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Optical characterization and thermal properties of CVD diamond films for integration with power electronics

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ABSTRACT

Studies of diamond material for thermal management are reported for a nominally 1- μm thick layer grown on silicon. Thickness of the diamond is measured using spectroscopic ellipsometry. Spectra are consistently modeled using a diamond layer taking into account surface roughness and requiring an interlayer of nominally silicon carbide. The presence of the interlayer is confirmed by transmission electron microscopy. Thermal conductivity is determined based on a heater which is microfabricated followed by back etching to produce a supported diamond membrane. Micro-Raman mapping of the diamond phonon is used to estimate temperature rise under known drive conditions of the resistive heater. Consistent values are obtained for thermal conductivity based on straightforward analytical calculation using phonon shift to estimate temperature and finite element simulations which take both temperature rise and thermal stress into account.

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1. Introduction

Device self-heating is a principal limiting factor in high-power electronics. Better thermal management, *e.g.*, through diamond heat spreading layers, is necessary for increasing power densities and efficiencies while reducing device size and mitigating device failure [1–8]. Diamond grown by chemical vapor deposition (CVD) produces thin films of good quality, but the first ~ 100 nm, called the coalescence layer, is generally characterized by smaller grain size and concomitantly higher grain boundary density. The latter often implies a higher non-diamond carbon content [9]. This layer is known to hinder heat flow from the device active region into the diamond [10], a process which is impeded even for ideal hetero-interfaces [11]. The polycrystalline grain structure and columnar texture of CVD diamond films also result in substantially lower in-plane thermal conductivity (κ) when compared to bulk crystals [10,12,13]. To understand and improve thermal management of devices incorporating polycrystalline diamond films as heat spreaders, it is necessary to reliably characterize their thermal properties.

Thermal conductivity for thin diamond films grown on silicon substrates have been measured using micro-fabricated test structures. Resistive heaters are formed on nanocrystalline diamond films grown on a substrate followed by back-etching of the silicon in an area directly below the heater to produce a diamond membrane [10]. The design is depicted in Fig. 1(a). Heat initially flows from the resistor and remains restricted to the diamond before eventually flowing into the substrate. Near the center of the membrane heat will primarily flow laterally from the heater at $x = 0$ toward the edges, *i.e.*, in the $\pm x$ directions of Fig. 1(a). With known steady-state heater drive power, assumed to diffuse into and through the diamond, Fourier's law

$$\dot{q} = -\kappa \nabla T \quad (1)$$

may be used to determine κ . Quantity $\dot{q}$ (W/m^2) is the lateral heat flux through the diamond layer, with cross-sectional area $L \times t$ in Fig. 1(a).

Thickness of the diamond layer t is important in applying Eq. (1), since it factors into the above computation, whether analytic or through simulation, and generally differs from the nominal value based on growth conditions. Therefore, thickness is determined *ex situ* and preferably using a non-destructive method. Furthermore, recent work illustrates that the lateral κ value depends

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Fig. 1. A schematic (a) and optical image (b) of the diamond membrane, and (c) depicts the model used to fit the spectroscopic ellipsometry data.

on thickness due, again, to the polycrystalline nature of the diamond layer [10,12].

Measurement of the temperature gradient, $\nabla T \rightarrow dT/dx$, is likewise a critical factor in using Eq. (1) to estimate κ . This may be established from positional micro-Raman mapping based on three prospective quantities. First is the dependence of the phonon energy on temperature, $\omega(T)$. For ideal bulk materials $\omega(T)$ is generally attributed to anharmonic (phonon decay) effects, which are intrinsic, and contributions from thermal expansion through

$$\frac{d\omega}{dT} = \left(\frac{\partial\omega}{\partial T} \right)_V + \beta_p V \left(\frac{\partial\omega}{\partial V} \right)_T \quad (2)$$

where $\beta_p = \frac{1}{V} \left(\frac{\partial V}{\partial T} \right)_p$ is the volume thermal expansion coefficient. With the help of scaling relation $\omega/\omega_0 = (V_0/V)^\gamma$, where γ is the Grüneisen parameter, Eq. (2) may be rewritten

