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TITLE:

Building Langmuir probes and emissive probes for plasma potential measurements in low pressure, low temperature plasmas

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KEYWORDS:

Langmuir probes, emissive probes, electrostatic probes, low temperature plasma, presheaths, sheaths, sheath formation, Bohm's Criterion, vacuum feedthroughs

SUMMARY:

The main goal of this work is to make it easier for research groups unfamiliar with Langmuir probes and emissive probes to use them as plasma diagnostics, especially near plasma boundaries. We do this by demonstrating how to build the probes from readily available materials and supplies.

ABSTRACT:

Langmuir probes have long been used in experimental plasma physics research as the primary diagnostic for particle fluxes (i.e., electron and ion fluxes) and their local spatial concentrations, for electron temperatures, and for electrostatic plasma potential measurements, since its invention by Langmuir in the early 1920s. Emissive probes are used for measuring plasma potentials. The protocols exhibited in this work serve to demonstrate how these probes may be built for use in a vacuum chamber in which a plasma discharge may be confined and sustained. This involves vacuum techniques for building what is essentially an electrical feedthrough, one that is rotatable and translatable. Certainly, complete Langmuir probe systems may be purchased, but they can also be built by the user at considerable cost savings, and at the same time be more directly adapted to their use in a particular experiment. We describe the use of Langmuir probes and emissive probes in mapping the electrostatic plasma potential from the body of the plasma up to the sheath region of a plasma boundary, which in these experiments is a negatively biased electrode immersed within the plasma, in order to compare the two

diagnostic techniques and assess their relative advantages and weaknesses. Although Langmuir probes have the advantage of measuring the plasma density and electron temperature most accurately, emissive probes can measure electrostatic plasma potentials more accurately throughout the plasma, up to and including the sheath region.

INTRODUCTION:

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During this first century of plasma physics research, dating from Langmuir's discoveries in the 1920s of the medium like behavior of a new state of matter, plasma, the Langmuir probe has proved to have been the single most important diagnostic of plasma parameters. This is true in part, because of its extraordinary range of applicability¹. In plasma encountered by satellites²⁻⁴, in semiconductor processing experiments, 5-8 at the edges of plasma confined in tokamaks, 9-11 and in wide range of basic plasma physics experiments, Langmuir probes have been used to measure plasma densities and temperatures spanning the ranges $10^8 \le n_e \le 10^{19} \, \text{m}^{-3}$, and $10^{-3} \le n_e \le 10^{19} \, \text{m}^{-3}$ $T_e \le 10^2$ eV, respectively. Simultaneously in the 1920s, he invented the probe now named after him and the emissive probe¹². This diagnostic is now primarily used as a diagnostic of plasma potential. Although it cannot measure the breadth of plasma parameters that the Langmuir probe can, it too is diagnostic of wide utility when it comes to the measurement of plasma potential, or, as it is sometimes called, the electrostatic space potential. For example, the emissive probe can accurately measure space potentials even in a vacuum, where Langmuir probes are incapable of measuring anything.

The basic setup of the Langmuir probe consists of putting an electrode with various voltage biases applied into the plasma and measuring the collected current. The resulting current-voltage (I-V) characteristics can be used to interpret plasma parameters such as electron temperature T_e, electron density n_e , and plasma potential ϕ^{13} . For a Maxwellian plasma, the relationship between collected electron current I_e (assumed to be positive) and probe bias V_B can be expressed as¹⁴:

collected electron current I_e (assumed to be positive) and probe bias V_B
$$I_e(V_B) = \begin{cases} I_{e0} exp[\frac{e(V_B - \phi)}{KT_e}] \text{, } V_B \leq \phi \text{ (1a)} \\ I_{e0} \text{, } V_B > \phi \text{ (1b)} \end{cases}$$
 where I_{e0} is the electron saturation current,
$$I_{e0} = Sn_e^\infty e\sqrt{T_e/2\pi m_e} \text{, (2)}$$

$$I_{e0} = Sn_e^{\infty} e \sqrt{T_e/2\pi m_e} , (2)$$

and where S is the collecting area of the probe, n_e^{∞} is the bulk electron density, e is the electron charge, Te is the electron temperature, me is the electron mass. The theoretical relation of I-V characteristics for the electron current is illustrated in two ways in Figure 1A and Figure 1B. Note, Eq. (1a,b) only applies to bulk electrons. However, Langmuir probe currents can detect flows of charged particles, and adjustments must be made in the presence of primary electrons, electron beams, or ion beams etc. See Hershkowitz¹⁴ for more details.

The discussion here takes up the ideal case of Maxwellian electron energy distribution functions (EEDF). Of course, there are many circumstances in which non-idealities arise, but these are not the subject of this work. For example, in materials processing etching and deposition plasma systems, typically RF generated and sustained, there are molecular gas feed stocks that produce volatile chemical radicals in the plasma, and multiple ion species including negatively charged

ions. The plasma becomes electronegative, that is, having a significant fraction of the negative charge in the quasineutral plasma in the form of negative ions. In plasma with molecular neutrals and ions, inelastic collisions between electrons and the molecular species can produce dips¹⁵ in the current-voltage characteristics, and the presence of cold negative ions, cold relative to the electrons, can produce significant distortions¹⁶ in the vicinity of the plasma potential, all of which of course are non-Maxwellian features. We prosecuted the experiments in the work discussed in this paper in a single ion species noble gas (argon) DC discharge plasma, free of these kinds of non-Maxwellian effects. However, a bi-Maxwellian EEDF is typically found in these discharges, caused by the presence of secondary electron emission¹⁷ from the chamber walls. This component of hotter electrons is typically a few multiples of the cold electron temperature, and less than 1% of the density, typically easily distinguished from the bulk electron density and temperature.

As V_B becomes more negative than ϕ , electrons are partially repelled by the negative potential of the collecting surface, and the slope of the $ln(l_e)$ vs. V_B is e/T_e , ie. $1/T_{eV}$ where T_{eV} is the electron temperature in eV, as shown in **Figure 1B**. After T_{eV} is determined, the plasma density can be derived as:

$$n_e^{\infty} = \frac{l_{e0}}{S e} \sqrt{\frac{2\pi m_e}{T_e}} = 3.74 \times 10^{13} \frac{l_{e0}}{S T_{eV}} m^{-3}.$$
 (3)

Ion current is derived differently than electron current. Ions are assumed to be "cold" due to their relatively large mass, $M_i >> m_e$, compared to that of the electron, thus, in a weakly ionized plasma, the ions are in fairly good thermal equilibrium with the neutral gas atoms, which are at the wall temperature. Ions are repelled by the probe sheath if $V_B \ge \phi$ and collected if $V_B < \phi$. The collected ion current is approximately constant for negatively biased probes, while the electron flux to the probe decreases for probe bias voltages more negative than the plasma potential. Since the electron saturation current is much larger than the ion saturation current, the total current collected by the probe decreases. As the probe bias becomes increasingly negative, the drop in current collected is great or small as the electron temperature is cold or hot, as described above in Eq. (1a). The equation for ion current then is:

$$I_{i}(V_{B}) = \begin{cases} I_{i0} & V_{B} < \phi \\ 0 & V_{B} > \phi \end{cases} \tag{4}$$

113 where

$$I_{i0} \approx -\frac{1}{2}eSn_0u_B,\tag{5}$$

114 and

$$u_B = \sqrt{\frac{T_{ev}}{M_i}}. ag{6}$$

115 We note that constant ion flux collected by the probe exceeds the random thermal ion flux due 116 to acceleration along the presheath of the probe and thus ions reach the sheath edge of the 117 probe at the Bohm speed¹⁸, u_B, rather than the ion thermal speed¹⁹. And they have a density 118 equal to the electrons since the presheath is quasineutral. Comparing the ion and electron saturation current in Eqn.5 and 2, we observe that the ion contribution to the probe current is smaller than that of electrons by a factor of $\sqrt{M_i/2\pi m_e}$. Such factor is about 108 in the case of argon plasma.

There is a sharp transition point where the electron current goes from exponential to a constant, known as the "knee". The probe bias at the knee can be approximated as the plasma potential. In the real experiment, this knee is never sharp, but rounded due to the space-charge effect of the probe, that is, the expansion of the sheath surrounding the probe, and also to probe contamination, and plasma noise¹³.

The Langmuir probe technique is electrostatic based on collection current, whereas the emissive probe technique is based on the emission of current. Emissive probes measure neither temperature nor density. Instead they provide precise plasma potential measurements and can operate under a variety of situations due to the fact that they are insensitive to plasma flows. The theories and usage of emissive probes are fully discussed in the topical review by Sheehan and Hershkowitz²⁰, and references therein.

For plasma density $10^{11} \le n_e \le 10^{18}$ m⁻³, the inflection point technique is recommended, which means to take the inflection points of series I-V traces and extrapolate to the limit of zero emission and get the plasma potential, as shown in **Figure 2**.

It is a common assumption that the two techniques agree in quasineutral plasma, but disagree in the sheath, the region of the plasma in contact with the boundary in which space-charge appears. The study focuses on the plasma potential near plasma boundaries, in low temperature, low pressure plasma in an effort to test this common assumption. To compare potential measurements by both Langmuir probe and emissive probe, plasma potential is also determined by applying inflection point technique to Langmuir probe I-V, as shown in **Figure 3**. It is generally accepted that the plasma potential is found by finding the probe bias voltage at which the second derivative of the current collected differentiated with respect to the bias voltage, $d^2I/dV^2 = 0$, that is, the peak of the dI/dV curve, with respect to the probe bias voltage. **Figure 3** demonstrates how this maximum in dI/dV, the inflection point of the current-voltage characteristic, is found.

Langmuir probes (collecting) and emissive probes (emitting) have different I-V characteristics, which also depend on the geometry of the probe tip, as shown in **Figure 4**. The space-charge effect of the probe must be considered before the probe fabrication. In the experiments, for the planar Langmuir probes, we used a ¼' planar Tantalum disk. We could collect more current and get bigger signals with a larger disc. However, in order for the analyses above to apply, the area of the probe, A_p must be kept smaller than the electron loss area of the chamber, A_w , satisfying²¹ the inequality $A_p \leq A_w$. For the cylindrical Langmuir probe, we used a 0.025 mm thick, 1 cm long Tungsten wire for the cylindrical Langmuir probe and a same thickness for the Tungsten wire for the emissive probe. It is important to note that for cylindrical Langmuir probes, for the plasma parameters of these experiments, the radius of the probe tip, r_p , is much smaller than its length,

 L_p , and smaller than the Debye length, λ_D ; that is, $r_p \ll L_p$, and $0 < r_p/\lambda_D < 1$. In this range of parameters, applying Orbital Motion Limited theory and Laframboise's development of it²² for the case of thermal electrons and ions, we find that for probe bias voltages equal to or greater than the plasma potential, the electron current collected may be parameterized by a function of the form $I_e = I_{eo}(1 + V_B/T_{eV})^{C_e}$, where the exponent $C_e \sim 0.4$. The important point here is that for values of this exponent less than unity, the inflection point method for determining the plasma potential, as described in the paragraph above, applies to cylindrical Langmuir probes too.

PROTOCOL:

173 1. Building Langmuir probes and Emissive probes to fit into a vacuum chamber

1.1. Planar Langmuir probe (see Figure 5 for more details)

1.1.1. Take a ¼" in diameter stainless steel tube as the probe shaft and bend one end to the desired 90° angle.

1.1.2. Cut the unbent side to a length so that the probe is can axially cover more than half of the chamber length.

1.1.3. Fit the unbent side of the shaft through the brass tube by a SS-4-UT-A-8 adapter in combination with a B-810-6 union tube fitting.

1.1.4. Use a ½" brass tube extending out of the customized flanges through a B-810-1-OR swagelok interface to provide axial support for the probe shaft.

1.1.5. Connect the unbent end of the probe shaft to the BNC housing through a B-400-1-OR swagelok fitting, as shown in **Figure 6**.

