Investigation of electrophysical and thermophysical characteristics of a low frequency nitrogen inductive discharge

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Abstract. A low-frequency (100 kHz) ferromagnetic enhanced nitrogen inductive discharge has been experimentally investigated in the nitrogen pressure range of 5–55 Pa and discharge current density of 20–80 mA/cm². Dependence of the discharge electric field strength $E$ on the gas pressure was measured for various discharge currents. Dependence of the neutral gas temperature on the gas pressure and discharge current was determined using optical emission spectroscopy, from the measured 0–0 vibro-rotational band of the second positive system of molecular nitrogen ($\text{C}_3\Pi_u$–$\text{B}_3\Pi_g$). Based on the determined neutral gas temperatures, values of the reduced electric field strength $E/N$ were calculated.

1. Introduction

Radio-frequency inductively coupled plasma (RF ICP) is widely used in various fields of science and technology as a source of ions and chemically active particles (ion implantation, plasma etching, plasma enhanced chemical vapour deposition, surface modification, etc.). The main disadvantages of RF ICP include poor magnetic coupling between the ICP coil and plasma ($k = 0.2–0.7$) and high frequency of discharge generation (typically 13.56 MHz). The use of a ferromagnetic core to improve the magnetic coupling between the ICP coil and plasma can significantly increase the efficiency of ICP generation, as well as reduce the driving frequency by two to three orders of magnitude [1]. The low driving frequency, in turn, allows the use of cheap and mass-produced power supplies for induction heating. Thus, a low-frequency inductive discharge with ferromagnetic enhancement of magnetic coupling between the ICP coil and plasma (FMICP) is of interest to develop new plasma sources, in particular, for plasma-assisted nitriding [2].

One of the main electrical parameters of gas discharge is the reduced electric field strength $E/N$, determining the electron energy distribution function $f(E/N)$, where $N$ is the concentration of neutral particles. Due to a high current density and a high frequency of electron-atom elastic collisions, the ICP neutral gas temperature $T$ can be significantly higher than room temperature even at a low gas pressure [3], which leads to a decrease in the gas density ($N = p/kT$) and an increase in the reduced electric field strength of gas discharge. Therefore, gas heating can have a noticeable effect on the ICP parameters. The aim of the paper is to measure the neutral gas temperature and to determine the reduced electric field strength of the low-frequency ferromagnetic enhanced nitrogen inductive discharge, in the gas pressure range typical of plasma nitriding technologies.
2. Experimental setup

A principal scheme of experimental setup is shown in figure 1. Gas discharge chamber 1 is made of quartz tubes with the inner diameter of 55 mm and the total length of 120 cm. Ferrite cores 2 with the total cross-section of 106 cm$^2$ enhance magnetic coupling between the discharge and the primary winding 3. A matching network 4 (variable LC circuit) is used for discharge ignition, discharge current stabilization and power regulation. As a power supply, 12 kVA 50–100 kHz power supply for induction heating is used. Discharge current $I$ is measured with a current transformer (Rogowski coil) 6. Discharge voltage $U$ is measured with a voltage loop 7 encircling the ferrite cores and collecting the alternating magnetic flux $\Phi$ that drives the discharge ($U=\frac{-d\Phi}{dt}$). Discharge electric field strength $E$ is determined as a ratio of the discharge voltage to the discharge path $L$: $E=\frac{U}{L}$ [4]. To determine neutral gas temperature, optical emission spectroscopy (OES) is used based on the analysis of N$_2$ molecular spectra. Optical emission spectrum is measured with a 0.12 nm resolution spectrometer AvaSpec-2048 8.

3. Numerical model of the diatomic nitrogen vibro-rotational spectra

The OES of the second positive system N$_2$ (C$^3\Pi_u$ – B$^3\Pi_g$) is used to determine the nitrogen rotational temperature, which, as shown in [5], is in equilibrium with the gas temperature. The algorithm for the molecular spectrum calculation is to determine the line intensities of the P, Q and R branches of a selected vibro-rotational band. As a result of spin-orbit interaction, the upper ($E'$, $\nu'$, $J'$) and the lower ($E''$, $\nu''$, $J''$) levels of the radiative transition are triply degenerate, where $E$ is a level energy, $\nu$ is a vibrational quantum number and $J$ is a rotational quantum number. Therefore, the transitions C$^3\Pi_0$–B$^3\Pi_0$, C$^3\Pi_1$–B$^3\Pi_1$ and C$^3\Pi_2$–B$^3\Pi_2$ should be considered. In addition, $\Lambda$-type doubling leads to the split of each branch into the relevant sub-band. However, since the observed vibro-rotational structure was not resolved by our spectrometer, in calculating the molecular spectrum, $\Lambda$-type doubling and spin splitting were neglected. In this case, the vibro-rotational band spectrum can be represented as a Dunham series [6]:

$$\lambda = \sum_{p=0}^{2} \sum_{q=0}^{2} Y_{pq}^C (\nu' + 1/2)^p [J'(J'+1)]^q - Y_{pq}^B (\nu'' + 1/2)^p [J''(J''+1)]^q ,$$  

where $n_a$ is the index of refractivity and $Y_{pq}$ are the constants related to the vibro-rotational transition taken from [7]. In the current paper, 0–0 vibro-rotational band is analyzed, therefore $\nu'$ and $\nu''$ in the equation (1) are equal to zero.

