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Thermal Diffusivity of Diamond Wafers Deposited with Multicathode dc Plasma-Assisted CVD[1]

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A thermal diffusivity map for diamond wafers of 10-cm diameter was obtained using a converging thermal wave technique in a nondestructive and noncontact manner. Diamond wafers were deposited by seven-cathode dc plasma-assisted chemical vapor deposition with different CH_4 concentrations in pure hydrogen and applied powers of the plasma. Six cathodes were located at the apexes of a hexagon with an arm distance of 4.3 cm about a central cathode. The wafer deposited at a low-power plasma (13.47 kW) and a low concentration of CH_4 (6%, by volume) shows three circular zones on the thermal diffusivity map. The thermal diffusivity shows the lowest value at the center. It increases to about 10% in a radius of 2 to 3 cm and then decreases with further increases in the radius. The optical photograph and the Raman lines of the wafer show patterns similar to those of the thermal diffusivity. These are affected by the locations of the cathodes in the deposition chamber when the plasma power is low. Diamond wafers deposited at a high-power plasma (20.58 kW) with high concentrations of methane (10%, by volume) show higher values of thermal diffusivity and better uniformity than wafers deposited at a low power and low methane concentration. A fine crack can be located on a wafer with the converging thermal wave technique.

KEY WORDS: converging thermal wave technique; CVD diamond; diamond wafer; thermal diffusivity.

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1. INTRODUCTION

Diamond has good physical properties such as a high thermal conductivity, a high electrical resistivity, a low thermal expansion coefficient, and good mechanical properties. Many studies have been performed on the use of chemical vapor deposition (CVD) diamond as a thermal management substrate of high-power electronic packages such as the heat spreader of an IC chip or a laser diode. Large-area diamond wafers are required for this application. Recently it was reported that diamond wafers of large areas could be deposited by the dc arc-jet method [1] and the dc PACVD (direct-current plasma-assisted CVD) method [2, 3]. As a result, evaluation of the quality of large-area diamond wafers became a major field of interest. Thermal diffusivity can be used to characterize the diamond wafer.

Thermal properties are difficult to measure accurately. In the case of diamond, it is more difficult because CVD diamond has a high thermal conductivity and has a rough surface. Many techniques have been developed to measure the thermal conductivity of CVD diamond. Among them, the steady-state heated-bar technique has been proved to be the most accurate method in second round-robin measurements [4]. However, this method is rather time-consuming and requires a sophisticated sample preparation and a well-defined sample geometry. A converging thermal wave technique to measure the in-plane thermal diffusivity is independent of the specimen shape and size [5, 6]. This technique is a nondestructive and noncontact method. An annular ring of a pulsed laser beam is irradiated on a specimen as the heating source, and the temperature variation at the center of the ring is measured with an IR detector.

In this paper, the thermal diffusivity of free-standing diamond wafers of 10-cm diameter is measured with a converging thermal wave technique at room temperature. To verify the accuracy of the converging thermal wave technique, we measured the thermal diffusivity of high-purity copper and aluminum. The diamond wafers were deposited by multicathode dc PACVD [2, 3].

2. MEASUREMENTS

2.1. Experimental Apparatus

A converging thermal wave technique was used to measure the in-plane thermal diffusivity of 10-cm diamond wafers without breaking the wafers. A schematic diagram of the experimental apparatus is shown in Fig. 1. A pulsed Nd:Yag laser with an energy of 0.9 J/pulse, a wavelength of 1.06 μm, a diameter of 1 cm, and a pulse duration time of 0.3 ms is used as the heating source. The pulse duration time is short enough to compare

