## Low Temperature Plasma III: Laser Diagnostics for Gases and Plasmas

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PPPL Graduate Summer School, August 2020



## **Applied Physics Lab – MAE Department Research Areas and Applications**



#### **Princeton University**



http://www.princeton.edu/~adogariu/

# Why Optical Diagnostics for Gases and Plasmas?

### **Desired properties**

- Non-intrusive
- Standoff/Remote
- Single shot (real time)
- Fast repetition rate
- High temporal resolution
- High spatial resolution
- High sensitivity and specificity
- Imaging capability

#### Measurements

- Species
- Concentration
- Density
- Temperature
- Pressure
- Velocity
- E-, B-field



## **PPPL-Princeton Collaborative LTP Research Facility (PCRF)**



#### **PCRF** Princeton Collaborative Low Temperature Plasma Research Facility

Links

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#### About



The Princeton Collaborative Low Temperature Plasma Research Facility (PCRF) is focused on low temperature plasma physics and is open to all users.

The PCRF provides state-of-the-art research capabilities and expertise for comprehensive characterization of low temperature plasma (LTP) properties with the goal to advance methods of predictive control of LTP with a focus on plasma-liquid and plasma-solid interactions, collective phenomena in LTP, and use of LTP in modern applications (e.g. material synthesis and processing).

The facility is formed from the existing low temperature plasma laboratories at PPPL and the Mechanical and Aerospace Engineering (MAE) Department of Princeton University (PU), with a total collective lab space greater than 7000 sq. ft., each located within 3 miles from each other.

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- https://pcrf.pppl.gov/
- https://pcrf.pppl.gov/facilities/advanced <u>diagnostics/index.html</u>

### **Princeton University**



#### Facilities Personnel

Information for Users Links

#### Personnel



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## Laser-based techniques

- TALIF (Two-photon absorption Laser Induced Fluorescence)
- Radar **REMPI** (Resonantly Enhanced Multi-Photon Ionization)
- FLEET (Femtosecond Laser Electronic Excitation Tagging)
- CARS (Coherent Anti-Stokes Raman Scattering spectroscopy)
- E-FISH (Electric field induced second harmonic generation)



## **LIF: Basic Principles**

What is Laser Induced Fluorescence (LIF)?

- Spontaneous emission resulted from the resonant absorption of the laser radiation by atoms or molecules
- A two-step process: excitation-emission



# **Typical Experimental Setup**



#### **Recorded data**

- Spatially resolved 2D imaging
- Temporally resolved gated intensifier

#### Measurements

- Species concentration
- Species composition
- Density
- Temperature
- Mixture fraction

## Simultaneous multi-species via PLIF



flame front structure visualization using simultaneous singleshot PLIF imaging of CH, OH, and CH<sub>2</sub>O in a piloted premixed jet flame

Z.S. Li \* 🖄 🖾, B. Li \*, Z.W. Sun \*, X.S. Bai <sup>b</sup>, M. Aldén \*

# 60 m/s 90 m/s 120 m/s 150 m/s

10 m/s

30 m/s

OH CH<sub>2</sub>O



## Two-Photon Absorption Laser Induced Flourescence (TALIF)

- If the transition to the excited state is higher than ~7eV, one photon excitation is prohibited in air (<180nm) – multiphoton transitions required.
- Laser sources deep in the UV (100-150nm) are prohibitive.
- Two-photon transitions: easy to achieve with short pulse lasers.



