Experimental design study by optical emission of a magnetron sputtering discharge.

M.F. Dony, A. Ricard*,J.P. Dauchot, M. Wautelet, M. Hecq,

Laboratoire de Chimie Inorganique et Analytique, Université de Mons-Hainaut, 23, avenue Maistriau, B-7000 Mons, Belgium,

*Laboratoire de Physique des Gaz et Plasmas, Université Paris-Sud, CNRS, Orsay, France.

Abstract.

A magnetron sputtering discharge is studied by optical emission and absorption spectroscopy, following an experimental design procedure. The 1.5 inch diameter target is made in an aluminum base alloy. The light is focused on an optical fiber and analyzed in a Jobin-Yvon computer-controlled 1.0 m Czerny-Turner scanning monochromator. The spatial resolution is 2 mm. Emission line intensities from the Ar sputtering gas and the sputtered species are studied as a function of geometrical and physical discharges parameters: excitation mode (RF or DC), target thickness, gas discharge pressure, electric power (RF) or current (DC), and the distance from the target. By use of hollow-cathode lamps, absorption due to aluminum atoms and argon metastable and resonant atom states densities are evaluated, as a function of the same parameters as in emission. The experimental responses (emission and absorption intensities) are fitted by a quadratic equation in which interactions between factors are included. The influences of the main factors are deduced and discussed.

1. Introduction.

Magnetron sputtering is widely used in thin film deposition, but only a few studies have been devoted to the diagnostics of magnetized plasmas. The emission spectroscopy allows the characterization of magnetron discharges for the studies of conducting and non conducting materials and the diagnostics of thin film deposition. DC magnetron discharges have been studied by S.Miyake et al.[1]. Three Ar I, Ar II and Al I lines have been investigated at 2 mTorr and 40 mA as a function of the

distance from the target surface. Mehdi et al.[2] have studied an RF planar magnetron discharge in Ar by optical emission spectroscopy. They have measured the emission lines from Ar, Al and other sputtered particles as a function of the RF power (50-200 W) and the discharge gas pressure (7-150 mTorr). Dony et al.[3] have considered the plasmas of DC and RF magnetron discharges. This discharge is analyzed by emission spectroscopy for Ar and metal atom radiative states and by optical absorption for the Ar metastable and resonant atoms. Their study was realized in the 6-200 mTorr pressure range for W=1-50 Watts in RF and I=2-50 mA in DC. and the spectral line intensities were studied as a function of the distance from the target surface at 25 mTorr in RF 50 W and DC 70 mA.

Several parameters (the target thickness (t), gas discharge pressure (p), electric power (W) or current (I) and the distance from the target (d)) can influence the optical emission of the glow discharge. The experimental design procedure [4] allows to envisage all parameters while reducing the number of experimental measurements. This technique supplies empirical relations between the emission intensity or absorption coefficient and the parameters. Here, the density of aluminium atoms and of metastable and resonant Ar atoms has been studied by optical absorption in DC and RF magnetron plasmas. The corresponding spectral line intensities have been measured for two cathode thickness (1.7 and 5 mm).

2. Experimental details.

The experimental system (Fig.1) has been described in a previous paper [3]. The magnetron cathode is an aluminium-based alloy (93.7% Al, 5.4% Cu, 0.44% Pb, 0.25% Fe) of diameter 33 mm and thickness 1.7 mm or 5 mm. The plasma is analyzed by emission spectroscopy via a quartz optical fiber (diameter 1 mm). The optical absorption is performed with hollow cathode lamps : -a S/N 63288 Ba-Ar cathodeon lamp for the Ar metastable and resonant atoms and a 124-E Al-Ne Perkin-Elmer lamp for the Al atoms. Two optical lenses of diameter 50 mm and focal 75 mm (L_1 and L_2 in Fig. 1) focus a nearly parallel beam into the plasma chamber. The spectral line intensities in emission and absorption are recorded by means of a Jobin-Yvon HR 1000 monochromator (focal length 1 m, grating with 2400 grooves mm^{-1}) working in the second order from 180 to 320 nm (resolution limit 5.10^{-2} Å) and in the first order from 320 to 800 nm. Two photomultipliers are used: a Hamamatsu 955 MA in the UV (180-320 nm) and Hamamatsu 166 UH in the visible (320-800 nm). Intensity data acquisition is achieved by means of the Jobin-Yvon software. Initially, the optical fiber, the cathode surface and hollow cathode lamp are aligned with a laser beam.

3.Experimental procedure.

