An inductive RF discharge in water vapor for atomic-emission spectrometry

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Abstract. The paper presents the results of experimental measurements of the emission properties of water inductively coupled plasma. The plasma temperature is calculated using the Boltzmann method. It is shown that the temperature decreases with increasing pressure. The plasma resistance, its conductivity, and the thickness of the skin layer are estimated. Analysis shows that the emissivity of water plasma can be optimized while maintaining the power consumption.

1 Introduction

Inductively-coupled plasma (ICP) has a number of distinctive properties, such as purity, high stability, and equilibrium, which makes it attractive for use in applications such as coating, etching, the emission analysis of the chemical composition of substances, purification, and disinfection [1]. Recently, there has been an increased interest in the properties of water vapor as a plasma-forming gas for the listed applications since water vapor is a relatively cheap raw material which dissociates in the discharge to form highly reactive oxygen and OH hydroxyl. The presence of atomic hydrogen in the plasma allows a relatively easy spectrometric analysis of parameters such as the plasma temperature and electron concentration from the hydrogen Balmer series [2].

The possibility of using an inductive discharge in water vapor for atomic emission spectrometry was studied in [3]. In comparison with electrode and diaphragm (capillary) discharges in a liquid, an inductive discharge does not suffer from drawbacks such as the effect of electrodes and the energy loss due to Joule heating of the liquid [4-5]. However, to excite an inductive discharge, it is necessary to reduce pressure in the discharge chamber to 10⁻⁴ – 10⁻⁵ bar, which leads to difficulties in introducing the sample into the discharge zone and additional requirements for tightness.

Although water ICP has been studied in a number of paper [6-8], appropriate methods for its diagnostics are not available, which makes it difficult to develop systems using water ICP for industrial and analytical applications. Therefore, the study of the energy and spectral characteristics and the search for regularities that allow estimating the parameters of water ICP remains an important problem.

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2 Experimental setup

A diagram of the experimental setup is shown in Fig. 1. Inductive discharge was excited in a quartz tube 1 of diameter \( D = 20–45 \) mm by a cooper inductor which had \( N = 2–5 \) coils. The inductor was supplied with a current with a frequency of 27.12 MHz from a CESAR 2710 high-frequency source. The load power output was \( P_s = 600 \pm 50 \) W. A vacuum pump was installed on one side of the quartz tube, and a vessel with tap water 2 with a metering valve 3 was placed on the other side. A check valve 4 was installed on the side of the vacuum pump.

Before the RF generator was turned on, air was evacuated from the system to a pressure of \( \approx 0.1 \) mbar with the valve 3 closed and the valve 4 open. After this, water vapor was supplied to the system using the metering valve 3. Then the RF generator was turned on, and the discharge was initiated. The discharge spectrum was recorded from the side of the quartz tube through an optical fiber cable 5 using a Solar Systems S-150 spectrometer having a spectral range of \( \lambda = 180–1100 \) nm and a spectral resolution of \( \approx 0.5 \) nm. The exposure of spectrum recording was varied depending on the brightness of the recorded lines. The pressure \( p \) in the discharge tube was monitored by a vacuum gauge 6 (Testo 552). The discharge burning time was 0.1–0.3 s.

Steady discharge burning in water vapor at a power input \( P_s = 600 \) W was observed at a pressure \( p \) of up to 5 mbar. When the pressure was raised above 10 mbar by increasing the vapor flow with the metering valve 3, the discharge extinguished due to insufficient power.

3 Results and analysis

The circuitry of the setup with the matching circuit is similar to that presented in [9]. Knowing the parallel matching capacitance \( C \), from the circuit parameters we can determine the water plasma resistance by the formula \( R = \frac{1}{2 \pi f r} \sqrt{r - R} \), where \( R \) is the resistance of the plasma reduced to the primary winding \( (R = R_{pl} N^2) \), \( r \) is the internal resistance of the generator (in our case, \( r = 50 \) Ohm), and \( f \) is the generator frequency.

The resistance of water inductively coupled plasma \( R_{pl} \) for a tube with \( D = 45 \) mm at \( p = 0.25 \) mbar was \( R_{pl} \approx 4 \) Ohm. For \( D = 20 \) mm and \( p = 1 \) mbar, we have \( R_{pl} \approx 1 \) Ohm.
Using the expression \( R_{pl} = \frac{\pi D}{\sigma l \Delta} \), where \( \sigma \) is the plasma conductivity, \( l \) is the width of the plasma loop (approximately equal to the length of the inductor), \( \Delta = \frac{1}{\sqrt{\pi \sqrt{\mu} \sigma}} \) is the depth of the skin layer (\( \mu \) is the magnetic permeability of the plasma) and knowing the resistance of the plasma, we can estimate its integral conductivity and the thickness of the skin layer.

In experiments with \( D = 20 \text{ mm} \) and \( D = 45 \text{ mm} \), the plasma conductivity was found to be approximately equal and equal to \( 10^3 \text{ Ohm}^{-1}\text{m}^{-1} \) and the depth of the skin layer was 3 mm. These values are close to the values obtained for argon plasma under the same conditions [10].

The temperature of the water ICP was determined from the hydrogen Balmer series using the Boltzmann method [11].

**Fig. 2.** Plasma temperature, determined from the relative intensities of spectral lines \( H_\alpha, H_\beta \) and \( H_\gamma \), as a function of pressure for \( D = 20 \text{ mm}, P_s = 600 \text{ W}, \) and \( N = 5 \).

Figure 2 shows the plasma temperature as a function of pressure for a tube of diameter \( D = 20 \text{ mm} \), a discharge power \( P_s = 600 \text{ W} \), a number of inductor coils \( N = 5 \). The temperature is determined from the relative intensities of the first three lines of Balmer hydrogen series (\( H_\alpha = 656.3 \text{ nm}, H_\beta = 486.1 \text{ nm}, H_\gamma = 434.1 \text{ nm} \)). It can be seen that the temperatures are not equal: the line \( H_\beta \) has the increased intensity as compared with the case of local thermodynamic equilibrium, so the temperature determined between \( H_\beta \) and \( H_\alpha \) is higher and between \( H_\gamma \) and \( H_\beta \) is lower than the actual one. For the tube with \( D = 45 \text{ mm} \) the dependency has the same character.

Despite of the impossibility of the accurate temperature determination nevertheless it can be seen that the plasma temperature decreases with increasing the pressure at the same heating power, which is natural since the particle concentration increases in proportion to the pressure.

**4 Conclusion**

Some characteristics of water ICP, such as resistance, integral conductivity, and temperature were determined.
It is shown that the resistance and conductivity of the plasma are close to those for argon ICP [10]. At an input power of 600 W and a diameter of the discharge tube of 20–45 mm, the maximum temperature of water ICP is 5000–6000 K.

These parameters, obtained at a sufficiently low input power, allow water to be considered as a promising medium for plasma-chemical and analytical applications.

Typical temperatures of argon ICP in emission spectral analysis are 6000–10000 K. To achieve these temperature parameters, it is necessary to increase the power mass density (the power per unit mass of plasma) introduced into the plasma. To do this, it is necessary to increase the power of the HF generator, reduce the volume of the plasma loop, and decrease the pressure.

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