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Microwave Powered Microplasmas: Applicator Designs, Characteristics, and Applications

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Backgrounds

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- Research group focuses on microwave plasma-assisted material processing with particular emphasis on the synthesis of diamond
- Previous students studied on the scaling properties of microwave plasmas

Motivations

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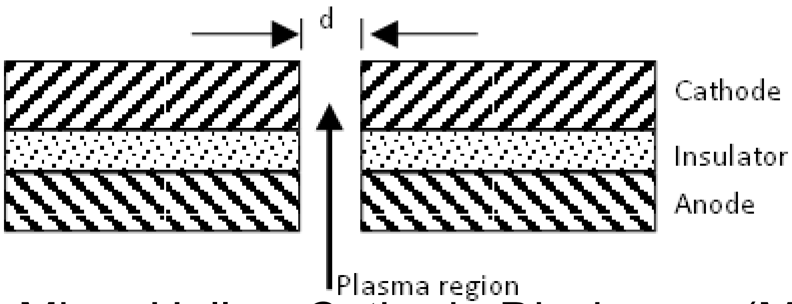
The field of microplasmas is an emerging technology within the past decade.

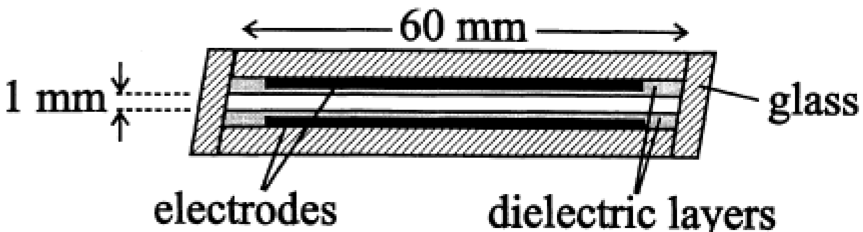
Various types of microplasma sources (DC, AC, RF, etc.) designed by many different research groups have diverse applications (sterilization, display panels, lighting, ozone production, microthrusters, spectroscopy, etc).

Only a few studies can be found in the literature on microwave generated microplasmas.

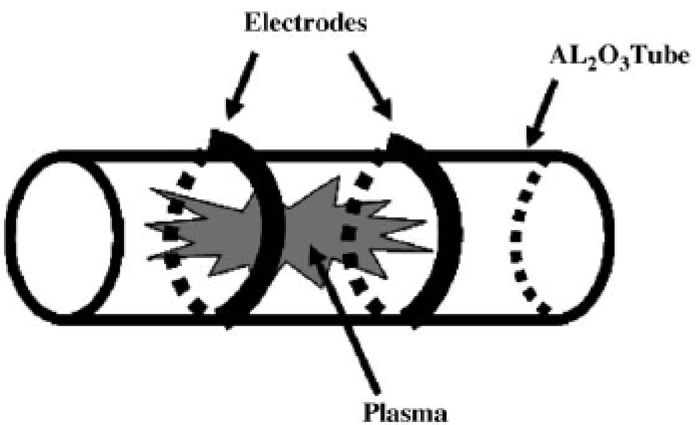
Non-microwave Microplasma Sources



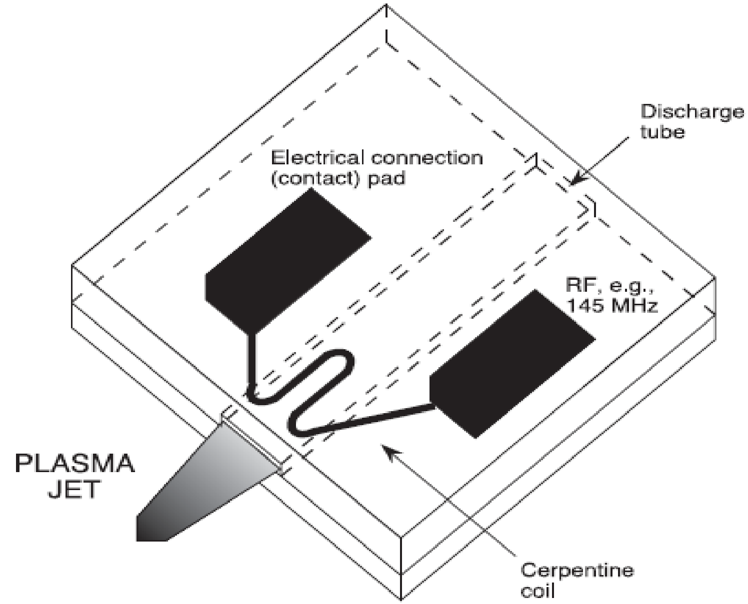
Micro Hollow Cathode Discharge (MHCD)
DC applicator



Dielectric Barrier Discharge (DBD)
AC – RF frequency (5 – 20 kHz)



Capillary Plasma Electrode (CPE)
AC – RF frequency



Miniature Inductively Coupled Plasma (mICP). RF – Microwave frequency

Miniature Microwave Plasma Sources

Why microwave?

- No electrodes
- High density
- Can generate surface wave plasmas (SWP's)

Potential applications of miniature microwave discharges include:

- Plasmas source for micro-system (chemical or biological analysis)
- Treatment or sterilization of tubes and other small parts
- Local area materials processing
- Portable, low temperature sterilization source
- Micro-thrusters for space propulsion

Objectives

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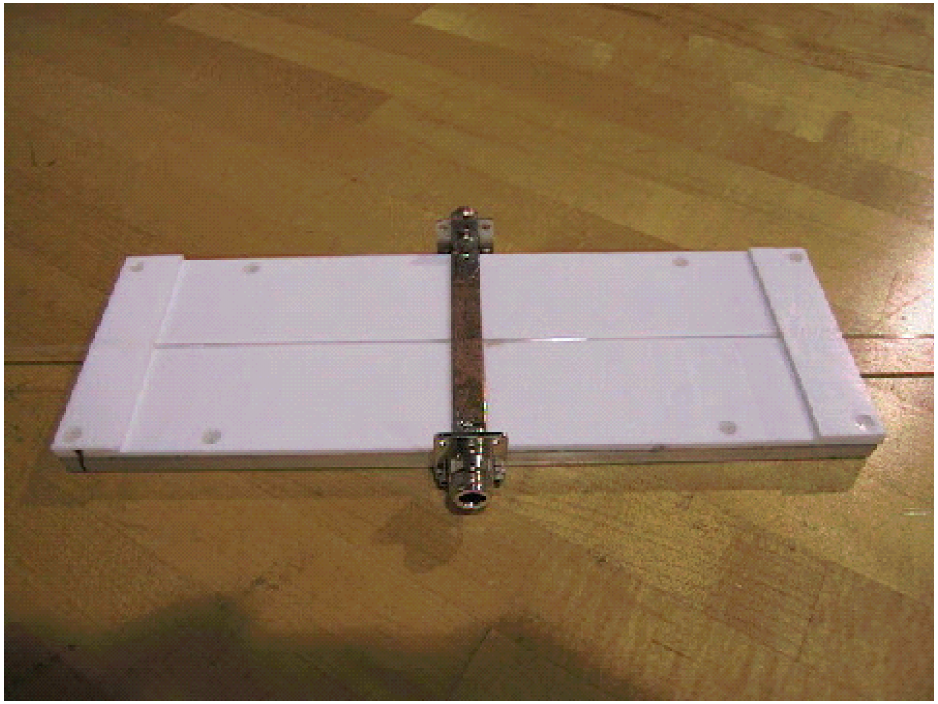
Explore microwave generated microplasmas

- Miniature microwave plasma sources mostly are based on:
  - Modified microstrip transmission lines
  - Modified waveguide or cavity
- Study the plasma behavior and the basic characteristics
- Demonstrate potential applications

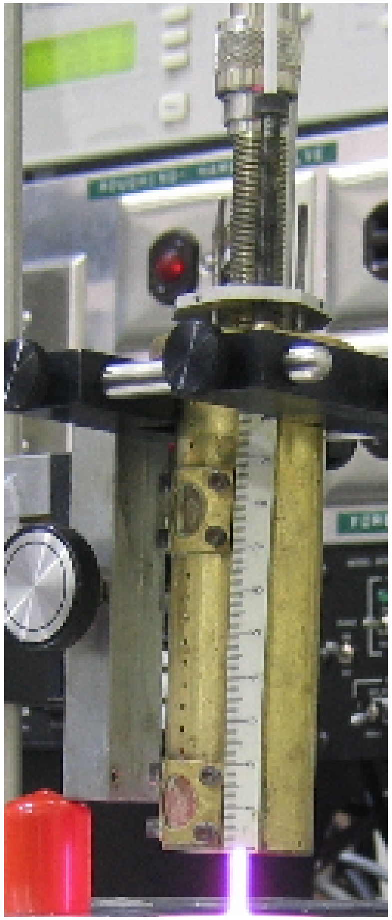
Presentation Outline

- Background, motivation, and objective
- Plasma source designs:
  - Microstrip based and coaxial cavity based
- Plasma diagnostics
  - Optical emission spectroscopy (OES) and double Langmuir probe (DLP)
- Applications
  - Etching of silicon and diamond, UV exposure of photoresist
- Summary

Plasma Source Designs

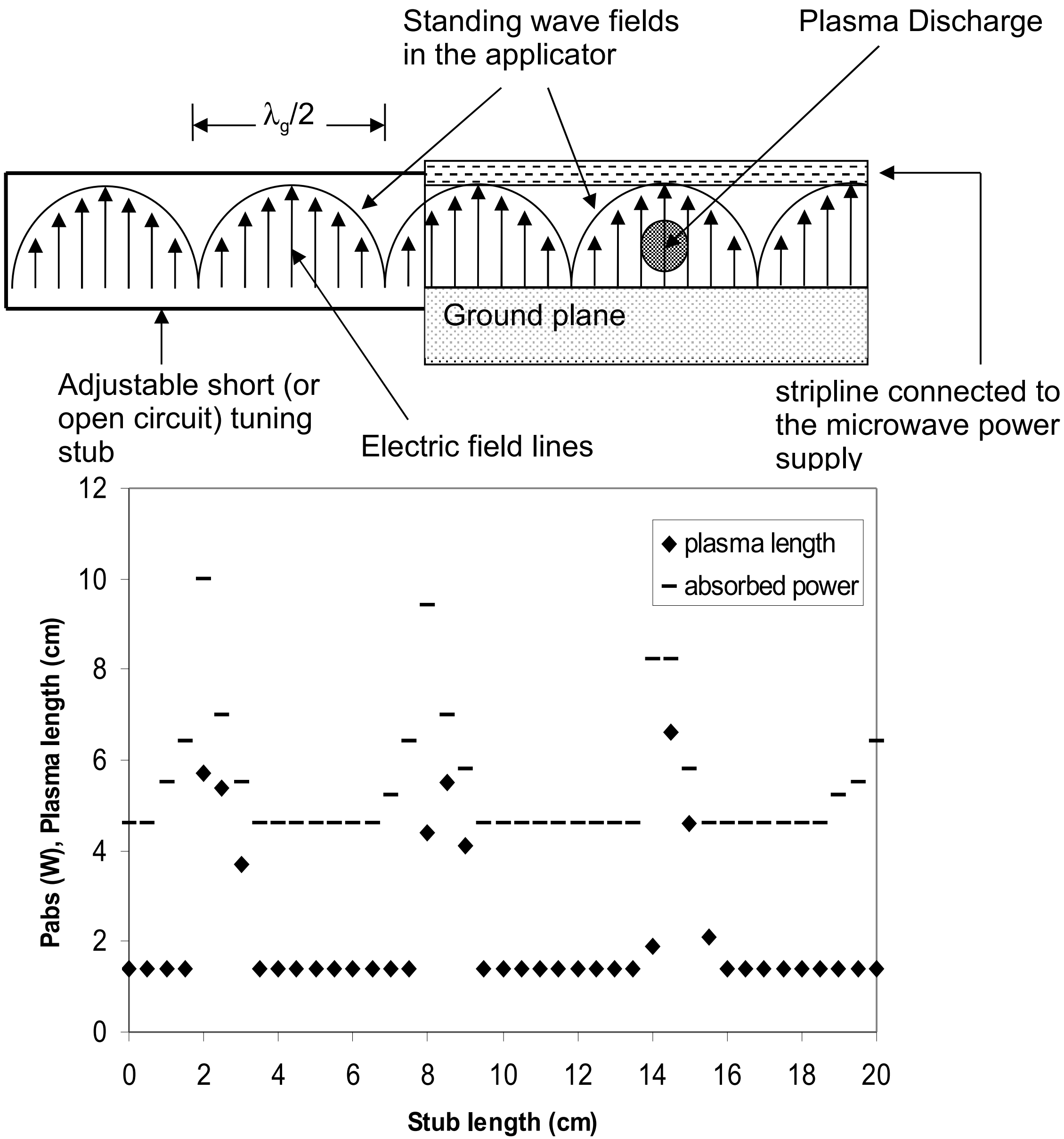


Microstrip applicator



Foreshortened coaxial cavity applicator

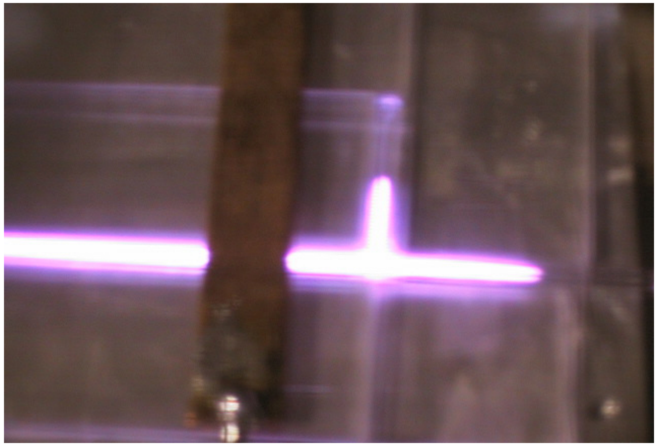
Microstrip Applicator Tuning



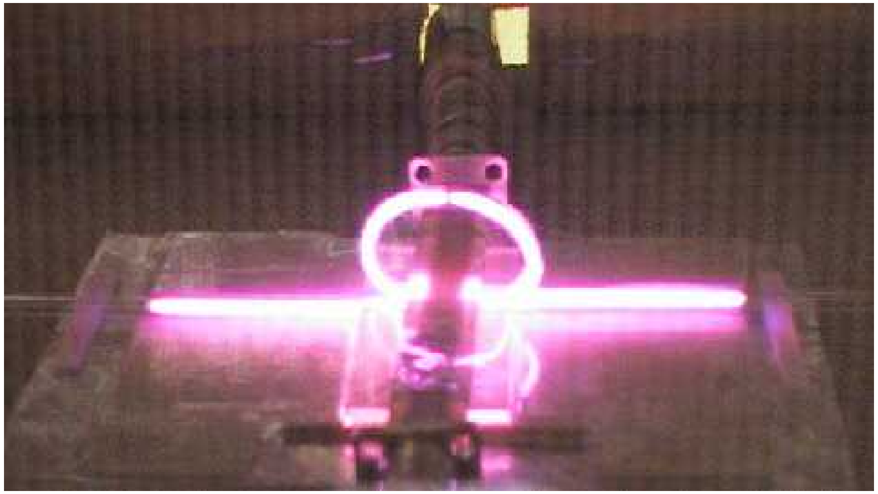
- The microwave energy creates a standing wave along the microstripline and the adjustable stub
- The maximum discharge length is achieved when the absorbed power is at the maximum
- The distance between the maximum power transfers to the load is at $\lambda_g/2$ or 6.12 cm.

