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Plasma Science & Technology Department Princeton, New Jersey (USA)

PPPL Graduate Summer School 2019



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What is a Plasma?

A *Plasma* is <u>more</u> than a collection of neutrals, ions, and electrons!

What characterizes a plasma (i.e. low temperature plasma)?

- 1. Quasi-neutral state of existence
- 2. Collective behavior due to Coulomb forces
- 3. Energy distribution (T_e is high, not T_i and T_g)
- 4. Collisional and radiative processes





Two types of plasmas

High-temperature plasmas or Hot (Thermal) plasmas $T_i \approx T_e \ge 10^7 \text{ K}$ e.g., fusion plasmas $T_i \approx T_e \approx T_g \le 2 \times 10^4 \text{ K}$ e.g. arc plasma at normal pressure

Low temperature plasmas or Cold (Non-thermal Plasmas)

 $\label{eq:T_i} \begin{array}{l} T_{i} \approx T_{g} \approx 300 \ \text{K} \\ T_{i} << T_{e} \leq 10^{5} \ \text{K} \\ \text{e.g. low-pressure glow discharge} \\ \text{high-pressure cold plasma} \end{array}$



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Plasma generation

A plasma is generated by supplying energy to a volume of gas to produce pairs of electrons and ions (charge carriers).

- most commonly electrical energy is supplied

Low temperature plasmas are usually excited and sustained electrically by applying

- Alternating Current (AC) power
- Radio Frequency (RF) power
- Microwave (MW) power
- Direct Current (DC) power
 - Pulsed DC power





How do we make plasmas?

Supply Energy!!! e.g. Heat transfer, radiation, electric power...

For many plasma applications, an Electric Field is applied to a gaseous environment

Plasma or Gaseous Discharge









DC Glow Plasma





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DC Glow Plasma



Reference: J.T. Gudmundsson & A. Hecimovic. *Foundations of DC plasma sources*. **Plasma Sources Science and Technology**, Volume 26, Number 12 (2017)

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PPPL's Remote Glow Discharge Experiment (RGDX)



https://www.pppl.gov/RGDX



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Low-Pressure Glow Discharge Plasmas





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Inductively Coupled Plasma

Frequency range: 10s kHz - 10s of MHz range









Pulsed DC generated plasmas



- During operation with pulsed DC the potential on the dielectric (target) is periodically modulated, at a pulsing frequency *f*, typically between 1 to 350 kHz.
- For a certain *reverse time*, τ_{rev} or *off time* at the beginning of each pulse, a positive reverse voltage is applied
 - this phase typically accounts for 10-50% of the pulse
- The remaining phase or *on time*, τ_{on} accounts for 50-90% of each pulse.



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Pulsed DC generated plasmas

• The primary reason in the development and establishing of pulsed DC was the suppression of arcs at the target during reactive deposition.



- Research indicates that pulsing the discharge modulates the intrinsic plasma parameters.
- One of the most important parameters influencing the quality of deposited films is the energy of ions impinging on growing films.
 - Studies have shown a dependency of film properties on the selected pulsing frequency and reverse times.



Pulsed DC generated plasmas

Charging up and discharging the dielectric surfaces



When no arcing regime is maintained, discharging during each off-time is complete



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Magnetron Introduction



Balanced Magnetron

- one pole is positioned at the central axis of the target and the other pole is placed around the outer edge of the target forming closed or balanced field lines.
- In the balanced magnetron, the dense plasma is constrained near the target racetrack (~60 mm from the target surface)

Unbalanced Magnetron

- outer magnets are stronger than ones in the center.
- some of the field lines from the edge magnets cannot be balanced by the center ones and have to be closed on themselves then causing the field lines to come down to the substrate
- Electrons move down to the substrate along these field lines with ions coming together as required by global plasma neutrality.
- higher ion flux onto the substrate compared to the balanced magnetrons.



