



# Deposition and characterisation of shear-mode ZnO sensor and micro-cantilever for contact sensing and nanoactuation



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## ABSTRACT

Integrated sensors in fitness and sports equipment have been developed in the last two decades. A zinc oxide (ZnO) thin film could be a suitable material for such applications due to its excellent physical and piezoelectric properties. In this paper, nanocrystalline ZnO films were deposited on silicon (Si) wafers with different substrate tilt angles (30° to 75°) and sputtering durations (2 to 4 h) using RF magnetron sputtering at room temperature. ZnO thin film topographical properties, film thickness (0.8 to 3.7 μm) and columnar inclined angle (14° to 34°) on the different locations of specimens were characterised by scanning electron microscope (SEM). Simulation and experimental results from the sensor arrays were also fabricated using fabrication techniques compatible with a standard micromachining process. The simulation and experiments for sensor arrays proved that sensor arrays were capable of distinguishing the differences of contact force values, contact locations and directions. Moreover, ZnO thin film micro-cantilevers were fabricated and tested by an AC impedance technique and a laser doppler vibrometer (LDV). The results exhibited good piezoelectric properties of shear-mode ZnO thin film driven cantilevers.

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## 1. Introduction

Equipment integrated with embedded sensors provides a revolutionary change in fitness and sports. For example, the nanometer lever built-in wireless pressure sensors in rackets, bats, clothes, and shoes could provide real-time contact force/pressure information. The nano-scale bio-sensor attached on an athlete could obtain the changes of cholesterol, glucose and other physiological data instantaneously. These analysed data could benefit coaches and nutritionists to draw more advisable training and diet plans, and finally improve the training qualities of athletes. As another example, contact force sensors could be placed on the boundary of a court such as table tennis, tennis, badminton and volleyball, to assist referee's judgement [1]. However, the technologies mentioned above have not been fully developed and commercialised yet.

In recent years, ZnO was applied to enhance the performance of different advanced materials, such as improvement of bonding behaviour and ultraviolet (UV) resistibility for glass fibre reinforced epoxy (GFRE) [2], anti-wear reinforcement of ultra-high molecular weight polyethylene (UHMWPE) [3], as well as thermal and mechanical properties of glass fiber reinforced polyester (GFRP) [4]. Due to its good piezoelectric property, cheap manufacturing cost, low temperature, low toxicity, and environment-friendly fabrication process, it also widely

used in microelectromechanical systems (MEMS), microfluidics, microbiotechnology, nanotechnology, etc. [5]. Since the (002) plane of ZnO needs the lowest surface free energy [6], the (002) oriented ZnO films are able to grow naturally in a hexagonal or wurtzite type crystalline structure without tilting substrate or external ion and plasma source. The sensor with a (002) film texture can perform in air, gaseous and dry environments [5], but is not suitable for the liquid environment due to the damping of the propagating wave of its longitudinal mode (L-mode) wave when sensing in liquid [7]. ZnO thin films with an inclined c-axis (shear) mode, which has both longitudinal and shear modes (S-mode) with different frequencies, could improve the sensitivities of sensor devices, such as bio-sensor, viscosity sensor, force sensor, as well as other different types of transducers [8–10].

Radio frequency (RF) magnetron sputtering is the most widely used technique for ZnO thin film fabrication due to its easily-controlled influence factors, low temperature (room temperature) and multiple choices of target bars. One of the common methods to realise inclined ZnO films is to change the substrate tilt angle in the sputtering process [11–14]. To achieve good quality crystallinity of ZnO, many crucial issues, such as the ZnO film formation mechanism and its evolution process, and the influence of various deposition parameters during ZnO growth, still remain to be solved.

This paper investigates the deposition and characteristics of inclined c-axis ZnO thin film first, mainly focusing on the effects of deposition duration and substrate tilt angle. Subsequently, an integrated contact force sensor is proposed and fabricated. Fabrication of ZnO sensors

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was integrated seamlessly into the microfabrication process flow. The characterisations of shear mode ZnO film micro-cantilever are presented in the final part of this paper.

## 2. Experimental work

ZnO thin films with different substrate tilt angles were deposited using an RF magnetron sputtering system in a gas mixture of argon (Ar) and oxygen ( $O_2$ ) with two substrate–target configurations. The ZnO target was 50 mm in diameter with 99.99% purity and the distance between target and substrate was 70 mm. The chamber was evacuated to a pressure less than  $5 \times 10^{-3}$  Pa before the introduction of a gas mixture and the generation of plasma, activated by an RF power at 13.56 MHz. Based on our previous studies [15], the optimal deposition working conditions had been investigated. The optimal ratio of Ar and  $O_2$  was 3:1 (9:3 sccm) and the working pressure was 0.8 Pa. In this study for deposition shear-mode ZnO thin films, the RF power values were set to be 200 W, and the deposition durations were varied between 2 and 4 h. Si samples that were 0.45 mm thick, 3 cm long and 1.2 cm wide were put on four aluminium (Al) plates with various tilting angles to substrate plane ( $30^\circ$ ,  $45^\circ$ ,  $60^\circ$  and  $75^\circ$ ) to grow ZnO thin films with different inclined c-axis angles. The deposited ZnO thin films were characterised using X-ray diffractometer (XRD, Panalytical Empyrean,  $Cu K\alpha \lambda = 0.15406$  nm, 40 kV/30 mA) and field-emission scanning electrode microscopy (FE-SEM, JEOL JSM-7600).

