This set of slides consists of a collection of short presentations on different topics, all related to plasma diagnostics. During my presentation I will use the first ‘introductory’ presentation as a guideline. I will discuss some diagnostics in more detail, and make a selection of the applications of diagnostics, depending on the audience.

Plasma Diagnostics
- how to study molecule formation in plasma ? -

Richard Engeln
“your working gas mixture $\neq$ input gas mixture”

*(at high dissociation degree)*

quote from Prof. J. Winter during his lecture during the 2005 Summer School on Low Temperature Plasma Physics: Basics and Applications
Molecule Formation in Plasma

Plasma source

O₂ plasma impinging on a substrate

O₂ plasma expansion

substrate

Introduction

taken from: A. Lebéhot *et al.* in ‘Atomic and Molecular Beams’, ed. R. Campargue
**Molecule Formation in Plasma**

\[ \text{O}^{(3P)}_{\text{atm}} + \text{NO}_{\text{ads}} \rightarrow \text{NO}_2\{^2B_1\} \rightarrow \text{NO}_2\{^2A_1\} + h\nu \]

N\(_2\) plasma with O\(_2\) injected in the background
Molecule Formation in Plasma

**Introduction**

**Vibrational Excitation of Hydrogen via Recombinative Desorption of Atomic Hydrogen Gas on a Metal Surface**

R. I. Hall, I. Čadež(a), M. Landau, F. Pichou, and C. Schermann

Groupe de Spectroscopie par Impact Electronique et Ionique, Université Pierre et Marie Curie, 75252 Paris Cedex 05, France

(Received 20 July 1987)

Recombinative desorption of atomic hydrogen on the walls of a gas cell has been observed to populate vibrational levels up to \( v = 9 \). The vibrational populations follow a Boltzmann distribution near 3000 K up to \( v = 3 \) and for higher levels the populations are well in excess of this temperature. These observations bring to light a new mechanism for vibrational excitation of \( \text{H}_2 \) in volume \( \text{H}^- \)-ion sources.

PACS numbers: 79.20.Nc, 34.80.Gs, 52.40.Hf

**FIG. 3.** Vibrational populations against vibrational energy on a semilog scale for tungsten and tantalum filaments.

\[ \text{H}_2^{r, v} \text{ detection via QMS detection of } \text{H}^- \]

\[ \text{H}_2 + e \rightarrow \text{H}^- + \text{H} \]
Molecule Formation in Plasma

Dark (dense) clouds
✓ 10-30 K / $10^4$-$10^8$ part./cm$^3$
✓ Universal molecule factory

Diffuse (translucent) clouds
✓ 40-100 K / 100 part./cm$^3$
✓ Unknown absorption features

(from: H. Linnartz, CRD meeting (2004))
Questions when studying molecule formation in plasma?
(when in contact with a surface)

- What particles are arriving at the surface?
- In which state are the particles arriving?
- New molecules are generated:
  - electronically and/or ro-vibrationally excited?
  - substrate material and temperature dependence?
- Is there flux dependence on the generation process?
Questions when studying molecule formation in plasma?
(when in contact with a surface)

What is needed to answer these questions?

- What particles are arriving at the surface?
- In which state are the particles arriving?
- New molecules are generated:
  - electronically and/or ro-vibrationally excited?
  - substrate material and temperature dependence?
- Is there flux dependence on the generation process?
Gas-phase optical diagnostics for the detection of stable molecules and atomic/molecular radicals

- **(VUV) Laser Induced Fluorescence**
  - relative densities, + spatial resolution

- **Fourier Transform IR/UV absorption**
  - line of sight, + absolute densities, + large $\lambda$-range (overview spectrum)

- **(Cavity Ring Down) absorption**
  - line of sight, + (very) high sensitivity

- **(spontaneous) Raman spectroscopy**
  - ‘low’ sensitivity, + every molecule Raman active, + spatial resolution
# Plasma Diagnostics (optical)

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<td>Cavity Ring Down absorption</td>
<td>n(v,J), T</td>
<td>NH, NH$_2$, NH$_3$</td>
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All diagnostics are complementary.
On diagnostics (general):


Laser Induced Fluorescence spectroscopy
Laser Induced Fluorescence (LIF)

