Using Bias Superposition to Test A Thick Film Conductance Sensor

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Abstract. A novel on-line monitoring technique for a range of MEMS and integrated sensor systems is presented based on the injection of a test stimuli into the bias structure of transducer functions. The technique “Bias Superposition” utilises both signal injection and signal extraction techniques to achieve an indication of structural integrity of the transducer and interface. The technique has been successfully applied to a thick film conductance sensor.

1. Introduction

Advances in monolithic systems integration is stimulating new applications for single and multi-chip smart sensors. Many of these new systems are highly complex, have limited test access and require a known value of the measurand signal for calibration and test of the unit. Implementation of Built-In Self-Test (BIST) techniques, which address both production and on-line test problems within these systems, has recently attracted considerable interest. However, the realisation of solutions is difficult as it is normally necessary to create a structure that doesn’t require a calibrated measurand source and is capable of detecting problems related to cross sensitivities, drift and long term stability in addition to traditional hard faults. In addition many integrated smart sensors are designed for harsh environments that add additional robustness requirements. In this paper, application of a novel on-line monitoring technique that is based on signal injection into the bias structure is presented. This will enable the sensor to perform both measurement of the physical parameter and generate information that supports the characterisation of its transfer function.

One of the most important parameters in water quality measurement is its conductivity. A form of conductivity sensor is incorporated into the Aqua 7 water quality sensor manufactured by Oxley Developments [1] for the water industry. The device manufactured using thick film technology is intended to measure drinking water quality. The electronics measures potentials and currents generated. Each of sensor’s responses are linearised by using the calibration coefficients stored on the memory chip encapsulated on the disc. The sensing elements on the Aqua 7 measure Redox, pH, Conductance, Free Chlorine, Total Chlorine, Ozone and Dissolved Oxygen as shown in Figure 1.

Figure 1 Assembly of the Aqua 7 sensor

The sensor head has an expected lifetime of 2 months and it would be advantageous to have a self-testing capability to notify the user/supervisory system when the device is out of calibration ( fouled).
This paper is organised as follows: In section 2 shows the thick film conductance sensor is introduced. Section 3 shows the application bias superposition to the conductivity sensor. Section 4 shows the results obtained and the paper is concluded in Section 5.

1.1. Bias Superposition Theory
A wide range of sensing systems are typically configured into an architecture where an electronic stimulus (bias) is applied to the sensor element to enable transduction of a physical stimuli. This signal is then fed into an analogue interface for signal conditioning (filtering, buffering etc) before being converted into the digital domain. See Figure 2 Typical Sensor interface

The output signal of the sensor is usually non-ideal and requires calibration and compensation to ensure it is directly proportional to the physical parameter being sensed. The Bias Superposition technique extracts additional information about the sensor function from information extracted from a test signal superimposed onto the sensor's bias structure. To enable on-line application, the test stimuli generated needs to have frequency content that is significantly different from the bandwidth of the system to enable filtering of the test response from either suitable nodes in the interface or the system output.

2. Thick Film Conductivity Sensor

2.1. Application
Conductivity sensors are crucial to many uFluidic applications including water quality sensing. It is of interest to embed these devices into systems that include readout and signal processing capability. In many applications, continuous monitoring of the integrity of the detectors is important for maintenance flagging and in some cases safety. This section applies the bias superposition to a device implemented in thick film technology.

2.2. Electrolytic Conductivity
The electrolytic conductivity \( \kappa \) is defined as the conductance between a pair of electrodes [2] which have an area of 1 cm\(^2\) and a distance apart of 1 cm (d). The unit is S/cm or S/m.

\[
\kappa = Gk = \frac{k\xi}{R} \quad (1)
\]

Where \( R \) is the measured solution resistance, and \( k \) is a constant which specifies the electrode pair. \( R \) needs to be found by calibrating the sensors against ‘standard solutions’, which have known concentration \( c \) and conductivity \( \kappa \) [3].

2.3. Conductivity Sensor Cell
The target sensor is a thick film device that uses two gold electrodes with active surface area of 10\( \mu \)m by 300\( \mu \)m on an Alumina substrate (Figure 3 (A)). The sensors were printed using thick film technology. The micro-channel was created using an ARG laser to expose the sidewall of the printed gold, thus creating the electrode structure. Figure 3 (B) shows two conductance sensors on a single substrate.
The advantages of using microelectrodes includes improvement to the sensor's accuracy and sensitivity by reducing the dielectric constant \( k \). \( k \) is required to be kept as small as possible. Conventionally it has been limited by the dimension of the sensor cells; however, by using micro processing techniques, it is possible now to achieve a fairly low \( k \) value in a small space. Use of microelectrodes also has less of an effect on the working environment and avoids changing the properties of the solution under measurement.

2.4. Equivalent Circuit

In solution conductivity measurements there are undesired effects e.g. hydration and polarization. There is also resistance introduced by the substrate-electrode interface and the connecting cable. The equivalent circuit is shown in Figure 4.

![Equivalent circuit diagram](image)

**Figure 4 Equivalent circuit**

R1/C1: **Contacts** – Electrode to solution interface

R2/C2: **Grain boundaries** – Electrode surface is not ideally flat and there are troughs and peaks on a micro scale.

R3/C3: **Polarization** effect due to a chemical change of electrode surface.

Rx: **Resistance** of the solution.

R5: **Resistance** of the substrate.

C: **Capacitance** due to electrode geometry.

In order to measure the solution resistance \( R_x \), a sufficiently high measurement frequency needs to be selected to shunt the undesired impedances of R1/C1 and R2/C2 and avoid electrolysis. In this design this frequency is 10KHz. When a step function is applied to the sensor the effects of R1/C1 and R2/C2 are observed in the sensor output waveform as a complex exponential fall. It is the analysis of this exponential fall time that provides information on the condition of the electrode-to-solution interface and hence to condition of the electrodes.

