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Plasma Studies in TÜBİTAK National Metrology Institute

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Abstract:

Over the past few decades, low-temperature plasma researches have gained momentum as they lie at the core of numerous applications in industry, environment and medicine. Recently, metrology community has also begun to take advantage of the unusual properties of plasma to develop new advanced measurement techniques and new measuring devices. In accordance with this purpose, in 2019, the Plasma Laboratory was installed in TÜBİTAK National Metrology Institute (UME) within the scope of infrastructure research and development program founded by Industry Ministry of Turkey for contributing to the ongoing worldwide research on plasma studies. In this work, the current advances in Plasma Laboratory of UME will be introduced. We present the installed experimental set-up for low-temperature and low-pressure gas discharge plasma. We present our preliminary measurement results in determining electron temperature by Langmuir probe and optical emission spectrometer.

Keywords: Thermodynamics of plasmas; Plasma properties; Plasma diagnostic techniques and instrumentation; Discharge in vacuum; Glow; Corona.

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1. INTRODUCTION

The term “plasma” was first introduced by Irving Langmuir in 1928 to describe the quasi-neutral ionized gas [1] where in addition to charged particles (electrons and ions) there also exist neutral atoms and molecules, metastable particles (excited atoms and molecules, radicals), and photons. Depending on the temperature of particles, it is referred as equilibrium, quasi-equilibrium and non-equilibrium plasma. The equilibrium plasma is composed of completely ionized gas at (10^6 – 10^8) K where the electrons and ions are in thermal equilibrium. The quasi-equilibrium plasma contains neutral atoms and molecules as well as charged particles at temperatures smaller than 2×10^4 K. Non-equilibrium plasma, on the other hand, consists of partially ionized gas at (300 - 1000) K where the electron temperature is much higher than the temperature of ions, neutral atoms and molecules. Over the past few decades, the competitive advantages of the non-equilibrium plasma have triggered its widespread applications in the industry, environment and medicine from manufacturing microelectronics and microprocessors to the decomposition of chemical pollutants and harmful microorganisms in water and air [2-11]. Recently, metrology community has also begun to take advantage of the unusual properties of plasma to develop new advanced measurement techniques and new measuring devices [12-16]. In accordance with this purpose, in 2019, Plasma Laboratory is installed in TÜBİTAK National Metrology Institute (UME) within

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Acta Materialia Turcica

Volume 5, September 2021, Pages 24-29

the scope of infrastructure research and development program founded by Industry Ministry of Turkey. This paper focuses on the ongoing research in TÜBİTAK UME on the non-equilibrium plasma generated in vacuum.
The non-thermal plasma can be generated under the low-pressure conditions where the collisions between the electrons, ions and neutral atoms and molecules occur rarely. MW, RF, low frequency, pulsed and DC power supplies are the common sources of plasma generation at low-pressures. The widespread applications of low-pressure plasma are manufacturing semiconductor components, decontamination and sterilization of medical devices, etc.

2. INFRASTRUCTURE IN UME PLASMA LABORATORY

Low-pressure non-equilibrium plasma is generated by a TDK-Lambda DC power supply in an evacuated stainless steel vacuum chamber of cylindrical shape with a diameter and a height of 500 mm. The discharge cell consists of two stainless steel parallel electrodes of 150 mm in diameter and 8 mm in thickness, positioned at the center of the vacuum chamber with 120 mm spacing in between. The vacuum chamber is pumped down to a base pressure of 10^{-7} Torr by a 15 m^3/h mechanical pump and a 400 L/s turbomolecular pump. A continuous dynamic flow (1-50 sccm) of ultra high purity Argon, Oxygen, Nitrogen or Helium gases were let into the chamber through a needle valve such that a DC glow discharge is activated between the electrodes at a pressure of up to 100 mTorr. Four flanges on the lateral surface of the chamber are available for invasive and non-invasive diagnostics of plasma (See Fig. 1). A Langmuir probe and an optical emission spectrometer are used for determining the plasma properties.

2.1 Langmuir Probe

A Langmuir probe is one of the widely used precision instruments for the characterization of non-equilibrium plasma. It is an invasive technique for plasma diagnostics where an electrically biased bare wire or metal disk with respect to the reference electrode is inserted into the bulk of plasma to collect electron and/or ion currents. The *I-V* characteristic of the probe is used deducing the plasma parameters. Among the key parameters measured by Langmuir probe are the plasma potential, the floating potential, the electron temperature, the electron current density, the electron density, the electron energy distribution function (EEDF), the ion current density, the ion density, the Debye length, the ion saturation current and the mean electron energy.