Pulsed DC Unbalanced Planar Magnetron

- Secondary electrons move in a direction perpendicular to both the electric and magnetic field.
- the E×B drift causes electrons to move parallel to the cathode surface in a direction 90 degrees away from the magnetic field.
- Secondary electrons lose their kinetic energy due to collisions with gas atoms (ionization) or with other electrons (electron heating), and the net result is an extremely dense plasma in this drift ring
- Ions in the drift region have a high probability of hitting the cathode. This results in even more production of secondary electrons and eventually very dense plasma







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Pulsed DC Unbalanced Planar Magnetron





Picture of inside the vacuum chamber with the magnetron and it's reflection in the mirror.

Planar rectangular HRC-873 magnetron with a Ti target $(3.5" \times 8")$ Base pressure 6×10^{-6} Torr Working atmosphere: Ar/O₂ mixture at a gas flow ratio of 1:1 and a pressure of 9 mTorr

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Time-Resolved Plasma Emission Images



Images taken Power=1 kW f = 20 kHz $\tau_{off} = 10 \text{ }\mu\text{s}$

Plasma distribution at the beginning of the on-time is different than during the rest of the cycle.



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Cross-corner effect in planar rectangular magnetrons



An image of the Ti target magnetron plasma racetrack taken approximately 0.2 μ s after the start of the on-time. Notice the cross-corner effect is clearly present at the opposing edges of the plasma racetrack.



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Cross-corner effect in planar rectangular magnetrons



Eroded rectangular target sputtered with a conventional magnetron cathode, showing faster etching in the upper left corner area.



Glow in opposite corners of a flat anode placed under a rectangular magnetron



Glow in opposite corners of dual anodes placed on the sides of a rectangular magnetron





Low-Pressure, Low-Temperature Plasma Processing



Plasma processing of silicon for semiconductor manufacturing.







Plasma processing to harden or coat materials.



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Plasmas are easier to be generated at low pressures

Low pressure plasmas (1 mTorr ~ a few Torr)

- ➤ are well understood
- are used extensively nowadays (e.g. in semiconductor industry for computer chips manufacturing)



However, to generate low pressure plasmas:

- vacuum chambers
- expensive vacuum pumps
- pressure monitoring and pressure control devices

Generate Plasmas at Atmospheric Pressure!!





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What happens at air pressure?

- No vacuum is involved
- Difficult to generate and sustain
- Run into some challenges such as glow to *arc* transition Non controllable

Arc Discharge: thermal plasma

- It's hot and detrimental
- -Gas temperature can reach as high as $2x10^4$ K
- Low voltage drop at cathode
- High cathode current density







Atmospheric (or higher) Pressure: Microplasmas



Pressure x Electrode Separation (or pressure for a fixed electrode separation)

Paschen Breakdown Curve

Stabilization of high-pressure plasmas: "pd scaling": "p" ↑, so "d" ↓ to keep breakdown voltage low and minimize instabilities after breakdown -

Microplasmas

Dimension: a few millimeter down to and below 100 μ m





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How do we create stable atmospheric pressure, low temperature plasmas?

Transient (pulsed) plasmas: *In atmospheric* plasmas, *for efficient gas heating at least 100-1000 colli*sions are necessary. Thus, if the plasma duration is shorter than $10^{-6} - 10^{-5}$ s, *gas* heating is limited. Of course, for practical purposes such plasma has to be operated in a repetitive mode, e.g., in trains of microsecond pulses with millisecond intervals.

Micro-confinement: Gas heating occurs in the plasma volume, and the energy is carried away by thermal diffusion/convection to the outside. If the plasma has a small volume and a relatively large surface, gas heating is limited.

Dielectric Barrier Discharges: These plasmas are typically created between metal plates, which are covered by a thin layer of dielectric or highly resistive material. The dielectric layer plays an important role in suppressing the current: the cathode/anode layer is charged by incoming positive ions/electrons, which reduces the electric field and hinders charge transport towards the electrode. DBD also has a large surface-to-volume ratio, which promotes diffusion losses and maintains a low gas temperature.

a.k.a Microplasmas





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Faraday's Dielectric Capacitors

Michael Faraday (1781 – 1867)



Faraday's Dielectric Capacitor (circa 1837)





Capacitance INCREASED!



