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Enhanced Structural, Thermal, and Mechanical Properties of YSZ Composites Co-Doped with ZnO and La₂O₃ for High Temperature Applications

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Opposed Reviewers:	

Enhanced Structural, Thermal, and Mechanical Properties of YSZ Composites Co-Doped with ZnO and La₂O₃ for High-Temperature Applications

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Abstract

The co-doping of yttria-stabilized zirconia (YSZ) with zinc oxide (ZnO) and lanthanum oxide (La₂O₃) was investigated to enhance its structural, mechanical, and thermal properties for high-temperature applications like thermal barrier coatings (TBCs). The YSZ samples were synthesized using the dry pressing and sintering method, with varying concentrations of ZnO (0.5%, 1.0%, and 1.5%) and La₂O₃ (10%), followed by sintering at 1400°C. The fabricated samples were systematically tested to evaluate their structural, mechanical, and thermal properties. X-ray diffraction (XRD) confirmed that co-doping with ZnO and La₂O₃ led to enhanced crystallinity and phase stability, with the most stable phase observed in samples containing 1.5% ZnO and 10% La₂O₃. Thermogravimetric analysis (TGA) demonstrated that the co-doped materials maintained thermal stability up to 700°C, though higher ZnO concentrations introduced slight thermal degradation. Density measurements revealed an increase from 74% to 92% with higher ZnO content, while porosity was significantly reduced from 26% to 2.6%, contributing to improved mechanical strength. Vickers hardness values increased from 620 HV to 690 HV, indicating more significant resistance to deformation. However, Charpy impact tests showed a trade-off in toughness as energy absorption decreased with higher dopant levels. Thermal conductivity measurements showed a reduction to 0.012 W/cm·K, making the material more suitable for Thermal Barrier Coating applications, though increased brittleness was observed in tensile stress tests, where higher ZnO concentrations led to earlier material failure. The study demonstrates that co-doping YSZ with ZnO and La₂O₃ significantly enhances its performance for high-temperature applications, improving hardness, density, phase stability, and thermal insulation. Future research should optimize the balance between brittleness and toughness by adjusting dopant concentrations to maximize the material’s long-term effectiveness in extreme operational environments.

Keywords: Yttria-stabilized zirconia (YSZ); Zinc oxide (ZnO); Lanthanum oxide (La₂O₃); Co-doping; Mechanical properties; Thermal properties; Electrochemical properties; High-temperature applications; Thermal barrier coatings (TBCs); Dry pressing and sintering; X-ray diffraction (XRD); Crystallinity; Phase stability; Thermogravimetric analysis (TGA); Density; Porosity; Vickers hardness; Thermal conductivity; Brittleness; Tensile stress tests; Toughness; Material failure; Thermal insulation; Dopant concentrations.

1. Introduction

It is crucial to have materials with the ability to sustain thermal and mechanical stability for use in high-temperature applications, such as thermal barrier coatings (TBCs). This research tackles these issues by studying the synergistic effect of co-doping Yttria-Stabilized Zirconia (YSZ), with Zinc Oxide (ZnO) and Lanthanum Oxide (La_2O_3) to improve its performance in extreme environments. Yttria-stabilized zirconia (YSZ) is a widely used ceramic material due to its ionic conductivity, thermal stability, and mechanical strength, which are requisite for high temperature applications, including gas turbines [1]. TBCs serve to isolate different parts of a turbine engine from excessive heat [2], [3]. Even though YSZ has good characteristics, recent studies suggest that additional doping using La_2O_3 and ZnO, which enhances thermal stability and mechanical properties, widens the scope of its favorable performance. [4][5]. In comparison to single-element doping, which has received considerable attention, the combination of La_2O_3 and ZnO is expected to result in greater phase stability and mechanical strength.

The potential synergistic effects arising from La_2O_3 's stabilization of the cubic phase combined with the enhancement of ZnO on sinterability suggest that these materials could possess super material traits, warranting their utilization for extreme temperatures [6]. Nonetheless, the synergistic nature of co-doping YSZ with both La_2O_3 and ZnO has not yet been studied and provides an opportunity to assess the effects of the two dopants together. It is evident that both La_2O_3 and ZnO individually showed remarkable improvement in the traits of YSZ, but it appears there is little to no information surrounding their co-doping. Multiple sources back the claims about the positive impacts of La_2O_3 and ZnO when applied separately, where La_2O_3 maintains stability of the cubic phase at elevated temperatures whereas ZnO improves sinterability and even contributes to the electrical conductivity [7] [8]. Most recently, it has been proposed that optimal properties of materials can be achieved through multi-element doping. Improved conductivity and sinterability has been observed with the co-doping of YSZ with ZnO and MnO_2 [9]. However, other experiments focusing on rare earth co-doping have been confirmed to lower thermal conductivity due to the phenomena of enhanced scattering of grains and porosity [10]. There has been very little study conducted with the incorporation of La_2O_3 and ZnO with YSZ, thus greatly exposing an unfilled puzzle devoid of explanation.

The development of new materials with the ability to endure exceptional conditions is pivotal in aerospace, energy, and automotive industries. For example, gas turbines function under significant thermal loads, which require coatings to provide thermal protection while resisting mechanical cracking. This study seeks to improve the thermal insulation, mechanical strength, and phase stability of YSZ to design advanced thermal barrier coatings and fuel cells that are more cost-effective and support wider sustainability objectives.

2. Materials and Methods

In this study, we focused on fabricating Zinc Oxide (ZnO) and Lanthanum Oxide (La_2O_3) doped Yttria-Stabilized Zirconia (YSZ) samples using pellet pressing and sintering. We selected extremely pure materials to ensure accurate and reliable experimental results. Our process of material selection and fabrication is illustrated in the upcoming sub-segments.

2.1 Materials

For our research, we imported the Lanthanum Oxide (La_2O_3), YSZ, and Zinc Oxide (ZnO), where the purity of these materials was our utmost concern. La_2O_3 had a molecular weight of 325.81, and the calcium content was negligible. ZnO, with a particle size of 20-30 nm, had a purity of 98.5%. The YSZ we used, an 8% Yttria-stabilized variant, had a particle size of 1 μm and a purity of 99.5%, with minor trace impurities. We used

ethanol (C_2H_5OH) with a purity of 99.99% for cleaning and as a dispersing agent in the preparation process. All handling of ethanol and La_2O_3 was conducted in a well-ventilated fume hood to minimize exposure to fumes and ensure safety during the preparation process.

2.1 Specimen Composition

We prepared four distinct batches for fabrication (We created eight samples for each batch, commulating 32 samples for different tests that we will be doing in this research). The compositions of each batch are given below:

Batch 1: 0.5% ZnO and 99.5% YSZ

Batch 2: 0.5% ZnO, 10% La_2O_3 , and 89.5% YSZ

Batch 3: 1% ZnO, 10% La_2O_3 , and 89% YSZ

Batch 4: 1.5% ZnO, 10% La_2O_3 , and 88.5% YSZ

2.2 Fabrication Process



Fig 1: Different steps of the fabrication process

We followed a detailed solid-state reaction technique to fabricate the ZnO and La_2O_3 doped YSZ samples. Each step was carefully executed to ensure a uniform and high-quality final composite. Preliminary experiments were conducted to determine optimal processing parameters, including milling duration and sintering temperature.

