Installation for Hard Carbon Films Deposition on Large Area Substrates[1]

N.S. Sochugov, A.A. Soloviev, S.V. Rabotkin, I.R. Arslanov, K.V. Oskomov, V.G. Podkovyrov, and N.F. Kovsharov

Institute of High Current Electronics, SD RAS, 4 Akademichesky Ave., Tomsk, Russia, 634055 e-mail: sochugov@lae.hcei.tsc.ru

Abstract – Description of the installation for vacuum ion-plasma deposition of hard carbon and hydrocarbon coatings on large-are substrates is presented. The installation vacuum chamber has the volume of 1 m^3 and is equipped with two extended unbalanced magnetron sputtering systems with cylindrical rotating cathodes and two extended ion sources with closed electron drift. The installation is equipped with two 8-kW power supplies; each of them can be connected to the magnetron or to the ion source. There is also a substrate bias power supply operating in the low-voltage (100–800 V) and high-voltage (1–10 kV) modes. Complex technology is realized in the installation including ion-plasma substrate cleaning by means of the Hall-current closed – drift ion source; creation of an intermediate layer between the film and substrate by the method of plasma immersion ion implantation from gas plasma; deposition of amorphous carbon films using methods of pulsed magnetron sputtering of graphite, or ion-plasma deposition from hydrocarbon plasma created with the closed – drift ion source.

1. Introduction

Amorphous diamond-like carbon (DLC) films are characterized by mostly tetrahedral geometry of strong covalent carbon-carbon bonds that is caused by sp^3 hybridization state of valence electrons in carbon atom. Such kind of structure is similar to diamond (except the existence of long-range crystalline order) that leads to unique combination of DLC properties. In particular, DLC is an attractive coating material for many applications in research and industry because of some important surface properties including high hardness, low friction, high wear resistance, good corrosion resistance, and more.

Problem of development of productive technology for DLC deposition has the following three main aspects. First, to create amorphous carbon structure rich with tetrahedral C-C bonds it is necessary to assist the growth of the film by ion bombardment providing approximately 100 eV per a deposited carbon atom Importantly, higher ion-to-neutral flux of the accelerated ions is favorable for deposition of harder and denser a-C and a-C:H coatings [1]. Second, hard DLC films are usually characterized by high intrinsic compressive stress level that leads to poor adhesion of the coatings. Thus, it is necessary to deposit an intermediate layer that is well-adherent both to the film and to the substrate. Third, to develop productive technology it is necessary to increase throughput of the process, i.e. increase deposition rate of the coating and/or treated substrates surface area. This can be achieved by generation of large volumes or extended flows of plasma, and/or by increase of plasma density.

At present time there is no technology that can meet all the requirements. For example, the hardest DLC (80–90 GPa) are usually deposited by means of fully-ionized carbon plasma of filtered vacuum cathodic arc [2]. However, this approach can not be applied for treatment of large-area substrates (or multiple parts simultaneously). Besides, for creation of interface adhesion layer it is necessary to use additional ion or plasma source. Another most frequently used deposition technique is plasma-immersion ion implantation (PIII) of a substrate followed by plasma-immersion ion-assisted deposition (PIID) of moderately hard (20–30 GPa) a-C:H films with use of hydrocarbon capacitive RF plasma [3] This method allows to create intermediate layer *in situ* for improving the film adhesion. Moreover, use of this approach makes it possible to process many large-area substrates of complicated shape simultaneously. The main drawback of the technique is low (~ 0.1 μm/h) film growth rate caused by low operating pressure (0.01 Pa). Increase of the pressure leads to sufficiently non-uniform spatial distribution of the plasma that is strictly unacceptable.

Alternative approach for large-area thin film deposition has been developed and used at [4]. It is based on extended cylindrical magnetron sputtering system and extended closed-drift Hall-current ion source. In spite of the rather big length (up to 2m-long) these devices can be adjusted to generate sufficiently uniform plasma along them ($\Delta n_i/n_i \sim 10\%$). This approach is very convenient for processing of flat large-area substrate, but the deposition set-up can be fitted to treat other kinds of substrates, for instance, cutting tools and dies. To improve quality of the coatings, during deposition of hard a-C films the magnetrons

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can operate in unbalanced mode to intensify ion bombardment of the growing film surface during PVD, and thus, to approach cathodic-arc PVD. The ion/plasma sources can be used for preliminary cleaning and implantation of the substrates, or for CVD of hard a-C:H films.

