

# Angular Divergence Control of Fast Neutral Molecules Produced through Charge Exchange Collisions of Ions Accelerated with the Use of a Single Emissive Grid

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**Abstract** – Experimental investigation results are presented of fast molecules angular divergence dependence on the beam characteristics of the molecules and emissive grid parameters of the beam source. The molecules are being produced in charge exchange collisions of ions accelerated by potential difference between a broad plasma ion emitter and a secondary plasma in a working vacuum chamber both plasmas being separated from each other with a single negatively biased emissive grid. At any grid parameters there is a threshold value of the beam permeance, which marks the angular divergence rise with a further growth of the beam current and/or with a decrease of the beam energy. Below the threshold the mean divergence angle is almost constant and amounts to several degrees, but at higher permeance it grows up to ~50 degrees. Such a high divergence angle is favorable for coating deposition on a substrate surface with high lugs and deep slots by means of universal sources of slow metal atoms and fast gas molecules, which simultaneously enter the working chamber through one and the same emissive grid.

## 1. Introduction

To assist with ions coating deposition on conductive substrates it is enough to immerse them in plasma, which contains all needed components ionized, and to apply to them an optimum negative bias voltage. This method is widely used in many industrial vacuum deposition systems. Its main advantage is a simple ion acceleration and simultaneous treatment of the whole complex-shaped substrate surface. As disadvantages a need to use arc-handling DC bias power supplies and a high heterogeneity of ion current density over the complex-shaped substrate surface should be noted. At the substrate sharp edges the current density is many times higher than in the slots. And ion sputtering often leads to blunting of the tool cutting edges.

The bias voltage cannot be applied to dielectric substrates and films. In this case broad-beam sources are indispensable for the beam-assisted deposition. As the bias power supply current of industrial systems in many cases reaches 10 A and more, the beam current should also reach at least the same value at any energy ranging from 10 eV to 1 keV to ensure characteristics optimization of deposited films.

The above parameters of the beam are available when the fast neutral molecule beam sources [1, 2] are used.

The fast molecules are being produced [2] as a result of charge exchange collisions in the working vacuum chamber near the emissive grid of the beam source between accelerated ions and slow molecules. The ion acceleration-deceleration in two space charge sheaths separated from each other with one grid allows beam current density up to 10 mA/cm<sup>2</sup> at any energy of accelerated particles beginning from 10 eV up to 10 keV. The sources offer up to 10<sup>4</sup> cm<sup>2</sup> cross-section of the beam, its equivalent current up to 10 A and lead to industrial-scale beam-assisted deposition at 0.05–0.6 Pa. As compared with ions the fast neutral molecules ensure better treatment stability, reliability, eliminate damage of conductive films with sparks and reduce the number of defects, which are usually induced in dielectrics and semiconductors with charged particles.

## 2. Beam-assisted deposition of metal evaporated with vacuum arc cathode spots.

For any beam-assisted deposition at least two sources are needed: an ion beam source and a source of metal vapor. The latter may be a cathodic arc evaporator, a magnetron sputterer or an electron beam evaporator.

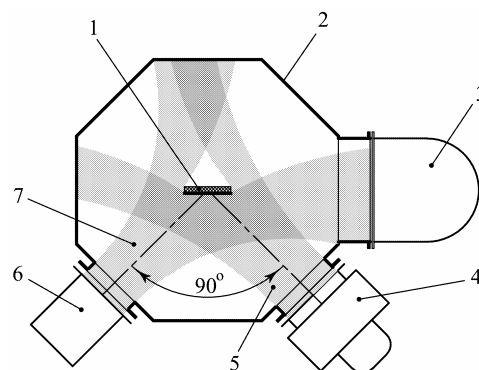


Fig. 1. Scheme of beam-assisted deposition: 1 – Al<sub>2</sub>O<sub>3</sub> substrate, 2 – chamber, 3 – diffusion pump, 4 – beam source, 5 – beam of fast nitrogen molecules, 6 – arc evaporator, 7 – flow of titanium vapor

The Fig. 1 presents a scheme of experimental film deposition using an arc source of metal vapor and a beam source of fast neutral molecules. A flat substrate 1 made of aluminum oxide is positioned in the center of a stainless vacuum chamber 2, which is pumped with a diffusion pump 3. Through a beam source 4 of fast neutral molecules the chamber 2 is being filled with a working gas. A substantial part of the gas molecules is converted into the beam 5 of fast neutral molecules.

Before deposition the substrate surface was etched during 10 minutes with 900-eV argon atoms, their beam current amounting to 0.5 A and their incidence angle amounting to 45 degrees. Then the argon was substituted for nitrogen, the beam equivalent current was increased up to 1 A, energy of fast  $N_2$  molecules reduced down to 200 eV and the arc evaporator 6 was switched on. At the arc current 100 A, gas pressure 0.4 Pa and distances from titanium target and from the beam source emissive grid to the substrate amounting to 50 cm a 5.8- $\mu$ m-thick TiN film during 2 hours was deposited its microhardness amounting to 2500HV50.

