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Procedia Engineering 120 (2015) 150 – 153

**Procedia  
Engineering**[www.elsevier.com/locate/procedia](http://www.elsevier.com/locate/procedia)

EUROSENSORS 2015

# Field-effect based pH sensors for cutting fluid condition monitoring

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## Abstract

Field-effect based Electrolyte Insulator Semiconductor (EIS) structures were used to monitor the degradation of cutting fluids under laboratory conditions. An initial set of experiments was carried out to evaluate the sensors' performance in terms of their sensitivity to degradation-induced pH changes. The results presented here show that the employed sensors are capable of differentiating between pristine and degraded cutting fluids as well as intermediate mixtures. We therefore suggest field-effect based pH sensors as promising candidates to be part of sensor arrays capable of delivering real-time information about the overall state of cutting fluids, which would enable a more cost efficient and environmental friendly fluid management.

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Peer-review under responsibility of the organizing committee of EUROSENSORS 2015

*Keywords:* pH sensing; cutting fluid monitoring; field-effect based sensors; water-based cutting fluid emulsion

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## 1. Introduction

Cutting fluids play a critical role in most metalworking processes. Their main functions are to remove chips and fines that occur during the process, as well as to cool, lubricate, and protect the machine, the machined part and the tool against corrosion [1, 2]. These fluids are divided in non-water-based and water-based subgroups of which the water-based fluids have the bigger market share [3]. During operation cutting fluids undergo changes in composition through contamination by metal chips and fines, tramp oil and dissolved salts. At the same time important components are removed from the fluid through evaporation or decomposition. In water-based fluids microorganisms introduced from the machines surroundings are an additional problem, since their metabolism decomposes organic components of the fluid to acidic products which increases the corrosion probability of the machine. In order to maintain the optimum performance of the cutting fluid its properties have to be kept within certain limits. This is achieved through the addition of water and chemicals to balance their composition during operation. Hence, close monitoring of the fluid with appropriate sensors in order to determine its degradation stage is required. For instance, parameters such as conductivity, solid fraction and oil concentration are accessible through physical sensors using electrical, acoustical or optical measuring principles. Chemical sensors can provide viable information about the concentration of ions and organic substances. A very important parameter to assess the overall state of cutting fluids that is accessible through chemical sensors is the pH value. All cutting fluids are designed to operate in the alkaline range at a pH between 8.0 and 9.5, because alkalinity helps

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to control corrosion and minimizes growth of microorganisms. As mentioned before, decreasing pH in cutting fluids is usually the result of bacterial activity and can serve as an indicator for the overall state of the fluid.

pH sensors based on potentiometric and optical principles are commercially available and are used today to monitor the acidity of cutting fluids during operation [2]. However, current solutions employ fragile sensors such as glass electrodes and are operated manually and therefore do not offer continuous information. Alternative sensor designs that are sturdy, require little maintenance and can be integrated into sensor arrays delivering real-time information about the degradation state, would help optimizing maintenance of cutting fluids and hence extend their lifespan. This in turn would reduce costs and minimize the impact on the environment. Field-effect based pH sensors [4] offer these features but have not yet been systematically tested for applications in cutting fluids. In this work, results of initial experiments with Electrolyte Insulator Semiconductor (EIS) structures (see inset in Figure 1 left) in cutting fluids are reported. In these devices the interaction of the electrolyte, e.g. cutting fluids, with the insulator, leads to a surface potential which is proportional to the pH value of the electrolyte. This surface potential then causes a shift in Capacitance/Voltage (C/V) curves measured over the EIS structure as shown in Figure 1 (left). Therefore the required substrate bias to reach a certain capacitance can be used as the output signal of pH sensors based on this principle.

## 2. Experimental

### 2.1. Materials and measurement setup

The sensing material in this study was a 160 nm thin  $Ta_2O_5$  layer deposited on an n-doped silicon substrate with a 55 nm thermally grown oxide layer in between. The back side contact is formed by a thin layer of aluminium. The structures were obtained commercially and the exact deposition parameters are unknown. Three 1.5 cm x 1.5 cm pieces were investigated and are henceforth labeled as sensor #1, #2 and #3. A water-based cutting fluid emulsion was used as the analyte. ZF Friedrichshafen AG provided a sample of pristine fluid and a sample that they deemed unusable after prolonged use. From these two solutions a third one comprising of a 1:1 mixture with respect to volume was prepared. Absolute pH values were determined with a freshly calibrated commercial glass electrode.

