



UDC 538.951:53.097

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EFFECT OF TEMPERATURE ON PROPERTIES OF DLC FILMS AND DLC-Ni:C SANDWICH GROWTH

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ВЛИЯНИЕ ТЕМПЕРАТУРЫ НА СВОЙСТВА DLC-ПЛЕНОК И НА РОСТ СЭНДВИЧ-СТРУКТУРЫ DLC-Ni:C

A possibility of formation of sandwich structures comprising of both DLC and nano-crystalline Ni-carbon films on silicon substrate is presented. The influence of DLC film exposition to high temperature during preparation of Ni:C layers on its properties has been investigated by means of isochronal thermal annealing in carrying gas used. All structures obtained were examined by atomic force microscopy, scanning electron microscopy and X-ray photoelectron spectroscopy. It has been established that annealing leads to decrease of the internal residual stress in the DLC films as well as causes swelling of samples with temperature rise. The fraction of sp^3 hybridized bonds increases and the sp^2 fraction symmetrically decreases with increase of annealing temperature. The different sequences of growth processes to obtain DLC-Ni:C-DLC sandwich structure were investigated. The best way to get good structure quality is to grow initial DLC film at 200 V and 100 W and Ni:C layer at 500 °C.

DIAMOND-LIKE CARBON, DLC, NANOCRYSTALLINE NICKEL, THERMAL ANNEALING, INTERNAL RESIDUAL STRESS, SANDWICH STRUCTURE.

Продемонстрирована возможность формирования сэндвич-структур, включающих слои алмазоподобного углерода (DLC) и слои углерода с нанокристаллами никеля на кремниевой подложке (ncNi:C). Исследовано влияние высокотемпературной выдержки DLC-пленки в процессе осаждения слоев ncNi:C на ее свойства. Изохронный отжиг проводился в среде технологического газа, используемого в CVD-процессе осаждения. Все полученные структуры были исследованы методами атомной силовой и сканирующей электронной микроскопии, а также рентгеновской фотоэлектронной спектроскопии. Установлено, что отжиг приводит к снятию внутренних механических напряжений в DLC-пленках, а также вызывает свеллинг образцов. При этом с увеличением температуры отжига возрастает доля sp^3 -гибридизованных связей и симметрично снижается доля sp^2 -связей. Были изучены различные последовательности процессов получения сэндвич-структуры DLC-Ni:C. Наилучший режим для достижения высокого качества структуры – это использование напряжения 200 В и мощности 100 Вт для выращивания DLC-пленки и получение слоев Ni : C при температуре не выше 500 °C.

АЛМАЗОПОДОБНЫЙ УГЛЕРОД, НАНОКРИСТАЛЛ НИКЕЛЯ, ТЕРМИЧЕСКИЙ ОТЖИГ, ВНУТРЕННЕЕ МЕХАНИЧЕСКОЕ НАПРЯЖЕНИЕ, СЭНДВИЧ-СТРУКТУРА.

I. Introduction

Diamond-like carbon (DLC) thin films have attracted significant attention because of their unique properties namely extremely high hardness, transparency in the infrared range, high density, low friction coefficient, chemical stability, etc. [1, 2]. All these provide a possibility to use the DLC thin films in many applications as protective coating for magnetic recording disks, anti-reflective coating on optics and IR windows, wear-resistant coating on cutting and abrasive tools, etc. Moreover, promising field emission properties of DLC thin films have been reported [3–5]. In this way, DLC films can find applications as electron emitters in vacuum microelectronic devices. The aligned array of conducting nanowires inside highly resistive DLC matrix can be used as nanofilaments in complex electronic devices. The formation of such conducting channels in different carbon matrices by irradiation with swift heavy ions is described in [6, 7]. Moreover, in a recent study, H.-G. Gehrke et al. [8, 9] have shown the fabrication of conducting vertical carbon nanowires in insulating Ta-C matrix together with a built-in self-aligned gate electrode which has potential application as a gated field emission cathode.

