RF driven Microhollow Cathode Discharge for Atomic Emission Spectrometry

R. Heming*, S. Tombrink*, M. Miclea**, M. Gruber***, J. Franzke*

*ISAS - Institute for Analytical Sciences; **Martin-Luther-Universität Halle-Wittenberg; ***Juniorprofessur für Optische Mikrosysteme OMS, FernUniversität in Hagen

mailto: heming@isas.de

The generation of a stable plasma in a microhollow cathode discharge structure powered by a RF generator is presented. The device is used for the detection of chlorine and fluorine in gas mixtures by means of atomic emission spectroscopy. Detection limits for CCl_2F_2 in He are 20 ppb, using either Cl 912.114 nm or F 739.868 nm. The RF plasma is compared to a plasma driven by a DC source.

1 Introduction

In the last few years, there is a strong interest in the development of miniaturized analytical measurement systems. Such systems, called “Lab on a Chip”, have their application fields in biotechnology, biomedicine and process analysis. Their main advantages are low power consumption, low demand of sample volume, fast and parallel detection and the fact that the analytical system can be brought to the sample instead of bringing the sample to the laboratory. The microstructure technology allows the integration of all necessary steps for sample preparation and separation on a chip.

On those chips, miniaturized plasmas in combination with spectrometers can be used as tools for the detection of molecular fragments and even for elemental analysis by atomic emission spectroscopy (AES). One of the main advantages of miniaturized plasmas is the benefit that the use of vacuum pumps is not required.

There are different ways to integrate plasmas in chips. One of them is a microhollow cathode discharge (MHCD). This plasma type, driven by a DC source has been demonstrated as a sensitive detector system before [2]. In this work, the MHCD structure is powered by low power RF source. RF driven MHCDs exhibit a longer endurance in comparison to DC excitation.

2 Setup

The MHCD setup is a multilayer system consisting of two metallic layers (Pt, 20 to 100 µm thickness) separated by a ceramic insulator (Al_2O_3, 170 to 390 µm thickness). A hole with a diameter of 100 to 300 µm is drilled through this multilayer structure.

The metallic layers are connected to a RF generator based on a low power design utilizing common high-speed CMOS logic devices. In the generator a bridged push-pull circuit is operating in D-mode to take full advantage of low power dissipation. A resonance tank is formed of the multilayer system and a matching network to provide up to 900 V_{pp} for ignition and operation of the plasma. With an DC input voltage of 4 to 7 V the power supply for the generator can be performed by a standard battery.

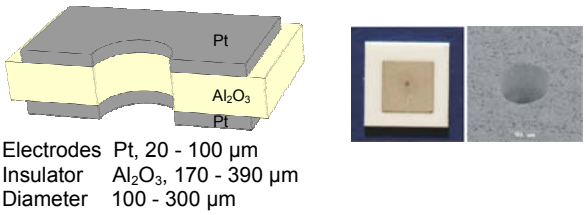

Fig. 1 *Layout, microstructure and SEM of the hole*

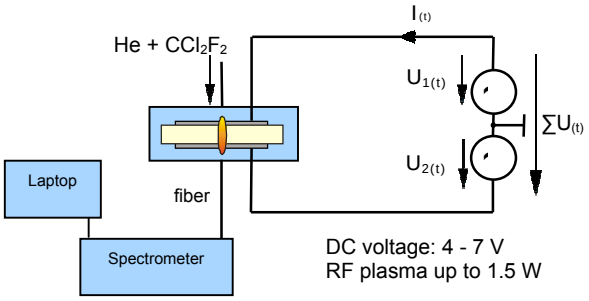

Fig. 2 *Experimental arrangement for AES*

An analyte gas is guided through a tube system into the chip which prevents a contamination with air. The discharge can be operated in a pressure range from 10 mbar to 2 bar. Emitted light is focused to a fused silica fiber (200 µm core diameter) connected to an Ocean Optics spectrometer (USB2000, sensitive from 350 to 1000 nm).

The signals are transmitted to a laptop and visualized on the screen.

3 Power measurement

In order to compare DC and RF plasma sources we want to predict the precise amount of RF plasma power. Two approaches are presented to estimate the plasma power.

The first approach is to measure the DC power supply of the entire amplifier. The transition from plasma off to plasma on is observed (1). The power is increased to the maximum (2) and decreased until the plasma is off again (3). At this point the plasma power is set to zero.

