Thin Solid Films, *137* (1986) 185–192
ELECTRONICS AND OPTICS

ZnO PROCESSING FOR INTEGRATED OPTIC SENSORS

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(Received April 10, 1985; revised July 12, 1985; accepted August 7, 1985)

ZnO thin films were sputter deposited onto oxidized silicon wafers. The film quality increased with increasing applied r.f. power. Characterization of the films was performed by measurements of the attenuation of the transverse electric TE_0 optical guided mode. For an applied r.f. power of 2000 W, the deposition rate was 220 nm min^{-1}, resulting in a mode attenuation of only 1.6 dB cm^{-1} for a film 0.8 μm thick.

The refractive indices of the films were 99.5% (ordinary index) and 99.8% (extraordinary index) of the single-crystal values.

The step coverage on etched SiO_2 profiles appeared to be very smooth, which simplifies the creation of waveguiding channels. The optical behaviour of multilayered waveguides consisting of ZnO and SiO_2 films was studied. A prototype of a mode coupler in such a multilayer showed the feasibility of efficient electro-optic modulation in ZnO-based waveguides.

1. INTRODUCTION

ZnO is often called a promising material for planar optical waveguides[1–3]. It is potentially low cost, can be sputter deposited onto many types of substrate and has large electromechanical, electro-optical and elasto-optical constants. However, in the last 2 years little attention has been paid to it. This could be explained by the difficulties which are met in the fabrication of single-mode waveguides with ZnO. ZnO is normally deposited onto amorphous substrates such as fused quartz, glass or oxidized silicon. The high refractive index contrast between such substrates and the ZnO film implies very small channel dimensions (less than 0.5 μm) for single-mode operation. For integrated optic sensors several sensitive processes are reported[4–6], depending on single-mode interferometers or coupled channel waveguides. Since single-mode channels can hardly be realized in ZnO, we focused our attention on the application of multilayered single-mode slab waveguides. Two waveguides separated by an intermediate layer can be used to exploit coupling phenomena, with no demands being made on precise planar geometrical structures. Only film thicknesses and refractive indices determine the coupling process, which are both easy to control.

0040-6090/86/$3.50

In this paper the results of high rate sputtering of ZnO films are reported and compared with results of other groups in this field. Characterization of the films was performed by optical waveguiding measurements and occasionally scanning electron microscopy (SEM). It follows from several papers[1–3] that these optical measurements give a good insight into the crystalline quality of the sputtered films. Conclusions can be drawn about the quality of the films from the absolute values of the refractive indices and the birefringence. Parriaux and Cochet[7] have shown that the *c* axis orientation can be calculated from the measured refractive indices of anisotropic films such as ZnO. The excellent step coverage of our sputtered films makes even the fabrication of low loss ridge waveguides possible.

We performed several experiments on multilayered waveguides. Results concerning attenuation and mode behaviour will be discussed in the subsequent sections.

2. SPUTTER DEPOSITION OF ZnO

2.1. *Equipment*

We deposited our ZnO films using an r.f. reactive planar magnetron sputter unit, an apparatus which is rather similar to those of some other groups[8,9]. The system is evacuated with a turbomolecular pump, backed by a sorption vessel and a rotary pump. An additional liquid nitrogen coil is mounted on the inside of the bell-jar enabling the chamber to be evacuated to 7×10^{-6} Pa. We use a 99.999% pure zinc target of diameter 6 in and 99.999% pure oxygen as the sputter gas. The gas pressure during sputtering is electronically controlled using a precision leak valve. The substrates are steam oxidized silicon wafers with a (111) orientation. The substrate holder is placed above the target and can be heated to 500 °C by means of an ohmic coil. If the substrate is heated to its maximum temperature, a minimum pressure of 1×10^{-5} Pa can still be obtained. The r.f. source is capable of supplying 3 kW and is coupled to the target by means of a manually operated matching network.

