



Article

Effect of Sintering Temperature on the Physical and Mechanical Characteristics of Fabricated $\text{ZrO}_2\text{--Cr--Ni--Ce--Y}$ Composite

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Abstract: The present study investigates the synthesis and characterization of a zirconium oxide (ZrO_2)-based metal composite doped with cerium (Ce) and yttrium (Y), using chromium (Cr) and nickel (Ni) as base metals. These constituents were selected for their superior mechanical properties and compatibility with the ceramic phase. High-purity powders were homogenized via high-energy ball milling, followed by cold pressing and sintering in a controlled atmosphere of hydrogen. The sintering process was conducted at temperatures ranging from 850 °C to 1350 °C to examine the evolution of microstructure, grain growth, and densification. Scanning electron microscopy (SEM) revealed a homogeneous distribution of phases, with distinct microstructural features attributed to each element at different sintering temperatures. The experimental results revealed that the composite's density was increased by 30% and porosity was reduced by 61% at a sintering temperature of 1350 °C. The hardness and flexural strength of composite were found to be 23% and 60% higher at 1350 °C, respectively, compared to that at 850 °C, suggesting enhanced mechanical properties due to cerium and yttrium reinforcement within matrix and efficient doping and phase transformation. Overall, incorporation of cerium and yttrium significantly improved mechanical behavior and phase stability of $\text{ZrO}_2\text{--Cr--Ni}$ composite, highlighting its potential for advanced engineering applications.



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1. Introduction

In the realm of advanced materials, the quest for novel compositions and fabrication methods has driven researchers to explore uncharted territories, seeking materials that exhibit remarkable mechanical properties and diverse functionalities. The mixing of metals and ceramics has been a focal point of this pursuit, yielding composites that combine the best of both worlds: the toughness of metals and the hardness of ceramics. Among these innovative combinations, $\text{ZrO}_2\text{--Cr--Ni}$ -based metal–ceramic blends with yttrium (Y) and cerium (Ce), synthesized through the versatile technique of powder metallurgy, have emerged as a captivating subject of study. The union of ZrO_2 , Cr, and Ni brings forth a triad of elements that encompass intriguing mechanical attributes. Zirconia (ZrO_2), a well-recognized ceramic, exhibits a rare phenomenon known as transformation toughening, wherein it undergoes a phase transition under mechanical stress, effectively retarding