- Number of laser photons $n_a$ absorbed in unit volume and time:

$$n_a = \sigma_{li} I_L n_l$$

- Number of fluorescence photons $N_f(\lambda_{ik})$ originating from $V$:

$$N_f = n_a V q_f = \sigma_{li} I_L n_l V \frac{A_{ik}}{A_i + R}$$

- Signal $S_f$:

$$S_f = N_f \cdot \Omega / 4\pi \cdot T \cdot q_{ph}(\lambda) \cdot G_{ph}$$

$$S_f \propto n_l$$
Laser Induced Fluorescence (LIF)

**Advantages**
- sensitive
- extra info from time behaviour
- experimentally straightforward
- possibility of 2D-imaging

**Disadvantages**
- not quantitative
- depending on gas composition (quenching)

How to detect the hydrogen atom in the ground state with LIF?
Advantages

- no demanding VUV-generation
- non-resonant fluorescence detection possible
- self-absorption can be avoided

Disadvantages

- low 2-photon cross sections require high laser intensities
- 2-photon cross sections often not known
Quantities deduced from LIF

- integrated intensity: \( n \)
- Doppler width: \( T \)

\[
\frac{\Delta \nu_D}{\nu} = \frac{1}{c} \sqrt{\frac{8 \ln 2 \cdot kT}{M}}
\]

- Doppler shift \( \nu - \nu_0 \): \( \nu \)
Applications:
- fast deposition of a-Si:H
- H-source
- surface passivation

Applications:
- deposition of a-C:N
- plasma etching (photo-resist)

2 photon LIF on atoms

monitoring H

- $3s, d$ to $2p$
- $205 \text{ nm}$ to $656 \text{ nm}$
- monitoring $H_\alpha$

monitoring N

- $3p$ to $3s$
- $207 \text{ nm}$ to $745 \text{ nm}$
(VUV) LIF on $\text{H}_2$ molecules

Excitation from $\text{H}_2(X, \nu=0)$ to $\text{H}_2(B)$

Photons with energy $\approx 11$ eV
($\lambda \approx 110$ nm, Vacuum UV)

Fluorescence of $\text{H}_2$ in B-state

$\lambda$ in the Vacuum UV
Absorption spectroscopy
Absorption spectroscopy

Lambert-Beer: \[ I(\nu) = I_0(\nu) \exp[-\kappa_\nu l] \]

Absorption: \[ \kappa_\nu = n(\nu,J)\sigma_\nu \]

If \( \kappa_\nu l \) is small: \[ \frac{\Delta I}{I_0} = \kappa_\nu l \]
IR laser absorption spectroscopy
IR laser absorption spectroscopy

Trace gas detection

Transmission

Wavenumber [cm\(^{-1}\)]

CH\(_4\), N\(_2\)O, H\(_2\)O
Fourier Transform absorption spectroscopy

interferogram
**O₂ FTIR measurement in a vessel**

![Diagram of FTIR measurement in a vessel]

**Graph 3**

**Intensity (a.u.)**

**Frequency (cm⁻¹)**

- **Background**
- **Sample**

FTIR absorption
O₂ FTIR measurement in a vessel
**IR laser absorption**

- Very high wavelength resolution
- High sensitivity

**FT IR absorption**

- Multiplex advantage
- Very large wavelength range

- Low wavelength resolution
- Sensitivity

Homo-nuclear diatomic species **not** detectable in IR
Sensitivity

\[ \frac{\Delta I}{I_0} \geq 10^{-3} \] (pulsed lasers)

\[ \kappa \cdot I \geq 10^{-3} \]

Example: \( l = 0.1 \ \text{m} \), \( \sigma = 10^{-18} \ \text{m}^2 \)

\[ n(v, J) \geq 10^{16} \ \text{m}^{-3} \]

\[ N_{tot} = \sum_{v,J} n(v, J) \geq 10^{18} \ \text{m}^{-3} \]

Alternative schemes:
- Fourier Transform spectroscopy (multiplex, but low sensitivity)
- Cavity Ring Down spectroscopy (high sensitivity)
Sensitive direct absorption technique


\[
d\frac{\ln R}{c\tau} = \frac{1}{d}\left(1 - R + n\sigma L\right)
\]

