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Low temperature and large area deposition of nanocrystalline diamond films with distributed antenna array microwave-plasma reactor

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ABSTRACT

Diamond films grown at low temperature (<400 °C) on large area of different substrates can open new applications based on the thermal, electrical and mechanical properties of diamond. In this paper, we present a new distributed antenna array PECVD system, with 16 microwave plasma sources arranged in a 2D matrix, which enables the growth of 4-inch nanocrystalline diamond films (NCD) at substrate temperature in the range of 300–500 °C. The effect of substrate temperature, gas pressure and CH_4 concentration in the total gas mixture of $H_2/CH_4/CO_2$ on the morphology and growth rate of the NCD films is reported. The total gas pressure is found to be a critical deposition parameter for which growth rates and crystalline quality both increasing with decreasing the pressure. Under optimized conditions, the process enables deposition of uniform (~10%) and high purity NCD films with very low surface roughness (5–10 nm), grain size of 10 to 20 nm at growth rates close to 40 nm/h. Nanotribology tests result in the friction coefficient of the NCD films close to that obtained for the standard tetrahedral amorphous carbon coatings (ta-C) indicating the suitability of this low-temperature diamond coating for mechanical applications such as bearing or micro-tools.

1. Introduction

Nanocrystalline diamond (NCD) films are outstanding material candidates that have attracted strong scientific and technological interests for optical, electronic, biomedical and tribological applications [1,2]. In fact, NCD films not only retain most of the extreme properties of microcrystalline diamond (MCD) but also exhibit a very low surface roughness due to their ultrafine (<20 nm) grains [3]. For instance, NCD coatings are highly desirable for the cutting tool industry [3,4] because smaller grain size enhances coating toughness and increases surface smoothness [3]. A smoother surface can facilitate chip evacuation, and thus significantly reduce cutting forces [4]. Recently, due to diamond's superior bulk thermal conductivity, NCD coatings on AlN or GaN [5,6] are being seriously considered as heat sinks on high-frequency and high-power devices where efficient heat removal becomes vital for maintaining the performance and reliability of these devices. However, low diamond adhesion to substrates and substrate damage during chemical vapor deposition (CVD) at high temperature are the limiting factors for its wider commercial use [7,8]. One of these limitations is in the typical CVD system conditions used for diamond growth such as hot filament or resonant-cavity microwave systems. As a matter of fact, these systems generally operate at temperatures as high as 800 °C in a harsh plasma environment. In addition, scaling up to larger deposition areas is particularly difficult and costly for microwave systems while hot-filament reactors suffer from contamination problems.

Therefore, there is an increasing demand for deposition systems that allow growing high purity NCD at lower temperatures and on larger areas. This demand leads to an increased interest and research effort in surface wave plasma systems for diamond synthesis that are more adapted to these requirements [9–12].

In this work, we present a new Distributed Antenna Array (DAA) CVD reactor for depositing NCD films at temperatures in the range of 300–500 °C by using $H_2/CH_4/CO_2$ gas chemistry. This reactor is based on 16 microwave coaxial plasma sources arranged in a 2D matrix. The reactor does not have physical limitations since the number of elementary sources can be increased allowing an easy up-scaling to grow large area diamond films. Moreover, lower pressures than in conventional diamond CVD processes have to be used allowing limiting plasma heating of the substrate, and thereby maintaining low substrate temperatures. However, much lower deposition rates are obtained.

In the DAA reactor, the influence of substrate temperature, total pressure and CH_4 concentration on the morphology, structure, uniformity, growth rate and friction coefficient of NCD films grown on Silicon was investigated using techniques such as scanning electron microscopy (SEM), atomic force microscopy (AFM), transmission electron microscopy (TEM), Raman spectroscopy, thin film reflectometry and rotational mode of a ball-on-disk nanotribometry.