Argon, 50 sccm, 20 Torr, 13 W

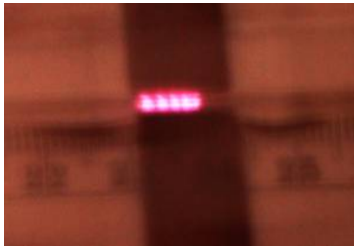
Plasma Behavior



Ar, 1 Torr inside 2 mm tube i.d. with branch



Ar, 1 Torr inside 2 mm tube i.d. modified to create a loop



Ar, 760 Torr inside 1 mm tube i.d.
P_{abs} = 20 W.

Plasmas have been created in tubes of various inner diameters including:

- 200 micrometers
- 450 micrometers
- 1 millimeter
- 2 millimeter

Plasma Volume and Power Density

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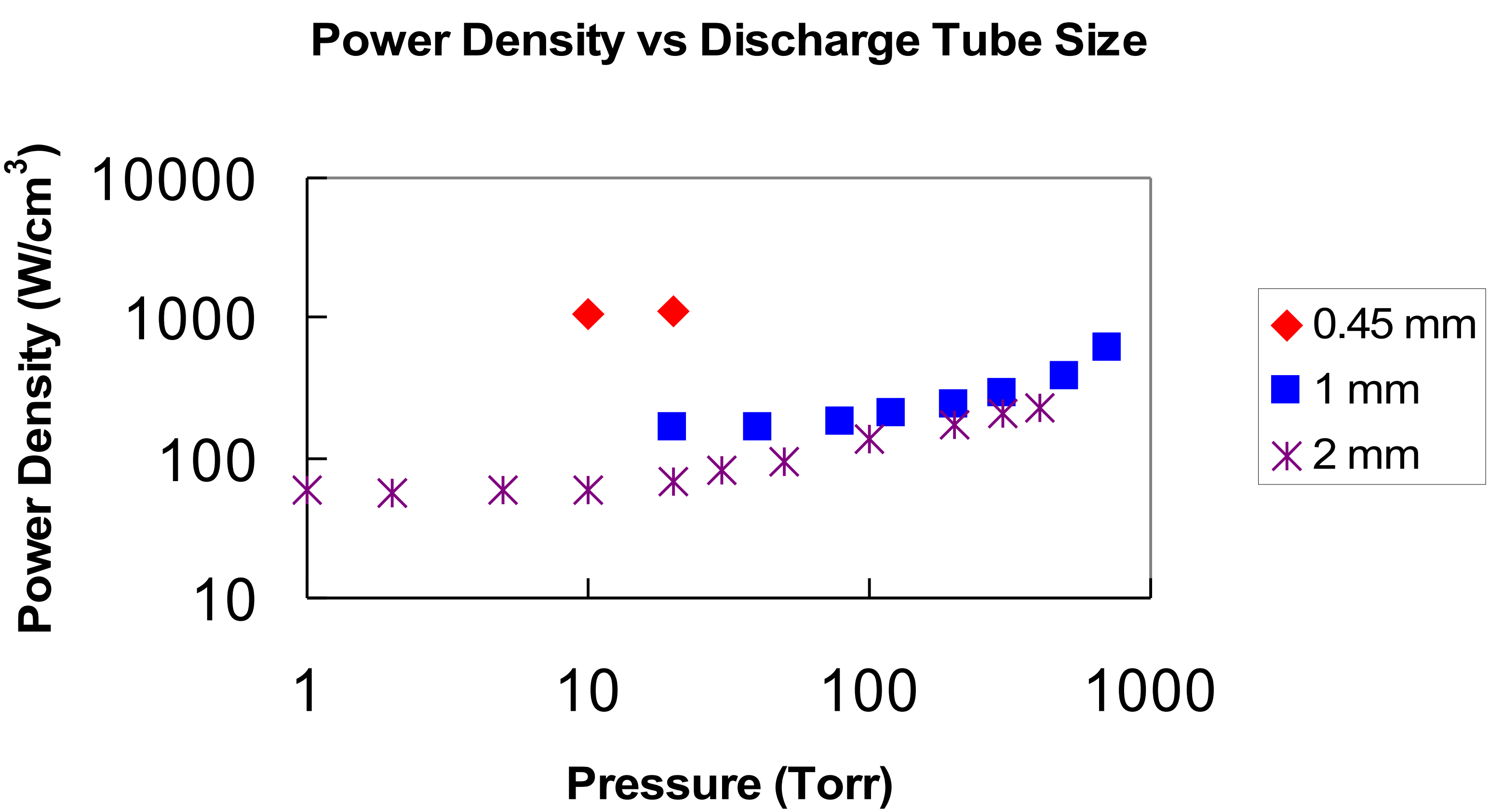

Ar, Microwave power = 30W

CW Microwave Plasma Discharges in tubes less than 1-2 mm in diameter require significantly higher microwave excitation power densities.

Plasma Volume and Power Density

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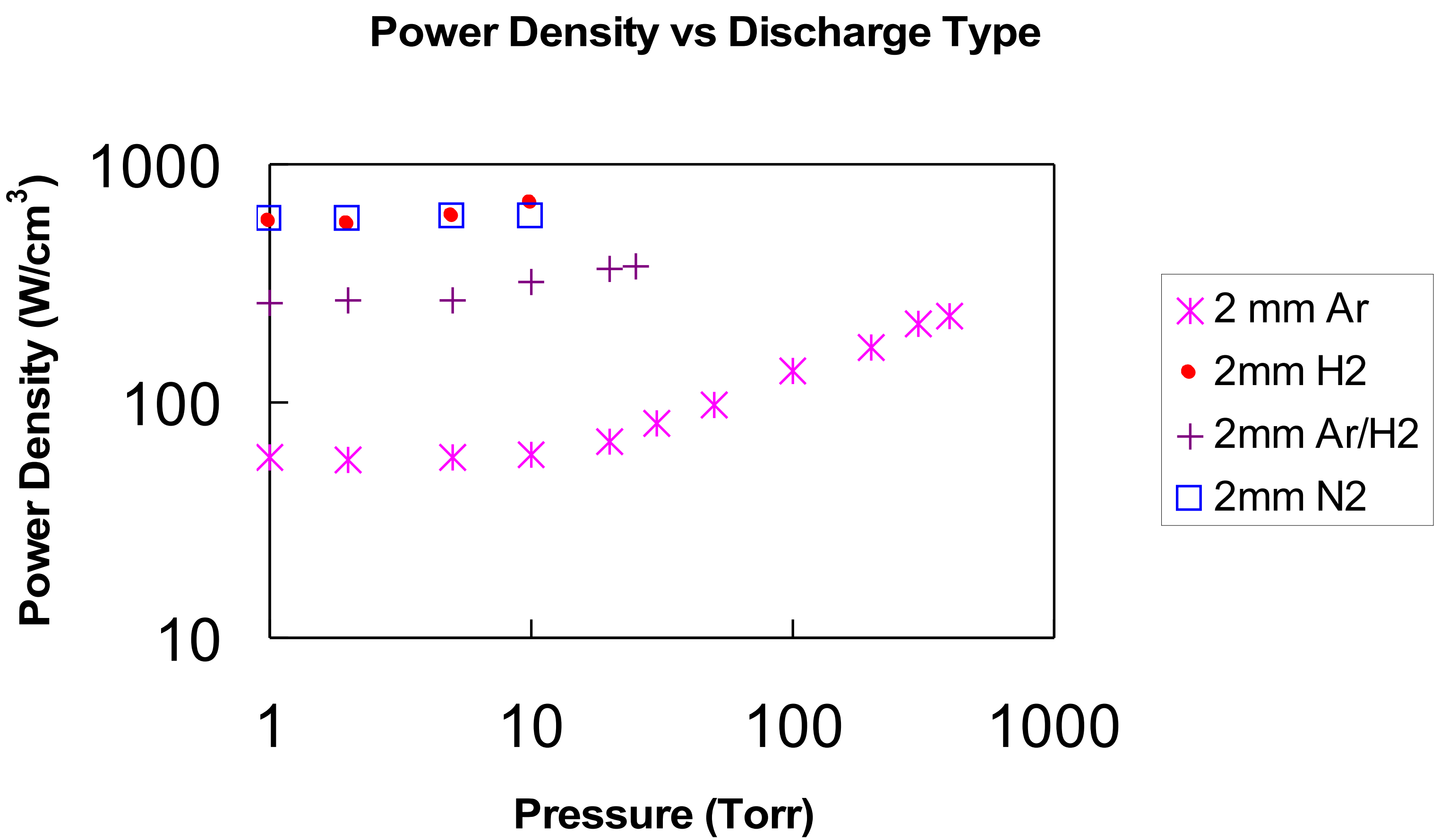
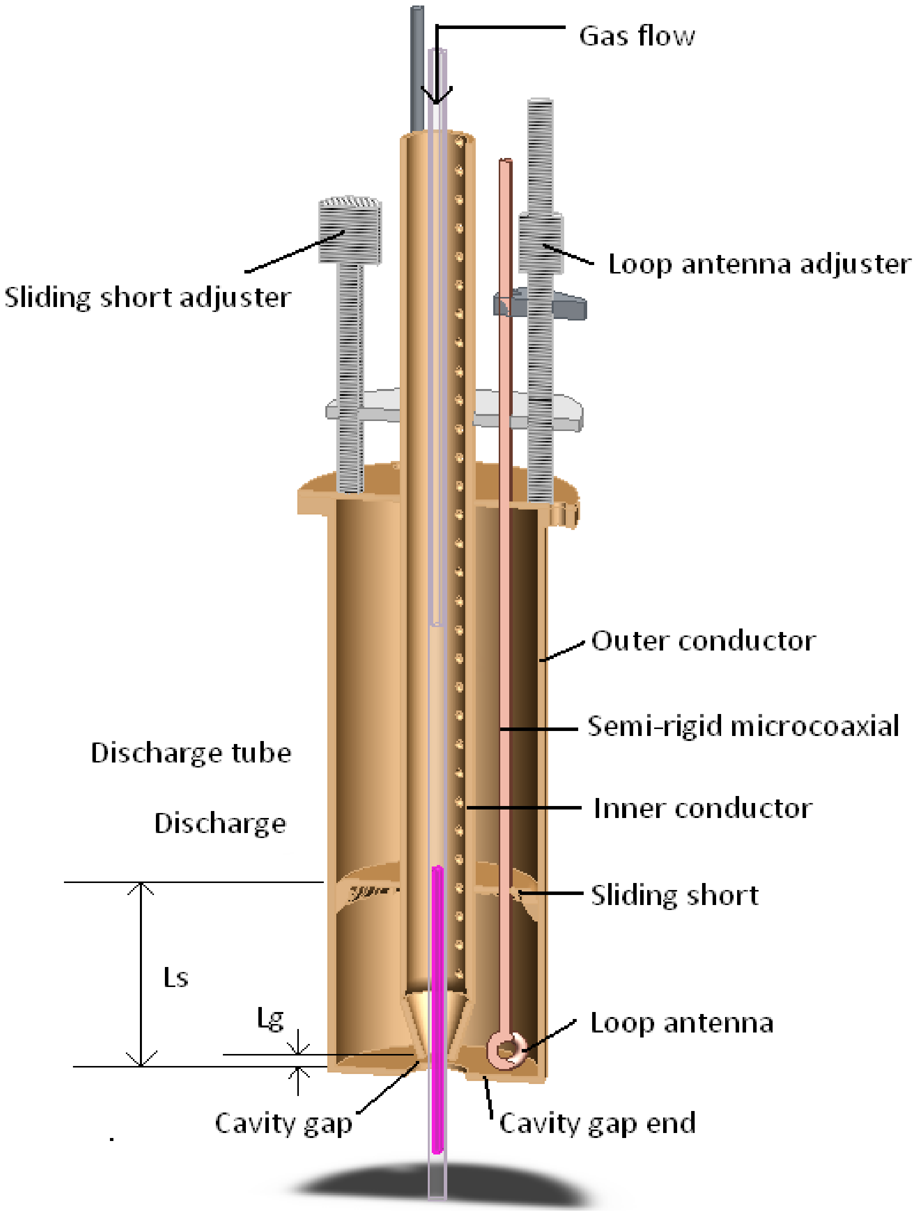
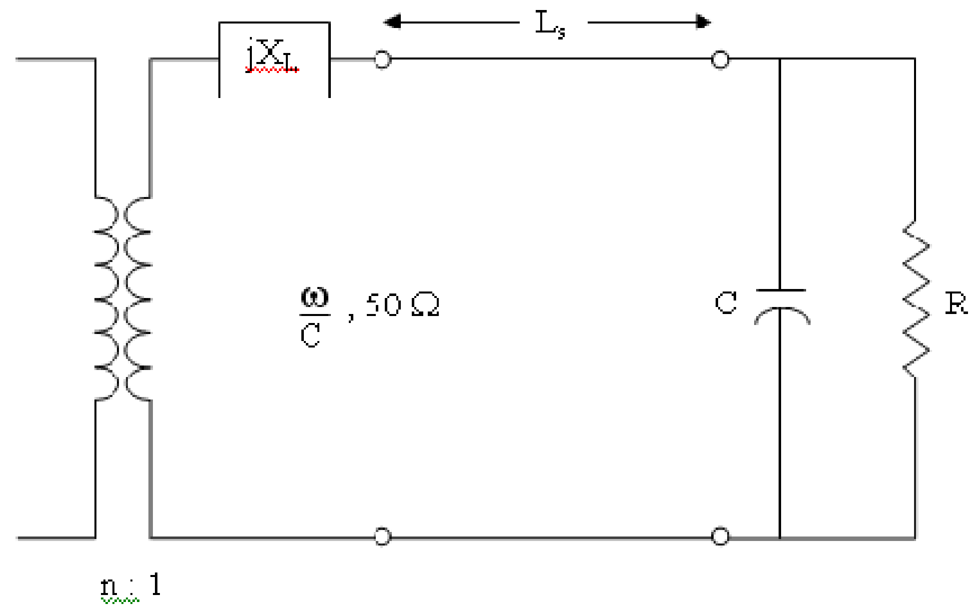


Discharge diameter 2mm with microwave power of 30 W.

