

Ion Source Based on Reflex-Discharge with Cold Hollow Cathode for Reactive Ion-Beam Synthesis of Diamond-Like Films

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Abstract – It is shown that ion source based on reflex-discharge with cold hollow cathode enables to produce film of amorphous hydrogenated diamond-like carbon $a\text{-C:H}$ by reactivity ion-beam synthesis method. Using propane as working gas results, with the course of time, in violation of conditions of stable discharge maintaining. Possible reasons for instability and operation time of the ion source, after which it is necessary to clean the discharge chamber, are defined herein. Synthesized thin carbon films were analyzed by transmittance IR-spectrometry.

1. Introduction

One of the most perspective methods of getting thin films in vacuum is reactivity ion-beam synthesis (RIBS) directly from ion beam [1]. It is known that polybrochate ion source (IS) “Radical-M250” [2, 3] is used with this purpose. It produces wide low-energy intensive beam with high uniformity of current-density distribution along beam cross section on the diameter of 150 mm, which provides coating with thin films of great smoothness on the specified surface. Herein it is proposed to use IS on the basis of reflex-discharge with hollow cathode to perform RIBS (Fig. 1).

The used IS modification of that type does not enable to get ion beams of great cross section, however

it has a number of advantages in other parameters. In particular, the width of energy ion spectrum in IS beam “Radical-M250” on half-height of ion energy distribution function is 540–580 eV [1], but the analogous parameter in the proposed IS does not exceed 30 eV [4]. The authors of the present paper think that such narrow energy ion spectrum will enable to control the properties of synthesized films with greater effectiveness and predictability. Besides, unlike IS “Radical-M250” the proposed source enables to independently control ion current density and ion energy, that makes it possible to study film synthesis more effectively and substantially increases technological possibilities of the ion source.

The present paper is an attempt to get diamond-like films of amorphous hydrogenated carbon ($a\text{-C:H}$) by means of RIBS with the help of IS based on reflex-discharge with hollow cathode. Synthesis of diamond-like $a\text{-C:H}$ currently represents an actual task of micro- and nanoelectronics, as these films have a number of important properties: biocompatibility, chemical inertness, endurance, high thermal conduction, resistance to γ -radiation, forbidden gap variation in accordance with synthesis conditions. Carbon films of various structures are perspective as a basis for producing efficient cathodes, using field electron emission effect [5], and as antireflecting [6], protective, and heat removing coatings.

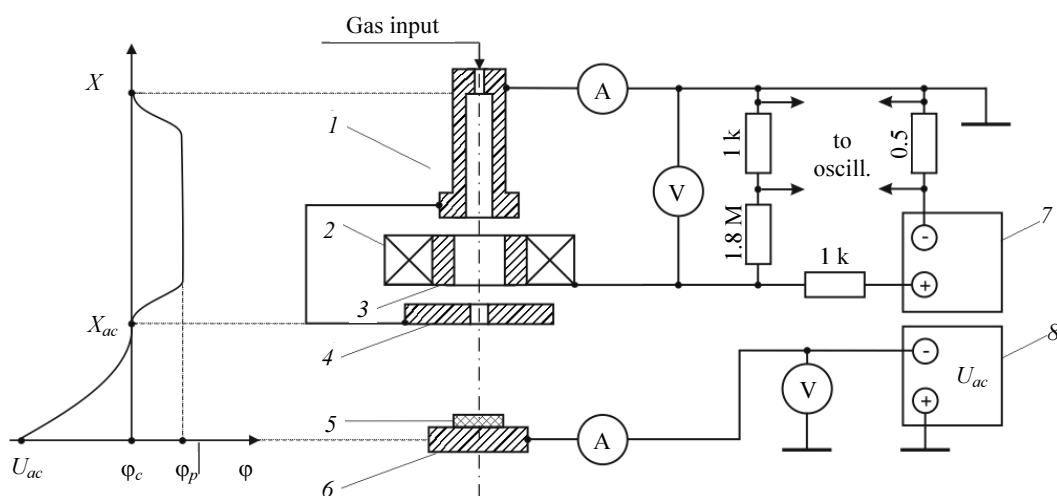


Fig. 1. Experimental installation scheme and diagram of potential distribution along the axis of discharge chamber and accelerating gap: ϕ_p – plasma potential; ϕ_c – cathode potential; U_{ac} – accelerating voltage

2. Experiment technique

Experiment scheme is presented in the Fig. 1.

