alpha h\nu)\ 1/n = B\ (h\nu - E_g)$

B: a constant related to a transition probability

$E_g$ : band gap

N: an index characterizing the transition step

n=2 is an indirect transition step

n= ½ is a direct allowed transition step

### 5.4 C-V Measurement

The most popular method for electrically characterizing high-k gate dielectrics and metal gate electrodes is the capacitance-voltage (C-V) technique. The C-V test is used to determine the dielectric constant, EOT, flat band voltage, fixed charges, bulk charges, and interface state density of high-k gate dielectrics, among other significant electrical parameters. When plotting the flat band voltage and EOT of MOS capacitors with different thicknesses, the work function of the metal gate electrodes can also be determined from the C-V measurement. The concentration of Si substrate doping can be determined by the inversion capacitance.

## 6. Parameters Affecting the Ceramics

### 6.1. Grain size:

One of the most significant elements impacting light scattering is the link between particle size and light wavelength size60). Light scattering increases significantly in a material with grain size that is similar to the wavelength of the light61). Generally speaking, light scattering in materials containing a matrix and particles is directly influenced by the refractive indices of the matrix and particles as well as the chemical makeup of the particles. When exposed to visible light wavelength, materials with particles smaller than roughly 0.1 m look less opaque because there is less reflection and absorption than in the case of materials with large particles60). Despite the fact that there are few particles per unit volume and that the opacity may be decreased by less reflection, materials with particles larger than 10 m have surface reflection, absorption, and refraction.

Grain size effect: Increasing the grain size gradually reduces the grain boundary light scattering effect, allowing the light beam to flow through the material and come into contact with the grain borders less frequently. Therefore, the diffuse-transmission mechanism determines the translucency of coarse-grained ceramics, and more light transmission occurs in thinner ceramics because there are less contacts between the light and the grain boundaries54). However, the relative strength of ceramics decreases as the size of the grains in the microstructure increases.

### 6.2. Oxygen vacancies:

Grain size effect: Increasing the grain size gradually reduces the grain boundary light scattering effect, allowing the light beam to flow through the material and come into contact with the grain borders less frequently. Therefore, the diffuse-transmission mechanism determines the translucency of coarse-grained ceramics, and more light transmission occurs in thinner ceramics because there are less contacts between the light and the grain boundaries54). However, the relative strength of ceramics decreases as the size of the grains in the microstructure increases.

### 6.3. Dopants:

The dopants used in the chemical composition of the Y-TZP ceramics are related to their translucency36). Dopant, an oxide that serves as a tool for designing grain boundaries and controlling the composition of $ZrO_2$ grain boundaries. For doping Y-TZP ceramics, a variety of oxides including $Al_2O_3$, $Sc_2O_3$, $Nd_2O_3$, and $La_2O_3$, MgO, and $GeO_2$ may be used. Alumina is typically used as a dopant in 0.25 wt% of Y-TZP ceramics. Less alumina is utilized in new generation ceramics, particularly monolithic Y-TZP, to provide a more translucent material. In Y-TZP ceramics, dopant oxides can separate at the $ZrO_2$ grain boundaries, and dopant segregation is a crucial factor in guaranteeing hydrothermal stability and excellent transparency

### 6.4. Sintering Temperature:

Ceramics' crystal composition and sintering process both affect the material's translucency. Shorter sintering periods ought to be taken into account if more translucent Y-TZP ceramics are desired. Grain growth in the Y-TZP ceramics is a result of longer holding (dwell) times during sintering, which affects translucency. However, it is not advisable to raise the sintering temperature in order to increase the amount of translucent ceramics produced by the grain size12. Higher sintering temperatures were shown to cause Y to migrate to grain boundaries and an uneven distribution of the Y-stabilizing ions, which causes cubic phases, which is undesirable. This would result in metastability and LTD of Y-TZP. Increasing the sintering rate is one method for enhancing the colour and translucency properties of monolithic $ZrO_2$.

