SYNTHESIS AND CHARACTERIZATION OF OXIDE NANOPARTICLES FOR ITS APPLICATION IN MEMRISTIVE DEVICES

Minor Research Project
Under
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Submitted by

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CERTIFICATE

This is to certify that the Research Article entitled *"Synthesis and characterization of oxide nanoparticles for its application in memristive devices"* being submitted by Abhijit Paul, Siddhartha Das, Deepjyoti Kalita and Barnali Karmakar for the fulfillment of Minor Research Project under Satya Nath Das Research Fund (SNDRF), B. H. College, Howly is an original piece of investigation and record of research is carried out by them under my supervision.

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DECLARATION

We hearby declare that the research work titled *"Synthesis and Characterization of Oxide Nanoparticles for its Application in Memristive Devices"* , has been carried out by us, submitted for the fulfillment of Students Minor Research Project under Satya Nath Das Research Fund (SNDRF). The Research Article is an authentic piece of work carried out under the supervision of Jyoti Prasad Roy Choudhury, Asst. Professor, Department of Physics, B.H. College Howly.

We also declare that the article has not been submitted to any other academic institution for the award of any degree or diploma.

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CONTENTS

SYNTHESIS AND CHARACTERIZATION OF OXIDE NANOPARTICLES FOR ITS APPLICATION IN MEMRISTIVE DEVICES

Abstract:

The last few decades have been revolutionary for semiconductor processing and applications due to their extraordinary optical and electronic properties. In this article, ZnO and PbO nanoparticles are prepared by chemical bath deposition technique. The structural morphologies of the prepared samples are verified with the help of XRD analysis and HRTEM technique. XRD analysis shows the hexagonal structure of ZnO nanoparticles and Orthorhombic structure of PbO nanoparticles. The average particle size calculated from the XRD data are found be 28.934 nm for ZnO nanomaterials and 63.11 nm for PbO nanomaterials. HRTEM imaging technique further verifies the structural characterization of the prepared samples. The electrical study is done with the help of an electrometer. The I-V measurement curve shows a zero crossing pinched hysteresis loop. The presence of such a pinched hysteresis loop suggests the existence of memristive properties in the semiconductor nanomaterials. The primary goal of this research project is to synthesize ZnO and PbO semiconductor nanomaterials and analyze their various physical and electrical properties for their applications in memristive devices.

Keywords: ZnO, PbO nanoparticles, quantum confinement, optoelectronics, memristor, hysteresis loop.

Chapter 1

Introduction

In the last few decades, semiconducting metal oxide nanoparticles have been a great course of study because of their huge range of optical, electrical, mechanical and chemical properties. In this case oxide nanoparticles have emerged as attractive building blocks due to their extraordinary properties, adjustable electrical conductivity, high surface area and quantum confinement effects. Zinc Oxide with chemical formula ZnO is an inorganic compound which is usually a white powder and is insoluble in water. ZnO is a wide band gap semiconductor with a band gap of 3.37 eV and high exciton binding energy of 60 MeV. It is also one of the most usable and easily accessible compounds for biomedical applications due to its eco-friendly properties [1]. Similar to Zinc Oxide, Lead Oxide (PbO) is also a semiconductor nanoparticle with a band gap of 2.59 eV with various applications. Prof. Dr. Amra Bratovcic, university of Tuzla had studied about numerous uses of PbO nanoparticles like glasses, stabilizers, storage batteries etc. Lead Oxide nanocomposites have also been a very trending research direction in the development of anti-cancer and anti-microbial drugs [2].

In recent years, researchers have shown a great attention towards the memristive properties of various nanocomposites. The memristor was originally first reported by Leon Chua in 1970. It is a two terminal electrical component relating electric charge and magnetic flux linkage [3]. Memristor is referred as the fourth fundamental electrical component along with resistor, capacitor and inductor [4]. Chua's theory was not proved for almost four decades. In 2008, Hewlett-Packard (HP) labs found the first experimental evidence for Chua's memristor based on an analysis of a thin film of Titanium di-Oxide (TiO_2) [5].

The Memory Resistor or Memristor property is based on its capability to remember the amount of current that had previously flowed through it i.e. its present resistance depends on how much current has flowed through it before [6]. Usually when the circuit is off, memristive devices can remember the most recent resistance until the circuit is turned on again [6]. Recombination of either ions or electrons of nanomaterials lead to a stage which suggests the capacitive behavior that exists in a memristor. The memcapacitive state is possibly the initial state of a memristor [7]. Memcapacitor basically has both memory and capacitive ability. By studying the V-I characteristics curve of the memcapacitor, pinched hysteresis loop is observed with advantage of being capable of storing energy [8]. Several reports by many researchers were published based on the memristive behavior in various nanocomposites but mostly the Oxide nanocomposites are preferred [9]. Ashish Kumar *et al* in 2012 fabricated ZnO based memristor device with platinum electrode [10]. The most recent study on memristive devices was done by Prof. B.K. Barnes *et al* in 2018 where they observed a hysteresis loop at a voltage range between +20V and -20V using ZnO based memristor [11]. Though many oxide nanomaterials individually show promising electronic properties, further works in manipulating the oxide materials is desirable. The core/shell type oxide nanocomposites may play a key role in modulation and further development of the Oxide nanomaterials to enhance their memristor characteristics [12].