The employed measuring setup is depicted in Figure 1 (right). The EIS structure is housed in a measuring cell with ports for the required fluidic and electrical connections. The measuring circuit is formed by a conventional Ag/AgCl reference electrode which provides the front side contact through the cutting fluid and a copper plate that is pressed against the conducting back of the EIS structure. Both electrodes are connected through shielded cables to a LCR meter (HP 4284A) which records the C/V curves. Fluid handling, including rinsing steps, was performed by syringe pumps. The cutting fluid samples were kept at 30 °C during the measurements by means of a thermostat. The entire setup is automated and controlled by a computer running a customized LabVIEW program.

### 2.2. Measurement routines

In a first experiment (experiment A) the performance and stability of the EIS sensors in cutting fluids were evaluated. For this purpose the measuring cell was filled with cutting fluid and after ten minutes a C/V curve was recorded. After a wait time of one minute, another C/V curve was taken and this procedure was repeated three times for a total of five curves. These measurements were performed with the pristine fluid first, then with the intermediate mixture and finally with the degraded sample. Between the fluid changes the cell was rinsed with deionized (DI) water.

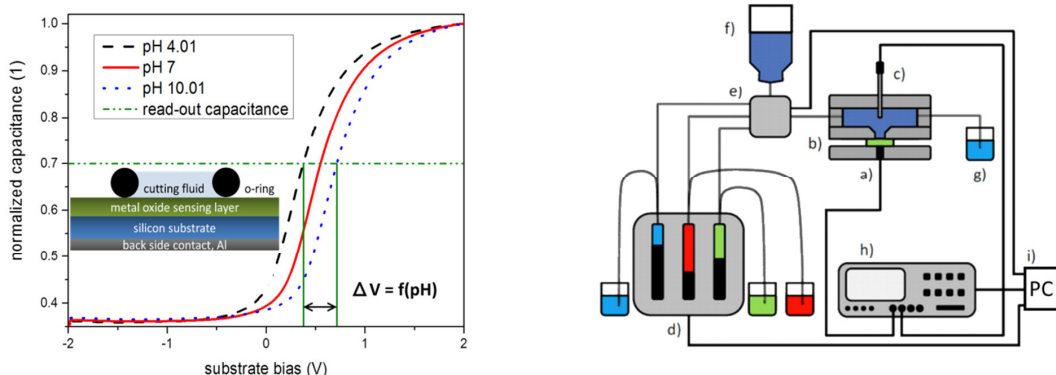


Figure 1. **Left:** Exemplary C/V curves recorded at different pH values. The proportional shift of the curves is clearly visible. The inset shows a schematic of the used EIS structures. **Right:** Schematic of the measurement setup with a) EIS structure, b) measuring cell, c) Ag/AgCl reference electrode, d) temperature controlled sample delivery system, e) valve, f) DI-water for purging, g) waste container, h) capacitance meter, i) computer for controlling and data acquisition.

A second experiment (experiment B) was conducted to see how the sensors reacted to repeated changes of the analyzed fluids. The measuring cell was firstly filled with the pristine sample and after five minutes a C/V curve was recorded. The cell was then rinsed with DI water, filled with the next sample and after additional five minutes the next C/V curve was measured. This procedure was repeated in the following sequence: pristine – intermediate – pristine – degraded – pristine – intermediate – pristine – degraded.

### 3. Results and discussion

#### 3.1. Experiment A: Performance and signal stability of EIS structures in cutting fluids

The results of experiment A are summarized in Table 1 and the data is plotted in Figure 2 (left). As expected it can be seen that the pH value decreases as the cutting fluid degrades. The overall difference between the pristine and the degraded sample is about one pH increment. All three analyzed EIS structures show a linear response to the varying pH values of the samples with different degradation stages. Furthermore the sensor output is stable over the five separate measurements resulting in a small standard deviation of 1 mV which indicates stability of the employed sensors, at least over a short time period. Long term drift has to be evaluated in future experiments. A linear fit was performed on the plotted data in Figure 2 (left) and the slope is reported as the calculated sensitivity. Since the overall change in pH is only one increment the accuracy of this value is questionable. Nevertheless the obtained values are reasonably close to the theoretical Nernst limit of 60.15 mV/pH at 30 °C. The differences in sensitivity between the three sensors and especially the observable offset of sensor #2 in Figure 2 (left) are most likely the result of slight geometrical deviations during the manual assembly of the measuring cell and related electrical contact issues. All in all it can be stated that the three cutting fluid samples can be clearly distinguished with the proposed method and the tested sensors.

Table 1. Results of Experiment A. The reported data is the mean of five separate measurements and the respective standard deviation. The given calculated sensitivity is the slope of a linear fit through the three data points.

