

## DEPOSITION OF NANOFILMS IN HIGH DENSITY PLASMA DEVICES

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### ABSTRACT

The results of experiments on deposition of metal nanostructured films in a microwave Electrone cyclotronre resonance (ECR) discharge and magnetron discharge with hollow cathode are presented. High plasma density in these devices ( $10^{17} \text{ m}^{-3}$ ) produced in large volumes ( $5 \cdot 10^{-3} \text{ m}^3$ ) increases the possibility of ionization of sputtered metal atoms on the way from the target to the substrate. The deposition of the films in these devices is stimulated large flows of ions, which allows to obtain nanofilms with unique properties. In addition, the flow of metal ions can be operated by an electric field near the substrate, which allows the film to deposit on submicron relief structures with a high ratio of depth (height) to width.

**Keywords:** Microwave ECR discharge, Hollow cathode magnetron, Nanofilms, Relief structures.

### INTRODUCTION

Among the various methods for producing nanostructured films we consider film deposition by sputtering in the low-pressure plasma (so called the physical deposition (PVD) method). In this method the bombardment of the cathode target with positive ions of inert or/and reactive gas is knocking metal atoms (Figure 1), which are deposited on the substrate. In electronic industry is most widely used magnetron discharge in crossed electric and magnetic fields. Plasma is created between the anode and cathode, at voltage of 400–800 V is applied.

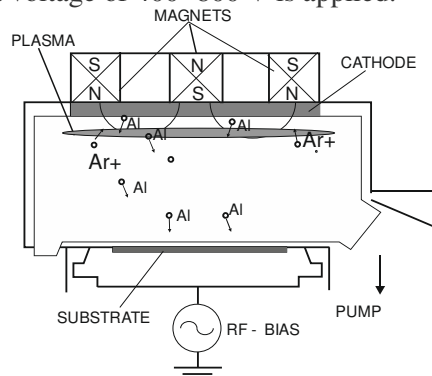


Figure 1. Diagram of magnetron discharge

Electrons are trapped by the magnetic field and perform complicated cycloid motion along the trajectories near the target surface. Region of the plasma with a high density (more than  $10^{17} \text{ m}^{-3}$ ) in the form of torus thickness of a about 1 cm, and outside it the density decreases to  $10^{15} - 10^{16} \text{ m}^{-3}$ . From the 80-s arises the high-density plasma sources ( terminology refers to the field of plasma processing). These sources create plasma with a density of more than  $10^{17} \text{ m}^{-3}$  in large volume (several liters). Fabrication of the films in these devices is stimulated by large flow of ions, what allows to synthesize nanostructured films with unique properties. These sources include inductively coupled, microwave ECR, helicon discharges and some others. Range of operating pressure is from a one tenths to ten of Pa. The low pressure, low temperature plasma is thermally nonequilibrium plasma – electron temperatures ( $T_e = 2 - 5 \text{ eV}$ ) are usually at least one order of magnitude higher than those of ions and neutrals ( $T_i, T_n = 0.1 - 0.3 \text{ eV}$ ).

Particular difficulties arise in the deposition of films on submicron relief structures. As an example, metallization of vias and trenches in the creation of modern integrated circuits with feature size 90–65 nm. In a conventional magnetron sputtered metal flux is almost 100% of the neutral atoms and they can not manage. Isotropic distribution of particles leads to the fact that condensing atoms

close the top of nanostructure and inside is formed the cavity (Figure 2a). To carry out the metallization of narrow and deep trenches and vias, it is required a flux of particles directed normally to the surface of the structure. One possible way of solving this problem is the ionization of sputtered atoms on the path from the target to the substrate (ionized PVD). Then by applying an electric field to the substrate can control the energy and motion of charged particles in the plasma sheath (Figure 2b).

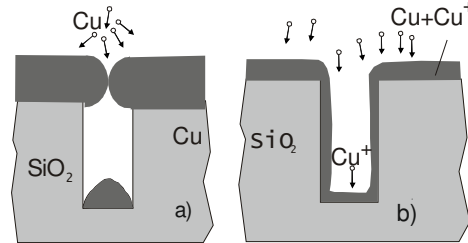


Figure 2. Metallization of the trench by sputtered atoms (a) and ions (b).