### 6.5. CS, MS and spark plasma sintering (SPS):

Conduction, convection, and radiation are the heat transmission modes for Y-TZP ceramics that are sintered conventionally. In CS, the Y-TZP ceramic's exterior surface receives heat application. Thermal conduction helps it get there during the sintering process, which results in strong temperature gradients and tensions inside the material. Thus, grain coarsening takes place in the microstructure, which worsens the material's final mechanical properties. However, MS permits both internal and external heating of the material to occur quickly and uniformly. Additionally, there are several restrictions on this sintering technique. For instance, silicon carbide susceptors, materials that absorb electromagnetic energy and transform it into heat, are necessary in microwave furnaces since many ceramics are unable to absorb microwaves well at ambient temperature.

### 6.6. Yttria content (3% or 5–8%):

The tetragonal phase of $ZrO_2$ is stabilized at room temperature using a variety of oxides. Typically, these include CaO, MgO, $Y_2O_3$, or $CeO_2$. In terms of transformability, phase stability, and mechanical qualities, their amounts should be managed. $ZrO_2$ is typically stabilized with 3 moles of $Y_2O_3$. The mechanical characteristics of this material are affected by the TM transition. The 3Y-TZP's tiny particle size microstructure and phase transformation process cause the cracks to begin to close during the volume expansion (4-5%), which stops them from spreading further. One of the most significant drawbacks of 3Y-TZP is its great opacity, which is in addition to its mechanical strength and phase stability. Monolithic $ZrO_2$ ceramics have therefore been found to exhibit less opacity when yttria content is between 5 and 8%60).

## 7. Applications of $ZRO_2$

### 7.1. Zirconia Ceramic Knives

High strength, wear resistance, no oxidation, no rust, acid and alkali resistance, anti-static, and no food reaction are all qualities of zirconia ceramic knives. They are the best high-tech green knives now available.

### 7.2. Zirconia High-temperature Heating Materials

Zirconia has a maximum operating temperature of 2400°C and can be utilized as a high-temperature heating element at temperatures as high as 1000°C. It has currently been utilised successfully in heating components and machinery in an oxidizing environment above 2000°C (table 3).

### 7.3. Zirconia Bio-Ceramic Material

Zirconia-based porcelain teeth have good transparency and outstanding gloss due to the lack of a metal inner crown, which can successfully prevent issues like tooth hypersensitivity and dark gum lines. Additionally, it is easy to clean, does not irritate the oral mucosa tissue, and has outstanding biocompatibility.

### 7.4. Zirconia Coating Material

High-performance turbine aero-engines are the primary application for zirconium oxide thermal barrier ceramic coating materials stabilized by $Y_2O_3$-based high-performance stabilizers.

### 7.5. Zirconia Communication Material

The zirconia pin body is a crucial part of the ceramic PC type optical fibre connector.

***Table 3.*** *Applications of Zirconium metal oxides*

| **S.No.** | **Zirconium metal oxides** | **Applications** |
|---|---|---|
| 1. | $Al_2O_3/ZrO_2$ | Catalytic uses |
| 2. | ZrO3/ZnO | Optical Use |
| 3. | $ZrO_2/CeO_2$ | Fuel Cells |

## 8. Conclusions

Numerous variables, including the synthetic process, the precursor, the temperature, and the kind of solvent employed, have been used to govern and control the sizes, forms, and intended applications of the synthesized nanomaterial. The generated nanomaterial was analyzed using a variety of characterization techniques, and the majority of the literature was used to calculate the particle size. There have been few reports on the antibacterial activity of zirconia mixed metal oxide nanoparticles, despite the fact that zirconia has been synthesized in combination with other metal oxides for a variety of applications. More research is needed to determine how zirconia can be combined with other metal oxides to improve antibacterial activity.

## Author Statements:

- **Ethical approval:** The conducted research is not related to either human or animal use.
- **Conflict of interest:** The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper
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- **Data availability statement:** The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

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