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Doping type influence on physical diamond properties

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Abstract

Diamond exhibits outstanding properties that make it a material of interest for the semiconductors industry. The industry required a silicon alternative that enabled devices to be smaller, cooler, faster, more powerful and cleaner. Diamond exhibit a large energy band gap that needs to be doped in order to observe any significant electrical conductivity. The purpose of this work is to compare the electrical properties of intrinsic and doped diamond based materials by impedance spectroscopy, I-V measurements and thermal conductivity measurements. The results of impedance measurement of the real and imaginary parts were presented in the form of a Nyquist and dielectric permittivity curves. In order to interpret the observed response, a resistor–capacitor circuit model was used for physical mechanisms interpretation. Direct evidence for the doping influence on the diamond, was obtained by I-V measurements. We describe Raman spectroscopy based method of measuring thermal conductivity of diamond doped samples, and review results achieved by line width correlation technique.

1. Introduction

In 1947 the invention of the transistor, brought extreme change in the modern world and it has paved the way for a digital world expansion. Since then, our digital devices kept getting smaller faster and cheaper. According to Gordon Moore’s law, every 2 years, the numbers of the components in the integrated circuit, will be doubled. Since Moore’s prediction, the electronics industry has been able to double the amount of the components in each circuit. However, as the components get smaller and smaller this prediction is designated to soon be over because of the critical heat flux generation from the conventional semiconductor materials on small substrate surfaces [1].

Diamond is a wide band semiconductor (5.45 eV) and has been established as a promising candidate for use in electronic device applications in the near future. Numerous papers have been published on the diamond’s superior properties for electronic industry, such as: negligible degrading performance, best heat transfer efficiency (thermal conductivity of 20 W cm⁻¹ K⁻¹), high voltage break down point (10 M V cm⁻¹) and electron–hole fast mobility (electron mobility of 4500 cm² V⁻¹ s⁻¹ and hole mobility of 3800 cm² V⁻¹ s⁻¹) and its low relative permittivity (5.87) [2, 3]. The exceptional intrinsic properties make the diamond a particularly attractive material in various fields of application as high power, high temperature electronics and high switching frequency [3, 4]. However, in the current electron industry the diamond based materials are mainly applied for heats-spreading purposes by incorporating it as an intermediate layer between a heat source and a heat sink [5].

The intrinsic carrier concentration of diamond is very low at room temperature and consequently the resistivity of pure diamond can exceed 10¹⁶ Ω cm only [6]. Therefore, diamond is a semiconductor that must be incorporated with extrinsic dopants, in order to reduce the electrical resistivity. Boron (B), which is an acceptor in diamond, has an ionization energy of 0.37 eV, and by very high dopants concentrations the activation energy of diamond approaches zero and the conduction becomes metallic. A resistivity of about 10⁻³ Ω cm at 300 K can
be achieved for boron concentrations above 10^{21} \text{ cm}^{-3} \[7, 8\]. Extrinsic n-type conductivity in diamond can also be achieved, by doping with phosphorus (P) with an ionization energy of 0.52 eV and nitrogen (N) with an ionization energy of 1.7 eV \[9\].

The dielectric function of wide gap materials is a fundamental quantity that is related to its electronic structure and describes its physical properties. However, almost all results published over the past two decades are focused on electrochemical dielectric measurements of diamond doped boron \[10–12\]. The overall objective of this work is to understand the doping influence on dielectric properties of diamond. Dielectric spectroscopy, combined I-V measurements are applied to gain quantitative information of doping influence on electrical diamond properties. Furthermore, the modification of carrier transport may reduce the superior thermal conductivity properties of diamonds. Therefore, we investigated the effect of doping on the thermal conductivity properties.

