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Stress in the Components of a Thin Film Silicon Monoxide Capacitor and its Relationship to Dielectric Loss

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The cantilevered substrate technique has been used to measure the stress in aluminium and silicon monoxide films deposited in sequence on glass substrates. The stress is compressive in aluminium and tensile in silicon monoxide although the latter may be changed by prior exposure of the aluminium film to atmosphere.

Electrical measurements were made on capacitors fabricated during the stress experiments. These showed that a definite relation existed between the dielectric loss and the stress.

Possible reasons are suggested for these results.

1. Introduction

Numerous papers have been published on stress in thin films (e.g. [1]) and the results for silicon monoxide have been particularly well documented [2-12], although incompletely understood [12]. Data are also available on the electrical properties of thin dielectric films, and silicon monoxide has been studied closely [23-25] because of its use in capacitors. Generally, the stress measurements have been made in isolation and no attempt has been made to relate them to other properties of the films. The present work was carried out on a more practical system in an effort to overcome this limitation.

The system chosen was the one often used in thin film capacitor fabrication and consisted of a silicon oxide film sandwiched between evaporated aluminium film electrodes. The stress was measured in each of these films and the electrical properties of the complete system were also investigated. However, it was impossible to measure both the stress and the electrical properties on the same substrate and the approach adopted was to deposit films simultaneously onto two substrates, one of which was used for the stress determination, and the other for the capacitor fabrication. Simultaneous deposition ensured that the composition of the dielectric film on the substrate used for stress measurements and for capacitor fabrication was the same.

The technique used for fabricating the capacitors differed in the following important respects from the usual one: (i) the films were deposited onto cold substrates; (ii) no annealing treatment was given after deposition.

Annealing is normally carried out on completed capacitors [3] and this has a marked effect on the electrical properties [2]. However, it would be exceedingly difficult to interpret stress measurements made onto hot substrates owing to the irregular thermal properties of glass [21]. Consequently, in order to allow a direct comparison to be made, all the measurements were carried out with the substrates at room temperature.

2. Experimental

The method used for the stress measurements was essentially the same as that described in an earlier paper [21]. The films were deposited onto thin cantilevered glass substrates and the resulting end deflections measured with a modified Talysurf probe after the lapse of a 3 min cooling period. The substrates were carefully annealed

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prior to the experiment to remove strain in the glass. The stress was derived from the Stoney formula [22] corrected to allow for bending across the width of the substrate [23], viz:

$$S = \frac{4L}{3w(1-\gamma)} \frac{\Delta}{m t_s t_f} \tag{1}$$

where Δ = deflection of substrate at distance L from the clamped end, w = width of substrate, γ = Poisson ratio of substrate, t_s = thickness of substrate, t_f = thickness of film, and m = slope of the deflection added weight line in the Young's modulus experiment.

The aluminium was evaporated from tungsten helices. The crucible for silicon monoxide was optically baffled to avoid spattering [24]. These sources were grouped symmetrically about a point immediately below the centre of the cantilevered substrate. The distances between the sources and the substrate were ~ 15 cm and the vapour impingement was at normal incidence. Excessive radiation was kept to a minimum by enclosing the sources as far as possible in a water-cooled copper box, and aluminium radiation baffles were also positioned between the sources and cantilevered substrate. Deposition rate was indicated by a quartz crystal thickness monitor and was controlled by manual adjustment of the source current; the crystal was positioned at the side of the substrate to minimise the effect of thermal drift. A subsidiary glass substrate, similarly placed, was used in conjunction with a movable mask to enable the films of aluminium and silicon monoxide to be deposited directly onto the glass for subsequent thickness measurements to be made with a Talysurf [25]. Preliminary experiments indicated that the thicknesses of these films were identical to those deposited on the cantilever.

The location of the rate-meter crystal meant that the rate could not be set in the conventional way: with the main shutter closed, the crystal was also isolated from the sources. The procedure was to heat the appropriate source to a temperature just below that at which evaporation was known to occur. The shutter was then moved aside and the rate set with the rate-meter. The short time occupied in performing this operation was insignificant compared with the total deposition time.

After the deflection had been measured, the system was let up to air, known weights were added to the end of the cantilever and the value of m deduced from the corresponding end deflections.

