ISSN 1726-5479

SENSORS & TRANSDUCERS

9 vol. 144 /12



IEEE 1451

TEDS Sensors, IEEE 1451 Standards

International Frequency Sensor Association Publishing

IFSA

Sensors & Transducers

Volume 144, Issue 9
September 2012

www.sensorsportal.com

ISSN 1726-5479

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Sensors & Transducers Journal

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Volume 144
Issue 9
September 2012

www.sensorsportal.com

ISSN 1726-5479

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Digital Sensors and Sensor Systems: Practical Design

Sergey Y. Yurish



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ISBN: 978-84-616-0652-8,
e-ISBN: 978-84-615-6957-1

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Conference Announcement



Topic E2: Transportation & Mobility

The Euromat conference series, organised by the Federation of European Materials Societies (FEMS), is one of the largest events of its kind in Europe, covering the full width of materials science and technology. We would like to direct your attention to the following Symposia which are focussing specifically on transport applications:

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Sensors & Transducers Journal, Vol. 144, Issue 9, September 2012, pp. 45-61

www.sensorsportal.com

Sensors & Transducers

ISSN 1726-5479
© 2012 by IFSA
http://www.sensorsportal.com

Effect of Firing Temperature on the Micro Structural Parameters of Synthesized Zinc Oxide Thick Film Resistors Deposited by Screen Printing Method

[a*] Ratan Y. BORSE, [b] Vaishali. T. SALUNKE and [c] Jalinder AMBEKAR

[a,b] Thin and Thick Film Laboratory, Department of Electronic Science, M. S. G. College, Malegaon Camp - 423105 (Nasik), India

[c] Centre for Materials for Electronics Technology (CMET) Pune, India

Tel.: (02554) 252077, fax: (02554) 251705

[a*] E-mail: ratanborse@yahoo.co.in

Received: 26 June 2012 /Accepted: 21 September 2012 /Published: 28 September 2012

Abstract: Zinc-oxide (ZnO) powder was prepared by synthesis of zinc nitrate using self propagation solution combustion method in air with the purpose of modifying the powders physico-chemical properties. Textured ZnO thick film resistors (TFRs) have been deposited on alumina using standard screen printing method. The films were fired at 500, 600 and 700 °C for 2h firing cycle in air. The influence of firing temperature on the structural and morphological properties of the TFRs was investigated. X-ray diffraction (XRD) studies indicate the formation of polycrystalline hexagonal (wurtzite) crystal structure with preferential orientation along (101) plane. X-ray line broadening technique is adopted to study the effect of firing temperature on microstructural parameters such as interplaner spacing, average grain size, microstrain, dislocation density due to microstrain, stacking fault probability and texture coefficient. The dependence of these values on the firing temperature was established, which enabled analysis of the evolution of the defect structure of zinc-oxide TFRs during the firing. Scanning electron microscopic (SEM) images of the films fired at 500 and 600 ^{o}C are more porous as compared to TFR fired at 700 ^{o}C. The grain size is little bit larger as the increase of firing temperature although the increase is not significant. *Copyright © 2012 IFSA.*

Keywords: ZnO, TFRs, Firing temperature, SEM, Texture coefficient.

1. Introduction

Zinc oxide has proven itself as one of the competitive and promising candidates to replace expensive materials like CdS, TiO_2, GaN, SnO_2, and In_2O_3 for applications such as solar cells [1], photocatalysis

[2], ultraviolet laser [3, 4], transparent conductive oxides [5], spintronics [6], and gas sensors [7]. Zinc oxide (ZnO) is an II–VI group n-type semiconductor material with a hexagonal Wurtzite crystal structure [8, 9], a strong cohesive energy of 1.89 eV [10], a high optical gain (300 cm^{-1}) [11], high mechanical and thermal stabilities [12, 13], and radiation hardness [14-18]. ZnO possesses a Wurtzite structure similar to GaN [19, 20], which is widely used in high-performance optoelectronic devices. Zinc oxide is a direct wide-band-gap semiconductor with a band gap of 3.37 eV at room temperature and has been investigated extensively for its electrical, optical and photocatalytic properties. It has been used in applications such as laser diodes, solar cells, gas sensors, field effect transistors, transparent conductive films, hydrogen production by mean to water photolysis and degradation of organic pollutants [21-25]. Their absorbing optical, electronic, and mechanical properties are highly creditable for promising nano devices. For example, epitaxial ZnO films have demonstrated enormous potential for developing blue lasers and light emitting diodes [26-28]. The films have been prepared by various dry processes such as pulsed laser deposition (PLD) [29], metal organic chemical vapor deposition (MOCVD) [30], chemical vapor deposition (CVD) [31], molecular beam epitaxy (MBE) [32], magnetron sputtering [33], and electron beam evaporation [34]. Wet processes such as electrochemical deposition [35], spray pyrolysis [36, 37], sol–gel [38, 39], and hydrothermal method [40-42] are also valuable for the preparation of the oxide film.

Various chemical synthesis methods have been employed by several workers to synthesize nano/micro crystals such as solvothermal, hydrothermal, self assembly and sol-gel, etc [43-47]. Various workers have been working on synthesis and characterization of different nanostructures of pure and doped zinc oxide phosphors. M. Jayalakshmi et al have explained the synthesis of nano crystals of zinc oxide using self propagation combustion method starting with products of zinc nitrate and dextrose [48]. Zinc Oxide nanostructures could be synthesized by several techniques such as vapor deposition, oxidation, sputtering, and pulse laser deposition. The self propagation combustion method was used for the preparation of ZnO powder, though there are several methods of preparation, as this method is easy with compared to other methods and the chemicals required for these methods are easily available and cheap. In this work, ZnO films were prepared by standard screen printing method on alumina substrates. The effect of firing temperature on crystalline structure of the film was investigated.

The aim of this work is to produce high-quality synthesized ZnO thick film resistors for structural properties. Special attention was paid to the influence of the firing temperature on the structure and morphology of the thick film resistors.

2. Materials and Experimental Methods

2.1. Synthesis of ZnO Powder

Zinc oxide nano structured powder was prepared by self propagating solution combustion method [48]. The starting materials are Zinc nitrate and Dextrose. Proper amount of zinc nitrate and dextrose are dissolved in water contained beaker and placed on a hot plate for 15 minutes as the solution dehydrates to form a deposition like a gel. Then the beaker was placed in a preheated muffle furnace at 500 ^{o}C temperatures. The solution boils, ignites with a flame and the entire reaction was completed within 5 minutes. The powder is amorphous in nature. Then the powder was calcined at 650 ^{o}C to get nanocrystalline ZnO powder. The XRD pattern of this confirms the formation of ZnO.