Chapter 2

Optoelectronic Properties of ZnO and PbO nanomaterials

Zinc Oxide (ZnO) and Lead Oxide (PbO) are two of the most interesting semiconducting metal oxides with different optoelectronic properties. Let us now discuss the various optoelectronic properties of ZnO and PbO separately:

2.1. Zinc Oxide (ZnO):

Zinc Oxide is one of the most usable and easily accessible semiconducting materials with wide band gap energy of 3.37 eV at normal temperature [13]. Because of this wide band gap energy, ZnO shows interesting optical properties in the Ultraviolet range [14]. This is also why these are mostly used in optoelectronic devices. Some of the key optoelectronic properties of ZnO are discussed below:

A. Transparent Behavior: Zinc Oxide (ZnO) is transparent in visible region, making it useful in the applications requiring light transmission such as solar cells and displays [15].

B. Photoluminescence: ZnO, when excited by photons or electrical current emits light through photoluminescence in the visible and Ultraviolet (UV) region. This property can be used in LEDs or UV light sources [16].

C. Piezoelectricity: ZnO can generate an electrical charge when mechanically stressed and vice-versa, his property is known as piezoelectric property. This property can be used in the application of sensors and detectors [17].

D. Electronic Mobility: ZnO shows exceptionally high electron mobility, this property maybe helpful in the construction of various high speed electronic devices [18].

2.2. Lead Oxide (PbO):

Lead Oxide (PbO) is also a semiconducting metal oxide similar to ZnO but with relatively narrower band gap compared to ZnO. It is generally a p-type semiconductor material. It has much lesser application in optoelectronics compared to Zinc Oxide because of its toxic nature. Lead Oxide is primarily used in ceramics and glass manufacturing [19]. Some of the key optoelectronic properties exhibited by PbO are discussed below:

A. Narrower band gap: Lead Oxide (PbO) has a narrower band gap energy typically around 2.59 eV making it more suitable for absorbing light as compared to Zinc Oxide [20].

B. Restricted transparency: Lead Oxide (PbO) is not transparent in visible range restricting its use in transparent devices.

C. Electrical conductivity: PbO can be doped to exhibit either n-type or p-type conductivity.

D. Optical behavior: PbO exhibits non-linear optical properties which can be used for optical signal processing.

Chapter 3

Experiments

3.1. Synthesis:

All the necessary chemicals required for performing our experiments were all purchased from Merck. The chemicals that were used are Zinc Acetate Dehydrate *[*$Zn(CH_3COO)_2\ 2H_2O$*]*, Lead Acetate[$Pb(CH_3COO)_2 4H_2O$**],** NaOH and Polyvinyl Pyrrolidone(PVP).A schematic for synthesis of ZnO and PbO are shown in Fig. 1.All our required solutions are prepared in 3% Polyvinyl Pyrrolidone(PVP) solution which is prepared by mixing PVP in distilled water under continuous stirring.

3.1.1. Preparation of ZnO:

First, Zinc Acetate Dehydrate *[*$Zn(CH_3COO)_2\ 2H_2O$*]*of 0.3M concentration is mixed and stirred in 50mL of PVP solution at room temperature. After the complete dispersion of the mixture in PVP solution, 50mL of 1M NaOH solution is added dropwise at 90°C with continuous stirring. After about 45-50 minutes, the solution turns milky white indicating the formation of ZnO nanoparticles. The prepared solution is then filtered and dried in hot air oven.

3.1.2. Preparation of PbO:

The previous method used in the preparation of ZnO nanoparticles is repeated in the similar manner for the preparation of Lead Oxide (PbO). Here Lead Acetate [$Pb(CH_3COO)_2 4H_2O$] of 0.3M concentration is mixed and stirred in 50mL of PVP solution at normal temperature. After that, 50mL of 1M NaOH solution is added dropwise and stirred vigorously at 90°C. After about 60-70 minutes, the solution slowly turns pale yellow indicating the formation of PbO nanoparticles. The prepared solution is then filtered and dried in hot air oven.

