Diamond nanocrystal thin films: Case study on surface texture and power spectral density properties

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ABSTRACT
Analyzing diamond nanocrystal (DNC) thin film morphology produced by the HFCVD technique is the main objective of the present work. Stereometric analysis of three-dimensional surface microtextures was carried out based on data obtained through atomic force microscopy (AFM), while the ISO 25178-2:2012 standard was applied to characterize surface topography. The Abbott–Firestone curve, peak count histograms, and Cartesian graphs, which were extracted through AFM images, gave valuable statistical information. As can be seen, the most isotropic sample was the Au catalyst (etched) deposited by the hot filament chemical vapor deposition method. Moreover, by increasing the time of DNC growth from 15 min to 60 min, the surface roughness was increased. In addition, the average power spectral density was calculated and furrows were determined for all samples.

I. INTRODUCTION
The unique properties of nano-materials make them suitable candidates to be applied in various types of technologies in the field of science, biology, and engineering. Among these types of materials, diamond nanocrystal (DNCs) thin films, because of their significant properties, motivated scientists to use them in industrial sectors over the last few decades. Diamonds, because of their high surface to volume ratio and significant properties like high breakdown field strength, high electron and hole mobilities, and large bandgap (5.5 eV), attract as much research interest as other carbon allotropes, i.e., graphite and carbon nanotubes. It is the hardest material that has ever been known with the maximum value of wear resistance.

In the process of producing DNCs, several methods such as the electrochemical method, conformal coatings for micro-electromechanical systems (MEMSs), and the chemical vapor deposition (CVD) method have been introduced.1 11 Between them, the chemical vapor deposition (CVD) method is considered the best method for the growth of diamond films. Hot filament CVD (HFCVD) has been used here for the growth of DNC films due to its high efficiency and low cost.12

Other methods require optimization of diamond properties including surface smoothness, field emission, and electrical...
TABLE I. The details of the prepared samples.

| Sample ID no. | Gas type | Filament temperature (°C) | Substrate temperature (°C) | Time (min) | Deposition method | Yield |
|---------------|----------|---------------------------|---------------------------|------------|-------------------|-------|
| 1             | Ar       | ...                       | ...                       | Room temperature | DC-magnetron sputtering | Au catalyst |
| 2             | H₂       | 100                       | 1700                      | 550        | 15                | HFCVD  |
| 3             | CH₄/H₂   | 100                       | 1800                      | 600        | 15                | HFCVD  |
| 4             | CH₄/H₂   | 100                       | 1800                      | 600        | 30                | DNCs   |
| 5             | CH₄/H₂   | 100                       | 1800                      | 600        | 60                | HFCVD  |

conductivity on the microscale; however, in the CVD method, the diamond film microstructure and crystalline size can be controlled in the nanoscale.

Recently, much research has been focused on the tribology of major applications of DNC films by focusing on their friction coefficients, wear rates, and hardness, but morphological studies of these films and their surface engineering are missing.

In order to investigate surface topography of these types of materials, atomic force microscopy (AFM) has been considered as the best tool in the modern surface-imaging area. Three-dimensional (3D) images of thin film surfaces at the microscopic level give valuable information about energy dissipation mechanisms and macroscopic friction. It should be noted that the geometry of DNCs can be extracted through AFM images by fractal and multifractal geometry. AFM has been applied to elucidate the surface geometry microtexture with high nanoscale resolution. Furthermore, stereometric, fractal/multifractal analyses, as well as analyses by power spectral density (PSD) functions, are accurate tools for characterization of the nano-scale patterns in 3D complex surfaces of thin films.

Here, we report the growth of nanocrystalline diamond thin films and study their 3D micromorphology prepared by using the HFCVD method. The fundamental properties of these films have been comprehensively investigated by AFM. Fractal/multifractal geometry along with statistical surface parameters was extracted from AFM images in order to obtain morphological information.

