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# Micromachining of Diamond-like Carbon Deposited by Closed Drift Ion Source for Cantilevers and Membranes

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In the present study production of the freestanding film structures of diamond like carbon (DLC) and DLC containing $SiO_x$ (DLC:$SiO_x$) are analysed. Role of the residual stress on the process of the micromachining of the "conventional" hydrogenated DLC and DLC:$SiO_x$ deposited by closed drift ion source has been investigated. Substantially reduced level of the internal stress (less than 0.5 GPa) as a result of the diamond like carbon film doping by $SiO_x$ was observed. DLC:$SiO_x$ free-standing film both cantilever and bridge-shaped with >1 μm thickness were successfully fabricated applying $SiO_2$ as a sacrificial layer by combination of the lift-off technique and wet chemical etching.

*Keywords:* diamond like carbon, free standing films, ion beam deposition, surface micromashining.

## 1. INTRODUCTION

Fast growth of the list of possible applications and markets of the microelectromechanical systems (MEMS) results in increased demand for novel MEMS fabrication technologies and materials. At present silicon is the main material for MEMS fabrication due to the well studied mechanical and electrical properties as well as possibility of the integration with IC's [1]. However, Si as a MEMS material has some inherent limitations such as relatively low Young's modulus and fracture toughness, relatively large coefficient of friction, high rate of wear and high surface energy [1]. Therefore search of new materials suitable for MEMS processing is in progress. In particular different thin film materials could be an advantageous choice due to the possibility to use surface micromachining instead of the bulk micromachining for different free-standing microstructures fabrication. In such a case use of the substrates with different properties such as optically transparent glass or flexible plastics would be possible. Substrates cheaper and much larger than monocrystalline silicon wafers could be applied in future. Due to the unique set of the mechanical, electrical, optical and hydrophobic properties as well as room temperature synthesis possibility diamond like carbon (DLC) films are very attractive materials for MEMS fabrication [2]. Recently found strong piezoresistive properties of the diamond like carbon films [3 – 5] increases its attractiveness as a MEMS based sensor material even more due to the possibility to make free standing film based high sensitivity piezoresistive sensors with mechanically strong and corrosion resistant sensing membrane. The properties of the diamond like carbon films depend on the ratio of the "diamond-like" ($sp^3$) and "graphite-like" ($sp^2$) bonds [6]. Higher $sp^3/sp^2$ bond ratio results in films with better mechanical properties (hardness, wear resistance, Young's modulus) [6], larger piezoresistive gauge factor [4] and higher optical transmittance [6]. However, such films suffer from the problem of the high internal stress as well [7 – 10]. Several techniques were used till now for reduction of the internal stress to the level enough low for fabrication of the free-standing diamond like carbon membranes and other micromechanical elements. Plasma immersion ion implantation [8 – 11] as well as doping by silicon [12] and stress-releaving annealing at 600 °C – 650 °C temperature [7, 13] were demonstrated to be efficient methods in the stress reduction. Both hydrogen-free [8 – 11, 13] and hydrogenated [12] free standing DLC films were fabricated in such a way. However, the stress reduction technologies mentioned above result in worsening of other mechanical properties such as hardness and Young's modulus [14 – 15]. From such a point of view, $SiO_x$ doped DLC films (DLC:$SiO_x$) seem to be good candidate as a material for the free standing film fabrication. In comparison with "conventional" hydrogenated DLC films, internal stress [16] and friction coefficient [17] of the $SiO_x$ doped DLC are lower. Other advantages of the DLC:$SiO_x$ films is better adhesion with different metallic substrates [18], higher wear resistance [16], higher optical transmittance [19], higher thermal stability [20] and increased hydrophobicity [21]. However, at our knowledge there are no reports on the fabrication of the $SiO_x$ containing diamond like carbon membranes or other microstructures with free standing film configuration.

In the present study mechanical properties (internal stress) of the hydrogenated DLC and DLC:$SiO_x$ films produced by direct ion beam deposition were investigated to use these films as possible structural elements of the microstructures. Both bulk and surface micromachining techniques were checked to produce free standing structures. It should be mentioned, that hydrocarbon ion beam deposition process has some advantages in comparison with the deposition techniques presently used for synthesis of the DLC films for free-standing film fabrication purposes. It is line-of-sight deposition (it means, that use of the lift-off technique is possible) and offers enhanced possibilities of the control of deposition

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process parameters in comparison with plasma enhanced chemical vapor deposition, and easier control of the ion energy and ion current density in comparison with plasma immersion ion implantation enhanced cathodic arc deposition and pulsed laser deposition.

