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Modelling and characterization of a multiparameter hot disk sensor for determination of fluid mixture ratios



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ABSTRACT

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1. Introduction

In many technical systems monitoring and control of binary fluid mixtures is important for correct operation. Often, low cost and robust sensors are required. One possible application can be seen in direct methanol fuel cells (DMFC) to enable the control of a constant methanol concentration in water of less than 4.2% (V/V) [1], depending on the currently required output power of the system. The exhaust after treatment of diesel engines using selective catalytic reduction (SCR) is another important application. Here, a mixture of 32.5% (W/W) urea dissolved in water (also known as AdBlue or diesel exhaust fluid, DEF) is injected in the exhaust stream where urea reacts at high temperatures with water to ammonia which reduces nitrogen oxide to nitrogen and water [2]. To ensure correct operation of the system a sensor is needed to monitor the urea concentration while a deception of the system using, e.g., only (salt-) water has to be prevented. Furthermore, a self-test of the sensor is desired to ensure correct operation.

Table 1 gives an overview of different physical properties of methanol/water-mixtures which can be used to determine their

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The mathematical modelling of a thermal sensor principle based on the transient hot disk method to measure the mixture ratio and flow rate of binary fluid mixtures simultaneously is presented, which helps to predict and understand the sensor response. Measurements of methanol/water-mixtures are shown with and without an applied fluid flow, while the measurement results are compared with the theoretical model. Using this sophisticated hot disk sensor and if no fluid flow is present, multiple thermal properties of the fluid mixture are determined, allowing a self-test of the sensor and the detection of a possible deception. The latter aspect is of interest especially for characterization of aqueous urea solutions used in selective catalytic reduction (SCR) systems to reduce exhaust emissions of diesel engines.

mixture ratio and their relative sensitivity. Electrical conductivity and relative permittivity show a very strong influence of the methanol concentration but they are also very sensitive to ions and dissolved CO_2 in the fluid mixture [9] which could be introduced by contaminants in the fuel cartridges or corrosion by formic acid that might remain as a by-product in the system [10]. Since viscosity also changes considerably with methanol concentration, a thermal sensor principle based on stationary vortices occurring behind a baffler placed in the fluid flow has been developed and successfully investigated [11] and is subject to a patent application [12]; however, viscosity is also strongly dependent on the temperature [4] making exact measurements in practical applications more demanding. In addition to these properties, the thermal properties show a strong sensitivity vs. changes of the methanol concentration. These can be measured using the hot disk method (see below) and their evaluation should be favourable compared to the measurement of other properties like density or speed of sound. Thus, the sensor principle presented in this work is based on the evaluation of thermal properties.

2. Sensor principle

The sensor principle that has been developed and successfully evaluated is based on the transient hot disk method [13], which is primarily used to thermally characterize solid materials. Here, a

Table 1

Relative sensitivity vs. changing methanol concentration of some physical properties for methanol/water-mixtures (at 25 $^\circ$ C).

| Property | Change in % per % (W/W) | References |
|---|---------------------------|------------|
| Electrical conductivity, σ | +5.1 to 10.9 ^a | [3] |
| Dynamic viscosity, η | +2.9 | [4] |
| Thermal diffusivity, α^{b} | -0.88 | |
| Thermal conductivity, λ | -0.87 | [5] |
| Relative permittivity, ϵ_r (at 0 Hz) | -0.55 | [6] |
| Thermal effusivity, b ^b | -0.43 | |
| Isobaric heat capacity, <i>c</i> _p | +0.18 | [7] |
| Density, ρ | -0.17 | [4] |
| Speed of sound, s | +0.11 | [8] |
| | | |

^a The cited reference does not specify the exact concentration notation.

^b Calculated from density, thermal conductivity and isobaric heat capacity.

small heater covered with the unknown binary mixture is heated with a short power pulse. If no fluid flow is present the temperature increase after a given time is affected by the thermal properties of the fluid, thus reflecting the mixture ratio if the two components in the mixture are known [14]. This sensor principle is well-known but has some limitations for the use in fluidic systems. First of all, if a fluid flow is present additional heat is dissipated from the heater due to forced convection, reducing the heater's temperature and thus prohibiting the measurement of the mixture ratio if the flow velocity is unknown. Furthermore, since only one parameter (the temperature of the heater) is measured, different fluids or fluid compositions can cause the same sensor response. Especially in the case of SCR systems a possible deception of the sensor by filling the DEF tank with an incorrect fluid must be detected to allow an on-board diagnosis of the exhaust aftertreatment system. Finally, when using only one measurement parameter a drift of the sensor cannot be detected.

To overcome these drawbacks, a sensor layout as shown in Fig. 1 has been realized, using one central heater with four-terminal sensing of its resistance and four surrounding temperature sensors to simultaneously measure the temperature increase of the heater and the spread of the induced temperature pulse. While the temperature increase is determined by the thermal effusivity $b = \sqrt{\lambda \rho c_p}$, the spread of the temperature pulse also depends on the thermal diffusivity $\alpha = \lambda/(\rho c_p)$. Thus, measuring both effusivity and diffusivity either allows an increase in resolution when determining binary mixtures, the detection of a possible sensor degradation or determination of ternary mixtures. Table 2 shows some rel-

Table 2

Important physical properties of the investigated solutions at $25\,^\circ$ C.

| | Water | Methanol/water, 10% (V/V) | Urea/water, 32.5% (W/W) |
|---|-------|------------------------------|----------------------------|
| λ (W/(mK)) | 0.61 | 0.55 | 0.57 |
| ρ (kg/m ³) | 997 | 983 | 1088 |
| $c_p (J/(gK))$ | 4.18 | 4.27 | 3.51 |
| α^{a} (10 ⁻⁸ m ² /s) | 14.6 | 13.1 | 14.9 |
| b^{a} (W $\sqrt{s}/(m^{2} K)$) | 1590 | 1519 | 1475 |
| References | [15] | [5,4,7] | [16] |

^a Calculated from λ , ρ and c_p .

evant properties of pure water and mixtures with methanol (10% (V/V)) and urea 32.5% (W/W), respectively, which will be used in the mathematical model shown later.

The flow velocity is measured independently using a modified pulsed wire anemometry approach [17]. Here, a heat pulse induced by the heater is transported downstream by the flow and can be measured with a temperature sensor placed behind the heater using the time-of-flight principle. The time required for the heat pulse to reach the sensor is inversely proportional to the flow velocity and nearly independent of the thermal properties of the fluid as will be shown later.

3. Experimental

The sensor has been realized with standard microtechnologies using a simple fabrication process to address low-cost applications. A 125 µm polyimide foil (Kapton HN, DuPont) was chosen as substrate because of the advantageous thermal properties (e.g., low thermal conductivity) and the excellent chemical resistance. Heater and temperature sensors are realized by sputtering a 500 nm aluminum layer on top of 40 nm titanium as adhesion promotion layer followed by wet etching with concentrated phosphoric acid at 50 °C. The achieved temperature coefficient of resistance for a reference temperature of $0 \,^{\circ}$ C is $3.25 \times 10^{-3} \,\text{K}^{-1}$. To passivate the aluminum structures an about 5 µm thick polyamide-imide layer (Durimide 32A, Fujifilm) is used. The whole process is described more precisely in [14]. Fig. 2 shows a picture of the realized sensor chip. Of course, platinum shows a higher resistivity and temperature coefficient of resistance and could be used instead of aluminum, also to achieve a higher chemical stability of the device.



Fig. 1. Layout of the realized sensors with central heater and surrounding temperature sensors [14].

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