

Surface characterization techniques of gold coated micro-electrodes

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Introduction: Gold electrodes have been extensively used as the base electrodes for monolithic integrated functional surfaces sensors on semiconductor devices (e.g. CMOS microchips with aluminum pads). The quality assessment of gold coating is crucial before further modifying the film with desired materials (e.g. recognition elements, nanostructures) for sensing applications. The quality assessment is typically performed using imaging techniques such as scanning electron microscopy (SEM), atomic force microscopy (AFM) and also via electrochemical surface characterization. However, these techniques either damage the chip or change the surface profile of electrode. We propose an alternative technique based on electrochemical characterization to study of the quality of gold-coated micro-electrodes with minimal changes to surface profile.

Materials and methods: square aluminum electrodes with dimension of $66\mu\text{m}\times 66\mu\text{m}$ on a microchip fabricated in CMOS technology were coated with gold film using the electroless chemical plating technique described in [1]. The chip was then glued to a printed circuit board (PCB) with silver conductive epoxy (MG Chemicals 8331D) and wire-bonded to the PCB. The wire-bonds were covered using epoxy (EP41S-5, masterbond). Electrochemical measurements using CV and/or square wave voltammetry (SWV) were carried out on each individual electrode using 10 mM sulphuric acid. The electrode conditioning potential and time prior to SWV were optimized to be 1.6 V and 10 seconds respectively. In SWV, the electrode voltage was swept once from 1.4 to 0 V at a with steps of 5 mV, scan rate of 0.1 V/s, and a modulation amplitude and frequency of 20 mV and 25 Hz respectively. The SEM and AFM were carried out at the end.

Results and discussion: The general electrochemical methods to find the effective surface area (ESA) of electrode employ CV with (i) ferro/ferricyanide redox couple at varying scan rates [2] and (ii) sulphuric acid. The latter is used specifically for gold electrodes [2]. The ESA is then estimated to be the area under the reduction peak. However, these methods may damage the electrode by causing the dissolution of the gold coating from the electrode surface. The hexacyanoferrate ion in the redox couple in method I has been found to cause irreversible changes to the surface of gold electrode [3]. Following technique II (Fig. 1.b), we observed an oxidation peak indicating that gold atoms have turned into gold ions and dissolved in the acid. Although method II provides an accurate measurement of the ESA, it leads to changes in the surface profile of the electrode. This was further confirmed using SEM (Fig. 1.c). To prevent the dissolution of gold, we propose two amendments to method II, inspired by [4]. These are, applying a conditioning potential in order to oxidize the surface, and replacing CV with SWV to reduce the oxidized layer. SWV further removes the charging current, simplifying the calculations of ESA. The area under the peak measured using this technique is observed to be identical to that of method II. We conclude these as same.

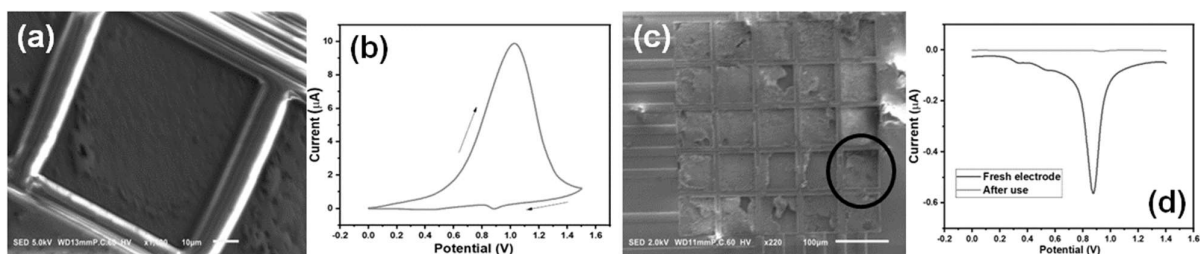


Fig.1.(a) SEM image of a gold-coated electrode (b) CV of an electrode in 10 mM sulphuric acid. (c) SEM image of damaged electrodes. (SWV and CV provided for highlighted electrode) (d) SWV voltammogram of electrode in 10 mM sulphuric acid.

Conclusion: The proposed surface characterization technique, based on conditioning and SWV can be used to measure the effective surface area of gold microelectrodes without causing dissolution of the gold.

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