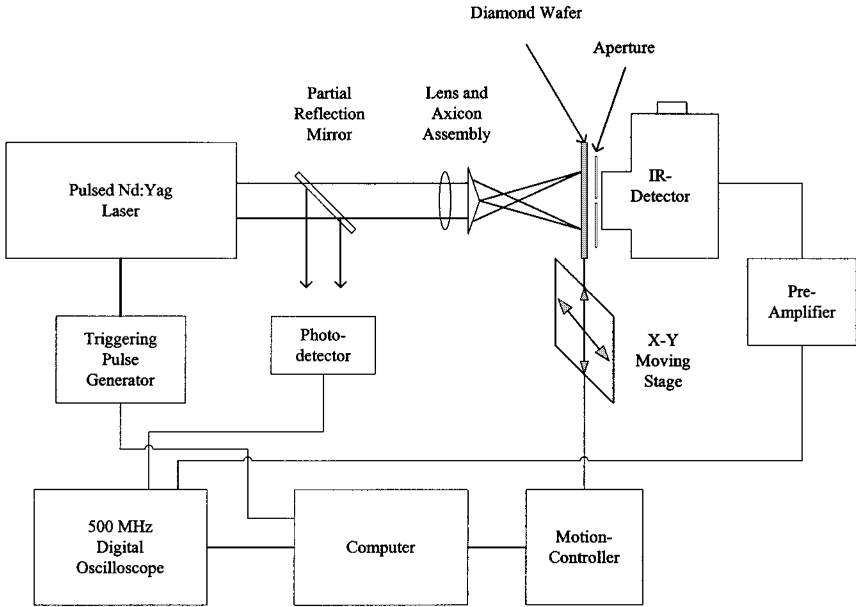

Fig. 1. Schematic diagram of the apparatus for the converging thermal wave technique.

with the characteristic thermal diffusion time. If the pulse duration time is too short, the laser pulse ablates the materials on the surface of the sample instead of providing thermal heating. For the annular laser beam to serve as a heating source, the laser beam is passed through a convex lens and an axicon (a lens of conical cross section) that has an apex angle of 5°. The focal length of the convex lens is 12.5 cm. The diameter of the annular ring is 13 mm, and the width is a few tens of micrometers. The diameter is measured to within ± 10 μm with a cathetometer. The diameter can be controlled by the focal length of the convex lens, the apex angle of the axicon, and the distance between the lens and the axicon. The annular laser beam is focused on the front surface of a diamond wafer. The temperature variation at the center of the heating ring on the back surface is measured with a HgCdTe (mercury cadmium telluride) detector that is cooled with liquid nitrogen. An aperture with a diameter of 1 mm is located in front of an IR detector. The output signal from the IR detector is measured with a 500-MHz digital oscilloscope with a preamplifier.

2.2. Data Analysis Methods

A detailed analysis of a thermal converging wave method is presented by Joo et al. [7]. A brief description of the data analysis method is as

follows. For a thin sample, the temperature variation at the center of the heating ring is given by [5]

$$T(t) = \frac{KR^2}{\alpha t} \exp\left(-\frac{R^2}{4\alpha t}\right) \tag{1}$$

where K is a multiplicative constant that is a function of E, ρ, and c. E is the energy absorbed per unit thickness, ρ is the density, and c is the specific heat. α is the in-plane thermal diffisivity, and R is the radius of the heating ring. The time to reach the maximum temperature at the center of the heating ring can be obtained by differentiating the above equation. The in-plane thermal diffisivity is given by

$$\alpha = \frac{R^2}{4t_{\mathrm{m}}} \tag{2}$$

where t_{m} is the time at which the temperature reachs a maximum. Nonlinear least squares is used to find the t_{m} on the trace of the temperature variation as a function of time. The fitting curve and the trace of the temperature variation are shown in Fig. 2. From the fitting curve, t_{m} can be determined within ± 0.2 ms.

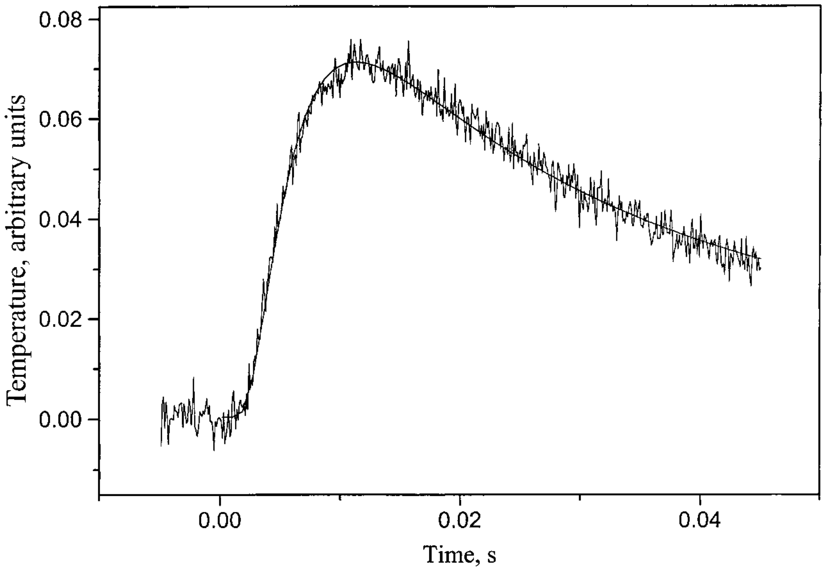

Fig. 2. Trace of temperature variation detected at the center of a diamond wafer. The line is the fitting curve.

