

# Plasmonic properties of silver nanoparticles embedded in diamond like carbon films: Influence of structure and composition

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## ABSTRACT

In the present study optical properties of hydrogenated diamond like carbon nanocomposite films containing silver nanoparticles (DLC:Ag) deposited by direct current (DC) unbalanced reactive magnetron sputtering were studied in 180–1100 nm range. Different substrate bias was used during deposition of the films. Structure of the films was investigated by multiwavelength Raman scattering spectroscopy and X-ray diffractometry (XRD). Chemical composition of the samples was studied by X-ray photoelectron spectroscopy (XPS), surface morphology was investigated by atomic force microscopy (AFM). Red shift of the surface plasmon resonance peak of DLC:Ag films with the increase of Ag atomic concentration was observed. It was found that high atomic concentration of oxygen in DLC:Ag films results in some redshift of the plasmonic peak, too. Such a behavior is explained by increase of the refractive index of the dielectric medium surrounding silver nanoparticle due to possible presence of the silver oxide interlayer at the Ag nanocluster and diamond like carbon matrix interface. It was demonstrated that influence of the increased Ag atomic concentration on position of the surface plasmon resonance peak of DLC:Ag films clearly prevails influence of the increased  $sp^3/sp^2$  ratio of the diamond like carbon matrix. Correlation between the structure of Ag nanocrystallites studied by XRD and position of the surface plasmon resonance peak position was observed.

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## 1. Introduction

Nanoparticles of the group I metals such as silver and gold received a huge interest of researchers due to strong surface plasmon resonance in these materials. Possible applications of these nanomaterials include such important and diverse areas as super-lenses [1], 3D plasmon rulers, which can be used to measure nanoscale distances [2], plasmonic nanoantennas [3], waveguides [4] and other elements of optical nanocircuits, selective photodetectors of the increased sensitivity [5], nanolithography [6], photocatalysts [7], photothermal therapy of cancer [8], plasmon lasers and light emitting diodes [4,9], advanced light trapping schemes for solar cells [4].

Silver nanoparticles as a plasmonic material have some advantages over gold nanoparticles due to higher intensity of the surface plasmon resonance [10], lower optical losses [11], and larger solar

energy conversion efficiencies [12]. However silver nanoparticles are prone to rapid surface oxidation when exposed to ambient conditions [13]. Stability of the silver nanoparticles can be increased by using ultra-thin protective coating [13–15]. Particularly nanocomposites containing silver nanoparticles can be used for such a purpose. Above all diamond like carbon is very good candidate as a matrix for plasmonic nanoparticles. Diamond like carbon (DLC) itself is amorphous nanocomposite consisting of the  $sp^2$  bonded (graphite-like) carbon nanoclusters embedded into the  $sp^3$  bonded (diamond-like) carbon matrix [16–18]. Two main groups of the diamond like carbon films can be distinguished: hydrogen containing (hydrogenated) and hydrogen-free DLC films [16–18]. DLC films received significant attention of the researchers and industry due to their unique properties such as high hardness, Young's modulus, low friction coefficient, wear resistance, corrosion resistance and biocompatibility [16,17]. Electrical and optical properties of the diamond like carbon films vary in a wide range and depend on the deposition techniques and conditions [16,17]. Using variety of plasma based methods of deposition, diamond like carbon films containing different metals can be deposited. Particularly diamond like carbon films containing silver nanoparticles (DLC:Ag) can be

