

Article

Synthesis and Characterization of MgO-ZrO₂ Heterostructure: Optical, Mechanical and Electrical Properties

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Abstract: The synthesis and characterization of MgO-ZrO₂ heterostructures are examined in this work. To promote the creation of nanowires, the Si substrate is first covered with a catalyst layer of various Au thicknesses. Sputtering is used to achieve this deposition. After that, chemical vapor deposition (CVD) with a Au catalyst layer is used to create MgO nanowire arrays on the silicon substrate. Second, MgO/ZrO₂ Core-shell Nanowire Arrays are created by applying ZrO₂ layers to the surface of MgO nanowires of different diameters using chemical vapor deposition (CVD) procedures. The presence of both magnesium oxide (MgO) and zirconium dioxide (ZrO₂) in their oxidized forms was shown by the detailed characterization of the MgO-ZrO₂ core-shell nanowire samples utilizing a variety of methods. Phase formation, mechanical homogeneity, optical characteristics, and topographical structure and roughness were all thoroughly examined at various stresses. MgO hardness values ranged from 1.4 to 3.2 GPa, whereas MgO-ZrO₂ ranged from 0.38 to 1.2 GPa. The I-V parameter study was a further step in the examination of the heterostructure's electrical properties. The structural, morphological, optical, mechanical, and electrical properties of the MgO-ZrO₂ heterostructure were all thoroughly described using these techniques.

Keywords: MgO-ZrO₂ heterostructure; optical properties; mechanical strength; electrical properties



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1. Introduction

Magnesium oxide (MgO) is a face-centered cubic crystal exhibiting a rock-salt structure. Owing to its non-toxicity, affordability, thermal stability, corrosion resistance, excellent thermal conductivity, and optical characteristics, MgO has been extensively utilized in solar cells, antibacterial agents, sewage treatment, building material additives, and electron emission materials. Due to the surface effect and size impact of nanomaterials, MgO nanostructures have enhanced physical and chemical characteristics compared to bulk materials. Nano-MgO has significant catalytic activity and is effectively used in water vapor and CO catalysis due to the active sites on its surface and edge that possess charge transfer potential [1]. At room temperature, pure ZrO₂ has a monoclinic crystal structure. However, at around 1170 °C, it transforms a tetragonal structure, and at temperatures

above 2370 °C, it converts to a cubic fluorite structure [2,3]. The t→m transformation in ZrO₂ is known to cause a large volume expansion of approximately 4.5%. This expansion can lead to the catastrophic fracture and structural unreliability of prepared components, which greatly limits the application of ZrO₂ as an engineering ceramic [4,5]. The addition of low-valent binary oxides, such as Y₂O₃, CaO, and MgO, can help to stabilize high-temperature polymorphs of ZrO₂ at room temperature. This is because these oxides can modify the crystal structure and control the grain size of ZrO₂, which in turn improves its mechanical properties and makes it suitable for a wide range of applications [6]. Out of the various stabilized zirconia materials, the ones stabilized with MgO have been found to possess superior fracture toughness, high chemical stability, and excellent ionic conductivity [7–9]. MgO-stabilized ZrO₂ has found wide applications in various fields such as refractory materials, thermal barrier-coating systems, ceramic electrolytes for oxygen sensors, and dental restorative materials due to its superior fracture toughness, excellent ionic conductivity, and high chemical stability. Consequently, significant research efforts have been directed towards the synthesis of MgO-stabilized ZrO₂ powders [10,11], but energy-saving and scalable methods for preparing MgO-stabilized ZrO₂ powders still need to be explored [8]. Photocatalysis has gained significant attention in recent years as an environmentally friendly and sustainable technology to address various environmental issues such as air and water pollution [12–14]. ZrO₂, on the other hand, exhibits high photocatalytic activity due to its unique structural and electronic properties, such as high surface area, large bandgap, and efficient electron–hole separation [15–17]. MgO-ZrO₂ nanomaterials are a class of mixed metal oxides that have attracted significant research interest due to their unique physical and chemical properties [18–21]. These nanomaterials are composed of magnesium oxide (MgO) and zirconium dioxide (ZrO₂) in varying ratios, depending on the synthesis method used. MgO and ZrO₂ are both well-known materials with distinct properties that make them useful in a wide range of applications. MgO is an alkaline earth oxide with a high melting point and excellent thermal stability [22,23]. It is widely used as a catalyst support due to its high surface area and basicity. On the other hand, ZrO₂ is a transition metal oxide with high thermal stability, mechanical strength, and excellent chemical resistance [24,25]. It has been utilized in a range of applications such as ceramic coatings, fuel cell membranes, and catalysis [26,27]. The combination of these two materials in a mixed metal oxide provides a material with unique properties that make it attractive for various applications such as catalysis, gas sensors, photocatalysis, and biomedical applications [28,29]. The synergistic effect of MgO and ZrO₂ in the mixed oxide can lead to enhanced catalytic activity, increased surface area, improved thermal stability, and higher mechanical strength compared to the individual components. The synthesis of MgO-ZrO₂ nanomaterials can be achieved using various methods such as co-precipitation [18,19,30], sol–gel [31], hydrothermal synthesis [32,33], and combustion synthesis [33]. These methods offer control over the composition, particle size, and morphology of the resulting nanomaterials, allowing for tailoring of their properties for specific applications. Overall, MgO-ZrO₂ nanomaterials have shown great promise in various fields, and their unique properties make them attractive for further research and development. The synthesis method can significantly influence the properties and performance of the resulting nanomaterials. Thus, the choice of the synthesis method is critical in obtaining MgO-ZrO₂ nanomaterials with desirable properties for photocatalytic applications. The MgO-ZrO₂ heterostructure has been a topic of interest for researchers due to its unique properties and potential applications. Various analytical techniques have been employed to extensively characterize this heterostructure. X-ray photoelectron spectroscopy (XPS) was used to investigate the surface chemistry and revealed the presence of Mg and Zr elements in their respective oxidation states. XPS has proven to be a powerful tool for analyzing