2.3. Specimen Preparations

The diamond wafers were deposited by multicathode (seven) dc PACVD [2, 3]. A schematic diagram of the dc PACVD setup is shown in Fig. 3. The six cathodes are arranged at the apexes of a hexagon with an arm distance of 4.3 cm, and one cathode is located at the center. Each cathode is connected independently to a dc power supply. The distance between the cathodes and the substrate is 4 cm. This array of the cathodes is designed to enlarge the deposition area with good plasma distribution. The substrate is rotated throughout the whole deposition process. The feed gas is varied with different CH_4 concentrations in pure hydrogen. The gas flow rate is 100 sccm (standard cubic centimeters per minute), and the pressure in the chamber is maintained at 100 Torr. When the substrate is cooled after deposition, the diamond wafer is self-extracted from the

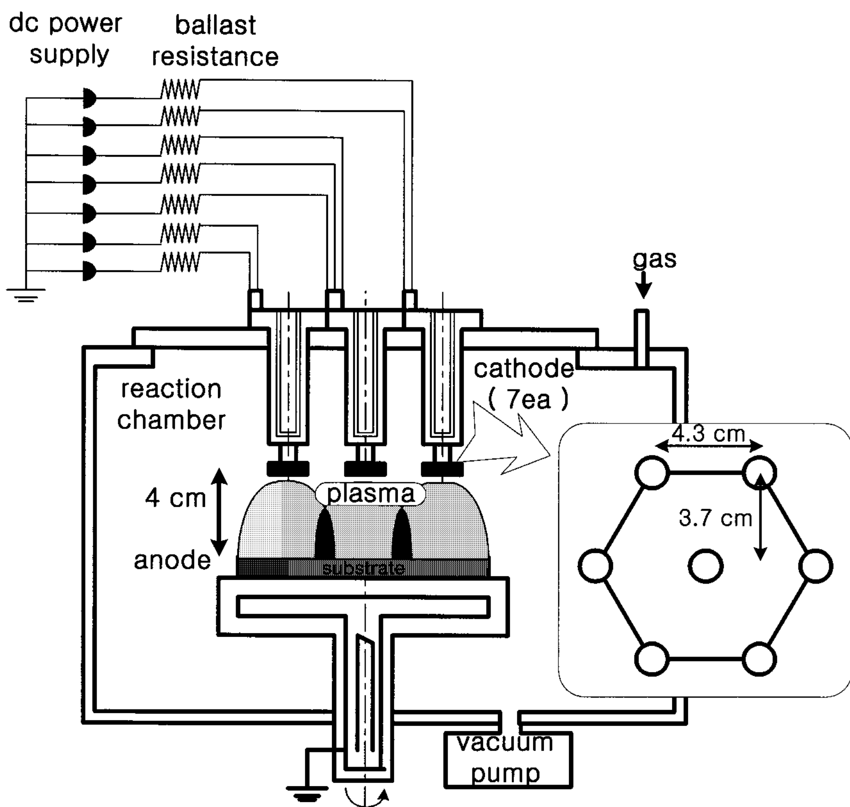

Fig. 3. Schematic diagram of the seven-cathode dc PACVD.

substrate by the difference in thermal expansion coefficients between the wafer and the substrate.

Two diamond wafers were deposited with different CH_4 concentrations and applied powers for the plasma. The diamond wafer with a CH_4 concentration of 6% was deposited at 1.9 kW for each cathode. Thus, the total power was 13.4 kW. The average deposition rate was 5 $\mu m \cdot h^{-1}$ throughout the deposition process. A pyrometer was used to monitor the substrate temperature. The average substrate temperature was 1250°C. The thickness range of the wafer is 850 to 1000 μm. The diamond wafer with a CH_4 concentration of 10% was deposited at a total power of 20.58 kW. The deposition rate was 9.4 to 9.6 $\mu m \cdot h^{-1}$, and the thickness was 800 to 876 μm. The deposition temperature was 1276°C. Dried graphite fluid was sprayed uniformly on both surfaces of the diamond wafer to a thickness of a few micrometers. This graphite-coated layer not only helps for uniform absorption of the incoming pulse but also gives a uniform emissivity to the IR detector.

