

A Methodology to Electrochemically Fabricate Fe-Ni-Co Nanotips

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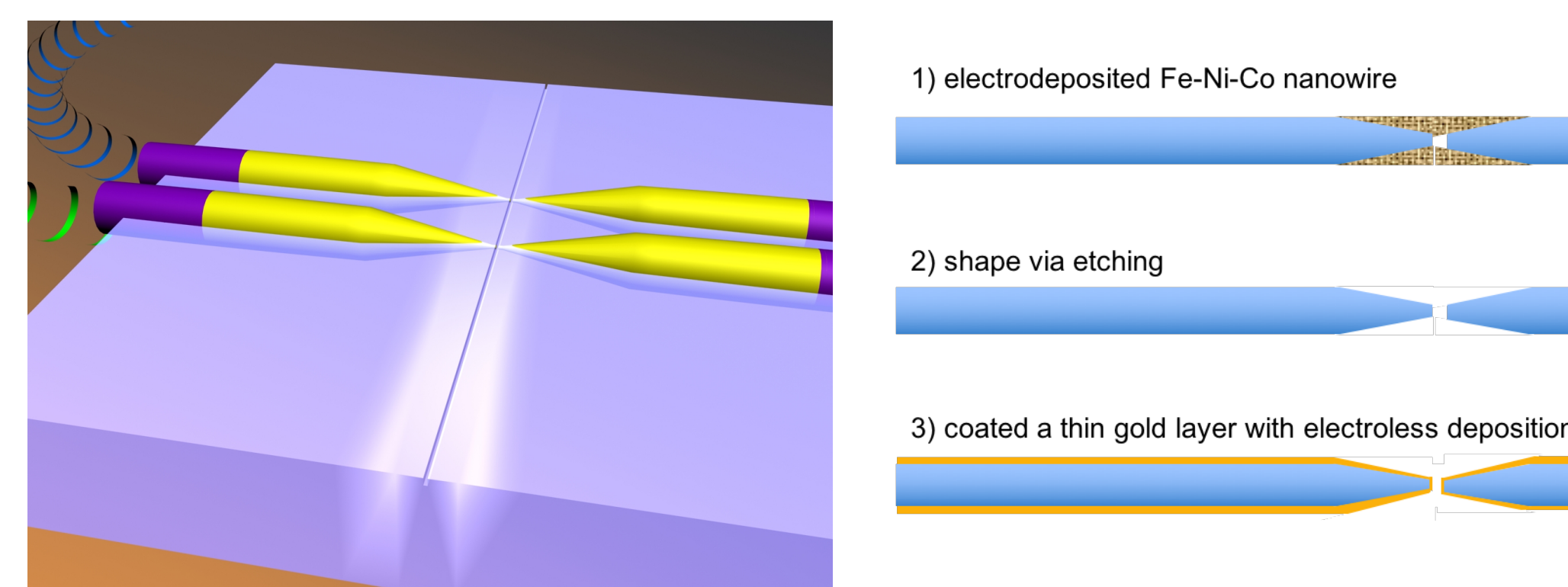
Opportunity

Introduction:

Fe-Ni-Co nanotips at the end of nanowires are fabricated at the interface of two Fe-Ni-Co regions via a combination of pulse electrodeposition, anodization and chemical etching. The conditions to electrodeposit and anodize the Fe-Ni-Co nanowires in polycarbonate templates were first investigated with a rotating cylinder electrode (RCE) to inspect the polarization behavior of the thin film deposition. The wires were fabricated with three, consecutive electrochemical conditions, where first an Fe-Ni-Co wire segment is deposited, followed by an anodic potential to induce growth of an iron oxide thin film, and then followed by an applied, pulse cathodic current density to reduce the oxide and deposit another layer of Fe-Ni-Co. Upon etching, tips formed at the end of the last Fe-Ni-Co region, as evidenced by SEM. Potential transients during the last applied cathodic pulse current step, suggests that both the reduction of the oxide and metal occur, and that TEM/SAED confirm changes in the crystalline Fe-Ni-Co structure at the interfacial region between steps that contributes to the tip formation.