In this paper we describe the high throughput bath-type installation for hard carbon and hydrocarbon films deposition by the PVD and CVD methods. The installation has been created at HCEI under ISTC project No. 2438 performances.

2. Installation

The installation created is intended to deposit hard amorphous carbon and hydrocarbon coatings on the substrates with dimensions up to 600×600×600 mm^3. Fig. 1 presents the installation schematics. The installation consists of a vacuum chamber *1* with a pumping-out system *2* and a control rack *3*. The vacuum chamber *1* has the cube form with the face dimension of 1 m and is made of stainless steel. Double walls of the chamber form a water cooling jacket. The chamber has two doors and is pumped-out with the vacuum system *2* based on the roughing-down pump NVR-90 and diffusive aggregate AVDM-400.

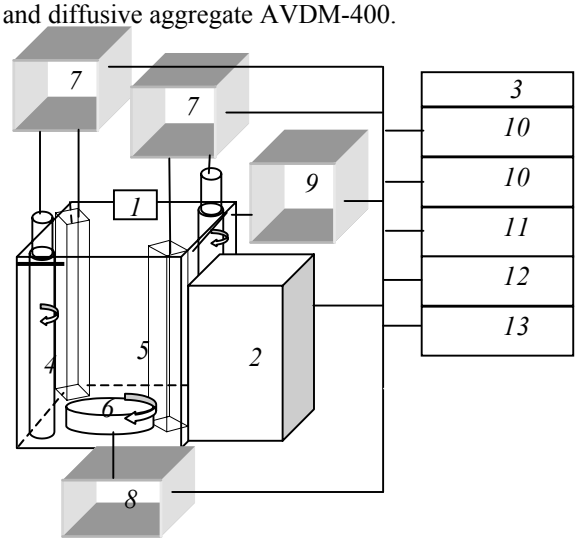

Fig. 1. Installation schematics: *1* – vacuum chamber; *2* – pumping-out system; *3* – control rack; *4* – magnetron; *5* – ion source; *6* – rotating table; *7*, *8* – power supplies; *9* – gas-filling system; *10*, *11* – power supply control consoles; *12* – vacuum system control console; *13* – gas-filling control console

Inside the chamber there are placed two unbalanced magnetron sputtering systems *4* with cylindrical rotating graphite cathodes with a 850-mm long sputtering part of the target and two extended closed electron drift ion sources *5* forming a 800-mm extension ion source. The treated parts can be placed on a rotating table *6* having 20 fixing positions with planetary rotation. Each of two power supplies *7* can operate either with the magnetron or with the ion source; hence, two technological sources can be turned-on simultaneously in any set. The pulsed power supply *8* is intended to apply the bias potential to the treated component parts. The installation is equipped with a two-channel gas-filling system *9*. In the control rack *3* the remote control consoles of the power supply, vacuum system and gas-filling are placed.

Figure 2 presents the view of a magnetron sputtering system with a graphite cathode. Cathode rotation is realized by means of a motor-reductor *1* through a vacuum-tight rotary motion feed-through *2*. The cathode magnetron *3* is made of high-density graphite. The total cathode length is 900 mm. The cathode consists of seven graphite tubes pasted to the common stainless steel base. Magnetic system of the magnetron is made of permanent SmCo magnets. An important characteristic of the extended magnetrons is the uniformity of the sputtering velocity along the target determining uniformity of the coating deposition velocity. To increase the zone of homogeneous coating deposition, magnets with increased magnetic field inductance are placed near the ends of the magnetron magnetic system. Our previous researches [5] have shown that use of such magnetic system allows extending the coating deposition range by 10–20 cm with homogeneity no worse than 2%.