the surface chemistry of materials due to its high sensitivity and ability to detect trace elements. Scanning electron microscopy (SEM) was utilized to analyze the surface morphology of the MgO-ZrO₂ heterostructure. SEM is a valuable technique for investigating the topography and texture of surfaces at high magnifications, providing a clear picture of the material's morphology. UV-spectroscopy was employed to evaluate the optical properties of the heterostructure. UV-spectroscopy is a non-destructive technique that allows for the characterization of the optical properties of materials, including the absorption and transmission of light. The results of UV-spectroscopy provided insight into the bandgap and the absorption properties of the MgO-ZrO₂ heterostructure. X-ray diffraction (XRD) was performed to assess the crystallinity of the materials. XRD is a powerful tool for the structural characterization of materials, providing information on crystal structure, phase purity, and crystallite size. The results of XRD analysis provided information on the crystal structure of the MgO-ZrO₂ heterostructure. Heterostructures are semiconductor structures with a unique natural structure. A heterostructure is a crucial structure composed of two distinct semiconductor materials mixed together [34]. A common method for assessing the nanomechanical characteristics of nanomaterials [35,36] and thin films [37–39] is nanoindentation. Because of its high sensitivity and excellent resolution, this approach makes it easy to determine properties such as nano-hardness, elastic modulus, and elastic/plastic deformation behavior. A diamond tip is used to apply pressure on the specimen until a predefined maximum load or depth is achieved, at which point the force is released. Both the weight placed on the indenter and the indenter's displacement are continually measured and recorded during the test.

Moreover, nanoindentation testing was carried out to assess the mechanical strength and hardness of the MgO-ZrO₂ heterostructure. Nanoindentation testing is a non-destructive technique used to evaluate the hardness of materials, providing an indication of the material's resistance to deformation under an applied load. The I-V characteristics of the heterostructure were examined to investigate its electrical properties. The results of the I-V characteristics revealed the electrical behavior of the MgO-ZrO₂ heterostructure. These various analytical techniques provide comprehensive insights into the structural, morphological, optical, mechanical, and electrical properties of the MgO-ZrO₂ heterostructure. This information is useful for understanding the properties and potential applications of the MgO-ZrO₂ heterostructure in various fields such as electronics, optics, and energy storage.

2. Materials and Methods

Sample Preparation

Nanowires were synthesized on a silicon (n-type) (100) substrate measuring 11 cm in length using chemical vapor deposition. The surfaces were cleaned with acetone and ethanol using ultrasonic cleaning for 20 min to remove any kind of contaminants from the surface, followed by rinsing with deionized water and air drying. A 2 nm thick gold layer was deposited on the silicon substrate using SEM grid sputtering as a catalyst in the nanowires growth process, helping to initiate and control the nucleation of the nanowires. The gold-coated silicon substrate was placed 10 cm away from 99.99% Mg₃N₂ powder in a quartz glass boat. The furnace was heated to 950 °C at a rate of 10 °C per minute for 60 min under a reaction pressure of argon flow 100 SCCM, and oxygen with a flow of 160 SCCM was introduced for 10 min after the furnace reached the highest temperature; then, it was cooled naturally, enabling the formation of MgO nanowires, as shown in Figure 1b. In the second step, the substrate coated with the MgO nanowires was placed in the quartz tube using ZrCl₄ as a precursor at 800 °C for 60 min, with a nitrogen flow of 100 SCCM and an oxygen flow of 6 SCCM, as shown in Figure 1c. Finally, a heterostructure was created by covering the MgO nanowires with ZrO₂. The MgO nanowires serve as the core while

the ZrO_2 forms the protective shell, resulting in enhanced material properties. Figure 1a shows the schematic of the CVD and deposition process of the MgO-ZrO_2 core-shell heterostructure. The growth conditions for the heterostructures are shown in Table 1.

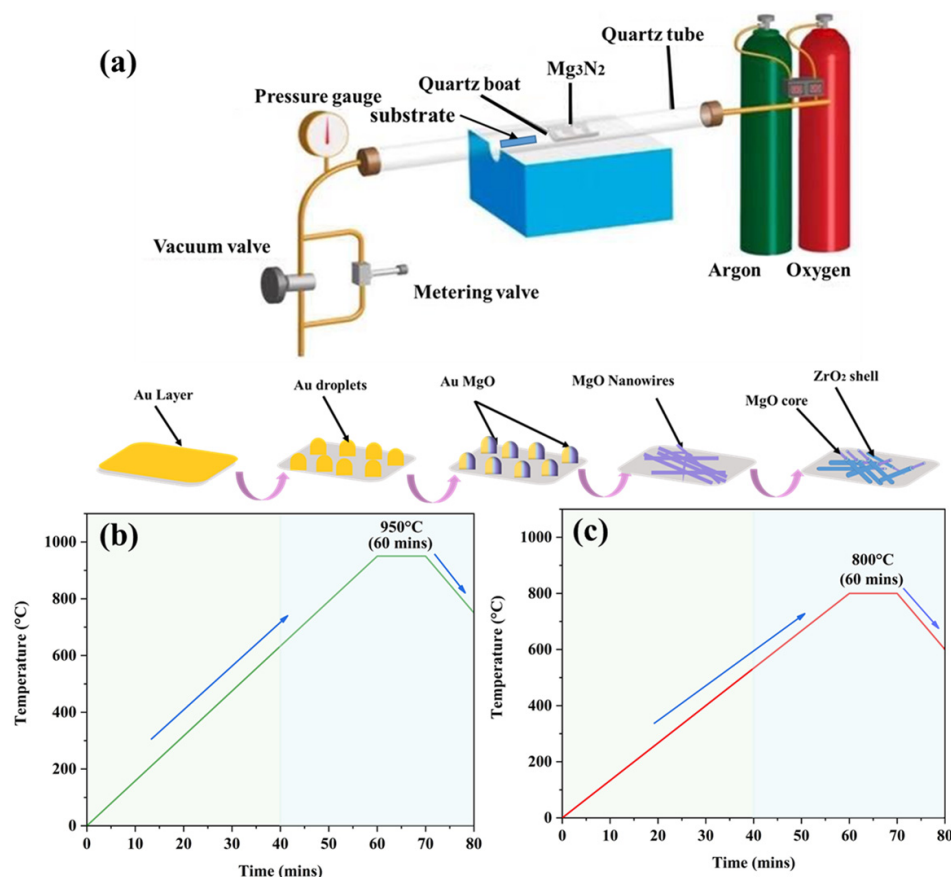


Figure 1. (a) Schematic process of the MgO-ZrO_2 core-shell heterostructure. Progression of deposition, where temperature is set in relation to time (b) for MgO (c) for MgO-ZrO_2 .

Table 1. MgO/ZrO_2 heterostructure growth conditions.

