DEPOSITION OF HIGHLY ORIENTED LOW-STRESS ZnO FILMS

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ABSTRACT

Several-micron-thick layers of ZnO films have been deposited on glass substrates. The films were grown by reactive DC magnetron sputtering from a zinc target. The films are highly oriented as indicated by x-ray diffraction analysis and the c-axis of the films is normal to the substrate. A moiré deflectometer has been built to determine the film stress by measuring the substrate curvature. The films have relatively low stress at higher deposition pressures. The acoustic coupling constant, k_t, of the films has been measured to be as high as .26, which is 90% the value for single crystal ZnO. The dependence of film quality and stress on various deposition parameters such as pressure and power are presented.

INTRODUCTION

Sputtered thin-films of ZnO have been widely used for acoustic wave generation and detection in surface-acoustic wave (SAW) and bulk acoustic wave (BAW) devices at frequencies ranging from megahertz to several gigahertz.[1,2] ZnO thin films have been implemented in micromechanical devices as sensors and actuators.[3,4] ZnO offers a relatively high electromechanical coupling constant (k_t) and reasonable compatibility with IC processing techniques. Various sputtering schemes to obtain good quality films have been reported.[5,6] Most of the previous work, however, has focused on relatively thin (<5 μm) layers over relatively small areas. Thicker films are required for microsensors and low frequency bulk acoustic wave devices. Furthermore, uniform films over large areas are needed to take advantage of batch IC processing techniques.

The purpose of this study is to investigate the mechanical properties of thick (>5 μm) ZnO films over areas as large as 100 cm^2. As the material thickness is increased, the residual stress in the ZnO films must be controlled to minimize substrate curvature and to prevent delamination of the films. Substrate fracture has also been observed on samples with thick, highly stressed ZnO. A moiré deflectometer has been built to determine the stress in the films by measuring substrate curvature. The k_t of the films were measured using pulse-echo techniques. Variations in the stress and k_t have been determined as a function of sputtering pressure and power. Films with relatively low intrinsic stress and high electromechanical coupling values have been obtained.

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DEPOSITION SYSTEM

The ZnO films were deposited by reactive DC magnetron sputtering from a zinc target with dimensions of 23 cm x 13 cm. The substrates are 100 mm x 100 mm x .075 mm 7740 glass plates. A 1400 Å-thick layer of gold with a thin (100 Å) layer of Ti was evaporated at 300°C on the substrates prior to ZnO deposition to serve as back electrodes. The Au film is highly <111> oriented as observed by x-ray analysis. The ZnO films were sputtered at a substrate temperature of 250°C in a 80% O_2 / 20% Ar ambient at power levels of 200 W and 400 W. The sputtering pressure was varied in the 5 to 10 millitorr range and the flow rate was approximately 100 sccm. The parameters used for ZnO deposition are summarized in Table I. The transducer deposition is completed by evaporating a 1000 Å layer of gold with a Cr adhesion layer through a shadow mask to define circular electrodes diameters of 550 μm and 1100 μm. Electrical contacts to the electrodes were made by bonding 50 μm diameter gold wires.

Parameter	Value
Target	Zn
Gas mixture	80% O_2 / 20% Ar
Target - sample spacing	9 cm
DC power	200 W / 400 W
Sputtering pressure	Variable (5 to 10 millitorr)
Flow rate	100 sccm
Substrate temperature	250 C
Deposition rate	1.0 μm/hr (200 W) 2.2 μm/hr (400 W)

Table I. Parameters used during ZnO depositions

MEASUREMENTS

The films deposited have been found to be polycrystalline with the c-axis oriented normal to the substrate as measured by x-ray analysis. Typical x-ray rocking curves had a half-width at σ (63% full height) of .75° to 1°. Some films have been found to have high compressive stress. When the films are relatively thick, and the substrates thin, the samples become significantly deformed. Under some circumstances localized stresses may even cause the substrate to fracture or the films may delaminate from the surface. Typically the stress deformation of a wafer can be characterized by a radius of curvature derived from a simple scanning measurement across the diameter of the wafer. If two-dimensional information is needed, a Fizeau or other type of optical interferometer is normally used. In our case the deformation to be measured is often too complex to characterize by a single number, and too extreme to measure with an optical interferometer. A quick, relatively insensitive method of obtaining two dimensional information is required.

