

Measuring the ionized flux fraction from high-power impulse magnetron discharges using a miniaturized passive ion energy spectrometer

M. Rudolph^{1,2}, T. Degoussé¹, T. Minea¹, V. Tiron³, C. Costin³,
L. Sirghi³, M.-C. Hugon¹, B. Bouchet-Fabre²

¹ *LPGP, Université Paris-Sud, UMR 8578, 91405 Orsay, France*

² *NIMBE, CEA Saclay, 91191 Gif-sur-Yvette, France*

³ *IPARC, Alexandru Ioan Cuza University, Faculty of Physics, Iasi, 700506, Romania*

High-power impulse magnetron sputtering is known to deliver a high flux of ions onto the growing film which affects it considerably in terms of crystalline structure and morphology. The characterization of this ionic flux has been the subject of some studies in the past. But while many of those studies investigated the ion energy distribution function, it is only half the picture, the other part being the ionized fraction of the metallic flux towards the growing film. This parameter is rarely investigated as no standard equipment is commercially available. We propose a modified Bainbridge spectrometer to separately collect ions from neutrals on monocrystalline Si surfaces. The ions are in addition dispersed as a function of their energy so that the instrument measures simultaneously the ion energy distribution function and thus provides a comprehensive image of the flux of atoms and ions onto the growing film. We present a description of the instrument focusing on its performance and limits. The discussion is followed by first results from direct current magnetron sputtering (DCMS) and high-power impulse magnetron sputtering (HiPIMS) of a tantalum target in an argon atmosphere.

1. Introduction

The particle flux from magnetron discharges onto a growing film contains neutral and charged species and although former are usually by far more abundant, the film morphology and texture largely depends on the ions [1]. This is because they gain significant energy in the sheath region in front of the substrate that forms either due to self-biasing in the case of an electrically floating substrate or due to an intentionally applied bias. The total energy flux provided by atoms and ions onto the substrate is thus largely dependent on the ionized flux fraction and its energy distribution.

Measurements of the ion energy distribution function (IEDF) from various discharge modes has been done in the past using energy-dispersive mass spectrometers. The classical direct magnetron (DC) mode is characterized by a low energy distribution peaking at a few eV. By pulsing the discharge at higher target potentials, larger instantaneous currents can be reached keeping the average power delivered to the target constant. This mode, called high-power impulse magnetron sputtering (HiPIMS), exhibits a second peak situated around 10 to 20 eV with a shoulder exceeding up to several 10s of eV [2]. This energetic peak is one of the reasons for the high interest in the HiPIMS thin film deposition.

The ionized flux fraction, although of equal importance for the total energy flux onto the substrate, is less quantitatively determined [3]. A typical number of 0.5 for the degree of ionization is

abundantly stated in literature [Pal13]. However, this value is misleading because firstly it varies quite significantly depending on the discharge conditions. Bohlmark et al. [5] defines a parameter denoted ionic contribution which is the recorded emission intensity from singly charged ions divided by the sum of the emission from singly charged ions and neutrals. This parameter, which approximates the degree of ionization grows steadily from 40% up to over 96% in the investigated range of different pulse energies between 1 and 10J for a HiPIMS discharge using a Ti target.

Secondly, the degree of ionization does not necessarily correspond to the ionized flux fraction onto the substrate, which is a more relevant parameter for film growth. Latter describes the actual ion-to-neutral ratio incident on the growing film. The thermal ion and neutral fluxes, Γ_i and Γ_n can be described as follows (n_n and n_i are the neutral and ion density, v_{th} is the thermal velocity and u_B is the Bohm velocity T_g and T_e is the gas and electron temperature) [Gud10]:

$$\Gamma_i = 0.61 n_m u_B \sim \sqrt{T_e} \quad (1)$$

$$\Gamma_n = 0.25 n_i v_{th} \sim \sqrt{T_g} \quad (2)$$

For a cold plasma discharge, in general, $T_e \gg T_g$, so that the ratio of ion-to-neutral fluxes does not

correspond any more to the degree of ionization in the bulk plasma [6].