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mentioned due to some interesting features. When silver atomic concentration in the film is more than 1–2 atomic percents (according to [19]), due to silver segregation silver nanoclusters embedded in the diamond like carbon matrix were detected [19–21]. Taking into account surface plasmon resonance in DLC:Ag films [22–26] and other properties of diamond like carbon films containing silver nanoparticles, such as hardness that is close to the hardness of the undoped DLC film [19], high wear resistance [27], antibacterial activity [28] and good haemo-compatibility [21] these films are prospective material for fabrication of different plasmonic devices. There are numerous studies on structure and composition of DLC:Ag films [19,22–35]. However optical properties of DLC:Ag films are investigated far less [22–26,29]. In [24] a broad absorption peak with maximum in 400–500 nm range was observed for hydrogen free DLC:Ag films deposited by pulsed laser deposition. In the case of the electrochemically deposited hydrogenated DLC:Ag films, surface plasmon resonance peak was observed in 650–750 nm range [23]. Position of the peak shifted to the higher wavelengths with the increase of silver atomic concentration in the films. For hydrogenated DLC:Ag films deposited by unbalanced DC reactive sputtering surface plasmon resonance related absorption peak was observed for the films with Ag amount higher than 5–6 at% and it was in 400–500 nm range [26]. However no surface plasmon resonance was observed in the case of the hydrogenated DLC:Ag films deposited by reactive radio frequency sputtering, despite that for some of the films Ag atomic concentration as high as 12 at.% was reported [29]. Thus data on the optical properties of DLC:Ag films and particularly on presence of the surface plasmon resonance phenomena are rather contradictory. There is lack of systematic studies on possible relation between the optical properties of DLC:Ag films and their structure and composition. In addition, influence of some key technological parameters on optical properties of DLC:Ag films was not studied till now. Particularly ion bombardment during film growth is essential for deposition of the diamond like carbon films and optimum ion energy must be set to achieve high  $sp^3/sp^2$  bond ratio [16]. However no studies on influence of ion energy (substrate bias) on optical properties of DLC:Ag films was performed till now.

Therefore in the present study the influence of the composition and structure on optical properties of hydrogenated DLC:Ag films deposited by reactive DC unbalanced magnetron sputtering was investigated. The effects of ion energy on optical properties of the deposited diamond like carbon layers containing silver nanoparticles were studied.

## 2. Experimental techniques

In the present study hydrogenated DLC:Ag films were deposited by DC unbalanced reactive magnetron sputtering of silver target. The diameter of magnetron was 3 in. Monocrystalline silicon and quartz substrates were used. Mixture of the hydrocarbons (acetylene) and argon gas was used in the reactive magnetron sputtering. In all experiments substrate–target gap was set at 10 cm, magnetron target current was 0.1 A, base pressure was  $5 \times 10^{-4}$  Pa and work pressure was  $(4 \pm 1) \times 10^{-1}$  Pa and thickness of the films was 100 nm. Negative substrate bias was varied in 0–200 V range. Substrates with no additional bias were grounded. Two sets of the samples containing different amount of silver were prepared by using different argon and acetylene gas flux ratios. Gas flux ratios were selected by using data of our previous experiments on unbiased deposition of DLC:Ag films [26]. For the first set of samples argon gas flux was 70 sccm and acetylene gas flux was 11.7 sccm (high  $C_2H_2/Ar$  ratio set, further – HC set). In such a case using no substrate bias, DLC:Ag films containing >20 at.% of silver can be deposited according to [26]. Samples of the HC set deposited by

**Table 1**

Deposition conditions of DLC:Ag films and name of the samples.

Sample name	$C_2H_2$ flux (sccm)	Ar flux (sccm)	Substrate bias (V)
LC0	7.8	80	0
LC50	7.8	80	–50
LC100	7.8	80	–100
LC150	7.8	80	–150
LC200	7.8	80	–200
HC0	11.7	70	0
HC50	11.7	70	–50
HC100	11.7	70	–100
HC150	11.7	70	–150
HC200	11.7	70	–200

using 0, 50, 100, 150, 200 V negative substrate bias are called as HC0, HC50, HC100, HC150, HC200 respectively. For the second set of samples Ar gas flux was 80 sccm and  $C_2H_2$  gas flux was 7.8 sccm (low  $C_2H_2/Ar$  ratio set, further – LC set). In such a case using no substrate bias, DLC:Ag films containing several atomic percents of silver can be grown [26]. Samples of the LC set deposited by using 0, 50, 100, 150, 200 V negative substrate bias are called as LC0, LC50, LC100, LC150, LC200 respectively. Deposition conditions of the samples are summarized in Table 1.

Optical properties of the films were investigated using an optical spectrometer Avantes that is composed of a deuterium halogen light source (AvaLight DHc) and spectrometer (Avaspec-2048). The absorbance of the films was analyzed in the wavelength region from 180 nm to 1100 nm.

Chemical composition of the films was established by recording X-ray photoelectron spectra with the KRATOS ANALYTICAL XSAM800 X-ray photoelectron spectroscopy (XPS) analyzer with  $Al K_{\alpha}$  radiation ( $h\nu = 1486.6$  eV) and by calculating atomic concentrations using original KRATOS XSAM800 software.