Ions were generated in plasma of reflex-discharge, which was initiated in discharge chamber, formed by hollow cathode 1, cylindrical anode 3 and reflex-cathode 4 with a hole of 4 mm in diameter. Magnetic field of ~ 80 mT was created in discharge chamber by annular constant magnet 2. Ions penetrated into accelerating gap through the hole in reflex-cathode and were accelerated by electric field in the direction of substrate 5. A special electrode or, as is shown in Fig. 1, a substrate holder 6 can be used as an accelerating electrode. Gas (propane C_3H_8) was let into the hollow cathode. Gas flow rate was about $400 \text{ atm} \cdot \text{cm}^3/\text{h}$. The ion source worked without stopping. Discharge current I_d was controlled in the range 0.1–0.4 A, discharge voltage $U_d = 350$ –450 V, beam current I_b did not exceed 40 mA. Voltage stabilizers UIP-1 7, 8 were used as a power source. Accelerating voltage U_{ac} was controlled in the range 0–600 V.

To analyze IS operation stability, current and discharge voltage oscillograms were registered with personal computer. Signals were supplied to the input of sound card AC'97. Emulation of double-beam oscillograph was carried out with the help of "Oscilloscope 2.51" program. Calibration was performed by supplying control signal with amplitude of 1 V from oscillograph RFT EO 213. By means of frame capture program "Snagit" oscillograms were uninterruptedly fixed with the speed of 50 frames per second and were stored in the computer memory in video format. Current and discharge voltage measurement was carried out by digital multimeters MXD-4660A.

Film deposition was performed directly from ion beam at vacuum chamber pressure of maximum 10^{-3} Torr on the substrate KEF 4.5 (111).

Film synthesis time did not exceed 10 min.

The received coatings were analyzed by transmittance IR-spectrometry on the installation IR-Fourier spectrometer FSM-1201.

3. Ion source analysis

Figure 2 presents IS voltage-current characteristic.

Beam current was controlled by means of discharge current changing, while ion energy was controlled by means of accelerating voltage changing.

The analysis showed that using hydrocarbon as plasma-forming gas results in unstable discharge burning, which is not detected while working with air and argon. Discharge current and voltage oscillograms show that beginning with the first seconds, on the background of stable glow discharge burning in propane, first small irregular current falling appear for 0.1–0.2 ms. Then value and duration of discharge current falls increase (Fig. 3, a).

It is possible to assume that the reason of the above-mentioned instabilities consists in formation of dielectric films on the surface of discharge chamber

electrodes. The films are formed of the products of propane molecule dissociation in discharge.

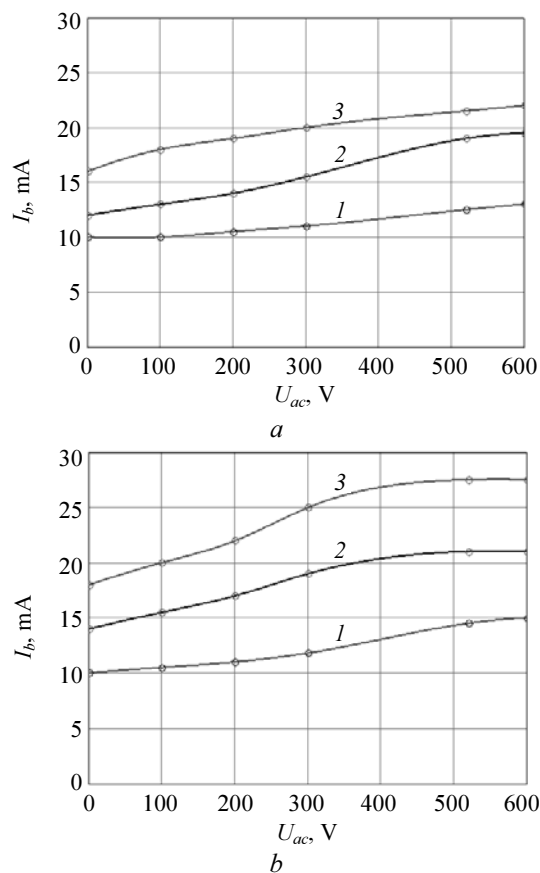


Fig. 2. IS voltage-current characteristic. The working gas: air (a), propane (b). Discharge current I_d : 1 – 175; 2 – 225; 3 – 275 mA

In ~ 40 min after discharge ignition, discharge current and voltage steps are detected; they indicate that for several ms glow discharge develops into arc (Fig. 3, b).

