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Electrophysical properties of diamond films deposited on silicon substrates by HFCVD method

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Electrically conductive diamond films were deposited on n-type Si(100) substrates by the hot filament chemical vapor deposition (HFCVD) method using a hydrogen–methanol gas mixture with ammonia addition. In contrast to conventional approaches, film growth was performed without preliminary hydrogen etching of the silicon surface, which simplified the technological process. The deposition conditions were optimized to suppress the formation of non-diamond carbon phases and improve film quality. Raman spectroscopy revealed a pronounced diamond-related peak at 1333 cm^{-1} with a relatively small contribution of sp^2-bonded carbon, indicating the predominance of the diamond phase in the deposited films. The films had thicknesses of 2.2–2.8 μm and were grown at a rate of 0.2–0.3 μm h^{-1}. The electrophysical properties were investigated in the temperature range of 300–500 K using Hall-effect measurements. The carrier concentration increased exponentially with temperature, while the carrier mobility decreased due to enhanced phonon scattering, confirming the semiconducting nature of the deposited films. The observed temperature dependences of carrier concentration, mobility, and resistivity were found to be consistent with the behavior expected for semiconductor materials. The results demonstrate that high-temperature HFCVD growth without preliminary hydrogen etching can produce diamond films with satisfactory structural quality and stable electrical characteristics, making them promising for potential electronic and optoelectronic applications.

Keywords: CVD technology, diamond film, silicon substrate, Raman spectra, charge carrier mobility, temperature dependence.

1. Introduction

Recently, there has been increasing interest in thin diamond films deposited on various substrates, particularly on silicon, due to their potential applications in micro- and optoelectronic devices [1–5]. Raman spectroscopy remains one of the most informative and widely used techniques for structural characterization of carbon-based and semiconductor nanostructures, providing information on crystallinity, defect formation, and phase composition [4, 8]. Diamond films on silicon are especially attractive because of the well-established silicon-based technology. In our previous studies [9–12], diamond films were successfully grown on silicon substrates using the HFCVD method. However, the obtained films were predominantly polycrystalline with grain sizes of 2–3 μm and contained noticeable amounts of graphite phases and a 15R-SiC interlayer, as revealed by Raman spectroscopy. In addition, the temperature dependence of electrophysical parameters such as carrier concentration, mobility, and resistivity were not investigated. Despite certain limitations related to filament degradation during operation, the HFCVD method remains attractive due to its simplicity and scalability. The film growth rate is typically about 1 μm/h and can be controlled by adjusting process parameters such as filament temperature and substrate distance [13]. In this work, diamond films were deposited on Si(100) substrates using the HFCVD method, and their electrophysical properties were investigated. The study of transport properties in nanostructured semiconductor materials remains important for understanding the relationship between synthesis conditions, structural quality, and functional characteristics [14]. Particular attention was paid to the temperature dependence of charge carrier concentration, mobility, and resistivity in order

to evaluate the suitability of this method for obtaining diamond films with properties close to those of single-crystal materials.

