

A STUDY OF THE EFFECT OF SURFACE METALIZATION ON THE THERMAL CONDUCTIVITY MEASUREMENTS BY THE TRANSIENT THERMO-REFLECTANCE METHOD

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ABSTRACT

This work is a numerical and experimental investigation of the effect of the use of a metallic absorption layer on the laser-based measurements of the thermal conductivity of dielectric, semiconductor, and highly-conductive materials. The numerical and supporting experimental results reveal the presence of behaviors associated with thermally-thin and thermally-thick absorption layers, depending on the ratio between the thickness of the absorption layer and the heat penetration depth. It is concluded that the TTR method performs optimally when the thickness of the metalization layer falls in the transition range between the identified thermally-thin and thermally-thick layers.

Keywords: *thermoreflectance, thin films, absorption layer, semiconductor, experimental, computational, heat transfer*

INTRODUCTION

The performance of electronic and telecommunication devices depends heavily on electro-thermal interactions, making the knowledge of material properties fundamental to the design process. The transient thermoreflectance method (TTR) [1, 2] is favored among experimental techniques [3] used to determine the thermal conductivity of thin-film and multi-layered materials. The main advantage of the TTR method is that it is a non-contacting and non-destructive optical approach, both for heating a sample under test and for probing the variations of its surface temperature [4, 5]. However, TTR measurements of the thermal conductivity can be hindered by less-than-desirable optical properties of the top layer material (i.e., low thermoreflectance coefficient, low reflectivity, high transparency, surface roughness, and oxidation), which degrade the measurement performance of a given system. In order to eliminate these difficulties, investigators have resorted to the use of a so-called metal "absorption" layer on top of the material under test (e.g., Au in [2] and Al in [6]). Metal films

are used because they exhibit high absorptivity and their optical properties are usually well known. Gold is a particularly attractive material for use as an absorption film because of good stability in its refractive index in a normal laboratory environment, sufficiently high thermoreflectance coefficient, and linear dependence between reflectivity and temperature in the range of up to 200 K change.

The motivation for this study arose from the discovery of a special behavior in the course of an experimental investigation of gold-covered SiO₂ samples. This behavior was later confirmed by a thorough numerical analysis for samples of differing thicknesses of SiO₂ and Au. It was suspected that this behavior might be caused by the significant difference between the thermal conductivity of Au and SiO₂, or in other words, that the thermal behavior is governed by the ratio of the thermal conductivities of the underlying material and of the metalization layer. Consequently, numerical studies were pursued for two underlying materials for which the thermal conductivity ratio would be closer to unity (e.g., Si) and much larger than unity (e.g., Diamond).

EXPERIMENTAL PROCEDURE

The schematic in Fig. 1 depicts the square heating and round probing spots produced by the TTR system in the SMU Submicron Electro-Thermal Sciences (SETS) Laboratory (<http://www.engr.smu.edu/sets1>). The source of energy in the TTR method is provided by a pulsed laser with short pulse duration. During each pulse, a given volume below the sample surface heats up to a temperature level above ambient due to the laser light energy absorbed into the sample. After each laser pulse is completed, the sample begins to cool down to the initial ambient temperature. During this process, the probing CW laser light reflected from the sample surface at the heating spot center (probing spot on Fig. 1) is collected on a photodetector that

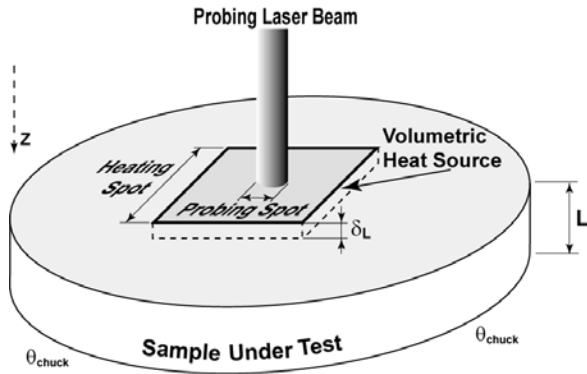


Fig. 1 Schematic of the heating and probing spot positioning on the sample under test.

reads the instantaneous surface reflectivity. The changes in surface reflectivity are linearly proportional to the changes in surface temperature, within a wide but finite temperature range. Finally, the photodetector signal, representing the variations in surface reflectivity, is acquired via a digital oscilloscope.

