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Study on the suitability of ZnO thin film for dynamic pressure sensing application

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Abstract

ZnO thin films were prepared by RF reactive magnetron sputtering on phynox substrate at room temperature for four different thicknesses by varying deposition duration. The structural and morphological properties and composition of these films were characterized using XRD, SEM, and EDS, respectively. Suitability of these films for dynamic pressure sensing applications and the effect of film thickness on dynamic pressure sensing were evaluated experimentally using a shock tube equipment. Shock tube test results show the pure dynamic behavior of ZnO films with fast rise and discharge. Sensors with higher film thickness showed improved sensitivity which is on par with commercially available dynamic pressure sensors. This work demonstrates that the cost-effective sensors based on ZnO thin film are capable of sensing dynamic pressures for different pressure ranges.

Keywords
ZnO, Thin film, Sputtering, Dynamic pressure sensor, Shock tube.

Rapid pressure variations with respect to time need to be measured in many fields such as aerospace (engine combustion studies, acoustics, shock waves, ballistics, etc.), automotive (airbag deployment), biomedical (arterial pressure pulse), etc., with demanding requirements which are completely different compared with those for static pressure measurements. Dynamic pressure sensors are used for precise measurement of time-varying pressure signals. Various technologies such as piezoresistive type (MEMS), optical methods, and piezoelectric effect are proven technologies for dynamic pressure measurement. Piezoresistive-type MEMS-based sensors offer higher output, low hysteresis, and miniaturization (Tran et al., 2018; Sidhick et al., 2007). However, sensors based on this technology suffer from drift due to temperature, have lesser bandwidth and low natural frequencies and need external voltage or current excitation (Fraga et al., 2014). Different fiber-based optical methods, such as single-mode fiber (Gan et al., 2008), fiber Bragg grating (FBG) (Sharath et al., 2014; Li et al., 2016), long-period fiber grating (LPFG) (Fu et al., 2016), etc., are being employed in optical-based dynamic pressure sensors. Though optical fiber-based pressure sensors are having advantages such as immunity to EMI, chemical inertness, higher bandwidth, etc., they have the disadvantages such as complex and bulky signal conditioning circuits (Interrogator), ageing of light source, etc. Sensors based on piezoelectric technology are being employed for dynamic pressure sensing due to the advantages such as self-generating, faster response, higher sensitivity, higher bandwidth, and low cost (Zarfl et al., 2016; Sirohi and Chopra, 2000; Wang and Qin, 2010). Because of these attractive features, sensors based on piezoelectric technology are most widely used for dynamic pressure sensing applications.

One of piezoelectric materials which attracted researchers in recent days is ZnO which is a biocompatible semiconductor type smart material with desirable properties such as suitability for harsh environment, high ‘d’ coefficient and high Young’s modulus, etc., making it suitable for pressure sensing applications (Fraga et al., 2014; Li et al., 2007; Jagadish and Pearton, 2006). ZnO has simple fabrication processes, low-temperature processing (Wallace et al., 2015) and is compatible with various substrates. In our present work, we have chosen ZnO thin film as the piezoelectric material for realizing dynamic pressure sensor.

Various methods such as sputtering (Hoon et al., 2011; Kutepova and Hall, 1998; Xia et al., 2013; Yoon and Kim, 2006; Rajan et al., 2017; Munje et al., 2017; Nowek et al., 2016; Okada et al., 2013; Joshi et al., 2012; Joshi et al., 2013a; Joshi et al., 2013b; Alias et al., 2013; Chang et al., 2002; Bachari et al., 1999; Kang et al., 2015), Solgel (Foo et al., 2014; Nurulfadzilah et al., 2014; Chan et al., 2016; Md Sin et al., 2011; Gupta et al., 2016; Nithya and Radhakrishnan, 2012; Salah et al., 2015), spray pyrolysis (Jayaraman et al., 2015; Mariappan et al., 2014; Rao and Santhoshkumar, 2009), metal organic chemical vapor deposition (MOCVD) (Deschanvres et al., 1992; Shi et al., 2012; Su et al., 2011; Gorla et al., 1999), plasma enhanced atomic layer deposition (PEALD) (Gong and Jackson, 2017), pulsed laser deposition (Shim et al., 2002) are widely used for the preparation of ZnO thin film. Among these, sputtering has a faster deposition rate, better adhesion to substrate and can provide high purity films (Hoon et al., 2011; Alias et al., 2013). In the present work, RF reactive magnetron sputtering has been used to obtain good quality films with proper stoichiometry. Four samples of ZnO thin films with different thicknesses were prepared by varying deposition duration and these samples were characterized for composition, structural and morphological properties. Suitability of the prepared films for dynamic pressure sensing was evaluated using the shock tube test facility.

