Advanced Electrical and Optical Diagnostics on Atmospheric Pressure Plasmas with Focus on Microdischarges

Klaus-Dieter Weltmann, Torsten Gerling, Hans Höft, Tomas Hoder, Andrei Pipa, Andrej V. Nastuta*, Ronny Brandenburg, Rene Bussiahn, Manfred Kettlitz

Leibniz Institute for Plasma Science and Technology, Greifswald, Germany
** Alexandru Ioan Cuza Universtity, Iasi, Romania*

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FROM THE IDEA TO THE PROTOTYPE

Need for atmospheric pressure plasmas

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New Fields of plasma technology …

- Plasma medicine
- Gas treatment
- Surface treatment
- Detection devices
- etc.

… require …

… Atmospheric Pressure Plasmas Sources:

- Dielectric Barrier Discharges (DBDs)
- Plasmajets
- Corona Discharges
- Gliding Arcs
- etc.

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Outline

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1 Atmospheric Pressure Plasmas Sources
- Principles and overview
- Need and concepts for diagnostics

2 Fast diagnostics
- Electrical diagnostics
- Optical and spectroscopic diagnostics

3 Discussion of examples:
Transient plasmas in „slightly reactive“ gas mixtures
- Single filament Dielectric Barrier Discharge
- Transient spark discharge
- Needle-to-plane discharge

4 Summary and outlook

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Atmosphreric Pressure Plasmas: Toolbox

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Non-Thermal (NT) Plasmas		Thermal Plasmas
"Cold" Non-Thermal Plasmas	Translational ("Hot NT") Plasmas	Thermal Plasmas
$T_i \approx T_g \approx 300 \ldots 400$ K $T_i << T_e < 10^5$ K (10 eV)	$T_i << T_e \lesssim 10^4 \ldots 10^5$ K $T_i \approx T_g \lesssim 4\ 10^3$ K	$T_i \approx T_g \approx T_e$ $T_x < 5\ 10^3 \ldots 10^4$ K

Barrier discharges

Gliding Arc

Arc

Coronas

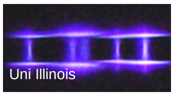
Arc jet

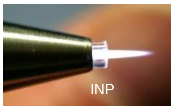
Microplasma-Arrays

Plasma Torch

Plasma jets

Microwave Driven Plasmas





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Discharges at atmospheric pressures

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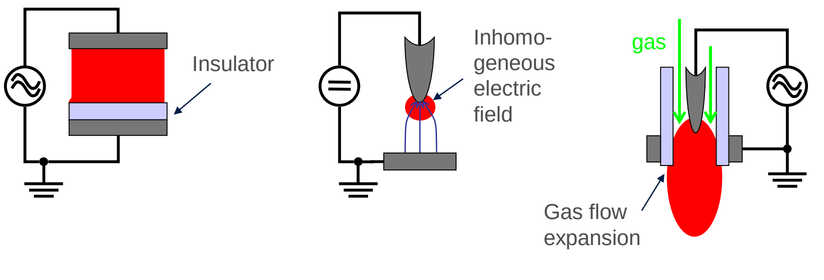
Non thermal plasma generation at 1 atm

($T_e \leq 10^5$ K *but* $T_e >> T_i \approx T_{gas} \approx 300 \ldots 10^3$ K)

Dielectric Barrier Discharge (DBD)

Corona

Plasma Jet



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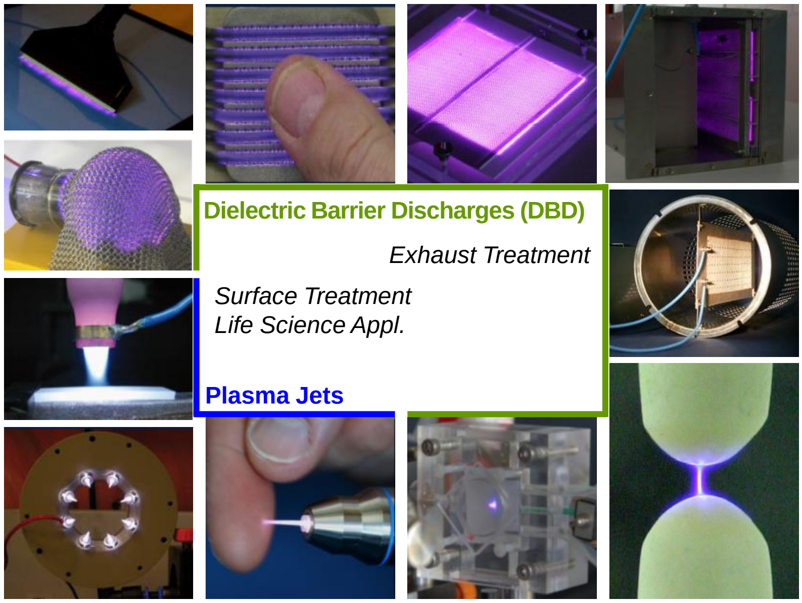
Atmospheric Pressure Plasma Sources



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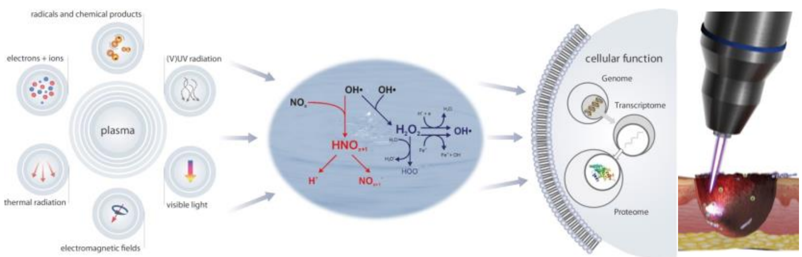
Example: From idea to prototype

Idea: *prototype:*

Plasma → liquid → cell → wound healing



Plasma diagnostics ↔ Plasma modelling

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Example: Plasma medicine

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Plasma sources

- development
- adaptation
- diagnostics
- optimization, control, monitoring
- experimental applications
- clinical applications

in vitro → **in vivo**

Biological effects

- physiological liquids
- cell: - microorganisms
  - mammalian cells
- cell and tissue cultures:
  - not contaminated/infected
  - contaminated/infected
- isolated tissues/organs
- organisms:
  - animal experiments
  - clincal trials/tests

Therapeutic Applications

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Example: Prototype/Product

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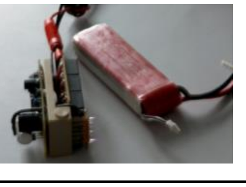
kinpen MED of neoplas tools GmbH

- CE Certification (EU RL 93/42/EWG Tests for Clinical Products)
- Certification as a medical product

- Future
- Intensifing clinical testings(different indications)
- Development for large-area treatment (3-pin Jet – product-line)

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Requirements for technological breakthrough

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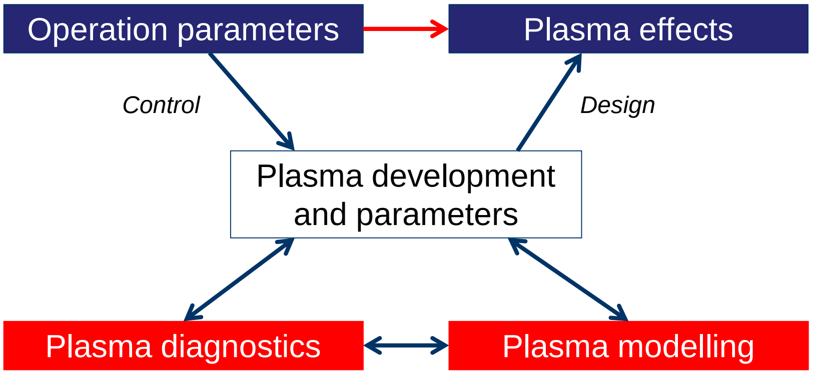
Reliable plasma sources are the key issue for
the development of new technologies and applications,
but need to be **fully charactarized**.



