Thin Film Thickness Measurement Using Conduction and Infrared Transmission

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ABSTRACT

Many applications in a diverse demographic of engineering fields require knowledge of the thickness of thin films and coatings. An example of some applications for which thin film thickness is critical are semiconductors, optics, and protective coatings. There are various methods for measuring thin film thickness, however, many of them are destructive, or are only capable of measuring thickness on a point-by-point basis. This work presents a full-field method of measuring film thickness by conductive or radiative heat transfer. The heat transfer models describing the response are explained, and potential applications of this technique are shown in experimental testing of a spray paint coating and gold thin films.

EXPERIMENTAL PROCEDURE

In the initial phase of testing, polymer plaques were painted with several steps of increasing thickness of Krylon® Ultra Flat Black (KUFB) spray paint. Two polymers were used: polymethylmethacrylate (PMMA), and Delrin®, a DuPont acetal resin. The KUFB spray paint was used as it had been used for previous work of the same objective, only using thermoelastic generation as the heating method [1]. The steps of increasing thickness were created by evenly spraying the entire specimen on one side, allowing for an appropriate drying time, and then shielding a portion of the specimen, and spraying the remainder of it again. This process was repeated to create several steps of increasing paint thickness, each time shielding an increasing length of the specimen. A Mitutoyo Contracer CBH-400 profilometer was used to measure the coating thickness to within ±4 µm. These measurements were made after testing was completed to ensure that the coating surface was not damaged. The thickness of the KUFB coating steps ranged from 15-71 µm.

A second set of specimen was made with 1 mm thick silicon wafer substrates, with a thin film of gold deposited on them by evaporation. The evaporator was a BOC Edwards Auto 301 Evaporator, with the deposition chamber pressure and specimen temperature being an average of 10⁻⁷ Torr and 72

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THERMAL MODELING

Previous work by McKelvie [2] focused on determining the amount of attenuation and phase lag of a thermoelastic heat source through a thin spray paint coating. This thermoelastic heating was created by an oscillated loading of the substrate material, as described by Equation 1,

$$\Delta T = \left(\frac{a}{\rho C_p}\right) T \left(\frac{\Delta \sigma_{kk}}{3}\right)$$  \hspace{1cm} (1)

where $\Delta T$ is the local change in specimen temperature, $a$ is the coefficient of thermal expansion, $\rho$ is the specimen density, $C_p$ is the specific heat at constant pressure, $T$ is the specimen temperature, and $\Delta \sigma_{kk}/3$ is the change in hydrostatic stress. As the thermoelastic generation of the coating is much less than that of the substrate, it is neglected by McKelvie, and the heat generation of the substrate is simply considered to be an oscillated temperature at the substrate / coating interface. This same model can describe the chopped light heating of the polymer specimen in this present work. This is because the polymers are transparent in the near to mid infrared, and the majority of the heat flux from the light source is therefore absorbed at the polymer / KUFB paint interface.

The McKelvie model describes the signal attenuation as a ratio of the temperature at the outer coating surface, $T_o$, to the temperature at the coating and substrate interface, $T_i$, as follows

$$\frac{T_o}{T_i} = \frac{1}{\cosh \left( \frac{\omega}{2 \chi} \right) \ast (1+i)}$$  \hspace{1cm} (2)

where $\omega$ is the test frequency, $\chi$ is the thermal diffusivity of the coating, and $t_i$ is the coating thickness. The phase lag of the surface temperature amplitude with respect to the testing frequency is described by the argument of this complex number. This model was developed from the Carslaw and Jaeger [3] model of the 1-D heat conduction through a slab having a periodic surface temperature on both surfaces. In the present application the paint coating is considered to be half the slab thickness, the outer surface of the paint being the center of the slab, and the thermoelastic generation of the substrate being the periodic surface temperature.

As the thermal diffusivity, density, conductivity, and other paint properties were not readily available, the coating was modeled as a particulate composite and these properties were calculated. A rule-of-mixtures approach was used to calculate the density and specific heat [4], and the Budiansky model used to calculate conductivity [5].

To obtain measurements of thin film thickness with resolution on the nanometer level, a model of heat transfer via the transmission of radiation is used. In the near to mid-infrared range, metals are greatly absorbent, causing the intensity of an infrared wave to be greatly attenuated. The amount of this decay is exponentially dependent on the thickness of the film. Using a fixed wavelength band of the spectrum, the intensity decay with thickness can be calculated for any material with known optical constants. Basic forms of these calculations can be found in radiative heat transfer texts [6]. They are the basis of any calculation of reflectivity, absorptivity, and transmissivity, and only require knowledge of the wavelength of the electromagnetic spectrum and the optical constants of the participating materials. The basic equation required for the calculation of the intensity decay through an absorbing medium is:

$$\tau = \exp \left( -\frac{4\pi kd}{\lambda_o} \right)$$  \hspace{1cm} (3)

where $\tau$ is the transmission through the material, $k$ is the material absorption constant, $d$ is the thickness or distance traveled, and $\lambda_o$ is the wavelength of the light in a vacuum. This most basic form is elaborated upon by considering variable angles of incidence, reflections, and re-transmission at interfaces due to differing optical constants, and the interference of these waves with the incident waves. However, the values of these other factors are all either known, can be calculated, or can be fixed to a known value.