2.2.1. Mixing

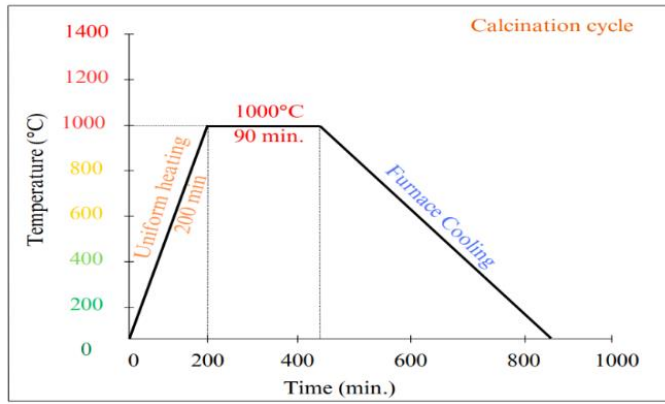
The first step involved mixing the starting materials according to their batch compositions. We employed wet ball milling to ensure uniform mixing, using ethanol as the wetting agent. We selected zirconia balls as the milling media, and the milling process was carried out for 24 hours in a pot mill (Model: G91) operating at 100 rpm. We chose wet ball milling to reduce the agglomeration of particles and create a homogenous powder mixture. Then, we chose Ethanol as the ideal wetting agent for our fabrication process as it minimised the hardness of the powder and reduced the drying time.

2.2.2 Drying

We transferred the mixture of ethanol-powder slurries into a beaker and dried it in a drying oven (Jisico, UK; Model: VARO/8P) at $100^{\circ}C$ for 24 hours. It allowed the ethanol to evaporate faster and more thoroughly. After drying the ethanol, we were left with a well-dispersed powder ready for the next phase.

2.2.3. Grinding

To achieve a fine and uniformly consistent powder, we ground the agglomerated powder manually using a mortar and pestle for approximately one hour. This process was for further breaking down agglomerates, which was essential for the calcination and compaction process.



- 1(0.5% ZnO, 0% La_2O_3)
- 2(0.5% ZnO, 10% La_2O_3)
- 3(1% ZnO, 10% La_2O_3)
- 4(1.5% ZnO, 10% La_2O_3)

Fig 2: Temperature Profile of the Calcination Cycle

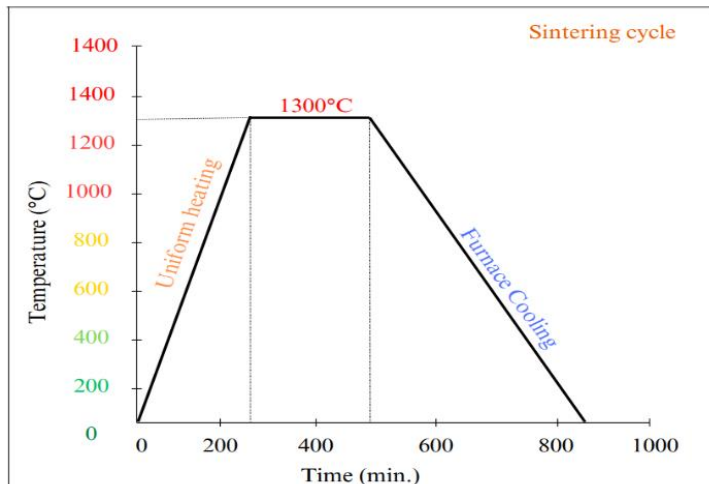
2.2.4. Calcination

Next, with a heating range of 5°C per minute, we calcinated the powder at 1000°C for 90 minutes. 1000 °C was chosen as the calcination temperature, as it was the most optimum temperature we found in the literature recommendations. It ensured the reduction of impurities and the retention of structural integrity. This was an essential step as it enhances the compaction properties of the powder and helps reduce the phase impurities. After calcination, we allowed the powders to cool gradually in the furnace and then deployed the powders for compaction.

2.2.5. Compaction

To form green bodies, we compacted the powders using a pellet press (Retsch, Model: PP25). We maintained a consistent weight of 1.6 g across all four batches to make pellets. We applied a pressure of 250 MPa for 3 minutes for each of our pellets, which we found optimal by the preliminary testing process. We chose 250 MPa to produce denser and more uniform pellets while avoiding cracking propagation in our green bodies. Our green bodies were inspected with a magnifying glass to confirm their integrity and uniformity before sintering.

2.2.6. Sintering



- 1(0.5% ZnO, 0% La_2O_3)
- 2(0.5% ZnO, 10% La_2O_3)
- 3(1% ZnO, 10% La_2O_3)
- 4(1.5% ZnO, 10% La_2O_3)

Fig 3: Temperature Profile of the Sintering Cycle

We sintered the green bodies in a Protherm furnace (manufactured in Germany). We gradually heated the samples at a well-controlled rate of 5 °C per minute. We selected 1300 °C for the sintering temperature, ideally 85% of the material with the lowest melting point in our composite. This temperature was optimal for the maximum densification of the YSZ and for maintaining the stability of both ZnO and La₂O₃. Once the temperature reached 1300°C, we kept this for 240 minutes to allow for proper densification and phase stabilization. The cooling process was carefully controlled over 420 minutes to relieve thermal stress and ensure the final structure was robust. The sintering cycle was optimized through literature review and repeated trials, accounting for the melting points and ideal processing conditions of the ZnO and La₂O₃ nanoparticle doped YSZ composites.

2.2.7 Final Composite Appearance



Fig 4: Final appearances of fabricated ceramic tablets

After sintering, we polished each sample manually to achieve the required surface smoothness for further testing. The dry polishing was performed using emery papers of various grades (100-2500) to obtain a mirror-like surface. We then used alumina powder for wet polishing to further refine the surface. Polishing was necessary for accurate hardness testing, thermogravimetric analysis (TGA), and X-ray diffraction (XRD) analysis as it ensured the surface quality required for these tests.

3. Results and Discussions

3.1 Structural Analysis

The phase structure and crystallinity of yttria-stabilized zirconia (YSZ) samples, which were doped with different amounts of ZnO and La_2O_3 , were determined using X-ray diffraction (XRD) analysis. Figure 5.1 exhibits the X-ray diffraction (XRD) patterns for Samples 1 to 4.

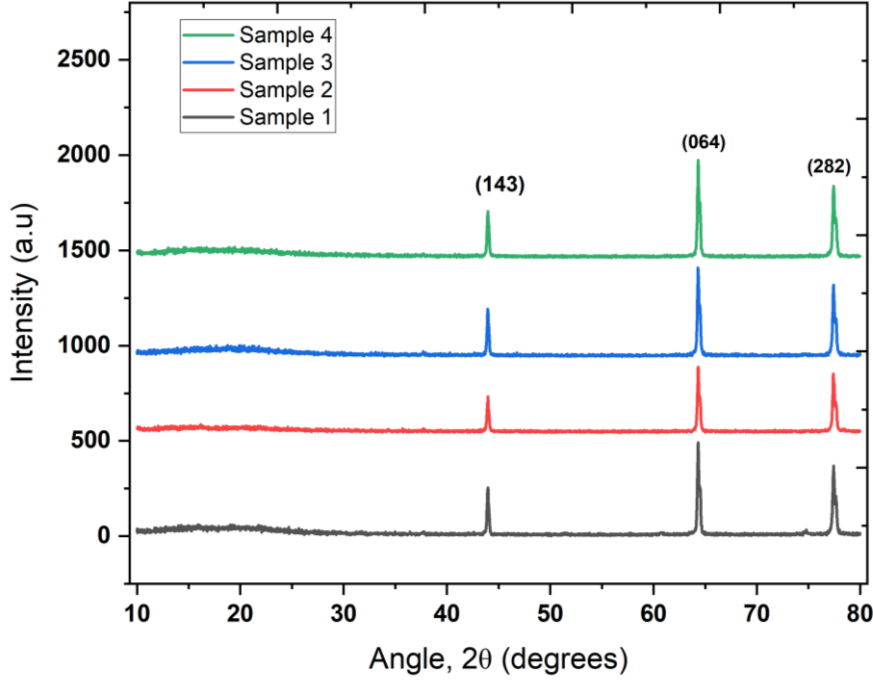


Figure 5: XRD patterns of La_2O_3 and ZnO doped YSZ for different weight ratios

The diffraction peaks are labeled with their respective crystallographic planes. Sample 1, containing 0.5 wt% of ZnO and lacking La_2O_3 , is used as our reference point for comparison. It displays essential peaks corresponding to the YSZ phase in its pure form. The considerably decreased magnitude of the peaks in this sample indicates a moderate crystallinity level or a reduced crystallite size. This observation aligns with research by Guo et al. (2019), where pure YSZ without rare-earth dopants exhibited limited enhancement in crystallinity and thermal stability [11].