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The challenge for plasma diagnostics

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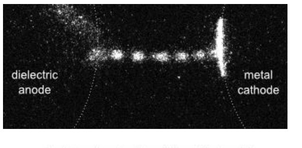
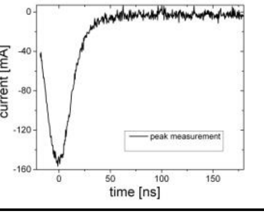
- Filamentary plasmas consiting of distinct discharge channels or **microdischarges** (MDs)
- Small scale (10 µm … 5 mm)
- Transient and short duration phenomena (1 ns … 10 µs)
- Erratic appearance (often)
- Quenching (→ low emission intensity)
- Gradients and instabilities





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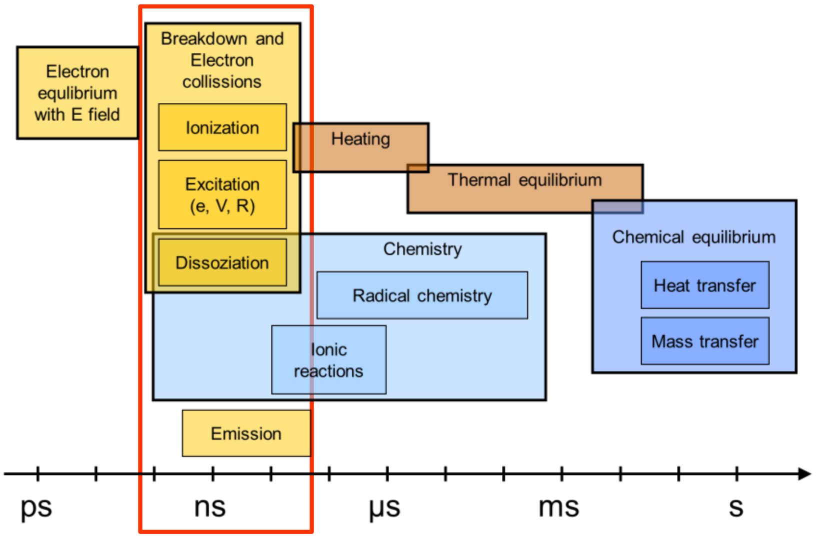
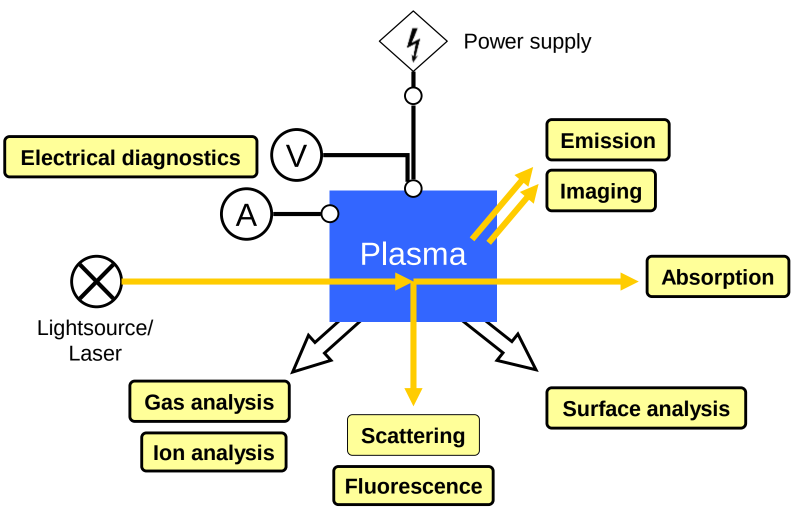
Timescales



P. Bruggeman and R. Brandenburg; JPD 46 (2013) 464001

Diagnostics interfaces



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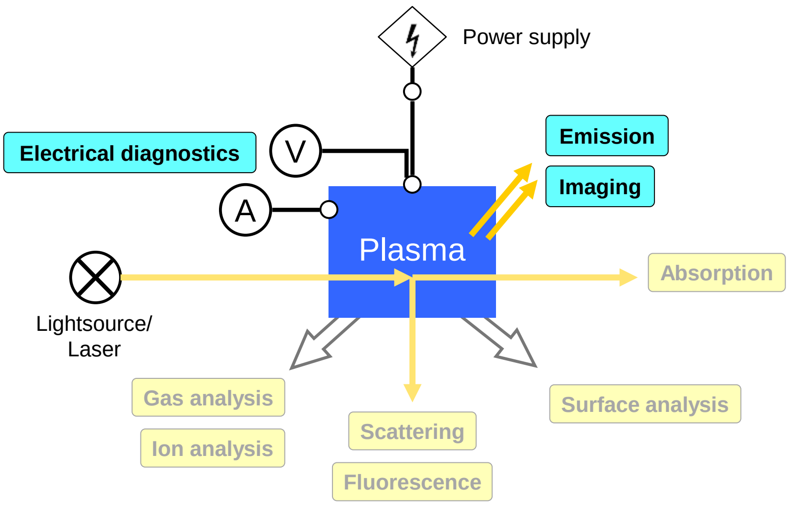
Diagnostics interfaces



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Methods for electrical characterization

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- Voltage-current recording using digital storage oscilloscopes with appropriate resolution and bandwidth
- High voltage probes with appropriate bandwidth and impedance
- Fast current probes (Rogowski coils)
- In case of DBD: Voltage-Charge-Plots (“Lissajous figure“)

Avoid ground loops and stray capacitances/impedances

Probes introduce probe impedance and capacity into electrical circuit

- Interpretation by means of equivalent circuits

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Methods for discharge morphology: ICCD

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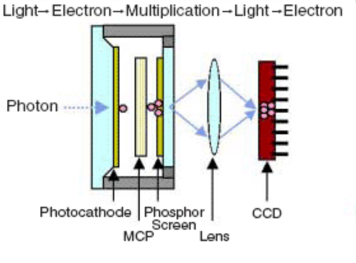

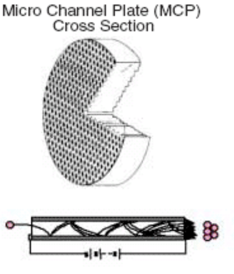

Principle	Image intensification by micro-channel plate; CCD-Sensor
Parameters	Δt down to 2 ns (stroboscopic ICCD go down to 80 ps) Gain 10^5 … 10^6
Pecularities	→ Temporally resolved measurement only if pulsed driven → Photos of individual MDs or discharge channels → Limiting: readout time

Figures from Hamamatsu

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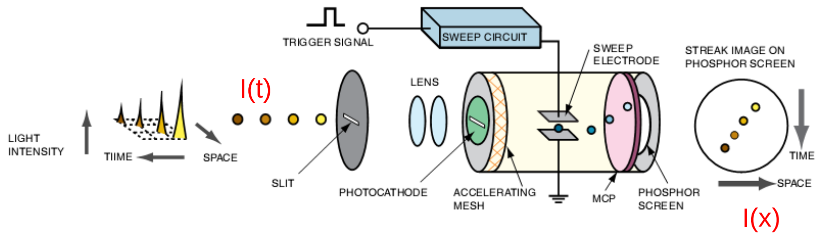
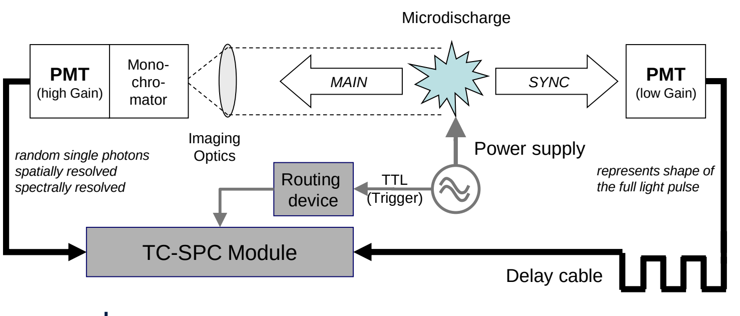
Methods for discharge morphology: Streak