In emission spectroscopy, two Ar I lines are studied Ar I 425.9 nm $3p^5 5p$ $(3p_1)$ - $3p^5 4s$ $(1s_2)$ and Ar I 451.1nm $3p^5 5p$ $(3p_5)$ - $3p^5 4s$($1s_2$) with respectively a sharp and a broad excitation cross section by electrons and also an Ar II line 442.6 nm $3p^4 4p(^4D_0)$ - $3p^4 4s(^4P)$. The first excited level configuration of Ar $(3p^5 4s)$ at

about 11.5eV contains two metastable ($1s_5$, $1s_3$) and two resonant ($1s_4$, $1s_2$) levels that are also studied in absorption spectroscopy. The density of these metastable and resonant levels has been determined by optical absorption of the $3p^5$ (4p-4s) spectral lines coming from an argon lamp. It should be noted that two lines with weak (696.5 nm) and strong (763.5 nm) oscillator strengths *f* have been chosen for the determination of the Ar ($1s_5$) metastable density. The Al excited and ground states have also been analyzed by emission and absorption spectroscopy of the Al I resonants $3p^2P^0$-$4s^2S$ (1/2-1/2) and Al I $3p^2P^0$-$4s^2S$ (3/2-1/2) transitions.

In the experimental design procedure, an experimental domain (Table 1) is defined for each factor (p, W (RF) or I (DC), d) . The original values of the factors are not directly used. They are proportionally transformed in coded values whose the origin of the coded factor is the center of the experimental domain (Table 1). The coded values of the factors are dimensionless and therefore can be compared.

Table 1. Correspondence between coded values and original values

Factors	Coded values	Original values	Units
Log p	-1.68	1	p (mTorr)
	1.68	2.3	
Log W	-1.68	1	W (Watt)
	1.68	1.78	
Log I	-1.68	1	I (mA)
	1.68	2.04	
d	-1.68	1	d (mm)
	1.68	15	

The program [6] fits the measured data following a quadratic polynomial:
$R = a_0 + a_1 \log p + a_2 d + a_3 X + a_4 (\log p)^2 + a_5 d^2 + a_6 X^2 + a_7 \log p.d + a_8 \log p.X + a_9 d.X$
Where R is the log of intensity in emission or the absorption coefficient A_1 ; X is the log I in DC or the log W in RF; a_i are the adjusted coefficients by the program and a_0 is the response in the "center" of the experimental domain in coded values (logp = d = X = 0). We have conducted four series of measurements (two cathode thickness for two excitation modes). Each series comprises 20 runs in order to have a central composite design with 6 replicate measurements [4].

4.Results and discussion.

In the first time, the principal components analysis (PCA) [5] is applied in order to obtain the groups of lines showing the same characteristics (in emission spectroscopy). For each line, a vector of n dimensions (n representing the number of adjusted factors) is attributed. The PCA is a data reduction technique: the n

dimensions are reduced in 2 dimensions while still preserving as much information as possible. With this procedure, it would appear that various groups behave quasi-identically: (1) Al I 394.401 nm and Al I 396.151 nm; (2) Ar I 425.936 nm and Ar I 451.074 nm although these lines have a sharp and a broad excitation cross section respectively; (3) Ar I 696.54 nm, Ar I 763.51 nm, Ar I 794.82 nm and Ar I 738.4 nm; (4) Ar I 750.387 nm and (5) Ar II 442.601 nm.

4.1 Emission

We have first studied the effect of the excitation mode and the cathode thickness on the logarithmic emission intensity in the center of the experimental domain (a_0). Table 2 shows that the lines intensity of Ar and Al are tenth higher in RF than in DC while the Ar II is not much influenced. For a given excitation mode, the intensity of Ar I lines is more intense for the thinner cathode while the inverse is found for the Ar II line.

Table 2. Logarithmic intensity in the center of the experimental domain (a_0).

Log(Line intensity)	1.7 d.c	5 d.c	1.7 r.f	5 r.f
Al I 396.2 nm	1.39	1.46	2.57	2.47
Ar I 425.9 nm	1.67	1.21	2.26	1.93
Ar I 696.5 nm	1.4	0.99	2.38	2
Ar I 750.4 nm	1.85	1.36	2.58	2.21
Ar II 442.6 nm	1.62	1.93	1.89	1.95

The lines intensities of Ar and Al decrease with d. The Al logarithmic lines intensities decrease linearly twice more in DC than in RF whatever the cathode thickness. In the case of the Ar lines, the decrease is nearly linear and independent on the excitation mode or cathode thickness. The interaction of d with logp and of d with power or current are weak, meaning that the decrease does not depend on pressure or power.

The Al lines increase with increasing p. The effect is nonlinear and the quadratic term is significant. This quadratic coefficient is negative and reduce the increase at high pressure or goes to a plateau. The pressure effect on the Ar lines is less obvious to analyze and is out of scope of this paper.

The logarithm of the lines intensity rises linearly with the logarithm of the power (RF) or the current (DC). The electric power coefficient is about twice higher for the Al lines than for Ar lines. The effect of the cathode thickness is not significant in DC while in RF, the line intensity increase with power is larger for the thinner cathode. The interactions are negligible.

