Simultaneous thickness and thermal conductivity measurements of thinned silicon from 100 nanometers to 17 microns

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Studies of size effects on thermal conductivity typically necessitate the fabrication of a comprehensive film thickness series. In this letter, we demonstrate how material fabricated in a wedged geometry can enable similar, yet higher-throughput measurements to accelerate experimental analysis. Frequency domain thermoreflectance (FDTR) is used to simultaneously determine the thermal conductivity and thickness of a wedged silicon film for thicknesses between 100 nm and 17 µm by considering these features as fitting parameters in a thermal model. FDTR-deduced thicknesses are compared to values obtained from cross-sectional scanning electron microscopy, and corresponding thermal conductivity measurements are compared against several thickness-dependent analytical models based upon solutions to the Boltzmann Transport Equation. Our results demonstrate how the insight gained from a series of thin films can be obtained via fabrication of a single sample.

Phonon scattering from boundaries and interfaces is an extensively studied source of thermal conductivity reduction in crystalline materials.4–5 As material dimensions are reduced in size, the frequency of scattering is increased.6 Systematic studies which experimentally demonstrate thermal conductivity size-dependence, however, are often challenged by the need to fabricate numerous samples that span an array of thicknesses.7–9 In this study, we show how materials fabricated with a wedged shape can allow for analysis of thickness-dependent thermal conductivity with a single sample. In particular, we utilize FDTR to simultaneously measure the thermal conductivity and thickness of a silicon wedge for film thicknesses between 100 nm and 17 µm by treating these features as fitting parameters in a thermal diffusion model. Beyond insight into the role of boundary scattering on the thermal conductivity, this method offers potential for experimental investigation of the thermal conductivity accumulation function which is subject to the mean free path spectra of materials.10–12

From an application perspective, there is also value in developing techniques which can simultaneously determine the thickness and thermal conductivity of a feature or device. Substrate thinning enables tighter integration between memory, processor, and analog technologies for heterogeneously integrated systems such as system-in-package and system-on-chip devices.13,14 However, tighter device integration requires efficient thermal management to keep junction temperatures within acceptable operating limits.15,16 Substrate thinning can lead to reduced thermal conductivity, and advanced packaging increases the number of thermal boundaries. Development of fast, accurate, and non-destructive characterization techniques for both thickness and thermal transport therefore offers benefit to the design of microelectronic assemblies with improved electrical and thermal performance.

While the most ubiquitous and accurate methods used to verify device dimensions remain microscopy techniques, such as cross-sectional scanning electron microscopy (XSEM), the creation of a cross section at a desired location requires localized removal of the region through focused ion beam (FIB) milling, which is both time-intensive and destructive. Prevalent optical techniques, such as white light interferometry, provide accurate non-contact surface measurements, but cannot measure subsurface features.17–19 Polarization and reflection-based techniques such as ellipsometry20 or reflectometry21 are able to resolve the thickness of subsurface layers, but measurements can be constrained by limitations in the material thickness or transparency. Furthermore, secondary measurements of thermal conductivity would still be required.

Time-domain thermoreflectance has been demonstrated as a well-suited technique for non-contact measurements of both thermal conductivity and thickness as it utilizes a pulsed laser heat source which can propagate strain waves (commonly referred to as picosecond acoustics, or picosecond ultrasonics) that can subsequently be used to resolve layer thicknesses.22–26 However, determination of film thickness through picosecond ultrasonic analysis requires accurate knowledge of the sound velocity of the film, and the analysis can be complicated in multi-layered structures where adjacent layers have similar acoustic properties.22 In this study, we demonstrate simultaneous thickness and thermal conductivity measurements of a thinned Si substrate, measured via FDTR, which are validated through comparison to XSEM thickness measurements and comparison to several models for the thermal conductivity.

A cross-section schematic of the silicon wedge sample is
shown in Fig. 1(a). A rectangular area (5 mm wide, 25 mm long) was cleaved from a (100) single-side polished n-type Si wafer (phosphorous-doped to $6.23 \times 10^{-15}$ cm$^{-3}$). The sample was coated with a metal film stack of 20/200 nm of Ti/Al by sputter deposition onto the polished Si surface (to aid in reflectometry measurements during polishing), and bonded metal-stack-down onto a 600 μm thick Si handle piece using EPO TEK 377 epoxy. Next, the sample was thinned with a precision lapping instrument. A reflectometer was used to periodically measure the Si thickness and adjust the lapping instrument to produce a wedge shape. The sample was lapped with progressively finer diamond lapping films until all the Si was removed at one edge of the sample. A final polishing step with a colloidal SiO$_2$ slurry produced a mirror surface finish.