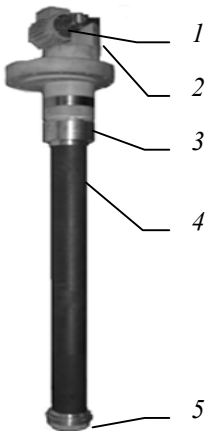

Fig. 2. External view of magnetron sputtering system with rotating graphite cathode: *1* – motor-reductor, *2* – rotary motion feedthrough, *3* – upper shield, *4* – graphite cathode, *5* – lower shield

Figure 3 presents the thickness homogeneity measurement results of the coating deposited to a substrate placed 6 cm apart the cathode. Non-homogeneity of the coating thickness in the central zone is in the limits of measurement accuracy and does not exceed ± 2%.

When depositing hard carbon coatings using method of graphite magnetron sputtering it is necessary to provide correspondent conditions of ion bombardment of the growing film surface. To increase plasma density near the substrate, additional unbalanced magnets placed at a 5-cm distance from the target surface were used in the described installation. Fig. 4 demonstrates influence of the additional magnets on the ion current density to a substrate.

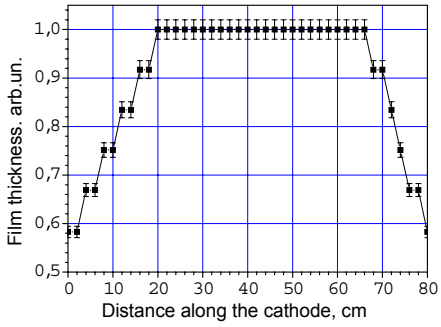

Fig. 3. Measurement results of coating thickness homogeneity along substrate 6 cm apart magnetron target

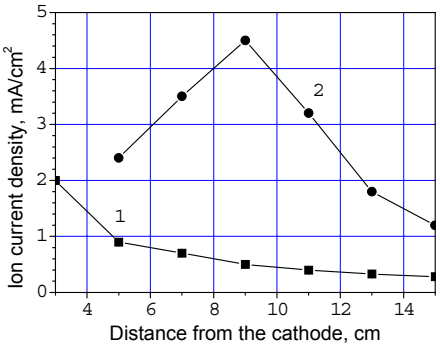

Fig. 4. Ion current density to substrate versus the cathode-substrate distance without additional magnets (1) and with additional magnets (2)

Use of additional magnets allows raising the ion current density by 2–4 times that allows increasing the depth of the substrate efficient ion bombardment from 5–6 cm (without using additional magnets) to 15–17 cm (using additional magnets). In its turn, this provides optimum conditions for hard film deposition at essential distances from the cathode and allows increasing the treated component parts.

Design and characteristics of the ion sources with closed electron drift are described in detail in [6]. In the described installation, the ion sources are applied both for preliminary surface cleaning and in the process of CVD of carbon films from the methane-argon mixture. Low-power mode of the ion source operation in which a collimated ion beam is formed is the most reasonable mode for surface cleaning. “Plasma” mode characterized by the presence of additional discharge in the vacuum chamber can be applied for PECVD.

Power supplies of technological devices are made on the modern element base and have microcontroller steering. Pulsed operation mode of magnetrons with adjustment of the pulse rate and pulse length as well as DC mode of the ion source operation are envisaged. The sources can operate in the modes of current and voltage stabilization, their power being of 8 kW. More detail description of the power supplies is given in [7].

3. Films Deposition

The first experiments on deposition of amorphous carbon films by the magnetron sputtering systems (assisted with pulsed high-frequency low-voltage negative substrate bias) and on deposition of hydrocarbon films by closed-drift ion sources have been performed. The films thickness has been measured by microscope-interferometer, their surface morphology has been studied by AFM, and their mechanical properties have been measured by nanoindentation with Berkovich indenter, correspondingly.

Parameters of pulsed magnetron discharge (current of 20 A, voltage of 660 V, pulse width of 300 μs, and pulse repetition rate of 1 kHz) have been fixed during a-C films deposition experiments. Argon pressure in the vacuum chamber was equal to 0.2 Pa. Silicon crystalline substrates have been placed 10 cm apart the magnetron target. Substrate bias pulse width (10 μs) and repetition rate (20 kHz) have been fixed while pulse amplitude has been varied from 0 to –600 V. In order to improve a-C films adhesion and to minimize their inherent intrinsic stresses, the substrate biasing of 2 min has been followed by pause of 5 s.

