The effect of substrate temperature on the morphology of diamond films grown under acetylene-lean and acetylene-rich conditions

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Abstract

The effect of substrate temperature on the morphology of diamond films grown by chemical vapor deposition is examined. Under some conditions increasing substrate temperature causes the film morphology to shift from {100} toward {111} faceting, whereas under other conditions exactly the opposite trend is observed. It is found that the factor that differentiates these two regimes is the abundance of acetylene relative to C radicals (mainly CH and in some cases C) at the growth surface.

Keywords: Diamond films; Acetylene-lean and acetylene-rich conditions; Thermal plasma

1. Introduction

For polycrystalline diamond films grown by chemical vapor deposition (CVD) it is commonly observed that under some growth conditions {100} crystal facets are predominant, while under other conditions {111} facets predominate, and that these trends are reproducible for a given reactor and flow configuration. For systems using methane and hydrogen as the diamond growth precursors, and within the window of conditions where well-faceted, continuous diamond films are obtained, increasing the input CH₄:H₂ ratio at fixed substrate temperature has always been reported—without exception, to our knowledge—to favor the appearance of {100} facets, while lower CH₄:H₂ input ratios favor the appearance of {111} facets. There have also been numerous reports in the literature regarding the effects on film morphology of substrate temperature. Here, however, the reported trends are seemingly inconsistent. Higher substrate temperatures have been reported to cause film morphology to shift from {100} toward {111} faceting in diamond grown by microwave plasma [1–4], hot filament CVD [5] and DC glow discharge [6]; while just the opposite—i.e. increasing substrate temperature promotes a shift from {111} toward {100} faceting—has been reported for films grown using oxy-acetylene flame [7–9], DC arc discharge [10,11] and RF thermal plasma [12,13].

Once a growing diamond film is sufficiently thick, relative to the initial nucleation layer, its further growth is affected, presumably, only by the local chemical environment (i.e. concentrations at the surface of various chemical species) and temperature. We hypothesize, therefore, that it should be possible to relate the morphology trends discussed above to the concentrations at the growth surface of key chemical species, and to substrate temperature.

In previous work [13] we reported experiments in which diamond films were grown using an atmospheric-pressure inductively-coupled RF plasma. In addition to observing film morphology trends vs. operating parameters, the gas composition at the growth surface was characterized by sampling gas through a small orifice in the film substrate and analyzing the sample with a gas chromatograph (GC). We hypothesized in Ref. [13] that a possible explanation for the effect of substrate tem-
perature on film morphology was the temperature dependence of surface-catalyzed H-atom recombination, which in turn could affect the concentrations of other species in the near-substrate region. This effect could be stronger in systems characterized by higher H-atom concentrations, which tend to be the higher growth-rate thermal plasma and combustion CVD systems.

Subsequently [14], we reported detailed kinetic modeling for conditions of the experiments in Ref. [13]. While the GC is capable of measuring the concentrations of stable species only, the model predicted the concentrations of both stable and radical species. The model predictions of stable hydrocarbons (CH, C₂H₂, C₂H₄ and C₃H₆) were found to be in quite satisfactory quantitative agreement with the GC measurements after account was taken of the perturbation due to the sampling orifice [15] and of chemical reactions that can occur in the GC sampling line. When the model predictions were compared to typical species concentrations from the literature for several other diamond CVD environments, it was found that the atmospheric-pressure RF plasma of Ref. [13] produced much higher concentration ratios at the growth surface of both H atoms and acetylene, relative to methyl radicals, than do these other systems. We thus hypothesized that C₂H₂ could play a much more important role in diamond growth in thermal plasmas than in most other diamond CVD systems.

