



## Spectroscopic diagnostic complex based on the magnetron sputtering device with a digital method of the data mining

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**Abstract:** A diagnostic complex based on a magnetron sputtering device is proposed for studying a magnetron discharge plasma parameter by optical emission spectroscopy, using two spectroscopic systems: photographic and photoelectric. Software for digital processing of the obtained emission spectra is developed. The results obtained by the two spectroscopic systems are compared.

**Keywords:** Data mining, Magnetron sputtering, Plasma, Spectroscopy.

### 1.0 Introduction

The great interest in studying of the magnetron discharge (MD) physics is due to its widely used for the formation of different kinds of film coatings [1] used in a variety of industry branches e.g. in microelectronic, cutting tools or reflecting layers manufacture [2]. That it requires to make systematic experimental studies of the spatial and temporal plasma parameters, in particular, the determination of the chemical composition of plasma particles, and their distribution to the excitation and ionization states, as well as the influence of the magnetron operating mode on these parameters.

Often, the magnetron discharges in the magnetron sputtering devices are specified by a small region of localization above the cathode surface, a great value and high inhomogeneity of the magnetic field, both in the vertical and radial directions [3]. Thus, the direct use of the most common plasma diagnostic method of Langmuir probe [4] is difficult due to the possible perturbation of the plasma [5]. Therefore, to study the MD plasma parameters, it is better to use those processes and phenomena that occur in the plasma formation itself, but can be detected at its periphery, for example, by analyzing the electromagnetic radiation of plasma particles in an

excited state. Optical emission spectrometry (OES) is one of the most widely used non-invasive plasma diagnostics methods [6, 7], as requires a simple and inexpensive experimental setup and permits to measure many plasma parameters in the most full range

From the intensity of the corresponding spectral lines the population of excited states of plasma particles and so electron temperature and electron density can be determine. The spatial distribution of the intensity of spectral lines along certain directions gives information about the distribution of particles in the studied excited states along these directions. The interpretation of the spectral lines gives information above the chemical composition of the plasma.

The application of the OES method supposed the use of one of two radiation detection systems - photographic (F) and photoelectric (FE). Each of them has both a number of advantages and certain disadvantages. The most complete information on the studied plasma parameters can be obtained with the simultaneous use of both these systems.

A complex that is presented in the work makes possible to study MD plasma by the OES method using both optical radiation detection systems (F and FE). Besides the description of the software for analyzing the obtained data is given.

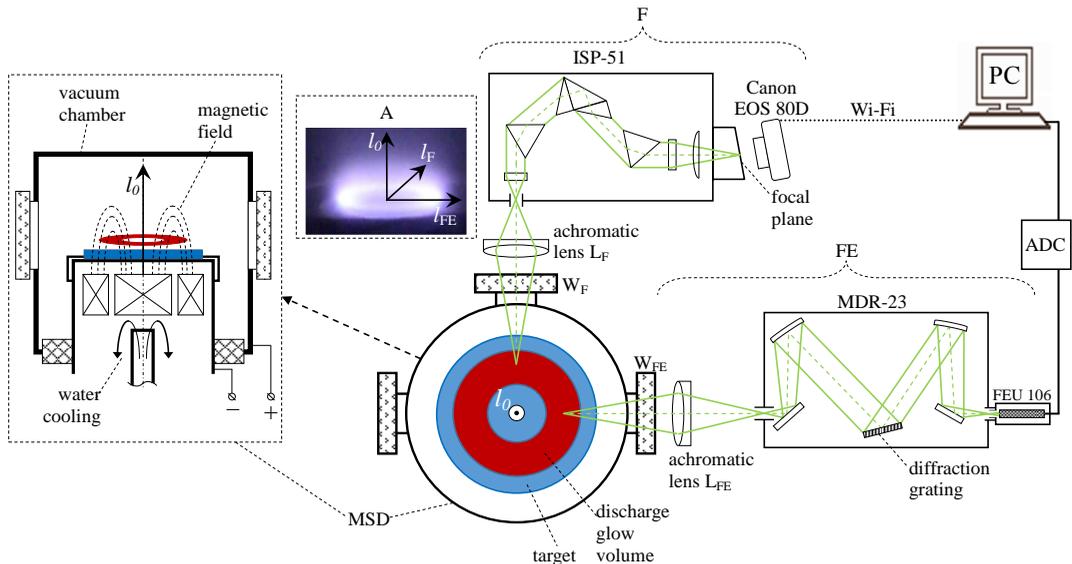
## 2.0. Schematic diagram of the diagnostic complex