With a constant ZnO content with the addition of 10 wt% La_2O_3 , we encountered an enhancement in the peak intensity, especially for the angle $2\theta=43.64^\circ$ with its corresponding lattice plane (JCPDS card number 96-100-9017). This enhancement implies an improvement in the crystal structure quality due to the addition of La_2O_3 to the YSZ matrix. It has been shown that the addition of La_2O_3 to the YSZ matrix expands the lattice parameters, consequently augmenting the grain sizes [11]. Attributed to the La_3^+ ions, the giant lattice enhances the phase stability that parallelly contributes to more extraordinary thermal properties such as higher thermal stability and lower thermal conductivity[12].

With greater enhancements in both the intensity and the sharpness of the peaks as the ZnO levels increase in samples 3 and 4, we encounter continuous improvement in the crystal structure quality and possibly an augmentation in the size of the crystal particles. Sample 4, with the highest dopant content, notably exhibits the

most significant peak intensities. This indicates the highest degree of crystalline order compared to our previous samples. Moreover, as doping ZnO is known to enhance lattice order and improve crystalline size, our results are homogeneous with what we found about enhancing the concentration of ZnO dopant in YSZ [13].

Diffraction patterns that we observed in our structural test of this research correspond to the tetragonal phase of YSZ, which is preferred due to its lower heat conductivity and decent thermal stability attributes. The optimum amount of concentrations and a successful characterization process using both ZnO and La₂O₃ dopants with YSZ are essential for improving the structural characteristics of YSZ. This is reflected in our XRD test, where the clear diffraction peaks demonstrate grain boundary stability and lattice integrity improvements. The grain boundary and lattice integrity improvements improved our ceramics in both structural and thermal sectors. In terms of harness, our ceramic composite has been hardened as corroborated in our hardness test, and also, the lattice integrity has made our fabricated composite material more resistive to thermal conductivity and thermal stability in high-temperature environments [14].

3.2 Physical Properties

3.2.1 Density:

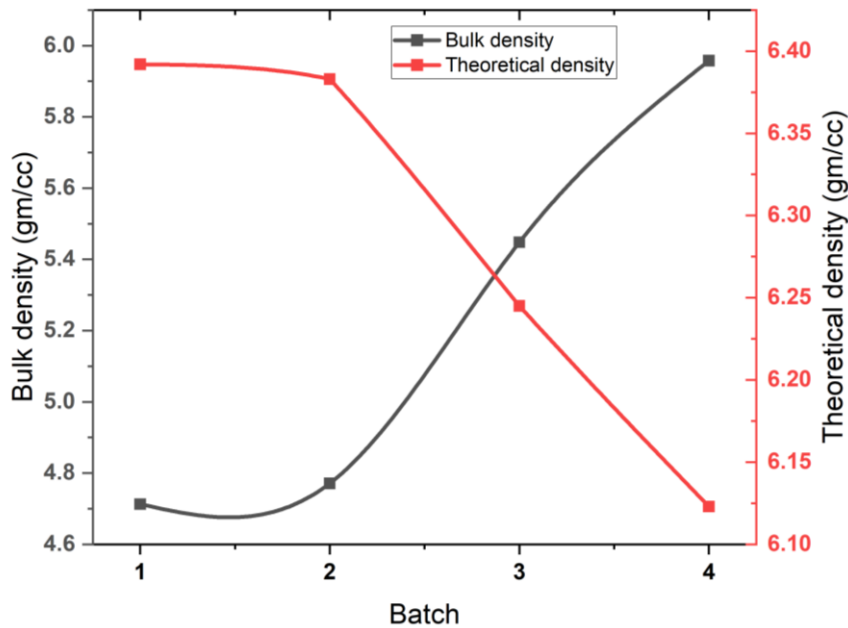


Figure 6: Variation of density with batches

We determined the bulk density using the Archimedes method, while we calculated the theoretical density by dividing the mass by the volume of each sintered sample. In Batch 1, which consisted of 0.5 wt% ZnO without La₂O₃ doping, we observed that the bulk density was lower than the theoretical density. This indicates the presence of porosity or incomplete densification in the sample. George et al. (2014) demonstrated that without sufficient sintering aids, such as La₂O₃, densification remains incomplete due to limited grain boundary diffusion, resulting in higher porosity [15]. In Batch 2, which included 0.5 wt% ZnO and 10 wt% La₂O₃, we found a stronger correlation between actual and theoretical densities, suggesting improved densification. Banerjee et al. (2006) showed that La₂O₃ acts as a sintering aid, promoting liquid-phase sintering and enhancing

densification by reducing porosity and facilitating grain boundary diffusion [16]. Yuan et al. (2017) further demonstrated that La_2O_3 significantly improves densification by forming dense microstructures [17]. In Batches 3 and 4, as we increased the ZnO content, we observed a significant increase in bulk density and a decline in theoretical density. Sankar et al. (2011) found that ZnO enhances densification through mechanisms such as liquid-phase sintering, promoting grain growth and reducing porosity [18]. Wang et al. (2016) confirmed that ZnO helps fill void spaces between particles, increasing density and improving sintering

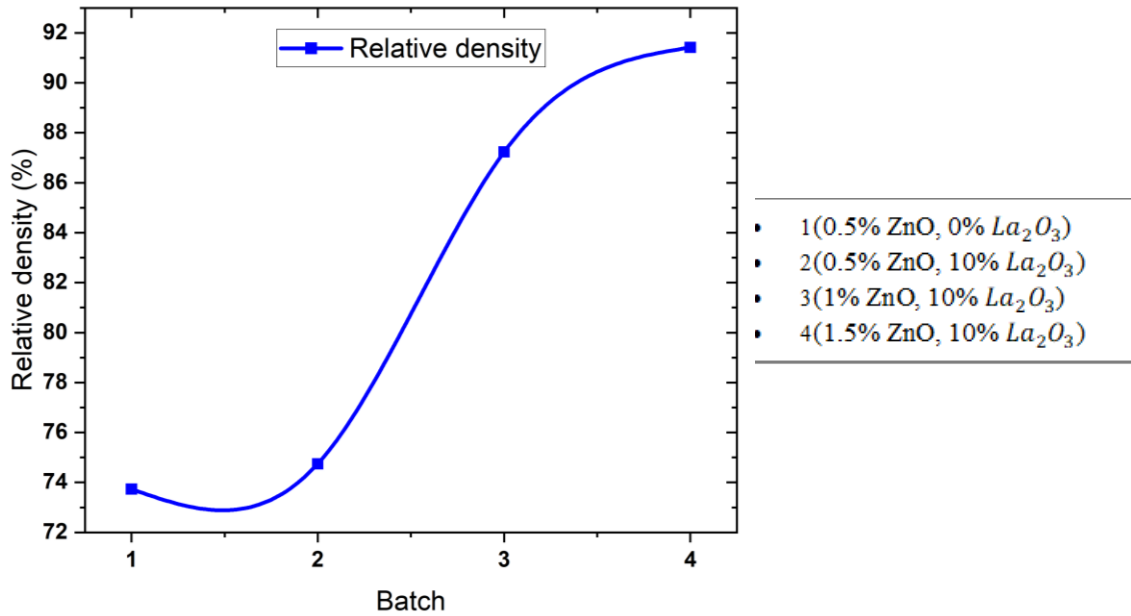


Figure 7: Variation of relative density with batches

performance [19]. Figure 7 shows a substantial rise in relative density from Batch 1 to Batch 4, with Batch 4 achieving a relative density of approximately 92%. Pérez-Coll et al. (2010) demonstrated that an increase in relative density correlates with reduced porosity, enhancing composite ceramics' structural integrity. This improvement is crucial for applications like thermal barrier coatings, where porosity can significantly affect thermal conductivity and mechanical properties [20].

