

Renovations to a Plasma Teaching Laboratory

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With the aid of a NSF Instrumentation and Laboratory Improvement Program Grant, replacement of dated equipment and the implementation of new experiments have upgraded and diversified our 25-year-old plasma laboratory course. The course teaches seniors and first-year graduate students methods of plasma production as well as plasma diagnostics for applications ranging from fusion to semiconductor processing. This paper describes four new experiments as well as the highlights of some of the equipment upgrades.

EDUCATIONAL SUMMARY

1. The paper describes new training tools or laboratory concepts/instrumentation/experiments in nuclear or electrical engineering.
2. The paper describes new equipment useful in a plasma laboratory or a general senior lab course.
3. Seniors and first-year graduate students are involved in the use of the equipment.
4. This contribution includes four new experiments.
5. The experiments were added to the number of experiments performed and in some cases replaced some labs.
6. Which texts or other documents accompany the presented materials? We have lab handouts for each experiment.
7. The concepts presented have been tested in the classroom? The students really enjoyed the new experiments and found them more relevant to current plasma research.

INTRODUCTION

THE DEPARTMENT of Nuclear Engineering at the University of Michigan has had a strong program in plasma and fusion for both undergraduate and graduate students for over 30 years. Over 25 years ago, with the help of an Atomic Energy Commission grant, a plasma course was developed to introduce experimental concepts in

plasma science to senior undergraduates and first-year graduate students. Many of the experiments were based upon surplus equipment available at the time. The primary goal of the course has been to teach methods of plasma production as well as plasma diagnostic techniques, particularly methods applied to fusion technology as well as basic plasma physics. The changes made to this course have allowed our undergraduates to obtain the skills necessary to continue their careers particularly in graduate school or industry, by providing them with the skills needed easily to work on current topics of plasma physics.

The plasma research field has changed dramatically over the past several years and has found new industrial applications, particularly plasma processing of materials [1, 2]. For example, deposition of thin films and etching of semiconductors have centered around glow discharges, which are relatively easy to produce in the lab compared to dense plasmas such as those found in fusion devices. Also new diagnostics which are based upon lasers are becoming relatively common [3]. We felt that it was necessary to upgrade the experiments to reflect the evolving field of plasma physics [4, 5]. The plasma lab course introduce four new experiments to achieve this goal: (i) probe measurements of the electron energy distribution function; (ii) laser/Schlieren photos of shock waves produced by a pulsed plasma; (iii) a laser deflection technique experiment for determining hot gas densities; and (iv) capacitively coupled RF plasma generation experiment such as those used in industrial reactive ion etching. The current set of experiments, including these new experiments is listed in Table 1.

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Table 1. List of experiments

1. Vacuum techniques
2. DC breakdown and the Paschen curve
3. RF parallel plate discharges at 13.56 MHz
4. Langmuir probes
5. Optical emission spectroscopy (OES) for species identification
6. Electronic temperature from OES
7. Microwave interferometry
8. Microwave cavity perturbation
9. Thermionic emission
10. Schlieren photography
11. Laser deflection method
12. Electron energy distribution function from probes

EXPERIMENTS

Electron energy distribution function measurements

A particle energy distribution function is an important concept, not only in plasma physics but in many areas of nuclear engineering (neutron distribution functions) and thermal sciences. Probe techniques such as the traditional Langmuir probe can be very useful for obtaining the electron and ion density and the electron temperature in low-pressure gas discharges if the electron energy distribution function (eedf) can be described by a Maxwellian distribution function. This, of course, may not be the case. There are very few techniques available for measuring energy distribution functions in plasmas. A variation to the traditional Langmuir probe technique can measure electron energy distribution functions with fairly inexpensive and relatively easy modifications.

The electron energy distribution function can be measured with a Langmuir probe using the Druyvesteyn [6] method. A Langmuir probe is simply a small wire inserted into the plasma. Various voltages are applied to the wire and the collected current is measured. Several analysis techniques can be used to convert the current-voltage data to plasma parameters. Druyvesteyn showed that the eedf, $g(E)$, is directly proportional to the second derivative of the current measured by the probe with respect to the voltage. This can be shown with an analysis found in [7] and [8].

$$g(E) \propto \sqrt{E} \frac{d^2 I_e}{dV_p^2} \quad (1)$$

where $E = V_s - V_p$, and $V_s =$ plasma space potential, $E =$ the electron energy, V_p is the probe potential and I_e is the measured current. Thus by measuring $d^2 I_e / dV_p^2$ as a function of probe potential, V_p , the eedf can be determined.