# TALIF of O in an atmospheric pressure plasma jet (APPJ)



- Capillary dielectric barrier discharge
- Two-photon excitation at 226nm
- Emission at 845nm
- Calibration with Xe



Atomic oxygen TALIF in a  $2\% O_2$ /He mixture APPJ

Schmidt et al 2017 Plasma Sources Sci. Technol. 26 055004



# Measurements of H density in RF helicon plasma

Goal: non-invasive measuring of neutral H concentration, dynamics of production and depletion under both steady state and pulsed RF plasma

- Quantify neutral density (H)
- Image the H density
- Time-resolve neutral concentration





Plasma mirror device for FRC (Field Reversal Configuration) – RF heating for quasisteady state magnetized cylindrical plasmas Sam Cohen group @PPPL



# **Fs-TALIF** in **H**

- Broadband two-photon excitation:
  - Very efficient (high intensity fs)
  - Low energy per pulse
  - Fast excitation (no quenching)
  - kHz dynamics
- H pump at 205nm, record at 656nm
- Kr seeding for density calibration







## H-density dynamics – CW helicon plasma





## H-density dynamics – pulsed RF plasma





## **REMPI for atomic spectroscopy**

#### **Resonantly Enhanced Multi-Photon Ionization:**

- An intense laser pulse ionizes the atom and creates charges/plasma.
  - The ionization is strongest when the photon(s) energy equals the energy difference between excited and ground state.
  - Extra photons bring the energy above the ionization energy of the atom (the energy required to remove one electron from an isolated, gas-phase atom).
  - Example: 2+1 REMPI = 2 photons to excite and 1 to ionize.
- Very high sensitivity and excellent selectivity
- Usually requires detection with electrodes or ion mass spectroscopy at low pressures.





# Radar REMPI – detection via microwave scattering





"Microwave Diagnostics of Small Plasma Objects," J. Appl. Phys. **98**, 0033301-1 – 033301-3 (2006).

**Radar REMPI -***The focused laser creates a small region of ionization which scatters the microwaves.* 

- Selectivity and sensitivity: independent!
  - Selectivity: laser wavelength ( $\Delta\lambda \approx \text{cm}^{-1}$ )
  - Sensitivity : microwave detection
- Truly standoff backscattering detection
- Non-intrusive, localized (laser spot)
- No daylight optical interference
- Bonus: sub-nanosecond temporal resolution!

Patent US7728295 (2010)





# Radar REMPI for remote sensing in plasma and gases



- Gas density and temperature, nanoparticle charge, negative ions
- Direct measurement of plasma density and of electron recombination and attachment in air

- Microwave (10-100GHz) probes the plasma.
- The mixer output is proportional with the scattering amplitude, hence electron density
- Linear signal from ppm to ppb
- Sub-nanosecond temporal resolution



## **Trace Species Detection – Nitric Oxide**

- Linearity from ppm to ppb
- High temporal resolution
- NO detection sensitivity: ~ ppb



A. Dogariu and R. B. Miles, Detecting localized trace species using Radar REMPI," Appl. Opt. **50**, A68 (2011)



## Direct measurement of electron attachment and recombination rates in atmospheric air



1. NO in N<sub>2</sub> - recombination only  $N(t) = \frac{N_0}{1 + \beta N_0 t} \quad N_0 = 2.5 \times 10^{14} \text{ cm}^{-3}$ Electron density measurement!

2. NO in air - recombination and attachment

$$N(t) = \frac{N_0 e^{-\nu_a t}}{1 + \frac{\beta N_0}{\nu} (1 - e^{-\nu_a t})} \quad \boldsymbol{\nu_a} = 0.76 \times 10^8 \, \text{s}^{-1}$$

**Electron attachment rate measurement!** Theoretical prediction  $v_a \cong 0.8 \cdot 10^8 s^{-1}$ 

Dogariu et. al, Appl. Phys. Lett. 103, 224102 (2013)

# Electron density and its dynamics in atmospheric pressure plasma jet (APPJ)

APPLIED PHYSICS LETTERS 96, 171502 (2010)

## Temporary-resolved measurement of electron density in small atmospheric plasmas

A. Shashurin, <sup>1,a)</sup> M. N. Shneider, <sup>2</sup> A. Dogariu, <sup>2</sup> R. B. Miles, <sup>2</sup> and M. Keidar<sup>1,a)</sup> <sup>1</sup>Department of Mechanical and Aerospace Engineering, School of Engineering and Applied Science, The George Washington University, Washington DC 20052, USA <sup>2</sup>Department of Mechanical and Aerospace Engineering, Princeton University, Princeton, New Jersey 08544, USA



FIG. 4. (Color online) Temporal evolution of average plasma density in atmospheric plasma jet for  $U_{\rm HV}$ =2.7 and 3.8 kV.





