Register impurities in the plasma forming gas helium in a large volume reactor using plasma electron spectroscopy method

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Abstract. The results of this article show the possibility of determining the qualitative composition of impurities in buffer helium in a high-volume vacuum reactor by initiating a short glow in a small (non-working) part of this volume and using the PLES method.

1. Introduction

The low-temperature plasma produced in a large volume can be of interest for the study of various phenomena occurring in plasma, and for applications [1–3]. This interest is motivated, in particular, by the development of technologies of surface treatment of materials, such as etching, plasma chemical synthesis, and production of thin films [4–8]. Inert gases such as argon and helium are usually used for this purpose. It is also worth noting that there are always impurities when discharge chambers of a large volume are used in real experiments. Their presence is associated with the use of fore-vacuum gas evacuation (only up to ~10^{-2}-10^{-3}) and a large number of metal parts in the discharge chamber, which produce impurities contaminating the original gases during discharge. The presence of these impurities can have a significant effect on plasma parameters: on the balance of excited and charged particles and on the formation of the electron distribution function (EDF) in the plasma parameters and, accordingly. The purity of the buffer gas, as a rule, is determined using optical methods, etc.

A promising method for the determination of impurities in high-volume metal chambers can be the PLES method [9] and gas-analytical detectors based on it, described in papers [10-14]. It is based on measurements of the spectra of Penning ionization reactions of atoms and molecules of an impurity A by metastable atoms of a buffer gas B*:

\[ A + B^* \rightarrow A^+ + B + e\{E_p\}, \]  

\[ B^* + e \rightarrow B + e\{E_m\}, \]  

(1a)  

(1b)

where the appearance energy of fast electrons in reactions (1) is expressed as \( E_p = E_m - E_i \) (\( E_m, E_i \) are, respectively, the excitation energy of a buffer carrier gas and the ionization energy of impurity). The best choice for \( B^* \) particles in reactions (1) are helium metastable atoms He* with an excitation energy of 19.8 eV, which are able to ionize any chemical compound (with the exception of neon).

The spectrum of the fast electrons produced in (1) consists of narrow peaks corresponding to their production energy values \( E_p \) in reactions (1) [9-14]. If we measure the energy \( E_p \) and knows the excitation potential of helium (\( E_m = 19.8 \) eV), then one can determine the ionization potential \( E_i \) of the sought atomic or molecular impurity \( A \) (\( E_i = 19.8 - E_e \)), thereby identifying them (qualitative analysis).
By absolute measurements and/or calibration of these peaks, it is ultimately possible to carry out a qualitative analysis of the impurities $A$.

The results of this article show the possibility of determining the qualitative composition of impurities in buffer helium in a high-volume vacuum reactor by initiating a short glow in a small (non-working) part of this volume and using the PLES method.

2. Experimental Setup

Experiments were carried out in a cylindrical grounded vacuum chamber made of steel with 15 cm in height and 20 cm in diameter. A glass tube with steel electrodes with a diameter of 5 mm and an interelectrode distance of 5 mm was placed inside the chamber. A cylindrical probe made of nickel with diameter 0.9 mm and length 5 mm was used for measurements.

A cylindrical probe made of nickel with a diameter of 0.9 mm and a length of 5 mm, which was located in the center of the tube at the same distance from the electrodes was used for measurements. Gas pressure in chamber volume 5 Torr were chosen so that a steady short glow discharge could be observed, and that a probe could be positioned in the negative glow region of the discharge. High voltage power supply was used for applying a voltage of up to 1 kV on the electrodes. The discharge current was limited by a 11-kΩ ballast resistor. Prior to experiments, the chamber was evacuated to $10^{-3}$ Torr using forevacuum pump, and then filled with helium up to the required pressure. Filling control was carried out using a needle inlet valve.

Probe measurements were carried out using the modern automated probe diagnostics system "Multifunctional Plasma Probe Analyzer" (MFPA) [14]. A more detailed description of the measuring circuit can be found in [15, 16]. The MFPA system allows for carrying out probe diagnostics in both stationary and nonstationary plasmas: obtaining probe current–voltage characteristics (CVCs), its first and second derivatives, measuring plasma potential, plasma density, electron temperature, and calculation of the electron energy distribution function (EEDF) by Druvestein’s formula [17,18]. A single probe acquisition time of the system is $t_{acq} = 0.5$ ms, which allows for obtaining a sufficiently high resolution for the EEDF (0.1 eV).

3. Results

During a series of experiments, growing current–voltage characteristics of the discharge were obtained (Fig. 3 a). The fact that the voltage between the electrodes increases with the current indicates that the discharge is in a stable mode.

Probe current-voltage characteristics, as well as their second derivatives with respect to the potential (slow part and first part, corresponding) for different values of the discharge currents, are presented in Figs. 1b – 2.

Plasma parameters such as electron density and electron temperature (see Table) were determined from CVC of the probe and their second derivatives of the probe current on the applied probe potential according to the method [18].

![Figure 1. a) DC discharge CVC in a small gap and b) Current-voltage characteristics (CVC) of the probe in a short glow discharge plasma for various discharge current values at a pressure of 5 Torr.](image)
Figure 2. a) The slow part and b) the fast part of the second derivatives of the probe current on the applied probe potential for different discharges currents.

As one can see from Figs. 2b, spectra of characteristic electrons are observed around 19.8-20 eV, which is typical of fast electrons arising in collisions of the second kind,

\[ \text{He (}^3S_1\text{)} + e \rightarrow \text{He} + e(19.82 \text{ eV}), \]
\[ \text{He (}^1S_0\text{)} + e \rightarrow \text{He} + e(20.61 \text{ eV}). \]

Besides the helium maximum at 19.8 eV, two peaks at approximately 3.1 eV, 4.2 eV and 6.2 eV are well expressed. They are related to electrons produced in Penning reactions of triplet metastable helium atoms with oxygen atoms and nitrogen molecules, respectively:

\[ \text{He}(^3S_1) + N_2 \rightarrow \text{He} + N_2^+(\text{}^3\Pi^+) + e(3.1\text{ eV}), \]
\[ \text{He}(^3S_1) + N_2 \rightarrow \text{He} + N_2^+(\text{}^3\Sigma^+) + e(4.24\text{ eV}), \]
\[ \text{He}(^3S_1) + O \rightarrow \text{He} + O^+(^3S_{1/2}) + e(6.20\text{ eV}). \]

4. Conclusions

Thus, the presented results demonstrate the possibility of determining the qualitative composition of a buffer gas in large-volume metal chambers by organizing a short glow discharge with a non-local plasma in a small (non-working) part of this volume and using the PLES method.

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