There are several techniques which can be used to determine $d^2 I_e / dV_p^2$. The method we use employs the standard Langmuir probe biased with various DC voltages with a small AC component

added. The electron current measured by the probe is thus:

$$I_e(t) = \text{function} [V_p + v_p(t)] \quad (2)$$

If the AC component $v_p(t) = A \sin \omega t$ is small for all values of t compared to V_p , then the current can be expanded using a Taylor series expansion around V_p to give:

$$\begin{aligned} I_e(t) - I_e = & \left(\frac{A^2}{4} \frac{d^2 F(V_p)}{dV_p^2} + \dots \right) \\ & + \left(A \frac{dF(V_p)}{dV_p} + \dots \right) \sin \omega t \quad (3) \\ & + - \left(\frac{A^2}{4} \frac{d^2 F(V_p)}{dV_p^2} + \dots \right) \cos 2\omega t + \dots \end{aligned}$$

The higher-order terms of the bracket in front of $\cos 2\omega t$ are small (8.3%) when $A \approx 1$ V (see [8]). Thus if we can determine the amplitude of the $\cos 2\omega t$ term, we can determine $d^2 F(V_p) / dV_p^2$ which in turn is related to the energy distribution function as

$$\frac{d^2 F(V_p)}{dE^2} \propto \frac{g(E)}{\sqrt{E}} \quad (4)$$

Note that it is very important to determine accurately V_s so that the electron energy, $E = V_s - V_p$, can be calculated. A common method is to determine the potential value where $d^2 I / dV^2$ equals zero, but this is only truly accurate for Maxwellian distributions. The other method is to determine V_s from the standard Langmuir probe technique.

In this experiment, a small AC voltage of about 1 V and 1 kHz is applied to the Langmuir probe using a sine wave generator. A lock in amplifier is used to measure the amplitude of the $\cos 2\omega t$ term at 2 kHz. Note that the plasma can be quite noisy and sometimes there are some fluctuations in the measured values. The circuit given in Fig. 1 is based on [9].

A convenient way to fit the data is to the following form (see [10]):

$$\frac{\ln g(E)}{\sqrt{E}} = \ln a - bE^x \propto \ln \left[\frac{d^2 I_e}{dV_p^2} \right] \quad (5)$$

x can be determined from a least-square fit to $d^2 I_e / dV_p^2$ as a function of E until the maximum correlation coefficient is found. $a =$ logarithm of intercept and $b =$ slope of the least squares fit with the optimal x . Figure 2(a) shows the lock in amplifier signal as a function of probe voltage for a nitrogen plasma at 7 torr. This plasma was generated between two brass electrodes, 3 in. in diameter, with a spacing of 0.5 in. and an applied DC voltage of 100 V (3 mA). Figure 2(b) shows the natural logarithm of the eedf as a function of energy

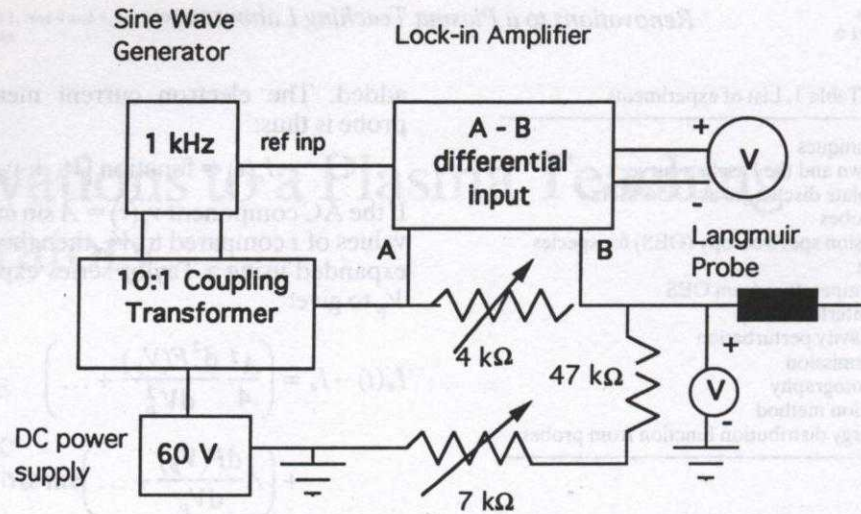


Fig. 1. Circuit diagram for the electron energy distribution function experiment.

