

# Analysis of Langmuir Probe Characteristics for Measurement of Plasma Parameters in DC Discharge

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## Abstract

There is a growing interest in the study of plasma parameters (density of charge particles and their energies) for their application in surface plasma ion nitriding. Nitrogen plasma were investigated using the Langmuir probe for dc-glow discharge plasma at operating pressures of 0.06-0.1mbar. Plasma diagnostics was carried out using the single electric probe with tip diameter of 0.5 mm and a length of 2mm, then the I-V characteristic of the probe were plotted at pressures of 0.06-0.1mbar. Plasma parameters such as electron temperature (Te), electron density (ne), ion density (ni), plasma potential and floating potential, were duly obtained. Paschen's curve was plotted, showing the breakdown voltage of nitrogen as a function of the product of the pressure in the chamber (P=0.06 to 0.1 mbar) and the distance between the two electrodes (d=4-6 cm) p\*d. The minimum breakdown voltage  $V_{(min)}$  was determined to be 280V at a pressure of 0.09-0.1 mbar.

**Keywords:** Plasma diagnostics, DC-glow discharge, Paschen curves, Langmuir probe

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## Introduction

### Basic plasma parameters

Glow discharges are used in various applications such as plasma thin films deposition (sputtering), plasma etching and modification of surfaces in semiconductor industry and materials technology (plasma ion nitriding). To use discharges, it is essential to have information about plasma and to have control on parameters. The processes and reaction rates occurring in plasma are generally dependent on density of charge particles and their energies. In order to analyze the plasma-surface interaction, various kind of diagnostic tools have been employed. The different species present in plasma and plasma parameters, especially those involved directly in the surface treatment processes, have been studied by the electrical and optical plasma diagnostic tools such as electrostatic probe, optical emission spectroscopy (OES), Laser-induced fluorescence (LIF) and absorption spectroscopy, etc. Plasma is defined as an ionized gas containing equal amounts of electrons and positive ions with a different numbers of non-ionized molecules [1]. The types of plasma are dictated by its electron density (ne) and electron temperature (Te) [2]. When electron and ion densities are averaged on par, the resulting density is known as the plasma density. electrons are faster than ions and neutral particles, given its overall lower mass. This is also the case in high temperature environment [3]. Light emitted by plasma is due to the excited being compelled to return to their unexcited electrons (ground).

Glow-discharge plasmas are generated by striking a high voltage electrical discharge in a low pressure gas environment. This is possible using a DC, AC, or more commonly high frequency AC operating in the KHz-MHz (radio frequency) or GHz (Microwave) regime. The most common type of glow discharge is a direct-current glow discharge, which is formed via passage of current at 100 V to several kV through a gas, usually argon (or other noble gases).

Friedrich Paschen came up a law in 1889[4], setting the breakdown voltage as a function of the product of the

pressure p and the inter-electrode distance d:  $V_b = f(pd)$ . Paschen described the breakdown voltage using the equation:

$$V = \frac{a(pd)}{\ln(pd) + b} \quad \dots\dots\dots (1)$$

where V is the breakdown voltage in Volts, p is the pressure, and d is the gap distance. The constants a and b are dictated by gas composition.

A glow discharge is a type of plasma that represents a partially ionized gas made up of (nearly) equal concentrations of positive/negative charges, accompanied by large amounts of neutral species. Glow discharge plasmas are utilized in plasma applications involving low/intermediate pressure regimes, such as materials surface processing [5,6,7,8,9,10].

### Single Langmuir Probe

The properties of plasmas can be determined using electrostatic probes [11]. This technique was created by Langmuir fifty years ago, and was called Langmuir probes. An electrostatic probe is a small metallic electrode, which is more often than not a wire carefully inserted into a plasma, and connected to a power supply to bias it to negative/positive voltages when needed. The probe collects current information, which is used to discern the condition of the plasma it is connected to. The electrostatic Langmuir probes are represents a basic plasma physicists' tool for determining electron densities, temperatures, and energy distributions.

