Preparation and characterizations of ZrO_2 thick films

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Abstract
Zirconium dioxide (ZrO_2), also known as zirconia, is a versatile material with applications in various fields, including ceramics, sensors, and catalysis. Thick films are generally defined as films with thicknesses in the range of a few micrometers to several hundred micrometers.
It is a versatile material with several optical applications due to its excellent optical properties, such as high refractive index and optical transparency. The present research work is focused on preparation of ZrO_2 thick films using standard screen printing method and investigation of electrical and optical properties of prepared ZrO_2 thick films. The electrical characterizations was carried out using electrical static system and half bride circuitry. The optical characterizations were carried out using homemade photoresponses system. The resistivity of prepared ZrO_2 thick films was found to be 6624735Ω.m. In optical characterization it is recorded that as intensity of light source is rise the photocurrent through ZrO_2 thick films is also increased, this results shows ZrO_2 thick films could be used optical devices development.

Keywords: Zirconium dioxide, thick films, electrical static system, photoresponses system, intensity.

1. Introduction

Optical sensors are devices that use light to detect and measure various physical properties. They play a crucial role in a wide range of applications and industries due to their versatility and ability to provide accurate and real-time data. It can be used to detect and monitor environmental factors like light levels, temperature, humidity, and air quality. They are commonly used in weather stations, building automation, and environmental monitoring systems [1]. There have been numerous semiconducting metal oxides sensors present in reported literature acts as potential applications like optical sensors, windows layer, solar cell, battery electrodes and gas sensing devices [1-2]. Zirconium oxide (ZrO_2) is a great candidature due to its low cost and wide band gap with n-type nature. It has high thermal expansion of coefficient, high ionic conductivity, good chemical stability and high resistivity to corrosion. Therefore ZrO_2 has been employed in gas sensing devices, dye sensitized solar cells, fuel cells, and protective layer for optical mirrors [3]. It has wide band gap metal oxide semiconductor with density 5.83 g/cm^3 for monoclinic phase and 6.10 g/cm^3 for tetragonal phase respectively [3, 4]. In accordance with the temperature, zirconia can have a monoclinic, tetragonal, or cubic crystal structure. Hydroxyl groups can be found on zirconia films. At ambient temperature, ZrO_2 forms a monoclinic crystal structure, which changes to tetragonal and cubic at elevated temperature [5]. ZrO_2-based optical waveguides are used in integrated photonics for guiding and modulating light in optical communication devices, photonic circuits, and sensors. The high refractive index of ZrO_2 allows for efficient light confinement and manipulation. ZrO_2-based optical sensors are employed in various fields, including environmental monitoring, biomedicine, and industrial applications. These sensors are sensitive to changes in optical properties (e.g., refractive index or fluorescence) and are used for chemical, biological, and physical measurements. It can be engineered to exhibit photoluminescence in certain applications, making it useful in optoelectronic devices and displays [6, 7]. The main gain of thick film sensors are simple construction principle, small size, good sensitivity and selectivity, low operating temperature, extraordinary stability, virtuous accuracy, easy processing, reproducibility, less expensive and low power consumption [8, 9]. The Monoclinic crystal structure of ZrO_2 is shown in Fig. 1 [10]. ZrO_2 has variety of distinctive properties few general properties of ZrO_2 are tabulated in Table 1.