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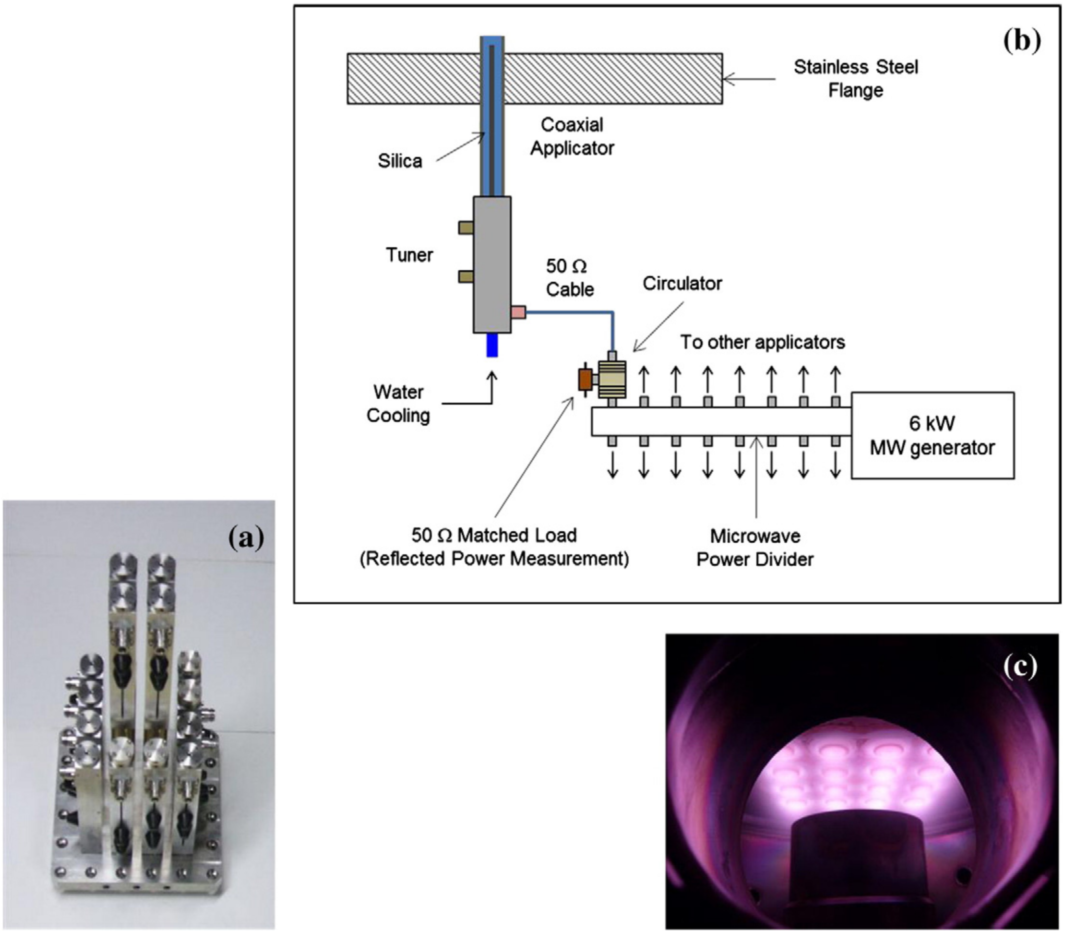

Fig. 1. (a) 16 coaxial plasma sources inserted in a square metallic flange arranged in a 2D-matrix to constitute the distributed antenna array (DAA), (b) Design of a coaxial plasma source, (c) View of the ignited plasma sources inside the chamber.

2. Experimental details

The DAA reactor consists of 16 coaxial plasma sources inserted in a square metallic flange and arranged in a 4 × 4 matrix (Fig. 1a) fed by a 6 kW microwave generator. Initially, this technology has been developed for ECR plasma [13] (i.e. the source extremity is equipped with a permanent magnet) but in our case, taking into account the working pressure close to 1 mbar, plasma sources are free from magnetic field. Microwave power is coupled to the system using antennas appropriately introduced into a rectangular waveguide. The microwave energy is then transported to the sources inside the chamber, thanks to coaxial cables (Fig. 1b). This arrangement leads to dividing power between the 16 sources (i.e. around 180 W/source). Discharge is then ignited around each source inside the chamber of the low-pressure reactor (Fig. 1c), and the plasma diffuses to the substrate. When the microwave power is increased, the localized plasmas expand and then meet together to produce a sheet of uniform plasma. Taking into account the distance between each elementary source, the substrate holder must be localized at least at 40 mm from the plane sources. Due to this configuration, even conductive substrates with sharp edges can be uniformly coated without disturbing the electric field. However, due to the low pressure operating regime, low plasma density and low molecular hydrogen concentration as compared to conventional high-power high-pressure CVD reactor are obtained. In order to compensate for the low etching of non-diamond phases due to the lack of atomic hydrogen, CO_2 is added to the gas mixture, as reported by other studies [9–12]. It is important to note here that reactors working at low pressure and low temperature have already been presented in the literature [9,11] but these studies are mainly based on surface wave plasma reactor which are well known to have a plasma density limited by the critical plasma density (7.5×10^{10} electrons·cm^{-3} at 2.45 GHz). Moreover, in such a kind of reactor, microwave applicators present large silica surfaces which, due to the use of hydrogen plasma, can be etched and lead to film contamination. In the case of DAA reactor, thanks to the small size and optimized design of each elementary source (coaxial applicator), the ignited plasma has been demonstrated to reach density up to 10^{12} electrons·cm^{-3} [14] which will lead, for similar microwave power, to a more efficient chemical species production. The reduced size of sources also allows limiting silica surfaces in the reactor and then contamination of grown layers. The last point that can be highlighted here is that there is no limitation to upscale the reactor by increasing the number of sources and a 3D distribution of the sources can be imagined in order to allow diamond deposition on complex shapes.

Prior to seeding, polished 2 inch Si wafers with thickness of around 250 μm (*p*-type, 1–3 Ω·cm, semiconductor grade) are ultrasonically cleaned in acetone, then etched by HF acid (20 vol.%) to remove the

Table 1
Sample ID and the corresponding growth conditions.

