Faulty Element Detection in Monolithically Integrated Sensing Electrode Array

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Introduction

Electrochemical methods for measuring the analyte concentration in fluidic and biological systems provide fast, low-cost, sensitive and selective measurement. The monolithic integration of electrochemical sensors with microchips on semiconductor technology is a promising technique to improve signal-to-noise ratio of the measurements through eliminating the sensors to instrumentation interconnects thus reducing electrical noise and parasitics. In addition, the integration creates the opportunity for fabricating miniaturised sensor arrays making new promising technologies (e.g. minimally invasive implantable biosensors) available for parallel analysis of multiple parameters from small quantities of biofluids. The exposure of the sensing electrodes to harsh and corrosive biofluid poses a risk of failure of electrodes, and misleading measurements. Therefore, there is a need for a mechanism to detect the faulty element in-situ and exclude it from the sensing system.

Methods

We observed that the most common types of physical damage to the gold-plated on-chip electrode, including corrosion, leads to a decreased electrochemical active surface area (ECSA) [unpublished results]. Therefore, ECSA has been selected as a marker of the health of the sensing electrode in this work based on which the electrodes may be classified into healthy and damaged. We propose a dedicated integrated circuit to allow the estimation of ECSA on-chip.

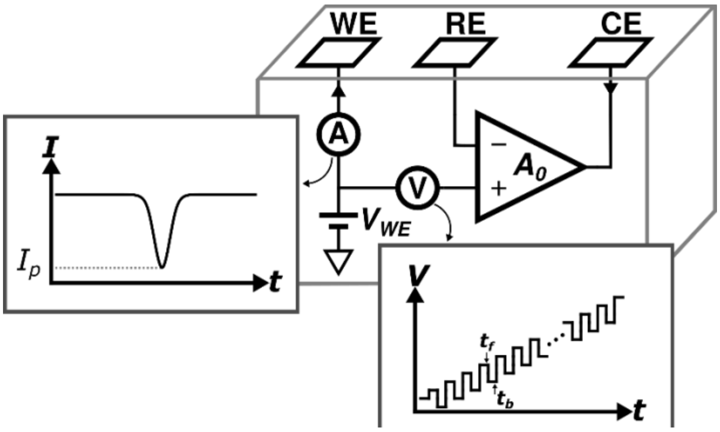

Figure 1 Block diagram for implementation SWV.

The square wave voltammetry (SWV) technique has been employed here, as it was shown to be a more sensitive technique compared to other techniques such as cyclic voltammetry[1] in measuring the ECSA. Precise ECSA calculation requires integrating the area of the voltammogram that corresponds to the reduction peak current of the electrode; We hypothesise that the peak current value (I_P in Figure 1) and a few consecutive samples are adequate to estimate the ESCA in-situ on-chip for the classification of electrode status. Figure 1 depicts the simplified form of the designed integrated circuit to perform SWV and estimate the ESCA. An error amplifier A_0 together with the electrolyte constitute a negative feedback loop to set the voltage difference between the reference electrode (RE) and the working electrode (WE) for SWV measurement through a programmable voltage source. The voltage generator is controlled by a digital block that allows reconfiguring and controlling the voltage amplitude, step voltage and frequency of the applied square wave signal from outside. The current flowing from WE to the counter electrode (CE) is sampled and digitised by the analogue-to-digital converter (ADC) with 10nA resolution for further computations.

Results & Discussion

To detect the peak current value, we swept the staircase potential from 0.4 V to 1.4 V with a square wave superimposed on it between WE and RE with the increment of step voltage in each period. The V_{OUT} is sampled and digitised at the end of the high and low phases of the applied square wave, as depicted in Figure 1, t_f and t_b for one period. A search for the peak value is then conducted through finding the smallest derivative value. That is, the difference between the two currents is then compared with the result of the previous cycle, and if the new value is larger than the previous value, the stored value is replaced with the new subtraction result. When the SWV reaches its end, I_P and four consecutive measurements are compared with values stored in a look-up table (to be implemented on-chip) that includes the binary classification model in order to classify the electrode's status. This process is applied to each electrode within the sensing array one after the other to identify the fabrication errors and failures in-situ at start-up time. As an example scenario, for a sensing array with 16 elements, scanning the faulty electrodes will take 177.92 seconds using one instance of the above circuit, if the SWV sweeps from 0 V to 1.4 V with 0.02 V amplitude, 0.005 V step and 25 Hz frequency.

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Reference

1. Qian, L. et al. *Analyst* 2021, *146* (14), 4525–4534.