

SOME ASPECTS OF DEPOSITION OF CONDUCTIVE, DIELECTRIC AND PROTECTIVE COATINGS ON INSULATORS WITH USING ARC DISCHARGE DC AND RF BIAS

V.S. Taran, R.M. Muratov, Y.N. Nezovibat'ko, A.V. Leonovych, M.A. Sergiiets
Institute of Plasma Physics of the NSC KIPT, Kharkov, Ukraine
E-mail: vtaran@kipt.kharkov.ua

Investigation results for technological process of low temperature plasma deposition of functional coverings for dielectric substrate at low temperatures (50...250 °C) are shown. Combined high frequency and arc plasma sources were used to provide high deposition rate and an opportunity to operate with heat sensitive substrate such as plastic, glass etc. Using this method there were obtained: pads on detectors of ionizing radiation, optically transparent protecting coverings for plexiglass, connecting coverings on mica for ultra-frequency emitter.

PACS: 52.77.Dq, 51.30.+i

INTRODUCTION

The aim of this work was to implement and test the method of applying different coatings onto dielectric substrate, operating with Arc discharge and RF bias.

RF bias of a certain frequency (5 MHz) and a variable constant voltage bias to a substrate with a different period of application were investigated.

To operate with dielectric substrate it is necessary to provide RF bias to a multi loop antenna which surrounds the sample. This assistant RF field provides additional ionization and removes the charge accumulated on the surface of the sample.

Experimental results prove that this method improves the coatings quality and keeps good adhesion while reduced temperature during workflow.

Nowadays the variety of thin coatings is commonly used in different technological applications. Coatings which are used in electronics as a contact for sensitive equipment are required to have an ideal surface morphology to maintain the accuracy of measuring devices [1], optically transparent protective coatings are widely used in optics and everyday life in the role of shielding for gadgets. Production such type of coating has its own features, considering the fact that most of the materials that are used for the substrate are dielectric or sensitive to overheating. In order to understand the problem completely it is very important to clarify the difference between process of sputtering onto metal and dielectric substrate.

In the first case we have conductive and more or less refractory substrate material, therefore it is implied that it is possible to provide a negative bias directly to substrate in order to improve the quality of the coating [2]. If the material is refractory enough we can simply increase bias to make deposited ions have more energy for better adhesion of the final coating.

In the second case we do not have such possibility course material is not conductive. So we can provide a negative bias to a base behind the sample to make needed potential in the area of the sample.

There still will be a problem of accumulated surface charge. To avoid it the RF field that relieves this charge can be applied to antenna surrounding the sample. But dielectric materials often are sensitive to overheating.

So increasing bias on the base behind the sample will not work in this case.

The technology of direct plasma deposition is well known. The next obvious step in improving the quality of coatings is, getting rid of the drop component in the plasma flow. There is a large growth of the droplet phase at the moment of arc ignition. The last inflict the most damage to the sample surface when reaching it. That's why we can observe such variety of plasma filters with different architecture to solve this problem [3]. The opposite effect of using filters is the reduction of flux density that affects the deposition rate.

Further improvement of the process is the use of both the high-frequency and arc plasma sources. Glowing discharge provide finishing cleaning by bombarding the substrate surface with ions of neutral gas. Simultaneous use of these two sources provides such bonuses as activation of the surface and additional ionization during the workflow. The RF bias can be applied rather to conductive substrate and vacuum chamber or to a multi loop antenna surrounding the dielectric sample. The antenna geometry and frequency of bias can variate from one work to another depending on the sample geometry and investigation condition [4]. Work with dielectric and heat sensitive materials should be focused on pulsed regimes that prevent overheating. Temperature of sample can be hold under control if the combination of spattering – not spattering phases is used. During the first phase the surface is heated and after that goes the cooling one. That process can be miniaturized by applying RF bias via impulse generator that can provide more sophisticated settings of the assisting RF field.

EXPERIMENTAL SETUP

The experiments were carried out on Bulat-6 type device, additionally equipped with a high frequency generator. Fig. 1 schematically shows the device for RF-cleaning and ion-plasma assisted deposition.