Aim:

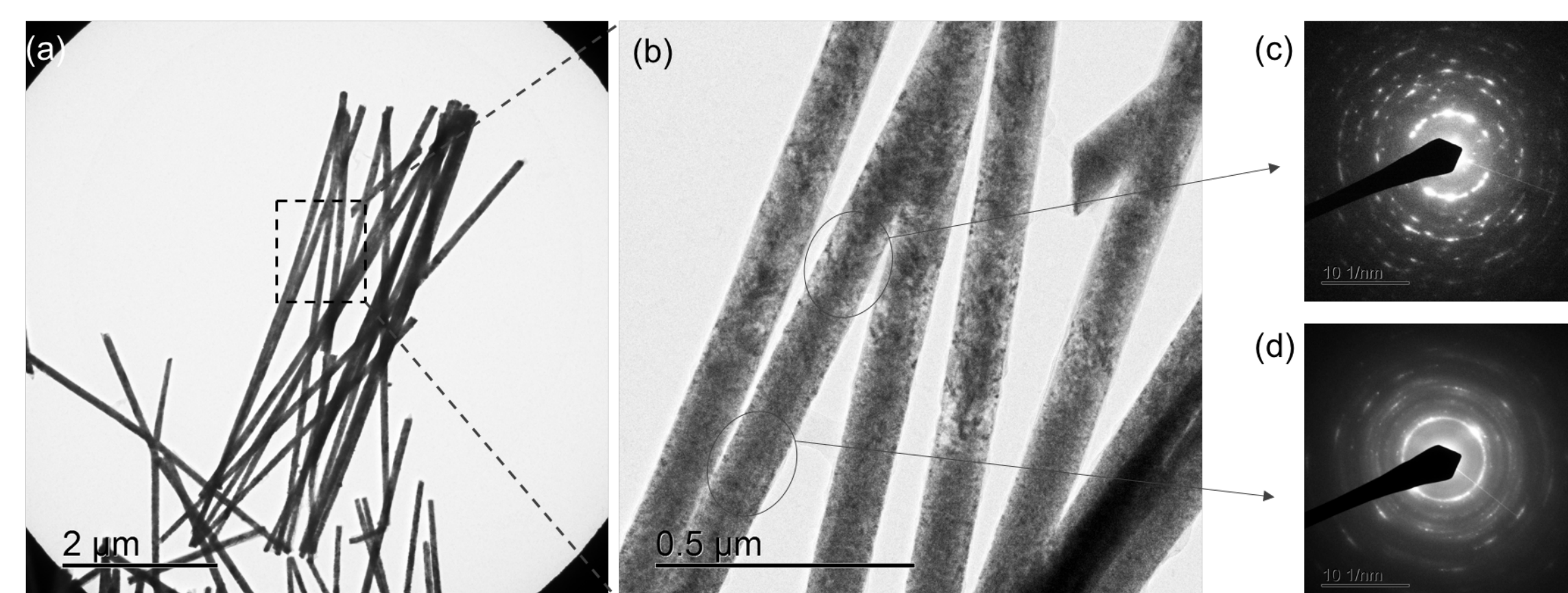
Fe-Ni-Co nanotips at the end of nanowires are fabricated at the interface of two Fe-Ni-Co regions via a combination of pulse electrodeposition, anodization and chemical etching. The conditions to electrodeposit and anodize the Fe-Ni-Co nanowires in polycarbonate templates were first investigated



Data or Results

Before Etching:

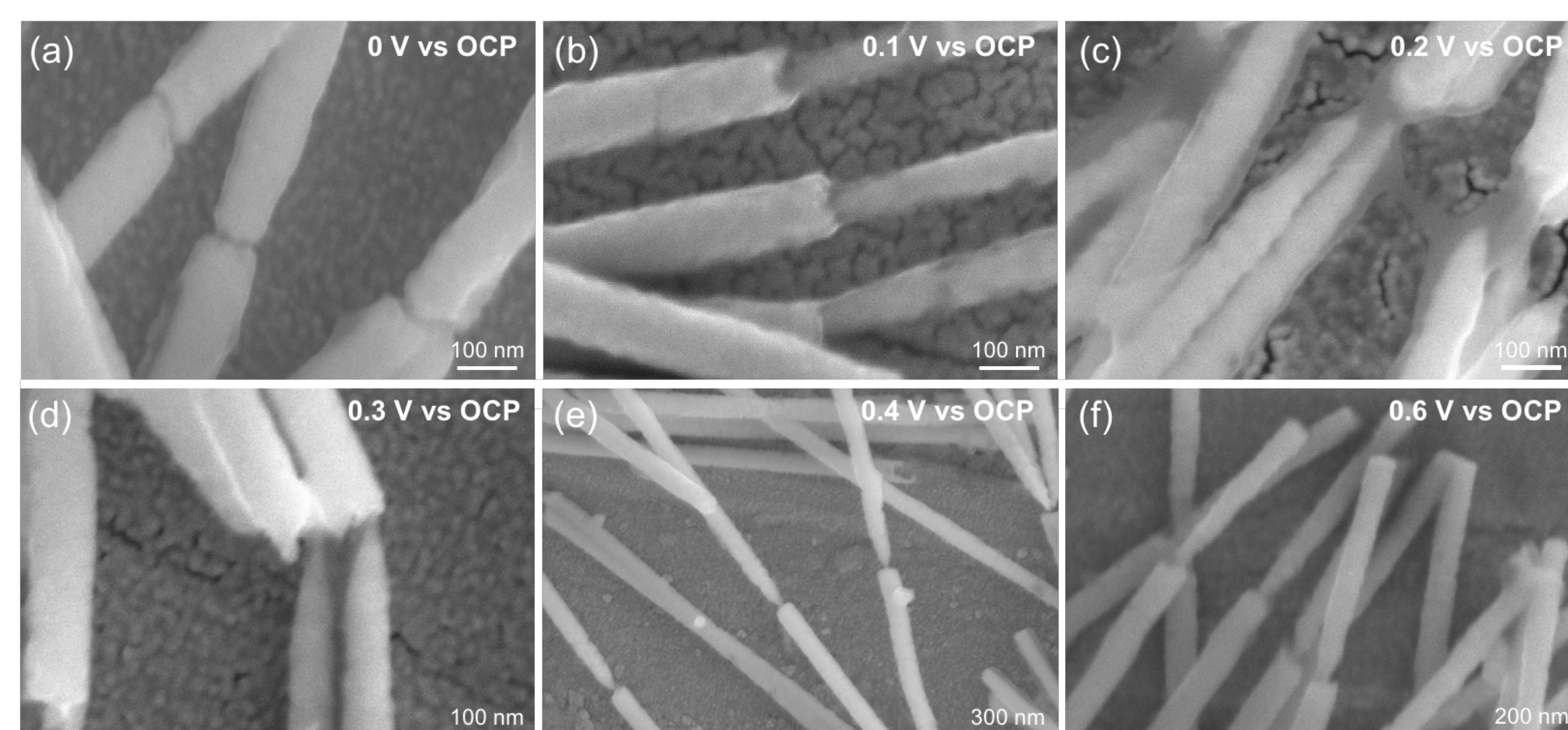
Without any subsequent etching step, all the nanowires are continuous. (b) is a high magnification image of the interfacial region, which was produced by reducing the iron-rich oxide created during the oxidation step; however, the boundary between the layers is not sharp. SAED results of the reduced oxide region is depicted in (c) indicates a polycrystalline structure, while in (d) the Fe-Ni-Co alloy region has a SAED result with less discrete points, and a more amorphous structure.



After Etching:

Once etched, either a gap or a nanotip is observed at the region where the anodic potential was applied. When there is no applied anodic potential, at the OCP, a gap structure is generated after etching (a), with a gap size around 10 nm. As the anodic potential increases from 0.1 V vs OCP to 0.6 V vs OCP, there is an evolution of nanotip structures fabricated as shown through (b) to (f).