2.2. Preparation of Thick Film Resistors

For ZnO thick film resistors, the inorganic to organic materials ratio was maintained as 70:30 %. In inorganic materials, the synthesized ZnO powder was used as a functional material. The ZnO powder was weighed and calcined in air atmosphere at 600 ^{o}C for 2 h. The ratio of active ZnO powder to permanent

binder was kept as 95:5 % in 70 % part. Glass frit (70 wt. % PbO, 18 wt. % Al_2O_3, 9 wt. % SiO_2 and 3 wt. % B_2O_3) was used as a permanent binder [49, 50]. Organic part consist of 8 % ethyl cellulose (EC, Loba Chemicals) as a temporary binder and 92 % butyl carbitol acetate (BCA liquid, Merck, Munchen, $C_{10}H_{20}O_4$, BP 245 ^oC) as a vehicle to make the paste. Butyl carbitol acetate was added drop by drop to obtain the proper viscosity and thixotropic properties of the paste.

The calcined ZnO powder was mixed and crushed thoroughly with glass frit and ethyl cellulose in an acetone medium in mortar and pestle. During this mixing process, BCA was added drop by drop to obtain the proper viscosity of the paste. This paste should have the thixotropic property for printing through the screen on the substrate. The paste was used to prepare thick film resistors on alumina substrate by using standard screen printing method using 140 s mesh no. 355. After screen printing, the films were dried under IR-lamp for 60 minutes [51] and then fired at temperatures of 500, 600 and 700^{o}C for 2 h firing cycle in muffle furnace.

2.3. X-ray Diffraction Analysis

To study the microstructural detail of the thick film resistors, X-ray diffractometer [Miniflex Model, Rigaku Japan] using CuKα, radiation (λ=0.1542 nm) with a 0.1^o/step (2θ) at the rate of 2 s/step was employed. A range of 2θ from 20–80° was scanned from a fixed slit type, so that all possible diffraction peaks could be detected. X-ray diffraction technique was used to determine the crystalline structure and preferential orientation of the crystallite materials and also to calculate the crystallite size. The average grain size of ZnO was calculated by using Scherrer's formula [52].

$$D = \frac{0.9\lambda}{\beta \cos\theta} \tag{1}$$

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

XRD-data is adopted to determine microstructural parameters such as texture coefficient, microstrain, dislocation density and stacking fault probability at different firing temperatures.

2.4. Surface Morphology by Scanning Electron Microscopy (SEM)

A scanning electron microscopy (Model SEM-JOEL JED-2300) was employed to characterize the surface morphology of s fired at 500, 600 and 700 ^{o}C. The composition of ZnO thick film samples were analyzed by an energy dispersive X-ray spectrometer (EDX) (JOEL-JED 6360 LA). For SEM all the ZnO thick film samples were coated with a very thin conducting gold layer (few100Å) using vacuum evaporation/sputtering technique to avoid charging of the samples. The thickness of the ZnO thick film resistors was measured using a Taylor-Hobson (Taly-step UK) system.

3. Results and Discussion

3.1. Characterization of Synthesized ZnO Powder

XRD profile of the synthesized ZnO powder prepared by using self propagation combustion method is shown in Fig. 1. It is clearly seen that powder has hexagonal wurtzite crystal structure (JCPDS 36- 1451). Grain size of the powder is 30 nm as calculated using Debye Scherrer method.

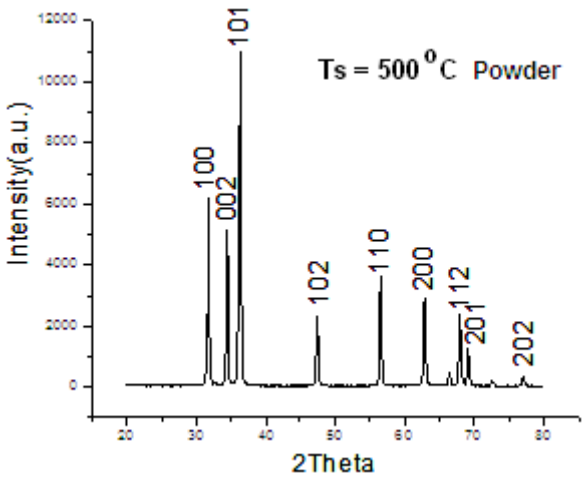


Fig. 1. XRD pattern of synthesized ZnO powder.

3.2. Fabrication of ZnO TFRs

ZnO Thick Film Resistors were prepared by standard screen printing method. The thick film resistor samples were good without crack. Thickness of the thick film resistors was observed to be uniform in the range of 25 μm to 35 μm.

3.3. Elemental Analysis (EDAX Analysis)

The elemental analysis of the synthesized ZnO thick film resistors fired at 500, 600 and 700 ^{o}C was carried out using EDAX (JEON, JED-6360 LA, Germany). The EDAX analysis shows presence of only Zn and O as expected, no other impurity elements were present in the ZnO thick films. From the EDAX spectra, it is found that wt% and at% of Zn and O is nearly matched. Table 1 gives quantitative elemental analysis of ZnO thick films**.**

Table 1. Composition of the ZnO Thick film resistors at different firing temperatures.

Firing Temperature	Element	At. Wt. %	Mass %
500 ^{o}C	Zn	77.33	93.47
	O	22.67	6.53
600 ^{o}C	Zn	79.67	94.12
	O	20.33	5.88
700 ^{o}C	Zn	80.39	94.37
	O	19.61	5.63

Fig. 2 (a, b, c) shows the EDAX spectra of ZnO thick film resistors fired at different temperatures. The mass percentage of Zn was found to increase with an increase of the firing temperature due to release of excess oxygen [53]. 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 looses 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 [54].