$$\frac{d\omega}{dT} = \left(\frac{\partial\omega}{\partial T} \right)_V - \gamma\omega\beta_p \quad (3)$$

where ω_0 represents the phonon energy at fixed reference conditions. For small ΔV in isotropic materials β_p may be approximated by 3α , with α the linear expansion coefficient. When materials are grown on a thick foreign substrate (S), the strain imposed in the layer requires the temperature dependence in Eq. (3) incorporate in-plane expansion by replacing β_p with $2\alpha_s + \alpha_c$ with subscript C denoting the grown layer and alluding to the direction perpendicular to the substrate plane. When integrating either Eq. (2) or (3) it is generally important to take into account the temperature dependence of the expansion coefficients. Practical direct application of Eq. (3) is limited to bulk materials and with $\beta_p \rightarrow 2\alpha_s + \alpha_c$ to thin films. More complicated geometrical configurations, such as the

membrane studied here, are conceptually related to these two important cases but application requires taking the position-dependent strain (or stress) state into account when interpreting the phonon shift. This is described further in Sections 3 and 5.

Because the linear thermal expansion of diamond and silicon differ, residual stress on a uniform CVD diamond film will occur following growth and cooling to room temperature. This thermal stress is compressive when diamond is grown at temperatures under 1220 °C, as is the case here [14]. Because the membranes are small in lateral dimension, the overall stress is expected to change little upon membrane formation. This is confirmed by our micro-Raman measurements. During calibration, the increasing temperature will produce thermal stress in the tensile direction due to expansion of the thick silicon substrate. The measured dependence agrees with this expectation. In contrast, when driving the micro-fabricated heater, the silicon temperature rise is negligible so that primarily the membrane will expand. Therefore, under drive conditions the membrane thermal stress will be in the compressive direction.

Raman line width Γ also depends on temperature making it a second potential choice for mapping temperature rise. Temperature dependence of the line width for natural diamond is well-described through the same anharmonic effects which influence phonon shift via a symmetric phonon decay $\Gamma(T) \sim [1 + n_B(\omega_0/2, T)]$ [15], where $n_B(\omega, T)$ is the Bose function. Finally, the relative intensity of the Stokes and anti-Stokes features in Raman spectra depend on temperature through phonon population according to $I_S/I_{AS} \sim [n_B(\omega, T) + 1]/n_B(\omega, T)$. This presents a third approach for mapping temperature. For this expression to be valid the measurements must be carried out in a range where the optical attenuation factor does not vary strongly with wavelength. The Stokes–anti-Stokes approach requires two spectroscopic ranges be measured—above and below the excitation wavelength—an approach which may be arduous and subject to the instrumental transfer function making it generally impractical.

In this paper, we describe spectroscopic ellipsometry measurements for determining thickness of CVD diamond. The method is further used to examine uniformity across a diamond layer grown on silicon. Micro-Raman measurements are reported for thermal test structures on diamond membranes determining thermal conductivity through the phonon shift. Concurrent line width measurements are also reported to exhibit a minor change for the temperatures induced during these membrane measurements.

2. Experimental details

The diamond layers studied here were grown on silicon substrates using microwave-enhanced CVD [10]. SEM analysis of the diamond surface (not shown), provides assessment of grain size which ranges from 140 to 250 nm across, with average ~ 197 nm for this sample. Heaters were fabricated from 10 nm titanium followed by 200 nm gold deposited on the diamond surface and patterned to have a 5 μm width. Back-side etching then fully removes silicon below the heater. A photograph of a representative membrane, 200 μm wide $\times$ 1000 μm long, appears as the dark region at the center of Fig. 1(b). In this structure, one heater is located near the center of the membrane and a second is near the edge for use as either a heater or resistance temperature device (RTD).

Spectroscopic ellipsometry measurements were performed across the 245–1688 nm wavelength range using a commercial instrument (J.A. Woollam, VASE ESM-300). Measurements and modeling were carried out at angles of incidence 55, 65, and 75°. We report here results from the 75° angle of incidence which is expected to reduce the effects the relatively rough diamond surface will have on modeling the data. Measurements at 75° angle

of incidence were found to reduce the mean square error in fits to our model by over 50% compared to analysis of data obtained at 55 and 65°.

