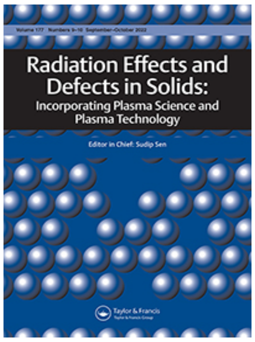

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ISSN: (Print) (Online) Journal homepage: https://www.tandfonline.com/loi/grad20

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https://doi.org/10.1080/10420150.2023.2195653

Obtaining plasma parameters by Langmuir probes and optical emission spectroscopy in low-pressure DC plasma

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ABSTRACT

The plasma environment in low-pressure systems allows coating different materials such as glasses, textiles, metals, etc. at the atomic level. Although vacuum coating is one of the best methods for thin film coating, the coating quality is closely affected by the spatial and temporal non-uniformities inside the reactor. Therefore it is quite important to determine the plasma properties within the chamber. The determination of plasma parameters such as electron temperature, electron excitation temperature, electron and ion densities can be realized using Langmuir probe systems, and by Optical Emission Spectroscopy (OES) by using Boltzmann plot method. Knowing such parameters can provide important information about specific reaction mechanisms, as well as it provides invaluable information that can increase reproducibility in various discharge process control in thin film coating. In this study, a homemade single Langmuir probe (s-LP) and a multiple Langmuir probe system (m-LP) were used to calculate electron temperature, electron density, saturation current, and plasma potential of the plasma at different pressures and at different voltages. One of the objectives of this work was to evaluate the electron temperature and ion density inside the plasma during a coating process. The second objective was to find the spatial change of electron temperature in the axial direction between cathode and anode by m-LP system including 8 independent tips. The last objective was to measure the axial plasma electron temperature distribution inside the vacuum chamber by using OES.

ARTICLE HISTORY
Received 19 November 2022
Accepted 17 March 2023

KEYWORDS
Langmuir probe; low pressure DC plasma parameters; optical emission spectroscopy; multiple Langmuir probe array

1. Introduction

Plasma applications can be used in surface treatments such as surface activation, surface cleaning, sterilization, coating processes, etching, and thin film deposition, etc. Understanding the physical and chemical processes in a plasma can be used in the reproducibility and optimization of such applications. Plasma diagnostics are thus rather important because it helps to understand the properties of the plasma filling in the vacuum chamber and the deposition process. Magnetron sputtering (MS) deposition is one of the standard methods in fabricating high-quality thin films on surfaces. The plasma parameters in the MS system

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affect the electrical, optical, and other properties of the deposited thin films (1). Since the plasma parameters play an important role in the efficiency and quality of the processes such as surface cleaning, surface activation, etching, sterilization, and film deposition, understanding the characteristics of these discharges are of great importance to obtaining the best coating properties. This close relationship between the plasma parameters and the performance of surface treatment makes analyzing the plasma parameters mandatory (2). The electron temperature is directly related to the kinetic energy of electron motion and the deposited film quality increases when higher-energy electrons are used in the coating process (3). Since the electrons are responsible for processes such as elastic and inelastic collisions taking place in plasma volume, it is critical to determine their temperatures and their spatial variation, which affect physical and chemical processes. Some of these plasma processes in which electrons play a significant role can be summarized as the excitation of atoms to higher energetic levels, dissociation of molecules in reactive sputtering processes, and creation of negative ions by attachment processes, etc (4). It is also possible to control the elemental composition of the deposited particles, which improves the deposition (5). If the inelastic collisions of electrons are lowered by reducing the pressure, the electron temperature will increase leading to the ability to tune electron temperature. This tuning will also affect the microstructure of thin film coating layers. Additionally, using a different inert gas will result in the change of the inelastic collisions again since the ionization cross-section of the discharge gas will be different so that the probability of inelastic collisions of electrons will also be different (6). It is important to note that if the process gas is chosen to be Ne instead of Ar (or Kr) the electron temperatures can be increased by up to a factor of 3. The conditions at the substrate will also be affected by incoming electrons, ions, etc. This incoming flux of energetic plasma species on the substrate surface plays an important role in surface processes such as adsorption and diffusion (7). During the last century, electrical probes have been extensively used to measure plasma parameters. In the beginning, probes were only used to measure the voltage distribution in gas discharges (8). Then, Langmuir and co-workers (9) developed the first theoretical and experimental foundations of electrical probes, which are now called Langmuir probes (LPs). They demonstrated that the probes could be used to determine many plasma parameters, such as plasma potential, electron density, ion density, and electron energy distribution function. Even though the parameters of plasma are measured by a variety of diagnostic techniques the electrostatic LP is one of the most widely used, versatile, and powerful plasma diagnostics tools. Additionally, they are the least expensive, simple to design, adaptable, easy to handle and use as long as their structures are carefully designed by taking the plasma size into consideration. For plasma diagnostics, single, and multi-needle designs were developed (10–14). LP measuring technique is based on applying a DC bias potential difference and measuring the current passing through the thin wire inserted into the plasma. LP method can be implemented only in the absence of an external magnetic field and the parameters are determined only at the immediate vicinity of the probe.