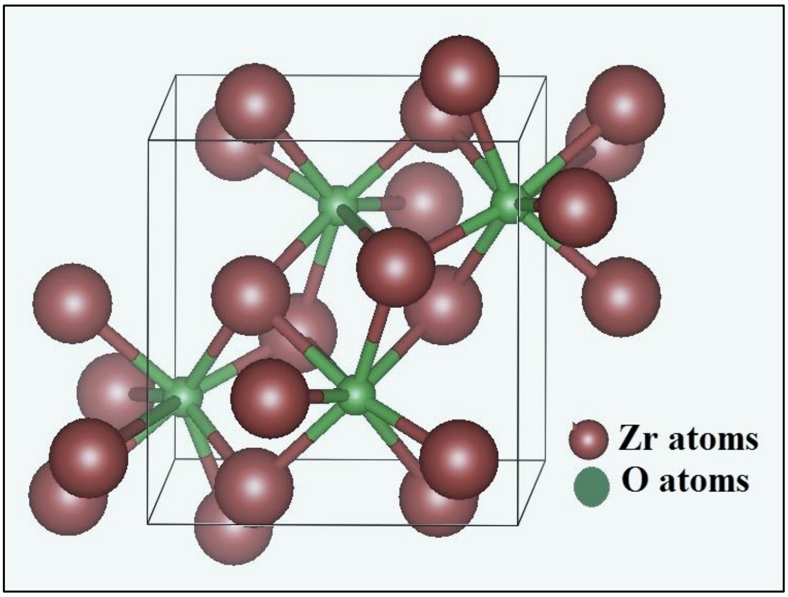

Figure 1: Monoclinic crystal structure of ZrO_2

Table 1: General properties of ZrO_2

Compound Formula	ZrO_2
Molecular Weight	123.218 g/mol
Appearance	White powder
Melting Point	2,715 °C
Boiling Point	4,300 °C
Density	5.68 g/cm^3
Band gap	5.8 eV

The current research present the preparation and characterizations of ZrO_2. In this work ZrO_2 thick films were prepared using standard screen printing method and electrical and optical properties of prepared ZrO_2 thick films were studied.

2. Experimental work

The commercial available ZrO_2 nanopowder is used for preparation thick films. By using standard screen printing method thick films of ZrO_2 were prepared on glass substrate [9, 10]. Initially, all glass substrate cleaned by double distilled water and acetone then dried under IR lamp. By using 70:30 organic (ZrO_2) and inorganic (EC and BCA) material ratio the thixotropic paste was formed for the preparation of thick films of ZrO_2. The paste is forced through a stencil onto a glass substrate. After deposition, the film is dried under IR lamp for 30 minutes at room temperature and annealed in a furnace for 2 hours at 300 °C to sinter the ZrO_2 particles and remove the organic components.

3. Result and Discussion

3.1 Electrical characterization:

The room temperature resistance and resistivity of pure ZrO_2 thick films was investigated using the variation of ambient temperature of across the films in static electrical system. The temperature across the sample was increased in interval of 10 °C and change in resistance of sample is recorded. It has been observed that as the temperature across the film increased the resistance of the film was drop as display in figure 2 [11, 12]. The resistivity of pure ZrO_2 thick films was estimated using Eq. 1.

$$\rho = \left(\frac{R \times b \times t}{l}\right)\Omega - m \qquad (1)$$

Where, ρ = Resistivity of prepared film, R = resistance at normal temperature, b = breadth of film, t = thickness of the film, L = length of the film.

