

## **Electrical and Structural Study of Screen Printed Thick Films of Tungsten Oxide Solid Films**

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### **ABSTRACT**

Nanomaterial's plays significant role in science and technology. The field has wide scope for different applications and methods. The nanomaterial is simple to use in different applications including sensors, optoelectronic devices, gas sensing applications, electronic devices. In current research article thick films of pure tungsten oxide ( $\text{WO}_3$ ) thick solid films were prepared by standard screen printing method on glass substrates and thick films are annealed at  $600^\circ\text{C}$  for 4 H in air atmosphere. Structural Characterization like X-Ray Diffraction (X.R.D), Scanning Electron Microscope (S.E.M), qualitative and quantitative elemental detection (E.D.A.X) was performed for prepared thick films. Structural parameters for thick films were performed by scanning electron microscopy, energy dispersive spectroscopy, X-ray diffraction for surface morphology and surface area, elemental detection, crystalline Phases of films and size of tungsten oxide ( $\text{WO}_3$ ) films. Electrical study was undertaken for Electrical parameters.

**Key words**-Nanomaterial's, structural parameters, element detection, electrical study etc.

### **1 INTRODUCTION**

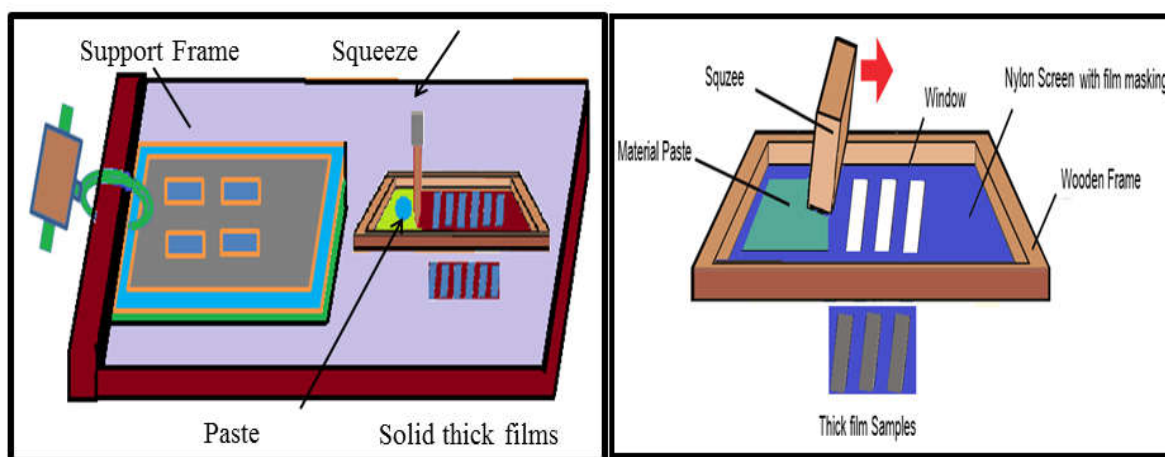
There are numerous methods have developed to fabricate thin and thick films in the material science. From the last fifty years several methods have investigated and developed day by day. The methods include spray pyrolysis evaporation, chemical vapor deposition, sol gel technique, magnetron sputtering pulsed laser deposition and screen printing technique. Screen printing technique was introduced in the later part of 20<sup>th</sup> century. It is versatile technique to prepare robust, compact and inexpensive hybrid circuit for different applications [1, 2]. Different methods have been investigated to grow tungsten oxide ( $\text{WO}_3$ ) films such as Spray pyrolysis, Vacuum evaporation, chemical vapour deposition, magnetron sputtering, pulsed laser deposition, sol-gel technique and screen printing technique [3, 4, 5]. The material tungsten oxide ( $\text{WO}_3$ ) in nanostructure form show remarkable physiochemical properties such as large surface area, small

size of particles and quantum size effect. Screen printing technique is simple to fabricate thick films of doped as well as base material. Among all screen printing method has found to gain popularity for the formation of superconducting oxide films. The greater advantages of thick film techniques include fast processing, economical use of paste and low cost [6-7].

## 2 MATERIAL AND METHODS

### 2.1 Preparation of thick films

Tungsten oxide thick solid films were prepared on glass substrate with particular dimension by using standard screen-printing technique as shown figure.