- absorption per unit of pathlength (cavity loss):
  \[ 1/c\tau = (1 - R + n\sigma L) / d \]
- non-intrusive and remote
- high sensitivity due to effective multipassing
- direct absorption \( \rightarrow \) line of sight measurement
Basic scheme of the pulsed CRD spectrometer

Ring-down time

\[ \tau = \frac{d}{c(1-R+n\sigma L)} \]

Cavity loss

\[ \frac{1}{c\tau} = \frac{1-R}{d} + \frac{n\sigma L}{d} \]
Performing a pulsed CRD experiment

Ring-down transient

CRD spectrum
Performing a pulsed CRD experiment

Cavity loss (1/cm)

$$\frac{1}{c\tau} = \frac{1-R}{d} + \frac{n\sigma L}{d}$$

If cavity length is 45 cm, determine R.

1. R = 99%
2. R = 99.9%
3. R = 99.99%
4. Not enough information

CRD absorption spectrum
+ optical technique
+ independent of intensity
+ direct absorption measurement:
  -- but: line-of-sight
+ high sensitivity due to effective multipassing
+ pulsed light sources: spectral range into the UV
+ experimentally straightforward (tunable laser, highly reflecting mirrors, PMT, ‘fast’ and ‘deep’ digitizer)

**high potential for diagnostics in plasmas**
ETP setup

Plasma created at high pressure (~400 mbar) in cascaded arc plasma source

Expansion into low-pressure chamber (0.2 mbar) + injection of e.g. SiH$_4$

Plasma in interaction with surface, leading to e.g. deposition or etching
CRD for the detection of SiH during a:Si-H deposition
CRD spectrum of SiH measured during a:Si-H deposition
CRD absorption during deposition

CRD spectrum of SiH measured during a:Si-H deposition
SiH detection: $A^2 \Delta \rightarrow X^2 \Pi$, 405 – 430 nm

- Line width
- Temperature
- Absorption
- Cross-section
- Density
TALIF spectroscopy on H atoms
Ar/H₂ plasma expansion

- Supersonic flow ($M > 1$)
- Subsonic flow ($M < 1$)
- Mach disk ($M = 1$)
- Stationary shock wave

Cascaded arc

Gas inlet

Anode (nozzle)

Cathodes (3)
Rayleigh scattering on H/H\textsubscript{2} plasma expansion

\[ \frac{1}{z^2} \]

\[ H_2 \text{ density (m}^{-3}\text{)} \]

\[ \text{axial position (mm)} \]
TALIF detection of H atoms

- produce 205 nm via THG of a Nd:YAG pumped dye laser
- from spectral scans data on: n, T, v, f(v)

Diagram showing energy levels and transitions:
- $3s^2 S \rightarrow 2s^2 S$ at 121.56 nm
- $3s^2 S \rightarrow 3p^2 P$ at 656.5 nm (Hα)
- $2s^2 S \rightarrow 2p^2 P$ at 10.2 eV
- $3d^2 D \rightarrow 3p^2 P$ at 13.6 eV

Additional details:
- Cascaded arc: 3.5 slm H₂, I=40A, V=150V
- Slitmask, filter, gates PMT
- 205 nm output
H atom density along the jet axis (TALIF)
Effect of nozzle-length on H density

[Graph showing the relationship between H density and axial position for different nozzle lengths (6 mm and 14 mm).]
Effect of nozzle-length on H flux

- short nozzle
- long nozzle (x5)

H flux density ($10^{25} \text{ m}^{-2} \text{ s}^{-1}$)

radial position (mm)
Effect of nozzle-width on H flux

![Graph showing the effect of nozzle-width on H flux](image)

- Triangles: large diam. nozzle
- Circles: short nozzle

H flux density \((10^{25} \text{ m}^2 \text{s}^{-1})\)

radial position (mm)
Conclusions

1. Large influence of nozzle geometry on H flux

2. Loss of H atoms due to surface association (volume association far too slow)

loss of $H$ atoms $=$ production of $H_2^{rv}$ at the surface

H flux: $\Phi_H > 10^{21} \text{ s}^{-1}$

Dissociation degree $= 0.4$

S. Mazouffre et al., Phys Rev. E 64 (2001) 066405

TU/e technische universiteit eindhoven
Doppler-LIF spectroscopy on Ar atoms
Expanding Thermal Plasma (ETP)