Experimental method

Deposition of ZnO thin films has been reported on various substrates such as glass (Hoon et al., 2011; Okada et al., 2013; Alias et al., 2013; Nurulfadzilah et al., 2014; Md Sin et al., 2011; Gupta et al., 2016; Nithya and Radhakrishnan, 2012; Salah et al., 2015; Jayaraman et al., 2015; Mariappan et al., 2014; Rao and Santhoshkumar, 2009), phynox (Joshi et al., 2012; Joshi et al., 2013a; Joshi et al., 2013b), silicon (Kutepova and Hall, 1998; Xia et al., 2013; Rajan et al., 2017; Chang et al., 2002; Kang et al., 2015; Deschanvres et al., 1992), sapphire (Gorla et al., 1999; Shim et al., 2002), polyamide (Munje et al., 2017), etc., by several researchers.

Since the present work is focused on dynamic pressure sensing, the substrate needs to act as the deflecting membrane. Hence, the selection of substrate should be such that the material needs to have high elasticity, high Young’s modulus, and high yield strength. These factors along with the necessity of ruggedness due to excessive vibration, transient peaks, pressure surge, etc., associated with dynamic pressure led to the selection of a flexible metallic substrate, namely, phynox (Joshi et al., 2012; Joshi et al., 2013a; Joshi et al., 2013b). In total, 50 µm thick phynox alloy foil cut into 25 mm × 7 mm strips were used as substrates in our present work. Since phynox is electrically conducting, it not only acts as a deflecting element but also as a bottom electrode, hence eliminates the need for the deposition of the separate bottom electrode.

Deposition and characterization of ZnO thin film

ZnO thin film deposition was carried out using RF reactive magnetron sputtering system (make: Hind High Vacuum (HHV) Pvt Ltd, India). Before loading the substrate into the chamber, it was cleaned thoroughly using the standard cleaning procedure. Impurities hideout in the pores were removed by ultrasonic cleaning for 10 min each in a soap solution, acetone and isopropyl alcohol followed by washing with De-Ionized (DI) water and drying. This process ensures the removal of organic and inorganic impurities. Target used is of sintered stoichiometric circular ZnO disc of 3-inch diameter × 3 mm thickness with 99.99% purity (make: Advanced Engineering Material, China).

The chamber was evacuated to an ultimate pressure of about 5×10^{-6} mbar. The 99.9% pure inert Argon gas followed by reactive gas oxygen (99.9% purity) was introduced into the chamber. Pre-sputtering of the target was carried out with a shutter in between target and substrate to avoid the contaminations and native oxide at the surface of the target getting sputtered on to the substrate surface. Later shutter was removed and the deposition of thin film on the substrate was initiated. Optimized sputtering process parameters are indicated in Table 1. The deposition was carried out for four different durations, i.e., 15, 30, 45, and 60 min with all other parameters being the same. Films were deposited on the substrate for an area of 20 mm × 7 mm. It is to be noted that entire film fabrication was carried out at room temperature and no annealing was carried out.

The sputtered samples (4 Nos) were studied for microstructure, morphology, film thickness and composition using the methods, namely, X-ray

Table 1. Sputtering parameters used for deposition of ZnO thin film.

Target – substrate distance	28 cm
Substrate temperature	24 ± 2°C
RF power	120 W
Ar-O_2 ratio	17.1–3.1 Sccm (85%–15%)
Ultimate pressure	5×10^{-6} mbar
Working pressure	1.4×10^{-2} mbar
Deposition time	15, 30, 45, and 60 min

diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive spectroscopy (EDS), etc. X-ray diffraction studies (XRD, Bruker D8 Advance Diffractometer, Model No: A18-A100/D76182) with Nickel filtered Cu Kα radiation, $\lambda = 0.15406$ nm, in the 2θ range 20° to 90° were conducted to evaluate the structural properties. Surface morphology and cross-section of the thin films were analyzed using field emission scanning electron microscope (FE-SEM, Carl Zeiss, Ultra 55) with an accelerating potential 5.00 kV/10.00 kV and magnification 100.00 kX. The thickness of the film was also measured using FE-SEM. Energy-dispersive spectroscopic analysis using X-ray (EDS) was performed to study the composition of the thin film. The equipment used for EDS is x-sight X-ray diffractometer of make Oxford INCA, equipped in FE-SEM.