## 2. EXPERIMENTAL

The hydrogenated diamond like carbon films as well as DLC:SiO$_x$ films were deposited by a gridless cold cathode closed drift ion source. The "conventional" DLC films were deposited using acetylene as a source of the hydrocarbons. The DLC:SiO$_x$ films were synthesized from a mixture of the hexamethyldisiloxane ($C_6H_{18}Si_2O$) and hydrogen. In this case hydrogen has been used as a feed (transport) gas. For all the synthesized samples ion beam energy was 800 eV, base pressure was $2 \cdot 10^{-4}$ Pa, work pressure was $2 \cdot 10^{-4}$ Pa. As we have shown in [18, 22] Raman spectra of the both "conventional" and SiO$_x$ containing DLC films were typical for diamond like carbon. The main peak of the Raman scattering spectra of SiO$_x$ containing DLC films was located at ~1530 cm$^{-1}$ for "conventional" DLC [23] and at ~1455 cm$^{-1}$ for SiO$_x$ containing DLC films [18, 22]. Concentration of Si and O in DLC:SiO$_x$ according to the XPS results was found to be 22 % and 26 % correspondingly [18]. $I_D/I_G$ ratio was 0.45 and 0.32 for "conventional" DLC [22] and at ~1455 cm$^{-1}$ for SiO$_x$ containing DLC films [18,22] respectively. More detailed information on the structure and chemical composition of the synthesized films versus different technological conditions can be found in [18, 22, 23].

In this study several techniques have been checked for fabrication of the freestanding DLC films. In all cases necessary pattern in the DLC films was formed by lift-off technique using photoresist as a sacrificial layer. The tested freestanding DLC structures were cantilevers and bridges.

In the first approach used for fabrication of the free-standing films we used DLC film deposited directly on Si(100) substrate. In such a case silicon substrate also served as a sacrificial layer. Silicon beneath of the membrane was removed by "wet" anisotropic KOH etching. Similar technique has been proposed for DLC free-standing films fabrication in [11, 24–28]. Both conventional and SiO$_x$ containing DLC films have been used.

The second tested technology included deposition of the conventional or SiO$_x$ containing DLC films on thermal silicon dioxide layer on top of crystalline silicon. In such a case thermal SiO$_2$ has been used as a sacrificial layer. It was etched by a buffered silicon dioxide etchant. Similar approach was reported for fabrication of the free-standing diamond like carbon films in [13, 29]. For some samples, subsequent silicon etching by KOH has been performed to make deeper "well" beneath the free-standing DLC film.

In the last technological approach applied in the present study, the SiO$_x$ containing DLC films were deposited on Si(100) substrate covered with a thin interlayer of the "conventional" DLC and silicon was used as a sacrificial layer.

Residual stress of both types of the diamond like carbon films has been evaluated by laser interferometry technique ($\lambda = 632.8$ nm) [30, 31]. Changes of the Si substrate curvature as a result of the DLC film deposition were measured by a conventional Michelson interferometer. Subsequently Stoney's equation has been used for calculations of the average stress in the film:

$$\sigma_f = \frac{1}{6}\frac{E_s h_s^2}{(1-\nu)h_f}\left(\frac{1}{R_2} - \frac{1}{R_1}\right), \tag{1}$$

where $E_s$ is Young's modulus of the substrate, $h_s$ is the thickness of the substrate, $\nu$ is Poisson's ratio of the substrate, $h_f$ is the thickness of film, $R_2$ and $R_1$ are the radii of the substrate after and before thin film deposition respectively.

Thickness and refractive index of the deposited films were measured using a laser ellipsometer Gaertner L115 ($\lambda = 632.8$ nm).

The fabricated different freestanding diamond-like carbon film structures were investigated using optical microscope and scanning electron microscope.