2. Experimental

The CVD polycrystalline diamond samples under investigation were deposited by micro-way plasma technology (Diamond Materials lab, Germany); Young’s modulus 1050 GPa, Atom density $1.77 \times 10^{23}$ 1/cm$^3$, Bandgap 5.45 eV, Thermal expansion coefficient $1.0 \times 10^{-6}$/K @300K. All of the investigated samples had an identical geometric area of 0.25 cm$^2$ where nitrogen doped diamond ($0.5 \text{ cm} \times 0.5 \text{ cm}, 300 \mu\text{m thickness}$), intrinsic diamond ($0.5 \text{ cm} \times 0.5 \text{ cm}, 300 \mu\text{m thickness}$) and boron doped diamond ($0.5 \text{ cm} \times 0.5 \text{ cm}, 300 \mu\text{m thickness}$) had been explored.

2.1. Time-of-flight secondary ion spectroscopy

ToF-SIMS analysis was carried out to characterize the doping density of the diamond bulks using a Physical Electronics TRIFT II ToF-SIMS instrument, 15-kV Ga primary ion gun.

2.2. Impedance spectroscopy and I-V measurements

Electrical performances of metal coated diamond electrodes were carried out using a LCR meter in the frequency range of 20 to $10^8$ Hz with AC amplitude of 1V and high voltage-1100 v, SMU. All measurements of the electrical properties were performed using a specially designed two-point probe in Faraday Cage system (Applied Mezo systems).

2.3. Raman experiment

Raman experiment was conducted using HR Evolution Horiba Raman instrument. The experiment was done with an excitation light wavelength of 532 nm, using 0.6–1.92 mW laser power, at room temperature and each spectroscopy was done for 5–20 s to collect the Raman scattering.

3. Results and discussion

3.1. Dielectric spectroscopy

Secondary-ion mass spectrometry (SIMS) is a technique used to analyze the composition of solid surfaces and thin films by sputtering the surface of the specimen with a focused primary ion beam and collecting and analyzing ejected secondary ions. The concentration of boron was $10^{17}$ cm$^{-3}$ and of the nitrogen concentration was $10^{17}$ cm$^{-3}$ in the doped diamond samples. The dielectric spectroscopy covers the wide frequency range by using computer controlled impedance analyzers, for each frequency is necessary to perform accurate dielectric measurements. The polarization of the sample, is being induced by a weak measuring electric field. The fundamental electrical property through which the interactions are described is the complex relative permittivity of the material, $\varepsilon(\omega)$. It is mathematically expressed as:

$$\varepsilon(\omega) = \varepsilon_1(\omega) + i \cdot \varepsilon_2(\omega)$$

$\varepsilon'(\omega)$ and $\varepsilon''(\omega)$ are, respectively, the real and imaginary parts of the complex dielectric permittivity.

The measurements of diamond dielectric properties were based on electrical impedance spectroscopy method, using LCR meter in a wide range of frequencies ($f = 10 – 10^6$ Hz) at room temperature.

The impedance as a complex function of capacitor cell, can be defined as:

$$Z(\omega) = R + \frac{1}{i \cdot \omega \cdot C} = Z'(\omega) + i \cdot Z''(\omega)$$
where the real part refers to resistive contributions and the imaginary part to the capacitive:

\[
Z_{\text{RC}}(\omega) = \frac{-i}{\omega \cdot \varepsilon''(\omega) \cdot C_0}
\]  

(3)

When:

\[
C_0 = \frac{\varepsilon_0 \cdot A}{d}
\]  

(4)

\[
\varepsilon^*(\omega) = \varepsilon'(\omega) + i \cdot \varepsilon''(\omega) = \frac{-i}{\omega \cdot Z_{\text{RC}}(\omega) \cdot C_0}
\]

(5)

\[
\begin{align*}
\varepsilon'(\omega) &= \frac{Z''(\omega)}{\omega \cdot (Z'(\omega)^2 + Z''(\omega)^2) \cdot C_0} \\
\varepsilon''(\omega) &= \frac{Z'(\omega)}{\omega \cdot (Z'(\omega)^2 + Z''(\omega)^2) \cdot C_0}
\end{align*}
\]  

(6)

Figure 1(a) shows the impedance Nyquist plot of an intrinsic diamond when \( Z' \) characterizes the reactive impedance part of capacitor sample and \( Z'' \), the resistance part. The impedance at low frequencies (20–90 Hz) shows a linear line (45°), which can be explained by Warburg element (W1) [13]. This region represents diffusion process of free charges between the layer of surface and metallic ohmic contacts. Experimentally, the low frequency dispersion corresponds to the grain boundaries polycrystalline diamond surface and to free carriers of electrodes' ohmic contacts (Warburg element (W1)), which cause significant resistance at the time of charges polarization between surface boundary with metal contacts [13]. The resistivity part at low frequencies was \( R_{W1} = 20 \) GΩ. The high frequency (90 Hz–1 MHz) dependence is completely different and it consists of a vertical line behavior. Only ideal capacitor behavior of the cell would exhibit the vertical line of the Nyquist plot.