1.1.6. Fit the gold-coated nickel wire through two single-bore alumina tubes (1/8" and 3/16" in diameter) with the thicker one fits inside the probe shaft, as shown in **Figure 7**.

1.1.7. Spot weld one end of gold-coated nickel wire onto a piece of stripped wire, which is soldered onto the pin of the BNC feedthrough at the end of the probe shaft.

1.1.8. Cut the gold-coated wire to length such that the joint with the stripped wire fits inside the alumina tube to prevent short circuit with the probe shaft.

1.1.9. Punch through a tantalum sheet to make a planar Langmuir probe tip (¼" in diameter)

1.1.10. Spot weld the other end of the gold-coated nickel wire onto the edge of the probe tip and set the probe tip to be normal to the axis of the boundary plate.

- 206 1.1.11. Position the probe tip a little forward so that the body of the probe does not touch the
 207 boundary plate while taking measurements inside the sheath.
- 209 1.1.12. Seal all joints with ceramic paste (e.g., Sauereisen Cement No. 31) to insulate the probe 210 circuit components from plasma. Use a heat guy to bake the ceramic joints for 5-10 min. 211
- 212 1.1.13. Use a multimeter to measure the resistance between the probe tip and the BNC connector. If continuity is demonstrated, the probe is ready to be put into the vacuum chamber.
- 216 1.2. Building a cylindrical emissive probe (see Figure 8 for more details)
- 218 1.2.1. Follow step 1.1.1-1.1.4 and repeat step 1.1.5-1.1.7 on the same probe shaft twice with the exception of using a 1/8", two-bore alumina tube instead of a single-bore one.
- 221 1.2.2. Cut the 0.025 mm diameter tungsten wire to about 1 cm.
- 223 1.2.3. Spot weld the tungsten filament onto gold-coated wires.224
- 225 1.2.4. Seal all the joints with ceramic paste and make sure the ceramic paste does not get onto
 226 the tungsten filament.
- 228 1.2.5. Check continuity between two BNC ends.
 - 2. Generate plasma

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- 232 2.1. Turn on the ion gauge to check the base pressure before putting gas into the chamber.
 233 Proceed with zeroing of the baratron gauge if the pressure is in the low 10⁻⁶ Torr range.
 234 Otherwise, check the leak in the system.
- 236 2.2. Use the pen to calibrate the baratron display until the number floats between ±0.01 237 mTorr.
- 239 2.3. Make sure that the needle valve is at a closed position.
- 241 Open the shutoff valve. Check that there is no pressure change on the baratron reading.
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- 2.5. Slowly turn the knob of the needle valve to release the gas into the chamber until the pressure reaches the requirement for the experiment. The typical working pressure stem from 10⁻⁵ ~ 2 x 10⁻³ Torr. Working gases have included argon, xenon, krypton, oxygen etc.
- 247 2.6. Turn on the KEPCO voltage power supply and set the voltage to -60 Volts to provide sufficient electron energy for the maximum ionization cross section of argon. Turn on the heating power supply for the filaments and slowly adjust the level until the discharge current

reads the required value. The discharge current tends to drop quickly in the first few minutes.

Keep adjusting the current level for about 30 minutes until the discharge stabilizes

2.7. Connect the voltage supply to the boundary plate and adjust the bias to desired level.

3. Take measurements

NOTE: I-V traces for Langmuir probes and emissive probes are acquired by a 16-bit DAQ board controlled by a Labview program. The details are not presented here since different users have different preferences for taking the data. However, there is a protocol for how to use the probes.

3.1. Take the load line: obtain an I-V trace without any plasma discharge in the chamber with all connections made between the probe and its measuring circuit (see **Figure 9-11** for the UW-Madison and the USD setup).

3.2. Langmuir probes

3.2.1. Clean the probe tip (this step is critical, as a clean probe exhibits a sharper 'knee' than a dirty probe) by biasing the probe positively so as to collect a large electron current.

3.2.1.1. Draw a current through the probe with a variable power supply and 50 Ohms to the machine ground to heat the tip so as to evaporate the layer of impurities that immediately attaches to the probe surface in the plasma and increase the surface resistivity of the probe.

3.2.1.2. Slowly increase the bias positively to surpass the plasma potential, permitting the probe to begin to draw the electron saturation current.

3.2.1.3. Continue to raise the potential; once one sees the probe tip glowing cherry red, the probe is clean. It is necessary to have a view of the probe tip in the plasma through a vacuum viewport.

3.2.1.4. Be careful and vigilant while varying the bias on the probe. If the probe is allowed to get too hot, the probe tip itself could become warped, and worse things can happen, such as the tip could have holes in it, it could evaporate, it could fall off; wires could melt and lose their insulation, and so forth.

3.2.1.5. Attach the probe to the data acquisition and control circuit (this is the part that will vary from lab to lab), and proceed to sweep the voltage applied to the probe while simultaneously measuring the current drawn by the probe. Save the I-V trace.

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3.3. Emissive probes 3.3.1. Repeat step 3.2.2 with the emissive probe's data acquisition and control circuit. 4. Data analysis 4.1. Langmuir Probes (See Figure 12, Figure 13 for more details). 4.1.1. Subtract the load line from the total I-V characteristic. 4.1.2. Fit the ion saturation current and subtract from the remaining I-V characteristics. 4.1.3. Take the natural log of current and plot it against the probe voltage. 4.1.4. Take linear fits of transitional region and saturation current separately. 4.1.5. Take the inverse of the slope of the transitional region and obtain the electron temperature value. 4.1.6. Obtain the plasma density by plugging the current at the crossing where the two fitted lines cross each other into Eq.3. 4.1.7. Apply the inflection point technique to the Langmuir probe trace and determine the plasma potential. 4.2. Emissive Probe (refer to **Figure 2**). 4.2.1. Repeat step 4.1.1-4.1.2 for individual I-V characteristics, then smooth each trace. 4.2.2. Differentiate each I-V trace and apply appropriate smoothing. 4.2.3. Locate the peak of each smoothed dI/dV (inflection point). 4.2.4. Apply a linear fit to the inflection points. 4.2.5. Obtain the plasma potential by locating the zero crossing of the fitted line. REPRESENTATIVE RESULTS: Langmuir probes, known to be sensitive to flows and to the kinetic energy of the particles they collect, have up till now have been considered to yield valid measurement of the plasma potential, except in sheaths. But direct comparisons of plasma potentials measured by Langmuir probes and emissive probes have demonstrated that in the quasineutral presheath region of the plasma immediately in contact with the sheath on the plasma side, Langmuir probes do not provide accurate measurements of the plasma potential²³. Plasma potentials from plasma bulk into the sheath measured by four different types of Langmuir probes were compared with the ones measured by an emissive probe for four different neutral pressures. Langmuir probes were built in four different configurations (see **Figure 14**) and were labeled as LP_j with j being an integer from 1 to 4. The cylindrical Langmuir probe is LP₁, LP₂, the double sided Langmuir probe, LP₃, the planar Langmuir probe with the side facing the boundary plate sealed by ceramic paste, and LP₄ stands for the planar Langmuir probe with the side facing away from the boundary plate covered by ceramic plate. The comparison between Langmuir probes and emissive probe potential measurements are shown in **Figure 15**.

In the work shown here, we compared how well cylindrical and planar Langmuir probes measure plasma potentials near plasma boundaries, a very important region of all bounded plasma systems. We were most concerned with the region called the presheath, which is that portion of the quasineutral plasma that boarders the plasma sheath itself. It is well known that in the simple plasma we diagnosed, that ions flow toward the boundary in order the set up the sheath structure, and that the speed of the ion flow ranges zero to the Bohm speed 18,20-21. We attempted to find out whether, under those conditions (see Figure 16C for the experimental setup), the manner in which Langmuir probes are used to measure plasma potentials give accurate results. We tried different designs of Langmuir probes, ones that were insulating on one side or the other, as well as that were conducting on both faces of the disc. We compared all the Langmuir probe measurements to emissive probe measurements of the plasma potential. We found that in the presheath, all Langmuir probes measure plasma potentials that differ from that measured by emissive probes, a difference that is positive relative to the plasma potential measured by emissive probes. The difference widens with proximity to the sheath edge, growing to a value of many electron temperatures. Representative results are shown in Figure 15A-C. This difference is an important result.

Plasma parameters such as Temperature, density, Debye lengths and Child-Langmuir sheath lengths obtained from the measurements by LP₂ in the bulk of the plasma are shown in Table 1. These parameters help establish good estimates of the nominal sheath thickness for planar Langmuir probes. Considering **Figure 15A-C**, one finds that the plasma potential profiles as determined by Langmuir probes deviate from those determined by emissive probes at a distance of three or four sheath thicknesses, which indicates that the difference between the two methods of measuring plasma potentials occurs in the plasma presheath, and not just in the sheath.

FIGURE AND TABLE LEGENDS:

Figure 1: Electron current collected by planar Langmuir probes. ideal electron current (I_e) versus probe bias (V_B) considering only bulk electrons are present in thermodynamic equilibrium at temperature T_{eV} and plotted with vertical axes as (A) linear and (B) logarithmic. Note that this data is acquired by subtracting the ion current from the probe current. The plasma potential is indicated by ϕ .

Figure 2: Emissive probe current - voltage characteristics and inflection point techniques. A) A

sample set of I-V traces by emissive probe in the linear scale and **B**) smoothed dI/dV curves. **C**) The plasma potential is determined by taking the inflection point in the limit of zero emission

Figure 3: Langmuir probe current-voltage characteristic and inflection technique for plasma potential measurement. plasma potential determined from the A) Langmuir probe I-V trace by B) inflection point method

Figure 4: Sheath expansion characteristics for planar, cylindrical, and spherical Langmuir probe tips for the case of collection and emission. Normalized I-V characteristics for the A) collecting probes and B) the emitting probes with different tip geometries (planar, cylindrical, and spherical). This figure has been modified from Sheehan and Hershkowitz²⁰.

Figure 5: Planar Langmuir probe tip mechanical schematic. A tungsten or tantalum tip is spot welded onto the wire (gold-plated nickel wire) exposed beyond the ceramic tubing. Ceramic past fastens the ceramic tubing to the stainless-steel tubing.

Figure 6: Langmuir probe body. Shown with part numbers and dimensions, the Langmuir probe body is design for vacuum seals at the vacuum chamber wall, at the coax cable connector (not shown here, see **Supplement Figure 6**), and a sliding, rotatable vacuum seal against the probe shaft. All tube fittings are listed in the table of materials.

Figure 7: Views of Langmuir probe tip fabrication and connection to probe shaft. A) Back View and **B)** side view of planar Langmuir probe. The probe tip is spot-welded to the gold-coated nickel wire which goes through two alumina tubes with the thicker one fitted in the metal shaft. All joints are sealed with ceramic paste.

Figure 8: Emissive probe tip schematic. Similar to Langmuir probe fabrication, the filament (tungsten wire) is spot welded to the gold plated nickel wire protruding from the small ceramic tubing covering each stalk. Ceramic past covers the exposed nickel wire and spot-weld, and fastens the ceramic tubing together and to the stainless steel tubing.

Figure 9: Langmuir probe measurement circuits at UW-Madison. A) A simplified measurement circuit for a Langmuir probe, **B**) The custom built DAQ and DAC board used at UW-Madison, and **C**) its circuit diagram.

Figure 10: Langmuir probe measurement circuits at USD. The bipolar operational amplifier power supply (4 quadrant power supply) and home built circuit to interface with 16-bit DAQ controlled by computer scripts, used at the USD.

Figure 11. Emissive probe measurement circuits at UW-Madison and USD. (A) A simplified measurement circuit diagram for the emissive probe, along with **(B)** a block diagram for the heating circuit used for emissive probes at both UW-Madison and USD. The heating circuit is described in more detail in Yan S-L et al.²⁶, from which this figure is adapted. The dotted line indicates the emissive probe circuit box, which has two inputs, one for the heating voltage and

one for the sweep voltage, and two outputs, for BNC cables that connect to the emissive probe. An interface circuit between the heating circuit and the DAQ used at USD, in (**C**).