Packaging of ZnO thin film for dynamic pressure sensing application

In order to evaluate the suitability of ZnO thin film for dynamic pressure sensing applications, it needs to be interfaced with test equipment. Toward this, sensor packaging was carried out with M14 × 1.5 h threaded pressure port which is welded to a 380 µm thick machined metallic diaphragm. The phynox substrate with ZnO thin film deposited on it (sensing element) was bonded on the top surface of this diaphragm. In this arrangement, the shock pressure will lead to the deflection of diaphragm which in turn transfers the strain to phynox substrate with ZnO film on it.

Double enameled copper wires of 70 µm diameter were used to attach electrical leads on to the sensing element films using silver epoxy paste. Figure 1 shows the schematic of ZnO thin film-based sensor in packaged condition.

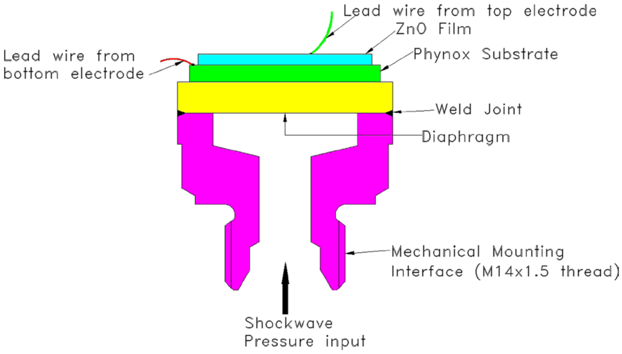

Figure 1: Schematic of ZnO thin film-based sensor packaged for dynamic pressure sensing application.

Test set up for characterization of a dynamic pressure sensor

A classic example of dynamic pressure is the supersonic pressure waves generated due to blast, explosions or ballistics in the air which are known as shock waves. An equipment called shock tube can be used for the generation of shockwaves (Theodoro et al., 2013). A shock tube can generate pressure pulses from few millibars to few hundred bars with a rise time in nanoseconds (Lally and Cummiskey). In a shock tube, the pressure generated can be computed either by using thermodynamic models or by using a calibrated reference sensor (Elkarous et al., 2016). Many research works in the area of metrology and scientific instruments define shock tube as a standard for dynamic pressure calibration (Diniz et al., 2006; Bartoli et al., 2012; Downes et al., 2014; Wang et al., 2015).

Shock tube used in our experiment (Figure 2) consists of a simple tube divided into two sections (driver and driven) by a membrane. When the membrane is made to rupture suddenly by increasing the pressure in the driver section, it leads to the propagation of shock waves through the gas in the driven section. This equipment can provide a pressure pulse of faster rise time and wide range (Reddy's tube) (Reddy and Sharath, 2013).

As can be seen in Figure 2, a ZnO thin film sensor was mounted on the end flange of the shock tube using the M14 × 1.5 pressure port mechanical interface. An industrial type quartz-based reference sensor (PCB Piezotronics make, model No: 113B22) of sensitivity 14.4 mV/bar was also mounted on the end flange. The output of the sensor under test and reference sensor were connected to a storage oscilloscope (make:- Agilent, model:- DS07024A)

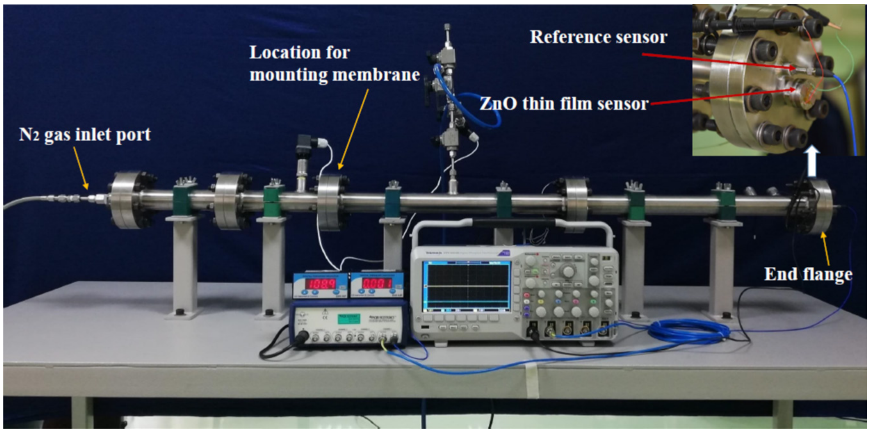

Figure 2: Experimental test setup used for the characterization of dynamic pressure sensor.