Estimates for the ionized flux fraction onto a substrate exist from measuring the difference in deposition rate between a negatively and a strongly positively (140V) biased substrate. In this way, Kouznetsov et al. [7] estimate the ionized fraction of deposited Cu flux to lie around 70%. In a different study, Macak et al. [8] observe two charge pulses onto a flat Langmuir probe positioned above the target and allocate them to the arrival of an Ar⁺ and Ti⁺ plume. From the total charge of the Ti⁺ plume and the total deposition rate constituted by the Ti atomic and ionic flux, they deduce the ionized fraction of the deposited flux to lie around 40% ± 20%. Although these indications may be valuable to pointing out the difference between the HiPIMS and the DC mode, they are insufficient to record small differences in the degree of ionization arising from changing parameters in the HiPIMS discharge.

In this study we report on the development and first test of a modified Bainbridge mass spectrometer that collects ions and neutrals separately from each other and at the same time disperses ions according to their energy. It is devised to function passively and to occupy a small volume only so that ordinary deposition reactors can accommodate it at their virtual substrate position.

2. Ion energy spectrometer

2.1 Experimental setup

The spectrometer setup with respect to the target is shown in Fig. 1. A variable entrance slit is positioned at a height $h = 10\text{cm}$ that corresponds to the virtual substrate position. Its radial position r with respect to the target centre can be shifted in or out to sample different regions of the magnetron. For this study the position r is kept fixed so that the entrance slit is positioned right above the racetrack.

2.2 General principle

The spectrometer consists of two neodymium (NdFeB) magnets kept at a mm distance and a homogeneous magnetic flux density of close to 1T in between (Fig. 2). The plasma species enter this magnetized region through a slit of 500 μm . Neutral atoms propagate straight on and settle on a Si wafer placed opposite the entrance slit as they are undisturbed by the magnetic field (Fig. 3). Charged ions on the other hand are forced on a circular trajectory with a radius that depends on both their mass and kinetic energy according to eq. (3) where r_g is the gyroradius, m the mass and q the charge of the

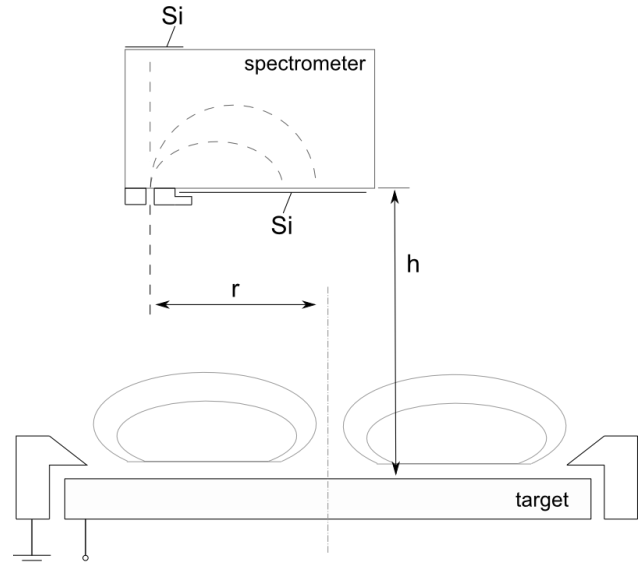


Fig. 1: Ion energy spectrometer mounted above the magnetron. The height h is fixed and corresponds to the virtual substrate position, the radius r can be adjusted to measure several points on the target.

ion, v_{\perp} is its velocity perpendicular to the magnetic field lines and B the magnetic flux density.

$$r_g = \frac{mv_{\perp}}{|q|B} \quad (3)$$

A clean polished Si wafer is placed to intercept their trajectory after an angle of 180° and collect the ions,

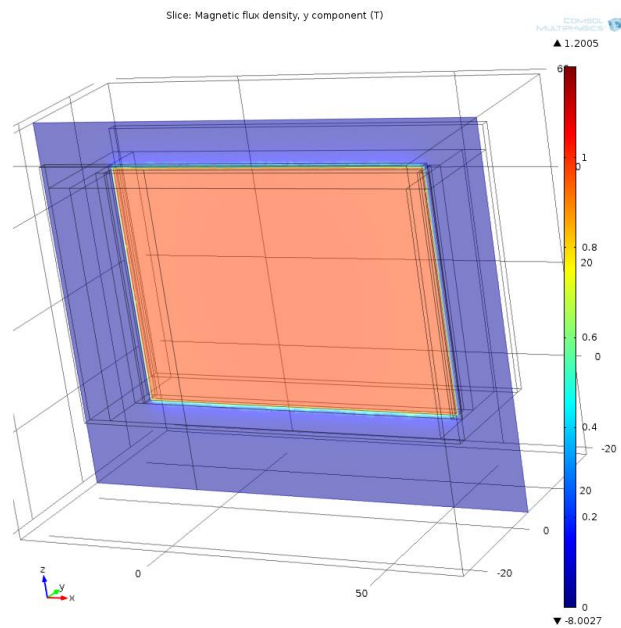


Fig. 2: Simulation of the magnetic flux density in y direction (into the plane) in between two neodymium magnets spaced at 1mm.