The thickness of the epoxy layer was determined from micrometer measurements at the base of the sample (with no top silicon layer) by subtracting the other constituent film thicknesses from the overall sample thickness, and determined as $98 \pm 8$ μm. In order to prepare the sample for measurement with FDTR, a coating of 5/120 nm Ti/Au was deposited via sputter deposition onto the polished Si surface (to aid in the signal was analyzed with a multi-layer heat diffusion equation, which takes into account the thermal conductivity ($\kappa$), volumetric heat capacity ($\rho c_p$), and thickness of each layer ($d$), as well as the thermal boundary conductance between layers ($G$). The determination of each parameter is detailed in the supplementary material. For reference, the parameters for each layer are consolidated in Table I. While the sample is physically comprised of six layers, it is treated as a four-layer system in the thermal model as the Ti/Al layers are considered as a single layer, and the silicon substrate is omitted. We find there is negligible measurement dependence upon the Epoxy/Si interface or underlying Si substrate properties due to the large thickness of the epoxy layer, in part attributed to using a lower bound of 10 kHz in our measurement of the data. This restricts the thermal penetration depth to shallower depths than are achievable at lower frequencies.

The thermal conductivity and thickness of the silicon, as well as the Au/Ti and silicon interface are treated as fit parameters in the thermal model using material parameters outlined in the supplementary material. For reference, the parameters for each layer are consolidated in Table I. While the sample is physically comprised of six layers, it is treated as a four-layer system in the thermal model as the Ti/Al layers are considered as a single layer, and the silicon substrate is omitted. We find there is negligible measurement dependence upon the Epoxy/Si interface or underlying Si substrate properties due to the large thickness of the epoxy layer, in part attributed to using a lower bound of 10 kHz in our measurement of the data. This restricts the thermal penetration depth to shallower depths than are achievable at lower frequencies.

![Diagram of the sample geometry](image)

**FIG. 1.** Diagram of the sample geometry, with a cross-sectional representation in (a) and a plan-view of the top (b) and showing the direction of FDTR measurements (blue arrow) and FIB cutouts. (c) displays a FIB cutout as viewed from the FDTR microscope. (d) displays a characteristic XSEM image of a FIB cutout.

**TABLE I.** Nominal values of the thermophysical properties for each layer used in the FDTR analysis. The thermal boundary conductance for a given layer corresponds to the interface of that layer and the layer beneath it. Additional details on the model parameters are provided in the supplementary material.

<table>
<thead>
<tr>
<th>Layer</th>
<th>$\kappa$ (W m$^{-1}$ K$^{-1}$)</th>
<th>$\rho c_p$ (MJ m$^{-3}$ K$^{-1}$)</th>
<th>$d$ (nm)</th>
<th>$G$ (MW m$^{-2}$ K$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Au/Ti</td>
<td>147</td>
<td>2.48$^{12}$</td>
<td>123</td>
<td>120</td>
</tr>
<tr>
<td>2. Si</td>
<td></td>
<td></td>
<td>Fig. 3</td>
<td>1.65$^{13,14}$</td>
</tr>
<tr>
<td>3. Ti/Al</td>
<td>100$^{34,35}$</td>
<td>2.43$^{32,33}$</td>
<td>220</td>
<td>50</td>
</tr>
<tr>
<td>4. Epoxy</td>
<td>0.3$^{34}$</td>
<td>$2.3^{32,33}$</td>
<td>semi-inf.</td>
<td></td>
</tr>
</tbody>
</table>

The thermal conductivity and thickness of the silicon, as well as the Au/Ti and silicon interface are treated as fit parameters in the thermal model using material parameters outlined in Table I. For small thicknesses, less than approximately one micron, the interface of the Au/Ti and silicon wedge, $G_1$, serves as the dominant source of thermal resistance, and we are unable to simultaneously fit for the thermal boundary conductance, thermal conductivity, and thickness. However, for wedge thicknesses in which the intrinsic thermal resistance approaches or exceeds that of the interface, the interface conductance may also be treated as a fitted parameter (see supplementary material). For thicknesses greater than 1.5 μm, an average value of $120 \pm 8.8$ MW m$^{-2}$ K$^{-1}$ is measured for the thermal boundary conductance, with no observable trend as a function of thickness. The average value of $G_1$, is therefore applied in subsequent fits, and we fit for the thickness and cross-plane thermal conductivity, considering isotropic transport. Representative data from an example measurement and
the corresponding model fit is displayed in Figure 2(a).

In order to assess the ability to simultaneously measure the thickness and thermal conductivity, sensitivity to the two parameters was determined from a sensitivity analysis calculation\(^2\), displayed in Figure 2(b)–d. Figures 2(b) and (c) display contours of the sensitivity as a function of modulation frequency for an array of silicon thicknesses. Because the thermal conductivity is variable as a function of thickness, for the sensitivity analysis, we assume the thermal conductivity to vary according to a model based on Callaway\(^3\). A qualitative estimate for the overall sensitivity to each parameter (as a function of thickness) is then determined by integrating the absolute value of the sensitivity over the measured frequency range (Figure 2(d)). Additional details on measurement sensitivity are provided in the supplementary materials. For the two parameters of interest, a higher overall sensitivity to the thickness is predicted for low film thicknesses \((d < 10 \, \text{nm})\), which transitions to a higher sensitivity to the thermal conductivity as the film properties becomes bulk-like. These differences in profile and magnitude suggest independence in measurement sensitivity, and lend credence to the ability to resolve the two parameters over the measured thickness range.

