

LASER OPTOGALVANIC ANALYSIS OF THIN FILM THICKNESS IN HOLLOW CATHODE DISCHARGE

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Abstract. In this experimental investigation comparative evaluation is made of the hollow cathode glow discharge possibilities for layer and surface analysis, in particular, for thin film thickness measurement by three approaches based on classical direct layer-by-layer spectral analysis, conventional optogalvanic effect and photoelectric optogalvanic effect.

1. Introduction

The new material development requires improvement of the existing methods for layer and surface analysis, and even elaboration of new ones. Due to its main advantages as high sensitivity and accuracy, simple apparatus and easy interpretation of the analytical signal, the classical direct layer-by-layer spectral analysis in a glow discharge, especially in Grimm's discharge, is an established method [1]. Due to its better stability and reproducibility the hollow cathode glow discharge has been successfully used recently for such analysis [1, 2].

The purpose of this investigation is to extend the possibilities of the classical layer-by-layer analysis in a hollow cathode glow discharge for thin film thickness measurement, and by this analysis to integrate with optogalvanic and photoelectric optogalvanic spectroscopy. The results obtained by us up to now are promising in this direction [3]. The possibilities of these three approaches, namely, using as analytical signal the spectral line intensity I , the optogalvanic signal (OGS) and the photoelectric optogalvanic signal (POGS), are illustrated by investigating Si_3N_4 thin film, deposited on Si substrate. This material is very interesting due to its large using as dielectric or masking layer in MOS-IC and sensor technologies.

2. Description of the Applied Approaches

We describe briefly the approaches used in this work.

The hollow cathode glow discharge is an efficient spectral source for direct layer-by-layer analysis since the entering and excitation of the investigated atoms takes simultaneously place in it and, thus, finally this causes emission of relevant spectral lines. The intensity of these spectral lines represents the analytical signal of this method.

The optogalvanic spectroscopy is based on the optogalvanic effect which is a change of the discharge impedance caused by absorbed resonant light [4]. The optogalvanic signal is purely electrical signal that is a reason for better sensitivity of the analyses. Therefore, the proposed by us replacement of the classical spectral line intensities by the OGS improves the layer-by-layer analysis in hollow cathode plasma.

The photoelectric optogalvanic signal can be obtained when suitable laser light emits photoelectrons from the illuminated surface. These photoelectrons, accelerated by the electric field in the discharge, turn into high-energy electrons able to produce additional plasma ionization, i. e. to change plasma conductance. Based on different work functions for different materials and due to the extremely high sensitivity of the photoeffect to the surfaces, POGS turns out to be a very selective method concerning layer-by-layer analysis. The use of the POGS for layer and surface analysis in high frequency discharge has recently given good results [5, 6]. It is for the first time that we investigate the possibilities of this method for such analysis in hollow cathode discharge.

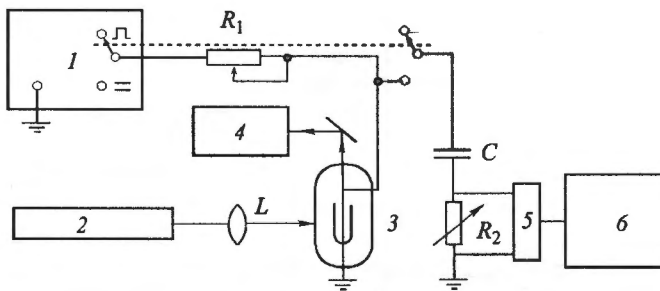


Fig. 1. Experimental setup: a hollow cathode tube (1); a d.c. or pulse supply (2); a nitrogen laser (3); a spectrograph (4); a preamplifier (5); a lock-in-nanovoltmeter (6)

3. Experimental Setup

The experimental setup used is composed by the following main components (Fig. 1): a hollow cathode discharge tube, supplied by a d.c. or pulse generator, a nitrogen laser, measuring RC-group and an analytical signal detection apparatus as spectrograph and a lock-in-nanovoltmeter with a preamplifier or an oscilloscope. The OGS and POGS are detected as voltage drop change over $R = 1 \text{ M}\Omega$ resistor. The capacitor C separates the measured from the high voltage. Two configurations of the applied hollow cathode tube

are developed: direct illumination of the cathode bottom, where the sample is placed, causes POGS while illuminating axially the hollow cathode through suitable channels, I and pure OGS are detected. The mean-square error was 0.10 for photographically registered intensity of the spectral lines and 0.01 for the measured OGS and POGS.