to capture the output voltage. Figure 2 shows the details of the complete experimental set up used for dynamic pressure sensing. Membranes of varying thicknesses were used in the shock tube to generate shock waves of different pressure. N_2 gas was admitted to driver section which resulted in sudden rise in driver section pressure and rupture of the membrane. Shock waves were generated which propagated through the shock tube toward the end flange. This pressure pulse led to the generation of piezoelectricity in the sensor under test as well as reference sensor.

Results and discussion

Structural characteristics

Figure 3 shows the XRD pattern of the thin films deposited for 15, 30, 45, and 60 min duration. Sharp peak at about 34° shows preferred grain growth along (002) plane for highly c-axis-oriented ZnO films (the other two peaks are corresponding to chromium and cobalt of substrate). This indicates a high crystallinity. This high degree of c-axis orientation and high crystallinity enhances the piezoelectric properties of the ZnO film (Joshi et al., 2012). The increase in the intensity of peak with an increase in deposition duration of the film indicates that crystallinity and grain size are enhanced with the film thickness (Chang et al., 2002). Grain size is calculated using Debye–Scherrer's formula (Salah et al., 2015), Grain size, $D = k\lambda/\beta \cos\theta$ where k is called shape factor, 0.94 for ZnO, λ is the wavelength of the x-ray used, β is full width at half maximum (FWHM) of the diffraction peak in radians, θ is the Bragg's diffraction angle. 2θ and FWHM are derived from XRD result and grain size of the film is calculated using Debye–Scherrer's Formula. Details are given in Table 2.

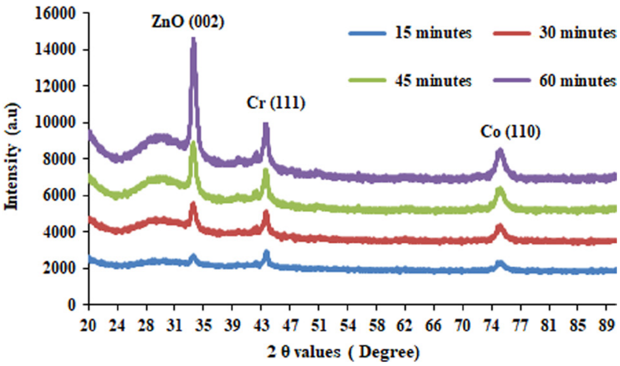

Figure 3: XRD pattern of ZnO thin film deposited for different durations.

Morphological analysis

The surface morphology of the ZnO thin films deposited for duration 15, 30, 45, and 60 min was characterized by FE-SEM and is shown in Figure 4A-D. Cross-section is shown in Figure 5A-D. The surface morphology shows the formation of dense and smooth homogenous films for all four deposition durations. As seen in topography, the average grain size is larger for longer deposition duration. This supports the results obtained from XRD. The individual grains are connected closely with each other. Cross-section indicates an average thickness of 160, 430, 532, and 683 nm for films deposited for the duration of 15, 30, 45, and 60 min. Films have a columnar structure perpendicular to the surface of the substrate as seen in cross-section. Figure 6

Table 2. Structural details of ZnO thin film derived from XRD.

Sl. No.	Deposition duration (min)	2θ (Deg)	FWHM (Deg)	Average grain size (nm)
1	15	33.950	0.796	10.905
2	30	33.890	0.684	12.694
3	45	33.890	0.601	14.435
4	60	34.014	0.517	16.777

shows the average thickness of ZnO thin film for different deposition durations.

Composition analysis

The EDS spectra of ZnO thin film for different deposition durations are shown in Figure 7A–D. Figure 8A, B shows the composition of ZnO films (in weight % and atomic %) as a function of deposition duration as obtained from EDS.