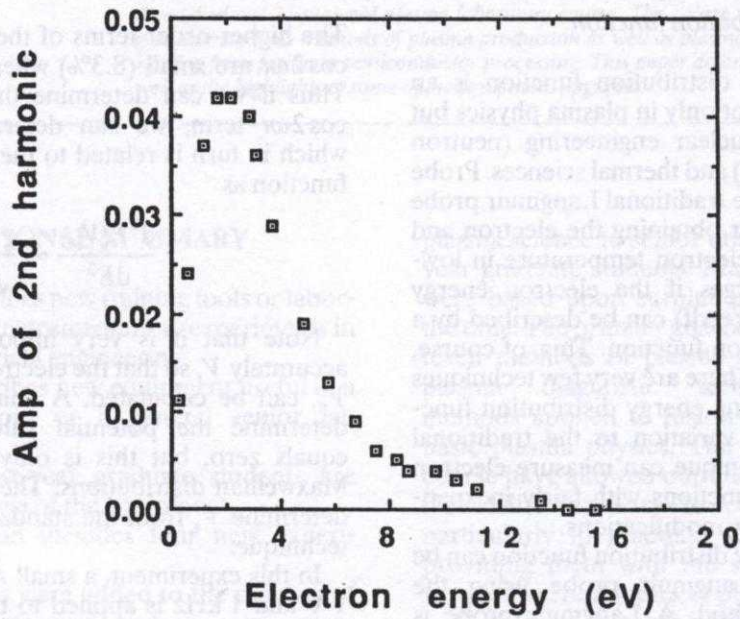


Fig. 2(a) Lock-in amplifier signal as a function of applied probe voltage for a nitrogen plasma at 7 torr.

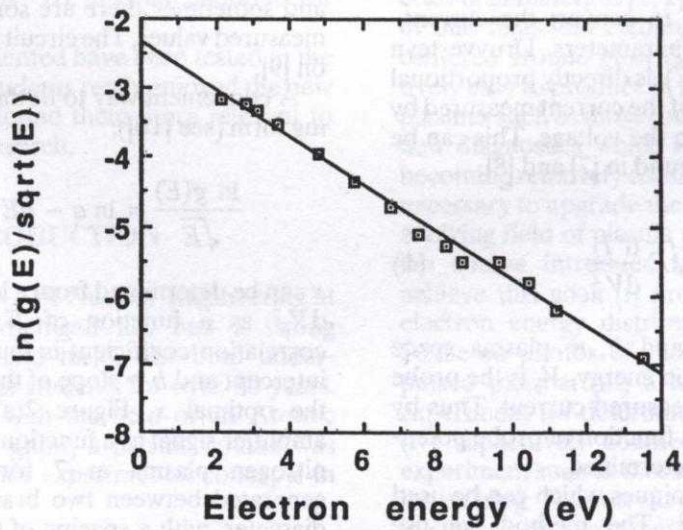


Fig. 2(b) Logarithm of the lock-in amplifier signal of (a) divided by the square root of the electron energy as a function of the electron energy.

assuming that x in equation (5) = 1. The straight-line fit indicates an excellent agreement with what would be expected from a Maxwellian distribution function.

Laser deflection technique

Reduced density channels in atmospheric pressure air have several important applications, e.g. to study lightning, particle beams transport for inertial fusion reactors, and as a means of enhancing electron beam propagation. There are several diagnostic techniques [4] that have traditionally been used to measure the radial density profiles of neutral density channels: Schlieren photography, laser holographic interferometry and Mach-Zehnder interferometry. In general, these methods are difficult to field, are insensitive to small changes in density and often require the use of relatively expensive equipment. The laser deflection technique provides an inexpensive and relatively easy to implement technique for measuring small changes in neutral gas densities.

This experiment also provides the student with an introduction to the physics of reduced density channels and to diagnostic techniques which use changes in index of refraction to infer density information. Whereas many experiments suitable for students are steady state in nature, this experiment introduces the student to fast-pulsed techniques.

A spark is generated between two sewing needles about 1 cm apart. An automobile battery and ignition system generate the spark. The hot gas channel is produced by the spark and deflects a CW He-Ne laser. A positive-sensitive detector measures these small deflections (microradians). An inexpensive x - y positioner moves the laser along the width of the spark. An Abel inversion program converts the angular deflection data as a function of width to gas density as a function of spark radius. Using the Rankine-Hugoniot equations, students learn about the hydrodynamics of heated channels. The details and the results of this experiment have been described previously [11].