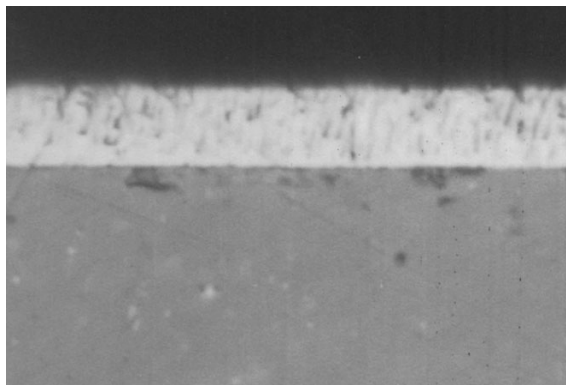


Fig. 2. Micrograph of a 5.8- $\mu$ m-thick TiN film synthesized on an  $Al_2O_3$  substrate using a flow of arc-evaporated titanium atoms and a beam of 200-eV  $N_2$  molecules

The film presented in the Fig. 2 was deposited on the substrate when incidence angles of slow Ti atoms and of fast  $N_2$  molecules both amounted to 45 degrees because the axis of the beam source 4 is normal to the axis of the arc evaporator. Films deposited at different position of the substrate are characterized with lower microhardness, density and thickness. The reasons are changes in incidence angles. For instance, when the substrate 1 (Fig. 1) is rotated 45 degrees clockwise the deposition of slow titanium atoms is not assisted with fast particles. When the substrate is rotated 45 degrees counter-clockwise the titanium atoms pass parallel to its surface and the deposition rate is very low. For this reason presented in the Fig. 1 beam-assisted deposition system with 90° angle  $\alpha$  between the axes of metal flow and beam sources cannot ensure an adequate film quality. To improve it a much lower angle is accessible. The Fig. 3 presents a

system with four 90-cm-long planar arc evaporators and two beam sources with rectangular 17×90  $cm^2$  cross-section and the angle  $\alpha=20^\circ$ . Metal plasma flows from each pair of arc evaporators 1 are mixed with the fast molecule beam from the source 2 in the region of substrates 4 loaded in the chamber 5 on the rotating system 6.

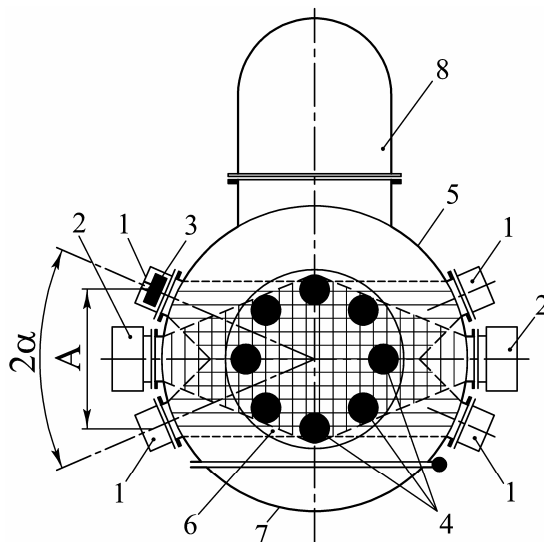


Fig. 3. Scheme of a beam-assisted deposition system: 1 – planar arc evaporators, 2 – 17×90  $cm^2$  cross-section beam sources, 3 – 90-cm-long targets, 4 – substrates, 5 – 80-cm-diam chamber, 6 – planetary rotating system, 7 – door, 8 – diffusion pump

It allows pre-treatment of the substrates 4 with 1–3 keV argon atoms and a subsequent deposition of wear-resistant or other films bombarded during their deposition with 50–200 eV gas molecules. Without any bias power supply the system ensures good quality of films deposited on the whole substrate surface except deep slots and high lugs. For a further quality improvement a further decrease is needed of the angle  $\alpha$  and of the distance A between the targets. But A is limited with the width of the beam sources 2.

For an overall overlapping of the metal flow and of the fast molecule beam it would be better to decrease the angle down to zero, to place the arc-evaporated target inside the beam source and to let the metal vapor and fast molecules enter the working vacuum chamber through one and the same emissive grid. It seems to be a very hard task to join the arc discharge, which produces metal vapor and glow discharge, which produces plasma emitter of the beam source in one and the same space.

