



Characterization of coplanar electrode structures for microfluidic-based impedance spectroscopy



Steffen O.P. Blume, Ridha Ben-Mrad, Pierre E. Sullivan*

Department of Mechanical and Industrial Engineering, University of Toronto, Canada

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ABSTRACT

Impedance spectroscopy has the potential for label-free integrated electrochemical detection in microfluidic lab-on-a-chip applications. Its capability to identify and discern between surface and bulk processes in solid–liquid systems finds particular use for the detection of biorecognition events or conductivity measurements. The electrochemical transducer can be in the form of interdigitated electrode structures to increase sensitivity. Experimental work was performed to characterize two different transducer designs. Applications included the monitoring of protein films on contact-less interdigitated electrode structures and conductivity detection of droplets on insulated two-electrode structures. The use of electrode passivation eliminated electrode degradation. Experimental results were compared to theoretical analytical models, and were found to closely correlate with one another. The analytical models were used to design the transducer for optimal conductivity detection. The results inform the current research efforts for the development of in-line impedance spectroscopy in digital microfluidics and confirm the use of simple analytical models for the first-order estimation of the frequency response of interdigitated electrode structures.

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1. Introduction

Impedance spectroscopy (IS) is a non-destructive electroanalytical method, that has been used for corrosion studies, coatings evaluation, batteries, fuel cells, and material characterization [1–3]. IS can characterize the kinetics of bound and free charges in electrochemical cells, as well as desorption and adsorption activities at surfaces in solid–liquid systems. In recent decades, IS has grown as a tool in analytical bioelectrochemistry [4–8].

The application of IS in analytical bioelectrochemistry is mostly concerned with label-free biorecognition processes at electrode surfaces, such that the impedimetric response of the fluid–electrode system is altered by immobilized macromolecules [4,6,9–12]. Yet other applications also include the detection of changes in the bulk fluid [6,13]. Biorecognition processes are usually differentiated into the detection of affinity binding events, the analysis of enzyme-modified electrodes, and cell and microorganism recognition.

The development of lab-on-a-chip devices and the associated need to downscale instrumentation for on-chip analyte detection and manipulation has driven innovation of miniaturized

transducers. Electrochemical transducers have been considered promising for miniaturization and microsystem-level analysis, due to their compatibility with common microfabrication technologies, without a reduction in sensitivity. This observation stands in contrast to optical methods that suffer from bulky external instruments [14] and loss of sensitivity with decreasing volumes [15]. In addition, electrochemical transducers can be combined with inexpensive instrumentation, and feature low power consumption, low detection limits, and adjustable selectivity [16–18].

A number of research groups have accomplished to combine impedance-based electrochemical detection methods with microfluidic systems [19–21], such as microchannels with integrated gold electrodes for impedance measurements in air, deionized water, and saline solutions [22], the use of two sensing electrodes to determine the impedimetric frequency response of different fluids in a digital microfluidic (DMF) device [19], a microfluidic chip for impedance sensing of cancer cell migration [20], an integrated impedance-based sensor for on-chip monitoring of cell growth in a DMF device [21], or insulated carbon microelectrodes for the monitoring of binding events between bovine serum albumin (BSA) and anti-BSA antibodies in microchannels [23].

Interdigitated electrode structures distribute the detection of localized changes to a larger sensing surface [4,24,25]. The integration of interdigitated electrode structures into microfluidic systems has seen developments toward impedimetric sensing of protein

* Corresponding author. Tel.: +1 416 978 3110.

E-mail address: sullivan@mie.utoronto.ca (P.E. Sullivan).

affinity binding behavior [26], dielectric spectroscopy with contactless microsensors to detect viruses [27] or perform quantitative cell analysis [14], the detection of hormone active chemicals by amperometric monitoring of anodic current and redox cycling between electrode fingers [28], detection of infectious parasites in water and determining their concentration by electrochemical impedance spectroscopy [13], and impedimetric detection of bacterial cells using antimicrobial peptides immobilized onto a microsensor array [12], to list only a few examples. Interdigitated electrode structures are not restricted to the detection of surface effects, but can also be used for bulk conductivity measurements. Theoretical and experimental work on optimizing the structure for conductivity detection has for example been addressed in [29–32]. Applications of conductivity detection included the use of potentiostats and microsensors for pH and conductivity measurements in a serpentine channel interface [33], and microfluidic devices with dual capacitively coupled contactless conductivity detection by impedance spectroscopy [34].

When immersed in a liquid containing an electroactive target of interest, the metallic electrodes can either be in direct contact with the liquid, or insulated, i.e. contact-less. The use of contact-less electrodes eliminates faradaic processes, which could be desired in the presence of an electrode-coupled redox probe, but can also be unfavorable. Exposed electrodes can suffer from measurement interferences, due to electrode polarization, electrode fouling, and electrolysis [14]. A dielectric insulator can prevent these undesired reactions. On the downside, it has ramifications on the sensor sensitivity. The design of the transducer shifts towards high performing passivation layers that are as thin as possible, have a high electric permittivity, and in the case of affinity binding have the appropriate molecular structure to immobilize biological probes. In this regard, advanced passivation geometries were evaluated by [35], based on three-dimensional numerical simulations to increase sensitivity of interdigitated capacitors and resulted in electric flux density guides and barriers.