## 3. EXPERIMENTAL RESULTS

### 3.1. Residual stress in DLC

The main experimental results of the residual stress of the diamond like carbon and SiO$_x$ containing diamond-like carbon films deposited on crystalline silicon (film thickness 200 nm – 1200 nm) are presented in Table 1. One can see, that the average stress level of the "conventional" DLC films synthesized from the acetylene gas in all cases (for films of the 200, 600 and 1200 nm thickness) is substantially (almost three fold) higher than the stress of SiO$_x$ containing DLC films. In the case of the SiO$_x$ containing diamond like carbon films, a slight increase of the stress level was found for the films deposited at higher ion beam current density (50 μA/cm$^2$ versus 100 μA/cm$^2$). However, no clear dependence of the residual stress on thickness for both "conventional" and SiO$_x$ containing DLC films was found.

It can be mentioned, that the stress values reported to be enough low for fabrication of the free standing structures in most cases were similar or even higher than in the present study – ~0.15 GPa [32], ~0.6 GPa [10], ~0.7 GPa – 0.8 GPa [8], ~0.9 GPa [24] and even 1.5 – 4 GPa [33]. Therefore, it can be supposed, that ion beam synthesized DLC films, especially SiO$_x$ containing DLC films, are suitable for fabrication of the freestanding DLC films.

**Table 1.** Residual stress of the diamond like carbon and SiO$_x$ containing diamond-like carbon films.

| Sample No | Ion beam current density (μA/cm$^2$) | Thickness (nm) | DLC structure | Stress (GPa) |
|---|---|---|---|---|
| 1 | 50 | 200 | DLC:SiO$_x$ | 0.14±0.07 |
| 2 | 100 | 200 | DLC:SiO$_x$ | 0.15±0.08 |
| 3 | 100 | 600 | DLC:SiO$_x$ | 0.27±0.19 |
| 4 | 100 | 1200 | DLC:SiO$_x$ | 0.07±0.02 |
| 5 | 100 | 200 | DLC | 0.80±0.21 |
| 6 | 100 | 600 | DLC | 0.79±0.24 |

## 3.2. Microstructures produced from DLC and DLC:SiO$_x$

Both DLC and SiO$_x$ containing DLC films were applied to produce microstructures with the necessary pattern employing the lift-off technique. However fabrication of the “conventional” freestanding DLC films simply by KOH undercutting of the Si(100) substrate appeared to be problematic (Table 2). Only some DLC cantilevers remained after the etching procedure. This fact can be explained by too high level of the residual stress according to the results presented in Table 1. On the other hand fabrication of the SiO$_x$ containing DLC membranes directly onto the silicon substrate was even less successful due to the under-etching and subsequent peeling of the diamond like carbon film (Table 2).

**Table 2.** Fabrication of the free-standing diamond like carbon films

| DLC film | Free standing film fabrication technique | Free standing films fabricated | Free standing film quality |
|---|---|---|---|
| DLC | Si bulk micromachining by KOH | some cantilevers | only some DLC cantilevers remained after the etching procedure |
| DLC | Surface micromachining using SiO$_2$ sacrificial layer | – | Problems of the adhesion between DLC and SiO$_2$ |
| DLC:SiO$_x$ | Si bulk micromachining by KOH | – | Peeling of the DLC:SiO$_x$ due to the under-etching |
| DLC:SiO$_x$ | Surface micromachining using SiO$_2$ sacrificial layer | bridges cantilevers | Good, some buckling of the bridge-like free-standing films |
| DLC:SiO$_x$ on 50 nm DLC interlayer | Si bulk micromachining by KOH | bridges cantilevers | Free standing films are deformed |

