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# FOULING MEASUREMENT USING A THERMAL-BASED MICROSYSTEM

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## Abstract

In process industries such as cooling systems, food and beverage and pulp and paper, fouling remains a complex and misunderstood phenomena reducing process efficiency (materiel lifespan, transfer limitation, environmental impact). The detection of fouling is of unquestionable scientific and economic interest. A thermal pulse analysis method developed by Neosens and based on INRA patent is presented. In this article, solutions using MEMS technologies are reported. Silicon implanted resistors and metallic resistances are being investigated and compared in term of sensitivity and measuring range.

**Keywords:** *fouling, sensor, thermal measurement, MEMS*

## I- Introduction

In the process industry, fouling stands as a complex and misunderstood phenomena. Heating, holding and cooling operations are carried out in continuous or batch processes and fouling occurs in the equipment with a large widespread of kinetics (from minutes up to years) and thickness range (from micrometers up to centimeters). The continuous control and understanding of fouling phenomena is of evident industrial interest: reduction of process performances, energetic consumption and water management.

Various devices have been reported in literature including rheological, electrical, chemical, mechanical, optical, sonic, ultrasonic and thermal methods. They exhibit specificities, advantages and disadvantages [1] but most of them appear to be complex in their design or their implementation.

A macro-sensor based on a thermal pulse analysis method, based on INRA patent [2], has been developed by Neosens company. The sensor measures the fouling status of equipment and stands as a real time, local and online measurement. The

use of microfabrication and microelectronics techniques enables the realization of sensor functionalities on a chip and the improvement of previous sensor performances (sensitivity and response time). In this paper, solutions using MEMS technologies are reported. In a first step, principle of sensor is presented. In a second step, chip design and working mode are detailed.

## II- Investigated methods

Three different methods (electrochemical current, electrical conductivity and heat transfer) have been investigated at Neosens company to monitor fouling phenomena. The deposit at the sensor surface may be seen either a chemical barrier, an electrical or a thermal resistance.

First method was dedicated to current measurement on a working electrode thanks to a tracer. When a deposit is found on the electrode, the tracer reduction is limited. However, this measurement is highly polluted by mass transfer and chemical composition and deposit structure.

Second method is based on electrical measurements and is an adaptation at the micro scale of a geophysical prospection technique used to determine the different layers composing the soil, known as Schlumberger technique. In this case, the increase of fouling thickness at the microsensor surface is linked to the variation of resistivity between two microelectrodes. However, this device exhibits two main deficiencies: (i) a measurement of deposit thickness remains difficult through electrodes located on a flat surface (electrodes design, 3D measurements) and (ii) the electrical resistivity of the bulk and the deposit must be known to estimate fouling thickness.

The third method, exposed here, uses the thermal-physical properties of generated deposit by determining its thermal resistance. So, if a thermal power is dissipated through the sensor, the temperature of its surface under the deposit can be correlated to the fouling thickness. Thermal sensor, used as a local deposit device, was compared to

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conventional global techniques (pressure drop, overall heat transfer coefficient) and validated as an accurate method. Fouling and cleaning monitoring was demonstrated at laboratory and industrial scale [3, 4].

### III- Fouling sensor and operating mode

The thermal sensor was made of a platinum resistance and two thermocouples (type K). The platinum probe acted as a heating element. The first thermocouple measures the wall temperature  $T_w$  at the sensor-product or deposit interface. The second thermocouple measures the bulk temperature  $T_b$  (Fig.1). The platinum wire was connected to a direct current generator (0–50 mA). The electric current (I) and potential (U) applied to the standard resistance were recorded and the heat power (P, 0–250 mW), the heat flux (j, 0–300 W/m<sup>2</sup>), and the electric resistance calculated. Each sensor signal (I, U,  $T_w$  and  $T_b$ ) was converted and recorded on a computer with specific acquisition software. Under clean conditions, a moderate heat flux (0 to 300W/m<sup>2</sup>) and permanent flow regime is applied. In these conditions, a thermally and hydrodynamically developing flow around the sensor is assumed, then the local heat transfer coefficient, h, reaches an important value (>1000 W.m<sup>2</sup>.K<sup>-1</sup>). It induces that the temperature difference between wall and bulk tends towards 0+ (in agreement with the precision of thermocouples). An overview of the heat transfer in the probe and the product leads to a calculation of thermal resistance of deposit or thickness if its thermal conductivity is known.