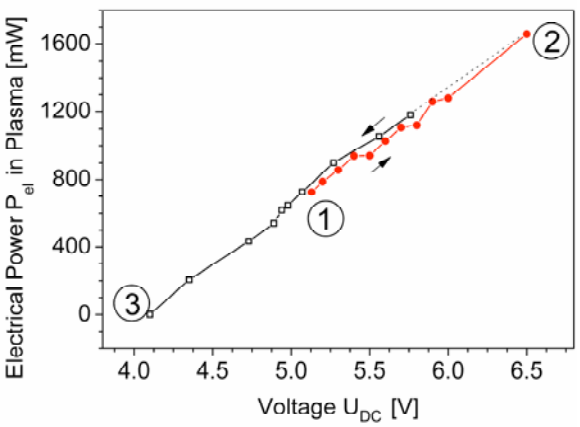

Fig. 3 *Input voltage and power demand of RF generator*

The second approach is to measure the plasma voltage and current with an oscilloscope

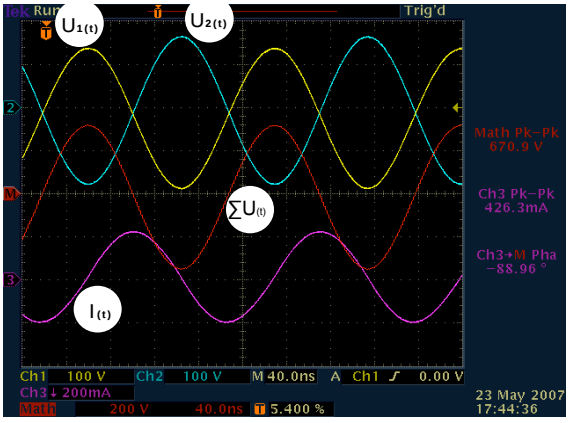

Fig. 4 *Voltage, current and power measured by scope*

and to calculate the power by [1]

$$P = \frac{1}{T}\int_{-T/2}^{+T/2} I(t)\cdot\sum U(t)\cdot dt$$

4 Detection of analytes

The RF amplifier is capable of igniting a stable plasma in a wide pressure range. It can operate in noble gases, gas mixtures and air. In this work, it is used for the detection of chlorine (Cl) and fluorine (F) in freon (CCl_2F_2) mixed with helium (He). All spectra were measured with 100 ms integration time of the spectrometer.

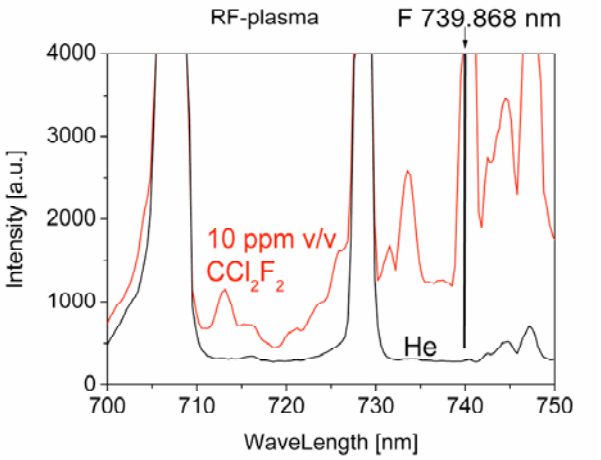

Fig. 5 *Spectra of He and CCl_2F_2 in He driven by RF*

In figure 5 the detection of 10 ppm v/v CCl_2F_2 using a plasma driven by a RF source is demonstrated. The detection limit is at 20 ppb v/v using either Cl 912.114 nm or F 739.868 nm.

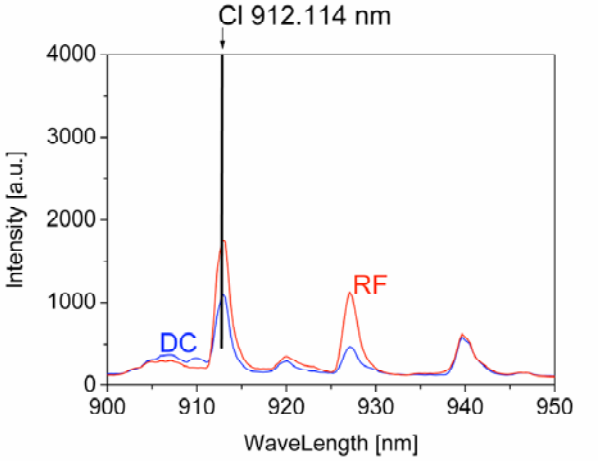

Fig. 6 *Comparison of spectra of plasmas driven by RF and by DC source*

In figure 6 the comparison of a DC and a RF plasma source at P = 1 W is shown for the wavelength region from 900 to 950 nm. At 912.114 nm the RF source shows a slightly stronger excitation of chlorine atoms than the DC source. Other analyte lines show opposite behaviour. The RF plasma source has the potential to compete with standard DC plasma sources and is able to improve the performance of MHCD.

Future investigations will include the design of an optimised RF transformer and detailed analysis of sputter defects for RF and DC driven MHCD structures.

References

[1] P. Gerdsen, *Hochfrequenzmeßtechnik,* Teubner, 1982.

[2] M. Miclea, M. Okruss, K. Kunze, N. Ahlmann, J. Franzke, 2007, Microplasma-based atomic emission detectors for gas chromatography, *Anal. Bioanal. Chem.* **388**, 1565-1572.