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X. Erfahrungsaustausch „Oberflächentechnologie mit Plasma- und Ionenstrahlprozessen“

# Time-Resolved Double Probe Measurements in Pulsed Magnetrons

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**(BMBF Project FKZ 13N8053)**

Outline:

- Introduction
- Double Probe Setup
- Time-Resolved Measurements
- Experimental Results
- Summary

Mühlleithen, 18 / 03 / 2003

# Introduction

- magnetron sputtering: well established technique for film deposition

- problems depositing dielectric (oxide) films
- films are highly insulating

- **r.f. sputtering:**

  \+ stable sputtering possible, e.g. from oxide targets
  \- low deposition rate

- **d.c. sputtering:**

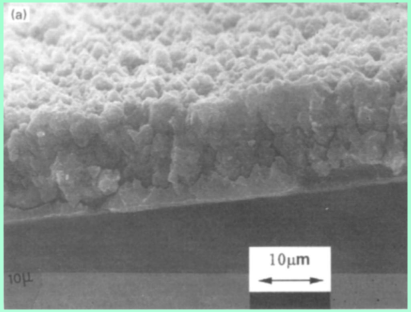
  \+ high deposition rate
  \- oxide formation on the metallic target

    ⇒ charging up, unstable process
    ⇒ electric breakthrough, micro-arcs
    ⇒ incorporation of metallic particles into the films

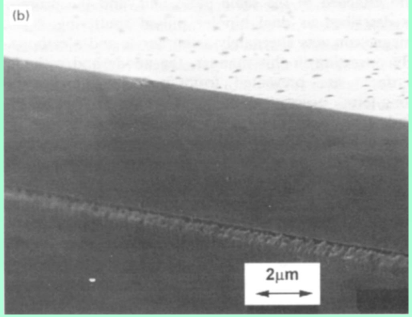
# Introduction

- to overcome the problem: pulsed d.c. discharge (10 - 1000 kHz range)
  - ⇒ surface discharging during off phase
  - ⇒ arc prevention and improved film quality

$Al_2O_3$:
[1,2]



d.c.



pulsed d.c.

  - unipolar: „self-discharging“ = target grounded/floating during „off“
  - **bipolar: active discharging = target positive during „off“**

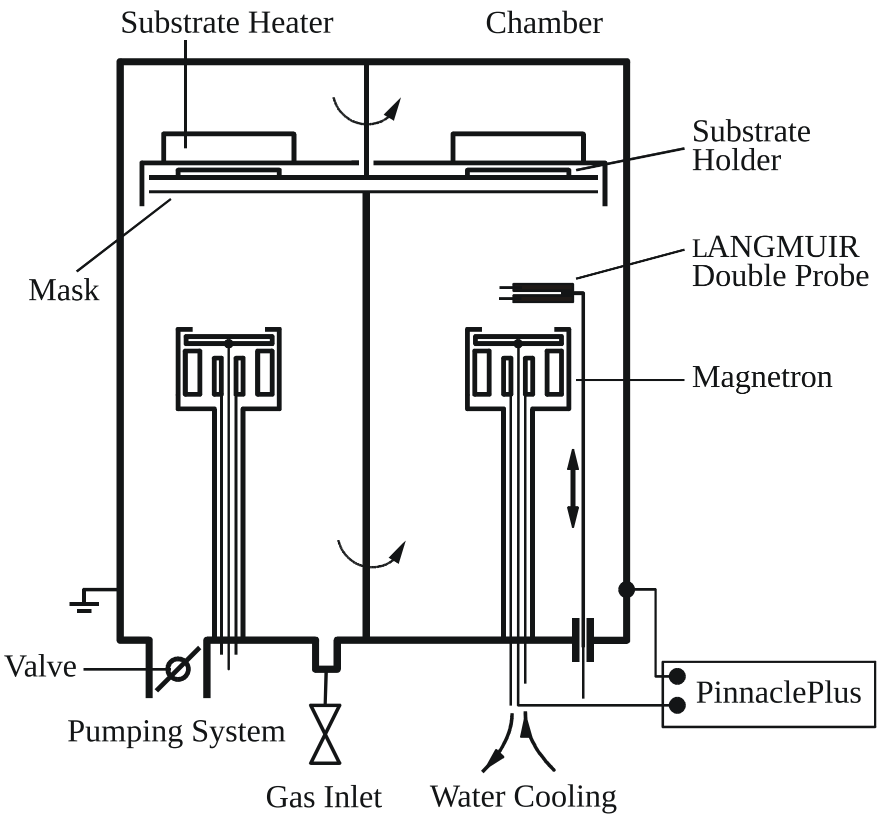
- further improvement of film properties reported

⇒ due to highly ionised and energetic plasma ?