Principle	Temporal profile transformed into spatial profile by defined deflection in streak tube and (I)CCD
Parameters	Δt down to 1 ps Gain $10^5 \dots 10^6$
Pecularities	→ Temporally resolved investigation of individual MDs → One spatial dimension

Figures from Hamamatsu

Methods for discharge morphology: TC-SPC



Principle	Time-correlated single photon counting (TC-SPC) with reference signal from MDs itself
Parameters	Δt down to 12 ps Gain up to 10^8 Δλ about 0.03 nm
Pecularities	→ highest sensitivity, lowest signal-to-noise ratio → temporally & spectrally res. investigation of erratic discharges → averaging over many MDs (stability & reproducebilty required) → 2D spatial resolution possible, but time consuming

K.V. Kozlov, H.-E. Wagner JPD 34 (2001) 3164-3176

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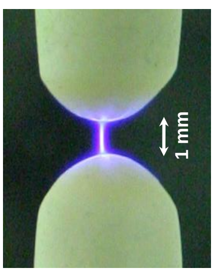
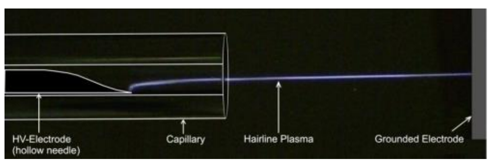
Plasma sources

Single filament volume DBD



- Pulsed operated (10 kHz)
- N_2 with 0.1 vol.% O_2

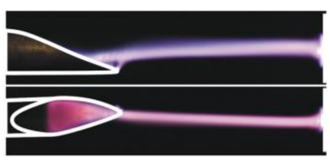
Self-pulsing transient spark



- -DC
- Ar in open air

Needle-to-plane discharge

- Sinusoidal (18 kHz)
- He (+O_2) in open air



Transient plasmas in „slightly reactive“ gas mixtures

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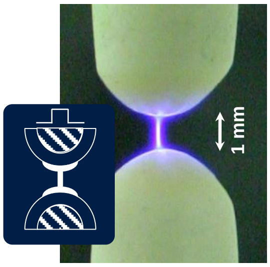
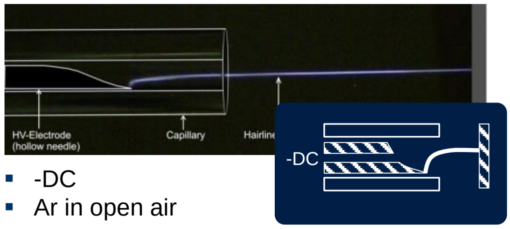
Plasma sources

Single filament volume DBD



- Pulsed operated (10 kHz)
- N_2 with 0.1 vol.% O_2

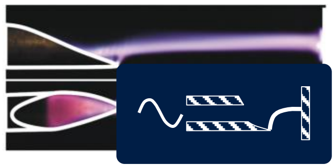
Self-pulsing transient spark



- -DC
- Ar in open air

Needle-to-plane discharge

- Sinusoidal (18 kHz)
- He ($+O_2$) in open air



Transient plasmas in „slightly reactive“ gas mixtures

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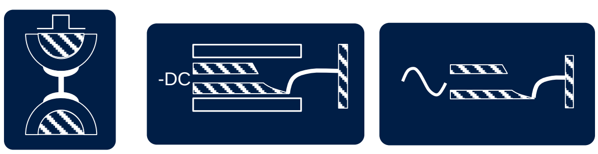
Methods discussed



	Single filament volume DBD	Self-pulsing transient spark (-DC)	Needle-to-plane discharge
Electrical meas.	☑	☑	☑
ICCD	☑		
Phase-resolved ICCD (PROI)	☑		☑
Streak	☑		
TC-SPC		☑	

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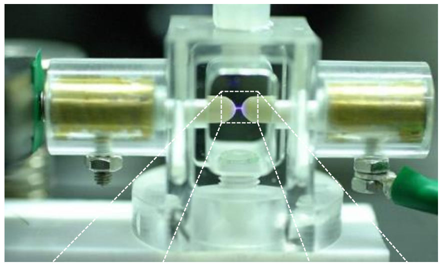
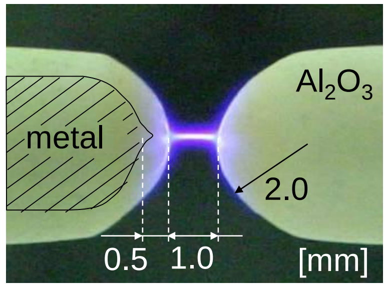
Single filament DBD

Single DBD:
Microdischarge (MD)

- Symmetrical single filament arrangement
- Al_2O_3 covered electrodes ($\varepsilon_r \approx 9$)
- Closed discharge cell flushed with defined gas mixtures

U	10 kV_P
Q	100 sccm
Gases	N_2 with 0.1 vol.% O_2
Duration	20 … 150 ns
Peak current	100 mA

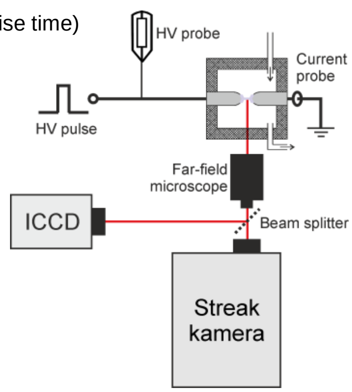




Single filament DBD: Set-up (1)

Diagnostics

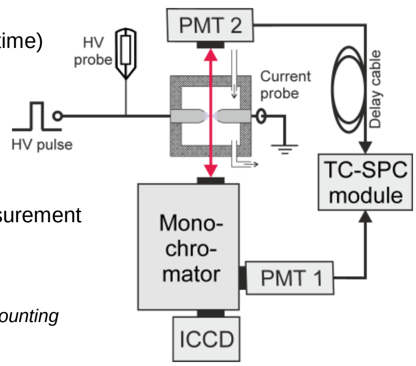
- Electrical probes (down to 350 ps rise time)
- 2D-imaging by ICCD camera ($\Delta x \approx 10$ µm, $\Delta t \approx 2$ ns)
- Spatio-temporal development along MD-axis by streak camera ($\Delta x \approx 10$ µm, $\Delta t \approx 50$ ps)



Single filament DBD: Set-up (2)

Diagnostics

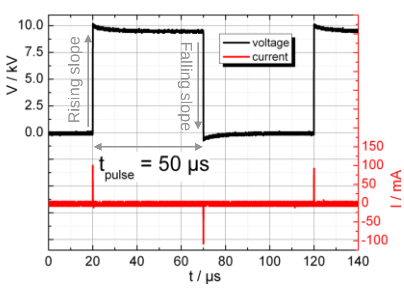
- Electrical probes (down to 350 ps rise time)
- 2D-imaging by ICCD camera
  (Δx ≈ 10 µm, Δt ≈ 2 ns)
- Spatio-temporal development along MD-axis by streak camera
  (Δx ≈ 10 µm, Δt ≈ 50 ps)
- Sensitive and spectrally resolved measurement by TC-SPC also known as Cross-correlation spectroscopy (CCS)
  (Δx ≈ 10 µm, Δt ≈ 12 ps)
  TC-SPC – Time-Correlated Single Photon Counting
  PMT – Photomultiplier Tube
- Optical emission spectroscopy (OES)