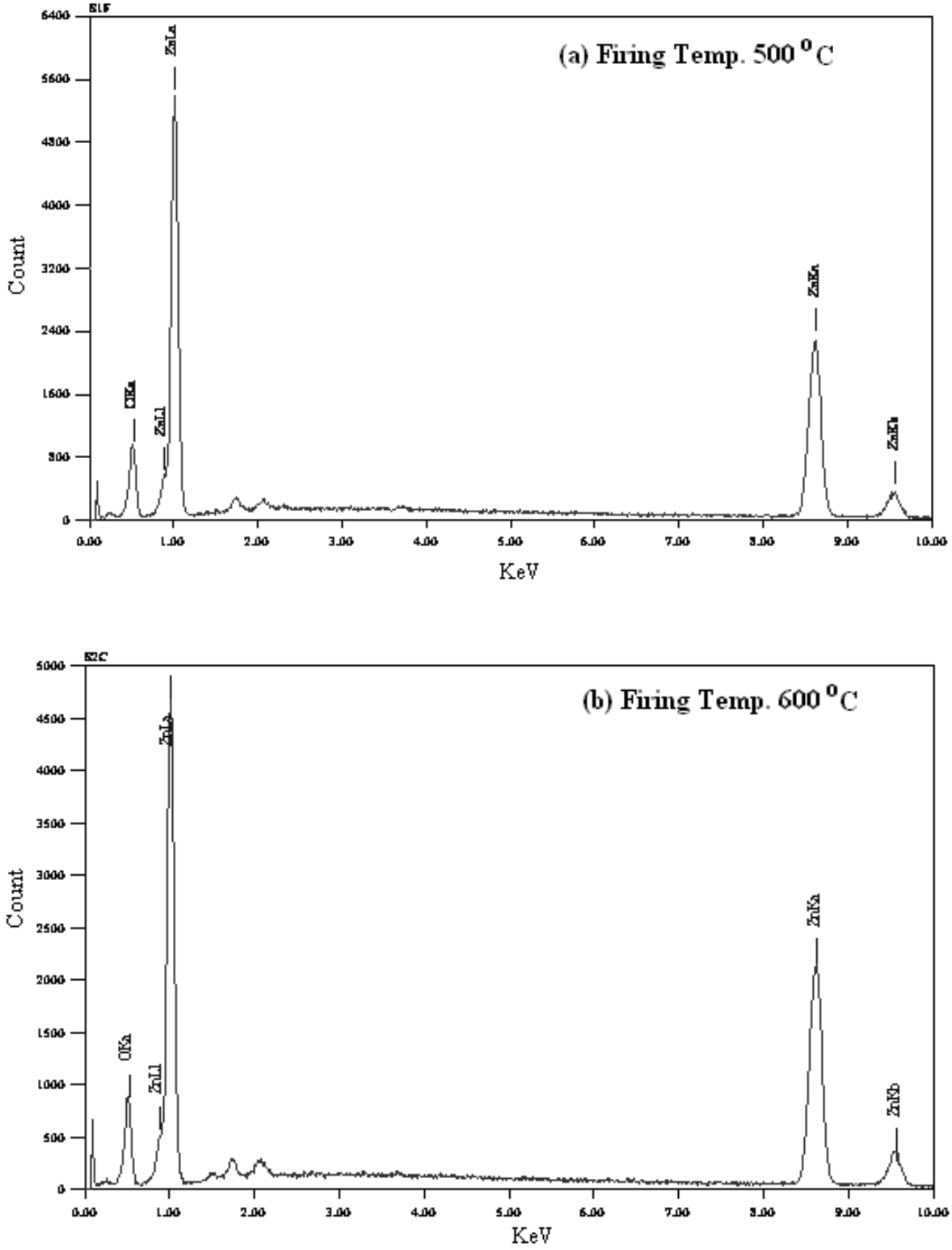


Fig. 2 (a, b). EDAX Spectra of ZnO thick films fired at (a) 500, (b) 600 ^{o}C.

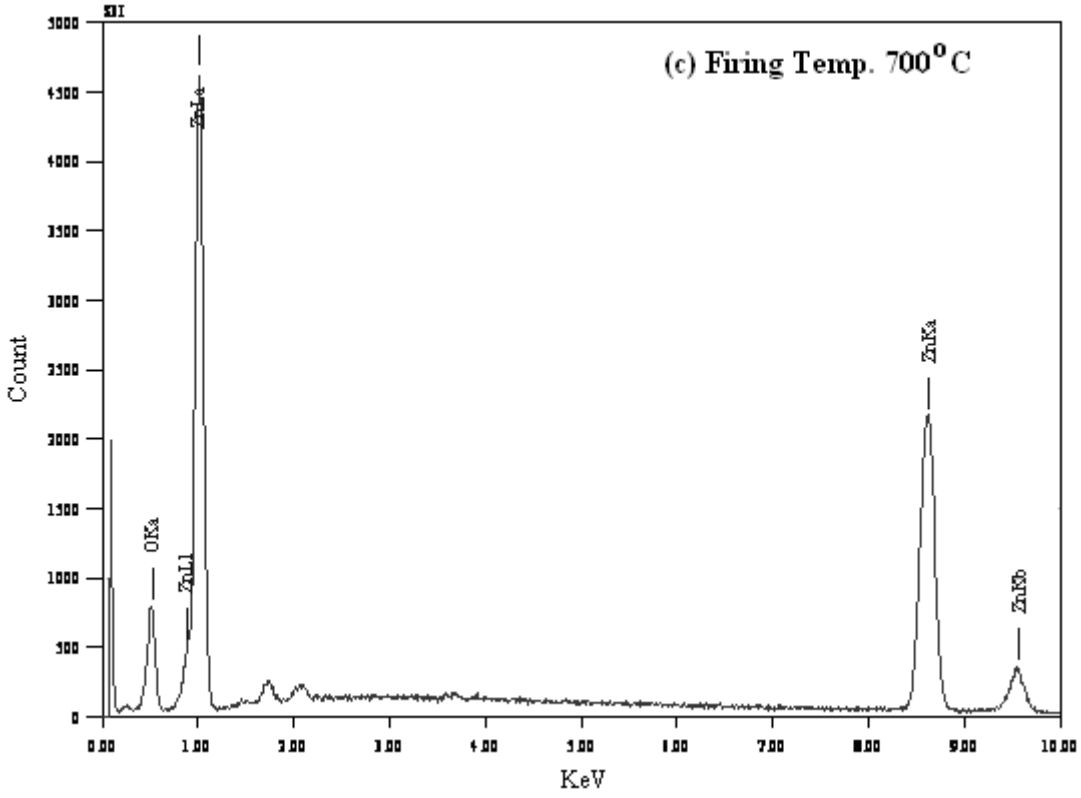


Fig. 2 (c). EDAX Spectra of ZnO thick films fired at (c) 700 ^{o}C.

3.4. Microstructural Parameters and their Analysis

3.4.1. XRD Analysis

The microstructure in TFRs is very complex and affected by a variety of parameters. The composition, softening point, viscosity, thermal expansion coefficient and wetting properties of the glass, the ratio of the size of glass particles to that of metal oxide grains and the sintering properties of the conductor material are some parameters known to affect the final microstructure of TFRs, besides the process condition (temperature and time in a defined firing cycle).

Fig. 3 shows the XRD profiles of the ZnO TFRs fired at 500, 600 and 700 ^{o}C prepared from synthesized ZnO powder. It is revealed that all the samples have diffraction peaks corresponding to (100), (002), (101), (102), (110), (103), (200), (112), (201) and (202) directions of the hexagonal wurtzite ZnO crystal structure [JCPDS 36- 145)] similar to Joseph et al [55]. Some peaks of alumina substrate (indicated with X) was also found in the XRD profile of the TFRs.