### 3. Beam-assisted deposition by means of universal beam sources

The above problem was solved by immersing of negatively biased targets into the plasma emitters of fast neutral molecule beam sources. It turns any source into a universal source of ~10 kVA broad be-

ams of secondary electrons from the target biased to 20 kV, of 1–3 keV argon atoms for the substrates cleaning and of the metal vapor flow from the target biased to –3 kV, which is mixed with 50–200 eV molecules bombarding the film during deposition [3].

The Fig. 4, *a* presents a universal source scheme with a flat target placed at a side opposite to emissive grid of a beam source. It is well known that the angular distribution of metal atoms sputtered with ions obeys the cosine law [4]. And the beam of fast neutral molecules is remarkable for a low divergence angle.

For this reason accelerated particles, which enter the working chamber together with the metal atoms do not assist the film deposition on perpendicular to the grid 5 trench sides of the substrate 8. To find the way to divergence equalization of both slow and fast particles a research was carried out of fast neutral argon atoms divergence angle dependence on the beam characteristics and on geometrical parameters of the grid.

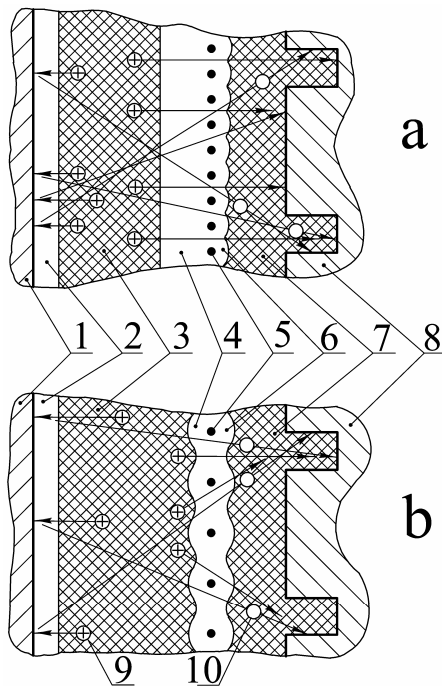


Fig. 4. Scheme of a universal beam source: 1 – negatively biased target, 2, 4 and 6 – positive space charge sheaths, 3 – plasma emitter of ions, 5 – emissive grid, 7 – secondary plasma, 8 – substrate, 9 – ions, 10 – sputtered metal atoms. a) low angular divergence of accelerated particles, b) high divergence of accelerated particles comparable with divergence of sputtered atoms

Fig. 5 presents an arrangement used to measure the angle of argon atoms divergence. It consists of a 7-cm-high, 4-cm-deep and 5-cm-wide stainless steel box with a replaceable 4×7 cm<sup>2</sup> target 2 made of 0.4-mm-thick oxidized sheet copper and two plates 3 with a slit between them. The slit width is adjustable from zero up to 5 mm using four screws 4. For the distance 4 cm from the slit to the target 2 it is easy to cal-

culate an angle  $\alpha$ , which corresponds to any width  $h$  of the print etched by fast atoms on the target surface.

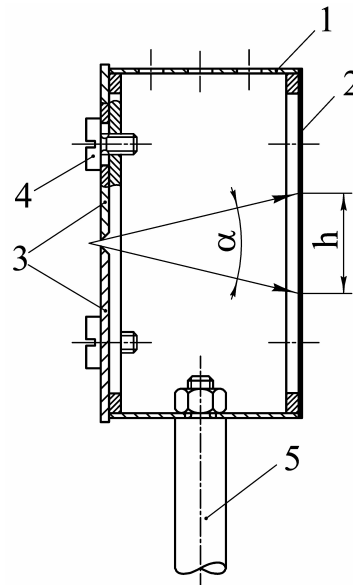


Fig. 5. Arrangement for measuring the divergence angle of fast neutral argon atoms: 1 – box, 2 – oxidized copper sheet target, 3 – plates with an adjustable slit between them, 4 – screws, 5 – rod

The Fig. 6 demonstrates dependence of the argon atoms divergence angle at a distance 20 cm from the center of a 17×90 cm<sup>2</sup> emissive grid made as a set of parallel 20-cm-long and 1.5-mm-diam steel wires and at argon pressure  $p=0.2$  Pa on the ion emission current  $I_{em}$ . The latter is the sum of ion currents in the working vacuum chamber circuit and in the grid circuit. The plates 3 were parallel to the beam source grid and the 2-mm-wide slit (Fig. 5) was parallel to the grid wires.