crack propagation. Chromium (Cr), on the other hand, introduces corrosion resistance and oxidation stability, characteristics particularly attractive for applications in demanding environments. Nickel (Ni), with its exceptional ductility and thermal conductivity, complements this amalgamation by enhancing the overall toughness and thermal performance. The addition of chromium (Cr) and nickel (Ni) to ZrO_2 has been shown to improve mechanical properties, but recent research has pushed the boundaries further by incorporating yttrium and cerium as reinforcing agents, giving rise to ZrO_2 –Cr–Ni-based metal–ceramic blends. Cho et al. (2019) ventured into the nuances of phase transformations, elucidating the impact of yttrium on the martensitic transformation of ZrO_2 , a phenomenon central to its transformation-toughening mechanism [1]. This intricate dance of phases was further deciphered by Li et al. (2020), who unveiled the role of cerium in influencing the kinetics of phase transitions and crystallite growth [2]. For instance, Tian et al. (2019) investigated the effects of yttrium oxide (Y_2O_3) on the mechanical properties of alumina-zirconia composites [3]. The addition of Y_2O_3 was found to enhance the strength and toughness of the composites, attributed to the formation of a fine-grained microstructure and phase transformation toughening. Similarly, Wu et al. (2020) examined the role of cerium oxide (CeO_2) as a sintering aid in zirconia ceramics. The inclusion of CeO_2 promoted densification and grain growth, resulting in improved mechanical properties [4]. Zhu et al. (2018) investigated the fracture toughness of ZrO_2 –Cr–Ni–Y–Ce composites and observed significant enhancement compared to the pure ZrO_2 matrix [5]. The researchers attributed these enhancements to the formation of yttrium-stabilized tetragonal zirconia phases, which transformed into monoclinic zirconia under stress, effectively impeding crack propagation. In a comprehensive study by Chen et al. (2020), cerium additions were found to increase the fracture toughness of alumina ceramics, a phenomenon attributed to the formation of cerium oxide precipitates at grain boundaries [6]. This intriguing phenomenon, coupled with the notable ability of cerium to scavenge oxygen vacancies, holds great promise in the context of metal–ceramic composites. The symphony of mechanical attributes resonates through the resonance of these blends, encompassing parameters such as fracture toughness, fatigue resistance, and wear behavior. The stage on which these compositions perform spans the spectrum of biomedical applications, where materials are thrust into the crucible of human well-being. Dental implants, bearing the weight of mastication, find their resonance in the enhanced mechanical stability of these metal–ceramic blends. Orthopedic implants, bridging the chasm of bone fractures, draw strength from the augmented load-bearing capacity of these composites. The biomedical orchestra extends further to include prosthetic components, surgical instruments, and drug delivery systems, all harmonizing to the tune of enhanced mechanical functionality. The mechanical behavior of ZrO_2 –Cr–Ni-based metal–ceramic blends with yttrium (Y) and cerium (Ce) prepared through powder metallurgy has emerged as a topic of significant interest in materials science and engineering. This literature review aims to explore the current state of research and advancements in this area, shedding light on the synthesis, mechanical properties, microstructural characteristics, and potential applications of these composites. Zhang et al. (2019) employed powder metallurgy (PM) to fabricate ZrO_2 –Cr–Ni-based metal–ceramic blends with yttrium and cerium. The study demonstrated the feasibility of the PM route in obtaining uniform distribution of Y and Ce within the matrix, leading to improved mechanical properties [7]. A key advantage of PM is the ability to tailor the composition and microstructure of the composites by controlling powder characteristics, mixing parameters, and sintering conditions. Several studies have explored the influence of powder characteristics, such as particle size and morphology, on the mechanical properties of the resulting composites (Jian et al., 2020) [8]. Ikoma et al. (2016) demonstrated that the addition of Y promoted the formation of a tetragonal zirconia phase in alumina-zirconia composites, imparting transformation-toughening capabilities and mitigating crack propagation [9]. Kumar et al. (2017) [10] demonstrated the successful fabrication of Y_2O_3 - and CeO_2 -doped ZrO_2 –Cr–Ni powders through co-precipitation and subsequent reduction processes. The addition of Y and Ce was found to enhance the sinter ability of the powders, resulting in denser and more homogenous compacts. Moreover, the

unique mechanical behavior of zirconia, such as transformation toughening, was further enhanced by yttrium addition. Besides the powder metallurgy technique, other techniques that are employed for synthesis of zirconia powders include co-precipitation, sonochemical, aqueous, and non-aqueous sol–gel methods [11]. Deng et al. (2019) [12] elucidated the toughening mechanisms in ZrO_2 –Cr–Ni-based composites with yttrium, showing that the transformation from the tetragonal to the monoclinic phase under stress effectively hindered crack propagation, leading to improved fracture toughness. Tensile tests have been conducted to assess the mechanical strength and ductility of the composites (Wang et al., 2019). The addition of yttrium and cerium has been shown to improve the tensile strength and elongation of the composites [13]. Hardness measurements are another essential aspect of the mechanical characterization, as hardness directly influences the wear resistance and load-bearing capacity of the materials. The incorporation of yttrium and cerium has been reported to enhance the hardness of the composites due to the formation of solid solutions and grain-boundary strengthening (Zhou et al., 2019) [14].

The aim of present investigation is to systematically examine the correlation between the sintering temperature and the resulting physical and mechanical properties of a ZrO_2 –Cr–Ni–Ce–Y composite. By subjecting fabricated composite samples to a range of sintering temperatures, this research aims to elucidate the optimal temperature for achieving desired mechanical characteristics. Understanding how sintering temperature affects the properties of ZrO_2 –Cr–Ni–Ce–Y composites can lead to the production of materials with superior mechanical characteristics. This research provides valuable insights into the microstructural evolution of ZrO_2 -based composites during sintering. ZrO_2 -based composites are widely used in various high-tech applications, including aerospace, automotive, and biomedical fields. Enhancing its properties through precise control of sintering parameters can expand its applicability and performance in these demanding environments.