It has been observed that (101) reflections are of maximum intensity, which indicates that ZnO TFRs have preferred orientation in the (101) plane, the intensity of which increasing with increase in firing temperature of the TFR. The higher peak intensities of an XRD pattern is due to the better crystallinity and bigger grain size. Crystalline nature increases as firing temperature increased to 700 °C. This is clear from the increase in peak intensity. Also in the present case the increase in Zn content and firing temperature is the reason for high preferential orientation along the (101) plane. A high degree of crystal orientation reduces the probability of the scattering of the carriers at the grain boundary.

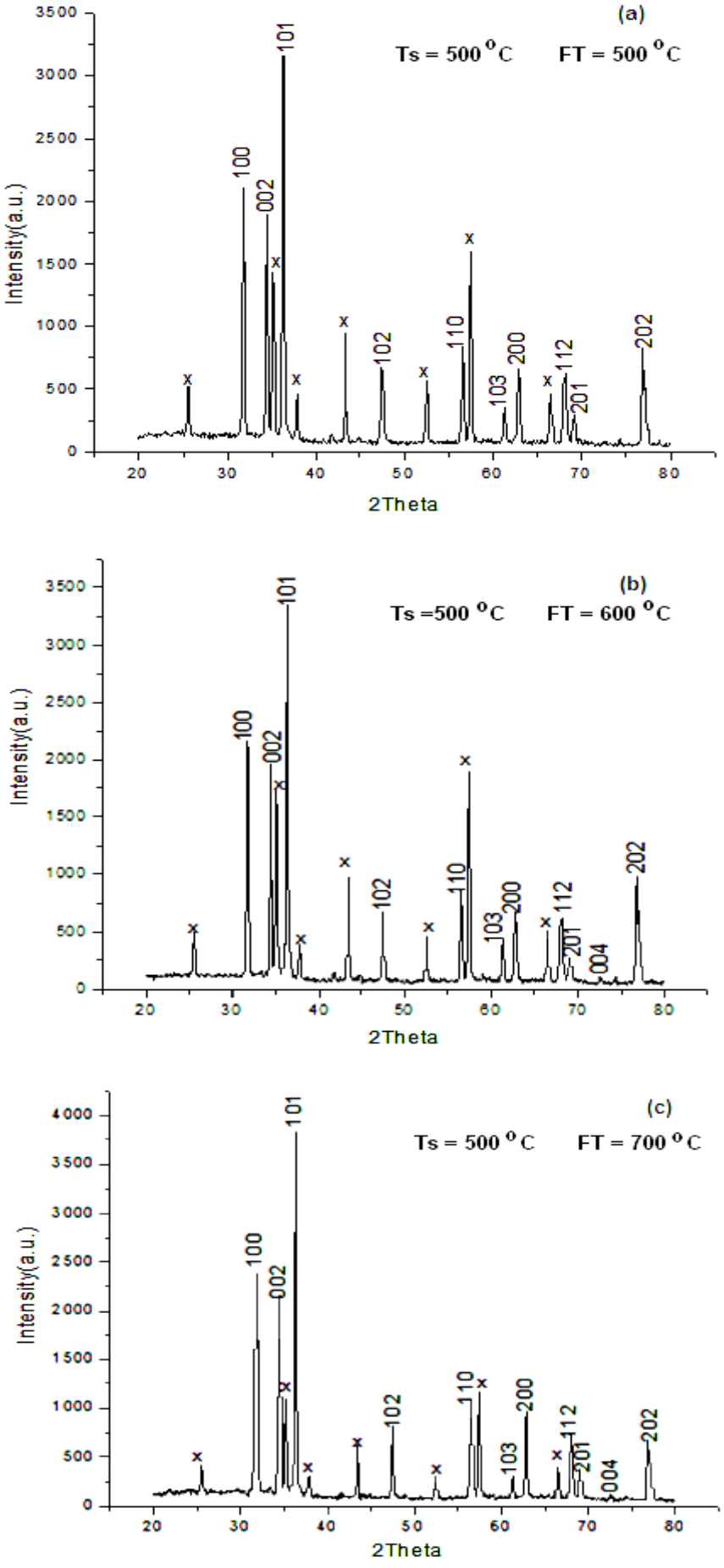


Fig. 3. XRD profiles of ZnO thick film resistors fired at (a) 500, (b) 600 and (c) 700 °C.

The XRD pattern was used to calculate the grain (crystallite) size of ZnO. The average grain size of ZnO TFRs fired at 500, 600, and 700 ºC was observed as 46.48, 51.47 and 53.39 nm respectively. It has been observed that the grain size increases with increase in firing temperature. The bigger grain size can be attributed to the agglomeration of particles due to increase in firing temperature [56-58]. While the XRD patterns of all the films seem to be qualitatively similar. Upon increasing the firing temperature from 500 to 700 ^{o}C, the 2θ increases and the FWHM value decreases, indicating that the (101) spacing decreases and the grain size of the ZnO TFR is improved with increasing firing temperature as indicated in Fig. 4 (a). These results are thought to be related to the reduction in oxygen atoms due to higher firing temperature. This change influences the stoichiometry of ZnO TFRs. The interplanar spacing of (101) plane is therefore 2.4797, 2.479 and 2.4777 Å for the TFRs fired at 500, 600, and 700 ºC respectively [Fig. 4 (b)], which is in good agreement with the standard value 2.4759 Å shown by JCPDS card 36-1451 file data. Table 2 shows the values of 2θ, interplanar (d) spacing and FWHM values of the ZnO TFRs deposited at different firing temperatures for (101) plane.