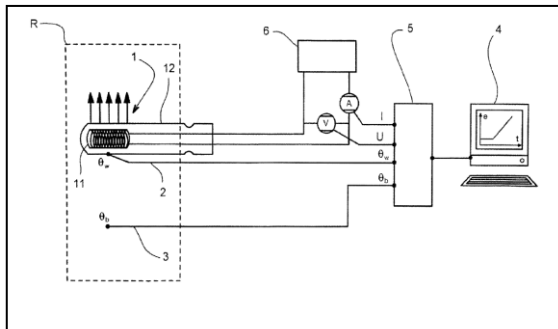


Figure 1: Principle of fouling sensor [2]

Heat generation inside the sensor (cylindrical shape) is assumed. In the platinum probe, we consider the heat transfer balance in cylindrical coordinates with the following equation:

$$\rho.C.\frac{dT}{dt} = \lambda \nabla^2 T + q \quad (1)$$

P, C,  $\lambda$  and q are mass density (kg/m<sup>3</sup>), specific heat (J/kg/K), thermal conductivity (W/(m.K)) and generated heat (W/m<sup>3</sup>) respectively. Geometric and thermal simplifications lead to the simplification of this formula in the platinum wire submitted to the Joule effect and stainless steel sheathes. Flux and power were determined with geometrical dimension and electrical parameters (I, U). Heat transfer may be formulated for a constant flux and in a system in which a deposit layer may exist. The temperature difference between wall and bulk,  $\Delta T$ , is formulated and correlated to fouling thickness e:

$$\Delta T = T_w - T_b = \frac{P}{2 \cdot \pi \cdot L} \cdot \left( \frac{1}{h \cdot (r+e)} + \frac{\ln\left(1 + \frac{e}{r}\right)}{\lambda_d} \right) \quad (2)$$

Parameters L, r and  $\lambda_d$  are sensor length (m), radius (m) and deposit thermal conductivity (W/(m.K)).

### IV- Miniaturized sensor principles

Miniaturization aims at reaching three main objectives: (i) early fouling detection (ii) small thermal inertia and then reduced response time (iii) decrease of unitary costs.

Silicon chip is composed of a heating element in order to dissipate a known heat flux towards the bulk and at least one sensor to perform temperature measurement at the chip/process interface. Chip design enables flux control: direction and surface to be crossed. The temperature sensor measures the surface temperature  $T_w$  of the chip that is a function of deposit thickness. Heating of the sensor can be constant or follow different phases: active or inactive. If the heating is constant, another thermal sensor is necessary in order to achieve bulk temperature  $T_b$  measurements. This thermometer must be insulated from the heating source. By comparing temperatures measured and recorded, it is possible to have information about the fouling status of equipment. If the heating is alternative, bulk temperature is measured during inactive phases when the heat flux is equal to zero.

### V- Performances improvement

From these functional principles, we realize micro-fabricated silicon-based components. Several aspects can be improved: precision of temperature measurement, detection range, size of the sensor and response time (Table 1).

Table 1: Comparison of specifications between macro-sensor and microsensor established for a theoretical deposit having a thermal conductivity  $\lambda_d=0,6W/(m.K)$

Technical specifications	Macro-sensor	Microsensor
Temp. precision (°C)	0,3	< 0,1
Thickness precision (µm)	10	1
Range (µm)	10-10000	1-1000
Response time (s)	20	< 5
Sensor surface (mm <sup>2</sup> )	100	10

We look for a component with a low resistivity, a high thermal coefficient of temperature (TCR) to improve the sensitivity and, in the best way, a linear variation in the interval 0-200°C. Two types of sensors are investigated:

- The first one is based on ion implantation process. Implantation doses and energies enable the choice of a large widespread of resistance values, through sheet resistance, and TCR [5-10]. Phosphorus implantation gives higher TCR up to 8000ppm/°C than boron implantation (Table 2). Another advantage of ionic implantation is the possibility to obtain resistors having high resistivity and then a very small size.

Table 2: Expected performances of N-doped and P-doped resistors

Dose [cm <sup>-2</sup> ]	N-doping (Phosphorus)			P-doping (Boron)		
	Resistivity [Ω.cm]	R□ [Ω/□]	TCR [ppm/°C]	Resistivity [Ω.cm]	R□ [Ω/□]	TCR [ppm/°C]
10 <sup>11</sup>	4		8000	15		
10 <sup>12</sup>	0,5	> 5000	7000	1		
10 <sup>13</sup>	0,1	600 - 1000	5000	0,2	5000	3000 - 5000
10 <sup>14</sup>	0,03	100 - 300	0	0,03	600 - 1000	250
10 <sup>15</sup>	< 0,01	15 - 40	< 2000	0,01	100	700 - 1500

- Another option is the use of metallic resistances, using Nickel as a gauge, to measure temperature variations. In this case, TCR is lower than for ion implantation but Nickel exhibits an acceptable TCR of 7000ppm/°C. The main advantage is the linear response R(T) that simplifies signal treatment between 0°C and 200°C. However, low resistivity of Ni leads to an enlargement of resistances and then the size of the chip.

## VI- Conclusion

We have developed a thermal-based sensor able to monitor fouling phenomena in real-time and continuous mode. MEMS technologies enable sensor miniaturization with a similar working mode. Small size of the chip leads to cost reduction

and performances improvement: better sensitivity, limit of quantification and response time. Today, two techniques for heating and measurement resistances realization are investigated. Implanted phosphorus-doped or metallic (Ni) resistances show a good TCR up to 7000ppm/°C but the lower resistivity of n-doped resistances makes the chip smaller resulting in the early fouling detection.

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