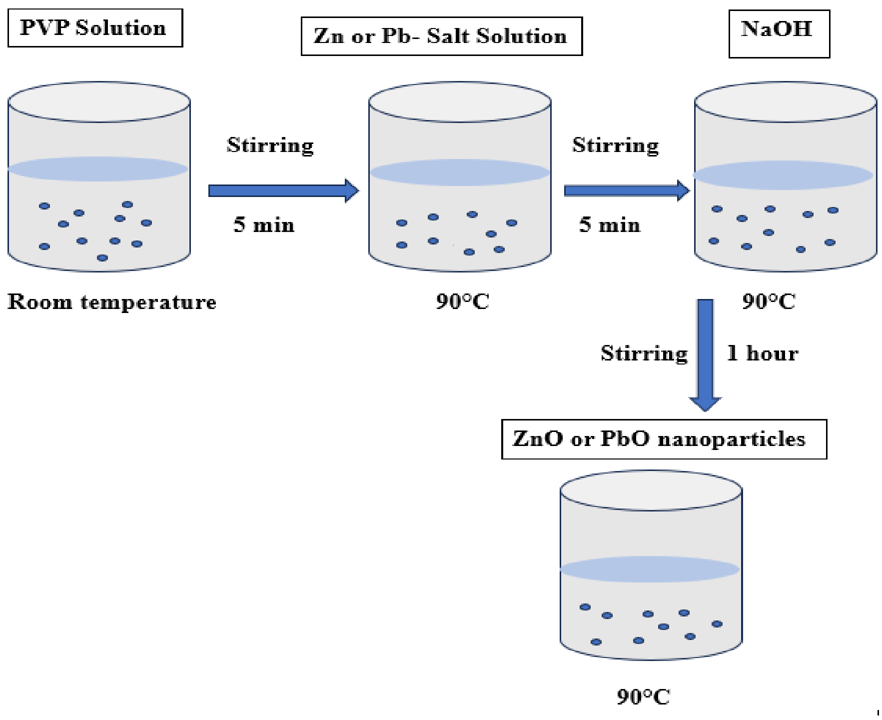


Fig: 1: Schematic diagram of preparation process of ZnO and PbO nanoparticles

3.2. Characterization:

Zinc Oxide (ZnO) nanoparticles provide versatility in the field of optoelectronics, while Lead Oxide (PbO) nanoparticles also offers interesting optoelectronic potential but with a limited use because of toxicity concerns. There are several characterization techniques such as XRD, Transmission Electron Microscopy (TEM) and spectroscopy which help in analyzing their crystal structure, size, composition, elemental and optical properties. The nanostructure of these semiconducting nano materials were mainly characterized by X-Ray Diffraction(XRD) technique(ULTIMA IV X-Ray diffractometer). Further studies on the crystal configuration of all our prepared samples were done with the help of HRTEM(JEOL, model: 2100F)**.** Using Keithley 2450 electrometer, the electrical properties of our prepared sampleswere studied by taking a voltage range between -10 V and +10 V. Memristor devices can be now fabricated in planar type electrode film geometry using the prepared samples to study their characteristics.

Chapter 4

Results and discussions

4.1. XRD Analysis:

The crystral structure of our prepared Zinc Oxide(ZnO) and Lead Oxide(PbO) semiconducting nanomaterials were analysed by usingX-ray powder diffraction technique. X-Ray Diffraction(XRD) technique is a very powerful technique used in the study and analysis of the atomic and molecular structure of crystals. Principle of XRD is based on the fact that when X-Rays are directed at a crystal, they are diffracted by the regularly arranged atoms in the lattice [21]. By analyzing the resulting diffraction pattern, we can determine various properties like arrangement of atoms within the materials, lattice parameters, crystallographic phases, crystal size, defects, orientation etc. The Bragg equation which relates the angles of incidence and diffraction to the interplanar spacing of crystals is the fundamental to XRD analysis [22].

The Bragg Equation,

$$2d\sin\theta = n\lambda \qquad (1)$$

Where **λ** is the wavelength of XRD, n is the order of diffraction and d is the interplanar spacing.

After analyzing the prepared samples, the following XRD patterns have been found **[**Fig. 2(a)for ZnO and Fig. 2(b)for PbO]. The XRD patterns suggest a hexagonal structure of ZnO [23] and orthorhombic for that of PbO [24] (According to the data collected from Joint Committee on Powder Diffraction Standards, JCPDS). The XRD spectrum confirms the presence of hexagonal diffraction peaks at (100), (002), (101), (110) corresponding to ZnO nanomaterials and orthorhombic diffraction peaks at (002), (201), (003), (131) corresponding to PbO nanomaterials. The value of lattice constants are calculated using The Bragg equation and other formulas and are found to be a = 3.238Å, c = 5.2Å for ZnO nanoparticles and for PbO nanoparticles the corresponding values are a = 5.49Å, b = 4.73Å and c = 5.86Å.

