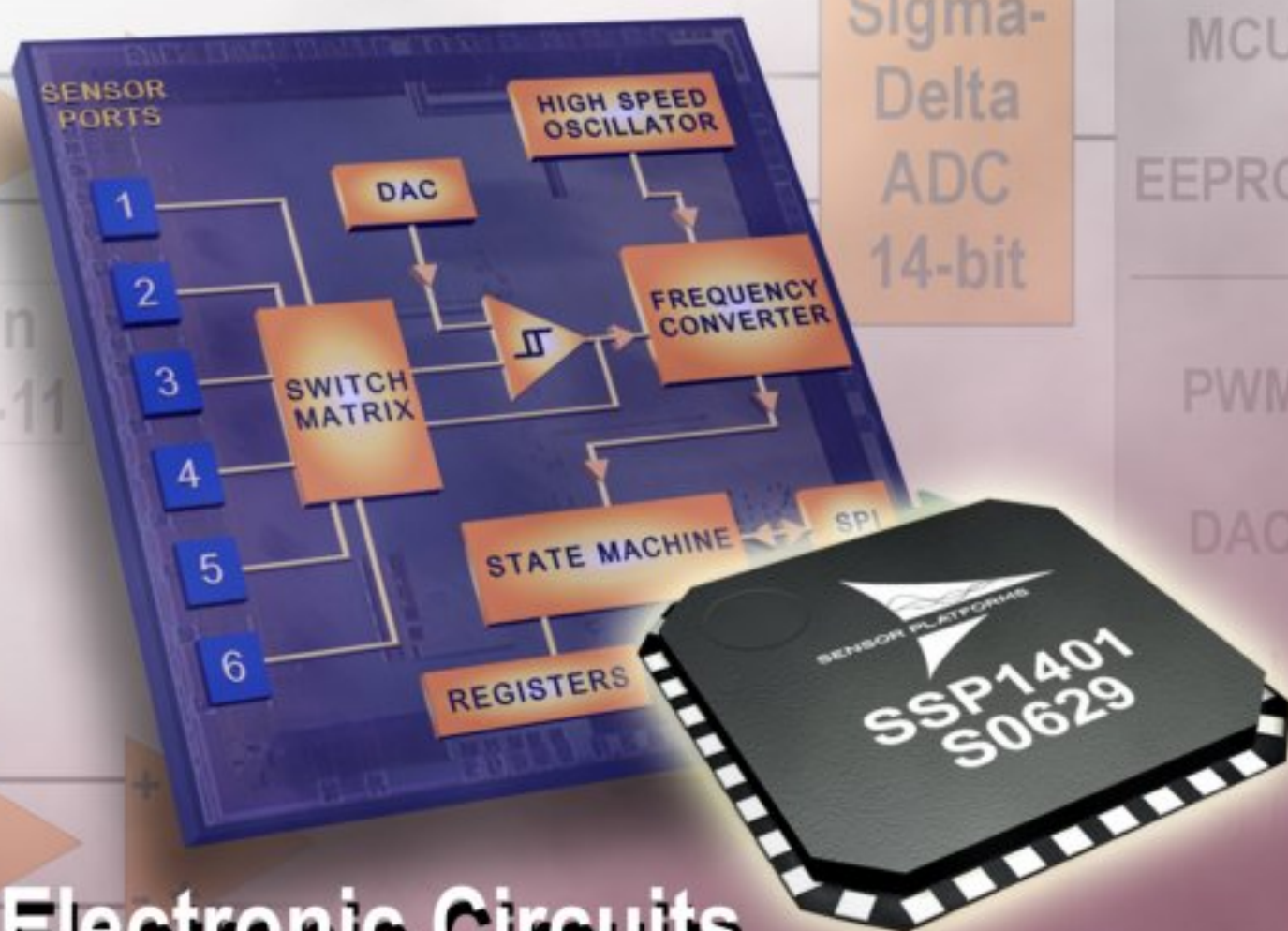


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Study of Microstructural Parameters of Screen Printed ZnO Thick Film Sensors