where the distance x between the point of intercept and the entrance slit corresponds to twice the radius r_g and is to first order a function of the mass and energy T of the particle. Measurements of x thus reveal the initial energy of the ion according to eq. (4).

$$T = \frac{q^2 x^2 B^2}{8m} \quad (4)$$

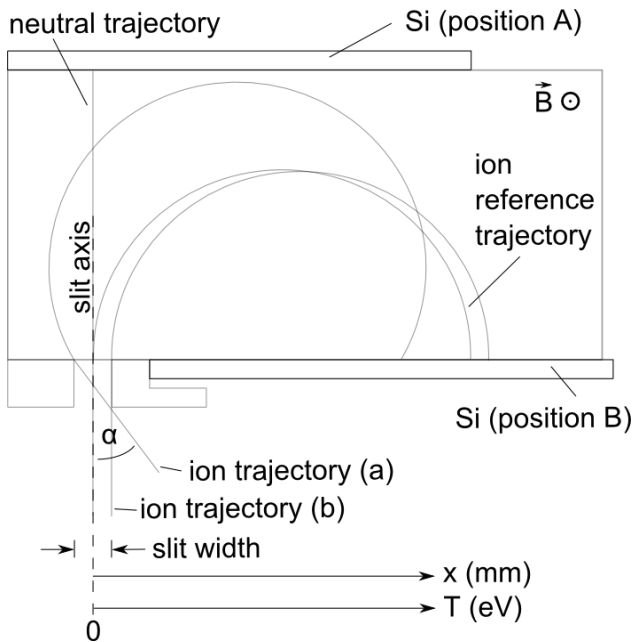


Fig. 3: Detailed sketch of the ion energy spectrometer. The two positions for the Si plates are to collect neutrals (position A) and ions (position B) separately. Trajectory (a) and (b) indicate the trajectories that result in the largest positive and negative departure from the position x on the Si plate with respect to the reference trajectory.

2.3 Uncertainties

Uncertainties associated to the energy measured by exploiting eq. (4) arise in particular from the design of the entrance slit. Consider a reference ion with kinetic energy T , mass m and charge q that is following a reference trajectory passing through the slit on its axis. This ion will be deposited at a position x on the Si plate. The same reference ion however may be deposited at a position different from x by either arriving at an angle or perpendicular but vertically displaced with respect to the reference trajectory or a combination of both. The trajectory of ions arriving at an angle still describe circles but the Si plate intersects those circles not through their centre anymore, so that those ions are deposited at smaller distances compared to x . The lowest possible distance for a reference ion is thus achieved by one

arriving at the maximum acceptance angle α with a trajectory almost touching the upper left corner of the entrance slit, denoted trajectory (a) in Fig. 3. On the other hand, the largest possible value of x for an ion of energy T is achieved by an ion propagating on a perpendicular trajectory almost touching the right corner of the slit, denoted by trajectory (b) in Fig. 3. A summary of the relative uncertainties as a function of the ion energy is given in Fig. 4. It can be seen that the uncertainty remains below 10% for ion energies $> 5\text{eV}$. Below this energy threshold, the uncertainty drastically increases. In principal, it is possible to decrease the uncertainty by using a different slit configuration with a smaller width and a smaller acceptance angles. Both measures, however would decrease the total incoming flux received per unit time, thus decreasing the total flux received by the spectrometer.

It should be noted that the effect of any local electric fields at the entrance slit is neglected. Typically the plasma bulk is at potential of a few volts positive so that ions would be subject to an acceleration towards the grounded entrance slit of the spectrometer which would deposit the ions at a larger distance x than that corresponding to their energy without acceleration. This potential drop can principally be taken into account by measuring the plasma potential.

