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Influence of RF ICP PECVD process parameters of diamond-like carbon films on DC bias and optical emission spectra

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The work presents the results of a research carried out with PlasmaLab Plus 100 system, manufactured by Oxford Instruments Company. The system was configured for deposition of diamond-like carbon films by ICP PECVD method. The change of an initial value of DC bias was investigated as a function of set values of the generator power (RF generator and ICP generator) in the constant power of the RF generator operation mode. The research shows that the value of DC bias nearly linearly depends on the RF generator power value and is affected only in a small degree by the power of ICP discharge. The capability of an installed OES spectrometer has been used to ensure the same starting conditions for the deposition processes of DLC films. The analysis of OES spectra of RF plasma discharge used in the deposition processes shows that the increase in ICP discharge power value results in the increased efficiency of the ionization process of a gaseous precursor (CH_4). The quality of deposited DLC layers was examined by Raman spectroscopy. Basing on the acquired Raman spectra, the theoretical content of sp^3 bonds in the structure of the film was estimated. The content is ranging from 30% to 65% and depends on ICP PECVD deposition process parameters.

Keywords: PECVD, diamond-like carbon layers, OES, Raman spectroscopy, AFM.

1. Introduction

Diamond-like carbon (DLC) films have received major attention over the last decade due to their unique properties. DLC films can be used in photonic structures as layers increasing sensitivity to certain environmental factors or as wave guiding layers.

Unique properties, such as high resistivity, thermal conductivity and chemical resistance, make DLC films interesting in electronic applications. For the above-mentioned applications these films are often deposited using plasma enhanced chemical vapour deposition (PECVD) method [1, 2].

In this work we focus on the influence of radio frequency (RF) inductively coupled plasma (ICP) PECVD process parameters, such as power values of RF/ICP generator on DC bias value and its stability, plasma optical emission spectra (OES) and film properties (ratio of sp^3 to sp^2 in the deposited layer and its surface roughness) [3]. The investigation was carried out in order to develop DLC deposition technology for future electronic applications.

2. Experimental method

2.1. Diamond-like carbon film deposition

The DLC films were deposited using PlasmaLab Plus 100 system manufactured by Oxford Instruments Company. The deposition processes were carried out in CH_4 atmosphere. The pressure in the working chamber is set on 50 mTorr and the methane flow at a rate of 100 sccm. The table temperature was controlled and stabilized around 20 centigrade. The used substrates were (111) 10 mm×10 mm polished silicon wafers located on 2″ silicon wafers (transport plates). The RF plasma was capacitively coupled by 13.56 MHz generator with supporting ICP generator (13.56 MHz). The power values of RF generator was ranging from 50 to 150 W and the power values of ICP generator were changed from 300 to 800 W.

2.2. Measurements

The initial values and the evolution of the DC bias during the deposition processes were measured by the integrated DC voltmeter and recorded in the log view of system main software. OES spectra were acquired in the SpectraSuite software for different plasmas (deposition and cleaning process) by the CCD spectrometer (200–900 nm) manufactured by Ocean Optics Company. The calculation of the relationship between sp^3 fraction content and sp^2 fraction content in the deposited films was based on Raman spectra analysis. The changes in film surface morphology were observed by AFM "tapping mode" investigation.

3. Results and discussion

It follows from the study that the RF power set as a parameter of a deposition process has a crucial effect on the DC bias values (Fig. 1), whereas the appropriate procedure of chamber cleaning ensures a repeatability of the process. ICP power influences in a minor degree the value of DC bias (Fig. 2). Experimental data were fitted by the linear approximation (method of least squares).