3. Application of Bias Superposition

An AC waveform consisting of the functional 10KHz signal is comprised with a low frequency 10Hz test signal to activate the transient response is used as a bias to the sensor. This bias allows the
simultaneous measurement of conductance and extraction of information regarding the conductance sensor properties. The bias waveform is shown in Figure 5. In this case it is not necessary to modify the physical architecture of the sensor.

**Figure 5** One period of the conductance sensor bias waveform

3.1. On-line Monitoring

Figure 6 (A) shows the normalized RC (network) response under different solution concentrations when a low frequency square wave has been superimposed onto the electrode bias. The RC time constant $\tau$ is defined as the time needed for the RC response to reach one third of its original value (compared to its value at $t=\infty$) after a step input is applied. Figure 6 (B) shows the normalised time constants measured by the sensor with $d=100\mu m$ for different salt (KCl) concentrations $c$.

![Figure 6](image)

(A) Normalized low frequency RC response & (B) Fit $\ln(\tau)$ to $\sqrt{c}$.

It is clear that there is a relationship between $c$ and $\tau$. It is clear from this plot that it is possible to use the time constant as a second method for determining the conductance of the solution. If both the low frequency and high frequency measurements produce the same conductance measurement then the sensor is working correctly. If there is a discrepancy between the two measurements then there must be a change in the electrode-to-solution interface. The validation algorithm is shown below:

1) The sensor is characterized by using the high frequency response $R' = f_h(c)$ and the low frequency RC response $\tau = f_s(c)$.

2) When the sensor is on-line it will measure the solution resistance $R_s'$ and the time constant $\tau$.

3) By rearranging $\tau = f_s(c)$ the concentration of ions $c_s$ is calculated. A second resistance $R''$ for the solution is calculated by substituting $c_s$ into the high frequency characteristic equation $R'' = f_h(c_s)$.

4) The measurement $R_s$ can be evaluated by comparing $R_s'$ with $R''$.

3.2. Experiment and Data Analysis

For this experiment two different fault models were developed:
1) Physical defects were added to the active area of the electrodes. In normal operating conditions they can be caused by improper deliver/storage methods or by force (crashes, pressure). To simulate this model, glue has been used to cover part of the electrodes.

2) Oxidation: This fault is caused by changes in the materials' properties. This mechanism has been induced by immersing the sensors in an acid solution for several minutes to become oxidized.

The manufactured sensors were calibrated using 15 different concentrations of KCl ranging from 0.001mol/L to 0.035mol/L, the relative conductivity range being 140µS to 4000µS.

4. Results

A comparison of \( R_x \) and \( \tau \) response between the calibration and the failed physically defective model are shown in Figure 7 (A) and (B) respectively.

![Figure 7](image1.png)

**Figure 7 (A) Measured resistance and (B) \( \tau \) response Vs Concentration KCl of sensor 25**

The validated status returned by the MATLAB function that compares \( R_x' \) with \( R_x'' \) to see if the difference is greater than the chi-square of probable values of \( R_x \) [4] see Table 1.

| State | \( R' \) | \( |R-R'| \) | \( \varphi \) |
|-------|--------|----------|------|
| 0     | 1.8017 | 1.3203   | 0.64058 |
| 1     | 1.686  | 1.2046   | 0.4307  |
| 2     | 1.4696 | 1.0129   | 0.28107 |

*0=healthy, 1=failed, 2=failed and \( \tau \) out of range

The response of the oxidized sensor is shown in Figure 8.

![Figure 8](image2.png)

**Figure 8 (A) Measured resistance and (B) \( \tau \) response Vs Concentration KCl of sensor 26**
From Table 2 it is clear that the function successfully distinguishes between the failed measurements and the correct one.

| State* | R' | [R-R'] | dR |
|--------|----|--------|----|
| 0      | 0  | 0      | 0  |
| 1      | 0.41066 | 0.91184 | 0.15578 |
| 1      | 0.53866 | 0.71754 | 0.068016 |
| 1      | 0.61072 | 0.43019 | 0.06911 |
| 0      | 0.50952 | 0.32096 | 0.099211 |
| 1      | 0.33026 | 0.16494 | 0.12311 |
| 0      | 0.56268 | 0.11016 | 0.12611 |
| 0      | 0.43214 | 0.14202 | 0.2338 |
| 0      | 0.66649 | 0.97376 | 0.00708 |
| 0      | 0.41077 | 0.043929 | 0.12547 |
| 0      | 0.38879 | 0.010726 | 0.069999 |
| 0      | 0.36616 | 0.019929 | 0.116833 |
| 0      | 0.29695 | 0.017098 | 0.055262 |
| 0      | 0.25008 | 0.004912 | 0.053776 |

*0=healthy, 1=failed, 2=failed and τ out of range

4.1. Discussion
By applying the combined waveform shown in Figure 9 both a high frequency measurement and a low frequency characterisation (step response) can be performed. The two measurements taken should, in a working device, yield the same effective resistance \( R'_s = R'_x \). By comparison of the measured resistance and \( τ \) (Figures 7&8) it is clear to see that once the network (RC) response is changed (due to alteration of the physical properties of the electrodes) \( R'_s \) will no longer equal \( R'_x \) thus change in the conductance sensor properties are detected.

5. Conclusion
The concept of Bias Superposition has been presented as a method for on-line monitoring of integrated sensors. This technique has been successfully applied to a conductance sensor fabricated using thick films technology. This has been achieved by using the RC response as a tool for self-validating the conductivity sensor, it has been proved by experiments that the validating function successfully distinguishes between the correct and faulty configurations of the sensor.

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