Ar/H_2 corresponds to 95% Ar and 5% H_2.

Foreshortened Coaxial Cavity Applicator

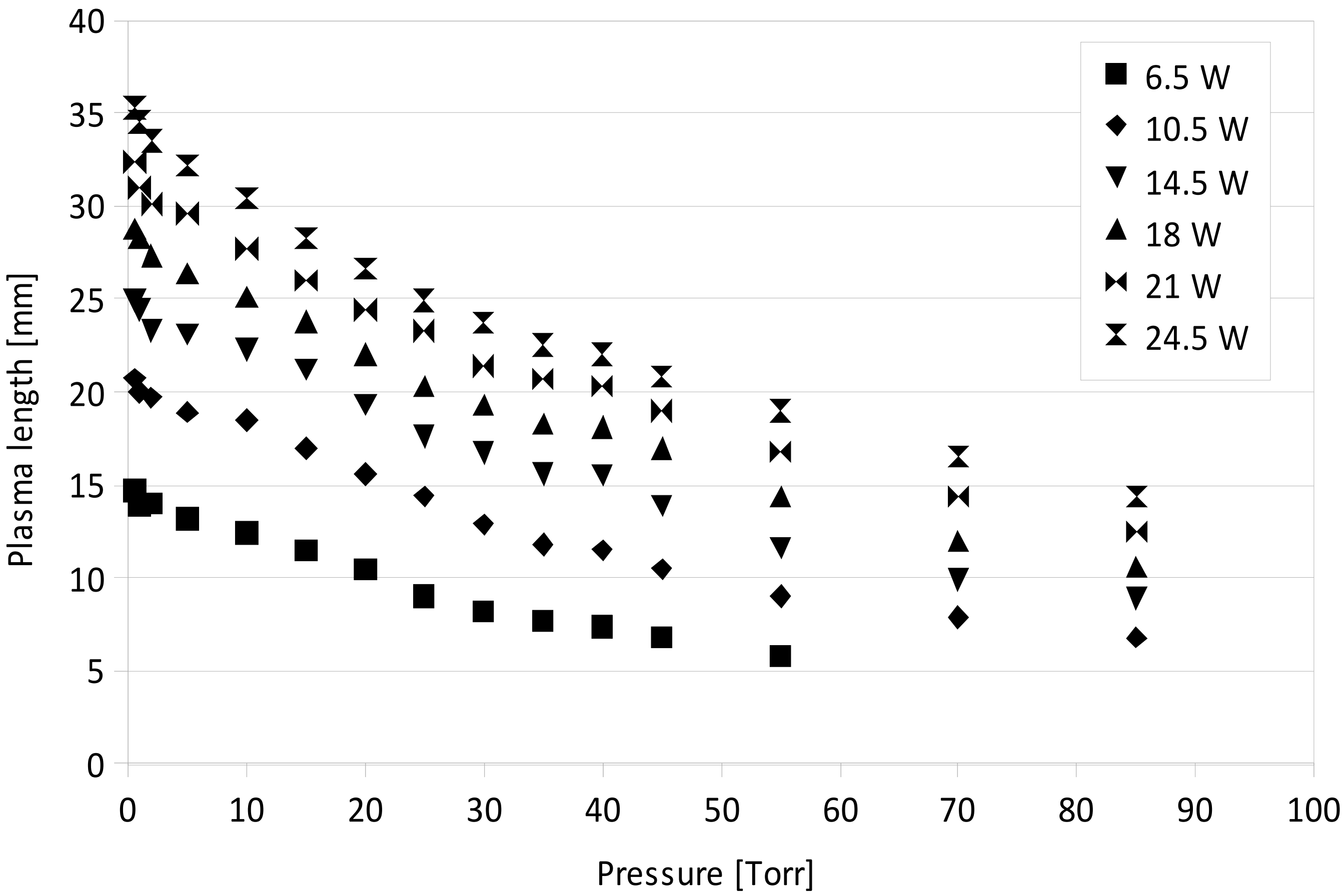




This is a modified coaxial cavity with a capacitive gap region.

The cavity can be adjusted in order to resonate by adjustment of the
- cavity length (L_s)
- gap distance (L_g)
- loop antenna coupling probe position and orientation.

Plasma Length



Variation of plasma length for Ar/O_2 discharge versus pressure. Total flow rate: 20 sccm, 98% argon, 2% oxygen. P_{abs}: 6.5 – 24.5 W.

- Plasma length decreases with increasing pressure
- Plasma length increases with increasing absorbed power
- Longer plasma columns can be generated with pure argon plasma. Addition of oxygen shortens the plasma length

Microwave Generated Microplasmas

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What have been established so far:

- Discharge tube sizes: power density is higher for smaller tube size
- Pressure: plasma length decreases as pressure increases
- Feed Gas: inert gas creates long plasma column. Addition of other gases reduces the plasma length
- Power: plasma length increases with increase of power.

Next: Plasma density, gas temperature, and electron temperature (note: using 2 mm i.d. Discharge tube)

Optical Emission spectroscopy (OES)

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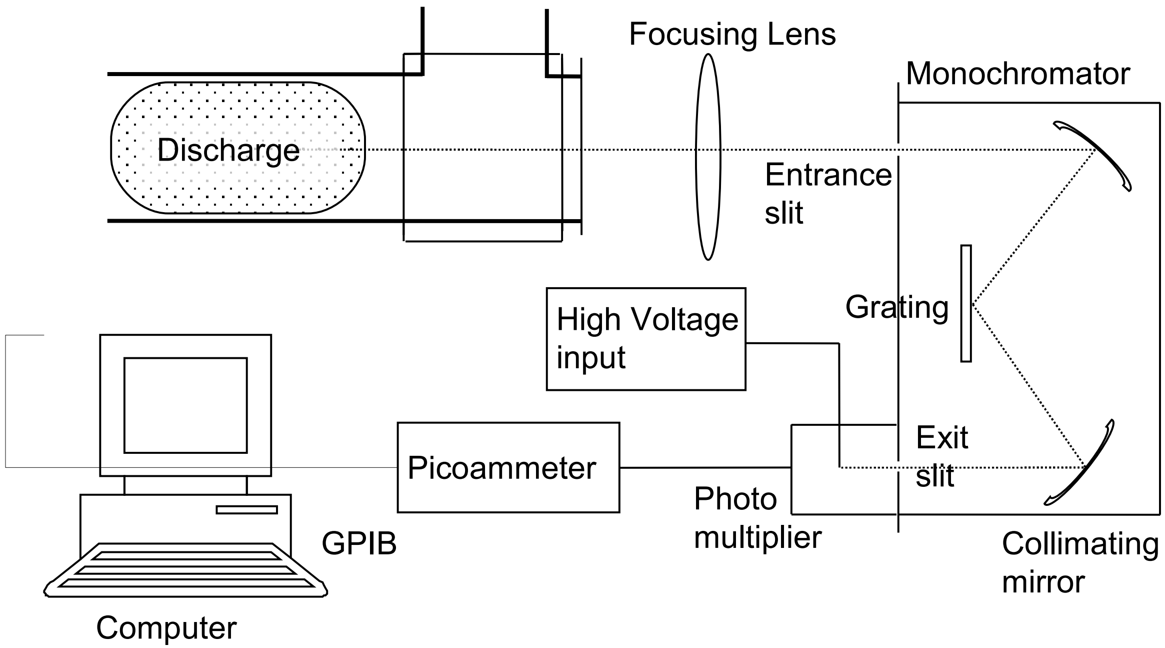

The relative rotational line intensities I of a Boltzmann distribution

$$I = Kv^4 S_{J'J''} \exp\left(-\frac{B_v' J'(J'+1)hc}{kT_r} \right)$$

Note: $B_v J'(J'+1)$ is the relative upper energy level

Theoretical background

Energy separation between rotational levels in a given vibrational and electronic state are typically small compared with the thermal translational energy. Thus, nearly all gas kinetic collisions produce a change in the rotational quantum number

Consequently, the relative rotational population distribution in a sufficiently long-lived vibrational state has a Boltzmann distribution and the rotational temperature reflects the gas kinetic temperature

The Honl-London formula for emission of the $\Delta\Lambda$=0, R-branch

$$S = \frac{(J'+\Lambda')(J'-\Lambda')}{J'}$$

OES – Gas Temperature Measurements

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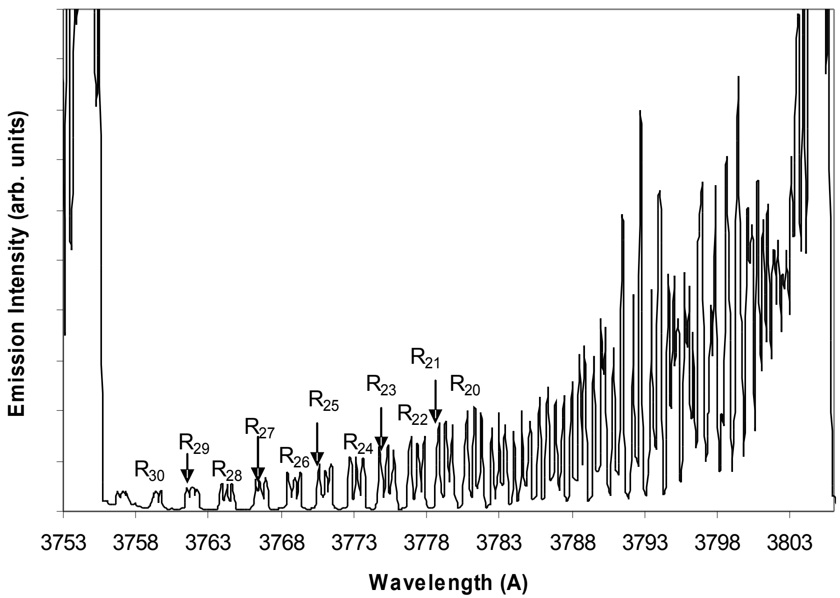
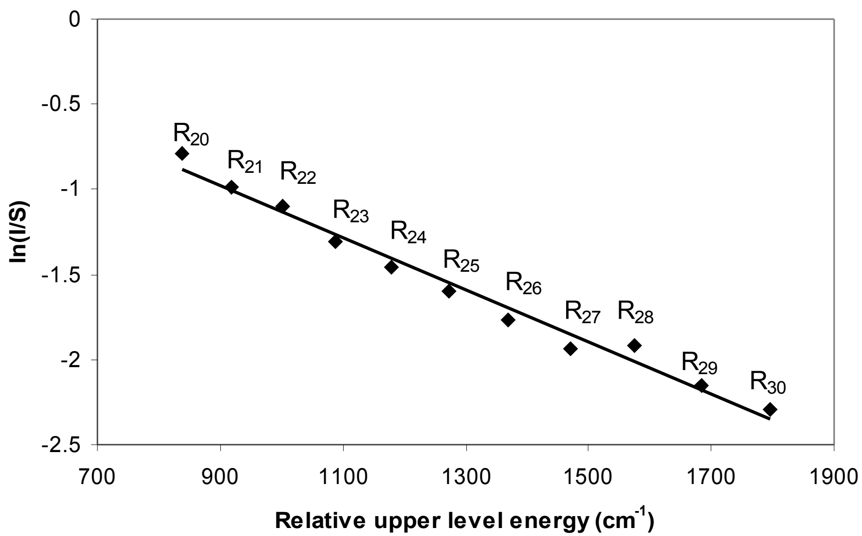
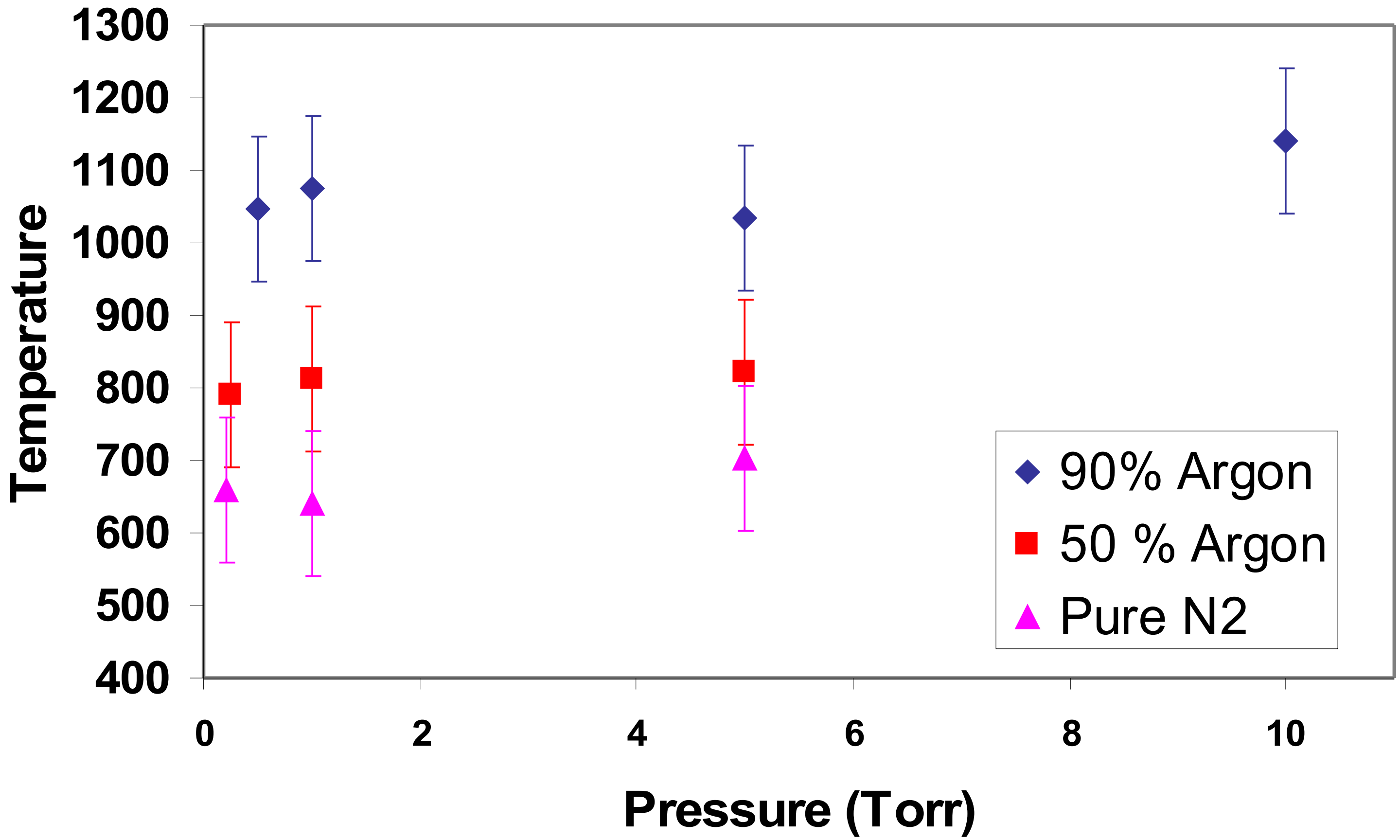




- The rotational temperature for nitrogen discharge was determined using the R branch of the $C^3\Pi_u \rightarrow B^3\Pi_g$ (2-0) band, commonly known as the Second Positive System (SPS)
- Eleven emission lines (R_{20} – R_{30}) in the spectrum range of 3758 Å – 3783 Å used to determine gas temperature
- The plot of *ln(I/S)* for this band is a linear function of the upper rotational energy

OES – Gas Temperature Measurements



Gas temperature of a mixture Ar-N2 discharges at 1 Torr in a 2 mm tube with 33 W microwave power was determined to be around 600K - 1200K.

