

Cleaning procedures in Ultrasonic Bath SONICA 3200 EP of glassware and electrochemical cell components of Electrochemical Scanning Tunneling Microscope.

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The electrochemical scanning tunneling microscope (EC-STM) is a microscopy technique couple with an electrochemical technique such as cyclic voltammetry. The EC-STM employed in our laboratory permits the imaging of smooth surface with atomic resolution at the solid-liquid interface; practically this technique permits to see the surface atoms in the presence of an electrolytic solution. Therefore, EC-STM allows an in-situ evaluation of the electrocatalysis processes and to follow the electrodes surface changes upon a chemical or an electrochemical reaction at atomic resolution.

In order to obtain high quality of in-situ STM images it is necessary to eliminate possibly contaminations coming from the solution, electrochemical cell, glassware, tweezers, glue used for coating of the tips etc. In Figure 1 a) and b) it is reported an example of a contaminated system; the surface has been not sufficiently cleaned so that atomic resolution is obtained but contaminants are clearly visible as light spots over the iodine-modified Au(111) surface. The presence of contaminants precludes in most cases the imaging of the surface and to reach atomic resolution. Furthermore the presence on nanocontaminants on the scanner tips or sample can produce fake images and therefore misleading results.

On the contrary, when a deep cleaning is assured, it is possible to obtain pictures such as those reported in in Figure 1 c) and d). In the example reported in Figure 1 c) and d) (I^- adsorbed on Au(111) single crystal) it is possible to see the surface atoms and there are no traces of contaminations. The easiest and most efficient method to clean all crucial equipment is to use ultrasonic bath. The equipment employed for the acquisition of the Figure c) and d) have been cleaned in Ultrasound Bath SONICA 3200 EP.

Ultrasonic cleaning uses Cavitation micro-to-nano-sized bubbles induced by high-frequency pressure (sound) waves to agitate a liquid. Stimulation induces a high force on the contaminants adhering to substrate such as metals, plastics, glass, rubber and ceramics. Due to the inherently small size of the bubbles and them relatively large energy ultrasonic cleaner is able to reach into

small crevices and remove entrapped soils very effectively. The contaminants can be dust, dirt, oil, pigments, rust, grease, algae, fungus, bacteria, lime scale, polishing compounds, flux agents, fingerprints, soot wax and mold release agents, biological soil like blood, etc. [1-3].

The adopted cleaning procedures involve the use of ultrapure water, bidistilled twice from a permanganate solution (KMnO_4) or piranha solution, a mixture of sulfuric acid (H_2SO_4) and hydrogen peroxide (H_2O_2).

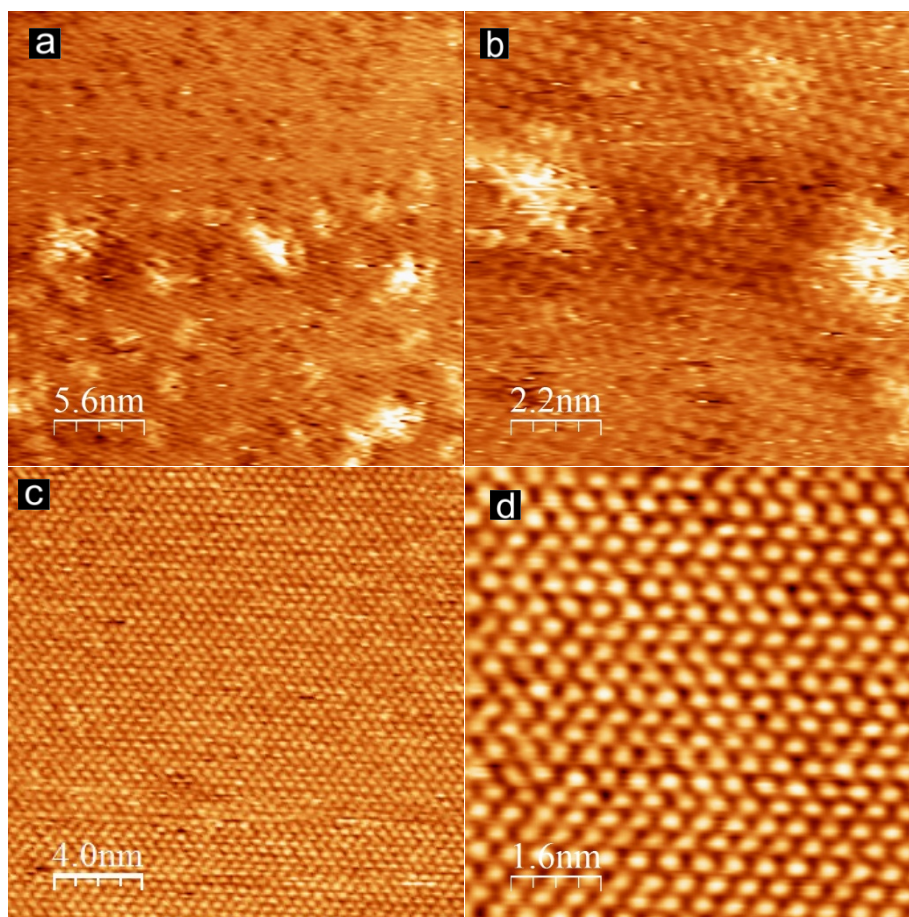


Figure 1: In situ STM images obtained from a Au(111) surface in 5mM H_2SO_4 + 1mM KI solution; image a) and b) represent an example of contaminated system, c) and d) high-resolution STM image of I/Au(111) surface without trace of contamination; images parameters: a) 28nm x 28nm, $I_t = 1\text{nA}$, $U_b = -3\text{mV}$, $E = -500\text{mV}$ vs Pt/PtI; b) 11nm x 11nm, $I_t = 1\text{nA}$, $U_b = -3\text{mV}$, $E = -500\text{mV}$ vs Pt/PtI; c) 11nm x 11nm, $I_t = 1\text{nA}$, $U_b = -15\text{mV}$, $E = -400\text{mV}$ vs Pt/PtI; d) 11nm x 11nm, $I_t = 1\text{nA}$, $U_b = -15\text{mV}$, $E = -400\text{mV}$ vs Pt/PtI.



Figure 2: Cleaning in Ultrasound Bath SONICA 3200 EP: a) Ultrasound Bath SONICA 3200 EP, b) STM electrochemical cell, c) tweezers, d) glassware, e) glue used for coating of the tips.

Cleaning procedure for:

- Electrochemical cell and tweezers (see Fig. 2 b and c): 5x sonication in ultrasonic bath for 15min with bidistilled water;
- Glassware (see Fig. 2d): 2x sonication in ultrasonic bath for 15min with bidistilled water, rinsing with piranha solution and then 3x sonication in ultrasonic bath for 15min with bidistilled water
- Glue used for coating of the tips (see Fig. 2e): 3x sonication in ultrasonic bath for 15min with bidistilled water.

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