Here we present results of two new sets of diamond CVD experiments, that again used an RF thermal plasma, but with a completely different plasma torch and reactor than in Ref. [13]. The RF generator plate power was 15 kW in the 200-Torr experiments and 20 kW in the 700-Torr experiments. The distance from the exit of the plasma torch to the substrate was 55 mm in the 200-Torr experiments and 17 mm in the 700-Torr experiments. In both sets of experiments argon was introduced into the torch as a swirling sheath gas at a flowrate of 30 slm, but otherwise the flow configurations differed. In the 200-Torr experiments, hydrogen, at 8 slm, and CH₄, at 80–320 sccm (CH₄:H₂ = 1–4%), were injected together with the main argon into the sheath flow, and an additional 15 slm of argon was coaxially injected through a ceramic tube located in the center of the torch head. In the 700-Torr

<table>
<thead>
<tr>
<th>Parameter</th>
<th>200-Torr experiments</th>
<th>700-Torr experiments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plate power</td>
<td>15 kW</td>
<td>20 kW</td>
</tr>
<tr>
<td>Sheath gas</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ar</td>
<td>30 slm</td>
<td>30 slm</td>
</tr>
<tr>
<td>H₂</td>
<td>8 slm</td>
<td></td>
</tr>
<tr>
<td>CH₄</td>
<td>80–640 sccm</td>
<td></td>
</tr>
<tr>
<td>Central gas</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ar</td>
<td>15 slm</td>
<td></td>
</tr>
<tr>
<td>Injection probe</td>
<td></td>
<td></td>
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<tr>
<td>Ar</td>
<td>5 slm</td>
<td></td>
</tr>
<tr>
<td>H₂</td>
<td>5 slm</td>
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<tr>
<td>CH₄</td>
<td>37.5 sccm</td>
<td></td>
</tr>
<tr>
<td>Torch-substrate distance</td>
<td>55 mm</td>
<td>17 mm</td>
</tr>
</tbody>
</table>

Table 1
Operating conditions for the 200-Torr and 700-Torr experiments

The RF plasma for these experiments was generated by a modified Tekna PL-35 induction plasma torch, powered by an RF generator operating at 3.2 MHz. The plasma exited the torch into a large deposition chamber, into which the film growth substrate was inserted, coaxial and normal to the plasma flow. For the two sets of experiments the chamber pressure was either 200 Torr or 700 Torr. For simplicity we will refer to these two sets of experiments as the ‘200-Torr’ and ‘700-Torr’ experiments, and to our previously reported experiments using a different reactor [13] as the ‘1-atm’ experiments.

Table 1 summarizes the operating conditions and configurations for the two new sets of experiments. The RF generator plate power was 15 kW in the 200-Torr experiments and 20 kW in the 700-Torr experiments. The distance from the exit of the plasma torch to the substrate was 55 mm in the 200-Torr experiments and 17 mm in the 700-Torr experiments. In both sets of experiments argon was introduced into the torch as a swirling sheath gas at a flowrate of 30 slm, but otherwise the flow configurations differed. In the 200-Torr experiments hydrogen, at 8 slm, and CH₄, at 80–320 sccm (CH₄:H₂ = 1–4%), were injected together with the main argon into the sheath flow, and an additional 15 slm of argon was coaxially injected through a ceramic tube located in the center of the torch head. In the 700-Torr
experiments hydrogen, at 5 slm, and CH₄ at 37.5 sccm (CH₄/H₂ = 0.75%; the reason for this choice is discussed below), together with an additional 5 slm of argon were injected through a coaxial, water-cooled injection probe that was inserted directly into the RF coil region of the torch, similar to the reactant injection scheme used in the 1-atm experiments [13]. In summary, the major difference in the flow configuration for the two sets of experiments was that for the 200-Torr experiments the reactants, hydrogen and methane, were injected around the circumference of the torch with the sheath gas, while for the 700-Torr experiments the reactants were injected through a central probe inserted directly into the plasma.

Molybdenum substrates were used, and were pretreated in all cases by sanding with progressively finer grit SiC paper, polishing with 1-μm diamond paste, ultrasonic rinsing in an ethanol solution of 60-nm diamond powder, and finally an ultrasonic rinse in acetone. The substrates were mounted on a substrate assembly that employed a previously described automatic temperature control system [16]. For the 200-Torr experiments the growth surface temperature was measured by a one-color pyrometer (Raytek, operating at 1.6 μm), and the substrate back surface temperature was monitored by an optical fiber interfaced to a separate one-color pyrometer. We estimate the uncertainty in the measured growth surface temperature to be ±25 K [17]. For the 700-Torr experiments only the temperature of the substrate back surface was available, due to limitations on optical access for the substrate location in that case. We estimate that the growth surface temperature in these experiments was ~100 K higher than the backside temperature.