The crystal sizes of all our prepared samples are determined using the Debye Scherrer's formula given by the equation [25] –

$$D = \frac{K\lambda}{\beta cos\theta} \quad (2)$$

Here, K is the shape factor (0.9), λ is the X-ray wavelength, β is the Fullwidth at Half Maximum(FWHM) and θ is the Bragg's diffraction angle.

We have determined the particle sizes of ZnO and PbO nanoparticles using the above formula, which are found to be 28.93 nm for ZnO and 63.11 nm for PbO. The datas collected from the XRD analysis are provided in the following page-

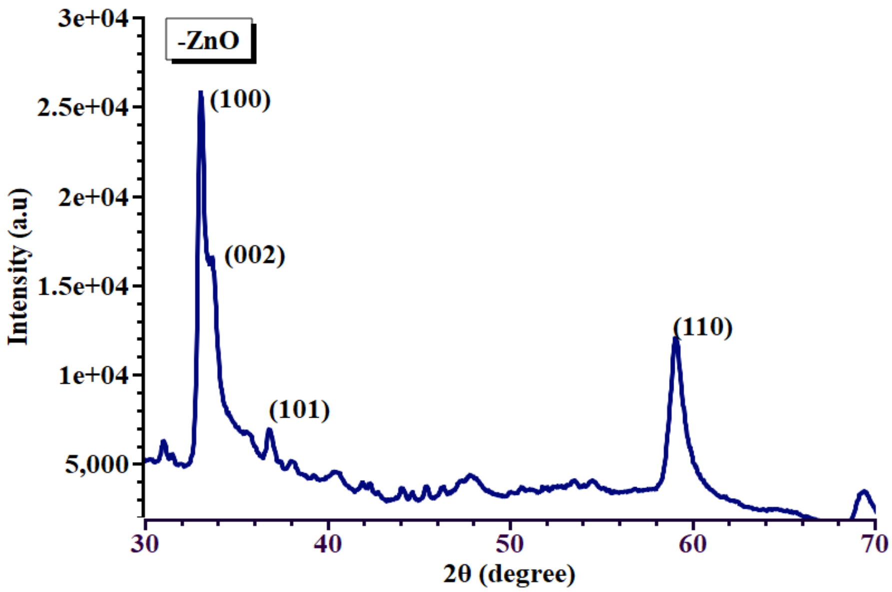


Fig. 2(a). XRD pattern observed for prepared ZnO

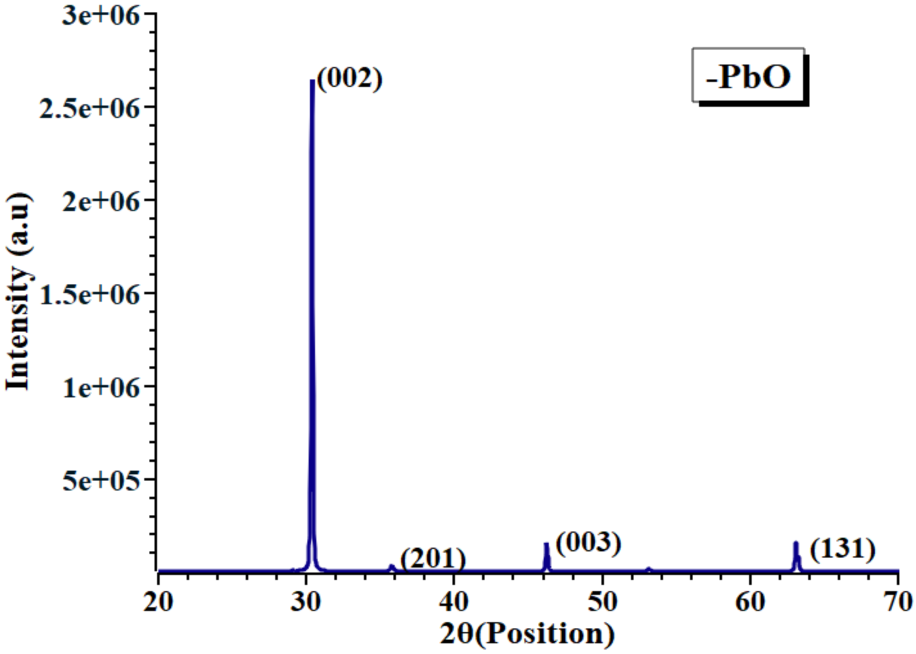


Fig. 2(b). XRD pattern observed for prepared PbO

2θ	FWHM	Size	Avg size
6.23	0.434	17.92019	28.93459
15.63	0.255	30.7399	
18.84	0.204	38.58835	
25.85	0.307	25.95356	
31.03	0.204	39.50763	
33	0.307	26.38243	
35.79	0.358	22.79518	
38.1	0.409	20.08757	
39.29	0.204	40.42073	
40.36	0.307	26.95038	

