# Microstructured Electrode Arrays: Optical analysis of the Glow Discharge in a Magnified Electrode Gap

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The characterisation of a discharge requires the knowledge about few parameters. The most important are the electron density, the debye-length  $\lambda_d$  and the voltage/current characteristic. Normally the electron density is obtained using a Langmuir Probe. This method delivers reliable results if the influence on the electric field can be neglected. In the analysis of microdischarges, this assumption is not fulfilled. J. Park et al. (J. Appl. Phys. **2001**, 89, 15), developed a model which allows to calculate the electron density using the plasma current, the area of the discharge and the electric field strength. The debye-length can be estimated using the dimensions of the dark space of the discharge. With this information it is possible to calculate the electron temperature. To measure the dimension of the dark space, it is crucial to take a close look at the discharge. We used an ocular of a microscope (40× magnification) to project an image of one electrode gap onto the sensor of an ICCD-Camera. The electrical parameters were obtained using an ENI V/I-Probe<sup>®</sup>. The experiments were conducted in helium and argon at atmospheric pressure. In order to cover the whole glow-discharge regime, the applied power was raised in steps of 1 W from the point of ignition to the glow-to-arc transition. Electron densities and temperatures as well as the debye-length could be obtained.

## Introduction

Over the last years, the generation of nonthermal gas discharges at atmospheric pressure has been investigated by many research groups. Massines et al.,<sup>[1,2]</sup> Okazaki et al.,<sup>[3,4]</sup> Trunec et al.<sup>[5]</sup> and Roth and co-workers<sup>[6]</sup> successfully generated atmospheric pressure glow discharges

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Institut für Mikrobiologie, Technische Universität Braunschweig, Spielmannstr. 7, D-38106 Braunschweig, Germany with a dielectric barrier array, and Selwyn and coworkers<sup>[7,8]</sup> developed an atmospheric pressure plasma jet producing a stable and homogenous plasma. There are two approaches based on the Paschen similarity law (pd = const.), which scale down the electrode dimensions to the µm range in order to ignite discharges at atmospheric pressure at moderate voltages working in the Paschen minima of the different gases. Schoenbach and co-workers,<sup>[9,10]</sup> Schmidt-Böcking and co-workers<sup>[11]</sup> and Eden et al.<sup>[12,13]</sup> use a microhollow cathode array. Our group introduced microstructured electrode arrays (MSE) consisting of an interlocked comb-like electrode system with  $\mu m$  gap widths.<sup>[14-17]</sup> In the last years, the MSE proved to be a versatile tool for various applications. We successfully tested their usage in surface modification experiments (coating and sterilisation techniques), waste-gas treatment (decomposition of CF4 and NO) and recorded the U/I-characteristic earlier.[18,19]





Figure 1. Geometry of the electrode gap (dimensions in μm).

However, the characterisation of the generated discharge was a hard task. Due to the very small dimensions of the MSE, well-known methods as the Langmuir Probe were not applicable. Therefore, it was not possible to measure important parameters such as the electron density or the electron temperature, which both affect the debye-length. The measurement of the electron density via line-shape analysis (Stark Broadening) failed as well. The debye-length can be approximated if one takes a close look at the discharge. The length of the dark space is a good yardstick for  $\lambda_d$ . The relation is valid for weakly ionised and nonmagnetised plasmas. It does not deliver exact values, but a lower limit for the electron temperature. Park et al. suggested that the electrondensity can be calculated using the plasmacurrent and the plasmacovered area.<sup>[10]</sup>

An ocular of a microscope was used to project a magnified image of the electrode gap onto the sensor of an ICCD-Camera. This simple setup enabled us to measure the thickness of the plasma's dark zone and thereby determine the debye-length. Simultaneously, the plasma current was recorded. With this data, electron densities and temperatures in helium and argon were calculated for a wide power range.