3.2.2 Porosity

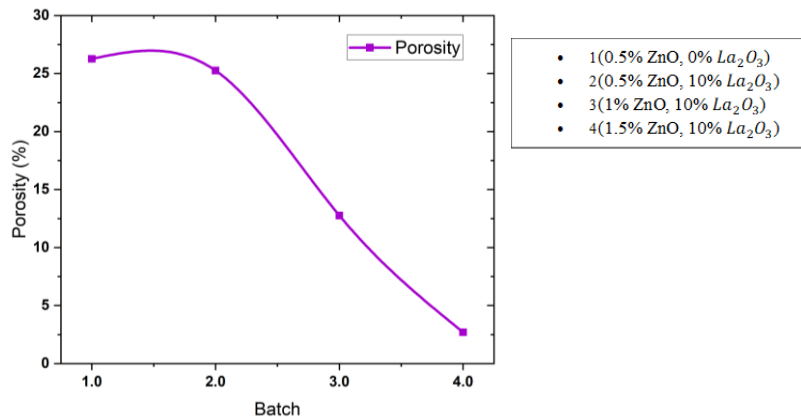


Figure 8: Variations of porosity with different batches

The following Figure 5.4 represents the trend of variation in the porosity with varying La_2O_3 , And ZnO particle concentrations were added into the 8YSZ. The graph shows that the X-axis represents the number of batches, and the Y-axis represents the percentage of porosity determined by the quantitative measure. For the first batch with the composition of 0.5% wt ZnO, 0% wt La_2O_3 , and 99.5% YSZ, the porosity is 26%. For the second batch with the composition of 0.5% wt ZnO, 10% wt La_2O_3 , and 89.5% wt YSZ, the percentage of porosity is 25.1%. Similarly, for the third batch with the composition of 1% wt ZnO, 10% wt La_2O_3 , and 89% wt YSZ, the percentage of porosity is 12.6%. Finally, for the fourth batch having a composition of wt 1.5% ZnO, wt 10% La_2O_3 , and wt 88.5% YSZ, the percentage of porosity is 2.6%. This graphical representation of the porosity test shows that the porosity is being reduced from batch 1 to batch 4. Therefore, it is evident that if we increase the amount of ZnO and La_2O_3 doping with the YSZ, the porosity is reduced substantially. At a position where the density was highest, the porosity was lowest. By filling the spaces between molecules with La_2O_3 and ZnO nanoparticles in YSZ, the densification increases with decreasing pore and void spaces or air bubbles. As a result, the porosity decreased [21][22]. Reducing porosity has many positive outcomes, such as improved mechanical strength, enhanced conductivity, increased density, and better sinterability. Reduced porosity implies a decreased presence of air-filled voids in the material, resulting in a reduced heat transfer through the coating. This enables the coating to provide more efficient protection to the underlying material against high temperatures [23][24]. Reducing the porosity of the coating enhances its mechanical strength and durability by decreasing the chances of crack propagation and rupture [25]. Thermal shock, or abrupt temperature fluctuations, is standard in TBCs and can be harmful if the material has a high porosity. Better thermal shock resistance is ensured by lower porosity, which creates a more homogenous and stable structure that can tolerate abrupt temperature changes [26]. Reduced porosity limits the space that corrosive species and oxygen can occupy inside the TBC before reaching the substrate underneath. This can strengthen the coated component's resistance to oxidation and corrosion, which is crucial under challenging working conditions [27]. In this work, 1.5 wt% ZnO, 10 wt% La_2O_3 . In addition, 88.5% YSZ provides the least porosity, so this composition could be a better material for TBCs than the other compositions studied in this work.

3.3 Mechanical Properties

3.3.1 Hardness Test

The hardness was measured via a Vicker's hardness tester (Manufacturer: Struers, Japan; Model: DK-2). This technique, commonly known as the mechanical method, is primarily employed for small components and thin sections. According to the ASTM E 384 standard, the micro-Vickers test requires an applied force ranging from 1 gf to 1000 gf. The Vicker's hardness value was measured using the following formula.

$$HV = \frac{1.854F}{d^2}$$

Where,

F= Applied load in kg

D= Average diagonal length in mm

$D = \frac{d_1 + d_2}{2}$ (figure 9)

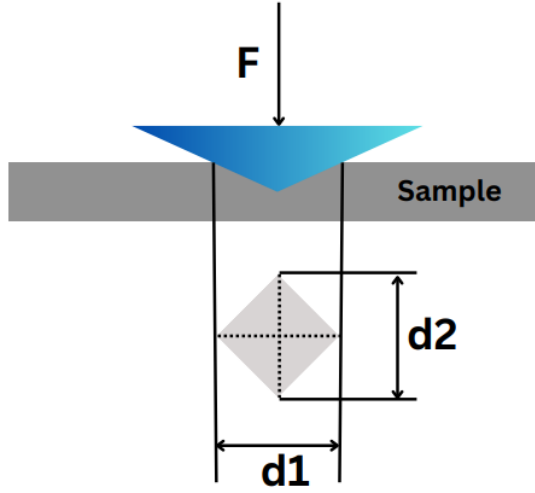


Figure 9: Schematic illustration of Vicker's hardness measurement

During the Vickers hardness testing, a weight of 1 kg was applied for 8 seconds. Before testing, the sintered samples underwent grinding using 800-2000 grit SiC sandpaper, followed by mechanical polishing with Al_2O_3 of 4-5 μm .

We evaluated the mechanical properties of Ytria-Stabilized Zirconia (YSZ) composites, doped with varying amounts of ZnO and La_2O_3 , using Vickers hardness testing. As shown in **Figure 10**, introducing ZnO and La_2O_3 significantly enhanced the hardness of the composites. This improvement was primarily due to increased densification and reduced porosity. The highest hardness values were recorded where density was maximized,

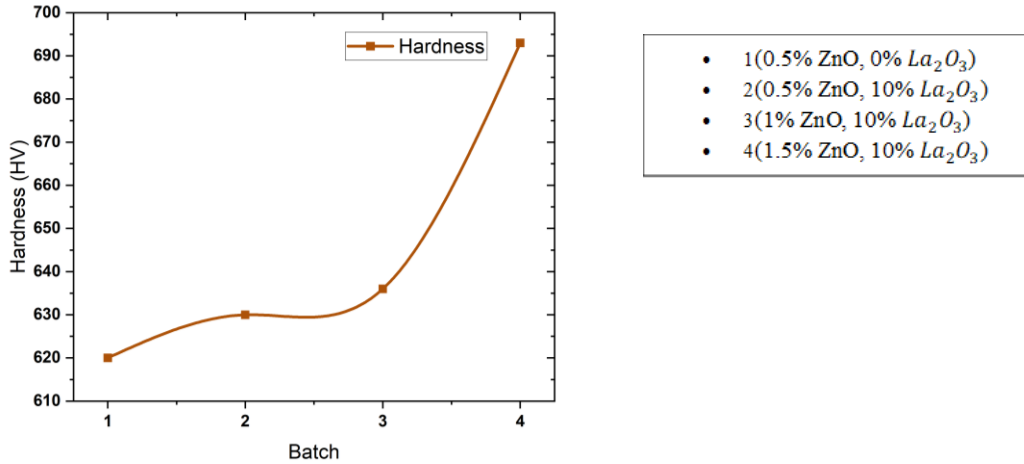


Fig 10: Variations of hardness with different batches

Porosity was minimized, confirming the well-established relationship between microstructure and mechanical performance. In our baseline sample, Batch 1, which contained 0.5 wt% ZnO and no La_2O_3 , the