Figure 12: The difference between the probe current and the electron current collected by a planar Langmuir probe. A) Sample of collected current vs. probe bias. The ion saturation current is linearly fitted from -85 V to -65 V. B) I-V trace after the ion current subtracted

Figure 13. Collected electron currents plotted on semi-log scales permitting electron temper and density measurements. A) a typical I-V trace in a semi-log scale obtained by a $\frac{1}{2}$ " planar disk Langmuir probe B) linear fitting of the transition region. Electron temperature is determined as 2.16 eV from the fitting between -1.9 and -2.2 V. Plasma density is determined by plugging the value of current at the crossing into Eq.3. The plasma potential V_P is determined this way to be about -0.4 V by locating the "knee", which is the location where two fitting lines cross. A more accurate method of measuring the plasma potential was shown in figure 3.

Figure 14: Multi-tip Langmuir probe detail. A) front view and **B)** top view of multi-tip Langmuir probe. The system (from left to right) consists of a cylindrical Langmuir probe, a 2-sided planar Langmuir probe, the planar Langmuir probe covered by ceramic paste in the front, the planar Langmuir probe covered in the back.

Figure 15: Results comparing various Langmuir probes to emissive probe measurements of the plasma potential near a plasma boundary. Plasma potential profiles for four different Langmuir probe configurations, and for an emissive probe, are displayed for four different neutral pressures; (**A**) 0.1 mTorr - (**D**) 1.0 mTorr. The boundary plate which created the sheath structure in the plasma was biased at -100 Volts. The discharge current was kept at 1.0 Amp. This panel of figures is adapted from Ref. 23.

Figure 16: Vacuum Chamber pumping scheme, magnetic confinement, and experimental design set up. The schematic of **A**) vacuum system and **B**) cross section of the multidipole chamber showing rows of magnets that help to confine thermionically emitted electrons, which are shown in **C**) being accelerated to the chamber wall so as to create ionization collisions with the neutral gas atoms, to make and confine the plasma. This figure was in part adapted from ref. 23.

Table 1: Plasma parameters for the experiments described in ref. 23, neutral pressure, electron temperature and density, Debye length, and Child-Langmuir length.

Supplemental Figure 1: Filaments for thermionic emission. A) The heating filament array and **B)** the wire setup on the chamber door

Supplemental Figure 2: Boundary plate support wire. Side view of the boundary plate setup from the vacuum viewport. Because of the laser beam dump welded onto the plate, the plate is heavy and needs support from above to maintain its orientation. The angle of the boundary plate is controlled by the length of the wire. The wire itself is attached to an empty Langmuir probe

shaft admitted from a flange on the top of the chamber.

Supplemental Figure 3: Boundary plate bias supply. Bias supply setup for the boundary plate, used to provide a negative bias leading to a sheath structure in the plasma surrounding the boundary plate.

Supplemental Figure 4: Tube fittings for a rotatable and translatable vacuum seal against the probe shaft. Tube fittings that come with O-rings are readily available and may be used for rotatable and translatable vacuum seals against a polished cylindrical tube. They can be improved upon with light machining to increase the inner diameter on the side opposite to the vacuum chamber. It is useful to order a brass fitting. Ferrules for ¼" tubing are used to separate 2 O-rings fit into the bore and compressed with the Cajon end nut and pusher, permitting the tube to twist and translate axially while maintaining the vacuum seal. The O-rings are lightly greased with vacuum grease.

Supplemental Figure 5: Langmuir probes for on-axis measurements, but which enter the vacuum chamber off-axis. Langmuir probe for smaller chambers before all joints sealed with ceramics. A single-bore alumina tube is inserted into the probe shaft until it bottoms out.

Supplemental Figure 6: BNC vacuum seal scheme. A) A vacuum sealed BNC to KF feedthrough is used to complete the vacuum seal for the probe (double and quad BNC connectors may also be purchased). **B)** A brass tube to pipe thread fitting may be used to connect to a KF fitting that completes the attachment as shown. Also note that BNC to KF feedthroughs are available with 2 and 4 BNC connectors. Custom flanges for emissive probes that require 2 BNC connectors, such as those used at UW-Madison, can be avoided if desired.

Supplemental Figure 7: The difference between raising or lowering the heating currents, consecutively. Inflection point technique to the limit of zero emission by A) high to low heating and B) low to high heating. The pressure is 0.25 mTorr, probe position is 30 mm from the boundary plate, which is biased at -90 Volts. The inflection points of high to low heating have less spread around the fitted line.

DISCUSSION:

Langmuir probes are used for particle flux measurements in an extraordinarily wide range of plasma densities and temperatures, from space plasmas in which the electron density is just a few particles $10^6 \, \mathrm{m}^{-3}$ to the edge region of fusion plasmas where the electron density is more like a few times $10^{20} \, \mathrm{m}^{-3}$. Moreover, electron temperatures between 0.1 and a few hundred eV's have been diagnosed with Langmuir probes. Langmuir probes are often used to measure plasma density and temperature. Finding the electrostatic plasma potential is intimately related to obtaining those two measurements. Emissive probes, on the other hand, are typically used solely to measure the plasma potential, and are of use in an even wider range of plasma parameters. This work describes in detail how to build and use both the Langmuir probes and the emissive probes in a laboratory setting in which a vacuum chamber is used to create and confine the plasma of interest, and discusses critical limitations to the use of Langmuir probes with respect

to their use in measuring plasma potentials accurately near plasma boundaries where sheaths and presheaths form.

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More rigorous steps of analyzing emissive probe I-V traces to obtain the plasma potential using the inflection point method in the limit of zero emission are discussed by Smith et al.²⁷. The user digitally controls the number of heating currents, one of which must be zero, and collects an I-V characteristic just like that described for Langmuir probes, for each heating current. By comparing the ion branch of I-V characteristics for the `cold sweep', that is, for zero heating current, to all the other characteristics (with positive heating currents), one can deduce to analog conversion I_c , collected current, and I_e , emission current determined Traces smoothed Traces differentiated, dI/dV smoothed and plotted vs. V, Maxima of dI/dV (inflection point of I-V traces) acquired Plot of I_e/I_c vs. V_{infl} (V determined by bias voltage of probe at inflection point) is fit with a linear extrapolation to the limit of zero emission to determine ϕ .

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Critical steps to building both probes are explained in detail, particularly drawing attention to vacuum seals that permit the probe shafts to be rotated and translated so that the probe tips may be positioned as needed by the researcher. We have indicated where suitable parts could be purchased by particular vendors, and where in-house machining may be required. We have also outlined the basic steps of analysis, more as a process of application of probe theory than as a software-dependent version of computational coding steps, recognizing that each lab may have different computational tools at their disposal.

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Langmuir probes, as is true of any diagnostic, have important limitations, some of which are central to the physics questions we have pursued in this comparison of probe techniques, a comparison which may be briefly summarized as follows: in relatively low temperature, low pressure plasmas, less than 10 eV, less than a few tenths of Pa of neutral pressure, planar and cylindrical Langmuir probe measurements of potential differ from the true plasma potential in the quasineutral presheath. But they have other limitations as well. The Langmuir probe technique is sensitive to plasma flows, and depending on whether the flow is signal or noise, this sensitivity may or may not be a limitation. Further, there can be problems with secondary electron emission, problems with plasma collisionality in higher pressure plasma, problems with ionization if biased too widely, and so forth. Emissive probes of course are not sensitive to plasma flows which make them superior to Langmuir probes in the measurement of plasma potential near boundaries where sheaths form concomitant with ion flows to the boundary. An active area of research regarding emitting surfaces at the boundary of plasma pursues the possibility of inverse sheaths²⁸ that might form if emission is sufficiently strong, and if the virtual cathode that can form around the emitting surface can indeed trap ions. There is some evidence that suggests inverse sheaths²⁹ could, where they form, cause emissive probes to float above the local plasma potential. Recent experiments with strongly emitting emissive probes in higher pressure plasma $(P_n > 3 \text{ mTorr})$ than that of the experiments reported here to some extent corroborates³⁰ this view. However, for low pressure, low temperature plasma, with modest heating currents, it appears that the inflection point technique in the limit of zero emission is not affected by this sort of phenomena. Finally we mention one last limitation common to both probe techniques, namely, that if the plasma is too dense and hot the probes cannot mechanically survive¹³, leading

to the upper limits quoted in the introduction.

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ACKNOWLEDGMENTS:

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DISCLOSURES:

566 The authors have nothing to disclose.

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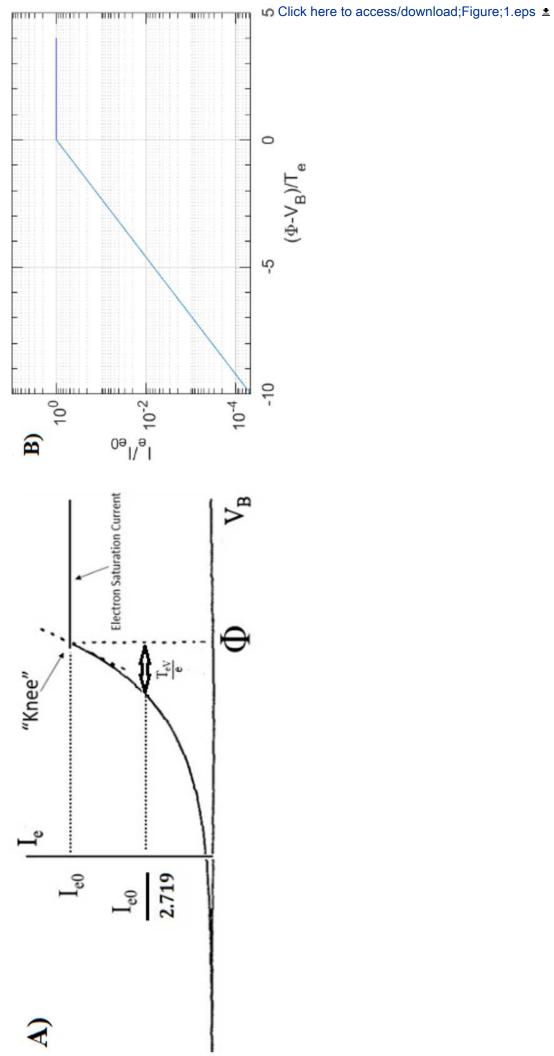
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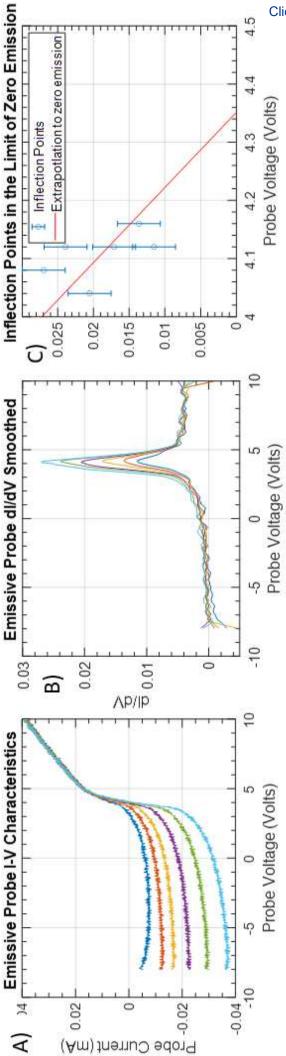
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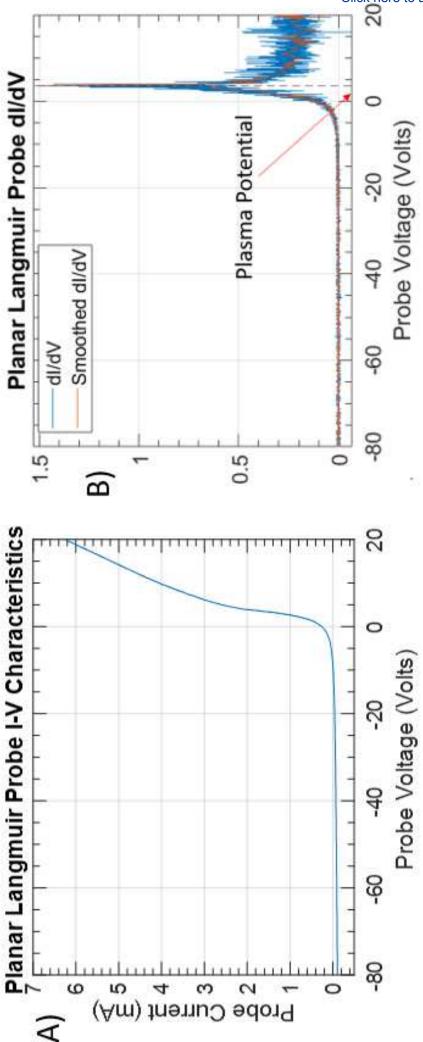
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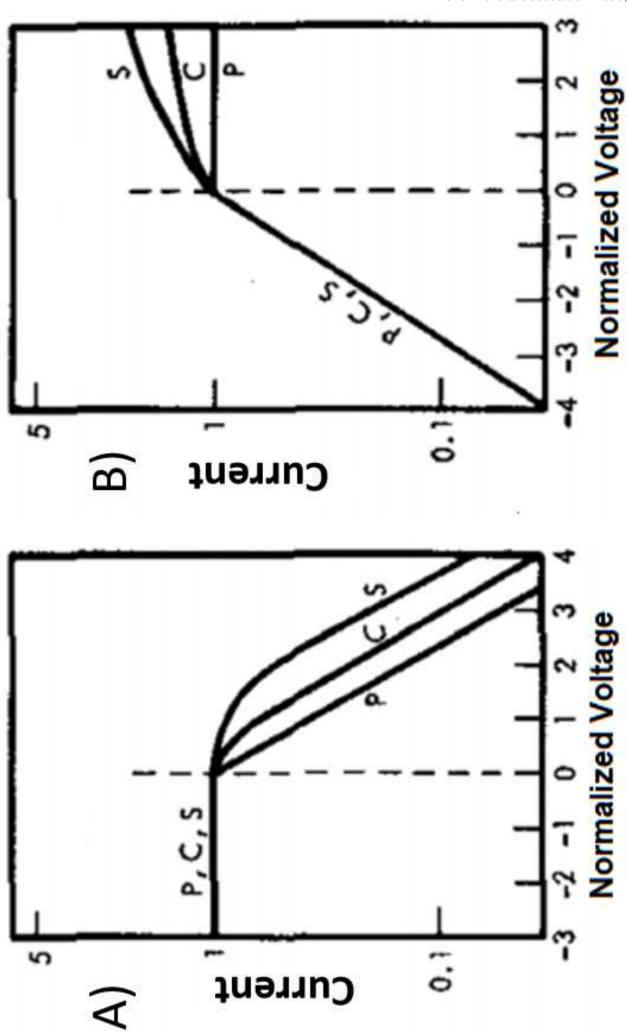
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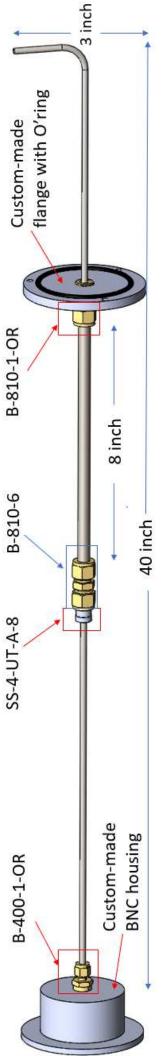
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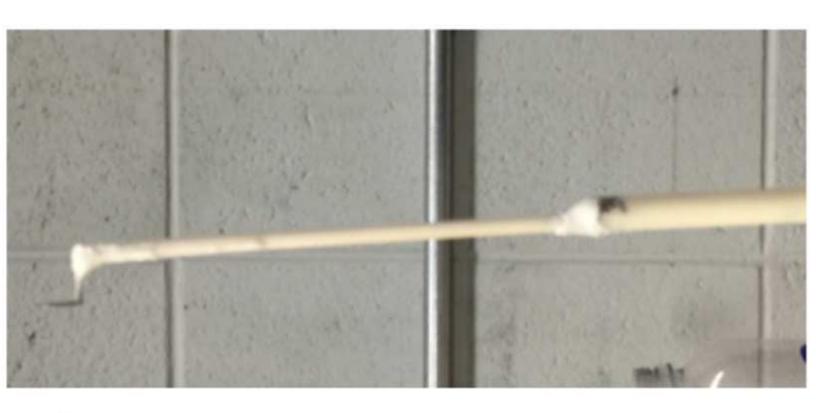








