Nonlinear I–V Characteristics and Thermal Stability of Nanocrystalline Titanium Oxide [1]

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ABSTRACT

Current versus voltage characteristics (I-V) of nanocrystalline Titanium oxide (TiO_2) has been investigated at various temperatures (from 50ºC to 350ºC) in air, measured by using a data acquisition system consisting of Keithley 6487 voltage source cum picoammeter. The nanocrystalline powder of $TiO_2$was prepared by the liquid phase method and samples were prepared via spray pyrolysis technique in the form of thin films on an optically plane and clean glass surface. X-ray diffraction studies showed a , mostly as rutile and anatase phases which both of them have the tetragonal structures of TiO_2. Surface morphological studies were performed with scanning electron microscopy. The nanocrystalline Titanium oxide exhibited nonlinear I-V characteristics of the negative resistance type with thermal stability.

Keywords: *Nanocrystalline Titanium oxide; XRD; FE-SEM; I-V characteristics; thermal stability*

INTRODUCTION

Titanium dioxide is a cheap, chemically stable, and non-toxic material. However, its electrical properties are unstable and it is a modest semiconductor and a mediocre insulator. For several applications, it would be interesting to make it either more insulating or more conducting. Titanium dioxide (TiO_2) is a material used in a wide range of common and high-tech applications. It is cheap, chemically stable, non-toxic, and last but not least bio-compatible. Titanium is successfully used as an implant material for dental, orthopedic and osteosynthesis applications, and its native oxide is mostly constituted of titanium dioxide [1]. TiO_2 powder is used as a white pigment in paint [2], replacing lead oxide which is toxic, and in toothpaste. Transparent single crystals or thin films have a high refractive index that makes TiO_2 suitable for optical applications [3-5]. Thus, research in many different fields is devoted to titanium dioxide under various forms such as single crystals, ceramics, and thin films. The goal of this work was to study the I-V characteristics, and structural, morphological properties of nano-crystalline TiO_2 thin films deposited by the spray pyrolysis technique.

EXPERIMENTAL

The methods of synthesis of nanoparticles can be broadly classified into three categories namely, liquid-phase synthesis, gas-phase synthesis, and vapor-phase synthesis. In the present work of the thesis, we have used the sol-gel method (which is under liquid phase synthesis) for the synthesis of pristine nanoparticles of TiO_2 [6-8].

Synthesis of TiO_2

Titanium tetra iso propoxide $[Ti(OCH(CH_3)_2)]_4$, iso-propanol $[(CH_3)_2CHOH]$ and nitric acid $[HNO_3]$ were used as received without any further purification. A 20 ml of solution of Titanium tetra isopropoxide was added drop by drop into the 22 ml of a solution containing 10 ml of iso-propanol and 12 ml deionised water under constant stirring at 80ºC into the round bottom beaker. After 1 hour, concentrated HNO_3 (.8 ml) mixed with deionised water was added into the TTIP

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solution and keep it under constant stirring at 60 ºC for 6 hours highly viscous sol-gel was obtained. The prepared sol-gel was heated at 300 ºC for 2 hours in the open atmosphere. After annealing, the TiO_2 nanocrystalline 2 g powder was obtained. In further preparation of TiO_2 film, the prepared powder was added in the ratio of 1:10 to the solution of isopropanol. The obtained powder is kept in a vacuum oven at 70 ºC for 24 hours so as to get completely dried powder.

Preparation of Samples

After the synthesis, obtained fine nanopowder of TiO_2 was calcinated at 800 ºC up to 5 hours in the auto-controlled muffle furnace, so that the impurities from product will be completely removed. The obtained product of fine nanopowder is further used for the preparations of samples. The obtained product of fine nanopowder of TiO_2 used for the fabrication samples was prepared via spray pyrolysis technique using Thin-film equipment: (Holmarc USA Make) in the form of thin films on an optically plane and clean glass surface [9-10].

Data Acquisition System

The data acquisition system consists of a Keithley 6487 source meter, GPIB cable, temperature controller, and computer as shown in fig. 1.1.