Plasma creation

Plasma chemistry

Material processing

SiH$_3$ reflection ~85%

SiH$_3$ reaction ~15%:
5-fold bonded Si

SiH$_3$ surface diffusion:
strong bond formation with dangling bond

dangling bond creation by Eley-Rideal H-abstraction
dangling bond creation by ion
dangling bond surface diffusion
Ar plasma expansion
Ar density as function of distance from the exit of the source

Axial position (mm)

Ar density (m$^{-3}$)

42 Pa

100 Pa

Rayleigh scattering
Doppler LIF experimental setup

- Diode laser
- Argon lamp
- Fabry Perot diodes
- Lock in amplifier
- reference
- trigger
- PMT
- filter
- z-axis
- BS
- chopper
Typical result of a Doppler LIF measurement

- **Argon lamp**
- **Fabry Perot**
- **LIF signal**

Graph showing intensity vs. piezo voltage with labeled components.
Ar velocity distribution functions

Doppler LIF
Evaluating Ar atom velocity and temperature with:

- $\gamma = 1.4$  
- $z_{\text{ref}} = 0.0025$ m  
- $T_0 = 6000$ K

Theoretically, $5/3$ for mono-atomic gas.
IR absorption spectroscopy on 
$\text{N}_2/\text{O}_2$ plasma
N$_2$/O$_2$ plasma setup
(IR diode laser absorption spectroscopy)
Molecule Formation in Plasma

\[ O^{(3P)}_{\text{atm}} + \text{NO}_{\text{ads}} \rightarrow \text{NO}_2^{2B_1} \rightarrow \text{NO}_2^{2A_1} + h\nu \]

\( N_2 \) plasma with \( O_2 \) injected in the background
NO formation in an Ar-N₂-O₂ plasma

\[ N + NO \rightarrow N_2 + O \]
\[ N + NO_{ads} \rightarrow N_2O \]
N$_2$O formation in an Ar-N$_2$-O$_2$ plasma

N$_2$O only formed at low O$_2$ flow
Time behavior of NO formation in Ar-N$_2$-O$_2$ plasma

$\tau_{res} \approx 1\text{s}$

- **no O$_2$ flow** ⇒ **no NO formation**
- **no N$_2$ flow** ⇒ **NO formation $>> \tau_{res}$**

N on/in the surface (no O storage)
Time behavior of N$_2$O formation in an N$_2$-O$_2$ plasma

$\tau_{res} \approx 1\text{s}$

$O_2$ off after 1 min
$O_2$ off after 5 min

no $O_2$ flow

$\downarrow$

N$_2$O formation $>> \tau_{res}$

NO on/in the surface
(depends upon $O_2$ conditioning)
Conclusions

1. Input gas mixture, N₂ and O₂, changes into a mixture of N₂, O₂ and NO, N₂O and NO₂.

2. Time-resolved measurements show that surfaces become saturated with N atoms and NO radicals.

3. In Ar-NO plasmas, up to 90% conversion of NO into N₂ and O₂.
VUV-LIF spectroscopy on $\text{H}_2^{r,v}$ molecules in plasma
Why study hydrogen plasma expansions?
(produced from a cascaded arc)

1. Use of H$_2$ gas in processing plasma application
   - etching and cleaning
   - passivation during deposition

2. Astrophysical interest
   - ‘hot’ H$_2$, formed at grains through surface association,
     and acts as precursor in astro-chemistry

3. Fundamental study of H$_2$/HD/D$_2$ Lyman transitions
   - extension of database

4. The cascaded arc might be used as H$^-$ ion source, because
   of high fluxes of H$_2$$^{r,v}$ at low T$_e$ (around 1 eV)
Plasma source and expansion

\[ I = 40 - 60 \, \text{A} \]
\[ P = 5 - 10 \, \text{kW} \]
\[ \Phi_{\text{arc}} = 3 \, \text{slm} \]
\[ P_{\text{arc}} = 0.2 \times 10^5 \, \text{Pa} \]
\[ P_{\text{bg}} = 100 \, \text{Pa} \]
Plasma expansion