Films were grown for 1 to 1.5 h in the 200-Torr experiments, and measured growth rates ranged from 29 to 69 μm/h. In the 700-Torr experiments all films were grown for 2 h, and measured growth rates ranged from 4 to 19 μm/h.

Gas composition at the substrate surface was characterized using a GC (Hewlett-Packard model 5890, series 2, equipped with flame ionization and thermal conductivity detectors). For the argon–hydrogen–methane plasma of interest, the GC provided measurements of Ar, H₂, CH₄, C₂H₂, C₂H₄ and C₂H₆. Gas was sampled through a critical orifice located at the center of each substrate, and delivered through a sampling line to the GC. For the 200 Torr experiments the orifice diameter was 100 μm and the sampling line was maintained at 25 Torr, dropping to 15 Torr across the GC sampling loop. For the 700-Torr experiments the orifice diameter was 70 μm, and the sampling line pressure was 40 Torr, dropping to 30 Torr across the sampling loop.

It is not necessarily the case that the Ar, H an C atoms introduced into the reactor with the argon, H₂ and methane flows are perfectly mixed at the substrate, and the different flow configurations employed could produce different results regarding departures from perfect mixing, especially in view of the lower diffusivities of hydrocarbons compared to hydrogen. To compare experimental results with kinetic modeling of the near-substrate region, it is crucial that the actual element fractions in this region are known, if possible, as opposed to the reactor inputs. The GC measurements allow us to make a straightforward determination of this effect [13, 14]. Thus, for example, a CH₄/H₂ input ratio of 0.75% in the 700-Torr experiments, in which the reactants were injected through a central probe inserted directly into the plasma, was found to produce the same C:H elemental ratio (approx. 0.0058) at the sampling orifice as a CH₄/H₂ input ratio of 2% in the 200-Torr experiments, where the reactants were injected into the outer sheath flow. This was the reason for running the 700-Torr experiments at a CH₄/H₂ input ratio of 0.75%, so that the morphology and gas composition trends vs. temperature could be fairly compared.

It is necessary to address the possible presence of nitrogen in our experiments. It is known that nitrogen can affect diamond film morphology [18–23], although to our knowledge no studies have explored the effect of nitrogen on the trend of morphology vs. substrate temperature. Based on the purity of the gases used and on measured chamber leakage rates, we estimate that the nitrogen mole fraction in the gas phase was ~1 ppm in our 1-atm [13] and 700-Torr experiments, and a few 10 s of ppm in our 200-Torr experiments. A review of the literature [18–23] indicates that 1 ppm of nitrogen is probably too low to affect diamond morphology, but 10 s of ppm probably could. However, as discussed below, the morphology trend vs. substrate temperature, as well as the ratio of C₂H₂ to C₁ radicals at the growth surface, was basically the same in the 200-Torr experiments as in other acetylene-lean systems, whereas it was the acetylene-rich 700-Torr and 1-atm [13] experiments where these trends were different. Therefore, we conclude that the presence of nitrogen, while it might have had some effect on the morphology results at 200 Torr, could not explain the basic trends reported here.

3. Results

3.1. Film morphology

The 200-Torr experiments involved two experimental series: one in which the input CH₄/H₂ ratio was varied from 1 to 4% at a fixed substrate temperature of 1140 K, and a second in which the substrate temperature was varied from 1043 to 1424 K at a fixed input CH₄/H₂ ratio of 2%. In the former series, increasing the input CH₄/H₂ ratio caused the film morphology to change from {111} toward {100} faceting, as illustrated in Fig. 1. This is consistent with our previously reported trend for the 1-atm RF plasma [13], as well as with the trend reported, to our knowledge, for all diamond CVD
Fig. 1. SEM images of films grown in the 200-Torr experiments at a substrate temperature of 1140 K. The input methane–hydrogen ratio equalled 2% (left) or 4% (right).

systems using methane and hydrogen as reactants. For each film we estimated the value of the film morphology parameter \( \alpha = \sqrt[3]{V_{100}/V_{111}} \), where \( V_{100} \) and \( V_{111} \) are, respectively, the growth velocities in the \( \langle 100 \rangle \) and \( \langle 111 \rangle \) diamond crystal directions \[24,25\]. The value of \( \alpha \) for each film was estimated by visual inspection of the SEM image, following the guidelines suggested by Tamor and Everson \[2\]. Obviously, these polycrystalline films show a distribution of facets and facet tilt angles, so that the value of \( \alpha \) is not sharply defined as it would be for a well-textured film. Nevertheless, the trends were found to be reproducible.