- development of time-resolved plasma diagnostics necessary
- Langmuir probe → charge carrier density and electron temperature

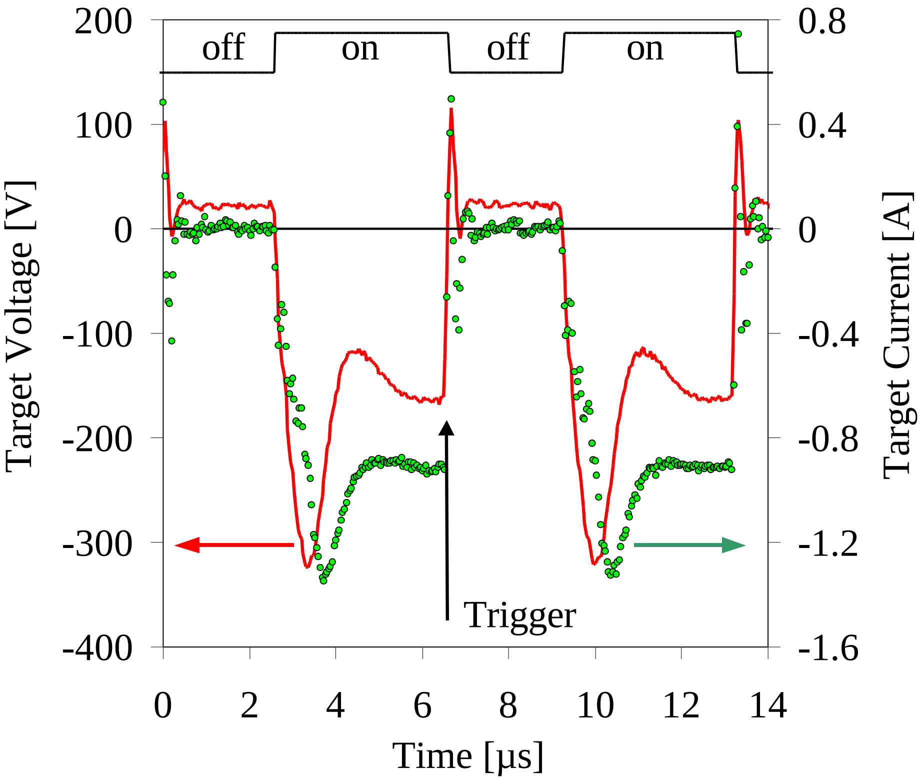
[1] Kelly, Arnell, Vacuum 56 (2000) 159-172
[2] Kelly et al., Surf. Coat.Technol. 86-87 (1996) 28-32

# Experimental Setup



- system: deposition of MgO
- cylindrical magnetron
  - Ø = 100 mm
  - target material : Mg
- pressure: 0.4 Pa (const.)
- gas: 50 sccm Ar + 10 sccm $O_2$ (oxide mode)
- **varied: frequency f, off time $t_{off}$, duty cycle**
- rotatable substrate holder: float., 80 mm from target
- probe position:
  - on-axis
  - 62 mm from target

# Experimental Setup



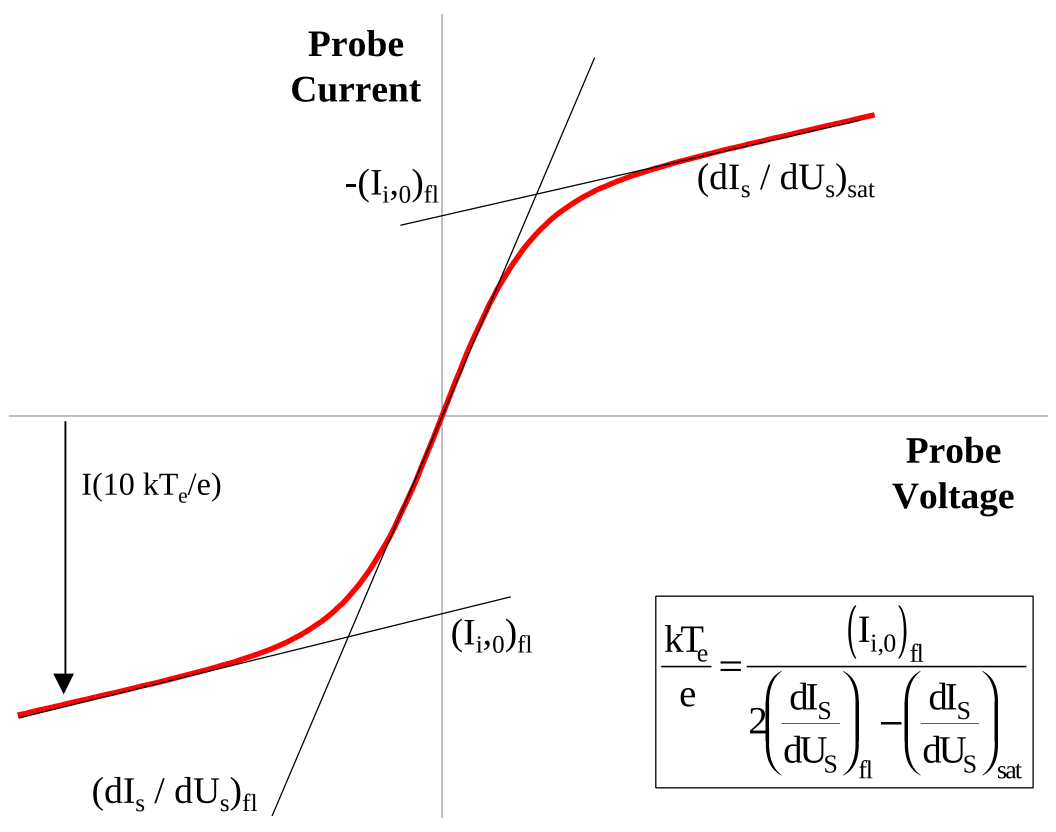
<P> = 100 W, f = 150 kHz, $\tau_{off}$ = 2.6 µs
U measured by potential divider (P5200)
I measured by current probe (A6306)