In this work, the electron temperature, electron density, and Debye length of Ar DC plasma were obtained using a single Langmuir probe (s-LP) and multiple Langmuir probe (m-LP) systems.

One additional technique used for plasma diagnostics is optical emission spectroscopy OES which is used to monitor plasma parameters by analyzing the emission lines of ionized gas (15). Due to its simple implementation, non-invasive nature and accuracy, OES is one of the attractive methods (16) to determine bulk properties of the plasma. Particularly, during plasma deposition, the excited atomic species of utilized gases can be monitored using OES (17) without any disturbance. Additionally, OES could play a crucial role in addressing the problems of RF coupling and plasma formation (18). Since OES is one of the most efficient techniques to measure the thermodynamic properties, it is also used in the experimental investigation of free-stream plasma flow at atmospheric pressures (19). In this study, by using the OES spectra, the excitation temperature of the plasma was obtained using the Boltzmann plot method (BPM).

This article is organized in the following way. The first part describes the plasma system, the second part describes the experimental setup, materials and methods used in this investigation. The working principles of the magnetron sputtering system, s-LP, m-LP, and OES studies were mentioned and described in detail. In the third part, how the results obtained from experiments were used in calculating the plasma parameters. These parameters are Debye length, electron temperatures at a single point and then at eight different points by using current–potential graphs, and excitation temperatures with the help of spectroscopic Boltzmann plot under different plasma pressures and potential differences.

2. Experimental setup

One of the widely used plasma thin-film coatings is the MS system but the understanding of the underlying physics that controls such plasma behavior is relatively little (20). In this study, a DC MS system was used to analyze the plasma properties inside the vacuum chamber. The working principle of this method is treating the energetic plasma ions created by external electric field between electrodes. To create an inert background plasma, a precursor gas was inserted into the deposition chamber to pressure from 10 to 100 mTorr. Argon gas was used because of its mass compatibility with most of the engineering materials and its relatively low cost.

A DC voltage of 250–550 V is applied between the anode (holding surface to be coated) and cathode (holding the sputtering target) to partially ionize Ar atoms introduced into the chamber. When this potential reaches the gas breakdown voltage, the ionized Ar plasma initiates sputtering on the Cu target surface. The process leads to the ejection of target atoms from the target surface because of circulating Ar ion bombardment (circular $\vec{E} \times \vec{B}$ force) to create the growth of the Cu thin film on the substrate surfaces. The atoms sputtered from the target material move in a volume from the cathode to the anode, performing thin coating on the surface attached to the anode. The MS vacuum system used in this study and coated glass, metal, and textile surfaces are shown in Figure 1. During such a coating process, the Langmuir probes that are placed in this volume are activated and the measurements were performed to obtain plasma properties such as electron and ion temperatures, plasma ion density, and plasma potential distribution.

Analyzing MS discharges is not complete because of the difficulty arising from applied strong magnetic and electric fields acting on the charged particles near the cathode surface holding the target (21). The directions of electric, magnetic fields, and the force$\vec{E} \times \vec{B}$ produced above the cylindrical cathode surface are shown in Figure 2. Here, the electric field produced from the external power supply faces into the page and the magnetic field obtained from Nd magnets on the cathode is directed from N to S radially. In low-temperature deposition processes, it is desired to have high-temperature electrons to get

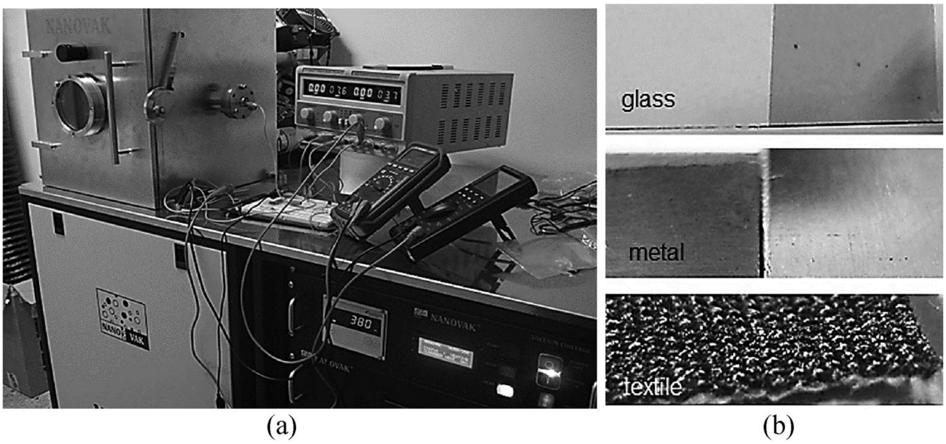


Figure 1. (a) Magnetron sputtering system used in this research and, (b) the coated glass, metal, and textile surfaces.