Samples[a]	A	B	C	D	E	F
Pressure (mbar)	0.5	0.35	0.5	0.5	0.5	0.5
Substrate temperature (°C)	400	400	400	400	300	500
[CH_4] (%)	2.5	2.5	1	5	2.5	2.5
[H_2] (%)	96.5	96.5	98	94	96.5	96.5
Film thickness (nm)	207	189	218	200	196	202
Deposition time (min)	417	281	477	360	440	332

[a] All samples were grown at a microwave power of 3 kW, 1% of CO_2 and total gas flow of 50 sccm.

natural SiO_2 layer and finally thoroughly rinsed in ultra-pure water. The substrates are then seeded by immersing in a bath containing nanoparticles of diamond suspension (average grain size ~ 5 nm). This seeding usually yields in a very high density of nuclei (~10^{12} nuclei/cm^2) [15]. Substrates are then placed on a molybdenum holder equipped with a graphite heater, and the substrate temperature is monitored by a thermocouple embedded in the holder.

In this study, all samples were grown at a microwave power of 3 kW, 1% of CO_2 and a total gas flow of 50 sccm. During NCD deposition the following process parameters were varied: substrate temperature (300–500 °C), total pressure (0.35–0.5 mbar) and CH_4 concentration (1–5%). Due to the thin thickness of the NCD films, an accurate in situ measurement was obtained by a single point one color-laser interferometer. The morphology of the films was investigated by top-view