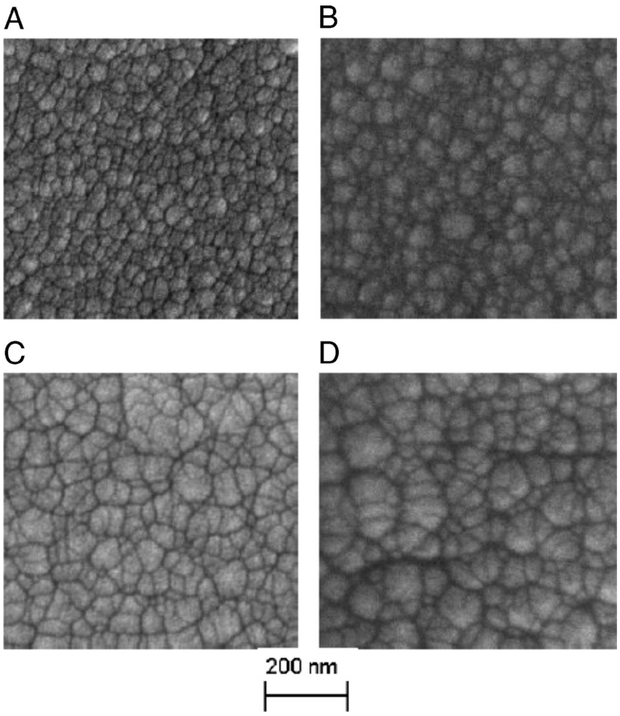

Figure 4: FE-SEM image: surface morphology of the ZnO thin films for deposition durations of (A) 15 min, (B) 30 min, (C) 45 min, and (D) 60 min.

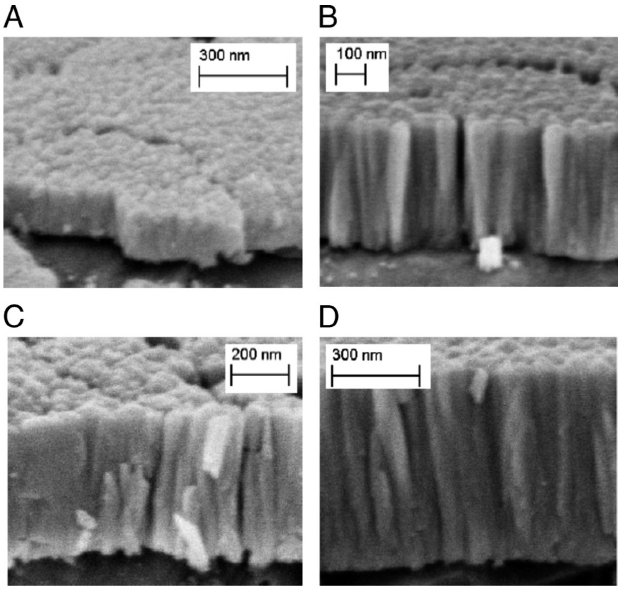

Figure 5: FE-SEM image: cross-section of the ZnO thin films for deposition durations of (A) 15 min, (B) 30 min, (C) 45 min, and (D) 60 min.

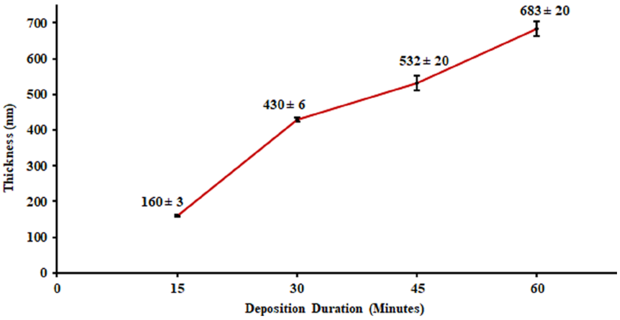

Figure 6: Variation of film thickness with deposition duration.