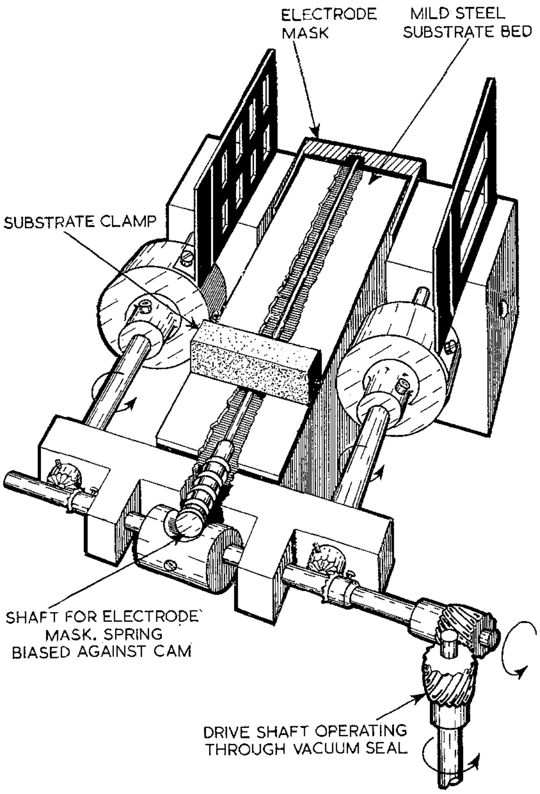

Figure 1 Mask changer for fabricating capacitors.

A special jig, illustrated in fig. 1, was constructed to enable electrical measurements to be made on the dielectric. This was clamped to the top plate of the vacuum system and could be activated through a rotary vacuum seal. It allowed eight small capacitors to be fabricated during the stress experiment. Of the three positions for the masks, two are for depositing aluminium electrodes and the third, with both masks rotated at right angles to the substrate surface, for silicon monoxide. In this open position, a subsidiary shutter protects the end of the bottom aluminium electrode from silicon monoxide. The capacitor jig was located at the side of the stress jig. The glass substrates used in the jig were the same as those used in the stress jig and had been subjected to the same pre-treatment. The total variation in thickness over the stress and capacitor jigs was 10%.

Capacitors were fabricated by ensuring that the masking was appropriate to the material being evaporated for the stress experiment. After the system had been let up to air, electrical measurements were made on each of the eight small capacitors with a Universal Bridge (Wayne Kerr A221) at a frequency of 1592 c/s ($w = 10^4$). Average values of capacitance and loss were tabulated and the curves plotted from these. For silicon monoxide, the pressure during evaporation was not greater than 5×10^{-5} torr. All depositions were made at a rate of less than 30 Å/sec. The aluminium was evaporated at similar pressures and rates of 15 to 20 Å/sec. For capacitor measurements, the aluminium electrodes were at least 500 Å thick. No attempt was made to investigate the properties of silicon monoxide in films less than 1000 Å thick. In some of the later experiments, the stress and electrical characteristics of the silicon monoxide were examined after the bottom electrodes had been exposed to the atmosphere for periods of up to 5 min.

3. Results

3.1. Stress in Aluminium Films

Fig. 2a illustrates the experimental readings plotted in terms of a force factor/thickness curve. Readings are shown for aluminium films deposited directly onto glass and for aluminium deposited on top of the silicon monoxide film. This factor F is defined by the equation

$$F = \frac{\Delta}{m t_s} \tag{2}$$

As previously discussed [21], this method avoids introducing any thickness error twice over, as would be the case if the stress had been calculated directly. Fig. 2b shows the corresponding stress/thickness curve. In the films examined, the stress was always compressive and showed little variation for thicknesses above ~ 400 Å. There were no significant differences between the stress values recorded on glass and on the silicon monoxide film.

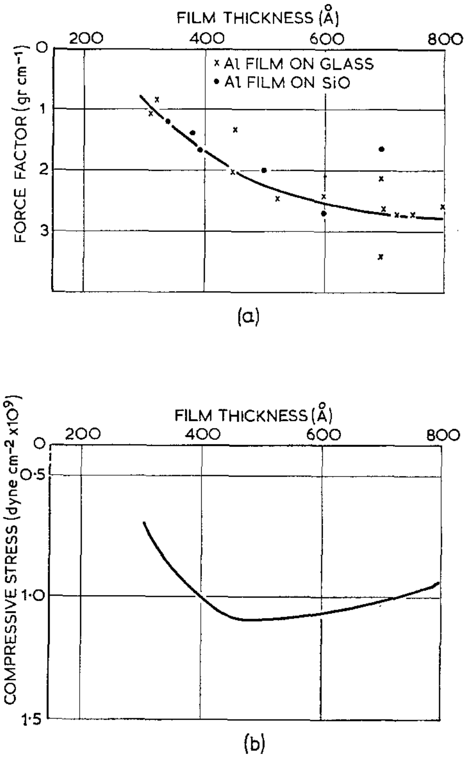

Figure 2 Stress data for aluminium: (a) force factor curve, × deposited on glass, ● deposited on SiO; (b) stress curve.