Raman measurements were performed with 325 nm, 442 nm, 532 nm and 633 nm excitations using the Raman microscope inVia (Renishaw). Dispersion of the G peak (Disp(G)) was calculated as a slope of the G peak position versus excitation wavelength graph. It is known that Disp(G) linearly increases with  $sp^3/sp^2$  carbon bond ratio [36,37]. Therefore this parameter of the Raman scattering spectra was used as a measure of structural quality of the diamond like carbon matrix of the DLC:Ag nanocomposite films.

Structure of the crystalline silver nanoclusters has been studied by X-ray diffractometer D8 Advance (Bruker AXS, Germany). Grazing incidence angle arrangement combined with parallel beam X-ray diffraction geometry was used. Multilayer Ni/graphite parabolic monochromator was placed in front of the sample. X-ray diffraction patterns were recorded using Cu cathode at 40 kV, anode current 40 mA, scanning step  $\Delta 2\theta = 0.04^\circ$ , average time of integration 15 s. Size of the silver nanocrystallites was defined by using Halder–Wagner graphic method using Ag(1 1 1) peak.

The morphology of the surface was analyzed with AFM NanoWizard®3 (JPK, Germany) working in AC Mode. Silicon probes with a reflective backside Al coating (ACTA-10, APPNano, USA) with a resonance frequency of 200–400 kHz and force constant of 13–77 N/m were used. Nominal tip radius was less than 10 nm. The scanning rate of 0.8 Hz was selected.

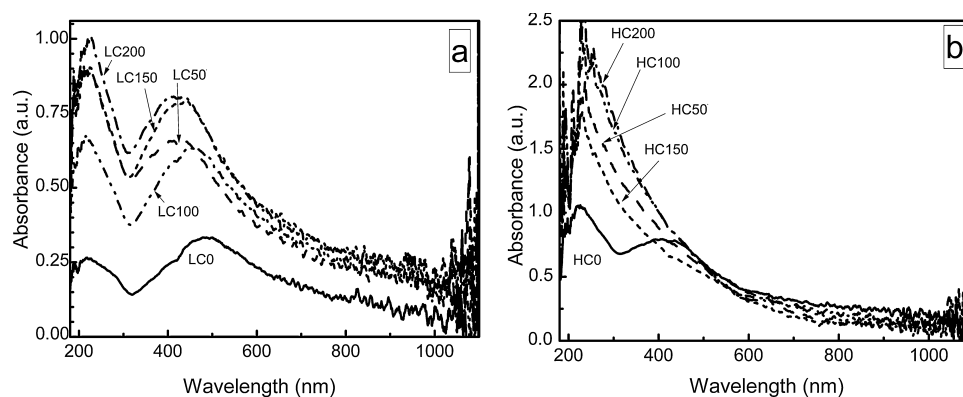
## 3. Experimental results and discussion

Summary of the parameters describing structure and composition of the DLC:Ag films with indication of the presence (absence) of the plasmonic peak (in 300–600 nm range) in the absorption spectra of the films is presented in Table 2. It can be seen, that Disp(G) increases and Ag atomic concentration decreases with the increase of substrate bias. Typical optical absorption spectra of DLC:Ag films deposited using different deposition conditions are presented in

**Table 2**

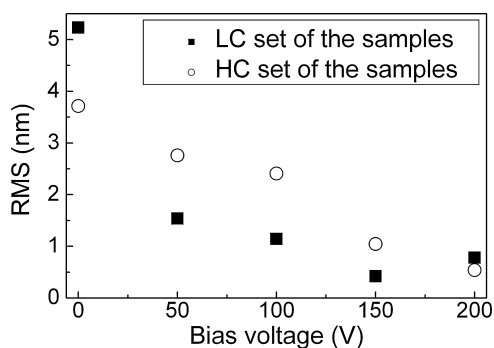
Structure and composition of DLC:Ag films and presence (absence) of the surface plasmon resonance peak.

