The Investigation of Piezoresistive, Optical and Electrical Properties of Diamond Like Carbon Films Synthesized by Ion Beam Deposition and PECVD

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In the present research ion energy effects on piezoresistive properties of hydrogenated diamond like carbon (DLC) films deposited by hydrocarbon ion beam deposition and radio frequency plasma enhanced chemical vapor deposition (PECVD) were studied. Comparative analysis of the influence of silicon oxide and silicon doping on gauge factor of DLC films was performed. Only weak dependence of piezoresistive properties of the DLC films deposited from acetylene gas by ion beam deposition using closed drift ion source on ion energy was observed and that was explained as a result of the competing effects of ion energy and ion/neutral ratio during the diamond like carbon film deposition process. In the case of PECVD deposited DLC films the dependence of gauge factor on substrate bias was observed. Doping by silicon and silicon oxide resulted in decrease of the DLC film gauge factor. Possible relations between the gauge factor of DLC films and their optical parameters such as optical bandgap, Urbach energy and B parameter were studied as well. It was found, that the gauge factor increases with the increased separation between valence and conduction π band in DLC films and/or decrease size of sp^2 cluster as well as with the decrease of the topological disorder (range in which vary size of sp^2 clusters) and/or density of sp^2 states and ammount of sp^2 bonded carbon phase. Resistivity of the investigated DLC films deposited by both methods decreased with the increase of the ion energy and no correlation between the piezoresistive properties of DLC films and their resistance was found. The observed behavior was explained by dependence of resistivity on hydrogen ammount in DLC films.

Keywords: diamond like carbon films, piezoresistance, optical properties.

1. INTRODUCTION

Diamond like carbon (DLC) is a metastable form of the amorphous carbon where big part of the carbon atoms is connected by sp^3 bonds similarly to diamond [1]. DLC films received considerable interest due to their exceptional properties such as high hardness and wear resistance, low friction coefficient, chemical inertness [1]. Despite significant efforts, diamond like carbon seems less promising as an electronic material due to high density of the defect related states [2]. Yet fabrication of the diamond like carbon based resonant tunnel diodes operating at microwave frequencies range [3, 4] as well as application of DLC for electrically active passivation of high power electronics devices can be mentioned [5]. However, diamond like carbon appears to be very attractive material for micromechanical sensor fabrication due to the recently found significant piezoresistive effect [6 – 11], possibility to make diamond like carbon free standing films (membranes) of the different shape [2, 11 – 14], exceptional mechanical properties [1] and chemical inertness [1] as well as room temperature synthesis possibility [1]. Despite the potential opened by discovery of the piezoresistive effect in DLC there are only few studies on piezoresistive properties of diamond like carbon films [6 – 11]. Possible relations between the piezoresistive properties and electronic structure of the diamond like carbon are still poorly investigated. Study of the optical absorption spectra of diamond like carbon films can

provide some additional information on electronic structure of that material. Particularly, the optical bandgap calculated according to the Tauc equation is a measure of the gap between the extended state in the valence band and conduction band [15]. In analogy to a-Si:H, the slope of the Tauc plot (*B* parameter) and Urbach energy (E_U), are usually determined for a-C:H films as well. In hydrogenated amorphous silicon these parameters are related to the band tail width and structural disorder of the films – B parameter decreases and Urbach energy increases with increased disorder [16 – 18]. In such a case *B* parameter is supposed to be related with the conduction-band-tail width [16, 17] and Urbach tail energy with the valence-band-tail width [18]. In the case of diamond like carbon, there are both sp^3 and sp^2 bonded atoms, not only sp^3 as it is in the case of silicon [1]. Optical properties of diamond like carbon are mainly determined by sp^2 clusters and, particularly sp^2 phase related π orbital [1], because π-electronic states are less spaced in energy than the sp^3 phase related σ states [19]. Therefore, there are different opinions on meaning of the optical parameters mentioned above in the case of the diamond like carbon. According to [1] optical bandgap is a measure of the separation between valence and conduction π band, while Urbach energy is a measure of the inhomogenous disorder in two phase (sp^3 and sp^2 bonded carbon) network. It (Urbach energy) measures range of sp^2 clusters present (topological disorder) [1]. On the other hand it was suggested in [19, 20], that in the case of DLC the Tauc gap should be associated not with the mobility edge as in a-Si:H, but with the number of rings forming the typical sp^2 aromatic cluster of a given DLC. According to that model, Urbach

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energy is not related to the topological disorder but rather to the Gaussian width of density of states of π band, peaked at ±$E\pi$ energies above/below the Fermi level [19, 20]. In other words, it is a function of the density of π states and percentage of sp^2 phase in DLC film [20]. In any case, information provided by optical parameters of diamond like carbon is interesting for study of piezoresistive properties of DLC films, because according to [7], piezoresistive properties of DLC depend on dimensions of the sp^2 bonded carbon clusters as well.

