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Research paper

Study of argon ions density and electron temperature and density in magnetron plasma by optical emission spectroscopy and collisional-radiative model

Kirill E. Evdokimov*, Maxim E. Konischev, Vladimir F. Pichugin, Z. Sun

Tomsk Polytechnic University, 30 Lenin Avenue, Tomsk, 634050, Russian Federation

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ABSTRACT

Optical emission spectroscopy (OES) combined with the models of plasma light emission becomes non-intrusive and versatile method of plasma parameters determination. In this paper we have studied the densities of charge carriers and electron temperature in Ar plasma of pulsed DC magnetron in different experimental conditions. Electron density and temperature were determined by fitting of relative emission line intensities calculated from collisional-radiative model (CRM) to experimental ones. The model describes the kinetics of the first 40 excited states of neutral argon Ar and takes into account the following processes: electron impact excitation/deexcitation, spontaneous light emission, radiation trapping, electron impact ionization, and metastable quenching due to diffusion to walls. Then, ions density was determined from relative intensity of 488 nm Ar^+ emission line and simple CRM accounting excitation from ground states of neutral Ar and ion Ar^+. The values of electron and ion density agree very well. To test the stability of results, we performed Monte-Carlo calculations with random variation of experimental spectrum as well as of excitation cross-sections and estimated confidence intervals and errors for plasma parameters. Also, we validated OES study by comparison with Langmuir probe measurements. The agreement between optical and probe techniques is satisfactory.

1. Introduction

Optical emission spectroscopy (OES) is widely used for low-temperature plasma diagnostics [1–6]. This method is comparatively cheap, versatile and non-intrusive. It allows determining such plasma parameters as electron temperature and density by so-called line-ratio technique. Each spectral line corresponds to optical transition between two quantum levels of atom/molecule, and a number density of gas species in the upper state determine spectral line intensity. In its turn the number density is the function of electron temperature and density. Having determined densities of species in various states by some population model, one can calculate dependence of different spectral line intensity ratios versus electron temperature and density and distinguish these parameters by comparison with experiment.

A population model should take into account two main processes in the case of low-temperature plasma: electron impact excitation and optical radiative transition. In general such models are called collisional-radiative and the simplest one called *corona model* takes into account only excitation and optical transition from one level [7]. For argon plasma there are some metastable states playing important role in kinetics, corona model is not valid for most states and one should use more comprehensive one. The review of usage of collisional-radiative models (CRM) for determination of plasma parameters can be found in [4].

This paper aims to study parameters of magnetron discharge plasma. We developed argon CRM, measured emission spectra of plasma in laboratory installation for reactive magnetron sputter deposition and proposed technique for determination of electron temperature and density by minimization of merit function composed from experimental and model line intensities. Also, we compared determined electron parameters with the results of double Langmuir probe measurements and Ar ions density calculated from simple CRM for Ar^+.

2. Experiment

Plasma of magnetron discharge in laboratory installation UVN-200MI with pulsed DC magnetron for reactive sputter deposition

* Corresponding author.
E-mail address: evdokimov@tpu.ru (K.E. Evdokimov).

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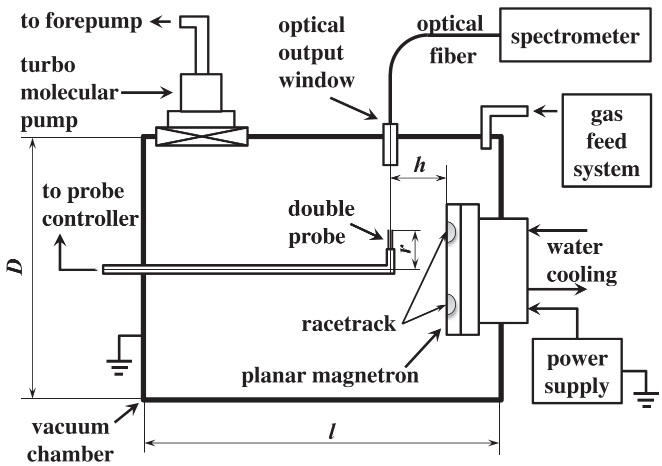

Fig. 1. Sketch of the experimental setup: $D = 380$ mm is the vacuum chamber diameter, $l = 450$ mm is chamber length, $h = 100$ mm is the distance between magnetron plane and optical window axis, $r = 65$ mm is the distance between chamber axis and the probe tips.

