Institute of Experimental and Applied Physics

Christian-Albrechts-Universität zu Kiel

*XXVIII. Erfahrungsaustausch OTPIP Mühlleiten*

*Tuesday, March 14, 2023*

# Characterization of a pulsed plasma and microparticles in an industrial scale ta-C laser-arc coating system


*Mathis Klette[1], Martin Kopte[2], Wolfgang Fukarek[2] and Holger Kersten[1]*

*[1]Institute of Experimental and Applied Physics, Workgroup Plasma Technology, Christian-Albrechts-University Kiel, Germany*

*[2]VTD Vakuumtechnik Dresden GmbH, Germany*

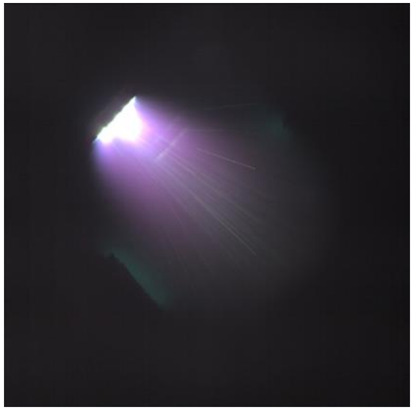

VTD Member of VON ARDENNE Group

# Project Prometheus

**sponsor:** Bundesministerium für Wirtschaft und Energie (BMWi) / Federal Ministry for Economic Affairs and Energy
**project executing organisation:** Forschungszentrum Jülich GmbH (PT-J.ESN2)

PROMETHEUS: **P**rojekt zur **R**eibungs-**O**ptimierung von **M**otoren durch **E**insatz von **t**riboaktiven **H**ochleistungskohlenstoff- sowie **E**isenbasisschichten und **S**chmierstoffen
loose translation: Project for friction-optimization of motors by use of triboactive highperformance carbon and ironbased coatings and lubricants

VTD (Vakuumtechnik Dresden GmbH): Manufacturer of PVD-Vacuum coating systems for industrial use
VTD: further development of the coating technology used to create optimized ta-C coatings



Arc discharge of the laser-arc module (LAM)

## Goals

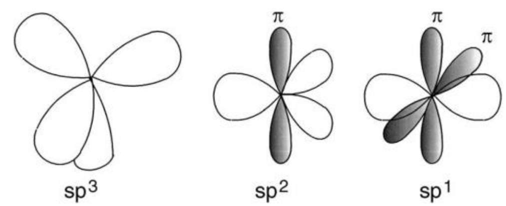
- Determine energy flux to substrate surface for generation of ta-C coatings
  - Spatially resolved profile of plasma parameters and ion distributions
- Suppressing particle generation and improving particle filtering
  - Analyze basic physics of cathode spot generation
  - Time and spatially resolved profiles of the plasma
- Enhancing deposition rates and uniformity
- Coating of nonconductive materials
  - What influences the ion energy?
  - What effect do other energy contributions (electrons, radiation…) have on ta-C coatings?

VTD Dresden

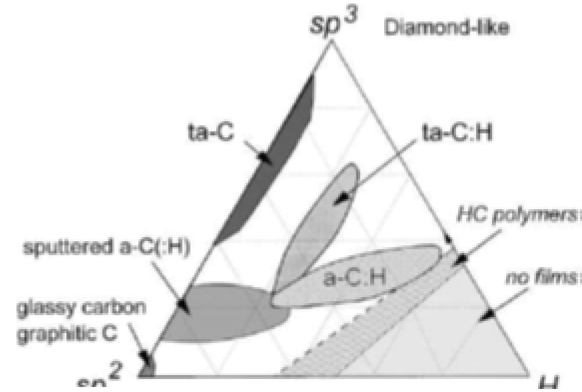
# Tetrahedral Amorphous Carbon (ta-C) / Diamond-like Carbon (DLC)

## Film growth

- Collision phase ($\sim 10^{-13}$ s)
  - Ion is implanted
  - Higher localized pressure
- Thermalization phase ($\sim 10^{-11}$s)
  - Heat distribution
- Relaxation Phase ($\sim 10^{-10} - 1$ s):
  - Orbitals stabilize / atomic bonds form



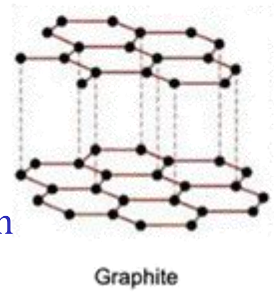
J. Robertson (2002)

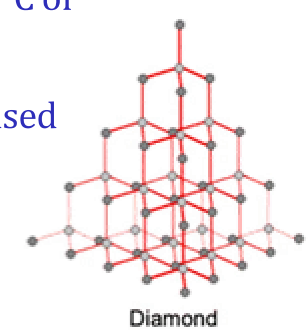


J. Robertson (2002)

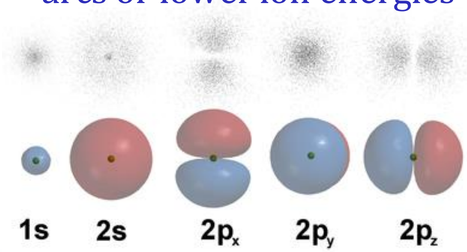
## Expected ideal deposition conditions:

- $E_{\mathrm{kin,ion}} \approx 100$ eV
- fully ionized plasma with ions of a single charge state (single or double ionized)
  - No macro particles or clusters which introduce defects
- Constant substrate temperature (150 °C or lower)
  - Substrate temperatures can be lowered significantly by using pulsed arcs or lower ion energies

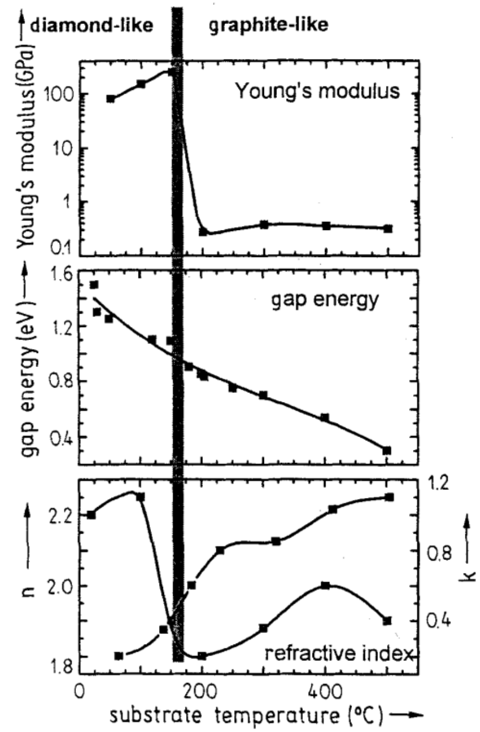


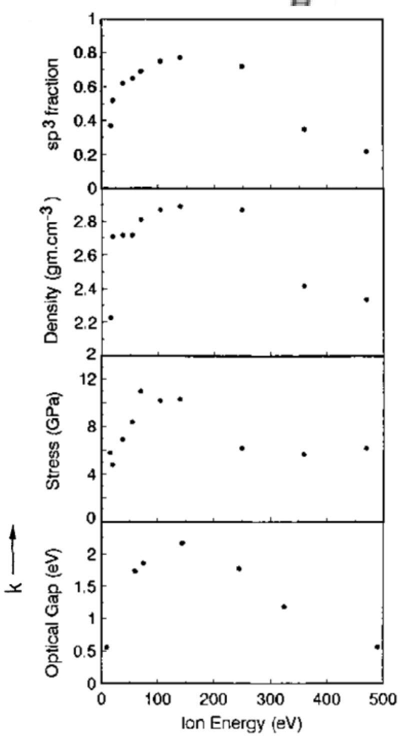
Negri (2020)



Wikipedia (2021)



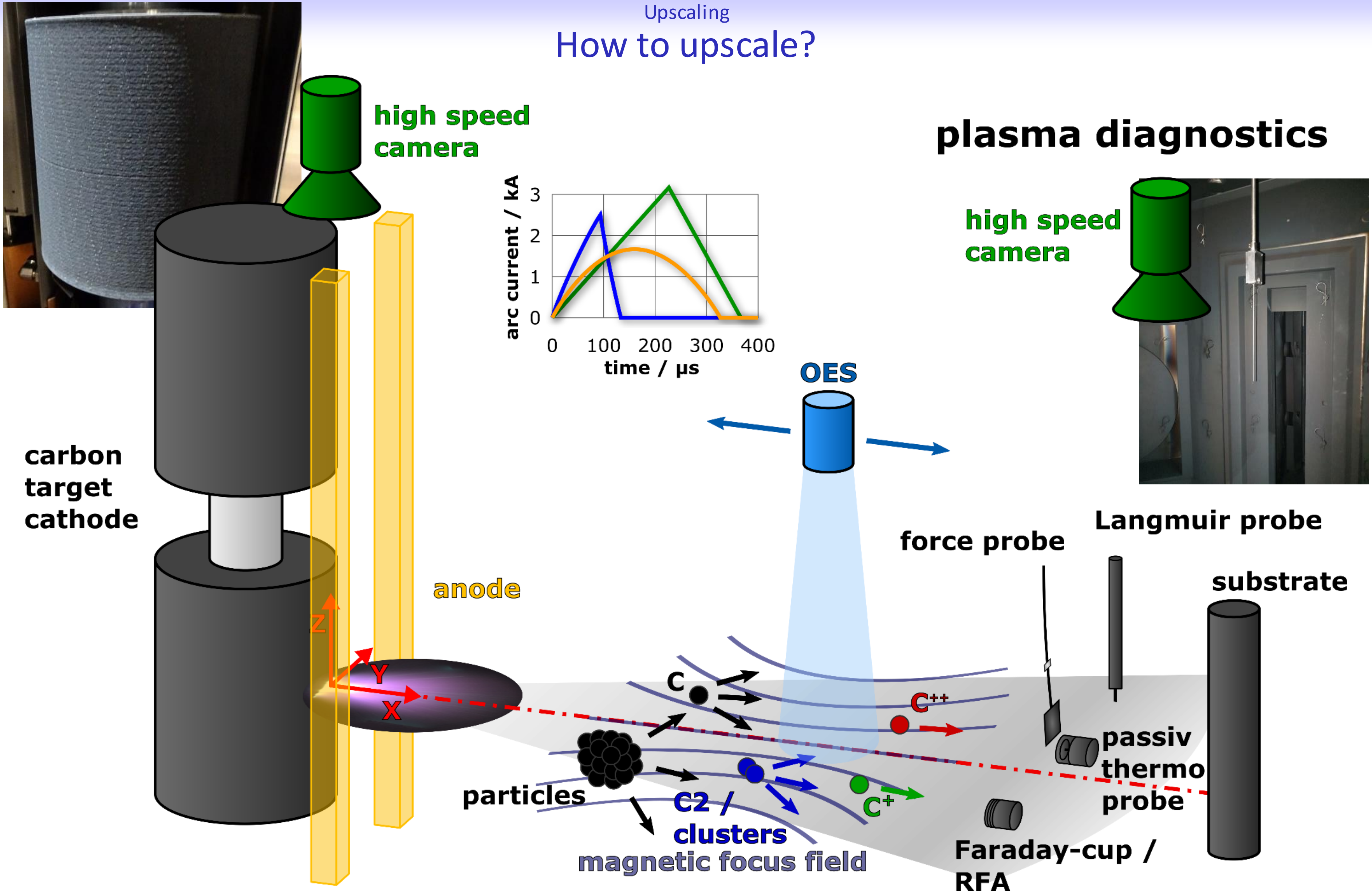
B. Schultrich (1994)



