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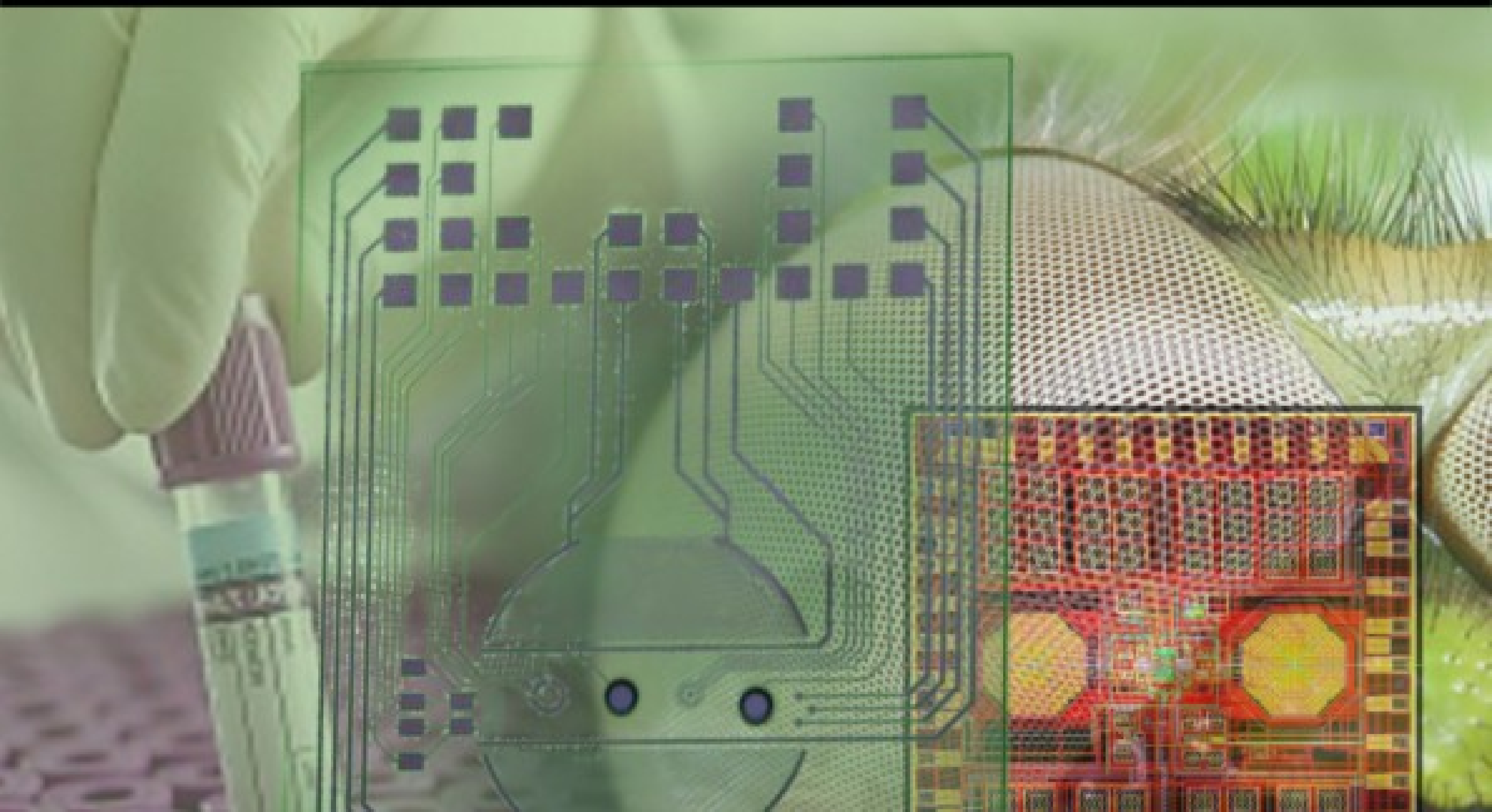

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Contents

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Effect of Firing Temperature on the Composition and Micro Structural Parameters of Screen Printed SnO_2 Thick Films Resistors

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Abstract: Tin Oxide (SnO_2) thick films were prepared on alumina substrate by using standard screen printing technique. Films were fired at different temperatures between 580 ^oC to 780 ^oC for 30 minutes in air atmosphere. The Morphological, Compositional and Structural properties of the SnO_2 thick films were performed by X-ray diffraction (XRD) technique, Scanning electron microscopy (SEM), Energy dispersive spectroscopy (EDX), Fourier transformation Infrared spectroscopy (FTIR) and Ultra visible spectroscopy. Structural characterization indicated the formation of polycrystalline SnO_2 films with tetragonal structure with preferred orientation along [211] plane. The X-ray line profile analysis has been used to evaluate the structural parameters such as crystallite size, texture coefficient, microstrain, and stacking fault probability. The effects of firing temperature on the structural parameters were analyzed. The crystallite size changes from 21 nm to 31 nm with increase firing temperature. Composition of Sn and O changes with firing temperature indicating non-stotiometric behavior of SnO_2 films. IR spectroscopy analysis at 2949.26 cm^{-1} showed the peak assigned to the –Sn-H vibration due to the effect of hybridization i.e. sp^3 and the sharp peak at 3734.31 cm^{-1} assigned to –Sn-OH stretching vibration due to hydrogen bonding. Optical absorption spectra of SnO_2 powder revealed that SnO_2 exhibits a shoulder at 202 nm along with an ill-defined band at 235 nm. *Copyright © 2010 IFSA.*