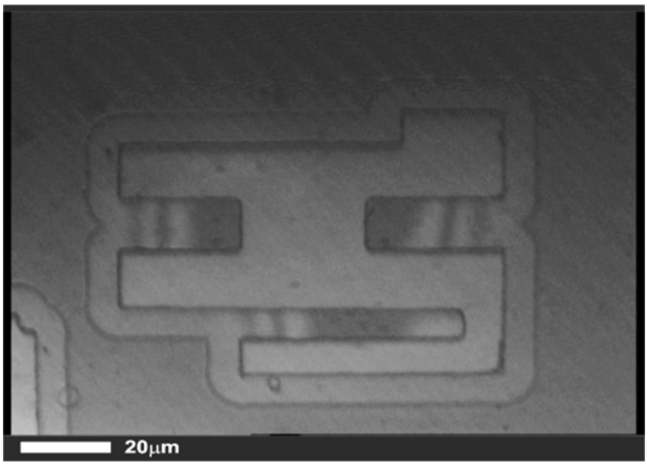

**Fig. 1.** Optical microscope images of the SiO$_x$ containing DLC free-standing films: the cantilever

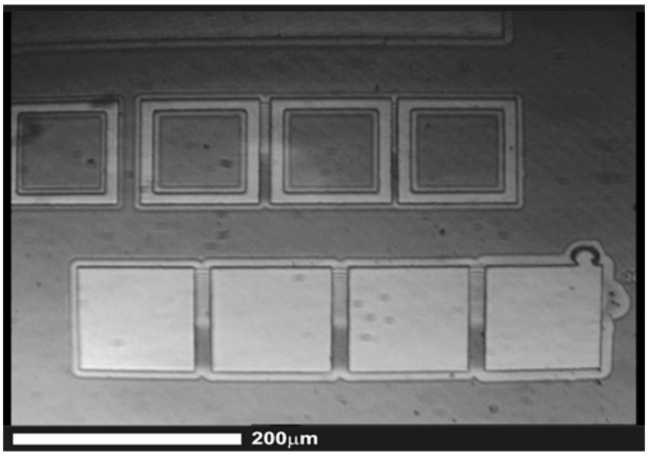

**Fig. 2.** Optical microscope images of the SiO$_x$ containing DLC free-standing films: overall view of the bridge-like structures

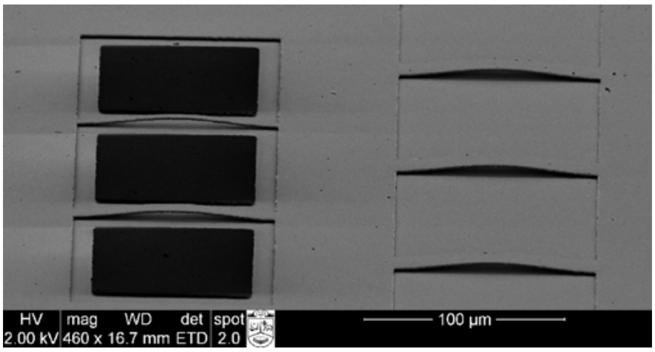

**Fig. 3.** SEM image of the SiO$_x$ containing DLC free-standing films (film thickness 1200 nm): overall view of the bridge-like structures

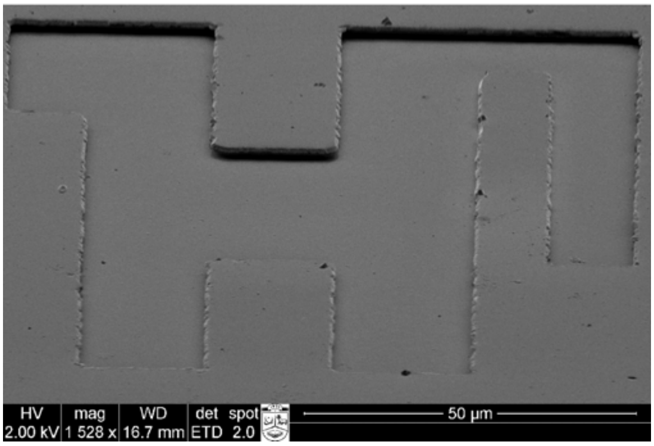

**Fig. 4.** SEM image of the DLC:SiO$_x$ free-standing films (film thickness 1200 nm): the cantilever

Having in mind problems mentioned above, one should use extra technological layers between the film and crystalline silicon. Fabrication of the “conventional” DLC (DLC deposited from acetylene gas) membranes using the second approach – where sacrificial thermal silicon dioxide layer was applied as a sacrificial layer – was unsuccessful due to the adhesion problems (Table 2). On the other hand, both cantilever-like and bridge-like free standing DLC:SiO$_x$ films of the thickness up to 1.2 micrometer were successfully fabricated using that approach (Figures 1, 2). SEM photos of the fabricated structures can be seen in Figures 3, 4. The additional "wells" fabricated by KOH silicon substrate etching more clearly revealed freestanding nature of the DLC:SiO$_x$ membranes (Figures 5, 6). The membranes successfully withstood through-etching of the monocrystalline silicon substrate by KOH (Figures 7, 8).