**Figure 1- Screen Printing Technique and its components**

The analytical grade Tungsten oxide chemical powder (99.99 %) was weighed. The calcined pure Tungsten oxide powder crushed thoroughly with glass frit which acts as permanent binder and ethyl cellulose acts as a temporary binder. The mixture was then blended with butyl carbittol acetate as organic vehicle to form the paste [8-9]. The paste was then screen printed onto the surface of glass substrate with dimensions 2 cm by 1 cm. After screen printing the solid films were dried under ordinary visible lamp of 200 watt for 2-3 hr. followed with firing into muffle furnace, dried under air atmosphere at 600°C for 4 hr. After preparation of pure tungsten oxide thick films were subjected to structural, morphological, electrical study.

## 2.2 Structural and Morphological characterization

Using X-ray diffraction (Miniflex Model, Rigaku, Japan) analysis from 20-80, 2 $\theta$  was carried out to examine the crystalline phases of the pure tungsten oxide thick solid films samples. The instrumental broadening was removed using silicon standard sample. The single line approximation method has adopted for the crystallite size determination using high intense peak. The average grain size of pure tungsten oxide were calculated by using the Scherer formula as referred [10],

$$D = \frac{0.9\lambda}{\beta \cos \theta}$$

The microstructure, EDAX, and chemical composition of the tungsten oxide films were analyzed using a scanning electron microscope [Nova nano SEM NPEP303] coupled with an energy dispersive spectrometer (EDS JEOL, JED-2300, Germany). The particle size was the sum of the diameter given in scanning electron microscope micrograph. The qualitative and quantitative analysis for element detection of tungsten oxide films were performed from the E.D.A.X spectra.

## 2.3 Electrical study

The resistance of the solid thick films of tungsten oxide sample was measured by using half bridge method at different temperatures in air. Variable D.C. power supply (APLAB 0-30V, 2A) is employed to enable the user to choose the required voltage; the voltage across the standard reference resistance was measured by digital multimeter. The thick film samples were heated slowly to avoid any thermal runaway. At the selected interval of temperature voltage across the reference resistance was noted. In this paper five thick films of tungsten oxide in under consideration for electrical study. In electrical study, resistance, activation energy and TCR is evaluated with increase in temperature.



**Figure 2- Static gas sensing and Electrical measurement set up**

The resistance of thick film samples of tin oxide was calculated by using the following equation [11].

$$R_F = R_{ref} [V_{supply}/V_{ref}]$$

Where,  $R_F$  –Resistance of thick film,  $R_{ref}$  – Resistance of Standard reference resistance,  $V_{supply}$  Applied voltage,  $V_{ref}$ –Reference voltage across standard resistance.

The activation energy ( $\Delta E$ ) is calculated with following equation

$$R_T = R_0 e^{-\Delta E/kT}$$

Where,  $R_T$  is resistance at  $T$ ,  $R_0$  is resistance at  $T_0$ ,  $\Delta E$  is activation energy and  $k$  is Boltzmann constant over a limited temperature range.

TCR is calculated with following equation

$$TCR = \frac{1}{R_0} \frac{\Delta R}{\Delta T} \times 10^6 \frac{ppm}{^\circ K}$$

Where,  $\Delta R$  is change in resistance between  $T_1$  and  $T_2$ ,  $\Delta T$  is temperature difference between  $T_1$  and  $T_2$ ,  $R_0$  is initial resistance of film sample

### 3 RESULT AND DISCUSSION

#### 3.1 X-Ray Diffraction (X.R.D)

The crystalline structure and crystallite size of pure tungsten oxide ( $\text{WO}_3$ ) thick film resistors were examined by X-ray diffraction measurement. X.R.D analysis of dried powder of thick films of tungsten oxide annealed at  $600^\circ\text{C}$  for 4 hrs is used for confirming the size of nanoparticles. The diffraction peaks located at different peak position are in good agreement with tungsten oxide phase structure indicating polycrystalline nature. Observed peaks are sharp and crystalline. Major peaks were found at  $2\theta = 23.12, 23.62$  and  $24.32^\circ$ , which were identified as corresponding to Miller index (002), (200) and (020) respectively, in monoclinic and some orthorhombic structure of  $\text{WO}_3$  (O-20-1324;M-43-1035) and other minor peaks in various directions. The crystallite size (D) was calculated from peaks using the Debye Scherer formula. The average crystallite size (D) of the pure  $\text{WO}_3$  thick film resistor sensors fired at  $600^\circ\text{C}$  was found to be 54.03 nm.