In the present study piezoresistive effect in different hydrogenated diamond like carbon films produced by two methods: direct ion beam and radio frequency PECVD was studied. Possible correlation between the optical parameters of DLC and piezoresistive properties was investigated. Resistance of DLC films was investigated as well to reveal possible relations with the piezoresistive gauge factor.

2. EXPERIMENTAL

Hydrogenated diamond-like carbon films were prepared using two different deposition methods: DC hydrocarbon ion beam deposition by closed drift ion source and capacitively coupled parallel plate radio frequency (RF) plasma enhanced chemical vapour deposition (PECVD). In the first case, DLC films were deposited using acetylene (C_2H_2) gas, while in the case PECVD grown DLC films methane (CH_4) gas was used as a source of carbon and hydrogen. SiO_x and Si containing DLC films were deposited by a closed drift ion source from hexamethyldisiloxane (HMDSO) vapour and from mixture of the hexamethyldisiloxane vapour and acetylene gas respectively. Chemical composition of these samples is presented in Table 1.

Table 1. Chemical composition of the DLC films deposited by closed drift ion source from hexamethyldisiloxane (HMDSO) vapour and from mixture of the hexamethyldisiloxane vapour and acetylene gas (according to [21, 22])

Film (gases used for synthesis)	Atomic concentration (%)		
	Si	O	C
HMDSO (E_{ion} = 400 eV)	23	20	57
HMDSO+C_2H_2 (E_{ion} = 800 eV)	2	10	88

The deposition was performed at room temperature. Thickness of the synthesized films was in 150 nm – 300 nm range.

In all experiments monocrystalline silicon (Si), polycrystalline alumina and quartz substrates were used. Before the deposition all the wafers were cleaned by boiling in dimethylformamide and acetone. Diamond like carbon films deposited on silicon substrates were used for the thickness control. Samples used in the study of the piezoresistive properties were fabricated on polycrystalline alumina substrates. In some experiments SiO_x containing DLC interlayers (interlayer thickness 50 nm) deposited by 800 eV energy ion beam using mixture of hexamethyldisiloxane vapour and hydrogen gas were used. Quartz substrates were used in the study of optical properties of DLC films.

Piezoresistive properties of the diamond like carbon films were evaluated by four point bending test [14]. Gauge factor has been calculated as a measure of the piezoresistive properties of the films:

$$k = \frac{\Delta R}{R} \cdot \frac{1}{\varepsilon} , \qquad (1)$$

where R is the resistance of the sample, ε is the applied strain and ΔR is the change of the resistance as a result of the applied strain ε. The diamond like carbon piezoresistors were fabricated on polycrystalline alumina substrates using Al-based interdigitated finger-like electrodes. The distance between the electrodes was 100 μm. More information about the samples used for investigation of the piezoresistive properties can be found in [8].

Measurements of the electrical parameters of DLC films were performed by a picoammeter/voltage source Keithley 6487.

Optical properties of the films were investigated using an optical spectrometer Avantes that is composed of deuterium halogen light source (AvaLight DHc) and spectrometer (Avaspec-2048). The optical properties were analysed in the region of wavelength from 250 nm to 1000 nm. The optical bandgap (E_{Tauc}) and B parameter were estimated using Tauc plot based on the equation known for interband transitions in amorphous semiconductors $\sqrt{\alpha E} = B(E - E_{Tauc})$ [15, 23]. Urbach energy was determined using semilog plot of the absorption coefficient in the range from 10^3 cm^{-1} to 10^4 cm^{-1} [18].

Laser ellipsometer Gaertner 117 operating with a He-Ne laser (λ = 632.8 nm) was used for the estimation of the thickness and refractive index of the films.