Double Langmuir Probe (DLP)

•Langmuir Probe diagnostics measures the current – voltage (I-V) characteristics of a discharge

•DLP were chosen since there was no well-defined ground electrode in the discharge

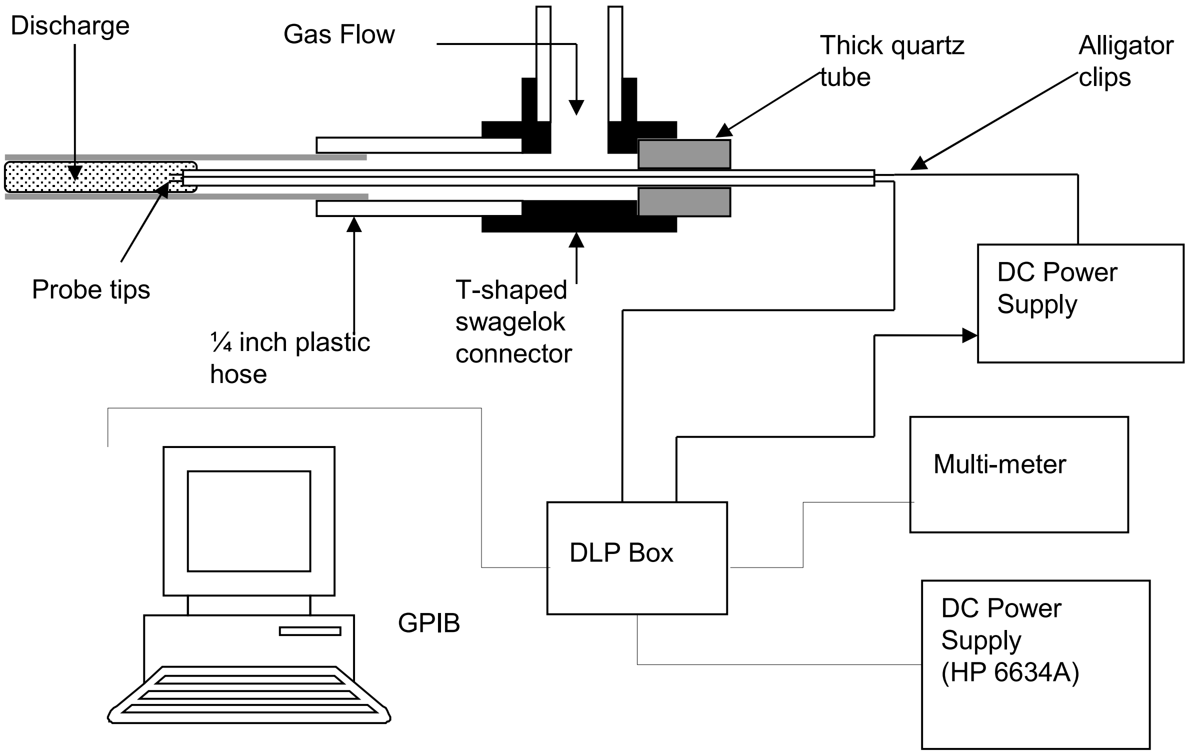
•The I-V characteristic is governed by the following equation:

$$\frac{I + I_1}{I_2 - I} = \frac{A_1}{A_2} \exp\left(\frac{V}{T_e}\right), \quad V = V_1 - V_2$$

•Note:

–I1 is the ion saturation current for probe 1

–A1 is the collection area of probe 1



- Collisionless sheath is assumed $(\lambda_i >> s)$
- Diagnostics were performed for argon discharges with pressure ranges from 3 – 10 Torr.
- Bias voltage applied range: -50 V – 50 V
- Probe area is bigger than the sheath thickness $(A >> s^2)$

DLP Measurements

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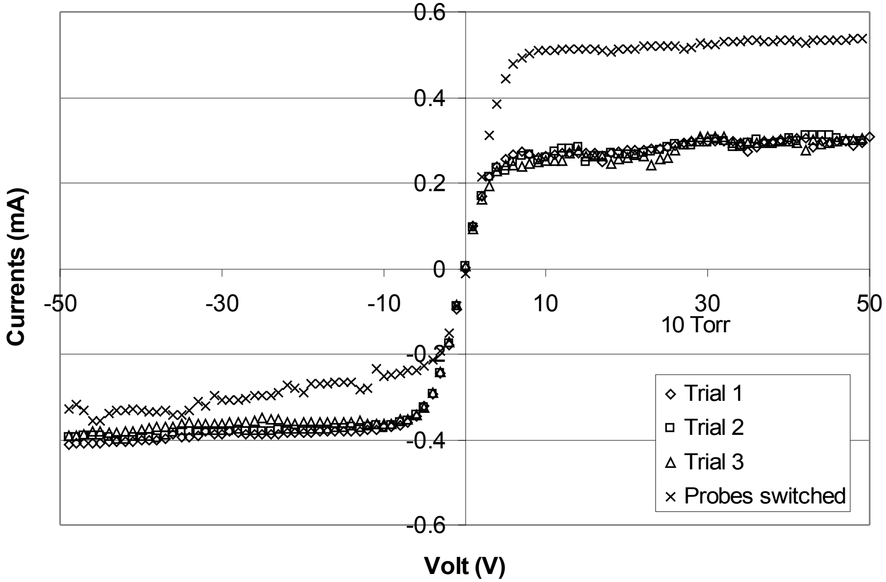
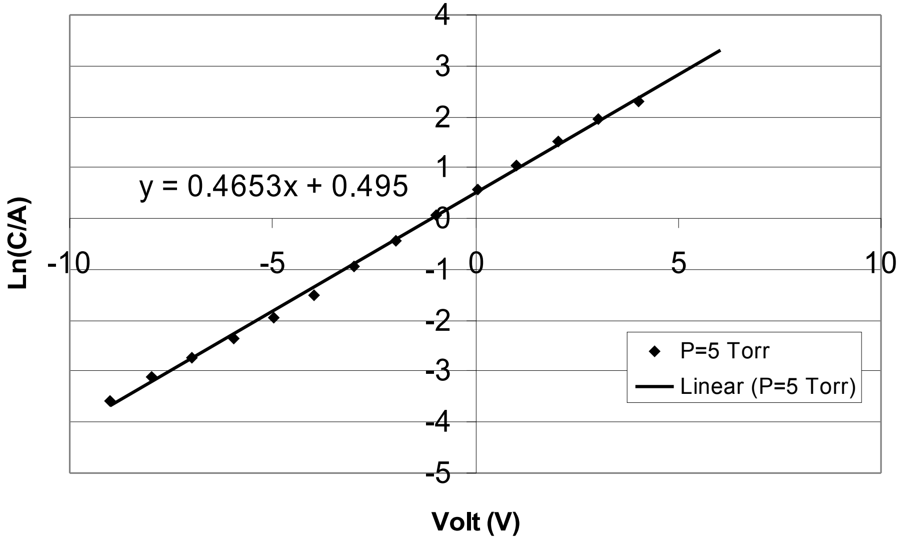


I-V curves obtained using DLP diagnostic. Pressure: 10 Torr, Pabs: 2.34 Watts, flow rate: 10 sccm



Log plot of the I-V characteristic from DLP diagnostic. $C=I+I_1/I_2-I$, $A=A_1/A_2$.

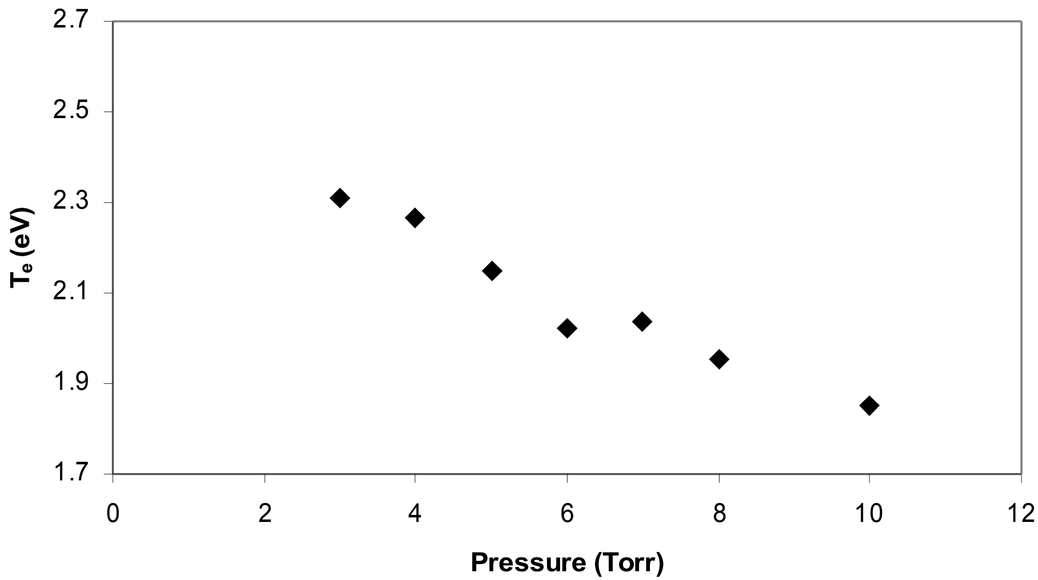
Re-arrange DLP I-V characteristic equation:

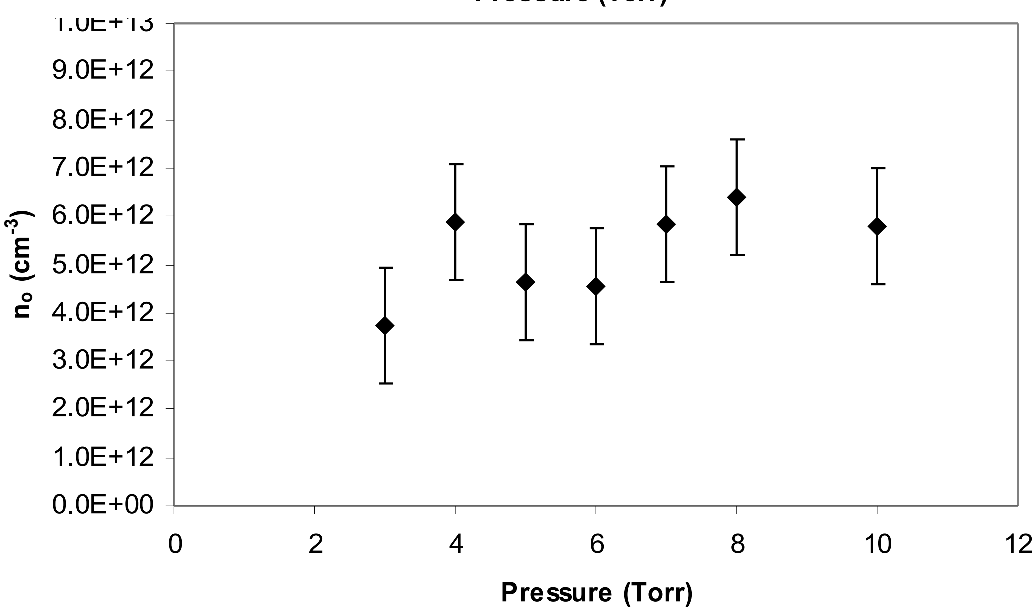
$$\ln\left(\frac{I + I_1 / I_2 - I}{A_1 / A_2}\right) = \frac{V}{T_e}$$

To find the electron density (n_o):

$$Ii = e\, n_s\, u_B\, Ap \approx e\, 0.61 n_o \left(\frac{e\,T_e}{M}\right)^{1/2} Ap$$