2. Materials and Methods

Zirconium oxide (ZrO_2), chromium (Cr), and nickel (Ni) were chosen as the base metallic constituents due to their better tensile properties and compatibility with the ceramic phase. The yttrium (Y) and cerium (Ce) powders were selected as dopants for the ceramic phase based on their ability to improve mechanical properties and phase stability [15]. High-purity zirconium dioxide (ZrO_2), chromium (Cr), nickel (Ni), and yttrium (Y) powders from suppliers, as shown in Table 1, were used for this procedure. The reason behind the selection of these materials was based on the theory of atomic radius of the elements. All the elements have very similar atomic radii, which would be an approval factor in the selection of appropriate matrix for the metal composite. Also, the dopants used for the composite have compatible atomic sizes to be fitted perfectly in the lattice spaces of the metal composite [16].

Table 1. Raw material details.

Powder	Supplier	Purity (%)	Particle Size (μm)	Apparent Density (g/cm^3)	Density (ρ) (g/cm^3)
ZrO_2	Sood Chemicals Haryana, India	95.01	1–5	5.5	5.89
Cr	Sood Chemicals Haryana, India	95.16	1–5	7.10	7.19
Ni	Sood Chemicals Haryana, India	95.10	1–5	8.02	8.91
Yttrium (Y)	Nano laboratories Punjab, India	98.20	1–5	4.4	6.96
Cerium (Ce)	Nano Laboratories Punjab, India	99.02	40	6.23	6.77

To prepare the composite, the specific volume percentages of each component taken are as follows: 20% ZrO_2 , 35% Cr, 35% Ni, 5% Cerium, and 5% yttrium. The powders were mixed and homogenized via high-energy ball milling in the depressurized steel chamber to ensure a uniform distribution of the dopants, i.e., cerium and yttrium within the ZrO_2 –Cr–Ni matrix. Before the compaction process, the composite was mixed with the binding agent. Iso-Propyl alcohol of a 90–95% concentration was used for obtaining a well-compacted and -matrixed product. The homogenized powder blend was compacted into a green body using a cold press (CP), with the compaction pressure and dwell time optimized to achieve the desired density and eliminate any defects in the green body. The raw powders of the composite were mixed separately via a high-energy mill with a ball-to-powder mass ratio of 10:1 at a speed of 200 rpm for a duration of 24 h to achieve a sustainable powder blend. The mixed powder was transferred to a die set for compaction. A punch of $\varnothing 7$ mm was used in the compaction. Under controlled pressure and through cold pressing, the powder mixture was compacted. The pressure applied during cold pressing ranges from 130 MPa to 660 MPa to achieve the desired density and compaction of the powder. The compaction time depends on the equipment, the density requirements, and the properties of the powder. The compaction time for cold pressing was 13 min. It was performed at room temperature to avoid sintering or densification due to heat. Therefore, the temperature was kept at or close to room temperature during the compaction process. A controlled and gradual pressurization is often employed to prevent sudden pressure fluctuations and ensure uniform compaction. A depressurization rate of 174 MPa per minute was employed in cold pressing.

The green body was sintered in a controlled atmosphere of hydrogen at specific temperature and duration. In sintering, the temperature is a critical parameter that determines the level of densification, grain growth control, the formation of the desired microstructure, and the mechanical properties of the composite. The compacted metal composite was sintered at 1350 °C for 200 min. However, a keen record has been maintained by observing the composite at different temperatures, i.e., at 850 °C, 950 °C, 1050 °C, 1150 °C, 1250 °C, and 1350 °C. A controlled and gradual heating rate of 5.5 °C per minute was employed to minimize the thermal stresses and prevent cracking. A holding time of 200 min was employed during the sintering, allowing for sufficient densification and bonding between the powder particles. After sintering, a controlled cooling rate was employed to avoid rapid thermal gradients and reduce the risk of cracking. During the sintering of the metal-based composite, a reducing atmosphere constituting hydrogen gas was prepared in the furnace to prevent oxidation for achieving better results. Figure 1 shows a graphical illustration of the composite fabrication procedure.