3. EXPERIMENTAL RESULTS

The piezoresistive gauge factor of the DLC films synthesized by a closed drift ion source varied within 12 – 21 range for the used energies (300 eV – 800 eV). The significant scattering of the results between the samples deposited under the same conditions during the same technological process was observed. Slow decrease of the gauge factor with ion beam energy can be seen in Fig. 1. Silicon and silicon oxide doping resulted in decrease of the gauge factor. Introduction of 2 atomic percents of silicon (Fig. 1, Table 1) resulted in decrease of the gauge factor by more than 1.5 times (see data for DLC films deposited from acetylene and from mixture of the hexamethyldisiloxane vapor and acetylene gas). On the other hand, it can be seen in Fig. 1, that gauge factor of the DLC films deposited on SiO_x containing DLC interlayer was lower than the corresponding gauge factor of the film deposited directly onto the polycrystalline alumina substrate. Probably it can be due to silicon doping of growing DLC film. Further increase of the concentration of the dopants in DLC film resulted in further decrease of the gauge factor (Fig. 1 and Table 1). In this case changes of the structure of carbon matrix of SiO_x containing DLC film in comparison with the diamond like carbon film deposited from acetylene gas should be taken into account as well. For example, presence of transpolyacetylene related chains was reported for SiO_x containing DLC film in [22].
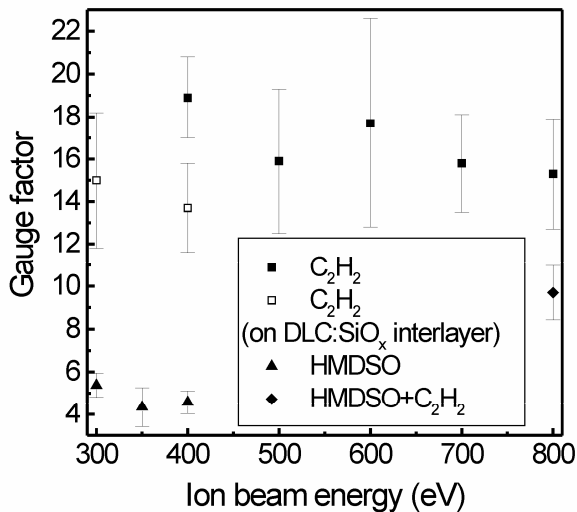
Fig. 1. The dependence of the gauge factor of DLC films synthesized by closed drift ion source on ion beam energy

Fig. 2. Bias effects on the piezoresistive properties of DLC films deposited by PECVD

Fig. 3. Gauge factor vs E_{opt}: DLC films synthesized by ion beam deposition (a) and PECVD grown DLC films (b)

Weak dependence of the gauge factor on ion energy for ion beam deposited DLC films was observed (Fig. 1) contrarily to the theories explaining piezoresistive effect in diamond like carbon films [7] and ion energy effects on structure of DLC films [1]. According to [7], DLC films are described as a composite of conductive sp^2 clusters in an insulating sp^3 matrix and piezoresistive effect occurs due to the changes of the distance between the clusters (and hence resistivity). Thus gauge factor of the DLC film should depend on sp^3/sp^2 ratio and size of sp^2 cluster – it should increase with the increase of sp^3/sp^2 ratio (sp^2 cluster size). On the other hand, according to [1], diamond like carbon films with the highest sp^3/sp^2 ratio are usually deposited at ~100 eV energy per carbon atom. The lowest ion energy per carbon atom used in present study is 150 eV and therefore, decrease of the gauge factor with the increase of ion beam energy should be expected. Such weak dependence of the piezoresistive properties observed in the present study can be explained by too low dependence of the size of sp^2 clusters (and sp^3/sp^2 ratio) on ion energy that is specific to the used deposition method and technique. Such an assumption can be supported by analysis of Raman scattering spectra measured with different exciting wavelength of the ion beam deposited DLC films as it was reported in [24]. In the case of the scattering spectra excited by 632 nm laser, which is relatively less sensitive to the variations of the sp^2 bonding configurations according to [24], no clear variation of the spectra parameters with ion beam energy was observed. Relatively significant variation of the Raman scattering spectra parameters with ion beam energy was observed only in the case of the Raman spectra excited by 785 nm laser that was non-monotonic – as it was demonstrated due to the competition between the effects of the ion energy and ion/neutral ratio [24].

