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Soot sensors for a healthy environment (SootSens)

- Nanosized soot particles are a serious health hazard in urban air
- Legislation continuously reduces allowable emission levels in diesel powered vehicles
- Good results from a new sensor that enhances soot deposition of submicron particles in diesel exhaust

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Abstract:
Nanosized soot particles are a serious health hazard in urban air. They can penetrate deep into the lungs and their fat solubility makes possible their accumulation in other organs such as the brain. Legislation has therefore continuously reduced the allowable emission levels and raised requirements for reporting the status of the exhaust system (OBD, on board diagnostics) in diesel powered vehicles.

In this project we develop a sensor based on thermophoresis (patent filed) to enhance soot deposition on the sensor. Thermophoresis is a force acting on particles located in a temperature gradient, such that a sensor held at lower temperatures than the surrounding exhaust gas will obtain enhanced deposition of submicron particles.

Sensors with finger electrodes (width/gap, 150/100 μm) and a heater for burn off of collected soot, were fabricated on one end of alumina substrates (90x5x1 mm). Thermal simulations initiated the construction of a sealed metal tube around the sensor rod (patent filed), which drained heat from the sensor surface allowing a temperature gradient of 50-100°C with respect to the exhaust gas temperature. This sensor layout showed preliminary good results in two runs of testing in diesel exhausts.

An aerosol based soot generation system was constructed which generates controlled soot atmosphere possible to use for testing and calibration of soot sensors in the lab.
In a recently approved one year project we will quantify and improve the thermophoresis effect, optimize the sensitivity of the soot sensor, test transistor devices and optimize the aerosol calibration system.

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Executive summary

Main Objectives:

Nanosized soot particles are a serious health hazard in urban air. Upon inhalation they can penetrate deep into the lungs and their fat solubility makes possible their accumulation in other organs such as the brain. For this reason legislation has continuously reduced the allowable emission levels and raised requirements for reporting the status of the exhaust system (OBD, on board diagnostics) in diesel powered vehicles. To meet the California Air Resources Board proposed legislation for diesel particulate filter, 17.5 mg/mile for light duty vehicles after 2013, will require the development of new sensors for monitoring the exhaust.

The objective in this project was to implement the idea of thermophoresis, that is a cold sensor surface, as a soot detecting sensor technology with improved sensitivity (patent filed). The goal was to perform proof of concept of thermophoretic soot sensors in order to get sensor manufacturers to pick up the technology and car manufacturers to implement it. Another goal was to develop an aerosol based soot generation system, by which soot sensors can be tested and calibrated in the laboratory before expensive testing in car and truck engines.

Method / implementation:

We have employed a resistivity sensor to show the potential of thermophoresis to enhance soot deposition on the sensor. Thermophoresis is a force acting on particles <100 nm located in a temperature gradient. Since gas molecules coming from the warmer area have a higher velocity than those coming from the cooler, aerosol particles receive a net momentum toward the cooler zone. Thus a sensor held at lower temperatures than the surrounding exhaust gas will obtain enhanced deposition of submicron particles and thus achieve higher sensitivities.

Sensors with interdigitated finger electrodes (width/gap, 150/100 μm) were fabricated by screen printing PtPdAu conductors on one end of alumina substrates (90x5x1 mm). A heater for burning off collected soot was located under the electrodes.

Soot particles are known to be charged. Therefore transistor devices are interesting to test as soot sensors. A first batch of transistor devices has been processed.

A large temperature gradient is required in order for the thermophoresis effect to be efficient. Thermal simulations of the heat distribution on the sensor surface led to the idea of using a “thermos” packaging layout, a sealed metal tube leaving only the finger structure protruding into the exhaust gas (patent filed).

A stationary diesel car motor was used to produce exhaust gas. This could be programmed to run in both steady-state and standard driving cycle modes. In both cases soot production was influenced by speed and torque of the engine. Resistance between

the fingers and temperatures of the sensor and exhaust gas were recorded throughout the measurements.

Aerosol technology was used to produce soot of diesel-like controlled size and concentration in the laboratory. A system for controlled deposition of soot on the sensor surface was constructed. The particle stream must be quenched to generate a controlled size of the particles and cooled in order to use the analysis system to quantify and characterize the soot particles. The particle stream was therefore actively heated by a heating gun and the sensor surface was cooled by air. It is also possible to generate soot of only one charge, which will be very useful for testing of the transistor devices

Concrete results and conclusions:

We have processed two batches of soot sensors in the project. The first batch was tested in diesel exhausts as described below. The second batch has important improvements, such as functioning conducting leads along the sensor rod, and an easy way to implement temperature sensors for precise testing of the technology. This will improve the packaging reliability and will be evaluated during the next planned test runs at VOLVO in mid December and in January 2010.

A first batch of transistor devices was processed. They did not function as intended since the soot created a short cut between source and drain. This was circumvented by a small adjustment in the sensor layout of the second batch of devices, which are now being processed.

Cyclic operation with repeated collection (resistance decreases from 120 to 20 Mohm) and burn off steps yielded sensor response curves for different soot concentrations and exhaust gas/sensor temperature combinations at steady-state conditions for soot concentrations 5-20 mg/m^3. Measurements during driving cycles where it was impossible to follow the instantaneous spikes in soot production from acceleration/deceleration were directed towards relating the sensor resistance decreases to the accumulated soot emission from the New European Drive Cycle.

The aerosol based soot generation system was shown to generate soot particles of similar size as those generated by a diesel engine. It was possible to collect soot in a controlled way on the cold sensor surface. The system has potential as a soot sensor testing and calibration system.

Recommendations:

In order to realize proof of concept for this soot sensor technology some critical test results and some development is missing. We need to quantify the thermophoresis effect and prove that the sensitivity of the soot sensor is high enough for the requirements of a sensor technology for the OBD application. Because soot particles are known to be charged, we will test silicon carbide based transistors as soot sensor devices. This technology has the potential to improve for example the sensitivity. We will also further

optimize the aerosol calibration system in order to make it possible to test and calibrate sensors in the laboratory before expensive tests are run in bench tests on real engines. When this have been established we are convinced that there is a high possibility that sensor producers will manufacture these soot sensors and car manufacturers will implement the technology in diesel vehicles.

It is important to get the results in the project exposed to the right community and one important conference will be the SAE conference in Detroit, the by far largest technical conference for the car industry.

We recently got a one year extension of the project approved. We think most of the above can be performed during this one year.

The project is also supported by a project at the VINN Excellence Center, FunMat at Linköping University. An important development of electrical contacts which stands very high temperature and corrosive environment are developed in a project within FunMat. These contacts will be implemented in the soot sensor layout, for finger electrodes and conducting leads.

During 2010 we intend to apply for a new project where the intention is to use the thermophoresis technology for a more general portable nanoparticle detector. The goal is to detect nanoparticles according to size, shape, concentration and content, since all these parameters have shown to influence their adverse effect. This will be a much larger project, which will require extensive development of the technology. However, we will greatly benefit from the results and methodology built up in the soot sensor project as well as from the consortium of expertise which now functions well together. The consortium will be increased in order to achieve the new goals.

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Introduction

Health effects of nanoparticles

Nanosized soot particles are a serious health hazard in urban air. Particles, typically of a size less than 10 or 2.5 μm (PM10 and PM2.5) are of growing concern as they contribute to a variety of health and environmental problems [1, 2]. The harmful consequences of soot on human health include respiratory and cardiovascular effects and the particles may be carcinogenic [3, 4]. Upon inhalation they can penetrate deep into the lungs and their fat solubility makes possible their accumulation in other organs such as the brain. Beside these health issues, soot deposition on buildings and vegetation contributes to their long-term degradation. Fine particles also cause reduction in visibility [5, 6], make lakes and streams acidic, deplete the nutrients in soil, and affect the diversity of ecosystems when settling on ground or water. Moreover, particles have both a direct and indirect effect on the earth's climate by either scattering or absorbing solar radiation and by acting as cloud condensation nuclei [7].

For this reason legislation has continuously reduced the allowable emission levels and raised requirements for reporting the status of the exhaust system (OBD, on board diagnostics) in diesel powered vehicles. To meet the California Air Resources Board proposed legislation for diesel particulate filter, 17.5 mg/mile for light duty vehicles after 2013, will require the development of new sensors for monitoring the exhaust.

Soot Sensor Technologies and Patent survey

Soot particle detection and measurement can be achieved by several methods. What has been described in the patent literature is soot collection on filters followed by the subsequent burn off with carbon dioxide measurement [8, 9], optical detection [10, 11] and the measurement of the electrical resistance of a soot layer deposited between two electrical contacts [12, 13]. None of these patents refers to the thermophoresis phenomenon as a basis for the soot deposition between the electrodes. The resistivity technique has been illustrated for both collection and regenerative burn-off for a sensor exposed to an open diesel flame [14]. Collection on the sensor's surface (interdigitated platinum electrodes on an alumina substrate) was done at 350°C, a temperature high enough to avoid water vapor condensation but low enough to prevent significant soot combustion on the platinum electrodes. The authors did not actively create a temperature gradient between the soot in the gas-phase and the sensor surface, nor did they mention the influence of thermophoresis. No information about the soot deposition mechanism was given.

Soot sensors may potentially be used to control the function and regeneration of the particle filter in diesel engine vehicles in order to for example avoid overheating due to an overload of soot.

Theory of Thermophoresis (ULUND)

Temperature is an important parameter for collection of aerosol particles. Thermophoresis is a force resulting from a temperature gradient being established in the gas phase [15]. Since gas molecules coming from the warmer area have a higher velocity than those coming from the cooler, aerosol particles receive a net momentum toward the cooler zone. A solid surface at lower temperature than the ambient thus can serve as a precipitator for aerosols.

When a given size distribution of generated soot particles passes through the deposition channel, the particles will be subjected to a force gradient towards the cold surface containing the sensor inducing a thermophoretic velocity, U_{th}. This process is independent of particle size while directly proportional to the temperature gradient in the free molecular regime. In the free molecular regime ($d<\lambda$), the thermophoretic velocity can be described by [15]:

$$v_{th} = -0.55 \frac{\mu_g \nabla T}{\rho_g T_p}$$

where ∇T is the temperature gradient in the deposition channel, T_p is the averge temperature of particle assumed to be same as the gas mean temperature, μ_g stands for the gas dynamic viscosity and ρ_g for the gas density.

