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Atmospheric plasma deposition of diamond-like carbon coatings

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ABSTRACT

The atmospheric pressure plasma-enhanced chemical vapor deposition of diamond-like carbon (DLC) has been investigated. The DLC coatings were grown with a mixture of acetylene, hydrogen and helium that was fed through a linear plasma source. The plasma was driven with radio frequency power at 27.12 MHz. Deposition rates exceeded 0.10 µm/min at substrate temperatures between 155 and 200 °C. Solid-state carbon-13 nuclear magnetic resonance revealed that the coatings contained approximately 43% sp^2-bonded carbon and 57% sp^3-bonded carbon. Coefficient of friction values for the coatings were found to be 0.24 ± 0.02, which is within the range observed for vacuum deposited DLC.

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1. Introduction

There is great demand for thin functional coatings in many industries, including semiconductors, medical devices, automotive and aerospace [1–13]. As fabricated components become smaller and more complex, the surface properties of the materials take on greater importance. Thin coatings play a key role in tailoring surfaces to give them the hardness, wear resistance, chemical inertness, and electrical characteristics needed in a desired application.

Diamond-like carbon (DLC) possesses an array of valuable properties: outstanding abrasion and wear resistance; chemical inertness; exceptional hardness; low coefficient of friction; and high dielectric strength [14–22]. Diamond-like carbon is considered to be an amorphous material, containing a mixture of sp^2- and sp^3-bonded carbon. Based on the percentage of sp^3 carbon and the hydrogen content, four different types of DLC coatings have been identified: tetrahedral carbon (ta-C), hydrogenated amorphous carbon (α-C:H) hard, α-C:H soft, and hydrogenated tetrahedral carbon (ta-C:H) [20,23,24]. Tetrahedral carbon films contain 80% to 88% sp^3 carbon and no appreciable hydrogen. They exhibit hardness values approaching 80 GPa. By contrast, α-C:H soft contains ~60% sp^3 carbon, a hydrogen mole fraction between 30 and 50%, and its hardness is less than 10 GPa.

Methods used to deposit diamond-like carbon include ion beam deposition, cathodic arc spray, pulsed laser ablation, argon ion sputtering, and plasma-enhanced chemical vapor deposition [25–35]. Researchers contend that several advantages exist when depositing DLC coatings in a low-pressure environment. For example, ion bombardment in vacuum is thought to promote denser sp^3 carbon networks. Moreover, argon ion sputtering is well suited for coating large parts [13,36,37]. On the other hand, the deposition of DLC in vacuum has several disadvantages, including high equipment cost and restrictions on the size and shape of material that may be treated.

The deposition of diamond-like carbon at ambient pressure has been demonstrated by several researchers. Izake et al. [38] and Novikov and Dymont [39] demonstrated that DLC can be made by an electrochemical process using organic compounds, such as methanol and acetylene dissolved in ammonia. This process requires that the substrate be immersed in liquid. The atmospheric pressure deposition of DLC with a plasma torch has been demonstrated by Kulik et al. [40]. However, this process subjects the substrate to gas temperatures in excess of 800 °C.

In this report, we show that diamond-like carbon films may be deposited using an atmospheric pressure plasma-enhanced chemical vapor deposition (PECVD) process at temperatures between 100 and 350 °C. The films were characterized by solid-state carbon-13 nuclear magnetic resonance, and found to have quantities of sp^2 and sp^3 carbon consistent with α-C:H soft. These films were tested for adhesion and coefficient of friction.

2. Experimental methods

A schematic of the atmospheric pressure plasma deposition system is shown in Fig. 1. The complete system, with modifications, was purchased from Surfx Technologies, LLC. It contained the following components: an RF VII, Inc. 1000 W RF power generator at 27.12 MHz, an RF VII, Inc. auto-tuning matching network, 3 mass flow controllers, a chamber that could be evacuated, a substrate tray with a resistive

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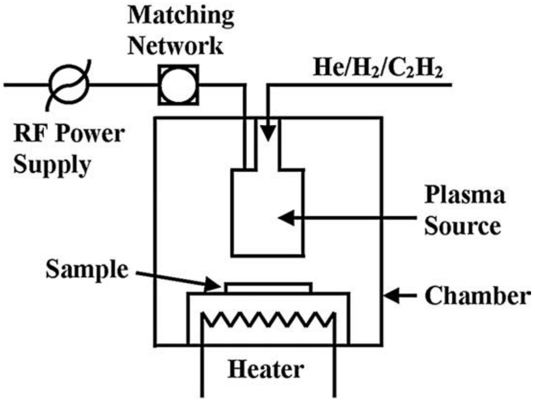

Fig. 1. Flow schematic of DLC deposition process.

