Interdigitated nanoelectrodes for sensing: fabrication and characterization

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Interdigitated nanoelectrodes (nIDEs) consist on two arrays of electrodes with comb shape which, under proper bias polarization, can be used for (bio)chemical sensing by measuring the change of impedance or electrochemical current.

In this work we present the fabrication of a complete device, and the first results of its characterization. The device can be used in different configurations: (i) measurement of the electrochemical current generated by chemical reactions occurring in the solution [1], and (ii) study of the impedimetric response of the system when the nature of the solution changes [2] or even further, with the inclusion of nanoparticles in between the digits [3].

Fabrication.

In order to combine micro and nanometer size features, the connection pads and lines are first fabricated by UV lithography, metallization (Ti/Au, 7nm/60nm) and lift-off. Aligning marks have been included in the design of the UV mask for a second lithography level which consist of electron beam lithography to define the digits, followed by metal evaporation and lift off. Figure 1 shows results of the fabrication process: the whole chip, fabricated in gold onto a SiO₂/Si substrate (a), and a detail of the digits (b), which are 180 nm wide and with a pitch of 230 nm. These devices can be used already for sensing or as the master to fabricate stamps for nanoimprint lithography (NIL). In the second case, a reactive ion etching (RIE) process is performed. Then, the fabrication of subsequent samples is easier and faster, since the two lithography levels (UV and e-beam lithography) are substituted by one single NIL step.

Once the electrodes have been fabricated, they are passivated with PMMA, leaving open only the large pads for external connections and the areas with the digits. Then, they are bounded to a printed circuit board (PCB), and encapsulated with *epotec*®. At this point, the sensor is ready to be connected to an external electronic system and the electrodes can be immersed in a liquid media (Figure 1 (c)).

Characterization.

Figure 2(a) shows the first results of <u>electrochemical current</u> measurements. The nIDE was immersed in a solution 1mM of $[Fe(CN)^{3-}]_6$ in KNO₃ (red line). A stabilization potential was applied during the first 30seconds, and then changed to 0V (^{*}). After the stabilization of the system when V = 0V, the current is due to the reduction processes occurring in the solution. The black line corresponds to the same measure, but in a KNO₃ solution. No electrochemical current is observed after the stabilization in this case. The sensors are being calibrated by adding known concentrations of $[Fe(CN)^{3-}]_6$, so subsequently can be used to determine the concentration of unknown solutions.

Figure 2(b) corresponds to results of the <u>impedimetric response</u> of the electrodes (total impedance of the media measured as a function of frequency). The electrode was immersed in DI water and then in solutions of NaCl with different concentrations (i.e., the resistivity of the media changes). The figure shows that the change from the low frequency capacity to the high frequency capacity depends on the concentration of Na^+ Cl⁻ ions in the aqueous media. Thus, the device can be also calibrated, to be used for sensing. Additional tests performed in liquids

^{*} $\operatorname{Fe}(CN)_6^{3-} + e^- \rightarrow \operatorname{Fe}(CN)_6^{4-}, E_0 = +0.35V$

with different permitivities (ε_r) have been performed, showing that the higher ε_r is, the lower the high frequency capacity.

Once the proper response of the sensors has been demonstrated, the devices are being currently used to detect and quantify the presence of insulating nanoparticles (Figure 3). The final goal is to functionalize the nanoparticles to perform specific detection of bioentities: an increase of sensitivity is expected since nIDES would allow the detection of a single nanoparticle. The results will be presented at the conference.

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References:

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Figures:



Figure 1. (a), optical image of a sensor, fabricated in gold on a SiO_2/Si substrate. The pads are defined by optical lithography, metallization and lift off, and the digits are fabricated in a second step, by e-beam lithography, metallization and lift off. A detail can be seen in the SEM image in (b). (c) once the electrode is fabricated, it is bounded to a PCB and encapsulated, so it can be connected to an external electronic system and the electrodes immersed in a solution, for characterization.



Figure 2. (a) **Electrochemical current** measurements, for an applied potential similar to the reduction potential of $[Fe(CN)_6]^{3-}$. When the electrode is immersed in a FeCN63- 1mM solution, a current due to the electrochemical reaction is measured (red line). For a non-active solution (KNO3), no current is observed (black line). (b) **Impedimetric characterization:** Dependence of the capacity of the system on the frequency when the electrode is immersed in solutions with different resistivity (i.e., aqueous solutions of NaCl at different concentrations). In this case, the transition from the high frequency capacity to the low frequency one depends on the concentration of ions in the solution.



Figure 3. SEM image of one of the electrodes (140nm width, 400 nm pitch), where silica nanoparticles (300nm diameter) have been precipitated.