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Research Paper

Raman Analysis of DLC Nanostructure Coating on AL 6061-T6 Made by DC Pulsed- PACVD

Seyed Mohammad Mahdi Shafiei^{1,2*}, Hamed Raeisifard¹

¹Department of Mechanical Engineering, West Tehran Branch, Islamic Azad University, Tehran, Iran

²Department of Mechanical Engineering, Roudehen Branch, Islamic Azad University, Tehran, Iran

*Email of Corresponding Author: shafiei.mohammad@wtiau.ac.ir

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Abstract

Plasma assisted chemical vapor deposition (PACVD) technique was used to make a diamond-like carbon (DLC) coating on the Aluminum 6061- T6 substrate. The deposition was carried out using CH₄ as the process gas, at different temperatures, 250°C and 300°C with constant power and flow rate. Characterization technique Raman spectroscopy was used to characterize these samples. Raman analysis of DLC coatings at different temperatures is carried out in detail for two different excitation wavelengths i.e. 514 and 785 nm and, results are presented in the paper. Blue shifts were observed in both D and G peaks of the Raman spectrum with an increase in deposition temperature, which indicates the formation of compressive strain in high-temperature deposited DLC coatings. Dispersion in both D and G peaks is observed for different excitation wavelengths suggesting that the coating is hydrogenated DLC. The degree of hydrogenation of the DLC coating appears to decrease for the deposition temperature. Nano-indentation study shows a marginal increase in hardness with an increase in deposition temperature.

Keyword

DLC, PACVD, Aluminum, Raman, Nano-indentation

1. Introduction

Diamond-like carbon (DLC) is a mixture of sp² and sp³ hybridization of carbon, where sp³ hybridization is more dominant, therefore has diamond-like characteristics [1-3]. DLC coatings showed good mechanical, optical, electrical, chemical, and tribological properties and can be used in applications for antireflective coatings for solar cells, IR optical materials, wear-resistant and low friction coatings, orthopedic implants, etc [4-8]. DLC coating is deposited by physical and chemical processes such as sputtering and microwave (MW) and Radio Frequency (RF) processes respectively [9-11]. Different process parameters such as reacting gases, bias voltage, etc.; can be used for the deposition of DLC films [12-14] in the chemical process.

Based on the degree of hydrogenation, properties of the DLC coatings such as hardness, coefficient of friction (COF), and anti-reflectivity can change. Therefore Hydrogenation plays an important role in biomedical applications to reduce cytotoxicity and COF in DLC coatings [15-17]. In this paper hydrogenated tetrahedral amorphous carbon (ta-C: H) coating was developed on an aluminum

substrate using plasma-assisted chemical vapor deposition (PACVD) technique using DC power. Raman analysis is a useful technique to understand carbonaceous materials such as DLC [18, 19]. In this paper, detailed Raman studies were carried out with excitation lasers of 514 and 785 nm to understand the DLC coating.

2. Experimental details

DLC films were deposited on the single-sided polished aluminum-T6 substrates (Table1).

Table 1. Chemical composition of aluminum-T6 substrates

element	Ti	Mn	Fe	Si	Cr	Cu	Zn	AL	
Wt%	min	-	-	-	0.7	0.04	0.15	-	95.56
	max	0.15	0.15	0.7	0.8	0.35	0.4	0.25	98.85

The deposition system consists of PACVD (model: HBS 500). DLC films were coated on aluminum-T₆ substrates. The substrates were first cleaned by soap water, distilled water, and in the end, by isopropyl alcohol in an ultrasonic bath. Before deposition, the substrates were cleaned in argon (Ar) plasma at a flow rate of 300 sccm and a constant bias voltage of 400 V for 20 minutes. DLC deposition was then carried out with 100 sccm of CH₄ as process gas in a constant power mode with a DC power of 200 W and at an operating pressure of 2.0×10^{-3} mbar. The deposition temperatures were varied at 250°C (Sample name - AL 250) and 300°C (Sample name - AL 300) to study the effect of deposition temperature on the structural and mechanical properties of the DLC coating.

The film thickness and refractive index of the DLC film were determined by scanning electron microscopy (SEM). Raman analysis of the DLC films was carried out by Seki Technotron, STR 300 Raman spectroscope with 514 and 785 nm monochromatic light. Hardness and Young's modulus were studied by the nano-indenter of Agilent make of model G200.

