



# SYNTHESIS OF CERIUM YTTRIUM CO-STABILIZED ZIRCONIUM DI-OXIDE BY SOL-GEL PROCESS

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**Abstract—** The Sol–gel route was used to synthesize Ceria–Yttria co-stabilized Zirconia (CYSZ) nanoparticles. The addition of stabilizing oxides to pure Zirconia, such as CaO, MgO, CeO<sub>2</sub>, and Y<sub>2</sub>O<sub>3</sub>, allows for the formation of multiphase materials, which are referred to as Co-Stabilized Zirconia. Cerium Oxide CeO<sub>2</sub> and Yttrium Oxide Y<sub>2</sub>O<sub>3</sub> are co-stabilized with Zirconium Oxide in this study. The creation of nanostructured coatings has been shown to boost the efficiency of TBCs by lowering thermal conductivity, increasing bonding power, and increasing thermal cycling lifetime, according to researchers. The crystallinity and stabilization of cubic crystalline phases were studied by energy dispersive X-ray spectroscopy (EDAX) at different calcination temperatures in the range of 500°C to 1200°C, and surface morphology and compositional analysis were studied by scanning electron microscopy using the sol-gel process (SEM). The research yielded interesting results, but it was discovered that when Zirconium Dioxide was synthesized using the sol-gel process, the tetragonal phase was not present; thus, other methods to obtain the tetragonal phase would be needed in the future for the application of Thermal Barrier Coating.

**Keywords—** Sol-Gel, nanoparticle, coatings, thermal barrier, co-stabilisation

## I. INTRODUCTION

Mixed metal oxide nanoparticles, also known as heterometal mixed oxide nanoparticles, are made by combining two or more metal oxides to produce synthesized materials with better properties. Mixed metal oxide nanoparticles can be made by combining various metals with different oxidation states in different ratios. The physical, chemical, and morphological properties of the synthesized mixed metal oxide nanoparticles differ, and they are used in a variety of fields. 1st. Because of the decrease in thermal conductivity, increase in bonding power, and improvement in thermal cycling lifetime, nanostructured coatings can improve the performance of TBCs [2–5].

### 1.1 Sol-gel Process

The metal alkoxide is hydrolyzed and then condensation polymerized to produce desired colloids. The viscosity of the system increases over time during this process, allowing it to be coated into thin films or directly formed into desired artefacts. This method produces a uniform and small-sized powder, allows for the development of new microstructures and compositions, the customization of objects or films with special porosity, and the coating of large-area or complex-



shaped objects [1]. Electronics, optics, electricity, space, biosensors, medicine (e.g. controlled drug release), reactive materials, and separation (e.g. chromatography) technology all use sol-gel derived materials. [number six]. Metals may be coated with sol-gel films to increase their resistance to oxidation and corrosion or to change their surface properties. However, issues inherent in sol-gel manufacturing or unique to coating/metal systems limit realistic applications. Since coatings are designed to increase oxidation and wet corrosion resistance, sol-gel coatings provide excellent oxidation protection. The sol-gel process entails hydrolysis of a metal-organic compound precursor to produce oxohydroxide, then condensation and polymerization to form a metal hydroxide network and a porous gel, respectively, with drying and heating of the gel to produce nanoparticles [7]. The effect of solvent and water addition rate on the synthesis and characterization of alumina-zirconia powders obtained by the sol-gel method was investigated. Following the experiment, it was discovered that using a particular solvent produces different textural properties and using a different water addition rate produces different structural properties in the synthesised material [8].

## 1.2 Thermal barrier Coatings (TBCs)

TBCs are widely used in gas turbine engines to shield metallic components from the hot combustion gases. Their use enables combustion temperatures to be raised well above the melting point of super alloys used in structural parts including blades and vanes. Despite the fact that TBCs were first introduced many decades ago, they continue to be the focus of intensive study in both academia and industry. While significant progress has been made in terms of reliability, lifetime, and temperature capability over the years, further work remains to be done in order to completely incorporate the TBC system into the design of a gas turbine engine and, as a result, increase the engine's performance. Thermal barriers are used as protective layers for steel pistons and cylinders in diesel engines, as well as components of compressor housing for aircraft jet engines, working surfaces of exhaust nozzles made of niobium alloys, gas turbines, and chemical plants made of Nickel based alloys [9]. As thermal barrier coatings, a variety of ceramic materials have been investigated (TBCs). High melting point, no phase transition between room temperature and operating temperature, low thermal conductivity, chemical inertness, thermal expansion fit with the metallic substrate, strong adherence to the metallic substrate, and low sintering rate of the porous microstructure are some of the basic requirements for TBCs materials [10-13].