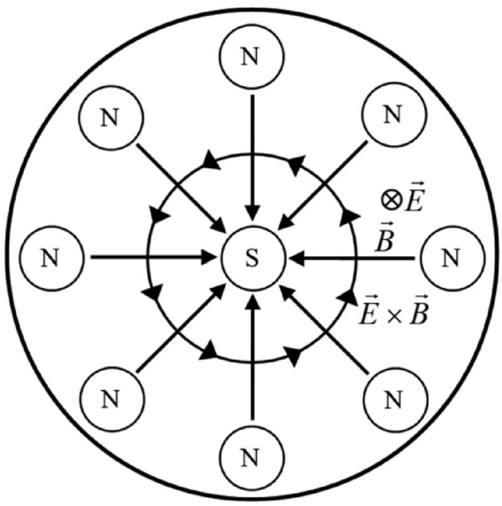


Figure 2. The schematic top view of cathode in the magnetron system. N and S are cylindrical Nd magnets.

high-quality films (*22*); this is provided by this $\vec{E} \times \vec{B}$ force. Note that the typical magnetic field values near the sputtering head is in the order of 100–300 mT.

2.1. Single langmuir probe experiments

In these study, the Cu thin film coating experiment was carried out inside at stainless steel cubic vacuum chamber (Figure 1) with side lengths of 400 mm (NANOVAK NVSS350, Turkey). The plasma was created by injecting Ar gas at 100 mTorr pressure by a sensitive flow meter into the chamber and applying a potential difference between the cathode and anode. During the coating process, single probe measurements were performed using a

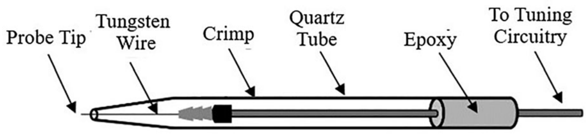


Figure 3. The schematics of a homemade Langmuir probe.

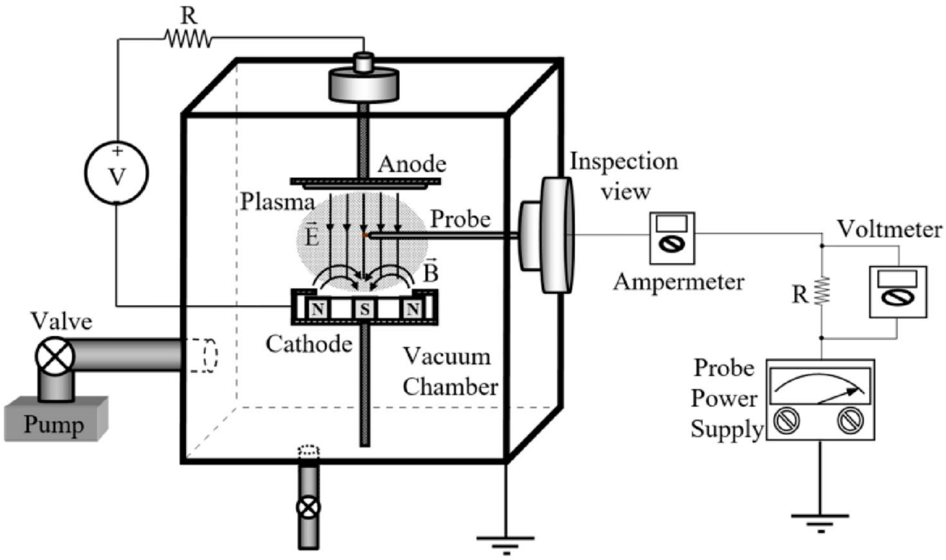


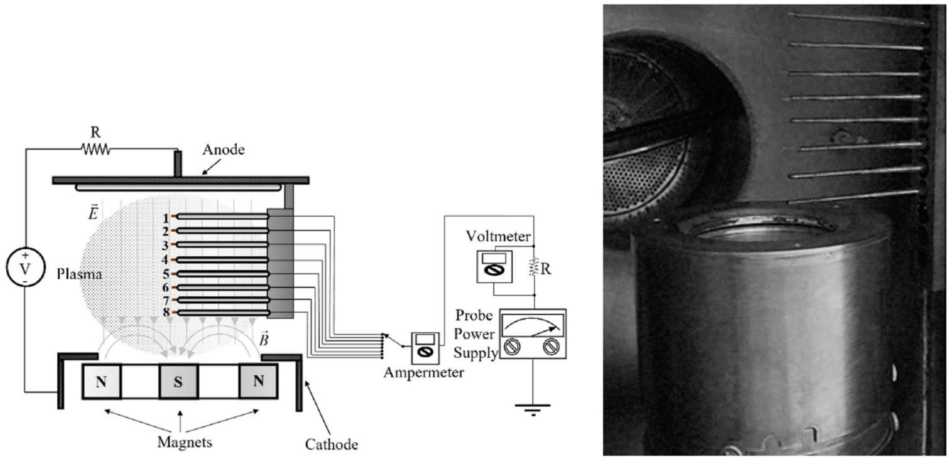
Figure 4. The block diagram of the experimental setup for s-LP experiments.

homemade Langmuir probe (see Figure 3) which was made of a quartz tube carrying tungsten wire of 0.1 mm in diameter along the centerline. The cylindrical probe tip exposed to the plasma was allowed to collect current from the plasma through its surface area of 0. 64 mm^2.