RESULTS AND DISCUSSION

The absorption layer within a substrate is depicted schematically in Fig. 2. Three length scales are sufficient to uniquely describe the heat transfer problem during pulsed laser heating in the TTR method; namely, the thickness of the absorption layer, h ; the optical penetration depth of the heating light, δ_L ; and the heat penetration depth during the pulse width, δ_H . Since the only absorption layer material considered in the present article is gold, it is reasonable to assume that the heat penetration depth is much bigger than the light penetration depth, i.e., $\delta_H \gg \delta_L$. It is equally reasonable to consider that the physical thickness of the absorption layer (e.g., gold) is bigger than the light penetration depth, i.e., $h > \delta_L$. Therefore, only two length scales need to be considered: h and δ_H .

Thermally-thick, thermally thin, and the transition regime for the absorption layer

In categorizing the heat transfer process in a layer of material, a distinction is made between layers that are *thermally-thin* and layers that are *thermally-thick*. When the thickness of the absorption layer, h , is larger than the heat penetration depth, δ_H , the layer has sufficient internal thermal resistance to support temperature gradients, i.e., the gold layer behaves as a thick plate. Thus, such a class of absorption layer is referred to as *thermally-thick*.

Two typical normalized temperature responses for *thermally-thick* layers of gold ($h_{Au} = 2 \mu\text{m}$ and $3 \mu\text{m}$) are shown in the middle part of Fig. 3. It is interesting to point out in reference to several of the curves in Fig. 3 that the temperature decay exhibits a sharp change of slope. The slope change corresponds to the time when the heat front reaches the less thermally conductive oxide layer, and is caused by the high temperature gradients developed at the interface between the

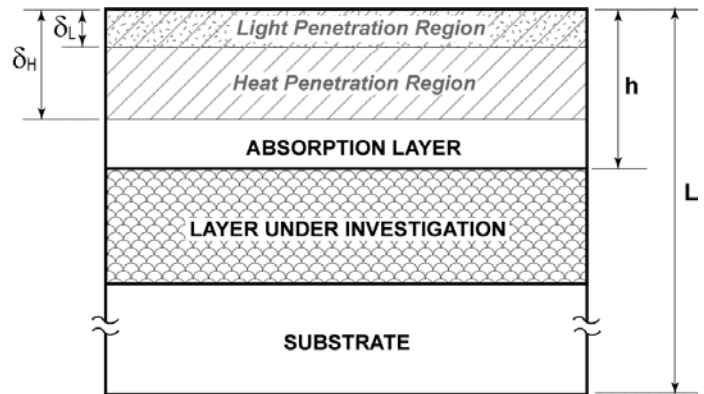


Fig. 2 Schematic of the absorption layer on the substrate.

highly conductive gold layer and the more resistive silicon dioxide layer. As expected, the time required for the heat front to reach the underlying oxide layer is longer for thicker absorption layers. At the upper limit, when the thickness of the absorption layer is bigger than h^* , the response shows that there is very little, if any, heat flow in the silicon dioxide layer, indicating that the bulk limit of the material has been reached.

The preceding discussion dealt with the thermal responses as the thickness of the Au absorption layer was decreased from infinity to $1 \mu\text{m}$. While the curve for $h = \infty$ represents the lower limit for the thermal response at the surface of the sample, the three upper curves in Fig. 3 ($h = 6000 \text{ \AA}$, 7000 \AA and 8000 \AA) represent the upper range. Further reductions in the thickness of the gold absorption layer will produce curves that fall below this upper range, depicting the behavior of *thermally-thin* absorption layers.