EXPERIMENTAL RESULTS

An example of the absolute thermal signal data taken from the PMMA sample with the KUFB coating is shown in Figure 1. The variation in this signal intensity is directly proportional.
to the radiative flux emitted from the surface. The absolute thermal signal data is scaled by the maximum thermal signal from the thinnest coating layer to obtain a relative intensity.

**Figure 1:** Absolute thermal signal of PMMA with stepped KUFB paint at 44 Hz.

The phase and normalized thermal signal experimental data are fit with McKelvie model, Equation 2, using the thermal diffusivity of the coating as a fitting parameter. The plot of the McKelvie predictions versus the normalized thermal signal and the relative phase lag are shown in Figures 2 and 3. The best fit was obtained using a thermal diffusivity of $0.6 \times 10^{-7}$ m$^2$/s for the KUFB coating on the PMMA substrate, and $0.9 \times 10^{-7}$ m$^2$/s on the delrin substrate. In order to validate the diffusivity values extracted from these fits, the thermal diffusivity was also calculated by modeling the paint as a particulate composite. After calculating the conductivity of the paint with the Budiansky model, and the density and specific heat with the rule of mixtures, the diffusivity was calculated from these parameters to be approximately $1.4 \times 10^{-7}$ m$^2$/s. Previous testing [1] relying on thermoelastic generation found the thermal diffusivity of the KUFB paint on a steel substrate to be $1.7-2.0 \times 10^{-7}$ m$^2$/s. The differences in diffusivity found by this testing, the testing using thermoelastic generation, and the composite model are likely due to differences in surface quality and interface conduction.

**Figure 2:** Normalized thermal signal through PMMA / KUFB specimen compared to McKelvie model.

Line scans of the absolute thermal signal were taken from each of the silicon wafer / gold thin film data images, and scaled by the average thermal signal from the uncoated portion of the wafer. This resulted in a relative measurement of the intensity as dependent only on the thickness of the gold thin film. The normalized thermal signal line scans for several different film thickness levels are shown in Figure 4.

**Figure 4:** Normalized thermal signal profiles from gold thin film specimen.

Figure 5 shows the anticipated fraction of infrared intensity transmitted through various thin metal films at an average wavelength of 4 µm from normally incident radiation. This predicted response is calculated from elaborations on Equation 3. The values of the absorption constant, k, used for the predicted response were taken from Palik [7]. As the films are greatly absorbent, interference fringes are not seen. Also shown on Figure 5 is the experimental data from this testing of gold thin films, and other experimental data compiled by Moses [8] of transmittance through thin metal films. It can be seen that the experimental data do not exactly correspond with the predicted response, but are offset from each of their respective curves by a similar amount. However, each experimental data set is well described by an exponential fit, and from this fit, the
Experimental absorption constant can be back-calculated, and has been found to differ from tabulated values by approximately a factor of 2. Factors that must be considered in light of this discrepancy in absorption constants are that thin film optical constants are known to vary given different deposition methods and substrate materials, and that this testing did not occur in a controlled vacuum immediately after deposition, possibly allowing the formation of a surface oxide or contaminant layer.

**Figure 5:** Theoretical and experimental transmission of normally incident mid-infrared (3-5 µm) radiation through various thin metal films.

**CONCLUSION**

It has been shown that the measurement of thin film thickness by conductive and radiative heat transfer is possible. For each of these methods, only a single parameter is required to fit the respective heat transfer model to the normalized experimental data. In a situation where these parameters could be accurately predetermined, this method could be used to measure film thickness. Given the simplicity of this testing scheme, only requiring a modulated heat or infrared source and an infrared camera system, there exists a great potential for this technique to be used to monitor thin film growth, or as a quality control tool.

In addition to the potential of measuring paint coatings and thin metal films, the thickness of other materials could be measured by selecting an appropriate frequency of oscillated heating, or a wavelength range over which the material to be measured is absorbent. Additionally, neither technique requires a substrate material, as the conductive method only requires a subsurface oscillated heating, and the transmission method only requires knowledge of the source intensity. As such, the method can be applied readily for measuring the thickness of free standing films.

**REFERENCES**


