

## THERMAL WAVE CHARACTERIZATION OF COATED SURFACES

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### INTRODUCTION

The experimental techniques and theory for utilizing the mirage effect, or optical probe beam detection, of thermal waves in opaque solids for determining their thermal diffusivities have been described in detail elsewhere. [1-4] An application to a coated nickel-based alloy has also been described elsewhere. [1] In previous papers [5,6] we presented a theoretical expression which describes the mirage effect signal in a three-layer medium (gas-coating-sample system), taking into consideration the effects of the sizes of the heating and probe beams. In this paper we extend the results of numerical calculations from that expression to the case of films which are thermally very thin (thicknesses of the order of  $10^{-3}$  thermal diffusion lengths). A model system of 100-500 nm thick Cu films on glass substrates was studied experimentally at thermal wave frequencies below kHz, and in this paper we compare the results of those measurements to the numerical calculations.

### DESCRIPTION OF TECHNIQUE

The experiment involves the measurement of the in-phase component of the transverse mirage effect displacement of a probe laser beam which skims or reflects from the coated surface in the vicinity of a modulated heating beam focal spot. This component is plotted as a function of the transverse offset distance of the two beams. These plots exhibit a zero-crossing at the center (corresponding to the coincidence of the two beams) and two zero-crossings at the 90° phase points on either side of the center. For a homogeneous material a measurement of the distance between the two 90° phase points is proportional to the thermal wavelength. Thus, the slope of a plot of this distance versus the reciprocal of the square root of the frequency is directly related to the thermal diffusivity of that material. For a coated surface, the behavior is more complex, but such plots still contain information about the lateral heat flow in the coating, and can be used in conjunction with the theory [5,6] to obtain information about the coating.

Using the theoretical expression provided in [6] one can easily compute plots corresponding to those of the experimentally measured zero-crossing distances versus the inverse square root of frequency. In Fig. 1 we show the theoretically computed slopes in the low frequency regions of such plots for different values of the height of the probe beam above the sample for uncoated materials whose diffusivities range from 0.1 to 1.3 cm<sup>2</sup>/sec. The isolated data points to the left of the curves in Fig. 1 indicate the values evaluated from the expression  $(1.4\pi\alpha)^{1/2}$ . One sees that this formula works quite well, provided that the height of the probe beam is kept low. At larger values of the probe beam height the slopes tend toward the value for air (taken to be 0.3 cm<sup>2</sup>/sec in this calculation). Thus, it is important in doing such experiments to use an experimental arrangement which minimizes the height of the beam. The present measurements are done using a technique[7] to bounce the probe beam from the sample surface has been developed to minimize the distance between the probe beam and the sample.

Previously the theory had been used to analyze measurements from uncoated samples. From the present calculations one also learns that the zero-crossing values are very sensitive to the thickness of the film on coated surfaces if the substrate has a much lower effusivity ( $\kappa c\rho$ ) than that of the coating. The reason is not difficult to understand from the point of view of thermal wave propagation. A change from a large effusivity film to a low effusivity substrate at an interface causes thermal waves to reflect strongly with no change in phase, resulting in essentially two-dimensional heat flow in the film. This causes the phase of the temperature at the surface, and hence the zero-crossing distance, to vary, even if the film thickness is small. Thus, thermal wave propagation can be a useful tool to measure the thicknesses of very thin metal films deposited on insulating substrates. In Fig. 2 we show some theoretical plots of the crossing distance of copper film on glass as a function of the film thickness. At these frequencies the thermal diffusion lengths in

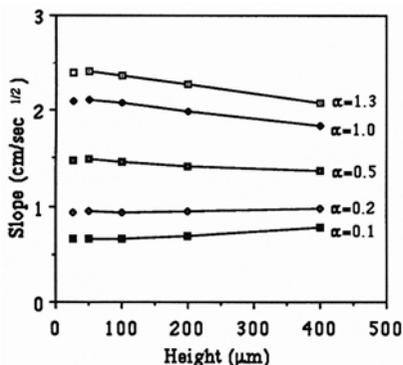


Fig. 1 Theoretical low frequency slopes of zero crossing distance versus inverse root frequency as a function of probe beam height for five different values of diffusivity of the solid. The isolated data points to the left of the curve represent the values evaluated from the expression  $(1.4\pi\alpha)^{1/2}$ .

copper are in the range of 230–450  $\mu\text{m}$ , about three orders of magnitude greater than the film thicknesses.