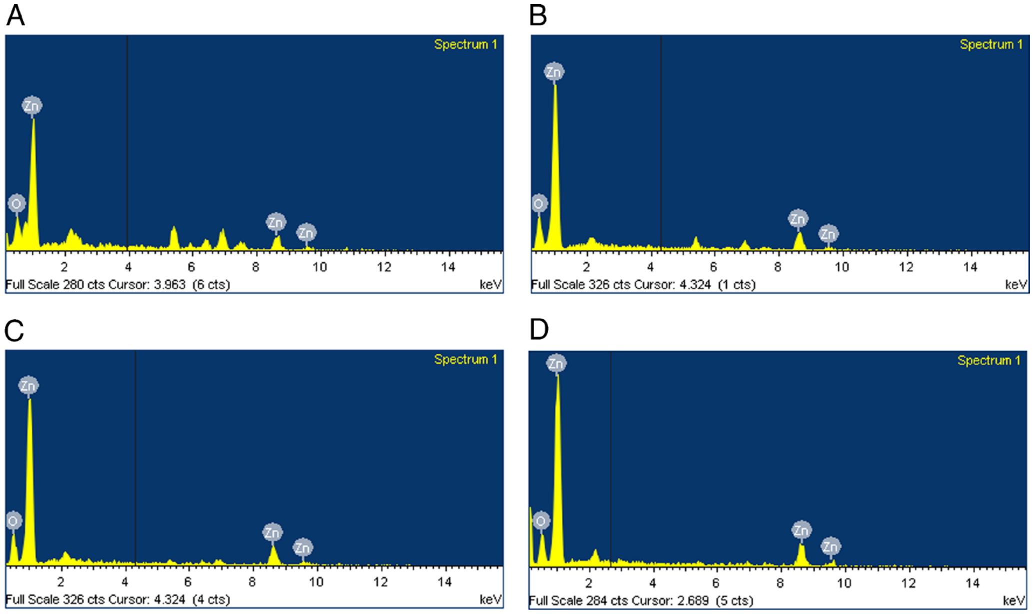

Figure 7: EDS spectra of the thin films for the deposition durations of (A) 15 min, (B) 30 min, (C) 45 min, and (D) 60 min.

The theoretical weight % of ZnO is Zn – 80.3397% and O – 19.6603%. The EDS results of four samples as per Figure 8(A) are closely matching with the theoretical values (Callister, 2007). The atomic % of four samples are also matching with the theoretical value (nearly 50% each for Zn and O) of a 1:1 compound like ZnO (Callister, 2007). This indicates that the ZnO films deposited using reactive RF magnetron sputtering maintained its stoichiometry irrespective of deposition duration when all other parameters were kept constant.

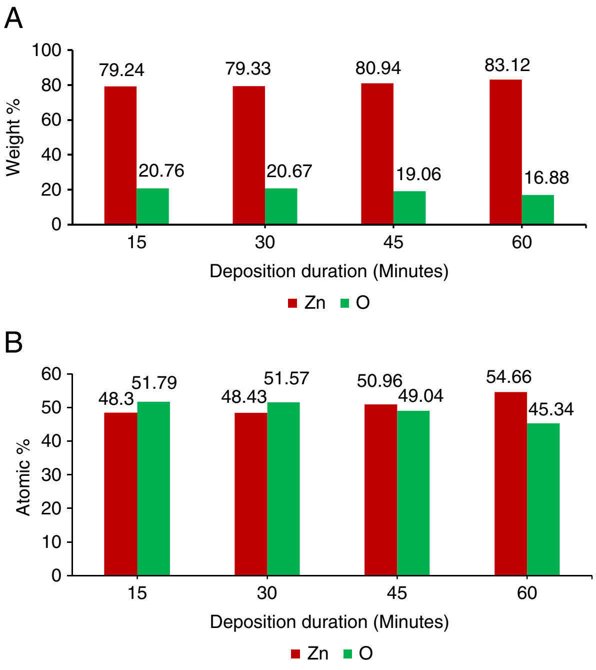

Figure 8: (A) EDS result – composition of ZnO film in weight %, and (B) EDS result – composition of ZnO film in atomic %.

Characterization of the dynamic pressure sensor

Sensors were mounted on the shock tube as shown in Figure 2. Stress reliving of the diaphragm and the thin film was carried out by subjecting each sensor to three shock pulses of about 10 bar each. Polycarbonate sheets of different thicknesses were used as the membrane in the shock tube to generate different levels of dynamic pressure. All the four thin-film sensors (with deposition duration of 15 min (Sensor 1), 30 min (Sensor 2), 45 min (Sensor 3) and 60 min (Sensor 4)) were tested for five different shockwave pressure levels ranging from about 4 bar to 15 bar. The output from test sensor and reference sensor were acquired using storage oscilloscope. The output of the reference sensor was used for computing the shockwave pressure level. All the four sensors responded to the shockwave with noticeable output. Figure 9 shows a typical response obtained from ZnO thin film-based sensor (Sensor 4) and the reference sensor to shock wave signal (14.24 bar pressure). ZnO thin film sensor demonstrated pure