## EXPERIMENTAL APPARATUS

In the laboratory of the Department of Physics of MSFU were produced two high-density plasma sources based on a microwave electron-cyclotron resonance (ECR) discharge and magnetron hollow cathode discharge. In the microwave ECR source plasma is produced by absorption of microwave in a magnetic field in condition of ECR, when the Larmor rotation frequency of the electrons is equal to frequency of incident wave (2.45GHz). From the source (Figure 3) (diameter 0.15 m, length 0.25 m) plasma flows into processing chamber (diameter 0.35 m, length 0.55 m), where mounts a substrate [1].

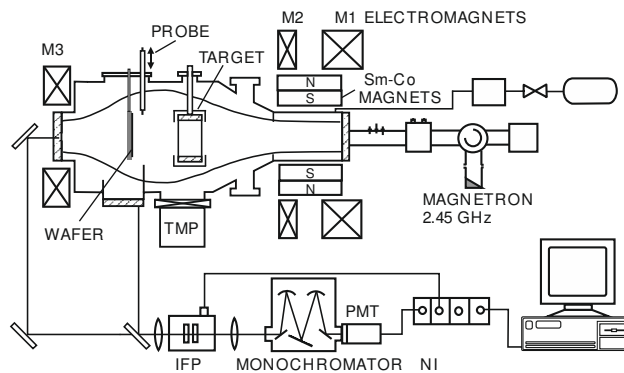
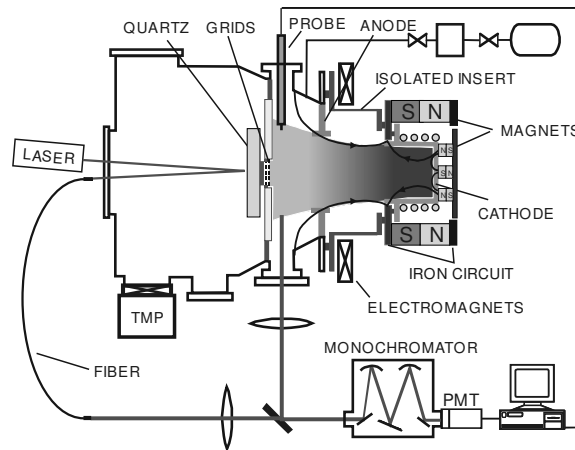


Figure 3. Schema of the ECR setup

This setup has several advantages. First, several cylindrical targets of various metals isolated from each other can install ahead the substrate. Only those targets will be sputtered, which voltage is applied. Second, it is possible to produce a preliminary plasma treatment of the substrate prior to sputtering targets. Third, the film can be obtained by the method of plasma chemical vapor deposition (eg, hydrogen, acetylene or with the addition of methane to carbon nanotubes, etc.). It can also produce the films etching in chemically active gases (discharges in fluorine and chlorine-containing gases). A feature of this discharge is that the cathode-target is operation in an ion-saturation current mode (order 50–100 A/m<sup>2</sup>). Detailed study of various plasma characteristics in this discharge is presented in articles [3-5].

Next, we consider in detail the characteristics of the plasma source of a new type – the hollow cathode magnetron (HCM). The current density in this source achieves more than of 1000

A/m<sup>2</sup> [2, 6]. The experimental setup of the magnetron discharge with a hollow cathode department of physics is shown in Figure 4 [6].