The measurement cell could be represented by the well-known equivalent circuit. In addition, the real and the imaginary parts of dielectric permittivity were measured as a function of frequency. \( \varepsilon'(\omega) \) (figure 1(b)) describes the real part and shows the changes in the available stored energy of ideal capacitor as a function of frequency. On the other hand, \( \varepsilon''(\omega) \) describes the imaginary part of the dielectric permittivity and corresponds to dielectric losses. The real dielectric permittivity measured (figure 1(c)) showed a certain amount of decrease at frequencies of 20–90 Hz, presumably due to the diffusion process of free charges between the layer of surface and metallic ohmic contacts. The high frequency (90 Hz–1 MHz) dependence is completely different and it consists only a dielectric constant behavior. The imaginary part of the dielectric permittivity goes through a maximum at a frequency of 21.1 Hz, which defines the dielectric relaxation of free charges between the layer of surface and the metallic ohmic contacts. At high frequency, the equivalent impedance tends to be \( R_0 = 230 \Omega \). As the frequency becomes large, the dependence of the free carrier contribution will become less important, and the interband charge-transfer processes will dominate [13–16].

An equivalent circuit model of intrinsic diamond is shown in figure 2.

The capacity \( C_1 \) calculation is made from the dielectric constant data (figure 2). The result corresponds to the geometrical data of the manufacturer's sample which includes an error margin that may influence the dielectric constant data. However, the result is consistent with the expectations of this experience (\( \varepsilon_{\text{pure diamond}} \approx 5 \)):

\[
C_1 = \frac{\varepsilon_1 \cdot \varepsilon_0 \cdot A}{d}
\]

(7)

\[
\Rightarrow C_1 = \frac{4.12 \cdot \varepsilon_0 \cdot \pi \cdot (1 \cdot 10^{-3})^2}{300 \cdot 10^{-6}} = 0.382 \, \text{pF}
\]

(8)

Figure 3(a) shows the impedance Nyquist plot of diamond doped nitrogen when \( Z' \) characterizes the reactive impedance part of capacitor sample and \( Z'' \), the resistance part. The impedance at low frequencies shows a smaller resistance impedance data compared to intrinsic diamond. The decrease in this region represents diffusion process of more free charges between the layer of surface and metallic ohmic contacts in doped diamond (figure 3(a)). The Nyquist plot at upper frequency's 100 Hz < f < 1 MHz (figure 3(a)) is completely different and it consists of two parts. The first part is a non–perfect semicircle while the second part shows a conventional diamond resistance at high frequency (1 MHz). In the medium frequencies, the impedance reveals a non–perfect semicircle. Such behavior corresponds to a low parallel capacity \( C_1 \) and resistance \( R_1 \).

The real dielectric permittivity measured (figure 3(b)) showed an exponential decrease up to pure dielectric permittivity of intrinsic diamond, (\( \varepsilon_{\text{pure diamond}} \approx 5 \)). The imaginary part of the dielectric permittivity goes through a maximum at a frequency, (51.1 Hz), which defines the dielectric relaxation of free charges between the layer of surface and metallic ohmic contacts. It corresponds to the free carrier's polarization of electrodes ohmic contacts and diffusion processes (Warburg). Furthermore, the imaginary part of the dielectric permittivity
showed a certain amount of decrease at all frequencies, presumably due to the donations of nitrogen electrons. The capacity calculation $C_i$ is made from the dielectric constant data according to the frequency (figure 3(b)). The result corresponds with $(\varepsilon_{\text{pure diamond}}^{-5})$. According to the equivalent circuit of Nyquist plot results
Calculation of nitrogen doped diamond:

\[ C_1 = \frac{\varepsilon_0 \cdot \varepsilon \cdot A}{d} \] (13)

\[ \rightarrow C_1 = \frac{5.34 \cdot \varepsilon_0 \cdot \pi \cdot (1 \cdot 10^{-3})^2}{300 \cdot 10^{-6}} \] (14)

\[ \rightarrow C_4 = 0.5 \text{ pF} \] (15)