The diagram of the complex for the diagnosis of MD plasma by the OES method is shown in Fig. 1. It includes a magnetron sputtering device (MSD), photographic (F) and photoelectric (FE) plasma radiation detection systems. A description of MSD is given in [8]. In Fig. 1 in two projections a schematic arrangement of the magnetized part of the MD relative to the diagnostic windows for outputting the optical radiation is shown. The MD region can conditionally be divided into three main parts [9]: the cathode dark space, the magnetized zone (the region of bright emission  $\sim 1$  cm length) and then the space up to the MSD camera that used as the anode. The optical radiation from the glow region of the discharge is output through the diagnostic windows  $W_F$  и  $W_{FE}$  of the magnetron chamber along the  $l_F$  and  $l_{FE}$  directions and is focused by achromatic lenses onto the entrance slit of one of the radiation detection systems.

Insertion A in Fig. 1 shows the glow of MD and the radiation detection systems axes relative to the MD axis  $l$ . The studied direction  $l$  along the MD glow is parallel to the axis  $l$  and is measured from the glow edge close-by to the cathode.

The diagnostic complex has two channels of radiation output for using two optical radiation detection systems (F and FE). In the first case (F), the optical spectrum of the MD plasma is registered in a wide range of wavelengths, and the intensity of the spectral line along its height ( $h$ ) represents the distribution of excited particles along the studied direction  $l$ . This is very relevant for a MD, since a magnetic field significantly affects the dependence of the plasma composition and its parameters on the axial coordinate. The signal recorded in this case is averaged over the exposure time. In the second case (FE), the integral intensity along the radiation collection height is determined for the studied wavelength, but it is possible to study

the changes in time of the recorded signal. It should be noted that for both registration systems, it is possible to study an arbitrary glow region along the direction  $l$  and  $l_{FE}$ . Thus, the use of two optical radiation detection systems allows obtaining information on the spatial-time characteristics of MD plasma.



**Figure 1. Schematic diagram of the diagnostic complex.**

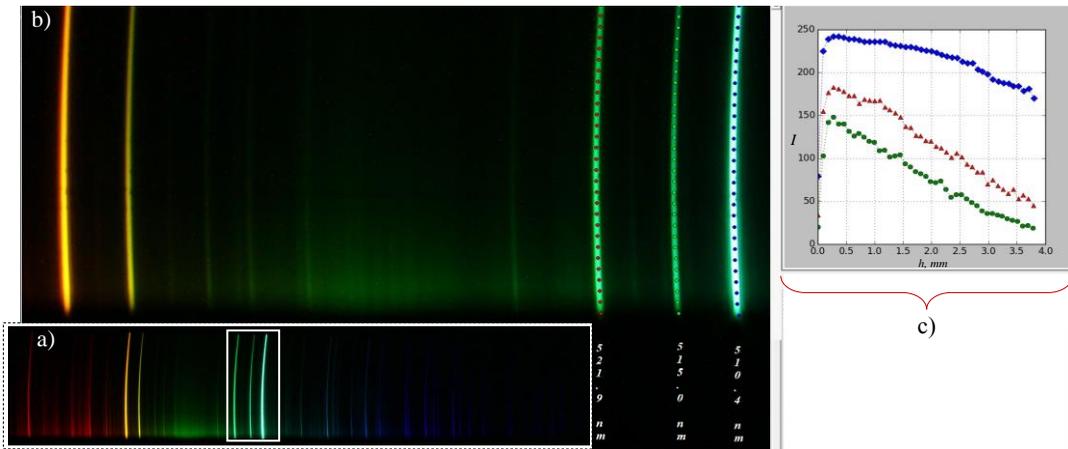
## 2.1. Photographic radiation detection system

Diagnostics of MD radiation by the OES method with a photographic system of registration (Fig. 1, F) was carried out using an ISP-51 spectrometer. The radiation of the bright glow region of the discharge is output through the  $W_F$  diagnostic window and with the achromatic lens  $L_F$  is projected onto the entrance slit (0.1 mm) of the ISP-51, where it is dispersed into the spectrum by 3 glass prisms. The total spectrum in the wavelength range of 390-700 nm of the investigated radiation is projected onto the focal plane of the output collimator with  $F = 120$  mm. At this, the intensity of the spectral line along its height ( $h$ ) represents the distribution of excited particles along the studied direction  $l$ . The spectrum image is captured using a Canon EOS 80D digital camera with a matrix size of 7000\*5000 pixels, having a maximum of relative spectral sensitivity at  $\lambda = 500.0$  nm. The resulting image of the radiation spectrum of the excited MD plasma particles is stored in electronic form in the \*.jpg format, which in the future allows you to create a database of optical spectra of various chemical elements under different experimental conditions.