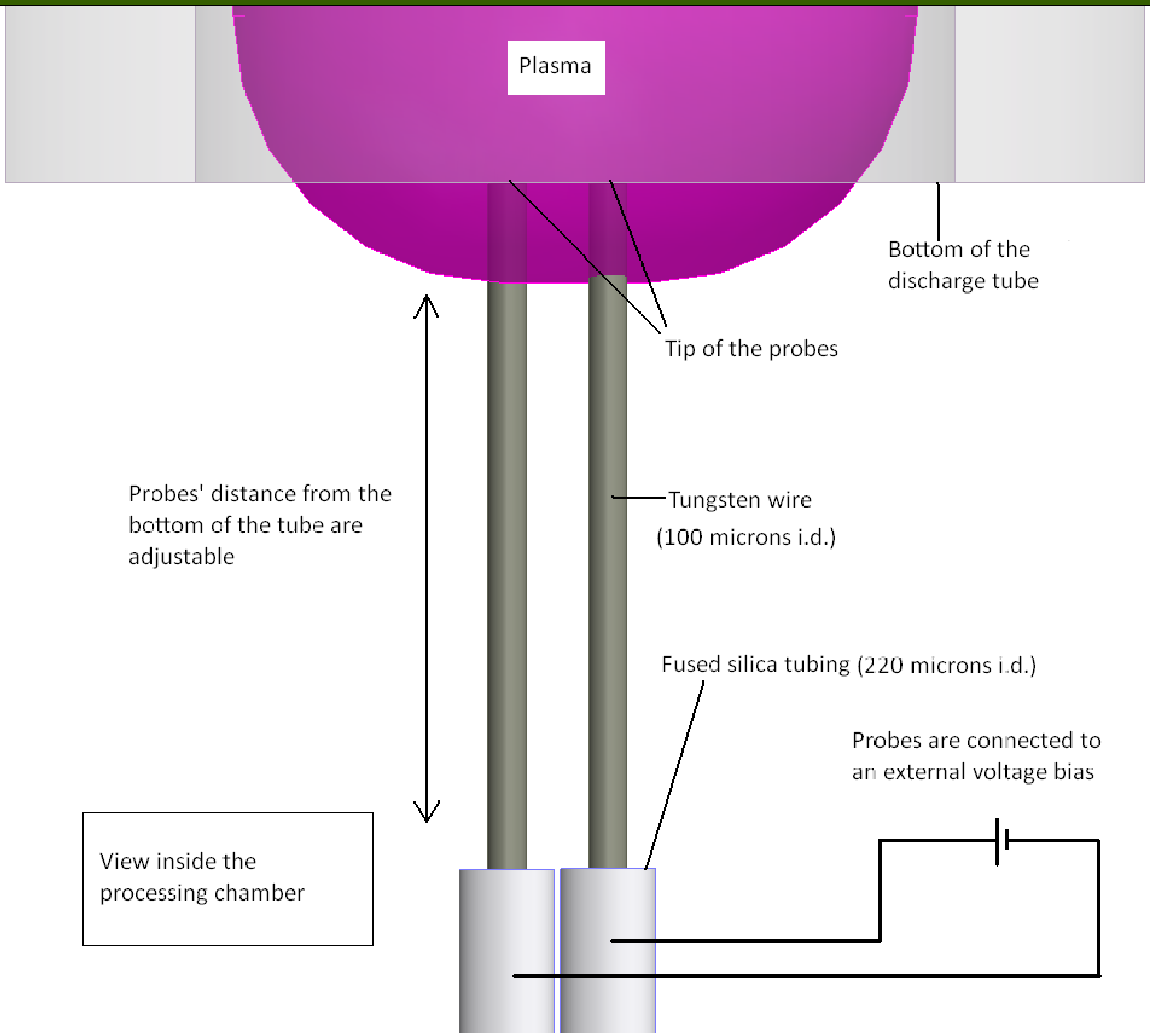
DLP Results





- The electron temperature ranges from 2.3 eV – 1.9 eV
- the electron temperature decreases as the pressure increases
- As the pressure increases, the charge density increases. The charge densities of argon discharges were measured to be 3 – 6×10^{12} cm^{-3}
- The results from electron temperature and charge density measurements confirmed that the assumed sheath thickness is valid

Spatial DLP Measurements



Charge density and electron temperature measurements using a double Langmuir probe. The probe distance relative to the tip of the discharge tube can be adjusted using the XYZ stage.

Bias voltage applied ranges from -50 to 50 V.

Experiment was performed for argon plasma at 0.9 Torr, P_{abs} 5.7 W, flow rate 20 sccm.

Spatial DLP Results

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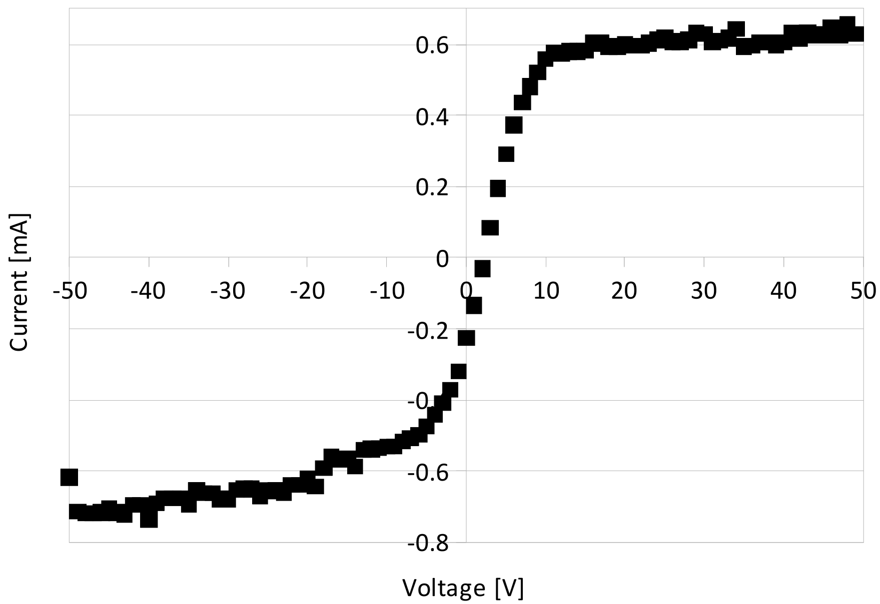
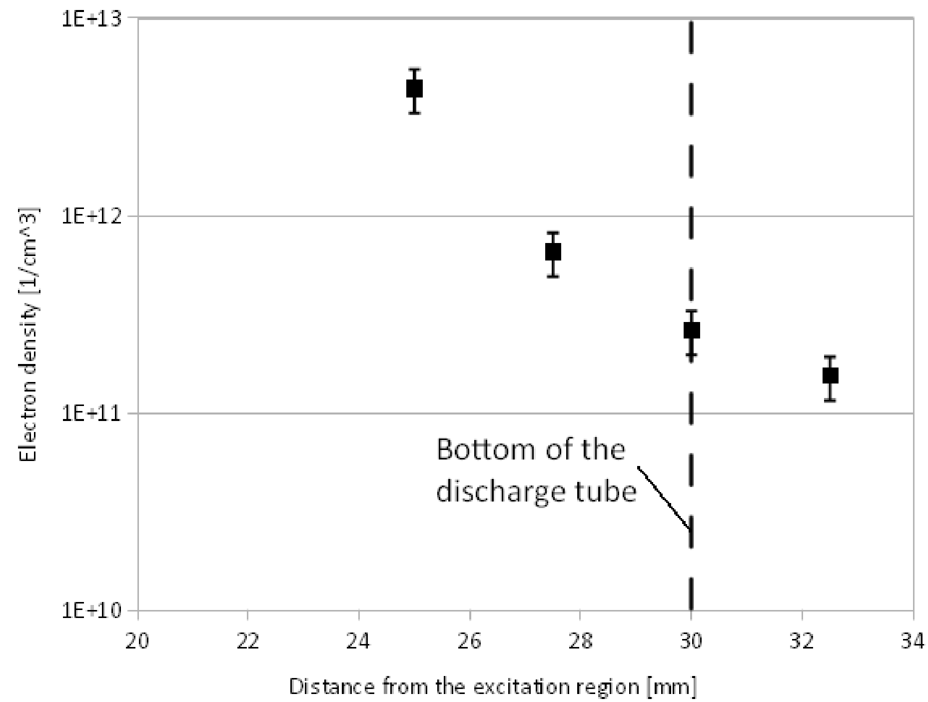
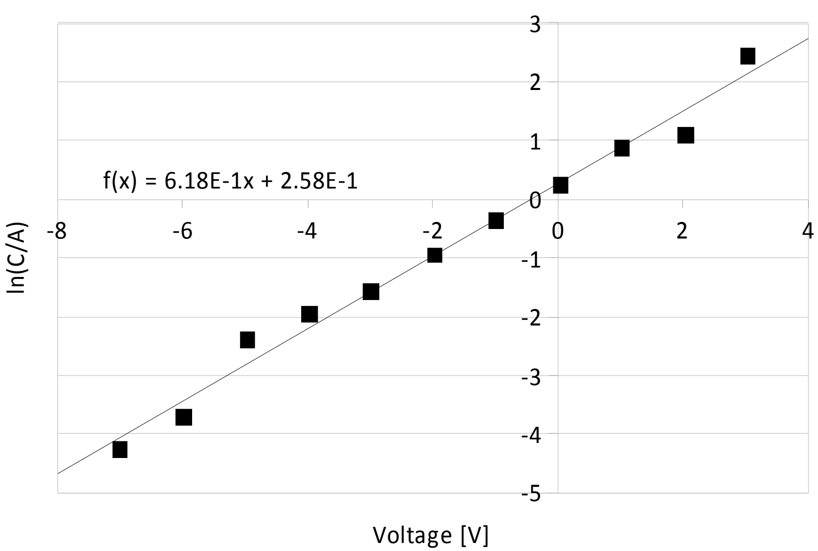






Probe tip location	T_e (eV)	n_e (cm^{-3})
5 mm inside the tube	1.28	4.41×10^{12}
2.5 mm inside the tube	1.62	6.55×10^{11}
At the tip of the tube	1.61	2.63×10^{11}
2.5 mm under the tip of the tube	2.62	1.54×10^{11}

Axial Density Profile of SWP's

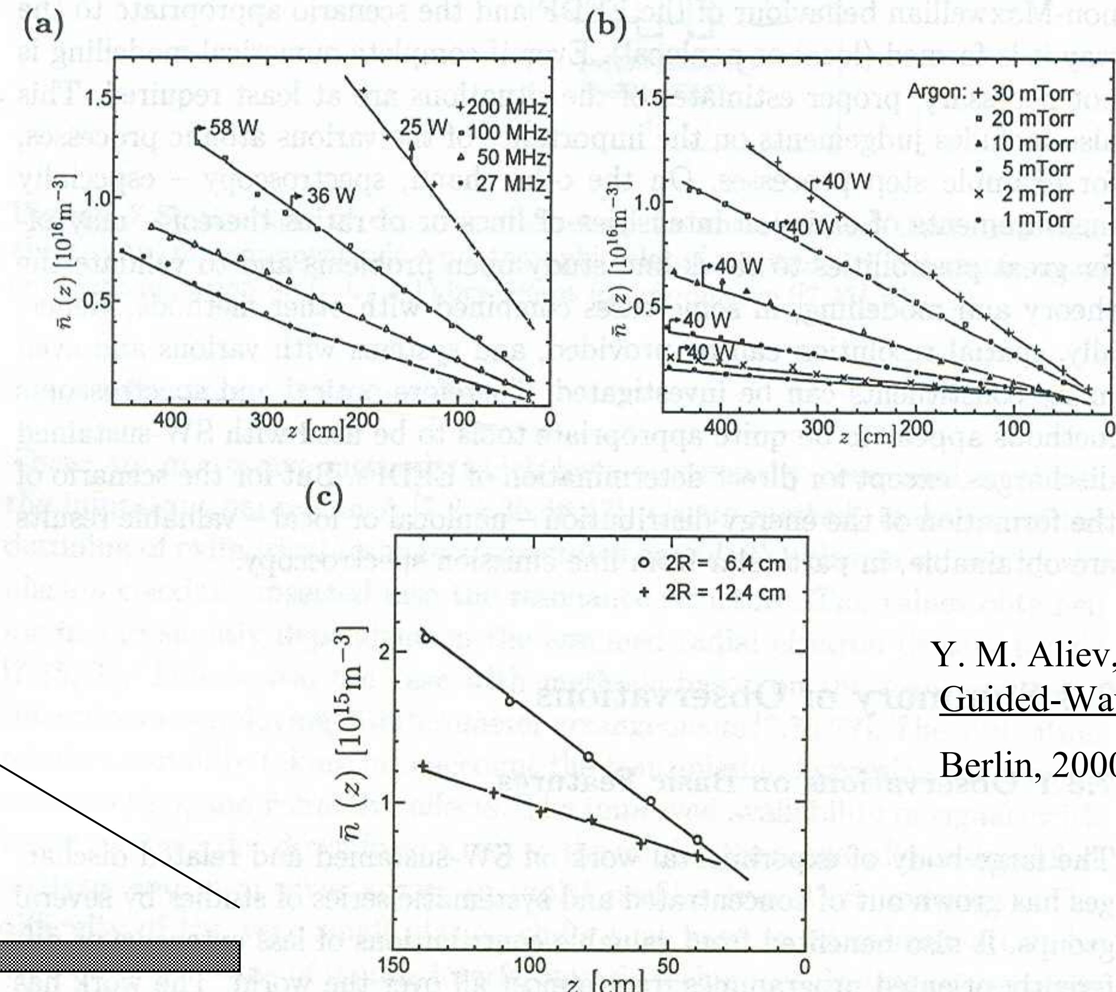
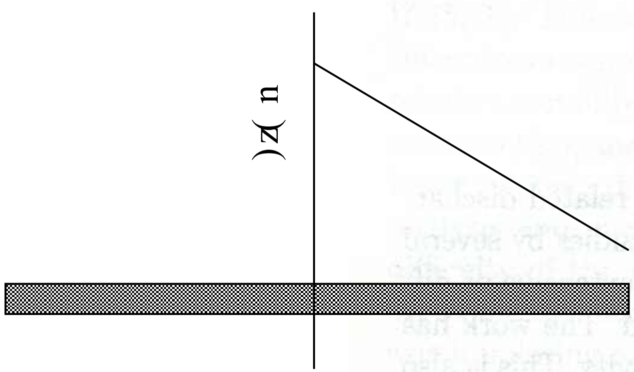


Figure 7.9. Axial distribution of the electron density $\bar{n}(z)$ with $R = 32$ mm (wall thickness 3.5 mm): (a) for various frequencies at 30 mtorr argon; (b) for various pressures at 100 MHz; (c) $\bar{n}(z)$ for various radii at 100 MHz and 10 mtorr argon ([7.5], Fig. 3–5)



Y. M. Aliev, H. Schulter, and A. Shivarova, Guided-Wave-Produced Plasmas, Springer, Berlin, 2000

Computational Argon Plasma Model

Plasma model was developed for pure argon plasma with operating pressure ranges from 0.1 – 10 Torr.