FIG. 1. (Color online) (a) The schematics of RMS experimental setup. (b) Typical scattering signals measured by RMS setup induced by calibrator bullet flying over the microwave horns along *z*-axis [in-phase (*I*) and quadrature (*Q*) components, and total amplitude of output signal *U*]. Teflon bullet of 8.5 mm length and 3.2 mm diameter was used. (c) Typical image of plasma jet for  $U_{HV}$ =3.8 kV, He flow of 15 1/min.

# **Radar REMPI for Hall Thrusters**

- Apply Radar REMPI to Hall thrusters for neutral xenon measurements
- Measure Xe density and temporal dynamics (sub-ns resolution)
- Use 2+1 REMPI in Xe:He mixtures at <1 Torr</li>
- New effect: measure magnetic field using depolarization of microwave scattering.



Magnetically induced depolarization of microwave scattering from a laser-generated plasma, *Phys. Rev Appl.* **12**, 034055 (2019).

## **Princeton University**



#### 2 kW Hall thruster at PPPL





## FLEET: A new molecular tagging diagnostic

- Femtosecond focused beam (filament)
- Using nitrogen for unseeded flow velocimetry : imaging N<sub>2</sub> emission
- N<sub>2</sub> dissociation ⇒ delayed N-N recombination into N<sub>2</sub><sup>\*</sup> → emission from excited nitrogen.

Femtosecond Laser Electronic Excitation Tagging

Femtosecond laser electronic excitation tagging for quantitative velocity imaging in air, Appl. Opt. 50, 5158-5162 (2011).

Patent US9863975 - Femtosecond laser excitation tagging anemometry (2018)



## **Mechanism for FLEET**



## Velocimetry – underexpanded sonic jet



- Unseeded air or nitrogen flows of gases and plasmas
- Single shot measurements
- High spatial resolution (tens of microns)
- Pressure range: from <1 Torr to</li>
   >> 1 atm
- Any gas temperature



## **Applications to hypersonic flows**

#### Hypersonic vehicles (Mach >5)

SCRAMJET engines (supersonic RAM)

- NASA X43 (Mach 9, 2004)
- AFRL X51 (Mach 7, 2013)
- Lockheed SR-72 (Mach 6, 2018)











## Hypersonic Wind Tunnel 9, AEDC White Oak, MD



#### **Optical diagnostics for hypervelocity flows**



Hypersonic wind tunnel: Nitrogen Run time: ~1-3 seconds! Simultaneous experiments:

- FLEET (flow velocity mapping)
- CARS (vibrational temperature)
  2016-2018 MACH 10 14
  2019-2020 MACH 18

Photos courtesy of AEDC

## **FLEET System installed at Tunnel 9**



#### Photos courtesy of AEDC



The laser system, optics and optomechanics components are installed on a transportable table placed in close proximity to the tunnel. Princeton University The gated intensifier, highspeed camera, and the zoom lens are placed on top of the tunnel.

## **Freestream Flow Velocimetry**



#### First direct flow measurement in Tunnel 9!

2017-2018 – Mach 10 and Mach 14 2019-2020 – Mach 18 Tunnel 9, AEDC Air Force Hypersonic Facility

## **Princeton** University



#### FLEET instantaneous mean velocity and Tunnel 9 velocity prediction







0.8 9 / 2 0.6

0.4

0.2

0 L 0

0.2

0.4

0.6

u/u

0.8

1



Dogariu et al., "Hypersonic Velocity Measurements in Large Scale Wind Tunnel Using FLEET", AIAA Journal 57, 4725 (2019)



## **APPJ - LTP plasma jet**

## Argon/Nitrogen flow imaging



Atmospheric pressure plasma jet (APPJ)



## APPJ time resolved imaging using FLEET: Argon and Nitrogen mapping

#### Argon flow velocity mapping



Nitrogen (entailed air) (100µs delay)