Particle deposition mechanisms governed by thermophoresis assumes that the measured temperatures are expected to be the same as the gas temperatures at the hot and cold-plate surfaces inside the deposition channel, producing a temperature gradient of $\Delta T = 1.4x10^5$ (ºC/m) across the deposition channel.

Thermophoresis has been used for size independent deposition of Ag particles from polydisperse aerosols [16] and removal of particles from diesel exhaust [17]. Such high collection efficiencies are not necessary for sensor purposes but the temperature gradient must be sufficient to obtain reasonable response times.

The Goal of the SootSens project

The objective in this project was to implement the idea of thermophoresis, that is a cold sensor surface, as a soot detecting sensor technology with improved sensitivity (patent filed). The goal was to perform proof of concept of thermophoretic soot sensors in order to get sensor manufacturers to pick up the technology and car manufacturers to implement it. Another goal was to develop an aerosol based soot generation system, by which soot sensors can be tested and calibrated in the laboratory before expensive testing in car and truck engines.

VOLVO concluded that priority number one was to position the soot sensor after the particulate filter and for example indicate soot filter failure.

Soot sensor development (LiU, SenSiC, IASI)

Pre-project initated by VOLVO

The soot sensor development was originally initiated by VOLVO Technology. Linköping University was invited to participate in a PFF (Programmet För Fordonsforskning (Program for Vehicle Research)) #P29148-1 project run by VOLVO and financed by VINNOVA. In this program VOLVO is testing several prototype soot sensors from different manufacturers. LiU was invited to use their sensor competence in order to develop a soot sensor. LiU involved Lund University as a consultant to use their competence in aerosol technology to construct a soot generation system for sensor testing in the laboratory and sensor calibration.

A project meeting at VOLVO created the idea to use thermorphoresis as a sensor technology. In the first approache Peltier elements was tested for cooling of the sensor surface, but this was not viable. Future development of new materials for Peltier elements may however change that.

The resources in the project were then increased by approval of the present project. A number of new partners and competences in for example packaging, electronics, sensors and processing of sensors were added to the project and this made it possible to implement the thermophoresis technology in sensors possible to test in diesel exhausts.

The PFF project gave resources for VOLVO to test state of the art soot sensors from different manufacturers including the soot sensors from the present project. Our sensor invention still holds as interesting.

Resistive sensors (LiU)

Sensors based on resistance change of soot collected on interdigitated electrodes have been introduced [14] where sensitivity depends on the known mechanisms of soot deposition (convection, diffusion, impact and thermophoretic effects).

In the present project we started by using finger electrode structures patterned on oxidized silicon at LiU. For the sensors used for measurements in diesel exhausts the electrodes were screen printed on alumina substrates at Selmic Oy, see below. The silicon based substrate for the soot detection consisted of a finger electrode structure of Ti and Au sputter deposited on a thermally oxidized Si substrate, Si / SiO_2 (100 nm)/ Ti (5 nm)/ Au (200 nm). The finger electrode structure on the wafer was obtained using lift-off technology. The width of the electrodes was 80 μm and the two gap distances tested were 80 and 300 μm.

The substrate was then cut into small pieces, each containing a single pair of interdigitated electrodes. These were mounted on a substrate positioned in a 16 pin holder where a Pt 100 sensor (Pt resistor with the resistance 100 Ohm at 0°C) allowed

temperature measurement, see Fig. 1. The holder was placed in a sealed cell and exposed to soot containing gas flows produced by aerosol technology at ULUND, as described below.

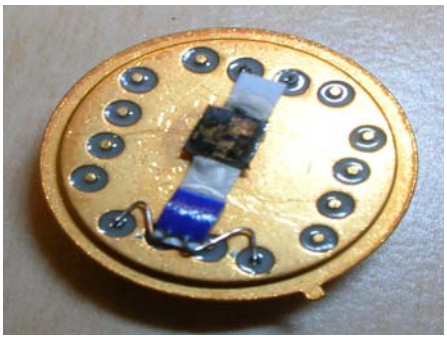


Figure 1. The sensor mounted on a heater with a Pt 100 temperature sensor (blue)

Sensor Processing by Screenprinting (Selmic Oy)

The sensor will operate in the exhaust pipe of the diesel engine which is an extreme hostile environment. Therefore certain thick film conducting materials were chosen. Expected operating environment was:

- Operational: -40 up to +350°
- Storage: -55 ... +125°C
- Harmful substances in the exhaust gas
- Large variation of operating temperature and its cycling nature causes risk of water condensation, (since exhausts contains up to 10% water, condensation may occur at temperatures < 100°C)

Aluminum oxide substrate and thick film pastes are known to work well in extreme temperatures. Base aluminum oxide (Al_2O_3) substrate is fired over 1300°C and thick film patterns above 850°C. Also a great advantage of these thick films are that the different thick film materials are TCE [18] matched not only to aluminum oxide substrate but also to each other and different layers integrates to each other almost seaminglesly which gives great reliability advantage at elevated or lowered temperatures.

The risk of corrosion due to harmful substances in the exhaust gas and risk of electromigration due to condensating water were main drivers when choosing materials to implement the thick film sensor. Also the selected materials are compliant to ELV – directive [19].

Silver is a commonly used material in thick film applications because of low cost and relatively low electrical resistance. Down side of the silver is that it is a very sensitive material in terms of electro migration and emission. Gold is used in applications where

high reliability is required. Gold is also not susceptible to ionic migration. A disadvantage of the gold is high cost. Copper is also used for thick film conductors but since the copper requires sintering in nitrogen atmosphere it's not compatible with all thick film materials.

The design of prototype sensors at Selmic followed the company specific design rules. Manufacturing was made by using existing equipment and production lines which are also used in volume production. All operations are quality certified according ISO9001-2001 and ISO/TS 16949:2002.

First batch of sensors was made with Au based materials containing also palladium except solid back side metallization on version 2, which was made with silver based conductor. Heater resistors were made in serpentine shape by using special silver containing low ohmic resistor pastes. Everything except the sensitive sensor area was protected with a dense, acid resistant protective overglaze layer.

Three different layout versions were made in the first batch. The main difference between the layouts was the pattern on the backside of the aluminum oxide rod. The first version employed integrated heaters on the backside for the three sensors to be assembled on the top side of the rod. Second version hold solid back side metallization and third version was without any thick film prints on the back side, just pure aluminum oxide.

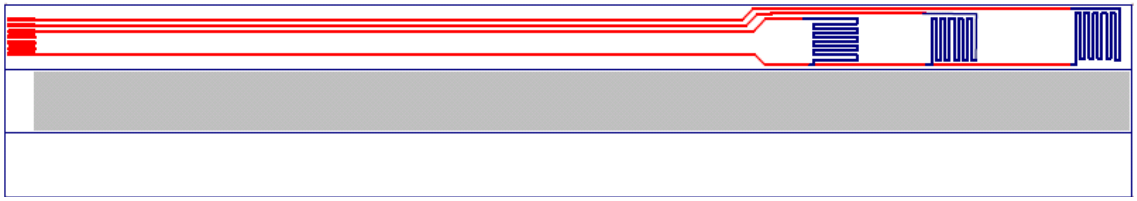


Figure 2. Batch 1, three different back side layout

1. Bottom side with integrated thick film heaters
2. Bottom side solid metallization
3. Bottom side solid with bare aluminum oxide surface

In the top side design there were spots for bonding three silicon based platinum sensors (red rectangle) and one integrated thick film sensor containing platinum (green rectangle). Also a heater resistor was designed around the top most sensor (yellow rectangle).

Figure 3. Batch 1, top side of the sensor

Figure 4. Batch 1, top side with heater resistor around top most sensor element and places for three optional silicon sensor elements

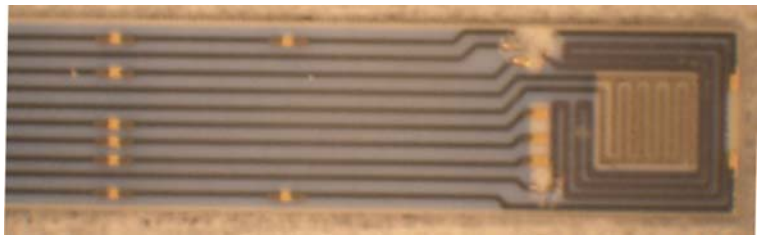


Figure 5. Batch 1, thick film sensor element circumvented by heater resistor

Conclusions from batch one was that the thick film sensors detected soot and thus was working but conductor resistance was too high. This was because of too narrow line widths and relatively high sheet resistance of the PtPdAu –paste used.

In batch 2 a new layout was designed based on experiences collected from batch 1. To gain lower conductor resistance from contact pins to sensor element silver based conductors were chosen. All conductor tracks were covered with dense, acid resistant over glaze again. The layout was optimized for only one sensitive thick film element or optionally silicon based element on top of the rod. This layout gave the possibility to widen conductor lines in order to decrease the conductor resistance. Heaters were removed from the design. The material and layout of the finger electrodes remained unchanged.

Figure 6. Batch 2 sensor rod with widened conductors

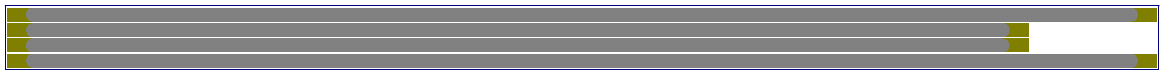


Figure 7. Batch 2 Bottom side

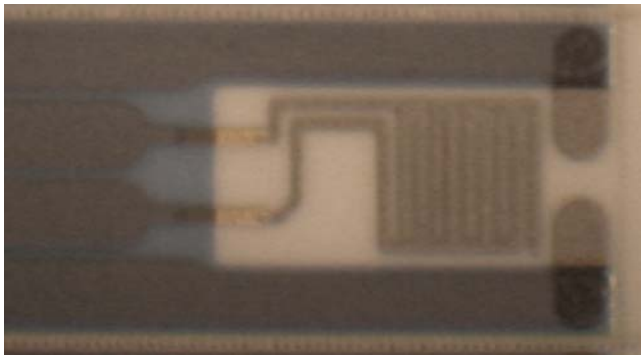


Figure 8. Batch 2 sensor element terminated with
silver conductors and covered with protective overglaze

Transistor devices (SenSiC, LiU)

The soot particles are known to be charged. Therefore we found it interesting in this project to also investigate the potential of transistor devices as soot sensors. The technology is based on silicon carbide, SiC, transistor devices, since this provides a technology known to be very resistant to high temperature and harsh environment [20]. These devices have been developed for example for combustion control in car exhausts and domestic boilers. The latter technology is now being commercialized by SenSiC AB.