Parameters	Nanowire Growth
Substrate	Si (n-type) (100)
Precursor	Mg_3N_2 , ZrCl_4
Gas Flow	Argon, Nitrogen, Oxygen
Growth Temperature	800 °C, 950 °C
Growth rate	10 °C/min
Growth Pressure	160SCCM, 100SCCM, 6SCCM

The condensation process can be regulated by adjusting the substrate temperature. At elevated substrate temperatures, the thermal energy and surface mobility of adsorbed molecules increase, promoting improved film diffusion and annealing. However, excessive heat can cause desorption and evaporation of the deposited layer. By carefully selecting the type of gas, growth temperature, catalyst material, and substrate position, various structural configurations of MgO/ZrO_2 heterostructures can be achieved. A summary of these possibilities is provided in Table 2.

Table 2. Survey of MgO and heterostructures by chemical vapor deposition.

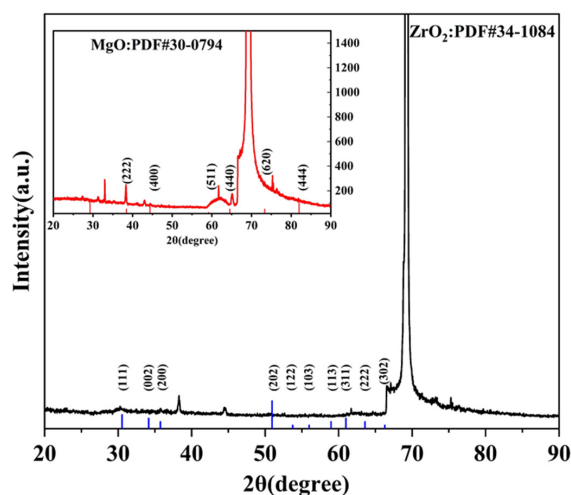
Precursor Used	Substrate	Carrier Gas	Time	Nanostructure	Ref
ZnO/MgO (800 °C)	c-Al ₂ O ₃	O ₂	30 min	NWs	[40]
Mg ₃ N ₂ (650 °C)	Si	N ₂ + O ₂	60 min	NWs	[41]
MgB ₄ (700 °C)	Si	Ar	2 h	NWs	[42]
Mg ₃ N ₂ (650 °C, 800 °C)	Al	N ₂ + O ₂	2 h	Nanobelts	[43]
Mg(thd) ₂ (150–400 °C)	Si (100)	Ar + O ₂	10 min	Films	[44]
Zn (500 °C)	Si	Ar + O ₂	60 min	NWs	[45]
MgB ₂ (900 °C)	Si	Ar + O ₂	8 min	NWs	[46]

3. Results and Discussion

3.1. XRD Analysis and Phase Identification

XRD analysis was performed using a Miniflex-600 diffractometer (Rigaku Americas Corporation, The Woodlands, TX, USA) with Cu K α radiation to examine the crystallinity of MgO-ZrO₂. A previous study revealed that pure MgO exhibited the same crystalline phase in all conditions, corresponding to the reference database (ICDD 30-0794). The XRD patterns exhibited characteristic peaks at 2 θ values of 37.93°, 41.91°, 61.3°, 65.64°, 71.93°, and 80° with preferred orientations in crystallographic planes (222), (400), (511), (440), (620), and (444), respectively, according to Figure 2. Additionally, the ZrO₂ phase (ICDD 34-1084) was identified at 2 θ values of 30.27°, 34.25°, 36.11°, 51.37°, 52.37°, 56.32°, 59.12°, 62.23°, 64.17°, and 66.20°, with planes diffracted in (111), (022), (200), (202), (202), (122), (103), (113), (311), (222), and (302). The research findings show that the ZrO₂ nanowires were fully encapsulated by MgO particles, which form the matrix constituent. The MgO-ZrO₂ binary diagram supports this observation, indicating that the tetragonal phase of ZrO₂ can contain up to 10% MgO, while the cubic phase can have as much as 27% MgO in solid solution. Furthermore, the XRD analysis revealed the presence of small peaks with low intensity, corresponding to the magnesium zirconate phase (MgZrO₃) (ICDD 35-0790). Moreover, the crystallinity of the MgO-ZrO₂ nanocomposite was determined by calculating the ratio of the sum of the areas of the most intense peaks to the total area of all the crystalline and amorphous peaks. The resulting crystallinity value was 99.99%. The size of the nanowires in the MgO-ZrO₂ nanocomposite was estimated using XRD line-broadening methods and Scherrer's equation.

$$d = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

**Figure 2.** XRD of heterostructure MgO-ZrO₂.

The equation used to calculate the nanowires size via XRD line-broadening methods involves the average crystallite size (d), the excess width line of diffraction peaks in radians (β), and the Bragg angle in degrees (θ). The average size of the crystallites was determined to be 130.75 nm and 128.12 nm.

The Williamson–Hall formula was employed to determine the crystalline size of the MgO-ZrO₂ nanocomposite, yielding sizes of 129.70 nm and 126.12 nm, respectively. In this equation, the grain size is denoted by d , the FWHM by β , the wavelength of Cu-K α by λ , and the constant values ϵ and A are both equal to one. The crystal strain is represented by η , while k is another constant with a value ranging between 0.8 and 1.39.

$$\beta \cos \theta = \frac{0.9\lambda}{d} + A \epsilon \sin \theta = \left(\frac{k\lambda}{d} \right) + (\eta \sin \theta) \quad (2)$$

The W-H equation simultaneously describes crystal strain and grain size, both of which are obtained from peak width. The equation defines the crystal strain as $1/\cos\theta$ and crystallite size as $\tan \theta$, where θ is obtained from peak width. The slope η is determined by plotting a linear line between ($\beta \cos\theta$) and ($\eta \sin\theta$), and the intercept of this equation is equal to ($k\lambda/d$). The results of the Scherrer and Williamson–Hall equations are approximately equal, with an R_2 value of 99%.