**Figure 4.** Hollow cathode magnetron

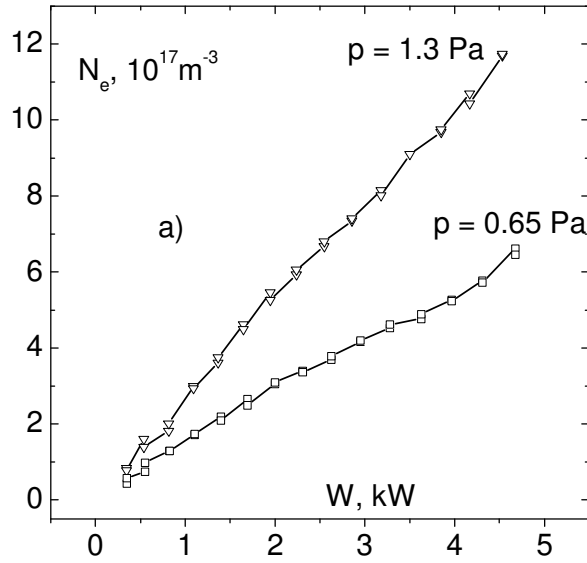
The cathode consists of a cup-shaped Cu target (diameter 0.14 m, length 0.1 m) made of copper and cooled with water. Cylindrical targets from different metals can be inserted into the cathode. The HCM is powered with inverter source up to 12 kW. The chamber was pumped to base pressure  $5 \cdot 10^{-6}$  Torr using a turbomolecular pump. Argon is used as the buffer gas. Pressure was within range of 0.065–1.3 Pa. Gas flow is provided by a gas flow controller. The magnetic field is produced by twelve columns of Nd-Fe-B magnets  $18 \times 20 \times 120 \text{ mm}^3$  surrounded the target with ring iron flanges on the end. The downstream of the HCM is located the electromagnet. The ends of the columns are connected by circular iron magnetic circuits. The magnetic field near the cylindrical surface of the cathode 500 Gauss. Magnetic field is directed along a sidewall surface of the magnetron and has a cusp at the mouth of the cathode. Magnetic field captures secondary electrons emitted from the cathode, which produce an ionization of the buffer gas and sputtering atoms of the target. Crossed ExB fields cause electron drift in an azimuthal direction, in result inside the hollow cathode plasma of high density (about  $10^{19} \text{ m}^{-3}$ ). The target utilization in such cathode is high, the zone of erosion occupies almost all cylindrical part. Those electrons and ions, which have an initial axial velocity, can leave the hollow cathode and propagate towards the substrate. For expansion of plasma stream and creation of more homogeneous radial distribution at a distance of several centimeters from a mouth was set the electromagnet, magnetic field of which was directed opposite to the magnetic field of the constant magnets. From the source plasma enters into the processing chamber in which is installed a water-cooled substrates.

The quality of the films depends on the magnitude and energy of the impinging flux of particles (ions, neutrals, and electrons) and substrate temperature. To determine these plasma parameters probe, optical emission spectroscopy (OES), optical absorption spectroscopy (OAS) and mass-spectrometry diagnostics were used by means of the data control and acquisition system based on National Instruments hardware and software LabVIEW.

## RESULTS AND DISCUSSION

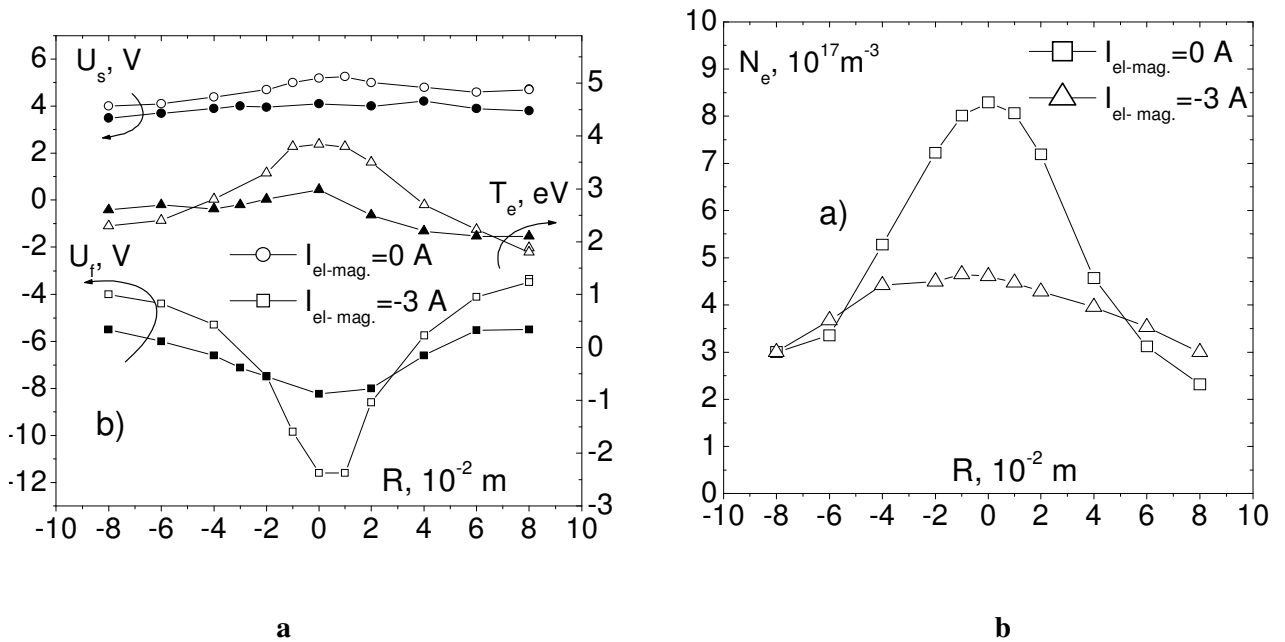
Effect of the magnetron power on plasma parameters on the discharge axis is shown in Figure 5.