The properties of DLC films mainly depend on their microstructure, which is an amorphous mixture of sp^2 and sp^3 hybridized bonds between carbon atoms [1 – 5, 8, 9]. The quality of DLC films and therefore their properties such as internal residual stress, resistance, hardness, friction coefficient, etc. can be changed by tuning the ratio of sp^2/sp^3 phases. One more way to change the film properties is its doping by metal (Fe, Cu, Ag, Sn) or non-metal (B, N, P, S, Si) impurities [10 – 13]. Hydrogenated amorphous carbon (α -C:H) films are grown in the processes with the use of hydrogen containing precursors. Therefore, hydrogen is always incorporated into the film and the amount of it also influences on film properties.

On the other hand, the formation of Ni-nanoparticle containing carbon films obtained by CVD technique has been reported recently [14]. Their field emission properties were investigated [15]. These films containing well separated 20 nm Ni particles showed the

best emission properties with the threshold field value close to 2 V/ μm .

It is possible to improve the conductivity of nanowires formed by swift heavy ions by incorporating metal phase, in particular nickel nanoparticles. At the same time, more or less thick films could not be obtained by the CVD process used for Ni-nanoparticle formation. Hence, it is necessary to grow structure consisting of alternate layers of pure carbon films (DLC) and NiC.

The growth temperature of Ni-nanoparticle containing carbon films is higher than DLC film preparation temperature. In this study an influence of thermal annealing in a carrying gas utilized for Ni-C layer formation on the properties of grown DLC films as well as the possibility to grow the sandwich structures comprising both DLC and Ni-C films on crystalline silicon substrates are presented.

II. Experiment

DLC films were grown on (1 0 0) silicon substrates of 4.30 mm in size in the RF plasma of methane (CH_4) gas. Plasma-enhanced chemical vapor deposition (PECVD) capacitive system with flat electrodes was used [16]. Positive bias was applied to the same electrode as the RF (40 MHz) power. The samples were mounted on the grounded electrode.

To provide good adhesion, three-step substrate pretreatment procedure was used. The first stage of this procedure was oxygen plasma exposition; at the second one, the substrate was annealed at 350 °C. At the third stage, hydrogen plasma exposition was used during sample cooling from annealing to the deposition temperature. Such a scheme is necessary to prevent exfoliating or bursting diamond-like carbon films. Methane pressure during the film deposition was 30 mTorr, substrate temperature was measured by a thermocouple and kept at 300 °C. It is possible to affect the phase composition and therefore the properties of films by the variation of process parameters such as DC-bias voltage in the range of 50 – 500 V, RF-power in the range of 50 – 450 W, and substrate temperature from room to 350 °C [17, 18]. The thickness of grown DLC films was measured by atomic-force microscopy at the film edge after the deposition and was $0.08 \pm 0.01 \mu\text{m}$.

The set of DLC film samples was annealed in argon ambient at the temperature of 400 – 600 °C using isochronal steps of 60 min. Thermal annealing was carried out using the same setup as for Ni-C film growth.

Ni-nanoparticles containing carbon films were prepared by chemical vapor deposition (CVD) technique with bis-(ethylcyclopentadienyl) nickel ((EtCp)₂Ni) as a precursor [14]. The deposition process was carried out in hot-wall horizontal low-pressure silica tube reactor. The temperature range was 500 – 600 °C, the deposition time was 5 min. Argon or hydrogen were used as a carrying gas. (EtCp)₂Ni partial pressure was 75 Pa. The total pressure in the reactor was 840 Pa. The obtained layers contained 10 – 20 nm Ni particles (presumably coated by nickel carbide shells), either isolated from each other or comprised into continuous coverage (it depended on some technological parameters, such as deposition time and substrate temperature).

Relative contents of the sp^2 and sp^3 phases in the obtained films were determined using X-ray photoelectron spectra measured by NANOFAB-25 system (NT-MDT). The curvature of the silicon wafer was measured using an interferometry method before and after film deposition, and after annealing. Internal residual stress in the film was determined with the use of Stoney equation [19]. All the grown films were compressed. The morphology of the deposited structures was examined using a scanning electron microscope Supra 55 VP. The film thickness was determined after deposition and annealing by atomic force microscopy with Nano-DST (Pacific Nanotechnology) machine.