Keywords: SnO_2, Thick films, Texture coefficient, Microstrain, SEM, XRD

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1. Introduction

Over the past two decades, solid-state gas sensors based on SnO_2, have become the predominant solid state devices for gas alarms. They are used for domestic applications and a wide variety of ceramic, thick film and thin film devices have been developed. Recent [1] investigators indicate that, nanosize SnO_2, is a promising candidate for the sensitive and long term stable detection of pollutant gases. The current trend of gas sensor technology requires continuous device miniaturization, low power consumption at usual working conditions device technology that is integrated circuit (IC) compatible to allow batch fabrication and reduction of single-device cost [2] Polycrystalline SnO_2 thick film is commonly investigated for gas sensor technology. Despite the amount of research completed to-date, some major-problems related to material; properties. An attempt has been made to contribute to solving problems by annealing at different temperatures and ambient, adding suitable dopant species to SnO_2 materials films.

SnO_2 is an anisotropic polar crystal, which crystallizes with tetragonal rutile structure. The unit cell contains six atoms, two tin and four oxygen. Each tin atom is at the center of six oxygen placed approximately at the corners of a regular slightly deformed octahedron and three tin atoms approximately at the corners of an equilateral triangle surround every oxygen atom[3]. It is an n-type semiconductor attracting tremendous attention due to its interesting properties like wide band gap of 3.57 eV at room temperature with large excitation binding energy, high chemical stability, low dielectric constant, large electrochemical coupling coefficient and high luminous transmittance [4, 5]. It is widely used in gas sensor application, varistors, surface acoustic wave devices, photovoltaic, electric transducers, piezoelectric materials and room temperature ultraviolet lasing [6-8]. Therefore it can be applied extensively to IT (Information tech.), BT (Bio-tech.), ET (Environmental tech.) [9]. Several deposition methods have been used to grow undoped and doped SnO_2 films such as Evaporation, chemical vapour deposition, spray pyrolysis, magnetron sputtering, pulsed laser deposition, sol-gel technique, screen printing technique, Pellet technique [10-12]. Screen printing technique was introduced in the later part of 1950's to produce compact, robust and relatively inexpensive hybrid circuit for many purposes. Later on thick film technique has attracted by the sensor field [13]. Screen printing is viable and economical method to produce thick films of various materials [14].

The main aim of the present work is to focus on the structural and surface morphological properties of SnO_2 thick films. We report the studies on structural parameters of SnO_2 thick films at different firing temperature. Such a detailed understanding of film properties are necessary if SnO_2 is to be developed to a degree which will enable its use in sensing devices.

2. Experimental Methods

2.1. Powder and Paste Preparation

Analar grade SnO_2 powder (99.9 %, Loba chem) was calcinied at 400 ^{0}C for 2hr in muffle furnace. Then this powder was crushed and thoroughly mixed with a glass frit (70 wt. % PbO, 18 wt. % SiO_2, 3 wt. % Al_2O_3, 3 wt.% B_2O_3 and 6% TiO_2) as a permanent binder. Organic vehicles ethyl cellulose (EC) and butyl carbitol acetate (BCA) were added to this mixture to achieve proper thixotropic properties of the paste. The ratio of inorganic to organic part was maintained as 70:30. (The ratio of active powder and permanent binder was kept at 95:5 in 70 % and ratio of EC –BCA was 92:8 in 30 %)

2.2. Thick Film Preparation

Thixotropic paste of SnO_2 was used to deposit thick films on alumina substrate (96 % pure) by using standard screen printing technique with 40 s mesh no.355.The details of the technique are described elsewhere [15,16]. The films were dried under IR radiation for 60 minutes in an ambient and fired at peak temperatures of 580, 680 and 780 ^{o}C for 30 minutes in muffle furnace.

2.3 Thickness Measurements

The thickness of the SnO_2 thick films was measured by using Taylor-Hobson (Taly-step UK) system. The thickness of the films was observed to be uniform in the range of 12-16 μm.

2.4. Material Characterization

The SnO_2 thick films were characterized by X-ray diffraction technique from 20-80^{0} [Miniflex model, Rigaku-Japan, DMAX-2500, CuKα (λ=1.542AU) radiation] with a 0.1^{O}/step (2θ) at the rate 2 s/step. The average crystallite sizes of SnO_2 thick film samples were determined by using Debye Scherer formula [17]

$$D = \frac{0.94\lambda}{\beta \cos\theta}, \tag{1}$$

where D is the grain size, λ is the wavelength of the X-ray radiation (1.542 Å), θ is the angle of diffraction and β is the full angular width of diffraction peak at the half maximum peak intensity.

The effect of the firing temperature on the orientation of the films was investigated by calculating the texture coefficient using the following equation-2 [18-20],

$$T(hkl) = \frac{I(hkl)/I_o(hkl)}{(1/N)\sum I(hkl)/I_o(hkl)}, \tag{2}$$

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.

Root mean square (RMS) microstrain (e) can be determined using a Williamson and Hall plot. The slope of the plot of (βcosθ/λ) versus (2sinθ/λ) gives the value of the RMS micro strain. The stacking fault probability was calculated using the following equation:

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

A scanning electron microscopy (SEM- JOEL JED-2300, Japan) was employed to characterize the surface morphology. The composition of SnO_2 thick film samples were analyzed by an energy dispersive X ray spectrometer (EDX) (JOELJED 6360 LA).