For determining the qualitative and quantitative characteristics of MD plasma, a multifunctional interactive GUI application OSA (Optical Spectrum Analyzed) was used [11, 12]. The OSA application was created in the Python programming language, taking into account the Tkinter graphics library, and uses a set of additional modules: PIL, SciPY, NumPy, and

Matplotlib. At the same time, mathematical algorithms and procedures have been developed, which allow you to transform the selected digital image into a numerical matrix, process this matrix and visualize the results. In parallel, the results are written to an external file.

The digital image of a part of the emission spectrum of particles excited in MD produced in argon with a copper cathode is shown in Figure 2a. The operation mode of the magnetron sputtering system was: buffer gas pressure  $P_{Ar} = 10$  Pa, anode voltage  $V_a = 320$  V, discharge current  $I_d = 25$  mA, magnetic field induction  $B = 0.05$  T, exposure time  $t = 90$  s.



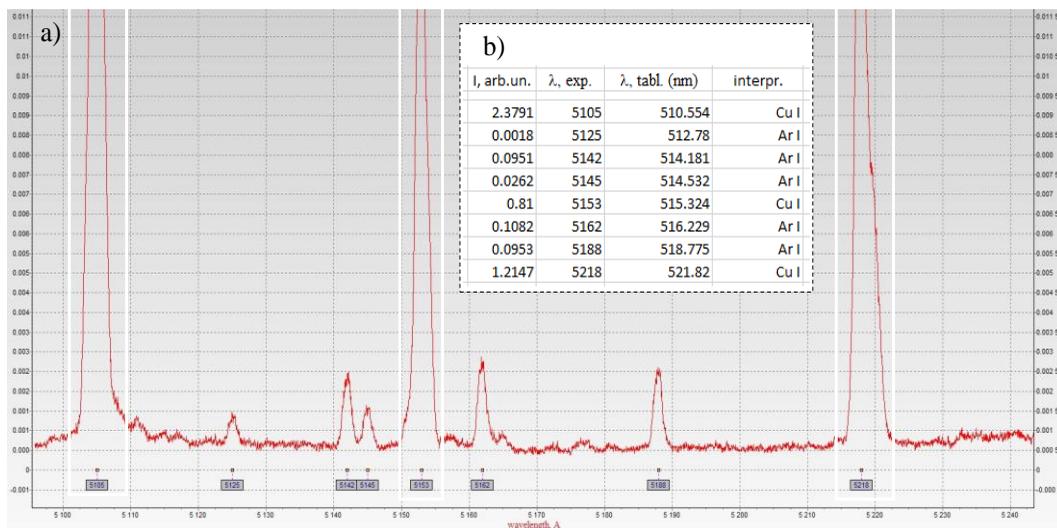
**Figure 2.** Functionality of OSA. Description in the text.

The OSA application permits to do the following:

- Determine the wavelength of an arbitrary spectral line and identify the optical spectrum by wavelengths [11] (Fig. 2b). In this case the nonlinear dispersion curve of ISP-51 has been taken into account. In Fig. 2b, the wavelengths of the detected lines, determined by OSA, are indicated as vertical labels.
- Interpret the identified spectrum lines [11] i.e. determine the attachment the observed in the work spectral lines to certain chemical elements. For this, a database of tabular data of spectral lines of various elements is connected to the software application [12].
- Determine the intensity distribution of the spectral line along its height  $h$  (corresponding points in Fig. 2b,c) [10], which reflects the distribution of the relative population of the corresponding excited states of particles of a certain chemical nature along the direction ( $h$ ) parallel to the discharge axis. It is also possible to determine the integral intensity of the spectral line by summing the value  $I$  of all the pixels forming this line.
- Information is displayed in two ways - in the form of a graph (Fig. 2c) and in the form of a text file that can be used for further processing by other programs.