hardness measured around 620 HV. This sample serves as a reference for assessing the effect of further doping on the YSZ matrix. The limited impact on hardness with this low ZnO content aligns with studies showing that small amounts of ZnO improve densification but don't significantly enhance hardness without secondary phase formation [28]. When we introduced 10 wt% La_2O_3 while keeping ZnO at 0.5 wt%, Batch 2 exhibited a noticeable hardness increase, reaching approximately 630 HV. This enhancement is attributed to the dispersion-strengthening effect of La_2O_3 , which refines the grain structure and improves resistance to plastic deformation. Research indicates that La_2O_3 enhances densification and grain refinement, supporting our findings [29] [30]. Moreover, La_2O_3 solid solution strengthening of secondary phases that impede dislocation motion enhances hardness further [31]. Upon increasing the ZnO content to 1 wt%, we noticed in Batch 3 hardness growing significantly to over 650 HV. This agrees with studies that have shown the strengthening effect of ZnO due to its promotion of densification and grain boundary strengthening[28]. The addition of ZnO and La_2O_3 together creates a synergistic effect of enhanced grain boundary strength and reduced dislocation mobility through solid solution strengthening like what has been noted in other doped ceramic systems. Batch 4 with 1.5 wt% ZnO and 10 wt% La_2O_3 exhibited the highest hardness of 690 HV. Use of these dopants improved the densification process and increased the volume fraction of secondary phases which in turn, resulted in greater restriction of dislocation motion. This agrees with previous studies where higher concentrations of the dopants increased hardness as a result of grain boundary strengthening and reduced porosity defect [30] [31]. The enhanced resistance to plastic deformation of materials, with the aid of secondary phases at the grain boundaries, is substantial. It is clear that a combination of solid solution strengthening, grain boundary strengthening, and reduction of porosity leads to an increase in hardness for all the batches. ZnO and La_2O_3 dope the material by refining the grain structure, introducing secondary phases, and pinning dislocation motion, thereby improving mechanical performance. The increase in hardness observed is in agreement with doped ceramic systems, where the rigidity is further elevated by increased densification and microstructural refinement.[28][29]

3.3.2 Charpy Impact Test

We conducted Charpy impact testing for the toughness and flexural shock resistance evaluation of ZnO and La_2O_3 - doped YSZ composites, focusing on their potential use in gas turbines operating at elevated temperatures. This test recorded the energy released during fracture which was an indication of the items being brittle or ductile. We specifically created 4 samples in compliance with ASTM A370 standards (10mm x 10mm x 55mm) for this study, embedding a notch to the surface to reproduce impact loading. The data from our investigation have been compiled in Table 1. As we analyze their values, we can observe a remarkable change in energy absorption for different tested specimens. After correction, the energy values ranged from 64.54 Joules to 42.69 Joules, suggesting that there was lots of difference in impacts resistance. We discovered that batch 1 with 0.5% ZnO and no La_2O_3 doping had the highest energy absorption at 64.54 Joules while the lowest value of 42.69 Joules was observed from the specimen with most doping which had 1.5% ZnO with 10% La_2O_3 .

Table 1. Charpy Test Results and Calculation

<i>Type of Specimen</i>	<i>Sam ple No.</i>	<i>Compositions of the Ceramic Composites</i>	<i>Cross sectional area (mm²)</i>	<i>Scale used</i>	<i>Initial Error i, (%)</i>	<i>Energy absorbe d (E) (Joules)</i>	<i>Corrected energy [E- i] (Joules)</i>	<i>Remarks</i>
<i>Charpy Simple Beam</i>	1	0.5%ZnO, 0% La_2O_3	100 mm ²	Charpy	0.5%	64.86	64.5357	Completely Broken
	2	0.5%ZnO, 10%	100 mm ²		0.5%	53.30	53.0335	Completely

		La ₂ O ₃						Broken
	3	1% ZnO, 10% La ₂ O ₃	100 mm ²		0.5%	47.52	47.2824	Completely Broken
	4	1.5% ZnO, 10% La ₂ O ₃	100 mm ²		0.5%	42.9	42.6855	Completely Broken

The results from our Charpy Impact Testing indicated a downward trend in energy absorption with the increasing concentrations of both ZnO and La₂O₃ nanoparticle dopants. This phenomenon suggests that the amount of dopants is paramount in the composite's impact resistance and energy absorptions. The sample1 with the lowest amount of doping (0.5% ZnO and 0% La₂O₃) exhibited the highest resistance in impact force while absorbing 64.54 Joules. This happens as ZnO helps to preserve material toughness by enhancing grain boundary cohesion and improving sinterability. [32].

We observed a steady decline in energy absorption when we increased the amount of dopants, as indicated by Table 1. The sample with the highest percentage of dopants (1.5% ZnO and 10% La₂O₃) absorbed the least energy. This indicates that a higher amount of ZnO and La₂O₃ nanoparticle dopants reduces YSZ's toughness. This phenomenon aligns with the previous research by Zhang et al., where he demonstrated that a higher amount of dopants causes a decrease in mechanical strength due to changes in microstructure that eventually cause brittle behavior in composites [33]. In our case, when we increased the amount of dopants, the interaction between the ZnO and La₂O₃ may have caused dopant-induced grain boundary segregation as indicated by Chen et al. that led to the reduction of the YSZ composite's fracture toughness and impact resistance [34]. As we observed from this impact test, increasing the ZnO and La₂O₃ nanoparticle in the composition while co-doping with YSZ reduces the ceramic composite's fracture toughness and impact resistance. Therefore, while applying this in high temperatures and extreme applications like ceramic coatings inside the gas turbines, the ceramic composite will be subjected to extreme temperatures for prolonged hours. In gas turbines, where the temperature is a significant factor, the flying debris at superheated temperature is another paramount factor for the gas turbines' overall performance, efficiency, and safety. Flying debris has been a concern for engineers as it causes pitting to the peripherals of the turbine blades, causing significant problems like vibrations, energy loss, excessive heating, and lower efficiency, which can jeopardize delicate machinery like gas turbines. As our impact test suggests while co-doping the YSZ with ZnO and La₂O₃, optimum concentrations of doping are required to ensure both lower thermal conductivity, good thermal stability, as well as good resistance to impact forces against prolonged hours of debris flying towards the blades of the turbines at superheated temperatures. Further research should examine the microstructural changes induced by varying dopant concentrations to optimize the balance between toughness and other essential properties, such as thermal stability and electrical conductivity, ensuring the material remains suitable for high-performance applications.