- pulsed power supply: Pinnacle Plus (AE), 5 kW
- asymmetric bipolar mode: small voltage during „off“
- operation at low power: 100 W to prevent probe contamination
- no rectangular waveforms for voltage or current
- sharpest transition = voltage during switching off

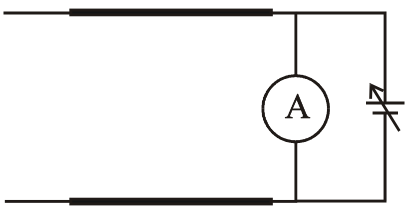
⇒ taken as trigger

# Langmuir Double Probe - Principle

Typical I-U Characteristics:



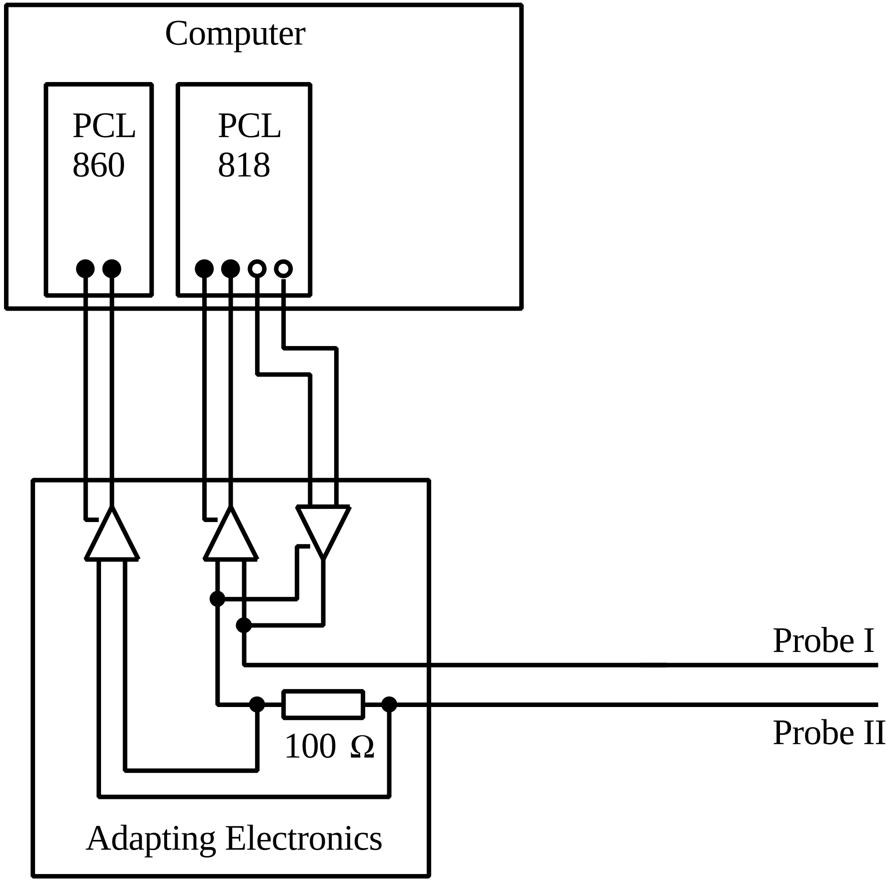
$$\frac{kT_e}{e} = \frac{\left(I_{i,0}\right)_{fl}}{2\left(\frac{dI_S}{dU_S}\right)_{fl} - \left(\frac{dI_S}{dU_S}\right)_{sat}}$$



- probe voltage between two (identical) probe tips
- current limited by ion saturation current
- $T_e$ from three slopes
- n after Sonin from current at $U_S = 10\ kT_e/e$
- symmetrical