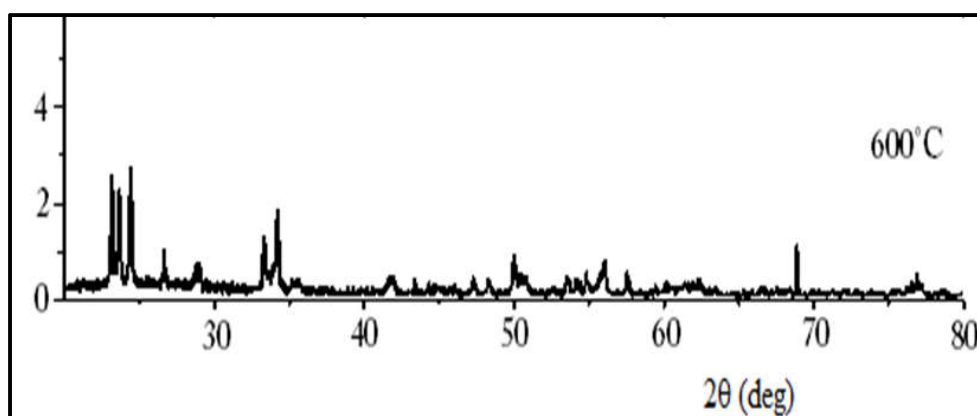


Figure 3 XRD Spectra of tungsten oxide thick solid films

#### 3.2 Scanning Electron Microscope (S.E.M)

The information about the surface morphology, crystalline shape and size of thick film materials is obtained by using Scanning Electron micrographs. SEM image were recorded at 100000X magnification for comparison. The SEM micrograph shows the distribution of particles and agglomerates to be affected with the firing temperature. It is evident that the material consists primarily of crystalline aggregates and is highly porous in nature. The SEM micrographs of  $\text{WO}_3$  thick films shows polycrystalline structure with less or more numbers of pores (voids) on the surface of the films, basically due to evaporation of organic binder during the firing of the films. The particle size distribution of the resulting films was affected by the firing temperature.

The average particle diameter was calculated was measured by the equation

$$S_w = \frac{6}{\rho d}$$

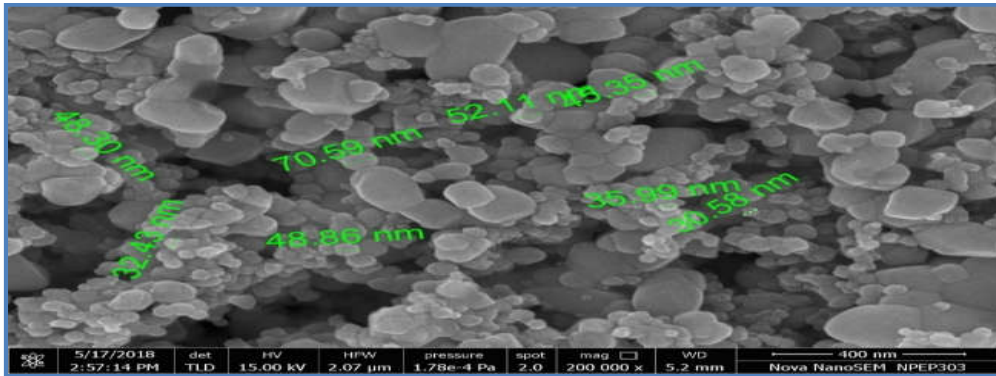


Figure 4-SEM micrograph of tungsten oxide thick solid films

Table 1: Particle size and specific surface area from XRD/SEM analysis

| Firing<br>Temperature WO <sub>3</sub><br>(°C) | Crystallite size<br>(nm) |       | Specific surface area (m <sup>2</sup> /g) |
|-----------------------------------------------|--------------------------|-------|-------------------------------------------|
|                                               | X.R.D                    | S.E.M |                                           |
| 600                                           | 54.03                    | 94.06 | 7.89                                      |

### 3.3 Energy Dispersive Spectroscopy (E.D.A.X)

The EDAX analysis of tungsten oxide WO<sub>3</sub> thick film resistors fired at 600°C was carried out using EDAX. The EDAX data indicate the appearance of peaks of the component in each WO<sub>3</sub> thick film resistive sensor material. The EDAX analysis showed presence of only W and O to be the only elements as expected, no other impurity elements were present in the WO<sub>3</sub> film samples. The EDAX results show lot of variation W/O ratio with firing temperature.

