PROGETTO DOTTORATO DI RICERCA 2014:
“Nano-structuring artificial photosynthesis for solar fuel production”

AGGIORNAMENTO DOPO IL PRIMO SEMESTRE
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1. First year project

**FIRST YEAR**

1. study of SICM/SECM technique and its applications in order to achieve the goals of the project;
2. 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
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.
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.
• 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.