The single probe current-voltage characteristics is shown in the case of plasma lacking magnetic fields in Fig. (1). The I-V define currents based on the applied probe voltage, and the I-V plot is divided into 3 distinct regions:- ion saturation, transition, and electron saturation region [12, 13, 14].

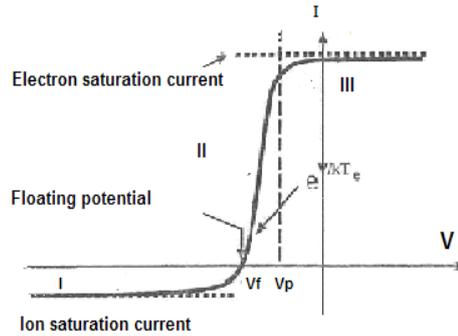


Fig. 1: I-V characteristics of single Langmuire probe.

Some of the assumptions made in this case is that the probe's current does not interfere with the plasmas' equilibrium, the probe's diameter is lower than that of the electrons' mid path, and that the electrons are within thermal equilibrium at a temperature,  $T_e$ , a Maxwellian kinetic energy distribution.

Region I: When  $V$  which is the probe's potential, makes a negative bias, electrons are rejected and only the ions are collected. The ion's current that passes via area  $A$  in the plasma is linked to both ion density and velocity:

$$J_p = \frac{I_p}{A_p} = \frac{n_i \cdot e \cdot v_{th}}{4} = \frac{n_i \cdot e}{4} \left( \frac{2kT}{m_i} \right)^{1/2} \quad \dots\dots\dots (2)$$

where  $I_p$  is the probe current (ion current), Amp.  $J_p$  is the probe current density (ion current density), Amp/cm<sup>2</sup>.  $A_p$  is the surface area of the probe.  $m_i$  is the ion mass,  $1.67 \times 10^{-27}$ kg for proton.  $n_i$  is the ion density no./cm<sup>3</sup>.

$$v_{th} = \left( \frac{2kT}{m_i} \right)^{1/2} = \text{mean kinetic velocity, m/sec.}$$

Increasing the probe's negative bias repel the electrons, which means that the current comes purely from ion.

Region II: Rendering the value of  $V$  less negative will compel the probe to collect both ions and electrons (high thermal energy). As  $V$  is made more positive, the collected ions and electrons are expected to cancel (or balance) each other out. This probe-plasma potential,  $V_f$  is called the floating potential. In the event of thermalized plasma, this voltage is  $1/2kT$  (expressed in eV). When  $V \gg V_f$  electron current increase.

The resulting current is exponentially related to  $V$ . However, it saturates at a plasma space potential value ( $\phi_p$ ), caused by the space charge limitation during current collection.

In region II, the electron current are defined by:

$$J_e = \frac{I_e}{A_p} = \frac{n_e \cdot e \cdot v_{th}}{4} e^{\frac{-eV}{k_B T_e}} \quad \dots\dots\dots (3)$$

$$I_e = \frac{A \cdot n_e \cdot e \cdot v_{th}}{4} e^{\frac{-eV}{k_B T_e}} \quad \dots\dots\dots (4)$$

$$\frac{dI_e}{dV} = \frac{-e}{k_B T_e} \quad \dots\dots\dots (5)$$

, where  $T_e$  in (K°).

The electron temperature can be calculated directly from the I-V characteristic plot of the probe.

The slope of the plot yields the electrons' temperature:

$$\frac{dI_e}{dV} = \frac{1}{T_e} \quad \dots\dots\dots (6)$$

The methods of calculating electron density are detailed in ref. (3, 12, and 16):

In region III, when  $V$  is positively biased, the probe collect the electrons and repelled the ions (completely). The current (from electrons) gets to a point where it is constant. This current is called the electron-saturation current  $I_s$ , and the electron density can be calculated:

$$I_s = \frac{n_e e A_p}{4} \left( \frac{2k T_e}{m_e} \right)^{1/2} \quad \dots\dots\dots (7)$$