## **Spontaneous Raman Scattering**



#### Chandrasekhara Venkata Raman (1888-1970)

1930 Nobel Prize in Physics





anti-Stokes scattering



## **Coherent Anti-Stokes Raman Scattering (CARS)**



## Hybrid CARS background free



#### Optimizing the Laser-Pulse Configuration for Coherent Raman Spectroscopy Dmitry Pestov, et al.

Science **316**, 265 (2007); DOI: 10.1126/science.1139055



- Femtosecond pumping allows broadband excitation, single shot spectroscopy
- Picosecond probing narrowband probe provides spectral resolution
- Delay eliminates background
- kHz repetition rate
- "Optimizing the Laser-Pulse Configuration for Coherent Raman Spectroscopy," Science 316, 256 (2007).
- "Real-time detection of bacterial spores using Coherent anti-Stokes Raman Spectroscopy," J. Appl. Phys. 103, 036103 (2008).
- *"Real-time monitoring of blood using coherent anti-Stokes Raman spectroscopy," J. Biomed. Opt.* **13**, 54004 (2008).
- "Coherent Anti-Stokes Raman Spectroscopy for detecting explosives in real-time," Proc. SPIE, 8358-27 (2012).



## **Hybrid CARS measurements**

- Gas density and temperature
- Solid/liquid molecular composition
- Surface changes under plasma interaction



Femtosecond hybrid CARS setup for measurements in solid/liquid/gas



# Application: Real-time *B. subtilis* spore detection 200 spores in 50 milliseconds



# Application: Real time CARS spectroscopy for trace explosives detection



- Backscattered CARS spectra are obtained in 100ms.
- Spectra can be analyzed at video rates.

#### Detection limit: ~ 20ng @ 1m (<1ng @30cm)

A. Dogariu and A. Pidwerbetsky, "Coherent Anti-Stokes Raman Spectroscopy for detecting explosives in real-time," Proc. SPIE, 8358-27 (2012).

### **Princeton University**



K. P. Pfeuffer, T. Le, L. E. Dogariu, D. Zipse, and A. Dogariu, "Characterization and applications of a fieldable single-laser standoff CARS detection system," Opt. Eng. **59**, 092007 (2020).

# **Hyper-Spectral Imaging**



#### AN + KClO<sub>3</sub>











500 x 500µm





8x4mm

\_

Spray deposit

Dry transfer

4x4mm

- Raster scanning at 1m standoff
- Fotal energy at the sample: 10µJ
- Sample density 100µg/cm<sup>2</sup>

Samples provided by DHS



## **Biomedical Applications**



#### **Mammalian Cancer**: real-time noninvasive spectroscopy of tumors



**Remote skin evaluation**: Hair and skin spectroscopy in real-time

## **Princeton** University







**Blood monitoring**: 20,000x faster, real-time, non-invasive, no sample preparation

Dogariu et al., J. of Biomed. Opt. 13(5), 054005 (2008)

## Gas and Plasma Applications: Remote CARS Thermometry

Equilibrium vibrational and rotational Raman spectra at equilibrium - Boltzmann distribution



using collinear hybrid CARS

43

Theoretical (Sandia CARSFIT code)

## **Vibrational Temperature in Hypersonic Flow**





- First ever real-time temperature measurements in Tunnel 9 (March 2018)
- Single shot, 1kHz (<1s run time)</li>
- Mach 14, 1,500m/s, 1 Torr Nitrogen
- Validate presumption of non-equilibrium temperature

## Validation of Non-Equilibrium Temperature



**Rotational temperature ~55K** 

Vibrational temperature ~1300K

- Rotational temperature as expected in equilibrium with translational T
- Vibrational energy frozen, high non-equilibrium predicted by Computational Fluid Dynamics (CFD) and measured by CARS in real time (@1kHz)

A. Dogariu, L. E. Dogariu, M. S. Smith, J. Lafferty, and R. B. Miles, "Single Shot Temperature Measurements using Coherent Anti-Stokes Raman Scattering in Mach 14 Flow at the Hypervelocity AEDC Tunnel 9," AIAA SciTech 2019 Forum, 1089 (2019).