The sensor chips are very small, a 3 inch wafer contains thousands of sensor chips, and therefore the cost is small even though a silicon carbide wafer is quite expensive, see Fig. 9.

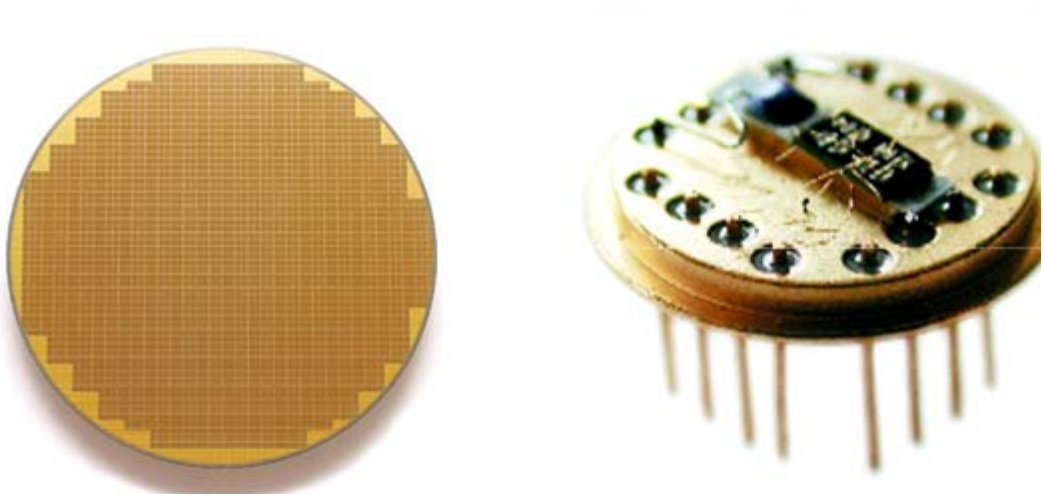


Figure 9. To the left: Silicon carbide wafer, 3 inch in diameter, with ~ 2000 sensor chip
To the right: Mounting on a 16 pin holder of one sensor chip and a temperature sensor (blue) on a substrate, which can be heated.

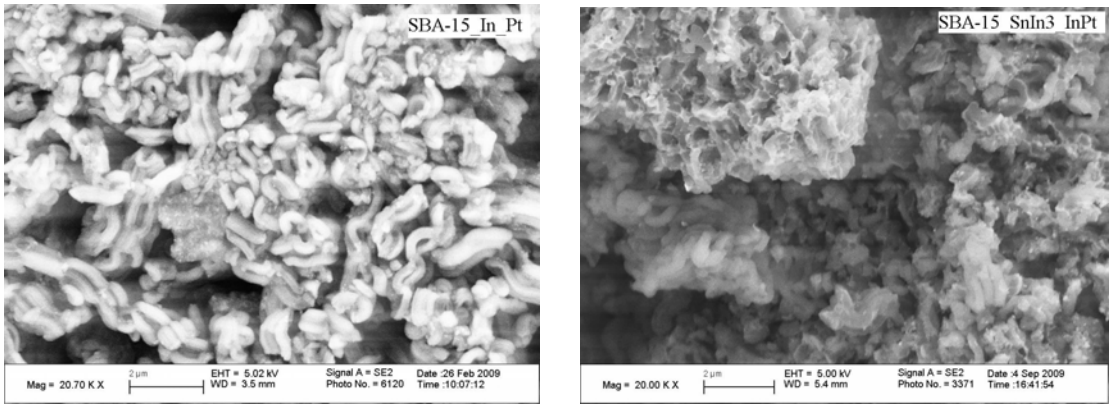
Layout changes will have to be performed of the device design used for gas sensors, otherwise the soot deposition will create a short cut between drain and source. The first batch of transistors, which was processed did not function as soot sensors. However, further improvement of the design has been performed and it is now very likely that the second batch of transistors will function. These will be tested during the SootSens II project.

Sensing layers (IASI, LiU)

The presence of a sensing layer on the deposition area can contribute to the improvement of soot adhesion. The role of this layer is to make a rougher surface and thus improve the soot deposition and to generate catalytic effects aiming to allow easier removal of the

soot after the deposition step. Soot measurement requires of course that the resistance of the sensing layer is still allowed to change during the soot deposition.

The sensing layer must contain a very active catalytic species for the oxidation reaction, based on noble metals, the best choice being platinum. Platinum was deposited on mesoporous materials as silica SBA-15, previously impregnated with different semiconductive oxide species, in order to make them catalytically active in oxidation reactions.



a b

Figure 10. SEM image of two samples of SBA-15 impregnated with 5% (wt) indium oxide then 5% Pt (a) and with Sn+In oxides (1/3) then In oxide and platinum (5% each) (b)

The SEM image indicates the preservation of the shape and size of SBA-15 after the impregnation with indium oxide and platinum. The impregnation products were located mainly on the outer surface of SBA-15, as confirmed by BET analysis of surface structure. The specific surface areas keep high values, several hundreds m^2.

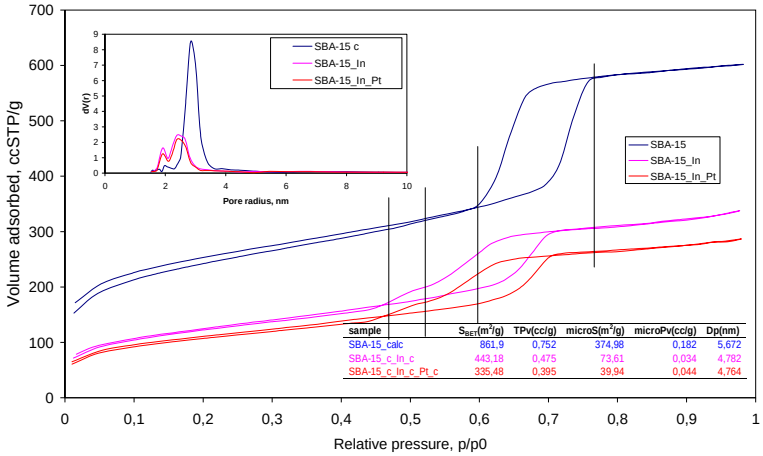


Figure 11. BET isotherms and pore distribution curves for SBA-15 (SBA-15), SBA impregnated with indium oxide (SBA-15_In) and SBA impregnated with indium oxide and platinum (SBA-15_In_Pt)

By the TEM technique, the location and state of the particles can be recognized. The grains of platinum and indium oxides are located mainly on the outer surface of the SBA-15 particles.

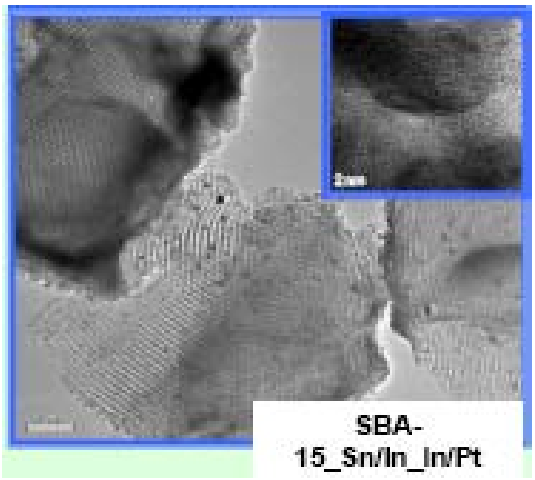


Figure 12. TEM image of a sample of SBA-15 impregnated with Sn+In oxides (1/3) then In oxide and platinum (5% each) (sample b in Figure 10)

Aerosol Technology (ULUND)

Soot generation

PM is a complex mixture of small airborne particles and liquid droplets typically consisting of agglomerates of primary particles (with 20-35 nm diameter) and smaller nucleation mode particles mostly in liquid phase. The primary particles in agglomerates are composed of soot (elemental carbon coated with traces of metallic ash) and onto those agglomerates, the heavier organic compounds and sulphuric acid are condensed. The nucleation mode particles on the other hand are dominated by condensed hydrocarbons and sulfates [21, 22].

The designed combustion soot generator, see Fig. 13, consists of three sections: a diffusion flame, flame quenching and particles mixing. Propane as fuel was fed into the inner of two co-axial stainless steel pipes of 7 and 28 mm i.d., respectively. While particle free dry air (PFA) was introduced through the outer pipe as an oxidant. The stability of the flame, and thus the generated soot, is very sensitive to small variations in the flow pattern. Two mass flow controllers (Bronkhorst High-Tech MFCs) were used to regulate the fuel and coating air flow rates with high precision. Additionally, the coating air stream was stabilized with a ceramic honeycomb monolith acting as a flow laminarizer to further enhance flame stability. By varying the fuel and air flow rates and hence the equivalence ratio, soot particles of different concentrations and size distributions was generated, see Table 1.

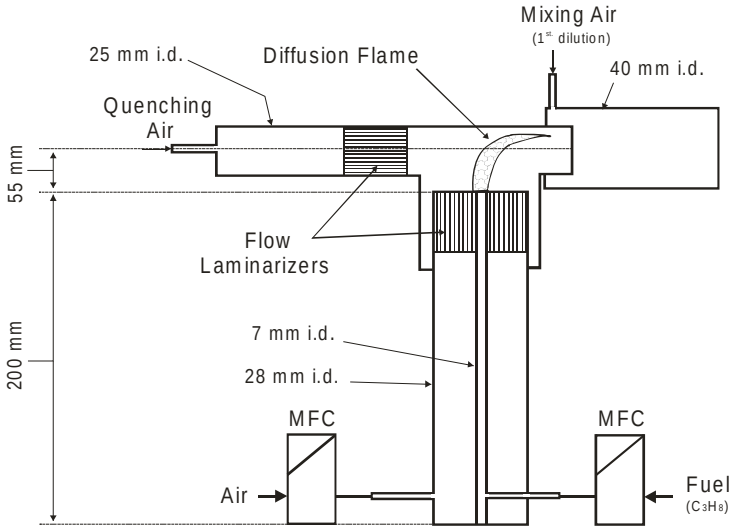


Figure 13**:** Shematic diagram of the experimental set-up

Table 1: Flow rates of fuel and coating air and geometric mean diameter (GMD) of particles produced.