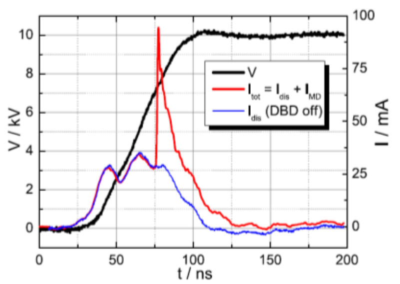


Streak camera and TC-SPC → complementary diagnostics

- Streak camera: fast recording, lower sensitivity
- TC-SPC: slow recording, higher sensitivity and spectral resolution

Electrical behaviour





Pulsed operation for higher power dissipation & density of active species:

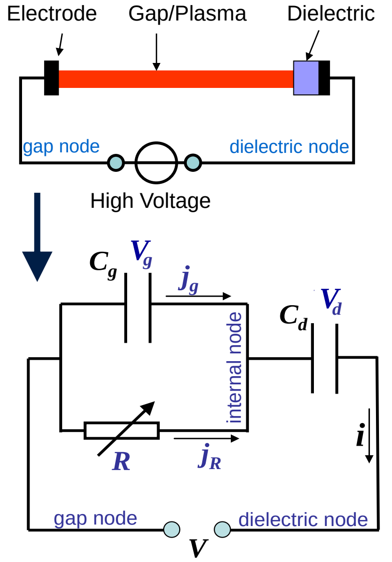
- V_{max}= +/- 10 kV with dV/dt= slope: 250 V/ns
- f= 10 kHz with variable duty cycle (t_{Pulse})

One MD per slope (rising slope - RS, falling slope - FS)

- Total current consisting of displacement and discharge current
- Higher power dissipation than in sinusoidal operated DBDs of same voltage amplitude (2 nC instead of 0.7 nC)

M. Kettlitz, H. Höft, T. Hoder, S. Reuter, K.-D. Weltmann, R. Brandenburg JPD (2012) 245201

Equivalent circiut



$$j_R(t) = \frac{1}{1 - \frac{C_{cell}}{C_d}} \left[i(t) - C_{cell} \frac{dV(t)}{dt} \right]$$

$$C_{cell} = \frac{C_d C_g}{C_d + C_g}$$

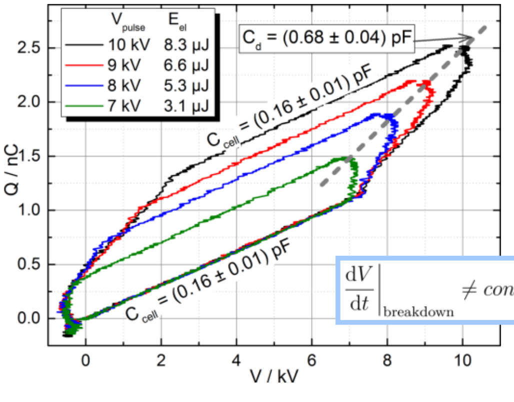
Applicability shown for:

- Large scale, sinusoidal driven DBDs
- Small scale, pulsed driven DBDs
- Micro-filament DBDs

Accuracy of C_{cell} and C_d crucial for description of instaneous characteristics

A. V. Pipa, T. Hoder, J. Koskulics et al. Rev. Sci. Instrum. 83, 115112 (2012) & CPP (2013)

Voltage-charge plots ("Lissajous figure")



$$\left.\frac{dV}{dt}\right|_{breakdown} \neq const. \quad \left(> 100\,\frac{V}{ns} \right)$$

Control of dissipated energy E_{dis} in pulsed MD by amplitude V_{pulse}

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Spatial structure & temporal development

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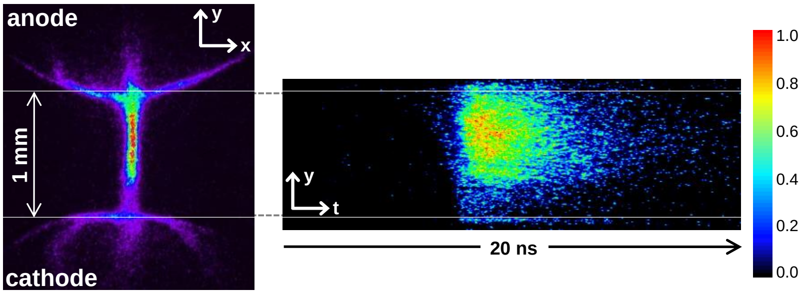
ICCD image | streak camera image



single MD | single MD

Fast optical recording on the sub-ns timescale even for individual events

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Reproducibility of MDs

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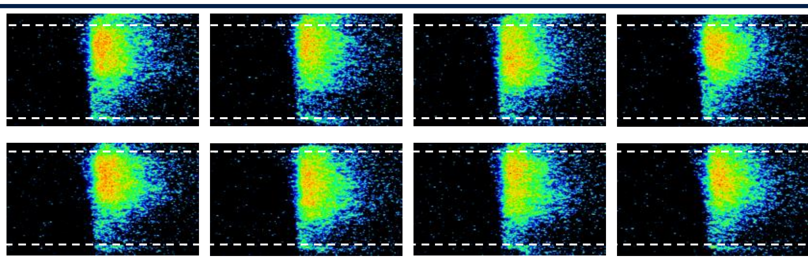
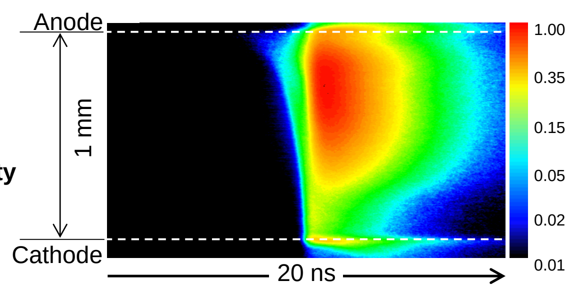


1,000 Microdischarges:

Good reproducibility



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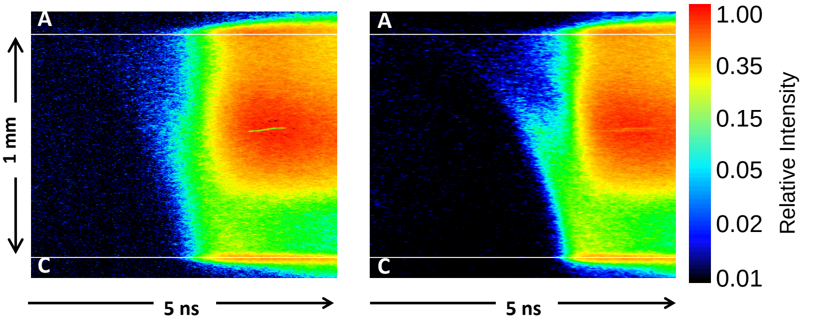
Jitter correction of streak photos

Uncorrected accumulation (1,000 MDs)

Jitter-corrected accumulation (1,000 MDs)



- temporal jitter in ns-range → jitter correction
- sum up of images/profiles shifted along time axis and overlaid with image processing function