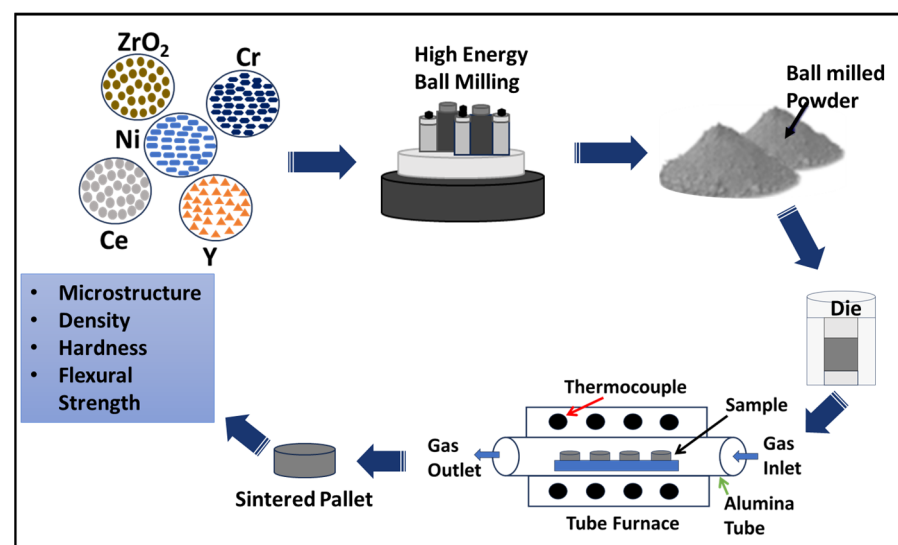


Figure 1. Flow diagram of the synthesis of ZrO_2 –Cr–Ni–Ce–Y composite.

3. Characterization and Testing Technique

3.1. SEM Analysis

The scanning electron microscope (SEM) was used to examine the microstructure of the sintered sample of size 7 mm × Ø7 mm. The sample was subjected to decarbonization with the help of dry, hot, compressed air for removing the iso-propyl alcohol which was used as the binding medium in the powder metallurgy process.

3.2. Measurement of Density

The Archimedes principle was applied to determine the density of the sintered composite. The following mathematical relation was used to determine the density:

$$\rho_{ex} = \frac{W_a}{W_a - W_w} \quad (1)$$

ρ_{ex} : density of sintered sample;

W_a : weight in air;

W_w : weight in water;

The rule of mixtures was utilized to compute the theoretical density of the fabricated composite. The inverse rule of mixtures is expressed as follows:

$$\frac{W}{\rho_{th}} = \left(\frac{W}{\rho}\right)_{ZrO_2} + \left(\frac{W}{\rho}\right)_{Cr} + \left(\frac{W}{\rho}\right)_{Ni} + \left(\frac{W}{\rho}\right)_Y + \left(\frac{W}{\rho}\right)_{Ce} \quad (2)$$

3.3. Mechanical Testing

The polished sintered composite sample was used for bulk hardness testing. To ensure accuracy, at least five indentations were made on the sample, and the average hardness was calculated. The hardness measurement was carried out via a Vickers hardness tester at a load of 1 kg and a dwell time of 15 s, as per ASTM E92. Indentation was made on the surface of the sintered composite sample. A 50 mm × Ø7 mm size of sample was ground and polished to transform it into a cuboid with a dimension of “30 × 5 × 5 mm” for hardness testing. The sintered sample was cut into smaller pieces of 10 mm × Ø7 mm with a diamond saw. A flexural test was carried out to determine the flexural strength of the sintered sample on a Universal Testing Machine (INSTRON make) at a cross speed of 1 mm/min as per ASTM: A-370. The flexural test specimen dimensions are 25 mm × 4 mm × 1.2 mm. The sample was loaded axially until failure, and the maximum applied load was recorded to calculate the flexural strength.

4. Results and Discussion

4.1. Phase Analysis and Microstructural Features of ZrO₂ Metal Composite

The X-ray diffraction (XRD) patterns of sintered ZrO₂ composite are shown in Figure 2. Figure 2a depicts the XRD pattern of the composite at a sintering temperature of 850 °C. Similarly, Figure 2b,c depict the XRD patterns of the composite at sintering temperatures of 1150 °C and 1350 °C, respectively. The XRD patterns at the three sintering temperatures are similar to each other. The XRD patterns clearly show four distinct peaks, indicating that the sintered ZrO₂ composite is polycrystalline in nature. It is clearly observed from the XRD patterns that the ZrO₂–Y phase occurs at a diffraction angle of 26°, the ZrO₂ phase at 41°, the Cr–Ni phase at 48°, and the CeO₂ phase at 75°.