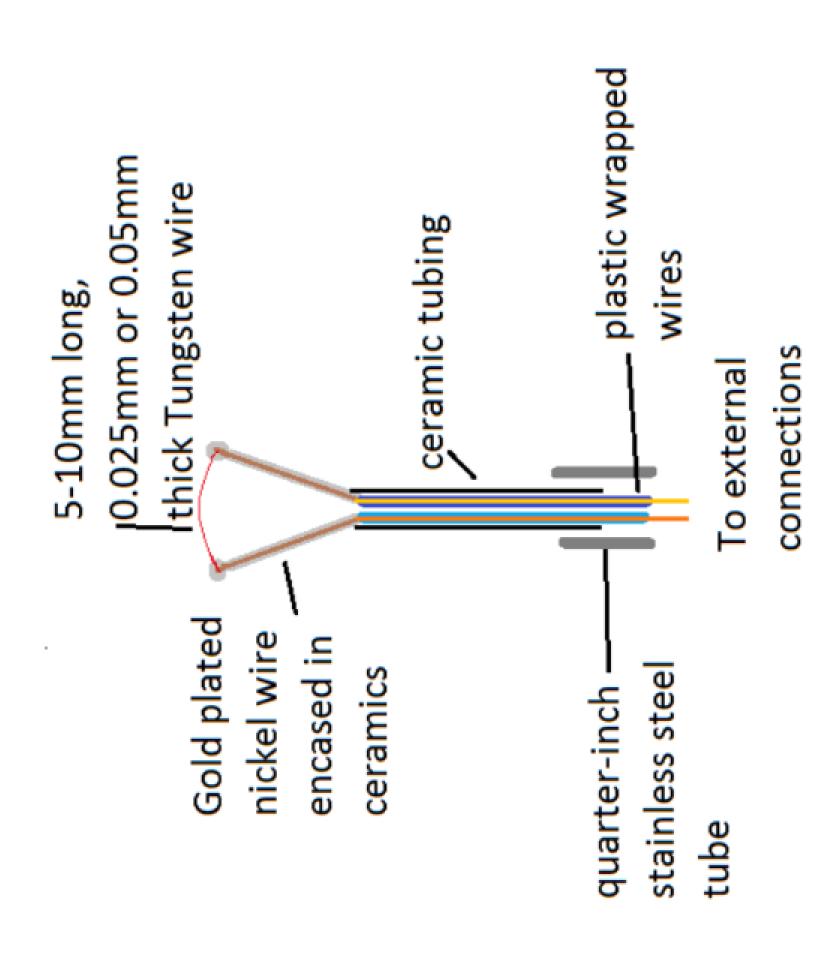


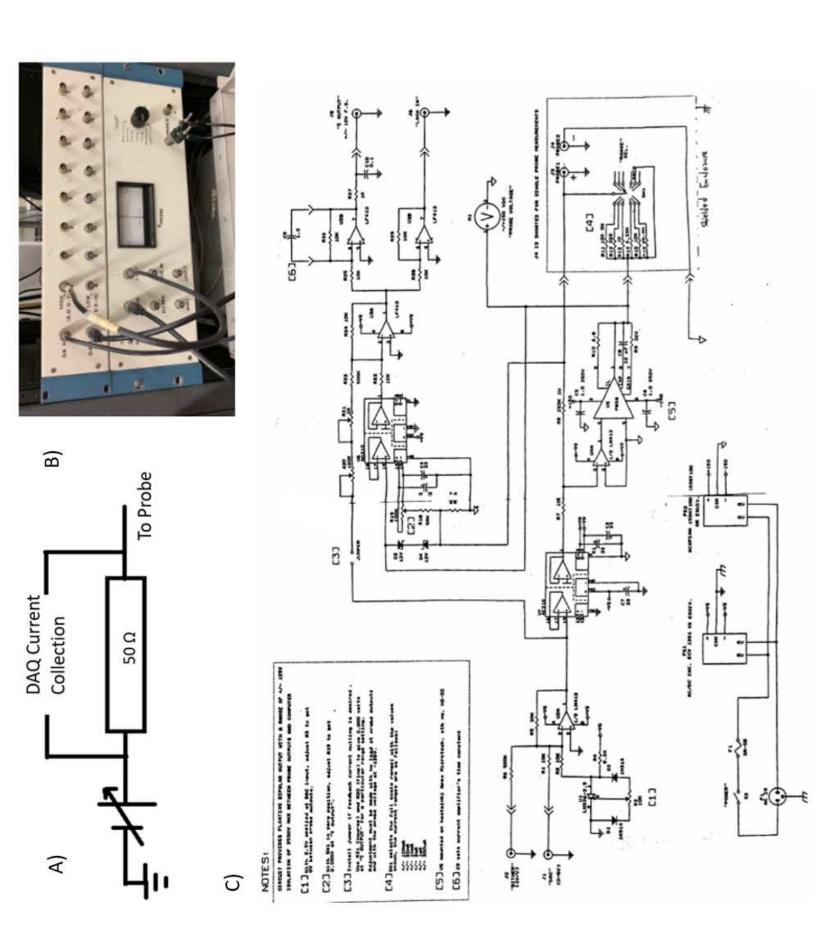






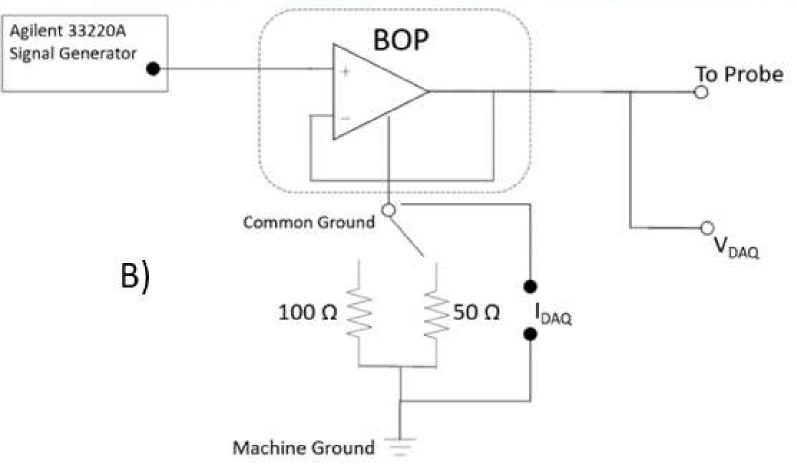


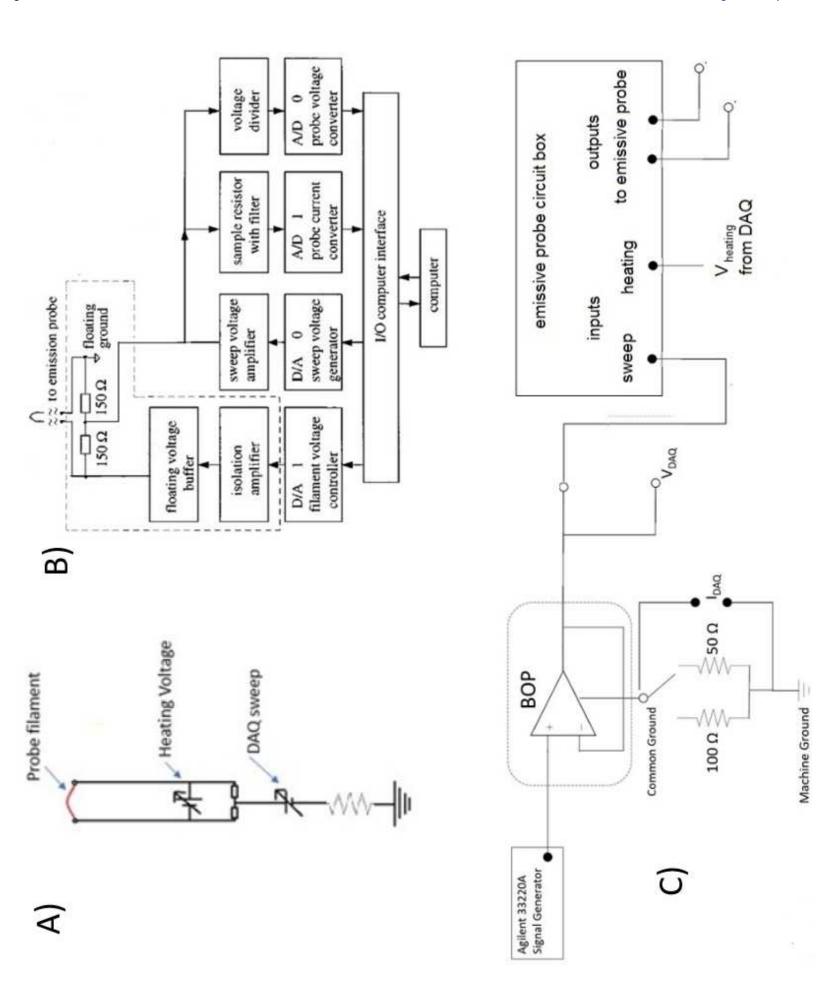




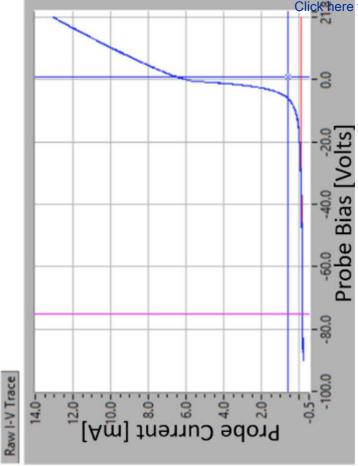




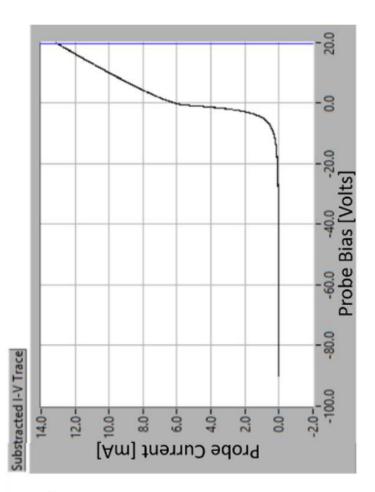






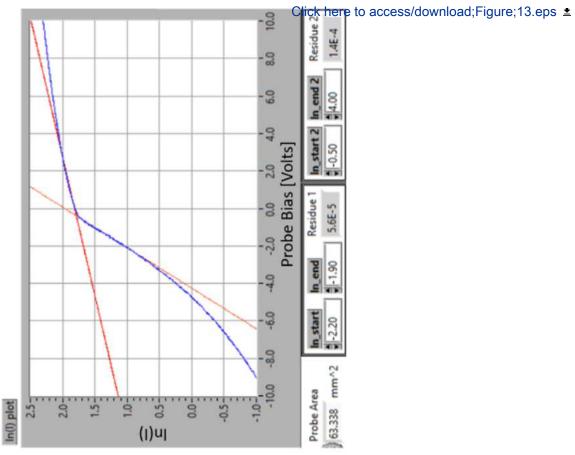




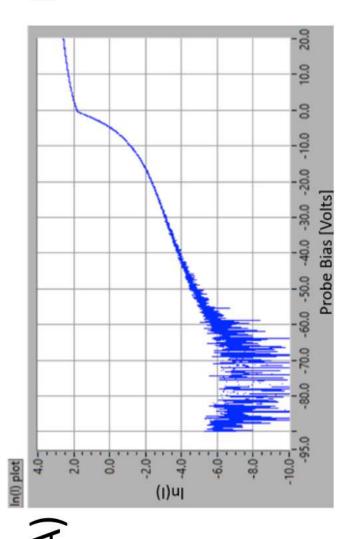








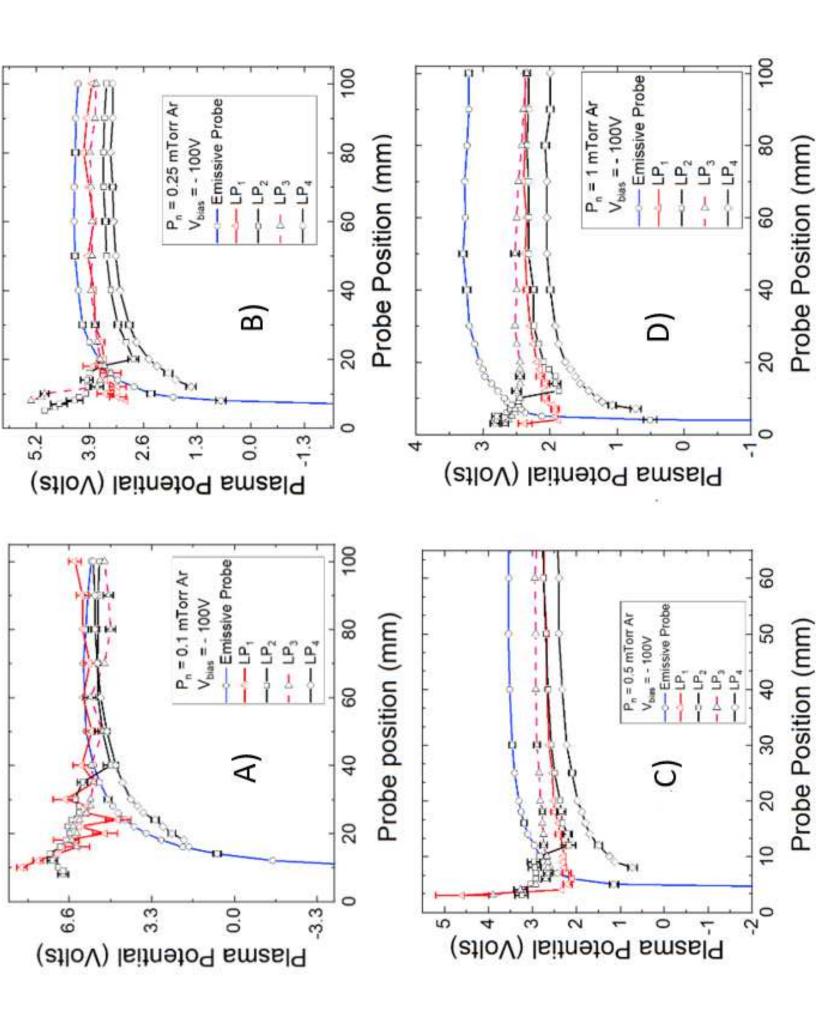


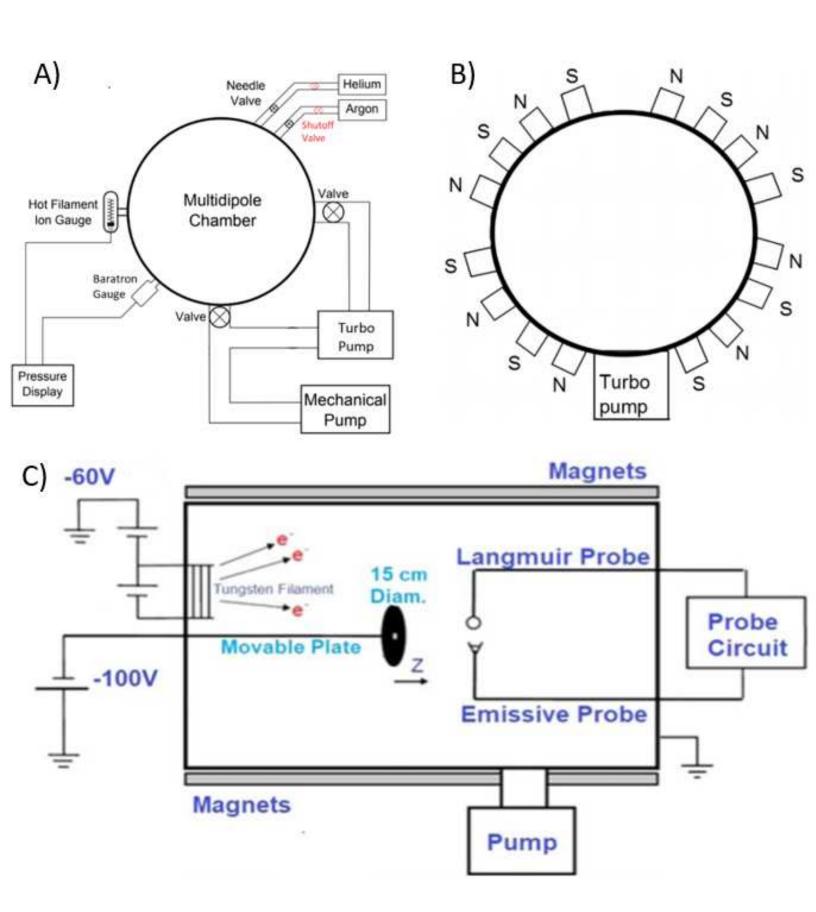




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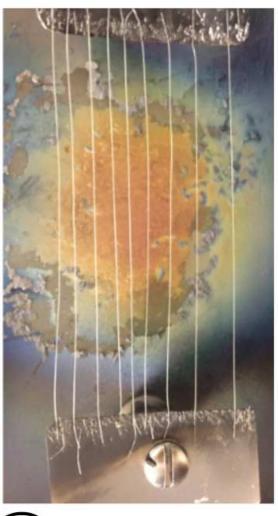