This Langmuir probe was positioned at the midpoint of the DC glow discharge volume between the cathode and anode (see Figure 4) to determine the plasma parameters around the location of the probe tip. The bias voltage of the probe was changed between −50 V and +40 V with 10 V steps by using an adjustable DC power supply (with 0.1% ripple accuracy) and the currents collected by the probe were measured by a sensitive multimeter. The probe current measurements were established at three different chamber pressures of 9, 20, and 60 mTorr respectively, which were maintained by using a turbo pump backed by a mechanical pump.

2.2. Multiple langmuir probe experiments

The spatial changes in plasma parameters are also important for plasma diagnostics since this information leads to the ion flux measurements, which are important for the physical properties of the thin film coating process. In this part of the experiments, a homemade multiple Langmuir probe assembly (see Figure 5) was used to measure spatial variation of



(a) (b)

Figure 5. (a) The block diagram of multiple probe apparatus, (b) its actual view above the magnetron cathode.

plasma properties. The probe array was made of 8 cylindrical Langmuir probes described earlier and was placed 8 mm apart along the centerline between the cathode and anode (separated by 110 mm) and the first probe was placed 25 mm away from the anode. For this experiment, the chamber pressure was kept constant at 20 mTorr. By applying the same bias voltage (varying from −40 to +40 V) to all these probes, respectively, and collecting probe currents, their independent I-V characteristics were obtained.

2.3. Measuring excitation temperature BY optical emission spectroscopy (OES)

Langmuir probe measurements in plasma provide local information around the tip location. To determine averaged bulk information over the volume of plasma, spectroscopic techniques are widely used (*23*). In OES, the light emitted by the Ar plasma is transferred into the spectroscopic grating and CCD camera system to obtain the relative light intensity as a function of the wavelength. This useful diagnostic tool, which can be applied inside the vacuum chamber or outside through a quartz window, has a non-invasive nature being able to detect both neutral and ionic species (*24*).

When the electron density is higher than the critical value given by the Griem criterion, the plasma is said to be optically thin and it obeys the local thermodynamic equilibrium (LTE) condition. Thus, the excitation temperature (T_{exc}) measured by OES is close to electron temperature (T_e) (*25*). The excitation temperature of the plasma atoms can be obtained from the slope of the Boltzmann plot by using the two-line intensity method. The single temperature of the plasma in LTE characterizes all internal energy modes such as vibrational, rotational, and electronics (*26*). Emission spectroscopy using the Boltzmann plot method is considered to be one of the best methods since the plasma is not disturbed during data collection. It must be noted that the shape and width of the emitted spectral lines are influenced by the collisional processes in plasma (*27*).

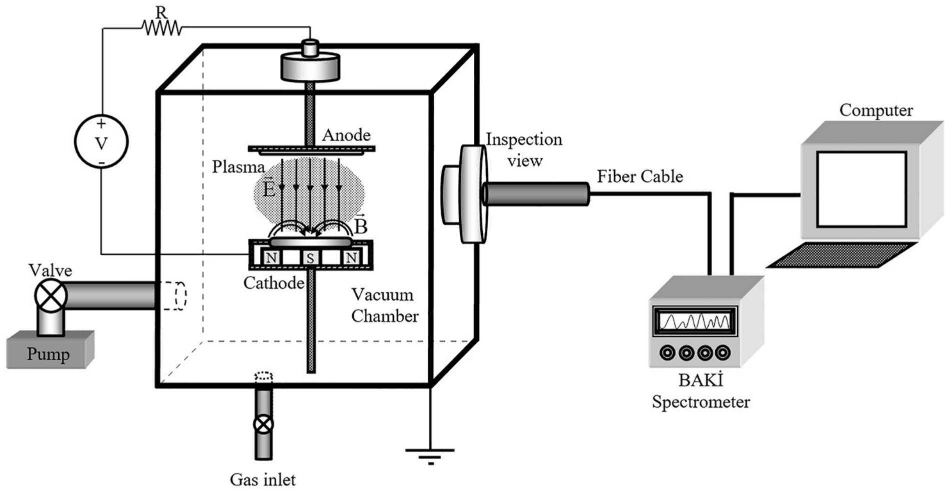


Figure 6. The diagram of OES system.

The quartz window mounted on one face of the vacuum chamber used in this study allowed the observation of the plasma radiation spectra from outside. The data were collected by a fiber cable, standing just outside this window, attached to the spectrometer (*28*). In this relatively low-temperature and low pressure plasma system, the dominating mechanism for the excitations is the collisions between neutral Ar atoms and sufficiently energetic electrons. Plasma photons emitted from these interactions were collected by BAKI emission spectrometer (Beam R&D, Co., Turkey) as shown in Figure 6, connected to the vacuum facility used in this research. This spectrometer included a grating of 1200 lines/mm, 1.6 nm resolution and 1–10 ms integration time. The fiber cable just outside the quartz window was fixed at a distance of 22 cm from the midpoint of the plasma within the chamber. Since the photons that fall into the solid angle of the fiber tip surface are collected, this OES measured averaged bulk emissions falling into this solid angle.

3. Results and discussion

3.1. The electron temperature at single point in plasma

By using the procedures explained earlier several single probe measurements were performed to collect I-V data. A sample I-V graph of a single probe measurement is presented in Figure 7.