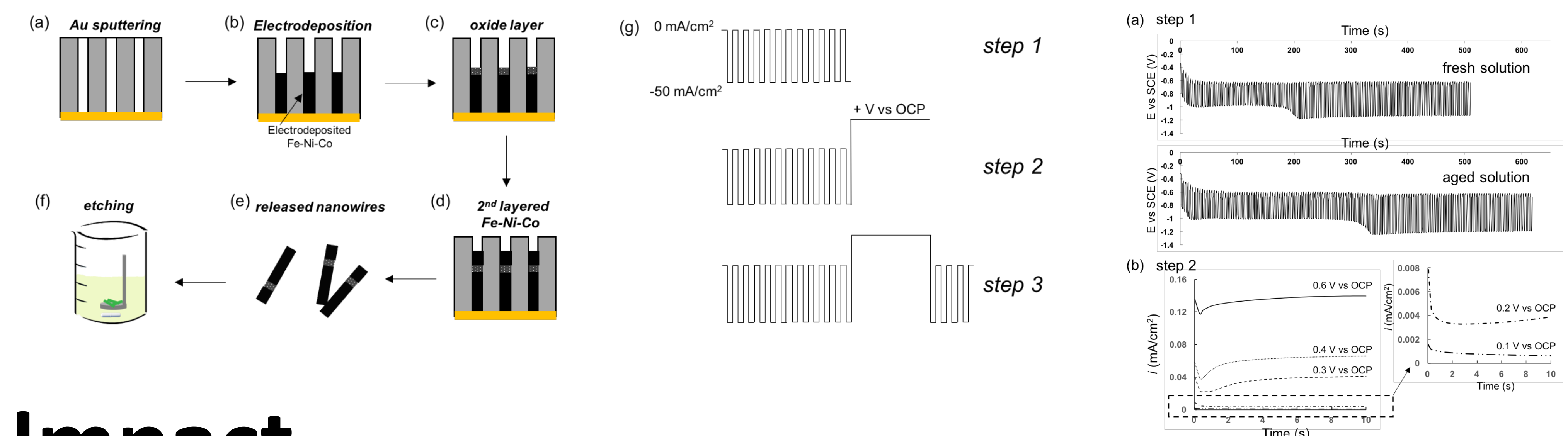
- Crystal structure
- Alloy composition
- Porous structure



Approach

Methods:

A summary of the electrodeposition and etching process of the nanowires is sketched in Figure. The nanowires were electrodeposited through a template assisted electrodeposition method using polycarbonate membranes that had a thin layer of gold sputtered onto one side of the membrane (a). The first layer of Fe-Ni-Co was pulse at 40 °C (b). The potential was stepped to its open circuit potential (OCP) value, for 10 min or to an anodic potential, at different values vs OCP, for 10 s (c), before the second layer of Fe-Ni-Co was deposited under the same condition (d). After deposition, the nanowires were released from the membrane by dissolving the membrane in dichloromethane under ultrasonic agitation (e), following by etching in a pH 5 citrate-acid solution for 10 min (f). A sketch of the applied current or potential waveforms is shown in (g). In step 1, the applied current is modulated with a square pulse for fabricating the first layer of the tri-layered nanowires; in step 2, an anodic DC potential, or its open circuit potential, is applied followed by another pulse cathodic current deposition in step 3.



Impact

- **The unique feature about my research is:** We developed a new methodology for creating nanotips at nanoscale, instead of just investment parameters
- **This address the problem of:** the mass producing of nanotips as nanosensors in an economical manner
- As an byproduct of this research (see image on the right), we developed a way of monitoring electrolyte aging during electrodeposition, this could be very useful for industry application

Step 1:

Fe³⁺ contaminate generate at the CE (counter electrode reaction, Fe²⁺ → Fe³⁺) increase after each running, Fe³⁺ deposition happens only after local (inside nanopores) Fe³⁺ been consumed entirely

Step 3:

At a pH 8.4 case (borate) during a non-pulse, reduction under constant current, both a solid state transformation Fe₂O₃ → Fe₃O₄ and dissolution, Fe₃O₄ → Fe²⁺ were found to both occur at a potential region that is near -0.8 V vs SCE until all the oxide was reduced.