## 2.2. Photoelectric radiation detection system

For diagnosis of MD by the OES method using a photoelectric radiation detection system (FE) the diffraction monochromator has been used (Fig. 1 (FE)). In this case, the radiation of the glow of the MD is output through the second  $W_{FE}$  diagnostic window and is projected using the achromatic  $L_{FE}$  lens onto the entrance slit (0.15 mm) of the MDR-23 diffraction monochromator (Optical Mechanical Association, St. Petersburg), in which the grating with groves per millimeter (600 lines / mm) is the dispersive element, and then is output through the exit slit (0.15 mm) of the monochromator. Typical resolution is 0.2 nm. Calibration of the sensitivity of the photon detection system was done with the tungsten standard lamp. The maximum of relative spectral sensitivity of the radiation detection system is observed for 460–470 nm. An optical signal is converted with the photomultiplier FEU 106 (Electro-Lamp Plant, Moscow) into an electric signal, amplified and recorded by the ADC. An adjustable automatic rotation of the diffraction grating permits to record the spectrum in the wavelength region of 320.0–800.0 nm.



**Figure 3.** Functionality of Spectr. Description in the text.

MD plasma parameters by the OES method using a photoelectric radiation detection system in the diagnostic complex are determined using the Spectr application created in the programming language Borland Delphi. The Spectr application has two operating modes: visualization and preliminary data processing directly during the experiment and full analysis after its completion. Pre-processing allows you to control the data during the experiment.

In Figure 3a there are shown a part of the emission spectrum of the glow MD region (the discharge mode is the same as in Fig. 2a).

Spectr allows you to:

- Determine the wavelength of the spectral line taking into account the linear dispersion of the monochromator.
- Determine the relative integral intensity of a selected spectral line (Fig. 3a).
- Using the tabular database on the spectral lines of various elements [12], interpret the decoded spectrum, i.e. to determine the correspondence of the spectral lines observed in the work to certain chemical elements (Fig. 3b).
- Scaling the dependence  $I(\lambda)$ , which increases the accuracy of processing the results and, therefore, significantly increases the information content of the experimental results.
- Display information in the form of a table (Fig. 3b), which shows the relative intensity of the spectral line (column 1), its calculated wavelength (column 2), the wavelength determined according to the spectral line tables (column 3), interpretation (column 4).

### 2.3. Comparison of the results obtained by two methods for radiation detection

To compare the results of evaluation of the relative intensity of the spectral lines determined by two methods of detection (F and FE), three lines of the Cu I spectrum were selected (highlighted by white rectangles in fig.2a and fig.3a). The table shows the wavelengths of the studied lines, the spectral transition and the normalized intensity of these lines, determined from the files shown in Fig. 2c) and Fig. 3b).

**Table 1.** Wavelengths, the spectral transition and the normalized intensity of the studied lines.

$\lambda$ , nm	Transition	Normalized line intensity *)			
		A	B	C	D
510.5	$4p \ ^2P^o_{3/2} - 4s \ ^2D_{5/2}$	1	1	1	1
515.3	$4d \ ^2D_{3/2} - 4p \ ^2P^o_{1/2}$	0.6	0.3	0.35	0.34
521.8	$4d \ ^2D_{5/2} - 4p \ ^2P^o_{3/2}$	0.71	0.53	0.52	0.51

\*) The method of radiation detection: A - photographic,  $h=0.5$  mm; B - photographic,  $h = 2$  mm; C - photographic, integral intensity; D - photoelectric, integral intensity.

The table 1 shows that the relative integral intensities of the studied lines of the Cu I spectrum are almost the same at the full intensities recording along the height  $h$  for two systems of radiation detection (C, D).

As previously indicated, the photoelectric system of radiation detection allows one to determine the relative integral intensity of the spectral lines, which depend from the intensity of radiation the whole investigated glow region along the selected direction. At the same time, the special feature of the photographic detection system is the localization of the studying region of glow, as the recorded spectrum has geometric dimensions - the height of the spectral line  $h$ . This allows one to determine the relative intensity of lines on any interval along the line height (Table 1 A, B). In this case, the distribution of the spectral line intensities along the height  $h$  reflects the

distribution of excited particles along the studied direction  $l$ . From fig. 2a and fig. 2c it is seen that the relative intensities of the studied lines change along the height of the spectral line  $h$ . Therefore, the relative particle distribution in the excited states  $4p\ ^3P_{3/2}$ ,  $4d\ ^3D_{3/2}$  и  $4d\ ^3D_{5/2}$  changes along the  $l$  axis in MD plasma. Thus, the data, obtained using the photographic radiation detection system, allow obtaining original scientific results and solving some problems that cannot be solved using the photoelectric system [13, 14, 15]. This, for example, is the determination of the distribution of excited particles depending on the distance to the target, which can be used to solve the fundamental problems of magnetron discharge physics.