### **Experimental Part**

#### **Plasma Reactor**

Main part of the experiment is the vacuum chamber, containing the MSE and the magnification optics. The used structures were described in detail before.<sup>[20,21]</sup>

The geometrical design of the electrode gap is shown in Figure 1. In order to withstand the thermal stress at high pressures, a material thickness of 100  $\mu$ m for the electrodes is necessary.

A Chromel Alumel thermocouple of K-type is directly placed under the plasma source measuring the temperature rise of the MSE during the plasma operation. A gas flow rate of 100 sccm was setup by means of mass flow controllers. The RF-power was generated using an RF power supply at 13.56 MHz (ENI, ACG-3B) equipped with a matching network (ENI, MW-5D). Between the matching network and the plasma source a special probe (ENI, VI-Probe) was inserted in order to measure voltage, current and phase angle of the system.

#### Detection

An intensified CCD-Camera system (LaVision Flamestar 2F) is used to record the images. The image intensifier can be gated with a minimum gate-width of 5 ns, therefore, time resolved measurements in relation to the RF are possible.

This setup can be amended with a monochromator (ISA Triax Serie 320) for optical emission spectroscopy (OES).

#### Measurements

The plasma chamber was evacuated to a pressure of  $10^{-3}$  mbar. Then it was filled with 1 000 mbar of the noble gas. A constant gas flow of 100 sccm was established. The applied power was raised in 1 W steps till ignition. Now the parameters of the camera system were optimised (gain, gate-width). A gain of 900 V together with a gate-width of 1 ms (13 500 periods) delivered the best results. At the same time voltage and current were recorded. After one image was taken the power was raised by 1 W. This was repeated until the discharge turned into a thermal arc. The debye-length was obtained from the recorded images by comparing the electrode gap width (70 µm) with the dark space using Adobe Photoshop. Figure 2 shows images of the discharge in helium and argon.

With this information it is possible to calculate the electron density and temperature.

First of all the electron density is calculated with the following equation:

$$\frac{I_{\rm P}}{A_{\rm p}} = n_{\rm e} e u_{\rm e} E \tag{1}$$

 $I_{\rm P}$  is the plasma current,  $A_{\rm P}$  the plasma-covered area,  $n_{\rm e}$  the electron density,  $u_{\rm e}$  the electron mobility and *E* the electric field-strength.

*Figure 2*. Images of the discharge in helium (left) and argon (right) at 1000 mbar.

um

70 um

4 μm

	Heliu	ım		Argon					
Power	n <sub>e</sub>	$\lambda_D$	T <sub>e</sub>	Power	n <sub>e</sub>	$\lambda_D$	$T_{\rm e}$		
w	m <sup>-3</sup>	(×10 <sup>-6</sup> ) m	eV	w	m <sup>-3</sup>	(×10 <sup>-6</sup> ) m	eV		
14	$1.59 \times 10^{18}$	8.7	2.2	27	$1.28 \times 10^{18}$	8.1	1.5		
15	$1.59 \times 10^{18}$	8.4	2.0	28	$1.26 \times 10^{18}$	1.3	3.7		
16	$1.63 \times 10^{18}$	12.0	3.9	29	$1.14 \times 10^{18}$	9.7	1.9		
17	$1.64 \times 10^{18}$	4.7	0.6	30	$1.50 \times 10^{18}$	9.2	2.3		
18	$1.68 \times 10^{18}$	3.7	0.4	31	$1.32 \times 10^{18}$	8.6	1.76		
19	$1.70 \times 10^{18}$	2.6	0.2	32	$1.60 \times 10^{18}$	1.0	2.92		
20	$1.76 \times 10^{18}$	6.7	1.4	33	$1.61 \times 10^{18}$	1.1	3.66		
21	$1.75 \times 10^{18}$	5.2	0.9	34	$1.60 \times 10^{18}$	9.6	2.66		
22	$1.83 \times 10^{18}$	3.7	0.4	35	$1.60 \times 10^{18}$	7.0	1.44		
23	$1.81 \times 10^{18}$	4.99	0.8	36	$1.59 \times 10^{18}$	6.8	1.32		
24	$1.85 \times 10^{18}$	8.2	2.2						
25	$1.83 \times 10^{18}$	11.0	4.1						

*Table 1.* Collected data for argon and helium at 1000 mbar.