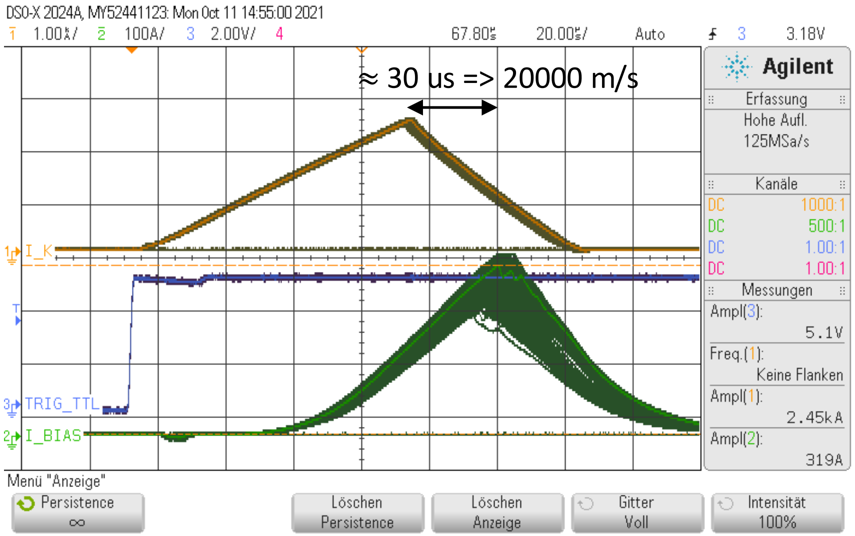
Fallon (1993)

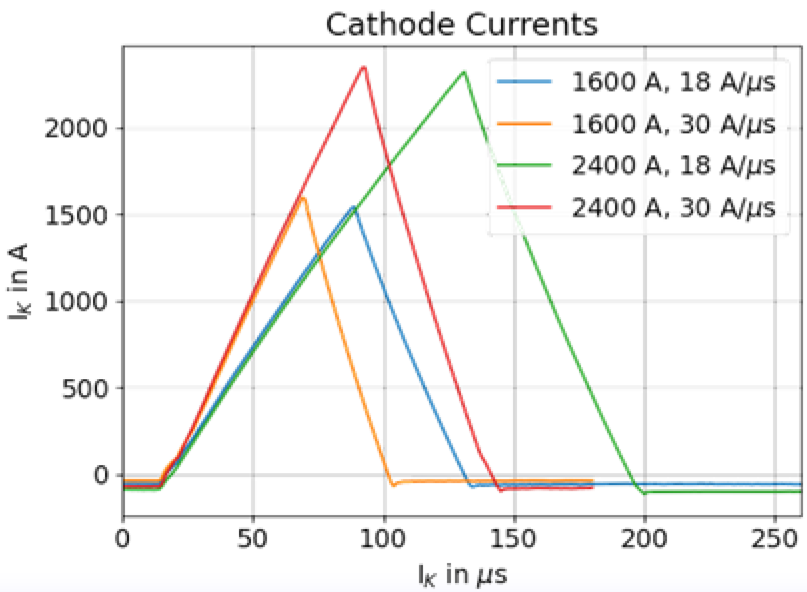
Upscaling

# How to upscale?



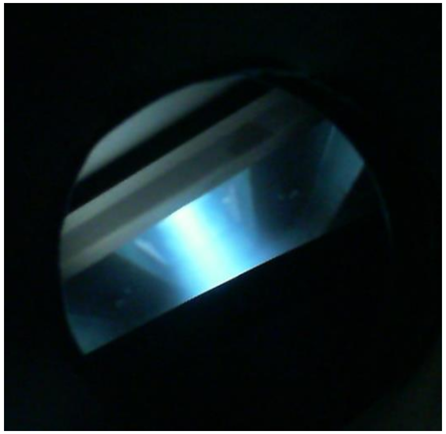
# Which conditions are characterized?




**no magnetic field**

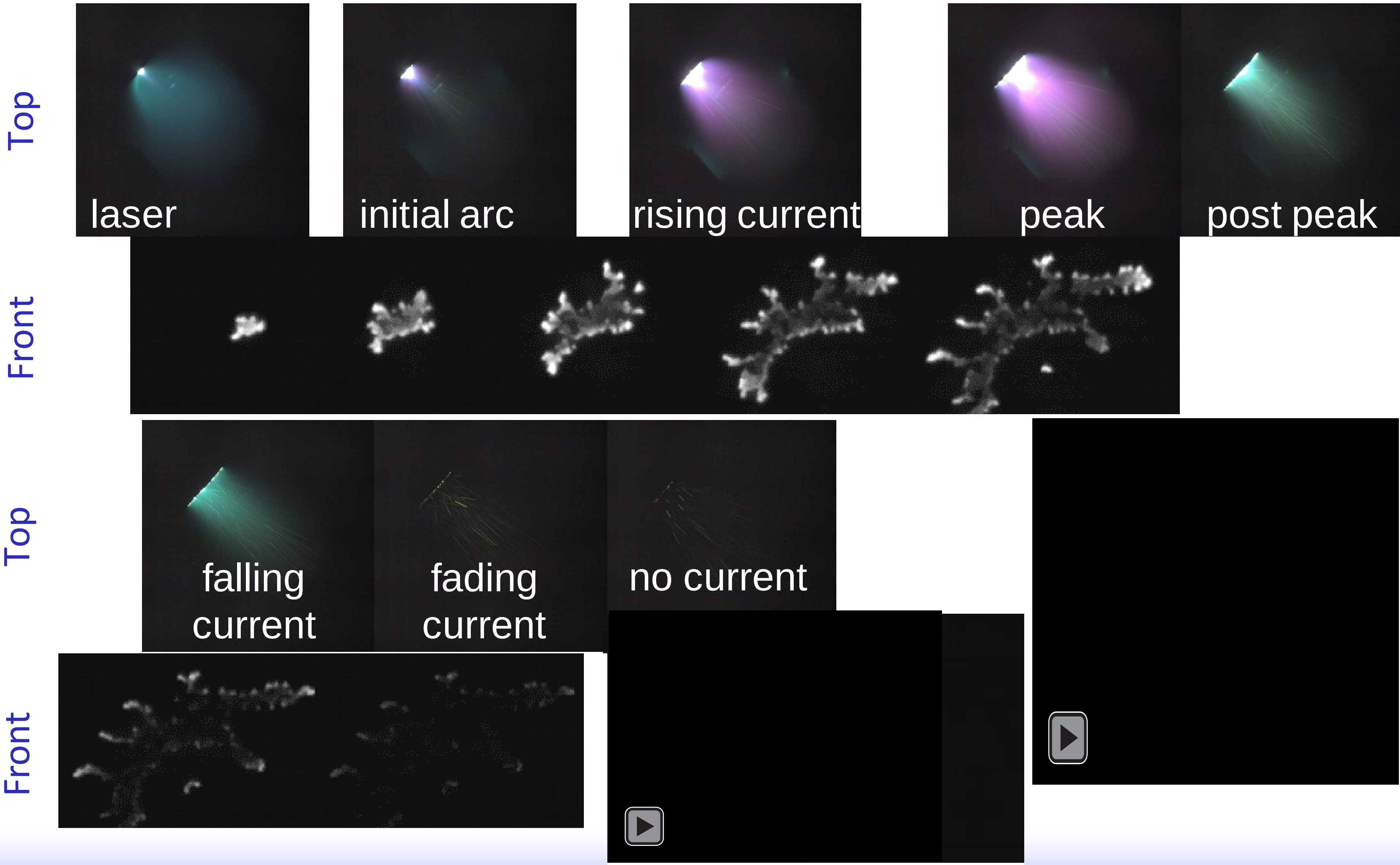
**with magnetic field**



| | | | |
|---|---|---|---|
| **Maximum cathode current** | $I_K$ / A | 1600 (low) | 2400 (high) |
| **Current ramp speed** | $\frac{dI}{dt}$ / $A\mu s^{-1}$ | 18 (slow) | 30 (fast) |
| **Magnetic plasma focus** | $B_{focus}$ | - (off) | A few mT (on) |

| $I_K$ / A | $U_K$ / V | $\frac{dI}{dt}$ / $A\mu s^{-1}$ | $Q$ / C |
|---|---|---|---|
| 1600 | 240 | 18 | 0.09 |
| 1600 | 400 | 30 | 0.07 |
| 2400 | 240 | 18 | 0.22 |
| 2400 | 400 | 30 | 0.15 |