Increasing substrate temperature at fixed input \( \text{CH}_4: \text{H}_2 \) ratio caused morphology to shift from \( \{ 100 \} \) toward \( \{ 111 \} \) faceting, as seen in Fig. 2. This is opposite to the trend reported in our previous 1-atm experiments \[13\]. Fig. 3 shows estimated values of \( \alpha \) vs. substrate temperature for these experiments, together with estimated error bars based on the distribution of morphologies seen in each SEM image.

In the 700-Torr experiments a single series of seven deposition experiments was run, all at fixed reactant flow rates, with backside substrate temperature varying from 950 to 1100 K, corresponding to an estimated growth surface temperature in the range 1050–1200 K. As substrate temperature increased, film morphology shifted from \( \{ 111 \} \) toward \( \{ 100 \} \) faceting, as illustrated in Fig. 4. This is consistent with our previous 1-atm experiments \[13\], where films were grown at a similar pressure but with a completely different RF plasma reactor.

In summary, the 200-Torr and 700-Torr experiments, both performed using the same RF plasma reactor, produced opposite trends regarding film morphology vs. substrate temperature. The trend for the 200-Torr experiments was similar to that reported for hot filament, microwave and DC glow discharge systems, while the trend for the 700-Torr experiments was similar to that reported for atmospheric-pressure RF plasma (using a different reactor), DC arc discharge and oxyacetylene flame CVD.

3.2. Gas composition measurements

In our previous 1-atm experiments \[13\], where GC measurements were made over a wide range of temperatures and input \( \text{CH}_4: \text{H}_2 \) ratios, \( \text{C}_2 \text{H}_4 \) was always by far the dominant hydrocarbon measured. Next most abundant was \( \text{CH}_4 \), at mole fractions that were 1–4% of the measured \( \text{C}_2 \text{H}_4 \). Ethylene, \( \text{C}_2 \text{H}_4 \), was always somewhat less abundant than \( \text{CH}_4 \), and the measured \( \text{C}_2 \text{H}_6 \) mole fraction was in general either several ppm or below the \( \sim 1 \) ppm detection limit of the GC.

A similar ordering of the measured hydrocarbons was observed in the present experiments for both the 200-Torr and 700-Torr cases. Fig. 5 shows a comparison of the results for these two cases for the measured concentration ratio of \( \text{CH}_4 \) to \( \text{C}_2 \text{H}_4 \) over a range of substrate temperatures. As noted above, the input \( \text{CH}_4: \text{H}_2 \) ratios differed between these two sets of experiments, but the actual C:H elemental ratios near the substrate were the same, \( \sim 0.0058 \). In addition, the total of all measured hydrocarbon mole fractions in both sets of experiments shown equalled close to 1000 ppm. In other words, the chemical inputs to the boundary layer in both sets of experiments were essentially the same, so that any differences in gas composition at the growth surface must be due to different boundary layer thicknesses, pressures and/or temperature profiles.
As seen in Fig. 5, the results for the measured concentration ratio of CH$_4$ to C$_2$H$_2$ differ considerably for the two cases. In the 700-Torr case this ratio equalled $\sim 0.03$, quite similar to the previous results for the 1-atm plasma [13], while in the 200-Torr case this ratio equalled $\sim 0.2$.

The relative abundance at the growth surface of C$_2$H$_2$ in diamond CVD systems is affected by several factors, including temperature, chemical inputs and pressure. Numerical simulations we performed, in which we ran our mechanism [14] for various assumed pressures, confirmed that the most likely explanation for the large difference in relative acetylene abundance in the 200-Torr experiments compared to the other two cases is the difference in pressure: reducing the pressure suppresses the recombination of C$_1$ species to C$_2$ species.