3. Results and Discussion

3.1. Calcinations, Drying and Firing of the Films

The calcinations 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 calcinations, grain boundaries are developed and the powder sinters to bigger agglomerates. This causes a higher surface area after firing and therefore a higher sensitivity of a layer. This powder was milled after calcination. The calcination took from 1 h to 10 h [21]. 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 70-100 ^{o}C by placing films under infrared radiation [21].

The high temperature firing cycle is designed to remove the remaining organic binders, to develop the structural and electrical properties of the film and bond the film to the substrate. 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 ^{o}C. As the temperature reached 580-780 ^{o}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 [21].

3.2. Chemical Composition

Table 1 shows the chemical composition of the films fired at different temperatures. The EDX spectrum showed the presence of only Sn and Oxygen. The mass percentage of Sn was found to be a maximum at 680^{o}C firing temperature and is due to release of excess oxygen [22]. From the analysis it was found that the SnO_2 films are nonstoichiometry. The deficiency or excess of any type of atom in the crystal results in a distorted band structure, with a corresponding increase in conductivity. Tin oxide looses oxygen on heating so that tin is then in excess. The oxygen, of course, evolves as an electrically neutral substance so that it is associated with each excess tin ion in the crystal; there will be two electrons that remain trapped in the solid material, thus leading to nonstoichiometry in the solid. This leads to the formation of the n-type semiconductor [23].

Table 1. Composition of the SnO_2 films at different firing temperature.

Firing Temperature	Element	At. %	Mass %
580 ^{o}C	Sn	50.23	88.22
	O	49.77	11.78
680 ^{o}C	Sn	53.97	89.69
	O	46.03	10.31
780 ^{o}C	Sn	53.65	89.57
	O	46.35	10.43

3.3. Structural Parameters and their Analysis

Fig. 1 shows X-ray diffraction patterns of SnO_2 thick films deposited on alumina substrates and fired at 580 °C, 680 °C and 780 °C. XRD pattern show the different peaks corresponding to SnO_2, SnO, Sn_3O_4 and Sn_2O_3 phases. It has been observed that [211] reflections corresponding to $2\theta = 52.200^o$ are of maximum intensities for all film samples thereby a strong orientation with stacking of the plane along the c-axis, which indicates SnO_2 film had preferred orientation in the direction of [211] plane. In addition two reflections at $2\theta = 58$ and 77.400^o corresponding to Al_2O_3 are also observed in all SnO_2 thick film samples. At reflection $2\theta = 35.600^o$ corresponding to SnO_2 [021] phase is observed. The intensity of [021] peak corresponding to SnO_2 phase increases from 680 to 780 °C firing temperature and is absent in film sample fired at 580 °C. The intensity of Sn_3O_4 phase decreases with increase in firing temperature. The observed 'd' values are found to be close to the JCPDS file for tetragonal SnO_2 [24]. This clearly indicates that the structure of SnO_2 films is polycrystalline in nature. Table 2 illustrates the presence of % relative phases of SnO_2 thick films [25].

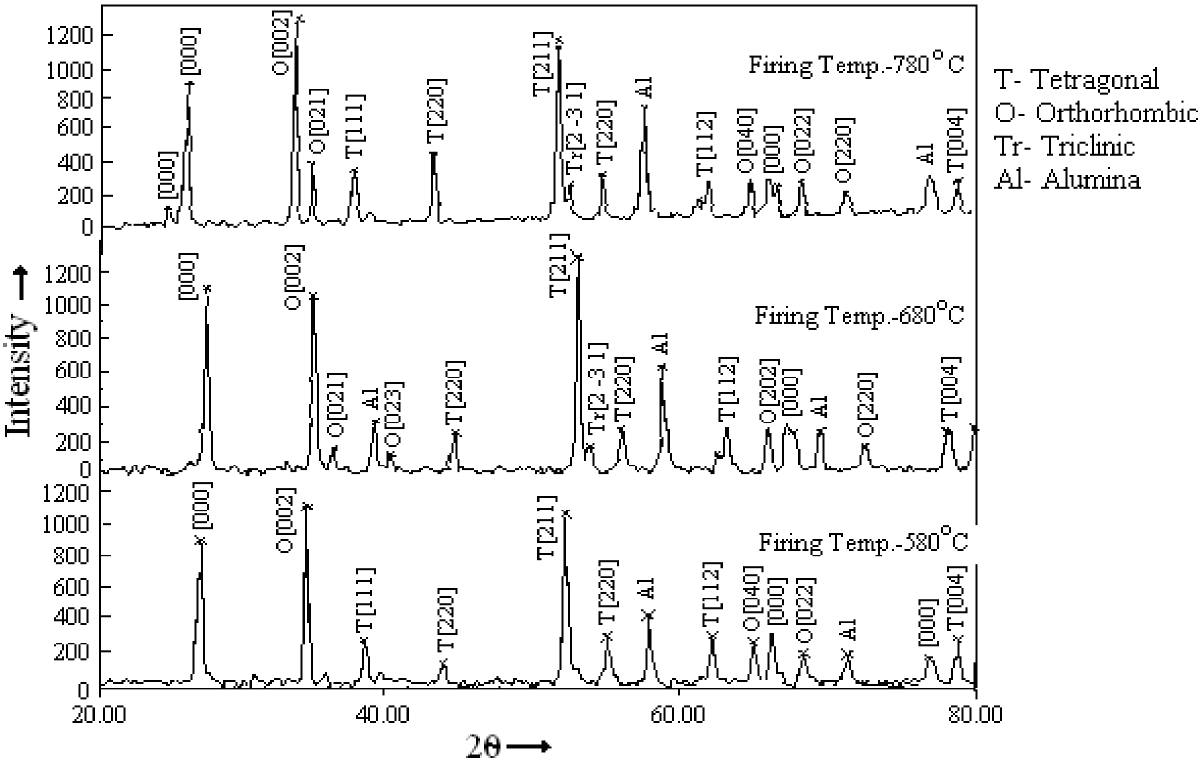


Fig. 1. X-ray diffraction pattern.