2.2. *Results*

As usual, three main parameters are varied to obtain optimum sputter conditions, namely oxygen pressure, temperature and applied r.f. power. The pressure was varied between 2 and 0.5 Pa, the lower value being limited by discharge instabilities. The temperature was varied from room temperature to 500 °C. Using the same r.f. power, two best values of 0.5 Pa and 470 °C were obtained for pressure and temperature respectively. The key parameter in the determination of these values was the attenuation of the TE_0 mode. Films sputtered with higher oxygen pressures or lower temperatures showed very high attenuation values, varying between 12 and 60 dB cm^{-1}. The films sputtered at low temperature looked bluish because of a rather rough surface. Figure 1 shows the influence of the applied r.f. power on the attenuation of the TE_0 mode. Attenuation decreases with increasing power, *i.e.* increasing rate. At about 2000 W the film quality stabilizes. Table I shows some data reported in several papers on ZnO for optical waveguides compared with our results. The loss figures concern the TE_0 modes.

The more recent results of Maniv *et al.*[11] are omitted from this table since they

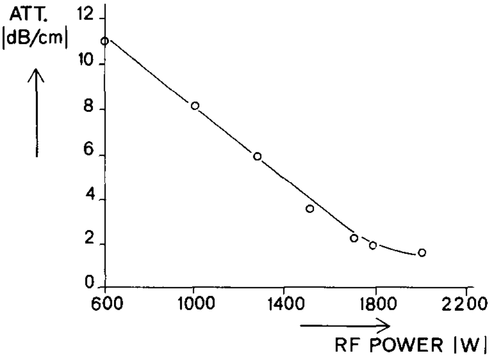

Fig. 1. Attenuation of the TE_0 mode *vs.* the applied sputter r.f. power.

did not attempt to obtain high quality optical films but merely studied the sputter process itself. Table I shows that our process has the advantage of the highest rate while retaining the quality of the films. It is very interesting to note that the results of Defranould[10] are almost the same with respect to all aspects of sputtering although he used a d.c. magnetron. Unfortunately he does not report the optical quality of his films.

Since we used a large target (diameter, 6 in) we were able to sputter onto relatively large areas. The films on the 2 in wafers have a thickness uniformity which is better than ±2%, which also compares with that obtained by Defranould[10].

The optical constants of the films sputtered at 1800 W or more are very close to the single-crystal values. We measured an ordinary index of 1.985 ± 0.004 and an extraordinary index of 2.007 ± 0.004. These values are respectively 99.5% and 99.8% of the single-crystal values (He–Ne light of wavelength 632.8 nm was used). The birefringence of our films is 0.85%, which is almost the same as the single-crystal value of 0.80%. These results indicate a high optical quality of the sputtered films.

For our purposes of integrated optic sensor applications we only need films up to 0.5 μm thick. These thin films can be grown with a very good reproducibility; however, we experienced problems with thicker films. Optical attenuation of the TE_0 mode increases sharply for films thicker than 1.3 μm. The appearance of these films becomes milky, indicating a rough surface. SEM pictures suggest a change in the film structure as the film becomes thicker. These observations could also have been made in the SEM pictures of other researchers. The negative comment of Maniv and Zangvilli[12] on the reliability of SEM observations should be noted here. Ambersley and Pitt[8] also experienced a change in the columnar structure of their films with increasing thickness (above 4 μm). One of the reasons for this change could be that many small columns begin to combine, resulting in different growth rates. Yamamoto *et al.*[3] used glass substrates with nearly the same thermal expansion coefficient as that of ZnO ($4.5 \times 10^{-6}\,°C^{-1}$ and $4 \times 10^{-6}\,°C^{-1}$ respectively) and produced very thick films without surface degradation. Since we used silicon substrates with a thermal expansion coefficient of $2.33 \times 10^{-6}\,°C^{-1}$ and a high sputter temperature, severe stresses built up in thick ZnO films. These stresses could be the cause of the change in crystalline structure. We shall shortly be using Corning 7059 substrates as well in order to check the influence of these stresses.