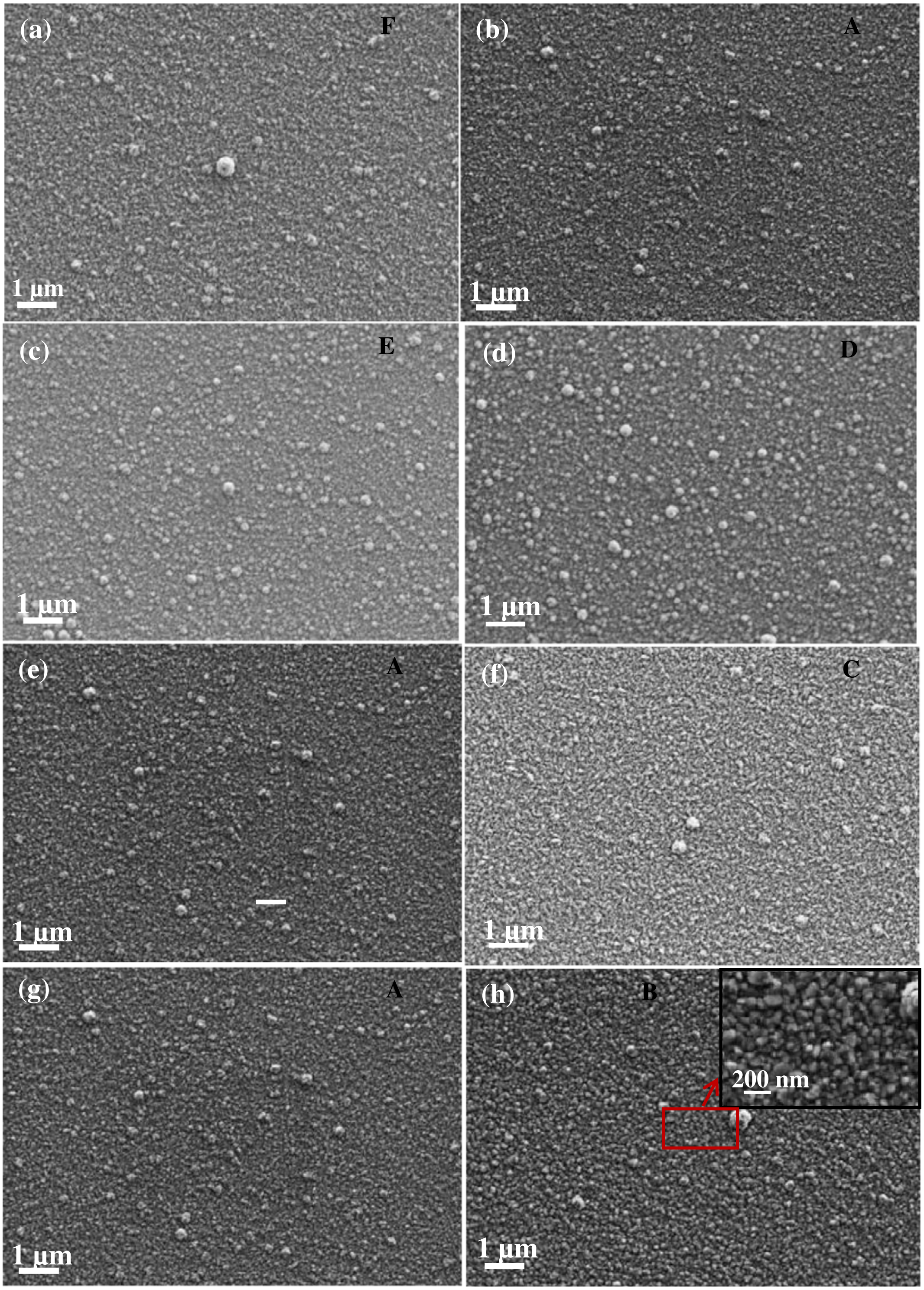

Fig. 2. Top-view SEM pictures of the NCD films showing the effect of (a–c) substrate temperature, (d–f) CH_4 concentration and (g–h) total gas pressure on the film morphology. Substrate temperatures for the NCD films shown in panels a, b, and c are 500, 400 and 300 °C respectively (corresponding samples: F, A and E). Similarly, CH_4 concentrations in panels d, e, and f are 5%, 2.5% and 1% respectively (samples: D, A and C). And total gas pressures in panels g and h are 0.5 and 0.35 mbar respectively (samples: A and B). The inset in panel h is the magnified image of the corresponding area.

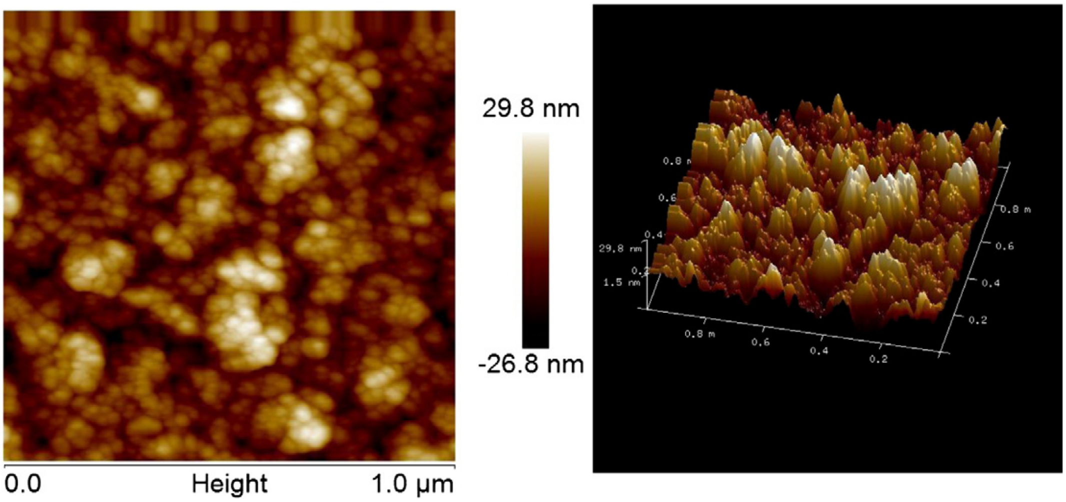

Fig. 3. Typical AFM images of the NCD film with thickness of around 400 nm deposited on Si substrates using the following growth conditions: 400 °C substrate temperature, 0.35 mbar pressure and 2.5% CH_4 in the gas mixture (sample B).

SEM images taken by a field emission *ZEISS ULTRA plus* SEM system. AFM was applied to get further information on the morphology and the surface roughness. These measurements were carried out with a *VEECO Dimension 3100* AFM system in tapping mode in air. HRTEM (200 kV, JEM-2100 F, JOEL) was used to get more insight into the film structure. The Raman spectra were obtained with a *HR800* (HORIBA Jobin-Yvon) working in a confocal mode and in the back-scattering configuration, using the 488 nm line of an Ar + laser (Coherent Innova 300) as an excitation source with a power of 10 mW. Film thickness and thickness uniformity was also measured ex-situ using a UV–visible reflectometer (NanoCalc, Ocean Optics).

Rotational mode of a ball-on-disk nanotribometer (NTR2, CSM Instruments, Switzerland), was used to carry out the tribological tests of the NCD films at room temperature and 60% humidity. A Sapphire ball with a diameter of 2 mm was used to slide against the diamond film. The sliding speed of the specimen against the ball and normal load were kept constant at 0.5 $cm \cdot s^{-1}$ and 10 mN, respectively. The total sliding distance for each measurement was 6.5 m. The wear rate of the ball, W, was determined according to the formula, $W = D/LV$, where V is the worn volume calculated from the diameter of the worn area and measured with an optical microscope, D is the sliding distance and L is the normal load.

3. Results

3.1. Evolution of microstructure and growth rate as a function of growth parameters

6 NCD films were deposited on Si substrates with the growth conditions listed in Table 1. The following growth parameters were varied in order to investigate their influence on the morphology, structure and growth rate of the films: CH_4 concentration in a mixture of H_2 and CO_2, substrate temperature during deposition and total gas pressure. All the films show strong adhesion to the Si substrates as no crack and delamination were observed in as-grown films even after a long period of time.