\( R_i \) resistivity represents parallel conductive process, presumably due to the donations of nitrogen. At high frequency, the equivalent impedance tends to \( R_2 = 551 \Omega \). As the frequency becomes large, the dependence of the free carrier contribution of nitrogen doping will become less important, and the interband diamond charge-transfer processes will dominate [13–15]. Dielectric permittivity results indicate that diamond doped nitrogen showed a certain amount of decrease at all frequencies, compared to Intrinsic diamond.

Figure 5(a) shows the impedance Nyquist plot of diamond doped boron when \( Z' \) characterizes the reactive impedance part of capacitor sample and \( Z' \), the resistance part. The Nyquist plot at all frequencies (20 – 1 MHz). The graph shows a semicircle which can be explained by parallel and series elements. This region represents parallel conductive process of free holes in doped boron diamond. Therefore, only three parameters of the system could influence the impedance: series resistance - \( R_2 \), parallel resistance- \( R_i \) and parallel capacitance - \( C_1 \). At higher frequencies (left part of the half circle), the - \( R_2 \) resistance is effectively shorted out by the \( C_1 \) capacitance, leaving only the series resistance \( R_2 = 193.53 \Omega \). At very low frequency (right part of the half circle), the imaginary part term in equation becomes negligible compared to \( R_2 \) and the real part tend toward \( R_1 + R_2 \).

The real dielectric permittivity measured (figure 5(b)) showed an exponential decrease for all frequencies, presumably due to the donations of boron free carrier acceptors. We couldn’t see the pure dielectric permittivity of intrinsic diamond, \( \varepsilon_{\text{pure diamond}} \) at 1 MHz, which can be explained by small concentration of free acceptors elements in this frequency region. The imaginary part of the dielectric permittivity showed a maximum decrease from the first frequency, presumably due to the resistivity nature of the diamond doped boron sample.

In terms of equivalent circuit (figure 6), \( C_4 \) acts as an open circuit and the equivalent impedance tends to \( R_1 + R_2 \). Consequently, \( R_1 + R_2 \) value can be determined directly from the intersection of the Nyquist plot with the x axis [13]. According to the experimental data:

\[ Z(\omega) = R_2 + \frac{1}{j \cdot \omega \cdot C_{W1}} + \frac{1}{j \cdot \omega \cdot C_{W2}} + \frac{1}{R_{W1}} + \frac{1}{R_{W2}} \] (10)

Resistance Warburg \( R_i \):

\[ Z(\omega \to 0^+) = R_2 + \frac{1}{R_{W1}} + \frac{1}{R_{W2}} = R_2 + R_{W1} + R_{W2} = 75.4 \text{ M}\Omega \] (11)

\[ R_{W1} = 75.4 \text{M} \Omega - R_{W2} - R_2 = 75.4 \text{ M} - 15.1 \text{ M} - 551 = 60.3 \text{ M}\Omega \] (12)
The equivalent circuit impedance of the sample is shown in figure 6 when:

\[ Z(\omega) = \frac{1}{j\omega \cdot C_1} \cdot R_1 + \frac{1}{j\omega \cdot C_2} + R_2 \]  

Figure 3. Nitrogen Doped Diamond: Impedance Nyquist Plot (a) and dielectric constant \( \varepsilon'(f) \) (b) - dielectric loss \( \varepsilon''(f) \) (c) according on the frequency.
The capacitance value \( C_1 \) can be obtained using the experimental data \( Z'(\omega) \) corresponding to a selected frequency according to the semicircle of Nyquist plot:

\[
Z'(\omega) = \frac{R_1}{(R_1 \cdot \omega \cdot C_1)^2 + 1} + R_2
\]

\[
Z''(\omega) = -j \cdot \frac{(R_1)^2 \cdot \omega \cdot C_1}{(R_1 \cdot \omega \cdot C_1)^2 + 1}
\]

\[
R_1 + R_2 = 340.2 \text{ k}\Omega
\]

\[
\Rightarrow R_1 = 340.215 \text{ k}\Omega - 193.53 = 340.021 \text{ k}\Omega
\]