The hardest coating (26 GPa) was deposited at the substrate bias amplitude of –400 V, and its elastic modulus was equal to 333 GPa. One can see very smooth surface morphology that is caused by amorphous structure of the coating (Fig. 5). Growth rate of the coating was about 1 micron per hour. It is necessary to note that mechanical characteristics of the coatings are close to those of a-C films deposited by laser ablation or unbalanced magnetron sputtering of graphite. Amorphous hydrogenated films deposited by PIID are also similar to these coatings. Only ta-C films deposited by filtered vacuum cathode arc are characterized by higher hardness and elastic modulus, as a rule.

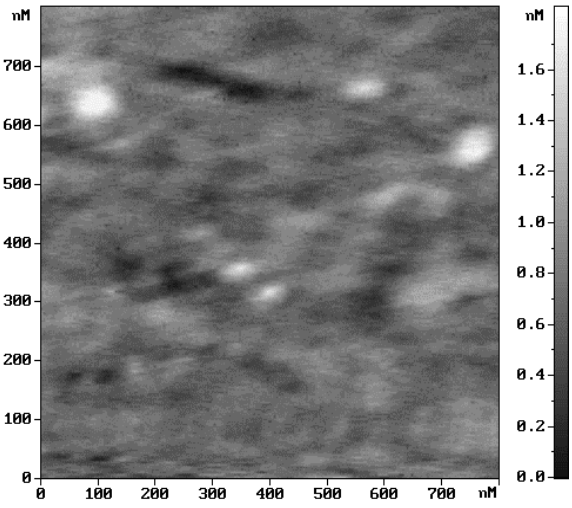

Fig. 5. AFM surface image for a-C film deposited by pulsed magnetron sputtering of graphite

Ion (or collimated) mode of operation of closed-drift ion source has been used for deposition of hard a-C:H films on this stage. Methane has been introduced into the vacuum chamber through the ion source, and its pressure was equal to 0.3 Pa. Anode voltage was

equal to +850 V, and ion beam current was about 0.3 A, correspondingly. Crystalline silicon and glass substrates were placed 10 cm apart the ion source.

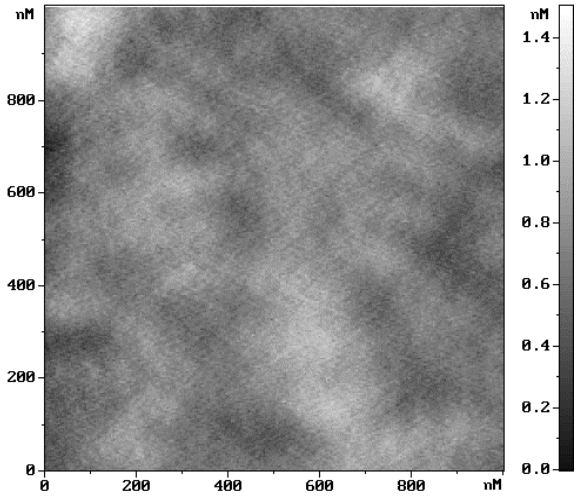

Fig. 6. AFM surface image for a-C:H film deposited by closed-drift ion source

The films deposited on both glass and silicon substrates were amorphous as it followed from their surface morphology (Fig. 6). As in the previous case, the films growth rate was about 1 micron per hour. Hardness of the a-C:H films deposited on silicon and glass substrates was measured as 13.5 GPa and 11 GPa, while their elastic modulus was measured as 134 GPa and 90 GPA, correspondingly. Probably, the reason of the a-C:H film softening in the case of glass is positive charge build-up at the dielectric substrate surface, which is followed by decrease of ion bombardment of the growing film.

4. Conclusion

Testing and carrying out of the first series of experiments on deposition of carbon and hydrocarbon coatings have shown that the created installation is promising for solving a series of applied technological tasks related to deposition of hard carbon and hydrocarbon coatings on large component parts.

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