⇒ well suited for deposition plasmas

# Usual (Static) Probe Circuitry



- voltage supplied by DA card

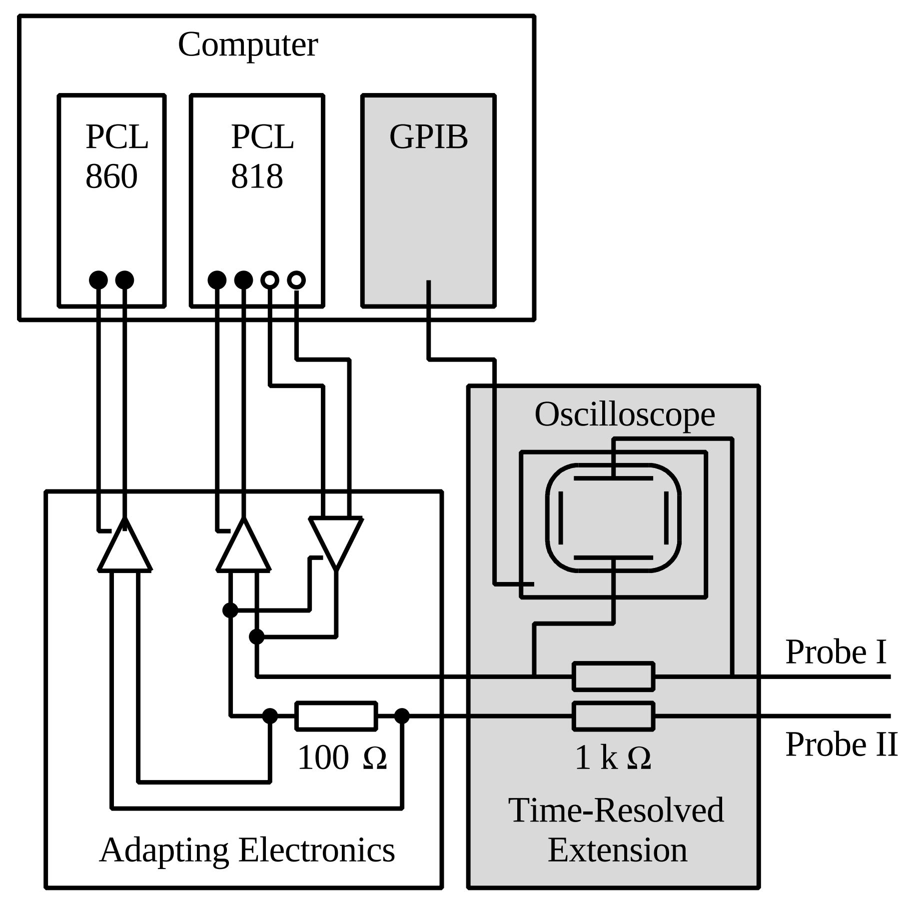
  (0 ... +10 V)
- transformed into probe voltage by adapting electronics

  (-100 ... +100 V)
- current measured across internal resistor (100 Ω) by high resolution AD card
- time for one current measurement: 200 ms

⇒ averaged over many cycles, integrated measurements

- whole system: powered via insulation transformer

# Extension for Time-Resolved Measurements



- approach: keep approved setup
- addition of two 1 kΩ resistors and digitising oscilloscope
- probe voltage varied manually
- for each voltage: probe current acquired with oscilloscope across external resistor
- averaged: typically 20 cycles
- scope output read by GPIB interface
- still: all components powered via isolation transformer

# Data Processing / Evaluation

- for each voltage $U_k$:
- current-time characteristics I(t)
- typically 119 files

$$\begin{array}{c|cccccc}
 & U_0 & U_1 & \dots & U_k & \dots & U_n \\
\hline
t_0 & I_{0,0} & I_{0,1} & \dots & I_{0,k} & \dots & I_{0,n} \\
t_1 & I_{1,0} & I_{1,1} & \dots & I_{1,k} & \dots & I_{1,n} \\
\dots & \dots & \dots & \dots & \dots & \dots & \dots \\
t_m & I_{m,0} & I_{m,1} & \dots & I_{m,k} & \dots & I_{m,n}
\end{array}$$

- for each time $t_i$:
- current-voltage characteristics I(U)
- 500 characteristics