The plasma density grows almost linearly with a power, the main contribution to the plasma density is argon ions, and the density of copper ions is 10–25%.



**Figure 5.** Electron density as function of magnetron power.  $I_{el} = -3$  A. 20 cm from the target.

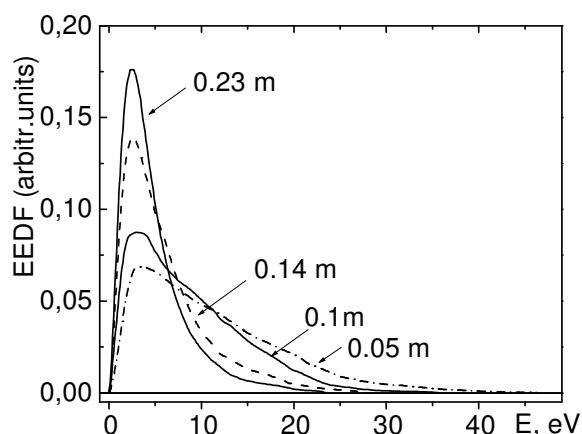
Figure 6a,b shows the radial plasma characteristics as function of a magnetic field of an electromagnet.



**Figure 6.** Radial profiles of: **a)** the electron density  $N_e$ ; **b)** electron temperature  $T_e$ , floating  $U_f$  and plasma  $U_s$  potentials. 20 cm from the target.  $p=5$  mTorr, 50 sccm,  $W=3$  kW.

The increase of a magnetic field of the electromagnet results in growth of uniformity of a stream and to a decrease of plasma density. At discharge power of 3 kW and pressure of 5 mTorr plasma density on an axis decreases from  $8.2 \cdot 10^{11} \text{ cm}^{-3}$ , when electromagnet is turned off, to  $4.5 \cdot 10^{11} \text{ cm}^{-3}$  when electromagnet current is equal to 3 A. Let's note also, that energy of ions on the isolated substrate, equal  $e(V_s - V_f)$  does not exceed 20 eV. By applying a bias voltage (accelerating voltage) on the substrate can cause resputtering of the films, what required for the deposition on the textured surfaces of complex shape. This acceleration of the ions is also necessary to obtain superhard coatings. Increase in the concentration and the number of high-energy electrons with increasing

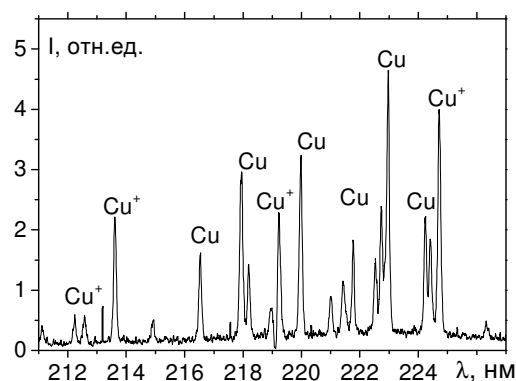
discharge power causes the effective ionization of sputtered copper atoms. Figure 7 shows the functions of the electron energy distribution at different distances from the source.



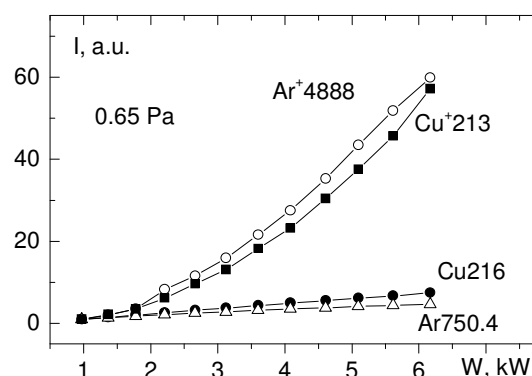
**Figure 7.** Electron energy distribution at different distances from the source

It is seen that near the source of a high proportion of electrons with energies above 10–16 eV, which can ionize the atoms of argon (15.7 eV) and metal (7.5 eV). With increasing distance the proportion of these electrons is reduced, however, even at a distance of 20 cm ionization takes place.