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Abstract: This paper explores the compositional, morphological and structural properties of ZnO thick films prepared by a standard screen printing method and fired between 650 °C to 900 °C for 2 hours in an air atmosphere. The material characterization was done using X-ray energy dispersive analysis (EDX), X-ray diffraction (XRD) and a scanning electron microscope (SEM). The deposited films were polycrystalline in nature having the wurtzite (hexagonal) structure with a preferred orientation along the (101) plane. The result shows that the wt. % of Zn was found to be 80.39, 82.66 and 83.47 % for firing temperatures of 700, 800 and 900 °C respectively may be due to the release of excess oxygen. The effect of the firing temperature on structural parameters such as the crystallite size, specific surface area, texture coefficient, RMSmicrostrain, dislocation density and stacking fault probability have been studied. The results indicate that grain growth can be increased by increasing the firing temperature which is responsible for decreasing the RMSmicrostrain, stacking fault probability and dislocation density in ZnO thick films. The crystallite size changes from 18.58 nm to 37.23 nm with respect to the increase in the firing temperature. *Copyright © 2010 IFSA.*

Keywords: ZnO, Thick films, SEM, XRD, EDX.

1. Introduction

ZnO is an n-type semiconductor. It is attracting tremendous attention due to its interesting properties like a wide band gap of 3.37 eV at room temperature with excitation binding energy (60 meV), high

chemical stability, low dielectric constant, large electrochemical coupling coefficient and high luminous transmittance [1]. It is widely used in photovoltaics, gas sensors, varistors, surface acoustic wave devices, electric transducers, as a piezoelectric material and for room temperature ultraviolet lasing [2, 3]. Therefore it can be applied extensively in IT (Information tech.), BT (Bio-tech.) and ET (Environmental tech.) [4]. Several deposition methods have been used to grow undoped and doped ZnO films such as spray pyrolysis, evaporation, chemical vapour deposition, magnetron sputtering, pulsed laser deposition, a sol-gel technique and a screen printing technique [5]. The Screen printing technique was introduced in the later part of the 1950's to produce compact, robust and relatively inexpensive hybrid circuits for many purposes. Later on a thick film technique has attracted attention for the sensor field [6]. Screen printing is a viable and economical method to produce thick films of various materials [7-12]. The main aim of the present study is to focus on the structural and surface morphological properties of ZnO thick films. We report studies on the structural parameters of ZnO thick films produced at different firing temperatures. Such a detailed understanding of film properties are necessary if ZnO is to be developed to a degree which will enable its use in gas sensing devices.

2. Experimental

Analar grade ZnO powder was calcined at 400 °C for 2 h in a muffle furnace. Then this powder was crushed and thoroughly mixed with a glass frit (PbO-70 %, SiO₂-18 %, Al₂O₃-9 % and B₂O₃-3 %) as a permanent binder. Organic vehicles such as butyl carbitol acetate (BCA) and ethyl cellulose (EC) were added to this mixture to achieve proper thixotropic properties of the paste. The ratio of inorganic to organic parts was maintained at 70:30 (the ratio of active powder to permanent binder was kept at 95:5 in 70 % and the ratio of EC to BCA was 98:2 in 30 %). ZnO thick films were prepared on alumina substrates using a standard screen-printing technique. The screen of nylon (40s, mesh no.355) was selected for screen-printing. The required mask (2 x 1.25 cm) was developed on the screen using a standard photolithography process. The paste was printed on clean alumina substrates (5 x 2 cm) with the help of a mask. The pattern was allowed to settle for 15 to 20 minutes in air. The films were dried under infrared radiation for 45 minutes and fired at temperatures of 650, 700, 800 and 900°C for 1.5 to 2 h (which includes the time required to achieve the peak firing temperature and then constant firing for 30 minutes at the peak temperature) in a muffle furnace. The structural properties of ZnO films were investigated using X-ray diffraction analysis from 20-80° [Rigaku diffractometer (Miniflex Model, Rigaku, Japan) with CuK α , $\lambda=0.1542$ nm radiation] with a 0.1°/step (2 θ) at the rate of 2 s/step. A scanning electron microscopy (SEM- JOEL JED-2300) was employed to characterize the surface morphology. The composition of ZnO thick film samples were analyzed by an energy dispersive X ray spectrometer (EDX) (JOEL-JED 6360 LA). The thickness of the ZnO thick films was measured using a Taylor-Hobson (Taly-step UK) system. The thickness of the films was observed to be uniform in the range of 20 μ m to 22 μ m. The effect of the firing temperature on the orientation of the films was investigated by calculating the texture coefficient using the following equation:

$$T_c(hkl) = \frac{I(hkl)/I_o(hkl)}{\left(\frac{1}{N}\right)\sum I(hkl)/I_o(hkl)}, \quad (1)$$

where

$T_c(hkl)$ is the texture coefficient of the (hkl) plane;

$I(hkl)$ is the measured intensity from the (hkl) plane;

$I_o(hkl)$ is the JCPDS standard intensity of (hkl) plane;

N is the number of diffraction peaks.