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Historical Ozone Tube of W. Siemens (1857)





Werner v. Siemens Poggendorf's Annalen der Chemie und Physik 102, 66 (1857) "Ozone Production in an Atmospheric-Pressure Dielectric Barrier Discharge"



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Dielectric Barrier Discharge



H.E. Wagner, R. Brandenburg, et. al. 'The barrier discharge: basic properties and applications to surface treatment'. *Vacuum*. 71 p417-436 (2003).



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Electric field strength *E* of first breakdown $\approx 150 \text{ Td} (p = 1 \text{ bar}, T = 300 \text{ K})$

Voltage $V_{\rm pp}$	3–20 kV
Repetition frequency f	50 Hz–30 kHz
Pressure p	1–3 bar
Gap distance g	0.2–5mm
Dielectric material thickness d	0.5–2mm
Relative dielectric permittivity ε_r	5–10 (glass)

B. Eliasson and U. Kogelschatz. IEEE Transactions Plasma Science. Vol. 19 Issue 6, 1063-1077 (1991)



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Single and double DBD





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The dielectric is the key for the proper functioning of the discharge.

Serves two functions:

1. Limits the amount of charge transported by a single microplasma

2. Distributes the microplasmas over the entire electrode surface area





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Microdischarge Activity and U-Q Lissajous Figure



B. Eliasson and U. Kogelschatz. IEEE Transactions Plasma Science. Vol. 19 Issue 6, 1063-1077 (1991)



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Fundamental Operation of the Dielectric Barrier Discharge

- Many of relevant plasma processes that are of importance to achieving our goal occur on time scales that allow us to study them.
- Optical emission spectroscopic studies will allow us to determine the temporal and spatial development of important plasma species such as radicals (OH, NO, various oxygen radicals) with high time resolution (less than 10 ns) and a spatial resolution on the scale of mm in the plasma volume following pulsed plasma excitation.



Time scale of the relevant processes of the DBD.





C (0-0) λ = 337.1 nm

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Fundamental Operation of the DBD

Electron Density

Outer Contour Line $n_e = 10^{10} \text{ cm}^{-3}$

Inner Contour Line $n_e = 10^{14} \text{ cm}^{-3}$



Streamer Propagation in 1 bar Air A.A. Kulikovsky, IEEE Trans. Plasma Sci. **25** 439-446 (1997). SETON HALL UNIVERSITY

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Numerical Results of Microdischarge Formation in Dielectric Barrier Discharges



Starting Phase of a Microdischarge (1 bar: 20% CO₂ / 80% H₂)





An electron avalanche propagates towards the anode

Reverse propagation towards the cathode





Numerical Results of Microdischarge Formation in Dielectric Barrier Discharges



Cathode Layer Formation



Just before the peak of the total current



Peak current



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Local Field Collapse in Area Defined by Surface Discharge



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Principals of DBD Microplasmas

Four Different Gap Widths



B. Eliasson and U. Kogelschatz. IEEE Trans Plasma Sci. 19(2) p309 (1991)



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How A Plasma Display Works!



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How A Plasma Display Work!



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Plasma Display Televisions





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Ozone Generator





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Properties of Ozone (O_3)

• Tri-atomic form of oxygen.



- Most powerful commercial oxidizing agent
- Unstable must be generated and used onsite
- Limited solubility in water, but more so than oxygen
- Leaves a dissolved residual which ultimately converts back to oxygen





Discharge Tubes in Ozone Generators





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Traditional Ozone Generator with Glass Tubes



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Generaling Ozone (son's)





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Generation of Ozone

Advantages of Enamel Dielectrics

Proven, Patented Design

- Simplicity
- Single Dielectric Component
 - Reduced number of Dielectrics
- Safety
 - Lower operating voltage
 (< 4000 V)
- Reliabilty
 - Fused Dielectrics ensure continuous production
- Lowest Power Consumption

 Operational Savings!