It can be seen in Fig. 2 that the gauge factor of DLC films deposited from methane gas by PECVD method depends on the used bias voltage. The highest gauge factor (41 ±17) was observed for the diamond like carbon films deposited at –400 V bias, while gauge factor of other films was substantially lower and was in the 10 – 20 range –
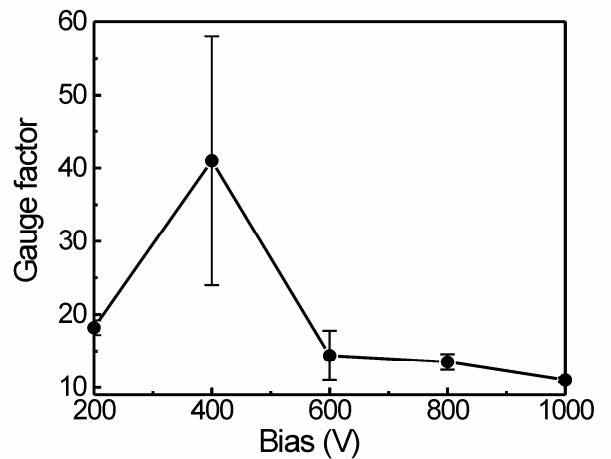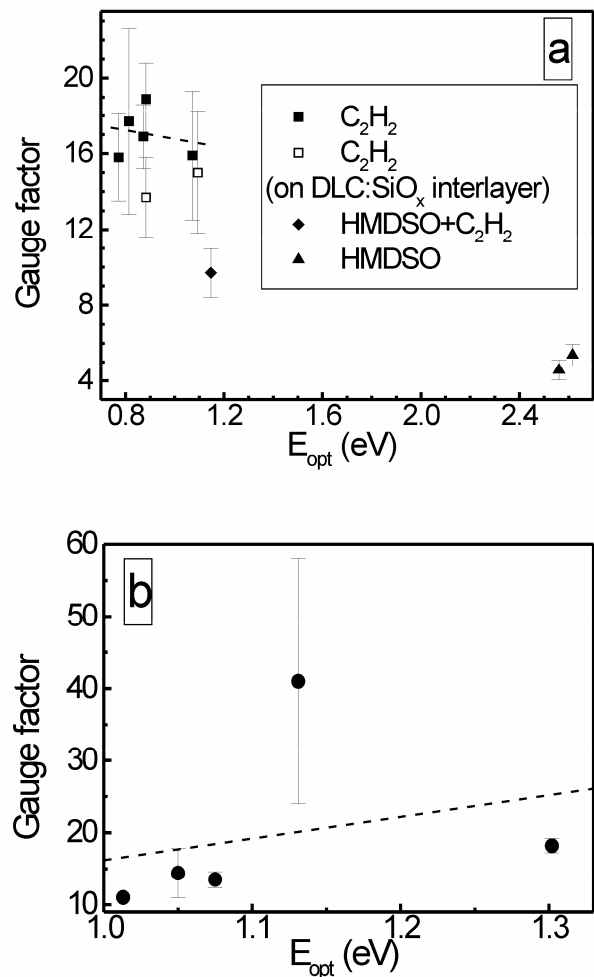
similarly to the DLC films deposited by closed drift ion source. It can be supposed, that at –400 V substrate bias DLC film with the highest sp^3/sp^2 bond ratio and smallest sp^2 clusters was formed. However, gauge factor of DLC films deposited at –400 V bias from methane gas was still lower than the highest gauge factor values reported for DLC films deposited by RF PECVD from acetylene and
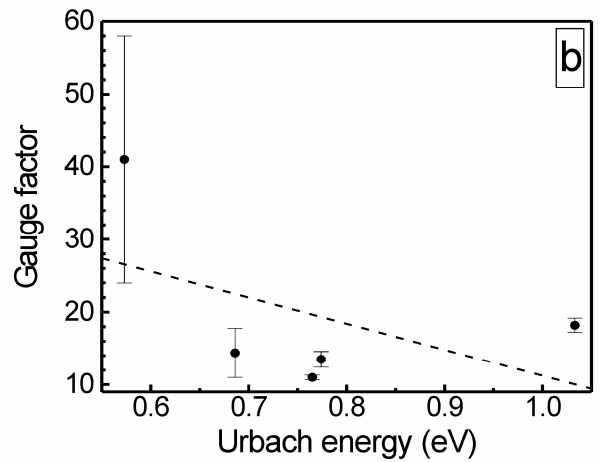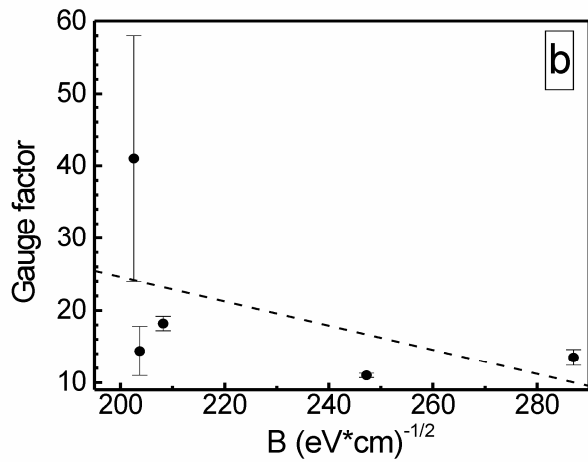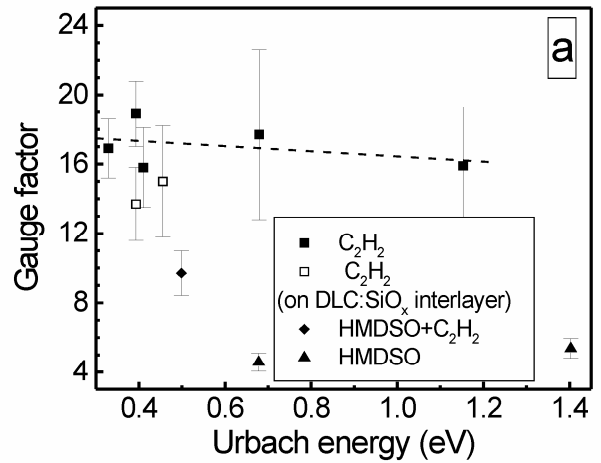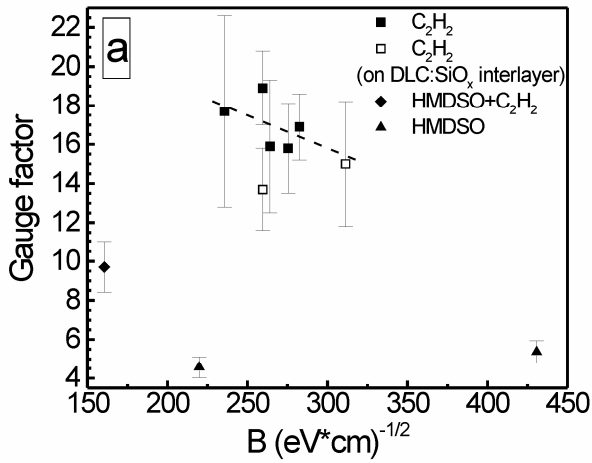
Fig. 4. Gauge factor vs B parameter: DLC films synthesized by ion beam deposition (a) and PECVD grown DLC films (b)