The instrument chosen is a moiré deflectometer as described by Kafri and Glatt.[7] The device detects ray deflections caused by height gradients of a reflective target. The apparatus utilizing this principle to measure wafer deformation is represented schematically in Fig 1. The output of a HeNe laser is expanded, brought to a focus and reflected from the beamsplitter to fill the collimating lens 1. The collimated laser beam reflects from the sample under test. The reflected beam whose wavefront has been distorted by the substrate passes through the beamsplitter to the collimating lens 2. The lenses are spaced to form a telecentric system forming an image of the substrate in the vicinity of the pair of Ronchi rulings. That image with superimposed moiré fringes is imaged onto a CCD camera for recording.

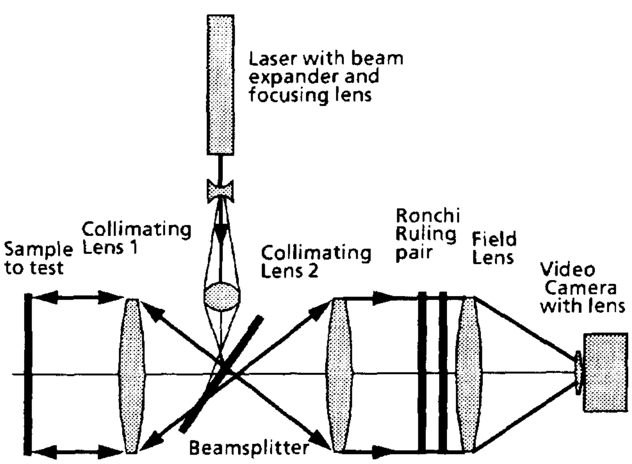


Fig 1. Optical schematic of the moiré deflectometer.

In use, a flat mirror is placed in the test position and the rotational position of the moiré fringes is noted. The flat is then replaced by the sample to be tested and the fringe pattern is recorded. The fringe rotation is related to the radius of curvature of the reflecting surface and the geometry of the apparatus by

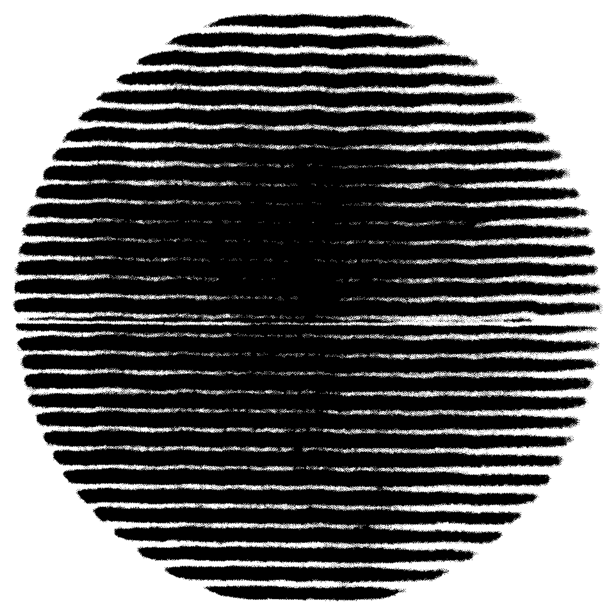
$$\frac{1}{R} = K \tan\alpha$$

where R is the radius of curvature, α is the tilt angle of the fringes, and K is a function of the geometry of the setup given by

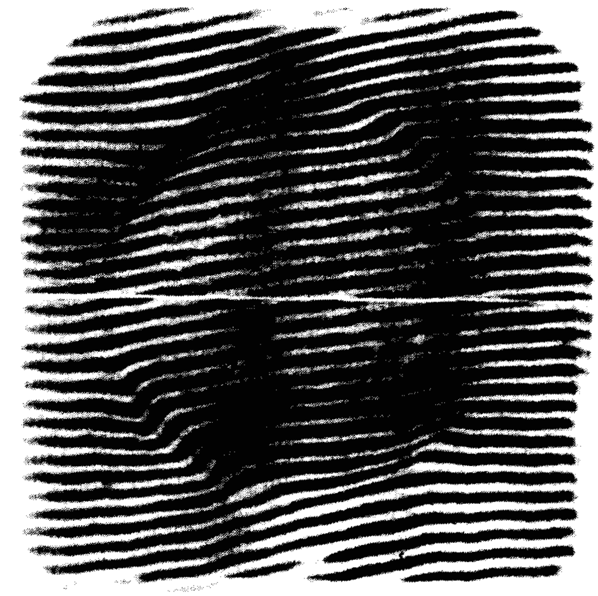
$$K = \frac{M\Theta}{d}$$

where M, the magnification, is the ratio of the focal lengths of collimating lenses 1 to 2, Θ is the angle between the rulings, and d is the separation of the rulings. In practice we simply use a mirror of known radius of curvature as a test object and determine K experimentally.