The spectral line intensities $I$ in the selected band can be presented as follows:

![Figure 1. Experimental setup:](image-url)
where $C$ is a constant, $T_r$ is a rotational temperature, $F_{J'}$ is an energy of the upper term with a rotational quantum number $J'$, $S_{J'J''}$ is a line strength [8].

For small values of the rotational number $J$, the transition refers to the Hund case (a). The increase of the rotational number value leads to the weakening of the spin-orbit coupling (Hund case (b) [9]). Since the difference between the cases (a) and (b) is insignificant, line strengths $S_{J'J''}$ for the Hund case (a) were used for the rotational number values $J$ of 0–50. The upper term energy was calculated by equation (3):

$$F_{J'} = \hbar c \sum_{p=0}^{5} \sum_{q=0}^{2} y_{pq}^C (v' + 1/2)^p [J'(J' + 1)]^q.$$  \hspace{1cm} (3)

The calculated discrete spectrum $I(J')$ was convoluted with the instrumental broadening function and compared with the measured spectrum. To determine the instrumental broadening function of the AvaSpec-2048 spectrometer, spectra of a helium glow discharge were measured, the shape of a He spectral line 388.8 nm was analyzed. Varying the rotational temperature $T_r$, the convoluted spectrum was fitted with the measured one using the least square method. The temperature corresponding to the best matching between the experimental and numerical spectra was considered as the gas temperature $T$. The samples of the calculated and experimentally measured molecular spectra of the 0–0 vibro-rotational band are shown in figure 2. The temperature accuracy is about ±50 K.

4. Results and discussion

In figure 3, dependences of the nitrogen FMICP electric field strength on the gas pressure are shown, for various discharge currents. With an increase in the FMICP current, the discharge electric field strength is decreasing, thus demonstrating a typical gas discharge negative volt-ampere characteristic. With an increase in nitrogen pressure $p$ and gas density $N$, the discharge electric field strength is increasing. To determine the gas density $N=\rho/kT$, the gas temperature was measured using OES and the above-mentioned numerical procedure for the N2 spectrum calculation. In figure 4, dependences of the gas temperature on the gas pressure are shown for various FMICP currents. With an increase in the gas pressure and FMICP current the gas temperature is rising due to the increase in the frequency of electron-molecule collisions and energy transfer between electrons and neutrals. As it is seen from the figure 4, even at the lowest gas pressure of 5 Pa FMICP gas temperature is noticeably higher (380–470 K) than the room temperature, therefore gas heating does affect the FMICP gas density and should be taken into account while determining the reduced electric field strength $E/N$. For comparison, in a low pressure (5 Pa) argon RF ICP the gas temperature determined by the same method is about 500–550 K [3].

Figure 2. Comparison of a calculated ($T_r=380$ K) and measured N2 spectra at $\rho=5$ Pa and $I=0.5$ A.

Figure 3. Dependency of $E$ vs. $\rho$ for various FMICP currents.
Figure 4. Dependence of gas temperature $T$ vs. gas pressure $p$, for various FMICP currents.

Figure 5. Dependence of reduced electric field strength $E/N$ vs. $N\Lambda$ values.

In figure 5, dependences of the reduced electric field $E/N$ on the $N\Lambda$ values are shown, where $\Lambda=0.94$ cm is a characteristic diffusion length. In contrast to the $E(I)$ dependence, the reduced electric field values weakly depend on the FMICP current. Therefore, the electron temperature does not change significantly with the FMICP current variation at a fixed gas density. With an increase in the $N\Lambda$ values the reduced electric field strength is decreasing, therefore the electron temperature is lowering too.

Conclusions

New experimental data on the plasma properties of a low frequency (100 kHz) low pressure (5–55 Pa) nitrogen ferromagnetic enhanced inductively coupled discharge have been obtained. Gas temperature was measured using optical emission spectroscopy by a 0–0 vibro-rotational transition of the second positive system of molecular nitrogen, significant gas heating was revealed even at the lowest gas pressure of 5 Pa. Taking into account gas heating, the characteristic values of the reduced electric field strength $E/N$ determining the electron energy distribution function were calculated.

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