**Experimental Setup (Reactor and Diagnostics)**

A home-made system was used in this work. It consist of a low-pressure gas discharge unit, which is made up of an evacuated chamber, a target (cathode), and a stainless steel anode disk. The cathode is made to face the anode, which results in an electrical field that allows gas discharge. Fig.(2), the schematic of the plasma chamber, the electrodes and DC-power supply of 4 kV. The bottom of the cathode electrode is shielded by an insulator disk (ceramic/ thermal teflon), while the top is shielded by the cathode space assembly, which included a ceramic insulator and stainless steel holder. The diameter of the top electrode is 14.5 cm, while the target electrode 7.5 cm. There is a gap distance of, 4 - 6 cm between the electrodes. The high voltage power supply operates at 300-2000 V. The voltage-current characteristics are shown in Fig.(3). The current-limiting resistor allows the current to be independently controlled.

This project utilized a custom-build Langmuir probe to characterize the plasma column of glow discharge in gases (N<sub>2</sub>) and reactive precursor channeled into the plasma chamber. The probe head is a 2 mm cylindrical fine metal wire with a diameter of 0.5 mm. The probe is coated by a protective ceramic coating, and indented

from the side of the chamber. It emits in a radial pattern from the edge to the center of the cathode. On top of the main power supply, which produces the glow discharge, a smaller auxiliary power supply links the anode and probe to the plasma chamber. The probe's current is determined as a function of the probe - anode voltage, via the variation of the probe's potential with respect to the anode potential in steps of 5-10 V. The generated I-V plot allows us to determine the electron density, electron temperature, and electron energy.

### Experimental Results

Paschen curve breakdown voltage is a function of both the working gas pressure and inter electrode spacing. A common Paschen curve for parallel electrodes of gas (nitrogen) can be determined by calculating the breakdown voltage using two planar stainless steel electrodes. The results of the gas pressures and distances are shown in Fig. (4). The trends in the shapes of the curves are examined and explained in the context of the electrochemical properties of the gas. In order to determine the breakdown voltage for a given pressure,  $p$ , and electrode spacing,  $d$ , the voltage applied to the electrodes was manually increased until a clear increase of current was observed on the ammeter, which indicate that the circuits had been closed due to the formation of plasma. Despite the voltage drop across the ballast resistor, it remains proportional to the current. Thus, the applied voltage for which the plasma was formed was reported as the breakdown voltage. This procedure was for each distance and pressure, and the average breakdown voltage determined afterwards.

To the left of the plot's minimum, the breakdown voltage is inversely related to the  $pd$ . At low pressures, the electrons' mean free path was longer and collision probability smaller relative to that of high pressure, which means less collisions and increase of the breakdown voltage to increase the incidence collision's probability for the breakdown. Here, the gas is not very dense or the plates are in close proximity; thus, even if a large number of secondary electrons are emitted, there is a low probability that any of them colliding with neutral atoms during the journey from cathode to anode. As the  $pd$  increases, collisions are more likely, and the breakdown voltage is lower; thus, the Paschen plot has a negative slope in this region.

When the  $pd$  exceeds the plots minimum at high pressure, the breakdown voltage becomes proportional to pressure (linear). This is when the electrons are constantly colliding, and breakdown conditions becomes almost equal to that of the energy gained from the electrons' intra-collision(s) [15]. The electron-neutral collision frequency and voltage required are proportional to the  $pd$ , which is represented by the positive slope of the Paschen plot during large  $pd$ s [15]. Nitrogen plasma produced using a glow discharge source can be characterized using a Langmuir probe. The I-V characteristics are shown in Fig. (5). Electron temperature can be determined by plotting the logarithm of the corrected probe's current against the probe's voltage in the electron retardation region. The slope of  $\ln I_e$  vs  $V$  plot, in its linear region, can be used to derive electrons' temperature. Once this is done, the floating potential can be calculated ( $I_a = 0$ ). Fig. (5) confirms the presence of two-electron population with electron temperatures. The variation of the electron temperature,  $T_e$ , and density,  $n_e$ , in nitrogen gas for different values of discharge current,  $I_a$ , as a function of gas working pressure is shown in Fig. (6). The common trends of the plots are that electron temperature tends to exponentially increase as pressure decreases. The pressure increase the mean free path, which also means that the energy acquired by the electrons