The resistance of the \( R_1 \) (340.021 k\Omega) was found to be orders time smaller than that of the diamond doped nitrogen material. This decreased resistance results from the highly conductive properties of boron at room temperature.

### 3.2. I-V measurement and breakdown voltage

The breakdown voltage is the level at which the wide-gap semiconductors begins to allow charge flow. Unlike conducting materials, this charge flow tends to be non-linear. It means that below a threshold voltage no charge will flow, and at or above that voltage a rush of charge will flow.

The diamond acts as a pure capacity with a high inner resistance, the sample does not conduct during I-V measurements. Until 1.2 DC kV (probe station limitation) there was no ionization process, only very small noise measurement. The breakdown voltage of an intrinsic diamond is 10MV/cm \[17\] and after normalization of the thickness parameter (300 \( \mu \)m), the breakdown voltage of the undoped sample is estimated to be:

\[
V_{\text{breakdown}} = 10 \frac{M}{\text{cm}} \cdot 30 \cdot 10^{-3} \text{ cm} = 300 \text{ kV}
\]

The I-V measurement of nitrogen doped diamond (thickness parameter, 300 \( \mu \)m) shows the beginning of ionization spike process in a voltage of 800 V (figure 7(a)). Several mechanisms give rise to free charge moving, one of which is by impact ionization. Impact ionization occurs when either an internal or external field, acting on the dielectric accelerates electrons towards the anode, thereby facilitating collisions with ubiquitous neutrals. The result is the formation of electron-hole pairs. Each electron-hole pair gets polarized by the electric field and is accelerated. The effect of boron doping on I-V behavior is also demonstrated in (figure 7(b)) where the current is plotted as a function of voltage. The I-V plot shows linear function performance, from small voltages, the voltage increase was limited, in order to prevent the breakdown voltage. The resistance of the sample is:

\[
R = \frac{U[V]}{I[A]} = \frac{0.02}{2 \cdot 10^{-7}} = 100 \text{ k}\Omega
\]

This result corresponds to scale resistance parameter from Nyquist plot (figure 5(a)) at low frequencies.

### 3.3. Raman spectroscopy

The Raman spectrum contains a wealth of information that correlates with many of the important material properties, such as optical transmission, thermal conductivity, diffusivity and electrical properties \[18\]. Diamond has the highest thermal conductivity of any known material at temperatures above \( \sim 100\)K. The
purest natural diamond single crystals reported so far, have a conductivity of \( \sim 25 \text{ Wcm}^{-1}\text{K}^{-1} \) at 300 K. A variety of experimental techniques have come into use for the measurement of the thermal conductivity of diamond materials, \([18, 19]\) such as dc-heated bar technique, Angstrom's thermal wave method and the mirage effect technique. However, the experimental methodologies for thin and small samples are very problematic. Raman spectroscopic can be a convenient and noninvasive technique to ascertain differences in thermal conductivity. A

![Figure 5. Boron Doped Diamond: Impedance Nyquist Plot (a) and dielectric constant (b)-dielectric loss (c) according on the frequency.](image-url)
few attempts have been made to relate thermal conductivity to the width of the 1333-cm$^{-1}$ (sp$^3$-bonded) diamond Raman line [20].

We performed this measurement for three diamond samples: intrinsic diamond, boron doped diamond and nitrogen doped diamond at room temperature environment. The experiment was done with an excitation light wavelength of 532 nm, using 0.6–1.92 mW laser power, and each spectroscopy was done for 5–20 s to collect the Raman scattering. The bands were fit by Lorentzian band shapes. In our treatment of the spectra, we obtained excellent agreement between the observed and calculated line shapes, and the statistical uncertainties of the fit are much smaller than the estimated systematic errors.

