A THIN FILM PREPARATION USING FOCUSED HIGH POWER ION BEAM.

Vit.M.Bystritskii, S.N.Volkov, A.V.Mytnikov and A.A.Sinebryukhov
Institute of Electrophysics RAS, 4 Academy ave., Tomsk 634055, RUSSIA
D.J.Rej
Los Alamos National Laboratory, Los Alamos, NM 87545, USA

Abstract.

Among the well known physical methods of thin film preparation as target evaporation by magnetron irradiation, by laser, electron and long pulse ion beams, the method of precipitation of evaporated target material with short pulse (<100 ns) and high power (>10^9 W/cm^2) ion beam seems to be less investigated. The advantages of such method are:

1. high precipitation velocity (1 μm per shot);
2. possibility of film formation at high area substrate (100 cm^2);
3. moderate vacuum conditions needed (10^{-4} - 10^{-5} Torr) - two order higher compared to other methods;
4. high melting temperature target usage.

These advantages are provided by action of pulsed ion beam at the target, when the effective energy transfer into target occurs, but irradiation energy losses are minimal. The thickness of evaporated layer of the target is determined by ion range in material (usually 1 - 10 μm) providing high velocity precipitation. Pulsed action of target plasma results in thermal and mechanical dynamic loading, that is important for film properties.

The usage of described method for formation of diamond like is of the most interest. In order to precipitate the carbon films we used high power ion beam with the following parameters: E = 600 keV, I = 60 kA , t =80 ns. Ion beam was generated in focusing B-applied diode at GIMN accelerator. The ion beam consisted of approximately 70% of protons and 30% of carbon ions. The ion current density in the focal plane reached 9 kA/cm^2 that corresponds to 300 J/cm^2.

Thickness of the precipitated films made up to 100 nm per shot at the distance between target and substrate 4 cm. In the case of glass substrate, its surface layer was strongly destructed. The electrical resistance of film was at the level of 100 Ohm. In the report the ellypsometric data obtained with scanning electron microscope are given.

Introduction.

Pulsed ion beams of wide range of pulsed power (10^9 - 10^{12} W and more) with nanosecond pulsed duration are the unique means for study of the processes beam-solid interaction. This is conditioned by high power density of ion beams, short ion range in the condensed matter with corresponding high volume energy density resulting in surface heating and evaporation. The characteristic time of the heat transition in the condensed matter do not exceed several microseconds but acoustic and shot wave velocity reaches several km/s. That is why the deposited energy remains in the near surface layer making the process to be adiabatic. Generated ablation plasma of a high density (up to 10^{20} cm^{-3} near the target) and of a high temperature (≈ 100 eV) can be deposed to a substrate, forming thin film of one component and composed materials, that is obtained firstly in[1].

There are other methods of target heating and ablation - laser and electron beam irradiation of a surface. But nevertheless those methods have some drawbacks compared to ion beam action. Despite of high laser beam energy density, the total energy of one reaches

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14. ABSTRACT
Among the well known physical methods of thin film preparation as target evaporation by magnetron irradiation, by laser, electron and long pulse ion beams, the method of precipitation of evaporated target material with short pulse (< 100 ns) and high power (>109 W/cm2) ion beam seems to be less investigated. The advantages of such method are: 1. high precipitation velocity (1 Jlm per shot); 2. possibility offilm formation at high area substrate (100 cm2); 3. moderate vacuum conditions needed (10"4 - 10"5 Torr) - two order higher compared to other methods; 4. high melting temperature target usage. These advantages are provided by action of pulsed ion beam at the target, when the effective energy transfer into target occurs, but irradiation energy losses are minimal. The thickness of evaporated layer of the target is determined by ion range in material (usually 1 - 10 Jlm) providing high velocity precipitation. Pulsed action of target plasma results in thermal and mechanical dynamic loading, that is important for film properties. The usage of described method for formation of diamond like is of the most interest. In order to precipitate the carbon films we used high power ion beam with the following parameters: E = 600 keV, I= 60 kA, t =80 ns. Ion beam was generated in focusing B-applied diode at GIMN accelerator. The ion beam consisted of approximately 70% of protons and 30% of carbon ions. The ion current density in the focal plane reached 9 kA/cm2 that corresponds to 300 J/cm2