4.2 Absorption

The absorption coefficient A_l of a spectral line along the absorption length is calculated following the equation :

$$A_l = 1 - \frac{I_t}{I_0}$$

where I_t is the transmitted intensity and I_0 is the incident intensity. A_l is correlated to the absorbing atom density [3].

In the center of the experimental domain (a_0) and for a given cathode thickness, the absorption coefficient A_l is higher in RF than in DC, this trend is the most important for the Ar I 738.5 and Ar I 750.4 nm (Table 3). For a given excitation mode and for Al lines, A_l increases with the cathode thickness while for the Ar lines the effect is opposed and more important (Table 3).

Table 3. Absorption coefficient (A_l) in the center of the experimental domain.

A_l	1.7 d.c	5 d.c	1.7 r.f	5 r.f
Al I 396.2 nm	0.56	0.65	0.69	0.77
Ar I 696.5 nm	0.41	0.14	0.42	0.29
Ar I 738.5 nm	0.21	0.1	0.51	0.35
Ar I 750.4 nm	0.08	0.04	0.3	0.19

The Al line absorption coefficient does not depend on the distance of observation (relative to the cathode) while, for Ar, A_l is decreasing.

A_l is increasing with the current (DC) and the power (RF) for Al and in the DC mode for Ar. In the RF mode, A_l (Ar) is nearly constant with the power.

For Al and Ar, the absorption coefficient rises with p in the low pressure range and reaches a plateau at high pressure.

By comparison with our previous results [3], the increase of Ar and Al radiative species when changing DC by RF excitation mode is confirmed. New results are here reported on the absorption coefficient on the ground states Al atoms. It is shown that the ground state Al atoms measured by A_l also increased as passing from DC to RF.

Conclusions.

An experimental design procedure, using a quadratic polynomial, has given reliable results for the Ar and Al excited states and for the Al ground states as the parameters of the magnetron discharge are varied from 10 to 200 mT, 10 to 60 W in RF and 10 to 100 mA in DC. The Ar and Al radiative species intensity, the Ar metastable and resonant atoms density and the Al absorption coefficient are found to increase when going from DC to RF mode.

Works are in progress to determine the Al ground state density from the presently measured A_l absorption coefficient.

References

[1]S. Miyake, N. Shimura, T. Makabe and A. Itoh J. Vac. Sci. Technol A 10(4), Jul/Aug 1992

[2]T. Mehdi, P.B. Legrand, J.P. Dauchot, M. Wautelet and M. Hecq, Spectrochim. Acta B, 48 (1993) 1023.

[3]M.F. Dony, A. Ricard, J.P. Dauchot, M. Hecq, M. Wautelet, to be published in Surface and Coatings Technology

[4] G.E.P. Box, W.G. Hunter and J.S. Hunter, Statistics for Experimenters, Wiley, New-York 1978.

[5]G.Kateman, L.Buydens, Quality Control in Analytical Chemistry, Wiley-Interscience Publication, New-York, 1993.

[6]Design-Expert 4.0, Stat-Ease, Mineapolis.

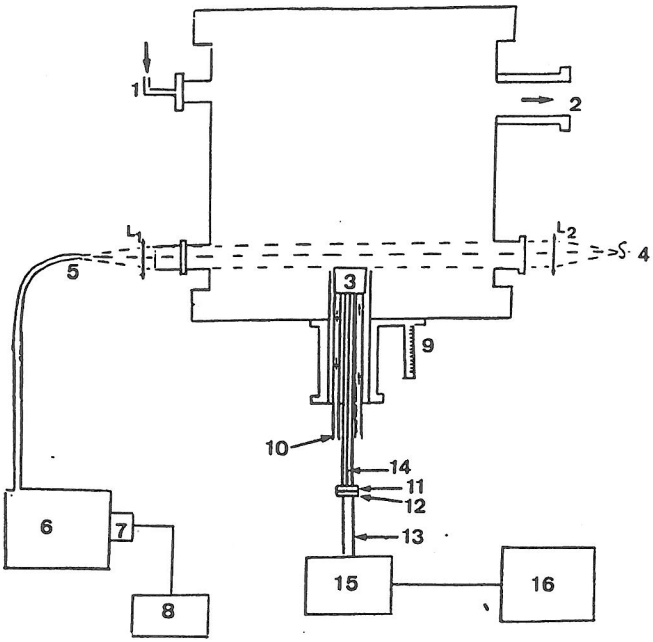

Fig.1. Experimental setup of the magnetron discharge with the optical arrangement for the emission and resonant absorption spectroscopy:
1. gas inlet; 2.turbomolecular pump; 3. cathode magnetron; 4. hollow cathode lamp; 5. optical fiber; 6. HR 1000; 7. photomultipliers; 8. PC control; 9.vernier; 10. cooling tubing; 11.female coaxial connector; 12. male coaxial connector (RF input: type "N"); 13. RG-393 coaxial cable; 14.copper conductor; 15. matching network; 16. 13.56 MHz RF generator.