Case	Equivalence ratio ϕ	Fuel / air flow rates (L/min) / (L/min)	Soot particles generated GMD, nm
Equivalence ratio 1	0.632	0.085 / 3.2	55
Equivalence ratio 2	0.653	0.085 / 3.1	85
Equivalence ratio 3	0.675	0.085 / 3.0	110

Thermophoretic soot sampler and deposition on sensor

The next step in this study was to investigate the capability of using thermophoresis as a method to deposit soot particles on the soot sensor and determine the key parameters within deposition and detection steps.

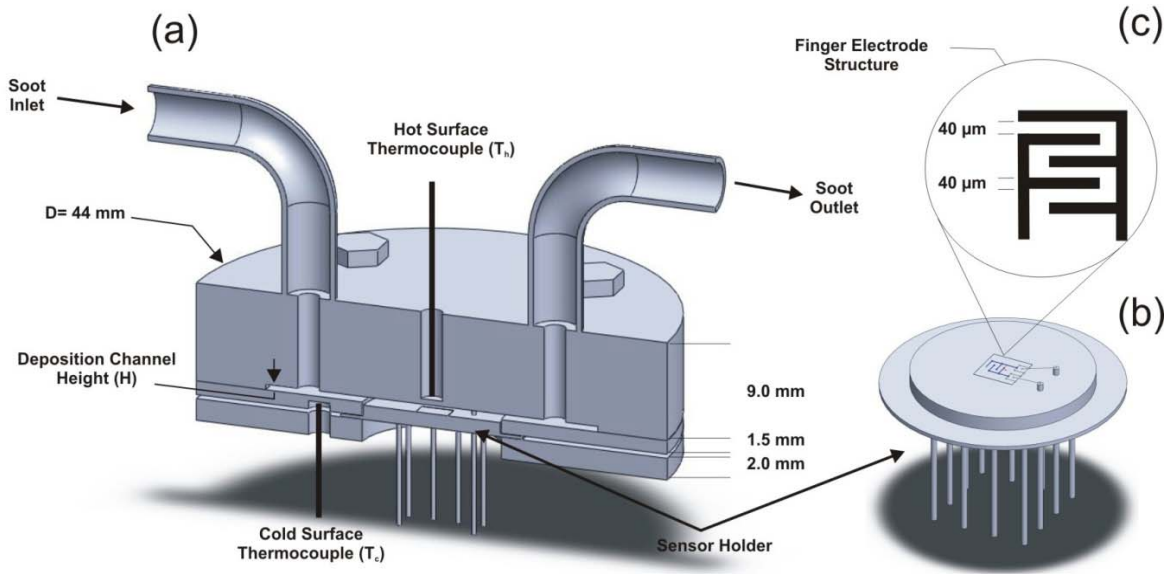


Figure 14: A three-dimensional cross section view of the soot sampler (a) and sensor holder (b) including design of the finger electrode sensor structure (c).

Figure 14 shows the thermophoretic soot sampler that was designed and used for the deposition of generated soot particles on the sensor. The sampler was manufactured based on a plate-to-plate thermal precipitator design [23, 24].

We applied a constant flow rate of 300 ml/min and in all deposition experiments the Reynolds number (Re = 23) indicates a laminar flow. However, as the inlet flow enters the deposition channel perpendicularly, a turbulent flow is expected initially and requires a certain length (hydrodynamic length) to become laminar. The hydrodynamic length can be calculated as

$L_h = 0.04 \cdot Re \cdot D_h$

Where D_h stands for the hydrodynamic diameter of the flow channel calculated as

$D_h = 2W \cdot H/(W+H)$.

Accordingly, $L_h = 0.9$ mm ensures a laminar flow over the sensor. It has been shown in the literature that thermophoretic deposition can be achieved under both laminar and turbulent flow conditions [25]. Moreover, earlier investigation by Tsai and Lu [24] showed that the thermophoretic deposition efficiency is only slightly decreased under turbulent conditions compared to laminar for Re values of 6580 and 1340, respectively.

Messerer et al. [23] showed that the deposition efficiency of agglomerated soot particles in a thermophoretic plate-to-plate deposition cell was within 5%, independent of particle size over the range of 30-300 nm (the size range relevant for this study). We also calculated the thermophoretic velocity and particle collection efficiency using both free molecular regime ($d<\lambda$) and continuum regime formulations (particle size larger than the molecular mean free path; $d>\lambda$). The thermophoretic velocity for $d<\lambda$ was found to be

0.52 cm/s and that for d>λ 0.38 cm/s. The efficiency of particle collection in the deposition cell was found to be 46.4% and 35% cm/s for d<λ and d>λ respectively [15].

Soot particle characterization

A scanning mobility particle sizer (SMPS 3934 form TSI Inc., USA) was used to characterize the number size distribution of the generated soot. It consists of a differential mobility analyzer (DMA 3071) and a condensation particle counter (CPC 3010) operating at a sample flow rate of 1.0 l/min and a sheath air flow rate of 6.0 l/min allowing a measurement range of 10-450 nm. Before the generated soot was analyzed with the SMPS system, it was diluted using an ejector diluter with a modified inlet nozzle (DI-1000 Dekati Diluter, Finland) to a ratio of 1:13 in order not to exceed the measurable range for the SMPS. After SMPS verification of a stable size distribution of the generated soot particles, a flow of 300 ml/min was established from the outlet of the burner through the thermophoreticusing a critical orfice at the thermophoretic sampler outlet, see **Fig. 13**.

Organic and elemental carbon analysis and TEM analysis

The organic and elemental carbon analysis (OC/EC) of the particles was performed by collecting samples on pre-baked quartz fiber filters (Tissue quartz, SKC Inc.) and subsequently analyzed according to the standard thermal method (VDI 2465/2) by a Thermal Carbon Analyzer (Model 2001, Desert Research Instruments Inc.). The set-up for the soot collection involved sampling soot directly from the burner outlet into two parallel lines at the same flow rates. In one line only a quartz filter was used, while in the other line a Teflon filter (Zeflour, SKC Inc.) was followed by another quartz filter. This configuration allows correction for possible gas phase organic vapors absorbed onto the quartz filter.

The morphology of the generated soot particles was investigated by a 60 KeV PHILIPS CM10 Transmission Electron Microscopy (TEM) using an electrostatic precipitator (NAS Model 3089, TSI Inc.) whereas the samples were collected on a carbon-coated copper grid of diameter 3 mm.

The Aerosol Particle Mass Analyzer (APM)

The APM coupled in series with a DMA (DMA-APM), was used for online mass determination of mobility classified particles, see Fig. 15. The system can be used for the determination of the particle effective density and mass-mobility relationship. The APM consists of an outer (r_2) and an inner (r_1) cylinder rotating at the same rotational speed ω. The aerosol is introduced in the annual gap between the cylinders and a potential (V_{APM}) applied to the inner cylinder while keeping the outer cylinder grounded. Thus, the force keeping the charged particles in orbit is the electrical force, balanced by the centrifugal force. Since the centrifugal force is mass dependent the particle mass can be determined according to:

$$m = \frac{qE}{r\,\omega^2} = \frac{qV_{APM}}{r^2\omega^2 \ln(r_2 / r_1)}$$

where r is the average radial distance to the gap between the cylinders from the axis of rotation ($(r_2 - r_1)/2$), q the particle charge and E the electrical field. The APM is described in more detail by McMurry et al. [26].

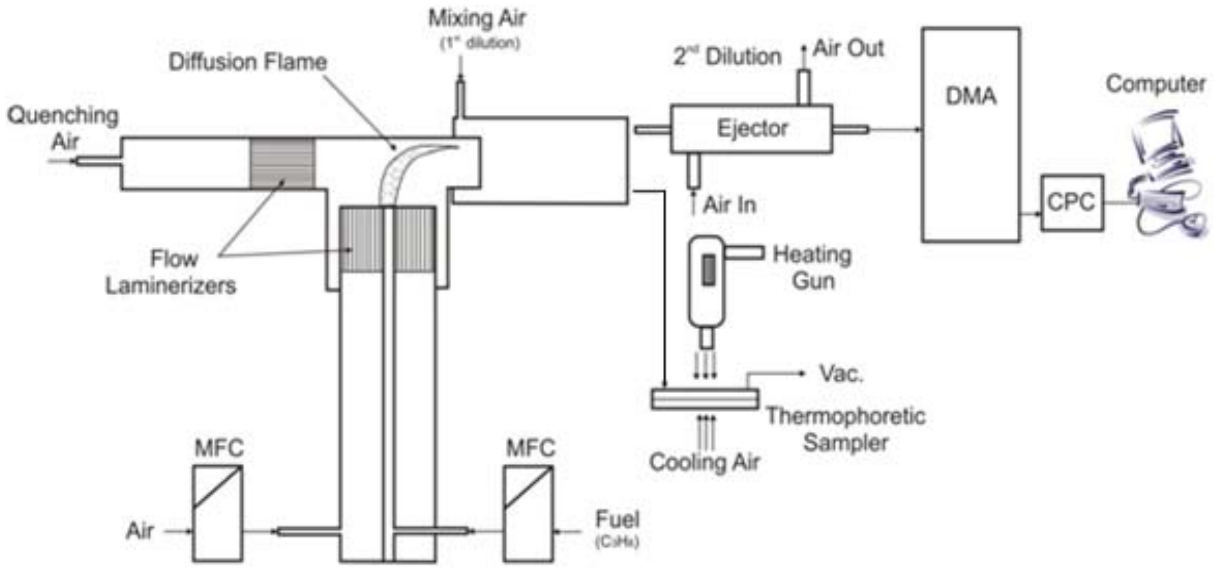


Figure 15 Schematic diagram of the experimental set-up

In the system, a DMA and an APM are coupled in series where the DMA selects particles of one mobility diameter at a time and the mass distribution of the selected particles is determined by stepping the APM voltage. The mobility diameter (d_{me}) and the particle mass are, in the transition regime, related as:

$$m = \underbrace{\frac{\rho}{\chi^3} \cdot \left(\frac{C(d_{me})}{C(d_{ve})} \right)^3}_{\rho_{eff}} \cdot \left(\frac{\pi}{6} \right) \cdot d_{me}^3$$

where ρ_{eff} is the density of the pure compounds, C the Cunningham correction factor, χ the dynamic shape factor and d_{me} the volume equivalent diameter. ρ_{eff} is the effective density defined as:

$$\rho_{eff} = \frac{m}{d_{me}^3} \cdot \frac{6}{\pi}$$

Thermal management design (SINTEF)