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usually less then tens of joules, that requires its reprate operation. Also the self screening of laser beam takes place especially in case of metal targets. Electron beam range in materials is several orders higher, that requires less electron energy in the beam and higher current density. Lower deposited energy density leads to lower current density at the target - 40 -150 A/cm^2, $t_p \approx 60$ ns, beam composition: 80\% of carbon ions, 20\%-protons. Accelerator repetition rate was of 0.3 Hz. The parameters of the ablation plasma are: $n = 10^{20}$ cm^{-3}, T_e =1 eV, plasma spot area - 20 cm^2. The distance from target to substrate was 2 cm, film deposition velocity 10^5 nm/s in the vacuum of 10^{-4} Torr and ion beam power density at the target 3 - 4.5.10^7 W/cm^2. Using the described technique, authors[2] , obtained Cd, Zn, brass films with thickness about 0.3 nm per shot, Pb films - 0.5 nm/shot. The substrate had temperature 20° C. The following study [3] showed the possibility of composite films deposition (YBaCuO) with above mentioned parameters of ion beams onto MgO substrates keeping the stoichiometric composition the same. The temperature of substrate was about 300 - 450 ° C in these experiments.

In[4] authors, using TEMP accelerator and ion beam with given parameters, tested the possibility to apply the ablation plasma from Pb target for active lashing media of metal vapor. The target vaporization energy (U_s = 0.96 J/g) was reached at 30 - 40 ns of pulse duration. Then the beam excited the created plasma, which moves with a velocity of 10^7 cm/s. The measured plasma parameters for Cu-target were the following: T_e = 0.6 eV, n_e = (2 - 5).10^{17}, vapor density -3.10^{19}, at the t=100 ns, heavy particles temperature - 5000 K, ionization degree -10^{-2}.

In[5-7] results of ion beam focusing in the B-applied ion beam diode are published. The accelerator parameters are: U = 0.5 MV, t = 80 ns, ρ = 8 Ohm, power in matched load regime - 10^{10} W. Beam power density at the target made up to 10^9 W/cm^2, ion beam current density - 1.5 kA/cm^2, beam composition: 80% of hydrogen ions, 20% of carbon ions.

Three regimes of beam-target interaction were found:

1. Thermal j < 40 A/cm^2
2. Intermediate 40 < j < 1000 A/cm^2
3. Gas-plasma dynamic regime at j > 1 kA/cm^2 with transformation over 70% of beam energy to gas-plasma phase and strong acoustic and shock waves formation.

It is shown, that ablation plasma moves with falling down velocity from 10^7 cm/s (at n =10^{16} cm^{-3} and t = 10^{-7} s) to (1 - 1. 5) 10^6 cm/s (at n = 10^{14} cm^{-3} and t=10^{-6}s).

The results of evaporated mass measurement under irradiation of ion beam with current density of 1.2 kA/cm^2 are following: Al - 2.06 mg/cm^2; Cu - 3. 99 mg/cm^2, Pb - 27.22 mg/cm^2. The estimate average pulsed pressure due to ablation plasma action made using evaporated mass measurement gives the value P_{abl} = 2 - 4 kBar.

The following study[8] was indicated that ion beam power increase in the focal plane due to modification of the diode hardware and due to neutralization of ion beam space charge applying external plasma source.

The following results were obtained:

1. 30% of beam energy can be focused into the spot with diameter of 10 mm.
2. Power and energy densities made up to 6.10^9 W/cm^2 and 300 J/cm^2 in plasma focusing experiment; and in vacuum: 3.10^9 and 200 J/cm^2 correspondingly.
3. Focus current density j = 10 kA/cm^2.

Protons with the energy over 500 keV carry 40% of total beam energy into focal plane.

Experimental set up and diagnostics.