3.2. Optical Properties

The UV-Vis spectra of pure MgO and MgO-ZrO₂ composites were recorded using an Escalab spectrometer (Thermo Fisher, Waltham, MA, USA) and are presented in Figure 3a. The absorption edge of MgO is observed at 468.3 nm. The MgO-ZrO₂ composites display a more intense absorption in the ultraviolet region at 264.7 nm due to the absorption contribution of MgO-ZrO₂ and the increase in the surface electric charge of the oxides as a result of the introduction of ZrO₂. This is beneficial for improving light utilization. The absorbance did not significantly increase as the MgO-ZrO₂ content increased from 0.25 to 0.85%. The bandgap of the prepared samples can be estimated using a Tauc equation.

$$\alpha h\nu = A(h\nu - E_g)^{\frac{n}{2}} \quad (3)$$

To estimate the bandgap energy of the MgO-ZrO₂ composites, the absorption coefficient α , Planck's constant h , and frequency of UV-visible light ν were used in the formula, where E_g is the gap between the bottom of the conduction band and the top of the valence band, and n is determined by the type of optical transition ($n = 1$ for direct and $n = 4$ for indirect). By plotting $(\alpha h\nu)^2$ versus $h\nu$ and finding the point where $\alpha = 0$ on the energy axis, the direct bandgap of pure MgO nanowires was calculated to be approximately 2.21 eV based on the nature of the observed absorption edge in the UV-visible spectra. This value is usual for MgO and corresponds to the material's capacity to mainly absorb light in the visible range; the presence of oxygen vacancies, other point defects, and possible unintentional impurities can introduce defect levels within the bandgap, leading to a substantial reduction in the effective optical bandgap observed in the absorption spectra. The bandgap energy of the MgO/ZrO₂ nanowires was 2.57 eV. The band gap of the MgO nanowires increased from 2.21 eV to 2.57 eV after depositing ZrO₂, as shown in Figure 3c. This is likely because the ZrO₂ layer acts as a barrier to electron tunneling, which effectively increases the band gap of the MgO nanowires, and the ZrO₂ layer affects the electronic structure of the MgO nanowires. ZrO₂, being a wide-bandgap material itself, can introduce changes at the MgO-ZrO₂ interface that influence electron behavior.

The incorporation of ZrO₂ affects the enhanced optical properties of MgO and mainly the absorptive coefficient for UV light. This change suggests that the substance is better suited to applications such as photocatalysis and UV sensors and shielding, where an interaction between the UV light is desired. The formation of the MgO-ZrO₂ heterostruc-

ture by electronic alteration may enhance charge separation and raise UV absorption for photocatalytic and optoelectronic application and enhance efficiency. As such, the optical characteristic determined herein afford insights into the enhanced performance of the heterostructure in various light-driven applications.

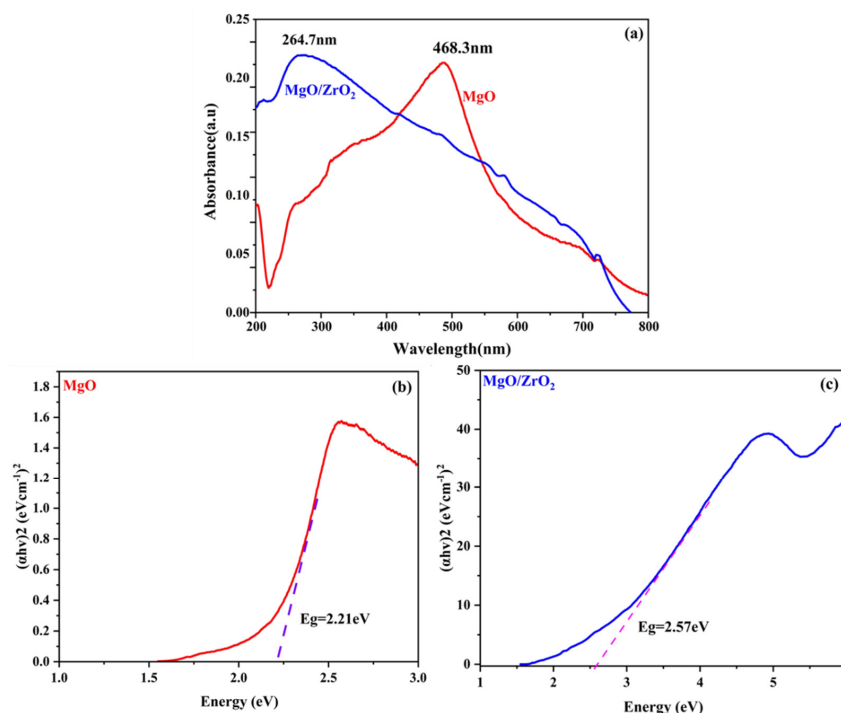


Figure 3. UV-visible spectra: (a) blue line shows the MgO-ZrO₂; red line spectra shows the MgO. (b) Energy band gap of MgO. (c) Energy band gap of MgO-ZrO₂.

3.3. EDS Analysis

It is crucial to understand the surface topology of the MgO-ZrO₂ nanocomposite material since it significantly impacts its catalytic performance. The nanomaterial content of the MgO-ZrO₂ nanocomposite was verified through an EDX spectrum analysis. Figure 4 displays clear peaks from the Zr atoms in the nanowires, which are observed at 2 keV. Additionally, the presence of the optical absorbance band at 1.5 keV reveals the presence of Mg nanowires. The element's composition was briefly studied via EDS analysis and further displayed in the figure.

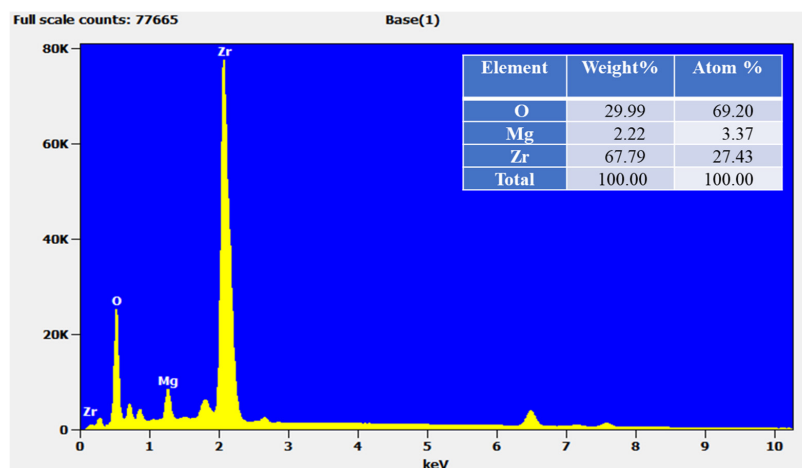


Figure 4. EDS analysis of MgO-ZrO₂.