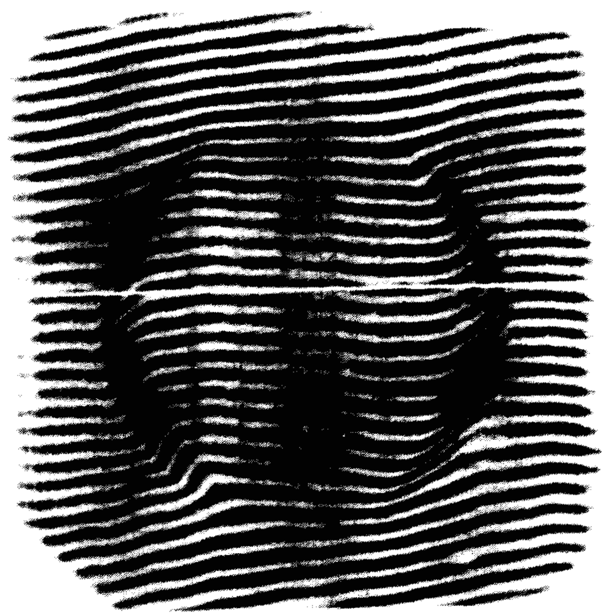
Fig. 2 shows typical results obtained with the deflectometer. The fringes obtained when a flat mirror is placed in the test position are shown in Fig. 2(a). Figs. 2(b) and (c) show the deflection of the substrate along two orthogonal directions parallel to the substrate edges. The ZnO film on this sample was deposited through a 7 cm-diameter shadow mask. These images show that the moiré deflectometer gives quick two-dimensional visualization and measurement of the substrate curvature.



(a)



(b)



(c)

Fig. 2 Moiré deflectometer fringes showing the curvature of substrates. (a) Flat mirror reference, (b) and (c) depict substrate deflections on a ZnO sample along 2 axes parallel to the substrate edges. The white line represents zero curvature.

For a uniformly stressed substrate with a corresponding uniform curvature, the film stress, σ_f, is[8]

$$\sigma_f = \frac{[E_s\, t_s^2]}{6\,(1-v)\, t_f\, R}$$

where E_s is Young's modulus of the substrate, v is Poisson's ratio of the substrate, R is the radius of curvature and t_s and t_f are the substrate and film thicknesses, respectively. An absolute uncertainty of 40% is estimated for stress values due to spatial variations in the intrinsic film stress and film thickness across the deposited area, uncertainties in the glass mechanical properties, and interpretation of non-uniform curvature. Relative values are within 20%.

The electromechanical coupling constant, k_t, of the films were measured using a pulse-echo detection system with the transducers untuned.[9] The system is capable of automated measurements with an accuracy better than ±.5 dB in the 100-1000 MHz frequency range and has been described elsewhere.[10] The thickness of the films were measured using a Tencor alpha-step profilometer and the low frequency capacitance of the transducers were measured using an rf impedance analyzer to correctly account for the transducer size. The contribution of stray elements in the electrical input to the transducer was also measured with the impedance analyzer. Fig. 3 shows a typical measurement of conversion loss as a function of frequency. A theoretical curve is fitted to the data to extract the k_t of the films. The absolute accuracy of the k_t values is ±.02 and the relative accuracy is ±.01.

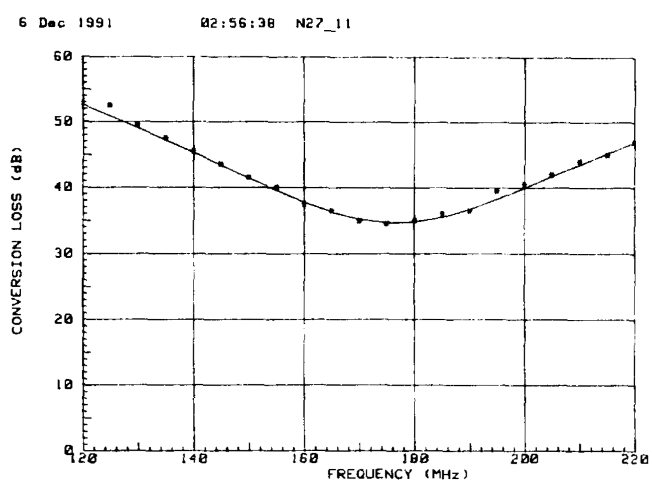


Fig. 3 Typical measured conversion loss as a function of frequency (points) and a theoretical fit to the data (line).