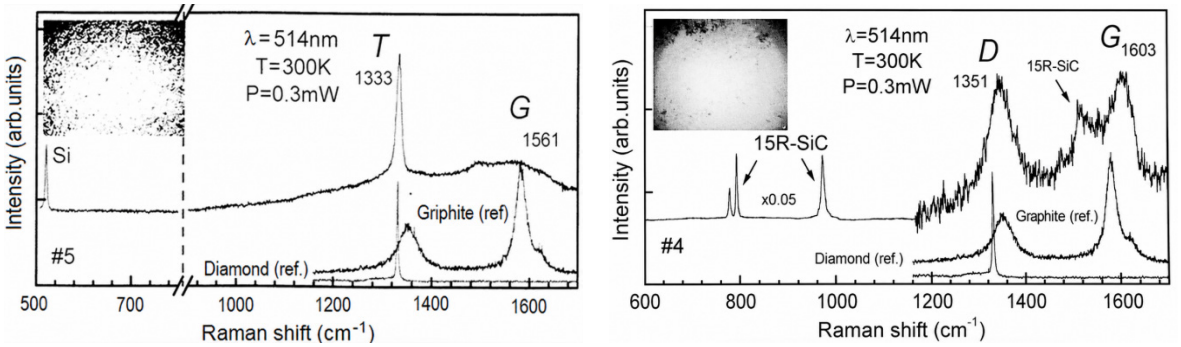
2. Materials and methods

Diamond films investigated in this study were deposited on Si(100) substrates using the hot filament chemical vapor deposition (HFCVD) method, as described in [1–3] and in our previous works [11, 12]. The substrates were n-type monocrystalline silicon wafers with a thickness of 300 μm and a resistivity of 0.1 Ω·cm. The gas phase consisted of a hydrogen–methanol (CH_3OH) mixture with the addition of ammonia (NH_3). In contrast to our previous studies, the deposition process was carried out without preliminary hydrogen etching of the silicon substrate, and the technological parameters were slightly modified. In the HFCVD process, a reduced pressure (typically 20–60 Torr) is maintained in the reaction chamber, and gas activation is achieved by a heated tungsten filament operating at temperatures of 2000–2200 °C. Before discussing the deposition process in detail, it is necessary to consider the key technological parameters influencing the properties of the diamond films. In HFCVD, diamond growth occurs in a hydrogen-rich atmosphere containing a carbon source (such as CH_4, CH_3OH, C_2H_6, or C_2H_5OH) and, in some cases, additional impurities such as NH_3. The addition of ammonia introduces nitrogen into the diamond lattice, which can affect both growth kinetics and film morphology. It is reported that nitrogen concentrations in the range of 0.1–0.6% can enhance the growth rate and stabilize the surface during deposition [15,16]. Nitrogen incorporation in diamond introduces deep donor levels (~1.7 eV below the conduction band), which influence the electrical properties of the films. In addition, nitrogen-related defects, such as NV centers, have been widely studied due to their potential applications in quantum technologies [17–20]. Diamond film growth on Si(100) substrates was carried out under the following conditions: filament temperature 2250–2300 °C, substrate temperature 870–900 °C, total gas flow rate ($CH_3OH + H_2 + NH_3$) of 65–70 cm³/min, and chamber pressure of 75–80 Torr. The methanol vapor concentration was maintained at 0.5–0.8% of the total gas mixture. The deposition time was 8–10 hours, resulting in film thicknesses of 2.2–2.8 μm and an average growth rate of 0.2–0.3 μm/h. It should be noted that, under identical growth conditions, approximately 50% of the samples exhibited a polycrystalline structure. This variability may be attributed to fluctuations in nucleation density, local temperature gradients, and variations in active hydrogen concentration during the deposition process. The selected growth conditions involve relatively high filament and substrate temperatures compared to those reported in [4, 5, 9]. These parameters were chosen to enhance the concentration and activity of atomic hydrogen, which preferentially etches sp^2-bonded carbon (graphite phase) more effectively than sp^3-bonded carbon (diamond). As a result, the graphite phase content is reduced, leading to improved film quality, although at the expense of a lower growth rate.

3. Results and discussion

The HFCVD method is based on complex gas-phase reactions leading to the deposition of synthetic diamond layers on a solid substrate [21]. In the present process, near the tungsten filament heated to 2250–2300 °C, methanol (CH_3OH) decomposes into carbon-containing species (primarily CO and H_2), while molecular hydrogen (H_2) dissociates into atomic hydrogen (H). Ammonia (NH_3) also undergoes thermal decomposition, forming reactive species such as NH radicals and hydrogen atoms. The decomposition of methanol can be described by the reaction: $CH_3OH \rightarrow CO + 2H_2$. According to [15], ammonia decomposition at temperatures above 1200–1300 °C may proceed via the formation of nitrogen and hydrogen ($2NH_3 \rightarrow N_2 + 3H_2$) or intermediate species. However, under typical HFCVD conditions, NH_3 predominantly decomposes into NH radicals and hydrogen ($2NH_3 \rightarrow N_2 + 3H_2$), where NH radicals play a key role in nitrogen incorporation into the diamond lattice. Diamond films are formed from carbon-containing radicals that reach the substrate surface. In this process, carbon atoms initially interact with silicon atoms at the substrate surface, forming a thin silicon carbide (SiC) interlayer. Subsequently, diamond nuclei are formed on the SiC surface, where both sp^2- and sp^3-bonded carbon species are deposited. The gas-phase reactions during HFCVD lead to the formation of both sp^2- and sp^3-hybridized carbon. However, sp^2-bonded carbon (graphite phase) is preferentially etched by atomic hydrogen due to its lower bond energy compared to sp^3-bonded carbon (diamond). This selective etching process plays a key role in promoting diamond growth. As a result, the growth conditions can be adjusted such that the etching rate of the non-diamond phase exceeds its formation rate. Under these conditions, the growth rate of the diamond film decreases, but the structural quality improves due to the reduc-