3.4. Morphological and Compositional Analysis

To characterize the size, shape, and morphologies of the formed MgO nanowires, scanning electron microscopy (EV018) was utilized. The resulting SEM image of the sample is displayed in Figure 5. The morphology of the MgO-ZrO₂ heterostructure material was observed more clearly, and the nanowires of the MgO were observed as shown in Figure 5a. In Figure 5b, MgO shows in the core and how the ZrO₂ nanowires spherically covered the MgO and act as shell around it. Figure 5c represents a systematic image which more clearly shows that the MgO nanowires act as a core while ZrO₂ acts as a shell indicated with a blue color. The ZrO₂ covers the MgO nanowires in a spherical manner, meaning that the ZrO₂ layer evenly coats the MgO rather than being patchy or irregular. Moreover, the MgO and ZrO₂ nanowires reveal circular and poly-dispersed particle shapes, which means the particles vary in size and shape, reflecting a certain level of heterogeneity in their formation. Despite being nanowires, they exhibit poly-dispersity, which refers to a mixture of sizes or shapes rather than to a uniform distribution. The observed nanowires have a diameter between 1.04 μm and 93 μm . This broad range of thickness implies that the nanowires are synthesized under conditions that permit their dimension to be controlled, to a certain extent, by factors such as temperature, time, or concentration of precursor, among others. The fact that the structures are larger in size also indicates that the wires could have perhaps grown longer as a result of the longer reaction time or higher growth rates. These findings provide valuable insights into the physical characteristics of the nanowires, allowing for a more thorough understanding of their potential applications.

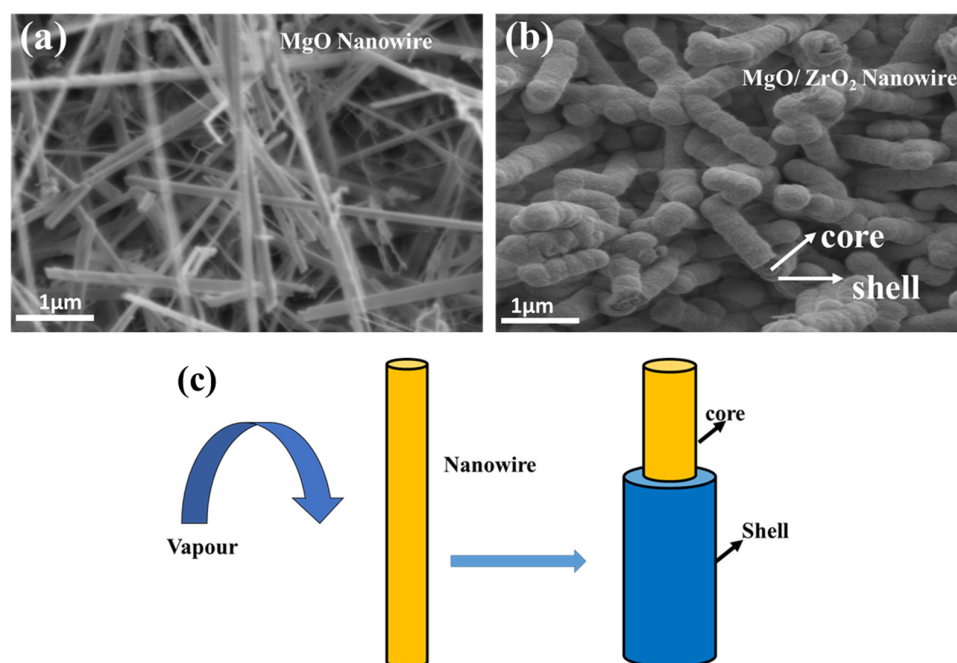


Figure 5. (a) MgO nanowire; (b) MgO and ZrO₂ nanowires show core and shell behavior; (c) systematic diagram illustrating the structural configuration of MgO nanowires and MgO-ZrO₂ core-shell nanowires.

The SEM analysis gives a clear understanding of the size, shape, and morphology of the MgO and MgO-ZrO₂ nanowires. The elemental configuration of MgO as the core layer with ZrO₂ as a layer which surrounds this core layer greatly improves the features of the material, including the surface area, electrical properties, and thermal co-efficient of the material. These morphological features put the heterostructure nanowires in a good

position for use in catalysis, sensors, and optoelectronics, where these characteristics are desirable to enhance performance.

Transmission Electron Microscopes (TEM)

The TEM (JEM-2100F and Tecnai G2 F30) images of the MgO-ZrO₂ solid base provide a clear view of the sponge-like mesoporous network. As shown in Figure 6, the particle size of the nanowires was approximately 10 nm and uniform in size. The particles were linked in a three-dimensional structure to form the mesoporous framework, with pore sizes ranging from 5 to 10 nm. A high-resolution TEM photograph demonstrates the presence of nanocrystals tethered to each other, which formed the mesoporous nanowires.

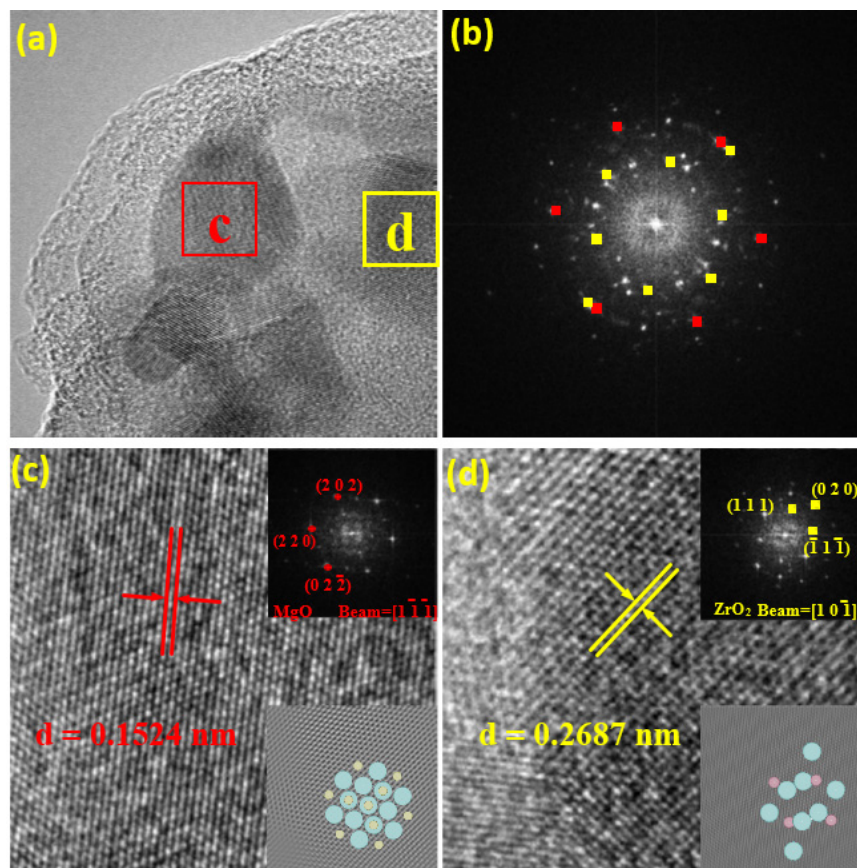


Figure 6. (a) TEM analysis of MgO and ZrO₂ nanowires highlighting the two materials labeled as c and d in the selected portion. (b) The SAED patterns of nanowires. (c,d) The HRTEM images of the nanowires of MgO and ZrO₂ respectively.