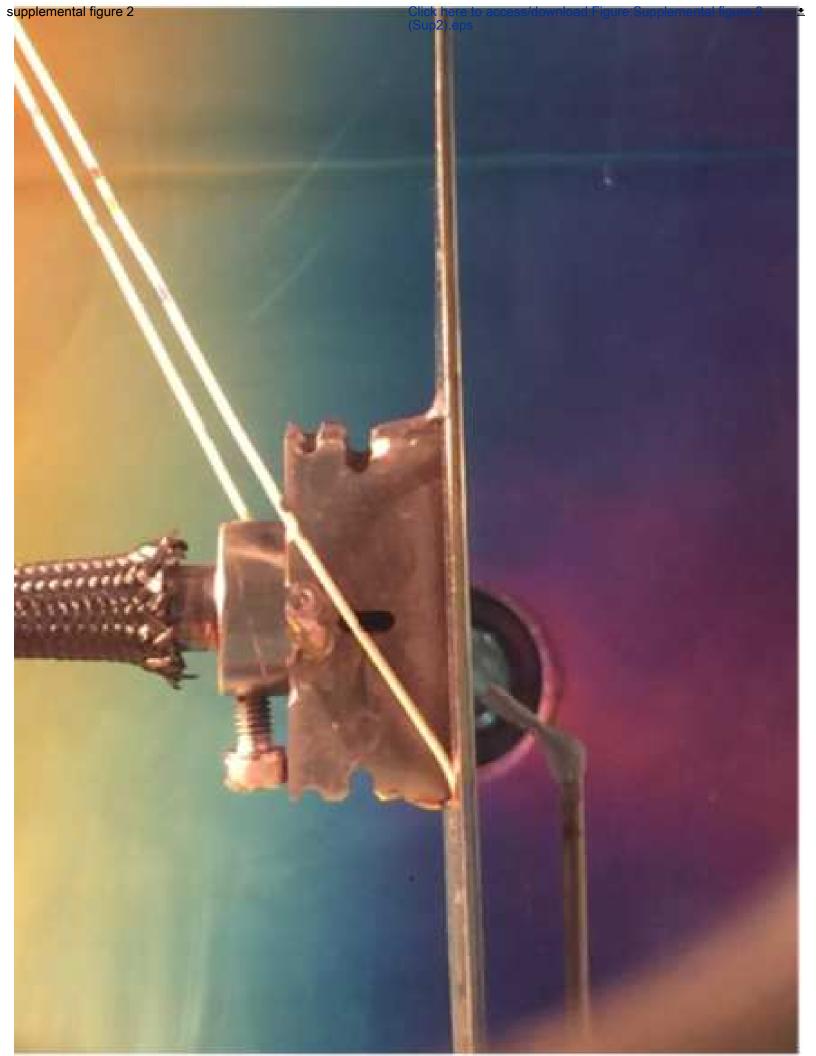














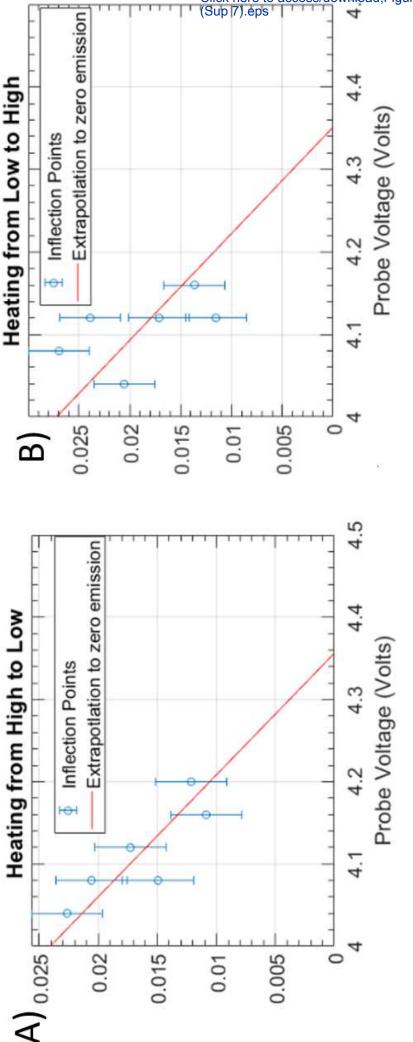






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Name of Material/ Equipment	Company	Catalog Number
0.001" thick tungsten wire	Midwest Tungsten Service	0.001"
0.005" thick tantalum sheet	Midwest Tungsten Service	0.005"
1/2" Brass supprting tube		
1/4" Brass Ferrule Set	Swagelok	B-400-SET
1/4" OD 304 or 315 stainless steel tube	Swagelok	SS-T4-S-035-20
Alumina tubes	COORSTEK	65655, single bore 0.156" OD 0.094 ID
Baratron gauge	MKS	Type 127
Brass Swagelok Tube Fitting	Swagelok	B-400-1-OR
Brass Swagelok Tube Fitting	Swagelok	B-810-6
Brass Swagelok Tube Fitting	Swagelok	B-810-1-OR
Ceramic liquid	Sauereisen	No. 31 Ceramic Encapsulant Liquid
Ceramic powder	Sauereisen	Cement Powder No. 31 Off-White
Gold plated nickel wire	SYLVANIA ELECTRIC PRODUCT	
Ion gauge controller	Granville-Phillips	270 Gauge controller
Mechanical pump	Leybold D60 D60AC	D60 D60AC
needle valve	Whitey	SS-22RS4
Power supply	Керсо	ATE 100-10M
Power supply	Sorensen	DCR 20-115B
shutoff valve	Kurt J. Lesker	Nupro SS-4BK
Stainless Steel Ultra-Torr Vacuum Fitting	Swagelok	SS-4-UT-A-8
Teflon coated wire	Geyer Systems	P31546
Turbo pump	PFEIFFER	TPH 240 C
Vacuum grease	APIEZON	L Ultra High Vacuum Grade Grease
Viton Orings	Grainger	#031
Viton Orings	Grainger	#010

Comments/Description

Emissive probe filament Heating filament to generate plasma

Interface between stainless probe shaft and swagelok tube fitting

Used to make the probe shaft, order seamless, sold in 20' lengths

single bore, double bore, quadruple bore, use for support structure for both emissive and Langmuir probes between the probe tip a

Display the pressure when there's gas flowing in the chamber

Tube fittings used on the probe

Tube fittings used on the probe

Tube fittings used on the probe

Mix with No.31 cement power to make the ceramic paste

There are Saureisen cements that cure with water, e.g. No.10 Powder

spod-welded to the probe tip to provide supports

Heat up the ion gauge and display pressure inside the chamber

Bring the pressure down to ~10 mTorr then serve as the backing pump for the turbo pump

Metering Micro-Needle Micrometer Valve 1/4" Tube Swagelok fittings

Voltage Bias supply of heating filament

Heating supply of heating filament

Knob handle, for 1/4" tubing, swagelok fittings

Tube fittings used on the probe

Connect the gold-coated wire to BNC pin

Bring the pressure down to 1E-6 Torr

Vacuum grease used to lubricate the oring

Round #031 Medium Hard Viton O-Ring, 1.739" I.D., 1.879" O.D

Round #010 Medium Hard Viton O-Ring, 0.239" I.D., 0.379"O.D

nd shaft



To: Nam Nguyen, Ph.D
Manager of Review, JOVE
From: Greg Severn, Ph.D
Professor of Physics, USD
Corresponding author, JOVE manuscript 1

Corresponding author, JOVE manuscript JoVE61804

Date: 20 August 2020.

On behalf of my colleagues, Noah Hershkowitz and Peter Li, we want to thank the editorial staff at JOVE and the three reviewers who carefully reviewed the manuscript and made insightful comments. We have addressed each comment, and the changes that we have made to the manuscript are recorded in colored font. We have reproduced the Editorial comments and the Reviewer's comments, and have placed our responses subordinate to each of these. As a result of the comments, the manuscript is much improved.

Editorial comments:

Changes to be made by the Author(s):

- 1. Please take this opportunity to thoroughly proofread the manuscript to ensure that there are no spelling or grammar issues.
- 2. Unfortunately, there are sections of the manuscript that show overlap with previously published work. Please use original language throughout the manuscript. Please revise the following: first paragraph of the introduction,

Langmuir probes have been one of the most important diagnostics of particle flux in the field of plasma science for almost a century¹. Used aboard satellites²⁻⁴, in plasma processing experiments, ⁵⁻⁸ at boundaries in tokamaks, ⁹⁻¹¹ and in a host of fundamental plasma physics experiments past and present spanning the range of plasma densities and temperatures represented by these applications $10^8 \le n_e \le 10^{19} \, \text{m}^{-3}$, and $10^{-3} \le T_e \le 10^2 \, \text{eV}$, Langmuir probes have been useful over a very wide range of plasma parameters. At the same time Langmuir invented the probe that bears his name (circa 1923), he also invented the emissive probe¹², now primarily used as a diagnostic of plasma potential, the accuracy of which has been improved by refinements in technique. It is a diagnostic of utility in an even wider variety of environments (for example, it can be used to measure space potentials in a vacuum).

(the paragraph above is essentially the first paragraph of the published work that caught the attention of the JOVE editorial staff, essentially verbatim. We have changed this paragraph as shown below)

During this first century of plasma physics research, dating from Langmuir's discoveries in the 1920's of the medium like behavior of a new state of matter, plasma, the Langmuir probe has proved to have been the single most important diagnostic of plasma parameters. This is true in part because of its extraordinary range of applicability¹. In plasma encountered by satellites²⁻⁴, in semiconductor plasma processing experiments,⁵⁻⁸ at the edges of plasma confined in tokamaks,⁹⁻¹¹ and in a wide range of basic plasma physics experiments, Langmuir probes have been used to



measure plasma densities and temperatures spanning the ranges $10^8 \le n_e \le 10^{19} \, \text{m}^{-3}$, and $10^{-3} \le T_e \le 10^2 \, \text{eV}$, respectively. Also during the 1920's, and at the same time he invented the probe now named after him, he invented the emissive probe¹². This diagnostic is now primarily used as a diagnostic of plasma potential. Although it cannot measure the breadth of plasma parameters that the Langmuir probe can, it too is diagnostic of wide utility when it comes to the measurement of plasma potential, or, as it is sometimes called, the electrostatic space potential. For example, the emissive probe can accurately measure space potentials even in a vacuum, where Langmuir probes are incapable of measuring anything.

second paragraph of the representative results.

The design of the Langmuir probes, indeed of the experiment itself, was to discover how cylindrical probes and planar Langmuir probes would work in the neighborhood of the plasma boundary that included the quasineutral presheath. The basic design of the experiment was to set up a presheath structure using a negatively biased electrode immersed within a large plasma volume, well known to produce directed ion flow toward the electrode, ranging from zero to the Bohm speed^{15,20-21}. We wished to observe if under those conditions (see figure 13c for the experimental set up), the usual determination of the plasma potential in the neighborhood of the sheath using Langmuir probe current—voltage (I—V) characteristics still hold valid, calibrated by emissive probe measurements. It is clear that cylindrical and planar (collecting on both sides) Langmuir probes measure an unphysically positive value of the plasma potential relative to emissive probe measurements, in the presheath, with the difference widening as the position at which the comparative measurements takes place gets closer and closer to the sheath edge. Representative results are shown in Fig.23. (a-c).

(much of this paragraph was flagged as overlapping with content on IOPSCIENCE.IOP.ORG, but without specifying which publication. To the credit of the complaint, however, this paragraph does borrow much from the first paragraph in section 2 of (our) ref. 3 cited in the iThenticate report! We have rewritten it as shown below[©]

In the work shown here, we compared how well cylindrical and planar Langmuir probes measure plasma potentials near plasma boundaries, a very important region of all bounded plasma systems. We were most concerned with the region called the presheath, which is that portion of the quasineutral plasma that boarders the plasma sheath itself. It is well known that in the simple plasma we diagnosed, that ions flow toward the boundary in order the set up the sheath structure, and that the speed of the ion flow ranges zero to the Bohm speed 18,20-21. We attempted to find out whether, under those conditions (see Fig.16C for the experimental set up), the manner in which Langmuir probes are used to measure plasma potentials give accurate results. And we tried different designs of Langmuir probes, ones that were insulating on one side or the other, as well as that were conducting on both faces of the disc. We compared all the Langmuir probe measurements to emissive probe measurements of the plasma potential. We found that in the presheath, all Langmuir probes measure plasma potentials that differ from that measured by emissive probes, a difference that is positive relative to the plasma potential measured by emissive probes. The difference widens with proximity to the sheath edge, growing to a value of many electron temperatures. Representative results are shown in Fig.15 (A-C). This difference is an important result.

I have attached the iThenticate report here as well.



3. Please revise the title to be more concise.

The current title: How to build Langmuir probes and emissive probes for plasma potential measurements in low pressure, low temperature DC plasmas, along with some surprising results at plasma boundaries

Has been changed to:

How to build, and use Langmuir probes and emissive probes for plasma potential measurements in low pressure, low temperature plasmas, especially near plasma boundaries.

4. Please ensure that all text in the protocol section is written in the imperative tense as if telling someone how to do the technique (e.g., "Do this," "Ensure that," etc.).