The experiments were performed at GIMN accelerator with output voltage of 600 kV, current of 150 kA and pulse duration of 80 ns in matched load regime. We used a magnetically

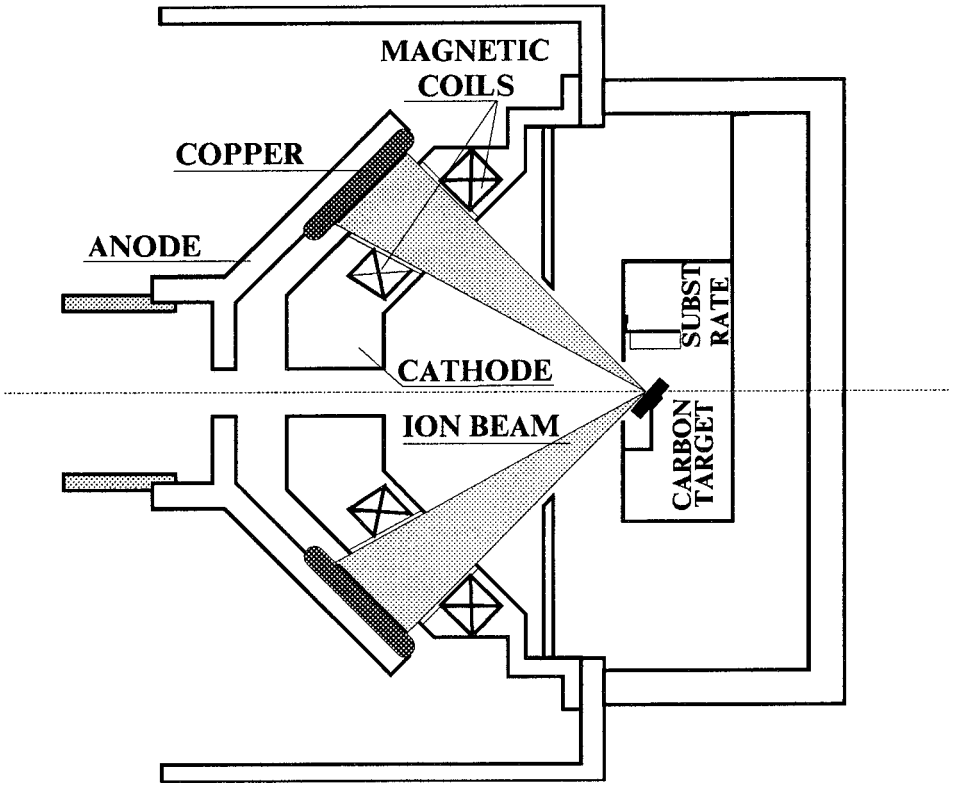
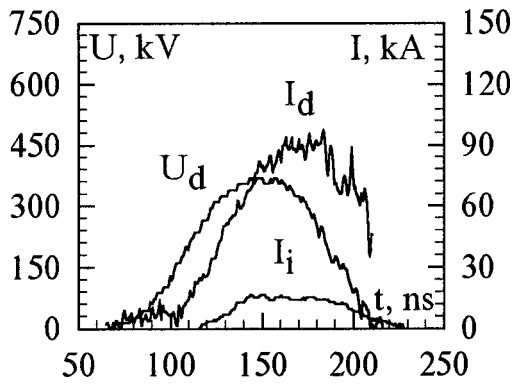


Figure 1. The drawing of the vacuum chamber.

insulated ion beam diode with applied B-field for the HPIB generation. The area of the plasma creating surface was equal to 120 cm^2, its curvature radius - 12 cm. We used as a dielectric perforated polyethylene. The schematic of the vacuum chamber is given at the Fig.1.

The electrical parameters are following: diode voltage - 500 kV, total diode current - 90 kA, ion current - 25 kA, pulse duration - 80 ns. The characteristical waveforms of the diode voltage, total and ion current are given at Fig.2.



Figire 2. The characteristical waveforms of the diode voltage, total and ion currents.