# Hybrid CARS thermometry in combustion



# Hybrid CARS thermometry in ns plasma discharge



- Non-equilibrium temperature and dynamics from vibrational spectrum of nitrogen in a nanosecond plasma discharge.
- Ns plasma discharge 18ns, 10-20kV
- Plasma glow discharge 10-40 Torr air





# Rotational and Vibrational Temperature in Plasma Discharge



- Non-equilibrium temperature and dynamics at 10 Torr.
- High vibrational temperature (~1400K)
- Low rotational temperature low (~400K)
- Non-equilibrium dynamics ms time scale
- Plasma glow discharge 10-40 Torr air





## **Second Harmonic Generation (SHG)**

$$P = \chi \varepsilon_0 E \qquad P = \varepsilon_0 (\chi^{(1)} E + \chi^{(2)} E^2 + \chi^{(3)} E^3 + \cdots)$$

• For SHG, second order polarizability:

 $P^{(2)} = \varepsilon_0 \chi^{(2)} E^2$ 

 Second order nonlinear interactions can only occur in non-centrosymmetric media

- **P**: Induced polarization
- *χ*: Electric Susceptibility
- ε<sub>0</sub>: Permittivity of vacuum
- E: Electric Field



- Harmonic generation in gases?
  - Second harmonic generation is not possible using gases as the nonlinear medium, since they are isotropic materials" Laser Fundamentals 2<sup>nd</sup> Edition by William T. Silfvast
  - While in a centrosymmetric medium second harmonic generation is impossible, applying an electric field destroys the symmetry and allows SHG



# fs E-FISH – use ultrafast lasers to measure electric field in gases and plasmas

#### Electric Field Induced Second Harmonic Generation (E-FISH)





## **Properties of E-FISH for E-field** measurements in gases and plasmas

2500

2000

Electric Field (V/cm)

Iormalized FLEM Response

## **Quadratic Dependence on E-Field**

- Sensitive down to 100's of V/cm
- **Time Resolution** 
  - Femtosecond laser pulsewidth
- **Spatial Resolution** 
  - Sub-mm, determined by the focal volume

## **Species Independence**

Non-Resonant technique that can be used in any gaseous mixture

## **Field Vector Sensitivity**

Nonlinear susceptibility polarization allows for<sup>100</sup> measuring the field vector components



**Princeton University** 

360

2000

20

## E-FISH in Atmospheric Pressure Plasma Jet (APPJ)

- Streamer front propagation in filamentary plasma jets
- 2D E-FISH mapping electric field with sub-ns temporal resolution



Space-Time resolved E-Field in cold Plasma Jet

## **Princeton University**

t= 10 ns

t= 15 ns



## 2D E-Field dynamics in ns Surface Dielectric Barrier Discharge (SDBD) using E-FISH





- Ns pulses applied to DBD measure/image E-field in atmospheric discharge
- Cylindrically focused beam allows for imaging of E-Field
- Fast gating allows sub-ns temporal resolution

## **Princeton University**





Meehan et al, "Two Component Electric Field Dynamics of a ns-SDBD Plasma with Sub-Nanosecond Resolution by Femtosecond EFISH," AIAA Scitech 1747 (2020)

#### Detection of freely propagating terahertz radiation by use of optical second-harmonic generation

#### Ajay Nahata and Tony F. Heinz

Departments of Electrical Engineering and Physics, Columbia University, New York, New York 10027

#### Received August 6, 1997

We report the application of electric-field-induced optical second-harmonic generation as a new technique for measuring the field of freely propagating terahertz radiation. Using silicon as the nonlinear medium, we demonstrate subpicosecond time resolution and a sampling signal that varies linearly with the terahertz electric field. This approach, which is attractive for centrosymmetric media, permits a significantly broadened class of materials to be exploited for free-space sampling measurements. 1998 Optical Society of America OCIS codes: 160.4430, 240.4350, 320.7110, 190.2620, 350.5610.