Fig. 5. Gauge factor vs Urbach energy: DLC films synthesized by ion beam deposition (a) and PECVD grown DLC films (b)

argon gas mixture at -350 V bias: $45-81$ in [7], $51-68$ in [9], $40-90$ in [10].

Piezoresistive properties of the synthesized DLC films were analyzed in parallel to the optical parameters calculated from the optical absorption spectra of DLC films (Figs. $3-5$). In the case of "conventional" hydrogenated DLC films both deposited from acetylene gas by closed drift ion source and from methane gas by PECVD, tendency of the gauge factor increase with the optical bandgap was observed (Fig. 3). It is in good accordance with the optical bandgap of DLC films increase with sp^3/sp^2 ratio reported by other authors [25, 26]. In such a way gauge factor increases with increased separation between valence and conduction π band in DLC films and/or decrease size of sp^2 cluster. It is in good accordance with the model describing piezoresistive effect in DLC films presented in [7]. However, analyzing possible relations between the piezoresistive properties of DLC films and their optical bandgap, increase of the optical bandgap with increased hydrogen amount in diamond like carbon film should be taken into account as well [27].

Some decrease of the gauge factor with increase of B parameter can be seen in Fig. 4 both for ion beam deposited (Fig. 4, a) and PECVD (Fig. 4, b) DLC films.

However, nearly the same B parameter value can be seen for the DLC films having very different gauge factor.

Decrease of the gauge factor with Urbach energy can be seen for the "conventional" hydrogenated DLC films deposited from acetylene gas by closed drift ion source and from methane gas by PECVD (Fig. 5).