Laser Schlieren photography

Schlieren photography is an extension of laser beam deflection. In the conventional deflection experiment, a single ray is deflected when there is a density gradient normal to this ray. In Schlieren photography, however, the source laser beam is expanded and collimated such that it is about the same size or larger than the density perturbation being investigated. Essentially, an infinite set of rays for deflection has been created.

Both methods respond linearly to the density gradient. Laser deflection is quantitative but provides density information only at one location. It is difficult to obtain quantitative density information from Schlieren photos but they provide time-dependent information in two dimensions, showing clearly the size and shape of such hydrodynamic features as shock waves or turbulence.

Both Schlieren and laser deflection use the fact that light travels slower in regions of higher density. This point is illustrated by equations (6) and (7a, b):

$$c = (1/n)c^* \quad (6)$$

where c = speed of light in media, c^* = speed of light in vacuum, n = index of refraction.

$$n - 1 = kr \quad (7a)$$

where k = Gladstone-Dale constant and r = density.

$$n = [1 - (N_e e^2 \lambda^2) / (2pm_e c^2)]^{1/2} \quad (7b)$$

where N_e = number density of electrons, e = electric charge, λ = wavelength of light rays being deflected, m_e = electronic mass and c = speed of light in vacuum.

Equation 7(a) describes the index of refraction for ions and neutrals; equation 7(b) describes the index of refraction for electrons. Figure 3 shows light beam wave fronts at various times t_i in a density gradient which increases in the positive x direction. The fronts travel slower in the region of higher density, resulting in a curved front. The rays which make up the beam are normal to the front

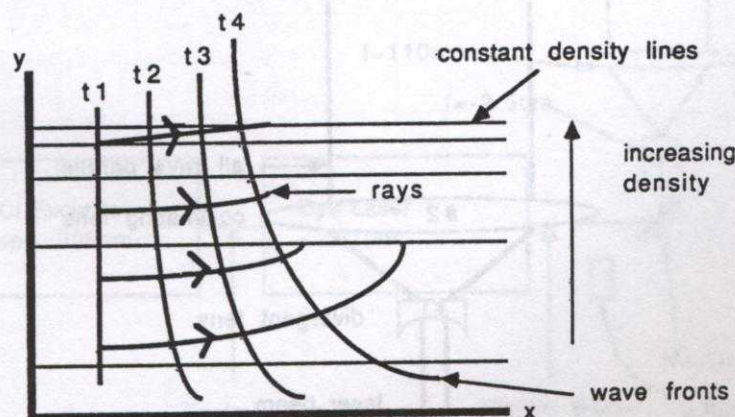


Fig. 3. Wave fronts at times t_i and rays bending through a density gradient.

and clearly bend in the direction of increasing density gradient.

As seen in Figure 4, a source is expanded in lens no. 1 and collimated with the desired magnification in lens no. 2. A test section is placed at aa. Approximately one focal distance away, one introduces a focusing lens, no. 3. The undeflected rays in aa come to a focus through an iris bb. The image of the test section aa is located on the film through focusing lens no. 4. If all the optics are properly aligned, all the light from the source passes through the iris and illuminates the film.

The details of the experimental details can be obtained from the authors and references [12-15].

An excimer-pumped dye laser takes Schlieren photographs of the spark generated between two sewing needles about 1 cm apart (see Fig. 5). An automobile battery and ignition system generate the spark. The timing of the spark and the laser are coordinated so that the laser is fired at specific times relative to the spark. A comparison of the photos taken at different times relative to the initiation of the spark gives the speed of the shock. See Fig. 6 for results of a typical spark.

Capacitively coupled RF plasma generator

In the early 1970s it was discovered that plasma could effectively etch integrated circuit patterns in

