THERMAL STRESS IN THIN FILMS USING REAL-TIME HOLOGRAPHIC INTERFEROMETRY

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INTRODUCTION

With the numerous applications of thin films, the mechanical properties of thin films are becoming increasingly important. Of these, the fact that internal stresses exist in thin films has been known for over a hundred years. Stresses in evaporated thin films have been a topic of study by many workers and many good reviews have come out (1,2). Besides the internal stress, the thermal stress arising out of the difference in thermal coefficient of expansion of the substrate and film material also becomes important. Though much data is available for internal stress in thin films, the data for thermal stress is scanty. It is in this context a study has been undertaken to set up a novel technique of measurement of thermal stress of thin films using real-time holographic interferometry. Using this technique thermal stress in conductive coatings has been studied and some results on iron and copper films are presented.

THERMAL STRESS

When a thin film is deposited on a substrate, due to the various physical conditions a stress is induced in the film. The stress manifests itself by bending the substrate. Hoffman (1) has analysed that the total stress in the film consists of (1) the contribution due to the differential thermal expansion of the substrate and the thin film material and (2) the stress induced due to the various parameters of nucleation and growth, which is usually termed as the intrinsic stress. Experimentally it is difficult to separate out the two.

Thermal stress is given by the equation

$$\sigma_T = \frac{E_f \ \varepsilon}{(1 - \nu_f)} \tag{1}$$

where ε = the strain due to differential thermal expansion

E_f = Young's modulus of elasticity of the film

ν_f = Poisson's ratio of the film

E is calculated from the equation

$$\varepsilon = \int_{T_M}^{T_S} (\alpha_f - \alpha_s)\, dT \tag{2}$$

where T_S and T_M are the substrate temperature during formation and measurement.

In most thin film devices the intrinsic stress is the predominant criterion. However in some devices the thermal stress is a matter of considerable concern. Examples of such devices are conductive coatings on glass for windshields and window glasses. These are subjected to thermal cycling effects. In such devices the increase of thermal stress beyond certain limits leads to the rupture and failure of thin films.

This paper concerns itself with thermal stress in this context. When current is passed through a conductive coating of a metal film or a dielectric film the temperature of the film increases due to the $i^2 R$ loss. The substrate temperature also increases. Due to the difference in thermal coefficient of expansion of the substrate and the film material, thermal stress is induced. The authors believe that the thermal stress in this case also can conveniently be represented by equation (1). Experimental determination of the thermal stress has been carried out for the first time by real-time holographic interferometry and an attempt has been made to compare theoretical and experimental results.

EXPERIMENTAL DETAILS

For the study of thermal stress of thin films the authors have used two configurations of the thin film object (1) reflection mode and (2) transmission mode. Earlier the authors have described a technique of measurement of intrinsic stress in thin films using real-time holographic interferometry (3). A brief description of the experimental details is given here.

Real-time holograms can be taken by in-situ processing using monobath and liouid pate (4). A demountable kinematic substrate holder which can be relocated with great accuracy is used for the study of the thermal stresses (3). Real-time holograms formed in the set up are of such high quality that the zero fringe condition can be obtained easily and is constant even after removing and relocating the substrate holder.

REFLECTION MODE

A microscope slide coated with a transparent conductive coating of Indium oxide is mounted as a cantilever on the kinematic substrate holder. Soldered leads are taken from thick evaporated gold films at the ends of the conductive film. This enables current to be passed through the film. A hologram using Agfa 10E75 plate **is** taken and processed in-situ. On looking through the hologram which is in real-time, with both the reference beam and object beam on, no fringes are seen on the substrate. The hologram is moved in its own plane using an X-Y translation stage. Straight interference fringes are superimposed on the object scene. Passing current in the film causes a rise in temperature of the film due to i^2 R dissipation. This results in an expansion of the thin film and the substrate. Initially a shift in the fringes on the cantilever is observed. As the current is increased temperature rises and the cantilever bends. It is observed that there is a change in the curvature of the fringes. At higher currents distorted fringes are seen. At still higher currents the film ruptures and most often the substrate breaks.