Control of the surface temperature at the sensor (finger structure) is essential for thermophoresis to work as intended. Thus, a thermal management design was requested. SINTEF was responsible for the design. The design process was aided with the finite element analysis (FEA) tool COMSOL Multiphysics 3.5a. The design method was a progressive approach, where the modeling started with simplified 2D finite element models and successively advancing towards more realistic 3D models. The design process lead to a new thermal management design concept; 'Thermos cooling', see Fig. 16. A result plot from the 'thermos' design is shown in Fig. 17. The result from the 'thermos' concept, compared to three other, more classical, thermal management designs is shown in Fig. 18. The design concept was applied for a patent and later presented at an international conference in Norway, see Publications (where more details can be found).

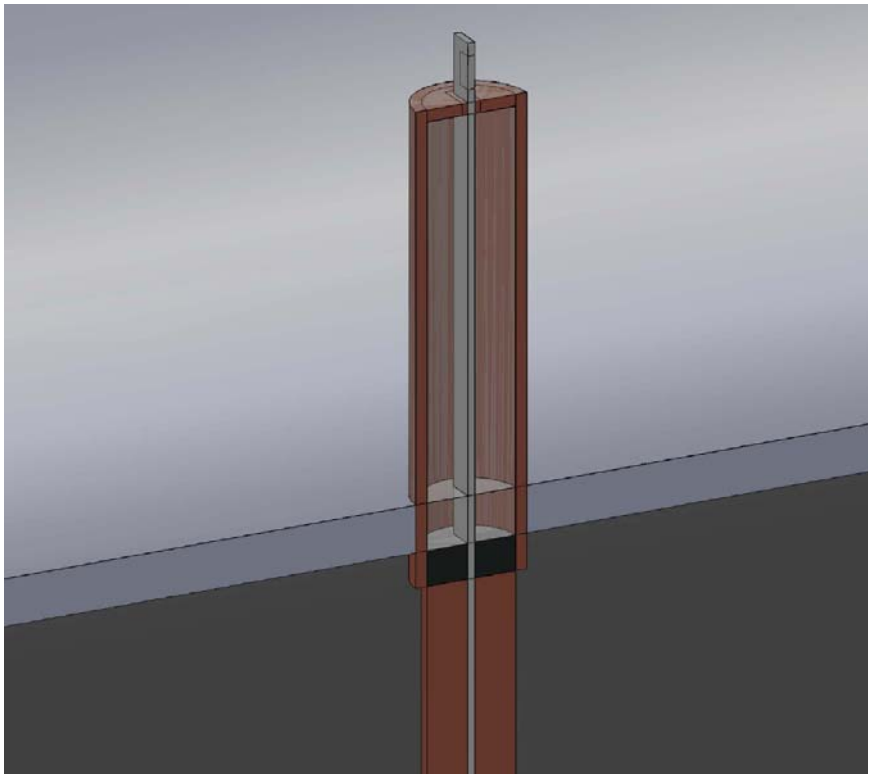


Figure 16. Cross−section of the 'thermos' concept

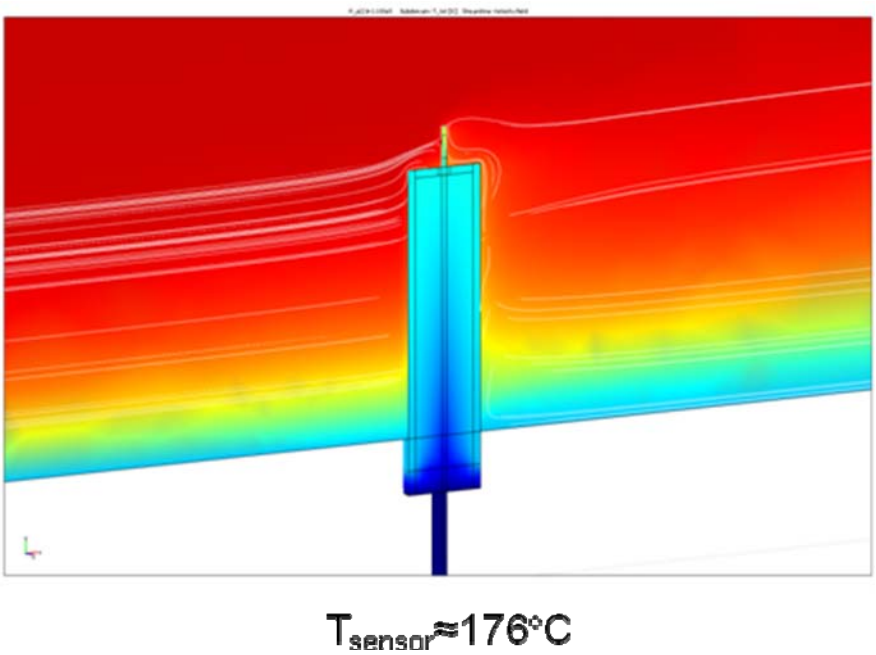


Figure 17. Result plot from the 'thermos' concept

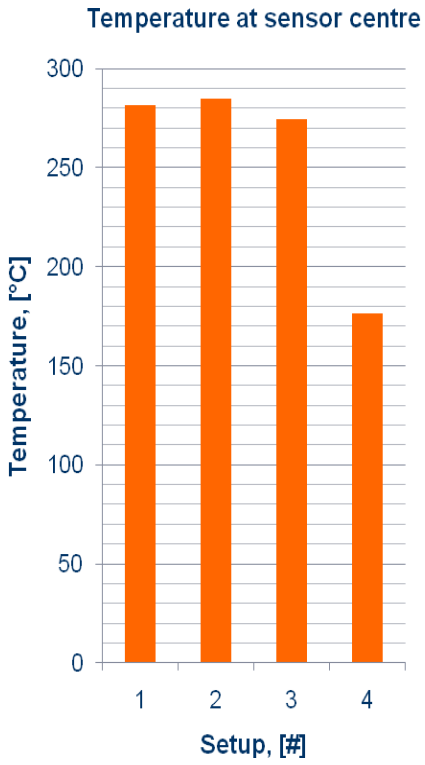


Figure 18. Comparison of four different thermal management solutions. The bars show the resulting temperature at the sensor surface. The 'thermos' concept is represented by setup no. 4.

Sensor Packaging (Mandalon Technologies AB)

The intention from the beginning was to make a general sensor rod in order to be able to adjust the components afterwards. This led to a complex structure with some drawbacks. As described in previous chapter we implemented backside heaters, around-sensor heater and several wire bondable connection points. This complexity led to narrow conductors and thus too high resistivity. For the first batch this has been taken care of with bypass wiring in order to reach low enough resistivity for the measurements. From a packaging point of view this patch is working but unreliable and also time consuming.

The experiences from batch 1 have led to the design of batch 2 which has focused on basic function of the sensor, backside heater and temperature measurement points.

All packaging considerations have to deal with the elevated temperature in exhaust pipes and to obtain the desired temperature gradients. Most of the electrical connections has been made with gap welding and, when applicable, normal soldering on colder areas. All attachments has been made with ***Resbond 907 GF***, a glass fibre reinforced putty designed for exhaust systems. However, due to the small system dimensions there is room for improvement as the fibres becomes relatively too large. All internal connections are secured with Resbond.

For the first measurements the sensor rod was mounted bare as in Fig. 19.

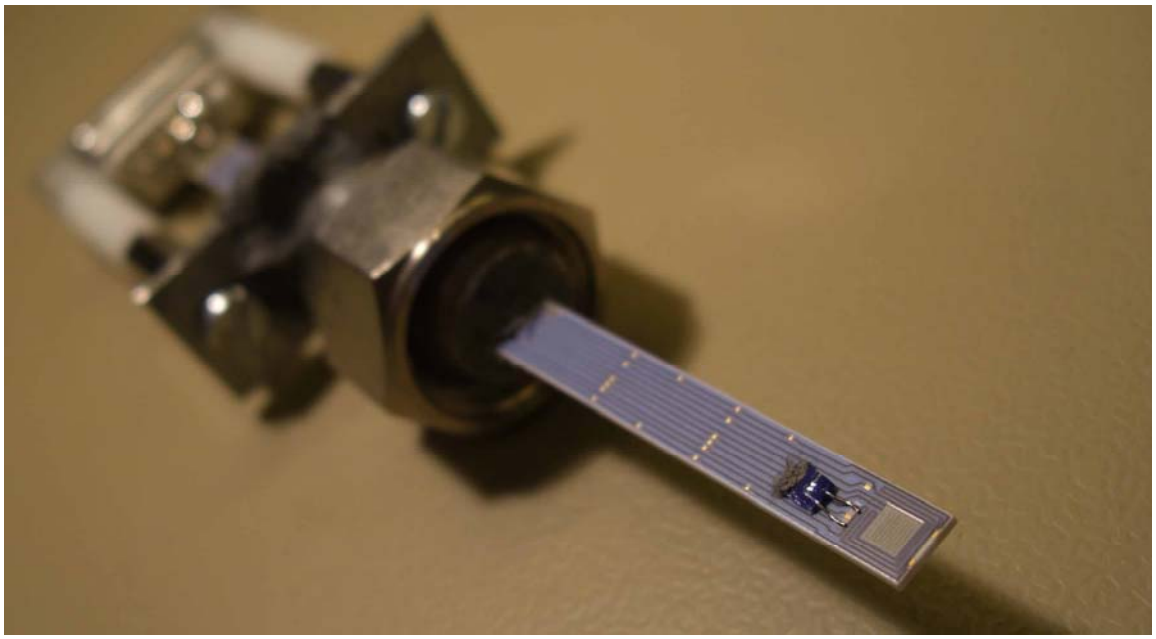


Figure 19. Bare sensor rod with Pt-100 attached

The later design for the whole component is made in accordance with the thermal simulations as a “thermos”. The thermos is built with 10 mm diameter stainless tubes of varying lengths, the sensor rod centered in the tube and fitted with the ceramic putty. The bypass approach lets us also apply temperature measuring Pt-100’s along the rod, inside the tube. We have also tested an active cooling of the sensor tip. This is performed with an applied air flow beneath the sensor tip, see Fig. 20.