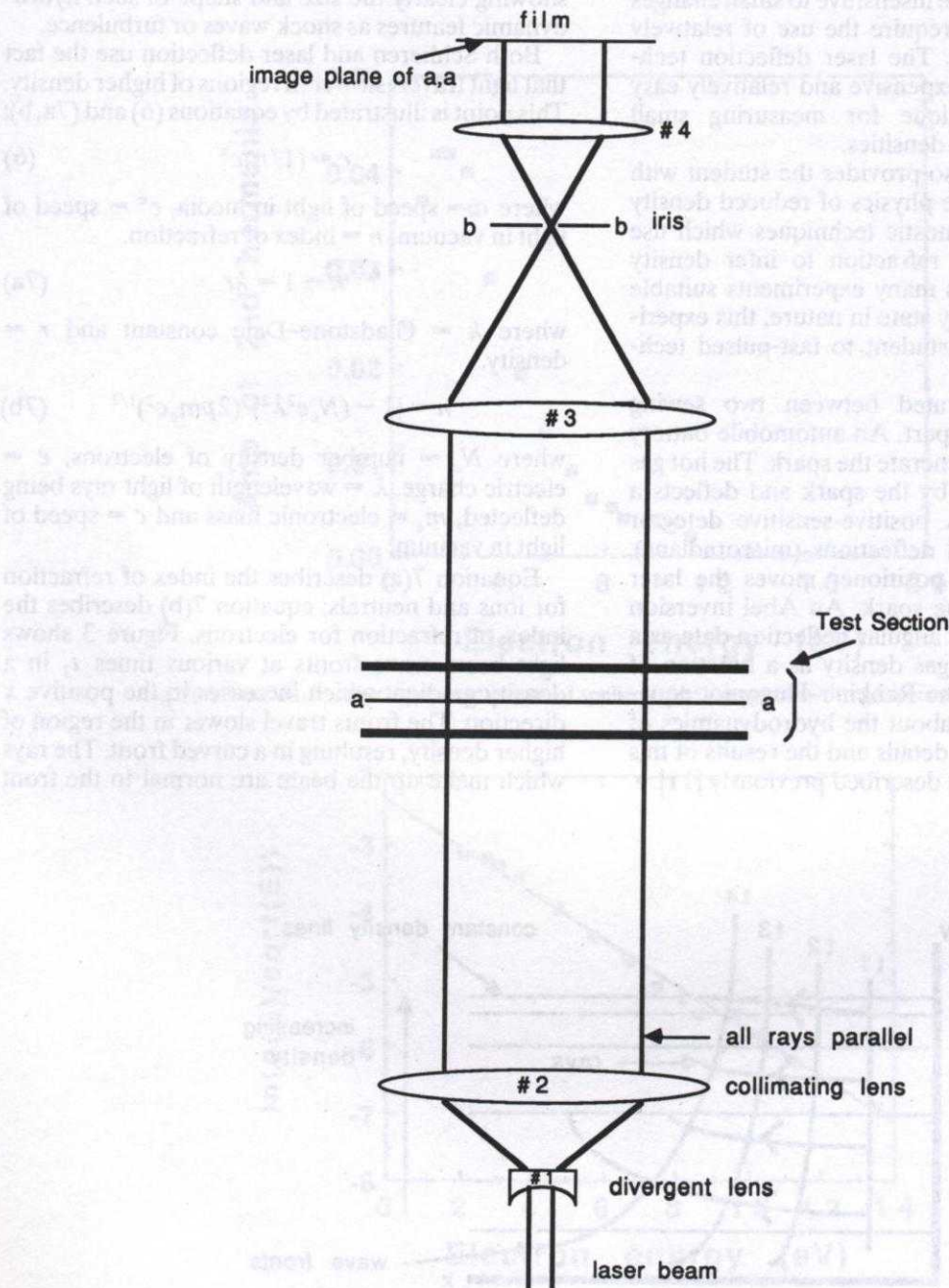


Fig. 4. Schematic of optical configuration for Schlieren photography.

semiconductor materials [2, 16]. Previously, his etching was done with chemical processes that used hot, corrosive, hazardous acids. In addition to the safety and environmental advantages of plasmas, plasmas were found to result in a better etch. Plasmas are now a routine element used in the manufacturing of integrated circuits.

There are many designs of plasma chambers used in industrial processing. A very common design, known as a reactive ion etcher, consists of two parallel metal electrodes where the bottom electrode is driven by radio frequency power at 13.56 MHz. In conjunction with a research project, we built such a capacitively coupled radio frequency plasma cell (see Fig. 7). Details of the

experimental design are given in [17]. This particular design (10 cm showerhead aluminum electrodes, 2.5 cm apart) was developed to provide a standardized reference for researchers studying industrial plasma processes such as reactive ion etching. Ideally, results obtained from this design should always be the same, independent of the individual reactor. Students can easily measure many of the important baseline parameters such as current and voltage.