The TEM analysis shows two materials labeled as c and d. The TEM image in Figure 6a shows these two materials, and an SAED pattern is shown in Figure 6b,c, which was used to determine the crystal structure of the nanowires. The SAED pattern was divided into two categories and labeled with yellow and red dots, as shown in Figure 6. In Figure 6c, the portion labeled as C (in red color) was recorded perpendicular to the nanowire long axis and can be indexed for the [2 0 2] zone axis of crystalline MgO. The length direction is supposed to be along the [2 2 0] direction, and the HRTEM image in Figure 6c shows the good crystallinity of material c. The interplanar spacings are about 0.1524 nm. In Figure 6d, the portion labeled as D (in yellow color) was recorded perpendicular to the nanowire long axis and can be indexed for the [2 0 2] zone axis of crystalline ZrO₂. The length direction is supposed to be along the [1 1 1] direction, and the HRTEM image in Figure 6d shows the good crystallinity of material d. The interplanar spacings are

about 0.2687 nm, corresponding to the (1 1 1) plane of monoclinic ZrO_2 . Overall, Figure 6 shows that materials c and d have different crystal structures, with material c identified as crystalline MgO with a cubic crystal structure, while material d is identified as crystalline ZrO_2 with a monoclinic crystal structure.

The SAED pattern and HRTEM image were used to determine the crystal structures and interplanar spacings of the materials.

3.5. Chemical State Analysis

To corroborate the results obtained from the XRD analysis, an XPS (Escalab 250Xi spectrometer Thermo Fisher) analysis was conducted in this study. After careful consideration, the sample was studied using high-resolution XPS spectra after the sintering process, as shown in Figure 7. The peaks of the binding energy of Mg 1s and O 1s for MgO are depicted in Figure 7b,c, respectively. On the other hand, Figure 7d exhibits the high-resolution spectra of the zirconia nanowires powders before they were integrated into the matrix (MgO). In this case, two peaks are visible, which are ascribed to the Zr 3d components of spin–orbit splitting (three $d_{5/2}$ and three $d_{3/2}$ orbitals), with binding energies of 181.85 eV and 184.30 eV, respectively. The energy difference between the Zr 3d doublets, ΔE , corresponds to 2.45 eV. The image inserted in Figure 7c presents the high-resolution O 1s peaks in the binding energies at 529.71 eV and 531.85 eV, corresponding to ZrO_2 nanowires and the carbon tape. Overall, the XPS technique identified changes in the chemical states of the analyzed samples' elements (Mg, O, Zr). The MgO- ZrO_2 phase formed during the sintering process led to significant changes in Mg, O, and Zr. According to the literature in Table 1, the bond energies detected at 529.71 eV and 531.85 eV for oxygen represent the formation of the MgO- ZrO_2 phase. Furthermore, Mg and Zr displayed shifts towards lower bond energies, corresponding to a reduction in each element. Binding energy corresponds to Mg 1s and Zr 3d are mentioned in Table 3.

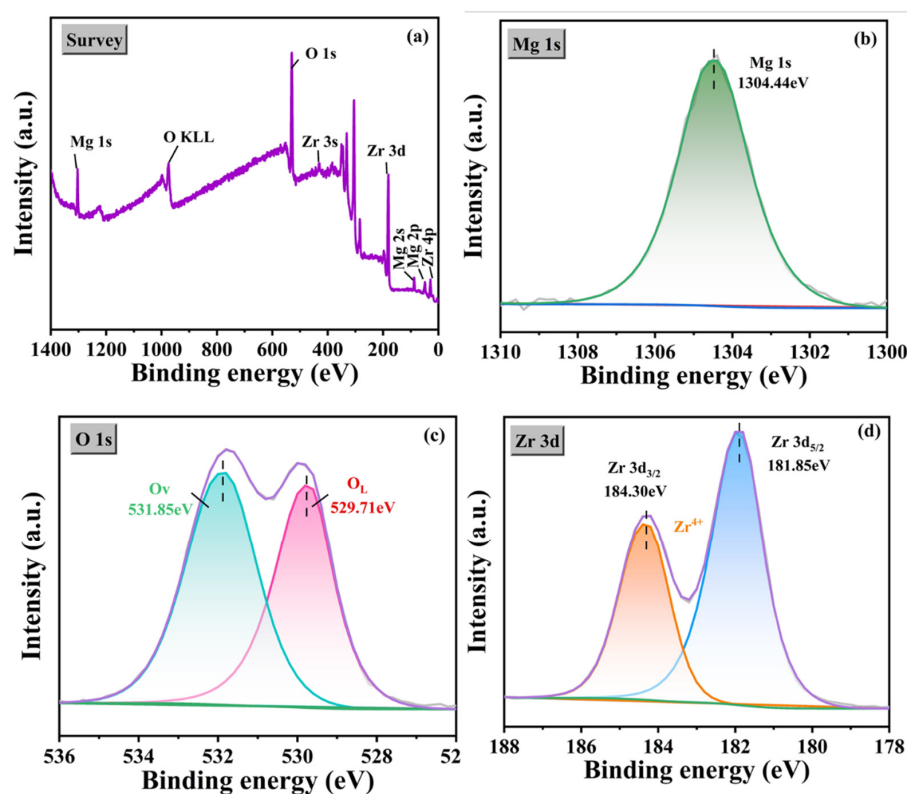


Figure 7. XPS spectra of MgO- ZrO_2 . (a) Combined survey. (b) Mg1s. (c) O1s. (d) Zr3d.

Table 3. Binding energy corresponds to Mg 1s and Zr 3d.

