Simultaneous thickness and thermal conductivity measurements of thinned silicon from 100 nm to 17 μm

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ABSTRACT
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.

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 nondestructive characterization techniques for both thickness and thermal transport, therefore, offer 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 noncontact surface measurements, but cannot measure subsurface features.\textsuperscript{15–19} Polarization and reflection-based techniques such as ellipsometry\textsuperscript{20} or reflectometry\textsuperscript{21} 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 noncontact 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.\textsuperscript{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.\textsuperscript{27} In this study, we demonstrate simultaneous thickness and thermal conductivity measurements of a thinned Si substrate, measured via FDTR, which are validated through a comparison to XSEM thickness measurements and a 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 × 10\textsuperscript{15} cm\textsuperscript{-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\textsuperscript{37} 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\textsubscript{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 ± 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 to serve as an opto-thermal transducer. Figure 1(a) shows a cross-sectional diagram of the finished sample.

Thickness and thermal conductivity of the wedged silicon were measured with FDTR. Details of the technique are discussed in prior publications.\textsuperscript{27–29} Here, the pump and probe beams were operated at wavelengths of 488 and 532 nm, respectively. The dimensions of the beams are determined through knife-edge measurements with a standard uncertainty of 0.1 μm. The average focused radii, measured between independent measurements over the course of the study, were found to be 6.7 and 5.9 ± 0.1 μm for the pump and probe, respectively. A pump power of 6 mW was utilized and modulated over a range of 10 kHz–40 MHz. The probe, also set to 6 mW, was focused concentrically with the pump onto the sample surface using a 5 × objective lens.

Measurements were performed in a linear path along the y-direction [illustrated by the blue arrow in Fig. 1(b)]. Near the base of the sample, on the thinned end, curved interference fringes could be observed through the optical microscope of the FDTR system and were used to establish a reference for determining the position of subsequent measurements. Microscope images were collected at each position where measurements were performed and aligned to create a contiguous image of the surface [example microscope image displayed in Fig. 1(c)]. Distance from the reference line was determined with digital image analysis: the number of pixels between the location of measurement and the reference line were mapped to a known distance scale (providing the number of pixels per unit of distance). After measurement of the frequency-dependent reflectivity, the signal was analyzed with a multi-layer heat diffusion equation, which takes into account the thermal conductivity (κ), 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.\textsuperscript{30}

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. While small thicknesses, less than approximately one micrometer, the interface of

\textbf{FIG. 1.} Diagram of the sample geometry, with a cross-sectional representation in (a) and a plan-view of the top (b), displaying 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>
<thead>
<tr>
<th>Layer</th>
<th>(\kappa) (W m\textsuperscript{-1} K\textsuperscript{-1})</th>
<th>(\rho c_p) (MJ m\textsuperscript{-3} K\textsuperscript{-1})</th>
<th>d (nm)</th>
<th>G (MW m\textsuperscript{-2} K\textsuperscript{-1})</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Au/Ti</td>
<td>147</td>
<td>2.48\textsuperscript{31}</td>
<td>123</td>
<td>120</td>
</tr>
<tr>
<td>2. Si</td>
<td>Fig. 3</td>
<td>1.65\textsuperscript{11–33}</td>
<td>Fig. 3</td>
<td>150\textsuperscript{34}</td>
</tr>
<tr>
<td>3. Ti/Al</td>
<td>100\textsuperscript{12,35}</td>
<td>2.43\textsuperscript{32,36,37}</td>
<td>220</td>
<td>50</td>
</tr>
<tr>
<td>4. Epoxy</td>
<td>0.3\textsuperscript{38}</td>
<td>2.39,40</td>
<td>Semi-inf.</td>
<td>...</td>
</tr>
</tbody>
</table>
the Au/Ti and silicon wedge, \( G_1 \), serves as the dominant source of thermal boundary resistance, and we are unable to simultaneously fit 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 the supplementary material). For thicknesses greater than 1.5 \( \mu \)m, an average value of 120 \( \pm \) 8.8 MW m\(^{-1}\) 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 are displayed in Fig. 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,\(^{3,28}\) displayed in Figs. 2(b)–2(d). Figures 2(b) and 2(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.\(^{41}\) 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 [Fig. 2(d)]. Additional details on measurement sensitivity are provided in the supplementary material.

For the two parameters of interest, a higher overall sensitivity to the thickness is predicted for low film thicknesses (\( d \leq 10 \mu \)m), which transitions to a higher sensitivity to the thermal conductivity as the film properties become 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.\(^{3,42}\) 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 [Figs. 1(b)–1(d)]. Uncertainty of the XSEM thickness measurements was considered as 5\% 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 \( \text{nm/\mu m} \) and offset of \(-56 \text{ nm}\), whereas the FIB measurements yield a slope of 1.54 \( \text{nm/\mu m} \) and offset of 250 nm. Through calculation of the variance in the linear model,\(^{3,1}\) the uncertainty, to within one standard deviation, for the slope and offset is \( \pm 0.03 \) nm/\( \mu \)m and 22 nm for the FDTR fit, and 0.08 nm/\( \mu \)m and 13 nm for the XSEM fit. Overlap in the uncertainty of the data with the fitted slope for FDTR and XSEM demonstrates 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 Callaway,\(^{43}\) Holland,\(^{44}\) Born–von Karman-Slack,\(^{45,46}\) and a model considering an experimentally derived phonon dispersion for silicon, determined by Weber.\(^{47}\) 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 cannot 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 nonaveraged results are provided in the supplementary material.

Variation in the thermal boundary conductance of the transducer/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 impurities. 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.

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.

REFERENCES


A. J. Schmidt, R. Cheaito, and M. Chiesa, “A frequency-domain thermoreflec-