Table 2. Presence of % relative phases of SnO_2 thick films.

Firing Temperature (°C)	%Relative Phases of SnO_2				
	SnO_2 (Tetragonal)	SnO (Orthorhombic)	Sn_3O_4	Sn_2O_3	Al_2O_3
580	32	34.82	22.00	1.937	9.248
680	32.98	26.92	19.139	2.172	18.79
780	34.38	29.38	17.07	2.928	16.24

3.3.1. Crystallite Size (D)

The XRD pattern was used to calculate the crystallite size of SnO_2 by using Debye Scherrer's formula [17]. The average crystallite sizes of SnO_2 films are 21, 25 and 31 nm (±2 nm) at 580 °C, 680 °C and 780 °C (±2 °C) respectively.

3.3.2. Texture Coefficient (T_C)

From the values calculated using equation-2, 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 [26]. 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 [211], [002] and [220] planes. From Fig. 2 it has been observed that the preferred orientation is the [211] plane for all firing temperatures. The increase in preferred orientation is attributed to an increased number of grains along the plane. From the variation of texture coefficient with firing temperature for [211] planes, it is seen that at firing temperature of 680°C, the texture coefficient Tc is maximum for SnO_2 thick film samples.

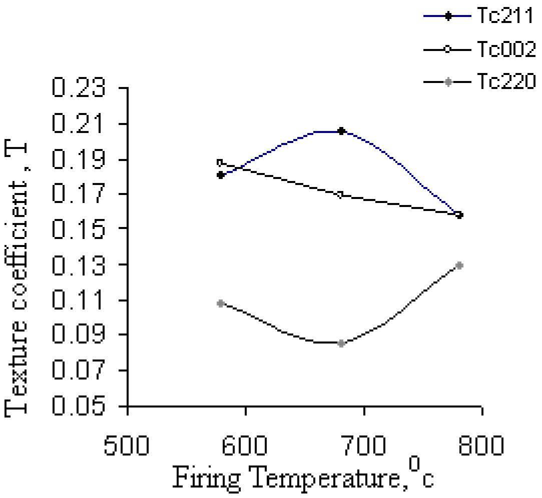


Fig. 2. Shows the variation of the texture coefficient with firing temperatures.

3.3.3. Microstrain (e)

Broadening of X-ray diffraction line profiles is mainly caused by non-ideal optics of the instrument, wavelength dispersion, and micro structural imperfections in the crystals. The micro structural 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 (macro strain) 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 (micro strain). A method to determine the micro strain by analyzing X-ray diffraction lines is described by Williamson and Hall [27, 28, 29]. 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 micro strain. Fig. 3 shows the Williamson-Hall plots for SnO_2 thick films with different deformations at different firing temperatures. The points in the W-H plot are scattered. Therefore micro strain 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 micro strains calculated at different firing temperatures indicates a decreasing trend with firing temperature as shown in Fig. 4. The RMS micro strain values calculated at different firing temperatures are given in Table 3.

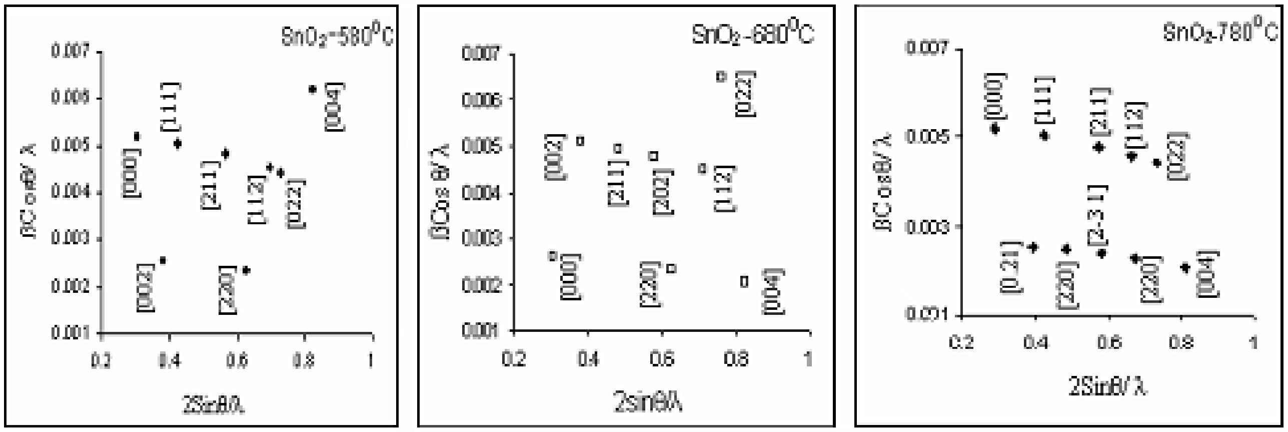


Fig. 3. Williamson-Hall plots for SnO_2thick films with different deformations at different firing temperatures.