Samples	Mg 1s eV	Zr 3d eV	
	MgO	ZrO ₂ 3d _{3/2}	ZrO ₂ 3d _{5/2}
Binding energy	1304.44 eV	184.30 eV	181.85 eV
From the literature	1303.8 [47], 1303.9 [47,48], 1303.4 [49]	183 [50], 184.9 [51]	181.1 [52], 182 [53]

3.6. Mechanical Properties

The mechanical properties of engineering materials include properties like strength, elasticity, plasticity, ductility, brittleness, stiffness, hardness, and toughness. These properties characterize the behavior of such materials when in use under different forces and conditions. The graph in Figure 8 displays the load–displacement behavior of the MgO and MgO/ZrO₂ nanowires, depicting a complete loading–unloading cycle. The loading phase can be characterized by three distinct stages. Initially, there is a gradual increase in the load applied on the nanowires, followed by a sudden drop in slope resulting in a flat curve. Finally, the third stage shows an increasing load being applied to the nanowires. Figure 8 exhibits the load–depth and hardness–depth that were obtained through the nanoindentation method using the continuous stiffness model. Figure 8a demonstrates a load–depth curve with a load of 100 mN, presenting both the elastic and plastic deformation that transpired when the indenter entered the sample, creating a hardness impression identical to the indenter’s shape. As the indenter was withdrawn, only the elastic part of the displacement returned to its original state, allowing for an elastic solution for modeling the contact process [25,26]. The maximum depth at the peak load ($P_{\max}$) is represented by $h_{\max}$ in Figure 8a, while S shows the initial unloading contact stiffness defined by Sneddon.

$$S = \frac{dP}{dh} = \frac{2\sqrt{A}}{\sqrt{\pi}} E_r = \frac{2\sqrt{24.5}h_c}{\sqrt{\pi}} E_r \quad (4)$$

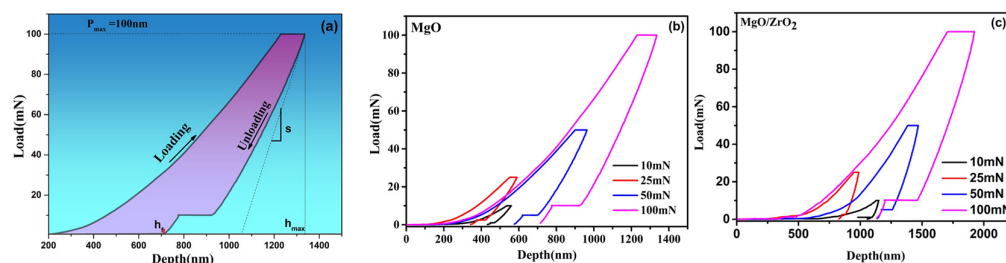


Figure 8. (a) Load–depth curves at 100 mN load. (b) Load–depth curves at different load levels for MgO nanowires. (c) Load–depth curves at different load levels for MgO/ZrO₂ core–shell nanowires.

The final depth of contact (h_f) after the complete unloading process and the contact area (A), which can be calculated using the equation ($A = 24.5h_c^2$), are important parameters to determine the mechanical properties of the sample. The contact depth (h_c) between the indenter and the sample under a load is crucial to calculate the maximum indentation depth. Meanwhile, the reduced elastic modulus (E_r) considers both the elastic deformation occurring in the indenter and the sample. Figure 8a,b shows the load and depth curves for MgO and MgO/ZrO₂. These essential parameters can be determined by using Equations (5) and (6), which, respectively, calculate the contact depth (h_c) and the reduced elastic modulus (E_r).

$$h_c = h_{\max} - \varepsilon \frac{P_{\max}}{S} \quad (5)$$

$$\frac{1}{E_r} = \frac{1 - \sigma^2}{E} + \frac{1 - \sigma_i^2}{E_i} \quad (6)$$

The equation for nanoindentation of samples involves several variables, including a constant, ϵ , which depends on the geometry of the indenter being used. For example, a Berkovich indenter has a value of ϵ equal to 0.75 [25]. Additionally, the equation includes two other key variables, the elastic modulus, E , and Poisson's ratio, σ , both of which are specific to the sample being tested.

To accurately measure the mechanical properties of different samples under varying conditions, micromechanical tests were conducted using nanoindentation. The maximum loads used during these tests ranged from 10 to 100 mN. The elastic modulus and Poisson's ratio for the diamond indenter were also taken into consideration to ensure the accuracy of the results. The elastic modulus for the diamond indenter, E_i , was measured at 1141 GPa while its Poisson's ratio, σ_i , was measured at 0.07 [25]. By conducting tests with different loads and verifying the consistency of the hardness and Young's modulus at various positions, researchers were able to obtain a comprehensive understanding of the mechanical properties of the samples under study.

MgO and MgO/ZrO₂ core-shell nanowires were subjected to nanoindentation testing to ascertain their hardness by using a nanoindenter (iMicro, Nanomechanic, Inc., Oak Ridge, TN, USA) with a diamond-shaped tip. According to the findings, the core (MgO) and shell (ZrO₂) possess dissimilar mechanical properties that cause variation in the hardness of the materials. MgO (magnesium oxide) is a slightly harder material which has hardness of 1.4–3.2 GPa, and it revealed the ability to protrude deformations and the regular structure in the lattice. On the other hand, the ZrO₂ (zirconium dioxide) shell, which is generally categorized as a hard material, reports lower hardness values in MgO/ZrO₂ core shell structure in the range of 0.38–1.2 GPa. This decrease appears to be attributed to the mechanical coupling between the MgO core and ZrO₂ outer layer, alongside structural flaws, such as porosity or stain on the outer layer. Collectively, the core offers solidity while the shell provides extra elements, although with a lower density of mechanical rigidity, as shown in Figure 9a,b. Peak load $P_{\max}$ (mN), maximum depth $h_{\max}$ (nm), contact stiffness S (mN/nm), contact depth h_c (nm), hardness H (GPa), elasticity modulus E (GPa), and reduced modulus E_r (GPa) of sample measured from load–depth, hardness–depth and modulus–depth curves for MgO and MgO/ZrO₂ are mentioned in Tables 4 and 5.