When the thickness of the absorption layer is smaller than the heat penetration depth during the heating pulse, the layer has insufficient internal thermal resistance to support temperature gradients, and as a result, the instantaneous

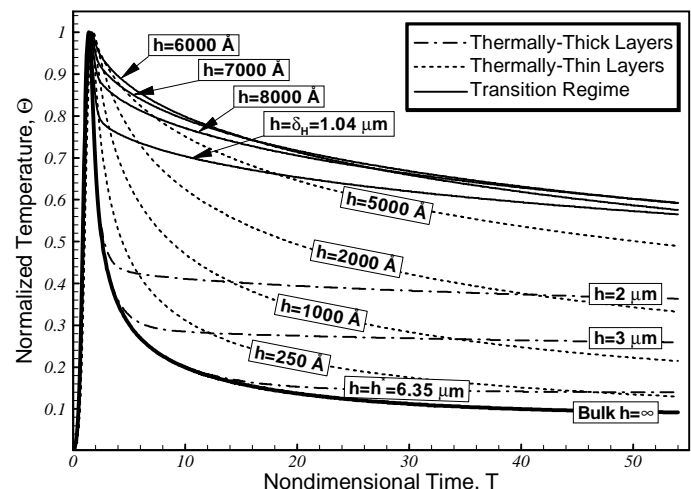


Fig. 3 Temperature responses of a SiO_2 bulk sample covered with Au.

temperature field is relatively uniform throughout the material. Consequently, this type of absorption layer will be referred to as a *thermally-thin* (also known as “lumped capacity”) layer.

Characteristic temperature responses for *thermally-thin* layers are also shown in Fig. 3, bracketed from above by the curve for $h = 6000 \text{ \AA}$ and from below by the curve for $h = \infty$. The light penetration depth, δ_L , is the lower limit for the thickness of an absorption layer. Therefore, layers thinner than δ_L are not considered since they are impractical for the TTR method.

The analysis performed in the present work has led to the association of the nondimensional thickness of the absorption layer, defined as $H = h(\alpha\tau)^{-1/2}$, with the different regimes of the normalized temperature response behavior. The computed normalized temperature responses for gold-covered silicon dioxide are plotted in Fig. 4, where a non-dimensional time based on the pulse width has been introduced, such that $T = t/\tau$. The temperature responses from Figs. 3 are shown at specific time instances, beginning with a time equal to twice the pulse width, i.e., $T = 2$, and ending with $T = 50$. This view of the results makes it possible to more easily identify the three different regimes which are entirely defined by the nondimensional thickness H . Behavior consistent with the *thermally-thick* regime appears for $H \gtrsim 1$, while behavior consistent with the *thermally-thin* regime occurs for $H \lesssim 0.4$. A *transition* regime between the *thermally-thin* and *thermally-thick* regimes is evident and occurs in the range of H that corresponds to the projection of the points for which $d\Theta/dH = 0$ (represented by the peaks of the curves plotted in Fig. 4). For the specific materials considered here, this transition occurs for $0.4 \lesssim H \lesssim 1$.

Results are presented for Si and diamond in Figs. 5 and 6, respectively, indicating that the defined regimes can be identified for materials within a wide range of thermal conductivity. The computed normalized temperature responses for silicon samples are plotted in Fig. 5 for different

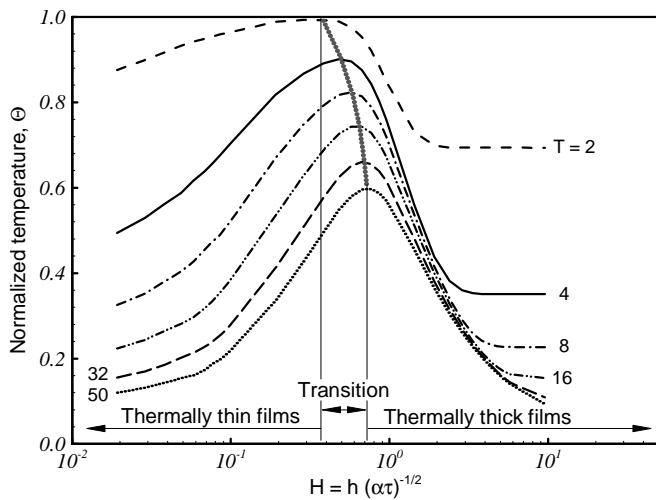


Fig. 4 Heat transfer regimes in Au covered SiO₂ samples.