While comparing all three diamond samples, it can be observed that in average the intrinsic diamond has the lowest full width at half maximum (FWHM), the nitrogen doped diamond has a higher FWHM than the
intrinsic, and the boron doped diamond has the highest FWHM of all three samples. According to this data it can be deduced that boron doped diamond has double degradation in heat conductivity versus to intrinsic diamond. The nitrogen doped diamond has a small degradation in heat conductivity versus to intrinsic diamond. These observations, also reported by other workers [21–23]. The dependence of the thermal conductivity of diamond samples versus the change of FWHM of the diamond Raman line (1332 cm\(^{-1}\)), may be correlated. The detailed analysis of the Raman spectra may provide a robust correlation of thermal conductivity of diamond samples [15]. The correlation is made through the determination of the FWHM (figure 8) of the diamond Raman line at 1333 cm\(^{-1}\) to the conventional thermal conductivity measurements.

Roughly this dependence can be given as follows [24]:

\[ \Lambda [\text{W cm}^{-1}\text{K}^{-1}] = 20 - a [\text{WK}^{-1}] \cdot \log(\Delta 1333\text{cm}^{-1}) \]

### Table 1. Results of thermal conductivity.

| Sample type          | \(\Lambda [\text{W cm}^{-1}\text{K}^{-1}]\) | FWHM cm\(^{-1}\) | \(a [\text{WK}^{-1}]\) |
|----------------------|--------------------------------------------|-------------------|------------------------|
| Intrinsic diamond    | 19.48                                      | 2.5               | 1.3                    |
| Diamond doped nitrogen | 17.8                                       | 3.5               | 4                      |
| Diamond doped boron  | 10.8                                       | 4.5               | 14                     |

Figure 8. (a) room temperature Raman spectroscopies for different diamond samples. (b) average full width at half maximum of the different samples at room temperature.
where \( a \) is the fitting constant. The fitting constants—range may be different, depending on the doping materials and the quality of a CVD process. For the correlation process, the data of thermal conductivity was taken from the data of Diamond Materials Lab. The results of the fit are shown in Table 1.

Deposited diamonds exhibit thermal conductivity properties that make it as a future material of interest for semiconductor industry. Despite correlation needs, our results show that the Raman method provides a useful, relatively fast and convenient optical-spectroscopic technique for the assessment of the thermal properties of CVD diamond. We can see that the Raman method represents an inherently simple and practical procedure for the determination of the room temperature thermal conductivity.

4. Conclusion

It has been observed that the intrinsic diamond acts as a capacity with very high resistance in low frequencies. Impedance results at low frequencies, indicate that the nitrogen-doped diamond leads to a much smaller resistance. This decreased resistance results from the nitrogen conductivity properties. The dielectric constant, of the nitrogen-doped diamond was \( \sim 2 \) times higher than that of the intrinsic diamond at low frequencies, an indication of the additional amount of free charges. The dielectric constant at high frequencies of the nitrogen-doped diamond was the same as the intrinsic diamond. This means that a nitrogen-doped diamond has a potential to conduct in certain conditions (depending on the doping concentration), keeping the characteristics of a diamond in high frequencies. The boron-doped diamond conducts well and it acts like a resistor with a capacitor in parallel. Our studies using, I-V and impedance spectroscopy techniques, enabled to identify the differences of various diamond bulk materials. An equivalent impedance model circuit that describes the differences of the various diamond bulk materials was proposed. I-V studies have shown that nitrogen doping changes the breakdown voltage performances of intrinsic diamond. It was also presented of very low breakdown voltage performances of diamond doped boron, direct evidence of the high contribution of free P type carriers in the room temperature. All the experiments were made at room temperature (300 K), temperatures influence will change the results. The detailed analysis of the Raman spectra provided room-temperature thermal conductivity of these samples. Despite thermal conductivity degradation, our results show that these materials still hold outstanding thermal conductivity properties. According to all physical experiments, the diamond materials characteristics can be changed depending on the required applications. The next challenge is to investigate the effect of doping in diamonds with variable concentration.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files). Data will be available from 14 October 2021.

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