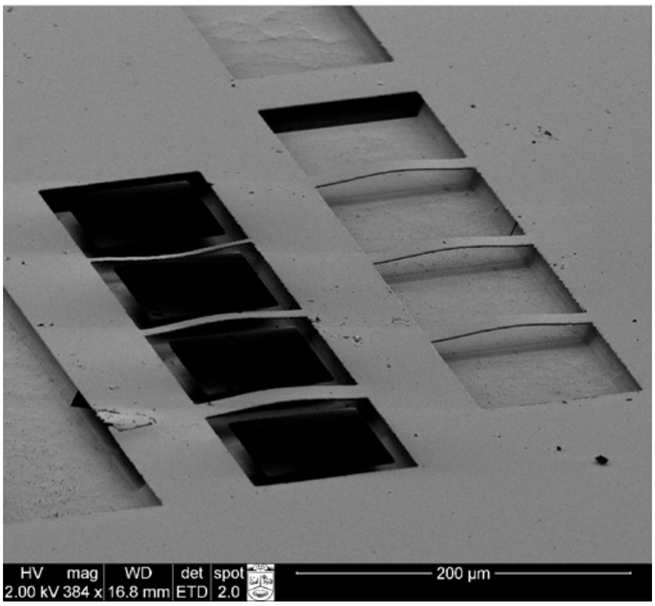

**Fig. 5.** SEM image of the DLC:SiO$_x$ free-standing films (film thickness 1200 nm): overall view of the bridge-like structures after the silicon etch-out by KOH

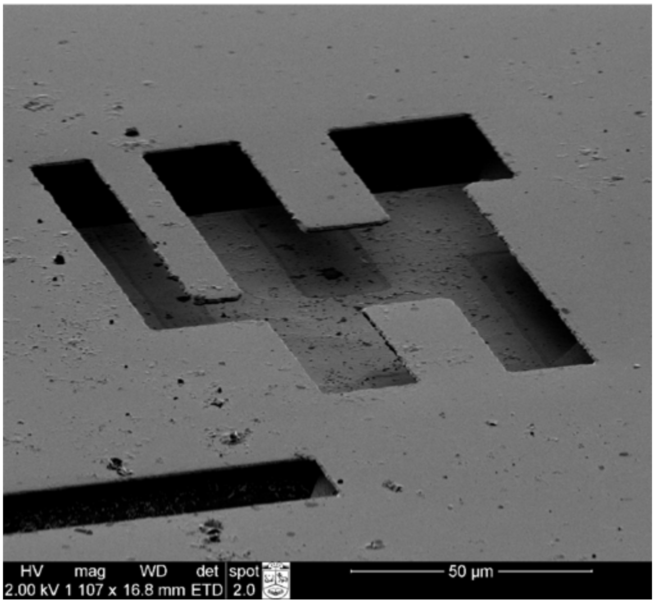

**Fig. 6.** SEM image of the DLC:SiO$_x$ free-standing films (film thickness 1200 nm): cantilever after the silicon etch-out by KOH

Some buckling of the free-standing bridges can be seen in Figs. 3 – 6. There are only few reports on fabrication of the DLC bridges [27, 28]. However, both in [27] and [28] similarly to present study buckling of the bridge-like free standing DLC films was reported. In addition it can be mentioned, that buckling was observed for DLC overhang produced by under-etching of the silicon substrate [24]. DLC free-standing foil of 20 µm thickness had a slightly curved shape as well [32], despite stress level of only 0.15 GPa was reported. In [28] buckling of the bridge-like free-standing diamond like carbon film was explained by recover of the length of the bridge to the unstressed value. In such a case centre of the bridge should deform due to the fixed ends of the bridge [28]. Free standing film buckling is well known problem [34]. Strain level at which free standing film will begin to exhibit significant transverse deflection (buckling) is described by equation [34]

$$\sigma_{critical} = \frac{E}{1-\upsilon} \frac{\pi^2 d^2}{3L^2_{critical}}, \tag{2}$$

where $E$ is Young's modulus, $\nu$ – Poisson ratio, $d$ – free standing film thickness and $L$ – length of the free standing bridge (unstressed value).