According to the experimental needs samples can be installed in special containers before being placed into multiloop-RF antenna. The form of antenna is various according to the geometry of the samples and experimental needs.

The presence of three arc evaporators provide the possibility to sputter multilayer in one working process. One evaporator slot equipped with a plasma filter with an open architecture.

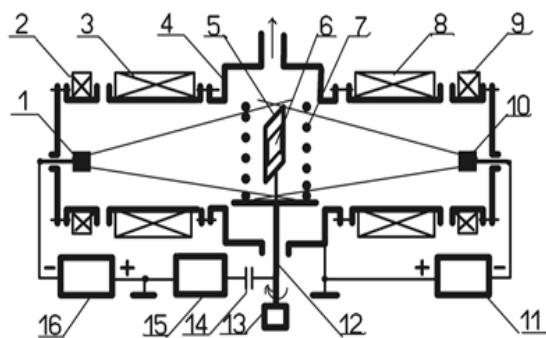


Fig. 1. Scheme of the experimental setup: 1, 10 – cathode; 2, 9 – stabilizing coils; 3, 8 – focusing coil; 4 – vacuum chamber; 5 – container (lining); 6 – sample; 7 – RF antenna; 11, 16 – power supply arcs; 12 – turning device; 13 – gear; 14 – capacitor; 15 – RF generator

RESULTS AND DISCUSSIONS

Deposition of metal contacts on to CVD (chemical vapor deposition) diamond samples was carried out on Bulat-6 type device. To perform in RF-cleaning and further metal applying crystal samples of the polycrystalline CVD diamond were placed in a special container. Container with samples was placed inside an electrode in the form of a multiloop RF-antenna. This construction was mounted on a rotating device in the center of the vacuum chamber against the Cr and SS arc evaporators. Rotating table with sample was connected to the RF generator via a capacitance.

The cleaning process was started simultaneously with rotating device. Neutral Ar gas inlets and supplies RF voltage to the target (sample container, RF-coil). Approximate cleaning mode: the shifting voltage $U_s = -(700 \dots 900) \text{ V}$; pressure $P(\text{Ar}) = 2 \cdot 10^{-1} \text{ Pa}$. The cleaning lasted for 3...5 minutes, and temperature was not higher than $60 \text{ }^\circ\text{C}$.

The advantages of plasma method of ion cleaning are in its uniformity allowed processing of details of a difficult form, and simplicity of technical realization. The application of coatings starts immediately after cleaning. Cr and then SS contacts applies in a single technological cycle. A characteristic feature is pulsed operation of the evaporator on a particular program. The plasma ion method lead to heating the substrate (container with the sample) up to a temperature above permissible, therefore, there was designed a pulse mode of operation of the evaporator with switched RF voltage (assisted sputtering). In such a mode, the temperature does not exceed $120 \text{ }^\circ\text{C}$. Combination of plasma arc discharge with RF discharge significantly increases the adhesion of the film, helps to seal texture coating and significantly reduces droplets formation

First priority task was finding suitable material to create metal contacts. It should have suitable properties for use in harsh conditions, but in the same time save

detector accuracy. Au and Cr were chosen as possible candidates. Comparison of materials for contact was carried out by analyzing the CVC and count rate of the samples with contacts obtained by magnetron sputtering.

Then Au contact was chemically removed and Cr contacts were applied instead. Afterwards, the study of electro-physical properties was repeated. Differences in characteristics of the detectors can be seen in Fig. 2.

The pulse-height distributions of the detector signal under α irradiation from ^{239}Pu source for detectors with different contact materials (Cr, Au) are shown in Fig. 3.