decreases, attributed to the reduction of the ionization process. It was also found that the electron temperature is inversely related to density and discharge current. The collision loss factor is proportional to the discharge current, which is due to the cumulative ionization effect enhancing the inelastic collision loss via lower energy electrons [3,16]. This result is good agreement with A. Qayyum et al (2006) studies, were pulsed plasma generated by electric power source. It is found that with increasing filling pressure electron temperature and density decreases. At higher pressures, mean free path is not large enough to accelerate free electrons. Because collisions among plasma species and electrons increase causing increase in species temperature by lowering electron temperature and hence high energy tail of electron energy distribution is quenched-, suggesting that number of energetic electrons at lower pressure is reduced [17].

The floating potential ( $V_f$ ) and plasma potential ( $V_p$ ) of  $N_2$  discharges can be calculated from the Langmuir probe I-V plot. The probe bias ( $V_p - V_f$ ) for  $I = I_e + I_i = 0$  is the probe's floating potential, which is illustrated in Fig.1. However, the probe's characteristics, determined at working pressures, show that the floating potential and electron temperature behaved similarly at different pressures and applied power (or cathodic potential). The floating potential obtained as a function of pressure for  $N_2$  gases discharge is shown in Fig. (8). The floating potential increased alongside the working discharge pressure. When the working Ar pressure was varied from 0.008 - 0.046 mbar, the floating potential for  $I=0$  decreased from -0.5V to 0.5V. The measurement of the  $N_2$  plasmas of the mean plasma potential are positive compared to the grounded electrode, independent of pressures Fig. (8). It should also be pointed out the  $N_2$  plasma potential decreased when the gas pressure was decreased from 0.08 to 1 mbar.

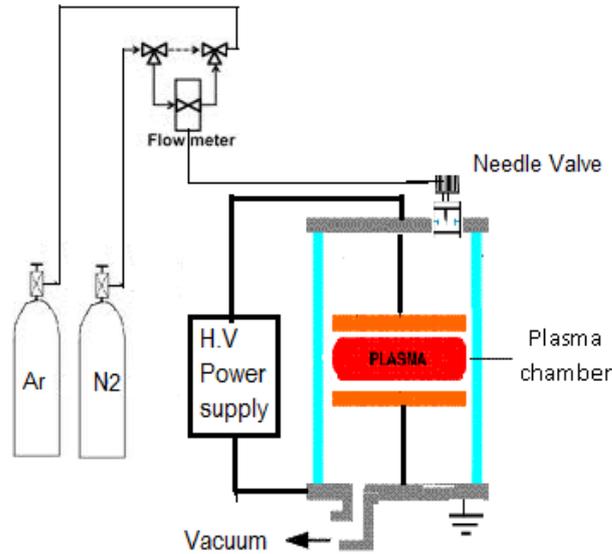
The ion density in  $N_2$  plasma can be calculated corresponding to orbital motion limit theory. The  $I_p^2$  vs  $V_B$ , plots for the ion-collection range ( $V_B < 0$ ) were obtained. The slope of the linear region of these  $I_p^2$  vs  $V_B$  curves was used to calculate the ion density. Fig.8 shows the evaluation of ion density in  $N_2$  plasma as a function of probe voltage for different working pressures. The ion density measured in the plasma bulk, with increasing pressure, the plasma density and frequency of collisions increases. Electrons suffer collisions with neutral particles, subsequently ionizing them. They lose their energy and are accelerated in electron field, gaining energy for ionization [3, 13,16]. The Langmuir probe normally determines higher temperature which may be due to the quick contamination (i.e. electrons, ions or impurities in plasma) of initially cleaned surface of probe. However, presence of this contamination layer reduces the amount of current collected from the plasma. Hence, Langmuir probe shows higher values as compared from spectroscopic measurements [18].

### Conclusion

We introduced a single electric (Langmuir) probe that is capable of measuring electron temperature, density, ion density, floating potential and plasma potential. It is also possible obtain other important coefficients characterizing the plasma system, such as electron energy distribution function (EEDF) and Ti ion temperature at specific operating conditions. From Plasma Langmuir probe analysis can be found that the electron temperature tends to increase steeply as the pressure decreases, while the electron density increases as pressure increases that is related to increase of discharge current.