# What does the discharge look like?

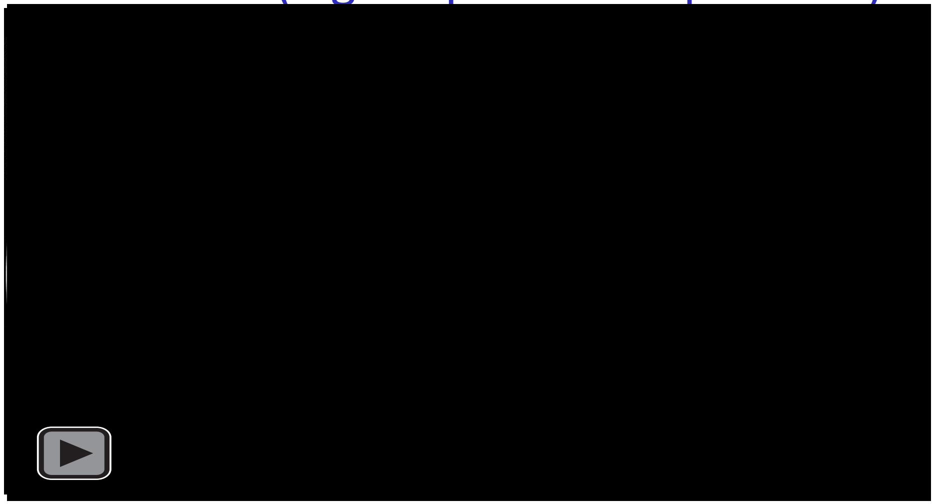


# Observations at Cathode for 2400 A, 18 A/us with mag. focus

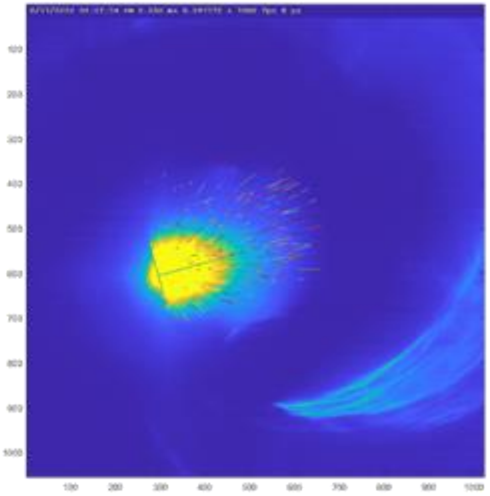
## Laser Plasma

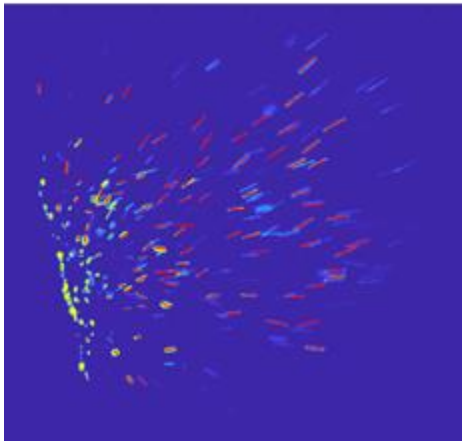
## Arc Plasma

## Arc Plasma (high exposure for particles)



## Particle Fits

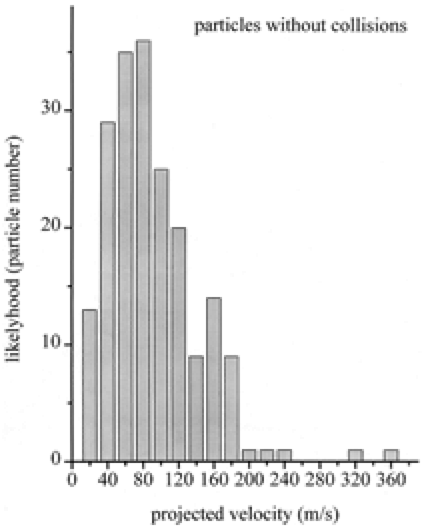


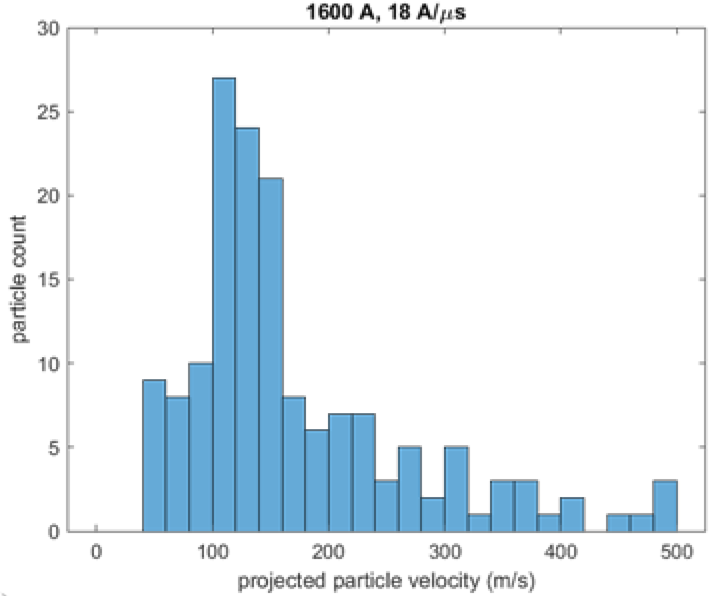


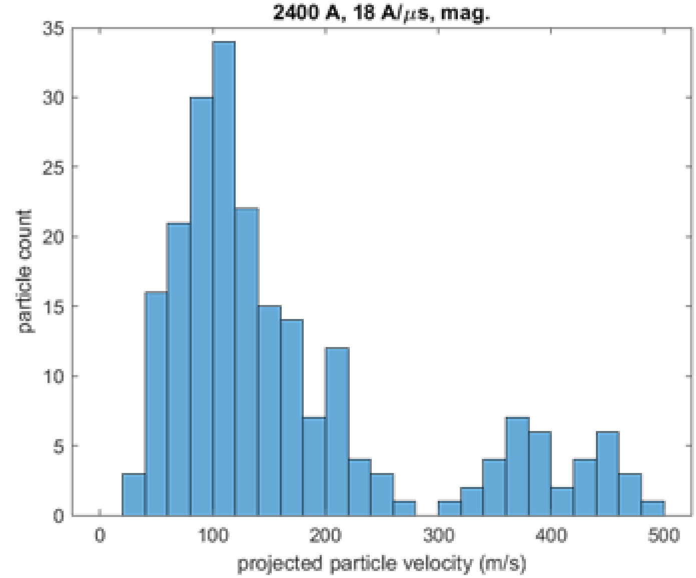
High-Speed Camera

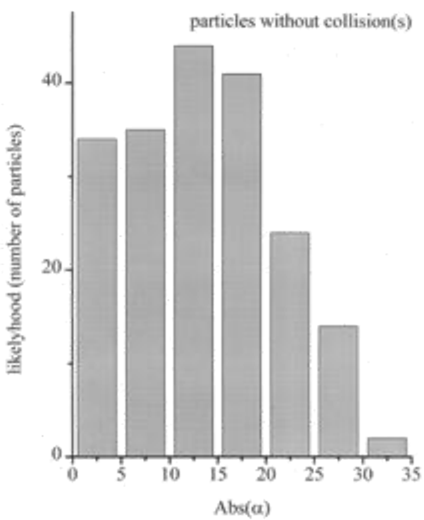
# Macro Particles - Statistics

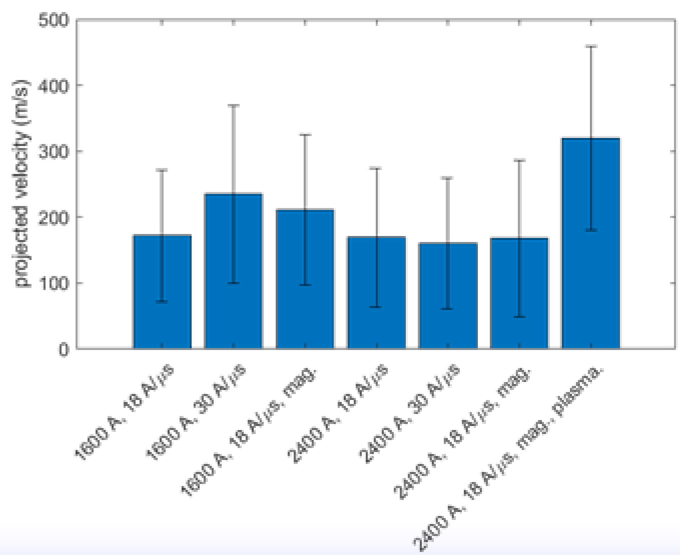
Thomas Schülke and André Anders,
Plasma Sources Sci. Technol. 8 (1999) 567–571