TABLE I

SPUTTER DEPOSITION PARAMETERS USED BY SEVERAL RESEARCHERS

Researcher(s)	*System*	*Target*	*Gas*	*Temperature* (°C)	*Pressure* (Pa)	*Deposition rate* (nm min^{-1})	*Attenuation* (dB cm^{-1})	*Reference*
Yamamoto *et al.*	R.f. magnetron	ZnO	Ar–O_2	400	1.3	150	2	3
Paradis and Shuskus	R.f. longitudinal magnetron	ZnO	Ar–O_2	500	1.3	10	2	2
Hickernell	D.c. triode	ZnO	Ar–O_2	300	0.4	25	1–2	1
Defranould	D.c. magnetron	Zn	O_2	450	0.3	150	—	10
Horsthuis	R.f. magnetron	Zn	O_2	450	0.5	200	1.6	This work

TABLE II

VALUES OF EFFECTIVE REFRACTIVE INDICES AND ATTENUATION FOR SEPARATE LAYERS AND A MULTILAYER

Mode	*Lower film*		*Upper film*		*Multilayer*		
					Measured values		*Calculated values*
	Effective refractive index	*Attenuation* (dB cm^{-1})	*Effective refractive index*	*Attenuation* (dB cm^{-1})	*Effective refractive index*	*Attenuation* (dB cm^{-1})	*Effective refractive index*
TE_0	1.9107	2	1.9153	2	Symmetric, 1.9190	4	1.9177
					Asymmetric, 1.9111	10	1.9122
TE_1	1.6882	5	1.7056	5	Symmetric, 1.7269	9	1.7220
					Asymmetric, 1.6858	17	1.6903

2.3. *Step coverage*

To create ridge-type waveguides we etched ridges in the SiO_2 layer on the silicon wafer. It appeared that our sputter process results in a very smooth step coverage, as shown in Fig. 2. We produced ridge waveguides with a width down to 25 μm without a measurable increase in optical attenuation. This width should not be taken as a lower limit. For our purposes we need slab-like waveguides, which have to remain relatively large in planar dimensions. These ridges cannot be formed by wet chemical etching since ZnO behaves very anisotropically in all etchants, leading to rough edges and consequently excess light scatter.

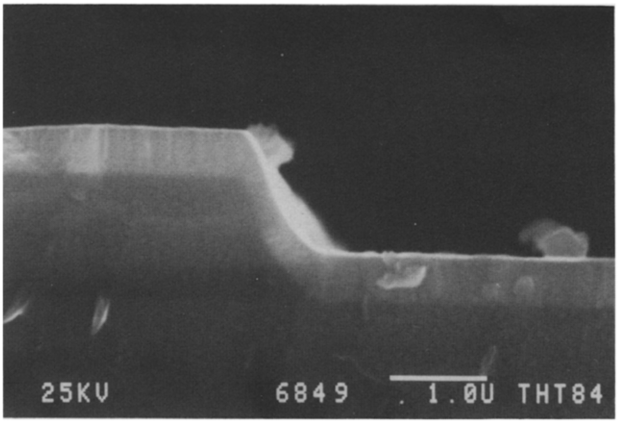

Fig. 2. Scanning electron micrograph of ZnO step coverage on etched SiO_2 profile.

3. MULTILAYERS

We studied the optical behaviour of multilayered waveguides consisting of two ZnO layers separated by a chemically vapour-deposited SiO_2 film. Structures as depicted in Fig. 3 were used; a cleaved part of a second oxidized silicon wafer is utilized as a shadow mask to sputter deposit ZnO only partially onto the first wafer. A computer program was written to calculate the modes for these multilayers. Propagation constants and attenuation were measured for both the separate layers and the multilayer.