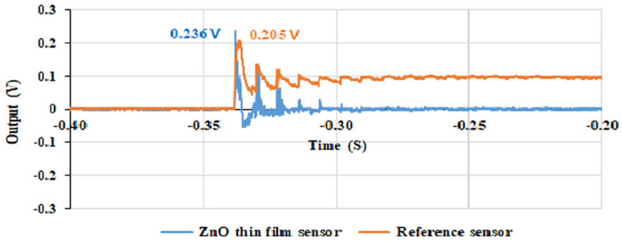

Figure 9: Response of ZnO thin film sensor (Sensor 4) and reference sensor to shockwave (14.24 bar pressure).

dynamic behavior with sharp output peaks, faster response, and faster discharge time, whereas a quartz-based reference sensor demonstrated a quasi-static behavior with slow discharge. The output of four sensors with respect to different shockwave pressure is plotted in Figure 10. Sensitivity of the four sensors to dynamic pressure is shown in Figure 11. Sensors showed an increasing trend in the output with an increase in film thickness and also with an increase in pressure.

Conclusions

Well-oriented ZnO thin films were deposited on phynox alloy substrate by RF reactive magnetron sputtering for four different thicknesses by varying deposition duration. These films were characterized by their composition, structural, and morphological properties. XRD indicated highly c-axis-oriented films with preferred grain growth along (002) plane. SEM showed the formation of dense and smooth homogenous films for all four deposition durations with columnar structure perpendicular to the surface of the substrate. The average grain size is larger for longer deposition duration. Cross-section of thin films showed the average thickness of 160, 430, 532, and 683 nm for films deposited for durations of 15, 30, 45, and 60 min. EDS indicated that the ZnO films maintained its stoichiometry irrespective of deposition duration. The effect of deposition duration of ZnO thin film on dynamic pressure sensing was also studied. All four sensors were packaged and evaluated for their suitability for dynamic pressure sensing by testing with shock tube equipment. All the four sensors exhibited noticeable output with increasing trend with respect to thickness and pressure for shockwave signals ranging from about 4 bar to 15 bar. Shock tube test results showed pure dynamic sensing behavior of ZnO films. The output obtained from four sensors is on par with the output of reference sensor of industrial standard. This study shows that the cost-effective ZnO films fabricated using RF reactive magnetron sputtering are efficient and sensitive to dynamic pressure. It is successfully demonstrated that sensors based on ZnO thin film are capable of sensing dynamic pressure.

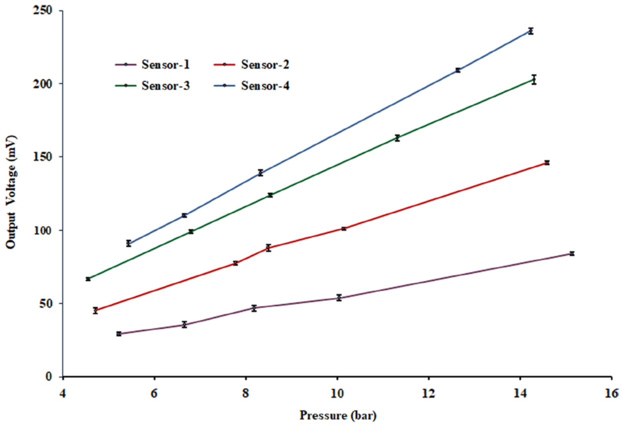

Figure 10: Output of ZnO thin film sensors vs shockwave pressure.

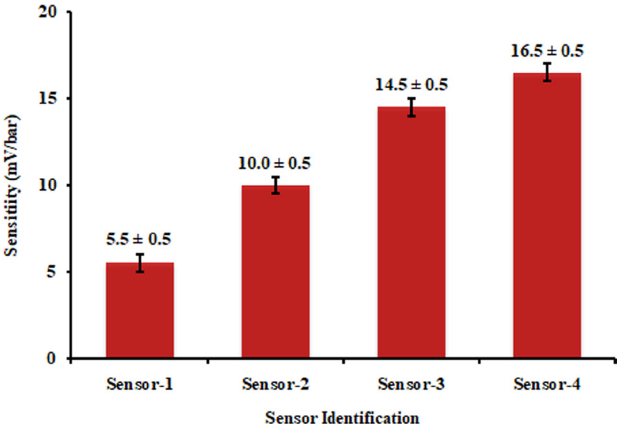

Figure 11: Sensitivity of ZnO thin film dynamic pressure sensors.

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