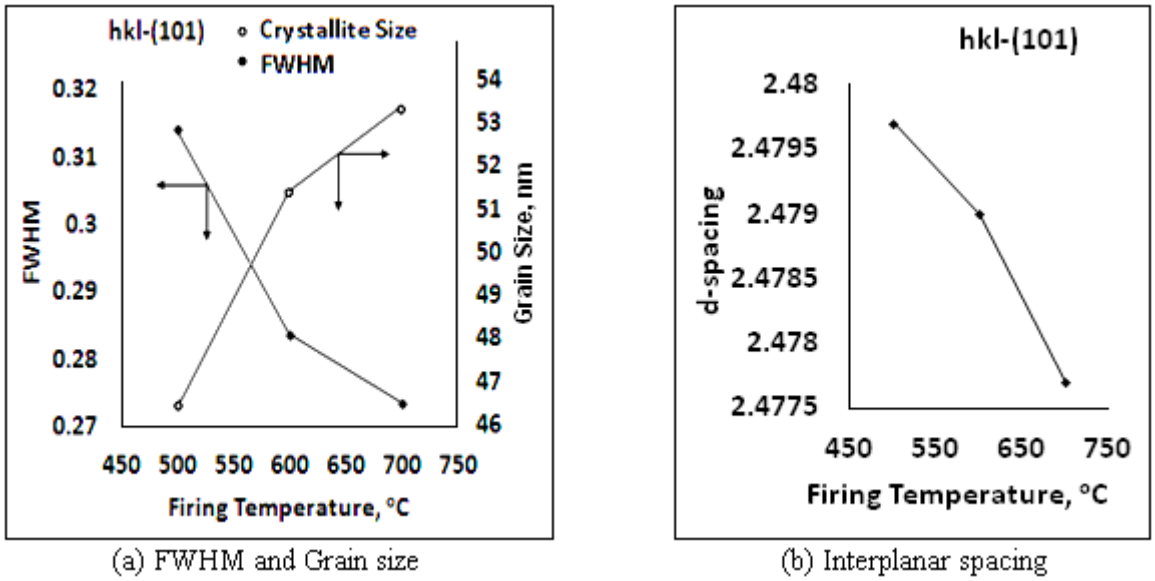


Fig. 4. Plot of FWHM, Grain size and interplanar spacing versus firing temperature.

Table 2. 2θ, interplanar spacing and crystallite size of the ZnO TFRs deposited at different firing temperatures for (101) plane.

Firing Temperature	(hkl) plane	2θ	Interplanar spacing, d (Å)	FWHM β
500 ^{o}C	101	36.23	2.4797	0.3141
600 ^{o}C		36.24	2.4790	0.2837
700 ^{o}C		36.25	2.4777	0.2735
JCPDS 36-1451		36.26	2.4759	--

3.4.1.1. Lattice Constants

From the XRD pattern, the lattice constants of synthesized ZnO TFR material can be calculated using the equation:

$$1/d^2_{(101)} = 4/3(1/a^2) + 1/c^2, \quad (2)$$

where *d* is the interplanar spacing, *a* and *c* are the lattice constants (being hexagonal structure, *a=b, c/a* =√8/3).

The calculated values of lattice constants are illustrated in Table 3. Lattice constants of ZnO are slightly depending on its stoichiometry. From Table 3, it has been observed that there is variation of lattice constants from JCPDS value (*a* = 0.3249 nm, *c* = 0.5205 nm). From XRD pattern it has been observed that there is shifting of diffraction peaks with firing temperature. It may be attributed due to slight variation of Zn and/or O stoichiometry in ZnO. This assumption is further confirmed by the results of EDAX analysis. As a semiconductor, in ZnO the radius of O^{2-}(0.132 nm) is larger than that of Zn^{2+} (0.074 nm). So the variation of O content in ZnO will lead to the variation of its lattice constants, which cause the shifting of the diffraction peaks [59, 60].

3.4.1.2. Texture Coefficient (T_c)

Texture coefficient (T_c) is used to quantify the preferential orientation of the films fired at different temperatures. The effect of the firing temperature on the orientation of the films was investigated by calculating the texture coefficient using the following equation [61, 62, 63]:

$$T_{C(hkl)} = \frac{I_{(hkl)}/I_{O(hkl)}}{1/N[\sum_N I_{(hkl)}/I_{O(hkl)}]} \tag{3}$$

where $h_{(hkl)}$ is the texture coefficient of the (*hkl*) plane, $I_{(hkl)}$ is the measured intensity, $Io_{(hkl)}$ is the *JCPDS* standard intensity and *N* is the number of diffraction peaks. It was observed that T_c is larger than unity for a preferentially oriented (hkl) plane [62, 63]. The lower values of T_c reveals that the films have poor crystallinity and this may be improved at a higher firing temperature. Fig. 5 shows the variation of the texture coefficient with firing temperatures for the (100), (002) and (101) planes. From figure 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 along the plane. The texture coefficient increases with increase in firing temperature for all planes.

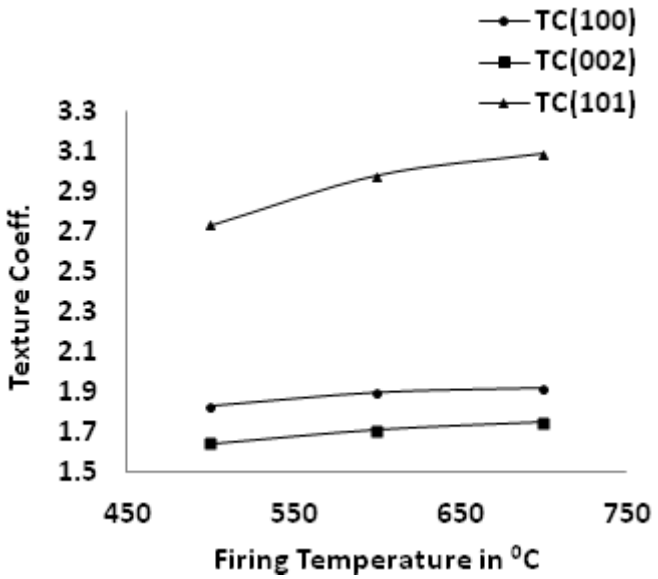


Fig. 5. Variation of the texture coefficient with firing temperatures.

3.4.1.3. Microstrain and Dislocation Density

XRD lines are usually broadened in their shape. These effects can be classified into instrument and specimen broadening. Instrument broadening originates from the non-ideal optical effects of the diffractometer and from the wavelength distribution of the radiation. In the present work instrumental broadening is corrected by using a standard defect free silicon sample.

Specimen broadening arises due to small crystallite (grain) size and strain (lattice distortion). Grain size causes the radiation to be diffracted individually [64]. The prepared ZnO thick film resistor is polycrystalline in nature, and hence large number of grains with various relative positions and orientations cause variations in the phase difference between the wave scattered by one grain and the others. The total intensity scattered by all grains is the sum of individual intensities scattered by each grain. On the other hand, lattice strain broadening is caused by varying displacement of the atoms with respect to their reference-lattice positions. A uniform compressive or tensile strain (macrostrain) results in peak shift [65] of X-ray diffraction lines, whereas a non-uniform tensile and compressive strain results in broadening of diffraction lines (microstrain). Thus grain size and microstrain effects are interconnected in the line broadening of peaks, which makes it difficult to separate. Many approaches exist for the evaluation and separation of size and strain parameters from the occurring line broadening. Williamson-Hall technique [64] is adopted in the present work where grain size D and micro strain ε is related as:

$$\frac{\beta_C \cos\theta}{\lambda} = \frac{1}{D} + \varepsilon\left(\frac{\sin\theta}{\lambda}\right) \tag{4}$$

where β_c is the instrumental effect corrected full width at half maximum of the peak measured in radian, θ is the diffraction angle and λ is the wavelength of X-ray.