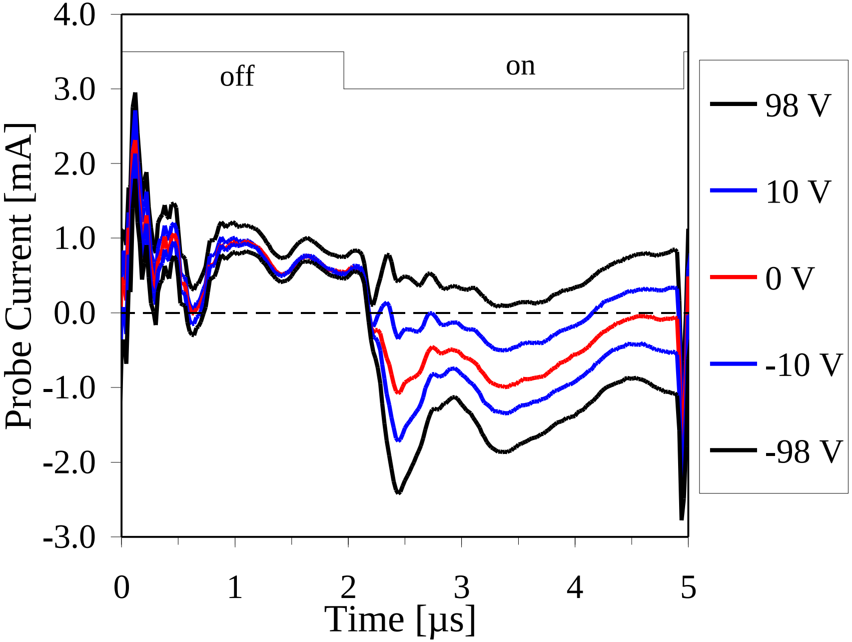
**matrix**

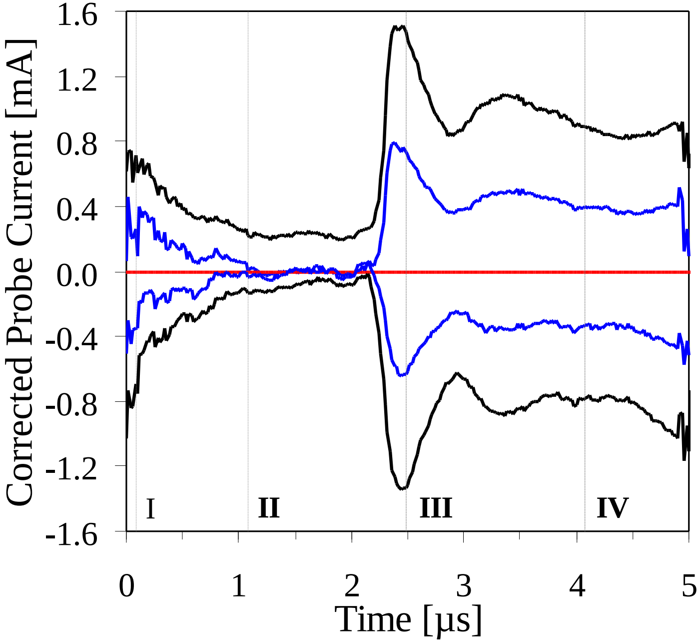
**transposed**

**computer-controlled**

same characteristics as for integral measurements, same treatment

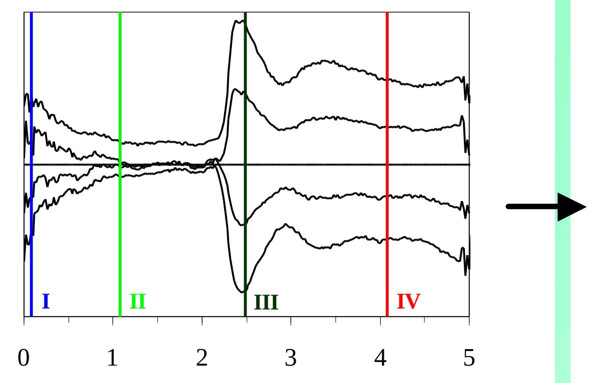
# Elimination of Noise





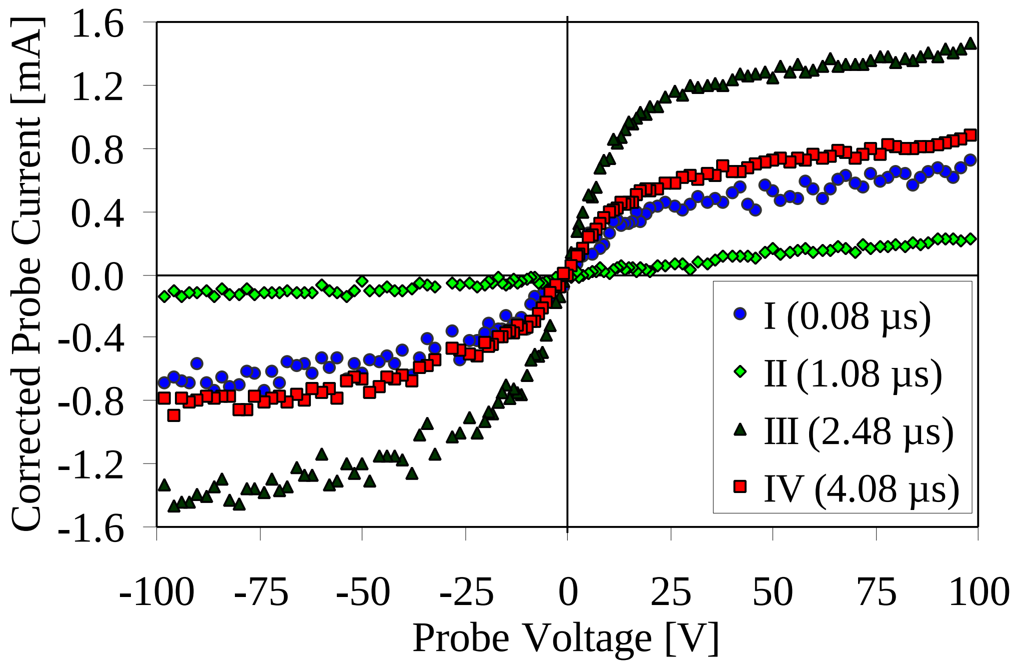
- U = 0 V: non-zero probe current signal = noise
- noise constant throughout whole voltage range
- subtracted from each measured current signal → **real probe current**