	Pristine fluid	Intermediate fluid (1:1 vol)	Degraded fluid	
pH	9.26 ± 0.01	8.79 ± 0.01	8.25 ± 0.01	calculated sensitivity
Output signal sensor #1	954 ± 1 mV	925 ± 1 mV	894 ± 1 mV	58.2 mV/pH, sensor #1
Output signal sensor #2	936 ± 1 mV	912 ± 1 mV	881 ± 1 mV	52.9 mV/pH, sensor #2
Output signal sensor #3	954 ± 1 mV	927 ± 1 mV	898 ± 3 mV	55.1 mV/pH, sensor #3

#### 3.2. Experiment B: Sensor response to alternating cutting fluid changes of different degradation stage

The goal of experiment B was to evaluate if the EIS structures show hysteresis when the fluid composition, and therefore its pH value, is changed back and forth between the three analyzed samples. Furthermore in a prospective industry-ready design this procedure could be used to calibrate the sensor since it is not necessarily the absolute pH value that is of interest but rather the change in pH compared to the initial value. Pumping a small volume of pristine cutting fluid into the measurement cell and recording the sensor output to which the following measurement of the actual fluid in use is then compared, would eliminate potential influences of long term sensor drift on the output signal.

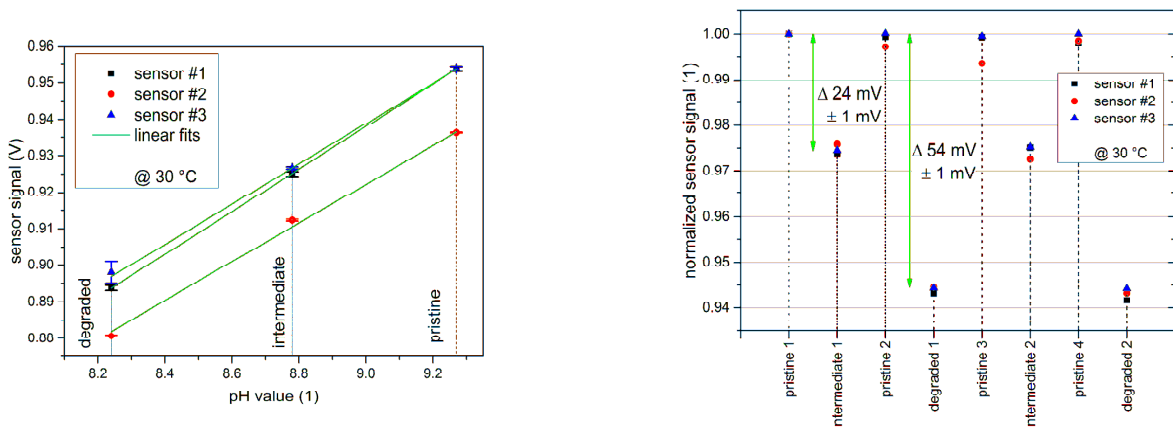


Figure 2. Left: Output signal of the three sensors for the three analyzed degradation stages of cutting fluid. Right: Normalized output signal for a series of measurements with alternation of the cutting fluid samples.

As shown in Figure 2 (right) the described procedure works well with all three sensors tested in this work. The data was normalized to the observed value of the first measurement in the pristine sample. This facilitates the analysis of the relative change in the sensor signal, which is about 24 mV for the change from pristine to the intermediate sample and roughly 54 mV for the change from pristine to fully degraded sample. These values are in good agreement with those obtained in experiment A and are sufficiently large to clearly distinguish between the three degradation stages.

#### 4. Summary and conclusions

The data presented in this work shows that field-effect based EIS structures can be used to monitor the pH value in water-based cutting fluids. Three degradation stages ranging from pristine to fully degraded fluid were reliably distinguished based on the sensor output. Since the pH value decreases with progressing degradation of the organic components of cutting fluids, these sensors could provide viable additional information about the overall state of cutting fluids if integrated into monitoring tools for other parameters such as conductivity and concentration. This integration requires sturdy and preferably miniaturized sensor solutions. Therefore the next step in our work is to replace the conventional Ag/AgCl reference electrode with an all-solid-state alternative and the physical combination of both electrodes on one substrate. Further future work includes the evaluation of other field-effect based pH sensors such as the extended-gate field-effect transistor (EGFET), the integration of the read-out electronics into the sensor and finally field tests on metalworking machines under industrial environments.

#### Acknowledgements

The authors thank ZF Friedrichshafen AG for providing the cutting fluid samples. The LabVIEW routine for controlling the measurement setup was written by Markus Haverkamp (IESM, TU Freiberg).

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