II. MATERIALS AND METHODS

A. Experimental details

Here, p-type Si wafers with (100) orientation were considered as the substrate, and Au was applied as the catalyst for Au thin film deposition. During the 15 min etching process of Au thin films by the HFCVD method, the working pressure and flow rate were 6.6 × 10⁻² Pa and 100 sccm, respectively, for H₂. The filament and substrate temperatures were 1700 °C and 550 °C, respectively. It should be noted that no common surface pre-treatment was used for increasing the nucleation density of diamond nanocrystals (DNCs).

Thereafter, nanostructured carbon was grown on the Au thin film. The ratio of CH₄/H₂ was set in the rate of 8%, while other mentioned parameters of the etching process were unchanged, except the filament and substrate temperatures which increased to 1800 °C and 600 °C, respectively. Growing DNCs were carried out for 15 min, 30 min, and 60 min at room temperature (297 ± 1 K). The details of each sample are summarized in Table I.
Here, 2.0 MeV He$^+$ ions were produced by a van de Graff accelerator to analyze the ion beam. Solid state detectors with 15 keV resolution were located at 162° for detecting the backscattered particle. The accelerator was located at Centro de Microanalisis de Materiales (CMAM). The angle between the surface and the incident ion beam was 90°, and scattered ions with a dose of 10 mC were detected at 165° by a mobile detector. The information about the atomic content and thickness of the thin films were obtained from Rutherford backscattering (RBS) analysis using SIMN RA software.

In addition, x-ray diffraction (XRD) with Cu-ka radiation ($\lambda = 1.54$ A) was applied to record the crystalline structure of DNC samples, where 2$\Theta$ was from 0° to 100°. Atomic force microscopy (AFM) (Veeco, Santa Barbara, CA) results were finally applied to provide quantitative data on surface roughness and morphology for studying the structural topography of samples. The experiments were carried out by cantilevers with the following specific properties for force–distance curve measurements: width 25 $\mu$m, length 180 $\mu$m, thickness 4 $\mu$m, quality factor Q = 100, tip radius 10 nm, Young’s modulus $E = 1.3 \times 10^{11}$ Pa, mass density $\rho = 2330$ kg/m$^3$, and Poisson’s ratio $\nu = 0.28$. The 3D surface topography of thin films was investigated through 256 × 256 pixel images recorded by a non-contact mode nanoscope multimode atomic force microscope (Digital Instruments, Santa Barbara, CA) with a scan speed of 10–20 $\mu$m/s.

![AFM images of DNC film surfaces](image-url)
B. Stereometric characterization of 3D surface microtextures

MountainsMap® premium software was used to analyze the surface properties of each DNC sample stereometrically, according to six quantitative parameters [functional, height, hybrid, functional (volume), spatial, and feature parameters] obtained through AFM images and ISO 25178-2: 2012. 3D surface roughness was estimated based on the objective parameters which are reported in Ref. 16. In the present study, fractal geometry and statistical parameters are the two main criteria for 3D surface roughness characterization.

III. RESULTS AND DISCUSSION

Figure 1 indicates Au and Si contents of gold thin films deposited on the Si substrate through RBS analyses. SIMN RA software (SIMNRA by Mayer, 1996) determined the Au film thickness as 90 nm.
In Fig. 2, the XRD patterns of the DNC films are illustrated for samples grown for 15 min, 30 min, and 60 min. A sharp Au(111) peak at 47° is observable in all samples. However, in sample nos. 4 and 5, with 30 min and 60 min growth, respectively, the existence of C(002) and C(111) peaks at 28° and 43°, respectively, demonstrates the fact that increasing the duration of DNC growth results in better crystalline quality of the samples.

3D AFM images of the surfaces of DNC films are shown in Fig. 3 for 5 × 5 μm² scanning square areas: (a) no. 1, (b) no. 2, (c) no. 3, (d) no. 4, and (e) no. 5.