The crystallite size was determined using Scherrer's formula:

$$D = \frac{0.94\lambda}{\beta_{20} \cos \theta}, \quad (2)$$

where D is the crystallite size, λ is the wavelength of the X-ray radiation (1.542 Å), β is the peak full width half maxima of the (101) peak of the XRD pattern and θ is the diffraction angle.

Root mean square (RMS) microstrain (e) can be determined using a Williamson and Hall plot. The slope of the plot of ($\beta \cos \theta / \lambda$) versus ($2 \sin \theta / \lambda$) gives the value of the RMS microstrain.

The dislocation density was calculated using the following equation:

$$\rho = \sqrt{12} (e^2)^{1/2} / dD, \quad (3)$$

where e is the RMS microstrain, d is the interplanar spacing and D is the crystallite size.

The stacking fault probability was calculated using the following equation:

$$\alpha = \frac{2\pi^2 \Delta(2\theta)}{45\sqrt{3} \tan \theta_{101}} \quad (4)$$

The specific surface area of ZnO thick films was calculated using BET method using the following equation:

$$S_w = \frac{6}{\rho d} \quad (5)$$

where d is the diameter of the particles, ρ is the density of the particles.

3. Result and Discussion

3.1. Calcination, Drying and Firing of the Films

The calcination of the powder before the paste preparation and the firing process of the printed film can determine the sensitivity of the active layer of the film if it is used as a gas sensor. With calcination, grain boundaries are developed and the powder sinters to bigger agglomerates. This powder was milled after calcination. This causes a higher surface area after firing and therefore a higher sensitivity of layer. The calcination took from 1 h to 10 h [13].

A drying stage is required to remove the organic solvents, make the printed film adhere to the substrate and be relatively immune to smudging. After printing, the film was allowed to settle in air for a few minutes so that some of the volatile solvents were evaporated slowly at room temperature. The organic agent was still present in the paste at this stage. Drying took place at temperatures between 70-180 °C either in a conventional oven or by placing films under infrared radiation [13].

The high temperature firing cycle is designed to remove the remaining organic binders, to develop the structural and electrical properties of the film and to bond the film to the substrate. Temperatures up to 1000°C are required to achieve these objectives. During this firing process the glass frit melts and grains of the functional materials are held together and also the film becomes bonded firmly to the

substrate. There are three distinct regions in this firing cycle. Firstly the temperature slowly was increased towards the peak firing temperature. During this time the remaining organics were removed. This occurred at 350-400 °C. As the temperature reached 600-900 °C, the glass frit softens. Secondly the temperature remained constant for about 30 minutes. During this time the active material sintered and various reactions took place. The electrical properties of the film began to develop. Finally there was a cooling stage to room temperature that allows the glass frit to solidify [13].

3.2. Composition

Table 1 shows the composition of the films fired at different temperatures. The EDX spectrum showed the presence of only Zn and Oxygen. The mass percentage of Zn was found to increase with an increase of the firing temperature due to release of excess oxygen [14]. From the analysis it was found that the ZnO films are non-stoichiometric. The deficiency or excess of any type of atom in the crystal results in a distorted band structure, with a corresponding increase in conductivity. Zinc oxide loses oxygen on heating so that zinc is then in excess. The oxygen, of course, evolves as an electrically neutral substance so that it is associated with each excess zinc ions in the crystal; there will be two electrons that remain trapped in the solid material, thus leading to non-stoichiometricity in the solid. This leads to the formation of the n-type semiconductor [15].

Table 1. Composition of the films at different firing temperature.