In this case, discharge burning voltage decreases up to ~ 100 V, and discharge current, limited by power source and ballast resistance, increases up to ~ 0.6 A. Apparently, the reason of cathode spot appearance consists in dielectric films charging by ions on the surface of cathodes, increasing of electric intensity and subsequent dielectric breakdown [7].

Visual observation of discharge chamber electrodes after working on propane speaks for the formation on them of coatings in the form of pressed powder, which apparently consists of various carbon modifications. On the anode which is located inside the annular magnet the above mentioned coatings have magnet properties. This can be explained by anodic deposition of products of spraying and vaporization of ferromagnetic material of cathodes.

In view of the conducted research discharge chamber was periodically cleaned. For this purpose gaseous discharge was ignited in the air. At discharge current of 0.5 A, the cleaning time did not exceed 30 min.

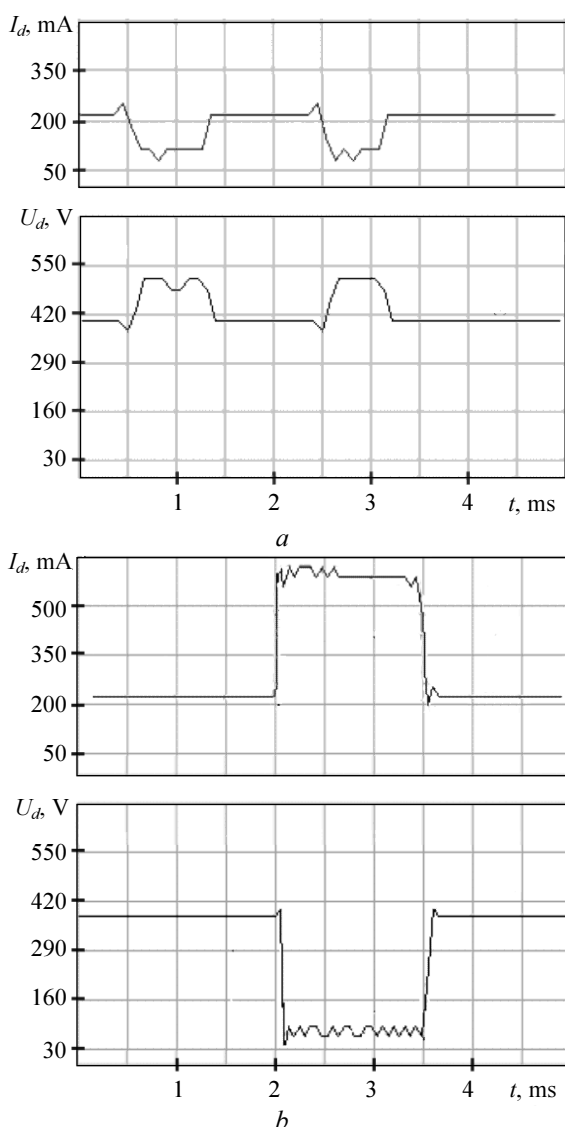


Fig. 3. Current and discharge voltage oscillogram in 5 (a) and 35 min (b) after the operation start

The total time of film synthesis between two cleanings of IS did not exceed 40 min to avoid formation of cathode spots and penetration of cathode erosion products into synthesized films.

4. Thin film analysis

Figure 4 presents transmission IR-spectrum of one of the synthesized samples.

This spectrum contains an area of deformation ($1330\text{--}1560\text{ cm}^{-1}$) and valence ($2850\text{--}3030\text{ cm}^{-1}$) vibrations of C–H bonds. To define that the received samples belong to *a*-C:H films, a modeling of absorption IR-spectrum in Hiperchem program using semi-empirical calculation algorithms was carried out (Fig. 5).

Comparison of Figs. 4 and 5 shows that the same areas of absorption corresponding to deformation and valence vibrations of *a*-C:H clusters are found in experimental and calculation spectra. Vibrations in the range of wave numbers $1600\text{--}1800$ in the spectrum of

experimental model may belong to C = O bonds. Apparently, in this case, the source of oxygen is the supplied hydrocarbon, which contains admixtures. Even small oxygen concentration in the film can result in appearance of intensive absorption bands, as carbon and oxygen atoms considerably differ in degree of electronegativity.