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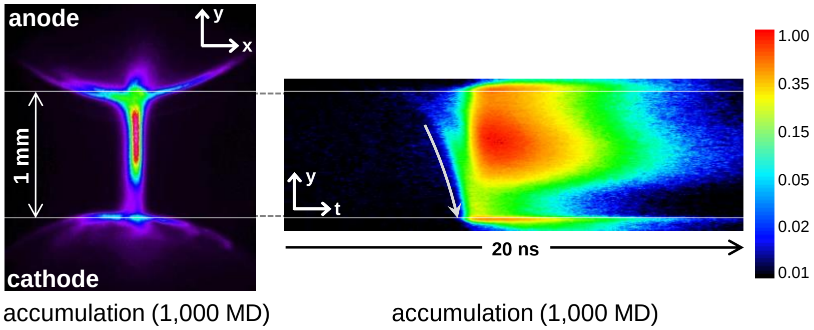
Spatial structure & temporal development

ICCD image

streak camera image



accumulation (1,000 MD)

accumulation (1,000 MD)

- ICCD image analysis:
  - → presence of volume and surface discharges
  - → filament radius r ≈ 50 µm
- Streak camera discharge characteristics:
  - → velocity of cathode directed ionisation front, streamer mechanism
  - → creation and decay of transient glow in the gap

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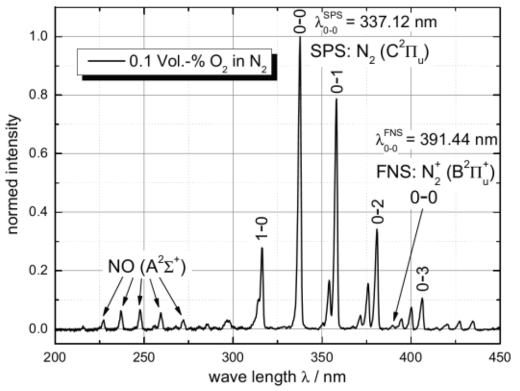
Typical optical emission spectrum for N_2/O_2



Molecular band radiation of nitrogen:

2nd positive system (**SPS**, 11 eV) $N_2(C^3\Pi_u)_{\upsilon'=0} \xrightarrow{A_{00}^C} N_2(B^3\Pi_g)_{\upsilon''=0} + h\nu$

1st negative system (**FNS**, 19 eV) $N_2^+(B^2\Sigma_u^+)_{\upsilon'=0} \xrightarrow{A_{00}^B} N_2^+(X^2\Sigma_g^+)_{\upsilon''=0} + h\nu$

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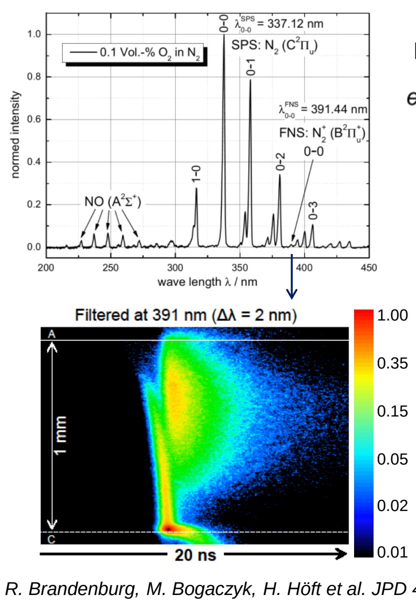
Spectrally selected streak image



FNS:

$e + N_2(X^1\Sigma_g^+)_{\upsilon=0} \xrightarrow{19eV} N_2^+(B^2\Sigma_u^+)_{\upsilon'=0} + 2e$

High electric field

Propagation of
cathode directed streamer
and
bulk plasma formation

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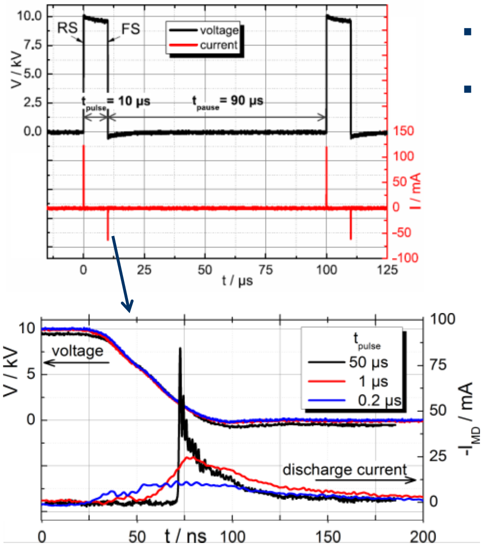
Power dissipation in falling slope MDs



- Asymmetric pulsing by variation of t_{pulse}
- Different delay times between MDs at rising and falling slope

Maximum and FWHM of current pulses depend on voltage waveform

M. Kettlitz, H. Höft, T. Hoder, K.-D. Weltmann, R. Brandenburg PSST 22 (2013) 025003

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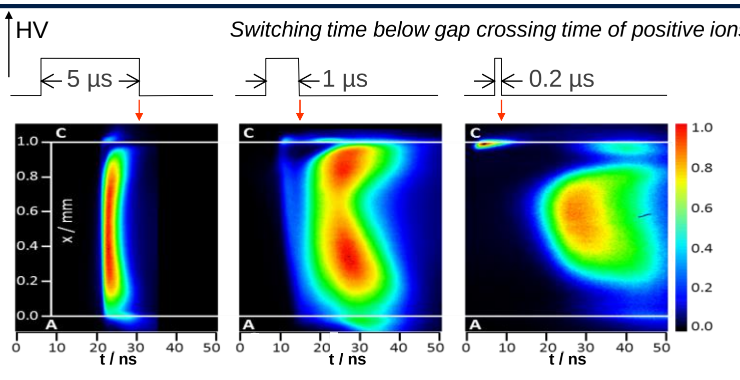
Manipulating volume breakdown

Switching time below gap crossing time of positive ions



Cathode directed streamer and bulk plasma

Transition

No streamer propagation phase

T. Hoder, H. Höft, M. Kettlitz, K.-D. Weltmann, R. Brandenburg Phys. Plas. (2012) 070701

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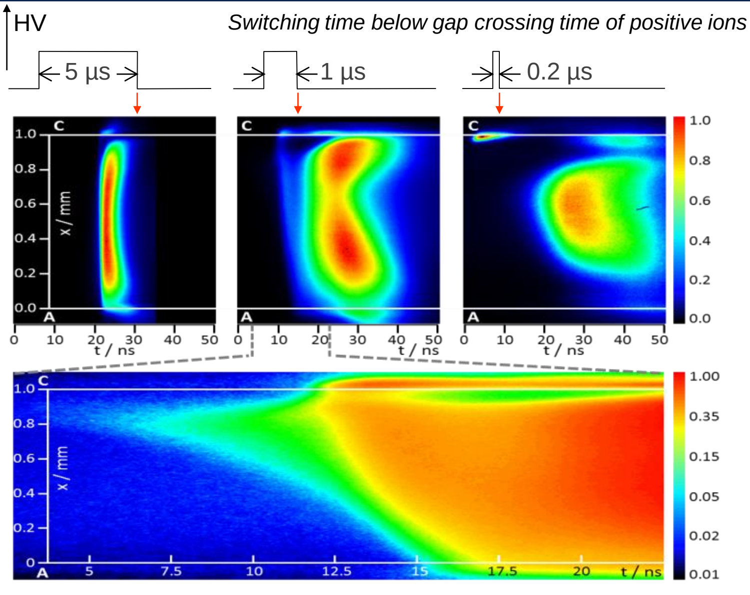
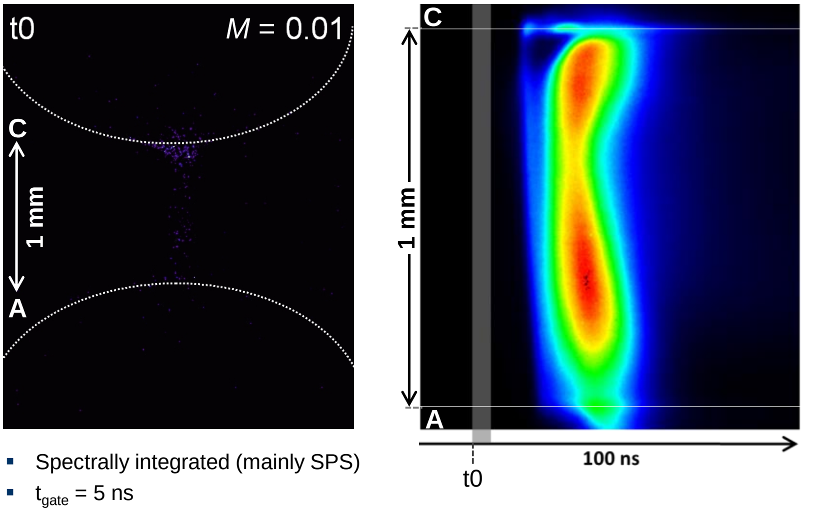
Manipulating volume breakdown