3.3.3 Tensile Stress Test

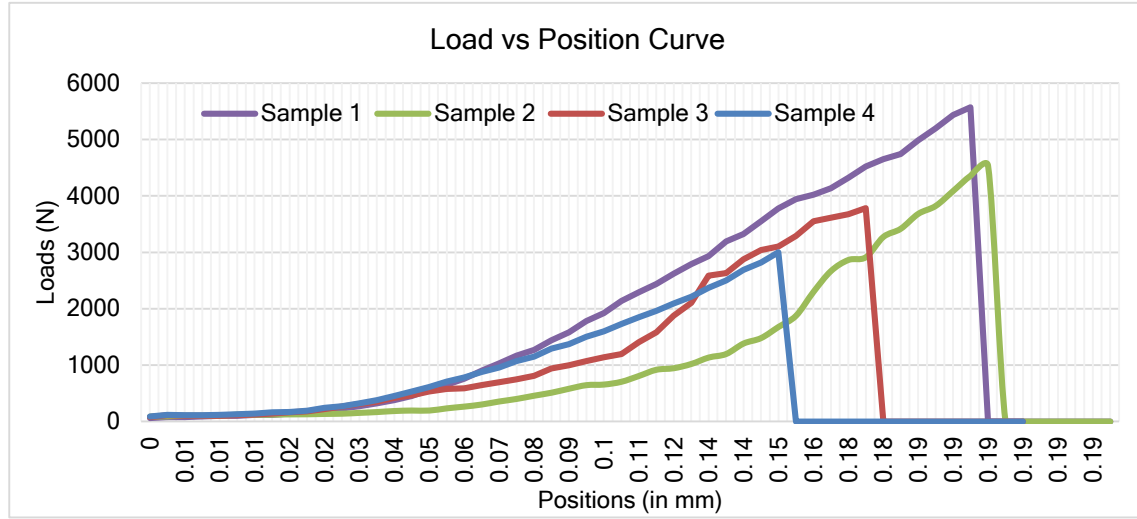


Fig 11: Variations in Loads in terms of positions of various YSZ samples

Sample 1 (0.5% ZnO, 0% La₂O₃) exhibited a gradual increase in load-bearing capacity with displacement, reflecting consistent structural integrity throughout the test. The smooth, linear progression of the load-displacement curve, culminating in a peak load of 5568.65 N at a displacement of 0.19 mm, suggests that minimal ZnO doping effectively preserves the inherent toughness of the YSZ matrix. This behavior is expected as previous studies demonstrated that the low concentrations of ZnO dopants enhanced the cohesion of grain boundaries and densifications [34]. The gradual increase in load further supports the idea that ZnO at low concentrations maintains the material's toughness by improving the overall microstructure and preventing premature failure. Sample 2 (0.5% ZnO, 10% La₂O₃) suggests that incorporating additional La₂O₃ (10%) into the composite provided a noticeable shift in the mechanical properties. Initially, the load progression was relatively flat, followed by a sharp ascent towards the peak load of 4357.73 N with a displacement of 0.19 mm, which suggests that while increased La₂O₃ contributes initially to early rigidity and densification enhancements, it also made our ceramic composite brittle in the end. That led to an increased brittleness, which was evident by a reduction in peak load with increasing concentrations of doping materials in the YSZ. This trend can be attributed to grain boundary weakening, as observed in studies where La₂O₃ doping causes dopant segregation at grain boundaries, reducing mechanical cohesion [35]. The early rigidity seen in Sample 2 matches previous findings on La₂O₃'s ability to enhance densification but at the cost of toughness, with sudden failure likely due to the brittle nature of the doped material under tensile stress [33].

In sample 3 (1% ZnO, 10% La₂O₃), increasing the ZnO concentration to 1% alongside 10% La₂O₃ altered the response of the material further by exhibiting a more pronounced early stiffness with a noticeable reduction in peak load of 3785.56 N at a displacement of 0.18 mm. The steeper curve with lesser peak load suggests that a higher amount of dopants causes enhanced densification of the YSZ, but it also compromises the toughness of the original compound. This phenomenon can be described by the weakening of grain boundaries at higher concentration levels of ZnO in YSZ materials, where previous studies have found that although composites perform well at lower ZnO doping levels, higher concentrations lead to a reduction in the overall toughness of those materials. [33] [36]. The reduction in peak load and earlier onset of failure in Sample 3 also aligns with the observed suppression of stress-induced phase transformation, a toughening mechanism in YSZ composites. Higher dopant levels tend to stabilize the tetragonal phase, inhibiting the transformation to the monoclinic phase and thereby reducing the material's ability to dissipate energy during tensile loading [37]. Finally, sample 4 (1.5% ZnO, 10% La₂O₃), with the highest concentration of ZnO (1.5%) combined with 10% La₂O₃ exhibited a

similar trend to Sample 3 but with a slightly lower peak load of 3126.87 N and a displacement of 0.16 mm. The plateau observed before the peak suggests that the material reached a threshold in its ability to handle stress, after which it rapidly deteriorated. This behavior highlights the trade-off between densification and fracture resistance. Materials become more rigid but also more brittle at higher dopant concentrations, as we observed in previous studies where the residual stresses and secondary phases are formed at higher concentrations of dopants, further reducing the toughness[38]. The early fracture in sample 4 with the highest amount of dopants can be linked to the forming of brittle secondary phases at grain boundaries. This phenomenon acts as a weak point, causing crack propagation and facilitation. This is further compounded by grain growth, as we found previously with our XRD analysis, which reduces the grain boundary densities and consequently increases the susceptibility of the material's brittle failure, which is a common phenomenon observed in higher dopant levels [33]. Our results demonstrate a clear trade-off between toughness and hardness in ZnO and La_2O_3 -doped YSZ composites. While increased dopant levels enhance densification and improve hardness, they also result in increased brittleness. This is evident in the steep load-displacement curves and early failure of Samples 3 and 4. Therefore, we encourage a more inquisitive look at this matter as an optimum amount of dopant concentrations must be found to find a more suitable range between the toughness and hardness where there are no tradeoffs.

3.4 Thermal Properties

3.4.1 Thermogravimetric Analysis Test

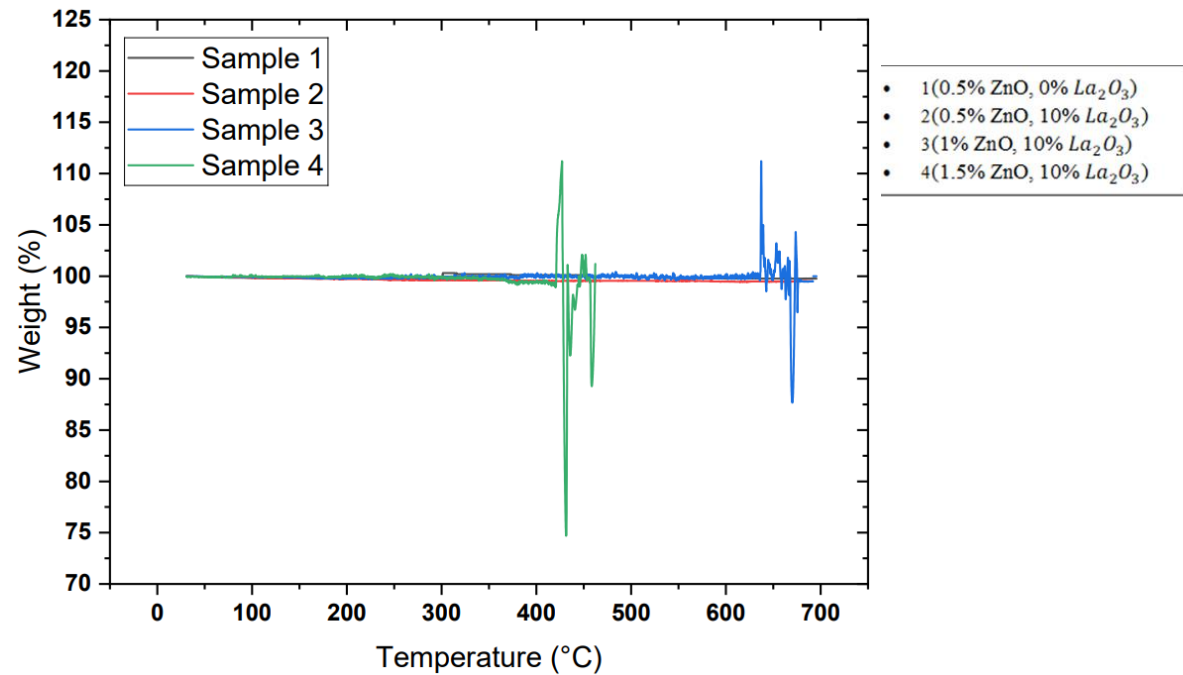


Fig 12: Weight (%) vs Temperature Ratio via Thermogravimetric Analysis

We used Thermogravimetric Analysis (TGA) to determine the thermal stability of the composites. As indicated in the graph, the weight loss curve shows the samples' thermal response throughout a wide range of temperatures