The top-view SEM pictures of the films presented in Fig. 2a–e show almost no discernable differences of the morphology for a range of temperature from 500 down to 300 °C (Fig. 2a–c) and CH_4 concentration from 5 to 2.5% (Fig. 2d − e) when pressure is kept at 0.5 mbar. Surprisingly, at 1% of CH_4, the grain shape changes from rounded to elongated shape (Fig. 2f). Nevertheless, whatever the temperature and CH_4 concentration, all films appear homogeneous, continuous and present small diamond nanocrystallites with no clear facets. The root–mean–square (rms) roughness over a 50 × 50 μm^2 area measured by AFM in

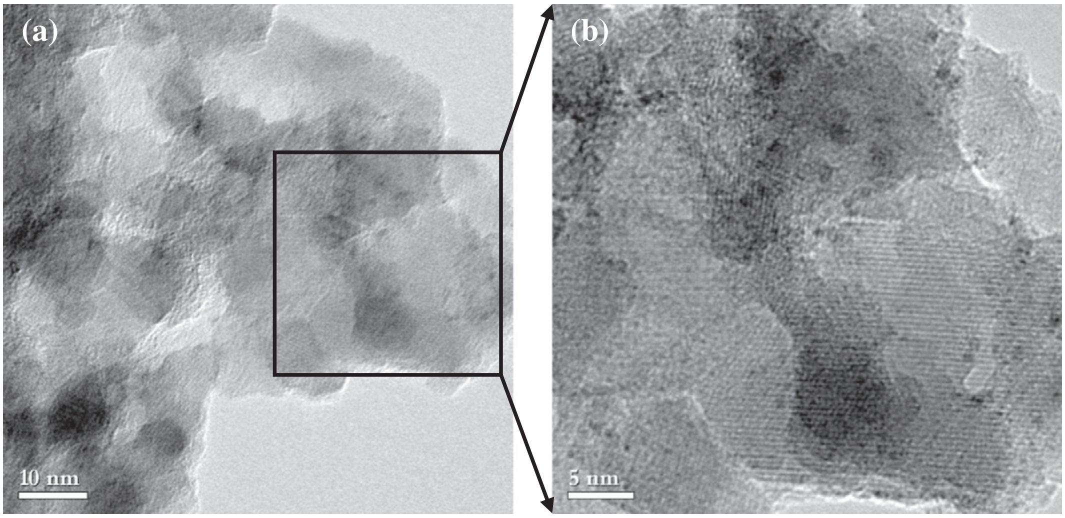

Fig. 4. (a) Plane view TEM image and (b) magnified view of the corresponding area of the sample B.

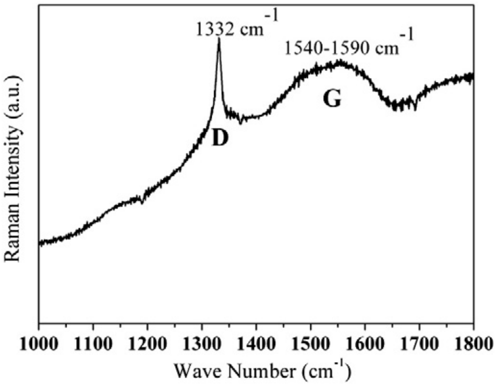

Fig. 5. Raman spectrum of the sample B recorded at an excitation wavelength of 488 nm.

several local areas of the NCD films is very similar from a sample to another and ranges from 5 to 10 nm (Fig. 3). To the contrary of variations in CH_4 concentration (in the range of 2.5–5%) and temperature for which no clear change in morphology was observed, a reduction of the total pressure from 0.5 to 0.35 mbar led to the appearance of small facets (Fig. 2g and h).

In the current investigation, it was found that the surface roughness does not depend on the film thickness (in the range of 100 nm–1 μm). This constitutes a characteristic of a NCD film for which continuous secondary nucleation occurs rather than columnar growth for microcrystalline diamond. This characteristic has been emphasized by TEM characterization. As is shown in Fig. 4, nanograins with a size between 10 and 20 nm can be clearly identified revealing that grown films are constituted of nanocrystalline grains.

Raman analysis confirms their NCD nature: the fundamental diamond line at 1332 cm^{-1} and the graphite-related G band at 1540–1590 cm^{-1} (Fig. 5) can be clearly observed. Low intensity of the G band in the spectrum of the NCD films ($I_D/I_G \approx 1.04$; Fig. 5) indicates the good crystalline quality of these films.

Fig. 6 shows the variation of the average full-width half-maximum (FWHM) of the first-order Raman line (peak position 1332 cm^{-1}) with respect to the substrate temperature, gas pressure and CH_4 concentration. The FWHM gets narrower with decreasing CH_4 concentration from 5 to 1% (Fig. 6a) and the total gas pressure from 0.5 to 0.35 mbar (Fig. 6b) suggesting the improvement in the crystalline quality of the NCD films with the reduction in CH_4 concentration and pressure. The opposite tendency is obtained with substrate temperature (Fig. 6c).

The growth rate as a function of growth parameters is presented in Fig. 7. It increases both with increasing the CH_4 concentration and substrate temperature (Fig. 7a and b). This tendency is usually observed for conventional CVD diamond growth. Unlike conventional CVD, the growth rate could be increased from 25 nm/h to 40 nm/h by reducing the total pressure from 0.5 to 0.35 mbar (Fig. 7c).