3. Results and discussions

From the scanning electron microscopy (SEM) result, it is found that the thickness of DLC coatings is decreasing for the deposition temperature, which means that the DLC process is falling in the depletion region in the same substrate (Figures 1 and 2). Figure 2 shows the morphology on the coating surface.

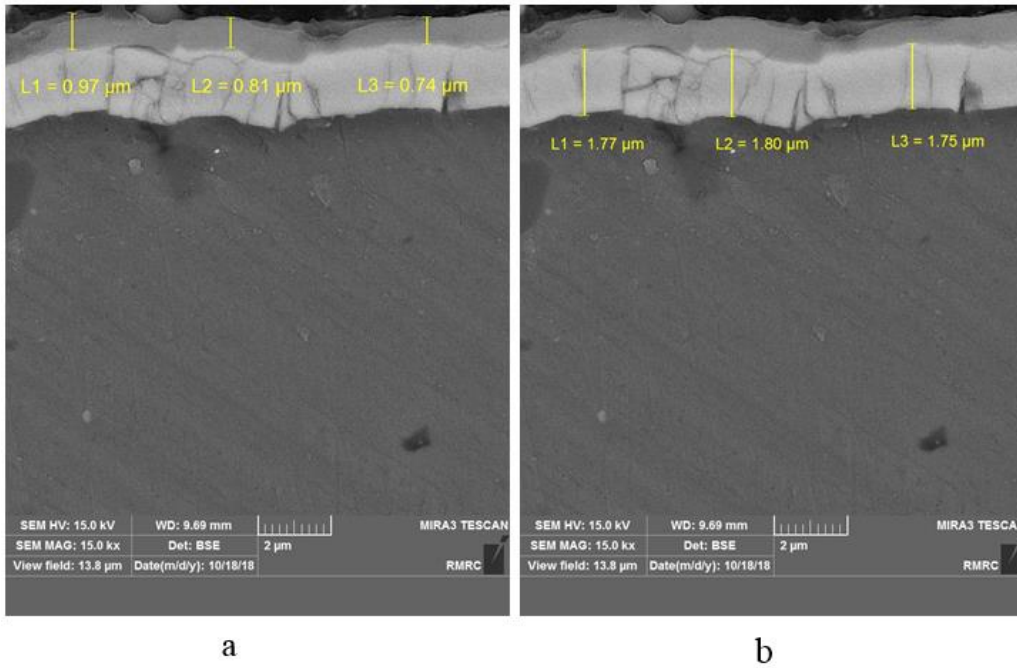


Figure 1. Thickness vs. deposition temperature variation of the DLC coating process a) 300 °C, b) 250 °C

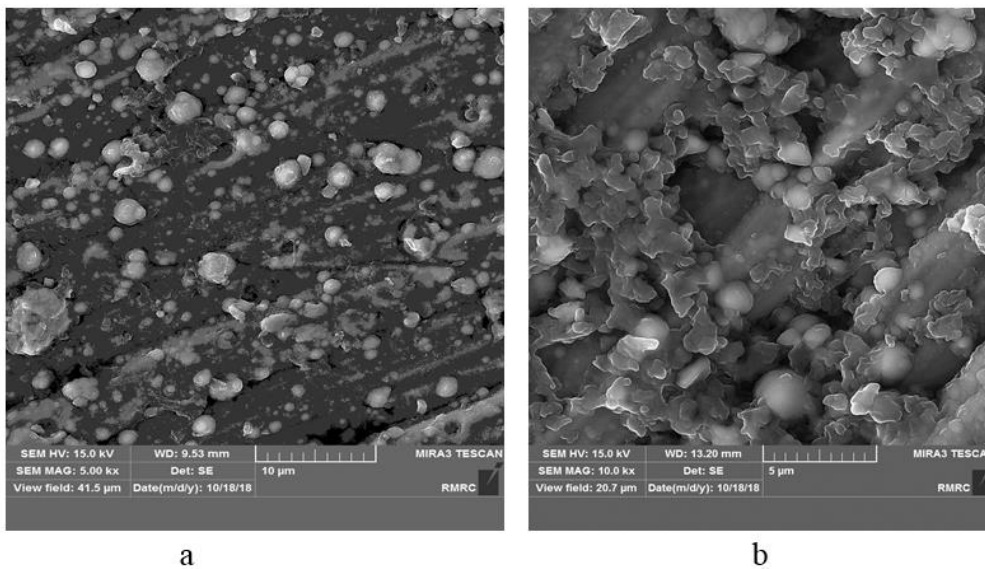


Figure 2. Morphology vs. deposition temperature variation of the DLC coating process a) 250 °C, b) 300 °C