- Cathode (3x)
- Cascade plates
- H₂ inlet
- Anode plate

Parameters:
- T = 6000 K
- T = 1000 K
- T = 2000 K
- T = 400 K

压强范围：10 - 100 Pa

1. Supersonic expansion (M >> 1)
2. Zone of silence
3. Shock front (M > 1)
4. Subsonic expansion (M < 1)

Mach disk (M = 1)
PLEXIS setup
PLEXIS setup

**Laser table**
Nd:YAG  
(450 mJ/shot @ 355 nm)

dye laser  
(50 mJ/shot @ 460 nm)  
(8 mJ/shot @ 230 nm)

**Vacuum chamber**
cylindrical (2m x 0.3m)  
9 Pa / 3000 sccm H₂

- Movable plasma source and substrate
- Axial magnetic field  
  \( B_{\text{max}} = 0.2 \text{ T} \)
Two photon Absorption LIF (TALIF) on atomic hydrogen

- Produce 205 nm via THG of a Nd:YAG pumped dye laser
- From spectral scans data on: n, T, v, f(v)

![Energy level diagram with transitions labeled 1s^2S, 2s^2S, 3s^2S, 2p^2P, 3p^2P, 3d^2D.](image)

- Ion transitions:
  - 121.56 nm Lyman α
  - 205.14 nm

- Cascaded arc:
  - 3.5 slm H₂
  - I=40A, V=150V

- Detection setup:
  - Slit mask
  - Filter
  - Gated PMT
  - 205 nm
H atom density in H₂ plasma expansion (TALIF)

Mazouffre et al. Phys. Rev. E 64, 016411 (2001)
VUV-LIF detection of H$_2^{r,v}$

**H$_2$ energy scheme**

- X $\rightarrow$ B transition in H$_2$ ($\sim$11 eV)
- Detection of fluorescence in the VUV range
- Excitation with 120 – 165 nm photons, produced via SARS

**Excitation and detection**

- X $\rightarrow$ B transition in H$_2$ ($\sim$11 eV)
- Detection of fluorescence in the VUV range
- Excitation with 120 – 165 nm photons, produced via SARS
SARS technique

Four-wave mixing process

VUV-LIF detection of $\text{H}_2^{r,v}$

**H₂ energy scheme**

- $X \rightarrow B$ transition in $\text{H}_2$ (~11 eV)
- Detection of fluorescence in the VUV range
- Excitation with 120 – 165 nm photons, produced via SARS
VUV-LIF detection of $H_2^{r,v}$

**H$_2$ energy scheme**

- Internal energy (eV)
- Lyman series absorption and emission
- $\Sigma^+_u$ and $\Sigma^+_g$

**VUV-LIF detection scheme**

- Plasma to pump
- PMT
VUV-LIF setup
Measured $\text{H}_2$ Lyman spectrum

![Graph showing the measured H$_2$ Lyman spectrum with SH frequency (cm$^{-1}$) on the x-axis and Fluorescence (a.u.) on the y-axis. Peaks at 76890, 77140, 81045, and 81295 cm$^{-1}$ are marked.](image-url)
VUV-LIF setup

Nd:YAG THG

Dye (C440)

BBO

220 nm

440 nm

LN$_2$

H$_2$

VUV mono

M

BS

NO cell

PMT

PMT

PMT

to pump

to pump
VUV-LIF setup

TALIF spectrum of NO
P = 2 mbar

Fluorescence (a.u.)

Fluorescence (a.u.)

Wavenumber (cm$^{-1}$)

NO cell

PMT

BBO

220 nm

440 nm

M

L

H$_2$

V

PMT

to pump

VUV mono

PMT
VUV-LIF spectroscopy

TALIF spectrum of NO
P = 2 mbar

Fluorescence (a.u.)
Fluorescence (a.u.)

VUV-LIF spectroscopy

H$_2$(v=4,J=7) via VUV-LIF

NO via TALIF

Nd:YAG THG

Fluorescence (a.u.)
Wavenumber (cm$^{-1}$)

73182 73184 73186 73188 73190

22048 22049 22050 22051 22052 22053

dye laser frequency (cm$^{-1}$)

0 44000 44020 44040 44060 44080 44100 44120 44140 44160 44180 44200

NO cell

44200 44220 44240 44260 44280 44300 44320 44340 44360 44380

22048 22049 22050 22051 22052 22053

H$_2$(v=4,J=7) via VUV-LIF

VUV mono

AS7 frequency (cm$^{-1}$)