The electron temperature of plasma can be found using the following equation, which gives probe current, I_{probe} due to the collected electrons by the probe tip surface area:

$$\ln(I_{probe} - I_{sat}) = \left(\frac{e}{kT_e}\right)(V - V_f) + C \quad (1)$$

where V is the probe voltage, I_{sat} is the electron saturation current, V_f is the floating potential of the plasma, k is the Boltzmann constant, e is the electronic charge, and C is a constant. If Eq. (1) is fitted to a straight line (using 4–5 data points in the exponential region shown in Figure 7), its slope gives e/kT_e from which the electron temperature, T_e can be obtained

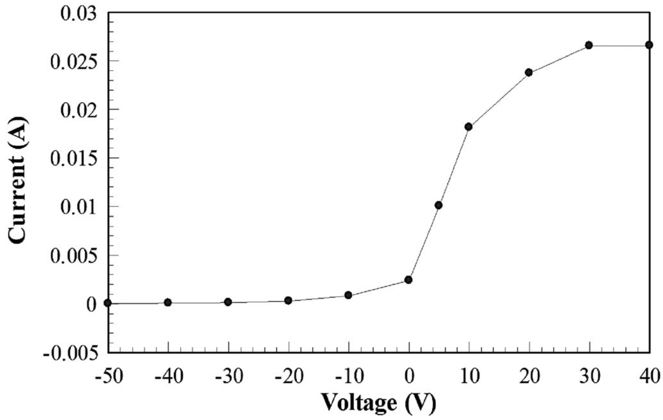


Figure 7. The current-voltage characteristics of obtained by s-LP system.

(*29*). Note that the line obtained from this equation should exclude the points at the saturation region, which occur at sufficiently high voltages. At relatively high voltages, excess amount of electron attraction to the probe tip causes a glow that cleans the probe surface, as shown in Figure 8. It is a known fact that as plasma pressure is increased, the electron density in the plasma should also increase due to thermodynamics and this process leads to more collisions and thus higher electron temperatures (*30*). For pressures 9, 20, and to 60 mTorr in magnetron sputtering Ar plasma coating of Cu, it was found that electron temperatures changed to 5.6, 6.8, and 14.3 eV respectively, as presented in Table 1. It is not difficult to show that the electron temperature is linearly dependent on the chamber pressure in the single probe measurements. Although the number of gas atoms in the chamber increases with pressure ($P = nkT$ where n is number density), the number of ionized electrons reduces since the energy provided by the constant electric field is shared by more gas atoms. This was presented as the reduction of electron density by increasing pressure in Table 1. If a positively charged metal wire is inserted in plasma, the electrons are attracted and a plasma sheath region following the shape of the charged metal is formed around it. The size of this region is approximately obtained from Debye length (λ_D) given by

$$\lambda_D = \left(\frac{kT_e}{4\pi N_i^2} \right)^{1/2} \tag{2}$$

where, T_e is the electron temperature, and N_i is the number of ions at the ion saturation region where voltages are negative. These values can be readily obtained from I-V data and calculated electron temperatures. Debye length values for magnetron plasma at 9, 20, and 60 mTorr pressures were found to be 3.3, 4.7, and 7.8 mm, respectively, resulting in an almost linear increase with pressure. These results indicate that the probe operation is at orbital motion limited (OML) regime, where the analytical expressions for the probe current are quite simple (*31*). When the probe potential is at excess positive voltage at pressures above a limit, the number of electrons around the probe is amplified, giving rise to a very hot and glowing region. The last measurement in Table 1 (at 60 mTorr) reflects this case since the electron temperature was intentionally made very high, *i.e.* 14 eV. Figure 8

Figure 8. The Langmuir probe in the cleaning process due to excess positive potential during MS coating.

Table 1. Calculated plasma parameters.

Parameters	Pressure (mTorr)		
	9	20	60
Electron Density: n_e ($\times 10^{18}$ m^{-3})	22.1	12.9	10
Debye Length: λ_D ($\times 10^{-3}$ m)	3.3	4.7	7.8
Saturation Current: I_{sat} ($\times 10^{-5}$A)	4.9	4	3.4
Electron Temperature: T_e (eV)	5.6	6.8	14.3

shows the Langmuir probe, which has a bright glow region around the tip due to the process described above. This process is generally used for probe tip cleaning since the vacuum coating process also produces coated layers around the probe tip which must be cleaned each time before sensitive measurements.

3.2. Spatial distribution of electron temperature and plasma potential

A homemade multipleLangmuir probe (m-LP) array was developed to measure the spatial variation of plasma properties along the centerline from anode to cathode (70 mm apart) in the plasma chamber. The positions of the probes are numbered such that the one closest to the anode is the first. The same bias potentials were applied to each of these probes and their associated current values were recorded. The I-V graphs obtained from these measurements are given in Figure 9(a) for the first four probes and in Figure 9(b) for the last four. The graphs were separated since there are many orders of magnitude differences between the two groups of probes.