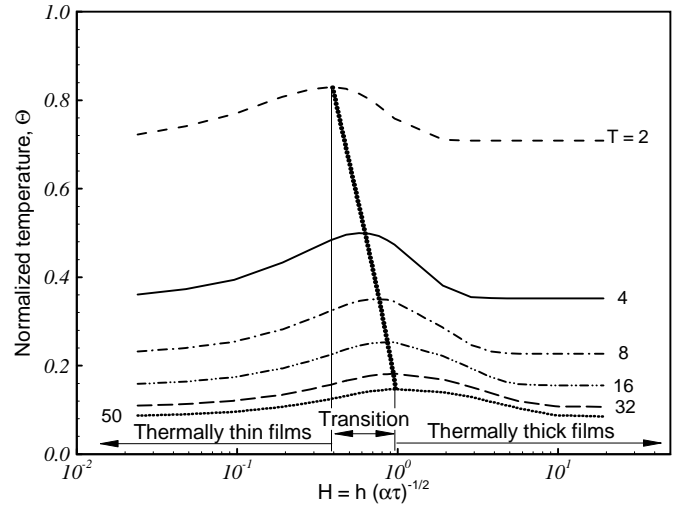


Fig. 5 Heat transfer regimes in Au covered Si samples.

nondimensional time $T = t/\tau$. As previously observed for gold-covered silicon dioxide samples, behavior consistent with the *thermally-thick* regime is evident for $H \gtrsim 1$, while behavior consistent with the *thermally-thin* regime occurs for $H \lesssim 0.4$. Transition between the *thermally-thin* and *thermally-thick* regimes for the gold-covered silicon sample is clearly visible and occurs again in the range $0.4 \lesssim H \lesssim 1$.

Corresponding results for gold-covered diamond are presented in Fig. 6. Two major differences are observed from the results shown above for Si and SiO₂. First, all of the transient surface temperature responses lie below the bulk gold curve (i.e., $h = \infty$). Second, increasing the thickness of Au in the *thermally-thin* regime results in the lowering of the transient temperature curve, while increasing the thickness of Au in the *thermally-thick* regime results in the raising of the temperature curve. The lowest curve exists in the transition regime. This behavior is the opposite of what was observed in the cases of Si and SiO₂ above.

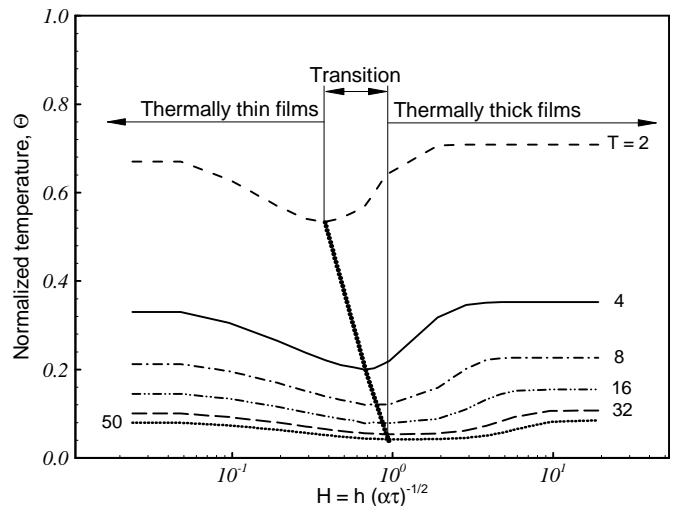


Fig. 6 Heat transfer regimes in Au covered diamond samples.