We have re-written the protocol section in the imperative tense. The new version is attached below: **PROTOCOL:**

- 1. Building Langmuir probes and Emissive probes to fit into a vacuum chamber:
- 1.1. Planar Langmuir probe (see Fig.5 for more details):
- 1.1.1. Take a 1/4" in diameter stainless steel tube as the probe shaft and bend one end to the desired 90 degrees angle.
- 1.1.2. Cut the unbent side to a length so that the probe is can axially cover more than half of the chamber length.
- 1.1.3. Fits the unbent side of the shaft through the brass tube by a SS-4-UT-A-8 adapter in combination with a B-810-6 union tube fitting.
- 1.1.4. Use a ½" brass tube extending out of the customized flanges through a B-810-1-OR swagelok interface to provide axial support for the probe shaft
- 1.1.5. Connect the unbent end of the probe shaft to the BNC housing through a B-400-1-OR swagelok fitting, as shown in Fig.6.
- 1.1.6. Fit the gold-coated nickel wire through two single-bore alumina tubes (1/8" and 3/16" in diameter) with the thicker one fits inside the probe shaft, as shown in Fig. 7
- 1.1.7. Spot weld one end of gold-coated nickel wire onto a piece of stripped wire, which is soldered onto the pin of the BNC feedthrough at the end of the probe shaft.
- 1.1.8. Cut the gold-coated wire to length such that the joint with the stripped wire fits inside the alumina tube to prevent short circuit with the probe shaft.
- 1.1.9. Punch through a tantalum sheet to make a planar Langmuir probe tip ($\frac{1}{4}$ " in diameter)
- 1.1.10. Spot weld the other end of the gold-coated nickel wire onto the edge of the probe tip and set the probe tip to be normal to the axis of the boundary plate
- 1.1.11. Position the probe tip a little forward so that the body of the probe does not touch the boundary plate while taking measurements inside the sheath.
- 1.1.12. Seal all joints with Sauereisen Cement No. 31 ceramic paste to insulate the probe circuit components from plasma. Use a heat guy to bake the ceramic joints for 5-10 min.
- 1.1.13. Use a multimeter to measure the resistance between the probe tip and the BNC connector. If continuity is demonstrated, the probe is ready to be put into the vacuum chamber.
- 1.2. Steps to build a cylindrical emissive probe (see Fig.8 for more details):
- 1.2.1. Follow step 1.1.1-1.1.4 and repeat step 1.1.5~1.1.7 on the same probe shaft twice with the exception of using a 1/8", two-bore alumina tube instead of a single-bore one.
- 1.2.2. Cut the 0.025 mm diameter tungsten wire to about 1 cm.



- 1.2.3. Spot weld the tungsten filament onto gold-coated wires.
- 1.2.4. Seal all the joints with ceramic paste and make sure the ceramic paste does not get onto the tungsten filament.
- 1.2.5. Check continuity between two BNC ends.

2. Generate plasma:

- 2.1.1. Turn on the ion gauge to check the base pressure before putting gas into the chamber. Proceed with zeroing of the baratron gauge if the pressure is in the low 10⁻⁶ Torr range. Otherwise, check the leak in the system.
- 2.1.2. Using the pen to calibrate the baratron display until the number floats between ± 0.01 mTorr.
- 2.1.3. Make sure that the needle valve is at a closed position.
- 2.1.4. Open the shutoff valve. Check that there is no pressure change on the baratron reading.
- 2.1.5. Slowly turn the knob of the needle valve to release the gas into the chamber until the pressure reaches the requirement for the experiment. The typical working pressure stem from $10^{-5} \sim 2 \times 10^{-3}$ Torr. Working gases have included argon, xenon, krypton, oxygen, etc.
- 2.1.6. Turn on the KEPCO voltage power supply and set the voltage to -60 Volts. Turn on the SORENSEN heating power supply and slowly adjust the level until the discharge current reads the required value. The discharge current tends to drop quickly in the first few minutes. Keep adjusting the current level for about 30 minutes until the discharge stabilizes
- 2.1.7. Connect the voltage supply to the boundary plate and adjust the bias to desired level.
- 3. Take measurements (I-V traces for Langmuir probes and emissive probes are acquired by a 16-bit DAQ board controlled by a Labview program. We won't go into too much detail here since different users have different preferences for taking the data. However, there is a protocol for how to use the probes):
- 3.1. Take the load line: obtain an I-V trace without any plasma discharge in the chamber with all connections made between the probe and its measuring circuit (see Fig.9-11 for the UW-Madison and the USD set-up).

3.2. Langmuir probes

- 3.2.1. Clean the probe tip (this step is critical, as a clean probe exhibits a sharper 'knee' than a dirty probe) by biasing the probe positively so as to collect a large electron current:
- 3.2.1.1. Draw a current through the probe with a variable power supply and 50 Ohms to the machine ground to heat the tip so as to evaporate the layer of impurities that immediately attaches to the probe surface in the plasma and increase the surface resistivity of the probe.
- 3.2.1.2. Slowly increase the bias positively to surpass the plasma potential, permitting the probe to begin to draw the electron saturation current.
- 3.2.1.3. Continue to raise the potential; once one sees the probe tip glowing cherry red, the probe is clean. It is necessary to have a view of the probe tip in the plasma through a vacuum viewport.
- 3.2.1.4. Be careful and vigilant while varying the bias on the probe. If the probe is allowed to get too hot, the probe tip itself could become warped, and worse things can happen, such as the tip could have holes in it, it could evaporate, it could fall off; wires could melt and loose their insulation, and so forth.
- 3.2.1.5. Attach the probe to the data acquisition and control circuit (this is the part that will vary from lab to lab), and proceed to sweep the voltage applied to the probe while simultaneously measuring the current drawn by the probe. Save the I-V trace.
- 3.2.2. Attach the probe to the data acquisition and control circuit (this is the part that will vary from

lab to lab), and proceed to sweep the voltage applied to the probe while simultaneously measuring the current drawn by the probe. Save the I-V trace.

- 3.3. Emissive probes:
- 3.3.1. Repeat step 3.2.2 with the emissive probe's data acquisition and control circuit
- 4. Data analysis:
- 4.1. Langmuir Probes (See Fig.12, 13 for more details):
- 4.1.1. Subtract the load line from the total I-V characteristic
- 4.1.2. Fit the ion saturation current and subtract from the remaining I-V characteristics
- 4.1.3. Take the natural log of current and plot it against the probe voltage.
- 4.1.4. Take linear fits of transitional region and saturation current separately.
- 4.1.5. Take the inverse of the slope of the transitional region and obtain the electron temperature value.
- 4.1.6. Obtain the plasma density by plugging the current at the crossing where the two fitted lines cross each other into Eq.3
- 4.1.7. Apply the inflection point technique to the Langmuir probe trace and determine the plasma potential
- 4.2. Emissive Probe (refer to Fig.2):
- 4.2.1. Repeat step 4.1.1~4.1.2 for individual I-V characteristics, then smooth each trace
- 4.2.2. Differentiate each I-V trace and apply appropriate smoothing.
- 4.2.3. Locate the peak of each smoothed dI/dV (inflection point)
- 4.2.4. Apply a linear fit to the inflection points.
- 4.2.5. Obtain the plasma potential by locating the zero crossing of the fitted line
- 5. The Protocol should contain only action items that direct the reader to do something. Please move the discussion about the protocol to the Discussion.

We cut a portion of our old protocol and moved a part of that to the discussion section. The new protocol section only has action terms.

6. Please highlight up to 3 pages of the Protocol (including headings and spacing) that identifies the essential steps of the protocol for the video, i.e., the steps that should be visualized to tell the most cohesive story of the Protocol. Remember that non-highlighted Protocol steps will remain in the manuscript, and therefore will still be available to the reader. This 3 page limit assumes a one line spacer between protocol steps.

The new Protocol section is about 2.5 pages. We highlighted all of them because every step is important to show audience how to build the probes and how to take and analyze the data.

7. Please ensure that the highlighted steps form a cohesive narrative with a logical flow from one highlighted step to the next. Please highlight complete sentences (not parts of sentences). Please ensure that the highlighted part of the step includes at least one action that is written in imperative tense.



We hope the new protocol section meets the requirement since every step is in imperative tense, and the entire section is highlighted.

8. There are currently 23 figures. Can some of the figures be combined into different panels of the same figure or be converted into supplemental figures?

We re-arranged the figures according to their reference in the edited manuscript. Sixteen figures are mentioned in the new introduction and protocol section. We listed all figures mentioned in the discussion section as supplemental figures and they were put into the "Supplementary Files" section.

9. Please combine all panels of one figure into a single image file.

All panels are combined into one image file.

10. Please remove the embedded Table from the manuscript. All tables should be uploaded separately to your Editorial Manager account in the form of an .xls or .xlsx file. Each table must be accompanied by a title and a description after the Representative Results of the manuscript text.

We used invisible tables to organize our figures. After combining all the panels into one, we got rid of all the tables and all figures and their captions are in-line with rest of the text.

Reviewer #1:

Author's response: We thank Reviewer #1 for the encouraging comments. We've revised our manuscript to address all the mentioned issues to make the protocol clearer to the audience. Manuscript Summary:

The manuscript describes construction and operation of Langmuir's collecting and emitting probes in low pressure plasma in a very descriptive way with typical example in an experimental device. Authors also provide useful information on vacuum production and maintenance, vacuum feedthroughs, plasma production process with hot cathode filaments using dc discharge. Methodically the description is proper and most of the construction and operational processes are well described in manner easily understandable to readers.

In brief the manuscript is well written aiming to educate and facilitate the beginners of basic plasma experiments. It will receive a good readership/viewership among plasma physicists because detail information on most of the equipment used in the experiment are provided. Personally, I enjoyed reading the manuscript.

Major Concerns:

NIL

Minor Concerns:

I have the following comments the authors may consider for incorporation/revision in the final version of the manuscript.

Page 2: The term for average velocity of electrons in equation (2) mentioned (although T_e and m_e are separately defines). Similarly in equation (6) in Page 3 it would be worthy to mention that

ion contribution to the probe current is smaller by a factor of (M_i/2.pi.m_e)^1/2 than that of the electrons.

Response: We rearranged the section of ion current added such comments after the discussion of presheath quasineutrality on page 4:

We note that constant ion flux collected by the probe exceeds the random thermal ion flux due to acceleration along the presheath of the probe and thus ions reach the sheath edge of the probe at the Bohm speed¹⁵, u_B , rather than the ion thermal speed¹⁶. And they have a density equal to the electrons since the presheath is quasineutral. Comparing the ion and electron saturation current in Eqn.5 and 2, we observe that the ion contribution to the probe current is smaller than that of electrons by a factor of $\sqrt{M_i/2\pi m_e}$. Such factor is about 108 in the case of argon plasma.

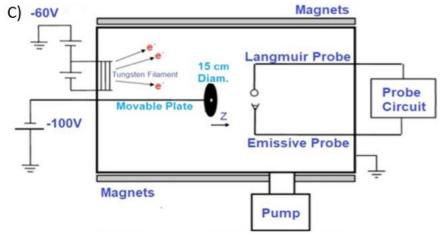
Fig. 4: Plasma density range 10⁵

We couldn't locate the density range in Fig.4. We think the reviewer means the density range "plasma density $10^5 \le n_e \le 10^{12}$ m⁻³" on page 4. We've change the range to $10^{11} \le n_e \le 10^{18}$ m⁻³

Page 7:

2.1.7. The discharge current variation range is mentioned as 0.70 - 1.5 A and referred to the figure 13. However, in figure 13, the specification of the power supply (-60V, 1 A) permits discharge current up to 1 A (max).

Response: The markup on the figure was a bit confusing. "-60 V, 1A" is the typical range for the voltage supply. The actual discharge current is varied by the current/heating supply. We revised the schematic figure to avoid the confusion.



2.2.4. It will be beneficial to mention the kind of gas typically can be used in such experiment in general and the one is used in this experiment in particular. (Because gas injection procedure is narrated here). The range of neutral gas pressure/working pressure at which such experiments are typically done may also be mentioned.

We added some comments about the gas type and working pressure in the protocol section 2.1.5. Now it reads:

Slowly turn the knob of the needle valve to release the gas into the chamber until the pressure reaches the requirement for the experiment. The typical working pressure stem from $10^{-5} \sim 2 \times 10^{-3}$ Torr. Working gases have included argon, xenon, krypton, oxygen, etc.