The maximum diameter of the beam in the focal plane was equal to 4.5 cm. The proton current density in the focal plane measured by the activation method in reaction $^{12}C(p,\gamma)^{13}N$ (E_p>500 keV) was about 3.8 kA/cm^2, that corresponds to total ion current density of 9 kA/cm^2 and energy deposition density of 200 J/cm^2. The radial ion current density distribution in the focal plane is given in the Fig.3.

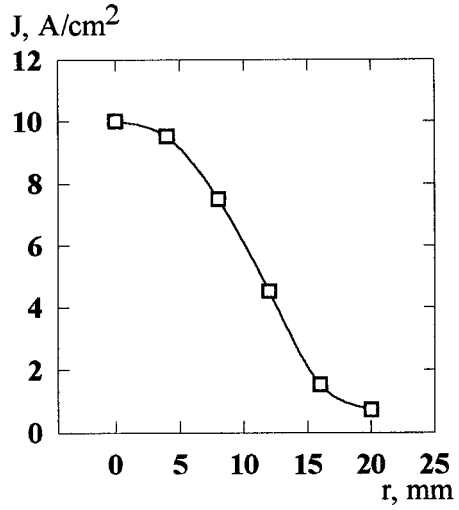


Figure 3. The ion current density distribution in the focal plane.

Experimental results.

We used pyrolythic carbon of MGP-6 type as a target and glass and silicon substrates. The thickness of the films was measured by laser ellipsometer with 10 Å accuracy. The film thickness after one shot increased on 40 - 200 nm, depending to the target - substrate distance.

The main two problems were following:

1. The soiling of the film surface by the evaporated elements of the diode.
2. Exfoilation of the deposited film from the substrate.

In the first case the Auger analysis showed the presence of Zn, Cu and o atoms in the surface layer. This fact may be coupled with evaporation of the brass field excluder by ion beam. We also obtained precipitation of the anode surface breakdown products ($(CH_2)_n$). To decrease of the soiling of the film we used various shields. The better results were obtained with the shield, shown in the Fig. 4.

The second problem may be explained with various processes:

- the soiling of the film surface;
- different values of the thermal expansion coefficient at film and substrate;
- accumulation of the electrical charge.

The second reason may be most probable. we obtained the exfoilation for all materials of substrates in case, when the substrate was placed near the maximal energy input of ion beam.

The main electrophysical parameter of studied films is the electrical resistivity (Ohm cm). We Used two methods - microwave method and resistivity measurement by using MIM structure (metal- insulator - metal). In the first case for film formation highly resistive substrates (Al_2O_3 and glass) were used. The thickness of a film deposited for one shot is estimated of 100 - 150 nm. The measured resistivity is of 3-9 kOhm cm. The area of the studied region is 1 mm^2. Inside this area, film uniformity in thickness is 10 - 15%. The measured resistivity does not depend of substrate material. The wear dependence of resistivity versus film thickness is obtained: thick films have lower resistivity.

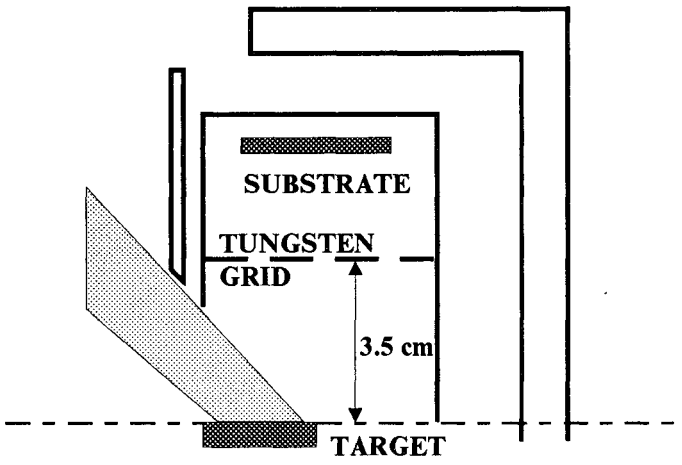


Figure 4. Drawing of the substrate - target location.

