Sputter Deposition of Nano and Submicron Layers with In Situ Thickness and Structure Control

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Abstract – Two-beam mass-monochromator allowing depositing by reactive sputtering thin layers with in situ control of layers composition and thickness is briefly described. The main ion channel based on the "Large MEPhI mass-monochromator" with ion energy range 1–40 keV produces ion beams with (EM/Z) ≤ 4000 keV×a.m.u. Additional ion channel with special decelerating ion system produces ions with (EM/Z) ≤ 60 keV×a.m.u in the energy range 0.05–10 keV. Both beams are directed to the axis of goniometer with rotatable two-target assembly and electrostatic analyzer of scattered ions and atoms. Computer controlled system of ion beams handling allows to use every beam for reactive redeposition and target surface analysis. Different diagnostics used for treated target analysis including control of deposited layer thickness and roughness are described.

1. Introduction

Thin and extra thin layers and capping on surface often define utility of products and pieces in engineering and electronics. As well, redeposition of materials in plasma, gas discharge and fusion devices strongly influence conditions of plasma-surface interaction changing erosion rate of plasma facing components, gas recycling and retention in materials, etc.

There is a lot of different vacuum, ion, laser and plasma technologies that allow generating layers and coverage with desired properties. But as a rule, every sort of such technology was a result of numerous experiments. So, generation of necessary deposit needs specific technology.

To understand physics of thin layer generation for different compositions of components and substrates as well as some important properties of modified surface, it is necessary to have well-determined and variable conditions of process. This means that well known Gunterschulz-Moor conditions are satisfied. To ensure the fulfillment of these conditions, using of mass, energy and angle separated beams of partials is preferable. We describe the experimental complex with well defied parameters of two ion beams, oil free vacuum system and in situ control of treated surface composition, layer thickness and roughness relevant to abovementioned conditions and designated for investigation of all stages of film deposition and removal under ion beam sputtering.

2. Ion Beam Channels

The main ion channel based on the "Large MEPhI mass-monochromator" [1] with 4-stage differential vacuum system turbo molecular and ion pumps. Electromagnet with central ion trajectory equal to 50 cm and inhomogeneous 90° sector magnetic field up to 0.6 T gives stigmatic focusing of ion beams with (EM/Z) ≤ 4000 keV×a.m.u. Duoplasmatron with total ion current up to 1mA with high voltage power supply system produces ions in the energy range 1–40 keV. Angle divergence of the beam ~ 1°, for the input diaphragm of 2 mm diameter energy spread in the beam is less than 0.003. Additional ion channel with special decelerating ion system produces ions with (EM/Z) ≤ 60 keV×a.m.u in the energy range 0.05–10 keV. This channel (see Fig.1) has inside liner at high negative voltage to provide higher ion current at low final energy of ions impinging target at ground potential.

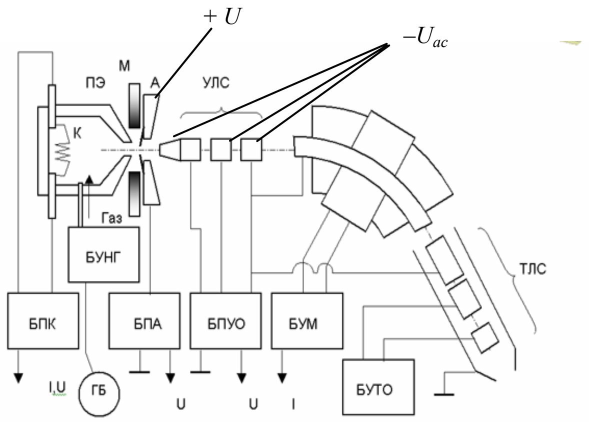

Fig. 1. Scheme and power supply of the low energy ion channel: УЛС – accelerating and focusing system, БПК – cathode supply, БУНГ – gas feed system, БПА – anode supply, БПУО – supply of accelerating optics, БУМ – electromagnet supply, БУТО – retarding system control, ТЛС – retarding lens system

Air-cooled duoplasmatron ion source with permanent magnet is used in this channel. Negative voltage bias U_{ac} relative to ground applied to extracting electrode and liner inside electromagnet. Both U that determines the final energy of ions and U_{ac} can be varied with computer controlled power supply blocks PSM10 up to 10 kV with accuracy ~ 1V. Electromagnet with central ion trajectory radius equal to 12 cm and magnetic field dropping factor –1/2 has high aperture and focuses ions in both horizontal and vertical directions. This channel is equipped with 70 l/s turbomolecular pump.