When the system was first completed, the students helped characterize the electrical properties of reactor both with and without a plasma present. The theory is quite complex [18], although the measurements are fairly easy to make. The students

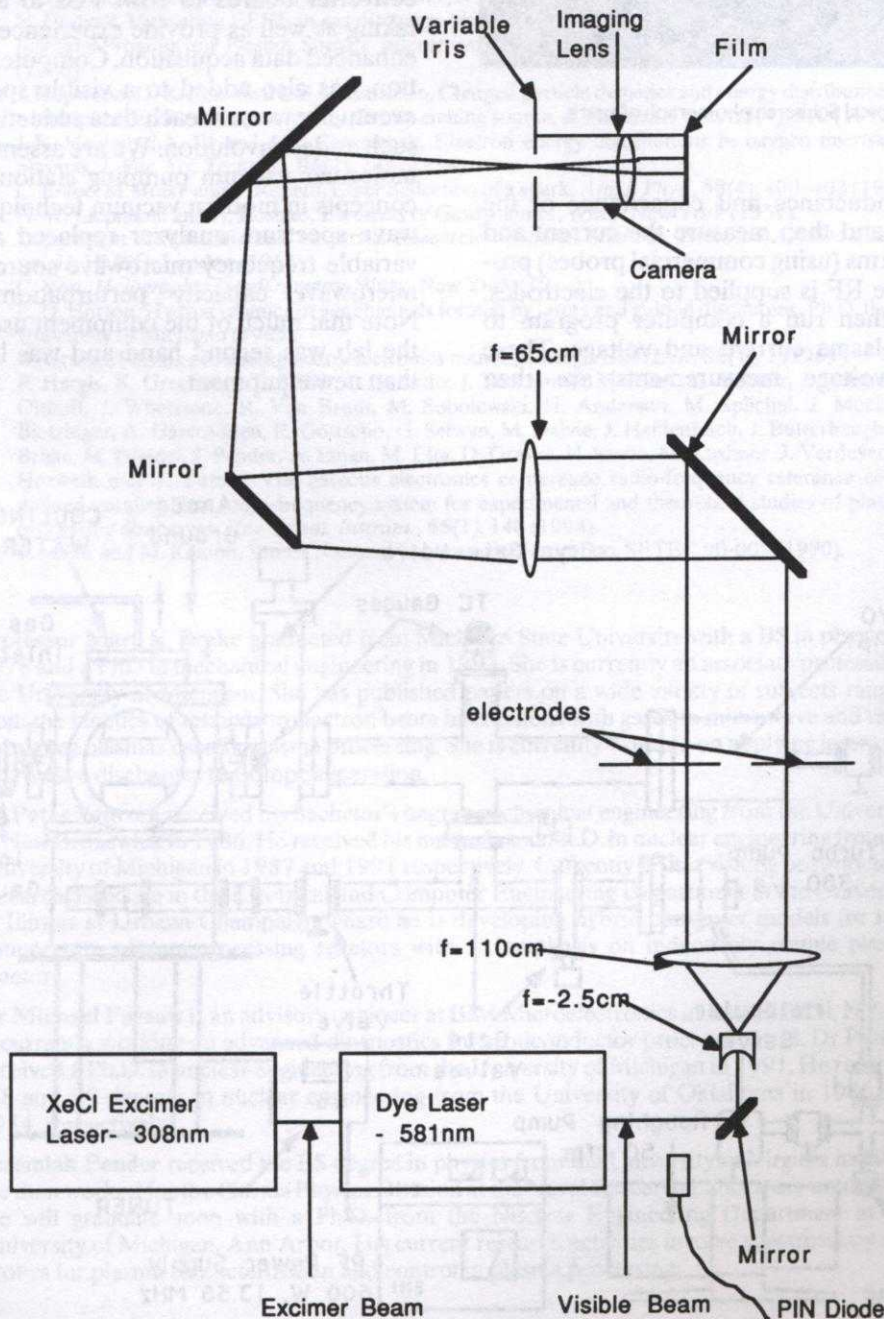


Fig. 5. Schematic for Schlieren photography.

