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Characterisation of nano-interdigitated electrodes

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Abstract. Interdigitated electrodes made up of two individually addressable interdigitated comb-like electrode structures have frequently been suggested as ultra sensitive electrochemical biosensors. Since the signal enhancement effects due to cycling of the reduced and oxidized species are strongly dependent on the inter electrode distances, since the nature of the enhancement is due to overlying diffusion layers, interdigitated electrodes with an electrode separation of less than one micrometer are desired for maximum signal amplification. Fabrication of submicron structures can only be made by advanced lithography techniques. By use of electron beam lithography we have fabricated arrays of interdigitated electrodes with an electrode separation distance of 200 nm and an electrode finger width of likewise 200 nm. The entire electrode structure is 100 micrometre times 100 micrometre, and the active electrode area is dictated by the opening in the passivation layer, that is defined by UV lithography. Here we report measurements of redox cycling of ferrocyanide by coupled cyclic voltammograms, where the potential at one of the working electrodes are varied and either an oxidising or reducing potential is applied to the complimentary interdigitated electrode. The measurements show fast conversion and high collection efficiency round 87% as expected for nano-interdigitated electrodes.

1. Introduction
1.1. Interdigitated electrodes in literature
Interdigitated electrode structures with feature size in the nanometer scale are popular in the solid-state physics community; they are either used for generation of surface acoustic waves (SAW), used for contacting nanowires [1] or even as sophisticated mechanical cantilever devices, as demonstrated by Lou Gang et al. [2]. Comparably fewer papers have addressed the use of the structure for electrochemical measurements despite appealing properties such as significant signal enhancement and possibility of easy lab-on-a-chip integration, both properties were recently demonstrated by Zevenbergen et al. [3] when they used a microfluidic system and nIDAs to detect as little as 70 molecules1.

Extensive theoretical work on the foundation of interdigitated array electrodes for electrochemical studies was done by Aoki [4–6; 19]. Experimental work on this kind of electrodes has been hampered by the high fabrication cost, since the advantages over other more established electrodes systems, such as the rotating ring disk electrode (RRDE), is only apparent for

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1 Example of other small IDA in litterature [4–18]
electrodes with feature sizes below a micron and hence strongly depend on electron beam lithography. However, Bech et al. demonstrated the possibility of replicating the structure in large quanta by nano-imprint lithography (NIL)\cite{7; 10}.

1.2. Redox cycling explained
The signal enhancement for IDAs is due to continuous cycling of the electrochemically active species between the two interdigitated electrode fingers \cite{6}. This process is denoted redox cycling. The diffusion layers for the cathode and anode will for very small interelectrode spacing overlap and electrochemical active species with homogenous kinetics will rapidly be converted between the oxidised and the reduced form due to the small diffusion length.

2. Device fabrication

Figure 1. Sketch of fabrication process. 1) Si/SiOx wafer is cleaned in ultrasonic bath for 3 minutes in acetone and isopropyl alcohol (IPA). 2) First ZEP resist and then PMMA resist are spun onto the wafer. 3) The wafer is then exposed by the electron beam and 4) after baking and development of the resist 5) the wafer get thermally evaporated a 5 nm Cr layer and a 40 nm Au layer onto the oxygen plasma treated wafer. 6) Most of the metal deposition is then removed in a lift off process in remover S1165 and the nanometre sized structures are left. 7) A bottom layer of LOR and a top layer of S1813 UV resist are then spun onto the wafer containing the nanostructres and 8) larger micro-metre sized connection and bonding pads are defined by UV-lithography. 9) After the UV exposure the resist is developed and 10) a Cr/Au layer are thermally deposited onto the wafer. 11) After a lift-off 12) the structures are finally covered by a layer of S1813 UV photoresist that serves as dielectric layer and 13)-14) are opened in the areas where electrodes and bonding pads are defined. 15) Finally the chip is wire-bonded to a chip carrier and a vial are mounted for containment of liquid.

The interdigitated electrodes were made by electron beam lithography (EBL) and interconnections and openings in the dielectric layer were made by UV-lithography, see figure 1. A Raith 150 EBL system was used to define the 200 nm line structures in a double layer resist consisting of a bottom layer of ZEP 520 A7 (spun for 30 s at 6000 rpm) and a top layer of PMMA.
950K A4 (spun for 60 s at 6000 rpm). The electron beam double layer resist were patterned through electron beam radiation with a dose of 57 mC/cm², then developed for 60 s in MIBK and subsequently for 5 min in o-xylene, thereafter rinsed 30 s in IPA and dried by blowing the sample with nitrogen. Metal (5 nm Cr and 50 nm Au) was thermally deposited and followed by a lift-off process in remover 1165.

Contacts to the nanometer-sized structures were made by UV-lithography in double layer resist (bottom layer LOR 3A and bottom layer S1813) and lift-off of thermally deposited metal (5 nm Cr and 50 nm Au) in remover 1165. Finally, S1813 were used as dielectric layer and the openings in the dielectric layer were also done by UV-lithography. The chip was then mounted and bonded to a chip carrier and a vial was glued to the chip by silicon glue.

3. Electrochemical measurements

The experiments were carried out in the flow cell (200 µl) with different concentrations of ferricyanide dissolved in a phosphate buffered saline (PBS) solution, as displayed in figure 2 and figure 3. All measurements were recorded in a four electrode setup2 by use of a Autolab bi-potentiostat. As pseudo reference electrode was use a chlorinated silver wire, that was lowered into the solution by a micrometer screw set-up. For the counter electrode was used a platinum wire.

![Figure 2. Double CVs for different concentrations of ferricyanide in phosphate buffered saline (PBS). All potentials are measured versus a pseudo Ag/AgCl reference electrode. The CVs with negative current values originates from the generator electrode and the scans with positive current are the recorded signal for the collector.](image1)

![Figure 3. Linear fit of endpoint data from figure 2 for various concentrations of ferrocyanide for a sweep rate of 100 mV/s. The linear fit for the generator give a gradient of $-0.117 \pm 0.002$ nA/µM and for the collector $0.103 \pm 0.002$ nA/µM. Dividing the gradient of the collector by the gradient of the generator give a collection efficiency of 87% ± 3%.](image2)

The cyclic voltammograms displayed in figure 2 have been recorded by applying a fixed potential of 500 mV vs. Ag/AgCl to the electrode serving as collector and then running a cyclic voltammogram from a starting point of 500 mV vs. Ag/AgCl to a potential of -300 mV vs. Ag/AgCl on the complimentary interdigitated electrode serving as the generator. The resulting cyclic double-voltammograms for various concentrations are displayed in figure 2. The CVs show typical behaviour of ultra micro electrodes and show no evidence of diffusion limited current flow.

2 Reference electrode, counter electrode and two working electrodes.
The detection limit was found to be 5 µM at a voltage sweep rate of 100 mV/s, which partly is due to the resolution of the potentiostat and partly due to electrochemical noise from the PBS solution that was used as supporting electrolyte and buffer.

4. Conclusion
An interdigitated electrode array (IDE/IDA) with a width and separation of the electrodes of 200 nm has been fabricated. For a concentration range from 5 µM to 4 mM there were observed a linear relationship between the concentration of a electrochemical reversible redox compound, ferrocyanide, and the endpoint current. The collection efficiency of the electrode has been calculated to be 87% ± 3% which is much higher then the round 30% collection efficiency reported for rotating ring disk electrodes.

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