Firing Temperature	Element	At.Wt. %	Mass%
700 °C	Zn	80.39	94.37
	O	19.61	5.63
800 °C	Zn	82.66	95.12
	O	17.34	4.88
900 °C	Zn	83.47	95.38
	O	16.53	4.62

3.3. Structural Parameters and their Analysis

Fig. 1 shows X-ray diffraction patterns of ZnO thick films deposited on alumina substrates and fired at 650 °C, 700 °C, 800 °C, 900 °C. XRD characterization demonstrates that all the structural variants correspond to the hexagonal wurtzite structure of ZnO. It has been observed that the XRD peak broadening decreases with an increase of the firing temperature. The intensity of reflections increases with a rise in the firing temperature. It has been observed that (101) reflections are of maximum intensity, which indicates that ZnO films have preferred orientation in the (101) plane. The observed 'd' values are found to be close to the JCPDS file for hexagonal ZnO [16].

Texture Coefficient (T_C)

From the values calculated using equation-1[17-18], it was observed that T_C approaches unity for randomly distributed samples whereas T_C is larger than unity for a preferentially oriented (hkl) plane. The lower values of T_C reveals that the films have poor crystallinity and this may be improved at a higher firing temperature. Fig. 2 shows the variation of the texture coefficient with firing temperatures for the (100), (002), (101) planes. From Fig. 2 it has been observed that the preferred orientation is the (101) plane for all firing temperatures. The increase in preferred orientation is attributed to an increased number of grains having this plane.

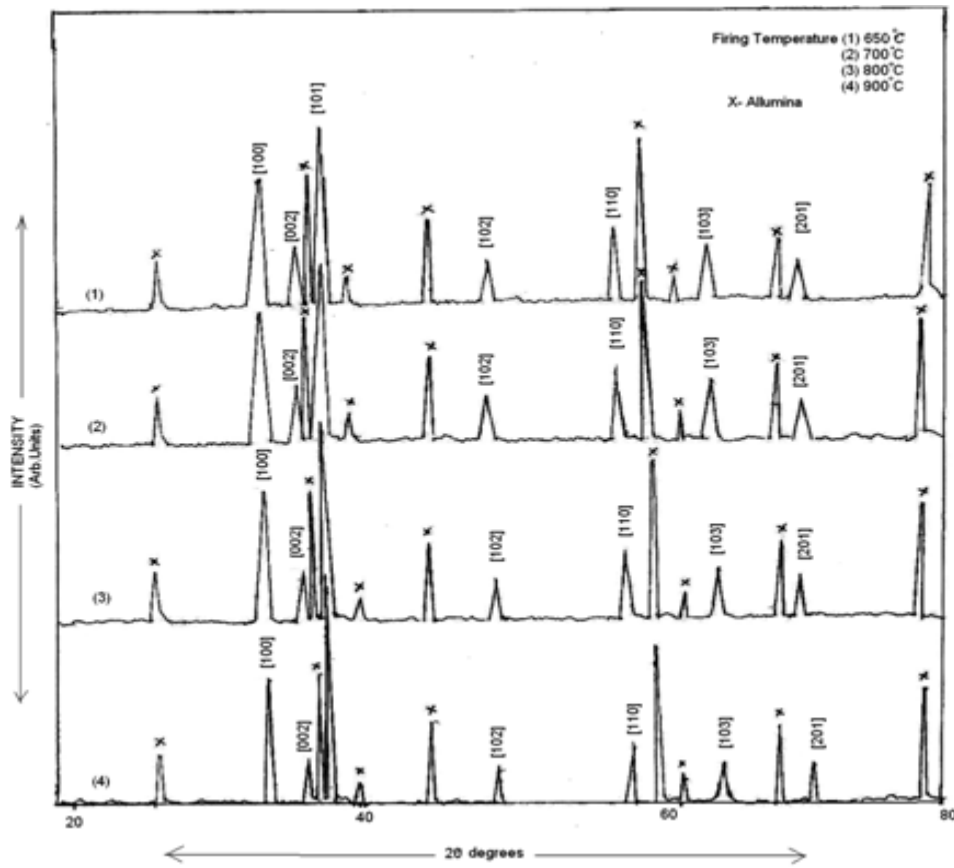


Fig. 1. XRD patterns of ZnO films fired at 650 °C, 700 °C, 800 °C, 900 °C.