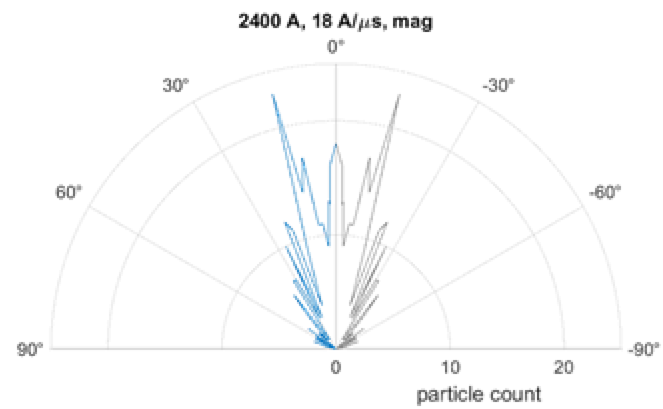


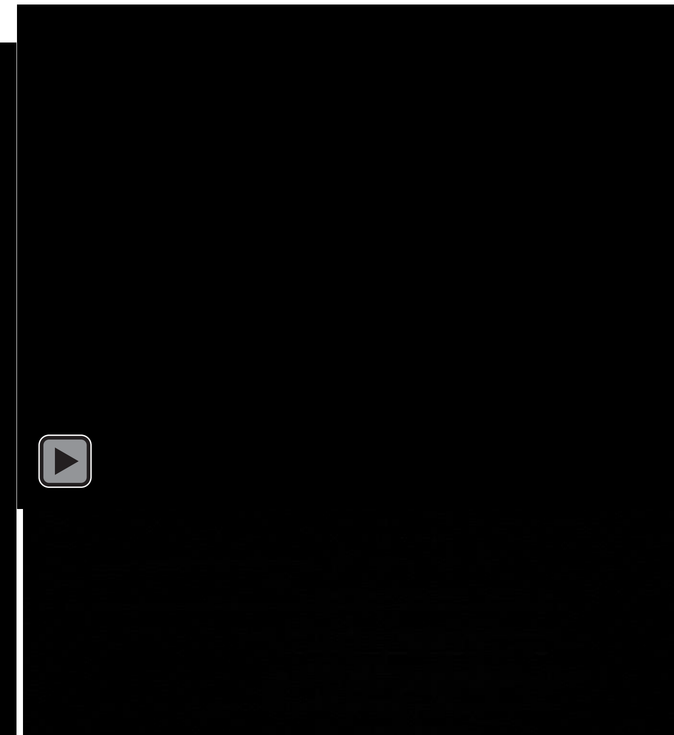



High-Speed Camera

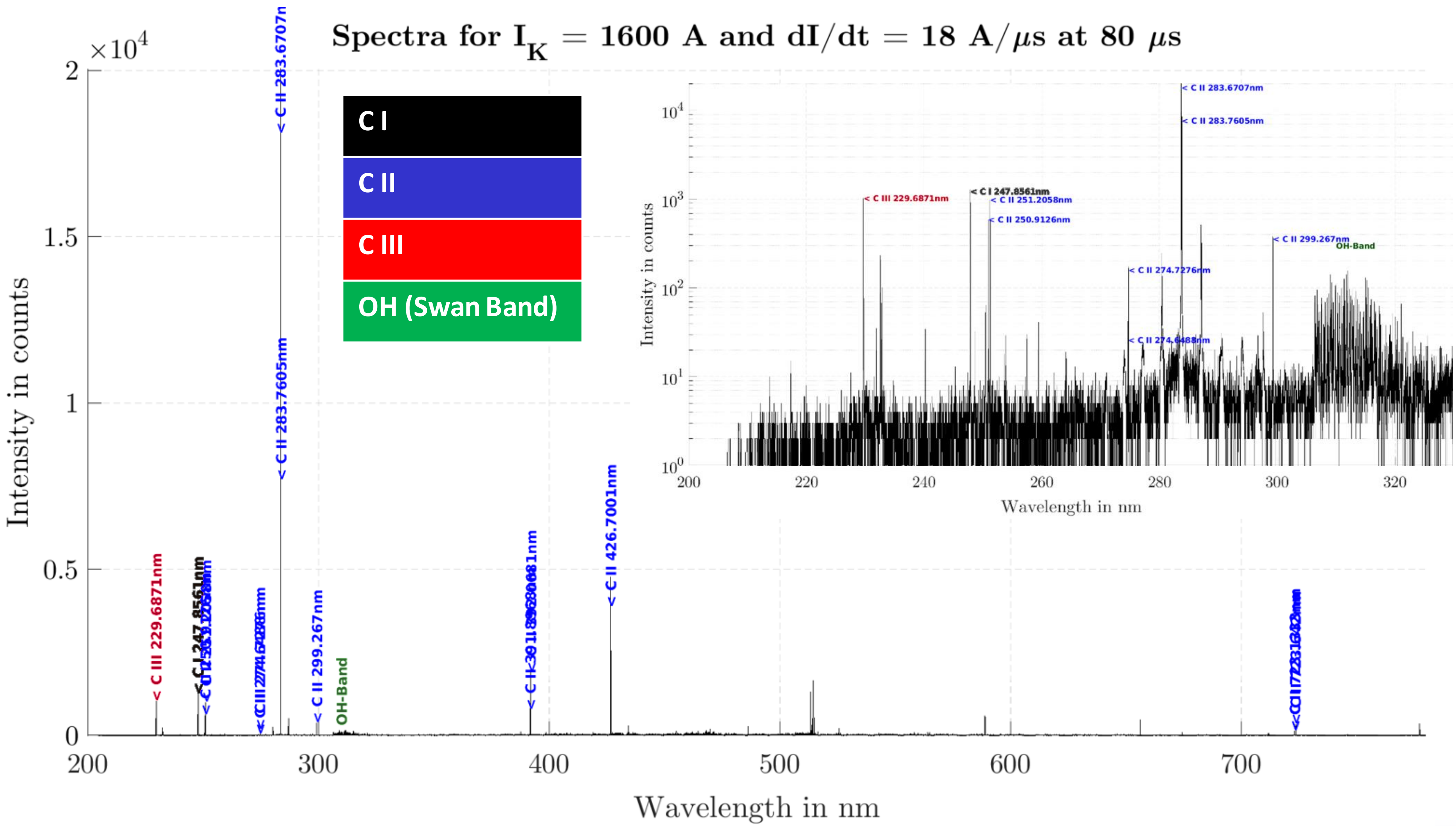
# Particle Collisions at Substrate

particle splits at substrate

particle velocity vs plasma frequency

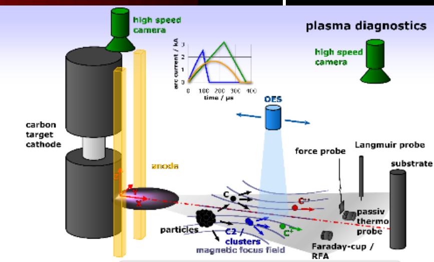


# Which excited species are present?

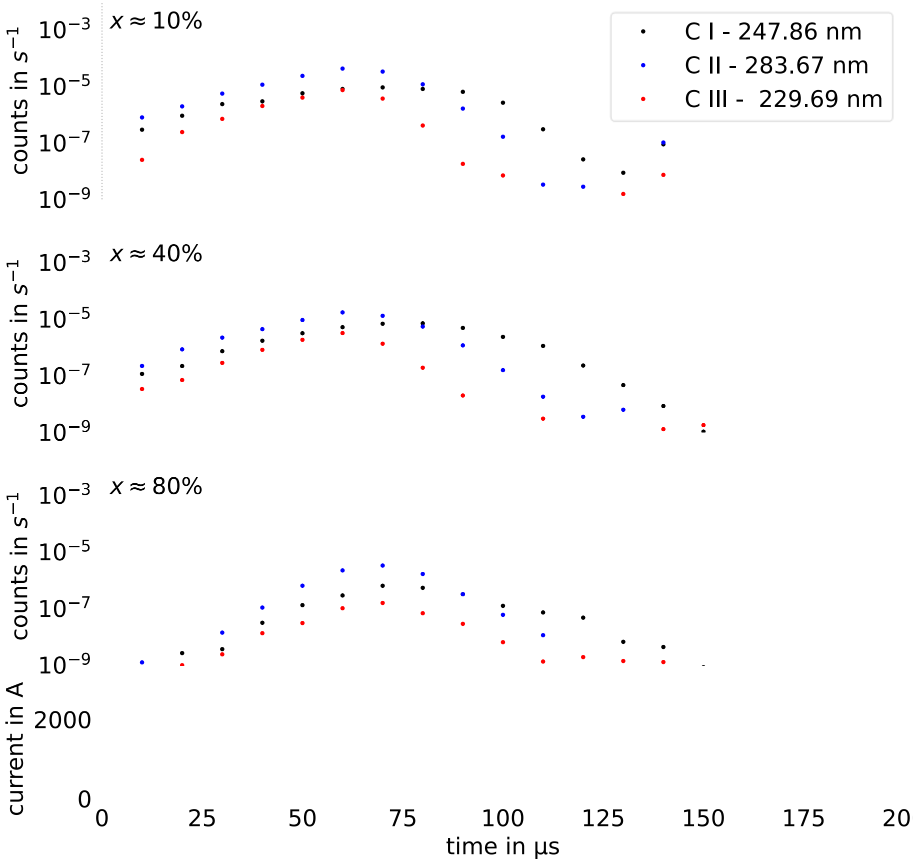


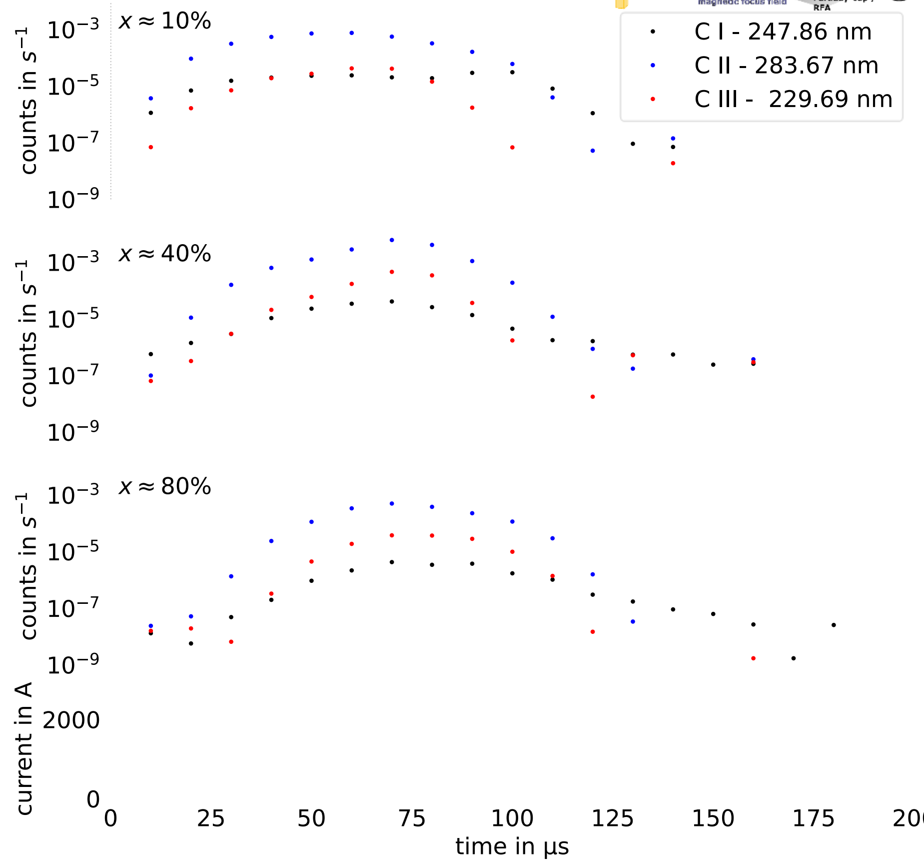
Echelle Spectrometer

# Time and spatially resolved measurement




## Arc at 1600 A, 18 A/µs magnetic focus off → on

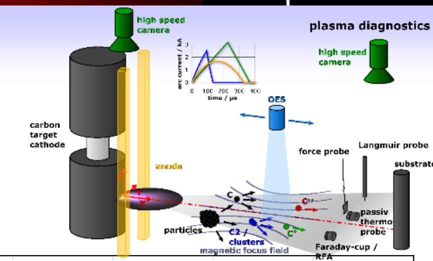


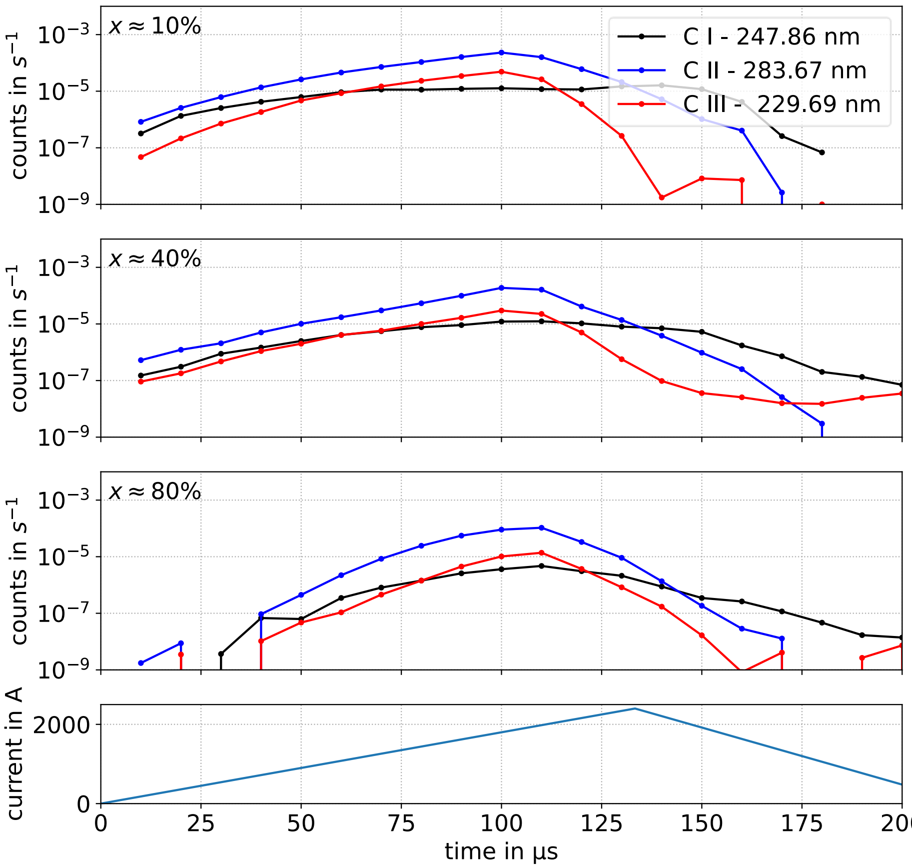

1. ion velocity ≈ neutral velocity
2. ion generation stops with current reduction.
3. neutrals keep getting generated
4. magnetic field increases ionization in the center