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**Fig.2: Experimental set up of dc-glow discharge plasma system.**

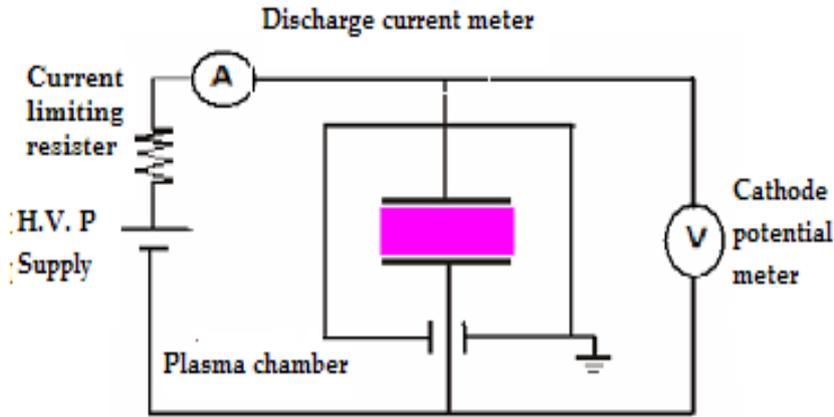


Fig.3: The dc-glow discharge plasma circuit

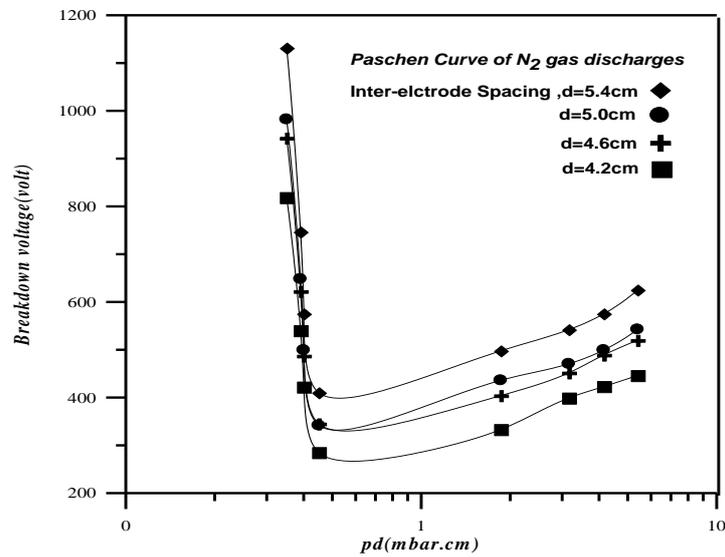


Fig. 4: The dependence of the breakdown voltage on  $pd$  parameter and the inter-electrode distance.

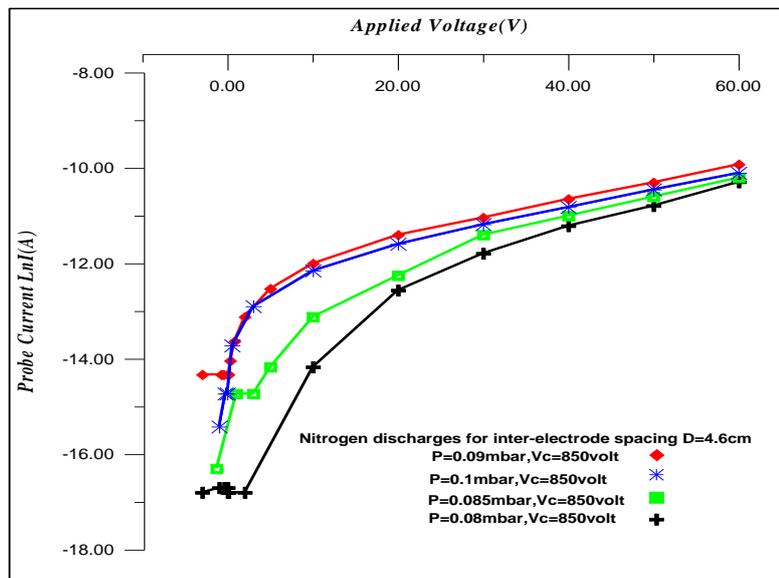


Fig. 5: Ln I-V characteristics of single Langmuire probe.