3.2. Stress in Silicon Monoxide Films

Figs. 3a and b show the data for the silicon monoxide films in terms of force factor against thickness and stress against thickness. The data show measurements made both with silicon monoxide layers deposited onto the initial aluminium electrode, and with silicon monoxide deposited on top of the upper aluminium electrode. The latter set of measurements corresponds to the situation arising when a

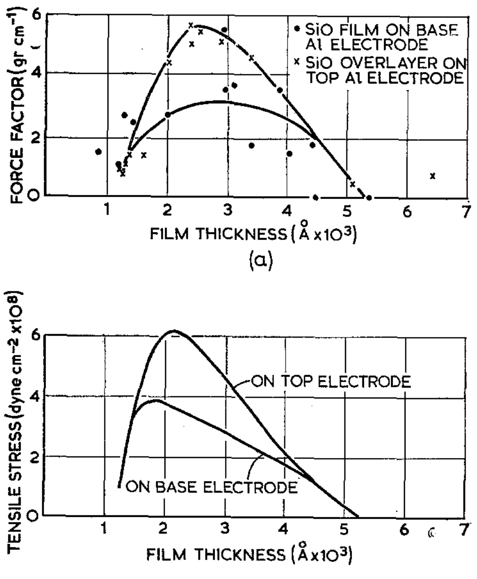

Figure 3 Stress data for silicon monoxide: (a) force factor curve, × SiO overlayer on top Al electrode, ● SiO film on base Al electrode; (b) stress curve.

protective silicon monoxide overlayer is deposited. A tensile stress is present in both cases, but it is somewhat larger when the films are deposited onto the top aluminium electrode. There is a fairly well pronounced stress maximum occurring in this case at a thickness ~ 2500 Å, and a tendency for much smaller stresses to occur in thicker films.

3.3. Stress and the Electrical Characteristics

Possible relationships between the electrical characteristics and the stress in silicon monoxide films were examined in another series of experiments. The thicknesses of the silicon monoxide films prepared in these experiments varied between 2000 and 4000 Å. The parameters are summarised by figs. 4 to 6 which show data for both air-exposed and unexposed aluminium films. Fig. 4 shows the stress values, obtained directly from equation 1, plotted for various film thicknesses as a function of the deposition rate/pressure ratio and is included to illustrate that for deposition on unexposed aluminium the stress was independent of this function. Fig. 5 shows a capacity/reciprocal of film thickness plot for the same set of readings. The slope of the straight line plot obtained in this way gives a value of 6.5 for the dielectric constant of the film. The most interesting result is illustrated by the loss/stress plot of fig. 6, for the same series of readings. The points lie on a fairly well defined curve with the high loss values corresponding to high tensile stress and low loss values corresponding to compressive stress. At zero stress the loss is about 1%. Furthermore, the silicon monoxide films which were deposited onto air-exposed aluminium are in much lower tensile, or even compressive stress, and these show low loss.

4. Discussion

Measurements made on aluminium films between 300 and 800 Å thick, deposited on both glass and aluminium, showed that the stress was compressive and ~ 10^8 dyne/cm^2. Little prior work has been reported on evaporated aluminium: Murbach [26] noted that films deposited on copper substrates were in tensile stress and attributed this to contraction of the film as it cooled from the recrystallisation temperature. Ennos [27] observed a compressive stress in a 400 Å aluminium film deposited on a glass substrate which was partly relieved at the end of evaporation but not affected by exposure of the film to moist air. Recently, d'Heurle [28] found that in the case of aluminium films deposited on oxidised silicon substrates, a changeover from tensile to compressive stress occurred as the residual oxygen pressure rose above 5 × 10^{-6} torr. These measurements were made on cold silicon-silicon oxide substrates which had been maintained at 200° C during deposition of the film—conditions which might have been expected to yield a tensile stress.

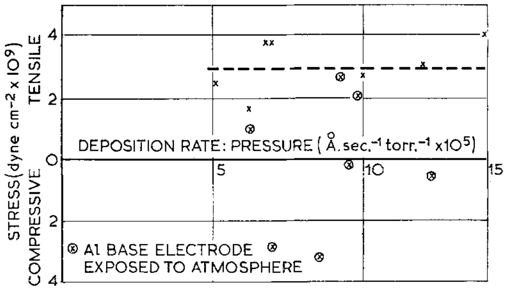

Figure 4 Stress in silicon monoxide as a function of the deposition rate/pressure ratio.