### 3.3. Numerical modeling

The discussion in the previous section emphasizes the different relative abundances of C$_2$H$_2$ and CH$_4$ measured by the GC for the 200-Torr and 700-Torr experiments. However, CH$_4$ is generally expected to be unreactive at the diamond surface. Most studies of diamond growth have concluded that the most important species are H and CH$_3$. Theoretical studies have also pointed to the potential role of C$_2$H$_2$ as a growth species (e.g. [26,27]), although experimental studies have indicated that its reactive sticking coefficient on diamond is roughly two orders of magnitude lower than that of CH$_3$ [28]. In addition, kinetic models have predicted that atomic C could be an important growth species in thermal plasmas under suitable conditions [29,30].

Unfortunately, the GC cannot measure H, CH$_3$ or C. Hydrogen atoms are expected to recombine rapidly to H$_2$ on the walls of the GC sampling line [31], while previous modeling [14] predicted that C and CH$_3$ are converted by homogeneous chemistry in the sampling line to CH$_4$, C$_2$H$_4$ and C$_2$H$_6$, in roughly equal amounts. Therefore, we rely on kinetic modeling to predict the concentrations of H, CH$_3$ and C at the substrate. While
these predictions cannot be directly validated by the GC measurements, the previous validation of our model for stable hydrocarbons [14] at least provides support for the model’s overall validity.

Our approach to modeling the new reactor used in these experiments was similar to that described in detail in Ref. [14] for the reactor used in the 1-atm experiments [13]. Namely, a global two-dimensional (axisymmetric) model that neglected the hydrocarbon input (approx. 0.1% of the total flow) was used to predict the location and velocity at the edge of the ‘chemically reacting boundary layer,’ defined to extend from the diamond growth surface to a location where the temperature equalled an arbitrarily chosen value, in this case 5000 K. Experimentally determined calorimetric balances for typical experimental conditions indicated that the average temperature and velocity at the exit of the plasma torch equalled 7500 K and 146 m/s, respectively. Solutions for temperature and velocity profiles were obtained using the commercial code CFD-ACE [32]. For the conditions and geometry of the 200-Torr experiments the two-dimensional model predicted that the 5000 K isotherm was located 1.4 mm from the substrate, and that the velocity at that location equalled 10 m/s. A one-dimensional stagnation-point-flow model was then used to predict flow and chemistry in the boundary layer. Chemical equilibrium was assumed at the 5000-K edge of the boundary layer, using Ar:H:C element fractions as measured by the GC. The CHEMKIN SPIN code [33] was used to solve the flow and species conservation equations. The same gas–phase and surface chemistry kinetic mechanism was used as described in Ref. [14].

Fig. 6 shows predicted profiles of several species and of temperature over the 1-mm gas layer adjacent to the growth surface, for the 200-Torr experiments at a substrate temperature of 1200 K and an assumed C:H element ratio of 0.0058, corresponding, as discussed above, to a 2% CH–H input ratio. Among the features of interest, we note the following: (1) the temperature gradient is extremely steep, dropping ~3300 K over 1
The predicted high abundance of atomic carbon is a unique feature of thermal plasma CVD environments with thin boundary layers [30]. Using the same diamond growth mechanism as in Ref. [31] the model predicts for this case a growth rate of 47 μm/h, in quite reasonable accord with the experimental result of 42 μm/h, with C predicted to be by far the dominant growth species.

4. Comparisons among different diamond CVD systems

We now address the question, are there differences in the chemical environment of different diamond CVD systems that could explain the two opposite trends of film morphology vs. substrate temperature?

To answer this question, we consider representative values of species concentrations at the growth surface for several diamond growth environments. The species of interest are H, C₂H₂, C and CH₃. The data we consider were taken from studies in which species concentrations at the diamond growth surface during CVD were either measured directly or calculated by an experimentally validated kinetic model. The systems considered include hot filament [34–37], microwave [38] and RF thermal plasma ([13,14] and the present
work). All the data considered are for a substrate temperature of 1200 K, or the closest temperature to that available, and all pertain to diamond growth conditions.

While C may be an important contributor to the growth rate in thermal plasmas, it seems plausible to assume, lacking information to the contrary, that all C radicals affect morphology evolution in essentially the same way, whereas the effect on morphology evolution of acetylene is fundamentally different. We therefore hypothesize that within the window of conditions where well-faceted diamond film grows, morphology evolution depends on the concentrations at the surface of three entities: H atoms, C\(_2\)H\(_2\), and the sum \(\sum_{n=0}^{3} [CH_n]\) of the concentrations of C\(_1\) radicals. In fact, while absolute growth rates must depend to large extent on the absolute concentrations of these species, we expect morphology to depend on their relative concentrations, at least within the usual diamond growth regime where growth is limited by mass transport from the gas phase.