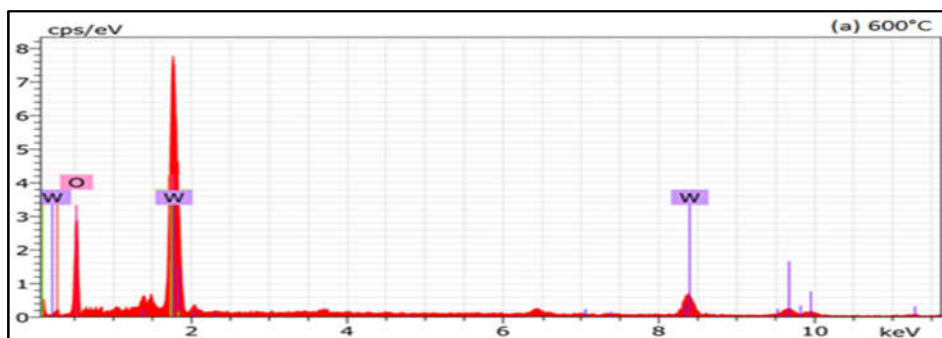


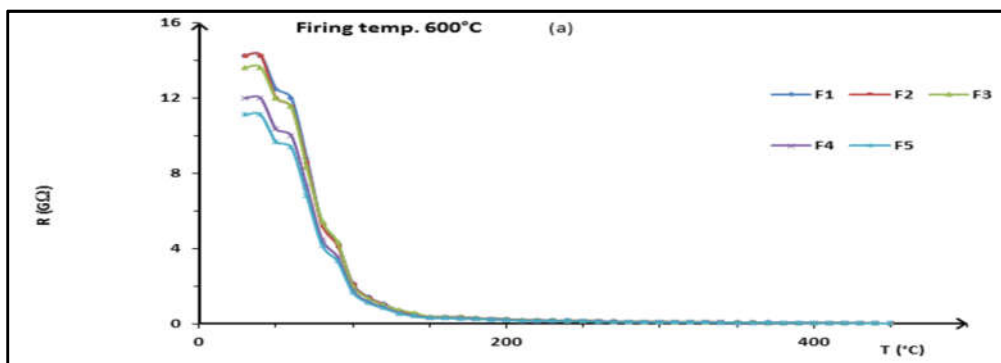
Figure 5 EDAX Spectra of tungsten oxide thick solid films

**Table 2: Element Composition**

| Firing temperature | Element | At. Wt. % | W/O ratio | Mass % | W/O ratio |
|--------------------|---------|-----------|-----------|--------|-----------|
| 600 °C             | W       | 65.19     | 1.9577    | 95.74  | 22.4741   |
|                    | O       | 34.81     |           | 4.26   |           |

### 3.4 Electrical Study

The electrical study was considered for five thick films of tungsten oxides. In this study resistance, activation energy and temperature coefficient of resistance is plotted against temperature. Following figure shows the variation in resistance of tungsten oxides thick films. The variation in sample to sample is observed due to different thickness of film, as thinner the film larger the electrical resistance of the thick film and vice-versa.

**Figure 6 Resistance Vs. Temperature Graph of sample to sample variations**

**Table 3: Sample to Sample Resistance variation of WO<sub>3</sub> thick films with temperature fired at 600°C**

| Firing temp.       | Film | Thickness | Resistivity | Sheet resistivity | TCR    | Activation energy (eV) |        |
|--------------------|------|-----------|-------------|-------------------|--------|------------------------|--------|
|                    |      |           |             |                   |        | LTR                    | HTR    |
| 600 <sup>0</sup> C | F1   | 22        | 137390      | 0.8243            | 908.37 | 0.3456                 | 0.4345 |
|                    | F2   | 21        | 121014      | 0.8046            | 908.34 | 0.3452                 | 0.4688 |
|                    | F3   | 19        | 189905      | 0.7290            | 907.80 | 0.3356                 | 0.4544 |
|                    | F4   | 18        | 158733      | 0.6486            | 908.33 | 0.3452                 | 0.4687 |
|                    | F5   | 16        | 133253      | 0.6444            | 903.26 | 0.3461                 | 0.4548 |

#### 4 Conclusion

Screen printing technique is reliable method to prepare thick solid films of tungsten oxides. The prepared thick films then subjected to electrical and structural characterization. From the research paper it has been concluded that thick solid films were successfully prepared. From XRD and SEM, the crystallite size was calculated. XRD spectra show that all peaks were matched for WO<sub>3</sub> phases. SEM image shows the nature of particles was agglomerated non homogeneous. Electrical data shows the n-type behavior of thick solid films.

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