tion of graphite inclusions. Ideally, diamond consists entirely of sp^3-bonded carbon, which determines its unique mechanical and electrical properties. To confirm the formation and structural quality of the diamond films, Raman spectra were recorded at 300 K using a laser with a wavelength of 514 nm (Figure 1a). A pronounced peak near 1333 cm^{-1}, characteristic of sp^3-bonded carbon and typically associated with diamond, is observed, which is close to the reference value of 1332 cm^{-1} [10]. Similar approaches based on Raman spectroscopy have been successfully applied for structural characterization of nanostructured silicon and semiconductor materials, where peak position and linewidth were shown to be sensitive to crystal quality and defect concentration [6, 20]. The full width at half maximum (FWHM) of the diamond peak is estimated to be approximately 10–12 cm^{-1}, indicating relatively good crystalline quality. The slight broadening of the peak suggests the presence of minor structural defects and small amounts of sp^2-bonded carbon. A weaker peak at ~1561 cm^{-1} is also observed, corresponding to the graphite phase (sp^2-bonded carbon), indicating incomplete etching during the growth process. The relatively weak G-band intensity, compared to the sharp sp^3-related peak near 1332 cm^{-1}, indicates that the film is predominantly sp^3-bonded carbon [10, 23]. Thus, the Raman analysis demonstrates that the selected HFCVD conditions favor the formation of diamond with a relatively low content of non-diamond phases.



a) *b)*

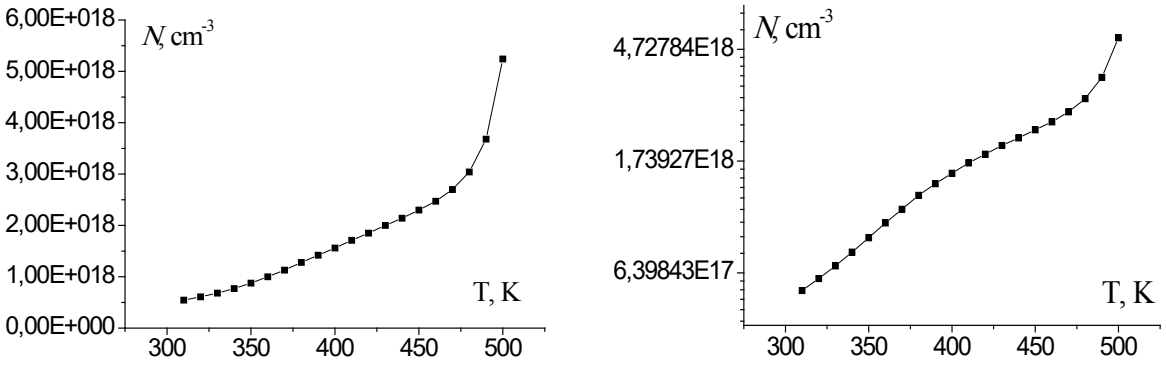
Figure 1. Raman spectra of diamond films grown by HFCVD on silicon substrates with (100) (a) and (111) (b) orientations under different technological conditions.

Figure 1a suggests improved structural quality of the deposited carbon film with a predominance of sp^3-bonded carbon.