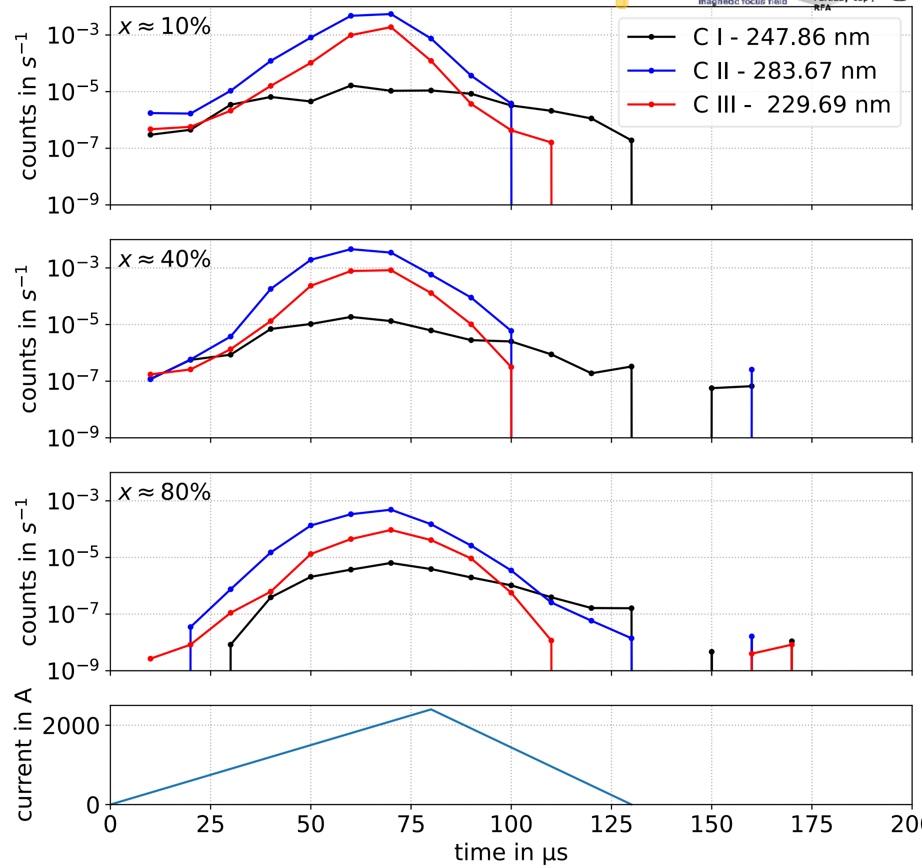
Echelle Spectrometer

# Time and spatially resolved measurements



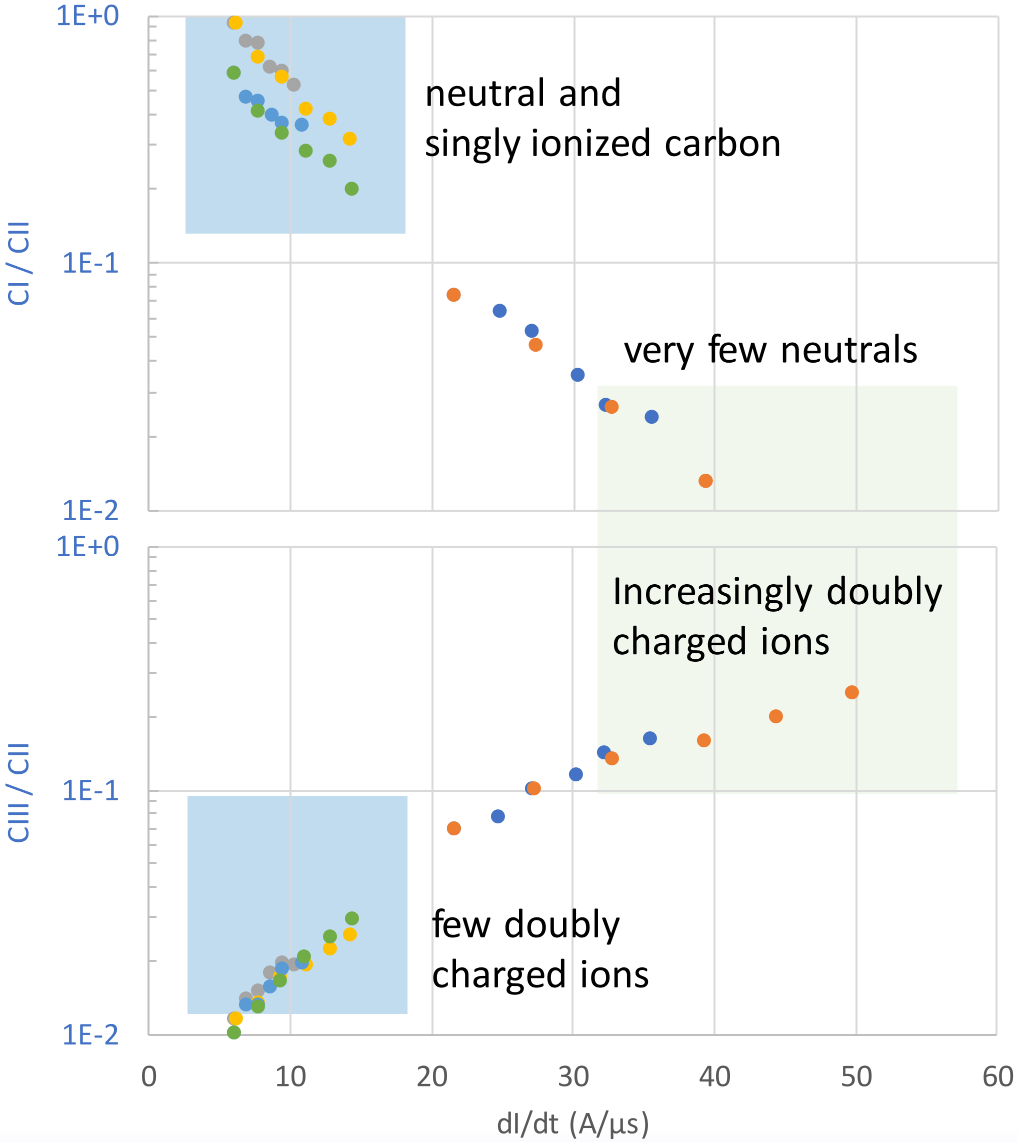

## 2400 A, 18 A/µs → 30 A/µs



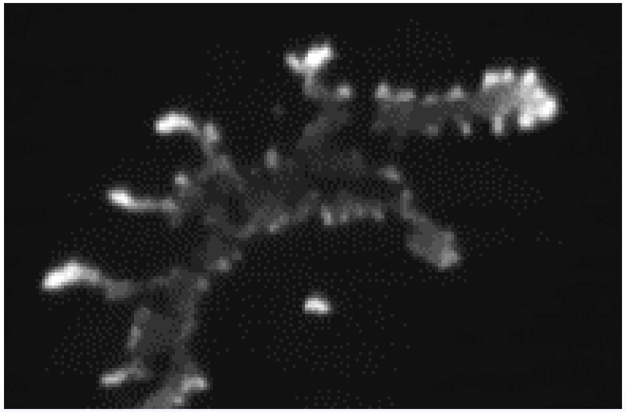

1. Higher current leads to stronger ionization
2. Increasing ramp speed / discharge voltage **drastically** increases ionization, esp. doubly charged

# Ratio of line intensities

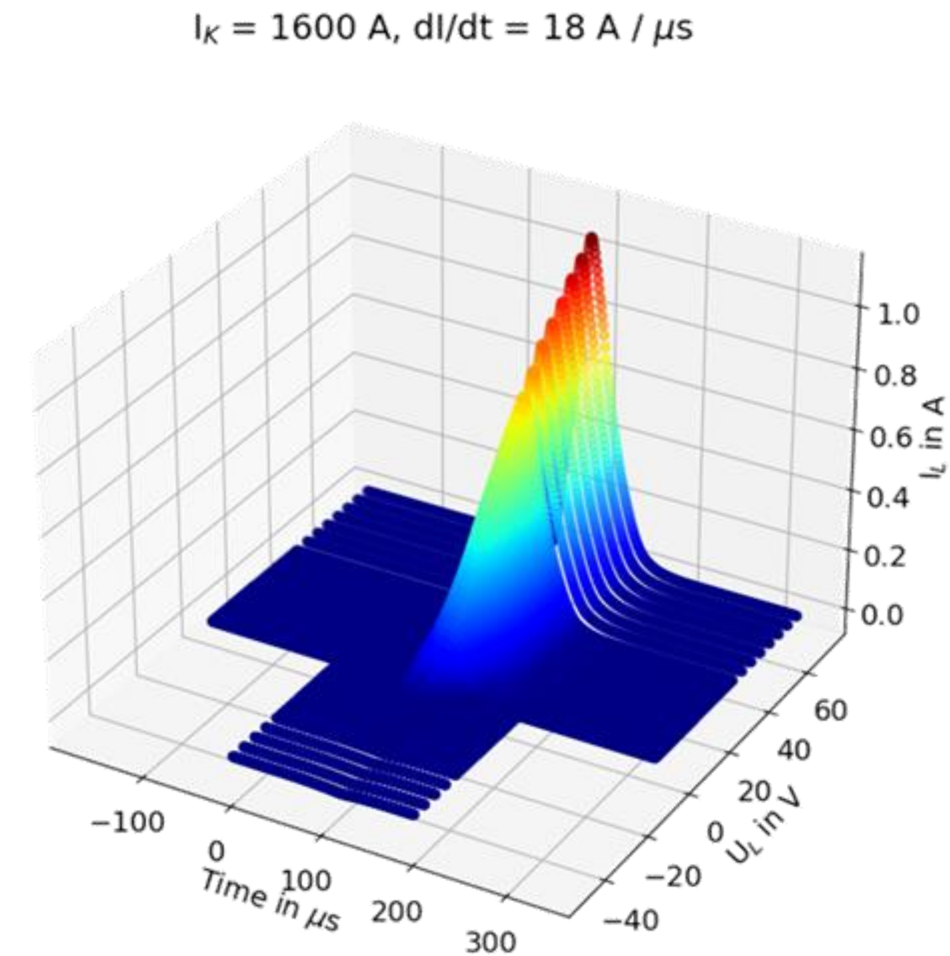


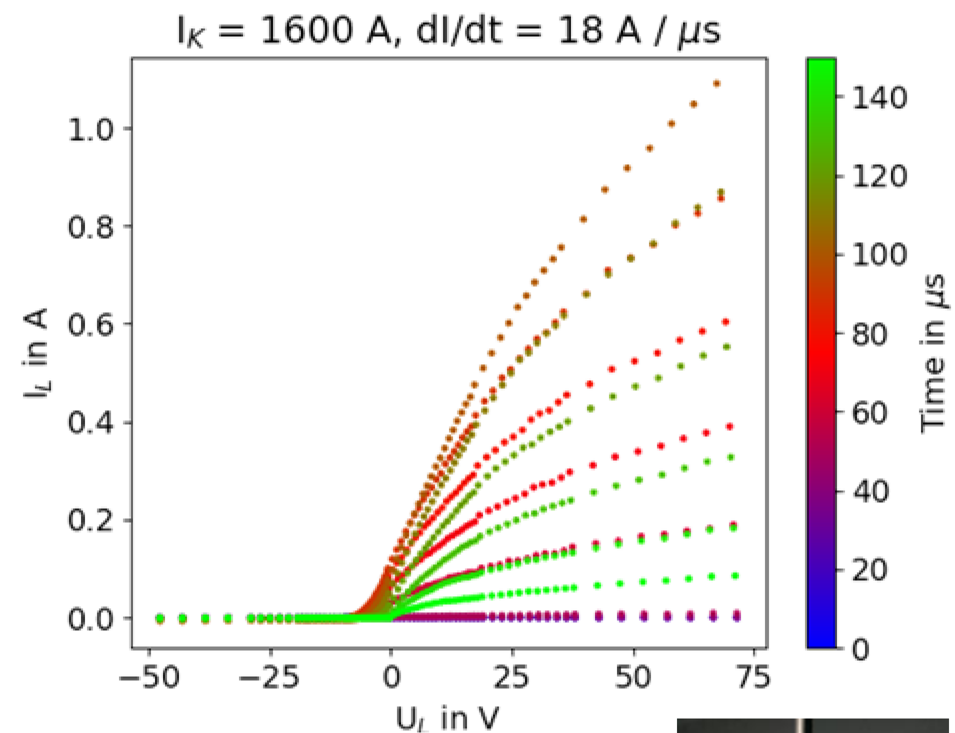

- Time integrated spectra
- High ramp speeds
  - Reduce neutral line intensities
  - Increase singly and doubly charged ion line intensities
    - In extreme cases the doubly charged lines are dominant
  - Dependency is almost exponential
- Maximum current
  - Increases ionization for low ramp speeds

- Absolute densities are to be determined using an absolute calibrated UV spectrum