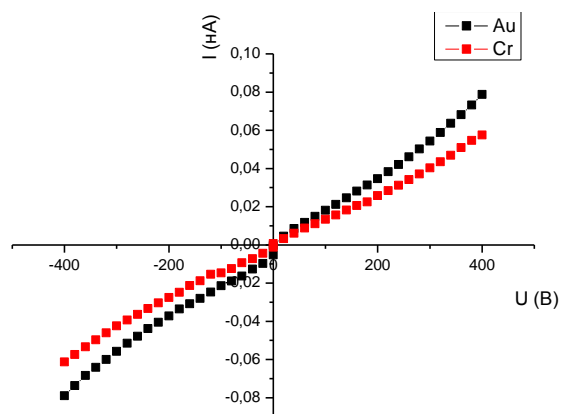


Fig. 2. Current-voltage characteristic polycrystalline diamond pattern with different contracts

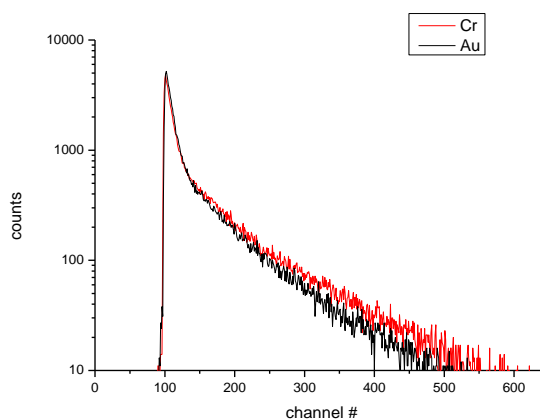


Fig. 3. The pulse-height distributions of the detector signal under α irradiation from ^{239}Pu source for different contact materials (Au, Cr)

These distributions could be considered to be equal, the existing differences are within the reproducibility of the experiment. Based on these results, in further experiments Cr was used as a main contact material for developing CVD diamond detectors, because of its significantly greater adhesion to the rough surface of unpolished CVD diamond.

The main purpose of depositing bilayer contacts is solving the problem of connecting detector to the readout electronics in the operating conditions, when heating above $T=120 \text{ }^\circ\text{C}$ is possible, because at this temperature the degradation of conductive adhesive contact occurs. One of solutions of this problem is the connection to readout electronics by soldering. Bilayer

contacts were deposited on CVD diamond samples, where the first layer was chromium, and the second layer – stainless steel.

In Fig. 4. it is shown the CVC of two samples with the smallest and largest values of electrical resistance. Registration of the pulse-height distributions of the detector signal under α irradiation from ^{239}Pu source performed with the following parameters of spectrometric tract: $U_{\text{bias}} = 400 \text{ V}$, the shaping amplifier gain – 1000, shaping time $\tau = 1 \mu\text{s}$, acquisition live time – 60 minutes.

In Fig. 5. presented an alpha particles counting rate for investigated detectors. The difference between investigated samples supposed to be due to inner defects.

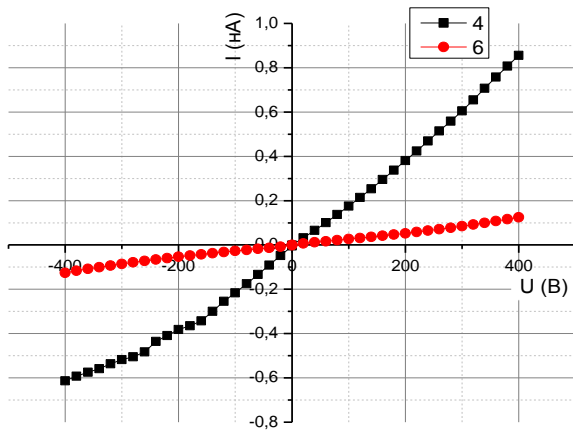


Fig. 4. CVC sample with the lowest and highest values of electrical resistance

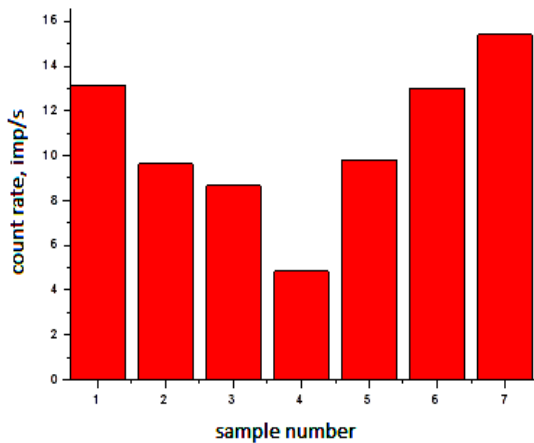


Fig. 5. Speed counting the registration of alpha particles

Analog sensitivity of detectors in the fields of electron radiation and bremsstrahlung with energy of $\sim 10 \text{ MeV}$ was measured on linear electron accelerator LU-10.