Modern Ozone Generator

Generating Ozone (con'i)





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Ozonia Advanced Technology Ozone Generator





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Generation of Ozone





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Generation of Ozone

Power Supply Unit





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IGBT Inverter Circuit

Main Circuit Breaker



- Latest in Power Semiconductor Technology, Modular Design
- Utilizes IGBT's (Insulated Gate Bi-Polar Transistors) Converter and Inverter Components





Ozone Water Treatment



Bubble Diffusion

Easy to use

Low energy usage

Mass transfer efficiencies to > 90%



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Municipal Ozone Installations

Key Ozonia Installations (Partial List)

Ozonia Installations	Ozone Plant Size [lb/day]	Start-Up Date
Los Angeles, CA	10,000	1986
Fairfax, VA – Corbalis	9,000	2003
MWD, CA – Mills	9,000	2003
Fairfax Co., VA – Griffith	9,000	2004
MWD, CA – Jensen	18,750	2005
Indianapolis, IN – Belmont	AWT 12,000	2007
Indianapolis, IN – Southpor	t AWT 12,000	2007
MWD, CA – Diemer	13,400	2008
MWD, CA – Weymouth	13,400	2009

Ozonia North America - Potable Water SummaryTotal Number of Installations:90Total Installed Production:> 265,000 lbs/day



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Ozone Water Treatment

MWD Mills WTP - California



3 x 3,000 lbs/day of ozone



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How do you optimize an ozone generator?



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Scientific Approach

Theoretical

$$P = \alpha \cdot 4 f \sum_{i=1}^{n} C_{\text{D},i} \frac{1}{1+\beta_i} U_{\text{min},i} \Big(U_{\text{peak}} - U_{\text{min},i} \Big) \quad [kW]$$

where

i	:	i th slice	
n	:	amount of slices per cylinder []	
Upea	k :	peak voltage [V]	
Umir	n,i :	minimum voltagei th slice [V]	
f	:	frequency [Hz]	
C _{D,i}	:	capacitance of dielectrics ith slice	
α	:	adjustable parameter $[0,\infty]$	
β_i	:	$C_{G,i}/C_{D,i}[]$	





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Experimental Test Rig





Outer grounded electrode (left picture) and the dielectric covered inner electrodes (right).



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Reference (Traditional) Arrangement







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Degrémont Technologies – Ozonia Intelligent Gap System







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Plasma Discharges in Water



liquid phase discharge



Pulsed Corona in Water



Spark Discharge in Water



Spark Discharge in Water



Gliding Arc Discharge with Water Spray



Plasma Arrays in Water



Pinhole Discharge in Water





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Applications of High-Pressure Microplasmas: Light Sources, Photonics, Sensors

Excimer and other non-coherent VUV/UV light sources

- efficiency
- intensity
- wavelength selectivity and control; monochromaticity
- lifetime and stability
- arrays

Photonic devices

- semiconductor devices
 - photodetectors
 - flexible devices and arrays
 - devices approaching cellular dimensions
 - nano-devices

Sensors

- sensor for chemical and biological agents
- sensor for explosives





<u>Capi</u>llary <u>D</u>ielectric <u>B</u>arrier <u>D</u>ischarge







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3-D Expansion







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Pulsed DC Homogeneous DBD



The Dielectric Barrier Discharge (DBD) cell.



A typical plasma in pure nitrogen environment.



Side view of the DBD cell experiment with the fast high voltage transistor switch connected to the bottom electrode.



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Most of the experimental studies are in rare gases and rare gas halide mixtures, with an increasing interest on atmospheric pressure air .