Figure 4 presents the depth histograms, which helps us investigate the distribution density of the data points on the surface. The vertical and horizontal axes indicate the depths and the whole population (%), respectively. The percentage of traversed
material in relation to the covered area is called the bearing ratio curve, and it is obtained through the Abbott–Firestone curve, which is the cumulative function of amplitude distribution. Here, the horizontal axis also represents the percentage of the bearing ratio.

In addition, the peak count histogram for each sample is shown in Fig. 5.

Cartesian graphs shown in Fig. 6 also represent surface texture directions whose corresponding value for each sample is summarized in Table II.

As can be seen, the highest and lowest values of the isotropy parameter are related to sample no. 2 (82.44%) and sample no. 4 (24.43%), respectively, while other samples have values of about 70%. The maximum value of the first direction

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| Sample nos. | Isotropy (%) | First direction (deg) | Second direction (deg) | Third direction (deg) |
|-------------|--------------|-----------------------|------------------------|----------------------|
| 1           | 70.60        | 44.98                 | 135.0                  | 26.46                |
| 2           | 82.44        | 90.00                 | 0.178                  | 44.99                |
| 3           | 74.83        | 163.0                 | 135.0                  | 146.3                |
| 4           | 24.43        | 11.93                 | 128.8                  | 153.5                |
| 5           | 67.68        | 0.208                 | 153.5                  | 44.99                |
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parameter is observed in sample no. 3 (163°), whereas the minimum value of this parameter was obtained in sample no. 5 (0.208°). For the second direction, however, the maximum and minimum value were related to sample no. 5 (153°) and sample no. 2 (0.178°), respectively. Finally, the highest value of the third direction parameter is achieved in sample no. 4 (153.0°), while the lowest value was achieved in sample no. 1 (26.46°).

Moreover, Table III summarizes all surface texture parameters deduced from 5 × 5 µm² scanning square areas of AFM images, according to ISO 25178-2: 2012, whose details are reported in Ref. 16. As can be seen, the lowest and largest root mean heights (Sq) belong to the Au catalyst as sample no. 1 (1.87 nm) and sample no. 3 with (158.9 nm), respectively. Furthermore, by increasing the time of DNC growth from 15 nm to 60 min, Sq is increased,