Figs. 4 and 5 show the stress and k_t measurements for films prepared at power levels of 200 W and 400 W, respectively. All films exhibit compressive stress. Both plots show a significant decrease in the stress as the pressure is increased. The k_t values also show a decrease as the deposition pressure is increased. At 200 W power and a pressure of 7.0 millitorr, the film had a relatively low compressive stress of -230 MPa and the corresponding k_t is .26, which is 90% the value for single crystal ZnO. Note that at 400 W and at the same pressure, the film has 4 times more stress with a similar k_t value.

CONCLUSION

We have studied the residual stress and electromechanical coupling constant of thick ZnO films grown with reactive DC magnetron sputtering. A moiré deflectometer has been implemented for quick two-dimensional measurement of substrate curvature. A pulse-echo system has been used to measure the conversion loss of the transducers and to calculate the k_t of the films. We have shown that the films grown at higher pressures exhibit lower residual stress with slightly reduced k_t values.

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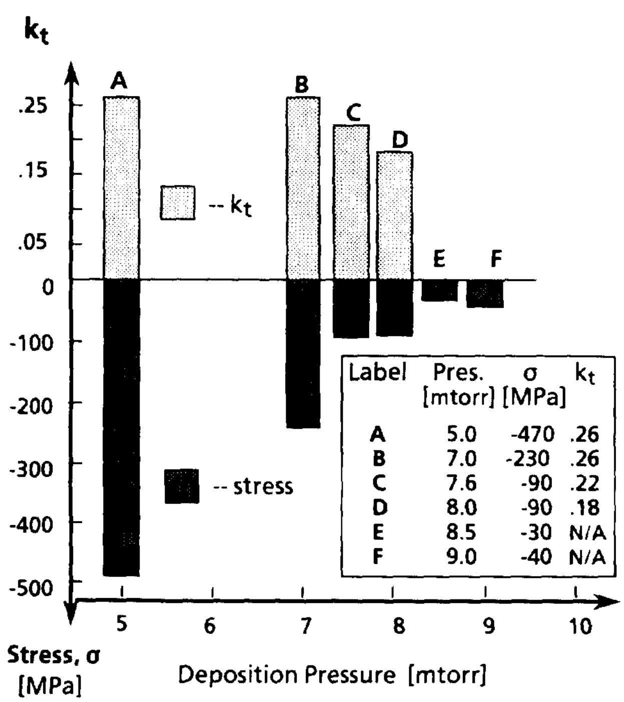

Fig. 4. The stress and k_t as a function of deposition pressure. The sputtering power is 200 W.

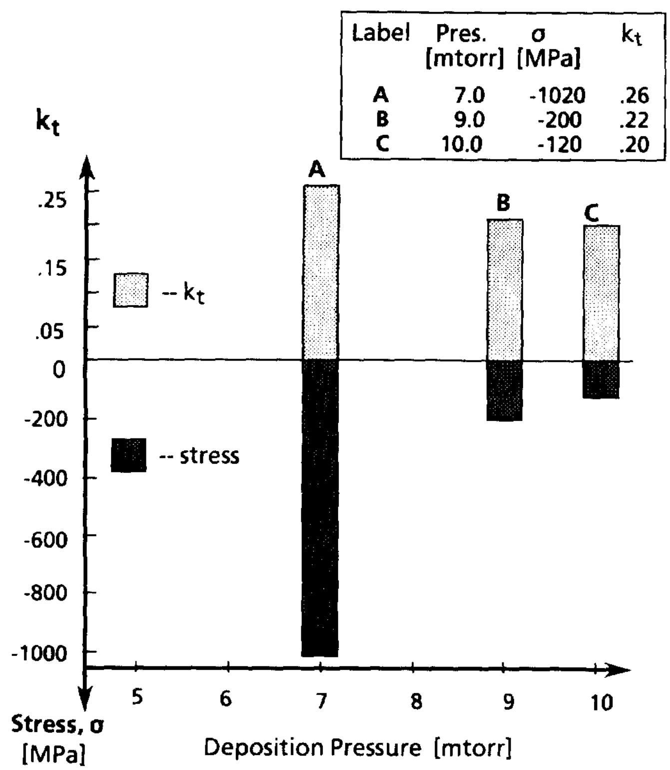

Fig. 5. The stress and k_t as a function of deposition pressure. The sputtering power is 400 W.