2.2 Optical Emission Spectrometer

The optical emission spectrometer covering a wavelength range of UV–visible–near-IR region (200 – 1100 nm) emission spectrum having a resolution of 0.2 nm is used for non-invasive diagnostics of the non-equilibrium plasma properties. From the emission spectra of excited atomic or molecular species by the electron-impact processes, under the assumption of Maxwellian EEDF, the electron temperature and the electron density is determined by applying line-ratio analysis on the two dominant excitation and spontaneous emission processes.

Figure 1. UME Plasma Laboratory infrastructure

Corresponding Author E-mail: *beste.korutlu@tubitak.gov.tr*

Acta Materialia Turcica

Volume 5, September 2021, Pages 24-29

3. ONGOING VACUUM PLASMA STUDIES

The objectives of the non-thermal plasma studies in UME Plasma laboratory are developing a primary standard for the realization of electron temperature and advancing in the sputtering techniques. The focal point of this paper is the construction of an electron temperature primary standard.

At this point it is necessary to give the definition of temperature unit in International System of Units (SI). Between the years 1954 and 2019, SI temperature unit Kelvin (symbol K) was defined by assigning a numerical value of 273.16 K to the thermodynamic temperature of the triple point of water where all three phases (solid, liquid and gas) coexist at thermodynamic equilibrium. This artifact based definition is abandoned for the benefit of ensuring the long-term stability and the traceability of the unit. Since May 20th, 2019 the unit is defined by the Boltzman constant as follows [17-19]:

> The Kelvin, symbol K, is the SI unit of thermodynamic temperature. It is defined by taking the fixed numerical value of Boltzman constant k to be $1.380\,649\ \times 10^{-23}$ when expressed in the unit of J K^{-1}, which is equal to kg m^2 s^{-2} K^{-1}, where the kilogram, meter and second are defined in terms of Planck constant h, speed of light c and Cesium hyperfine transition frequency $\Delta\nu_{Cs}$.

The assigned numerical value of the Boltzman constant is determined with three fundamentally different methods: Acoustic Gas Thermometry (AGT), Dielectric Constant Gas Thermometry (DCGT) and Johnson Noise Thermometry (JNT) (See [20] and references therein).

3.1 Primary Realization of Electron Temperature

For an ideal gas of monoatomic non-interacting particles at the thermodynamic equilibrium, the mean kinetic energy is given by $\langle E_k \rangle = 3/2\, kT$. Consequently, the Boltzman constant is a conversion factor between the mean energy and the thermodynamic temperature of the system.

A vacuum plasma system provides us with such an environment thereby allowing the primary realization of Kelvin via Langmuir probe. As mentioned above, EEDF can be deduced from the *I-V* characteristics of the Langmuir probe. Then, the average kinetic energy of electrons obtained from EEDF together with the fixed value of the Boltzman constant yields the electron temperature. Therefore, a non-equilibrium vacuum-plasma and a Langmuir probe allow the primary realization of electron temperature.

The optical emission spectroscopy may be used for the validation of the Langmuir probe results. It is not possible to think of it as a primary standard, because the electron is determined under the assumption of Maxwellian EEDF. In fact, vacuum plasma with a Langmuir probe could be used effectively for the calibration of optical emission spectrometers. We aim to render optical emission spectrometer calibration service to its users.

Below a comparison of two systems in determining the electron temperature is given for the Argon gas. Figures 2 and 3 are given for illustrative purposes of Langmuir probe and optical emission spectrometer measurements at P = 60 mTorr and V = 588.4 V. The spectral lines were identified using the NIST Atomic data table [21].

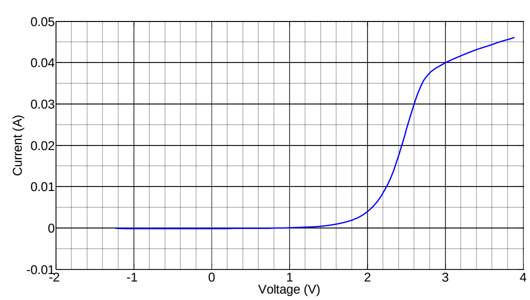

Figure 2. The I-V curve of Langmuir Probe.