The picoammeter is controlled by the installation of GPIB (General Purpose Interface Bus) card. This GPIB card has an IEEE-488.2 bus, which is common to the testing meter. Communication with the temperature controller is done using Rs-232 port.

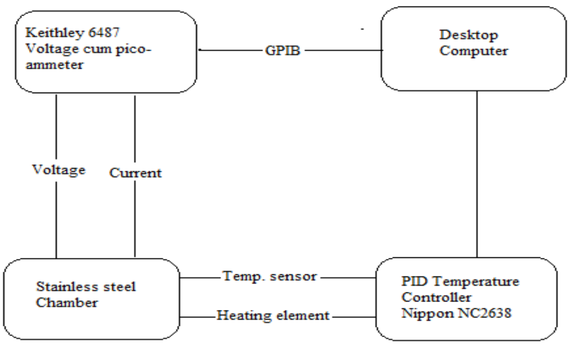

Fig.1.1 Flow Chart of Data Acquisition System

The language used for controlling the source meter and temperature controller is lab view graphical programming language, which can provide a friendly user interface. To study the VI characteristics of the sample element, special software was designed.

RESULTS AND DISCUSSION

X-Ray Diffraction : Nanocrystalline TiO_2

Titanium dioxide or Titania (TiO_2) is widely nominated for three main phases of rutile, anatase and brookite . Among them, the TiO_2 exists mostly as rutile and anatase phases which both of them have the tetragonal structures. However, rutile is a high-temperature stable phase and anatase is formed at a lower temperature .

Figure 1.2 shows the XRD pattern of pure Titanium Dioxide (TiO_2) which is calcinated at temperature 800ºC, which shows crystalline annealed with 2θ peaks lying at planes (1 0 1), (1 0 3), (0 0 4), (1 1 2), (2 0 0), (1 0 5), (2 1 1), (2 1 3), (2 0 4), (1 1 6), (2 2 0), (1 0 7), (2 1 5), (3 0 1), (0 0 8), (3 0 3), (2 2 4) and (3 1 2) respectively. The sharp diffraction peaks were clearly seen and they perfectly match with crystal structure of TiO_2 therefore, we get perfectly crystallinity of TiO_2 particles in Anatase phase. The calculated crystallographic parameters are a = 3.7300 (Å), b = 3.7300 (Å) and c = 9.3700 (Å) [Crystal system: Tetragonal ; Space group: I41/amd ; Space group number: 141, Reference Code: 01-075-1537] . All the peaks match well with the standard tetragonal type structures of titanium dioxide (TiO_2) in the Anatase phase and well agree with the JCPDS card No. 21-1272 . The average crystalline size was calculated by using the Scherrer formula found to **82.75** nm. The stick pattern of crystalline TiO_2 is as shown in fig. 1.2. It was found that the thick films consisted only of the tetragonal structure TiO_2 with no structural change and they were well crystallized during deposition [11-12].