On the microfabrication process of the integrated force sensors as shown in Fig. 1, the integration processes of the ZnO sensors were conducted on wafer level. 4 inch p-type  $\langle 100 \rangle$  Si wafers with 450  $\mu\text{m}$  thickness were used. Firstly, both sides of a wafer were sputtered with 1  $\mu\text{m}$   $\text{SiO}_2$ , to provide insulation between the bottom electrode and the silicon substrate. Then a 200 nm Au/Ti layer was sputtered as the bottom electrode, followed by a 2  $\mu\text{m}$  thick ZnO thin film and finally a 200 nm Au/Ti layer as the top electrode. The vias were also designed and fabricated for bonding application.

The micro-cantilevers were also fabricated on a 4 inch p-type (100) silicon wafer with 450  $\mu\text{m}$  thickness. The fabrication process flow is shown in Fig. 2. Similar to the sensor microfabrication shown in Fig. 1, the fabrication process started with oxidation of the silicon wafer (Fig. 2(a)). Au/Ti of 100 nm thickness was deposited as the bottom electrode (Fig. 2(b)). A ZnO layer 1440  $\mu\text{m}$  long  $\times$  540  $\mu\text{m}$  wide  $\times$  2  $\mu\text{m}$  thick with a  $45^\circ$  inclined substrate was then sputtered at room temperature (Fig. 2(c)), followed by the deposition of the top electrode, which is Au/Ti with a thickness of 100 nm (Fig. 2(d)). After deposition of the thin films, the wafer underwent deep reactive ion etching (DRIE) to carve out the mechanical structure. There were two steps during the etching: (i) the cantilever pattern was defined by etching from the front side (Fig. 2(e)); and (ii) cavity was cut from the back side to free the cantilever (Fig. 2(f)). The thickness of the silicon beam was controlled by the etching time. The etching speed is around 3  $\mu\text{m}/\text{min}$ .

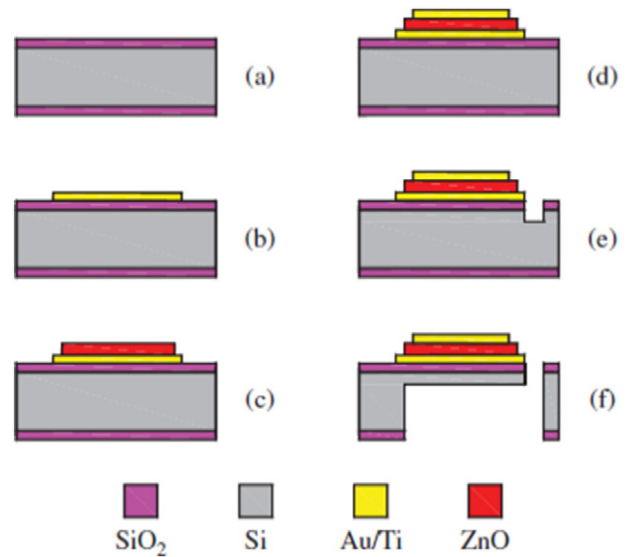


Fig. 2. Fabrication process flow: (a) oxidation, (b) Au/Ti deposition, (c) ZnO deposition, (d) Au/Ti deposition, (e) front-side etching, and (f) back-side etching.

## 3. Results and discussion

### 3.1. Results of ZnO film deposition

When the samples were placed on the tilted substrate, different locations on the samples had a different ‘target-to-substrate distance’ (TSD). Since TSD has a strong influence on ZnO film thickness and columnar inclined angles, the Si samples with ZnO thin films were cut into three parts for investigation, top (T), middle (M) and bottom (B) after deposition. ZnO film thickness and columnar inclined angles were measured by SEM. Their typical result images with different deposition times and tilt substrate angles are shown in Fig. 3. It can be observed from Fig. 3 that the ZnO films obtained with a deposition time of 2 to 4 h and substrate tilt angles of  $30^\circ$  to  $75^\circ$  present compact columnar structures in all conditions. As expected, ZnO crystals grow in a hexagonal wurzite structure and form long columns along the c-axis resulting in a columnar grain structure.

Fig. 4 presents ZnO thin films obtained from different regions of a sample under the same deposition time. The top region (Fig. 4(c)) has less TSD and produced higher ZnO film thickness than the middle and bottom parts. On the lower part of ZnO films, the long rod columnar grain structure can be obviously seen from all of the three SEM images without significant difference. On the upper part of ZnO films, some parts of ZnO films merge together to form a large region of plate. This finding is quite close to the theory of ‘texture development of non-epitaxial polycrystalline ZnO films’ proposed by Kajikawa [13]. After a

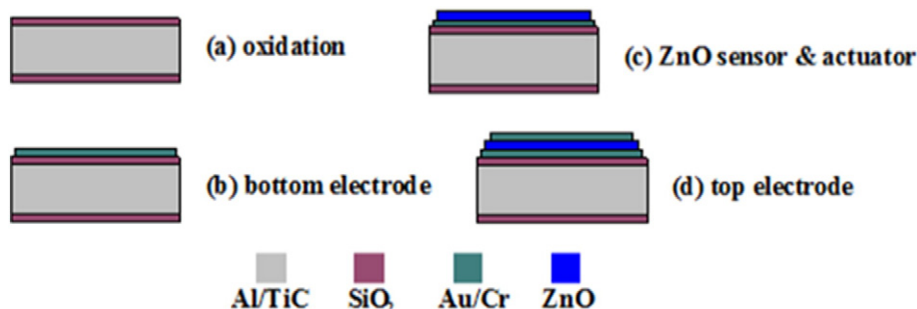


Fig. 1. Integration processes of ZnO sensors.

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