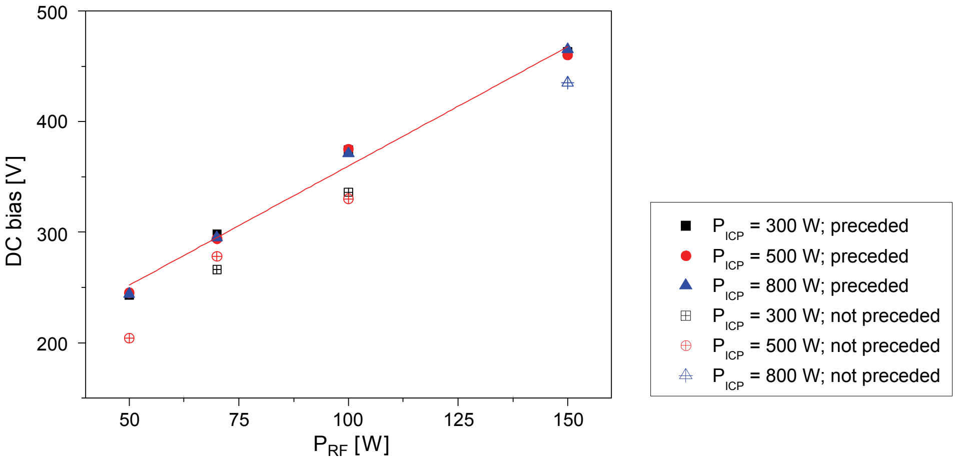

Fig. 1. Initial values of DC bias in deposition process of DLC layers as a function of the applied RF generator power.

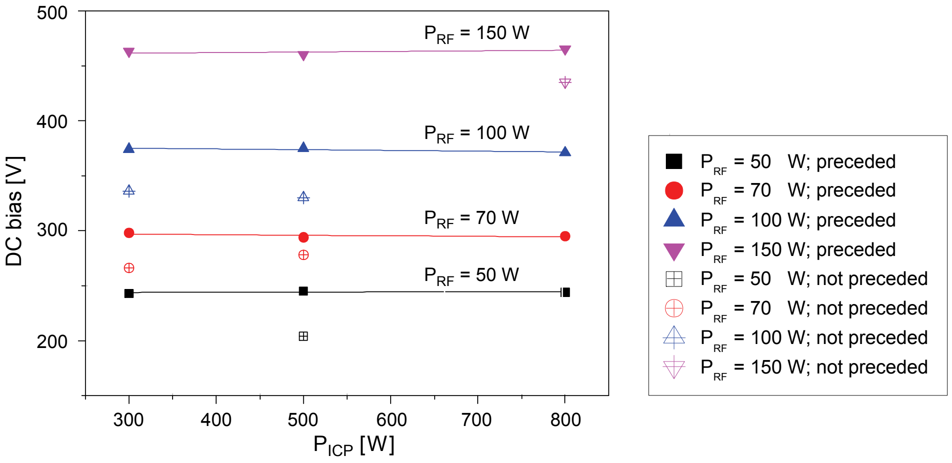

Fig. 2. Initial values of DC bias in deposition process of DLC layers as a function of the applied ICP generator power. Full marks – deposition process preceded by the chamber cleaning process; hollow marks – not preceded.

The initial value of the DC bias differs significantly depending on whether the DLC deposition process is conducted directly after the chamber cleaning process in oxygen plasma or it is a subsequent deposition process (Fig. 3).

In the acquired emission spectra, the presence of the same types of excited particles and ions (H_α, H_β, H_γ, H_δ, H_2, H^+, CH, CH^+, C_2) is observed [4]. Their activity results in the growth of the DLC films with quality demanded in the new applications and electronic devices. The increase in RF and ICP generators power values results in

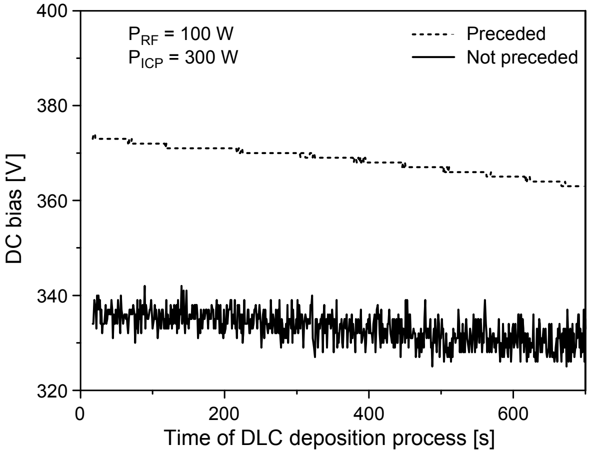

Fig. 3. Changes in DC bias value during the deposition process which was preceded and not preceded by the chamber cleaning process.

the increase in spectral lines intensity. The activity of the particles, present in the discharge, is rising with the ICP power value. The observed feature confirms that high density plasma, which is inductively coupled by the ICP generator, greatly increases the effectiveness of the ionization process. The improvement in the quality of the deposited films in these conditions, is confirmed by the investigation of the samples with Raman spectroscopy. The intensity of oxygen and other gases peaks and the background level of OES diagnostics by CCD spectrometer in the processing chamber cleaning process were used to determine the quality of the processing chamber "purity level", which means the thickness of the deposited layer on the working chamber walls during previous deposition processes (Fig. 4).