(a) Table for ZnO

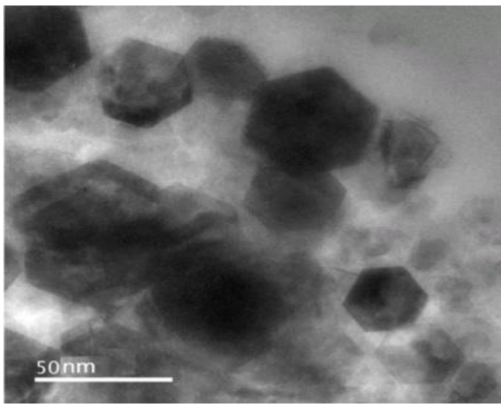
2 θ	FWHM	Size	Average Size
7.159	0.1023	76.06109	63.11082699
15.1097	0.1023	76.57744	
17.7069	0.1279	61.45046	
22.1745	0.1023	77.35653	
26.739	0.1535	52.00127	
27.3836	0.1023	78.13301	
28.6524	0.1535	52.21576	
29.1286	0.1535	52.27168	
30.3757	0.1535	52.42301	
31.9026	0.1535	52.61801	

(b) Table for PbO

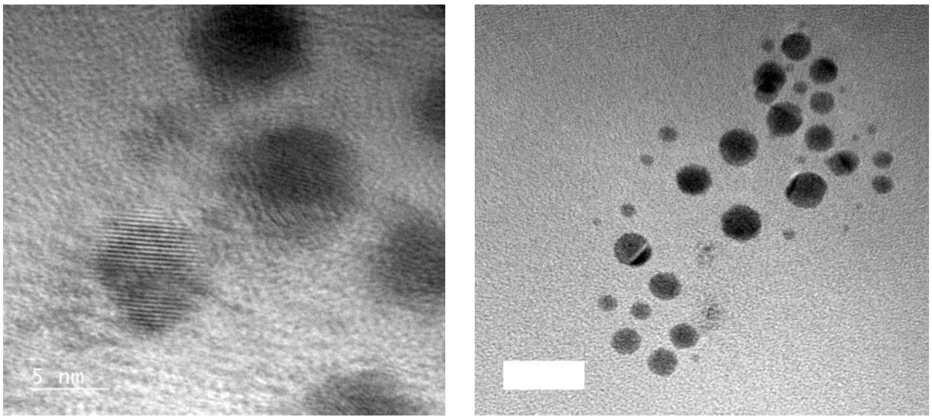
4.2. HRTEM Analysis:

High Resolution Transmission Electron Microscope(HRTEM) is a very powerful and cutting edge imaging method used to study nanotechnology and material science which can provide great insights into the atomic and molecular structures of materials. HRTEM works by directing a focused electron beam into a sample to analyse its atomic and molecular level morphology. HRTEM can achieve resolutions down to the sub-angstrom level, providing informations such as individual atoms, crystal defects and nanoscale interfaces with exceptional precision [26 , 27].

The structural aspect and surface morphology of prepared ZnO and PbO samples were studied using HRTEM(JEOL, model: 2100F). A well distribution of nanomaterials could be clearly seen in the HRTEM images. HRTEM images of ZnO and PbO for different magnification are Shown in the figures (Fig. 3). The HRTEM images verify the hexagonal crystal structure of ZnO nanoparticles and tetragonal morphology for PbO, which definitely shows a good agreement with the X-Ray Diffraction(XRD) analysis of our samples. The hexagonal morphology of ZnO nanoparticles can be clearly seen in the lower magnification image as shown in the below images-



(a)



(b) (c)

Fig. 3 HRTEM images – (a) & (b) for ZnO for different magnifications and (c) for PbO Nanomaterial

4.3. I-V Characteristics:

Electrometers are used to measure very low electric charge or voltage in a circuit [28]. Their working is based on the fact that when an electrical charge is applied to a conductor, it causes a separation of charges within the conductor.