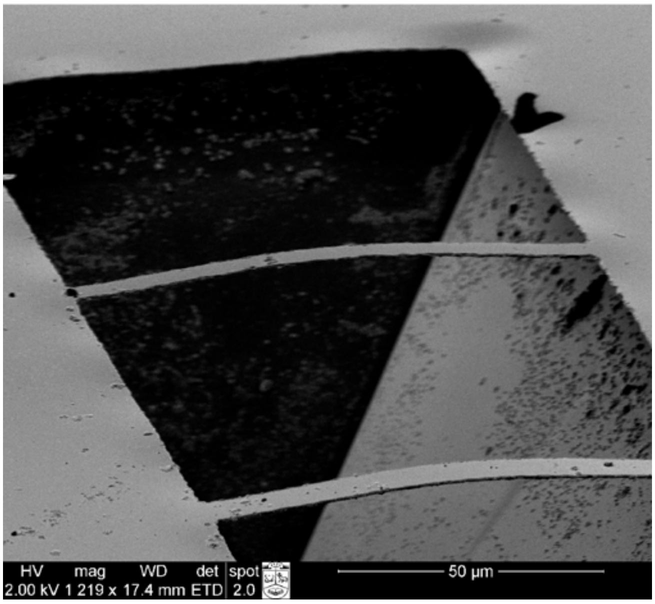

**Fig. 7.** SEM image of the DLC:SiO$_x$ free-standing films (film thickness 600 nm): bridge-like structure after the silicon substrate through etching by KOH from the top side

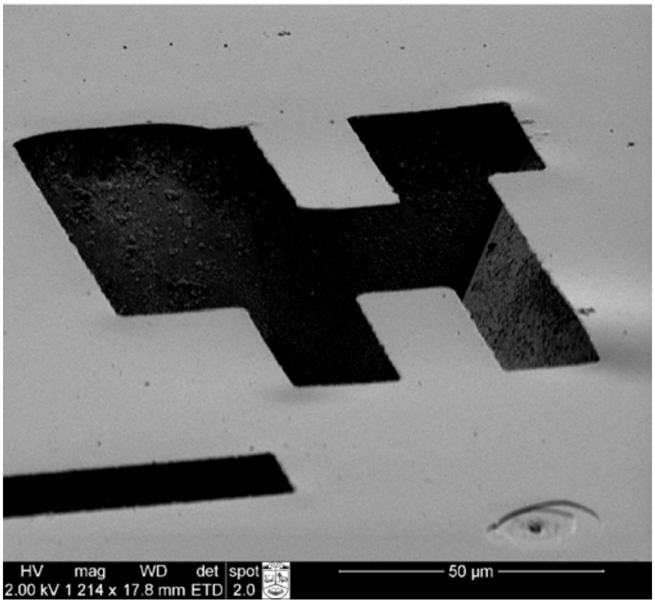

**Fig. 8.** SEM image of the SiO$_x$ containing DLC free-standing films (film thickness 600 nm): cantilever after the silicon substrate through etching by KOH from the top side

According to equation (2) in our case to avoid buckling of the $SiO_x$ doped DLC film with stress level as low as 0.07 GPa, DLC film thickness should be at least 1.4 μm to avoid free standing bridge-like film buckling. While in the case of the stress level of 0.27 GPa, DLC film thickness should be ~2.6 μm to avoid free standing bridge-like film buckling. It means, that the stress level of the synthesized $SiO_x$ doped DLC films is low enough for successful fabrication of the free standing films. However, DLC films thicker than 1.2 μm should be fabricated to avoid buckling problem.

The last technological approach applied in the present study included use of the DLC:$SiO_x$ films on Si(100) substrate with thin interlayer of the "conventional" DLC. In such a way, both free-standing bridges and cantilevers were fabricated. However, the membranes were deformed in comparison with the $SiO_x$ containing DLC membranes fabricated using the sacrificial silicon dioxide interlayer. Such a behavior possibly is related to the additional internal stress induced in a multilayer membrane by "conventional" DLC film, because these free-standing films crumbled after the several weeks holding in the air ambient.

## CONCLUSIONS

In conclusion the residual stress of the "conventional" hydrogenated DLC and $SiO_x$ containing DLC films deposited by closed drift ion source has been investigated. The substantially reduced level of the internal stress (less than 0.5 GPa) as a result of the diamond like carbon film doping by $SiO_x$ was observed. The achieved residual stress level enabled producing of the $SiO_x$ containing DLC free-standing films with >1 μm thickness with both cantilever and bridge-shaped configuration. The thermal $SiO_2$ as a sacrificial layer in production of free standing film structures DLC:$SiO_x$ on silicon was found to be the best choice in between the checked materials.

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