All characterizations performed in this study clearly show that the growth conditions used in DAA reactor are very close to those used in surface wave reactors since the evolution of morphologies and growth rates are very similar [15–17].

3.2. Mechanical behavior: nanotribology

Rotational mode of a ball-on-disk nanotribometer was used to carry out the tribological tests of the NCD films at room temperature and 60% humidity. The measured residual stress and friction coefficient of the films were compared with that of standard NCD and ta-C coatings

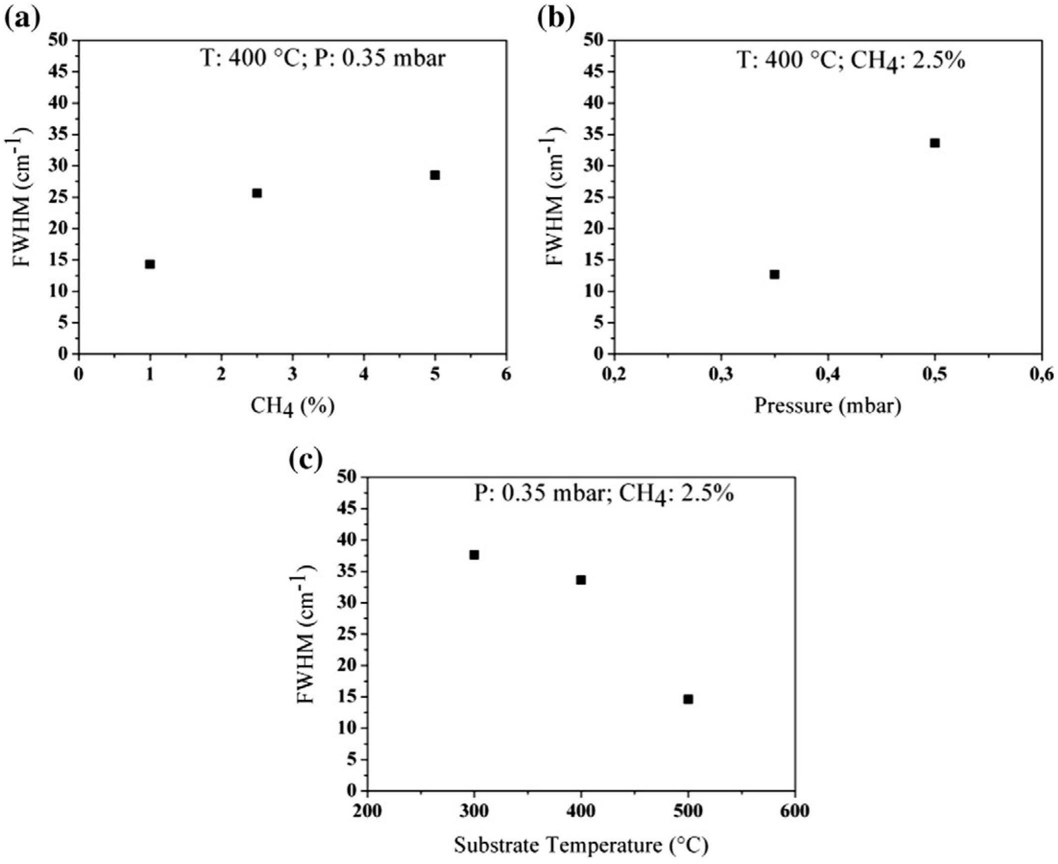

Fig. 6. The variation of avg. FWHM of the first-order Raman line with (a) CH_4 concentration from 1 to 5%, (b) total gas pressure of 0.35 and 0.5 mbar and (c) substrate temperature from 300 to 500 °C used in the deposition of the NCD films.