Raman spectrum for different deposition temperatures is shown in Figure 3. Raman data is developed. Both D and G Raman peaks are found to be blue shifting for the deposition temperature of DLC processing. It is concluded that high deposition temperature leads to an increase in compressive stress. The FWHM value of both G peaks is found to be decreasing, while the D peak is found to be increasing with an increase in the deposition temperature. Ad/Ag (peak area ratio of D and G peaks) ratio is found to be increasing, for the deposition temperature, which suggests that ring structure concentration is getting increased for linear structure.

Dispersion of both G and D peaks are studied in different excitation wavelengths of 514, to study the variation of deposition temperature over the structural property of the DLC coatings (Figure 3).

Dispersion takes place when peak shift occurs at different excitation wavelengths, which is generally associated with the disorder in the system [18-20].

More dispersion in the G peak confirms the formation of DLC coating. However, the D peak also appears to shift with the excitation wavelength, which is an indication of hydrogenation of the DLC coating. The dispersion seems to reduce for D and G peaks when the deposition temperature is raised to 300°C. It means that both hydrogenation and disorder in the DLC coating are reducing with the increase in the deposition temperature. Similar results are obtained through non-Rutherford backscattering (non-RBS) and elastic recoiling detection analysis (ERDA) where the hydrogen to carbon ratio was calculated for the DLC coating at room temperature is found to be 32:68, which decreases slightly at a deposition temperature of 300°C to 30:70.