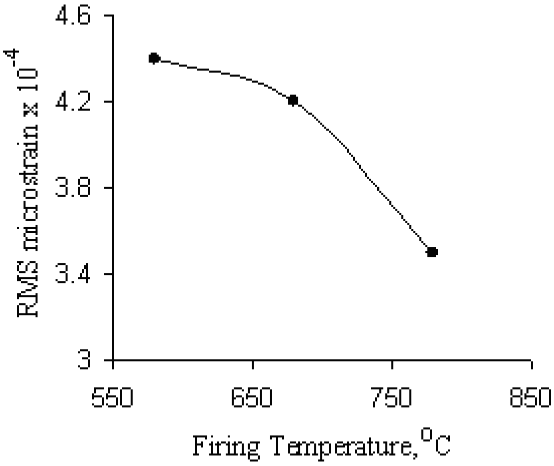


Fig. 4. Plot of micro strain versus firing temperature.

Table 3. Microstructural Parameters of SnO_2 thick films fired at different temperatures.

Firing temperature, (ºC)	average grain Size (D), nm (XRD)	Particle size (D),nm (SEM)	Texture Coefficient, T_c			RMS Microstrain, (ε) x 10^{-4}	Stacking fault Probability, (α) for [211]
			[211]	[002]	[220]		
580	21 nm ±2 nm	152 ±2 nm	0.1801	0.1876	0.1088	4.4	0.0492
680	25 nm ±2 nm	194 ±2 nm	0.2057	0.1687	0.0864	4.2	0.0822
780	31 nm ±2 nm	226 ±2 nm	0.1579	0.1579	0.1295	3.5	0.0497

3.3.4. 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 [30]. From the XRD patterns of SnO_2 films, the peak shift $\Delta(2\theta)$ for the oriented [211] plane was observed with a change in firing temperature. The stacking fault probability was calculated using equation-3 [31] at different firing temperatures. The stacking fault probability is large for the SnO_2 film fired at 680 °C, indicating more defect/dislocations in the film as shown in Fig. 5.

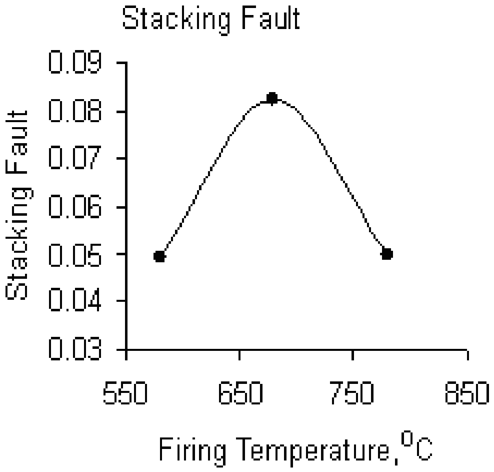


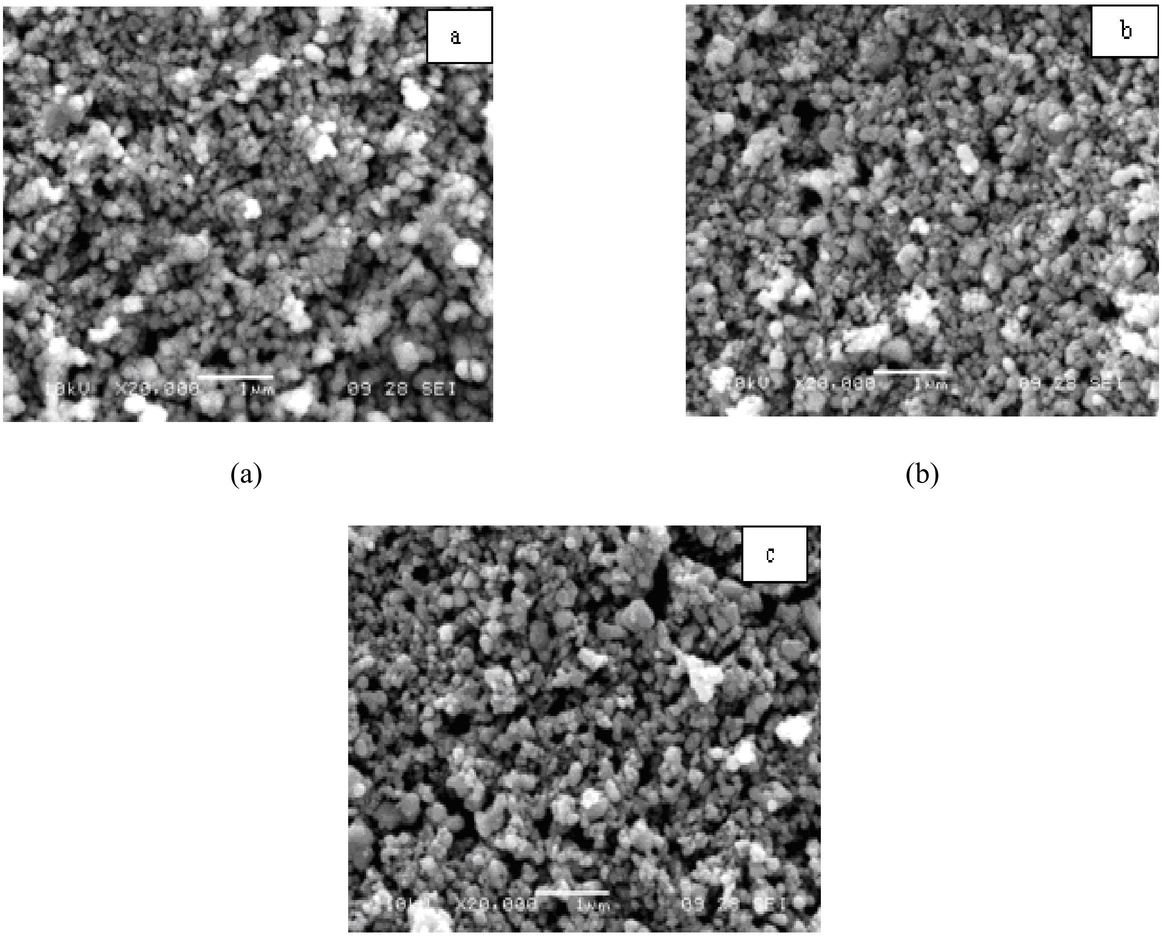
Fig. 5. Variation of stacking fault probability as function of firing temperature.

