

Centro interdipartimentale di ricerca
«CENTRO STUDI DI ECONOMIA E TECNICA DELL'ENERGIA
GIORGIO LEVI CASES"

PROGETTO DOTTORATO DI RICERCA 2014:

"Nano-structuring artificial photosynthesis for solar fuel production"

AGGIORNAMENTO DOPO IL PRIMO SEMESTRE Giovedì 14 maggio 2015

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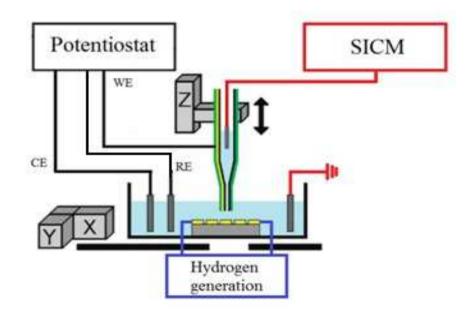
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1. First year project

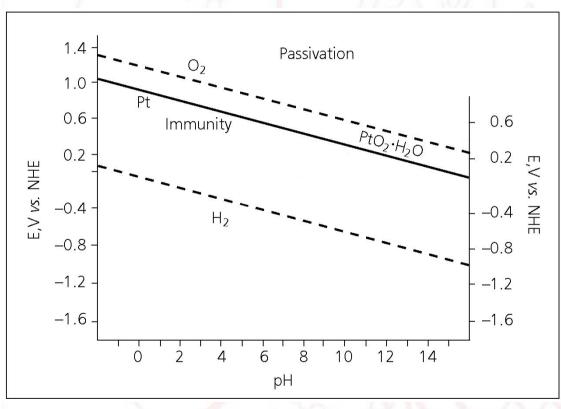
FIRST YEAR

- study of SICM/SECM technique and its applications in order to achieve the goals of the project;
- first approaches to water splitting through bibliographic study and preliminary experiments.
- November-April: production of an electrochemical probe for hydrogen measurement with parallel & integrated surface topography through the SICM technique

Experimental set-up



2. Pipettes coated with Pt



- To perform electrochemical analysis, a conductive layer must be deposited on the surface of pipettes.
- Pt is a very common used catalyst for oxidation of gaseous hydrogen in fuel cells
- As a noble metal, it's potential range of application is wider than other materials used as electrodes for electrochemical analysis

- Coating with Pt via CVD:
 - Pt(acac)₂ as precursor, water as co-reagent; N_2 as carrier; T growth = 280-285 °C; p = 100 Pa.
 - Thickness of the layer: 150 nm.
 - Good conductivity & reproducible results.

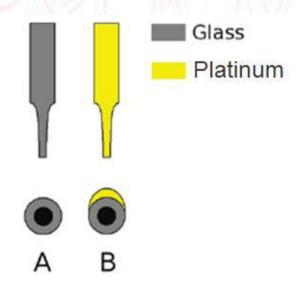


Figure 1. Schematic of integrated SECM—SICM nanopipet probe fabrication viewed normal to and along the nanopipet axis.

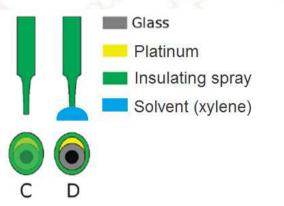
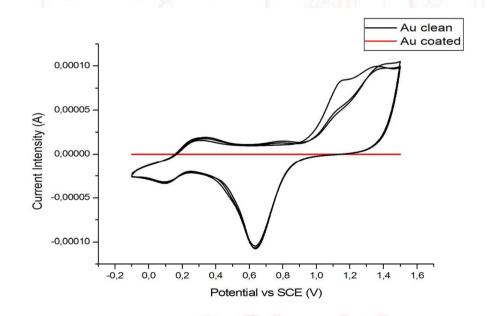


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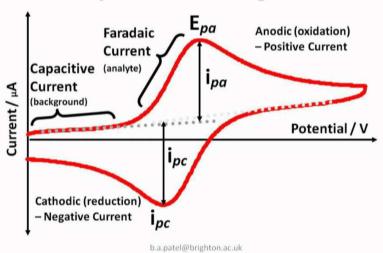


- To perform electrochemical analysis coupled with SICM, only the conductive tip must be exposed to the solution.
- A thin layer of insulating coating is deposited over Pt, then the tip is "re-opened" using a small drop of solvent to dissolve the very top of the coating (xylene was very effective).

3. Characterization procedure

- An useful way to evaluate the quality of our prototype is through electrochemical analysis
- A cyclic voltammetry (CV)
 consists in a potentiodynamic
 electrochemical
 measurement. The current at
 the working electrode is
 plotted versus the working
 electrode's potential to give
 the cyclic voltammogram
 trace

Cyclic Voltammogram



- A comparison must be done between the CV of the nanopipette and the CV of a macroscopic Pt wire in order to verify the reliability of the product.
- A set of solutions with different concentrations of a redox specie (ferrocene) is used.
- The linear correlation between the two systems, or deviations from linearity at certain concentrations, must be evaluated.