Both ion channels are equipped with beam deflection systems to scan by beam over target. Special controllers are developed for programming of beam position on a target. The axis of both beams are directed onto the center of working chamber with angle between them equal to 45°. The main chamber is equipped with 1000 l/s turbomolecular pump, gas feeding system, different analytical tools and two-target assembly.

3. Two Target Assembly

Two targets of 25 mm in diameter are mounted in the chamber in such a way that one of the targets is positioned on the center of goniometer, while another is situated 4 cm away (see Fig. 2). Temperatures of Both targets are controlled with tungsten ohmic heaters in the range 300–900 K. Another one during experiment can replace the target in the center of the chamber.

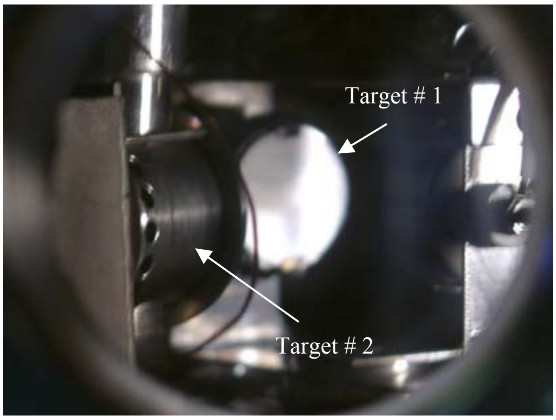

Fig. 2. Positional relationship of two targets in the chamber

4. Analyzing System

The main analytical tool of the complex is the computer controlled electrostatic energy analyzer of ions and neutrals [1]. Stripping sell and two CEM (ВЭУ-6) for ions and neutrals are used for energy analysis of particles in the energy range 0.1–40 keV providing surface composition analysis with low ad middle energy spectroscopy of scattered ions recoils (LEIS+MEIS). This analyzer can be turned around axis of goniometer for scattering angles in the range $0 < \theta < 135°$ allowing registration of energy spectra of scattered particles for both ion channels. This energy analyzer can be also used for thin layer thickness measurements using small angle scattering of protons or deuterons providing for light atoms on layer of heavy atoms substrate depth resolution less then 0.1 nm [2]. Using different primary ions and angles of registration it is possible to realize very high sensitivity for some combinations of deposited atoms and material of substrate. For example, $\sim 10^{-4}$ of monolayer of tungsten atoms on carbon substrate can be detected.

A special laser system (Fig. 3) is developed for measurements of target surface reflectivity. This simple device allows to monitor surface roughness and thin films thickness on target surface. Secondary ions mass spectrometry (SIMS) for measurements of ionized sputtered positive ions is realized using small sector electromagnet equipped with SEM (ВЭУ-1) as a detector. This method is very sensitive for some elements and can be considered as complementary for LEIS and MEIS analysis.

The variation of the scattering angle with accuracy ~ 1° in combination with the possibility of analyzing target rotation (relative one or two axes) gives it possible to deduce the reciprocal position of different atoms in compounds and alloys with LEIS or MEIS. Gas feed into the chamber is available through computer control gas flow system. General scheme of the chamber with ion beams and analyzing tools is shown in Fig. 3.