Sample name	$\lambda_{\text{plasmonic peak}}$ (nm)	Disp(G)	Ag at. conc. (%)	O at. conc. (%)	C at. conc. (%)
LC0	Yes	0.100	22.0	11.3	66.7
LC50	Yes	0.226	8.2	10.7	81.2
LC100	Yes	0.196	15.2	7.4	77.5
LC150	Yes	0.251	4.5	7.7	87.8
LC200	Yes	0.210	2.9	7.4	89.7
HC0	Yes	0.233	3.1	8.6	88.3
HC50	–	0.243	3.1	8.2	88.6
HC100	–	0.277	0.3	5.9	93.8
HC150	–	0.266	0.3	5.7	94.0
HC200	–	0.220	0.2	5.8	94.1

**Fig. 1.** Optical absorbance spectra of DLC:Ag films deposited using different negative bias voltage (0, 50, 100, 150, 200 V): LC set (a), HC set (b).

**Fig. 1.** In the case of LC set of samples, surface plasmon resonance related absorption peak is seen for all layers investigated. While in the case of HC set of samples, surface plasmon resonance related absorption peak can be observed only for DLC:Ag film deposited without substrate bias (grounded substrate). Plasmonic peak is absent as well in the absorption spectra of the films containing low amount of silver (Table 2 and Fig. 1). The critical atomic concentration of Ag when plasmonic peak started to be registered was  $\sim 3$  at.%. For some films containing such an amount of silver surface plasmon resonance peak was detected in the optical absorbance spectra (Fig. 2), while for some it was absent. No correlation between the Disp(G) of the films and presence (or absence) of the plasmonic peak was observed.

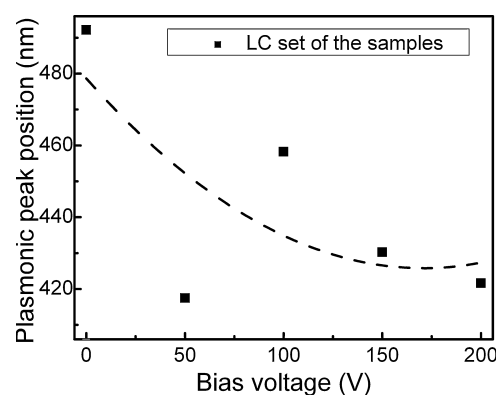
**Fig. 2** illustrates that for both sets of the DLC:Ag films surface roughness (RMS) decreases with the increase of the substrate bias i.e. increase of ion beam energy during DLC:Ag film growth results in decrease of the surface roughness. The observed trend is similar to the results reported in [38], where it was shown that DLC films deposited by using low energy ions exhibited substantially higher roughness than those deposited by using high energy ions.

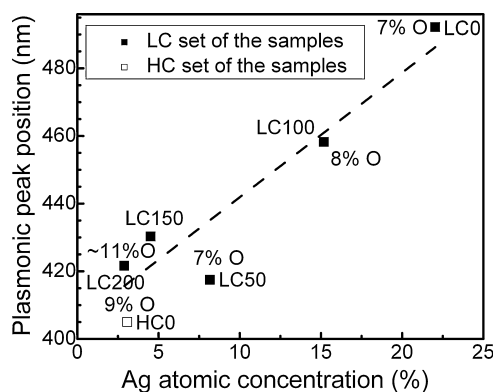
**Fig. 2.** The dependence of DLC:Ag film surface roughness on negative substrate bias.

According to [38] ion impingement energies higher than  $\sim 50$  eV tend to lead to smoother surfaces because the ions become implanted beneath the surface, where the impact energy is less efficient in promoting surface diffusion. However, no correlation between the absence or presence of the surface plasmon resonance peak and surface roughness of DLC:Ag films was observed.

To describe plasmonic properties of silver nanoparticles in DLC, complex approach is needed. Position of the plasmonic peak can be analyzed in terms of the technological conditions either in terms of the resultant composition and structure of the films.

Some blue shift of the plasmonic peak with the increase of the substrate bias (for low  $C_2H_2/Ar$  ratio, LC set of the samples) can be seen in Fig. 3. It should be mentioned that blueshift of the plasmonic peak position with negative substrate bias is nonmonotonic. Nonmonotonous decrease of Ag atomic concentration in film with substrate bias can be seen, too. Such a behavior can be explained by competition between DLC:Ag film growth and sputtering effects. DLC:Ag films are nanocomposites. Thus different sputtering yield of Ag and hydrogenated amorphous carbon matrix should be taken