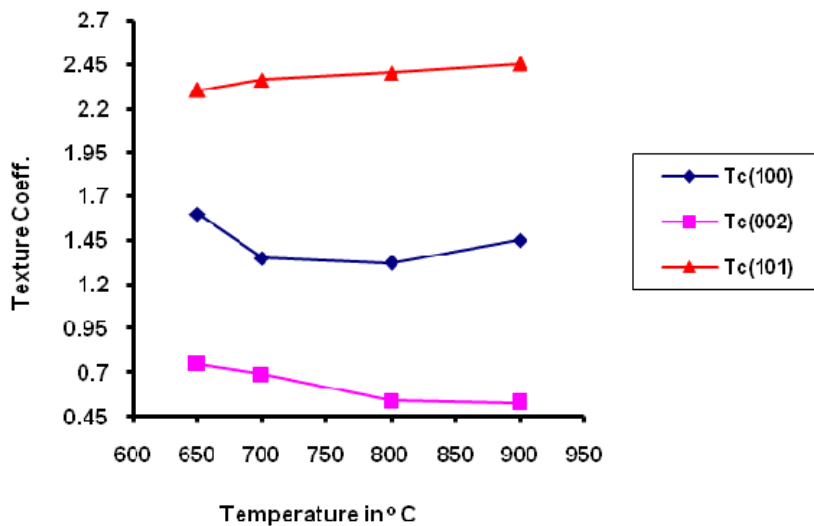


Fig. 2. Variation of the texture coefficient with firing temperature.

Crystallite Size (D)

The XRD pattern was used to calculate the crystallite size of ZnO by using equation-2 (Scherrer's formula) [19]. The crystallite size of ZnO films at different firing temperatures is given in Table 2.

Table 2. Variation of structural parameters with firing temperature.

Firing Temp. °C	Crystallite (grain) Size, D nm (XRD)	Particle Size, d nm (SEM)	Specific Surface Area in m ² /g	Micro Strain, e	Dislocation Density, $\rho \times 10^{13}$ Lines/cm ²	Stacking Fault probability α
650	18.58	381	2.75	0.6926	5.1985	0.0092
700	18.637	381	2.75	0.6926	5.1811	0.0092
800	24.79	470	2.25	0.5205	2.925	0.00797
900	37.23	617	1.715	0.3476	1.298	0.0066

Microstrain (e)

Broadening of X-ray diffraction line profiles is mainly caused by non-ideal optics of the instrument, wavelength dispersion, and microstructural imperfections in the crystals. The microstructural line broadening can be subdivided into size broadening and strain broadening. Size broadening is due to the finite size of domains surrounded by stacking faults, by twins or other imperfections, which diffract incoherently with respect to one another. Strain broadening is caused by a varying displacement of the atoms with respect to their reference-lattice positions. A uniform compressive or tensile strain (macrostrain) results in a peak shift of the X-ray diffraction lines, whereas a uniform statistical distribution of tensile and compressive strain results in a broadening of the diffraction lines (microstrain). A method to determine the microstrain by analyzing X-ray diffraction lines is described by Williamson and Hall [20]. A Williamson and Hall (W-H) plot is a classical method to obtain qualitative information of the anisotropy in broadening. The slope of the plot of $(\beta \cos \theta / \lambda)$ versus $(2 \sin \theta / \lambda)$ gives the value of the RMS microstrain. Fig. 3 shows the Williamson-Hall plots for ZnO thick films with different deformations from planes (100) to (201) at different firing temperatures. The points in the W-H plot are scattered. Therefore microstrain is RMS. If the points in the WH plot are scattered, i.e., if $(\beta \cos \theta / \lambda)$ is not a monotonic function of $(2 \sin \theta / \lambda)$, the broadening is termed anisotropic. The RMS microstrains calculated at different firing temperatures indicate a decreasing trend with firing temperature is given in Table 2.

Dislocation Density (ρ)

Dislocation density is defined as the length of dislocation lines per unit volume of the crystal [21]. A dislocation is an imperfection in a crystal associated with the misregistry of the lattice in one part of the crystal with an other part [21]. The dislocation density calculated using equation-3 [21] at different firing temperatures indicates a decreasing trend with firing temperature and is given in Table 2.