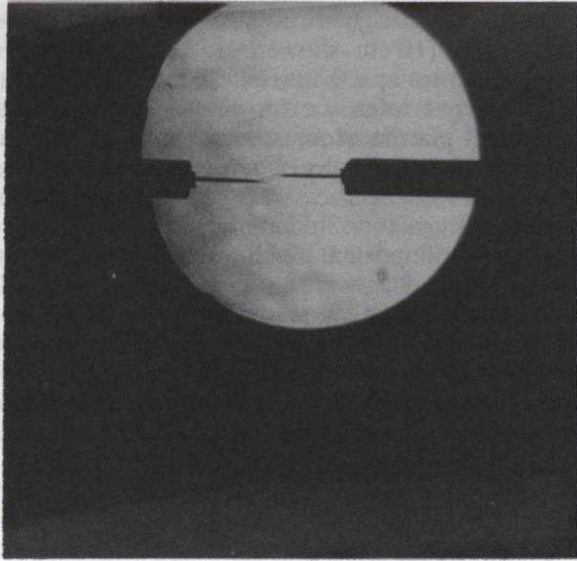


Fig. 6. Typical Schlieren photograph of spark.

measure the inductance and capacitance of the reactor circuit and then measure the current and voltage waveforms (using commercial probes) produced when the RF is supplied to the electrodes. The students then run a computer program to calculate the plasma current and voltage. These current and voltage measurements are then

compared to those made by researchers at other institutions.

During the past year, students have studied the manufacturing of integrated circuits by etching a pattern into polysilicon using a CF_4/O_2 plasma. The students used optical emission spectroscopy to monitor the progress of the etch. Specifically they measured the time rate of change of fluorine lines to help them determine when to stop the etch process. When the polysilicon has been etched away, the SiO_2 layer is exposed and the fluorine signal increases. In the future years we plan to add other diagnostics to this system.

Other upgrades

Other upgrades include the addition of A/D converter boards to IBM PCs to automate data taking as well as provide experience in computer-enhanced data acquisition. Computer data acquisition was also added to a visible spectrograph so eventually we can teach data reduction techniques such as deconvolution. We are assembling a turbomolecular vacuum pumping station to introduce concepts in modern vacuum techniques. A microwave spectrum analyzer replaced an antiquated variable frequency microwave source used in the microwave capacity perturbation experiment. Note that much of the equipment used to upgrade the lab was second hand and was less expensive than new equipment.

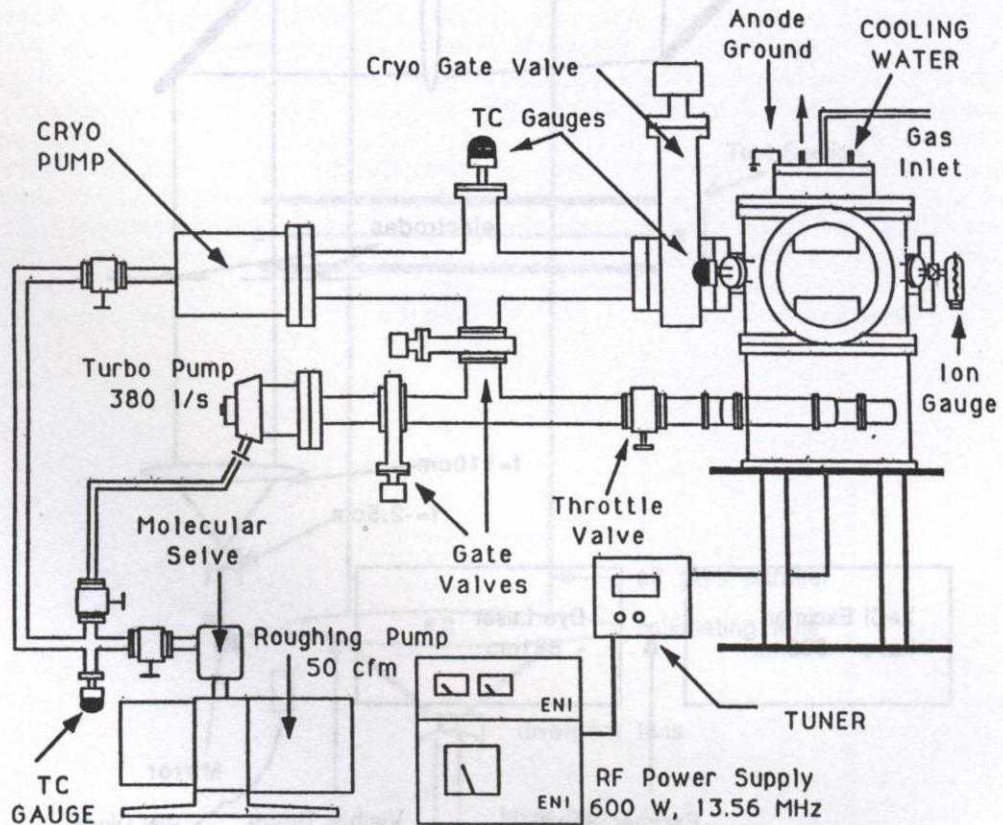


Fig. 7. Schematic of the RF parallel plate reference reactor.