Electron temperatures for all these probes along the centerline were calculated using the same procedure explained earlier and plotted in Figure 10. As seen from this figure, the electron temperature of the MS plasma increases from anode to cathode and saturates near the cathode region. This is an expected result since there is an effective $\vec{E} \times \vec{B}$ trap in the region near the cathode where the most intensive ionization occurs and the density of the plasma is higher than that of bulk plasma. By moving away from this intensive ionization region

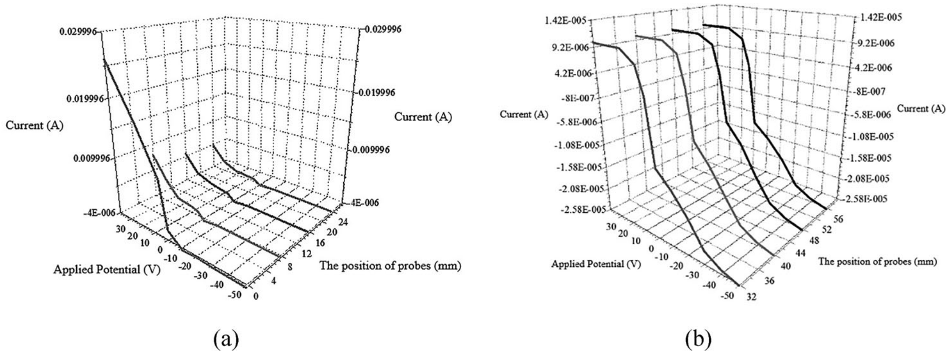


Figure 9. (a) The current-voltage characteristics of the first four probes from anode to cathode, (b) The current-voltage characteristics of the last four probes from anode to cathode.

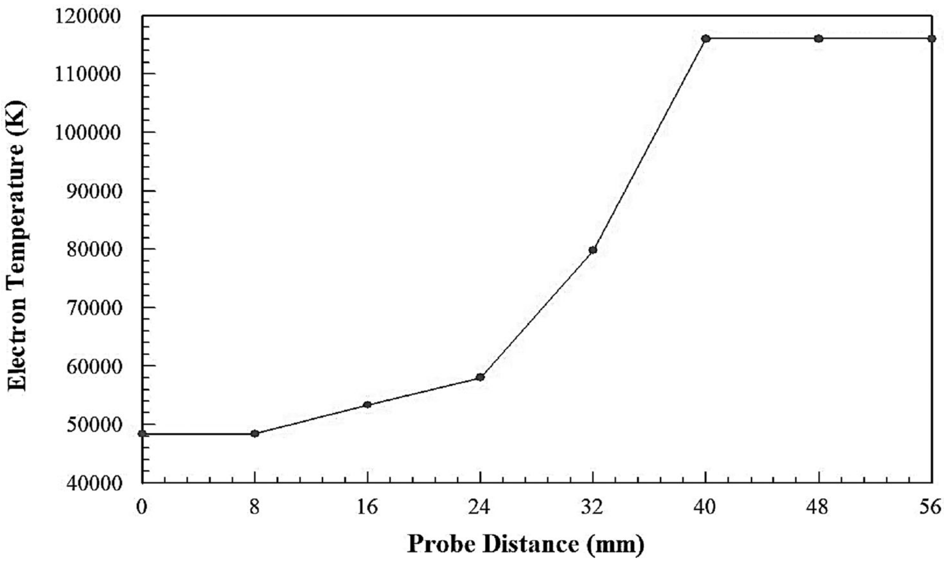


Figure 10. Spatial electron temperature distribution along centerline from anode to cathode.

toward the anode, the electron temperature decreases as it is expected (*32*). Additionally, the discharge is primarily confined in the vicinity of the cathode target, so that plasma species diffuse toward the walls and the anode lose energy and get cooler (*33*). Plasma potentials were measured from these data for each probe from the I-V points at which probe currents vanish, see Figure 11. This graph shows that plasma potential throughout plasma remains almost constant at a small negative potential (*i.e.* −0.001 V here). It is expected that this potential has a gradient near the anode and cathode (boundary layer) but this process was not observed here since the first probes near the cathode and anode were placed more than 20 mm away.

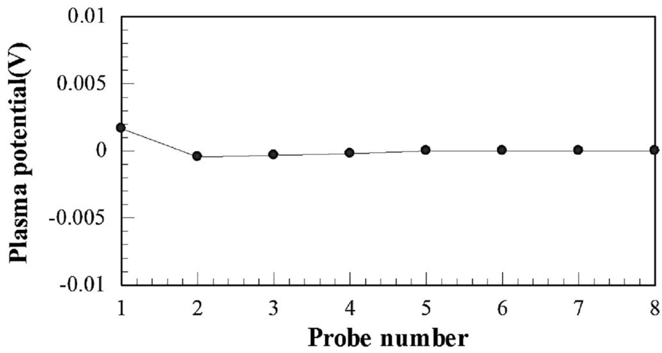


Figure 11. Plasma potential measured by each probe when $I_{probe} = 0$.