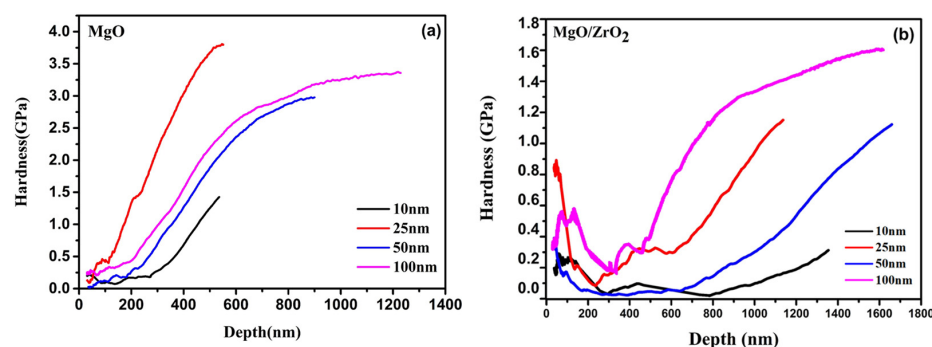


Figure 9. Hardness and depth curves at different load levels: (a) MgO, (b) MgO/ZrO₂.

Table 4. Peak load $P_{\max}$ (mN), maximum depth $h_{\max}$ (nm), contact stiffness S (mN/nm), contact depth h_c (nm), hardness H (GPa), elasticity modulus E (GPa), and reduced modulus E_r (GPa) of sample measured from load–depth, hardness–depth and modulus–depth curves for MgO.

$P_{\max}$ (mN)	$h_{\max}$ (nm)	S (mN/nm)	h_c (nm)	H (GPa)	E (GPa)	E_r (GPa)
9.9858	1596.3	0.18	1554.6	1.402	58.37	59.17
24.978	1293.9	0.23	1212.4	2.826	73.48	73.52
49.96	2134.8	0.14	1867.2	1.758	54.15	54.94
99.89	1281.1	0.66	1167.6	3.284	90.11	88.73

Table 5. Peak load $P_{\max}$ (mN), maximum depth $h_{\max}$ (nm), contact stiffness S (mN/nm), contact depth h_c (nm), hardness H (GPa), elasticity modulus E (GPa), and reduced modulus E_r (GPa) of the sample measured from load–depth, hardness–depth and modulus–depth curves for MgO/ZrO₂.

$P_{\max}$ (mN)	$h_{\max}$ (nm)	S (mN/nm)	h_c (nm)	H (GPa)	E (GPa)	E_r (GPa)
9.988	1650.5	0.22	1616.4	0.38	44.97	46.08
18.757	1991.4	0.43	1958.6	1.00	59.32	59.9
49.96	1616.9	0.23	1454	0.759	60.25	60.97
99.87	1944.6	0.77	1847.3	1.241	68.36	68.49

The researchers found that the hardness of the thin film reduced as the indentation depth grew throughout the first phases of indentation measurement. However, after a certain amount of time, the hardness gradually increased to its actual value. It is vital to remember that when applying an excessive indentation depth, the substrate effect might change how hard the film is. At larger indentation depths, the substrate effect becomes more pronounced. In the nanoindentation of thin films, if the indenter penetrates too deeply, it may start to probe the underlying substrate or support material rather than just the nanowire itself. This can artificially increase or decrease the measured hardness, depending on the relative hardness of the substrate compared to the nanowire. For instance, if the substrate is harder than the nanowire, the hardness value will increase as the depth grows. The hardness of the nanoindentation on nanowires is shown in Figure 9a,b, along with the findings from this study.

3.7. I–V Characteristics of the MgO-ZrO₂ Heterostructure

Figure 10 illustrates the I–V characteristics of the fabricated MgO and MgO-ZrO₂ nanowires. We used a Hall effect instrument for measuring the current voltage values of the nanowires, where the Ag electrode was connected to the positive voltage. A nonlinear rectifying behavior was observed, wherein the reverse current gradually increased with the bias voltage, consistent with findings in previous studies. Additionally, an increase in the applied voltage resulted in a corresponding increase in the current. At 56.5 V, the current flow in the MgO nanowires approached 5.0×10^{-4} ; after depositing ZrO₂ at 58.4 V, the current reached 1.14×10^{-4} .

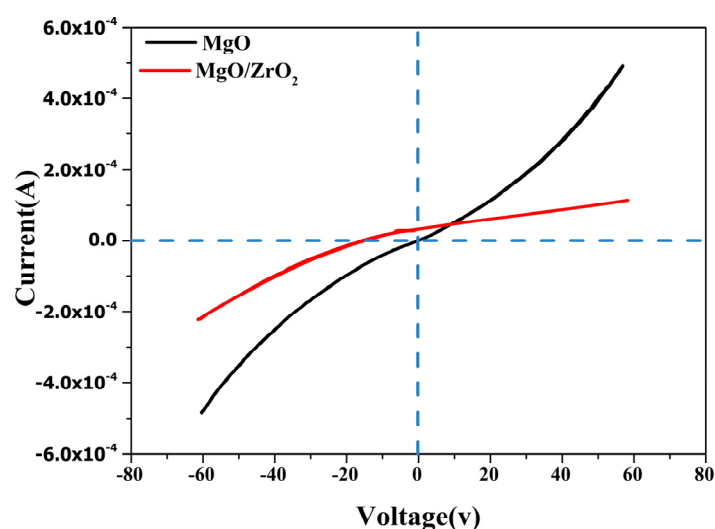


Figure 10. I–V characteristics of the MgO-ZrO₂ heterostructure.

The reason that the current and voltage values of the MgO nanowires are higher may be explained by research which has shown that MgO nanowires exhibit greater electrical

conductivity and lower resistivity compared to their ZrO_2 counterparts due to their unique crystal structure. MgO nanowires have a face-centered cubic structure, while ZrO_2 boasts a monoclinic or tetragonal structure. Moreover, the size and shape of these nanowires can also impact their electrical properties. The I–V characteristics of the studied MgO and MgO– ZrO_2 heterostructure are depicted in Figure 10.

4. Conclusions

In conclusion, we successfully fabricated MgO/ ZrO_2 core–shell nanowire heterostructures using an Au catalyst-assisted CVD technique employing Mg_3N_2 and ZrCl_4 as precursors under oxygen and argon gases. The mechanical properties of the nanowires were tested using nanoindentation, which shows that the hardness range of the MgO nanowires is higher than of the MgO/ ZrO_2 nanowires. The I–V characteristics of the heterostructure were also examined to investigate its electrical properties, showing that the values of the MgO nanowires are greater than the values of the MgO– ZrO_2 nanowires. Research has shown that MgO nanowires exhibit better electrical conductivity and lower resistivity compared to their ZrO_2 counterparts due to their unique crystal structure. The results obtained from these various techniques provide comprehensive insights into the structural, morphological, optical, mechanical, and electrical properties of the MgO– ZrO_2 heterostructure.

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