For the measurements in the second case, the following MIM structures were used: thin tungsten film was deposited onto glass substrate by cathode souttering. Then, the carbon film was deposited, with thickness of 400 - 500 nm. The film resistivity makes up to 5.10^5 - 10^6 Ohm/cm inside 1 cm^2 of deposited film. The applied voltage reaches 100 mV, upper metal layer was not deposited. After this layer formation, the resistivity dropped to 5.10^4 Ohm/cm. The difference in ρ measurement between described methods can be explained as existence of metal impurities in the near surface layer of a film as was described above.

The main problem in carbon film formation appeared to be its exfoilation off a substrate. During a fast vacuum chamber opening, the fast cooling of a system film- substrate occurs (the similar process was seen at the surface of calorimeter used for ion beam measurement. The difference of coefficients of thermal expansion of a film and a substrate should result in this process. The exfoilation is seen for films with thickness over 200 nm. In some cases, the exfoilation occurs during 10 - 20 min.

We also performed the micro hardness analysis of one sample surface. The micro hardness was measured in four points - at initial Si surface (point 1), initial film surface (point 2), film surface, eaten with acid (point 3) and film surface, eaten with ion beam (point 4). The data (in kGr/cm^2) are given in the able 1.

TABLE 1
The microhardness of the sample surface.

Measurement No.	Point 1	Point 2	Point 3	Point 4
1	642	1290	1530	2290
2	724	1290	1850	2290
3	946	1100	1850	2900
Mean value	770	1226	1743	2493

Conclusion.

The performed investigation shown the possibility of thin film preparation using high power ion beam. The growth velocity of the film reached 200 nm per shot for ion current density about 10 kA/cm^2 and ion energy of 600 keV. The film resistivity was equal to 10^5 Ohm cm, that is any order higher than initial graphite resistance. The presence of the metal impurities in the near surface layer, that required better substrate insulation from the diode. This shield may be fabricated with the gas valve.

References.

1. Shimotori, Masugata et. al J. Appl. Physics, V. 63, N. 3, 1988.

2. Goncharov O. I., Isakov I. E., Remnev G. E. 300-keV High-Power Ion-Beam Source: Practical Apllications. Proc. of 8 Int. Conf. on High-Power Particle Beams, July 2-5, 1990, Novosibirsk, USSR, V. 2, p. 1243-1248.
3. Isakov L. F., Remnev G. E., Zakutaev A. N. High-Rate Deposition of Thin Films by High-Power Ion Beam. Proc. of the 9th Int. Conf. on High-Power Particle Beams, May 25-29, 1992, Washington DC, USA, V.3, p.1966-1970.
4. Chistjakov S.A., Pak V. S., Remnev G. E. Radiation haracteristics of Plasma Produced by HPIB Interaction with Metallic Targets. Proc. 7 Int. Conf. on High-Power Particle Beams, 4-8, 1988, Karlsruhe, W. Germany, V.1., p.808-813.
5. Bystritskii V. M., Boiko V. I., Volkov S. N. et al. Generation and focusing of high-power ion beam in magnetically insulated diode with applied B-field. Proc. of 7 All Union Simposium on High Current Electronics, Tomsk, 1988, p. 97-99.
6. Bystritskii V. M., Volkov S. N. Generation and focusing of high-power ion beam in the magnetically insulated diode. Proc. of 13 Int. Sumposium on Discharged and Electrical Insulation in Vacuum, Paris, France, 1988, V.2, p.410-412.
7. Bystritskii V. M., Volkov S. N., et al. Generation and focusing of high-power ion beam in magnetically insulated diode. Fizika Plazmy (Sov.), 1989, V.15, vip.11, p.1337-1345.
8. Bystritskii V. M., Volkov S. N., et al. Generation and focusing of high-power ion beams in a magnetically insulated diode with apllied B-field. Laser and Particle Beams, 1991, vol.9, N 3, pp. 699-711.