<u>Assumptions</u>

- Continuum mechanics
- Maxwellian EEDF
- Non-equilibrium plasma, T_{gas} = 800 K
- Ambipolar diffusion
- Quasi-neutral plasma
- Sheath boundary condition

<u>Solving</u>

- Continuity equation for charge species
- Continuity equation for metastable species
- Electron heat equation

Process	**Reaction**	**Activation Energy**
Ground State excitation	$Ar + e \rightarrow Ar^* + e$	11.56 eV
Ground state ionization	$Ar + e \rightarrow Ar^+ + 2e$	15.6 eV
Step-wise ionization	$Ar^* + e \rightarrow Ar^+ + 2e$	4.14eV
Superelastic collision	$Ar^* + e \rightarrow Ar + e$	-11.56 eV
Quenching to resonant	$Ar^* + e \rightarrow Ar^r + e$	
Metastable pooling	$Ar^* + Ar^* \rightarrow Ar^+ + Ar + e$	
Two-body quenching	$Ar^* + Ar \rightarrow 2Ar$	
Elastic scattering	$Ar + e \rightarrow Ar + e$	

Computational Plasma Model

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Continuity equation for the charge species

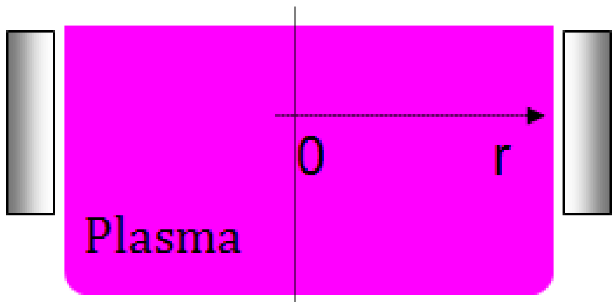
$$\frac{\partial n_e}{\partial t} + \nabla \bullet \Gamma = R_{iz} + R_{step} + R_{pool} \qquad \Gamma \approx \frac{-k_B T_e}{m_{Ar} \nu_{iN}} \nabla n_e$$

Continuity equation for the metastable species

$$\frac{\partial n^*}{\partial t} + \nabla \bullet \Gamma^* = R_{exc} - R_{step} - R_{s\_coll} - R_{quench} - 2R_{pool} - R_{quench2} \qquad \Gamma^* \approx -D_i \nabla n^*$$

Electron heat equation

$$\frac{\partial \left(n_e \frac{3}{2} e T_e \right)}{\partial t} + \nabla \bullet Q_e = W_{abs} - W_{coll}$$



1-D simulation domain

Boundary conditions for n_e at the sheath edge

$$\Gamma_s = n_s u_B \approx 0.61 n_e \sqrt{\frac{e T_e}{M_{Ar}}}$$

Initial condition: n_e=constant

Boundary conditions for n_{exc} at the wall (sheath edge)

$$n_{exc} = 0$$

Boundary conditions for T_e at the sheath edge

$$Q_w = (2T_e + 5.2T_e)\Gamma_s$$

COMSOL software was used
Number of element in mesh: 100
Boundary condition at r = 0 is symmetrical B.C.

W_{abs} is a constant value and is approximated from the power density (PD) measurements. For argon plasma below 10 Torr, PD ~ 10 W/cm^3

1-D Plasma Simulation Result

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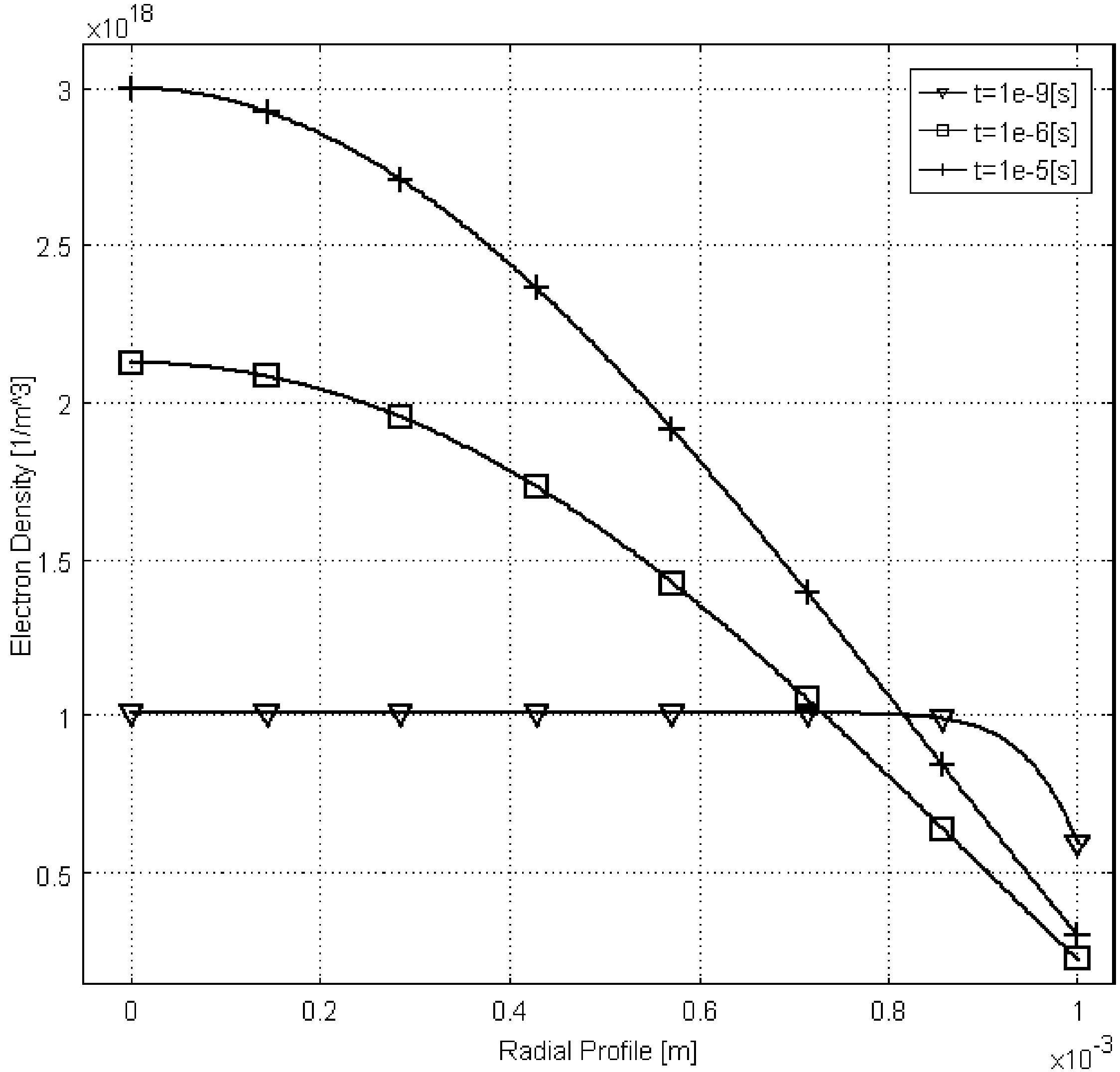


1-D simulation results of the computational model of argon plasma is in a good agreement with the experimental results.

Electron density range: 10^{11} – 10^{13} cm^{-3}
Electron temperature range: 1.2 – 2.6 eV

Radial profile of electron density in 1-D argon plasma simulation. Power density: 10 W/cm^3, pressure: 1Torr.

Characteristics of SWP's

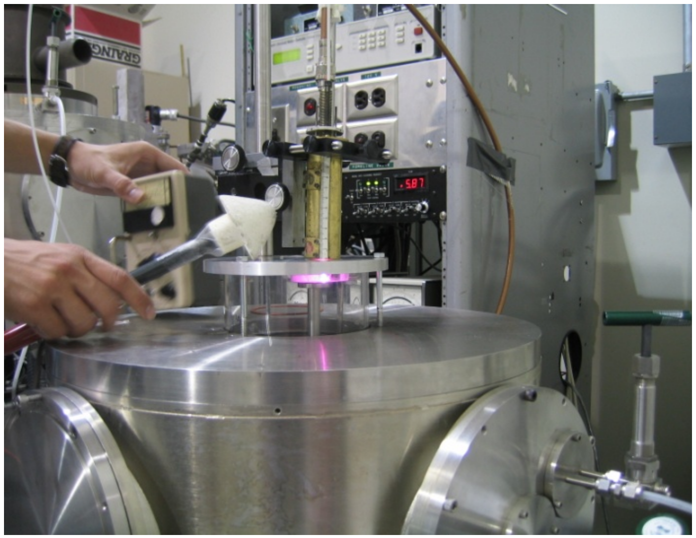
Gas Temperature range: 600 K – 1200 K

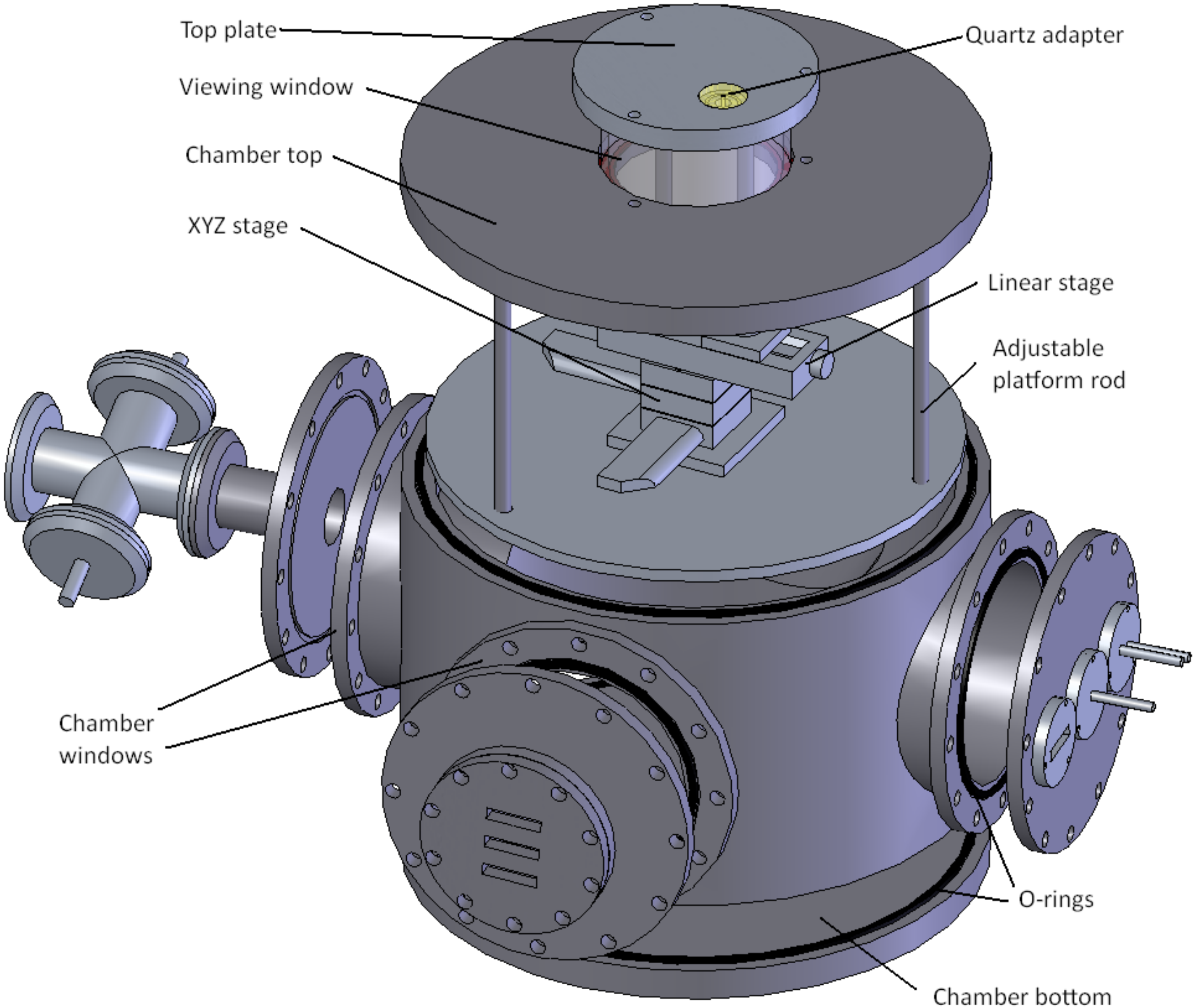
Charge density falls off at the edge of the plasma column

Electron density range: 10^{11} – 10^{13} cm^{-3}

Electron temperature range: 1.2 – 2.6 eV

Micromachining System

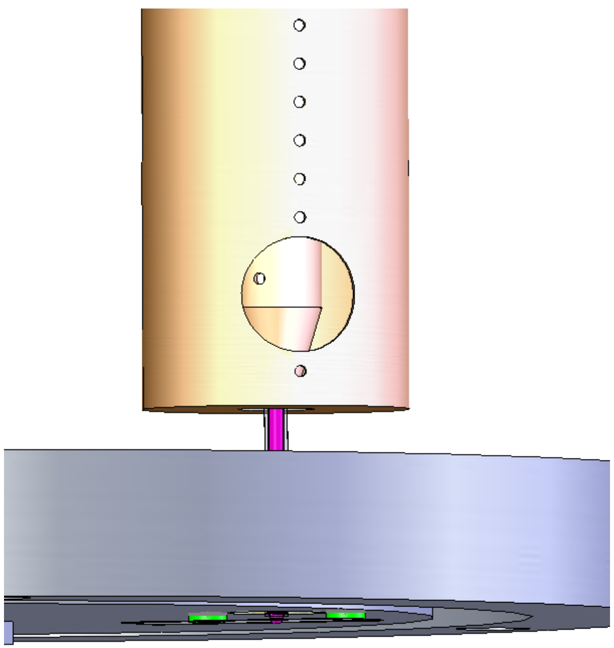




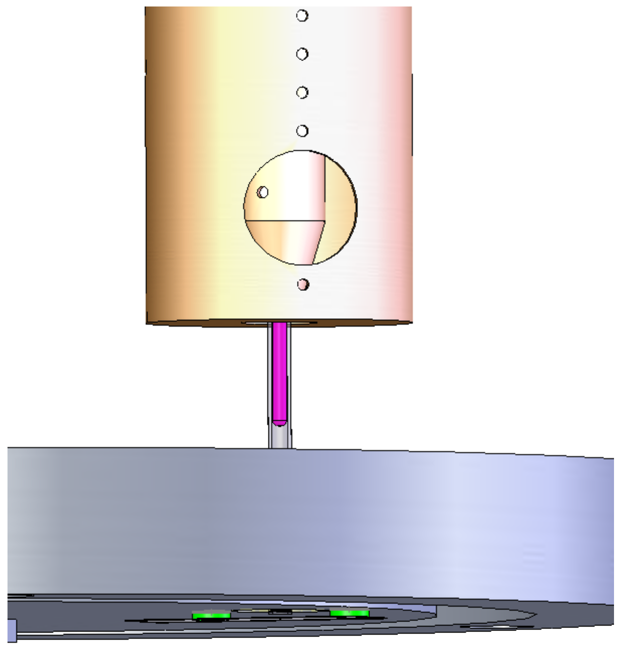
Micromachining System
-Local area materials processing
-The plasma applicator is used as either an
  ion source, and/or
  UV light source