# Sample Characteristics for Different Times



**4 examples**
**for the transformation**
**of real data**

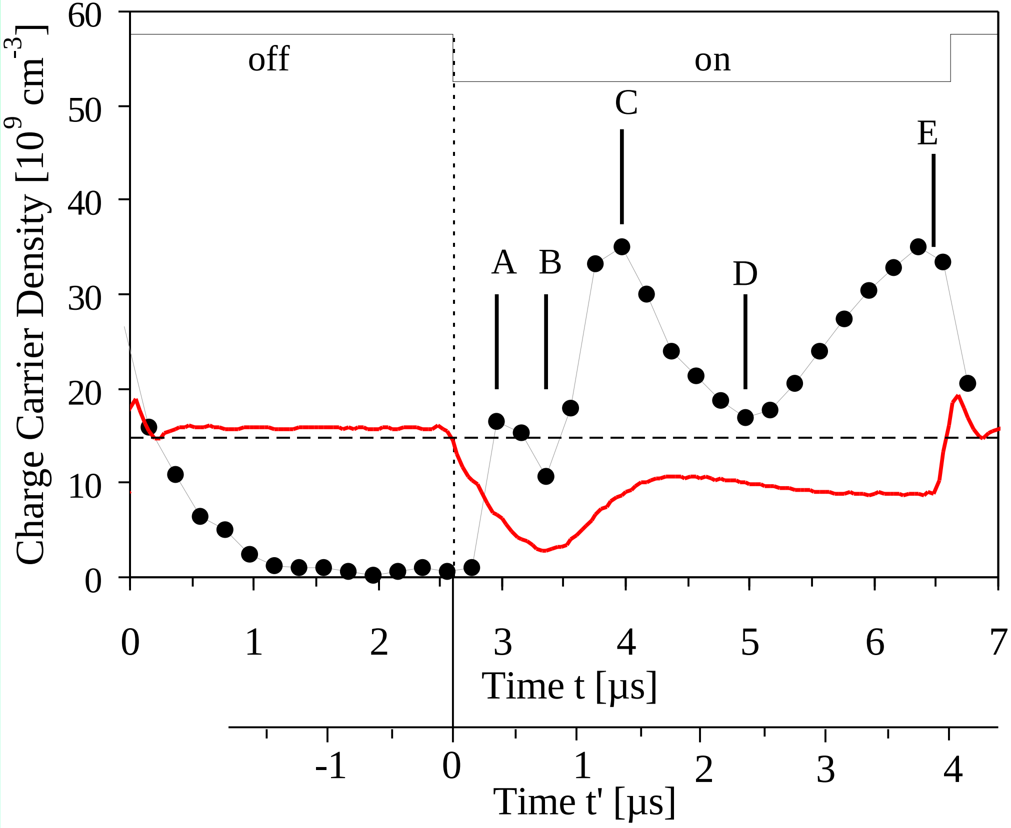
f = 150 kHz, $\tau_{off}$ = 2.6 µs
duty cycle = 0.6



- on phase: qualitatively good double probe characteristics
- off phase: low current and noisy characteristics at the end

⇒ at most points in time: fairly evaluable

# Time Dependence of the Charge Carrier Density (150 kHz)



**off phase:**

- exponential decay
- time constant: 490 ns (independent of f, $\tau_{off}$)