The slope of the plot of $\beta_c \cos\theta/\lambda$ and $\sin\theta/\lambda$ gives the microstrain and the inverse of intercept on y-axis gives grain size value. Fig. 6 shows the Williamson-Hall plot of ZnO TFRs fired at different temperatures. It shows grain size increases from 46.48 nm to 53.39 nm as firing temperature increases but microstrain value deceases from 2.237 to 1.363.

Fig. 7 shows the variation of RMS microstrain and dislocation density as function of firing temperature. Dislocation density is defined as the length of dislocation lines per unit volume of the crystal. A dislocation is an imperfection in a crystal associated with the misregistry of the lattice in one part of the crystal with another part [66]. The dislocation density was calculated using the following equation:

$$\rho = \sqrt{12}\,(\boldsymbol{\varepsilon}^2)^{1/2}/(d \times D), \tag{5}$$

where $\boldsymbol{\varepsilon}$ is the RMS micro strain, d is the interplanar spacing and D is the crystallite size.

It was observed that the microstrain and dislocation density decrease with an increase in the firing temperature. This leads to a reduction in the concentration of lattice imperfections [58, 67]. A similar trend has been reported by Mahalingam et al. [66] for electrodeposited ZnO thin films.

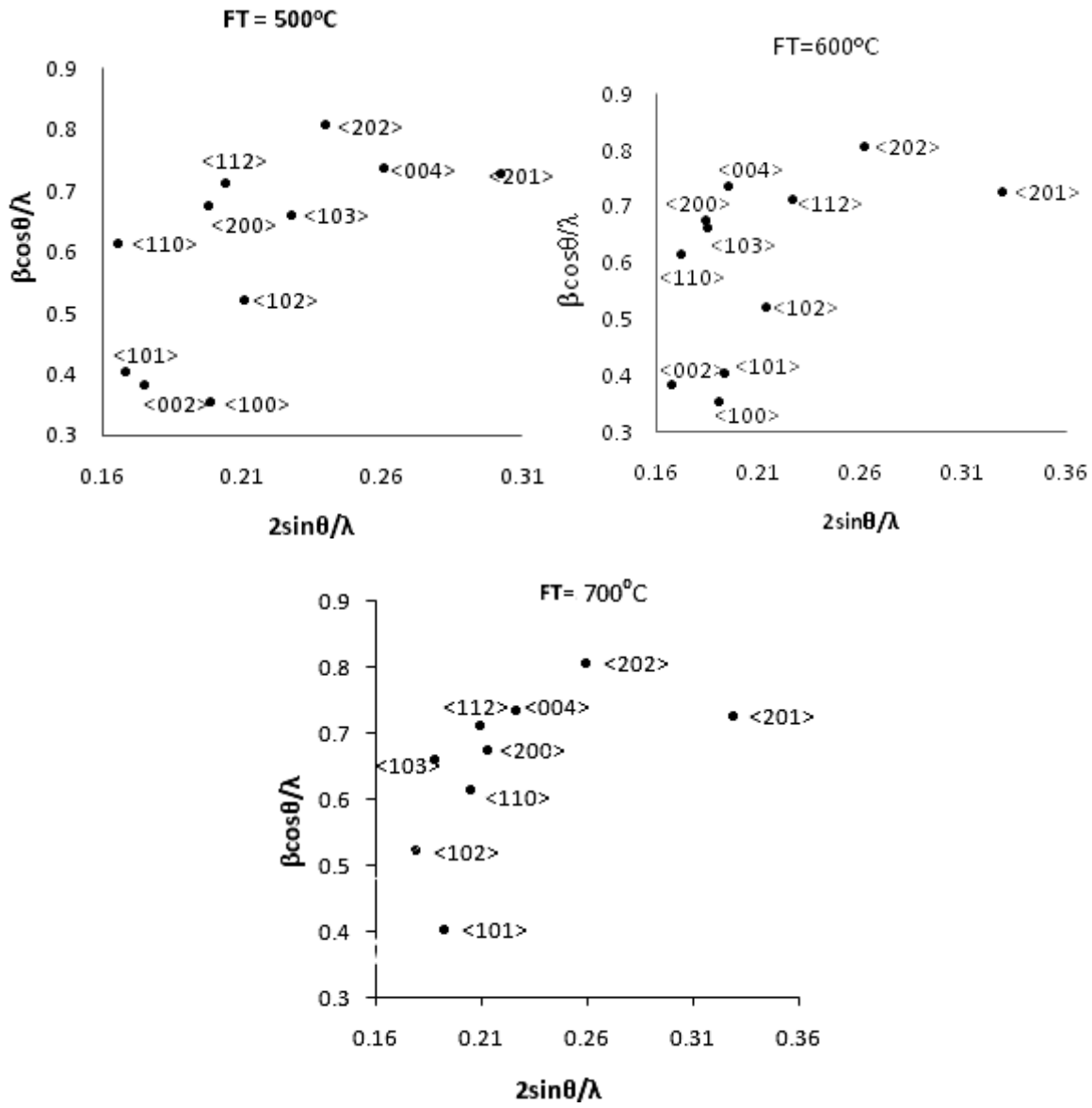


Fig. 6. Williamson-Hall plot to determine grain size and microstrain of ZnO TFRs fired at different temperatures.

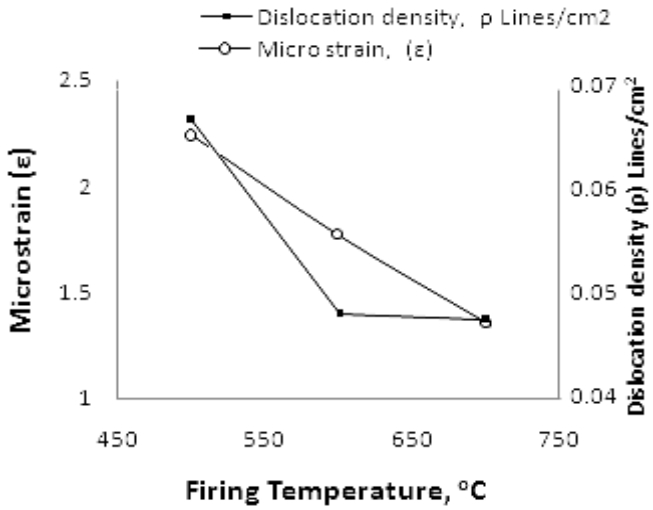


Fig. 7. Variation of micro strain and dislocation density with firing temperature.