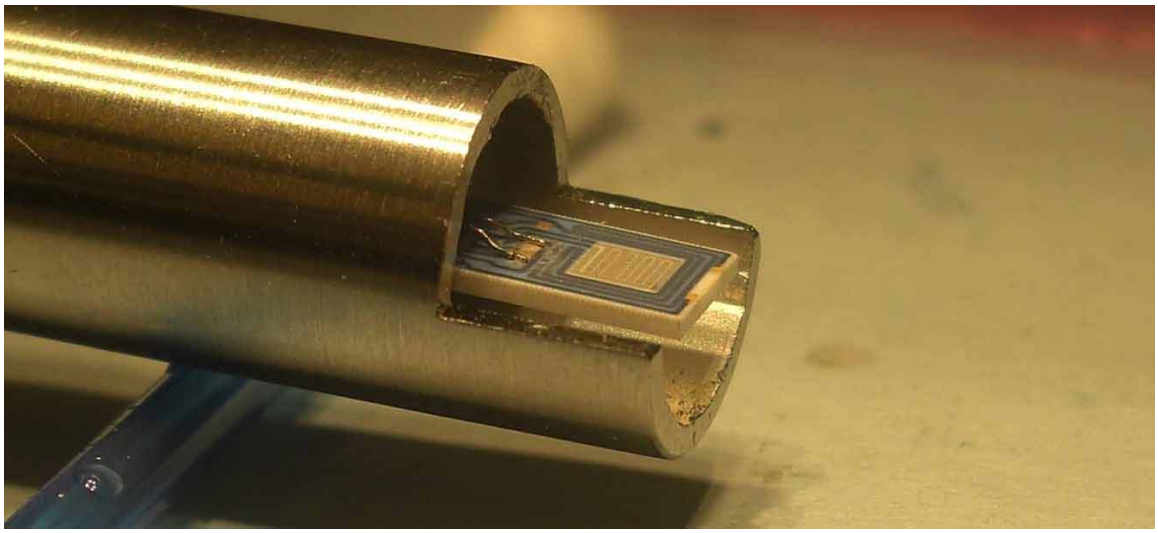


Figure 20. Active cooling design

In order to regenerate the sensor we need to heat the tip. This is made with a Heraeus heater mounted on the back of the sensor tip. At the back end all electrical connections are made thru a D-sub connector.

Figure 21. Test of heated tip of sensor rod

Fig. 22. Electrical connections

Sensor Electronics (SINTEF, LiU)

SINTEF have tested the electronics required to control the sensor in a cyclic operation between soot capturing and regeneration. An off-the-shelf integrated circuit specified for automotive applications has been identified as a key component for this purpose. An electronic test board based on this component has been developed. The cyclic mode is shown schematically in the graph below. The x-axis show the resistance of the SootSens sensor and the y-axis show the voltage signal read by this integrated circuit.

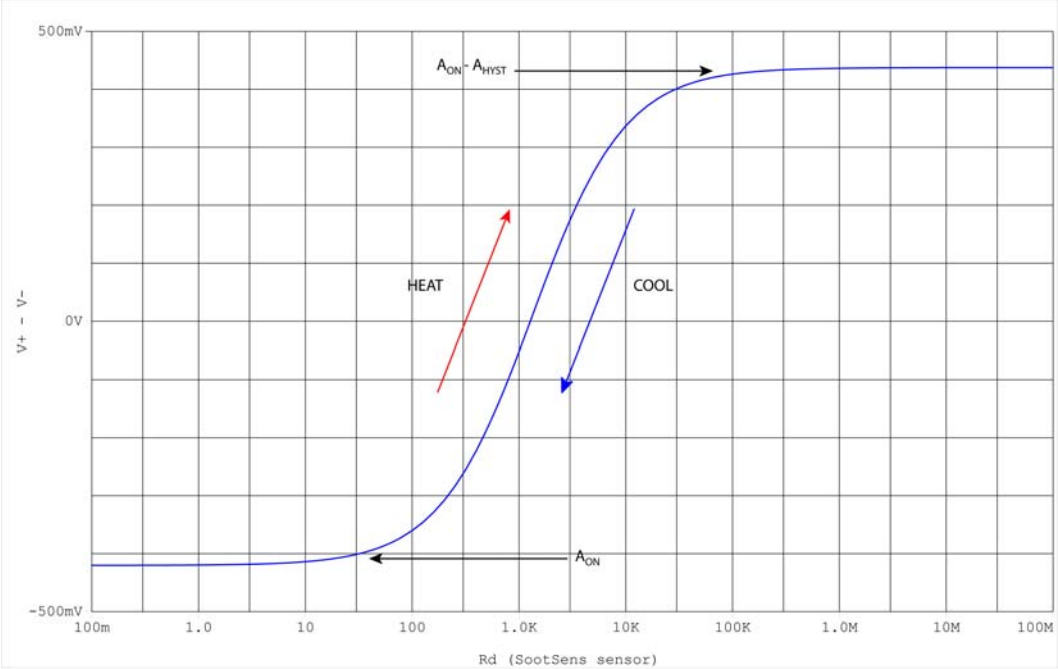


Figure 23. Voltage versus resistance of the soot sensor operated in a two temperature cyclic mode.

This first prototype is large and bulky and only suitable for proof-of concept measurements in the laboratory, see Fig. 24. The final electronics will of course be much smaller and integrated in the sensor unit.

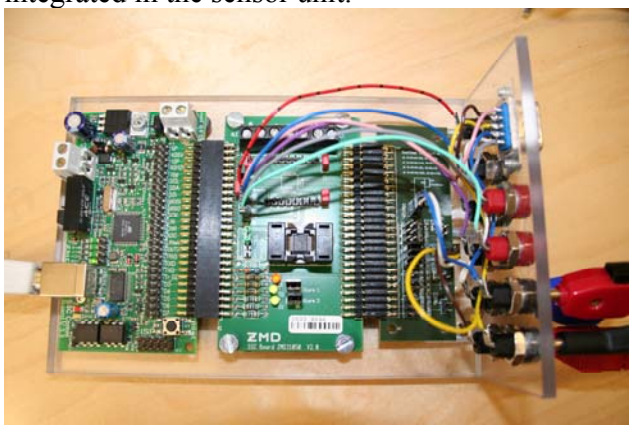


Figure 24. The electronics for cyclic temperature operation of the soot sensor

Some problems remains to be solved in order to use this standard equipment for the soot sensor in this project. SINTEF had rather limited financial resources in the project and therefore had to focus on the Thermal management simulation, which at this stage in the project was the most important. For the first testing in diesel exhausts at VOLVO the necessary electronics was therefore developed at LiU.

Sensor Testing

Sensor testing in the laboratory (LiU, ULUND)

Soot generation – stability and characterization

One of the aims of this investigation was to produce a set of soot particle size distributions and concentrations with high stability. Average particle size distributions used for the deposition experiments are shown in Fig. 25 a**.**

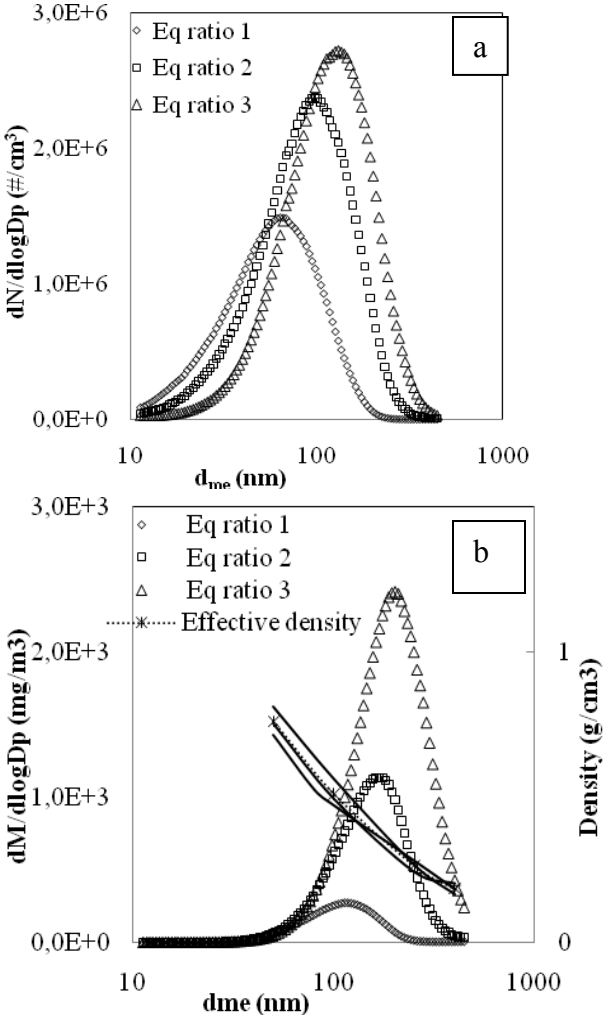


Figure 25: SMPS measurement of number and mass distributions

All distributions are unimodal, with slight deviations from lognormal distributions. The geometric mean diameters (GMDs) based on number concentration were 55, 85 and 110 nm and the geometrical standard deviations (GSDs) were 1.77, 1.80 and 1.81 respectively for the three cases. The smallest GMD (55 nm) is considered to be similar to a typical diesel soot size distribution.