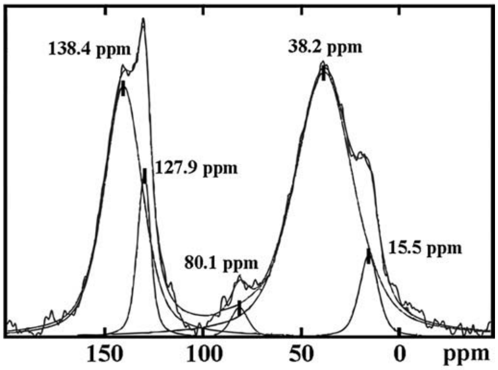

Fig. 2. Solid-state ^{13}C NMR spectrum of an atmospheric pressure plasma deposited DLC coating.

coil heater, a Surfx plasma source, and an LED display and user interface to control the system operation. The chamber featured a removable lid, which the plasma source was housed in, and a glass viewport. Ultra-high purity helium and acetylene (Scott Specialty, purity 99.6%) were fed to the plasma source using nylon tubing. Stainless steel tubing was utilized for the hydrogen gas line as well as for feeding the gas mixture from the control unit to the plasma source.

The coatings were deposited onto two types of substrates: p-type Si (100) wafers and 304 L stainless steel disks, 1.12″ in diameter. The substrate was placed in the chamber with the plasma source mounted directly above it. The glass viewport was sealed onto the chamber, and the chamber evacuated to approximately 50 Torr, followed by backfilling it with helium to 800 Torr for safety purposes. Next, the resistive coil heater was turned on and after the substrate reached the desired temperature, helium was introduced at 30.0 L/min through the plasma source. The RF power was applied at 120 W, and hydrogen was introduced at a flow rate of 0.05 L/min. Then the RF power and hydrogen flow rate were simultaneously increased to a final value of 160 W and 0.50 L/min. After the plasma stabilized, acetylene was introduced at 0.01 L/min and slowly increased to 0.05 or 0.10 L/min. The deposition time was varied between 5 and 25 min. After this period, the acetylene flow, hydrogen flow, RF power and heater were turned off, and the chamber was evacuated to 50 Torr and backfilled with helium to 760 Torr. Then, the glass viewport was removed, the helium flow was turned off, and the substrate retrieved from the chamber.

The carbon films were analyzed by nuclear magnetic resonance with a Bruker DSX 300 solid-state NMR spectrometer with operating frequencies of 300.13 MHz and 75.14 MHz for ^{1}H and ^{13}C, respectively. Chemical shift anisotropy was minimized using magic-angle spinning. The ^{13}C cross-polarization/magic-angle spinning (CP/MAS) spectra were obtained with a standard Bruker MAS probe using 4 mm (outside diameter) zirconium oxide rotors with Kel-F caps [41]. For maximum sensitivity, the ten-milligram samples scrapped from Si wafers were confined to the middle of the rotor by placing sodium chloride above and below the sample. Potassium bromide (KBr) was used to adjust the magic angle with the upfield methine resonance of adamantine at 29.46 ppm, serving as the external chemical shift reference.

To reduce experimental time as well as enhance the sensitivity, cross-polarization from the abundant ^{1}H nuclei to the dilute ^{13}C nuclei was utilized. The recycle delay between scans in the cross-polarization experiment depends on the spin-lattice relaxation time of the protons. As ^{1}H nuclei possess significantly faster relaxation times than ^{13}C nuclei, data acquisition time is reduced in comparison to direct observation of the ^{13}C signal. All experiments were performed at ambient temperature. A 3.8 μs $\pi/2$ pulse length was used with a contact time of 1.5 ms. The recycle delay was 1.0 s with 75,000 as the average number of scans acquired. By using a magic-angle spinning frequency of 10 kHz, it was possible to identify and reduce the intensities of spinning side bands [42–44].