The results for one multilayer are presented in Table II. From the measure-

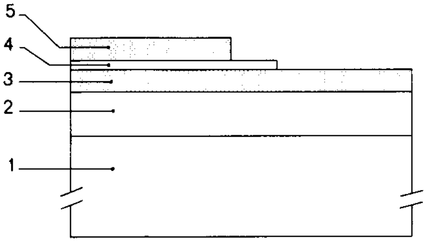

Fig. 3. Cross section of a multilayered waveguide: 1, silicon wafer (280 μm); 2, thermally grown SiO_2 (1 μm); 3, sputtered ZnO (0.2–0.5 μm); 4, partially chemically vapour-deposited SiO_2 (0.2 μm); 5, partially sputtered ZnO (0.2–0.5 μm).

ments of the separate layers, the refractive indices and thicknesses of these layers can be determined. These values together with the thickness and refractive index of the separating layer are fed into the computer which then calculates the mode effective indices of the multilayer itself, which are given in the last column. Results for other multilayers are quite similar.

We also calculated the spatial intensity profiles of the electric fields of the modes and plotted them as shown in Fig. 4. The intensity profiles of the symmetric and antisymmetric modes mainly depend on the thickness ratio of the two ZnO layers, and on the thickness of the intermediate layer. Figure 4(a) presents the results for a multilayer with equal ZnO layers. In Fig. 4(b) the thickness of the upper layer is decreased by 10%. As can be seen, the spatial distribution of the propagated power is greatly changed. It can be easily explained from these plots why the attenuation coefficients for the symmetric and antisymmetric modes are different. In the case of the antisymmetric modes, a larger part of the energy propagates in the upper ZnO layer. This layer exhibits larger losses since it is sputtered onto a poor quality intermediate layer. For reference, we sputtered ZnO films onto chemically vapour-deposited SiO_2 films which were directly deposited onto cleaned silicon wafers. The loss of the TE_0 mode was about 7 dB cm^{-1}, compared with 2 dB cm^{-1} which is typical for films on steam-oxidized wafers.

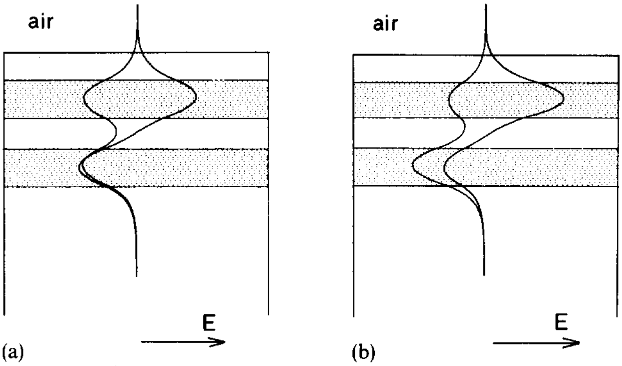

Fig. 4. Electric field strength of guided TE modes in multilayers (shaded areas, ZnO; white areas, SiO_2): (a) symmetric multilayer (thicknesses: ZnO layers, 0.25 μm; separating layer, 0.2 μm); (b) asymmetric multilayer (thicknesses: upper ZnO, 0.23 μm; lower ZnO, 0.25 μm; separating layer, 0.2 μm).

For the asymmetric case the energy of both modes is more clearly split over the two layers. Consequently the difference in attenuation of both modes is also increased. These sandwiched structures can be used for mode coupling purposes, such as switching and modulating or for sensor applications. We developed a guided TE-to-TE coupler for sensor applications, the details of which are presented elsewhere[13]. Figure 5 shows a cross section of the mode coupler. This device could be regarded as a modified directional coupler, where the two channel waveguides are replaced by two slabs. The advantage of this configuration is the reduction in the accuracy required for the fabrication.