Switching time below gap crossing time of positive ions

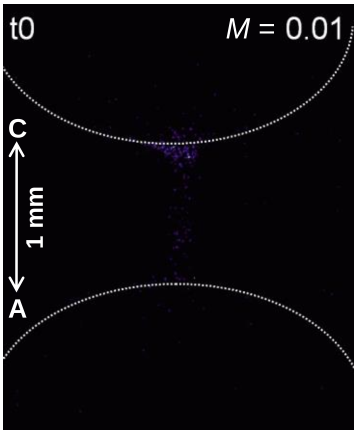


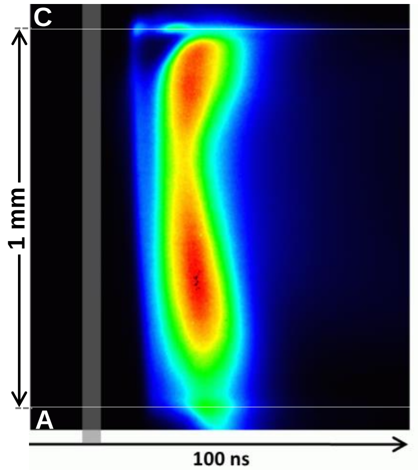
Development for t_{pulse}= 1 µs



- Spectrally integrated (mainly SPS)
- t_{gate} = 5 ns
- 100 accumulations
- Normalised intensity, scaling factor $M = I / I_{max}$

Development for t_{pulse}= 1 µs





- Pre-ionzation changes pre-phase and discharge development
- Short cathode directed streamer **and** anode directed streamer
- Bulk plasma with two maxima

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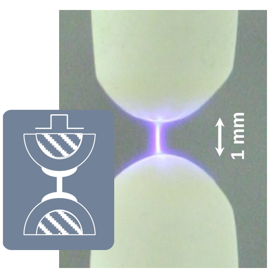
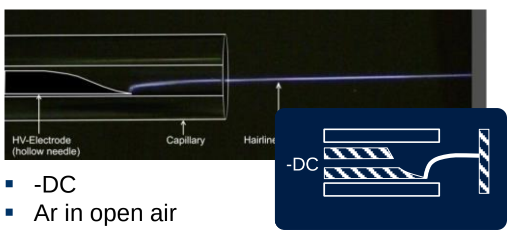
Plasma sources

Single filament volume DBD



- Pulsed operated (10 kHz)
- N_2 with 0.1 vol.% O_2

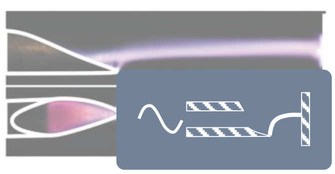
Self-pulsing transient spark



- -DC
- Ar in open air

Needle-to-plane discharge

- Sinusoidal (18 kHz)
- He ($+O_2$) in open air



Transient plasmas in „slightly reactive“ gas mixtures

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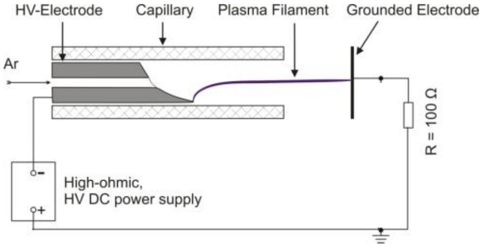
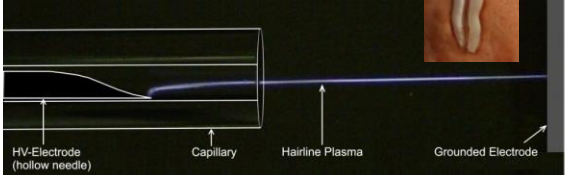
Self-pulsing transient spark



U	-5 … -10 kV
Q_{Ar}	200 … 500 sccm
Gases	Argon in air
Duration	10 ns
Peak current	0.2 … 2.3 A

$r_{Filament}$ = 30 µm



T. Gerling, T. Hoder, R. Bussiahn, R. Brandenburg, K.-D. Weltmann PSST 22 (2013) 065012

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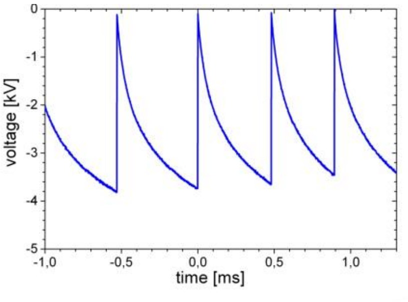
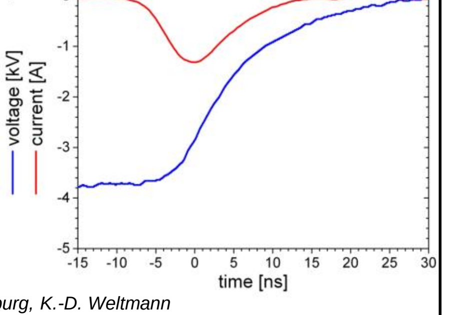
Electrical behaviour



Subsequent current pulses with
- I_{max} = 0.2 – 2.3 A
- FWHM = 9.8 ns
- f = 0.5 – 3 kHz (20% jitter)
- P_{mean} = 0.1 – 0.5 W

↓ TC-SPC

Electrical parameters determined by operation parameters Q and position of capillary relative to needle



T. Gerling, T. Hoder, R. Bussiahn, R. Brandenburg, K.-D. Weltmann JPD 46 (2013) 145205; PSST 22 (2013) 065012

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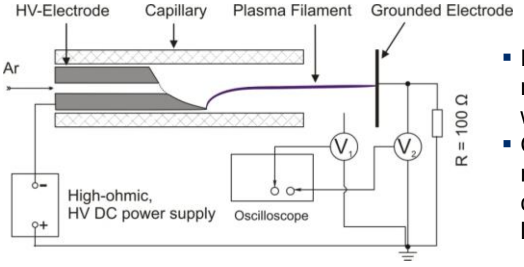
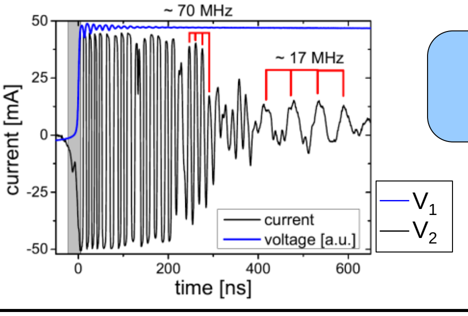
Electrical measurements (1)