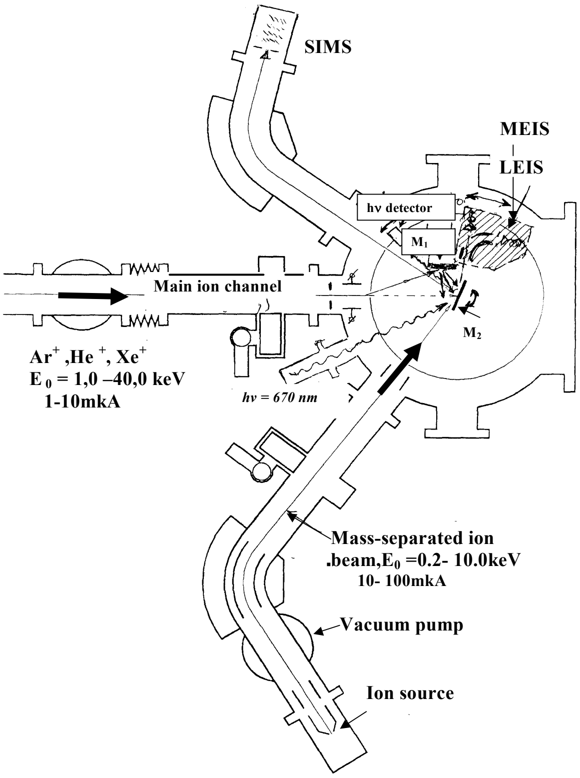

Fig. 3. Scheme of the analytical chamber with two ion beams and different diagnostics tools, M_1 and M_2 are targets for sputtering and deposition

5. Procedure

The computer simulation of experiment precedes measurements with ion irradiation. The Monte Carlo code SCATTER [3] based on the binary collision approximation and taking into account the real surface microrelief of used targets (as it measured with scanning tunnel microscope) and positions of both targets relative to ion beam directions allows to calculate coefficients and differential distributions of scattered and sputtered particles. So, sputtering and deposition of sputtered atoms can be evaluated including secondary processes of redeposition and reflection.

For the reactive deposition of chosen material onto substrate the main ion channel with the 5–40 keV heavy noble gas ions is mainly destined. Deposition rate up to 1 monolayer per second (Ar 30 keV $\rightarrow$ Cu) can be achieved. Scanning of primary ion beam provides homogeneity of deposition. As well, simplified version of the second ion channel (without mass-separator unit) providing Ar^{+}10 keV ion beam 100 μA/cm^2.

Analysis of the surface composition by LEIS is available with the "low energy" ion channel. The main ion channel is very convenient for MEIS. In the letter case ion beam can be directed to the sputtering target as well to substrate target for analysis. Computer controlled ion beam deflection system makes it possible to vary irradiation time and time for analysis. Fig. 4 shows as an example the energy spectra of ions.

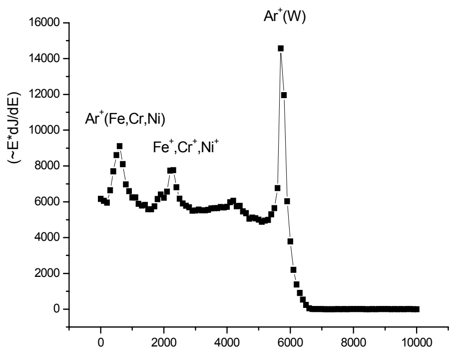

Fig. 4. Energy spectra of ions scattered and knocked out from the stainless steel target after deposition by reactive sputtering of tungsten atoms detected by energy analyzer during sputtering of stainless steel target (substrate target) after reactive deposition on it (at temperature ~600 °C) of several monolayers of tungsten

Measurement of the thickness of deposited layer can be realized with ion beam profiling. But for noticeable difference of atomic numbers of substrate and deposited atoms, using of hydrogen ions with energies up to 40 keV allows to evaluate deposited layer thickness analyzing energy spectra of protons scattered from the be-layer target. Depth resolution and maximum layer thickness depends on the energy, stopping power of protons, scattering angle and the different layer atomic mass ratio. This method of layer thickness measurements looks like RBS but applicable for smaller thickness. For example, D^{+} ion with primary energy 40 keV allows detecting carbon layer on tungsten substrate with thickness up to 0.3 μ. Very thin (of order of 1 nm) layer of heavy atoms created before deposition of layers with less averaged atomic umber can be also used as a mark allowing to control sputtering or growth of the surface layer with the help of fast hydrogen ions. As well, ion mixing resulted in the spread of thin layer of heavy atoms under ion bombardment can be detected during such sort of non-destructive analysis.

Various configurations of the deposition-analytical system are also available as well as it's upgrading due to using of standard flanges ∅50 mm and ∅100 mm. For example, the additional build-in small-scale plasma-beam generator [4] can be used for high flux treatment of targets.

6. Conclusion

The described analytical complex demonstrates different possibilities that can be used for investigation of different stages of deposition and sputtering of nano and submicron layers.

References

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