While numerical models can provide an accurate representation of the physical, chemical, and electrodynamic characteristics of an electrochemical cell, they impose high computational costs on the design and optimization process. Therefore, inexpensive analytical solutions that can estimate the impedimetric coupling between the electrode fingers, and are subject to simplifying assumptions about the structure of the cell, present as useful surrogates for numerical simulations under certain conditions. Such models have been discussed in [36–38].

Presented here are experimental results with simple contactless interdigitated electrode structures and impedance spectroscopy to study conformation changes in the sensitive region above the electrode fingers due to the presence of a hydrated protein film. The results are compared to values from an analytical model for interdigitated electrode structures. This work also presents a two-electrode contact-less structure for conductivity sensing and discusses an approach for the optimization of conductivity cells based on the same analytical model. It was found that the analytical models correlated well with the experimental data and are therefore helpful tools in the first-order estimation of the impedance of interdigitated electrode structures and conductivity cells.

1.1. Test device fabrication

Test devices were fabricated in the facilities of the University of Toronto Nanofabrication Centre (TNFC). Chromium coated glass substrates were purchased from Deposition Research Lab, Inc. (St. Charles, MO, USA). The substrates were made of 50 mm × 75 mm × 1.1 mm borosilicate glass slides coated with a 100 nm chromium layer. An additional 50 nm gold layer was

deposited using a custom TES12D Thermal Evaporator. The gold target material (99.999% purity gold pellets) was purchased from Kurt J. Lesker Company (Clairton, PA, USA). Typically, chromium adhesion layers are considerably thinner (~5 nm) than in this case, in which the available chromium coated glass slides were repurposed. The substrates were patterned using standard photolithographic fabrication techniques. Photomasks were made of high resolution films with a minimum feature size of 13 μm (Pacific Arts & Designs, Markham, ON, Canada). Experience with the fabrication of devices had shown that a minimum feature size of 20 μm was more feasible.

The substrates were cleaned in an acetone (Fisher Scientific, Pittsburgh, PA, USA) bath (5 min) and washed in methanol (Fisher Scientific, Pittsburgh, PA, USA) (2–3 min). Next, the substrates were rinsed in deionized water (DIW), dried with a nitrogen spray gun, and dehydrated on a hot plate for at least 5 min at 110 °C. An approximately 12.5 kÅ thin layer of Shipley S1811 photoresist (Marlborough, MA, USA) was spin coated (3000 rpm for 30 s), followed by solvent-removal baking for 2 min at 110 °C. After exposure in a Suss MicroTec MA6 mask aligner (Garching, Germany) for 7.5 s (flood exposure), the substrates were developed in MF-312 (Rohm & Haas, Dow, Midland, MI, USA) for approximately 30 s. The substrates were again rinsed in DIW, dried with nitrogen, and post-baked for 1 min at 115 °C. The substrates were next gold-etched in potassium iodide (Transene Company, Inc., Danvers, MA, USA) for no longer than 10 s to prevent overetching. The etching was stopped by rinsing in DIW and spraying the substrates with nitrogen. The slides were then placed in a chromium etch bath with CR-7S10 etchant (Cyantek Corp., Fremont, CA, USA) for approximately 90 s. The bath was agitated to obtain a more uniform etch rate across the entire substrate. The slides were again rinsed in DIW and dried with a nitrogen spray. The remaining photoresist was stripped in AZ-300T (Branchburg, NJ, USA), while slightly agitating the bath for at least 10 min. The slides were DIW–nitrogen treated and cleaned in acetone and methanol. The substrates were dehydrated on a hot plate at 110 °C for 5 min after being rinsed in DIW and dried with a nitrogen spray gun. A 1 μm Parylene C layer was vacuum deposited in a Specialty Coating Systems Labcoater (PDS 2010) (Indianapolis, IN, USA). The final step was to coat the substrates with an amorphous fluoropolymer. Teflon AF-1601S (DuPont, Mississauga, ON, Canada) solution was diluted to 1.5 wt% in Fluorinert FC-40 (Sigma–Aldrich, St. Louis, MO, USA). A thin film of approximately 50 nm was spin coated in a two-step process (Step 1 – Ramp up: 2000 rpm, 20 s; Step 2: 2500 rpm, 60 seconds). The substrates were then baked for 20 min at 175 °C.

1.2. Experimental apparatus

Images were recorded with an Optronics Macrofire monochrome camera (Muskogee, OK, USA) installed on a Zeiss SteREO Discovery.V12 microscope (Jena, Germany). Impedance measurements were acquired with an Agilent Technologies E5061B ENA Series Network Analyzer (Santa Clara, CA, USA) with an impedance analysis option. The network analyzer was set to the Port 1-2 Series mode, configuring the two S-parameter terminals (port 1 and 2) of the analyzer to be used for impedance measurements with a frequency sweep from 5 Hz to 300 MHz. Before each measurement series the analyzer was calibrated using a non-inductive high precision through-hole 50 Ω resistor and was connected to the test devices via BNC cables and alligator connectors.

The electrochemical cell can be considered as a system that takes an input signal and transfers it into an output signal [39]. Most if not all systems are nonlinear. It is however possible to use a linear approximation for most systems, especially if the input signal amplitude is small. The input to output dependence of a

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