**Fig. 3.** Surface plasmon resonance peak position vs negative substrate bias voltage.

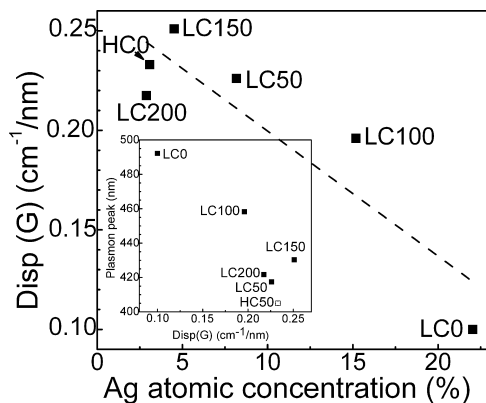


**Fig. 4.** The dependence of the surface plasmon resonance peak position of DLC:Ag on silver atomic concentration and oxygen content in the films.

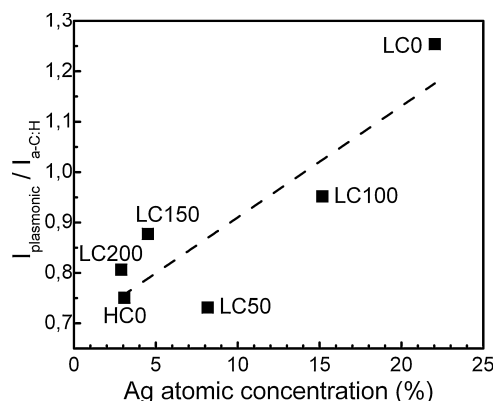
into account [39]. It must be taken into account that structure of the diamond like carbon matrix depends on substrate bias (ion energy):  $sp^3/sp^2$  bond ratio is maximal at the optimum ion energy, while hydrogen atomic concentration usually decreases with the increase of ion energy [16]. That is additional variable. Thus sputtering rate of DLC:Ag film can depend on ion energy in complicated way. It can result in nonmonotonic dependence of the film composition and properties on ion energy.

The dependence of the plasmonic peak position on the resultant chemical composition (silver, oxygen concentration) and structure of the DLC:Ag films ( $sp^3/sp^2$  bonding) is substantially more pronounced and steady. On the other hand, this kind of information (dependence on the composition) is more general and it can be applied for the films deposited using different technologies.

Following this approach one can observe that plasmonic peak shifts to the higher wavelengths with the increase of Ag atomic concentration (Fig. 4). This shift has a quasilinear character. Position of the plasmonic peak is sensitive to the oxygen concentration too. In the case of high atomic concentration of oxygen in DLC:Ag films, some redshift of the plasmonic peak was detected. It should be mentioned, that in [40] oxidation of the silver nanoparticles embedded into the silica films resulted in red shift of the surface plasmon resonance peak, too. According to [41,42] refractive index of  $Ag_2O$  ( $>2.8$ ) is higher than refractive index of the hydrogenated diamond like carbon ( $<2.8$  according to [43]). In such a way, the registered redshift of the plasmonic peak of DLC:Ag films containing high amount of oxygen can be explained by oxidation of silver nanoparticles and resultant increase of the refractive index of the dielectric medium surrounding the metallic silver nanoparticles.



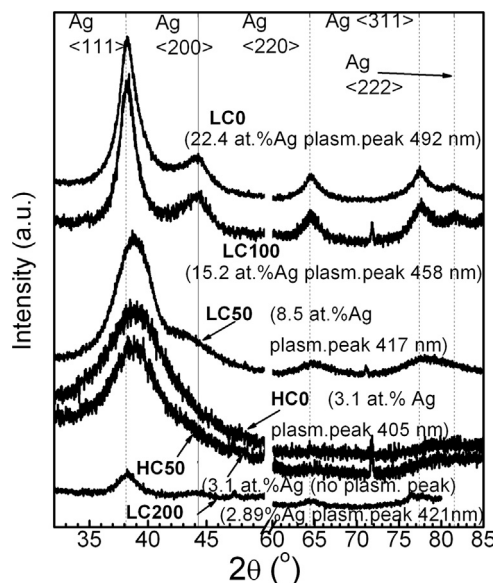
**Fig. 5.** The dependence of Disp(G) of DLC:Ag films on Ag atomic concentration. The inset shows the dependence of the surface plasmon resonance peak position on Disp(G) of DLC:Ag films.



**Fig. 6.** Dependence of intensity of the plasmonic peak normalized to a-C:H absorption peak intensity vs Ag atomic concentration in DLC:Ag film.