The SEM micrograph of the sintered composite is shown in Figure 3. Notably, it revealed a homogeneous distribution of ZrO₂, Cr, Ni, Ce, and Y phases across various sintering temperatures, indicating the effectiveness of the fabrication process. The microstructure consists of gray, black spot, grayish-white, polar white, and silverish white particles. The grayish areas were attributed to the presence of Ni having a BCC phase (Figure 3c), while the black spots indicated Cr having an FCC phase (Figure 3d), and white spots represented the ZrO₂ phase (Figure 3d). Additionally, the polar-white cotton-shaped

structures (Figure 3c) suggested the presence of yttrium, whereas the silverish white coloration depicted the presence of cerium in the metal composite (Figure 3d). At a sintering temperature of 850 °C, the stone-shaped structures that are formed are attributed to the slow fusion rate of Ni and Cr into ZrO_2 , being influenced by the addition of cerium and yttrium (Figure 3a). This phase persisted up to a sintering temperature of 950 °C.

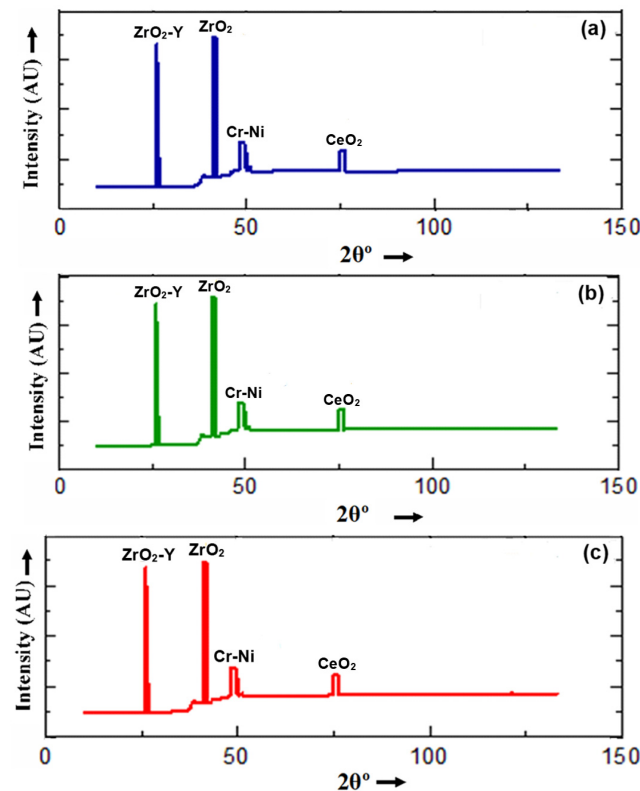


Figure 2. X-ray diffraction patterns of sintered ZrO_2 –Cr–Ni–Ce–Y composite at sintering temperatures of (a) 850 °C, (b) 1150 °C, and (c) 1350 °C.

Subsequently, significant changes occurred between 1050 °C and 1150 °C, characterized by smaller stone-shaped structures, reflecting the impact of increased sintering temperature (Figure 3b). The kinetics of sintering process is influenced by the sintering temperature. At higher temperatures, sintering occurs more rapidly, leading to accelerated densification and structural changes in the material [17]. At 1250 °C, the stone-shaped structures are fused to form layered structures, resembling a flowing fluid but maintained the solidity upon closer inspection (Figure 3c). The higher temperature promoted additional atom transport and reorganization in the material, which resulted in the fusing of discrete structures into stratified structures. The smooth, continuous look of these layered formations gave the impression that they were flowing fluid, simulating the fluid-like behavior of a molten material. But upon closer examination, it was clear that these formations were actually solid, and although they looked fluid, they still had structural coherence and integrity. This phenomenon highlights the dynamic nature of phase change in designed materials by illuminating the intricate interactions between temperature, material composition, and microstructural evolution throughout the sintering process [18]. After the completion of sintering process at 1350 °C, a layer of silverish white coloration, alongside polar-white cotton-shaped structures, emerged, indicating the fusion of cerium and yttrium with the boundaries of the ZrO_2 –Cr–Ni matrix. Furthermore, the micrograph developed at 1350 °C highlighted the formation of strong metallurgical bonds between the grains, resulting in a sudden decrease in porosity and an increase in the degree of dissolution. This densification process, occurring relatively late, led to the formation of a compactly packed microstructure within the metal composite.