3.4. SEM Analysis

Surface morphology of SnO_2 thick films at different firing temperature were analyzed using scanning electron microscope (JOEL JED 2300(LA) Japan). Fig. 6-(a), (b) and (c) show SEM images of SnO_2 thick films fired at 580 °C, 680 °C, 780 °C respectively. The micrograms show uniform polycrystalline nature of the films indicating increased particle size with firing temperature. The micrograms show voids between the particles basically due to evaporation of the organic solvent during the firing of the films. The firing temperature increases the atomic mobility. The atoms can be moved to more energetically favoured sites such as voids, grain boundaries and interstitial positions. An increase in firing temperature increases the mobility of atoms at the surface of films and thus improves the crystallinity. The particle sizes of SnO_2 thick films are 152 nm, 194 nm and 226 nm (±2nm) at 580 ^{0}C, 680 ^{0}C and 780 ^{0}C (±2 ^{0}C) respectively. Fig. 6 shows that variation of Grain/ particle sizes with different firing temperature. It is seen that the value of particle size observed by the SEM is greater than the value of average grain size observed by the XRD. The higher value observed by SEM may be due to the agglomeration of the grains during observation under SEM as electron probe is high energy probe compared to that of XRD.

3.5. Fourier Transformation Infrared Spectroscopy Studies

FTIR spectroscopic measurements of pure SnO_2 powder sintered at 400 ^{0}C in air has been studied in the wave number range between 400-4000 cm^{-1}, using PC Shimadzu spectrophotometer. Fig. 7 shows that FTIR spectra of pure SnO_2 powder sample. It is seen that the broad band peak assigned in aromatic region to the –Sn-O vibration were observed at 675cm^{-1} which shows maximum absorption. Broad band with intensive peak at 1454.38 cm^{-1} bending at 1430 cm^{-1} assigned to O-Sn-O vibration was observed. This shows 38 % of transmittance [As reported value of pure SnO_2 in –C-H vibration is 1456 cm^{-1}]. The stretching peak was assigned to the –Sn-O stretching vibration at 1514.17 cm^{-1} was observed to be sharp and intense which shows 37 % of transmittance bending at 1500 cm.$^{-1}$ which indicates the formation of O_2 adsorption on SnO_2 Surface. A doublet at 2333.94-2359.02 cm^{-1} assigned to Sn-H stretching vibration and bending at 2264.51 has been identified and it requires more energy for maximum absorption. Weak band peak at 2949.26 cm^{-1} assigned to the –Sn-H vibration was observed due to the effect of hybridization. The sharp peak at 3734.31 cm^{-1} assigned to –Sn-OH stretching vibration due to hydrogen bonding was observed.



(a) (b)

(c)

Fig. 6. Scanning Electron Micrograms of SnO_2 thick film samples fired at (a) 580 (b) 680 and (c) 780 ^{o}C.

3.6. Optical Studies

UV-Vis spectra were carried out on the Shimadzu UV-2400pc. Fig. 8 (a):-shows the optical absorbance spectra of SnO_2 powder sintered at 400 ^{0}C in the wavelength range 200–1000 nm. From figure it is seen that the UV spectrum of SnO_2 exhibits a shoulder at 202 nm along with an ill-defined band at 235 nm. The variation of optical density with wavelength was analyzed to find out the nature of transition involved and the optical band gap.