### 3.0. Conclusions

A diagnostic complex based on a MSD for studying MD plasma by using two radiation detection systems (photographic and photoelectric) and digital processing of the obtained emission spectra is proposed.

The photographic system allows to study the spectrum of radiation in a wide spectral range (390.0–700.0 nm), which provides information on the population of excited states of plasma particles of various chemical nature. The analysis of the spatial distribution of radiation shows the distribution of particles of a certain chemical nature in a selected excited state along a direction parallel to the discharge axis. This information is very relevant for determining the influence of various kinds of collision processes [13] that occurring in a magnetron plasma discharge.

The photoelectric recording system gives the information about changes in time of the integral intensity of the studied spectral line. The developed software allows qualitatively and efficiently obtaining and analyzing experimental results.

### 4.0. References

- [1] C.H. Shon, J.K. Lee, Modeling of magnetron sputtering plasmas, *Applied Surface Science*, 192(2002) 258-269.
- [2] A. Kolpacova, P. Kurda, M. Tichy, Study of Plasma System by OES (Optical Emission Spectroscopy), *WDS`11 Proceeding of Contributed Papers, Part II*, (2011) 180-185.
- [3] D.O'. Connell, T. Gans, D.L. Crintea, U. Czarnetzki and N Sadeghi, Plasma Sources Science and Technology Plasma dynamics in an inductively coupled magnetic neutral loop discharge, *Plasma Sources Science and Technology*, (2008) 16 543.
- [4] S. Pfau, M. Tichý, Langmuir probe diagnostics of low-temperature plasma, in R. Hippler, S. Pfau, M. Schmidt, K.H. Schoenbach, Low temperature plasma physics, *WILEY-VCH, Verlag, Berlin*, (2000) 131–172.
- [5] Yu.A. Zheleznov, V.Yu. Khomich, (2011) *Priladnaya Fizika* 3 60 (In russian).
- [6] V.N. Ochkin 2010 *Spektroskopiya Nizhkotemperaturnoi Plazmi* (Moscow: Fizmatlit Press) p 421 (In russian)

- [7] Xi-Ming Zhu, Yi-Kang Pu, Using OES to determine electron temperature and density in low-pressure nitrogen and argon plasmas, *Plasma Sources Science and Technology*, 17(2008) 024002.
- [8] I.A. Afanasieva, V.V. Bobkov, V.V. Gritsyna, Y.E. Logachev, I.I. Okseniuk, A.A. Skrypnyk, & D.I. Shevchenko, On excited particle formation in crossed  $E \times H$  fields. *Vacuum*, 149(2018) 124-128.
- [9] T.E. Sheridan, M.J. Goeckner, J. Goree, Model of energetic electron transport in magnetron discharges. *Journal of Vacuum Science & Technology A: Vacuum, Surfaces, and Films*, 8(1990) 30-37.
- [10] I.A. Afanasieva, S.N. Afanasiev, N.A. Azarenkov, V.V. Bobkov, V.V. Gritsyna, Yu.E. Logachev, I.I. Okseniuk, A.A. Skrypnyk, D.I. Shevchenko, V.M. Chornous, Digital Processing of Optical Emission Spectra of Magnetron Sputtering Plasma System, *Problems Atomic Science Technology*, 120(2019) 164.
- [11] I.A. Afanasieva, S.N. Afanasiev, V.V. Bobkov, V.V. Gritsyna, D.R. Drozdov, Yu.E. Logachev, A.A. Skrypnyk, D.I. Shevchenko, Digital Identification of the Emission Spectrum Lines of Magnetron Discharge, *Problems of Atomic Science and Technology*, 122(2019) 35-38.
- [12] A.N. Zajdel', V.K. Prokofev, S.M. Rajsikij [i dr.] 1977 *Tablicy spektral'nyh linij* (Moscow: Nauka Press) p 800 (In russian)
- [13] Fantz U, Basics of plasma spectroscopy, *Plasma Sources Science and Technology*, 15(2006) S137.
- [14] Xi-Ming Zhu, Yi-Kang Pu, Optical emission spectroscopy in low-temperature plasmas containing argon and nitrogen: determination of the electron temperature and density by the line-ratio method, *Journal of Physics D: Applied Physics*, 43(2010) 403001.
- [15] G. Castro, M. Mazzaglia, D. Nicolosi, D. Mascali, R. Reitano, B. Zaniol, L. Celona, O. Leonardi, F. Leone, E. Naselli, L. Neri, G. Torrisi, S. Gammino, Application of optical emission spectroscopy to high current proton sources, *Journal of Physics: Conference Series*, 874 012033.

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