The combination of the measured debye-length and the calculated electron density delivers the electron temperature

$$T_{\rm e} = \frac{\lambda_{\rm D}^2 e^2 n_{\rm e}}{\varepsilon_0 k} \tag{2}$$

Table 1 shows the results in helium and argon, Table 2 and 3 summarise the findings.

#### Discussion

During the experiments it was obvious that measurements at the extreme ends of the glow-discharge-regime are not reliable. At low power (directly after ignition) the discharge is not homogeneous and the discharge does not cover the whole length of the electrode gap. Therefore, the plasma-covered area cannot be determined exactly. Because of this problem the first data point in argon (26 W) was not taken into consideration. When the power is raised, the plasma fills the whole electrode gap and the

Table 2. Electron density, temperature and energy range in helium.

experiment delivers a good result (in argon between 27 and 36 W). If the power is raised beyond 36 W, another problem occurs. The plasma area extends, the discharge no longer stays inside the electrode gap but starts to cover the electrode surface. Therefore, the plasma area is not known. When the power exceeds 39 W the abnormal-glow-regime is reached, between 40 and 41 W the glow-to-arc transition takes place. So the experimental data for applied powers over 36 W were discarded.

The results prove that the MSE is capable of generating a glow discharge at atmospheric pressure. The delivered electron densities and electron temperatures, as well as the debye-length are typical for a nonthermal glow-discharge, even if the electron density is relatively low.

The relatively low electron density is also a good explanation why the determination of the electron density via Stark-Broadening failed. Dong et al.<sup>[22]</sup> showed, that this method is only applicable to discharges with an electron-density of about  $10^{21}$  m<sup>-3</sup>. If the electron density is significantly lower (like in our discharge), the doppler effect is much stronger.

*Table 3*. Electron density, temperature and energy range in argon.

Helium				Argon				
	Min	Max	Average		Min	Мах	Average	
$n_{\rm e}  ({\rm m}^{-3})$	1.59	1.85	$1.72 \pm 0.096$	$n_{\rm e}~({\rm m}^{-3})$	1.14	1.61	$1.45 \pm 0.168$	
$T_{\rm e}$ (eV)	0.2	4.1	$1.6 \pm 1.32$	$T_{\rm e}$ (eV)	1.32	3.66	$2.3 \pm 0.889$	
<i>T</i> <sub>e</sub> (K)	1547	31720	$12000\pm10282$	$T_{\rm e}$ (K)	10 212	28 625	$18000\pm 6877$	

## Conclusion

The applied method proved to be a promising alternative to measure important parameters of microdischarges where common methods fail because of the small dimensions of the plasma source. The experimental setup is simple and inexpensive. The electron density could be measured with a maximum uncertainty of 5% in helium and 12% in argon. The determination of the electron-n-temperature showed a much wider range of variation (83% in helium, 38% in argon). Compared to the range of electron temperatures that were discussed for glow-discharges ( $10^4$ – $10^5$  K), the obtained results allow a good characterisation of the examined discharge.

There are two big advantages of the applied method. There is no intrusion into the discharge and therefore no influence on the electric field and the relevant parameters are very easy to obtain. Furthermore, no assumptions ignoring some aspects of the plasma (e.g. the local equilibrium that is discussed if the electron temperature is measured via OES) have to be made.

In order to minimise the uncertainty of this method it is crucial to improve the optical system. A camera with a higher sensitivity would allow to record a single period of the RF. This would make it much easier to determine the size of the dark space. A higher 'per-pixel-resolution' would be a huge step forward as well. This could be achieved with a CCD-sensor with a smaller pixel pitch, or — much easier and cheaper — with a higher magnification of the microscope optic.