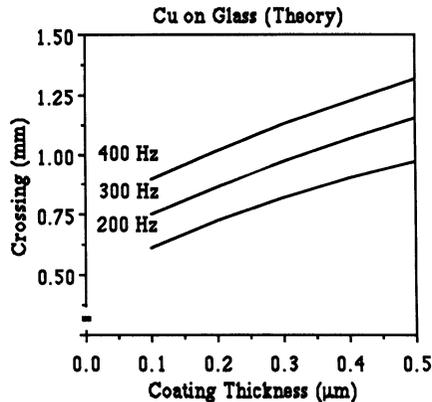


Fig. 2 Theoretical plots of the dependence of the zero crossing distance on film thickness for Cu on glass, for three different frequencies.

## RESULTS AND DISCUSSION

We have prepared some samples of copper films on glass by rf sputtering. The thicknesses of the deposited copper films were measured by Rutherford back scattering (RBS) with 2 MeV alpha particles. Figure 3 shows a typical RBS spectrum. It shows that there is considerable diffusion at the copper-glass boundary due to the high temperature of the sputtering process. This lack of definition of the boundary is the principal source of error in the measurement. The thermal wave zero-crossing distances measured on these samples are shown in Fig. 4, which is in good agreement with the theoretically computed values shown in Fig. 5. The RBS estimated thicknesses are summarized and compared with thermal wave measurements in Table 1.

The excellent agreement between the two methods of measurement indicates that the thermal wave technique provides a good way to measure film thicknesses of thermally thin conducting films on less conducting substrates. The idea of using a thermal wave technique to measure thicknesses of thin films depends on the reflection of thermal waves at an interface. Thus, one might expect the same idea to work on a thin film of low effusivity material deposited on a substrate of higher effusivity material. However, in the latter situation the lateral heat flow is dominated by the substrate rather than the film. Consequently, the high sensitivity to film thickness for films much thinner than a diffusion length would not be present.

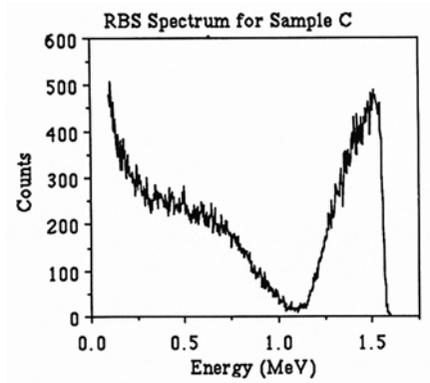


Fig. 3 Rutherford Back Scattering spectrum of sample C (see also Fig. 4 and Table 1).

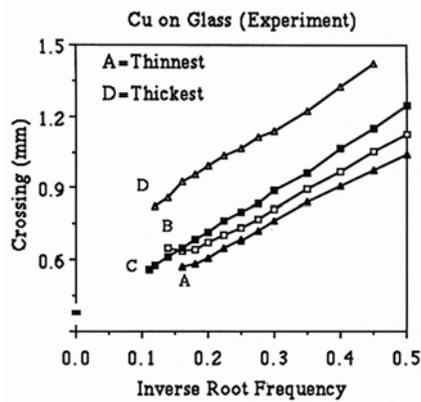


Fig. 4 Experimental plots of zero crossing distance versus inverse root frequency for four Cu/glass samples, whose thicknesses increase from A to D.

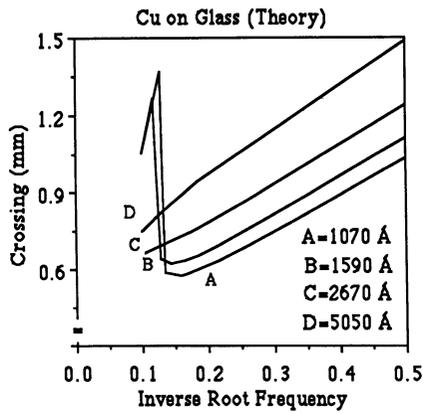


Fig. 5 Theoretical plots of zero crossing distance versus inverse root frequency with thicknesses which provide the best fits to the data of Fig. 4.

Table 1. Thicknesses of Cu films as determined by RBS and Thermal Waves

Sample	Thickness (Angstroms)	
	<u>RBS</u>	<u>Thermal Wave</u>
A	935 ± 130	1070
B	1450 ± 450	1590
C	2460 ± 660	2670
D	4470 ± 620	5050

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