III. Results

To investigate the influence of thermal annealing on DLC film properties, we prepared a series of samples at 300 °C. DC-bias voltage and RF-power were 200 V and 100 W, correspondingly. The thickness of as-grown films was 70 – 90 nm. In our previous work it was shown that annealing in air in the temperature range of 250 – 350 °C had no effect on the properties of DLC films [17]. Here we present investigating the influence of isochronal annealing in argon ambient in the temperature

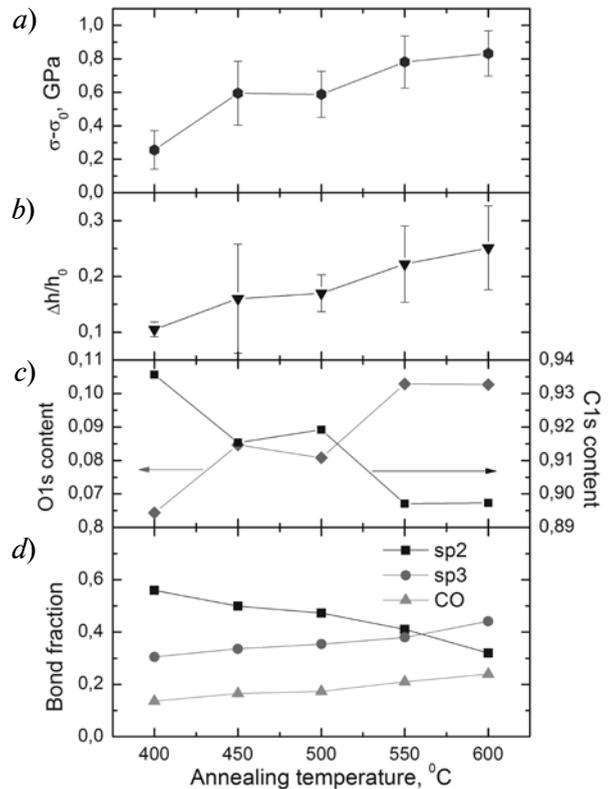


Fig. 1. Dependence of the stress change in the DLC films (a), the relative increase of the film thickness (b) and the change in the O and C content (c) and fraction of sp^2 and sp^3 hybridized bonds (d) after isochronal annealing in argon ambient as a function of temperature

range of 400 – 600 °C (see Fig. 1) on DLC film properties. These temperatures are typical in the CVD process used to get Ni containing carbon films.

In Fig. 1, a we present internal residual stress in the DLC film, initial stress value being preliminarily subtracted from the whole value ($\sigma - \sigma_0$), as a function of annealing temperature. As it was pointed out above, all as-grown films were stressed. Fig. 1, a shows that the stress value decreases with temperature.

The influence of thermal annealing on the DLC film thickness is presented in Fig. 1, b. Almost linear temperature dependence of the film swelling is observed. The film thickness increases up to 25 % of initial thickness. Interestingly, internal stress as well as film thickness exhibit similar trends with the increase of accelerated ion fluence, as it was shown in

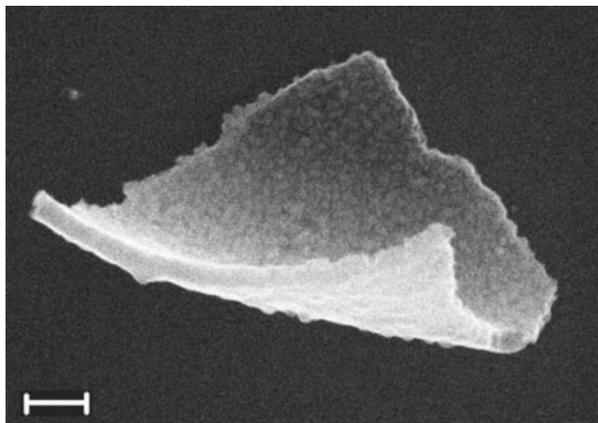


Fig. 2. SEM micrograph of peeled flake of NiC-DLC structure. Marker length is 200 nm

Ref. [20]. This finding let us conclude that the mechanisms of the film properties change are very close. To support this idea we need to investigate the change in film structure.