The percentage of carbon hybridization was calculated by deconvoluting the sp^2 and sp^3 resonance, found in the NMR spectrum at approximately 140 and 40 ppm, respectively [45–49]. The method fitted the observed resonances with 50% Gaussian and 50% Lorentzian lineshapes. As a reference check, a solid-state ^{13}C NMR spectrum was acquired from diamond powder. A sharp resonance with a chemical shift at 34.9 ppm was observed in agreement with the literature value of 36.0 ppm [45].

Thickness measurements of the diamond-like carbon films on the silicon wafers were obtained using a Dektak 7 profilometer. To obtain a step height measurement, the samples were masked and then mechanically scratched. This data also leads to the determination of the deposition rate. These measurements could not be made with sufficient accuracy on the stainless steel coupons due to their high surface roughness.

The adhesion of the DLC coatings to the stainless steel and Si wafer was determined using a stud pull test [49]. In an effort to remove surface contaminants, samples were briefly treated with an oxygen plasma for 5 min at 250 W RF power, 30 L/min helium flow, and 0.3 L/min O_2 flow. The test device was a Romulus III from Quad Group Inc. A metal stud, 0.1″ diameter, was affixed to the coating surface using epoxy glue. The stud was then pulled perpendicular to the substrate at a rate of 6 lb/s. Adhesive strength is defined as the amount of pressure required to remove the stud from the substrate. After the pull test, the fractured surface was examined by scanning electron microscopy (SEM) using a Leo 1455VP. No conducting coating was applied. All images were obtained at 25× with an accelerating voltage of 25 kV and a working distance of 8 mm. Quad-backscattering detection was employed.

Pin on disk testing, per ASTM G99, was performed to obtain the coefficient of friction. A 3.75 mm radius wear track was created at a speed of 2.8 cm/s. Coefficient of friction values were calculated by dividing the normal load (1.0 N) by the force obtained after 100 cycles.

3. Results

3.1. Carbon bonding

A solid-state ^{13}C NMR spectrum of the carbon film obtained from several samples is displayed in Fig. 2. The process conditions were

Table 1
Position and area of sp^2 and sp^3 carbon peaks

Carbon type	Peak no.	Position (ppm)	Fraction of total area
sp^2	1	138.4	0.327
sp^2	2	127.9	0.076
sp^3	3	80.1	0.054
sp^3	4	38.2	0.525
sp^3	5	15.5	0.018

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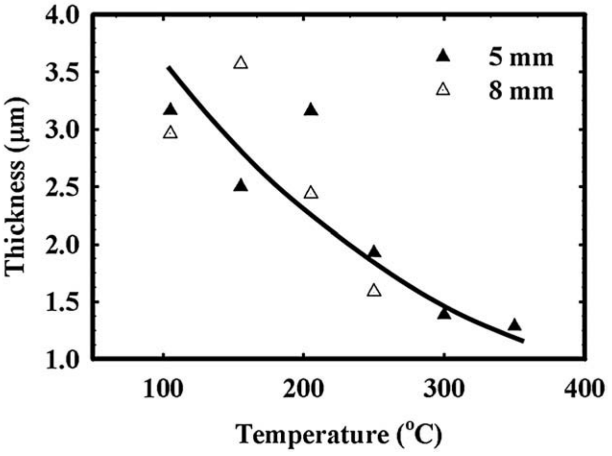

Fig. 3. Thickness of DLC coatings as a function of deposition temperature with 0.10 L/min of C_2H_2 and a source-to-sample distance of 5 mm and 8 mm.

160 W RF power, 155 °C substrate temperature, source-to-sample distance of 8 mm, 0.10 L/min acetylene flow, 0.50 L/min hydrogen flow, and 30.0 L/min helium flow. As seen in the figure, two main resonance bands are observed, centered at approximately 128 and 38 ppm. These bands are due to sp^2 and sp^3 carbon bonding, respectively, and demonstrate the successful deposition of a diamond-like carbon film at atmospheric pressures. The sp^2 band was deconvoluted into two peaks, while the sp^3 band was deconvoluted into three peaks. Table 1 lists the position of each deconvoluted peak as a fraction of the total band area. The sp^3 peaks centered at about 80 and 15 ppm are due to spinning side bands and were not included in the area calculation. Based on the area under the bands, the fraction of sp^2 and sp^3 carbon was 43 and 57%, respectively. In summary, diamond-like carbon coatings have been deposited using the atmospheric pressure plasma fed with acetylene, hydrogen and helium at a substrate temperature of only 155 °C.