# Measurements Overview

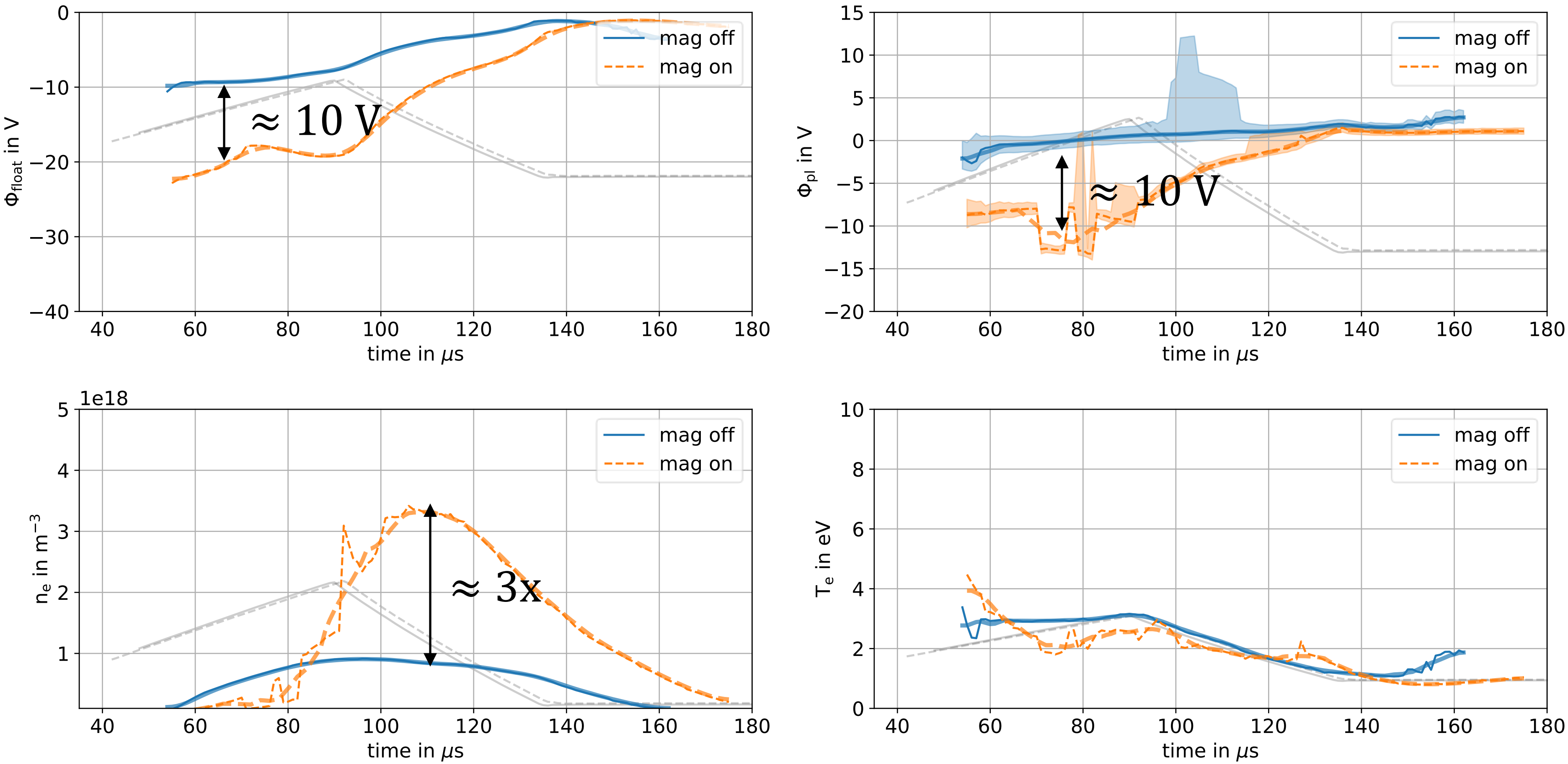



Time and spatially resolved Langmuir measurement:
DC Voltage, averaging over multiple pulses, then increasing voltage
Many unknowns in advance, requires cleaning current for negative voltages

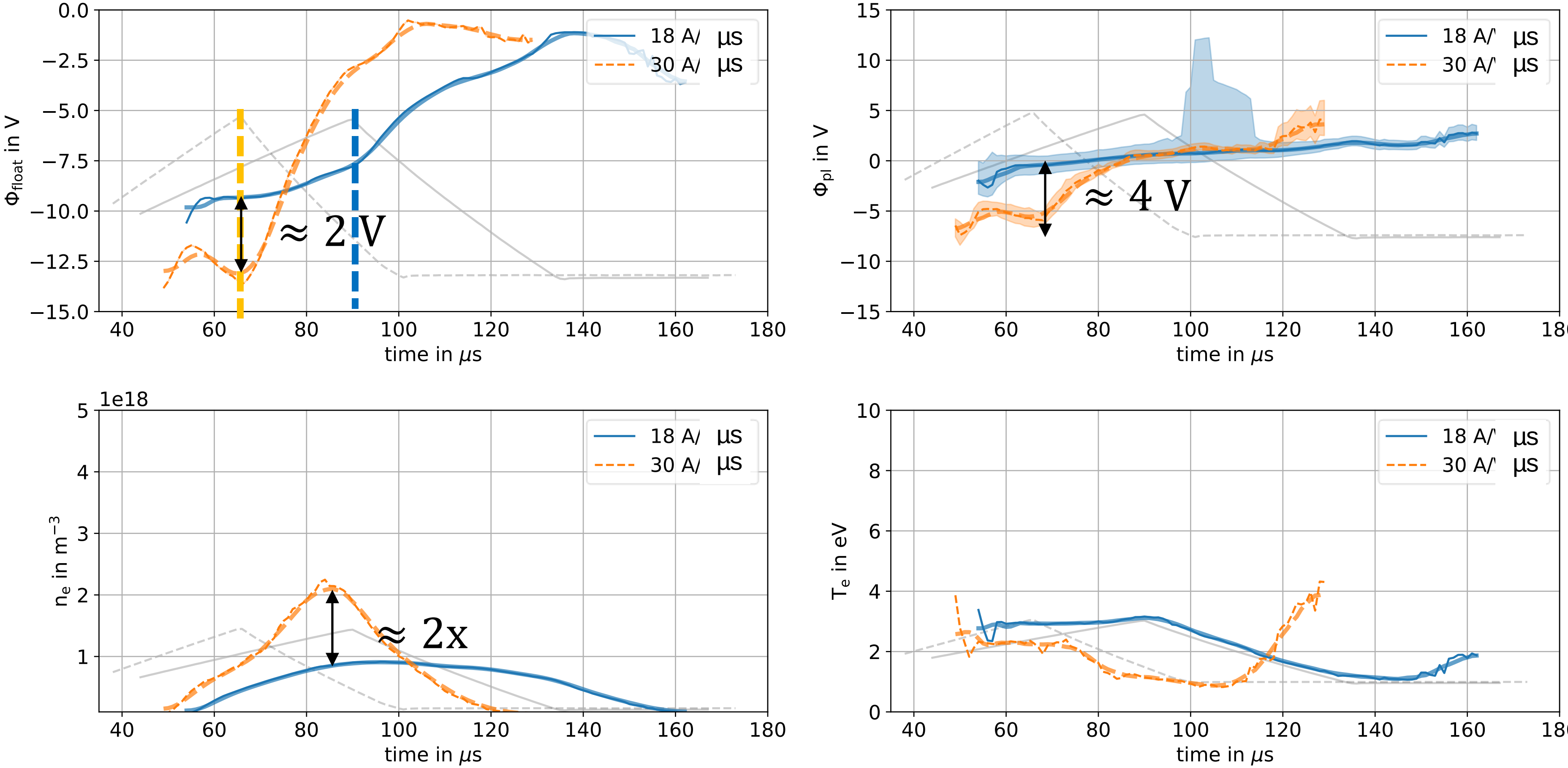
Langmuir Probe

# Plasma Parameters (1600 A, 18 A/s, magnetic variation)



Langmuir Probe

# Plasma Parameters (1600 A, ramp speed variation)



# Setup for a Retarding Field Analyzer

**Data from Langmuir measurements**

$n_e \approx 0 - 2.6\ e^{-18}$

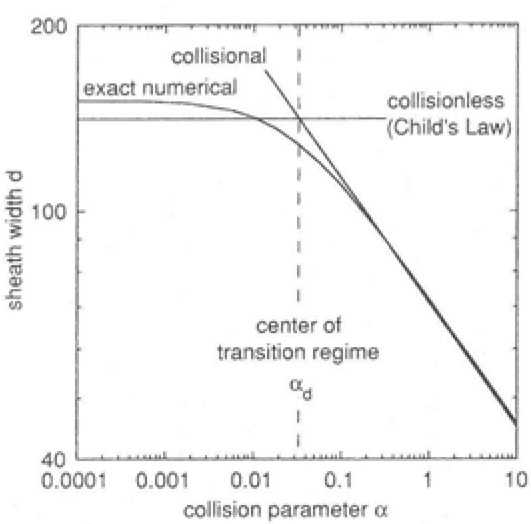
$T_e \approx 1 - 2\ eV$

$\Phi_{pl} \approx -5..5\ V, 0\ V\ @\ n_{e,max}$

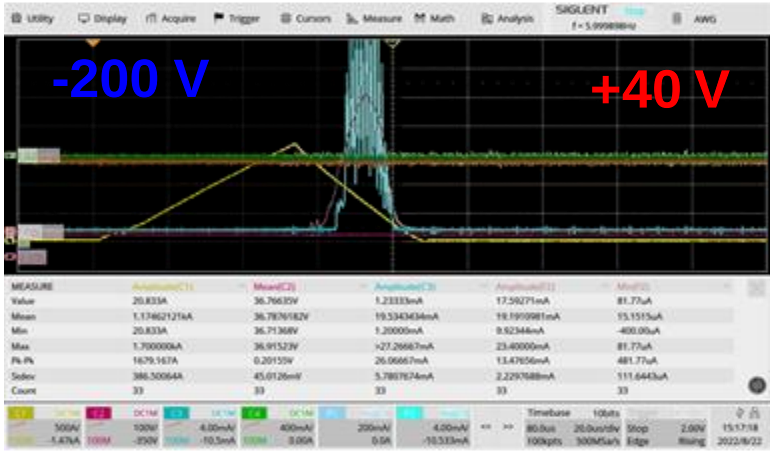
$\lambda_{De} \approx 4.6$ um im Dichte maximum

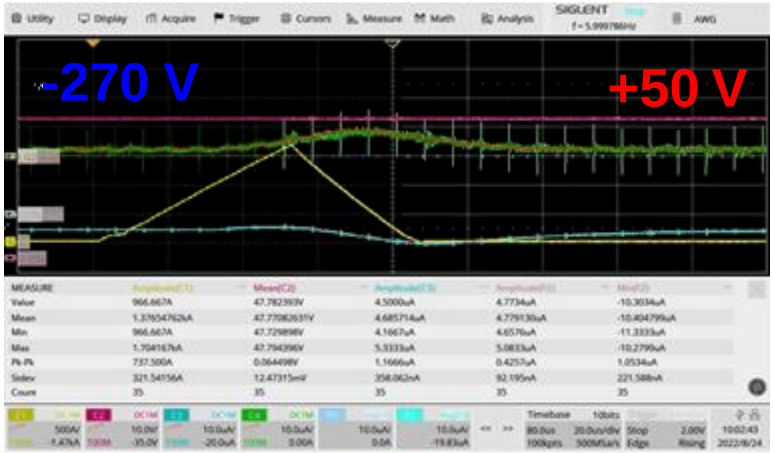
$s_{\text{coll. less}}(200\ V) \approx 2$ mm

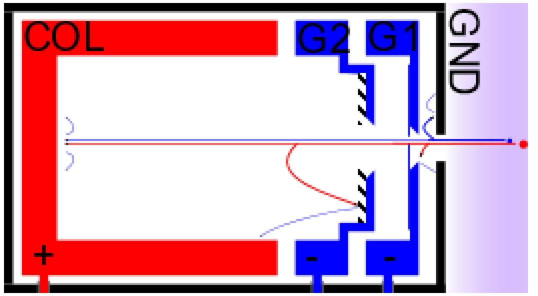
$s_{\text{coll. less}}(270\ V) \approx 2.5$ mm