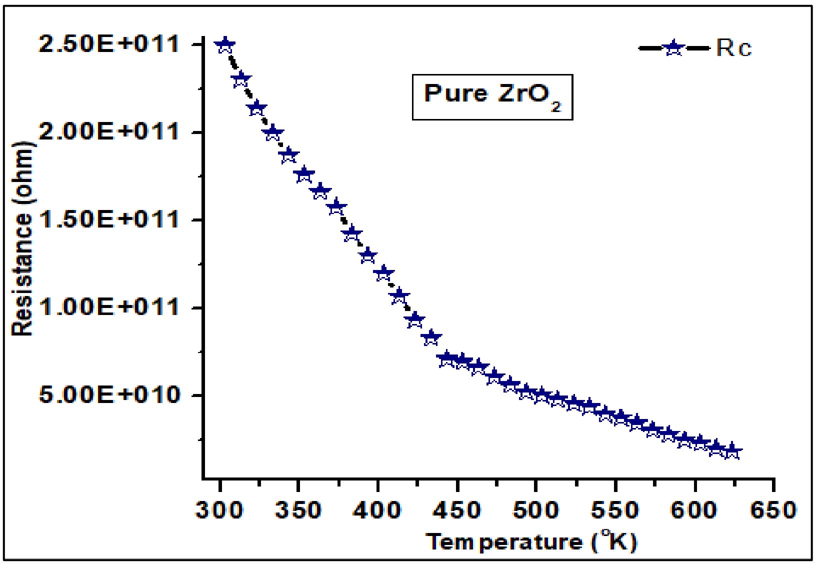

Figure 2: Temperature v/s resistance plot of pure ZrO_2 thick films

The Temperature Coefficient of Resistance (TCR) of films is a measure of how a material's electrical resistance changes with temperature. Thick film resistors are a type of resistor that has a thick, paste-like resistive material, typically a ceramic-based compound, applied to a substrate, such as a ceramic or glass. It is essential in applications where stable resistance values over a range of temperatures are required [12, 13]. The resistivity of pure ZrO_2 thick films was estimated using Eq. 2.

$$TCR = \frac{1}{R_o}\left(\frac{\Delta R}{\Delta T}\right) \; /^{O} C \qquad (2)$$

Where, ΔR = change in resistance between temperature T_1 and T_2, ΔT = temperature difference between T_1 and T_2 and R_o = room temperature resistance of the film.

$$\Delta E = Ae^{-Ea/kBT} \quad \text{eV} \qquad (3)$$

Where, ΔE = Activation energy, T = Temperature in Kelvin and A = Arrhenius prefactor.

The activation energy in the context of electrical and optical properties of materials typically refers to the energy required for charge carriers or photons to transition from one energy level to another within the material. It is particularly important for understanding the behavior of semiconductors and the absorption of light in various materials. By employing Eq. 3 the activation energy is calculated for pure ZrO_2 thick films at lower and higher temperature regions [14, 15]. The Arrhenius principle was used to compute activation energy plot as illustrated in figure 3. The obtained electrical outcomes of pure ZrO_2 thick films are tabulated in Table 1.

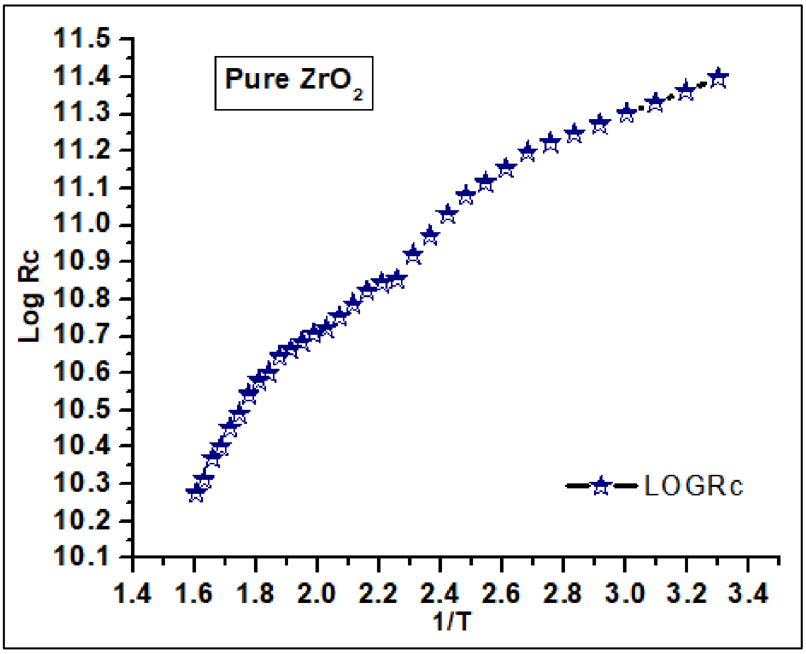

Figure 3: Arrhenius plot of pure ZrO_2 thick films

Table 2: Electrical outcomes of pure ZrO_2 thick films

Sample	Thickness (μm)	Resistivity (Ω.m)	Conductivity (Ω^{-1}.m)	TCR (/°C)	Activation Energy (eV)	
					HTR	LTR
ZrO_2	53	6624735	1.5095E-07	-0.0026	0.111827	0.063845

In optical studies, activation energy is often related to the energy difference between electron energy levels in atoms, molecules, or crystals. When photons are absorbed, they impart energy to electrons, causing them to jump from lower energy levels to higher ones. The energy required for this transition is associated with the material's bandgap energy.