3.2. Effects of process conditions

Shown in Fig. 3 is the effect of temperature on the DLC coating thickness for two different source-to-sample distances. Deposition was carried out for 25 min at 160 W RF power, flow rates of 0.10 L/min C_2H_2, 0.5 L/min H_2, and 30.0 L/min He, and source-to-sample distances of 5 and 8 mm. A loosely bound amorphous carbon by-product is observed at temperatures below approximately 200 °C and 155 °C for 5 and 8 mm source-to-sample distances, respectively. At temperatures above, the coating thickness drops rapidly as the substrate temperature increases to 350 °C. The maximum thickness achieved was 3.16 μm and 3.54 μm for the 5 and 8 mm source-to-sample distances, respectively.

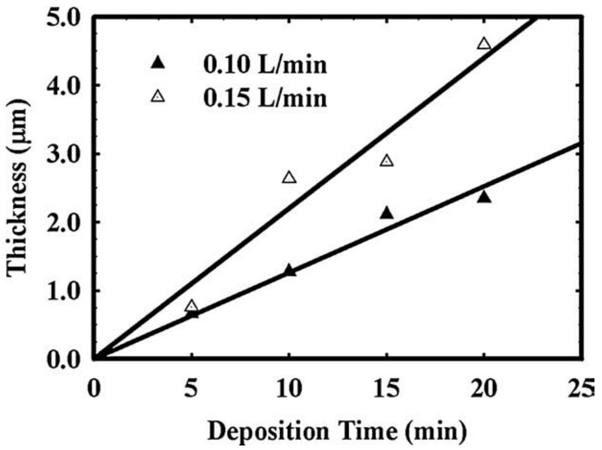

Fig. 4. Thickness of DLC coatings as a function of deposition time at two C_2H_2 flow rates, a substrate temperature of 200 °C, and a source-to-sample distance of 10 mm.

Table 2
Adhesion data for DLC coatings

Sample	Adhesion (psi)	
	No treatment	O_2 plasma clean
A	3650	2300
B	5370	3820
C	4550	520

The dependence of the coating thickness on deposition time at the different acetylene flow rates is shown in Fig. 4. The process conditions are the same as reported earlier, except that the substrate temperature is 200 °C and the source-to-sample distance is 10 mm. The thickness varies linearly with time with the lines extrapolating back to the origin. This indicates that there is no induction period for the onset of DLC deposition. The film growth rate is obtained from the slope of the lines, which equals 0.13 ± 0.04 mm/min for 0.10 L/min acetylene flow and 0.22 ± 0.07 mm/min for 0.15 L/min acetylene flow. These results indicate that the deposition rate is positive order in acetylene concentration.

3.3. Adhesion, wear and dielectric strength

The adhesive strength of the DLC coatings, measured by a stud pull test, is shown in Table 2. Samples A and B were generated on stainless

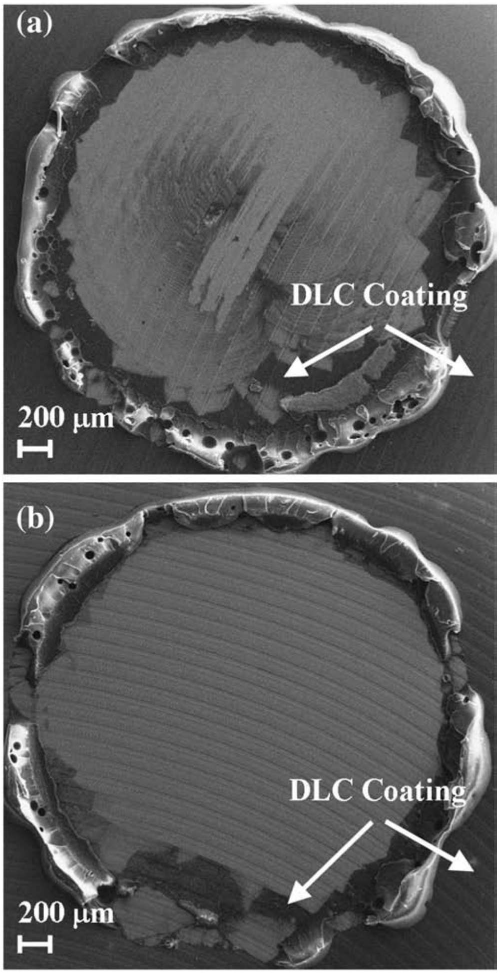

Fig. 5. Scanning electron micrographs of DLC coatings after the adhesion test: (a) without; and (b) with plasma treatment prior to deposition.