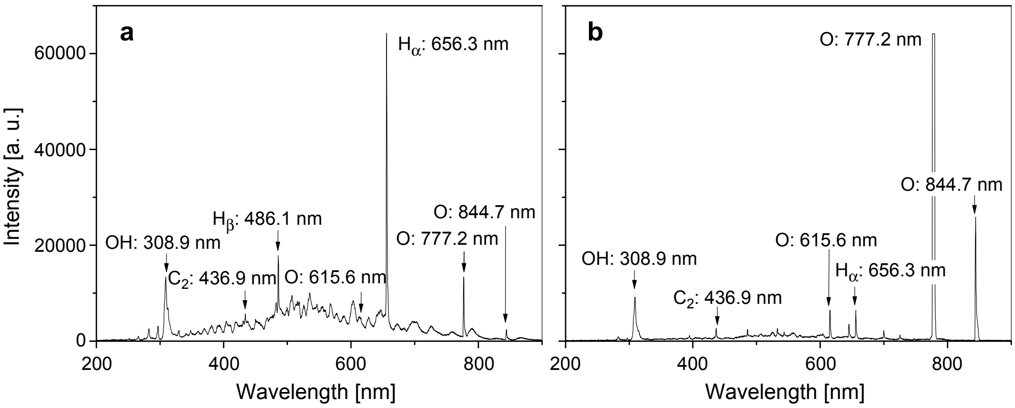

Fig. 4. OES spectra of the chamber cleaning process in oxygen discharge: beginning of the chamber cleaning process (**a**), and chamber cleaned (**b**).

The quality of DLC layers deposited on transport plate (2″ silicon wafer) and/or on silicon (111) substrate (10 mm×10 mm) was examined by Raman spectroscopy. In each deposition process, the silicon square substrate was located in the middle of the transport plate. Raman spectra were measured in the middle of the investigated substrate and around its edges. The growth of DLC film during the deposition process occurred at different planes (on the top of the substrate and on the top of the transport plate). Acquired Raman spectra were measured both on the square silicon substrate and on the transport plate. Basing on the acquired Raman spectra, the theoretical content of sp^3 bonds in the structure of the film was estimated. The content is ranging from 30% to 65% and depends on ICP PECVD deposition process parameters. For the film deposited during the same process, the change in the sp^3 content across the square substrate and the transport plate, is shown in Fig. 5.

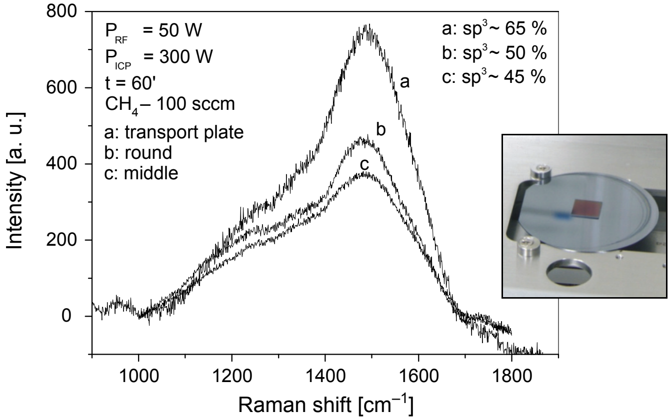

Fig. 5. The changes in sp^3 content in the deposited layers in different locations of the substrate.