3.2 Optical characterization:

3.2 Photoresponse study of pure ZrO_2 thick films:

A photoresponse study of pure ZrO_2 thick films involves investigating how the electrical properties of the material change when exposed to light, particularly in the context of its semiconductor or insulator behavior. Photoresponse study of pure ZrO2 thick films can provide valuable insights into the material's behavior under light exposure and its suitability for various optoelectronic applications. Additionally, the results may contribute to the understanding of its electronic properties and its potential in emerging technologies. In his investigation, author explored the light-dependent conductance swings of developed thick films as an optical sensor [16, 17].

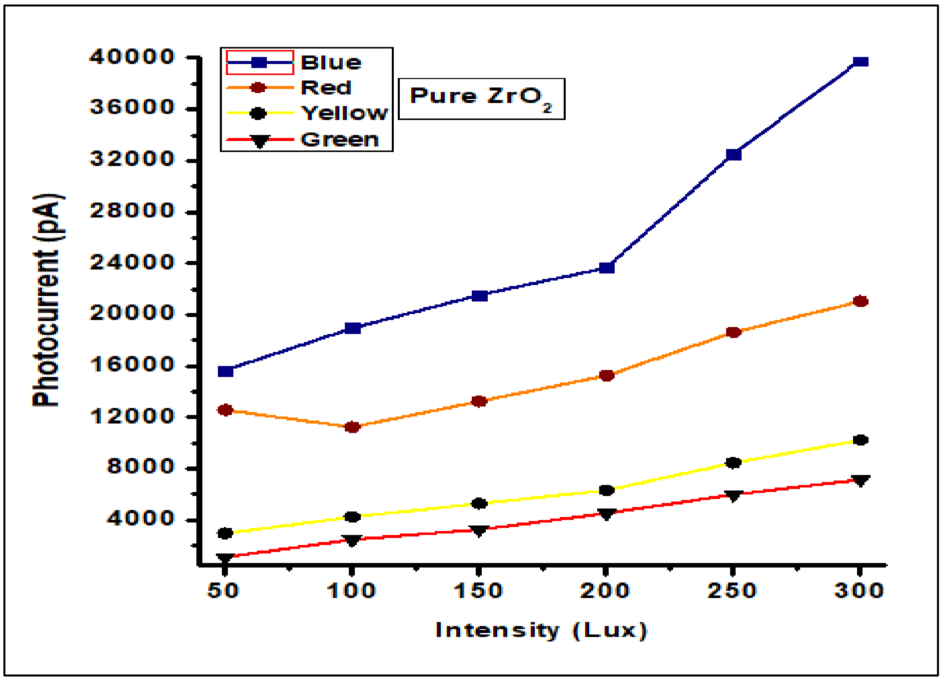

Figure 4: Intensity versus photocurrent plot of ZrO_2 thick film

Using red, yellow, green, and blue color filters and a Photoresponse characterisation system, the light sensing properties of produced thick films were investigated at various wavelengths of 640, 550, 510, and 450 nm. The +5VDC fixed power supply, a color filter holder, a sample holder, a 5 Watt light source, and a distance adjustment function make up the photoresponses characterization static system. By rotating the light source from the thick film in this setup, the change in current was examined in response to the light intensity. Using a Lux meter, a change in film current with an alteration in intensity over a distance ranging from 2 cm to 40 cm was detected. The photocurrent through the film was measured by fixing the location of the filter close to the ZrO_2 film and moving the filter, or varying the distance from the light source, to a different point, and measuring the photocurrent response [16]. Figure 4 shows how pure ZrO_2 thick films are responding right now to various color filters that are being applied. It has been noted from Fig. 4 that the photocurrent of the films reduced as distance was increased. The developed pure ZrO_2 thick film has an optical or light response, and this plot demonstrates that it can be applied to the creation of optical devices.