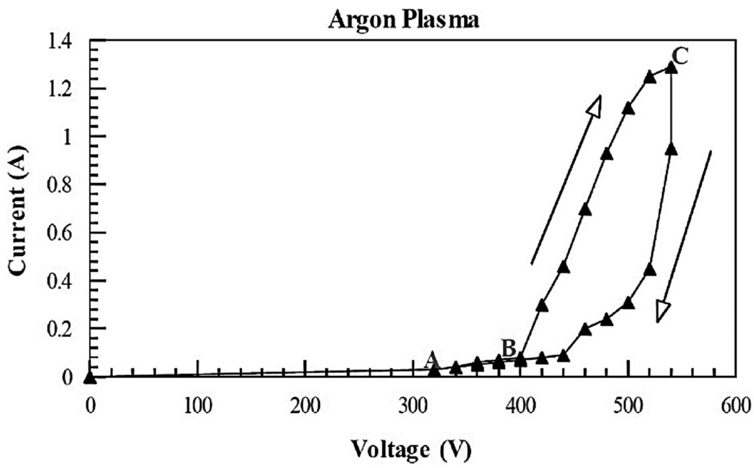


Figure 12. Hysteresis effect in I-V characteristics in the magnetron plasma.

3.3. Hysteresis effect on I-V characteristics

I-V characteristics of a plasma system give the impedance behavior of the plasma since the inverse of the slope of the I-V curve leads to resistivity. In this study, the voltage between the cathode and anode is increased slowly to initiate the ionization of Ar gas to trigger the sputtering process. Just after the discharge starts at a specific voltage, the resistivity starts changing linearly (see linear resistive region between A and B in Figure 12). As the voltage is increased further, the resistivity changes nonlinearly between points B and C, after which the saturation region starts. If the voltage is reduced just before this saturation region, the curve follows a different path of a lower resistive regime (points between C and B). This shows that cathode to anode voltage should be adjusted carefully during voltage increase or decrease to get the same sputtering yield on the target so that a constant coating rate on the sample surfaces is obtained in each case.

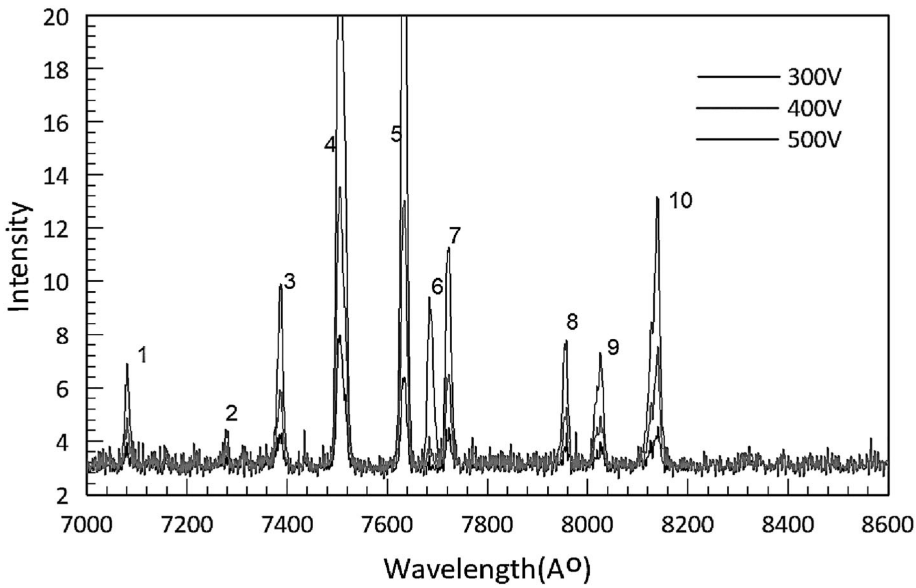


Figure 13. The emission spectra of plasma at applied different cathode-anode potentials.

3.4. Temperature obtained from OES and Boltzmann plot method

With an Ar gas pressure of 9 mTorr and cathode-anode voltage of 300 V, the plasma light intensity (I) was obtained using the data collected by the computer attached to the OES measuring system (see Figure 6). The temperatures were calculated from the slopes of $\ln(I\lambda/gA)$ versus energy difference (E_k) between atomic transitions, which was obtained from the Boltzmann plot equation given by:

$$\ln\left(\frac{I_{k_i}\lambda_{k_i}}{g_i A_{k_i}}\right) = -\left(\frac{E_{k_i}}{kT_{exc}}\right) + C \tag{3}$$

where $I_{k_i}, \lambda_{k_i}, g_i, A_{k_i}, E_{k_i}$ and T_{exc} are the atomic emission intensity, the wavelength of the corresponding transition, the statistical weight, transition probability, energy, and the excitation temperature, respectively. The energy E_k and gA values in Eq. (3) were found using atomic spectra database of NIST (*34*) by obtaining the transition wavelengths shown in Figure 13.

When the voltage between the cathode and anode is increased, it is expected that the ionization rates of Ar atoms are increased. This is exactly what is seen in Figure 13 since intensity peaks due to Ar atomic transitions get stronger as this voltage is increased from 300 to 500 Volts. By examining the intensity levels, it was found that this relationship was linear. As presented in Table 2, the wavelengths and corresponding peaks were found to be 7077, 7273, 7384, 7635, 7683, 7724, and 8151 Å for the first ionizations (ArI) as 7503, 7944, and 8018 Å for the second ionizations (ArII) 2 and their corresponding energies and log functions were obtained. These peaks show that the discharge is pure and no impurity atoms exist in Ar plasma.

Table 2. The wavelengths and corresponding data for the plasma.