A gap type planar electrode film geometry was used for measueing I-V characteristics. For this purpose, an electrometer (Keithley 2450 Electrometer) was used for the measurement and analysis of I-V characteristics. The Keithley 2450 Electrometer is a very sophisticated and high precision device used in the measurements of low currents and resistances in electronic circuits. Its advanced features help in the fields of scientific research by providing cutting edge technology to detect minute electrical signals making it an essential tool in semiconductor testing and material characterization. A simple schematic of measurement using Keithley electrometer is given below -

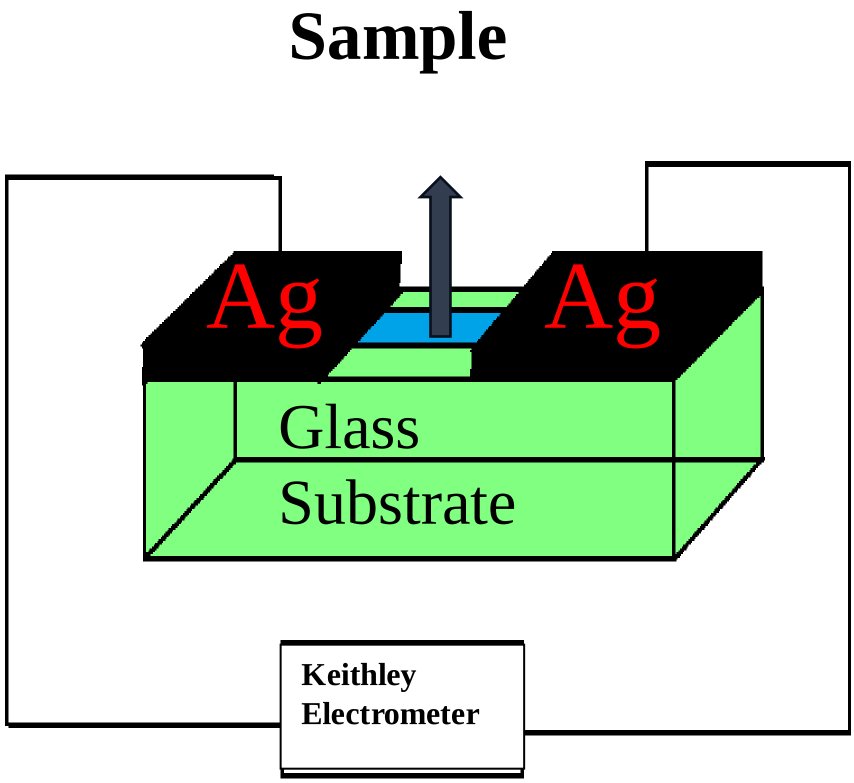
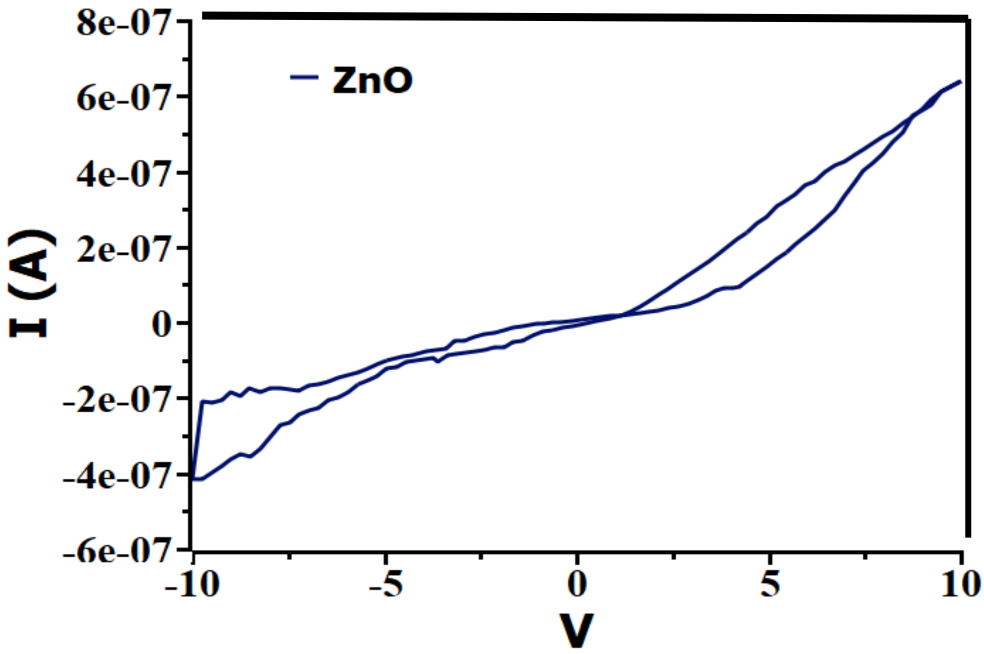
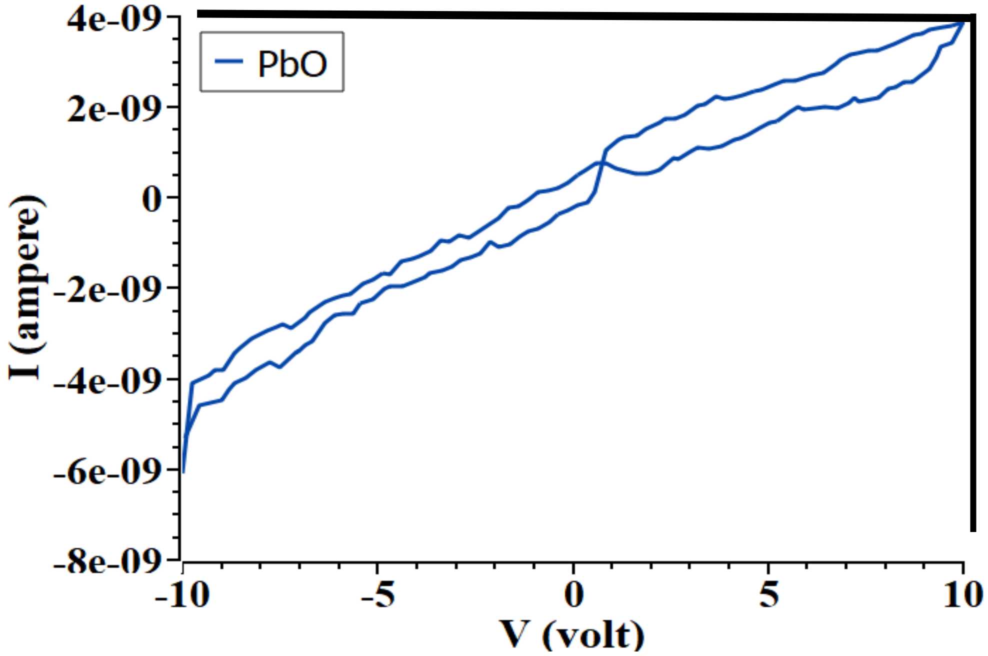