from 0 to 700°C. Sample 1 shows the maximum thermal stability with minimum weight loss throughout the thermal cycle, retaining approximately 99% percent of its initial weight at the highest tested temperature. This is consistent with the high YSZ content and negligible doping of ZnO, whereas, at 528°C, around 80% of the weight was lost only for YSZ without dopants. [29]. Several studies confirm the high thermal stabilities of YSZ-based composites where strong phase stability and minimum degradation of pure YSZ composites have been recorded, particularly under high thermal conditions. For instance, Parchovianský et al. (2022) found that YSZ composites retain phase stability and mechanical strength at elevated temperatures when lightly doped. [39]. Sample 2 showed a more significant weight loss than Sample 1 with 10 wt% La_2O_3 and 0.5 wt% ZnO. At 700°C, weight retention was around 89.5%, suggesting that La_2O_3 slightly affects thermal stability. Previous studies indicate that La_2O_3 incorporation can indeed improve the phase stability and sintering resistance of YSZ composites, as seen in research by Fabrichnaya et al. (2009), which demonstrated that La_2O_3 forms solid solutions with YSZ, improving its structural integrity. However, La_2O_3 volatilization at higher temperatures can contribute to weight loss during thermal analysis [40]. Sample 3 exhibited additional degradation, with just 89% of the weight preserved at 700°C. This composition has a greater ZnO concentration (1 wt%) and 10 wt% La_2O_3 , indicating that higher ZnO content may lead to lower thermal stability. Studies by Pihlatie et al. (2009) and Robles-Arellano & Bichler (2014) demonstrate that higher dopants of ZnO contents can increase grain boundary conductivity in YSZ composites. However, it leads to the formation of secondary phases that propagate degradation during the thermal cycling process [41] [42]. We found that sample 4 exhibited the greatest weight loss out of the 4 batches, retaining only 88.5% of its original weight at 700°C. This batch had the highest ZnO content (1.5 wt%) and 10 wt% La_2O_3 , suggesting that increasing ZnO concentration while maintaining La_2O_3 levels may threaten the composite's thermal stability. The reason behind this phenomenon can be described by the research by Parchovianský et al. (2022), who observed that while La_2O_3 enhances the structural integrity of composites, increasing the amount of ZnO can form phases that eventually lead to the thermal degradation of composites [39]. Although integrating both ZnO and La_2O_3 into the YSZ can improve phase stability and sintering resistance for sure, it can cause weight loss at high temperatures due to the volatilization of the Lanthanum-based compounds [43]. In addition, adding ZnO has been shown to increase the grain boundary conductivity of the YSZ composites. This creates secondary phases that contribute to weight loss during thermal analysis. It is evident from this test that while the incorporation of both ZnO and La_2O_3 improves the thermal stability of YSZ significantly, superfluous usage of ZnO into this co-doping mechanism can indeed contribute to weight loss. This phenomenon is supported by our mechanical tests on this ceramic composite, where we have encountered lower impact resistance, fracture toughness, and tensile stress of the sample with the most amount of ZnO. This is consistent with studies like that of Nagaraju & Annamalai (2019), who found that La_2O_3 stabilizes the cubic phase of YSZ but also reduces fracture toughness, which may exacerbate weight loss at higher temperatures [43]. The findings are consistent with prior research, demonstrating the trade-offs between phase stability and temperature degradation in these doped YSZ systems. However, thermal stability at higher temperatures is mostly required for thermal barrier coating applications, which can be gained for sample 1(0.5% ZnO) rather than only for YSZ.

3.4.1 Thermal Conductivity Test

Thermal conductivity results for YSZ doped with ZnO and La₂O₃ are illustrated in the graph for a temperature range from 100°C to 900°C. Four samples show distinct trends:

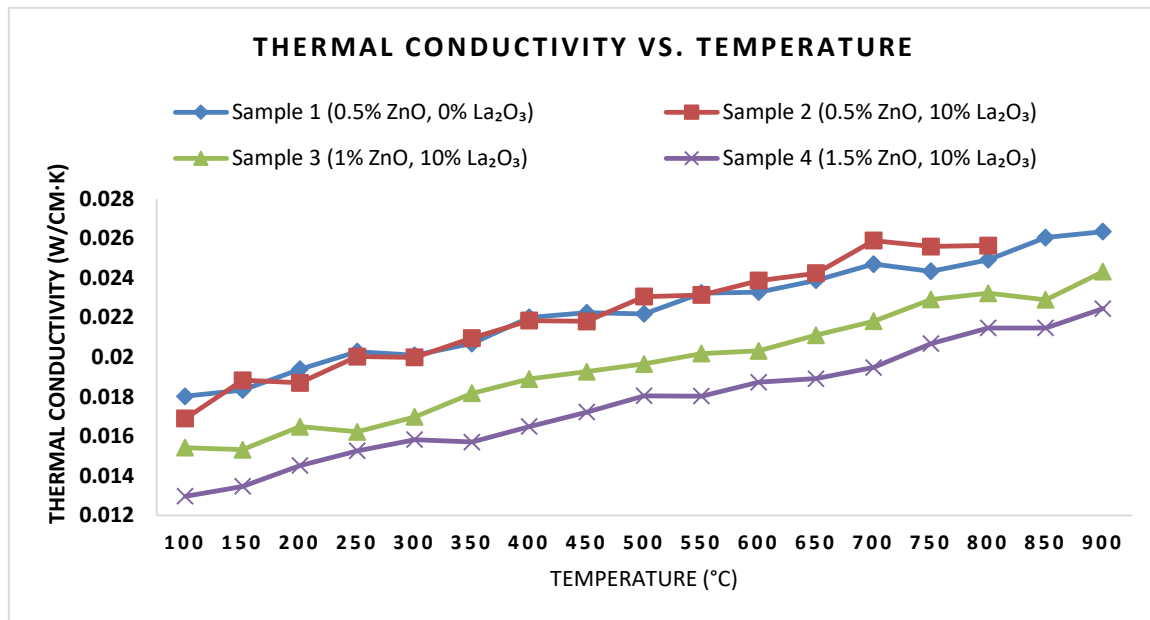


Figure 13: Thermal conductivity vs temperature curves for 4 samples of co-doped YSZ

Sample 1 (Blue line) maintains the highest conductivity throughout the temperature range, starting at about 0.018 W/cm·K at 100°C and increasing to approximately 0.026 W/cm·K by 900°C. This sample, with minimal La₂O₃, showcases the typical behavior of YSZ with enhanced heat conduction due to fewer dopants that impede phonon transfer. This trend corresponds with findings by Schlichting et al. (2001), who noted that dense YSZ without significant dopants retains higher thermal conductivity due to reduced phonon scattering [44]. Similarly, Guo et al. (2016) found that La₂O₃-doped YSZ exhibited lower thermal conductivity than pure YSZ, but minimal doping would reduce the extent of phonon scattering and result in higher heat conduction [45]. Sample 2 (Red line) shows a consistent but lower conductivity than Sample 1, indicating the initial impact of La₂O₃ doping. Starting at about 0.016 W/cm·K and rising to 0.022 W/cm·K, the presence of La₂O₃ helps stabilize the cubic phase but slightly reduces conductivity due to enhanced phonon scattering. This finding aligns with Liu et al. (2014), who found that La₂O₃ co-doped with YSZ decreased thermal conductivity by promoting phonon scattering due to lattice distortions [46]. La₂O₃ reduces densification and increases phonon scattering when used as a dopant, lowering thermal conductivity [5]. Sample 3 (Green line) demonstrates a more prominent reduction in thermal conductivity that started at 0.014 W/cm·K and increased towards 0.018 W/cm·K. Enhanced with ZnO dopant, this sample demonstrates the higher dopant levels in YSZ that scatters the grain boundary that inhibit thermal conductivity. Yang et al. (2017) found that ZnO doping in YSZ significantly improved densification and reduced thermal conductivity by generating oxygen vacancies [8]. Piticescu et al. (2021) showed that La₂O₃ and ZnO co-doping further reduced thermal conductivity by increasing grain boundary scattering and porosity [47]. Sample 4 (Purple line) exhibits the most substantial drop in conductivity that started slightly above 0.012 W/cm·K and ended around 0.016 W/cm·K. Due to the high levels of both ZnO and La₂O₃, photon scattering and phase stability happen at the maximum amount, thus resulting in the best thermal insulation properties. Chen et al. (2020) found that co-doping YSZ with La₂O₃ and Gd₂O₃ resulted in a 26.5% reduction in thermal conductivity compared to pure YSZ, primarily due to enhanced phonon scattering and reduced grain size [4]. Matsumoto et al. (2009) also highlighted the synergy between La₂O₃ and

ZnO in reducing thermal conductivity by stabilizing phases and enhancing phonon scattering [5]. The consistent increase in conductivity across all samples as temperature rises can be attributed to the increased mobility of phonons at higher temperatures despite the scattering effects introduced by the dopants. The results underscore the effectiveness of ZnO and La₂O₃ co-doping in reducing YSZ's thermal conductivity, making it highly suitable for thermal barrier applications where lower thermal conductivity is beneficial.