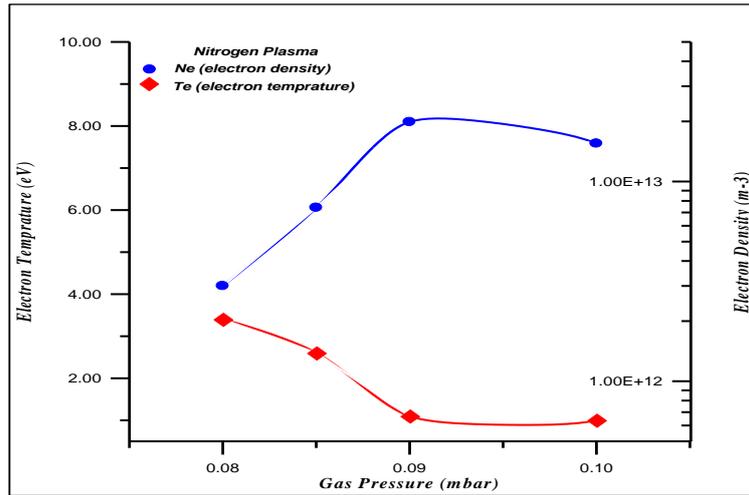


Fig.6. The variations of electron temperature, density as a function of pressure in  $N_2$  discharge plasma.

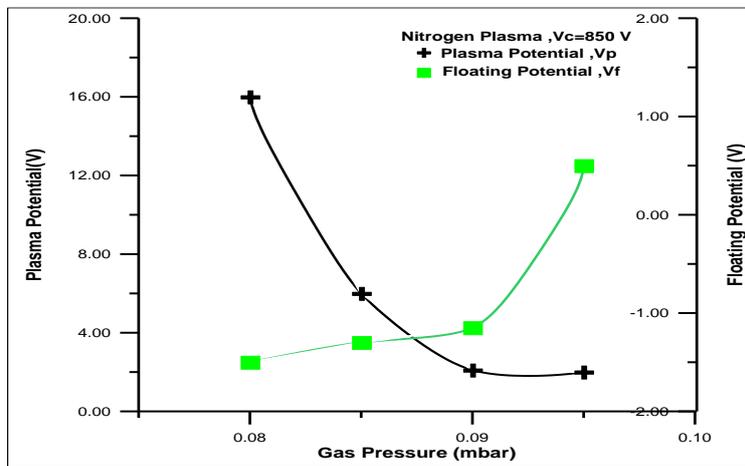


Fig.7: The variations of plasma potential and floating potential as a function of pressure in  $N_2$  discharge plasma.

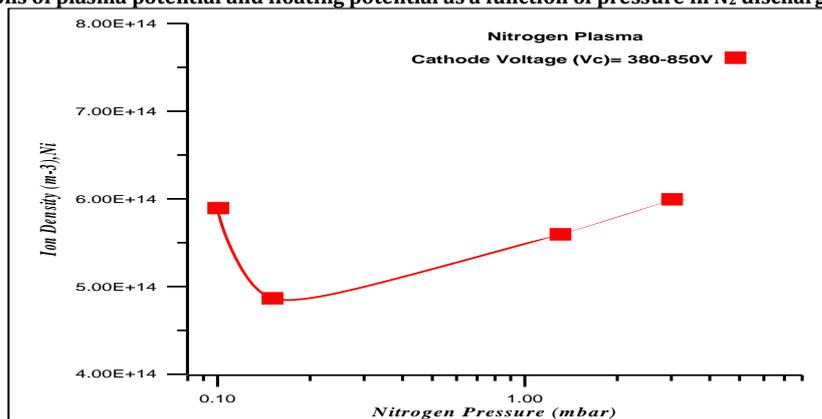


Fig. 8: The variations of ion density as a function of pressure in  $N_2$  discharge plasma.