The inert gas He at 2.5 Torr pressure is chosen as working gas because of its "transparency" to $\lambda = 337.1$ nm spectral line emitted from nitrogen laser. The mean discharge current value is about 10 mA. The diameter of the hollow cathode, made of Al, is 6 mm and its length — 40 mm.

The Si_3N_4 thin film has been deposited by low pressure chemical vapor deposition on n -(4–9 Ωcm) type Si(100) substrate in industrial type reactor. These thin film characteristics, connected with its applications in the semiconductor industry, have been investigated in [7, 8].

4. Results and Discussion

The dependencies of the analytical signals (I , OGS and POGS) on the layer sputtering time t are shown in Figs 2–4. The correct place finding of the interface between layer and substrate by these dependencies is one of the most important tasks for such analyses. The interface position is indicated in the figures by an arrow. The weakly expressed minimum around the 45th minute of the $I = f(t)$ function for two investigated nitrogen spectral lines can be attributed to the interface position between Si_3N_4 thin film and Si substrate (Fig. 2). It is also clear from Fig. 2 that both layer and substrate contain

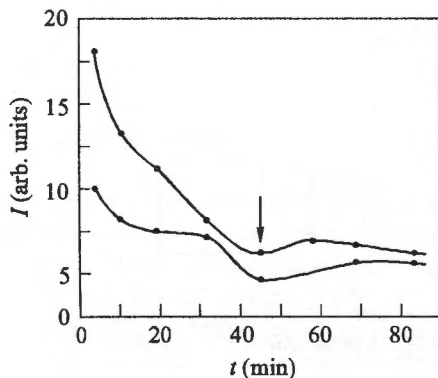


Fig. 2. $I = f(t)$ dependence for NI4109.98A (1) and NI4358.27A (2) spectral lines

nitrogen. Obviously, this nitrogen atom diffusion from the surface into the substrate is caused by the high temperature (about 800°C) which is necessary for the layer deposition. The same fact can be also seen from the $\text{OGS} = f(t)$ function (Fig. 3), registered simultaneously with $I = f(t)$. The measured OGS arises as a result of 337.1 nm spectral line absorbance by N_2 molecules, i. e. this signal also gives an information about the nitrogen depth profile. Because of the low OGS amplitude at the region around the interface in this particular case investigated by us, the OGS sensitivity is insufficient to "feel" the interface position so as the present in Fig. 3 dependence is not suitable for interface position determination. However, this position can be defined

very precisely from $POGS = f(t)$ function (Fig. 4) due to better sensitivity of the photoelectric part of this signal with respect to the changed material characteristics in the interface region. In contrast to the POGS used by authors in [5, 6], our POGS is a complex one. In our experiment, it consists of two parts: a pure OGS, caused by $\lambda = 337.1$ nm laser light absorbed by N_2 molecules in the hollow cathode plasma and another signal, caused by the accelerated photoelectrons. Taking into account the high work function for photoelectron emission from Si_3N_4 and the insufficient photon energy (3.7 eV), corresponding to $\lambda = 337.1$ nm spectral line, the second part of this complex POGS has quite little amplitude under our experimental conditions even at two photon photoeffects, taking place in our experiments especially at laser flux intensity $> 10^2$ kW/cm².