| Statistical parameters | Symbol | No. 1 | No. 2 | No. 3 | No. 4 | No. 5 |
|------------------------|--------|-------|-------|-------|-------|-------|
| **Height Parameters**  |        |       |       |       |       |       |
| Root mean square height | Sq (nm) | 1.87  | 492.9 | 18.19 | 20.51 | 158.9 |
| Skewness               | Ssk    | 8.31  | −1.75 | −0.241| 0.084 | −7.86 |
| Kurtosis               | Sku    | 132.3 | 4.12  | 3.442 | 3.227 | 63.67 |
| Maximum peak height    | Sp (nm)| 38.76 | 324.7 | 55.81 | 77.72 | 66.78 |
| Maximum pit height     | Sv (nm)| 25.11 | 1190  | 75.64 | 73.80 | 1303 |
| Maximum height         | Sz (nm)| 63.87 | 1515  | 131.5 | 151.5 | 1370 |
| Arithmetic mean height | Sa (nm)| 0.87  | 367.2 | 14.25 | 16.17 | 40.7  |
| **Functional Parameters** |        |       |       |       |       |       |
| Areal material ratio   | Smr (%)| 100   | 83.18 | 100   | 100   | 98.51 |
| Inverse areal material ratio | Smc (nm) | 1.07 | 279.4 | 22.06 | 25.80 | 41.15 |
| Extreme peak height    | Sxp (nm)| 2.67  | 1346  | 39.06 | 41.22 | 42.63 |
| **Spatial Parameters** |        |       |       |       |       |       |
| Auto-correlation length | Sal (µm) | 0.0837 | 0.1502 | 0.2624 | 0.2442 | 0.1348 |
| **Hybrid Parameters**  |        |       |       |       |       |       |
| Root mean square gradient | Sdq | 0.0798 | 16.95 | 0.1755 | 0.2067 | 6.266 |
| Developed interfacial area ratio | Sdr (%) | 0.2937 | 378.3 | 1.498 | 2.091 | 53.50 |
| **Material Parameters** |        |       |       |       |       |       |
| Material volume        | Vm (µm³/µm²) | 0.000146 | 0.001337 | 0.000858 | 0.0011 | 0.000775 |
| Void volume            | Vv (µm³/µm²) | 0.001217 | 0.2807 | 0.02292 | 0.02689 | 0.04192 |
| Peak material volume   | Vmp (µm³/µm²) | 0.000146 | 0.001337 | 0.000858 | 0.0011 | 0.000775 |
| Core material volume   | Vmc (µm³/µm²) | 0.000805 | 0.05801 | 0.01606 | 0.01821 | 0.0158 |
| Core void volume       | Vvc (µm³/µm²) | 0.001041 | 0.07176 | 0.02062 | 0.0246 | 0.0208 |
| Pit void volume        | Vvv (µm³/µm²) | 0.00175 | 0.2090 | 0.002295 | 0.002298 | 0.0212 |
| **Feature Parameters** |        |       |       |       |       |       |
| Density of peaks       | Spd (1/µm²) | 1.492  | 0.1209 | 3.104 | 3.910 | 0.0403 |
| Arithmetic mean peak curvature | Spc (1/µm) | 9.839  | 5.308 | 2.428 | 4.017 | 6.427 |
| Ten-point height       | S10x (nm) | 50.26  | 1312  | 118.4 | 127.1 | 1214 |
| Five-point peak height | S5p (nm) | 32.12  | 287.2 | 52.18 | 66.57 | 58.32 |
| Five-point pit height  | S5v (nm) | 18.14  | 1185  | 66.22 | 60.51 | 1301 |
| Mean dales area        | Sda (µm²) | 0.5398 | 0.09417 | 0.5640 | 0.3439 | 0.0304 |
| Mean hills area        | Sha (µm²) | 0.3699 | 0.05836 | 0.3360 | 0.2747 | 0.06124 |
| Mean dales volume      | Sdv (µm³) | 4.27 × 10⁻⁵ | 0.08648 | 0.001101 | 4.54 × 10⁻⁴ | 0.03145 |
| Mean hills volume      | Shv (µm³) | 1.39 × 10⁻⁴ | 5.39 × 10⁻⁴ | 0.001009 | 9.32 × 10⁻⁴ | 6.24 × 10⁻⁴ |
| **Functional parameters** (Stratified surfaces) | | | | | | |
| Core roughness depth   | Sk (nm) | 2.15   | 186.5 | 44.0  | 50.86 | 43.14 |
| Reduced peak height    | Spk (nm) | 2.91   | 15.3  | 17.09 | 22.09 | 15.69 |
| Reduced valley depth   | Sv1 (nm) | 1.90   | 2404  | 21.56 | 20.29 | 331.8 |
| Peak material portion  | Smr1 (%) | 10.03  | 2.583 | 9.294 | 10.27 | 10.11 |
| Peak valley portion    | Smr2 (%) | 86.77  | 83.17 | 88.30 | 89.61 | 87.94 |
TABLE IV. The fractal dimensions ($D_f$) for analyzed samples. The average results were expressed as standard deviation and mean value. Statistical difference: $P < 0.05$.

| Fractal dimension | No. 1   | No. 2   | No. 3   | No. 4   | No. 5   |
|-------------------|---------|---------|---------|---------|---------|
| $D_f$             | 2.43 ± 0.01 | 2.34 ± 0.01 | 2.17 ± 0.01 | 2.65 ± 0.01 | 2.28 ± 0.01 |

FIG. 7. The average PSD of surface textures for (a) no. 1, (b) no. 2, (c) no. 3, (d) no. 4, and (e) no. 5.
TABLE V. The average PSD parameters for the analyzed samples.