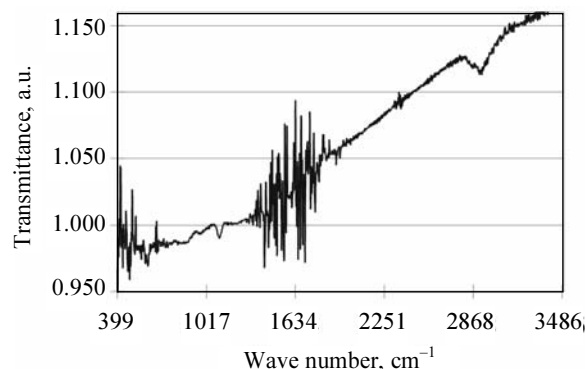


Fig. 4. Spectrum of IR-radiation transmittance by the synthesized film, $U_{ac} = 450\text{ V}$

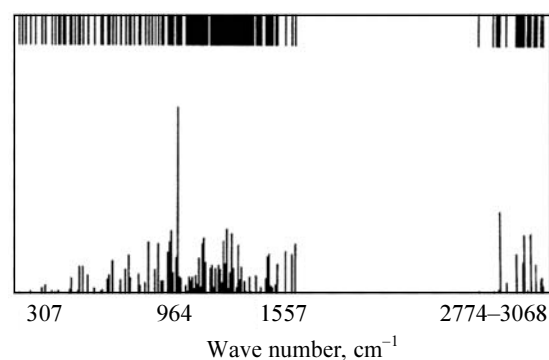


Fig. 5. Spectrum of radiation absorption by *a*-C:H clusters generated in Hiperchem program

To analyze IR-spectra of *a*-C:H material the area of valence vibrations of C–H bonds is usually used. Interpretation of spectrum of synthesized film in the area of valence vibrations of C–H bonds on Fig. 6 is as follows [8]: $sp^3CH_3(a)$ (2960), $sp^3CH_2(a)$ (2925), $sp^3CH(a)$ (2900), $sp^3CH_3(c)$ (2870), $sp^3CH_2(c)$ (2850), $sp^3CH(c)$ (3030) (sp^3 , sp^2 , and sp are hybridization types, figures in brackets represent wave number (according to reference table) in cm^{-1} , corresponding to characteristic frequency of bond absorption; (a) and (c) designate antisymmetric and symmetric oscillations). Absorption bands 3000 and 3045 cm^{-1} correspond to valence vibrations of aliphatic and aromatic sp^2CH groups, respectively.

Preliminary study determined the dependence of fixed carbon concentration in the film on ion energy. At $U_{ac} = 200\text{ V}$ soft polymer film is produced, and at $U_{ac} = 600\text{ V}$ solid film is produced, however absorption band intensity in IR-spectrum in the area of valence vibrations of C–H bonds decreases, that speaks for decreasing of fixed hydrogen concentration in the

film. In this case, it is possible to assume the formation of *a*-C or *ta*-C film.

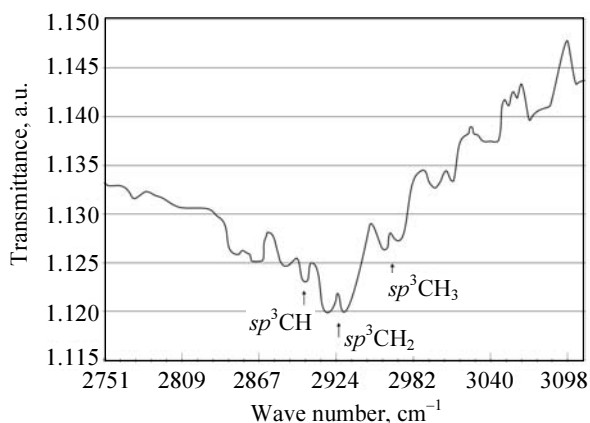


Fig. 6. Spectrum of radiation transmission by the *a*-C:H film in the area of valence vibrations of C–H bonds, $U_{ac} = 450$ V

A number of parameters (good adhesion, high mechanical strength, domination sp^3 -hybridized C–H groups, moderate hydrogen concentration) enable to

rank the coatings, deposited at accelerating voltages $U_{ac} = 400$ – 500 V, among *a*-C:H diamond-like films of carbon.

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