3. RESULTS

The thermal duffusivity of high-purity copper and aluminum foils of varying thicknesses were measured with the converging thermal wave technique to investigate the thickness effect. In our calculation, the thermal diffusivity of diamond is decreased when the thickness is larger than 2.5 mm [7]. The thermal diffusivity of high-purity copper at thicknesses of 50, 100, 300, and 500 μm is 1.163 ± 0.023 $cm^2 \cdot s^{-1}$. These values agree with the literature value of 1.1234 $cm^2 \cdot s^{-1}$ within 4%. The thermal diffusivity of aluminum foils with thicknesses of 50 and 100 μm is 0.916 ± 0.018 $cm^2 \cdot s^{-1}$. The literature value for aluminum is 0.8418 $cm^2 \cdot s^{-1}$ and agrees within 8.8% [8].

The specimen size effect was studied by Joo et al. [7]. When the sample size is smaller than twice the diameter of the heating ring, the t_m increases rapidly, with a broader peak, and the temperature decreases very slowly after the maximum is reached. These results were confirmed in the experiment with copper. For this reason, the region inside 1 cm from the edge of the wafer was excluded from the measurements. For the same reason, the thermal diffusivity near a crack in the diamond wafer is lower than the thermal diffusivity measured far from the crack.

The thermal diffusivity distribution on the diamond wafer with a CH_4 concentration of 6% is shown in Fig. 4 as a contour map of the octagonal shaded area. The measured points were 175 on this wafer at an interval distance of 5 mm in the X and Y directions. The dark-colored region on the contour map represents a higher thermal diffusivity than the whiter region.

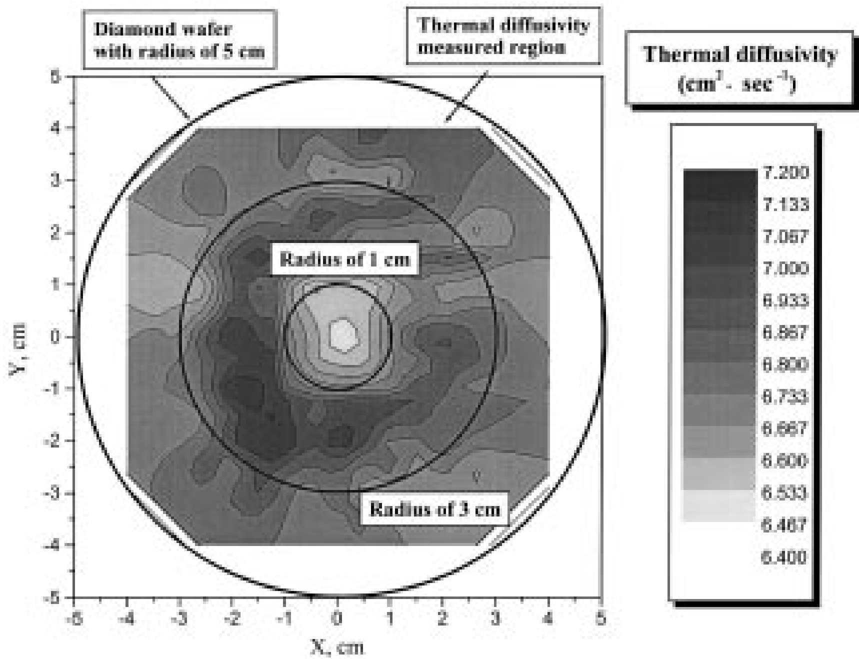

Fig. 4. Thermal diffusivity distribution on a 6% CH_4 diamond wafer.

Three diffusivity regions are shown. The minimum thermal diffusivity is 6.4 $cm^2 \cdot s^{-1}$, near the center of the wafer. The maximum thermal diffusivity is 7.2 $cm^2 \cdot s^{-1}$. The highest thermal diffusivity region is located between 1 and 3 cm from the center of the wafer. The thermal diffusivity shows intermediate values outside the radius of 3 cm from the center of the wafer. These regions are shown in a symmetric ring pattern. Raman peaks showed similar trends [3]. At 2 cm from the center, the Raman peak has a FWHM (full width at half-maximum) of 4.0 cm^{-1} and has the sharpest peak shape. Raman lines at other locations show higher FWHM values. The FWHM is 4.5 cm^{-1} at the center and 5.0 cm^{-1} at 4 cm from the center of the diamond wafer.