3.4.1.4. Stacking Fault Probability

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 [67]. From the XRD patterns of ZnO films, the peak shift Δ(2θ) for the oriented (101) plane was observed with a change in firing temperature. The stacking fault probability (α) was calculated using equation-6 [66] at different firing temperatures.

$$\alpha = \frac{2\pi^2 \Delta(2\theta)}{45\sqrt{3}\,\tan\theta} \tag{6}$$

The stacking fault probability decreases with an increase in the firing temperature. This leads to a reduction in the concentration of lattice imperfections [58, 68] in the ZnO TFRs, indicating less defect/dislocations in the film as firing temperature increases as shown in Fig. 8. A similar trend has been reported by T. Mahalingam et al [66] for electrodeposited ZnO thin films.

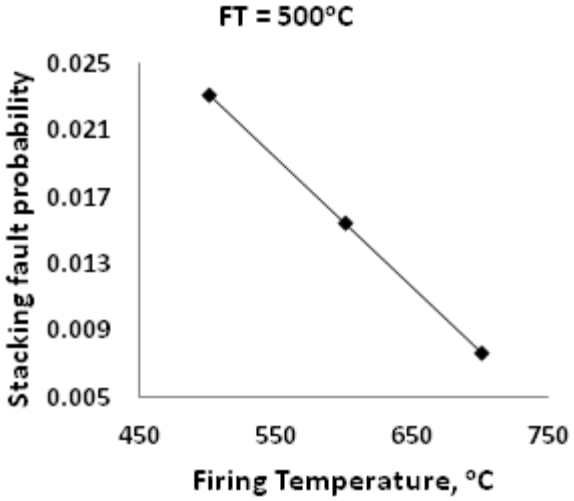
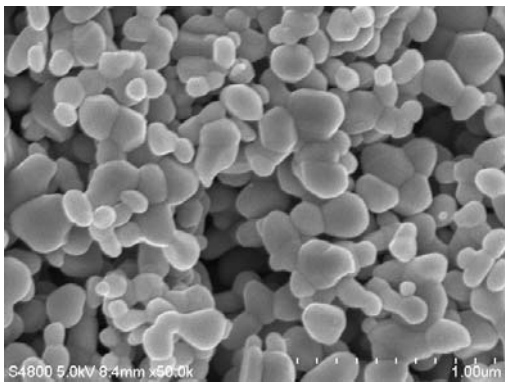


Fig. 8. Stacking fault probability versus firing temperature.

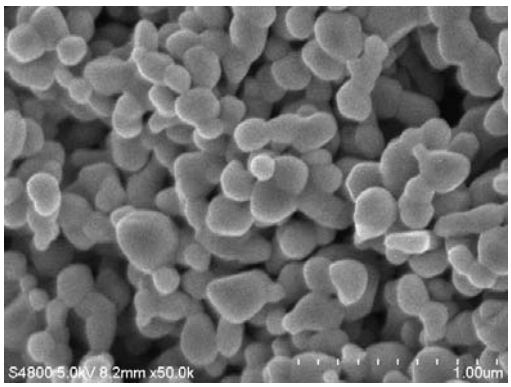
3.4.2. SEM Analysis

SEM images of Fig. 9 show that all of the films are porous with small grains (sub micron). The films fired at 500 and 600 ^{o}C are more porous as compared to film fired at 700 ^{o}C. The grain size is little bit larger as the increase of firing temperature although the increase is not significant. All the SEM images are recorded at 50 k magnification for comparison.

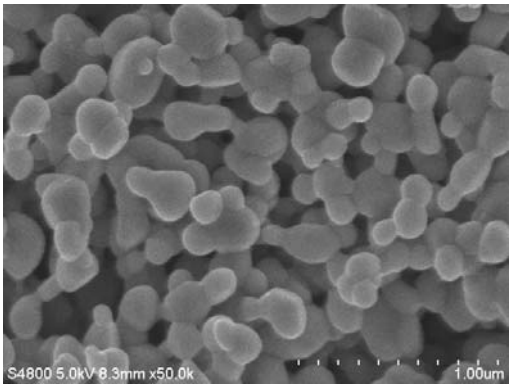
The microstructure consists of primarily irregularly shaped of 1 to 6 μm aggregates of fine particles oriented randomly leading to a moderate porosity. The material is characterized by some intergranular porosity (about 20 %). Some large open pores of several micrometers in diameter and small open pores of several tens of nanometers in diameter are present in sample. Microstructure of TFR fired at 600 ^{o}C shows that some necks are formed within the structure. Agglomeration of small crystallites also seems to be present in the certain region. The contrast difference is due to different orientations of the crystallites. At 500 ^{o}C firing temperatures the surface morphology of the ZnO film showed individual grains clearly and the grain size enlarged above firing temperature of 500 ^{o}C.



(a) SEM image of ZnO TFR fired at 500 ^{o}C



(b) SEM image of ZnO TFR fired at 600 ^{o}C



(c) SEM image of ZnO TFR fired at 700 ^{o}C

Fig. 9. SEM images of ZnO TFRs fired at different temperatures.

The microstructure of ZnO TFR fired at 700 ^{o}C shows the formation of submicrometer crystallites distributed more or less uniformly over the surface with very small number of open pores and several particles connected with each other and shows the strongly agglomerated structure with neck growth. The TFR fired at 700 ^{o}C also reveal lower surface porosity, large particle size and smaller specific surface to volume ratio than the films fired at 500 and 600 ^{o}C. As seen in micrographs, the grain size increases with the firing temperature. The number of particles which has hexagonal structure increased with increasing firing temperature. The grain size in ZnO thick film resistors increases with increasing firing temperature**.**

Table 3 illustrates the variation of Microstructural parameters calculated at different firing temperatures.

Table 3. Microstructural parameters of ZnO TFRs fired at different temperatures.

Firing Temp. (^{o}C)	**Grain size, (from XRD) D, (nm)**	**Grain size (from SEM) D, (μm)**	**Lattice Constants (101)**		**Texture Coeff. T_C(101)**	**Micro strain, ε**	**Dislocation density, ρ (Lines/cm^2)**	**Stacking Fault probability, α**
			a (nm)	**c (nm)**				
500	46.48	1.25	0.3241	0.5293	2.3713	2.237	0.0670	0.02322
600	51.47	1.69	0.3240	0.5291	2.9790	1.771	0.0482	0.01548
700	53.39	2.063	0.3238	0.5288	3.0896	1.363	0.0476	0.007736
ASTM			**0.3249**	**0.5205**				

4. Conclusions

ZnO thick film resistors can be deposited by a screen printing method on alumina substrates. The structural and morphological properties of the ZnO TFRs were influenced by firing temperature. XRD pattern confirms polycrystalline wurtzite ZnO with a preferential orientation along (101) plane. Grain size and microstrain is obtained using Williamson–Hall plot method. As firing temperature increases grain size increases and microstrain decreases. It is observed that the texture coefficient in the ZnO thick film resistors increases along the (101) direction with the firing temperature. SEM images indicated that the TFR fired at 700 °C has the highest crystallite size.