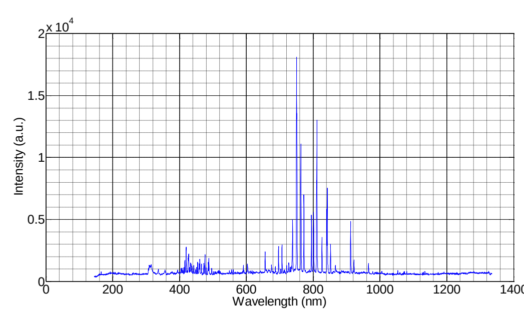

Figure 3. The emission spectra of Argon.

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Acta Materialia Turcica

Volume 5, September 2021, Pages 24-29

In determining the electron temperature from optical emission spectrometer, corona model is assumed and spectral line ratio analysis is performed [21]. The strong emission lines with wavelengths of 750.4 nm and 425.9 nm are chosen since the excitation cross section of these states from ground state is relatively high compared to the excitation cross section of metastable states. The ratio of emission intensities reads

$$\frac{I_{Ar(2p_1)}\, n_{Ar(2p_1)}}{I_{Ar(3p_1)}\, n_{Ar(3p_1)}} = \frac{Q_{ecx}^{Ar(2p_1)}}{Q_{ecx}^{Ar(3p_1)}} \tag{1}$$

where I is the emission intensity, n is the population density, Q_{exc} is the excitation rate coefficient from the ground state which is proportional to EEDF. For a Maxwellian EEDF, the ratio on the right hand side is approximated as

$$\frac{Q_{ecx}^{Ar(2p_1)}}{Q_{ecx}^{Ar(3p_1)}} \sim \frac{Q_0^{Ar(2p_1)}\,\exp\left(-E_a^{Ar(2p_1)}/T_e\right)}{Q_0^{Ar(3p_1)}\,\exp\left(-E_a^{Ar(3p_1)}/T_e\right)} \tag{1}$$

where Q_0 is a pre-exponential factor, E_a is the activation energy. The electron temperatures obtained by commercial Langmuir probe and by spectral line ratio analysis from optical emission spectrometer are given in Fig. 4. The results are consistent with each other within the uncertainty of the Langmuir probe. There is an ongoing work for the verification of the Langmuir probe results at different temperatures by the optical emission spectrometer. Once this is achieved, vacuum plasma with Langmuir probe could be used for calibrating the optical emission spectrometer.

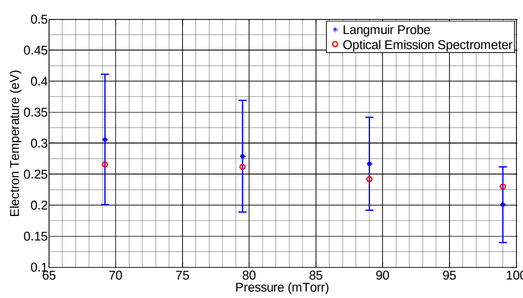

Figure 4. The comparative analysis of electron temperature by Langmuir probe and optical emission spectroscopy.

4. CONCLUSION

The plasma laboratory infrastructure in TÜBİTAK UME has been constructed to permit a variety of non-equilibrium plasma applications. Based on the environment where plasma is generated, the ongoing and planned studies are classified as plasma in vacuum and plasma in liquid. Electron temperature primary standard, UME-made Langmuir probe and hot-cathode magnetron reactive sputtering are among the studies of vacuum plasma while water purification is a study of plasma in liquid. The focus of the paper is mainly on electron temperature determination at primary level as for the rest of the studies the infrastructure is still under construction. Now that the SI unit of thermodynamic temperature is determined via Boltzman constant, plasma in vacuum could be used for the primary realization of the electron temperature. The two instruments available in our infrastructure for electron temperature determination are the commercial Langmuir probe and the optical emission spectrometer. As a Maxwellian EEDF is assumed in the determination of the electron temperature the optical emission spectrometer cannot be used as primary realization of electron temperature. Instead, the electron temperature obtained by Langmuir probe in vacuum plasma is verified by the optical emission spectrometer within the uncertainty of Langmuir probe. We present the preliminary results at fixed pressure and voltage values. The results are consistent within the uncertainty of Langmuir probe. The extended study of a range of temperatures is left as a future study. There are ongoing studies on building a UME-made Langmuir Probe. Once this is achieved, it will be used for calibrating the optical emission spectrometers.

5. ACKNOWLEDGMENT

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