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Human Hair: 60 – 100 μm



<u>Cathode Boundary Layer Discharges (CBLD)</u>



Materials: Electrodes: Molybdenum Dielectric: Alumina

Dimensions:

Electrode Thickness: 100 μm to 250 μm Dielectric Thickness: 100 μm to 250 μm Opening Diameter: 300 μm to 4.5 mm

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Up Scaling

Maintain the sandwich structure and scale up in one direction – *Micro-slit structure*





Parallel operation of multiple openings – *Multi-CBL structure*



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Parallel operation without individual ballast



- Cathode: Mo ~0.25 mm thick
- Dielectric: $AI_2O_3 \sim 0.25$ mm thick
- Anode: Mo ~0.25 mm thick
- Hole diameter: ~0.75 mm
- Center to center distance: ~1.5 mm



Visible Picture of parallel operation of 9 holes (Operating gas: xenon (scientific grade) Base pressure: ~1 mTorr; Working pressure: 200 Torr Cathode voltage: -398 V; Discharge current: 6 mA)



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Self-organization





Xenon (100 Torr)

Xenon (250 Torr) (ignition assisted with mechanical switch)



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More on Self Organization



Self-organization (Visible images) of a microslit CBL discharge:

- (a) 50 Torr;
- (b) 150 Torr;
- (c) 245 Torr;
- (d) 354 Torr and
- (e) homogeneous discharge at 100 Torr (249V and 4 mA)

(The images are at different magnification for a better demonstration purpose)



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Planar Si Electrode

Inverted Pyramidal Electrode

DRIE Electrode









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EXCITATION OF A GREEN PHOSPHOR (Mn:Zn₂SiO₄)

University of Illinois Laboratory for Optical Physics and Engineering

PHOSPHOR EMBEDDED MICROCAVITY







10 % Xe/Ne, 700 Torr



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University of Illinois Laboratory for Optical Physics and Engineering

Microdischarge Array Flat Lamp : Basic Design



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University of Illinois Laboratory for Optical Physics and Engineering







Capillary Plasma Electrode (CPE) Realizations



Solid Pin Electrodes (Cross Flow)



Hollow Pin Electrodes (Flow-Through)



Cylindrical Electrodes (Longitudinal Flow)



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Multi-Capillary Plasma Electrode







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1 Capillary Plasma Electrode



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Capillary Plasma Electrode - Operation





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Solid Oxide Fuel Cell Chemistry

Research and Technology Initiatives

Idea: Use low-T plasma to generate hydrocarbon feed gas for cell



300 kW Fuel Cell

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Microplasma-Assisted Combustion



(Courtesy of M. Gundersen – USC)

USC static reactor for studies of pulsed plasma induced ignition



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Plasma-Aero Experimental System University of Wisconsin (Madison) - Noah Hershkowitz exposed electrode dielectric ~3 mm 0.1 mm **HV** connection edge plasma substrate 12 mm 0.1 mm dielectric buried electrode plasma wide electrode substrate 15 mm 1 mm 🔺

- Planar electrodes are 0.08 mm aluminum tape
- Wire electrode is 0.38 mm diameter copper wire
- Dielectric layer is 0.1 mm polyethylene, $\epsilon \approx 3.2 \epsilon_0$

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Plasma Actuators – The future of Flying!?!



Wing-less planes!!!



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Surface Effects of Microplasmas

For instance, if we want to modify the surface of a material (e.g. a silicon wafer)





Small changes at the surface



Energy & reactive species can change the surface



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Plasma Application in Medicine

Direct Plasma – Charges on Tissue, Produced <u>In</u> Air or Oxygen



Indirect Plasma – Jet, Often <u>NOT</u> in OXYGEN





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DC MHCD Plasma Micro Jet



- Dimensions of the device are:
- Opening: 0.8 mm in diameter
- Separation: 0.5 mm
- Depth of exit opening: 1 mm
- Electrode material: copper
- Dimension of the plasma jet are
- •~ 800 μ m in diameter
- •8 -10 mm in length
- Flow rate: 2-3 SLM
- Power consumption: 8 W (400 VDC, 20 mA)







Plasma Micro Jet Inside Water







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Inactivation of Bacteria

Experimental Set-up



Experimental Procedure

Total path length:120 mmPositiveMoving speed:4 mm/sTime per path:30 sTotal treatment time:30s / 60s / 90 sArea exposure/path:< 1 s (visible plasma),~10 s (radical exposure)</td>