## 1.3 Materials

**1.3.1 Zirconium Dioxide( $ZrO_2$ )** undergoes disruptive phase shifts into three phases, which are referred to as allotropic forms: monoclinic below 1170 °C, tetragonal

between 1170 °C and 2370 °C, and cubic above 2370 °C. The thermal, mechanical, and electrical properties of phase changes are superior [14]. Pure  $ZrO_2$  is polymorphic at ambient pressure, with cubic (fluorite) structure (Fm3m) at high temperatures (2,370°C), tetragonal structure (P42m/nmc) at intermediate temperatures (1,200°C–2,370°C), and monoclinic symmetry (P21/c) at low temperatures (950°C) [15]. Pure zirconia ( $ZrO_2$ ), on the other hand, has little utility as a thermal barrier coating. This is due to the displacive tetragonal (t) to monoclinic (m) phase transition in pure  $ZrO_2$ , which occurs at 950°C on cooling and is followed by a shear strain of 0.16 and a 4 percent volume expansion. This change in shape in the transforming volume, if not accommodated, can lead to catastrophic failure and, as a result, structural unreliability of applied coatings. The ability to stabilize the high temperature (tetragonal and cubic) phases at room temperature has led to the addition of a variety of oxides such as MgO, CaO,  $Y_2O_3$ ,  $Sc_2O_3$ , and  $CeO_2$  in order to reduce or eliminate the high temperature transition(s), with the most common system being yttria stabilized zirconia (YSZ) [15, 16].

**1.3.2 Yttrium Dioxide( $Y_2O_3$ )** A vital starting point in the inorganic synthesis of compounds, it can be used as an additive in high-temperature coatings, paints, and plastics to protect them from UV degradation, as well as in the production of permanent magnets.

**1.3.3 Cerium Dioxide ( $CeO_2$ )** Cerium Dioxide (Cerium Dioxide) ( $CeO_2$ ) Cerium is found in its main ores, bastnaesite and monazite, in a mixture with other rare earth elements, and has a fluorite structure. High-capacity capacitors, conductor buffer layers, fuel cells, polishing materials, UV blocks, and optical devices are only some of the applications.  $CeO_2$  doped with  $ZrO_2$  has shown to have significantly increased durability and a strong thermal barrier coating property.

**1.4 Sol-gel Techniques** The Sol-gel process, shown in Figure 1, has several advantages, including cost-effectiveness and simplicity. Until now, thin films made using these methods were primarily used for optical and defensive purposes and had a thickness of 100 nm or more. The 'sol' (or solution) gradually progresses towards the development of a gel-like diphasic system with both a liquid phase and a solid phase with morphologies ranging from discrete particles to continuous polymer networks in this process. The volume fraction of particles (or particle density) in the colloid can be so low that a large amount of fluid must be extracted before the gel-like properties can be recognized. This can be achieved in a variety of ways. Allowing time for sedimentation and then pouring off the remaining liquid is the easiest process. Centrifugation can also be used to speed up the phase separation process. Drying is needed to remove the residual liquid (solvent) phase, which is usually followed by substantial shrinkage and densification. The distribution of porosity in the gel essentially determines

the rate at which the solvent can be extracted. Changes placed on the structural template during this phase of manufacturing would obviously have a significant impact on the final component's microstructure. Then, via final sintering, densification, and grain growth, a thermal treatment, or firing phase, is often needed to promote further poly-condensation and improve mechanical properties and structural stability. One of the major benefits of this approach over more conventional processing methods is that densification can also be accomplished at a much lower temperature. The precursor sol may be deposited on a substrate to form a film (for example, by dip coating or spin coating), cast into a suitable container with the desired shape (for example, to obtain monolithic, fibres, membranes, or aerogels), or used to synthesize powders (e.g. microspheres, Nano spheres). The sol-gel technique is a low-cost, low-temperature method for finely controlling the chemical composition of a product.

## II. EXPERIMENT

In this paper, two samples of CYSZ with varying percentage of each of the constituents were processed, with increasing percentage of cerium, yttrium and zirconium the samples of varying percentage were synthesized by calculating with molecular weight, samples 1 and 2 tabulated below in Table 1:

**Table 1: Synthesized samples with varying compositions**

Sample	Amount of Oxides to synthesize Nanoparticles(grams)		
	ZrO <sub>2</sub>	Y <sub>2</sub> O <sub>3</sub>	CeO <sub>2</sub>
1	19.7149	2.2591	3.4426
2	12.3218	2.4205	3.6885

. In a container of nitric acid, the three constituents were dissolved separately (HNO<sub>3</sub>). 30ml (HNO<sub>3</sub>) is added and constantly stirred, followed by an equivalent volume of H<sub>2</sub>O. The beaker is then filled with 40 mL of ethylene glycol and Nitric acid, which is cooked on a hot plate at 1100C. As heated, the color of the constituents changed, and nitric acid vaporized in the range of 1400 C to 2800 C. After continuing to heat for around 6 hours, a gel-like substance was left over.