The effective density of soot particle was also measured using the DMA-APM technique. The effective densities for a given size varied by less than 10% for the three different the equivalence ratios. The effective density decreased with increasing particle size from ~0.8 g/cm3 for 50 nm particles down to below 0.2 g/cm3 for 400 nm particles. The results are presented in Fig. 25 b.

The first sensor testing by thermophesis

A simple deposition cell where a thermal gradient was established between the sensor surface and the gas stream was constructed. Since thermophoretic deposition of the soot particles was favored by the contact of the gas flow with a cold surface, the diluted soot flow was heated to 240-270^{o}C before deposition by passing it through a steel coil heated by an electrical heater. The sensor was heated by the gas stream and the temperature, as measured by the Pt 100 sensor, was in the range 105-125°C. These temperatures were sufficiently high to prevent water condensation at the dilution levels used. Soot layer morphology was investigated by SEM using a Leo 1550 VP emission field scanning electron microscope, see Fig. 26.

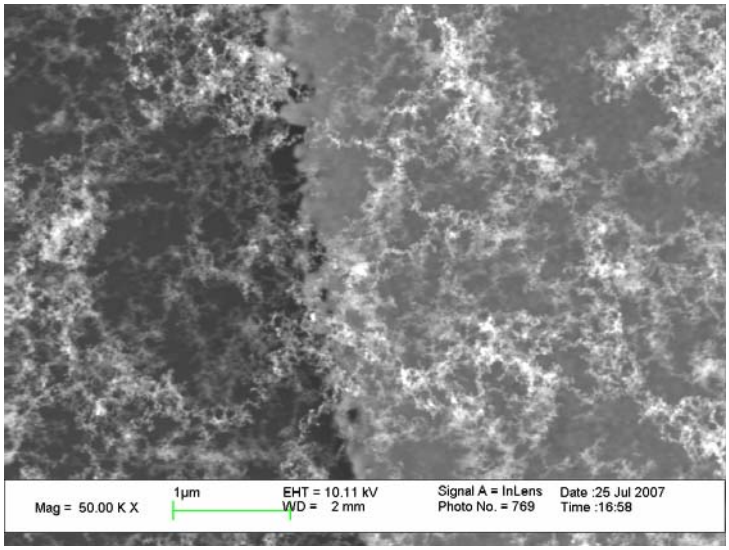


Figure 26. Typical SEM image of soot deposited onto SiO_2 (dark grey, to the left) and the gold electrode (grey, to the right)

For sensing purposes, the deposition of soot leads to formation of multiple disordered bridges between the metallic fingers creating a conductive layer as stipulated by the perculation theory. Since initial deposits are insulated from one another, a delay is expected before a resistance decrease is registered. The resistance measurements were performed using a multimeter device (TTI 1604) capable of measuring in the resistance range from1 kOhm to 20 MOhms.

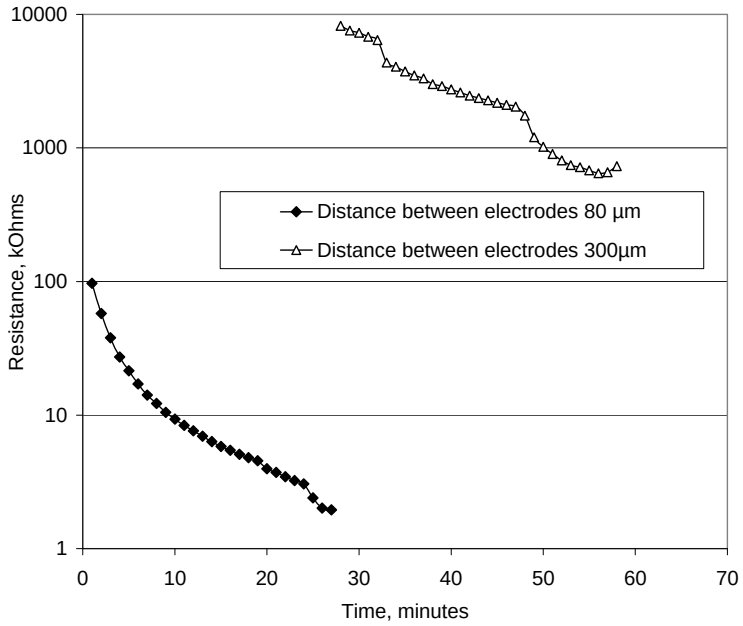


Figure 27. Resistance values evolution in time for different distances between electrodes - logarithmic scale (dilution ratio: 1/100; gas flow temperature: 240^{o}C, sensor temperature: ~ 100 - 125°C)

The time delay for the initial resistance decrease as well as the total resistance decrease was observed to be dependent on the distance between the metal finger electrodes as shown in Fig. 27.

Improved deposition by thermophoresis and resistivity measurements

As the soot particles, which contain conducting elemental carbon, deposit on the sensor surface they will start building a network of disordered bridges in the gaps between the metallic fingers until the first conductive layer is established. This process will take a certain time (referred to as on-set time) where no resistance decrease is recorded. There will also be a certain time before the resistivity decreases enough to fall into the measureable range of our system.

Figure 28 shows the electrical resistance over time when depositing soot generated with equivalence ratio 1 (55 nm), 2 (85 nm) and 3 (110 nm) on fresh sensors continuously for 2h. Before the first conductive layer of soot was established on the substrate surface, the resistance values between the finger structures were out of the measurable range and set to the maximum measuring range in Figure 7, 40 Mohms. The figure shows a significant

difference in on-set time between the three different soot size distributions, The on-set times of 27.0, 7.0 and 1.6 minutes were observed when exposed to soot using three different cases of equivalence ratios 1, 2 and 3 (GMD = 55, 85 and 110 nm), respectively. After that time, the resistance started to decrease as indicated by the multimeter. Different exposed soot size distributions did not only show a difference in the on-set time but also in the resistance levels during later part of exposure time, which went on decreasing slowly. For equivalence ratios 2 and 3, the resistance profile seems to be similar, starting with a relatively fast decrease for the first 20 min and then beginning to almost level off, reaching a final resistance of 34 and 8 kOhm for set exposure time, respectively. For the equivalence ratio 1 case, the resistance profile follows a smoother decline ending up at a considerably higher resistance of 1.4 Mohm.

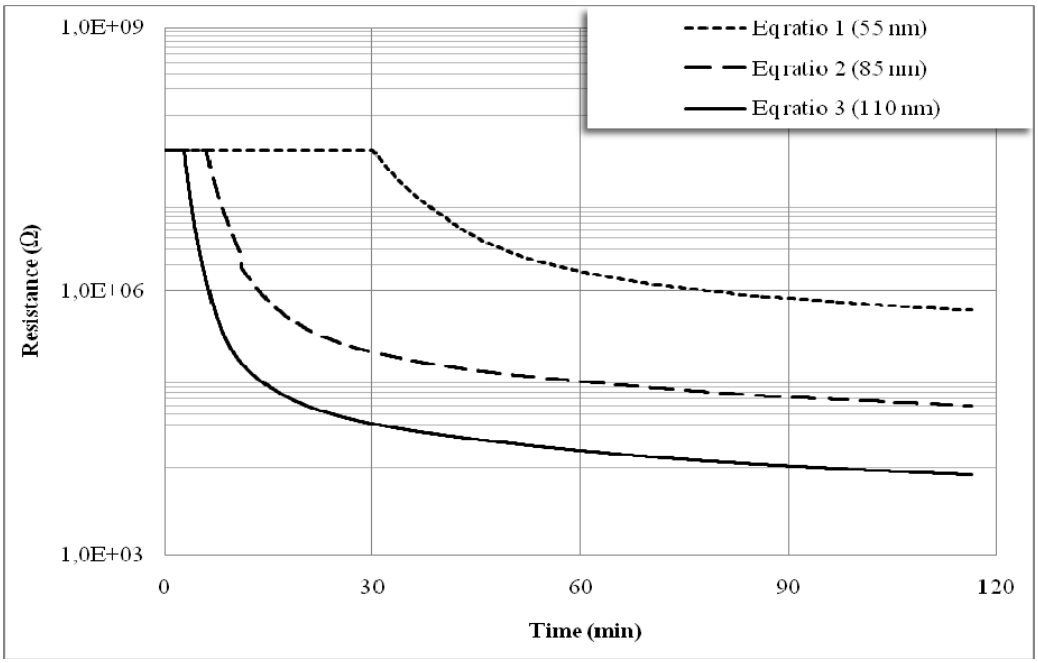


Figure 28: Examples of a single measurement showing the resistance change over time for fresh sensors exposed 2h to generated soot particle modes with GMDs of 55, 85 and 110 nm, respectively.

The difference in resistivity and on-set time can largely be explained by different mass exposures per minute for the three different cases. After normalization of the resistivity with cumulative mass-exposure, the three curves appeared more similar, see Fig. 29. Still, there was a difference in resistivity between the cases: The case with the largest agglomerate mobility size (110 nm) showed the fastest decrease in resistivity. There are several possible reasons for this: The particles in the size distribution with GMD = 55 nm are more compact as shown by the higher effective density compared to 85 nm and 110 nm. Depositing a larger number of more compact agglomerates, compared to fewer larger agglomerates with more open structures but with the same total deposited mass will require a longer on-set time as it will take longer to create an electrically conducting bridge between the electrodes. One can expect that the fractal-like conducting branches

between two electrodes are being longer for the cases of larger agglomerates as an effect of the decreased effective density. However, it is interesting to note that the relative difference between the three cases increases with time.