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 F. Massines, A. Rabehi, P. Decomps, R. Ben Gadri, P. Ségur, C. Mayoux, J. Appl. Phys. 1998, 83, 2950.

- [2] F. Massines, P. Ségur, N. Gherardi, C. Khamphan, A. Ricard, Surf. Coat. Technol. 2003, 174–175, 8.
- [3] S. Okazaki, M. Kogoma, M. Uehara, Y. Kimura, J. Phys. D: Appl. Phys. 1993, 26, 889.
- [4] T. Yokoyama, M. Kogoma, T. Moriwaki, S. Okazaki, J. Phys. D: Appl. Phys. 1990, 23, 1125.
- [5] D. Trunec, A. Brablec, J. Buchte, J. Phys. D: Appl. Phys. 2001, 34, 1697.
- [6] K. Kelly-Wintenberg, A. Hodge, T. C. Montie, L. Deleanu, D. Shermann, J. Reece Roth, P. Tsai, L. Wadsworth, J. Vac. Sci. Technol. 1999, 17, 1539.
- [7] J. Park, I. Henins, H. W. Hermann, G. S. Selwyn, J. Appl. Phys. 2001, 89, 15.
- [8] J. Park, I. Henins, H. W. Hermann, G. S. Selwyn, J. Appl. Phys. 2001, 89, 20.
- [9] R. H. Stark, K. H. Schoenbach, J. Appl. Phys. 1999, 85, 2075.
- [10] R. H. Stark, K. H. Schoenbach, Appl. Phys. Lett. 1999, 74, 3770.
- [11] C. Penache, A. Bräuning-Demian, L. Spielberger, H. Schmidt-Böcking, Proceedings of the Seventh International Symposium on High Pressure Low Temperature Plasma Chemistry (HAKONE VII), Greifswald 2000, 501.
- [12] J. G. Eden, S.-J. Park, N. P. Ostrom, S. T. McCain, C. J. Wagner, B. A. Vojak, J. Chen, C. Liu, P. von Allmen, F. Zenhausern, D. J. Sadler, C. Jensen, D. L. Wilcox, J. J. Ewing, J. Phys. D: Appl. Phys. 2003, 36, 2869.
- [13] S.-J. Park, C. J. Wagner, C. M. Herring, J. G. Eden, Appl. Phys. Lett. 2000, 77, 199.
- [14] C. Geßner, P. Scheffler, K. H. Gericke, Proceedings of the Seventh International Symposium on High Pressure Low Temperature Plasma Chemistry (HAKONE VII), Greifswald 2000, 112.
- [15] C Geßner, P. Scheffler, K.-H. Gericke, Proceedings of the International Conference on Phenomena in Ionized Gases (XXV ICPIG), Nagoya 4 2001, 151.
- [16] H. Schlemm, D. Roth, Surf. Coat. Technol. 2001, 142–144, 272.
- [17] K.-H. Gericke, C. Geßner, P. Scheffler, Vacuum 2002, 65, 291.
- [18] L. Baars-Hibbe, P. Sichler, C. Schrader, C. Geßner, K.-H. Gericke, S. Büttgenbach, Surf. Coat. Technol. 2003, 174–175, 503.
- [19] C. Schrader, L. Baars-Hibbe, P. Sichler, T. Cordes, K.-H. Gericke, S. Büttgenbach, *Proceedings of CHEMREACTOR* 2003, 16, 371–374.
- [20] L. Baars-Hibbe, C. Schrader, P. Sichler, T. Cordes, K.-H. Gericke, S. Büttgenbach, S. Draeger, *Vacuum* 2004, 73, 327.
- [21] L. Baars-Hibbe, P. Sichler, C. Schrader, K.-H. Gericke, S. Büttgenbach, Plasma Process Polym. 2005, 2, 174–182.
- [22] L. Dong, J. Ran, Z. Mao Appl. Phys. Lett. 2005, 86, 161501.