A periodic electrode is deposited on top of the multilayer. The reverse side of the wafer is metallized to form the ground electrode. The wafer itself may be regarded as

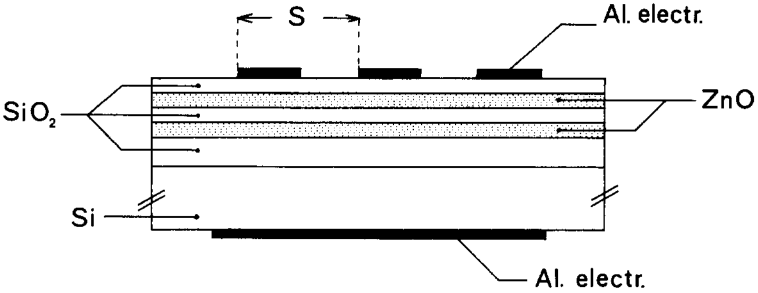

Fig. 5. Cross section of a guided TE-to-TE mode coupler.

a ground electrode since it has a very low resistivity (3 Ω cm). The spacing between the two opposite electrodes is therefore very small, usually being about 1.8 μm, which results in rather homogeneous electric fields in the multilayer. The antisymmetric mode is coupled to the waveguide by means of a rutile prism. An applied voltage on the electrodes couples energy to the symmetric mode. Using a second prism, light is again coupled out and both modes can be measured separately. In order to couple, the spatial period Λ must match the effective index difference between the two modes according to

$$\Lambda = \frac{\lambda_0}{\Delta N_{\mathrm{eff}}}$$

By rotating the wafer with respect to the prisms this period can be varied as follows:

$$\Lambda = \frac{s}{\sin \alpha}$$

where α is the angle between the light beam and the electrodes and s is the deposited electrode periodicity.

Figure 6 shows the response of the mode coupler. The interaction length L was 2 mm or 124 periods. The sensitivity is dependent on the interaction length. The electrode pattern can be optimized for a maximum electric field modulation depth,

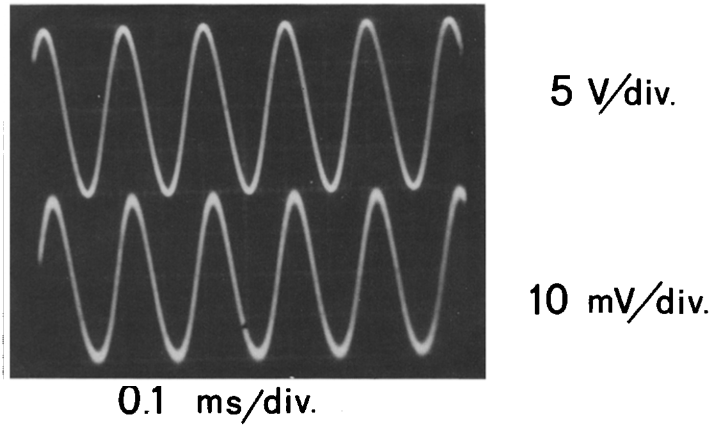

Fig. 6. Oscilloscope picture of mode coupler response: upper trace, driving voltage; lower trace, response of the symmetric mode (antisymmetric mode was launched).

and also to improve the sensitivity. We expect to improve the coupler to a 100% modulation for about 10 V.

4. CONCLUSIONS

We found that the optical quality of the sputtered ZnO films increases approximately linearly with increasing deposition rate (up to 220 nm min^{-1}). Results are very reproducible with respect to refractive indices and optical attenuation.

The sputter process gives excellent step coverages on etched SiO_2 profiles, making the production of low loss waveguiding channels feasible.

We developed a guided TE-to-TE mode coupler based on a multilayered waveguide. Good results were obtained with a first prototype which had a very short interaction length (2 mm). Increasing this length and optimizing the electrode pattern is expected to improve the sensitivity to a full-modulation voltage of 10 V.

ACKNOWLEDGMENT

These investigations were supported (in part) by The Netherlands Technology Foundation (STW).

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