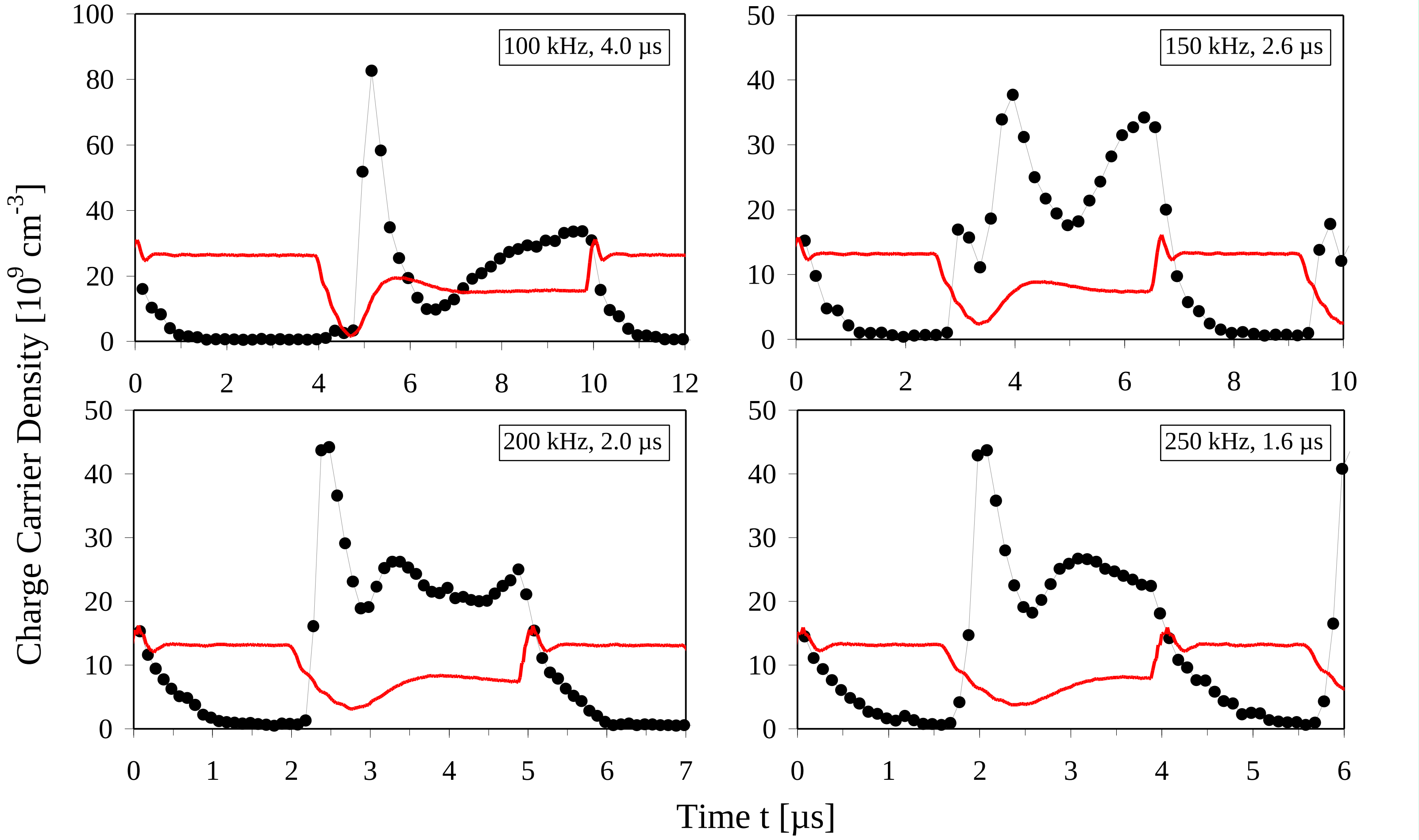
**on phase:**

- typical extrema Y
- D-E: transition to stationary discharge
- A-C: „switching structures“

**analysis:**

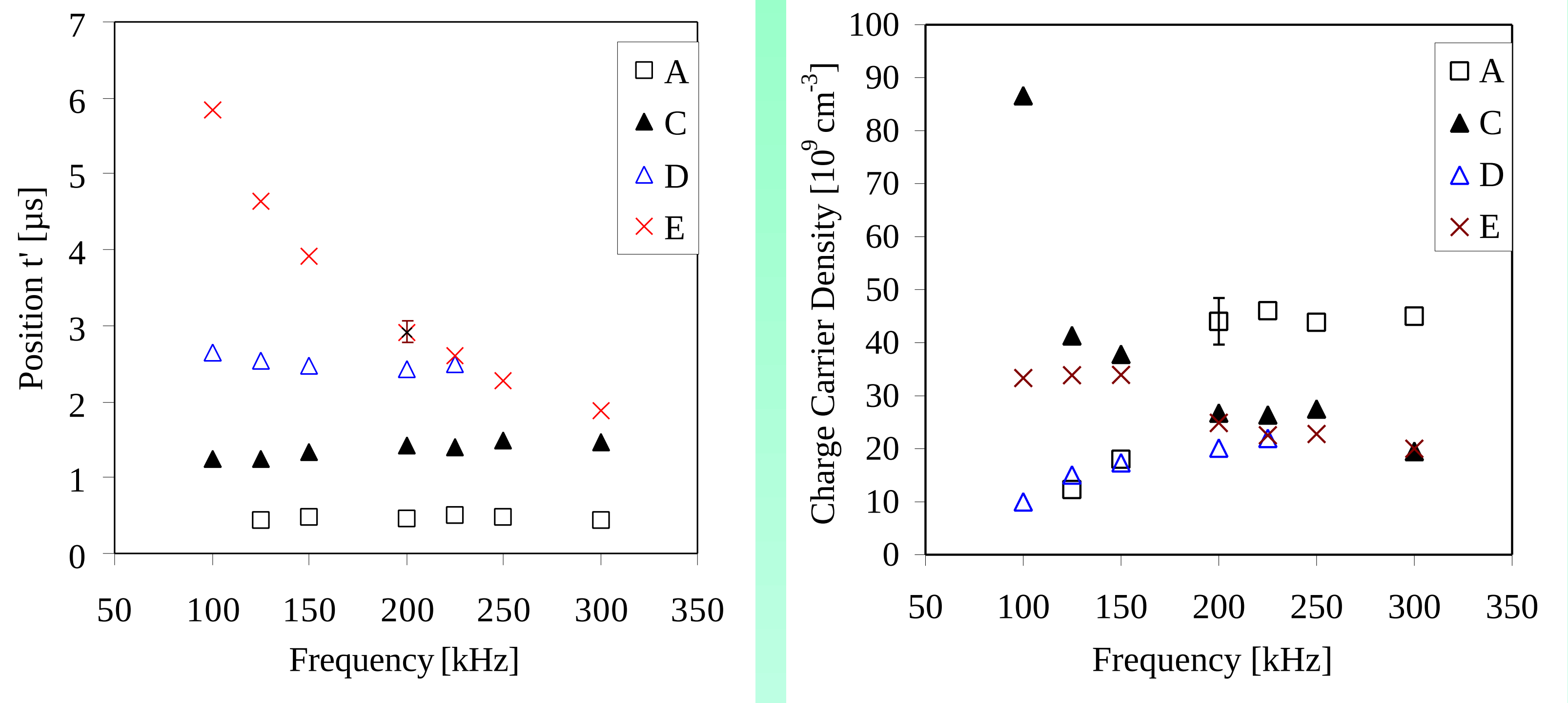
- n(Y) and t‘ (Y)
- second time axis (t‘ = 0 at switching on)