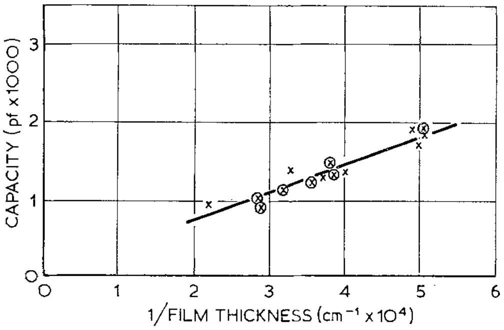

Figure 5 Capacity as a function of the reciprocal of thickness.

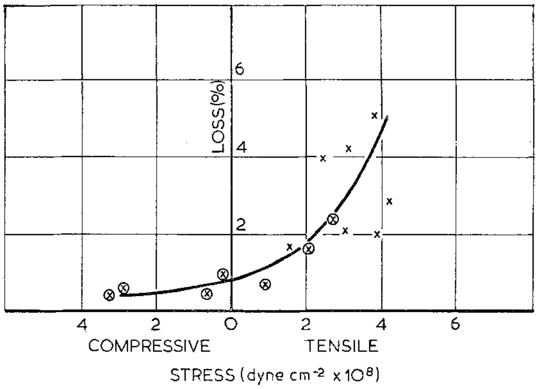

Figure 6 Loss as a function of stress.

An explanation of the compressive stress on the basis of a thermal model seems to be unlikely. Murbach's [26] model always gives rise to a tensile stress. The other thermal effects possibly leading to stress are the bulk temperature rise and the temperature gradient resulting from the incident heat flux. The latter effect is small and produces a tensile stress [29]. The bulk temperature rise also leads to a tensile stress; however for a typical rise of 10° C in an aluminium film deposited on glass, the calculated value of 10^8 dyne/cm^2 is an order smaller than that observed.

The most likely explanation for the observed stress is the presence of oxide impurities which produce strain in the metal lattice as they occupy a greater volume than the equivalent mass of aluminium. The oxide could form as inclusions in the metal lattice or as a film at the free surface [30, 31].

The stress observed in silicon monoxide films is in agreement with the observation that the stress is a function of the ratio of the arrival rates of silicon monoxide to oxygen molecules [12]; under the experimental conditions used, a tensile stress would always be expected.

The stress/thickness curves, which are derived from experimental points showing a wide scatter, are not well defined. Maxima occur at ~ 2000 Å for deposition onto both first and second aluminium films, but the maximum is sharper in the latter case. No satisfactory explanation can be offered for the presence of maxima at this particular thickness. Exposure of the aluminium underlayer to atmosphere prior to deposition, results in a more compressive stress. Hill [12] has observed a similar tendency when silicon monoxide films themselves are exposed to air. It is probable that the change arises from adsorption of water vapour on the aluminium film [4, 30] and a subsequent tendency to form the higher oxide of silicon, which has a compressive stress because of the inclusion of oxygen into the structure. No difference could be detected between the measurements of the dielectric constant in films deposited on exposed and unexposed aluminium, so it must be concluded that such oxidation is on a small scale: the value for SiO is 6.0 and for SiO_2 3.8, so a lower value might be expected for the air-exposed films. The high value measured (6.5) could result from the presence of adsorbed water or silicon impurity [13] in the films.

The interesting aspect of this work is the relation between the dielectric loss and intrinsic stress illustrated in fig. 6. A typical unannealed silicon monoxide film contains many holes, as revealed by etching [32]. Annealing eliminates most of these holes and produces a sharp drop in the loss. This indicates that the main conduction paths in unannealed films are through the holes. The actual conduction mechanism is not known [33], although a thin adsorbed surface layer of water could provide the conduction path. On this hypothesis the conduction can be thought of as occurring through the presence of weak paths and not as a bulk effect. Elimination of these weak paths would tend to reduce the loss. Films deposited in compressive stress tend to contract and would be expected to contain fewer holes and thus result in a lower loss.

5. Conclusions

The results of this study of the silicon monoxide-aluminium capacitor system are as follows:

(*i*) The stress in the aluminium films deposited both on glass and silicon monoxide is compressive and probably results from the presence of oxide impurity.

(*ii*) The stress in silicon monoxide is normally tensile and is in agreement with previous work. Exposure of the initial aluminium film to air results in the stress becoming more compressive. The compressive stress probably arises from increased oxidation of the silicon monoxide resulting from water vapour adsorbed on the aluminium.

(*iii*) Capacitance measurements do not indicate any systematic difference between the dielectric constant of films deposited onto exposed and unexposed aluminium films. The enhanced oxidation of the silicon monoxide must therefore be on a relatively small scale.

(*iv*) A relationship exists between the dielectric loss and stress in unannealed films. Films in compressive stress show lower loss. A self-consistent model based on conduction through holes in the film is proposed to account for this observation.

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