With voltage probes



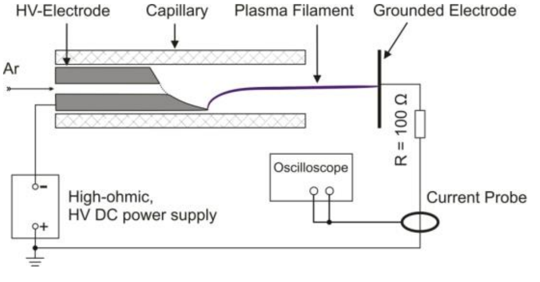
- Each voltage probe measurement causes weak oscillations
- Oscillation frequencies mainly influenced by cable lengths/ground loops

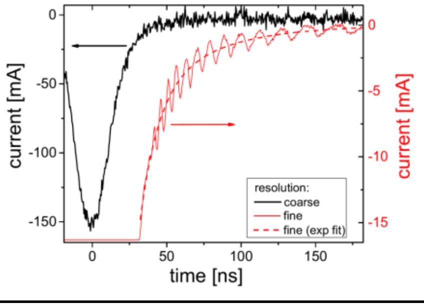


Alternative setup for sensitive signal measurement necessary

Electrical measurements (2)

With current probes





Frequencies invariant to cable length

Identification of ion acoustic waves

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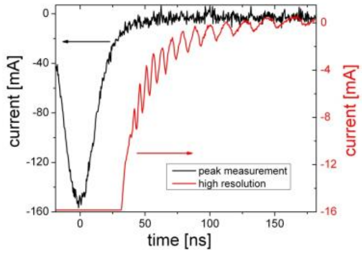
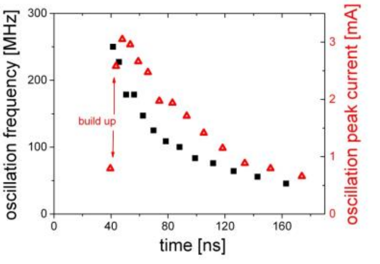
-DC





- Every 10th or 15th discharge if Q= 350 … 500 sccm
- Why ?: → Fast current pulses (10 ns)
  → Rapid change of discharge conditions

→ (x,t) - oscillation of Ar_2^+ ion density in a plasma

$$f_{Ar_2^+} = \frac{1}{2\pi}\sqrt{\frac{e^2 n_{Ar_2^+}}{\varepsilon_0 m_{Ar_2^+}}}$$

Measured molecular argon ion density
$n_{Ar_2^+} = 1.10 * 10^{14} \ldots 4 * 10^{12}\ cm^{-3}$

T. Gerling, R. Bussiahn, C. Wilke, K.-D. Weltmann EPL 105 (2014) 25001

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Scheme of spatio-temporal development (CCS)

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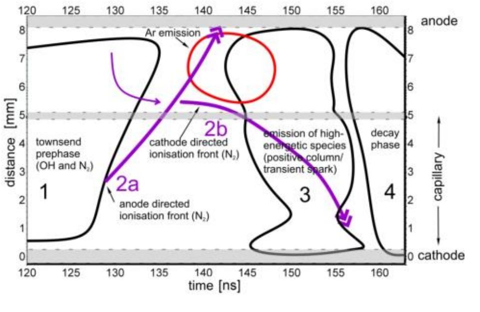
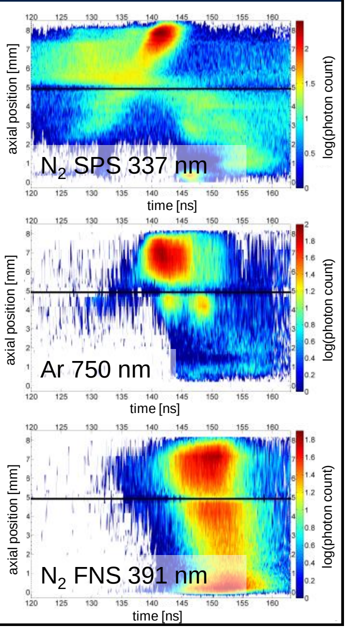
-DC

Development in four phases

1. Pre-phase
2. Phase of ionising fronts
   a. Anode directed
   b. Cathode directed
3. Transient spark
4. Afterglow

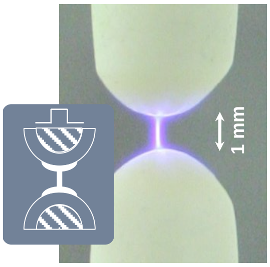




T. Gerling et al.; PSST 22 (2013) 065012

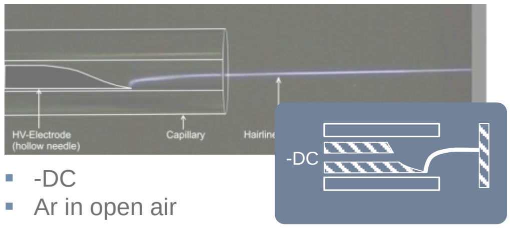
Plasma sources

Single filament volume DBD



- Pulsed operated (10 kHz)
- N_2 with 0.1 vol.% O_2

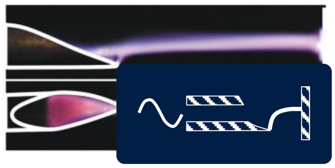
Self-pulsing transient spark



- -DC
- Ar in open air

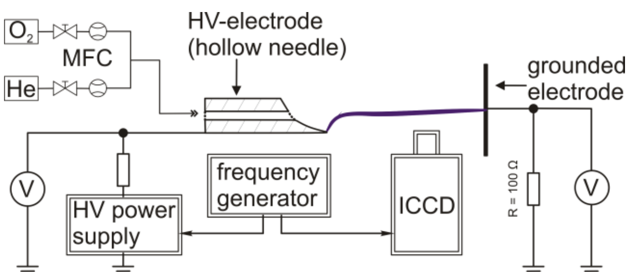
Needle-to-plane discharge

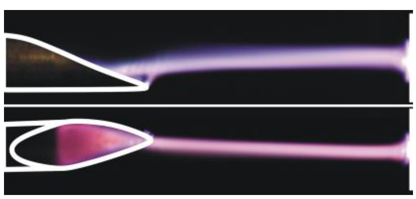
- Sinusoidal (18 kHz)
- He (+O_2) in open air



Transient plasmas in “slightly reactive” gas mixtures

Needle-to-plane discharge

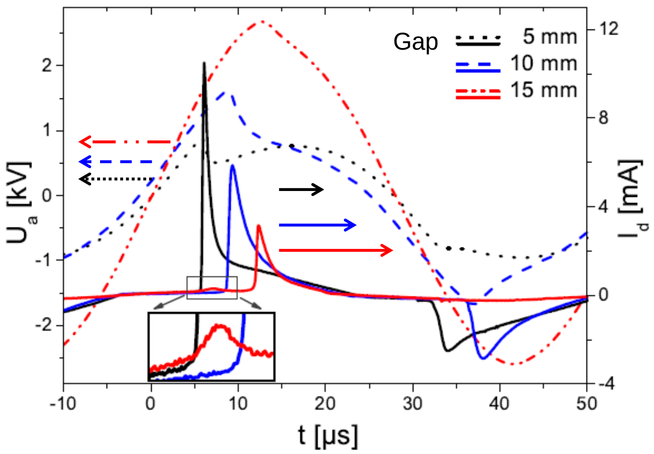




U	2 … 5 kV_{pp}
Q_{He}	3 slm
Gases	Helium in air
Duration	1 µs
Peak current	3 … 10 mA