Stacking Fault Probability (α)

A stacking fault is a planar imperfection that arises from the stacking of one atomic plane out of sequence with another while the lattice on either side of the fault is perfect. The presence of a stacking fault gives rise to a shift in the peak positions of observed reflections with respect to the ideal JCPDS positions of the sample [22]. From the XRD patterns of ZnO films, the peak shift $\Delta(2\theta)$ for the oriented (101) plane was measured with a change in firing temperature. The stacking fault probability was calculated using equation-4 [21] at different firing temperatures indicating a decreasing trend with firing temperature and is given in Table 2.

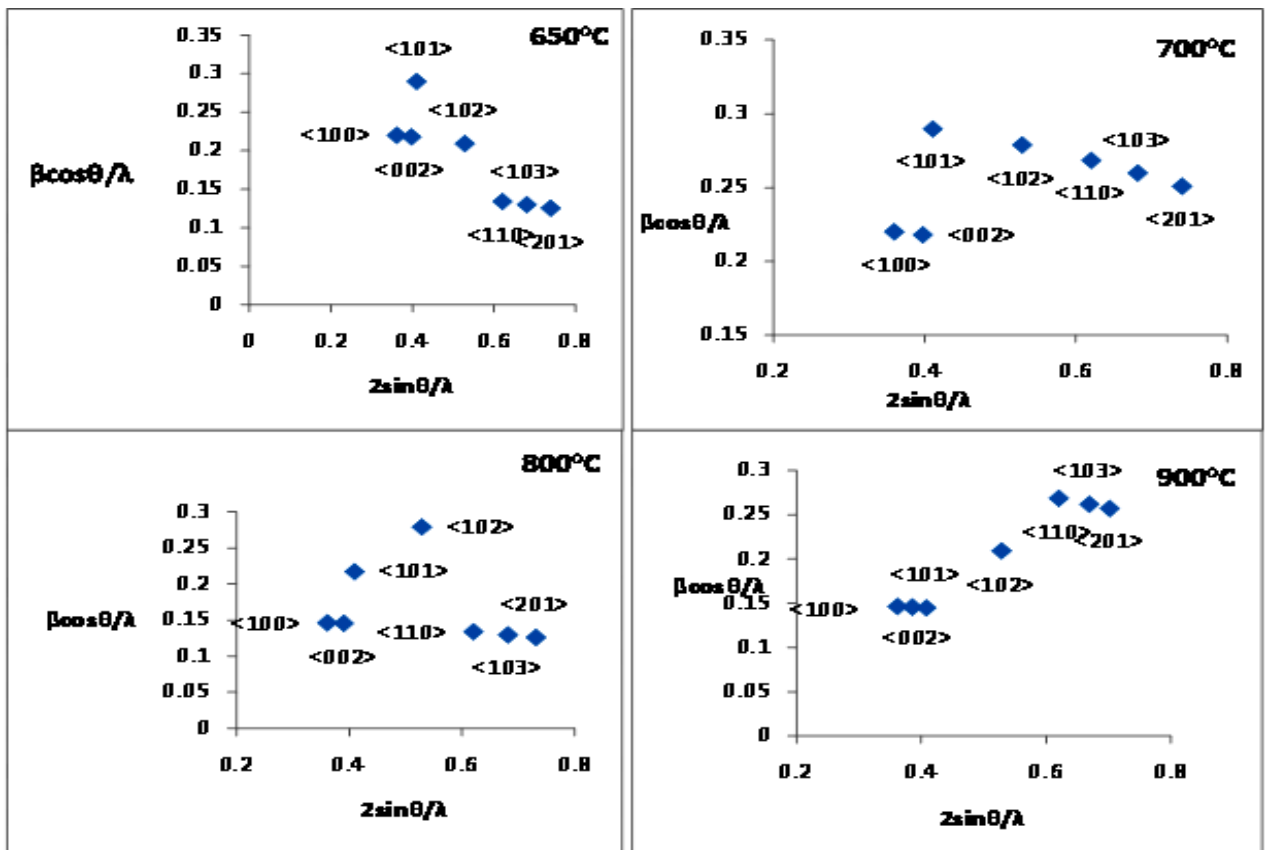


Fig. 3. Williamson-Hall plots for ZnO thick films with different deformations at different firing temperatures.

Fig. 4(a) shows the variation of RMS microstrain and dislocation density as function of firing temperature. Fig. 4(b) shows the variation of stacking fault probability as function of firing temperature. It was observed that the microstrain, dislocation density and stacking fault probability decrease with an increase in the firing temperature. This leads to a reduction in the concentration of lattice imperfections [23-24]. A similar trend has been reported by Mahalingam et al. [21] for electrodeposited ZnO thin films.