Further investigation of this method was carried in work when transparency in visible spectrum was required for protective coatings for plexiglass. The first stage is cleaning process ion bombarding in Neutral Ar gas $5 \times 10^{-1} \text{ Pa}$, $U_{\text{RF}} = -500$ during 20 min. The voltage was to the target (sample container, RF-coil). Approximate cleaning mode: the shifting voltage $U_s = -(900 \dots 700) \text{ V}$; pressure $P(\text{Ar}) = 2 \cdot 10^{-1} \text{ Pa}$. The

cleaning lasted for 3...5 min, and temperature was not higher than $60 \text{ }^\circ\text{C}$. Deposition phase $U_{\text{bias}} = -(230 \dots 360) \text{ V}$, $I_a = 100 \text{ A}$.

Variety of exposition time during the deposition process gives us different transparency Fig. 6. Conductive contoured coatings were also carried for mica details required for use in the windows of microwave energy output.

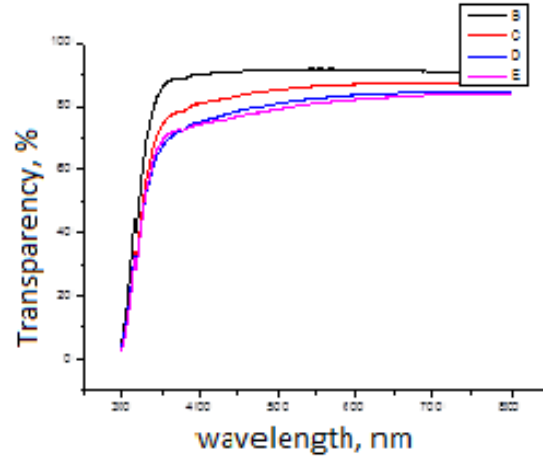


Fig. 6. The dependence of transparency of the wavelength. Depositing time: B – 3 min; C – 4 min; D, E – 5 min

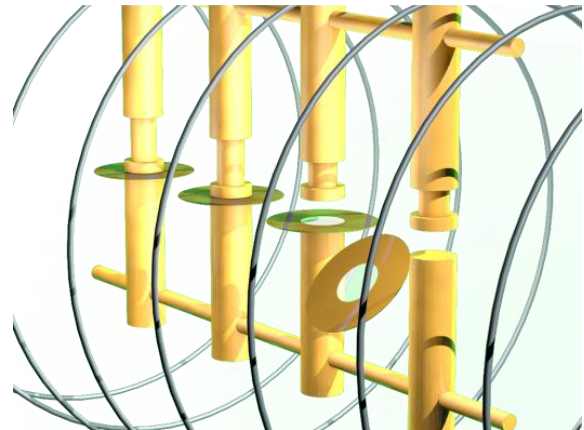


Fig. 7. Masking container for mica samples

Windows of energy output are used in RF frequency electrovacuum devices with millimeter and submillimeter diapasons such as: klinotrones, generators of the diffraction radiation etc. The use of the produced parts will improve metal-glass output windows that are used now. It improves their manufacturability and reliability, and in its turn improves the structural reliability of the device as a whole.

Approximate cleaning mode: the shifting voltage $U_s = -400 \dots 450 \text{ V}$; pressure $P(\text{Ar}) = 2 \cdot 10^{-1} \text{ Pa}$. The cleaning lasted for 3...5 min, and temperature was not higher than $60 \text{ }^\circ\text{C}$. Deposition phase $U_{\text{bias}} = -(90 \dots 50) \text{ V}$, $I_a = 100 \text{ A}$.