An optical photograph of the wafer is shown in Fig. 5. A pattern similar to the thermal diffusivity contour map can be found in the optical photograph. The black ring is the same area of the highest-thermal diffusivity region as in the contour map. The white central area and the outer ring correspond to the lowest and intermediate thermal diffusivity contours. These white regions are directly underneath the cathodes. The region

Fig. 5. Photograph of a 6% CH_4 diamond wafer.

of the highest thermal diffusivity is located between the central cathode and the outer cathodes.

Figure 6 shows a contour map of thermal diffusivity on a diamond wafer with a 10% CH_4 concentration. There are 37 measured points on this specimen. The thermal diffusivity is slightly higher than that for a diamond wafer with a 6% CH_4 concentration. The thermal diffusivity distribution is 5.0 to 7.6 $cm^2 \cdot s^{-1}$ and does not have ring patterns like the

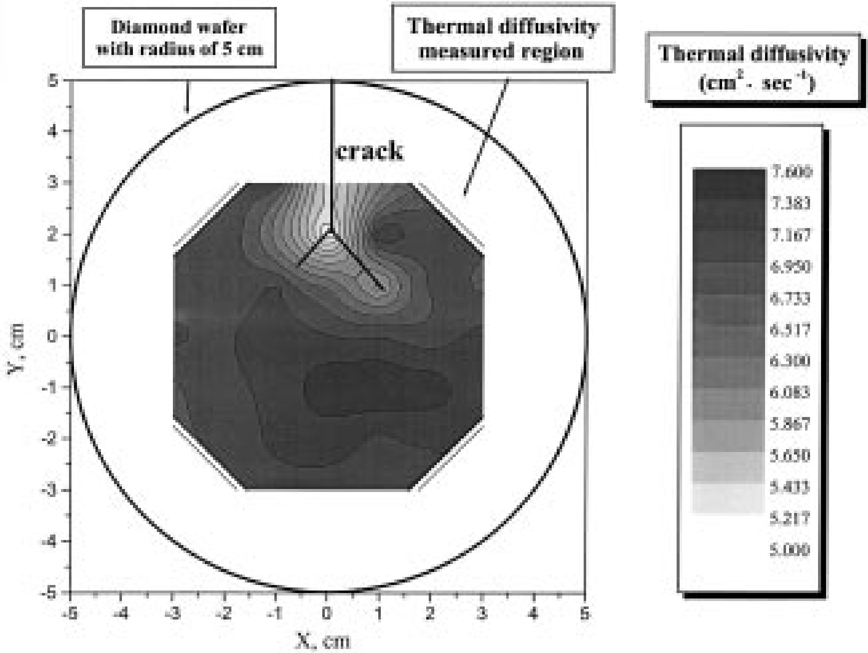

Fig. 6. Thermal diffusivity distribution on a 10% CH_4 diamond wafer.

diamond wafer of 6% CH_4 concentration. The variation of thermal diffusivity might be caused by the higher plasma power. A diamond wafer of better quality could be obtained by increasing the power of the plasma with a higher concentration of CH_4 with a multicathode dc PACVD.

The region near the positive Y axis in Fig. 6 shows a relatively lower thermal diffusivity than the other regions do. The locally low thermal diffusivity means that a crack exists near the measured point. It was not easy to locate the fine crack by visual inspection just after the deposition was completed. However, the crack location can be seen clearly on the diffusivity contour map.

4. CONCLUSION

The thermal diffusivity of 10-cm diamond wafers has been measured by the converging thermal wave technique. The diamond wafers were synthesized by seven-cathode dc PACVD. The thermal diffusivity distribution on the diamond wafers is given as a contour map. Three circular concentric thermal diffusivity patterns are found in the 6% CH_4 diamond wafer.

These patterns are related to the locations of the seven cathodes. The thermal diffusivity of a 10% CH_4 diamond wafer is higher than that of a 6% CH_4 diamond wafer deposited at a lower plasma power. The higher power and CH_4 concentration give a more uniform distribution of the thermal diffusivity values on the diamond wafer, along with a faster growth rate. The location of a fine crack barely visible on the diamond wafer could be detected by the converging thermal wave technique.

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