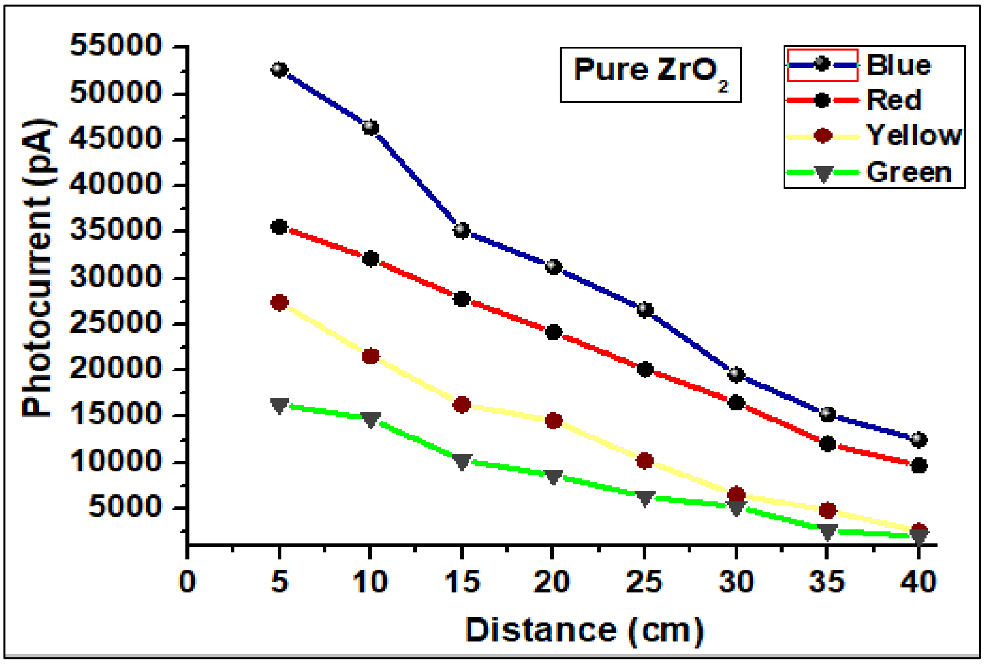

Figure 5: Distance versus photocurrent plot of ZrO_2 thick film

A distance versus photocurrent plot for a ZrO_2 thick film can be used to visualize the response of the material to incident light as a function of the distance between the light source and the film's surface. Such a plot is especially relevant in optoelectronic studies and device characterization. From figure 5 it is observed that, as decreasing in photocurrent with increasing distance from the light source [18, 19]. This indicates a strong and direct dependence of photocurrent on the light intensity. The distance versus photocurrent plot can provide valuable insights into the ZrO_2 thick film's light-sensitive behavior. It helps in characterizing the material's response to incident light and can be informative for designing optoelectronic devices like photodetectors or sensors based on ZrO_2 thick films [19, 20].

4. Conclusion:

ZrO_2 thick films were successfully prepared using standard screen printing method and electrical and optical properties of prepared ZrO_2 thick films were studied. The prepared ZrO_2 thick films has 6624735 Ω.m, -0.00261 /°C and 0.111827 eV resistivity, TCR and activation energy respectively. In optical study, the blue filter shows maximum current variation compared to other selected filter. It is also found that as intensity of light is increased the photocurrent through ZrO_2 thick films is also increased, this results shows ZrO_2 thick films could be used optical devices development. The optical properties and versatility of ZrO_2 make it a valuable material in the field of optics, enabling a wide range of applications across multiple industries, including telecommunications, aerospace, healthcare, and scientific research.

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