Experimental results

To validate the numerical results, experiments were carried out to investigate the influence of the thickness of the absorption layer deposited on a given semi-infinite thickness of SiO₂ with the expectation of observing the three different thermal behaviors, namely, *thermally-thin*, *transition*, and *thermally-thick*. The experimental and corresponding numerical results are presented in Fig. 7. The thickness of the absorption layer measured by a profiler for each sample is shown in the bottom left. The numerical methodology consists of solving the heat transfer problem for each given finite layer configuration and varying the unknowns until the RMS error between the numerical and experimental curves is minimized.

The behavior of the temperature responses of samples 1-3 is consistent with the previously discussed behavior of *thermally-thin* absorption layers, where the gold layer thickness is considerably smaller than the heat penetration depth during the pulse, and the cooling phase of the temperature response is completely dependent on the thermal properties of SiO₂. The decrease in the level of the temperature response evident as the gold layer becomes thinner is explained by the fact that the amount of energy accumulated during the pulse decreases accordingly with the reduction of the thermal capacitance of the absorption layer.

Samples 4 and 5 exhibit a behavior consistent with the *thermally-thick* regime whereby the diffusion of heat through the underlying oxide layer is discernable by the presence of an abrupt change of slope at small values of T (T ≈ 2.5 for sample 4 and T ≈ 3.5 for sample 5).

Responsivity of the TTR method

To further assess the performance of the TTR method, we introduced the *responsivity*, R_s , of the thermal conductivity measurement defined as $R_s = K(d\Theta/dK)_{max}$, where Θ is the normalized temperature response of the sample surface and K is

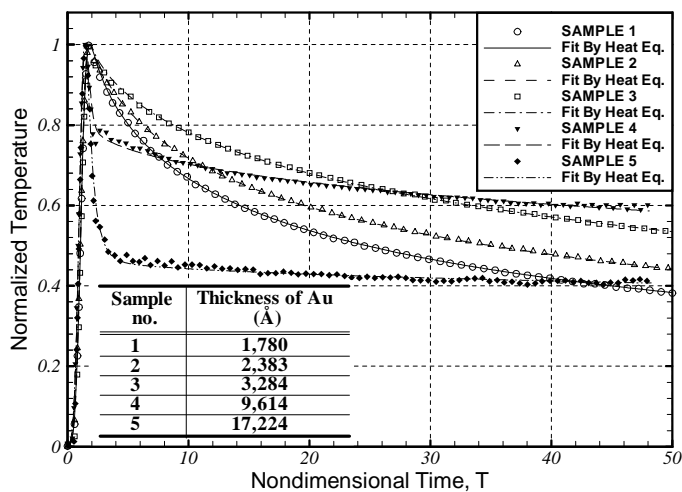


Fig. 7 Measured and computed temperature responses of a SiO₂ layer covered by different thicknesses of gold.

the thermal conductivity of the material. R_s is directly connected with the accuracy of the method by the equation $\sigma_K = R_s^{-1}\sigma_\Theta$, where σ_K is the random measurement uncertainty of the thermal conductivity, K , and σ_Θ is the random apparatus uncertainty related to detecting the temperature response. While σ_Θ depends on the apparatus signal-to-noise ratio and can be considered as a conservative value for a particular setup, the latter equation shows that the measurement uncertainty, σ_K , of the TTR technique decreases with increases in the responsivity value, R_s . Hence, the responsivity, R_s , which is dependent on the properties and geometry of the materials making up a sample as well as the functional characteristics of a given TTR system, can characterize the performance of the TTR method and be useful for optimizing an experiment. By numerically solving the heat equation for Θ , it was possible to compute R_s and determine the thickness of the absorption layer for each material sample that would yield the optimal performance of the TTR method. It was determined that the optimal thickness falls within the thickness range that corresponds to the *transition* regime.

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