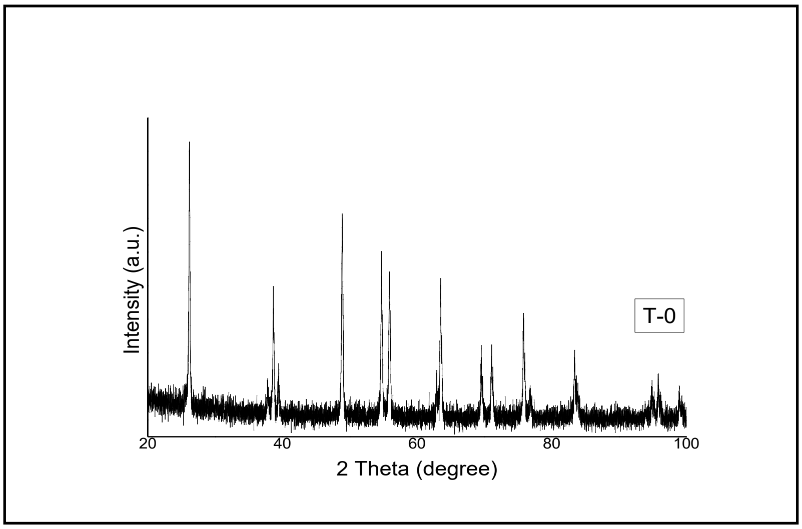

Fig. 1.2 XRD of crystalline pristine TiO_2

Field Emission - Scanning Electron Microscope

The surface morphology and the nanocrystalline particle size of titanium oxide (TiO_2) were examined by using Field Emission Scanning Electron Microscope. Figure 1.3 show the FE-SEM micrograph of pristine TiO_2, thin films. To verify the morphology scheme data obtained from the scanning electron microscopy will confirm the structural analysis obtained by the X-ray diffraction pattern [13-14].

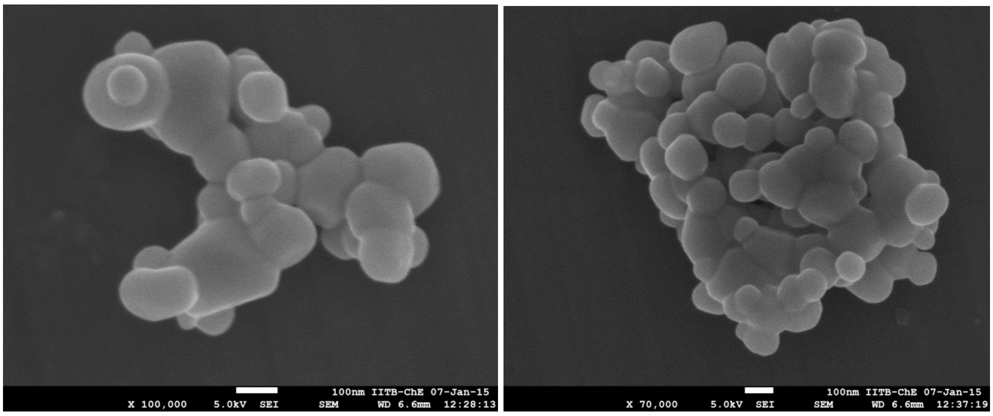

Fig. 1.3 FESEM of Nano-crystalline pristine TiO_2

Figure 1.3. shows the micrograph of sample pure TiO_2 thick films. TiO_2 particles are found to be tetragonal and semi spherical shape with the average size in the range of 85-94 nm. The average particle size observed in FE-SEM is in good agreement with the calculated value from XRD analysis [15].

Thermal Stability of TiO_2 thin films

Figure 1.4 shows the variation of current Vs. temperature from 50ºC to 350ºC at constant source voltage (10 V DC) by using Keithley (6487) voltage source cum picoammeter. i.e. thermal stability for different samples of pristine TiO_2

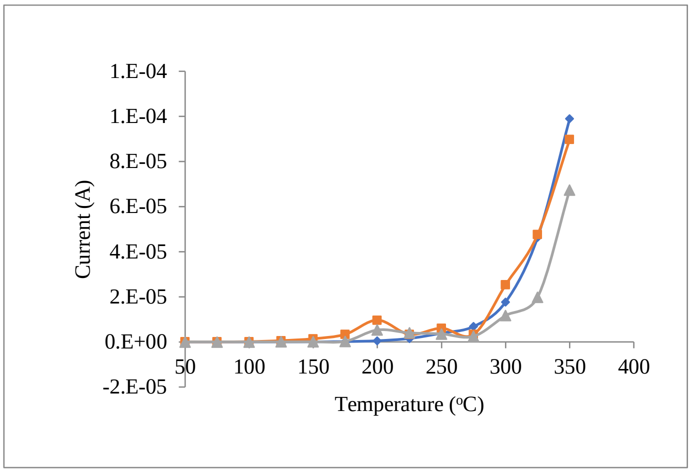

Fig. 1.4 Thermal Stability Curves of pristine TiO_2 Samples.