CONCLUSIONS

In this article it was shown the usage of combined high frequency and arc plasma sources for applying conducting, dielectric and protecting coatings onto

different dielectric substrates at low temperatures (50...250 °C). The aim of the study was to find the most optimal technological regime of deposition depending on the substrate material. This method provided high deposition rate and gave an opportunity to operate with heat sensitive substrates such as plastic, glass etc. Plasma filter with open architecture was used to decrease droplet fraction which is the main problem of obtaining high-quality coatings. Assisting RF bias played the primary role in deposition process. In case of dielectric as a substrate material there is a problem of accumulated surface charge and usage of the RF bias could handle it.

The obtained samples possessed all required properties and were tested in working regimes.

This work has been performed in part within the STCU-NASU project #6183.

REFERENCES

1. A.V. Leonovych, V.S. Taran. Combined high frequency and arc plasma sources for contacts

application // *Europhysics conference abstracts*, 2015, vol. 39E ISBN 2-914771-98-3, P1.305.

2. V.P. Tabakov. *Formation of wear-resistant ion-plasma coatings of the cutting tool*. Moscow: "Engineering", 2008, p. 59-62.

3. A.I. Ryabchikov, I.A. Ryabchikov, I.B. Stepanov, Y.P. Usov. High-frequency short-pulsed metal plasma-immersion ion implantation or deposition using filtered DC vacuum-arc plasma // *Surface and Coatings Technology*. 2007, v. 201(15), p. 6523-6525; doi: 10.1016/j.surfcoat.2006.09.053.

4. Nigamananda Samal, Hui Du, Russell Luberoff, Krishna Chetry, Randhir Bubber, Alan Hayes, and Adrian Devasahayam. Low-temperature ($\leq 200^\circ\text{C}$) plasma enhanced atomic layer deposition of dense titaniumnitride thin films // *Journal of Vacuum Science and Technology A*. 2013, v. 31, p. 01A137; doi: 10.1116/1.4769204.

Article received 22.01.2017

НЕКОТОРЫЕ АСПЕКТЫ ОСАЖДЕНИЯ ПРОВОДЯЩИХ, ДИЭЛЕКТРИЧЕСКИХ И ЗАЩИТНЫХ ПОКРЫТИЙ НА ИЗОЛЯТОРЫ С ИСПОЛЬЗОВАНИЕМ ДУГОВОГО РАЗРЯДА И ВЧ-СМЕЩЕНИЯ

В.С. Таран, Р.М. Муратов, Ю.Н. Незовибатько, А.В. Леонович, М.А. Сергиец

Показаны результаты исследования технологического процесса низкотемпературного плазменного осаждения функциональных покрытий для диэлектрической подложки при низких температурах (50...250 °C). Для обеспечения высокой скорости осаждения и возможности работать с теплочувствительными подложками, такими как пластик, стекло, были использованы комбинированные высокочастотные и дуговые источники плазмы. С помощью этого метода были получены: детекторы ионизирующего излучения, оптически прозрачные покрытия для плексигласа, соединительные покрытия на слюде для ультрачастотного излучателя.

ДЕЯКІ АСПЕКТИ ОСАДЖЕННЯ ПРОВІДНИХ, ДІЕЛЕКТРИЧНИХ І ЗАХІСТНИХ ПОКРИТТІВ НА ІЗОЛЯТОРИ З ВИКОРИСТАННЯМ ДУГОВОГО РОЗРЯДУ ТА ВЧ-ЗМІЩЕННЯ

В.С. Таран, Р.М. Муратов, Ю.М. Незовибатько, А.В. Леонович, М.А. Сергієць

Показано результати дослідження технологічного процесу низькотемпературного плазмового осадження функціональних покриттів для діелектричної підкладки, при низьких температурах (50...250 °C). Комбіновані високочастотний і дуговий джерела плазми були використані для забезпечення високої швидкості осадження і можливість працювати з теплочутливою підкладкою, такою як пластик, скло і т. д. За допомогою цього методу були отримані: детектори іонізуючого випромінювання, оптично прозорі захисні покриття для плексигласу, з'єднувальні покриття на слюді для ультрачастотного випромінювача.