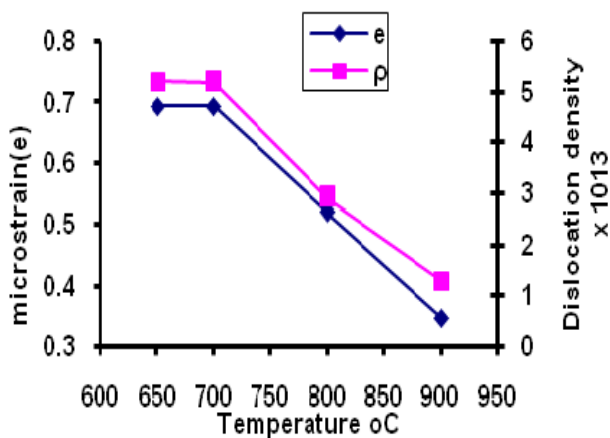


Fig. 4(a). Variation of microstrain ϵ and dislocation density ρ with firing temp.

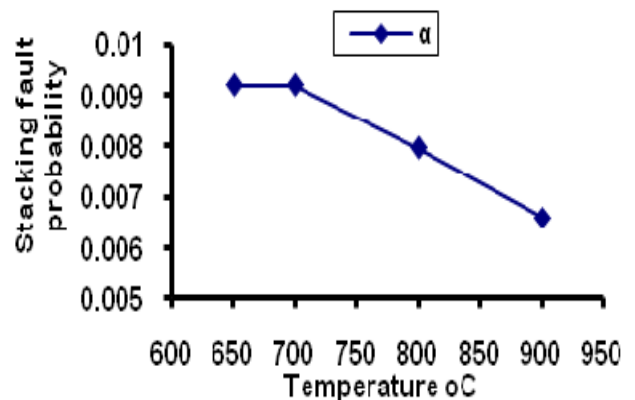
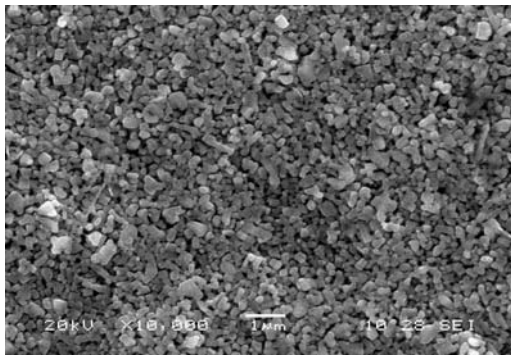


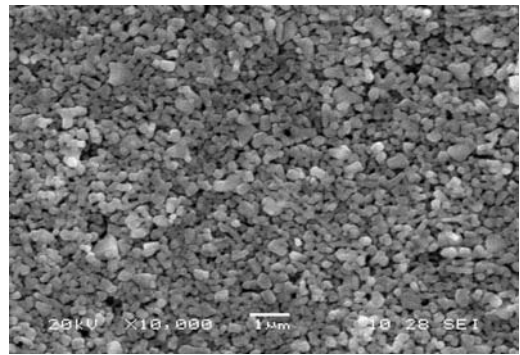
Fig. 4(b). Variation of stacking fault probability with firing temp.

3.4. Surface Morphology Analysis

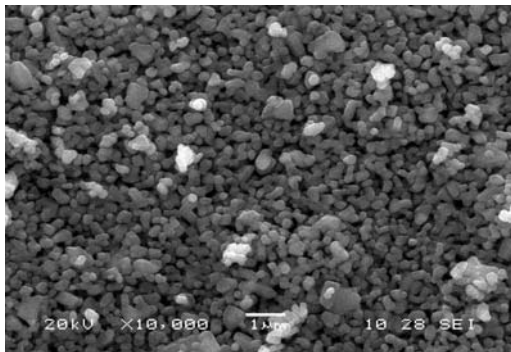
Fig. 5-(a), (b), (c) and (d) show SEM images of ZnO thick films at firing temperatures of 650 °C, 700 °C, 800 °C, 900 °C respectively. Fig. 5 (a) and (b) shows that the film structure is uniform and consists of a large number of spherical grains leading to a high porosity and large effective area available for the adsorption of oxygen species. Fig. 5 (c) shows that some necks are formed within the structure. Fig. 5 (d) shows the strongly agglomerated structure with neck growth. Due to this strong agglomeration, the effective surface- to- volume ratio would be decreased and oxygen adsorption-desorption capability of the film structure would be decreased. It has been observed that an increase in the firing temperature leads to an increase in the crystallite size. It is thought that the increase in crystallite size was affected by the promotion of the crystalline phase in the film and neck growth between particles as the firing temperature was increased. From the SEM images the average particle size has been measured and the specific surface area was calculated using equation 5 [25]. As the firing temperature increases, the grain and particle size increases and the specific surface area decreases.