73182 73184 73186 73188 73190

to pump
state-selective
spatially resolved
non-intrusive
dynamic range $> 4$ orders
detection limit $\sim 10^{13}$ m$^{-3}$

Measured $H_2$ Lyman spectrum

Advantages multiplexing:
- spectrum more dense
- efficient measurement
Measured H$_2$/HD/D$_2$ Lyman spectra

100 Pa
1500 sccm H$_2$ / 1500 sccm D$_2$
45 A, $z = 8$ mm

Measured and calculated $\text{H}_2/\text{HD}/\text{D}_2$ Lyman spectrum

Spectroscopic data for $\text{H}_2$

All $\text{H}_2$ Lyman transitions

Spectroscopic data for HD

HD Lyman transitions $J < 11$

Spectroscopic data for $\text{D}_2$

$\text{D}_2$ Lyman transitions $J < 12$
New calculated Lyman transitions including higher rotational states (J > 10), in collaboration with Abgrall and Roueff

Non-Boltzmann distribution for H$_2$

- 700 K for low J
- 3800 K for high J
Non-Boltzmann distributions in H$_2$/D$_2$ jet

Low J: $T = 700$ K
High J: $T = 3800$ K

Low J: $T = 300$ K
High J: $T = 3000$ K

Results on H⁻ production through DA process

Production rate of H⁻ ions by dissociative attachment

CRD spectroscopy on $N_2/H_2$ plasma
NH₃ production in N₂/H₂ plasma

- SiH₃ reflection
- ~85% SiH₃ reaction
- ~15%: 5-fold bonded Si
- SiH₃ surface diffusion: strong bond formation with dangling bond
- dangling bond creation by Eley-Rideal H-abstraction
- dangling bond creation by ion
- surface diffusion

NH₃ production in N₂/H₂ plasma
NH₃ production in N₂/H₂ plasma

Plasma chemistry leading to e.g. NHₓ

NH₃ formation?
Cavity Ring Down: principals and features


- absorption per unit of pathlength (cavity loss):
  \[
  1/c\tau = (1 - R + n\sigma L) / d
  \]

- non-intrusive
- high sensitivity due to effective multipassing
- direct absorption –> line of sight measurement
Cavity Enhanced Absorption detection scheme

NH₃ production in N₂/H₂ plasma

CEA measurement recorded in a vessel in which N₂/H₂-plasma expands

- Scanning frequency: 30 Hz
- Frequency range: 15 GHz
- Averages: 1000
- Measurement time: 30 s

The absorption coefficient $\kappa(\nu)$ from intensity by:

$$\kappa(\nu) = \left( \frac{S_0(\nu)}{S(\nu)} - 1 \right) \times \left( \frac{1 - R}{d} \right)$$
NH₃ production in N₂/H₂ plasma

Part of the absorption spectrum of NH₃ as measured in an expanding N₂/H₂ plasma

\[ \sigma \approx 10^{-22} \text{ m}^2 \]

\[ T_{tr} = 600 \text{ K} \]

\[ N \approx 10^{19} \text{ m}^{-3} \]
Ammonia density produced in expanding N$_2$ plasma in which H$_2$ is injected in the background

Saturation behavior explained by rate determining steps:

\[ \text{N}^+ + \text{H}_2 \rightarrow \text{NH}^+ + \text{H} \]
\[ \text{NH}^+ + \text{e} \rightarrow \text{N} + \text{H} \]

Total N$^+$ flow is consumed

\[ P_{bg} = 100 \text{ Pa} \]
NH₃ production in N₂/H₂ plasma

Ammonia density as function of background pressure at constant gas flow (N₂-arc/H₂-background)

Linear with pressure (dNH₃/dt constant)

3 particle reaction
stable intermediate

Wall production (?)
NH₃ production in N₂/H₂ plasma

NH₃ production in two different vessels

- Total gas flow of 2 slm through cascaded arc
- At maximum 12% of the background gas is NH₃

larger surface-to-volume ratio

Conclusions

Input gas mixture, \( \text{N}_2/\text{H}_2 \), changes into \( \text{N}_2/\text{H}_2/\text{NH}_3 \) mixture

(12 % of the background gas is \( \text{NH}_3 \)).

\( \text{NH}_3 \) is formed at surfaces.