Figure 1 shows the typical experimental results (5). The distortion in the fringes can be attributed to the non-uniformity of film thickness which results in a non-uniform resistivity. Fringes seen at the loading end of the cantilever are due to the relaxation of the loading which occurs if sufficient time is not allowed between fixing the cantilever and taking the hologram.

TRANSMISSION MODE

In the former case the substrate holder had a metal background and due to the temperature change of the substrate the background would also get heated and show distorted fringes. A refinement of the technique has been made by having only a vertical post on the demountable substrate holder on which the cantilever substrate can be fixed. Now it is possible to transmit the object beam through the substrate. Instead of transparent microscope slides used in previous experiments slides ground on one side with carborundum 120 grade abrasive has been used as the substrate. Metal films of iron and copper **of** different thicknesses have been coated on these substrates. Leads have been attached using conductive silver paint. A copper-constantan thermocouple is also attached on the film for the measurement of temperature. A Hewlett Packard 5 1/2 digit multimeter is used for the measurement of the thermo emf.

Real-time holograms are taken as described earlier. Increasing voltages applied to the thin film results in the bending of the cantilever which is manifested in increasing number of fringes from the free end. observed through the hologram. For different voltages the temperature of the substrate and the number of fringes are noted. Figure 2 shows experimental results for an iron film.

The theoretical thermal stress is calculated from the equation (1) . The values of the coefficient of thermal expansion, Young's modulus of elasticity and the Poisson's ratio is taken from the tables.

Experimental thermal stress is calculated as follows.

For a cantilever

$$F = \frac{3EI}{l^3} \Delta \qquad (3)$$

where F is the force causing the maximum deflection A at the free end of the cantilever. 1 is the length of the cantilever and I is the moment of inertia given by

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where b is the width of the substrate and h is the thickness of the substrate.

$$F = \frac{3Ebh^3}{12\, l^3} \Delta = \frac{E b h^3}{4 l^3} \Delta$$

Thermal stress of the film

σ_T = Force per unit area of the film

$$= \frac{E tah^3}{4 l^3 b h_f} \Delta$$

where h_f = thickness of film

$$\sigma_T = \frac{E h^3}{4 h_f l^3} \Delta \qquad (4)$$

To prove that the stress in the film causes the bending of the cantilever a glass slide without the film is subjected to a temperature change. No fringes are observed for the reason that holographic interferometry is less sensitive to in-plane displacement. The thermal coefficient of expansion mismatch causes the bending to occur.

The deflection of the substrate is measured in terms of the fringes that occur on the substrate for different temperatures. Theoretical and experimental results for iron and copper films are compared and presented in Tables 1 and 2.

CONCLUSION

From tables 1 and 2 it can be observed that there is a fairly good agreement between the theoretical and experimental thermal stress values measured using real-time holographic interferometry. The small deviations occur because of the ambiguity in the counting of the fractional fringe and the measurement of the temperature. It has been demonstrated here that thermal stress can be quantitatively measured using holographic interferometry. Though it has been applied to the case where thermal stress predominates over intrinsic stress, it is believed that it would be equally applicable where the total stress includes both types of stresses.

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TABLE 1 - Comparison of theoretical and experimental thermal stress for iron film

AT °C	Theoretical σ_T (equation 1) x 10^{6} dynes per cm^2	Number of fringes	Experimental σ_T (equation 2) x 10[illegible] dynes per cm^2
8.5	37.79	1	36.2
18.1	80.02	2	72.4
35.75	158.01	4	144.8
44.8	198.01	5.5	199.0
60.5	267.0	7	253.0
73	322	8.5	307.0
85	375	10	362.2
100.5	444.2	12	434.0
116	512	14	506
136	601	17	615

Substrate length 5 cms, thickness 0.115 cm, iron film thickness 300 Å.