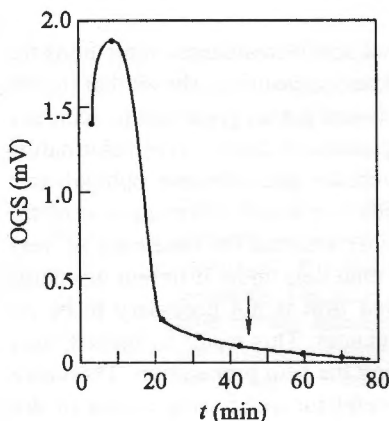


Fig. 3. Dependence of $OGS = f(t)$

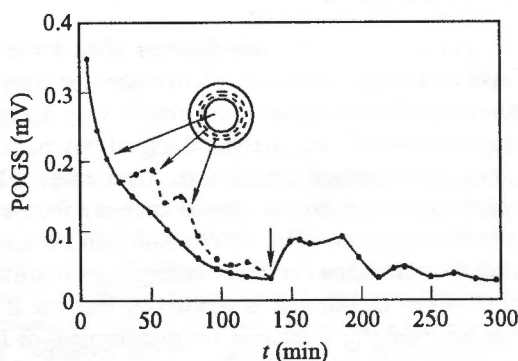


Fig. 4. Dependence of $POGS = f(t)$ for sample with diameter equals to cathode diameter (dashed lines) and for sample diameter/cathode diameter $< 2/3$

Si_3N_4 thin film thickness of $0.21 \mu\text{m}$, measured by a laser ellipsometer with 2 \AA accuracy, gives the possibility for cathode sputtering velocity estimation as $0.005 \mu\text{m}/\text{min}$ and $0.0016 \mu\text{m}/\text{min}$, obtained by our experimental dependences shown in Fig. 2 and Fig. 4, respectively. This difference between the estimated velocities (connected with the different time position of the interface) is due to smaller discharge current density in the last case. According to the correlation between the velocity of the sputtered layer and the presence of its atoms in the hollow cathode plasma, we can take $(0.02-0.05) \mu\text{m}$ layer step in depth, corresponding to a unit exposition of 10 min. There are not any principle limitations for cathode sputtering velocity varying in the hollow cathode plasma since this discharge works with high stability in a wide range of gas pressure and discharge current values.

In principle, the dependences in Figs 2-4, drawn for some layers with known thickness, allow layer thickness/time calibration curve to be obtained. Obviously, the better accuracy of POGS registration in comparison with spectral line intensity is good prerequisite for sensitivity enhancement of the carried out analyses of layer thickness.

Fig. 4 shows an interesting peculiarity of $POGS = f(t)$ function when the sample diameter is equal to a cathode diameter. During the first minutes, POGS amplitude

decreases what corresponds to sputtering from the central region of the investigated sample. Later on, but before the final sputtering of Si_3N_4 thin film on the central region, consecutive concentric regions begin to sputter in direction to the cathode walls. This process is expressed by the consecutive maximums and minimums of POGS, shown in Fig. 4. The registered peculiarity is caused by the radial distribution of the ion current in a hollow cathode discharge. This distribution is well-known to be homogeneous only at the central region of the cathode bottom. Therefore, the experimental result obtained above is very important because its neglecting may cause wrong POGS interpretation in some cases. In order to prevent our experiments from such confusion, the maximal sample diameter was chosen to be $2/3$ from the cathode diameter.

5. Conclusion

The comparison of the possibilities of the three applied in this investigation methods for layer and surface analysis, particularly for layer thickness measuring, shows that the replacement of the spectral line intensity by the optogalvanic signal gives rise to accuracy improvement of the analytical signal but such a replacement cannot give information about the interface position in some cases. However, the photoelectric optogalvanic signal combines this improved analysis accuracy with very exact determination of the interface position. The latter result can be successfully utilized for obtaining of very useful information about sputtering rates of different materials under different discharge conditions. Besides, it is obviously that the deposited film is not necessary to be removed from the discharge for measurement of its thickness. This result is, indeed, very important for some technological improvements during the film preparation. Therefore, the proposed by us approach turns out to be very useful for monitoring *in-situ* of different thin films in a hollow cathode discharge when it is used as a source for thin film deposition.

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