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# Laser based measurement of water film thickness for the application in exhaust after-treatment processes



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#### ABSTRACT

In this work, an absorption based laser sensor for the investigation of dynamically changing liquid film thicknesses is developed and validated. For the wavelength selection of the single-ended, fibre-coupled, diode-laser sensor, near-infrared spectra of liquid water are measured at various thicknesses and temperatures. To reduce the influence of the film temperature, the evaluation is supported by a calibration procedure. Unknown film thicknesses of up to 440  $\mu$ m could then be measured. To adapt the sensor to particular boundary conditions of different systems, the single-ended setup can be changed to a transmissive configuration. The sensor is validated in both configurations against a commercially available chromatic confocal resonance (CHR) sensor. A comparison with respect to the CHR sensor leads to an accuracy of 2.8  $\mu$ m and a precision of 3.9% of the new laser based sensor.

### 1. Introduction

The formation and development of thin liquid films over time plays a major role in many industrial processes and in numerous research sectors, for example falling film evaporation and distillation, coating, printing, wetting or cleaning of solid surfaces. In mechanical seals and other lubricated components, the surveillance and measurement of the oil film is crucial for monitoring the aging and the functionality of components (Dwyer-Joyce et al., 2004; Reddyhoff et al., 2006). The knowledge of liquid film thickness is also relevant for the investigation of downsizing issues of internal combustion engines, like pool fires (Drake et al., 2003; Foucart et al., 1998; Porter et al., 2010), arising when gasoline impinges and forms a film on the piston head, where it burns under non-ideal conditions which leads to higher pollutant concentrations. For the numerical simulation of pool fires, the thickness of the gasoline film on the piston is an important input parameter. Similarly essential is the liquid film analysis in the selective catalytic reduction (SCR). SCR catalysis is used to reduce NO<sub>x</sub> concentration in Diesel exhaust gas. A 32.5% mass urea-water solution (UWS, AdBlue® or DEF) is injected into the exhaust gas, where it reacts to NH<sub>3</sub> and reduces NO<sub>x</sub> to N<sub>2</sub> and H<sub>2</sub>O. During transient driving conditions, the injected UWS can wet the wall and later carbonize to solid deposits. For a comprehensive understanding of the carbonisation and the reaction between wall, film and exhaust, not only the film thickness, but also the urea concentration in the film and the film temperature have to be determined (Grout et al., 2013; Dong et al., 2008).

Due to the fact that the investigation of film thickness of diverse liquids is that important, multiple techniques have been developed over the past few years (Alonso et al., 2010; Yang et al., 2010; Greszik et al., 2011; Pan et al., 2016; 2017). One of these techniques is based on non-invasive robust ultrasonic sensors (Dwyer-Joyce et al., 2004; Reddyhoff et al., 2006), where the transit time of short acoustic pulses is measured to determine the thickness of the liquid. Also, several optical and laser based techniques have been developed. For example, laser induced fluorescence (LIF) (Alonso et al., 2010; Schubring et al., 2010; Schulz et al., 2010; Schagen and Modigell, 2007), Raman spectroscopy (Greszik et al., 2011), internal reflection (Shedd and Newell, 1998) or direct absorption spectroscopy (Yang et al., 2017; Yang et al., 2010) have all been used to measure film thickness.

From this diverse selection of techniques currently only laser absorption spectroscopy has the potential to measure concentration, thickness and temperature of the film simultaneously. Here a proof of concept of an approach based on and Pan et al. (2016) to measure the thickness of free liquid films is presented. We extended the concept of Pan et al. with a calibration and validated this approach with a chromatic line scan sensor (CHR sensor) as an independent measurement

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technique. The calibration has the decisive advantage that temperature and concentration changes of UWS no longer influence the film thickness measurement. This is especially important, since the exhaust temperature in real SCR systems rises up to 500  $^{\circ}$ C, resulting in continuous heating of the films and a concentration increase of the urea. To be able to investigate the formation of AdBlue films in harsh environments such as SCR-systems in the future, the concept of direct absorption spectroscopy in combination with a calibration needs to be further developed. In a first step, we present a laser-based sensor that can measure water films without dependence on temperature.

