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Thermal Characterization of Polycrystalline CVD Diamond Thin Films

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Abstract

An experimental thermal characterization method is developed for high thermal conductivity thin films. The method utilizes Ta/Pt resistors on microfabricated free-standing thin film structures both for heating and temperature monitoring at different positions on the structures. The steady-state temperature at the heater and the sensor positions are monitored as a function of the power dissipated by the heater under vacuum environment, and the thermal conductivity is estimated by comparing these results to FEA and/or analytical models. The developed method is used to characterize the thermal conductivity of various different CVD diamond films of different grain sizes and films thicknesses. The measured thermal conductivity values range from 15 W/m·K to 300 W/m·K, which are at least one order of magnitude lower than that of natural diamond. It is also shown that the thermal conductivity of such films in the in-plane direction increases with increasing grain size and film.

Keywords: CVD Diamond, Thermal Conductivity

1. Introduction

Natural diamond is an exceptional material with outstanding thermal and mechanical properties. In addition to its supreme mechanical strength ($E \approx 1000$ GPa), natural single crystalline diamond has the highest thermal conductivity of all solids (2000-2400 W/m·K). The ability to synthesize diamond on flat surfaces via the chemical vapor deposition (CVD) technique facilitated the use of diamond as a key material for applications including, but not limited to, microelectronics, high power optical elements, and recently, micro-electro-mechanical systems. Even though most of the physical properties of CVD diamond are the same of its natural counterpart [1], it has been shown that its thermal properties may significantly differ, depending mostly on to the grain boundary density and films thickness [2]. There have been several attempts at measuring the actual thermal properties of CVD diamond films using different methods, such as, the 3ω [3], laser induced transient grating [4], or flash [5]; however, such efforts were mainly limited to relatively thick films ($t_d \ge 100 \mu m$). Angadi, et al. have specifically investigated the thermal conductivity of thinner (1-8 μm thick) ultra-nano-crytalline (UNCD) films, but in the through-the-film direction [6], which might be significantly different from the conductivity along the film, depending on the grain orientation. In this work, we present a thermal characterization method based on the comparison of empirical and FEA data of temperature drop along locally heated various cantilever and bridge type free-standing structures

microfabricated on CVD diamond films as thin as 1 µm. The free-standing structures bear multiple Ta/Pt resistors for local heating and temperature monitoring at different locations.

2. Methodology

The thermal conductivity of the CVD diamond films was measured with a standard analytical method, and with the FEA method proposed in this article.

2.1. Analytical Method

A straightforward method to measure the through-the-film thermal conductivity of thin films is to use simple doubly clamped or cantilever type beams, heated on one end, and the temperature measure at the other end. Given that is the heat flow between the heater and the sensor is sufficiently unidirectional. By measuring the temperature difference between the two elements on the cantilever, the thermal conductivity k can be deduced by the following expression:

$$k = \frac{P \times L}{S \times \Delta T} \tag{1}$$

where, P is the power dissipated at the heater, L is the distance between the measurement points, S is the crosssectional area, and ΔT is the measured temperature difference between two points. The accuracy of this method is limited by the sensitivity of the resistive sensors (width of line versus gradient along it), and large temperature gradient between the heater and sensor is desired for accurate measurements.



Figure 1: FEA thermal simulation result for the micro-bridge designed for high thermal conductivity thin film characterization (left), and the temperature distribution along the device for three different thermal conductivity values (right). The temperature distribution within the heater plate is very uniform; the distribution within the sensor plate is, while more evident than the heater plate, lower than the gradient along the beams, slowing more precise temperature measurement.