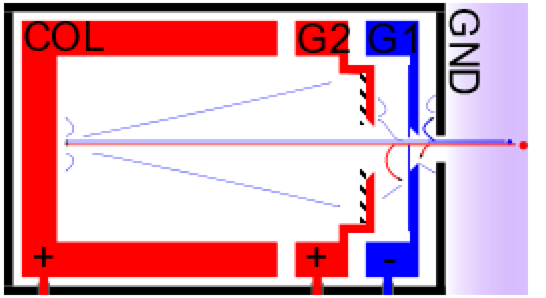


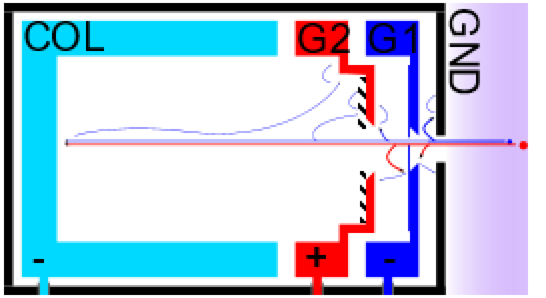

T. E. Sheridan and J. Goree
Physics of Fluids B: Plasma Physics **3**, 2796 (1991)



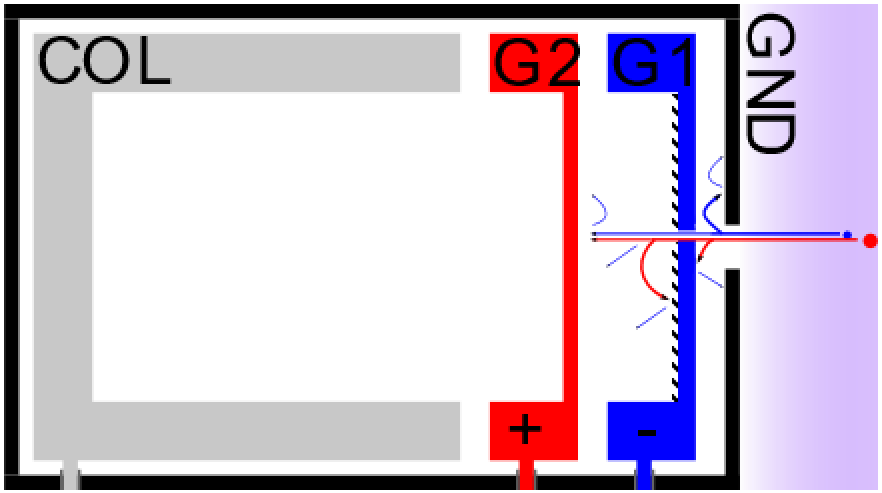



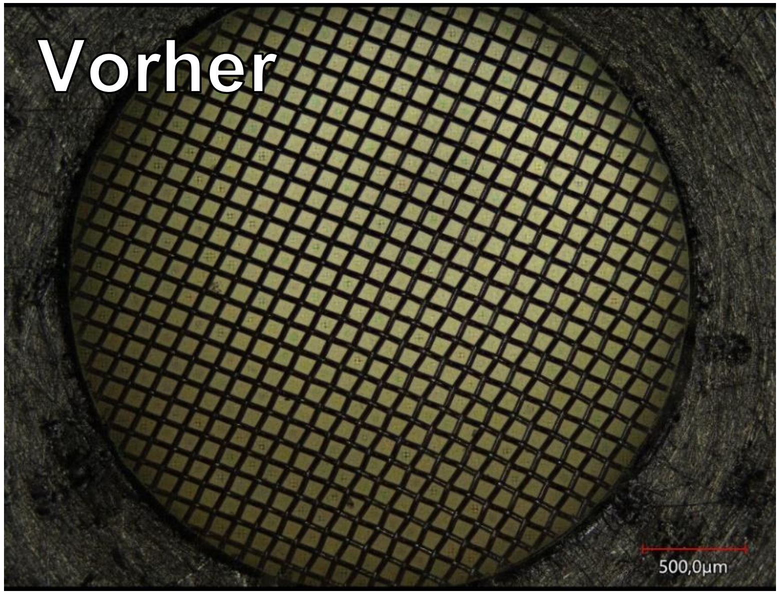



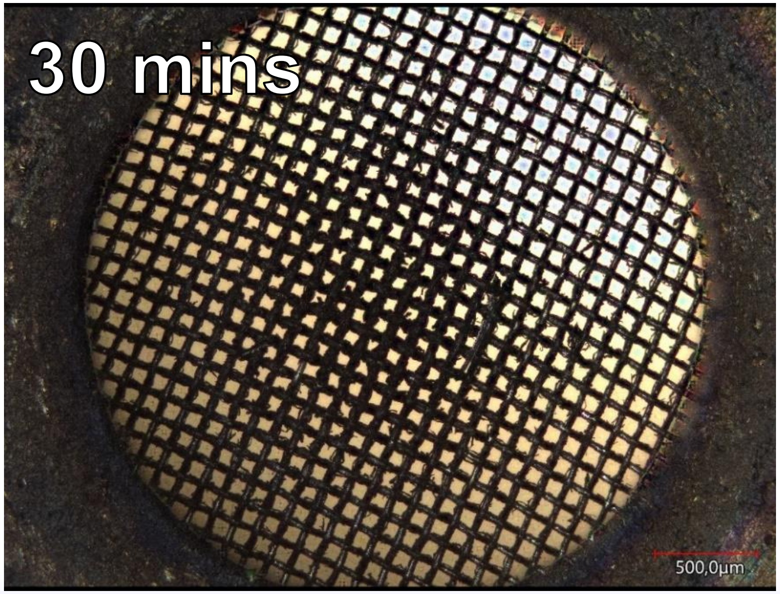



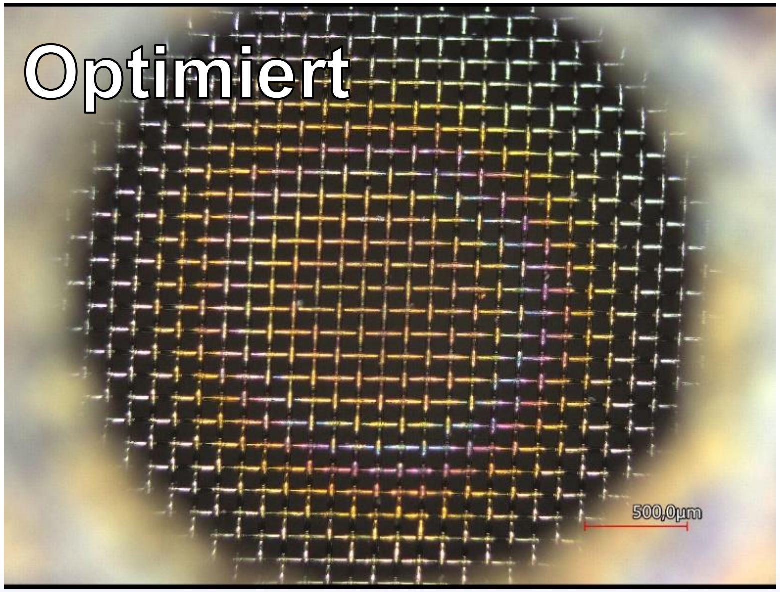



# Setup for a Retarding Field Analyzer



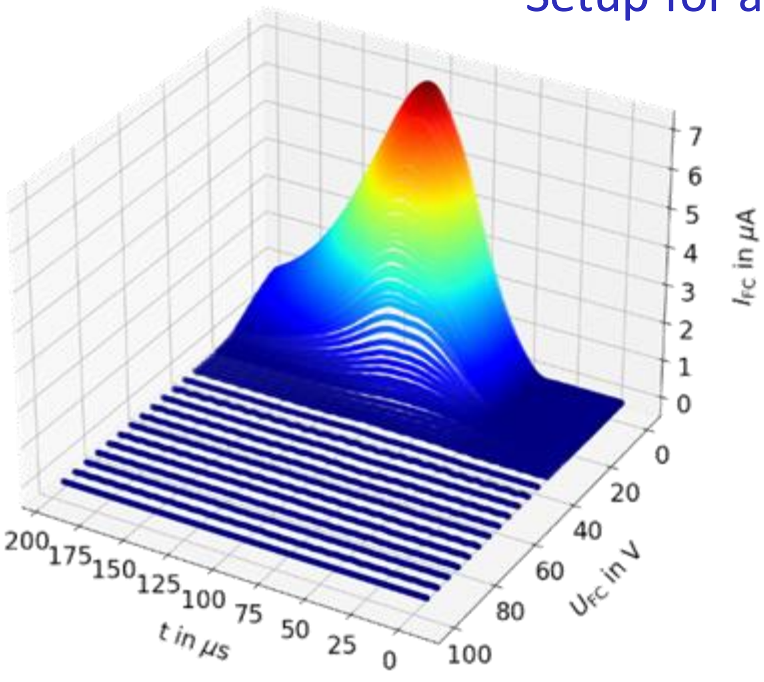



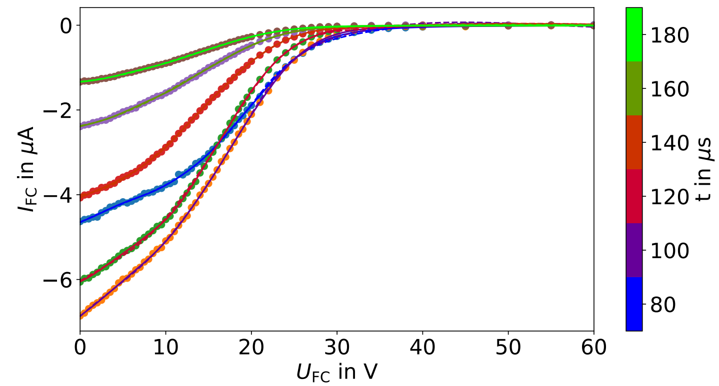



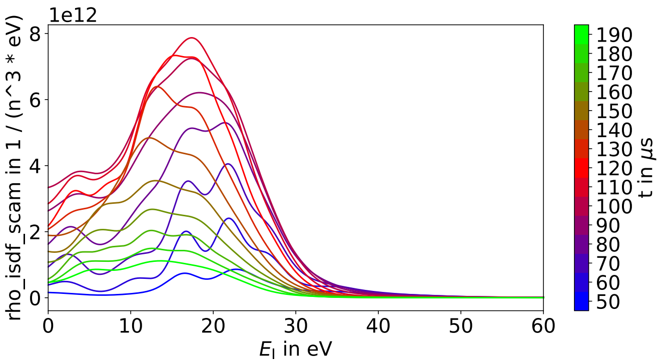



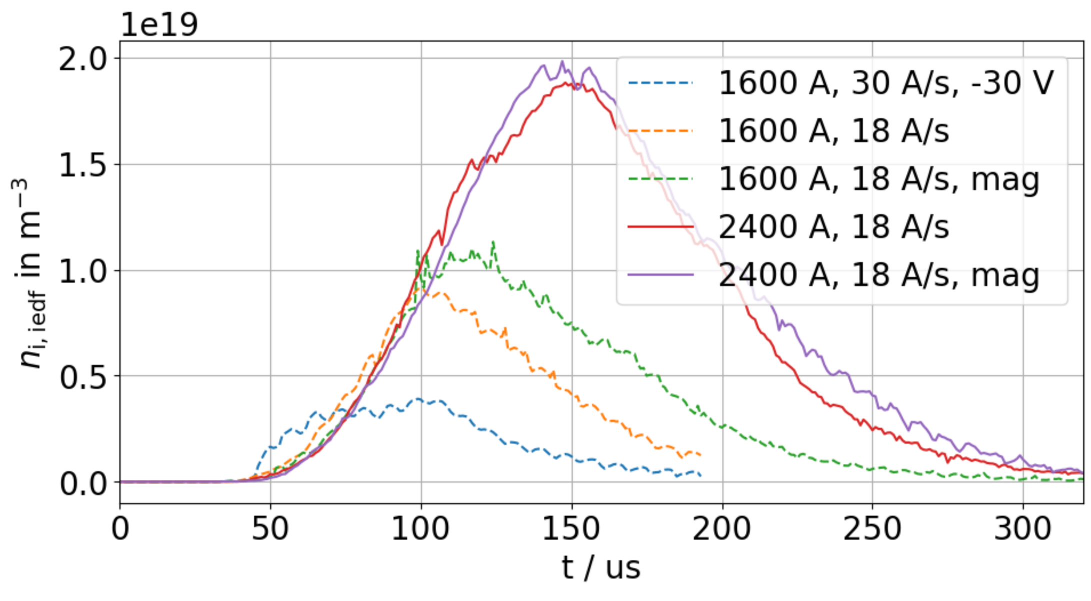
RFA / Faraday Cup

# Setup for a Retarding Field Analyzer

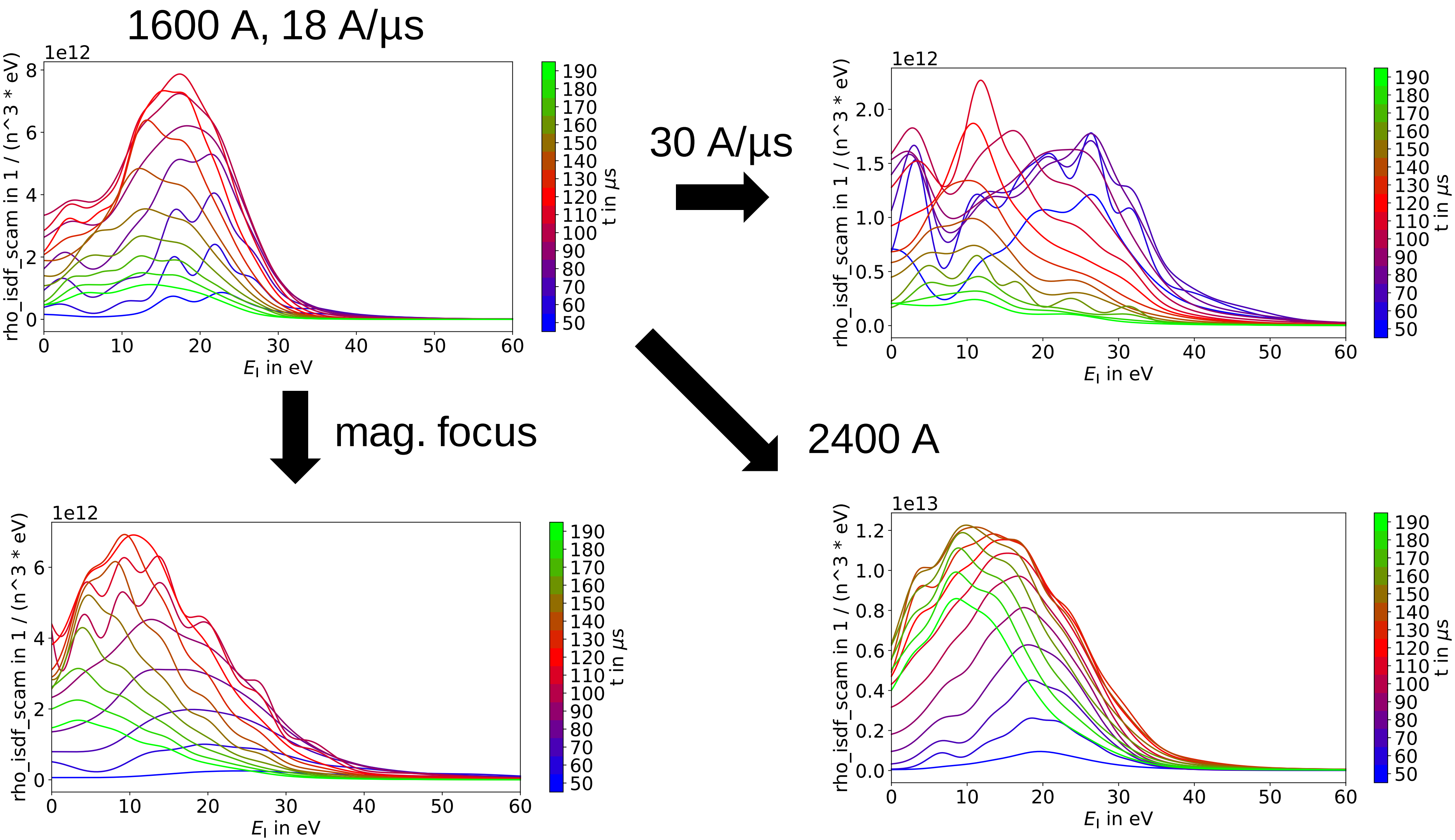


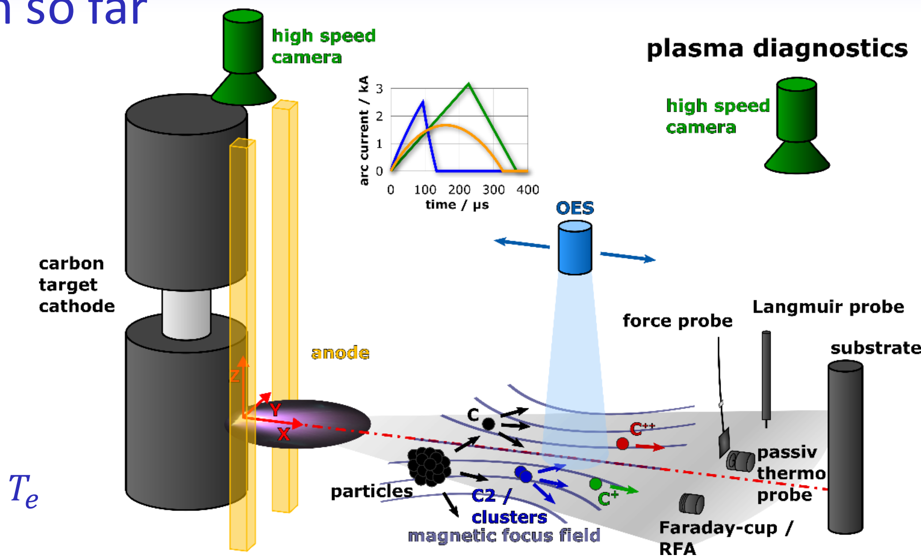
# Setup for a Retarding Field Analyzer



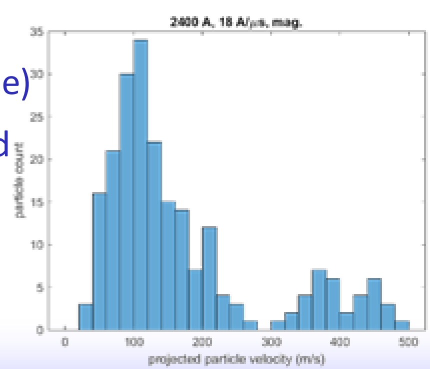
Conclusion and Outlook

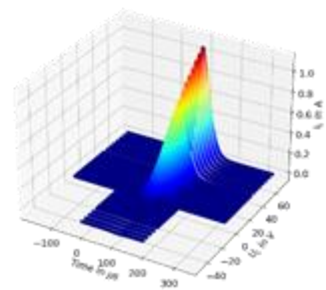
# Conclusion so far

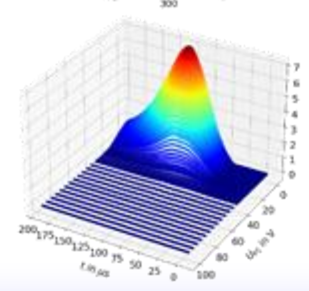
- Ionization is heavily depended on discharge ramp speed
- Magnetic fields push the plasma towards the center increasing ionization and density
- Faster projectiles than other sources, no very slow particles
- High BIAS and double Ionization should allow for coating of non conductive materials