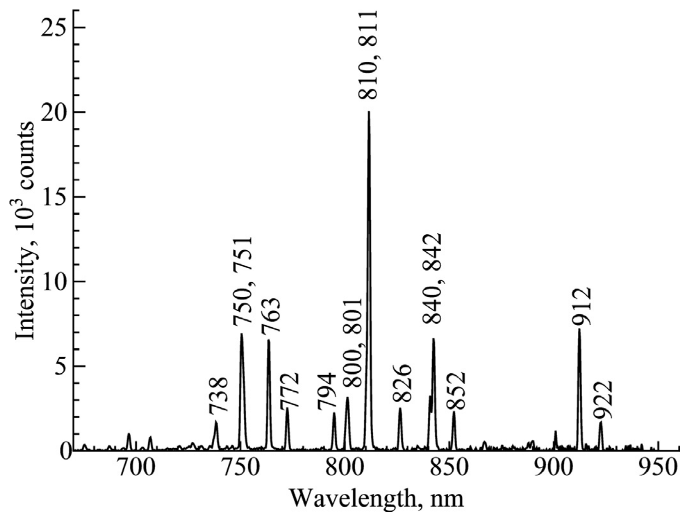

Fig. 2. Emission spectrum of Ar plasma in laboratory installation UVN-200MI for reactive magnetron sputter deposition. Most intense lines of Ar atom are marked by corresponding wavelength. Operation parameters: discharge power 1 kW, pressure 0.06 Pa.

was investigated. [8]. Cylindrical vacuum chamber of the installation has diameter 380 mm and length 450 mm. Vacuum pumping speed is 150 l/s and working pressure varies from 0.04 to 0.5 Pa. A titanium target with 200 mm diameter was sputtered in argon atmosphere. Magnetron power source PS MS1 provides modulated magnetron supply with frequency 60 kHz and fill factor 80%, and works in the average power stabilization regime.

Plasma emission spectra were detected by Avantes Avaspec 3648 spectrophotometer with spectral resolution (FWHM) of 1.4 nm. Optical system including spectrophotometer and optic fiber was intensity calibrated with a tungsten-ribbon lamp. Optical fiber was connected to light output window straightly without lens. Output window was protected from contamination by steel tube with 6 mm inner diameter and 42 mm length placed inside the chamber. Taking into account 18 mm thick chamber walls, the effective acceptance angle can be estimated as 6° giving the effective collecting volume of $1.5 \cdot 10^{-4}$ m^3.

Simultaneously, double probe measurements were performed. Cylindrical probe tips are made from tungsten and have 23.4 mm length and 0.5 mm diameter. The distance between the tips is 3 mm. The probe was positioned near magnetron racetrack area in front of optical output window. Overall experiment schematic is shown in Fig. 1.

Probe data was interpolated and averaged by the algorithm described in [9] and then plasma parameters were determined using technique from [10]. First, we registered 80–100 voltage–current characteristics for each experimental instance. In our case I–V curves usually have high frequency noise due to vicinity of magnetron power source frequency and the frequency of probe data acquiring controller. To diminish this effect, all I–V curves was interpolated, averaged and additionally smoothed by Savitzky–Golay filter. Then, electron temperature was determined by voltage difference between two extrema of double derivative of I–V curve. And finally, ion density was determined by fitting experimental curve to theoretical one in orbital-motion limit approximation.

We neglected the effect of surface contamination and magnetic field because sputtered material (Ti) is a conductor and magnetic field is relatively weak. An estimate of magnetic field strength in the probe region based on characteristics of magnetic system gives value about 5 mT and corresponding electron gyration radius is much larger than the probe tip radius. However, these factors may influence determined plasma parameters, especially charge carrier density.

Fig. 2 represents characteristic emission spectrum of Ar plasma in the installation.

The most bright spectral lines in registered Ar emission spectrum correspond to $2p \to 1s$ transitions and lay in 660– 930 nm range where upper limit is determined by spectrophotometer sensitivity. We have chosen 20 spectral lines in this range belonging to $2p \to 1s$ manifold with Einstein coefficient exceeding 10^6 s^{-1} and determined experimental intensity I_{ij}^{OES} for each. Here indexes i and j denote the upper and the lower states of Ar atom corresponding to certain transition. Intensities of unresolved spectral lines were determined by curve-fitting method using pseudo-Voigt instrumental line shape (ILS). Parameters of ILS were derived from most bright resolved spectral lines.

3. Argon collisional-radiative model

In this work we adopted CRM proposed in Gangwar et al. [11] and extended it by taking into account metastables quenching due to collisions with vacuum chamber walls. The latter process was accounted similarly to Iordanova and Koleva [3]. CRM describes homogeneous plasma and space non-uniformity is accounted by introducing characteristic plasma length determining self-absorption and diffusion. The model considers steady-state low-temperature and low-density argon plasma and describes kinetics of ground state and first 40 excited states of Ar atom. Table 1 represents the states along with excitation energy E_i and statistical weight g_i taken from NIST Atomic Spectra Database [12]. Here, the excited states are in Paschen notation [1] and "gs" denotes the ground state.

Population of the states is determined by the following set of particle balance equations:

$$\sum_{j \neq i} n_e n_j K_{ji}^{ex} + \sum_{j>i} n_j A_{ji} \Lambda_{ji} = n_i \sum_{j \neq i} n_e K_{ij}^{ex} + n_i \sum_{j<i} A_{ij} \Lambda_{ij} + n_e \, n_i \, K_i^{iz} + \nu_i^d \, n_i, \tag{1}$$

where n_e is the volume averaged electron density, n_i is the volume averaged density of atoms in ith state, K_{ij}^{ex} is the electron-impact excitation/deexcitation coefficient from ith to jth state, K_i^{iz} is the electron-impact ionization coefficient for ith state, A_{ij} and Λ_{ij} are transition probability (Einstein's coefficient) and escape fac-