As at  $p=0.2$  Pa the free pass of argon ions  $\lambda_i \gg d$ , where  $d$  is width of the sheath 4 between the plasma emitter 3 and the grid 5 (Fig 4, *a*), the width  $d$  may be calculated for known accelerating voltage  $U$  and ion current density  $j=I_{em}/S$ , where the grid area  $S=1530$  cm<sup>2</sup>, using the Child-Langmuir law. Analysis of the data presented in the Fig. 6 shows that at any grid slit width there is a threshold of the beam perveance, which marks the angular divergence rise with a further growth of the beam current and/or with a decrease of the beam energy. The threshold corresponds to the width  $d$  being approximately equal to the doubled slit width. Below the threshold the mean divergence angle is almost constant and amounts to several degrees, but at higher beam perveance it may grow up to  $\sim 50^\circ$ .

When the slit between the plates 3 (Fig. 5) is perpendicular to the grid wires then in the same ranges of beam and grid parameters the angle  $\alpha < 3^\circ$ . It means that at high current and low energy of the fast atoms the beam is "polarized", i.e. angle  $\alpha$  within a plane perpendicular to the grid slits is an order of

magnitude higher than within a plane parallel to them. This result explains some peculiarities of the source presented in the Fig. 6. When it is positioned at the axis of a 80-mm-diam vacuum chamber and produces a beam of 1.5-keV argon atoms with 1 A equivalent current then the beam leaves on the chamber wall 30-cm-distant from the grid a homogeneously etched 20-cm-wide print. Equality of the print width and of the grid height may be explained with a low divergence angle in the plane parallel to the grid wires. And absence of any shadows of three 10-mm-diam rods (see the Fig. 7), which connect two cathode discs of the source to each other may be explained with a high divergence angle in the plane perpendicular to the grid wires.

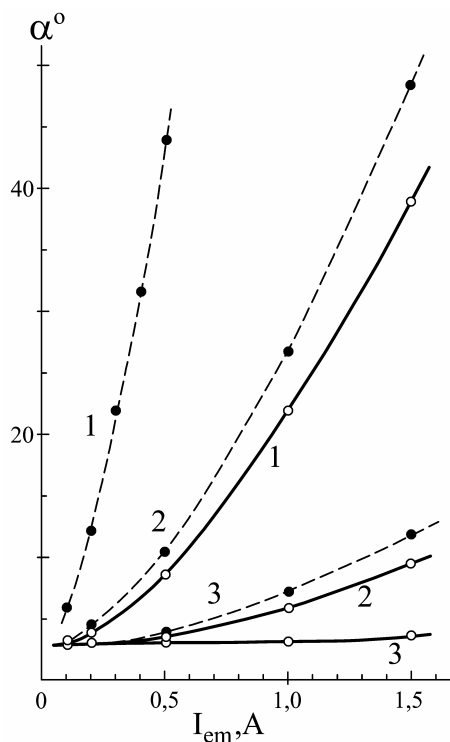


Fig. 6. Dependence on ion emission current  $I_{em}$  of fast argon atoms divergence angle  $\alpha$  at the grid slit width 7.5 mm (full curves) and 13.5 mm (hatch curves) and accelerating voltage between the plasma emitter and the grid.  $U=0,75$  kV (1), 1.5 kV, (2) and 3 kV (3)

As to the grids with a great number of equidistant holes homogeneously distributed on a metal sheet the divergence angle  $\alpha$  depends in the same way on the beam and grid parameters as presented in the Fig. 6.

## 6. Conclusions

The above results demonstrate abilities of fast neutral molecule beam sources. Generally a high-energy beam for substrate etching is remarkable for a



Fig. 7. Beam source with a cylindrical 20-cm-diam and 20-cm-high grid made of parallel 1.5-mm-diam steel wires

low divergence angle. But a low-energy beam produced using the same emissive grid and intended to increase deposited atoms mobility on the substrate surface is remarkable for high divergence angle. The latter is favorable for a beam-assisted deposition on complex-shaped substrates with lugs and slots using universal sources of metal atoms and fast molecules, which simultaneously enter the working chamber through one and the same emissive grid.

The investigation results help to choose parameters of the grid, which can make available similar angular distributions of slow metal atoms sputtered from a target and of fast particles, which assist the metal deposition. One and the same device produces a broad electron beam for heating the substrate in vacuum, a metal vapor to be deposited and a broad beam of fast molecules for pre-cleaning the substrate surface and for modification of the film during its deposition.

## References

- [1] A. Metel, S. Grigoriev, *US Patent No 6,285,025*, 2001.
- [2] S. Grigoriev, Yu. Melnik, A. Metel, *Surface and Coating Technology* 156, 44 (2002).
- [3] S.N. Grigoriev, A.N. Isaikov, Yu.A. Melnik, A.S. Metel, in *Proc. 7<sup>th</sup> Int. Conf. on Modification of Materials with Particle Beams and Plasma Flows. Tomsk, 26–29 July, 2004*. Tomsk, 2004, p. 29–33.
- [4] N.V. Pleshivtsev, *The cathode sputtering*, Moscow, Atomizdat, 1968, 347 pp. (in Russian)