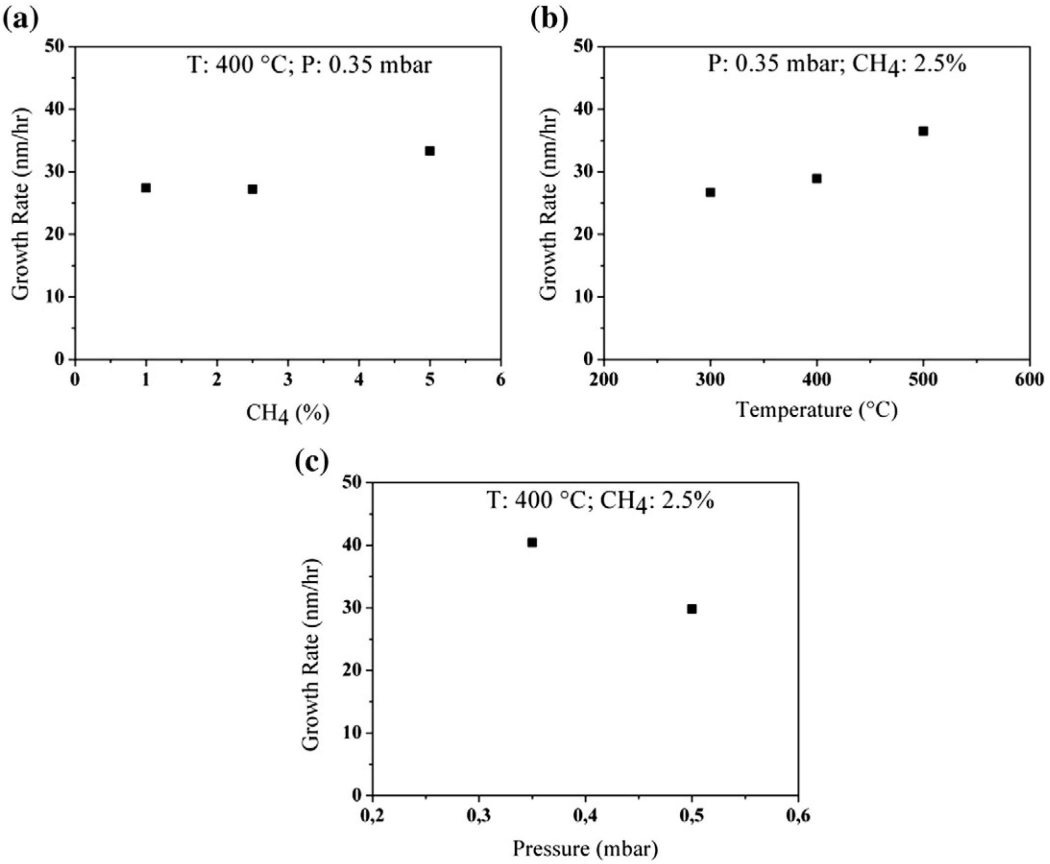

Fig. 7. Avg. growth rate variation of the NCD films with (a) CH_4 concentration from 1 to 5%, (b) substrate temperature from 300 to 500 °C and (c) total gas pressure of 0.35 and 0.5 mbar.

deposited by HFCVD and Arc-Filtered PVD techniques respectively (these samples were prepared at NeoCoat SA).

The total residual stress, σ_{tot}, on silicon has been determined by the following Stoney equation:

$$\sigma_{tot} = E_s^{'} \cdot \frac{t_s^2}{6t_f} \cdot \left(\frac{1}{R} - \frac{1}{R_0}\right) \tag{1}$$

where E_s' is the biaxial Young's modulus of the substrate (180 GPa for silicon), t_f and $t^2{}_s$ are the film and substrate thickness (~275 μm). R_0 and R is the initial and final radius of curvature of the substrate respectively measured by a Taylor–Hobson stylus-type profilometer (a 50 MPa resolution was estimated from the curvature measurement).

Residual stresses of diamond films are composed of thermal mismatch stresses and intrinsic tensile film stresses induced during film growth. The residual stress of our NCD films was found to be compressive, and it varied from −800 to −1000 MPa. The deposition parameters (Table 1) did not show any significant influence on stress level. We calculated the thermal contribution of residual stress that was around −550 MPa at 400 °C. The intrinsic stress was also found to be compressive with a value of −300 MPa.

The friction coefficient was measured as a function of the sliding distance for different NCD and ta-C coatings at a constant applied load of 10 mN and is listed in Table 2. The friction coefficient seems to principally depend on the surface roughness, and the smoother is the NCD coatings the lower is the friction coefficient and wear rate, which is a typical behavior of polycrystalline hard coatings [3].

4. Discussion

We have investigated the effect of substrate temperature, CH_4 concentration and total pressure on the morphology, structure and growth rate of the NCD films in a new DAA microwave plasma CVD reactor. The crystalline quality of the NCD films is measured qualitatively from the FWHM of the first-order Raman line. In fact, FWHM is often taken as a measure of the 'perfection' of the diamond crystallite, and the narrower is the linewidth, the better is the crystallinity [18]. Theoretical FWHM of the line in perfect single crystal diamond is 1.5 cm^{-1} while for polycrystalline diamond films it varies from 5 to 10 cm^{-1} [18]. In disordered synthesized diamonds the width of the line can even exceed 14 cm^{-1} [18]. The process parameter study performed here shows that the crystallinity is improved with decreasing CH_4 concentration in the feed gas. Similar tendency is commonly observed in conventional CVD growth and is attributed to an increase of non-diamond phases at higher methane concentration, this phenomenon being more sensitive at low microwave power density. The slight increase in crystalline quality with increasing growth temperature can be attributed to an increase in etching efficiency of non-diamond phases.

Similar to conventional CVD growth, we have also observed a reduction in the growth rate with decreasing substrate temperature while a linear increase is observed with increasing CH_4 concentration. It is well known that diamond growth is a complex process including several temperature-dependent surface reactions (adsorption and/or desorption processes, surface diffusion and migration, etc.) [19]. Therefore, a reduced substrate temperature usually results in substantially lower growth rates. At constant temperature and pressure, the number of carbon radicals arriving at the growth surface depends mainly on the methane concentration in the source gas and, as a consequence, the growth rate may increase with carbon concentration.