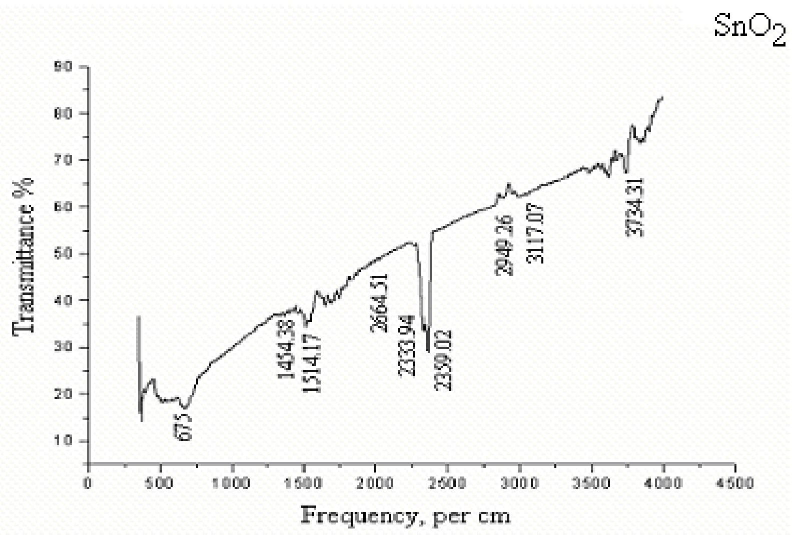
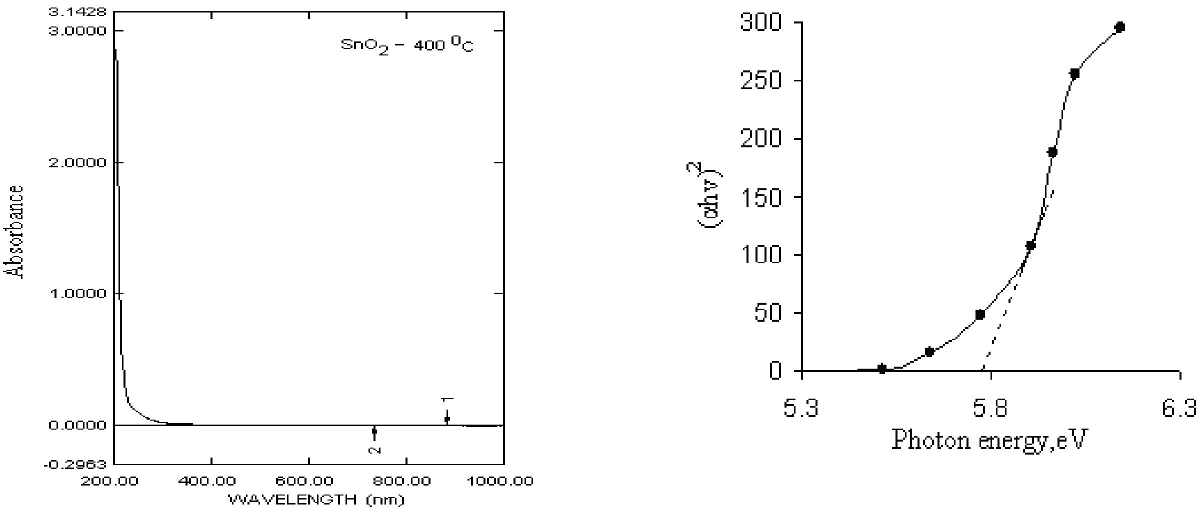


Fig. 7. FTIR spectra of pure SnO_2 powder sample.



(a)

(b)

Fig. 8. (a) Absorption spectra of SnO_2 powder sintered at 400^0C (b) Plots of $(\alpha h\nu)^2$ against hν for different SnO_2.

The wavelength at which 'α' rises suddenly corresponds to the band gap energy that was estimated. The absorption coefficient is found to be 2.8 supporting the presence of direct band gap. [32] The nature of the transition is determined by using the relation-4 [33]

$$\alpha = A\,(h\nu - E_g)^{1/2}/h\nu\,, \qquad (4)$$

where E_g is the optical band gap energy, A is a constant.

The plot of $(\alpha h\nu)^2$ as a function of the energy ($h\nu$) of the incident radiation has been shown in Fig. 8 (b). It is found that the plot is nonlinear indicating absence of indirect transition. The band edge can be evaluated from the intercept of the extrapolated linear part of the curve with the energy axis. The corresponding value of E_g is found to be 5.75 eV. The presence of direct band gap of SnO_2 has been reported with E_g = 3.57 eV [34]. The difference in present case may be attributed to the different mechanism and technique.

4. Conclusion

SnO_2 thick films were prepared on alumina substrate by standard screen printing technique which is simple and inexpensive. From EDAX and SEM it was confirmed that SnO_2 films were non-stoichiometric, which are suitable for sensing application. XRD analysis shows the structure of SnO_2 films is polycrystalline in nature having improved crystnallity with increase in firing temperature. The structural parameters of SnO_2 films have been evaluated. The RMS microstrain decreases with increase in firing temperature. IR spectroscopy analysis at 2949.26 cm^{-1} the peak assigned to the –Sn-H vibration due to the effect of hybridization i.e. sp$^{3.}$ The sharp peak at 3734.31 cm^{-1} assigned to –Sn-OH stretching vibration due to hydrogen bonding. From optical studies, the direct band gap value was found to 5.75 eV. The UV spectrum of SnO_2 exhibits a shoulder at 202 nm along with an ill-defined band at 235 nm.