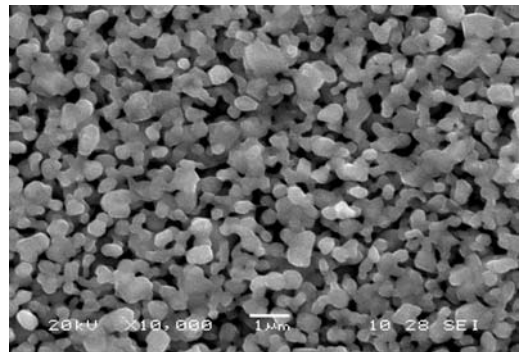
(a) SEM of ZnO film at 650 °C.



(b) SEM of ZnO film at 700 °C.



(c) SEM of ZnO film at 800 °C.



(d) SEM of ZnO film at 900 °C.

Fig. 5. SEM images for ZnO thick films fired at different firing temperatures.

4. Conclusion

ZnO thick films were prepared on alumina substrates by a standard screen printing technique which is a simple and in expensive method. From EDX and SEM it was confirmed that ZnO films were non-stoichiometric, which are suitable for gas sensing applications. XRD analysis reveals the hexagonal structure having improved crystallinity with an increase in the firing temperature. SEM indicates an

increase in crystallite size and particle size with an increase in the firing temperature. We have also studied various structural parameters of ZnO films. The microstrain, dislocation density and stacking fault probability, specific surface area decrease with an increase in the firing temperature.

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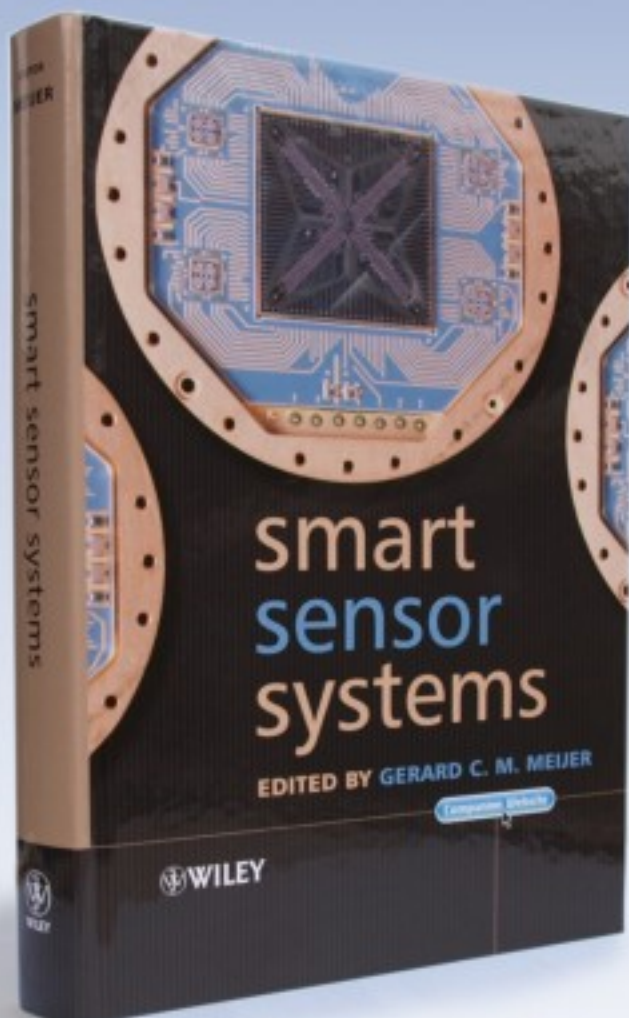
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