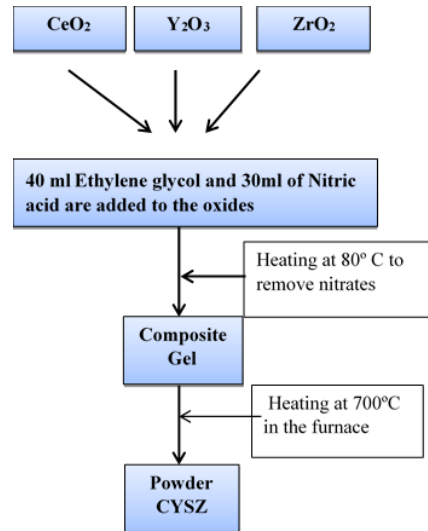


Figure 1 Methodology adopted for preparation of CYSZ powder by Sol-gel Process

## 2.1 Characterisation

Scanning electron microscopy (SEM) model Philips XL-30 with accelerating voltage 30 kV was used to examine the morphology and particle size of the CYSZ powders. Since ceramics are electrically non-conducting, a conducting surface coat must be applied to provide a path for event electrons to flow to ground [17]. Using image analysis software, over 100 particles from the SEM image were counted to calculate the average particle size and size distribution (Image Tool, VEGA3-TESCAN). This gel was heated for 6 hours in the furnace, and the nanoparticles obtained were examined using SEM and EDAX.

## III. RESULT AND DISCUSSIONS

### Energy Dispersive X-ray Spectroscopy Analysis (EDAX)

The dopants Cerium and Yttrium are found in the Zirconium lattice, according to EDAX. EDAX analysis was used to evaluate the phase of all synthesized samples. Figure 2 shows the EDAX pattern of the powders. When Zirconium Dioxide was synthesized using the sol-gel process, the tetragonal phase was not present, as previously stated in the introduction [18–21].

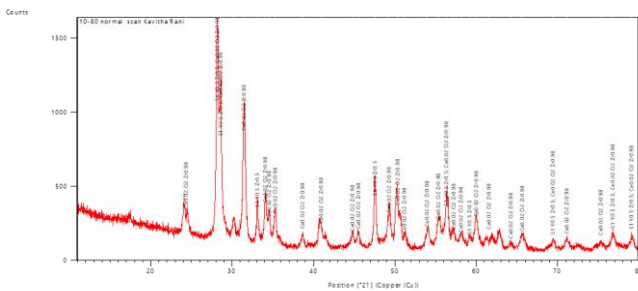
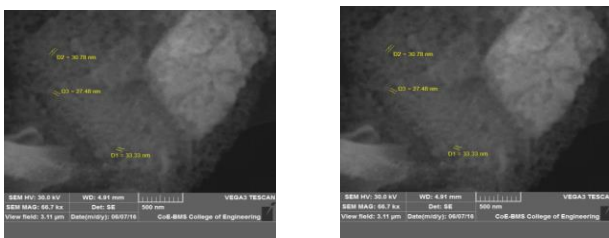


Figure 2 EDAX Analysis of CYSZ powder

### Scanning Electron Microscope (SEM)

SEM images were analysed to investigate the morphology, and the results are shown in Figure 3. Because of densification, the particles are more or less uniform, and the crystals are tightly packed. As a result, the Nano size particles needed for TBC application are undervalued. The SEM micrographs of CYSZ calcined at 1,000°C for 2 hours are shown in the figure. The particle size distribution is almost homogeneous, and the average particle size is well below 100 nm, as can be shown. Particles with a nearly spherical morphology are homogeneous and well defined, with no strongly aggregated particles visible in the picture. The estimated average particle sizes are roughly within the range of 20–30 nm, according to Image tool software (Image Tool, VEGA3-TESCAN). The average particle size is less than 25 nanometers.



Sample 1

Sample 2

Figure 3 SEM analysis of CYSZ samples with varying compositions

### IV. CONCLUSION

The synthesis of CYSZ by sol-gel route was carried out, the results were studied, and it was concluded that by Sol-gel process, the required phases with the mentioned composition of Zirconium dioxide are not obtained; therefore, the tetragonal structure of Zirconium dioxide can be achieved using other methods such as combustion, ion Exchange, solid

state reaction, and so on. The crystal structure, morphological assessment, and thermal behavior of the final nanopowder produced by this method were evaluated using EDAX analysis and SEM technique in order to evaluate the crystal structure, morphological assessment, and thermal behavior of the final nanopowder produced by this method for further thermal barrier coatings applications. This method does not achieve the tetragonal crystal structure of zirconia, which is ideal for TBCs applications, according to crystal structure investigations. The development of nanoparticles with an average particle size of 25 nm and a narrow particle size distribution was verified by scanning electron microscopy of CYSZ powder.

### Acknowledgements

The present work was supported by department of Industrial Engineering and Management, Chemistry Department and Mechanical Engineering Department, B.M.S. College of Engineering, Bengaluru, India.

**Conflicts of Interest:** The authors declare that there are no conflicts of interest regarding the publication of this paper.

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