The results of the FDTR-determined thickness are displayed as a function of distance from the reference, illustrated in Fig. 3(a). The measurement uncertainty is calculated from the root sum square of the residual of the fit, uncertainty in the controlled parameters, and the standard deviation in the fitted values between measurements\(^1,4\). In general, the thickness as a function of distance from the reference exhibits a high degree of linearity. For validation of the measurement,
The thickness was compared against direct measurements obtained from XSEM. Cross-sections were prepared via FIB milling at intervals spaced approximately 1 mm in distance starting from the reference location (Fig. 1(b-d)). Uncertainty of the XSEM thickness measurements was considered as 3% of the measured thickness. The result of a linear fit for the FDTR- and XSEM-determined thickness is displayed in Fig. 3(a). The FDTR measurements provide a slope of 1.53 mm/µm and offset of -56 nm, whereas the FIB measurements yield a slope of 1.54 nm/µm and offset of 250 nm. Through calculation of the variance in the linear model, the uncertainty, to within one standard deviation, for the slope and offset are ±0.03 mm/µm and 22 nm for the FDTR fit, and 0.05 mm/µm and 13 nm for the XSEM fit. Overlap in the uncertainty of the data with the fitted slope for FDTR and XSEM demonstrate good agreement between the two thickness determination methods.

In addition to the thickness, we also investigate the thermal conductivity of the silicon along the thickness gradient of the wedge. A number of additional measurements were made along the y-direction of the sample surface and the thermal conductivity and thickness were simultaneously fit in the manner previously outlined. The thermal conductivity is plotted as a function of FDTR-determined thickness in Fig. 3(b). For reference, we compare the measured results against several models for the expected thermal conductivity as a function of thickness. These models are based upon analytical solutions to the Boltzmann Transport Equation under the relaxation time approximation, and follow the approaches of Callaway43, Holland44, Born-von Karman-Slack45,46, and a model considering an experimentally-derived phonon dispersion for silicon, determined by Weber47. Details regarding the calculation of each model are provided in the supplementary material.

Compared to measured thickness, there is higher uncertainty in the measured thermal conductivity, which is attributed to reduced sensitivity as the film becomes thinner (see Fig. 2(d)). This gives rise to the large uncertainties for films on the order of 100 nm, and for smaller thicknesses, direct measurements can not be made. To reduce scatter in the data, measurements are averaged at equal spacings along the gradient. Specifically, we average the thermal conductivity data by grouping measurements into equal, logarithmically spaced bins and take the average value from within each bin, shown in Fig. 3(b). The non-averaged results are provided in the supplementary material. Variation in the thermal boundary conductance of the transistor/silicon interface can contribute to uncertainty in the thin film regime as the interface becomes the dominant source of thermal resistance relative to the intrinsic resistance of the silicon layer (plotted in the supplementary material). Thus, the thermal conductivity of smaller thicknesses may be resolvable for more thermally resistive films in which the resistance intrinsic to the film remains higher than that of the interface at smaller thicknesses. For thicknesses greater than 100 nm, the uncertainty of the measurement is reduced, and is generally within error of that expected from the thin film models. For each model, the total degree of scattering is determined from the combination of impurity, Umklapp, and boundary scattering, added via Matthiessen’s rule. The magnitude of the impurity scattering term is dependent upon a number of features including impurity concentration, mass differential, and also localized strain induced from impurities48. We note that the models in Fig. 3(b) utilize the impurity scattering coefficients taken directly from the corresponding publications.

In summary, we demonstrate the application of frequency domain thermoreflectance for simultaneous determination of the thermal conductivity and thickness of a silicon wafer fabricated with a linear gradient in thickness. The thickness measurements were compared against results from XSEM, demonstrating good agreement between the two techniques. Furthermore, for films above 100 nm, the size dependent thermal conductivity of the system was also found to compare favorably with prevalent analytical models. Taken together, these results demonstrate how materials prepared in this geometry enable a faster, yet equally comprehensive analysis of the size-dependent thermal properties with a single sample, as compared to a series of films with varying thickness.

**SUPPLEMENTARY MATERIAL**

See the supplementary material for additional information on the parameters used in the thermal model, sensitivity analyses, and details on the models of thickness-dependent thermal conductivity.

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**DATA AVAILABILITY**

The data that support the findings of this study are available from the corresponding author upon reasonable request.


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(a) $\kappa_2: 109 \pm 5.1 \text{ W m}^{-1} \text{ K}^{-1}$
$d_2: 821 \pm 55 \text{ nm}$

Data vs. Model

(b) Frequency (Hz)
Silicon Thickness (nm)

(c) Frequency (Hz)
Silicon Thickness (nm)

(d) Integrated Sensitivity (a.u.)
Thickness (nm)
(a) Thickness (nm) vs. Distance (μm)

(b) Thermal Conductivity (W m\(^{-1}\) K\(^{-1}\)) vs. Thickness (nm)

- Thin film model (based on Callaway)
- Thin film model (based on Born-von Karman-Slack)
- Thin film model (based on Holland)
- Thin film model (based on Weber dispersion)