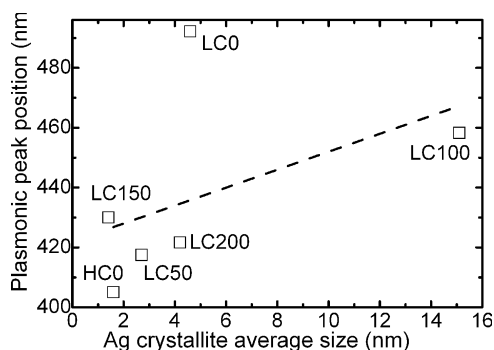
Influence of the structure of the diamond like carbon matrix on position of plasmonic peak was studied recording Raman scattering spectra of DLC:Ag films. However, such a study was complicated due to different amount of silver in the investigated DLC:Ag films. In can be seen in Fig. 5, that Disp(G) decreased with the increased of Ag atomic concentration. According to [36,37] Disp(G) increases with  $sp^3/sp^2$  ratio in DLC films. It is known as well that refractive index of DLC films increases with  $sp^3/sp^2$  ratio [44]. In such a way, one can expect that refractive index of diamond like carbon matrix of DLC:Ag films will increase with Disp(G) and one could expect red shift of the surface plasmon resonance peak. In our experiment, we have the opposite trend (see Fig. 5 inset). It seems that influence of the increased Ag atomic concentration prevails over the influence of the increased  $sp^3/sp^2$  ratio (increased refractive index) of the diamond like carbon matrix. As a result red shift of the plasmonic peak was detected even for the films with high  $sp^3/sp^2$  ratio (see inset of Fig. 5).

Analyzing intensity of the surface plasmon resonance peak (normalized to a-C:H matrix related peak at  $\sim 215$ – $225$  nm) one can see (Fig. 6) that it in the case of high concentrations of Ag it increases with the increase of Ag atomic concentration. In the case of the Ag concentrations below 5 at.% no clear dependence was found.



**Fig. 7.** XRD diffractograms of DLC:Ag films and position of the surface plasmon resonance (SPR) peak. In all cases linear intensity scale was used.





**Fig. 8.** The dependence of surface plasmon resonance peak position on Ag crystallite size.

These results allow to assure that plasmonic properties of DLC:Ag are defined by both concentration and crystalline structure of Ag nanocrystallites. To check this assumption we performed the XRD analysis of the nanocomposite films. X-ray diffractograms of the DLC:Ag films are presented in Fig. 7. It should be mentioned, that similar structure of the silver crystalline phase was reported by other authors for hydrogenated DLC:Ag films deposited by reactive magnetron sputtering [20,34]. In our study we were trying to correlate plasmonic properties of nanocrystalline silver with the structural measurements results. According to Fig. 7 in all cases Ag(1 1 1) related XRD peak was detected. XRD peaks related to other orientations of silver were substantially weaker, while for some samples only Ag(1 1 1) related peak was seen. These observations are summarized in Fig. 8 where plasmonic peak position versus average Ag(1 1 1) crystallite size evaluated according to Sherrer equation is plotted. One can see that shift of the plasmonic peak position to the higher wavelength with the average crystallite size calculated using Ag(1 1 1) peak can be observed.

#### 4. Conclusions

In conclusion plasmonic properties of DLC:Ag films deposited by DC unbalanced reactive magnetron sputtering were investigated by analyzing absorbance spectra in 180–1100 nm range. Redshift of the surface plasmon resonance peak of DLC:Ag films with the increase of Ag atomic concentration were observed. Higher atomic concentration of oxygen in DLC:Ag films resulted in oxidation of Ag and contributed to some redshift of the plasmonic peak. Influence of the increased Ag atomic concentration on position of the surface plasmon resonance peak of DLC:Ag films clearly prevailed over influence of the increased  $sp^3/sp^2$  ratio of the diamond like carbon matrix. Ratio of intensities of the surface plasmon resonance peak and a-C:H matrix related peak at ~215–225 nm increased with Ag atomic concentration in the case of DLC:Ag films containing higher amounts of the silver (>5 at.%). In the case of the Ag concentrations below 5 at.% no clear dependence of the surface plasmon resonance peak intensity versus Ag concentration was found. Tendency of the shift of plasmonic peak position to the higher wavelength with the average Ag crystallite size was found.

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