Fig. 4: Electrical measurement using Electrometer

The I-V characteristic curves for both the prepared samples are then plotted with voltage ranging between -10 V to +10 V. The curves are shown below-



(a)



(b)

Fig. 5: (a) I-V characteristic curve of ZnO

(b) I-V characteristic curve of PbO

4.4. Memristive behaviour characteristics:

A memristor or memory resistor is a double terminal passive electrical component that exhibits an extraordinary ability where it can remember its resistance value even after the power is turned off. Unlike resistors, capacitors and inductors, the memristor's resistance changes is based on the amount of current that has previously flowed through the circuit.

During the I-V measurements of the prepared samples, a pinched hysteresis loop could be clearly obserbed in the characteristic curve. Pinched hysteresis loop, however, is a prominent property of memristive devices [29]. The resulting hysteresis loop clearly exhibits mem-behaviour as the point of pinching occur in the origin of the I-V plane. However, some nanomaterials even exhibit mem-capacitive and mem-inductive behaviour because of their capacitive and inductive properties and they particularly show non-zero crossing hysteresis loop. Li. Qingjiang and others researched about the non-zero crossing pinching behaviour of TiO_2 based metal-insulator-metal devices [30]. The results obtained clearly indicates that if this pinching occurs in the 1st quadrant then the nanomaterials show mem-capacitive property, whereas pinching in the 3rd quadrant indicates mem-inductive property [30 , 31].

Here, we will only be concentrating on the memristive properties exhibited by our prepared nanomaterials. The patterns shown in Fig. 5 indicates the zero crossing pinching property of the memristive devices. To obtain the pinched hysteresis loop in the I-V plane, the voltage was initially ramped up from 0 V to +10 V, +10 V to 0 V, 0 V to -10 V and finally -10 V to 0 V. In ramping up the voltage from 0 V to +10 V, the current flowing through the memristor device increases as initially the device was in the High Resistance State (HRS) at 0 V. The device then started to change its resistive switching state at +10 V which is also known as the ON state or Low Resisance State (LRS) of the devide and the corresponding voltage can now be referred as the SET voltage [32]. The LRS continues all the way upto -10 V. After reaching -10 V, the device again started to change its resistive switching state and the corresponding voltage can now be referred as the RESET voltage [32]. A general I-V plot representing the resistive switching between LRS and HRS is shown in Fig. 6 for better understanding. After many consecutive analysis of the I-V plot of prepared samples, we found the same repetitive pattern in the I-V plot suggesting that ZnO or PbO film memristive devices possesses repeatability in the measurements.