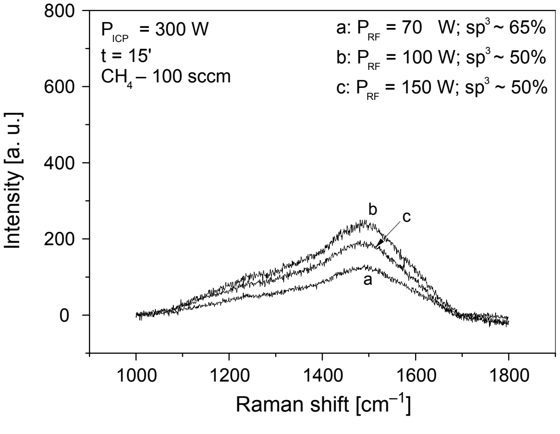

Fig. 6. The influence of RF generator power value on the acquired Raman spectra (middle of the sample).

Figure 6 shows the influence of RF generator power value on the intensity of the acquired Raman spectra from the middle of the substrate. In the constant ICP power value conditions (P_{ICP} = 300 W), the increase in RF generator power value from 70 to 100 W results in the increase in the intensity of *D* and *G* peaks. The increase in peaks intensity is not correlated with the increase in sp^3 content in the film. On the contrary, the increase in RF power value resulted in the reduction of sp^3 content from 65% to 50%. For RF generator power value set at 150 W, the decrease in spectrum intensity was observed, but with preserved relationship between peaks intensity. Due to an unchanged relationship between the intensity of *D* and *G* peaks, the relationship between sp^3 content to sp^2 content in the sample is also unchanged. The observed tendencies are consistent with subject literature [1, 2]. The process of the film growth in the investigated system by PECVD method should be carried out at the lowest RF discharge power value at which the ignition of discharge is still possible.

In the investigated range of deposition process parameters, in which the ignition of stable plasma discharge is possible, the surface roughness (minimal value R_a = 0.1 nm, RMS = 0.14 nm) (Fig. 7), measured in the center of the sample improved

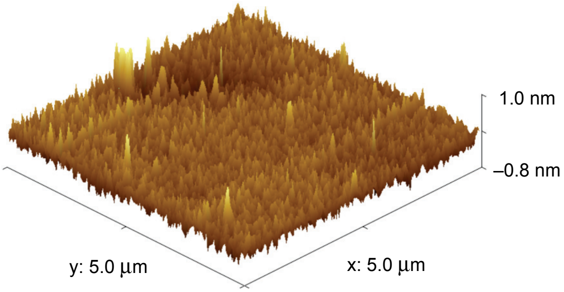

Fig. 7. AFM image of DLC film deposited at P_{RF} = 70 W and P_{ICP} = 300 W with sp^3 ~ 65%.

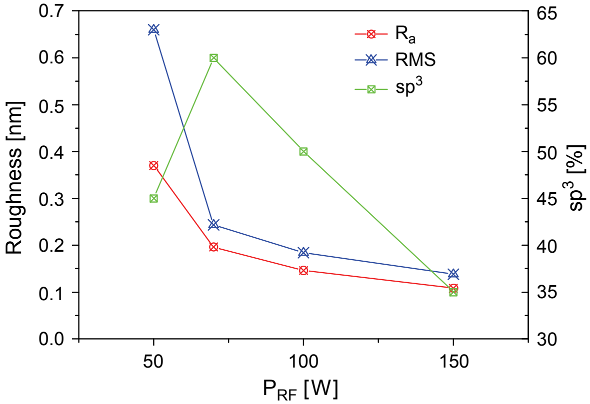

Fig. 8. Surface roughness and sp^3 content of DLC layers deposited at different RF generator power values in ICP PECVD.

with an increasing RF power value (Fig. 8). For DLC layers deposited at RF generator power value above 70 W the significant decrease in sp^3 content is observed.

4. Conclusions

Results of the investigation can be summarized in the following way:

– Deposition process of DLC layers must be carried out in conditions of stable plasma discharge depended on total gas pressure, gaseous precursor flow rates, RF power and ICP power.

– In order to achieve the repeatability of technology, it is necessary to ensure the same initial conditions of the DLC deposition process by the chamber cleaning process in O_2 discharge.

– In DLC deposition process, the RF power value has the most important influence on DC bias value, which cannot exceed the value of 500 V.

– Chemical reaction rates depend on location of the substrate surface relative to the plasma volume. Therefore, it is necessary to prepare individual procedures for 2″ silicon substrate and for substrates with different shapes and surface area.

– The growth of the DLC layers with good quality surface and the highest sp^3 content occurs at RF power value set on 70 W.

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