Micromachining System Feature

Direct plasma processing

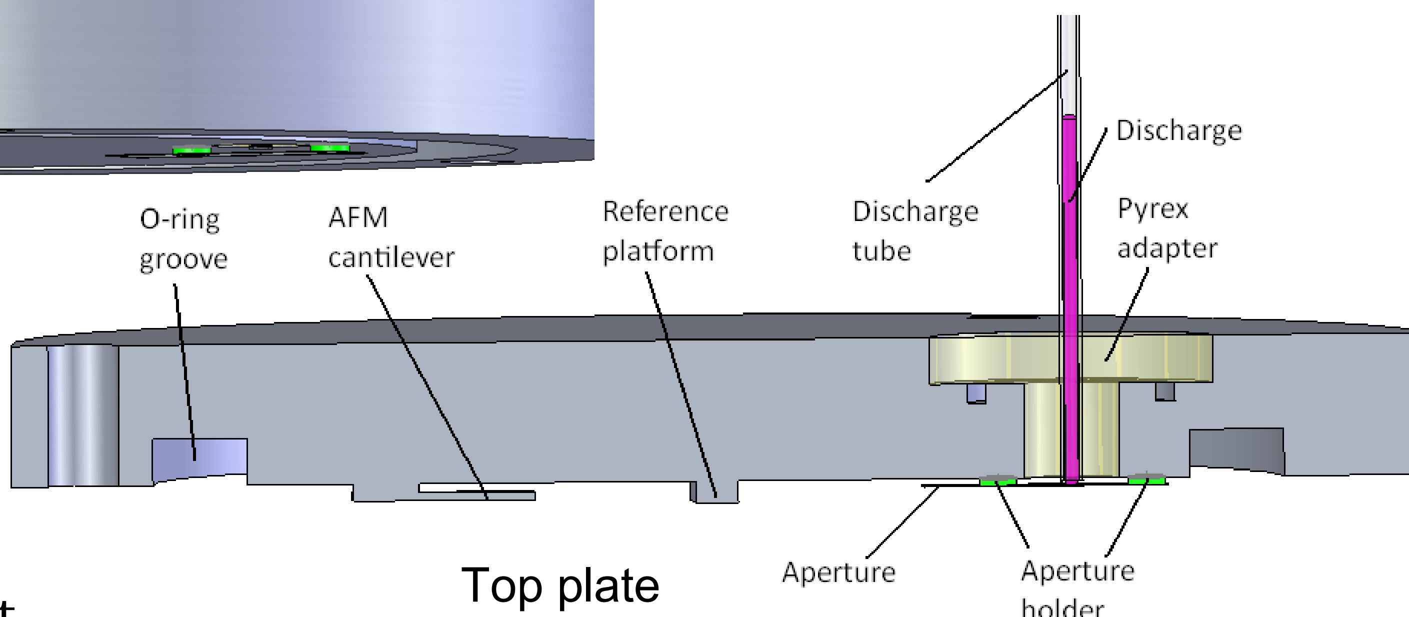


Remote plasma processing



Operating parameters:

- 2.45 GHz 1 – 50 Watts
- 2 mm i.d. quartz discharge tube
- Pressure 0.5 – 100 Torr
- Feed gases: Ar, O_2, SF_6, Ne, H_2 N_2
- Flow rates: 0.5 – 20 sccm
- Aperture size: 25 – 800 μm
- RF bias : 20 V_{pp}, 1 – 4 MHz



Direct plasma processing:
- Ion source
- Ion/radical source

Remote plasma processing:
- Radical source
- UV exposure of photo resist

Micromachining Plasma

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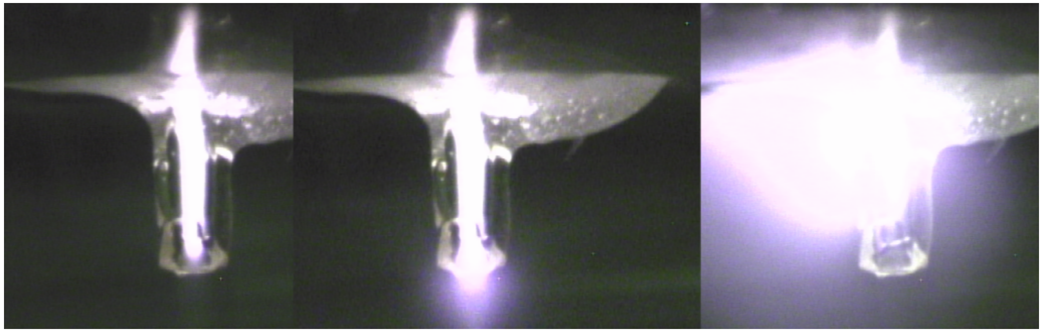
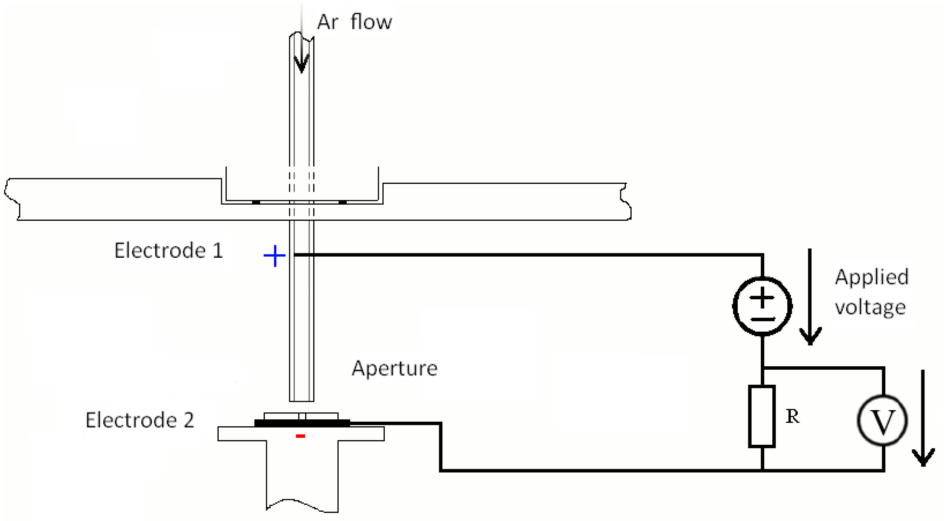


Argon plasma
P_{in} = 10 W
P = 0.5 Torr

Argon plasma
P_{in} = 20 W
P = 0.5 Torr

Argon plasma
P_{in} = 30 W, P = 0.5
Torr

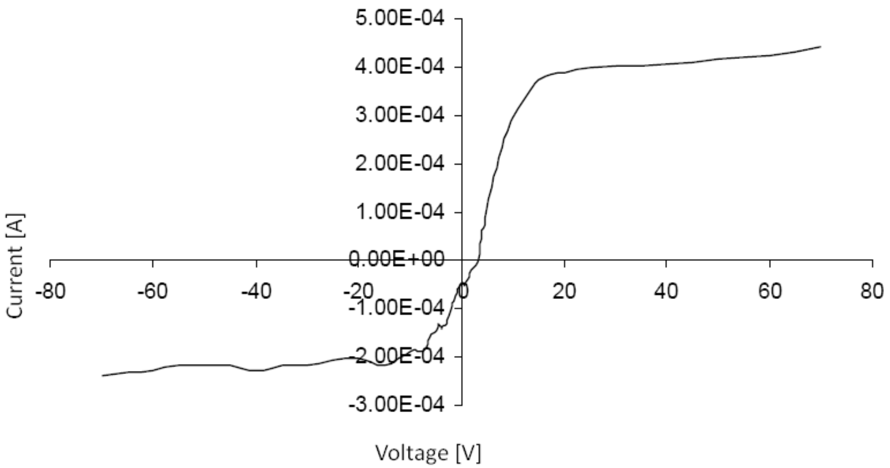


Increasing the absorbed power increase the length of the plasma column. However, when the applied microwave power is too big, plasma can be generated outside the discharge tube in the processing chamber.

Charge particles collected on the substrate holder can be measured without an aperture or with a 450 μm aperture. However, using 15 μm aperture, the I-V characteristic could not be produced.

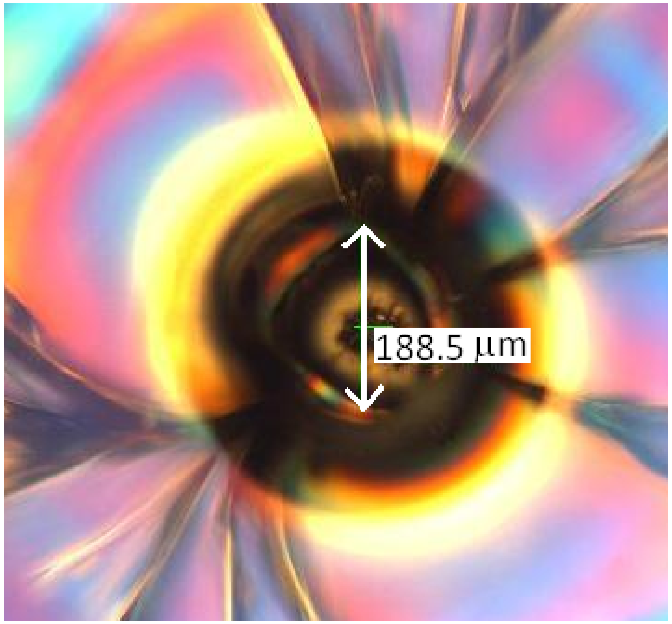
For argon plasma, plasma sheath can be approximated As several λ_{De}.

$$\lambda_{De} = 743\sqrt{\frac{T_e[eV]}{n_e}}\,[cm]$$



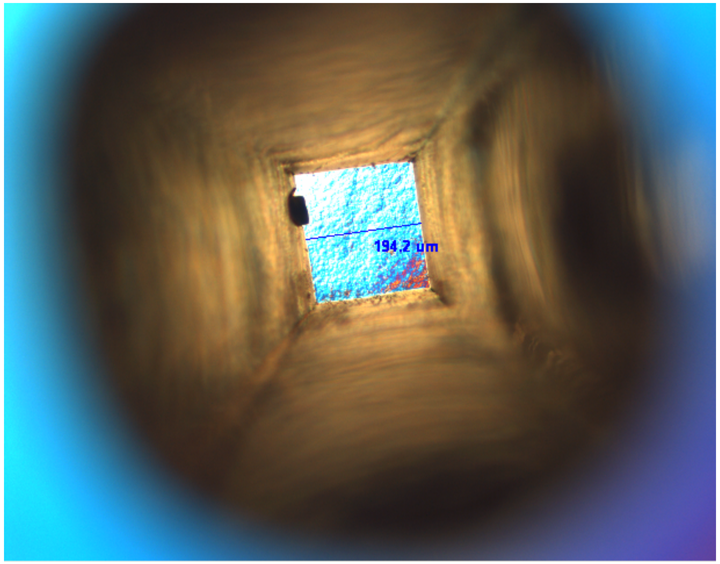
I-V characteristics of the charge particle observation with no aperture

Aperture Designs



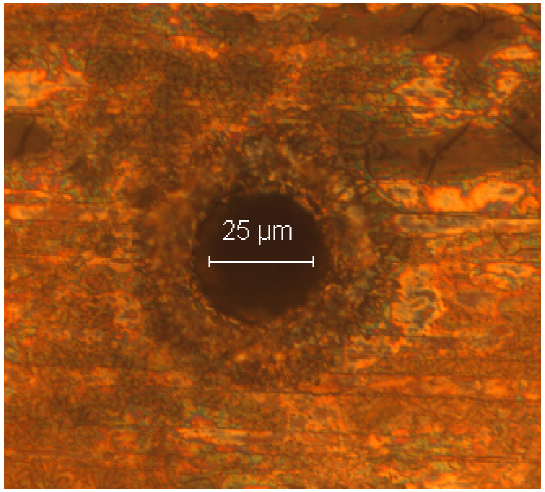
Pyrex aperture

Thickness: 100 µm
Opening size: 10 – 500 µm
Laser drilled
(Fraunhofer USA CCL)



Silicon wafer aperture

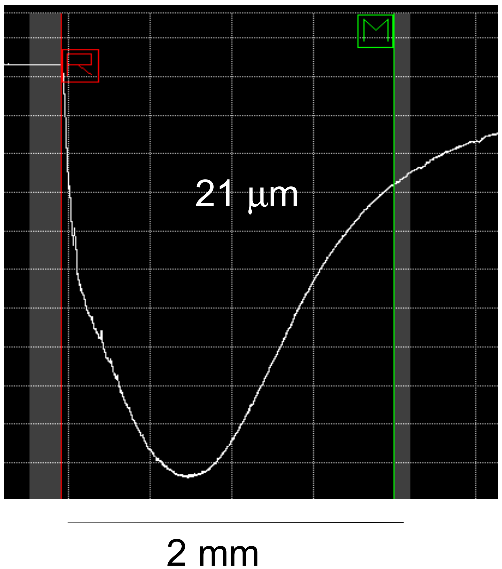
Thickness: 300 µm
Opening size: 50 – 800 µm
KOH wet etching
(Fraunhofer USA CCL)



Steel aperture

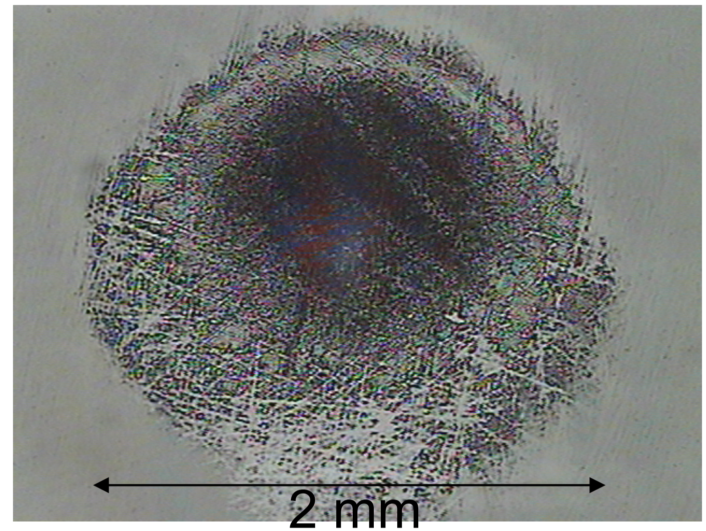
Thickness: 13 µm
Opening size: 25 -30 µm
(Melles-Griot)

Ion Source Application: Silicon Etching



Dektak surface profile
Without aperture
Stationary

- Ar/SF_6 (35/0.5 sccm)
- Pabs = 5 W
- Pressure = 1 Torr
- t = 10 min
- Etching rate = 2.1 µm/min