When the substrate was pre-etched with hydrogen before film deposition, diamond peaks were not detected in the Raman spectrum. Instead, peaks corresponding to a graphite-like phase were observed. The Raman spectrum of this film also contains an additional peak associated with the 15R-SiC polymorph, which is absent in Figure 1a. The formation of the 15R-SiC interlayer is likely related to the initial interaction between carbon species and the silicon substrate, resulting in a transition layer at the interface. As the film thickness increases, the contribution of this interlayer decreases, while graphite growth becomes dominant. The sharper peak in Figure 1a confirms the improved structural quality of the diamond film obtained under the present growth conditions. Figure 2 shows the temperature dependence of the charge carrier concentration in a diamond film in both normal (a) and semilogarithmic (b) scales. The Raman spectrum of this sample is shown in Figure 1a. As seen from the semi-logarithmic plot, the carrier concentration increases exponentially with temperature, indicating thermally activated conduction typical of semiconductors [23]. At temperatures up to approximately 480–485 K, the concentration increases gradually, corresponding to partial ionization of impurity centers. At higher temperatures (above ~490 K), a more pronounced increase is observed, which can be attributed to the activation of deeper impurity levels or defect-related states, possibly associated with residual sp^2-bonded carbon. The temperature dependences of carrier mobility (Fig. 4a) and resistivity (Fig. 4b) were also investigated. The increase in carrier concentration leads to a corresponding decrease in resistivity. A slight increase in resistivity observed in the low-temperature region

(300–320 K) may be associated with carrier localization or compensation effects. The mobility decreases with increasing temperature: at lower temperatures the change is weak, while at higher temperatures it becomes more pronounced due to enhanced phonon scattering and increased carrier–carrier interactions.

Overall, the observed temperature dependences of carrier concentration, mobility, and resistivity are generally consistent with the behavior expected for semiconducting materials.

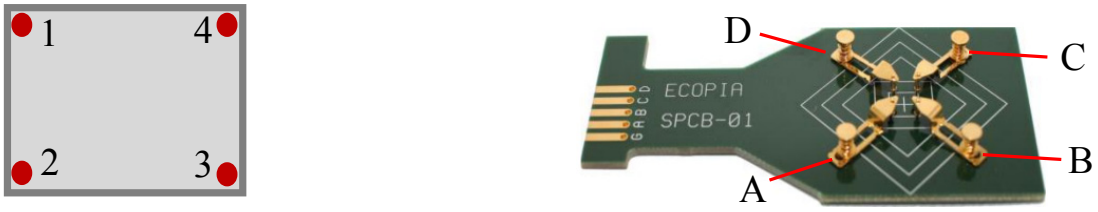


a) b)

Figure 2. Temperature dependence of the charge carrier concentration (N) in the diamond film in normal (a) and semi-logarithmic (b) scales.

The temperature dependence of the carrier concentration reflects impurity ionization processes and the activation of intrinsic defects. Analysis of the N(T) behavior allows estimation of the activation energy of impurity levels and the presence of deep defect states.

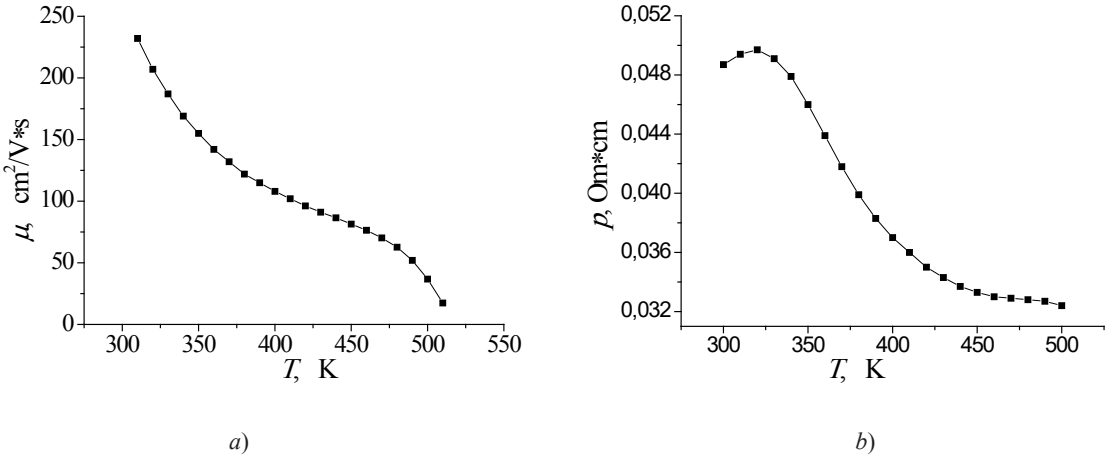
The resistivity of the semiconductor is related to the carrier concentration and mobility by the expression $\rho = 1/q(\mu_n n + \mu_p p)$, where n and p are the concentrations of electrons and holes, respectively [22]. Electrical measurements were performed using the Van der Pauw method with an HMS-7000 Hall measurement system, enabling simultaneous acquisition of temperature-dependent data. Ohmic contacts were formed by thermal evaporation of silver (Ag) in vacuum (~10^{-5} Torr) at a sample temperature of approximately 450 °C, followed by annealing for 10 minutes. Four contacts were placed at the sample edges, as shown in Figure 3a. The experimental sample mounted in the measurement holder used for Hall-effect characterization is shown in Figure 3b. The overall uncertainty of resistivity, Hall mobility, and carrier concentration measurements was approximately 2%.