# Change of Time Dependence with Frequency



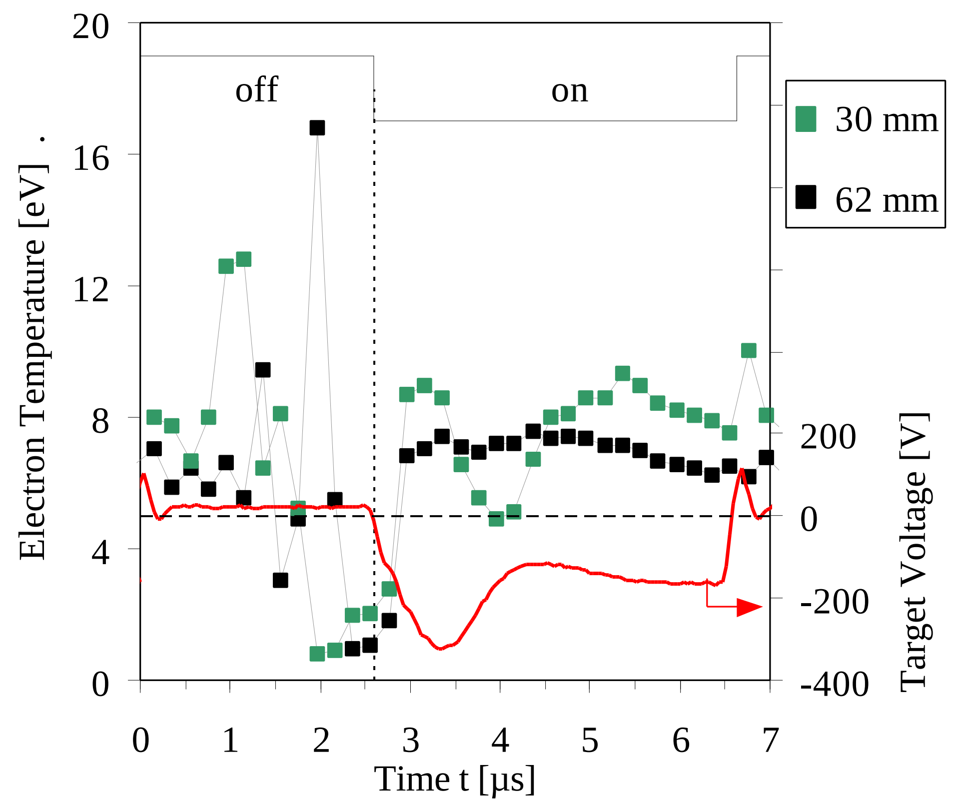
**duty cycle = const. = 0.6**, <P> = 100 W, p = 0.4 Pa, $F_{Ar}/F_{O2}$ = 50/10

# Temporal Positions and Heights of the Extrema



- except of E (switch off) position almost unaffected by frequency
- f < 200 kHz: strong variations (A,C), f > 200 kHz: almost no changes
- n(C): increasing with increasing off time

# Time-Resolved Electron Temperature



f = 150 kHz, $\tau_{off}$ = 2.6 µs, duty cycle = 0.6

- on: nearly constant (6 eV)
- off: hardly accessible due to low probe current
- literature: significant peak after switching on (~ 13 eV) but close to target [1]
- reduced distance to target (30 mm):
  - variation during on phase
  - increased at initial stage (9 eV)

[1] J. Bradley et al., Surf. Coat. Technol. 142-144 (2001) 221

# Summary

- time-resolved double probe measurements in pulsed (100-350 kHz) magnetron
- proper modification of existing static probe system
- time resolution of better than 50 ns

- typical decay of the charge carrier density in the off phase (490 ns)
- although integrated charge carrier density constant ($2 \cdot 10^{10}$ cm$^{-3}$)
- strong variations in the on phase of $(1\text{-}8) \cdot 10^{10}$ cm$^{-3}$
    - characteristic peak structure: first two peaks correlated to switching
    - second peak connected density at the end of the off phase

- electron temperature time dependence (~ 6 eV)
    - difficult to access
    - small variations observed close to the target

# Outlook

- explanation of the time-dependence
- increased discharge power (deposition conditions)

- improvement of the measurement system
  - minimising floating “stray” current
  - compensating for voltage drop across 1 kΩ resistors

- improvement of $T_e$ measurement

- combination with other techniques (OES)

- treatment of negative ions ?