T. Gerling A.V. Nastuta, R. Bussiahn, E. Kindel, K.-D. Weltmann PSST 21 (2012) 034012

Electrical behaviour



- Main characteristics allow discussion of discharge inception
- Pre-pulse for 15 mm gap distance (needle-to-plane)

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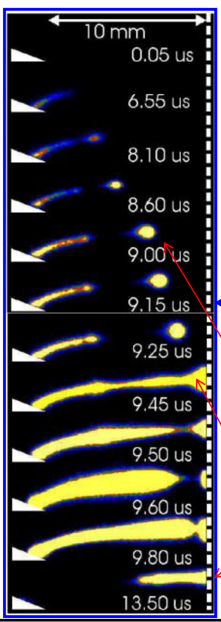
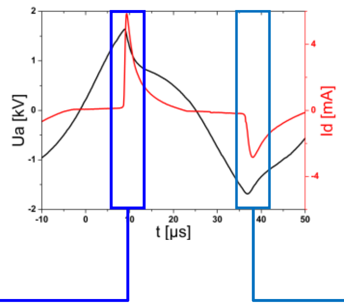
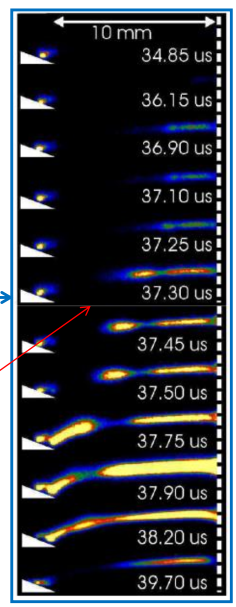
Phase resolved ICCD-imaging







Four types of Bullets observed

(a) Forward bullet (positive guided streamer)

(b) Reverse bullet (against gas flow)

(c) “Return stroke” (faster electron cloud)

(d) Backward bullet (negative guided streamer)

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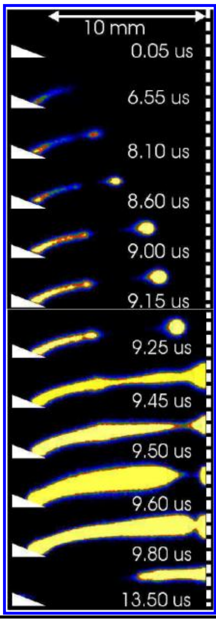
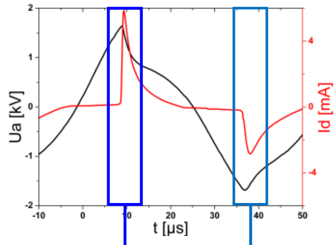
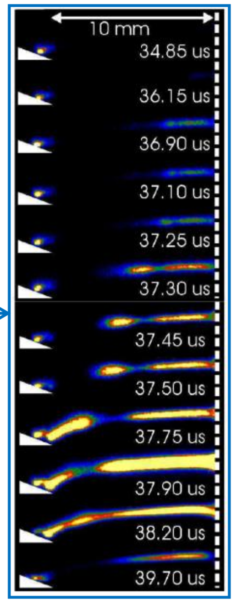
Phase resolved ICCD-imaging







- Positive and negative voltage cycles similar
- Complex development with propagation in and against external electrical field and flow direction

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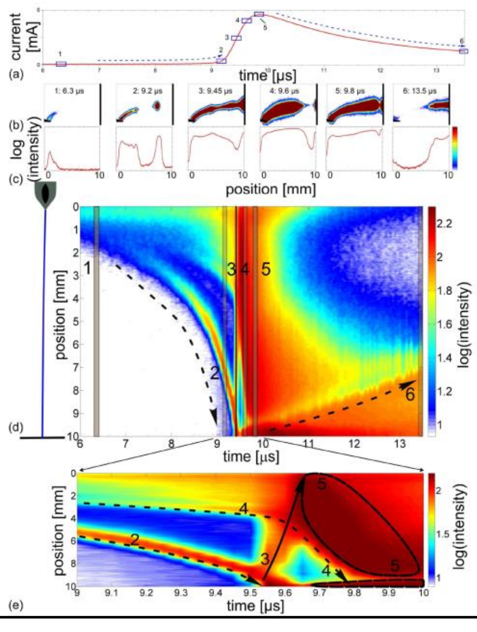
Contour plot from phase resolved ICCD



"3D" – representation of ~150 images in one diagram

Development divided in six phases

1. Townsend pre-discharge
2. First forward bullet
3. First backward bullet ("return stroke")
4. Second forward bullet
5. Atmospheric pressure glow discharge
6. Second backward bullet

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Outline

1 Atmospheric Pressure Plasmas Sources
- Principles and overview
- Need and concepts for diagnostics

2 Fast diagnostics
- Electrical diagnostics
- Optical and spectroscopic diagnostics

3 Discussion of examples:
Transient plasmas in „slightly reactive“ gas mixtures
- Single filament Dielectric Barrier Discharge
- Transient spark discharge
- Needle-to-plane discharge

4 Summary and outlook

Summary

Reliable plasma sources provide profound diagnostics
- Discharge development
- Basic plasma parameters
- Benchmark of modelling
- Estimation of plasma treatment effects

Examples given:
- combined use of electrical, optical and spectroscopic diagnostics on plasma filaments/microdischarges
- Streamer mechanism after pre-phase
- Power dissipation and discharge inception determined by memory effects

On the way to more reliable and controllable plasma sources such activities must be increased in connection with the application of other diagnostics and simulation giving further access to basic plasma parameters.

Outlook

- Systematic measurements at conditions as realistic as possible (regarding gas mixtures, materials etc.)
- Combination with surface diagnostics and laser diagnostics
- Correlation between operation parameters and plasma properties
- Determination of fluxes
- Combination with simulation ("mutual benchmarking") including plasma chemistry

Application/indication oriented development of plasma sources

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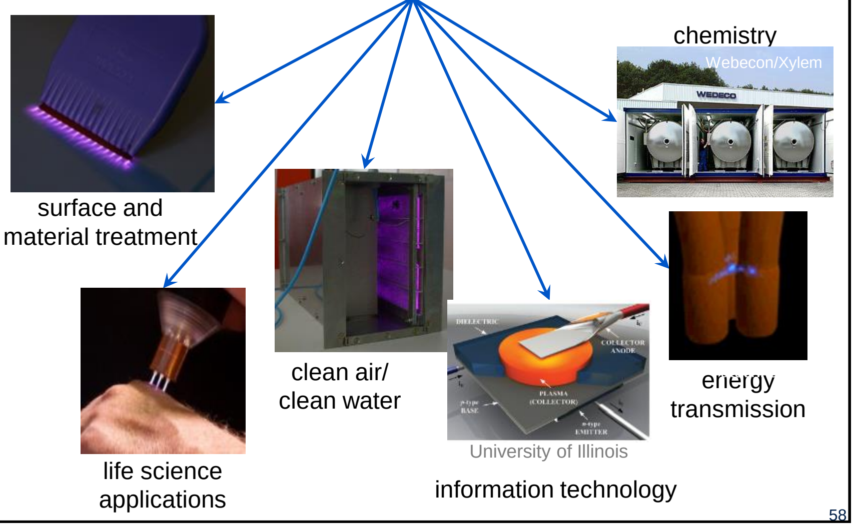
Outlook

Application/indication oriented development of plasma sources



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CAMPUS PlasmaMed

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