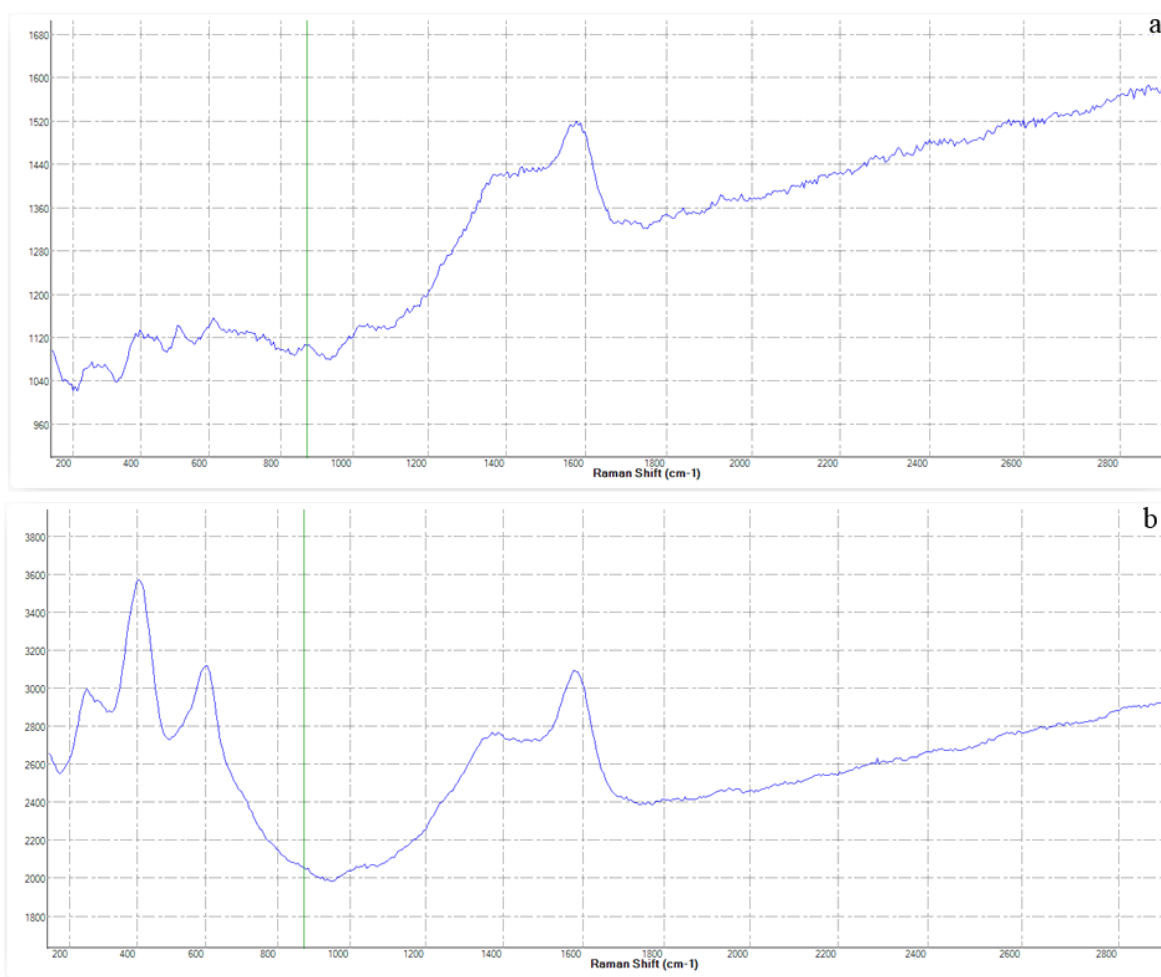


Figure 3. Raman spectrum of DLC coating collected at an excitation wavelength of 514 nm and deposited at a) 250 °C and b) 300°C

Nano-indentation analysis shows an increase in the hardness and Young's modulus with an increase in the deposition temperature. The data is consistent with the Raman results that reveal the increase in deposition temperature leads to a decrease in disorder, hydrogenation, and, an increase in ring structure content and compressive strain, which helps in increasing the hardness and Young modulus of the DLC coating (Figure 4).

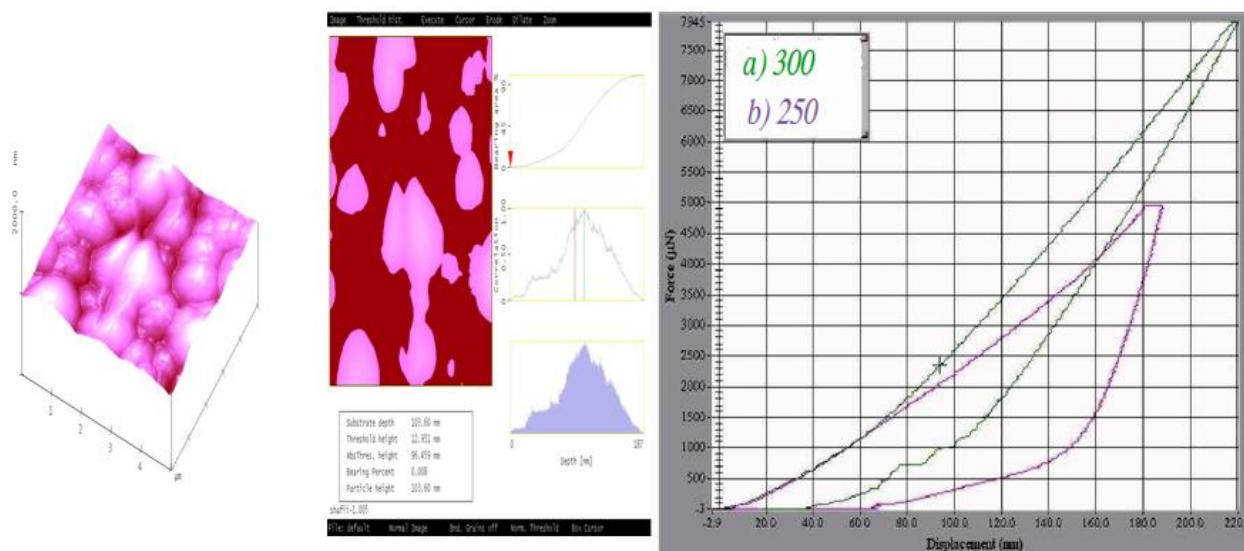


Figure 4. AFM image of nano-indentation obtained with 100mN load on the polished top surface of the coating a) 300°C and b) 250°C

4. Conclusions

The DLC coating made by DC- the PACVD process is found to be disordered and partially hydrogenated based on a detailed study of Raman analysis. With the increase in the deposition temperature, it is observed that disorder and hydrogenation are slightly get reduced and compressive strain is getting increased. Ad/Ag ratio is also found to be increasing with the deposition temperature, which indicates the increase in ring structure content for linear structure content. Overall structural properties contribute to the increase in the hardness of the DLC coating.

5. Acknowledgement

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6. References

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