A typical emission spectrum of wavelength between 210 and 228 nm from Cu/Ar plasma is shown in Figure 8. Figure 9 shows the normalized intensities of Ar and Cu spectral lines as function of a magnetron power (intensity at  $W = 0.9$  kW is accepted for 1).



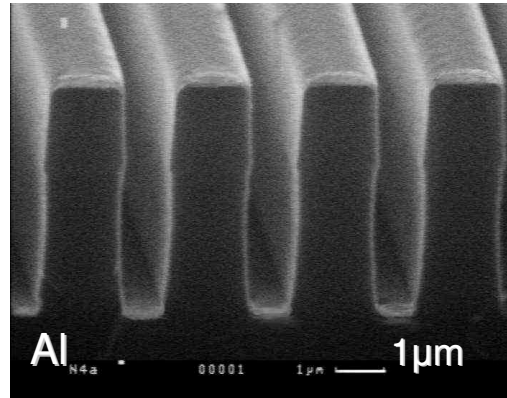
**Figure 8.** An optical emission spectrum from Ar/Cu plasma at wavelength between 210 and 228 nm.  $p=0.65$  Pa,  $I_{el}=-3$  A,  $W=3.36$  kW.



**Figure 9.** Normalized intensities of Ar and Cu spectral lines as function of a magnetron power,  $I_{el}=-3$  A

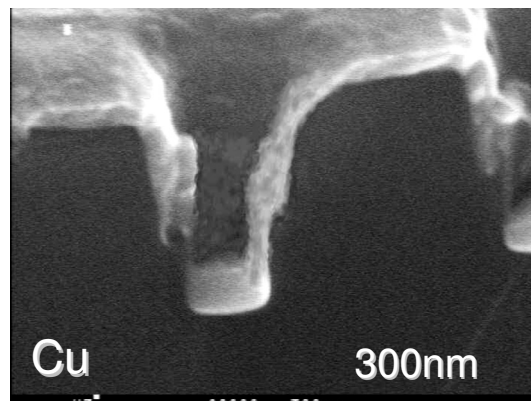
These data do not depend on the spectral sensitivity of PMT. It is seen that intensities of radiation of argon and copper atoms increase nearly linear with power. The intensities of argon and copper ions are proportional to about square of the magnetron power. As shown measurements, the EEDF of electrons depends weakly on a power. Therefore dependence of intensity emission is determined mainly by the electron density.

Figure 10 presents the electron microscope photographs of trenches and vias, etched in silicon dioxide and filled with metal.



**Figure 10.** Metallization of the trench with aspect ratio 5:1. Film thickness: top = 270 nm, bottom = 220 nm, side = 100 nm. Ar,  $p=0.08$  Pa, ECR discharge,  $W_{\mu} = 970$  W.

The substrate was under floating potential, energy of the ions on a substrate was in a range 15-25 eV. Cross sections show an anisotropic character of a trench and vias filling, which defined by a stream of metal ions. There is no overhang on top of the structures. Figure 10 presents trenches with width of 1 μm and depth of 5 μm, filled with aluminum. From the ratio of film thickness at the top and bottom we can estimate the metal ion flux fraction – ratio an ion flux to total flux of ions and atoms. We assume that deposition at the bottom of the deep trench is mostly due directed ions and at the top is by atoms and ions. From figure 10 the metal ion flux ratio is equal to 80% for Al. Figure 11 shows metallization of the vias with stepped configuration known in IC processing as dual damascene.



**Figure 11.** Metallization of the vias. Dual damascene. HCM discharge.  $P = 1$  Pa.  $W = 3$  kW

Metal ion flux fraction measured by means of the gridded energy analyzer in front of a substrate, biasing the grid to either repel as admit ions. The ionized flux fraction was attained 80 % for aluminum and titanium and 50 % for copper at power of 3 kW. Deposition rate reached 100 nm/min for Al and 150 nm/min for Cu at substrate located 0.2 m from the target.

## CONCLUSION

The description of two sources of high-density plasma - a microwave ECR discharge and magnetron discharge with hollow cathode is presented. These sources are used for effective ionization of atoms sputtered from the target. The advanced diagnostic system allows to determinate optimal plasma parameters, conduct real-time monitoring of process for producing nanostructured films with desired properties.

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