The trend of diamond morphology vs. substrate temperature has been reported for all of the systems considered. In Table 2, we summarize characteristic results from the literature regarding the morphology trend as well as the concentration ratios at the growth surface of both H atoms and C\(_2\)H\(_2\) to C\(_1\) radicals.

<table>
<thead>
<tr>
<th>CVD system: 200-T orr</th>
<th>Increasing substrate temperature causes:</th>
<th>(\frac{[H]}{[C]+[CH_3]})</th>
<th>(\frac{[C_2H_2]}{[C]+[CH_3]})</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hot filament {100} (\rightarrow) {111} [5]</td>
<td>6 to 15</td>
<td>0.1 to 10 [34–36]</td>
<td></td>
</tr>
<tr>
<td>Microwave {100} (\rightarrow) {111} [1–4]</td>
<td>4</td>
<td>9 [38]</td>
<td></td>
</tr>
<tr>
<td>RF thermal plasma at (200) Torr</td>
<td>40</td>
<td>2 (this work)</td>
<td></td>
</tr>
<tr>
<td>RF thermal plasma at or near (1) atm</td>
<td>90</td>
<td>110 [14]</td>
<td></td>
</tr>
</tbody>
</table>

Regarding the ratio of C\(_2\)H\(_2\) to C\(_1\) radicals, however, the two RF thermal plasma systems are dramatically different from each other, with characteristic ratios equal to \(\sim 2\) in the 200-Torr case and \(\sim 110\) in the 1-atm case. The 200-Torr experiments lie in the ‘acetylene-lean’ regime (concentration ratio of C\(_2\)H\(_2\) to C\(_1\) radicals \(\sim 0.1–10\)) similar to the hot filament and microwave systems, while the 1-atm experiments lie in the ‘acetylene-rich’ regime (concentration ratio of C\(_2\)H\(_2\) to C\(_1\) radicals approx. 100). We do not have a calculation of the C\(_1\) radical concentration in the 700-Torr experiments, but, as noted above in connection with Fig. 5, the measured ratio of CH\(_4\) to C\(_2\)H\(_2\) in those experiments was quite similar to its value in the 1-atm experiments [13,14], suggesting similar levels of C\(_1\)–C\(_2\) interconversion chemistry. C\(_1\) radical concentrations roughly scale on the CH\(_4\) concentration through partial equilibrium of fast hydrogen abstraction reactions [39]. In addition, some of the CH\(_4\) measured by the GC, as noted above, could be ascribed to methyl radicals that were destroyed by chemical reactions in the sampling line. It is thus reasonable to expect that the 700-Torr experiments had a similar value of the C\(_2\)H\(_2\)-to-C\(_1\)-radical ratio as did the 1-atm experiments, i.e. one can reasonably deduce that the 700-Torr experiments also lay in the acetylene-rich regime.

The films grown in the 700-Torr experiments, as previously discussed, followed the same morphology trend as did those in the 1-atm experiments. Considering this together with inspection of Table 2, it thus appears that a possible explanation for the different morphology trends is the relative abundance of acetylene: diamond films grown in acetylene-lean environments undergo a transition from \{100\} to \{111\} faceting as substrate temperature increases, while films grown in acetylene-rich environments do just the opposite.

This correlation, of course, does not prove that it is acetylene that causes this effect. However, there have been theoretical studies suggesting that the presence of acetylene can indeed have a strong influence on film morphology. In particular, atomic-scale simulations by Battaile et al. [40,41] indicate that C\(_2\)H\(_2\) affects growth
of order unity (approx. 0.1–10), then lower substrate temperature favors \{100\} facets and higher substrate temperature favors \{111\} facets. When, however, this ratio is much greater than unity (e.g., approx. 100)—as can occur, depending on conditions, in high-temperature gas environments such as DC arc torches, RF thermal plasmas and oxyacetylene flames—then the trend of film morphology vs. substrate temperature is reversed.

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