SUMMARY

The new experiments and equipment both update and diversify our plasma lab course. Students learn basic plasma laboratory techniques that are important to a wide variety of plasma

applications from fusion to semiconductor processing.

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REFERENCES

1. B. Chapman, *Glow Discharge Processes*, Wiley, New York (1980).
2. D. Manos and D. Flamm (eds), *Plasma Etching: An Introduction*, Academic Press, New York (1989).
3. W. Demtroder, *Laser Spectroscopy*, Springer-Verlag, New York (1982).
4. R. Huddleston and S. Leonard, *Plasma Diagnostic Techniques*, Academic Press, New York (1965).
5. I. H. Hutchinson, *Principles of Plasma Diagnostics*, Cambridge University Press, New York (1987).
6. M. J. Druyvesteyn, *Der Niedervoltbogen*, *Z. Phys.*, **64**, 781 (1930).
7. J. D. Swift and M. J. R. Schwar, *Electric Probes for Plasma Diagnostics*, Elsevier, New York (1969).
8. V. Godyak, Measuring EEDF in gas discharge plasmas, lecture given at a NATO Advanced Study Institute Program on Plasma Surface Interaction and Processing of Materials, Alicante, Spain (1988).
9. J. Hopwood, D. K. Reinhard and J. Asmussen, Charged particle densities and energy distributions in a multipolar ECR microwave-cavity plasma etching source, *J. Vac. Sci. Tech.*, **A8**(4), 310 (1990).
10. J. R. Heidenreich, III and J. R. Paraszczak, Electron energy distributions in oxygen microwave plasmas, *J. Vac. Sci. Technol.*, **B6**(1), 288 (1988).
11. C. Enloe, M. Brake and T. Repetti, Laser deflection of a spark, *Am. J. Phys.*, **58**(4), 400-403 (1990).
12. H. W. Liepmann and A. Roshko, *Elements of Gasdynamics*, Wiley, New York (1957).
13. Department of Scientific and Industrial Research, *Schlieren Methods, Notes on Applied Science no. 31*, HMSO, London (1963).
14. C. Vest, *Holographic Interferometry*, Wiley, New York (19).
15. L. D. Horton, Hydrodynamics of gas channels formed by lasers and guided discharges, Ph.D. thesis, University of Michigan (1985).
16. D. Graves, Plasma processing microelectronics manufacturing, *AICHE J.*, **35**(1), 1 (1989).
17. P. Hargis, K. Greenberg, P. Miller, J. Garardo, J. Torczynski, M. Riley, G. Hebner, J. Roberts, J. Olthoff, J. Whetstone, R. Van Brunt, M. Sobolewski, H. Anderson, M. Spichal, J. Mock, P. Bletzinger, A. Garscadden, R. Gottscho, G. Selwyn, M. Dalvie, J. Heidenreich, J. Butterbaugh, M. Brake, M. Passow, J. Pender, A. Lujan, M. Elta, D. Graves, H. Sawin, M. Kushner, J. Verdeyen, R. Horwath and T. Turner, The gaseous electronics conference radio-frequency reference cell: a defined parallel-plate radio-frequency system for experimental and theoretical studies of plasma-processing discharges, *Rev. Scient. Instrum.*, **65**(1), 140 (1994).
18. P. Miller and M. Kamon, Sandia National Laboratories Report no. SETEC 90-009 (1990).

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Dr Michael Passow is an advisory engineer at IBM Microelectronics in East Fishkill, NY. He is currently working on advanced diagnostics for semiconductor process control. Dr Passow received a Ph.D. in nuclear engineering from the University of Michigan in 1991. He received MS and BS degrees in nuclear engineering from the University of Oklahoma in 1986 and 1984, respectively.

Jeremiah Pender received the BS degree in physics from the University of Virginia in 1985. He then worked for the Plasma Physics Division at the Naval Research Laboratory until 1988. He will graduate soon with a Ph.D. from the Nuclear Engineering Department at the University of Michigan, Ann Arbor. His current research activities involve spectroscopy and probes for plasma characterization and control in plasma processing.