Electrical Conductivity Measurements in Combination with Raman Microspectroscopy and Temperature Programmed Oxidation for Analysis of Microstructure and Reactivity of Soot

B. Grob[1], F. Knoller[1], N. P. Ivleva[1], R. Niessner[1]

[1]Chair for Analytical Chemistry, Technical University of Munich, Munich, 81377, Germany
Keywords: electrical conductivity, Raman microscopy, temperature programmed oxidation, soot reactivity, real-time detection
Presenting author email: benedikt.grob@mytum.de

Soot particles emitted by diesel engines are a well known problem especially in urban areas due to their influence on the human health but they can have a significant influence on the climate as well. Therefore the emission regulations for light and heavy duty vehicles prescribe a strict limit of diesel soot particle emission. This can only be accomplished by diesel particle filters (DPF). To control the back pressure of this filters a continuous or periodically gasification of the deposited particles is necessary. The efficiency of this regeneration step is mainly affected by the oxidation behaviour of the soot, which is directly connected to its microstructure.

To optimize the engine in the way that they produce highly reactive soot, tools are required to analyse soot samples regarding their oxidation behaviour. Currently, the temperature controlled oxidation of soot samples is used as a common method for engine optimization. But as we could show in early publications Raman microspectroscopy as well as the electrical conductivity measurement are a promising tool for soot microstructure characterization (Schmid 2011 & Grob 2012) Especially the conductometric measurement method is very attractive, because it is a simple, rapid and cheap tool compared to the other techniques. So far we could show in basic experiments the possibilities and the limitations of this method (Grob 2012).

Our approach is now to develop a device which makes it possible to investigate the influence of the microstructure and the oxidation behaviour of soot on the electrical conductivity combined with the monitoring of the processes by the Raman microspectroscopy (RM) and the temperature programmed oxidation in one setup. Therefore we developed a flowcell (Fig. 1) which can be heated up to 1273 K and can be placed under the Raman microscope, due to a good isolation and a effective cooling of the cell frame. Through a quartz glass window it is possible to observe the soot layer on the conductometric sensor (Fig. 2) inside the cell. On the outlet of the cell a FTIR can be plugged as a gas detector of the controlled oxidation. The soot particles are collected by a thermophoretic particle precipitator on the conductometric sensor surface, which consists of a oxidized silicon substrate with interdigitated AlSiCu electrodes on it.

The setup allows us to change the graphitization of a soot sample under controlled atmosphere. By cooling down the sample under protective gas atmosphere in the same device a contamination or further oxidation of the sample can be prohibited. In the second step the measurement of the electrical conductivty of the sample follows and can be correlated with the fingerprint spectra of the RM provided through the window of the cell.

So far the conductometric sensor principle is only used for on board particle detection to monitor the DPF in light duty vehicles. We hope that we are able to extend the use of the principle by this experminental setup for soot microstructure and reactivity analysis.

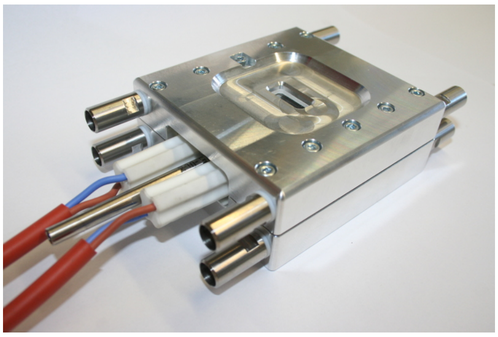

Figure 1: The heatable flow cell covered with a water cooled aluminum block.

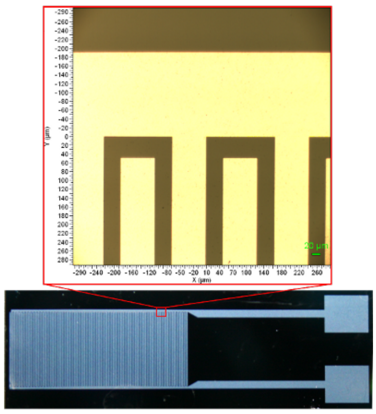

Figure 2: The interdigitated electrodes with a distance of 50 µm between the fingers.

Grob, B., Schmid, J., Ivleva, P. N., Niessner, R. (2012) *Anal. Chem.* **84**, 3586–3592.

Schmid, J., Grob, B., Niessner, R., Ivleva, N. P. (2011) *Anal. Chem.* **83**, 1173–1179.