4. Conclusion

Doping yttria-stabilized zirconia (YSZ) with zinc oxide (ZnO) and lanthanum oxide (La₂O₃) was aimed towards improving the structural, mechanical and surface properties of YSZ for high temperature applications including thermal barrier coatings (TBCs). The study strategically investigated the combined impacts of these dopants, which uncovered notable enhancements in efficiency on multiple levels. Maintaining the thermal and structural stability of YSZ in harsh conditions, remains an important concern because of the requirements from sectors like aerospace and energy.

The research revealed that YSZ co-doped with ZnO and La₂O₃ showed remarkable improvements in crystallinity and phase stability for higher levels of 1.5% ZnO and 10% La₂O₃ additions. X-ray diffraction (XRD) analysis provided evidence for enhanced phase stability as the material remained in the stabilized tetragonal phase, which is critical for high thermal resistance. Also, thermogravimetric analysis (TGA) showed the material having thermal stability up to 700 degrees without significant weight loss, although higher concentrations of ZnO resulted in higher degradation relative to lower concentrations. This indicates that structural integrity is enhanced with co-doping but requires optimal levels of the dopant to ensure prolonged thermal stability.

Furthermore, density measurements indicated that co-doping enhances bulk density, improving from 74% to 92% as ZnO content increased. This increased density, alongside the significant reduction in porosity (from 26% to 2.6%), demonstrates that the co-doped material is more structurally compact, contributing to improved mechanical strength and durability. Vickers hardness testing confirmed that adding both ZnO and La₂O₃ contributes to increased hardness from 620 HV to 690 HV, improving the material's resistance to wear and deformation. However, this increase in hardness was accompanied by increased brittleness, as reflected in the results of Charpy impact tests, where energy absorption decreased with higher dopant levels, indicating reduced toughness.

Thermal conductivity tests further supported the material's suitability for TBC applications by showing a reduction in thermal conductivity, with the lowest value of 0.012 W/cm·K observed at higher ZnO concentrations. This lowered electrical conductivity increases thermal insulation, thus improving protection from overheating for other components. Nevertheless, this also leads to increased brittleness, as shown from the tensile stress tests which revealed increased concentration of ZnO led to lower failure stress. This indicates increased densification comes at a cost of decreased fracture toughness.

The results of this study underline key considerations that must be made for the design of materials that need to withstand severe mechanical and extreme thermal conditions. The co-doping approach allows for the desired increase in hardness, density, phase stability, and thermal conductivity making the material appropriate for gas turbines and energy systems where durability is critical. Nonetheless, the increase in brittleness at greater dopant concentrations illustrates the need for better failure prediction when the material is subjected to operational stresses.

Despite the remarkable improvements in the co-doped YSZ material, challenges remain. Thermal stability and toughness were not considered in the wider range of dopant concentration that yields other results. Moreover, longer-term performance under sustained operational stresses needs other investigations to know if the material loses its enhanced properties over time.

All in all, the co-doping of YSZ with ZnO and La₂O₃ has proven to significantly improve its mechanical and thermal properties, positioning it as a strong candidate for more efficient and durable thermal barrier coatings and solid oxide fuel cells. The enhancements in hardness, density, phase stability, and thermal conductivity are promising, but future research is needed to fine-tune the balance between brittleness and toughness, ensuring the material's long-term reliability in high-performance systems.

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Appendix

A.1. Lanthanum Oxide Powder



A.2. YSZ Powder



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A.3. Zinc Oxide Powder



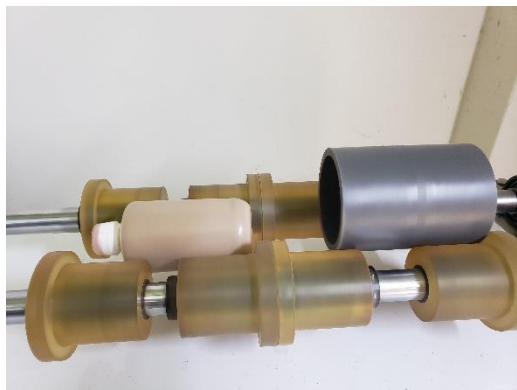
A.4. Morter Pestle



A.5. Ethanol



A.6. Ball Milling Setup



A.7. Drying in Oven for Evaporating Ethanol



A.8. Dried Powders



A.9. Dried Powder for Calcination



A.10. Hydraulic Pressing



A.11. Tablet Formation



A.12. Protherm Oven for Sintering



Dear Editor,

I am writing to submit my research manuscript titled “*Co-Doping Yttria-Stabilized Zirconia (YSZ) with Zinc Oxide (ZnO) and Lanthanum Oxide (La₂O₃) for Enhanced Structural, Mechanical, and Thermal Properties in High-Temperature Applications*” for consideration for publication in **Ceramics International**. This study explores the synergistic effects of ZnO and La₂O₃ co-doping on YSZ, a ceramic material commonly used in thermal barrier coatings (TBCs), and presents significant findings that enhance the material's performance under extreme conditions.

In this manuscript, we provide an in-depth analysis of the influence of ZnO and La₂O₃ doping on the structural, mechanical, and thermal properties of YSZ, focusing on applications such as gas turbines and energy systems. Through systematic testing, we demonstrate how co-doping can improve crystallinity, phase stability, density, hardness, and thermal conductivity, while also addressing the trade-offs between toughness and brittleness at higher dopant levels.

Our study provides novel insights into the optimization of dopant concentrations, and offers a potential solution for improving the reliability and efficiency of materials exposed to high thermal and mechanical stresses. This research contributes to the growing body of knowledge in materials science and has important implications for industries that rely on high-performance ceramics, such as aerospace, energy, and automotive sectors.

I believe that this paper is well-suited for **Ceramics International**, as it aligns with the journal's focus on **Ceramics**. The findings discussed in this research are both novel and highly relevant to current advancements in material design for extreme operational environments.

Thank you for considering my manuscript for publication. I look forward to your feedback and am happy to make any necessary revisions to ensure the paper meets the journal's standards. Please do not hesitate to contact me should you require any additional information.

Sincerely,

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27/02/2025

Declaration of Interest

The authors of this manuscript hereby declare that there are no financial, personal, or professional relationships that could be perceived as potential conflicts of interest regarding the research presented in this paper. Specifically:

- The research was conducted independently without any financial support from external organizations or sponsors that could influence the content or outcome of the study.
- There are no personal, academic, or professional relationships between the authors and any entities, individuals, or companies that could affect the integrity of the research or its findings.
- The authors do not have any proprietary interests in the materials, methods, or products discussed in this paper, and there is no commercial involvement related to the research outcomes.
- No competing interests exist in terms of the interpretation of data or the conclusions drawn from the results presented in this study.

We confirm that all ethical guidelines and academic integrity standards were adhered to during the design, conduct, and reporting of the research, ensuring that the findings are presented in an unbiased and transparent manner.