The Micro-Raman measurements were carried out using near-ultraviolet (NUV) 363.8-nm excitation from an argon-ion laser. Excitation was focused on the sample using a 10× objective. Scattered light was passed through a notch filter, dispersed by a grating spectrometer with 0.78-m focal length, and detected using a liquid-nitrogen cooled CCD array. NUV excitation is found to reduce the fluorescence background from the diamond allowing simultaneous observation of the broad non-diamond carbon (NDC) Raman features from 1500 to 1600 cm^{-1} [9]. This Raman excitation wavelength will also probe silicon to very shallow penetration depth of ~ 10 nm, corresponding to the region where substrate temperature rise is expected to be highest.

Computing temperature rise from phonon shifts relies on calibration of the specific geometrical sample configuration of interest. We calibrate for each membrane by measuring the temperature-induced phonon shift using a commercial stage at a series of set-points. Across the relevant temperature range this shift is linear. The line slope, $d\omega/dT$, depends on whether calibration is conducted either directly on the membrane or above the silicon substrate. This difference is attributed to substrate-induced stress arising from mismatches in the thermal expansion coefficients of diamond and silicon. Consequently, we must account for thermal stress in the net phonon shift according to

$$d\omega/dT = k_T + k_S(d\sigma/dT) \quad (4)$$

Temperature (k_T) and stress (k_S) coefficients for diamond can be obtained by measuring the diamond phonon energy, i.e. $d\omega/dT$, over the membrane and above the silicon substrate. Eq. (4) recognizes that the stresses induced during the calibration procedure from uniform temperature rise are different from what is produced by the line heater. Thermal stresses for the calibration condition of uniform heating are simulated as a function of temperature using finite element analysis. From these we obtain $d\sigma/dT$ over the membrane and over silicon, each corresponding to the same position where the calibration measurements were conducted. From the two $d\omega/dT$ measurements we can now obtain unknowns k_T and k_S . Here we obtain $k_T = -0.015 \pm 0.003 \text{ cm}^{-1}/\text{K}$ and $k_S = -0.5 \text{ cm}^{-1}/\text{GPa}$. These values are close to prior reports with variations plausibly attributed to the diamond layer morphology [16]. Finite element simulations described below account for temperature rise and thermal stress when the heater is driven and use the k_T and k_S coefficients to compare expected with measured phonon shift.

3. Spectroscopic ellipsometry measurements of diamond thickness

Shown in Fig. 2 are representative measurements of ellipsometric angles Ψ and Δ across the full wavelength range. Spectra were successfully modeled using a structure comprised of the silicon substrate and diamond layer incorporating root-mean square (RMS) surface roughness, as depicted in Fig. 1(c). It was found necessary to incorporate an intermediate layer in the model which we identify as intermixed silicon and carbon. This layer, denoted Si/C, was optically modeled using Cody-Lorentz oscillators with optical gap of ~ 2.7 eV. Results of the modeling are shown in Fig. 2 relative to the raw data exhibiting good agreement.

The diverse polytypes of SiC (e.g., 2H, 3C, 4H, and 6H) [17] have wide band gap making them of interest for various applications [18]. Dielectric functions of SiC in different phases have been derived using first principle calculations [19] and measured using spectroscopic ellipsometry [20,21]. Results have shown that the imaginary part of the dielectric function in polytypes of SiC is neg-

Fig. 2. Directly measured ellipsometric angles Ψ and Δ across the full wavelength range (solid lines) along with modeling (dashed lines).

ligible for photon energy less than 3 eV [19–21] and reporting energy gap in the range of 2.4–3.3 eV [17]. The ~ 2.7 -eV gap of the intermediate layer in our model is consistent with ranges reported for the energy gap of SiC.

Since the presence of this interface layer is attributed to inter-diffusion of silicon and carbon during diamond growth, it is reasonable to expect variable stoichiometry and/or mixed phase rather than an ideal material structure. Nevertheless, we carried out modeling of the data using the optical properties of known SiC phases. These models resulted in higher mean square error values confirming that the ideal phases do not adequately describe the optical properties of our interface layer.