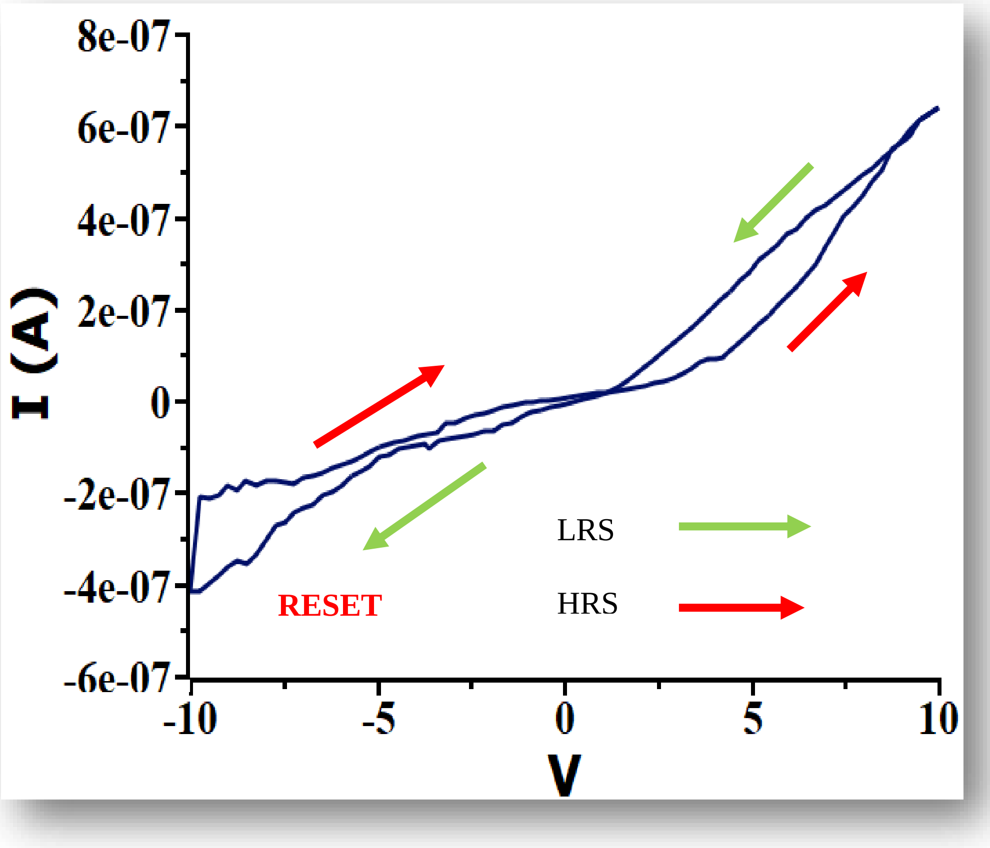


Fig. 6: Zero crossing behavior of memristive devices with arrows Suggesting the direction of resistive switching.

A memristor is usually characterised by the presence a pinched hysteresis loop in the I-V plot. This zero crossing loop is generally divided into two resistance states i.e. Low Resistance State (LRS) and High Resistance State (HRS). The transition from HRS to LRS and vice-versa is necessary in memory applications and it represents the application domain of memristors [33].

Chapter 5

Conclusions

The synthesization of Zinc Oxide (ZnO) and Lead Oxide (PbO) nanomaterials and their application in memristive devices have received significant attention in recent years due to their promising potential and unique structural and optoelectronic properties. These nanoparticles can be used in the fabrication of memristive devices. Further ZnO and Pbo nanomaterial based memristive devices can promise enhanced reliability and non-volatile memory applications. The repetitive transition between multiple resistance states of these memristive devices helps in emulating synaptic behaviour in neuromorphic computer systems and other advanced applications. The ability to tune resistance states makes these nanoparticles a very crucial component for understanding biologiccal synapses.

In our study of these nanomaterials, we have synthesized ZnO and PbO nanoparticles dispersed in PVP matrix via chemical bath deposition method. For analysing different properties of the prepared samples, XRD analysis, HRTEMimaging technique (for structural study) and Electrometer (for electrical study) were used. The structural morphology of the prepared samples were verified with the help of HRTEM technique. Further, the average particle size was calculated from XRD analysis and is found to be 28.934 nm for ZnO and 63.11 nm for PbO. The diffraction peaks (100), (002), (101), (110) corresponding to ZnO nanoparticles and diffraction peaks around (002), (201), (003), (131) corresponding to PbO nanomaterials are seen from the XRD data. It may also be noted that XRD always measures the average size of the samples from a collection of small quantum dots because of which the size obtained from the XRD data is much larger. The electrical properties were studied using a gap type planar geometry. The analysis of the obtained I-V plot shows a zero crossing pinched hysteresis loop for all our samples confirming that our prepared samples behaves like a memristive device. The occurrence of such memristive behaviour is due to the transition from HRS to LRS and vice-versa. Therefore, these kinds of nanomaterials may be a key component in the fabrication of memory devices.

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