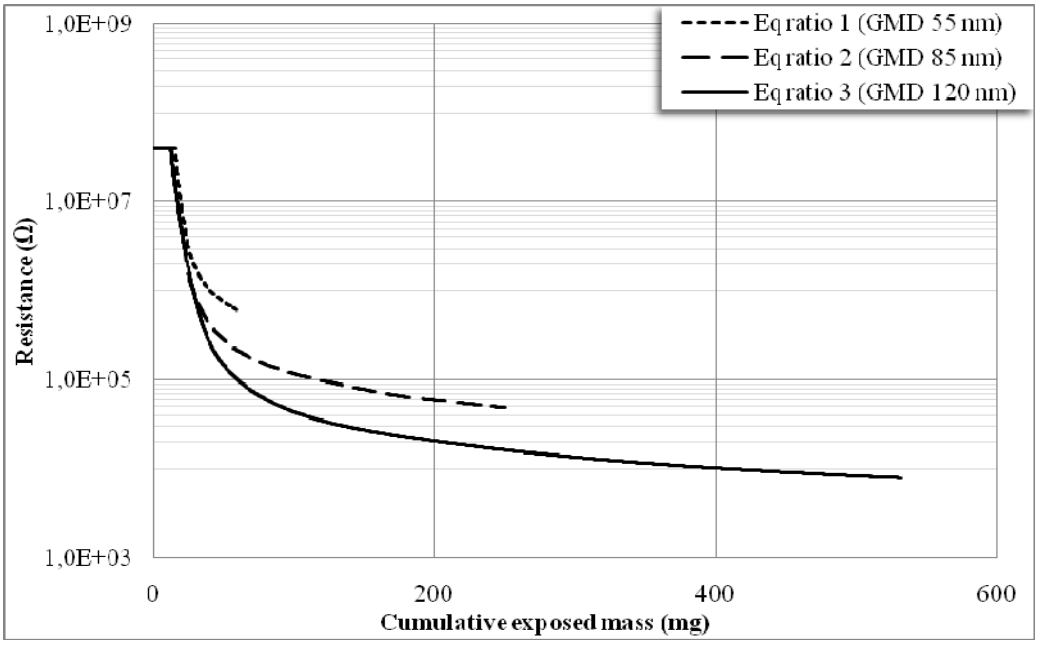


Figure 29: Experiments showing the cumulative mass of soot exposed and the resultant change in resistance for fresh sensors for 2h test by particles with GMDs of 55, 85 and 110 nm.

Sensor testing in diesel exhausts (LiU, VOLVO)

For these measurements sensors processed by Selmic Oy were used. Two test runs of the soot sensors in diesel exhausts have been performed. In the first run two sensor mountings were tested, perpendicular or parallel to the exhaust stream. The former gave much better result.

Sensors were positioned in the center of an exhaust pipe with sensing area facing the gas flow. Reference soot concentrations were measured using a Cambustion DMS500 Differential Mobility Spectrometer. The engine was driven in both steady-state and cyclic operation.

Resistance changes were observed during steady-state operation of the engine when the carbon particle concentration in the exhaust was 6-8 mg/m^3 as shown in Fig. 30. The decreases corresponded to soot collection on the electrodes when the sensor temperature was about 40°C lower than the exhaust. Increases occurred when the soot was burned away at elevated temperature.

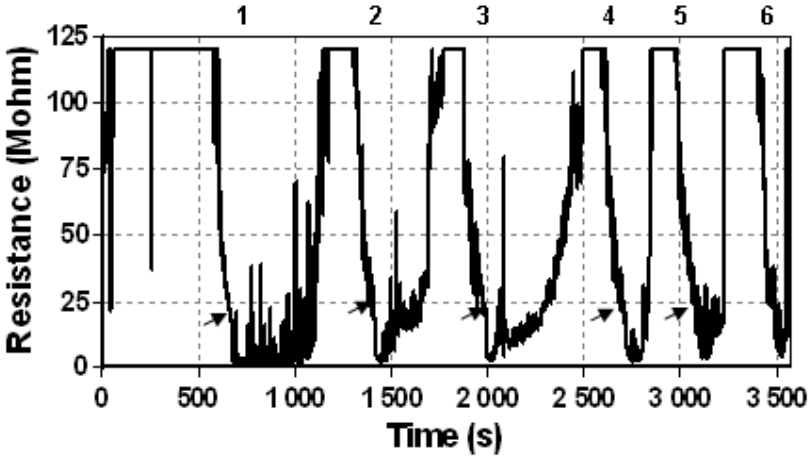


Figure 29: Consecutive resistance decreases(1-6): sensor temperature 162°C in exhaust gas containing 6-8 mg/m^3 and carbon particles at 202°C. Heater started at arrows raising the sensor temperature to 600°C and turned off at 120 MOhm.

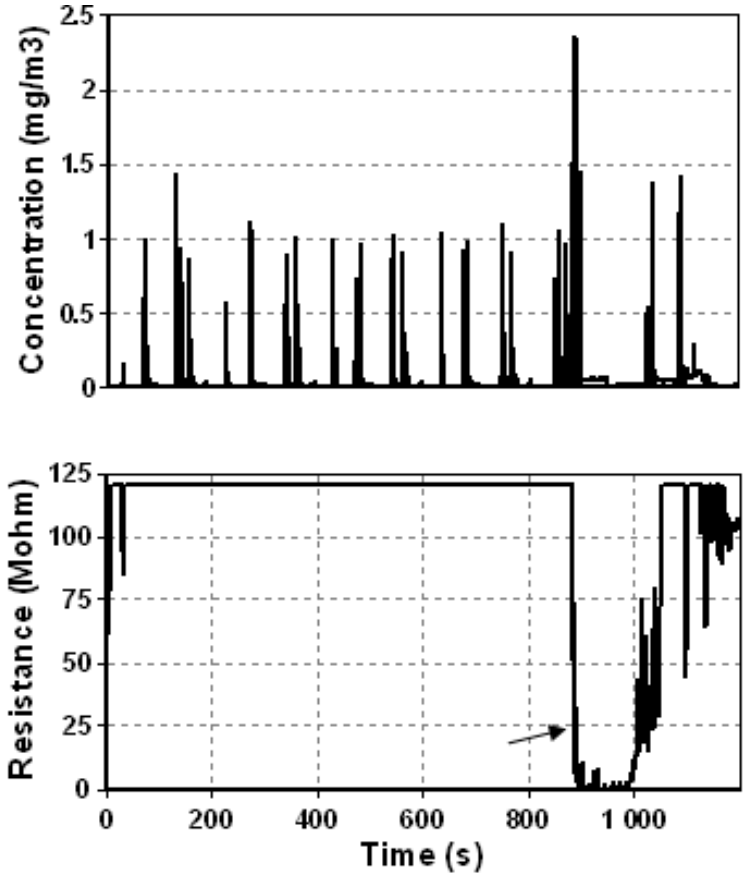


Figure 31: DMS carbon particle concentrations (upper) and sensor resistance (lower) during an NEDC driving cycle. Exhaust gas temperature: <150°C during the low speed first half of the cycle, 216°C during the high speed second half. Just before the drop in resistance of the sensor there was a 30% increase in the soot concentration of the exhaust over a period of 1 minute.

Driving cycles are important criteria for evaluating engine performance and pollution abatement procedures. The sensor responded toward the end of an NEDC (New European Driving Cycle, adopted in 2000 for cold start) cycle when higher speeds raised the exhaust temperature sufficiently to promote soot collection on the electrodes as shown in Fig. 31.

Cyclic operation with repeated collection (resistance decreases from 120 to 20 Mohm) and burn off steps yielded sensor response curves for different soot concentrations and exhaust gas/sensor temperature combinations at steady-state conditions for soot concentrations 5-20 mg/m^3. Measurements during driving cycles where it was impossible to follow the instantaneous spikes in soot production from acceleration/deceleration were directed towards relating the sensor resistance decreases to the accumulated soot emission from the New European Drive Cycle.

Conclusions

- We have qualitatively showed that thermophoresis is a viable sensor technology for soot detection with high sensitivity
- Soot can be produced by aerosol technology for testing of sensors in the laboratory and calibration of soot sensors
- A thermos concept for efficient cooling of the sensor surface has been demonstrated

Work to be continued in SootSens II

The SootSens II project was recently approved and the work in this project will focus on:

- Optimize the cooling of the sensor surface
- Quantification of the thermophoresis effect
- Fast and efficient regeneration of the sensor surface from soot
    - Catalytic sensing layers to lower the regeneration temperature
    - Testing other sensing layers
- Optimization of the aerosol technology calibration system
- Testing of transistor soot sensor device
- Electronics for cyclic operation of the sensor at two different temperatures
- Further testing in diesel exhausts

This development will considerably increase the interest from companies to commercialize the soot sensor. But we also want to improve the sensor technology with a future project in mind, see below.

Future development of nanoparticle sensors

A soot sensor project also proceeds within the VINN Excellence centre FunMat. The development in the FunMat centre regards advanced materials for the metal contacts of the soot sensor. Contacts are required, which are long term stable during cycling of the operation temperature between about 50 - 150°C below the diesel exhaust temperature

and a regeneration temperature of about 600°C. The MAX materials are identified as especially interesting for the development of these contacts.

During 2010 we intend to apply for a new project where the intention is to use the thermophoresis technology for a more general portable nanoparticle detector. The goal is to detect nanparticles according to size, shape, concentration and content, since all these parameters have shown to influence their adverse effect. This will be a much larger project, which will require extensive development of the technology. However, we will greatly benefit from the results and methodology built up in the soot sensor project as well as from the consortium of expertise which now functions well together. The consortium will also be increased in order to achieve the new goals.

Publications

Journal papers

D. Lutic, J. Pagels, R. Bjorklund, P. Josza, J. Visser, A. Grant, M. L. Johansson, P. Jasko, P.-E. Fägerman, M. Sanati, A. Lloyd Spetz, Detection of soot applying sensor device with thermophoretic deposition, Sensors, to be submitted

A. Malik, H. Abdulhamid, J. Pagels, J. Rissler, M. Lindskog, R. Bjorklund, P. Josza, J. Visser, A. Spetz, and M. Sanati, Generation of diesel like soot particles and thermophoretic deposition on resistivity soot sensor, AST Journal, submitted.

Conference contributions

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R. Bjorklund[1], M.L. Johansson[2], A. Grant[3], P. Jozsa[3], P.E Fägerman[4], J. Paaso[5], A. Larsson[6], D. Lutic[7], A. Lloyd Spetz, Resistance sensor based on thermophoresis for soot in diesel exhaust, MRS Spring meeting, submitted.

Patents

PCT/SE2008/050215 Method and arrangement for detecting particles, Doina Lutic, Anita Lloyd Spetz, Mehri Sanati, Jaco Visser, Peter Jozsa, patent filed by Volvo CC (2008)

20092713/2009.07.20, Lokal termisk styring, SINTEF IKT, Andreas Larsson

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The Nordic Innovation Centre initiates and finances activities that enhance innovation collaboration and develop and maintain a smoothly functioning market in the Nordic region.

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