#### 2. Instrument design

#### 2.1. Measurement technique

Laser absorption spectroscopy, utilizing a tuneable diode laser is based on the Beer-Lambert Law (Eq. (1)). Here, the attenuation of light is a function of the incident light intensity I<sub>0</sub>, the path length or film thickness  $\delta$ , the temperature-dependent line strength S(T), the number density n and the normalized line shape function  $\phi(\nu - \nu_c, T, p)$  at the central frequency  $\nu_{c}$ , which is a function of temperature (T) and pressure (p). To avoid wavelength independent influences and to ensure robustness, a second wavelength is used, yielding the Beer-Lambert Law given in Eq. (2) Rearranging Eq. (2) allows the thickness of the absorbing media to be calculated (Eq. (3)). In general, this requires detailed knowledge of the wavelength  $(\nu)$  dependent absorption coefficient and the absorption influencing species concentration. The problem at this point is, that the absorption coefficient varies as soon as the fluid under investigation changes for example due to concentration differences. Thus, measurement in transient systems such as SCR systems is not possible. In order to avoid this problem, the absorption coefficient is replaced by the calibration constants a and b (e.g. Fig. 7) determined by a previous calibration measurement. Hence a direct relation between measured intensities and liquid film thickness can be established (Eq. (4)). The calibration constants describe the straight line equation that connects the individual measuring points of different film thicknesses. "a" refers to the slope of the line and "b" is the offset of the line which refers to the roughness of the surface beneath the film. To minimize the influence of the surface quality, a flat gold mirrors is used as reflective target.

$$I(\nu) = I_0(\nu) \cdot \exp[-S(T) \cdot n \cdot \phi(\nu - \nu_c, T, p) \cdot \delta] = I_0(\nu) \cdot \exp[-\alpha(\nu, T) \cdot \delta]$$
(1)

$$\ln\left(\frac{I(\nu_l)}{I_0(\nu_l)}\right) - \ln\left(\frac{I(\nu_k)}{I_0(\nu_k)}\right) = \delta(\nu_l, \nu_k) [\alpha(\nu_k, T) - \alpha(\nu_l, T)]$$
(2)

$$\delta(\nu_{l},\nu_{k}) = \frac{\ln\left(\frac{I(\nu_{l})}{I_{0}(\nu_{l})}\right) - \ln\left(\frac{I(\nu_{k})}{I_{0}(\nu_{k})}\right)}{\left[\alpha(\nu_{k}, T) - \alpha(\nu_{l}, T)\right]}$$
(3)

$$\delta = a \cdot \ln \frac{I(\nu_1) \cdot I_0(\nu_k)}{I_0(\nu_1) \cdot I(\nu_k)} + b$$
(4)

Nonetheless, the calibration can be influenced by concentration and temperature changes for a certain wavelength combination ( $\nu_k$ ,  $\nu_l$ ). For this reason, the wavelength combination must be chosen carefully and the independence from temperature and concentration has to be demonstrated. For a proof of concept, the influence of solvents on the absorption can be neglected by measuring film thickness of pure water. Since film formation in SCR processes is associated with changes in urea concentrations, such an investigation is necessary in a subsequent step.

#### 2.2. Cell for wavelength selection and calibration

Prior to its operation under real process conditions, the wavelengths of the lasers must be selected and the sensor has to be calibrated. The wavelength selection is conducted based on FTIR (Fourier transform



**Fig. 1.** The calibration cell consists of a top (1), which is protected by an O-seal (2), a sapphire window ( $1^{\circ}$  wedged) (3), a calibrated spacer (4), a reflective target (which can be substituted by an additional window for FTIR measurements) (5). The cell body (6) can be heated with cartridges (7).

infrared) spectrometer measurements. The calibration cell design is suitable for measurements in transmission and in single-ended mode and compatible with the FTIR. For the calibration measurements in the single-ended configuration the cell is equipped with a reflective target. The cell shown in Fig. 1 consists of a window (3) and a reflective target (5). Between window and target, a metal spacer (4) is placed. The spacer defines the thickness of the liquid layer. For transmission measurements, the reflective target (5) can be replaced with a second window. The thickness of the spacers is manually measured with a micrometre with an accuracy of 1  $\mu$ m.

### 2.3. Wavelength selection

The selection of the wavelength for the liquid film thickness sensor is a central part of the measurement technique. For selecting the wavelength, the same cuvette as in the calibration measurements is used and the infra-red transmission spectrum of water is recorded. Fig. 2 shows the transmission spectra of pure water taken with an FTIR spectrometer (Bruker Vertex 80v) with liquid film thicknesses ranging from 0.05 mm to 0.44 mm (T = 294 K). The strongest absorption



Fig. 2. FTIR spectra of liquid H<sub>2</sub>O films with different thicknesses.

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