TABLE 2 - Comparison of theoretical and experimental thermal stress for copper film

AT °C	Theoretical σ_T(equation 1) x 10^7 dynes per cm^2	Number of fringes	Experimental σ_T(equation 2) x 10^7 dynes per cm^2
4.5	7.128	1.5	7.38
8	12.672	2.5	12.3
11.75	18.612	3.5	17.22
16.5	26.132	5.5	27.06
23.0	36.432	7.5	36.9

Substrate length 5 cms, thickness 0.11 cm, copper film thickness 200 Å.

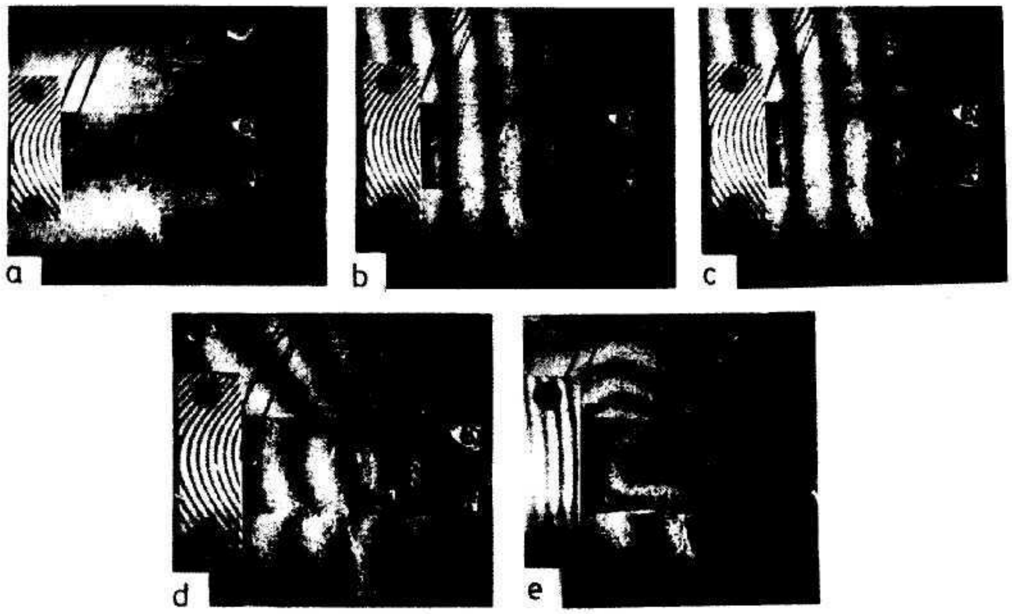

Figure 1 Thermal stress in Indium oxide thin film by real-time holographic **interferometry**: Reconstruction of Cantilever substrate (a) Zero fringe, zero current condition. (b) Fringes introduced by raovinp hologram in its own plane, (c) Current 40 mA, voltage 60 V, (d) Current 100 mA, voltage 130 V, (e) Current 150 mA, Voltage 190 V.

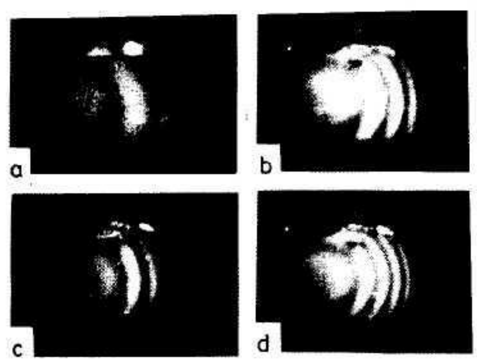

Figure 2 Thermal stress in Iron thin **film**: **(a)** AT = 37 C [b] AT = 45°C (c) AT = 68.5°C (d] AT = 90.5°C.