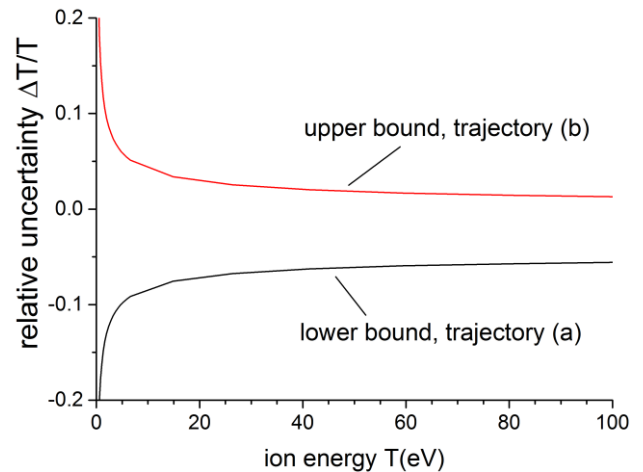


Fig. 4: Relative uncertainties in energy arising from the finite slit width ($500\mu\text{m}$) and acceptance angle ($\pm 12^\circ$).

2.4 Magnetron

A balanced magnetron is used to test the spectrometer. It is contained in a vacuum chamber equipped with a throttle valve between the reactor and a turbomolecular pump to regulate the working pressure. The target is connected to a high-voltage pulsing unit with preionization capability [9] which can reach currents up to 100 A. Fig. 5 shows a typical

HiPIMS voltage and current pulse recorded at 0.5Pa reactor pressure using 100% Ar as working gas and a 99.95% pure Ta target.

2.5 Readout of Si surfaces

The deposited Ta atoms on the Si surfaces form a profile from which the amount of deposited material can deduced separately for ions and atoms. The readout will be performed using a mechanical profiling. Current profilometers have a vertical resolution of 6nm which is sufficient to read-out the Si plates with high precision. Alternatively, Rutherford backscattering spectrometry (RBS) can be used. Sampling the profile with a beam of α particles at 1800 keV and measuring the spectra of the back-scattered α allows to deduce the areal density of Ta atoms over the beam size.

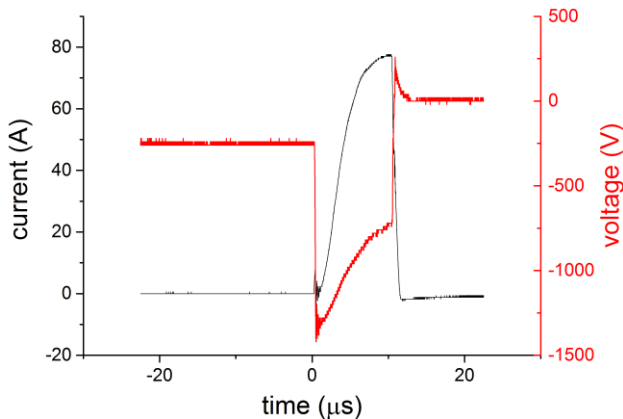


Fig. 5: Typical voltage and current pulse for sputter deposition in HiPIMS mode.

3. First results

First test cases have been run using a sputtering reactor presented above in non-reactive DC and HiPIMS mode. For both cases the entrance slit was positioned above the center of the circular target. Results will be presented on the International Conference on Phenomena in Ionized Gases ICPIG 2015 in Iași, Romania.

4. Acknowledgements

The authors are grateful for the funding of this activity by the French National Research Agency (ANR) under the contract ANR-13-IS09-0003-01. Furthermore, they thank in particular Joël Baudet for technical support with manufacturing the spectrometer.

5. References

[1] I. Petrov et al., *J. Vac. Sci. Technol.* A12 (5), pp. 2846 – 2854, 1994.

[2] M. Aiempnakit et al., *Surf. Coat. Technol.* 205, pp. 4828 – 4831, 2011.

[3] J.T. Gudmundsson et al., *J. Vac. Technol.* A30(3), 038001, 2012.

[4] M. Palmucci et al., *J. Phys. D : Appl. Phys.* 46, 215201, 2013.

[5] J. Bohlmark et al., *J. Vac. Sci. Technol. A* 23, 18, 2005.

[6] J.T. Gudmundsson et al., *Vac.* 84, pp. 1360 – 1364, 2010.

[7] Kouznetsov et al., *Surf. Coat. Techn.* 122, pp. 290 – 293, 1999.

[8] K. Macak et al., *J. Vac. Sci. Techn. A* 18(4), pp. 1533 – 1537, 2000.

[9] M. Ganciu-Petcu et al., US Patent 7927466 B2, 2011.