Acknowledgements

The authors are thankful to Management authorities of M. G. Vidyamandir, M. S. G. College Malegaon (Nasik), for providing all the required facilities for doing the work and also like to thank the authorities of Physics Department, Pune University and NCL Pune for cooperation in the field of XRD and Scanning electron microscopic analysis respectively. The authors also gratefully acknowledge the financial support from the University Grant Commission, New Delhi.

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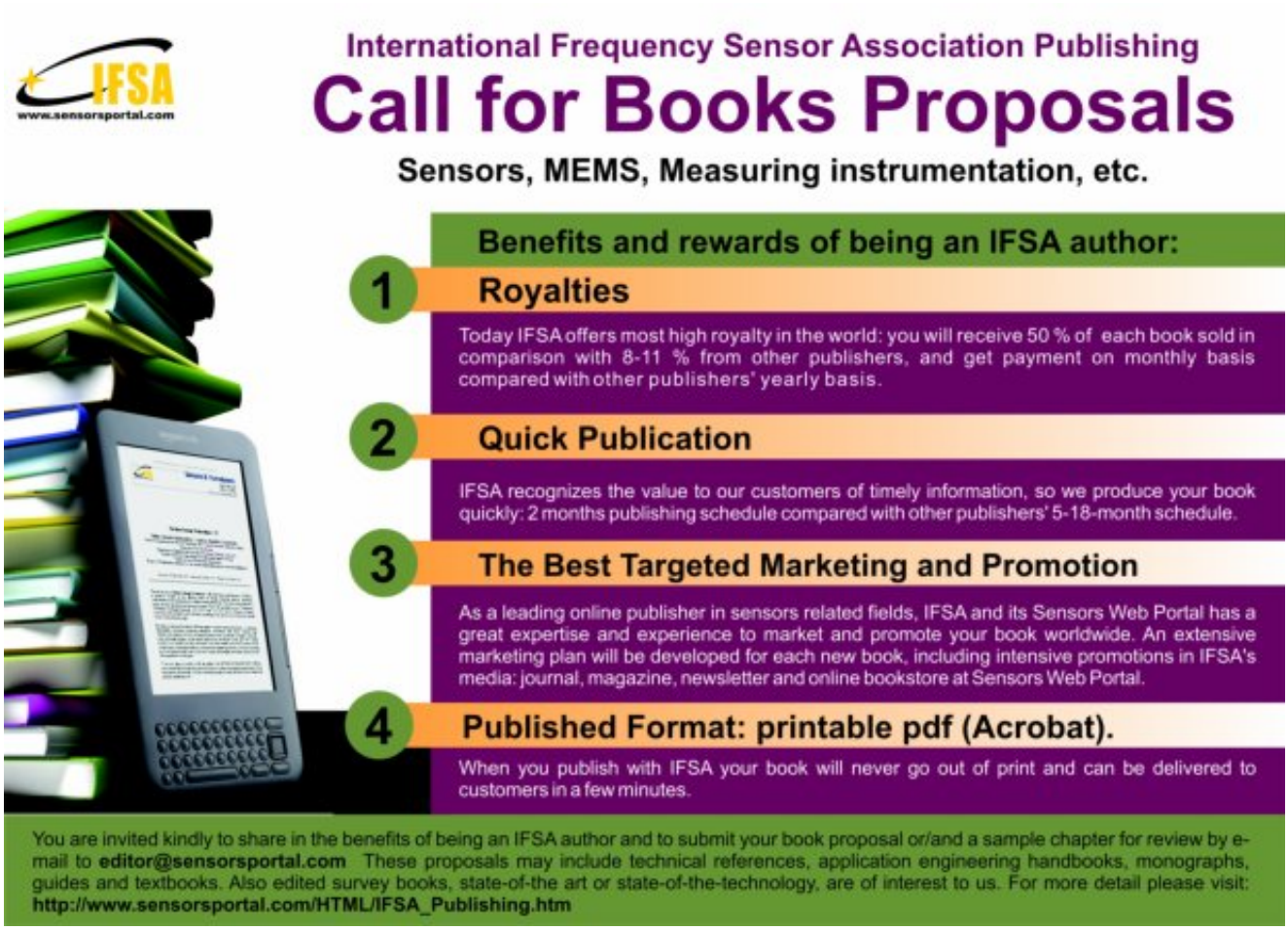
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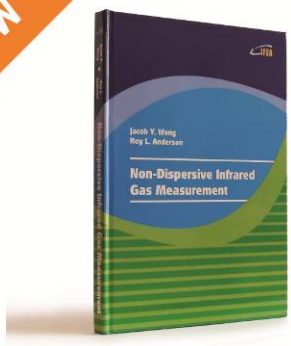
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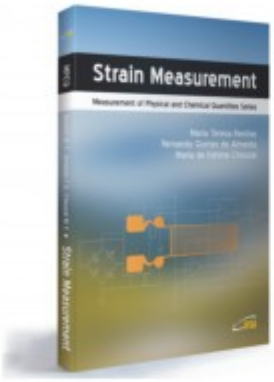
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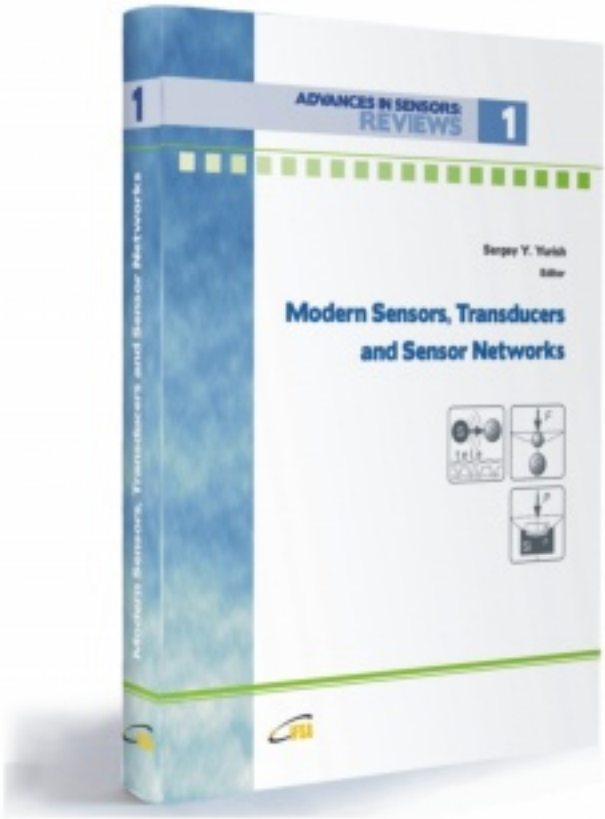
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