a) b)

Figure 3. Contact configuration for Van der Pauw measurements on the diamond film, showing four ohmic contacts placed at the sample edges (a), and the experimental sample holder (b).

The temperature dependence of charge carrier mobility μ(T) is governed by the competition between different scattering mechanisms. At low temperatures, mobility is mainly limited by scattering on ionized impurities, resulting in $\mu \propto T^{3/2}$. At higher temperatures, scattering by acoustic and optical phonons becomes dominant, leading to $\mu \propto T^{-3/2}$ [23]. In non-polar semiconductors such as Si, Ge, and diamond, the dominant scattering mechanisms are impurity scattering and phonon scattering. The overall mobility can be described by Matthiessen's rule, which combines the contributions of different scattering processes. The observed decrease in mobility with increasing temperature in this work indicates that phonon scattering plays a dominant role at elevated temperatures, while impurity scattering is more significant at lower temperatures.



a) *b*)

Figure 4. Temperature dependence of charge carrier mobility μ (a) and resistivity ρ (b) of the diamond film.

Figure 4 shows the temperature dependence of charge carrier mobility and resistivity of the diamond film. As seen from Figure 4a, the mobility decreases with increasing temperature. This behavior is typical for semiconductors and is mainly attributed to enhanced phonon scattering at elevated temperatures. At lower temperatures, the decrease in mobility is relatively gradual, indicating a contribution of impurity scattering. However, at higher temperatures, the mobility decreases more rapidly due to the dominant role of lattice vibrations. Figure 4b demonstrates that the resistivity decreases with increasing temperature, which is consistent with the increase in carrier concentration observed in Figure 2. A slight increase in resistivity at low temperatures may be associated with carrier localization or compensation effects. Overall, the observed temperature dependences of mobility and resistivity confirm the semiconducting nature of the obtained diamond films. These results can be explained by the increase in thermal energy with temperature, which leads to higher electron velocities and enhanced scattering by acoustic and optical phonons. As a result, phonon scattering becomes the dominant mechanism at elevated temperatures, causing a decrease in charge carrier mobility [24, 25]. As shown in Figures 2 and 4, the temperature dependences of carrier concentration (N), mobility (μ), and resistivity (ρ) are generally consistent with the behavior expected for semiconducting materials.

4. Conclusions

Diamond films were successfully deposited on Si(100) substrates using the HFCVD method under the selected growth conditions. The obtained films had thicknesses of 2.2–2.5 μm and were formed at a growth rate of 0.2–0.3 μm/h. Raman analysis indicated the presence of predominantly sp^3-bonded carbon with a characteristic peak near 1333 cm^{-1}. The temperature dependences of charge carrier concentration, mobility, and resistivity were investigated. The results demonstrate typical semiconductor behavior: the carrier concentration increases with temperature, while mobility decreases due to enhanced phonon scattering. Overall, the obtained results indicate that the HFCVD method enables the

growth of diamond films on silicon substrates with stable structural and electrical properties, making them promising for potential electronic and optoelectronic applications.

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Conflicts of interest

The authors declare no conflict of interest.

Author contributions

Conceptualization: A.S.S and A.K.; Methodology: A.K. and U.Kh.R.; Software: U.Kh.R.; Validation: A.S.S., A.K. and D.V.S.; Formal Analysis: A.S.S. and D.V.S.; Investigation: U.Kh.R. and O.Z.T.; Resources: A.K. and D.V.S.; Data Curation: U.Kh.R.; Writing – Original Draft Preparation: U.Kh.R.; Writing – Review & Editing: A.S.S and A.K.; Visualization: U.Kh.R.; Supervision: A.S.S.; Project Administration: A. K.; Funding Acquisition: A.S.S. All authors have read and agreed to the published version of the manuscript.

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