When samples are kept to a thermal treatment in an inert atmosphere (N_2), from 50°C to 350°C under the small interval of time, there occurs also a mass loss on heating in air atmosphere but it happens at higher temperature, around 500°C and in a smaller temperature interval (the mass loss begins in the neighborhood of 430°C and considerably slower down around 550°C). It is noteworthy that the temperature at which we observe oxidation in as – grown films is significantly lower than temperatures mentioned in for samples deposited by screen printing technique, a fact that seems to confirm the sensitivity of oxidation temperature on sample texture. In present study, thermal treatment of samples is less than temperature 500°C, so there is no problem of mass loss of thick film samples. Generally, the grain size of nanocrystalline materials increases by increasing annealing temperature. This could be attributed to the effects of evaporation of absorbed water and reorganization of the grain. Uniform distribution of the grain is also observable . This is important to note that, due to thermal treatment to the films, which show better and excellent sensitivity and stability results without any further mechanical deformations obtained or seen in the films [16-17].

Current-Voltage (*I–V*) Characteristics :

The current-voltage (I–V) characteristics of the sample of pristine TiO_2 and their temperature dependence have been investigated in the air by using a data acquisition system consisting of a Keithley 6487 source meter, GPIB cable, temperature controller, and computer. Fig. 1.4 shows the current-voltage (I–V) characteristics sample of pristine TiO_2 measured at different temperatures (50°C, 100°C, 150°C, 200°C, 250°C, 300°C and 350°C). All the samples show semiconducting behavior as the resistance of samples is decreasing with an increase in the I-V measurement temperature. The nature of the plot is initially linear and then it becomes nonlinear beyond threshold voltage, said to be exponential in nature. The resistance is drastically falling at temperature 350°C and is found to be in the range of 5-15 M□□□ The nanocrystalline titanium oxide exhibited nonlinear I-V characteristics of the current-controlled negative resistance type [18].

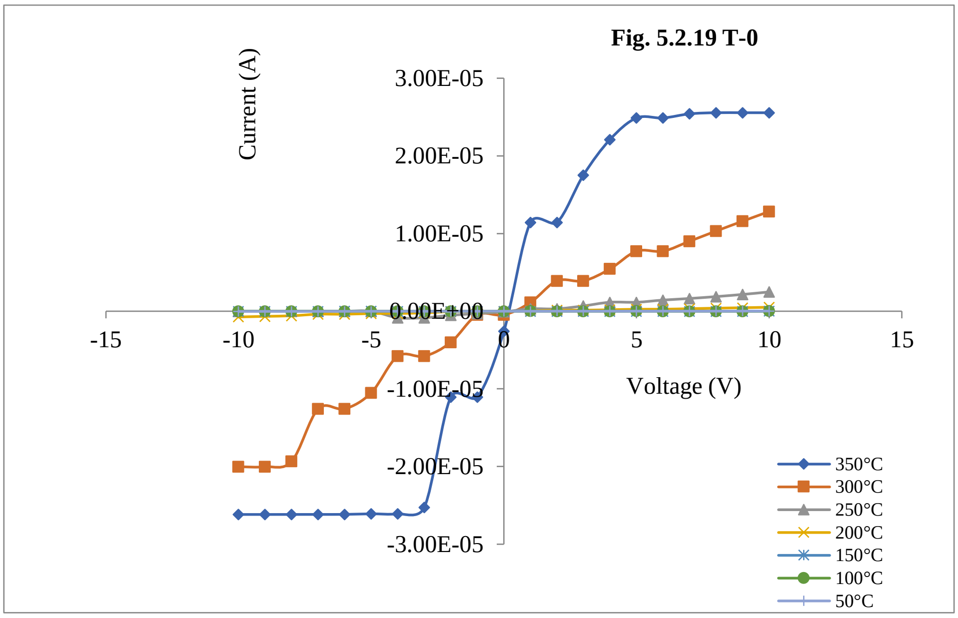

Fig. 1.4 Current–Voltage (I–V) *Characteristic* of pristine TiO_2

CONCLUSIONS

Current versus voltage characteristics (I-V) of nanocrystalline TiO_2 has been investigated at various temperatures (from 50°C to 350°C) in air and characteristic curves are generally used as a tool to determine and understand the basic parameters of a component or device and can also be used to mathematically model its nonlinear behaviour within an electronic circuit.

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