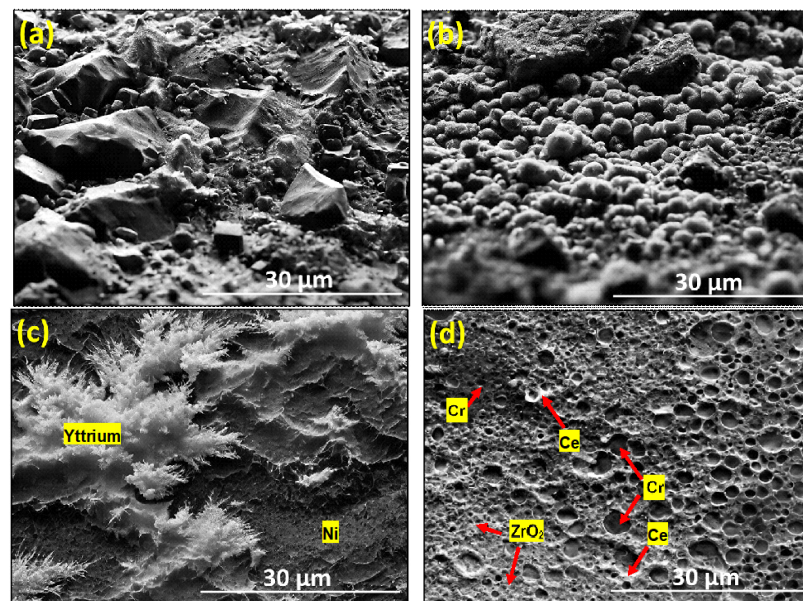


Figure 3. The SEM micrograph of the sintered $\text{ZrO}_2\text{-Cr-Ni-Ce-Y}$ composite at temperatures of (a) 850–950 °C, (b) 1050–1150 °C, (c) 1250 °C, and (d) 1350 °C.

4.2. Variation in Density and Porosity of Composite with Sintering Temperature

Figure 4 illustrates the effect of sintering temperature on the density and porosity of the sintered composite. It is evident that both theoretical and experimental densities increase significantly with rising sintering temperature during the initial phase of the sintering process. Solid-phase sintering occurs at temperatures below the melting point of Ce (796 °C). During this phase, enhanced atomic diffusion improves particle contact, reduces porosity, and facilitates the formation and growth of sintering necks, thereby increasing the density. When the temperature surpasses the Ce-melting point (796 °C), a bridged Ce network forms as the liquid Ce phase bonds and links different composite particles. Figure 4 demonstrates the relationship between porosity and sintering temperature. The reduction in porosity occurs because of the liquid-phase Ce filling the pores between the $\text{ZrO}_2\text{-Cr-Ni}$ grains. Before sintering, many pores are located at particle contact points and around grains. At 850 °C, the Ce liquid phase rearranges to fill the pores, further reducing porosity. However, from 850 °C to 1350 °C, porosity values decrease significantly due to the rapid diffusion of Ce into the $\text{ZrO}_2\text{-Cr-Ni}$ composite [19].

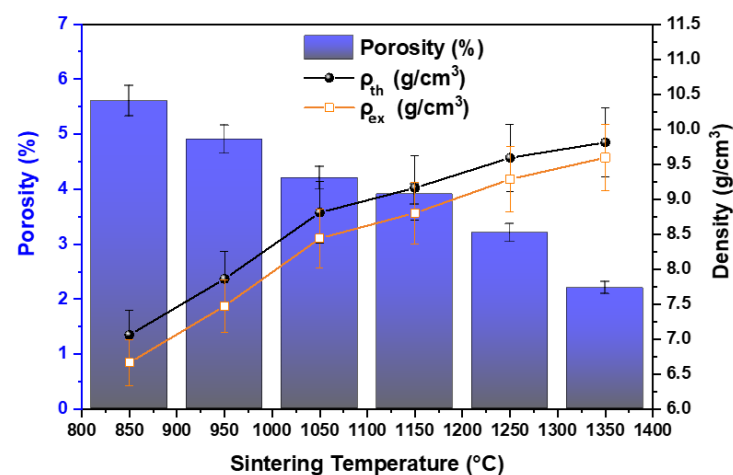


Figure 4. Variation in density and porosity of $\text{ZrO}_2\text{-Cr-Ni-Ce-Y}$ composite with sintering temperature.