Wavelength (nm)	Intensity	gA (s^{-1})	E_k (eV)	ln ($I\lambda/gA$)
727.293	3.5	5.49 E + 06	13.327	−7.68 E + 00
738.398	4.75	4.24 E + 07	13.302	−9.40 E + 01
750.386	8	4.45 E + 07	13.479	−8.91 E + 00
763.510	6.5	1.22 E + 08	13.171	−1.01 E + 01
772.420	4	3.51 E + 07	13.327	−9.34 E + 00
815.118	4.5	9.90 E + 04	14.848	−3.29 E + 00

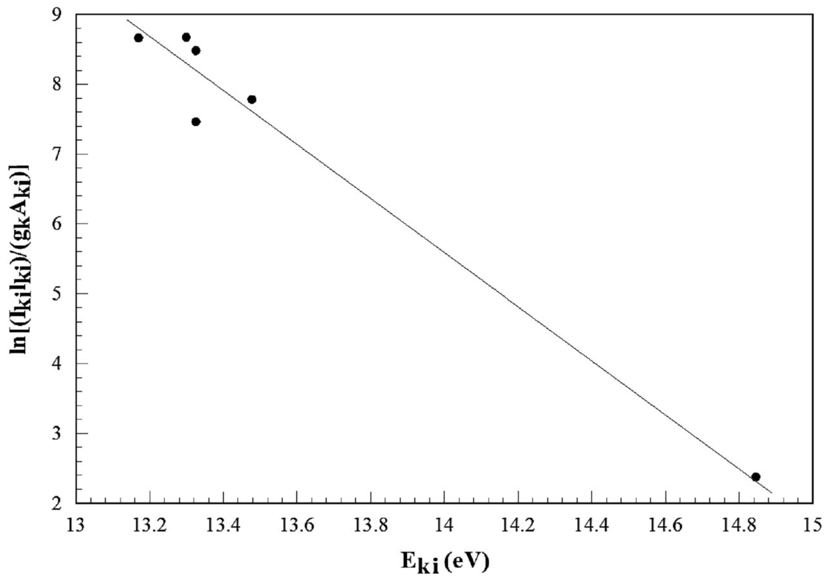


Figure 14. ln($I\lambda/gA$) versus E_k for plasma at 300 V cathode-anode potential.

Note that although the probes measure local plasma properties, OES provides the bulk properties of the plasma falling into the solid angle by the optical tip. Therefore, the averaged electron temperature was expected to be much lower than that obtained from the Langmuir probe measurements. The negative slope of the line in Figure 14 (obtained from the collected data) was found to be 3.9, which is equal to *E/kT*. Since electron temperature is inversely proportional to the slope, the bulk electron temperature in the plasma volume was found to be nearly $T = 1.3$ eV. This value is nearly one-fourth of the local electron temperature (see Table 1) that was found by a single Langmuir probe measurement at 9 mTorr pressure. Similar differences were also observed in (*35*). Since there is a significant amount of difference between the Langmuir and OES temperature measurements, it can be concluded that the plasma is not at LTE state (*36*, *37*). The information obtained from OES is not complete since it concerns only ions, although there are also neutral atoms in low-temperature plasmas (*38*) whose existence plays an important role during plasma processes. The uncertainty in the values of A_{k_i},and the selection of lines with small energy differences in the upper energy levels are also the sources of excitation temperature calculation errors by using the Boltzmann Plot method (*39*). Furthermore, the contamination of the probe tip is certainly another reason for the discrepancy in the results. When a clean probe is inserted into the MS plasma during coating, quick contamination develops and creates a resistive surface layer, which affects the results (*40*). The diversion of electron energy distribution from the Maxwellian distribution is another source for the errors in T_e calculation (*41*).

4. Conclusion

The plasma diagnostics were carried out in a DC magnetron sputtering system including Ar gas at pressures between 9 and 60 mTorr by applying a DC voltage cathode to anode from 300 to 500 V. This study included single and multiple Langmuir probes and OES during the coating process and their results were compared. It was observed that the electron temperature increases from the anode to the cathode and saturates near the cathode. The probes allow local plasma measurements, but they may disturb the plasma when a sufficient voltage is applied. In contrast, the OES measurement technique is a passive measurement from which bulk properties are obtained by observing the light emission so that plasma is not disturbed during measurements. The study showed that the number of electrons (which shows a degree of ionization) is reduced while the electron temperature is increased with increased pressure. For such pressures and voltages, it was found from the probe measurements where the electron temperatures varied between 5.5 and 11 eV while OES measurements produced smaller temperatures. The results revealed a deviation from the local temperature equilibrium state (LTE) in the magnetron sputtering plasma. It was also shown that the position of the probe is important and that the surface contamination of the probe can be eliminated by applying high values of probe bias voltage. Another important outcome of this plasma diagnostic was that varying cothode-anode voltage resulted in a hysteresis effect to which attention must be given. By using these results, the optimization and process control for enhancing the reproducibility in various discharge configurations and fabrication mechanisms of functional thin film coatings using the magnetron sputtering method can be achieved.

Disclosure statement

No potential conflict of interest was reported by the author(s).

Notes on contributors

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