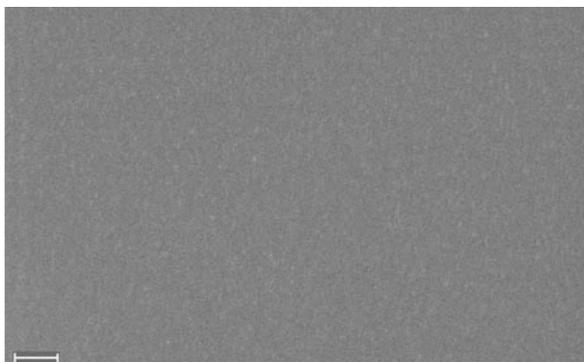
Influence of annealing on the film structure was studied by *X*-ray photoelectron spectroscopy. This technique lets us investigate content and bond fraction in ~5 nm thick subsurface layer. The contents of carbon and oxygen in the films are presented in Fig. 1, *c*. C1s peak was decomposed into components corresponding to graphite, diamond phases and C–O bond. The positions of corresponding maxima were kept close to 284.6, 285.2, 286.7 eV according to data from Ref. [1]. The results are presented in Fig. 1, *d*. With the rise of annealing temperature, the fraction of *sp*³ hybrid-

ized bonds increases, and simultaneously the *sp*² phase symmetrically decreases. Moreover, it is seen that the oxygen content at the sample surface increases with the increase of annealing temperature. We explain this high oxygen content as follows. The increase of C–O bond fraction could be associated with the oxidation of the surface when DLC films were taken out the reactor. The driving reason for oxygen trapping can be activation of carbon bonds due to the hydrogen loss during film annealing.

For the preparation of sandwich structures we used two reverse sequences of the film growth processes: Ni : C-DLC and DLC-Ni : C. In the first case, after the deposition of DLC film grown without pretreatment procedure, the Ni : C layer peeled off the substrate. SEM micrograph of a piece of the sample is presented in Fig. 2. The figure shows that the stressed DLC film fixes a Ni nanoparticle containing carbon film, rolls up and peels off. To improve adhesion, the three-step substrate pretreatment procedure was used. But it led to the oxidation of Ni nanoparticles. Thus we were not able to obtain the sandwich structure by this method.

In the second case, we prepared two series of DLC films at 300 °C and different values of RF-power and DC-bias voltage: 100 W, 200 V and 400 W, 400 V, which gave high and low values of compressive stress in the DLC films [17, 18]. The morphology of the films grown at 400 W and 400 V was nonuniform. Partial peeling on the edges of some samples was observed.

a)



b)

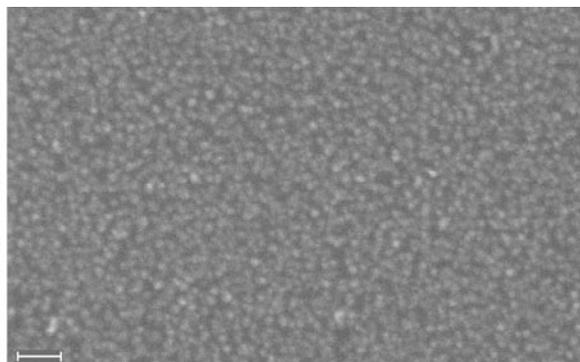


Fig. 3. SEM micrograph of DLC-NiC structure. DLC film was grown at 100 W and 200 V, Ni-C layer was grown using argon as a carrying gas at 500 °C (a) and 570 °C (b). Marker length is 100 nm

The Ni-C layers were grown on the top of the DLC films at different temperatures (500 or 570 °C) and carrying gases (argon or hydrogen).