4.3. Hardness

Figure 5 shows a plot of hardness versus sintering temperature. The specimen obtained at the temperature range of 850–1350 °C was tested via the Vickers hardness tester. The obtained Vickers hardness value was closely matched with the estimated hardness. Figure 5 revealed that as the sintering temperature increased, the hardness of the ZrO₂–Cr–Ni–Ce–Y metal composite increased. However, at sintering temperatures between 1050 °C and 1150 °C, only a slight increase in hardness was observed. This is due to the densification of cerium and yttrium within the lattices of the ZrO₂–Cr–Ni metal composite [20]. During doping of non-metallic and metallic elements, stable hardness occurred due to the reinforcement provided by the doped material to the matrix of metal composite, which would require high amounts of energy for complete densification to obtain a homogenous mixed composite. Moreover, the cerium and yttrium addition provides a significant reinforcement to the ZrO₂–Cr–Ni metal composite, which requires an extra amount of latent energy during the sintering process for full densification. The addition of cerium and yttrium to the ZrO₂–Cr–Ni matrix can lead to solid-solution strengthening, wherein the atoms of the added elements occupy interstitial or substitutional positions within the crystal lattice, effectively impeding the movement of dislocations and increasing the material hardness [21]. The formation of smaller stone-shaped structures was also observed in the sintering temperature range of 1050–1150 °C, as depicted in Figure 3b, compared to the larger structures shown in Figure 3a. The hardness increased with the increase in sintering temperature. High sintering temperatures facilitate grain growth within the material, resulting in the larger crystal structures. The larger crystal structures formed at the higher sintering temperature contribute to the material's increased hardness by reducing the grain boundary area, enhancing the dislocation interaction, and promoting strain hardening. This mechanism collectively improves the material resistance to deformation and makes it harder [22].

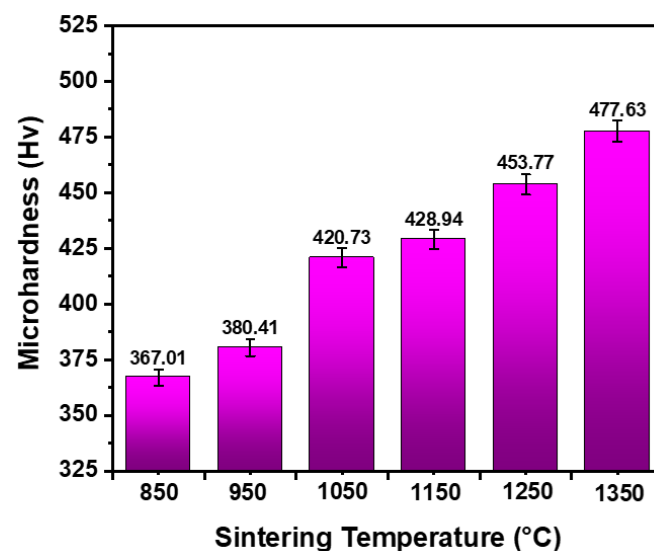


Figure 5. Variation in microhardness of ZrO₂–Cr–Ni–Ce–Y composite with sintering temperature.

4.4. Flexural Strength

Figure 6 illustrates the flexural strength for various porosity levels of the sintered ZrO₂–Cr–Ni–Ce–Y composite at different sintering temperatures ranging from 850 °C to 1350 °C. It was observed that the addition of cerium and yttrium in the ZrO₂–Cr–Ni metal composite increases the flexural strength that attain a maximum value of 964 MPa at 1350 °C. Also, it was observed that at this value the rate of porosity also decreases quite swiftly. With the propagation of sintering temperature, the rate of porosity decreases instantly. The addition of yttrium enhanced the strength of the fabricated composite. During compaction and sintering process, the doping agents need some time to densify completely into the