Fig. 1. Schematic of the experimental setup. PMT, photomultiplier tube. Inset: Detail of the electric-field-induced SHG configuration.





Fig. 3: (a) Mechanisms generating THz waves by intense twocolor laser pulses, distributed according to the optical intensity. The first region involves the Kerr effect (four-wave mixing) and photoionization. The second region accentuates the contribution of photoionization in the tunnel regime (photocurrents) and involves plasma waves created by ponderomotive forces. (b) Photocurrent process: the two-color electric field generates free electrons via tunneling ionization occurring near the field extrema at  $t = t_n$ . This builds a slow component of the current that acts as a THz source.



EPL, **126** (2019) 24001 doi: 10.1209/0295-5075/126/24001 www.epljournal.org

April 2019

#### Perspective

## Terahertz spectroscopy from air plasmas created by two-color femtosecond laser pulses: The ALTESSE project

L. BERGÉ<sup>1</sup>, K. KALTENECKER<sup>2</sup>, S. ENGELBRECHT<sup>3</sup>, A. NGUYEN<sup>1</sup>, S. SKUPIN<sup>4</sup>, L. MERLAT<sup>3</sup>, B. FISCHER<sup>3</sup>, B. ZHOU<sup>2</sup>, I. THIELE<sup>5</sup> and P. U. JEPSEN<sup>2</sup>

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<sup>5</sup> Department of Physics, University of Gothenburg - SE-412 96 Göteborg, Sweden





## **Optical Diagnostics for Gases and Plasma**

- Remote backwards air lasing
- Remote trace gas detection using Radar REMPI
- Remote magnetometry with atomic Xe
- Two-photon absorption laser induced fluorescence (TALIF)
- Flow velocimetry -Fs Laser Electronic Excitation Tagging (FLEET)
- Standoff real-time molecular detection and imaging using coherent Raman (CARS)
- Remote gas thermometry (CARS)
- Femtosecond Localized Electric Field Measurement (EFISH)
- Slow Light Imaging Spectroscopy (SLIS) Spectroscopy without spectrometer



## Acknowledgements



Richard Miles (Texas A&M, Princeton)



Mikhail Shneider (Princeton)



Ben Goldberg (Sandia Nat. Lab, CA)



Chris Galea (Princeton)



T. L. Chng (Ecole Polytech., Paris)



Matthew Edwards (Lawrence Livermore National Laboratory)



James Michael (Iowa State Univ.)



Stephan Reuter (Polytechnique Montreal)

## **Princeton** University



#### REAL PLASMA REAL PLASMA TEC

**Collaborators:** 









Homeland Security

Science and Technology



Chemring

Sensors & Electronic Systems





University of Pennsylvania School of Medicine University of Pennsylvania Health System



#### **PCRF** Princeton Collaborative Low Temperature Plasma Research Facility

Facilities Personnel Information for Users Links

## $PCRF \ \ \, Princeton \ \, Collaborative \ \, Low \ \, Temperature \ \, Plasma \ Research \ \, Facility$

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#### Information for Users

#### **Proposal Information**

A copy of the joint call and submission process instructions can be downloaded here.

A copy of the user proposal template can be downloaded here.

A copy of the available equipment at PCRF can be downloaded here.

Currently users can submit applications to Yevgeny Raitses via email (yraitses@pppl.gov). In your email, please include:

- your name
- affiliation
- title of proposal
- attached completed proposal document

#### **Current Call for Proposals**

Opening call for proposals: November 4, 2019

Closing call for proposals: December 20, 2020

Review of proposals: December 21, 2019 to January 20, 2020

Deadline for decisions: by February 3, 2020

The facilities will consider out-of-cycle proposals throughout the year depending on facility utilization. Interested applicants should contact the respective facilities.

- <u>https://pcrf.pppl.gov/</u>
- <u>https://pcrf.pppl.gov/user%20info/index.html</u>