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	Bacteria	stain
Α	Escherichia coli	Negative
В	Staphylococcus aureus	Positive
С	Micrococcus luteus	Positive
D	Bacillus megaterium	Bositive
Е	Bacillus subtilis	Positive
F	Bacillus natto	Positive



Plasma Dose Effect



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SEM Pictures

SEM pictures of S. aureus before and after PMJ treatment



Control

PMJ treatment

SEM of PMJ treated S. aureus show clear poration on cell membrane as well as the change of the cell morphology.

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Living tissue sterilization without harm: Recent pig experiments



Courtesy: Drexel Plasma Institute



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Hemostasis and coagulation in Hairless mice, not immunocompromised (SKH₁)





Saphenous vein cut: without plasma animal continues to bleed for 10-20 minutes. 15 seconds of FE-DBD clots the blood and seals the vessel <u>without damaging</u> <u>tissue</u>, preventing additional bleeding.

Courtesy: Drexel Plasma Institute



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Biological Mechanisms: Plasma Interference into Natural Intracellular Biochemistry

Biological sample





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Dental Application







Cleaning of Dental Cavities Other Applications

- Bio Decontamination
- Sterilization of Medical Instruments and Wounds





- The plasma jet did not heat tooth surface over 37 degrees.
- Heating the tooth over 42 degrees can causes severe damages to the nerves inside a tooth.

"No thermal-damages"

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Teeth Whitening with low temperature plasma















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A Brief Collection of Atmospheric Pressure Plasma Jets (APPJ)



Gases used: Helium, Argon... or mixed with reactive gases $(O_2, CH_4...)$ AC, pulsed DC, rf or microwave



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Our Version of the Atmospheric Pressure Plasma Jet



Interaction with aqueous environments



Interaction with organic surfaces



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Jet Length vs. applied voltage



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Plasma in a Curved Teflon Tubing







Distance the streamer can travel inside the insulating tubing depends on applied voltage, location of the powered electrode, type of working gas.





Move plasma jets in multiple directions





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3 holes (diameter: 1/16") on side wall



Further Extension of these Plasma Jets







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Water irrigation in fields and greenhouses





Plasma irrigation for agriculture

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Plasma Seed Treatments



(a) Side-view of basil seedlings grown from plasma treated seeds (left) and untreated seeds (right). (b) Top-view of basil seedlings grown from plasma treated seeds (left) and untreated seeds (right).



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Basil: Plasma Treated vs. Untreated



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Basil: Plasma Treated vs. Untreated



Graph demonstrating average final height of twelve treated and nontreated sweet basil plants after a month of growth from seeds.



Low Temperature Plasma interaction with biological materials???

PLASMA



Many unanswered questions as to the role of plasma in the biological interactions with biological materials.

- What are the low temperature plasmas doing to the live biological materials?
- Can low temparture sources be tailored to better control interactions with biological materials?



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Acknowledgements

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IEEE Transactions on Plasma Science





IEEE TRANSACTIONS ON PLASMA SCIENCE





Jose L. Lopez – Seton Hall University Senior Editor of Industrial, Commercial, and Medical Applications of Plasmas



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LABORATORY OF ELECTROPHYSICS & ATMOSPHERIC PLASMAS (LEAP)

11





Two M.S. in Physics Degree Tracks:

- 1. Course track (33 credits) for educators / doctoral degree (Ed.D.) and business tracks (M.B.A)
- 2. Master's Thesis (30 credits) for R&D research or scientific research doctoral degree (Ph.D.)

Research Areas:

- 1. Plasma Physics Science & Technology
- 2. Condensed Matter / Complex Matter Physics
- 3. Biophysics & Environmental Physics
- 4. Environmental Systems & Technologies



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The future ain't what it used to be...Yogi Berra





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Questions???





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Thank You!

Jose L. Lopez, Ph.D.



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