metal composite matrix which require an extra amount of latent energy [23]. However, the flexural strength did not get affected, as it has always maintained a linear relationship with sintering temperature. But, the values of flexural strength were observed to be much higher than 578 MPa in the case of the $\text{ZrO}_2\text{-Cr-Ni}$ composite in the range of sintering temperature from 1000 °C to 1200 °C. From this, it is confirmed that to some extent, the flexural strength was also affected by doping $\text{ZrO}_2\text{-Cr-Ni}$ metal composite with cerium and yttrium in the sintering temperature range of 1000 °C to 1200 °C. Finally, it could be concluded that the doping of cerium and yttrium enhances the mechanical behavior of the metal composite ($\text{ZrO}_2\text{-Cr-Ni}$) and correlates with the changes that occurred in the microstructure and phase transformation with the mechanical behavior of the metal composite. The increase in flexural strength may be due to the high density of the composite. It has been observed from Figure 6 that the flexural strength of composite increases sharply with an increase in the sintering temperature of up to 1350 °C. This may be attributed to the increase in transformable tetragonal phase in the material due to CeO_2 concentration [24]. The values of flexural strength signify that the flexural strength is controlled by stress-induced transformation toughening.

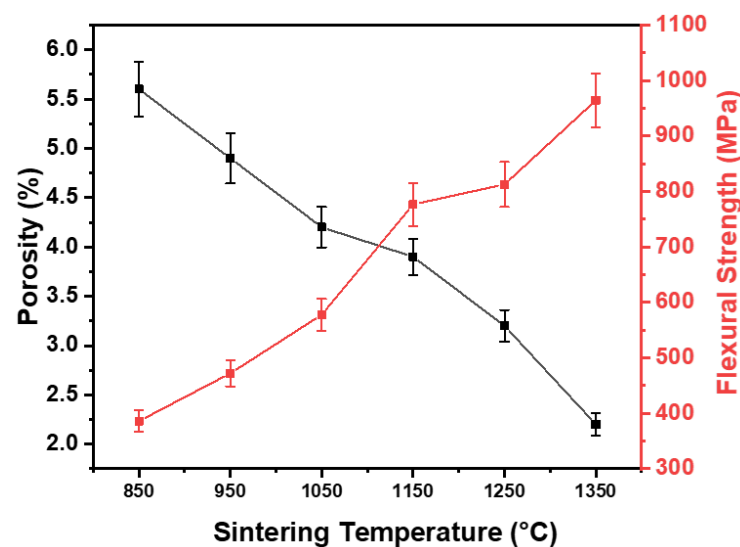


Figure 6. Variation in flexural strength of $\text{ZrO}_2\text{-Cr-Ni-Ce-Y}$ composite with sintering temperature at different levels of porosity.

5. Conclusions

The following conclusions may be drawn from the present study on $\text{ZrO}_2\text{-Cr-Ni-Ce-Y}$ composite:

- The SEM micrographs depict a homogeneous distribution of ZrO_2 , Cr, Ni, Ce, and Y phases, indicating the effective fabrication of the composite.
- At lower sintering temperatures (850 °C to 950 °C), the microstructure features stone-shaped structures due to slow fusion rates, which evolve into smaller forms between 1050 °C and 1150 °C.
- The microstructure of the composite has been refined from a coarse, stone-shaped structure at lower temperatures to finer, layered formations at higher temperatures, culminating in a dense, compact structure at 1350 °C.
- The density of the composite increased with temperature from 6.7 gm/cm^3 at 850 °C to 9.7 gm/cm^3 at 1350 °C, primarily due to filling of pores by liquid Ce phase.
- The micro-hardness was found to be 367 HV at 850 °C and 478 HV at 1350 °C. This is due to the solid solution strengthening and the grain refinement.
- The flexural strength steadily increased with temperature, from a value of 386 MPa at 850 °C and reaching a peak value of 964 MPa at 1350 °C, demonstrating the composite's potential for high-performance applications.

- The addition of cerium (Ce) and yttrium (Y) played a crucial role in enhancing the mechanical properties of the composite through solid-solution strengthening and densification.

Author Contributions: B.C.S. and D.K.R. conducted experimental tests on composites and performed data processing; N.J., V.S. and A.V. helped with the collection of data from the scientific literature review, organized the results obtained from the scientific community, and wrote the manuscript; D.M.G., K.R. and D.G.P. contributed to result interpretation and the discussion of the selected results. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflicts of interest.

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