The total pressure seems to be a critical process parameter that strongly influences both the growth rate and crystallinity. However, the pressure effect on the morphology and growth rate disagrees with what is commonly observed in conventional CVD diamond deposition systems. Indeed, it is now well accepted that an improvement both in

Table 2
Friction coefficient and ball wear rate of different NCD and amorphous carbon coatings.

Sample	Type of film	Roughness (nm)	Friction coefficient	Ball wear rate (10^{-14} $m^3 \cdot N^{-1} \cdot m^{-1}$)
B	NCD in DAA reactor	10	0.32	4
D	NCD in DAA reactor	5	0.12	0.3
LD1298	NCD in HFCVD reactor	20	0.22	20
ARG300	Filtered-Arc ta-C	<5	0.10	3

growth rate and diamond quality is obtained by increasing the microwave power density coupled to the plasma through an increase of pressure [20]. Nevertheless, this new deposition system is different from conventional resonant-cavity reactors since the chemical species are created close to the 16 plasma sources and then diffused toward the substrate. The improvement of both the crystallinity and the growth rate with the pressure reduction from 0.5 mbar to 0.35 mbar could be explained in the following ways:

(1) a decrease of the pressure leads to an increase of H_2 dissociation by electronic impact that increases as pressure decreases; (2) a decrease of the pressure leads to an increase of the mean free paths (λ) of active species (CH_3, atomic hydrogen, etc.). For instance at 0.13 mbar λ is 500 times than at 60 mbar [21,22]. As the active species are generated at or near the plasma source, a lowering of pressure might facilitate species transport to the surface. Further works based on plasma spectroscopy and plasma modeling must be carried out to better understand this new CVD system.

Finally, in order to quantify more accurately the thickness homogeneity of NCD material using DAA reactor, a 100 nm thick film has been grown on a 4 inch silicon wafer following the same seeding procedure. The evolution of the film thickness along the diameter is presented in Fig. 8. The film shows a variation in thickness lower than 10%, validating the homogeneity of this process for NCD deposition.

5. Conclusion

We have demonstrated the deposition of uniform NCD films with very low surface roughness (5–10 nm) and grain size (10–20 nm) at substrate temperature as low as 300 °C and pressure lower than 1 mbar by using a new DAA CVD reactor. The main feature of this reactor is that the plasma sources are far from the substrate holder and distributed in a 2D-matrix allowing an easy up-scaling by increasing the number of sources. The deposited NCD films exhibit relatively good crystallinity (high sp^3 content). The thickness uniformity of a NCD film deposited over a 4 inch substrate has been found to be about 10%. The effect of growth parameters on growth rate and film quality has been studied. Increasing temperature and decreasing CH_4 content are found to lead to an improvement in both growth rate and crystallinity. Surprisingly, higher quality films and higher growth rate are achieved with decreasing the total pressure, whereas the opposite trend is usually observed in resonant-cavity reactor. This might be related to a more efficient transport of atomic hydrogen to the growth surface. In addition, the films have been characterized from a tribological point of view, and a friction coefficient close to that obtained for standard ta-C coating (~0.12) has been obtained. This low-temperature diamond coating could therefore be used, in the first instance, in mechanical applications such as bearings or micro-tools.

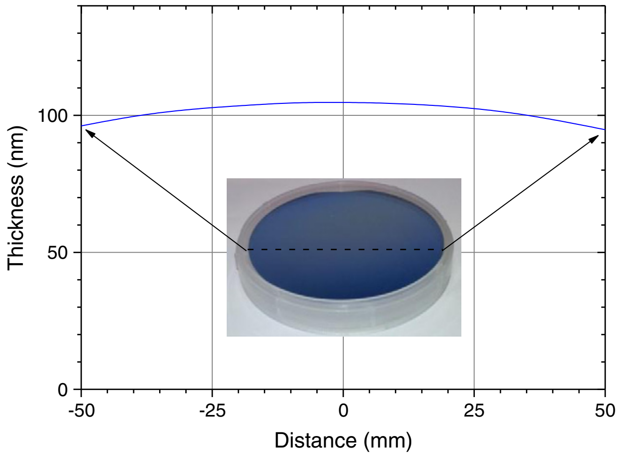

Fig. 8. Thickness variation along the diameter of a NCD film deposited on a 4 inch Si wafer at a substrate temperature of 400 °C, total pressure of 0.35 mbar and CH_4 concentration of 2.5%.

Prime novelty statement

In this paper, we present a new distributed antenna array PECVD system, with 16 microwave plasma sources arranged in a 2D matrix, which enables the growth of 4-inch nanocrystalline diamond films (NCD) with good crystallinity and uniformity (~10%) at substrate temperature in the range of 300–500 °C and pressures < 1 mbar. The reactor does not have physical limitations since the number of elementary sources can be increased allowing an easy up-scaling to grow large area diamond films.

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