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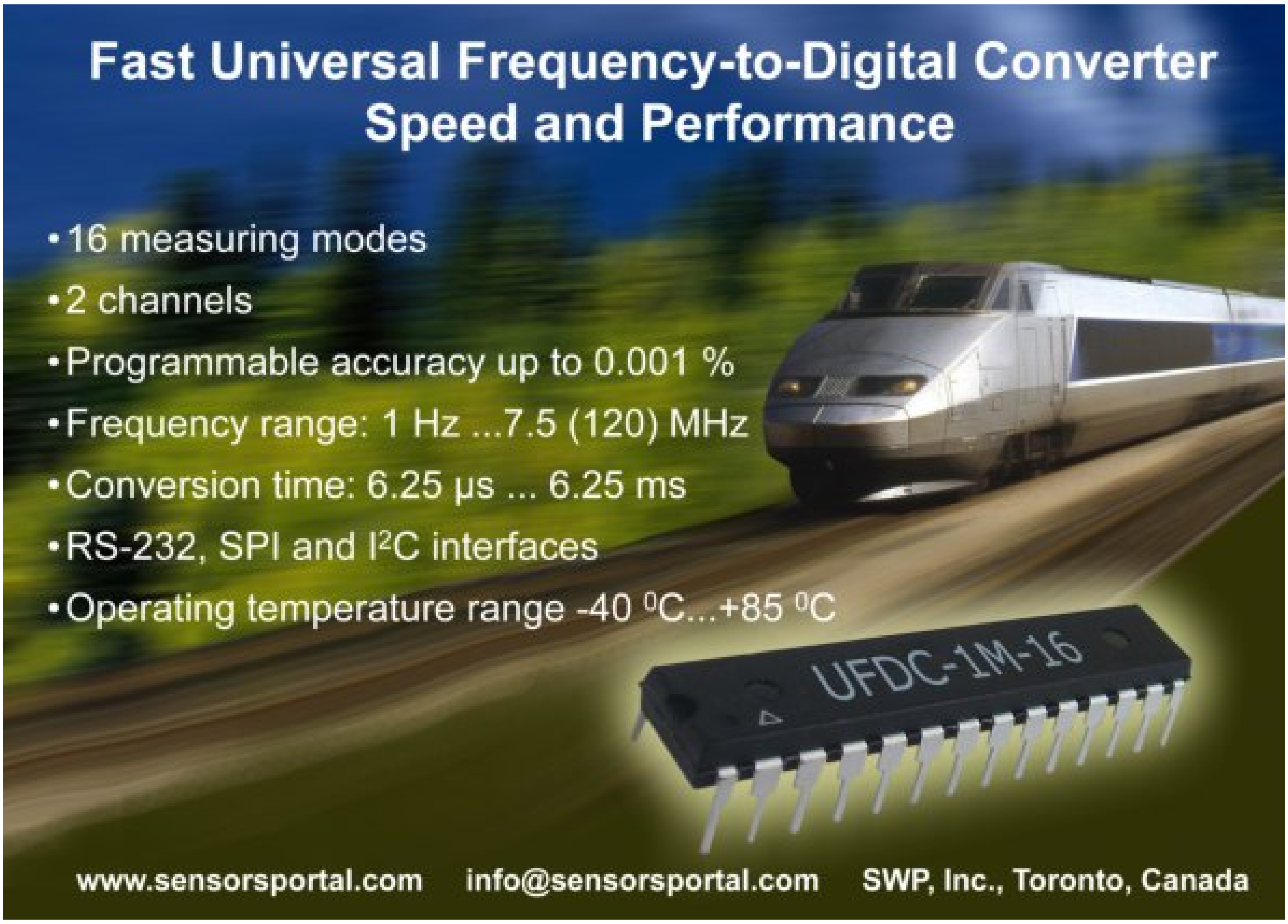

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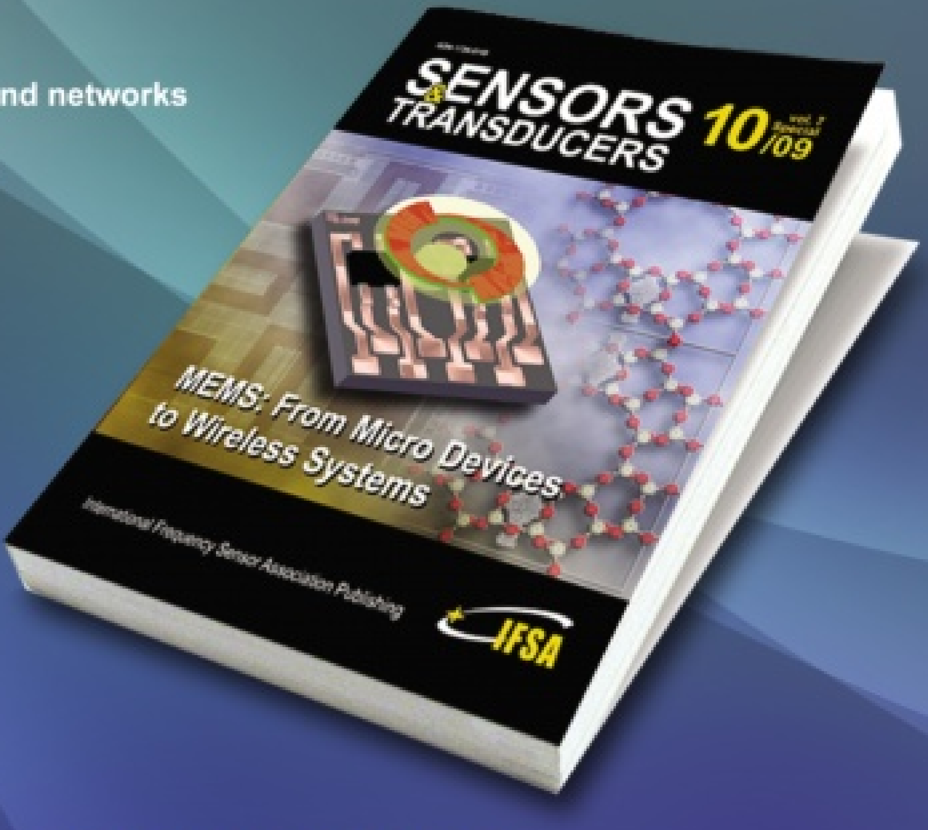
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