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steel substrates, whereas sample C was prepared on a silicon substrate. On substrates not initially treated with the O_2 plasma, the adhesion ranged from 3650 to 5370 psig. By contrast, substrates initially exposed to the plasma yielded DLC films with lower bond strengths of 520 to 2300 psig. The oxygen plasma treatment of the samples was intended to remove surface contaminants; however, due to the decrease in adhesion from plasma treated samples, it is apparent that a surface oxide was formed. After failure the surfaces of the steel coupons were examined with the SEM, and these results are shown in Fig. 5. The SEM images reveal small amounts of DLC remaining around the edges of the circular test region and very thin layers of coating within the center region. These pictures reveal that failure occurs at the interface of the carbon film with the steel surface. Similar results were observed for DLC deposition on silicon.

Coefficient of friction values for DLC deposited on 304 L stainless steel coupons were found to be 0.24 ± 0.02, per ASTM G99. It was found that the oxygen plasma treatment prior to deposition had no impact on the coefficient of friction values. This may be compared to a coefficient of friction of 0.55 for stainless steel [50].

4. Discussion

It has been found that diamond-like carbon can be deposited at low-temperature and atmospheric pressure using a downstream acetylene, hydrogen and helium plasma. As shown in Fig. 2, solid-state ^{13}C NMR peaks are observed at 138.4 and 127.9 ppm for sp^2 carbon and at 38.2 ppm for sp^3 carbon. These chemical shifts are in good agreement with the values reported for diamond-like carbon deposited in vacuum processes [45–49]. For example, Merwin et al. [45] report chemical shifts of 120.0 and 34.5 ppm for sp^2 and sp^3 carbon, respectively, whereas Bustillo et al. [47] report chemical shifts of 140.0 and 40.0 ppm for sp^2 and sp^3 carbon. The broadness of the NMR resonance bands reflects the effect of dispersion in the observed chemical shifts, i.e. different types of sp^2 and sp^3 carbon bonding in the films [48,51]. The peak at 138.4 ppm may be assigned to unsaturated CH groups, while the peak at 127.9 ppm is due to CH_2 groups. On the other hand, the broad band at 38.2 ppm may be assigned to saturated carbon atoms with 1, 2 or 3 hydrogen atoms attached. The fraction of sp^2 and sp^3 carbon bonding was determined to be 43 and 57%. Based on the four classifications of DLC coatings, the material produced in this study may be designated as α-C:H soft [23]. This seems reasonable given that the films were deposited by a downstream, low-temperature plasma process with the substrate held at 155 °C.

The results shown in Figs. 3 and 4 reveal that the rate of DLC deposition decreased rapidly with increasing substrate temperature up to 350 °C, but increased with the partial pressure of acetylene. The optimal temperature for growth is around 200 °C, since this yields a high rate while at the same time avoiding the formation of a loosely bound carbon deposit. At the higher acetylene flow of 0.15 L/min, a deposition rate of 0.22 μm/min is achieved. This may be compared to vacuum processes where rates have been reported as low as 10^{-3} μm/min to as high as 0.6 μm/min, depending on the method utilized [52,53].

The coefficient of friction for the DLC obtained in this study was 0.24 ± 0.02. Previous studies report coefficient of friction values for vacuum deposited coatings to be within the range of 0.05–1.00 [1,49,54–57]. For uncoated stainless steel, the coefficient of friction is 0.55 [50].

The work presented above is the first successful demonstration of diamond-like carbon deposition using a downstream, low-temperature atmospheric pressure plasma source. Additional work is needed to improve the process and enhance the properties of the DLC coatings.

5. Conclusions

The deposition of diamond-like carbon using a low-temperature, atmospheric pressure plasma process is reported for the first time. Using solid-state ^{13}C NMR spectroscopy, the coatings were found to possess 43% sp^2 carbon and 57% sp^3 carbon. A deposition rate of 0.22 μm/min was observed at 160 W RF power, 200 °C substrate temperature, 10 mm source-to-substrate distance, and with feed rates of 0.15 L/min acetylene, 0.50 L/min hydrogen and 30.0 L/min helium.

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