Fig. 3 shows the results of measurements at different die locations from the wafer. Each measurement was carried out post-heater fabrication, back etching, and dicing. Good consistency is obtained for all model parameters examined: diamond thickness, RMS roughness, and Si/C thickness. Measurements from several positions on given die likewise produced good agreement. The diamond thickness values obtained a range from 959.3 to 983.1 nm with mean value 972.1 nm, in good agreement with the nominal 1- μm from the CVD growth. Measurements taken on the same die (not shown) were likewise consistent. Spot checks of thickness and roughness from electron microscopy cross-sections are in good agreement, with the RMS roughness estimated at ~ 18 nm. Our results show desired cross-wafer uniformity is obtained from the growth.

To check the presence of a Si/C interface layer, scanning transmission electron microscopy (STEM) measurements were carried out on cross-sections of the sample prepared by focused ion beam (FIB) milling. The sample was milled to electron transparency at

Fig. 3. Die locations of a diamond/Si wafer where SE measurements are done. Non-uniformity in the diamond thickness is less than 1%.

30 kV followed by successive polishing steps at 5 and 2 kV. Analysis was performed using a JEOL ARM 200F operating at 200 kV. Fig. 4 shows a bright field STEM image oriented along the Si [110] zone axis. The crystal lattice of the Si substrate is seen in the bottom of the image. Regions can be seen in the top portion which correspond to the polycrystalline diamond. Between the diamond and silicon there is a disordered layer which is confirmed to contain both C and Si using electron energy loss spectroscopy. The layer observed by STEM is 1–2.5 nm thick, which is considerably thinner than the Si/C we obtain from ellipsometry measurements. We preliminarily attribute the differences in Si/C layer thickness to the possibility of extended interdiffusion which is

Fig. 4. Bright field scanning transmission electron microscopy image of the sample at the diamond/silicon interface.

not evident in the STEM but may have an effect on the optical properties. Details of TEM measurements focusing on the near interface region will be discussed in a forthcoming publication.

4. Micro-Raman measurements of diamond membranes

Micro-Raman spectra of the diamond membrane near the heater structure are shown in Fig. 5. Clearly seen is a shift in the diamond $O(\Gamma)$ -symmetry phonon when comparing heater off and with drive power ~ 380 mW. This shift is primarily attributed to increased temperature, although changes in the membrane thermal stress also contribute to the overall measured peak position. Shown in Fig. 6(a) is the position-dependent shift in phonon energy

$$\Delta\omega(x) = \omega(x) - \omega_0(x) \quad (5)$$

at this same drive power. In addition to the diamond data points (squares) we include the data for the silicon $O(\Gamma)$ -symmetry phonon which is observed where the membrane ends and the diamond continues over the substrate. This is useful as a reference point because temperature rise can be observed in the silicon due to excessive heater temperature, poor heat sinking, or excessive laser power. We also note that the positional dependence of $\omega_0(x)$ in Eq. (5) is important because local stresses present in the polycrystalline diamond may shift diamond peak position [16]. Across the membrane we observe ~ 1 cm^{-1} variation in the diamond peak position. This variation is significant when compared with the maximum heater-induced shift of slightly more than -2 cm^{-1} . Thus, using an average value for ω_0 measured from the membrane will not compensate for the large variation when computing temperature rise from position to position based on Eq. (5) and the calibrated $d\omega/dT$.

The data presented in Fig. 6(a) may now be used to estimate thermal conductivity. Using Eq. (1) and the slope dT/dx directly from fitting the data and using $-0.015 \pm .003$ cm^{-1}/K for diamond phonon temperature coefficient, we obtain a quick estimate of $\kappa = 136 \pm 9$ W/m K. Further analysis of the results is carried out using a custom two-dimensional finite element (FE) simulation (COMSOL 5.2) including the silicon substrate, diamond membrane layer, and heating structure. The known drive power is supplied and the boundary conditions maintain the silicon support struc-

Fig. 5. Micro-Raman spectra of the diamond membrane near the heater structure with heater OFF and ON.