The growth of Ni-nanoparticle containing carbon film was successful at all cases. The size and concentration of Ni nanocrystals did not depend on the type of underlying DLC film but changed with the parameters of CVD process, namely the carrying gas and process temperature. Moreover, if we used hydrogen as carrying gas, the concentration of Ni nanoparticles increased with temperature while their size did not change. For the case of argon as a carrying gas, the size and concentration of nanocrystals increased with temperature (Fig. 3). At the same time, the CVD process temperature had significant influence on DLC sublayer, as described above. Capping DLC layers were grown on all the samples using the same process parameters as were used for initial DLC layer growth. The upper layer of DLC film was grown without pretreatment procedure. Only hydrogen plasma exposition during 1 – 2 min was used to remove oxygen from the surface. Partial peeling and formation of blisters were observed after the capping layer growth in all the cases if high temperature CVD was utilized. The initial DLC films grown at 400 W and 400 V had some cleavages. Moreover, the area and quantity of such cleavages significantly increased after the growth of the third layer (Fig. 4). Thus, it was very difficult to obtain uniform films using these conditions. In the

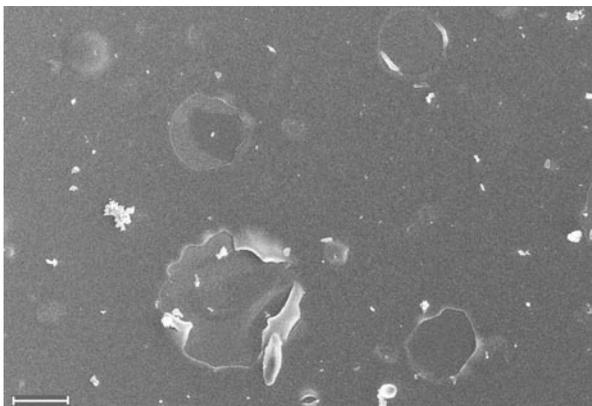


Fig. 4. SEM micrograph of DLC-NiC-DLC structure. DLC film was grown at 400 W and 400 V, Ni-C layer was grown using hydrogen as a carrying gas at 570 °C. Marker length is 10 μm

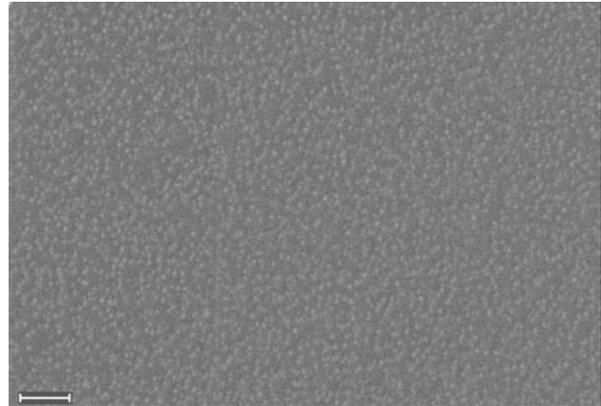


Fig. 5. SEM micrograph of DLC-NiC-DLC structure. DLC film was grown at 100 W and 200 V, Ni-C layer was grown using argon as a carrying gas at 500 °C. Marker length is 100 nm

case of Ni-C layer grown at low temperature and the initial DLC film grown at 100 W and 200 V, the obtained structures were uniform and of good adhesion (Fig. 5). Therefore, these conditions corresponded to the best results of growing a sandwich structure.

IV. Conclusion

It has been found that isochronal annealing in argon ambient leads to the decrease of the internal residual stress in DLC films. Moreover, with the rise of annealing temperature, the relative concentration of sp^3 hybridized bonds increases, and simultaneously the sp^2 phase symmetrically decreases. Also, annealing causes sample swelling. Film growth in sequence NiC-DLC leads to peeling the whole structure off the substrate. For the reverse sequence, the size and concentration of Ni nanocrystals do not depend on the type of the underlying DLC film (with high or low stresses) but depend on the process parameters such as carrying gas and temperature. For both hydrogen and argon carriers, the Ni nanocrystal concentration increases with temperature. Moreover, if we use hydrogen as carrying gas, the size of Ni-nanoparticles does not change while in the case of argon their size increases with temperature. The rise of temperature during the growth of NiC layer leads to all the structure peeling irrespectively of the initial DLC film. We have found that the sequence DLC-NiC-DLC, where the initial DLC film is of high stress and NiC layer

is deposited at low temperature, is the best way to grow a good sandwich structure.

This work was supported by Russian RFBR grants 12-08-01197 and 13-02-92709.

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