2.2. FEA Method

Conventional micro-electro-mechanical structures, such as the cantilever in Figure 2, with resistive heaters/sensors for thermal characterization of thin films has been proposed before [7]; however, such structures cease to be useful for extremely conductive films: Large resistors are required to be able to deliver enough power to increase the temperature on the sensor side, such that the sensing SNR is sufficient. However, large resistors consume large areas (and beam width), and in turn, leads to a wider thermal path, which makes it difficult to increase the sensor temperature. To facilitate the thermal characterization of extremely conductive films, a novel structure, as shown in Figure 1, was designed. This novel structure has two large membranes bearing the Ta/Pt

heater and the sensor. These membranes are suspended via thin beams to reduce the thermal path; hence, the sensor temperature can be elevated enough with moderate current densities. The temperature gradient on the heater and sensor membranes are much lower compared to the beams; therefore the temperature measurements are more accurate. Simulations showed that this structure provides significant enhancement of measurement accuracy compared to the standard cantilever method for films with thermal conductivity higher than 800 W/m·K. The thermal conductivity can be estimated by measuring the temperature at the heater and the sensor membranes and fitting the thermal conductivity value used in the FEA analysis to meet the measurement results conductivity measurement results are presented for both the novel bridge and conventional cantilever structures.

3. Fabrication of Test Structures

CVD diamond is deposited on a thermally oxidized $(1\mu m)$ 500 μm thick Si wafer (Figure 5 a). In this work, two CVD diamond layers were initially used. First one is polycrystalline (1.35 μm thick) and second one is amorphous or nanocrystalline (1 μm thick). These layers had grain sizes of 180 nm and 5 nm, respectively. Measurements for other CVD diamond layers with different thicknesses are in progress. A lift off with an image reversal resist is done to pattern the Ta/Pt (10/210nm) layer. 1 μm of PECVD SiO₂ is then coated to act as a mask for the diamond etching. PECVD SiO₂ is pattern by RIE etching with a photo resist mask. The PR is then stripped. The structure is released by a 2 step processes. First step is DRIE Si backside etch to release the structure, followed by a HF vapor etching to release the chip. SEM photos of a cantilever and bridge type test samples are shown in Figure 3.



Figure 2: SEM pictures of fabricated test samples.

4. Experimental Results

The first step of the measurement procedure is to calibrate the Ta/Pt resistors by measuring the Temperature Coefficient of Resistance (TCR). These measurements were performed under a temperature controlled oven, and the TCR was found to be 0.002564 K^{-1} . A dedicated vacuum setup, which was able to attend a minimum chamber pressure of 10 Pa, was built for the actual thermal conductivity measurements to eliminate convection losses.

Averaging several measurements, the thermal conductivities of the polycrystalline and nanocrystalline diamond layer was found to be 303 W/m·K, and 13 W/m·K, respectively. Main reason for this large difference is due to grain size and is in agreement with results on thicker layers [2]. Due to the low conductivity of the roughly 1µm thick layers measured here, the 53% improvement in measurement accuracy, which improves linearly with increasing temperature increase at the sensor, for both the cantilever and bridge type were not drastic, but still significant, as shown in Figure 5b. Same value was obtained with FEA method for the cantilever structure at same power.

5. Conclusions

A dedicated micro-bridge structure that facilitates the thermal characterization of highly conductive thin films has been designed and fabricated. The measurement principle is based on Ta/Pt resistors located on different locations on the free standing micro-bridge structure and used for heating and/or temperature monitoring. The thermal conductivity along the films then can be estimated by fitting the experimental data with FEA analysis results. Due to the low thermal conductivity of $\sim 1 \,\mu m$ thick CVD diamond films compared to natural diamond, the enhancement of temperature measurement accuracy attained by the proposed structure was limited to %53.



Figure 3: Resistivity increase of the Ta/Pt resistors with (a) temperature and (b) input power. The discrepancy from the linear behavior in (a) is attributed to the poor temperature stability of the oven at low temperatures.



Figure 4: (a) Temperature increase at the heater and sensor locations for a cantilever type as a function of input power. (b) Temperature increase at the heater location of a cantilever and a bridge type test sample with identical width and length. The bridge type sample yields 53% higher temperature increase for a given input power

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