Fig. 6. (a) Diamond phonon shift across the membrane (left axis) which is used in simulation (solid red line) with drive power ~380 mW. The right axis shows the temperature rise in diamond (black squares) and silicon (blue circles). Bright and dark rectangles show positions of the membrane and surrounding silicon support. (b) Summarizes the shift in the diamond line width from “Heater ON” relative to “Heater OFF”. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

ture at ambient temperature far from the heater and membrane. Unknown parameters in the analysis are κ and the thermal boundary resistance (TBR) between the diamond and silicon. Other quantities in the model, such as elastic constants and thermal expansion coefficients, are literature values. The simulation computes the temperature rise (ΔT) and stress (σ) distribution in the structure. From these quantities and the calibration results, $\Delta\omega$ is calculated and compared pointwise to the experimental values. Quantities κ and TBR are varied via a Monte-Carlo optimization approach to find the minimum in the reduced chi-square, denoted χ^2 . We ignore in our simulations any temperature dependence of κ . This topic has been previously investigated by Anaya et al., where only a weak temperature dependence of Kappa is observed for diamond at temperatures above room temperature [10]. Simulations of our data comparing a temperature dependent with constant value of κ across the membrane show that, in the temperature range studied here, there is no definitive improvement when using the former.

The optimization minimum results in $\kappa = 141$ W/m K and $TBR = 7.6 \times 10^{-7}$ m² K/W. The corresponding fit to $\Delta\omega$ vs. x is also included in Fig. 6(a). The resulting value of κ agrees with our rough estimate from the slope in the data. This shows that both the straightforward analysis using Eq. (1) and the simulations are useful in determining the membrane thermal conductivity. To examine the uncertainties in κ and TBR from the simulation, we calculated the 90% confidence intervals using the full χ^2 shape [22]. Because the measurement approach is not sensitive to the TBR, due to the negligible temperature gradient across the diamond/silicon interface, the dependence in the simulated χ^2 is

very shallow resulting in a large 90% confidence interval of $\kappa = 141^{+46}_{-21}$ W/m K. However, if we consider the χ^2 dependence along the κ axis where the global minimum resides, we obtain 141^{+10}_{-21} W/m K for the 90% confidence range. In studying several membranes under various drive conditions and membrane thickness, TBR varies from as high as 1×10^{-5} m² K/W to as low as 1×10^{-8} m² K/W. In the current study and for the 90% confidence interval, TBR value ranges from 6×10^{-6} m² K/W to 6×10^{-8} m² K/W. Our analysis also shows that a very small TBR value (e.g., 1×10^{-8} m² K/W) results in an unacceptable fit to our data. We conclude from this that the membrane approach is not sufficiently sensitive to TBR to reliably extract a value from these measurements.

Since Raman line width depends on temperature we examine whether this quantity can be similarly used in our measurements to obtain dT/dx and subsequently estimate thermal conductivity according to Eq. (1). We show in Fig. 6(b) the measured change in peak width, as a function of position, when comparing the heater on versus off conditions according to $\Delta\Gamma(x) = \Gamma(x) - \Gamma_0(x)$. The results show no trend in line width following the temperature rise seen in peak position, Fig. 6(a). Given the gradual dependence of line width on temperature, only ~ 0.5 cm⁻¹ increase is expected between the edge and center of the test structure under these drive conditions [23]. This variation is within the uncertainty ranges shown. We conclude that peak position is useful in measuring temperature-induced trends, for this range of temperature rise, but the line width is not reliable.

5. Conclusions

We have studied CVD diamond on silicon for measuring thermal conductivity. Thickness measurements are carried out using spectroscopic ellipsometry. Good agreement is obtained between these results and thickness reported based on diamond growth condition. RMS roughness of the diamond is in good agreement with electron microscopy. Additionally, a silicon carbide interlayer is necessary to model the ellipsometry data. The presence of the interlayer is confirmed by TEM. Near-UV micro-Raman mapping is used to measure phonon redshift across a diamond membrane when drive current through a micro-fabricated resistive heater induces a temperature rise. The optimization minimum in our finite element simulation results in $\kappa = 141^{+10}_{-21}$ W/m K.

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