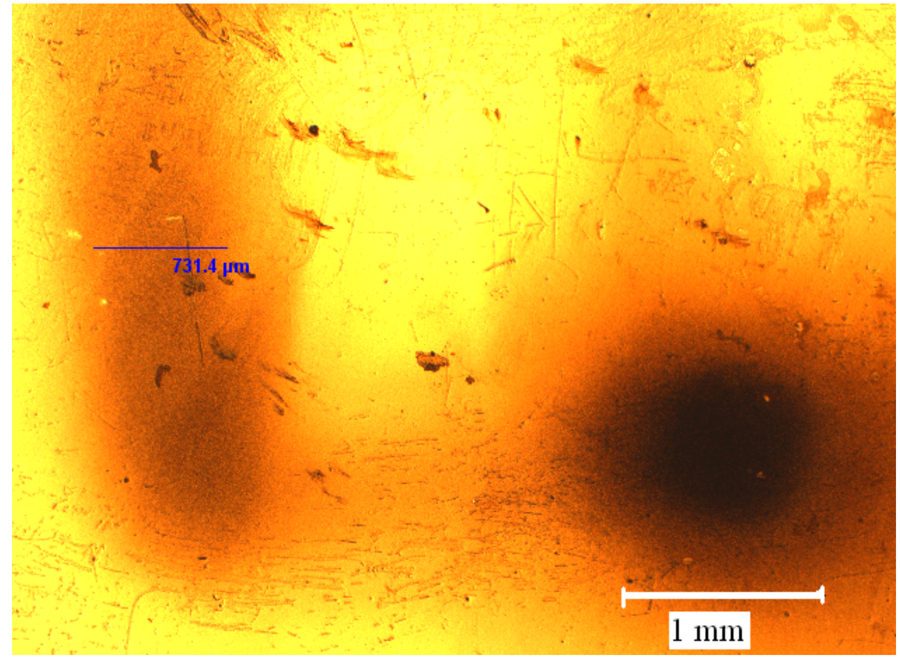
Optical image of an etched Si surface
Without aperture
Stationary

Discharge tube etched by SF_6

Ion Source Application: Silicon Etching

Dektak surface profile
100 µm aperture
Stationary

Ar/SF_6 (35/0.5 sccm)
Pabs = 5 W
Pressure = 1 Torr
t = 3 min
Etching rate = 0.6 µm/min



Optical image of localized Si etching using Ar/SF_6 microplasma.
100 µm aperture
Ar/SF_6 (15/1.5 sccm)
Input power = 16 W
Pressure = 1.35 Torr
RF bias = 4 MHz, 20 V_{p-p}
Speed: 100 – 500 µm/s,
Stationary for 3 min

Ion Source Application: Diamond Etching

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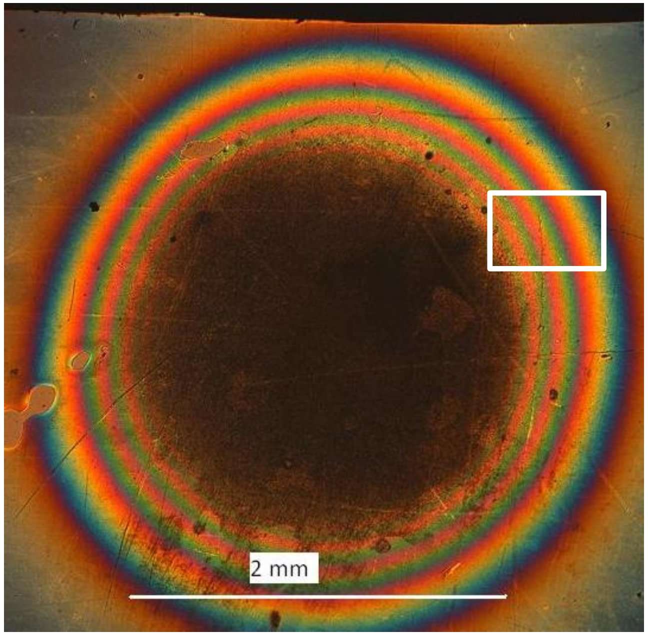

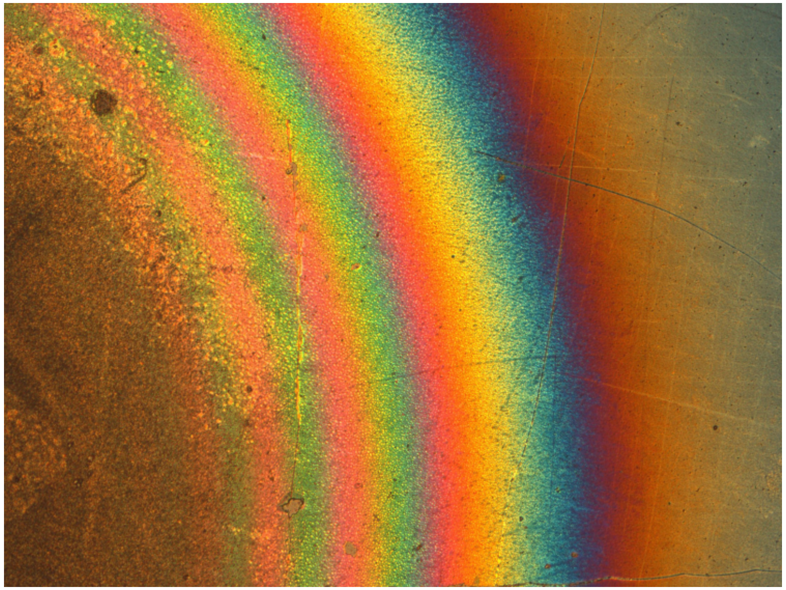

Image of UNCD surface etched using micromachining system Ar/O_2 plasma

No aperture
Ar/O_2 (17/3 sccm)
P_{abs} = 20 W
Pressure = 1.18 Torr
RF bias = 4 MHz, 20 $V_{p\text{-}p}$
t = 40 min

10x magnification of the fringes area.

The relative vertical depth between the consecutive fringes of the same color for diamond is 130 nm.

Etched depth ~ 650 nm
Etching rate ~ 1 μm/hr

Ion Source Application: Diamond Etching

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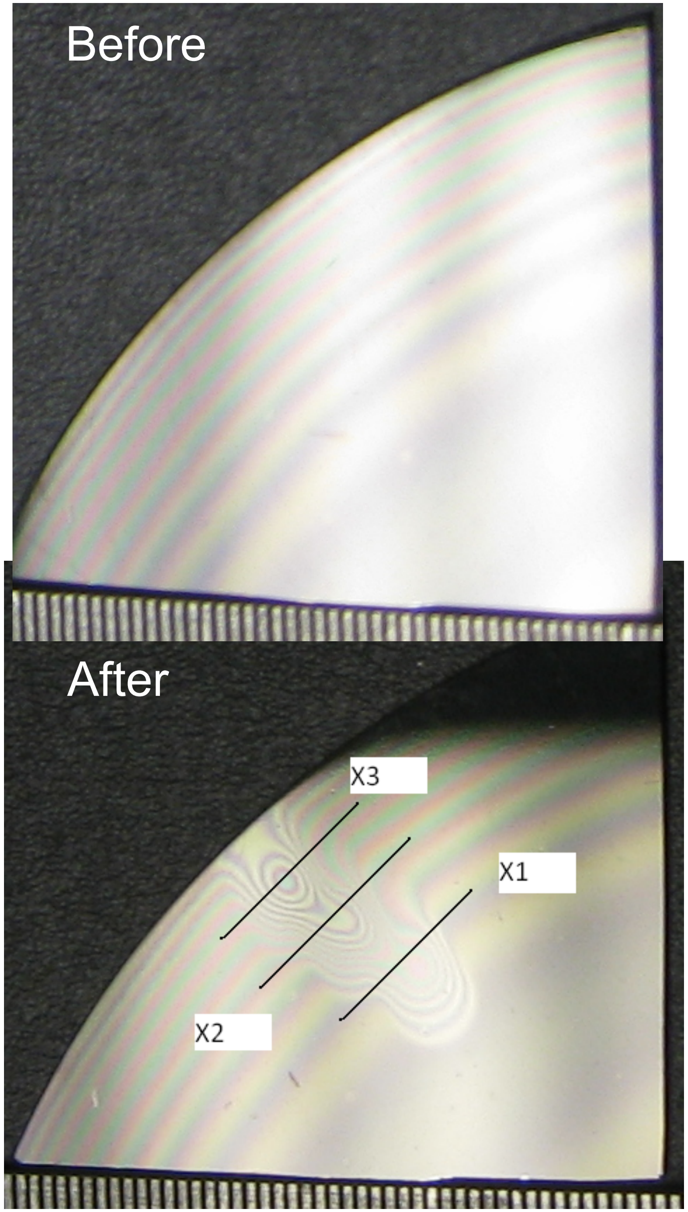


25 μm steel aperture
Ar/O_2 (20/3 sccm)
P_{abs} = 25 W
Pressure = 0.78 Torr
t = 60 min
RF bias =
X1 : no rf bias
X2 : 1 MHz, 20 $V_{p\text{-}p}$
X3 : 4 MHz, 20 $V_{p\text{-}p}$

Distance between the substrate to the aperture ~ 500 μm.

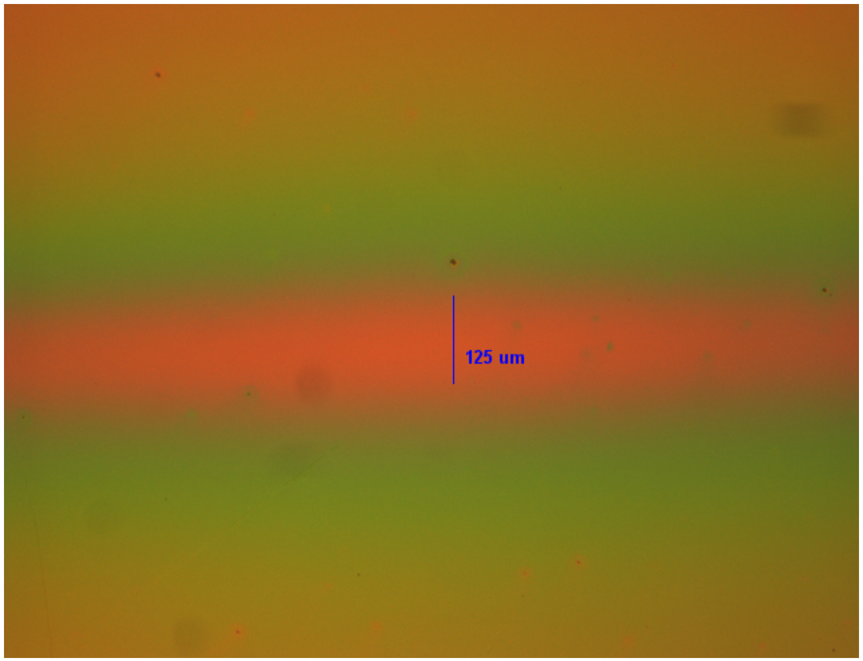
Diamond etching rate: 0.5 – 2 μm/hr

Experiment	Diameter (mm)		Depth (μm)		Etch Rate (μm/hr)	
	Fringe	Dektak	Fringe	Dektak	Fringe	Dektak
X1 (no RF bias)	1.75	1.6	0.52	0.6	0.5	0.6
X2 (1 MHz)	2	2.7	0.91	1.9	0.91	1.9
X3 (4 MHz)	2	2.5	0.78	1.7	0.78	1.7

Distance between ruler marks is 0.5 mm

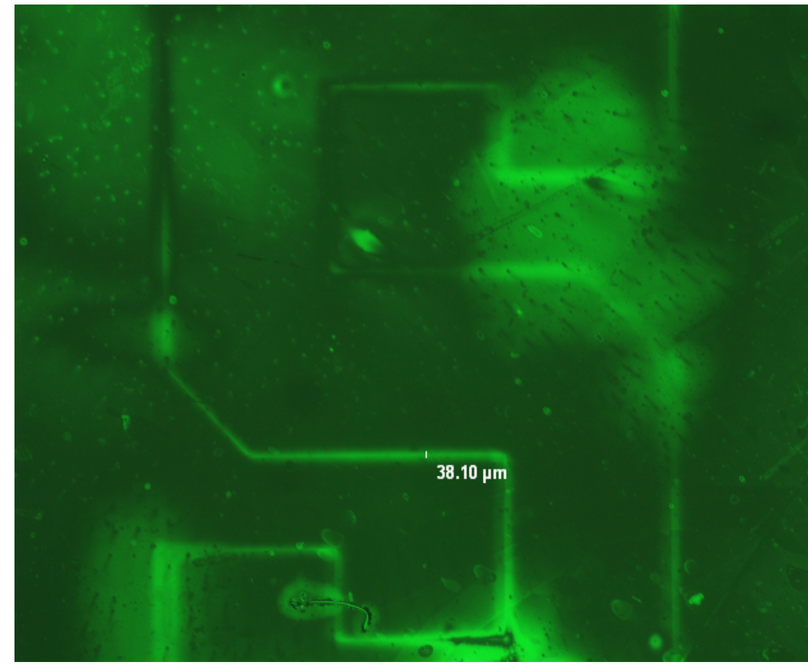
Material Processing Results (Remote)

Plasma ashing of photo resist



P_{abs} = 34 W,
Pressure = 0.84 Torr,
Ar/O_2 (20/10 sccm),
aperture size 30 μm
v = 1 μm/s,
line width = 125 μm

UV exposure of photo resist



Optical microscope image of photo resist exposed to Ne plasma under 30 μm aperture. P_{abs} = 14 W, Pressure = 25 Torr, v = 10 μm/s

Summary

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Microwave generated microplasma applicators have been built: microstrip and cavity based.

Plasma length decreases with increasing pressure and increases with increasing microwave power. Power density for argon and Ar/O_2 discharges are in the range of 10 – 450 W/cm^3.

The electron density and electron temperature of an argon plasma in the microplasma system at 1 Torr was experimentally measured using a double Langmuir probe. The electron density is on the order of 10^{11} – 10^{13} cm^{-3} and the electron temperature is 1.2 – 2.6 eV.

Summary

A computational model for low pressure argon plasmas was developed. 1-D simulation results for the argon plasma model are in a good agreement with the experimental results.

Several applications of the micromachining system have been demonstrated. Silicon etching was performed using Ar/SF_6 plasma with etching rate 0.2 – 2.1 μm/min. Diamond etching were performed using Ar/O_2 plasma with etching rate in the range of 0.5 – 2 μm/hr.

Application of the micromachining system as a free radical source and a light source were also demonstrated.

Current Work

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XG Sciences produces exfoliated graphene nanoplatelets (xGnP)

Results:

- Built a system with a reduced power consumptions,
- Performed continuous operation,
- Maintained the yield quality

Current project:

- Assembling the next generation system that should double the yield volume.

Acknowledgments

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Thank You