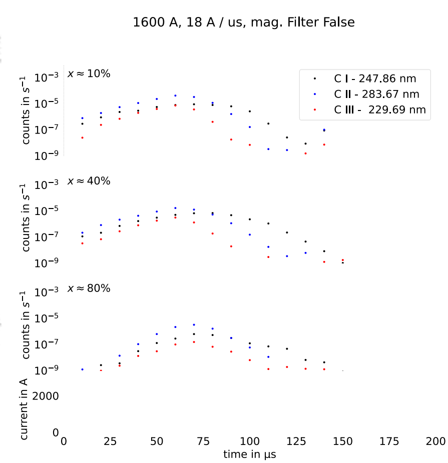



- Langmuir measurements were done to obtain EEDFs, $n_e$ and $T_e$
- RFA measurements used to determine ISDFs, $n_i$ and $T_i$
- Echelle spectrometer measurements obtained spatially and time resolved charge density evolution
  - Different species are traveling at the same speed but are generated at different stages
- Not shown today
  - Energyflux measuerments (PTP)
  - Impulsflux measurements (force probe)
  - Spectrometer absolute calibration and density profiles
  - Cathode spot evaluation



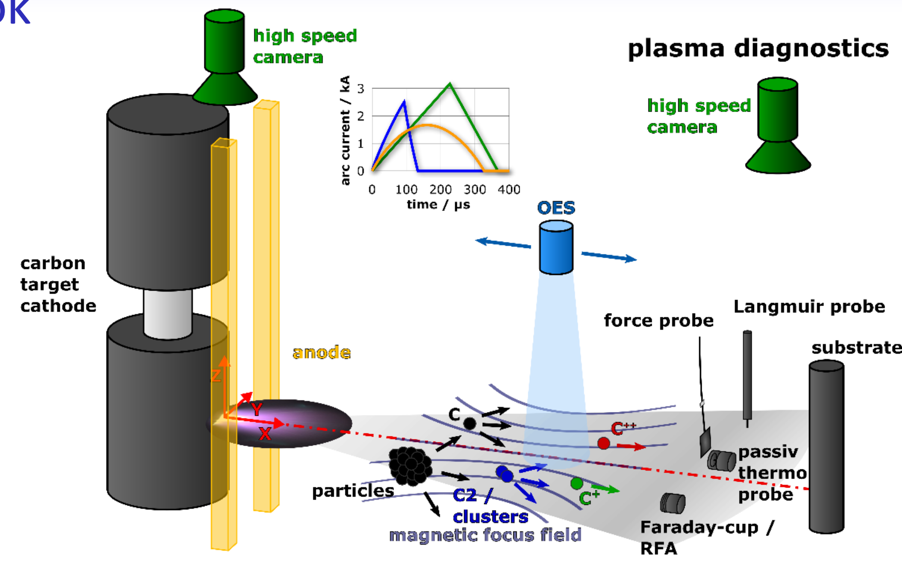


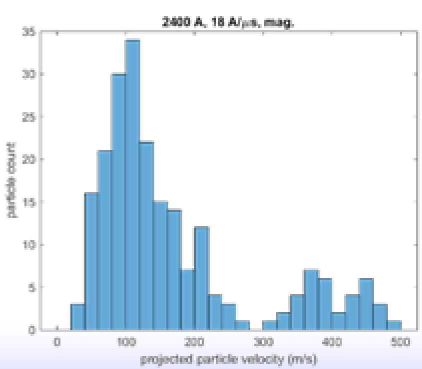


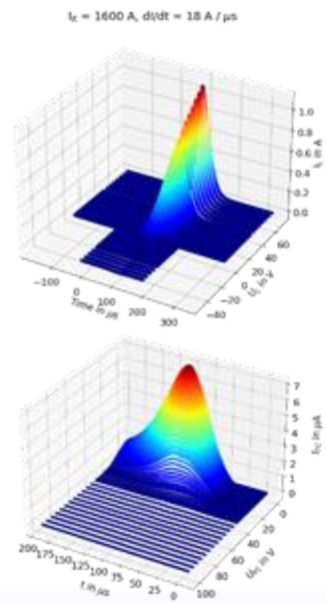
# Outlook

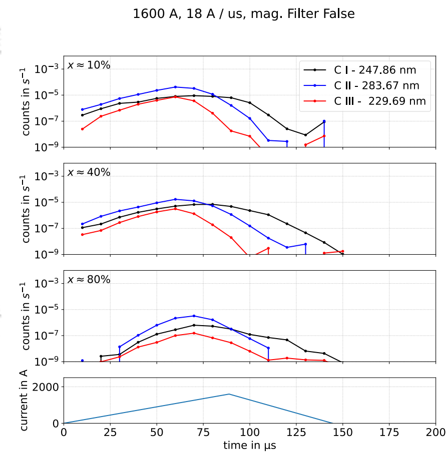
## Goals and Outlook

- Suppressing particle generation and improving particle filtering
  - Analyze basic physics of cathode spot generation
- Enhancing deposition rates and uniformity
- Coating of nonconductive materials
  - What effect do other energy contributions (electrons, radiation…) have on ta-C coatings?
- Force probe measurements to determine neutral flux contributions
- PIC Simulations



# Thank you for your attention