| Average PSD parameters | No. 1 | No. 2 | No. 3 | No. 4 | No. 5 |
|------------------------|------|------|------|------|------|
| Wavelength (μm)        | 0.8813 | 0.8813 | 0.8813 | 0.8813 | 0.8813 |
| Amplitude (nm)         | 0.0244 | 7.516 | 1.063 | 1.163 | 2.849 |
| Dominant wavelength (μm) | 0.2679 | 0.0352 | 0.8531 | 1.382 | 0.0705 |
| Maximum amplitude (nm) | 0.5015 | 1195 | 3.746 | 7.403 | 205.1 |

TABLE VI. The furrow parameters of samples.

| Parameters of furrows | No. 1 | No. 2 | No. 3 | No. 4 | No. 5 |
|----------------------|------|------|------|------|------|
| Maximum depth (nm)   | 6.209 | 1539 | 81.01 | 71.96 | 1354 |
| Mean depth (nm)      | 1.623 | 483.1 | 30.48 | 33.55 | 123.6 |
| Mean density (cm/cm²) | 52 012 | 37 427 | 31 369 | 34 316 | 34 821 |

FIG. 8. The furrows of samples for: (a) no. 1, (b) no. 2, (c) no. 3, (d) no. 4, and (e) no. 5.
which indicates an increase in root mean square. The negative value of surface skewness (Ssk) in sample nos. 2, 3, and 5 confirms the dominance of pits on their surfaces, while its positive value in sample nos. 1 and 4 confirms peak dominance.

Surface kurtosis (Sku) is another parameter whose value for all samples is above three and points to the existence of high peaks or valleys on the surface. The maximum and minimum values of Sku are observed in sample nos. 1 (132.3) and 4 (3.227), respectively. In addition, Stv and Stp are the maximum valley depth and maximum peak height, with the maximum value in sample no. 5 (1303 nm) and sample no. 2 (324.7 nm), respectively, and their variation is not non-monotonic. Meanwhile, Stz is defined as the sum of the maximum pit height and the maximum peak height, with the greatest value in sample no. 2. In addition, it can be seen that by increasing the time of DNG growth, Ssz has increased. The same routine is observable for the arithmetic mean height (Sa), which is the mean of the vertical deviations from the mean surface, and also for Stoz, whose values are increased by increasing the time of DNG growth. The maximum value of Stoz is obtained in sample no. 2 (1312 nm).

Here, fractal dimensions were calculated by the enclosing boxes method with coefficients of correlation (R²) and are summarized in Table IV. As can be seen, the values of R² for all linear fits were 0.998 ± 0.001, which confirm the excellent data fit by linear functions.

On the other hand, the average power spectral density (PSD) of surface textures is shown in Fig. 7, and its values are summarized in Table V.

The furrows of samples (including mean depth of furrows and mean density of furrows along with the maximum depth of furrows specified in Table VI) are shown in Fig. 8.

IV. CONCLUSIONS

We mainly focused on the synthesis of DNG thin films on a Si substrate in the present approach and then used AFM and multifractal analysis to investigate their morphological features. In this aim, an Au catalyst was applied, and DNG growth was carried out by the HFCVD method for different durations: 15 min, 30 min, and 60 min.

The 3D surface microtexture characteristics of DNG thin films were quantitatively investigated by the Abbott–Firestone curve and fractal geometry along with other stereometric analyses such as furrows and average PSD. According to these parameters, microstructures and surface texture will be investigated with high precision and easy implementation. According to the results of the present study, a new insight into diamond nanocrystallines can be achieved in the field of thin film morphology. Studying these statistical parameters demonstrated that among the prepared specimens, sample no. 2, which was the Au etched thin film produced by the HFCVD method, was the most isotropic sample with the maximum surface roughness (492.9 nm). Moreover, by increasing the DNG growth duration, surface roughness was increased, and the surface became irregular. In addition, the most regular topography (Df = 2.17 ± 0.01) was found in sample no. 3, while the most irregular topography (Df = 2.65 ± 0.01) was found in sample no. 4.

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