- 2.2.5. Operation of voltage power supply and current power supply for discharge initiation is not very clear to me.
- * The voltage power supply is meant for discharge (to accelerate the thermionic electrons from the filament to 60 eV).
- * The other one current power supply is for heating the filaments (for 9 filaments of 0.125 mm dia, each 5 cm long) with heating current may be 10-20 A (I suppose).

Response: The original text "through which they are connected to two DC power supplies in series" may be the cause of confusion. The voltage supply is in series with the parallel structure of filaments and the current power supply, as shown in Fig.13. Two power supplies serve different purposes. The voltage supply is used to raise the energy of primary electrons and the current supply is used to provide heating and vary the discharge current. We rewrote the protocol about the power supplies and below is the new version:

Turn on the KEPCO voltage power supply and set the voltage to -60 Volts to provide sufficient electron energy for the maximum ionization cross section of argon. Turn on the SORENSEN heating power supply for the filaments and slowly adjust the level until the discharge current reads the required value.

The sentence [Turn on the current power supply......] may be revised as "Turn on the current power supply and slowly adjust the level until the discharge current (in the voltage power supply) reads the required value (0.7- 1.5 A)".

Response: The edited version is shown below. We specified the voltage and heating power supply in the protocol.

Turn on the KEPCO voltage power supply and set the voltage to -60 Volts. Turn on the SORENSEN heating power supply and slowly adjust the level until the discharge current reads the required value.

Reviewer #2:

Manuscript Summary:

The paper presents the construction and use of Langmuir and emissive probes for low pressure LTPs. A focus is give to the measurement of plasma potential in the pre-sheath with both probes.

Major Concerns:

The inflection point technique is mentioned multiple times as a way to analyze the I-V curves, but how it's done is not discussed in the text.

The reviewer makes an important critical comment. To address it, with respect to the inflection point techniques as touching the Langmuir Probe analysis, we have added the following sentences at the first notice of the technique, p4, fourth paragraph. The colored text is the added text.



To compare potential measurements by both Langmuir probe and emissive probe, plasma potential is also determined by applying the inflection point technique to the Langmuir probe I-V characteristic, as shown in Fig.3. It is generally accepted that the plasma potential is found by finding the probe bias voltage at which the second derivative of the current collected differentiated with respect to the bias voltage, $d^2I/dV^2 = 0$, that is, the peak of the dI/dV curve, with respect to the probe bias voltage. Fig. 3 demonstrates how this maximum in dI/dV, the inflection point of the current-voltage characteristic, is found.

Minor Concerns:

Some overly long sentences.

pg 2, 1st paragraph - "We describes the use of ... relative advantages and weaknesses." Long sentence that would be better to break into 2.

The very long sentence referred to is here:

We describe the use of Langmuir probes and emissive probes in mapping the electrostatic plasma potential from the body of the plasma up to the sheath region of a plasma boundary, which in these experiments is a negatively biased electrode immersed within the plasma, in order to compare the two diagnostic techniques and assess their relative advantages and weaknesses.

It has been changed to two sentences:

We describe the use of Langmuir probes and emissive probes in mapping the electrostatic plasma potential from the body of the plasma up to the sheath region of a plasma boundary, which In these experiments, is created by a negatively biased electrode immersed within the plasma. The measurements permit a comparison of the two diagnostic techniques in order to assess their relative advantages and weaknesses.

pg 2, 2nd paragraph - "Used aboard satellites ... wide range of plasma parameters" Long sentence that would be better to break into 2.

It is a long sentence. It is now three sentences:

During this first century of plasma physics research, dating from Langmuir's discoveries in the 1920's of the medium like behavior of a new state of matter, plasma, the Langmuir probe has proved to have been the single most important diagnostic of plasma parameters. This is true in part, because of its extraordinary range of applicability¹. In plasma encountered by satellites²⁻⁴, in semiconductor processing experiments, ⁵⁻⁸ at the edges of plasma confined in tokamaks, ⁹⁻¹¹ and in wide range of basic plasma physics experiments, Langmuir probes have been used to measure plasma densities and temperatures spanning the ranges $10^8 \le n_e \le 10^{19}$ m⁻³, and $10^{-3} \le T_e \le 10^{2}$ eV, respectively.

pg 3, 2nd paragraph - "while the electron flux ... decreases to negative probe current." Confusing line here. I think it's saying the electron flux, which is larger than ion flux, starts decreasing due to



being repelled by the negatively biased probe. This in turn decreases the negative probe current due to electron collection.

Here is the awkward bit,

while the electron flux to the probe, a much larger flux than the ion flux, begins to be repelled, and this leads a marked decrease to negative probe current.

We agree with the reviewer's suggestion here:

The collected ion current is approximately constant for probe biases more negative than the plasma potential, while the electron flux to the probe decreases for probe bias voltages more negative than the plasma potential. Since the electron saturation current is much larger than the ion saturation current, the total current collected by the probe decreases. And as the probe bias becomes increasingly negative, the drop in current collected is great or small as the electron temperature is cold or hot, as described in Eq. (1a).

Reviewer #3:

Author's response: We thank Reviewer #3 for very thoughtful, very insightful comments. With the exception of #4 and to some extent #5 (see detailed responses), each of these comments require us to change the manuscript to make it better.

The Journal of Visualized Experiments

Manuscript ID: JoVE61804

Title: How to build Langmuir probes and emissive probes for plasma potential measurements in low pressure, low temperature plasma, a comparison of techniques

Author(s): Peixuan Li, Noah Hershkowitz, and Greg Severn

The paper presents an experimental procedure to build Langmuir and emissive probes and use them as low-temperature plasma diagnostics. The paper fits the scope of The Journal of Visualized Experiments but should not be accepted in this form. The authors need to take into consideration the remarks below and re-submit the paper:

1) Please comment the difficulties of using Langmuir Probes when the electron energy distribution function is not Maxwellian (cf. page 2), e.g. nitrogen discharges.

The referee brings up a very important point. There are discharges in which the electron distribution function is not expected to be Maxwellian, including the multi-dipole hot filament discharge plasmas described in this paper. It is an excellent suggestion to make readers aware of examples of plasma discharges in which Non-Maxwellian EEDFs have been observed, for example, in discharges in molecular gases particularly at sufficiently high pressure (typically much greater than 1 mTorr), or in atomic gas discharges of sufficiently high energy, typically rf plasmas, in which inelastic electron-neutral collisions are important. We agree, too, that the comment about



electronegative plasma also needs to be considered and for the same reason, namely, that non-Maxwellian features arise in such plasma and the use of Langmuir probes alone cannot produce valid measurements of negative ion densities. We bundle our responses to items 1 and 3, as shown below, which is an insertion, an new paragraph on page 3, after ``.... beams, etc. See Hershkowitz¹⁴ for more details.''

The discussion here takes up the ideal case of Maxwellian electron energy distribution functions (EEDF). Of course there are many circumstances in which non-idealities arise, but these are not the subject of this work. For example, in materials processing etching and deposition plasma systems, typically RF generated and sustained, there are molecular gas feed stocks that produce volatile chemical radicals in the plasma, and multiple ion species including negatively charged ions. The plasma becomes electronegative, that is, having a significant fraction of the negative charge in the quasineutral plasma in the form of negative ions. In plasma with molecular neutrals and ions, inelastic collisions between electrons and the molecular species can produce dips¹⁵ in the currentvoltage characteristics, and the presence of cold negative ions, cold relative to the electrons, can produce significant distortions¹⁶ in the vicinity of the plasma potential, all of which of course are non-Maxwellian features. We prosecuted the experiments in the work discussed in this paper in a single ion species noble gas (argon) DC discharge plasma, free of these kinds of non-Maxwellian effects. However, a bi-Maxwellian EEDF is typically found in these discharges, caused by the presence of secondary electron emission¹⁷ from the chamber walls. This component of hotter electrons is typically a few multiples of the cold electron temperature, and less than 1 % of the density, typically easily distinguished from the bulk electron density and temperature.

References:

¹⁵Lee, H-C., Lee, J-K., and Chung, W-C., Evolution of the electron energy distribution and E-H mode transition in inductively coupled nitrogen plasma, *Phys. Plasmas* **17**, 033506 (2010).

¹⁶Amemiya, H., Plasmas with negative ions-probe measurements and charge equilibrium, *J. Phys. D: Appl. Phys.* **23** 999 (1990).

¹⁷Andreu, J., Sardin, G., Esteve, J., and Morenza, J., L., Filament discharge plasma of argon with electrostatic confinement, *J. Phys. D: Appl. Phys.* **18** 1339-1345 (1985).

2) When working in low-pressure discharges, the cylindrical probes are regulated by the Orbital Motion Limited regime, developed by Laframboise. Please discuss in the text this issue.

We agree that this is an important omission. Because this paper is about plasma potential measurements, the applicability of the inflection point technique to cylindrical Langmuir probes is the pertinent part of OML theory, and the Laframboise's development of the same using Maxwellian EEDFs, and IEDFs. We have added, at the end of the paragraph (p.5) where the physical characteristics of the cylindrical Langmuir probe are given, the following....

It is important to note that for cylindrical Langmuir probes, for the plasma parameters of these experiments, the radius of the probe tip, r_p , is much smaller than its length, L_p , and smaller than the Debye length, λ_D ; that is, $r_p \ll L_p$, and $0 < r_p/\lambda_D < 1$. In this range of parameters, applying Orbital Motion Limited theory and Laframboise's development of it^{17c} for the case of thermal electrons and ions, we find that for probe bias voltages equal to or greater than the plasma potential, the electron current collected may be parameterized by a function of the form $I_e = I_{eo}(1 + e(V_B - \phi)/T_{eV})^{C_e}$, where the exponent $C_e \sim 0.4$. The important point here is that for values of this exponent less than unity, the inflection point method for determining the plasma potential, as described in the paragraph above, applies to cylindrical Langmuir probes too.

²²Mausbach, M., Parametrization of the Laframboise theory for cylindrical Langmuir probe analysis, *J. Vac. Sci. Technol. A*, **15** 2923-2929 (1997).

3) In electronegative plasmas, e.g. oxygen discharges, how Langmuir probe theory should be modified to consider the presence of negative ions?

See response to item 1) above.

4) A video showing the Langmuir and Emissive probes in operation would improve the quality of the paper.

The reviewer is right of course. It is the point of the work under review to lead to the creation, with the help of the JOVE production staff, of such a video.

5) Please discuss the limitations to use Langmuir and emissive probes.

Limitations concerning the use of Langmuir and emissive probes are discussed in the last half of the discussion section of the manuscript, pages 26 and 26, beginning with the sentence, "Langmuir probes, as is true of any diagnostic, have important limitations,....". However, we have omitted something of obvious importance, something that Hutchinson points out, namely, that the use of them is, "limited to plasmas that the probe itself can survive". We have added the sentence at the end of that section:

Finally we mention one last limitation common to both probe techniques, namely, that if the plasma is too dense and hot the probes cannot mechanically survive¹⁴, leading to the upper limits quoted in the introduction.

6) For single Langmuir probe the area of the reference electrode is important when compared with the area of the probe. Please comment in the text of the manuscript.

We agree that this is an important issue! We have added a sentence recognizing this issue in a passage detailing the size of the Langmuir probe tip that we typically use, near the bottom of page 4.... We include the sentence below:

Of course we could collect more current and get bigger signals with a larger disc. However, in order for the analyses above to apply, the area of the probe, A_p must be kept smaller than the electron loss area of the chamber, A_w , satisfying 17b the inequality $A_p \leq A_w$.



Reference

²¹Barnat, E., V., Laity, G., R., and Baalrud, S., D., Response of the plasma to the size of an anode electrode biased near the plasma potential, *Physics of Plasmas* **21**, 103512 (2014).

In conclusion, the paper is interesting, but authors should consider the remarks listed above in the new version of the manuscript.