Analyzing B parameter and Urbach energy in a manner typical for hydrogenated amorphous silicon, it seems, that these two parameters represent different aspects of the disorder. In one case, gauge factor increases with the increased disorder (B parameter), in another case gauge factor increases with the decreased disorder (Urbach energy). In the frames of this model, gauge factor increases with increase of the conduction-band-tail width and decrease of the valence band tail width. In the frames of the models describing physical meaning of the Urbach energy for the diamond like carbon films, gauge factor increases with decrease of the topological disorder (range in which size of sp^2 clusters vary) and/or density of sp^2 states and ammount of sp^2 bonded carbon phase. Such a relation between the gauge factor on one hand and optical bandgap and Urbach energy reveals, that optimization of the piezoresistive properties of DLC films can be complicated process, because increase of the Urbach energy with optical bandgap was reported for DLC films in

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[20, 28]. It means, that topological disorder of DLC films increases with decrease of the sp^2 cluster size (decrease of the π states density).

It should be taken into account, that in the present study during measurement of the gauge factor and optical properties data have been taken from the different areas. In the case of the measurement of gauge factor, active area was substantially larger than in the case of the optical absorption measurements. In addition it should be mentioned as well, that presence of some defects (conductive channel) in active area of DLC film during the measurement of the gauge factor can have many more substantial influence on the measurement data than presence of the macroscopic defects (e. g., pinholes) in DLC film in the case of the optical measurements.
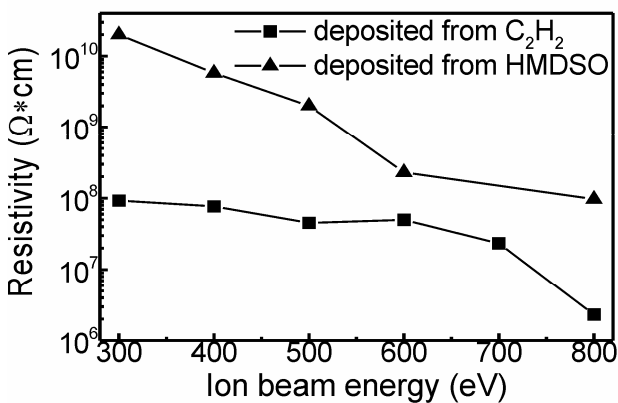

Fig. 6. The dependence of resistivity of ion beam synthesized hydrogenated DLC films deposited from acetylene gas and SiO_x containing DLC films deposited from hexamethyldisiloxane (HMDSO) on ion beam energy
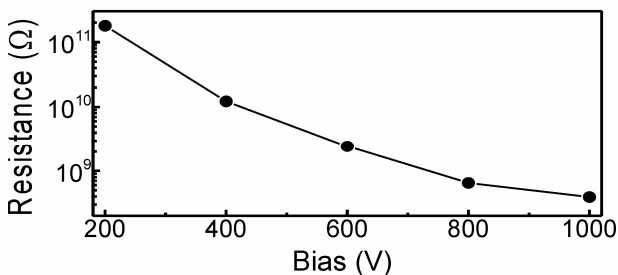

Fig. 7. Resistance of the piezoresistive element fabricated using PECVD grown DLC vs bias voltage

The resistivity of ion beam synthesized hydrogenated DLC films deposited from acetylene gas and SiO_x containing DLC films deposited from hexamethyldisilox-ane (HMDSO) decreased with applied ion beam energy (Fig. 6). Resistance of the piezoresistive element fabricated using PECVD grown DLC decreased with bias voltage as well (Fig. 8). It means, that no correlation between the piezoresistive properties of DLC films and their resistance can be found. The observed behavior can be explained mostly by decrease of the ammount of hydrogen in DLC films with increase of the ion energy [32]

CONCLUSIONS

In conclusion, ion energy and doping effects on piezoresistive properties of DLC films were investigated.

Weak dependence of the gauge factor on ion beam energy in the case of DLC films deposited from acetylene gas by closed drift ion source was explained by competition between the effects of the ion energy and ion/neutral ratio during the DLC film growth process. Doping of the diamond like carbon films by silicon oxide and silicon resulted in decrease of the gauge factor. Dependence of the piezoresistive properties of DLC films deposited by PECVD from methane gas on substrate bias was observed as well. It was explained by the influence of the substrate bias on structure (sp^3/sp^2 ratio and sp^2 cluster size) of DLC films.

The relations between the gauge factor of DLC films and optical parameters such as optical bandgap, Urbach energy and *B* parameter were studied. It was found, that the gauge factor increases with the increased separation between valence and conduction π band in DLC films and/or decrease size of sp^2 cluster as well as with decrease of the topological disorder (range in which size of sp^2 clusters varies) and/or density of sp^2 states and ammount of sp^2 bonded carbon phase.

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