INVESTIGATION OF SCALE ELECTRODEPOSITION BY COUPLING IN SITU MICROSCOPE OBSERVATION AND EIS.

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In recent papers, a new experimental device was presented to investigate the *in situ* scale electrodeposition through a transparent quartz crystal microbalance (QCM) (1,2). As shown in figure 1, a microscope lens was set behind the transparent working electrode leading a 400 x 300 µm field of view to be observed from a 5 mm in diameter electrode. An impinging jet cell was used in order to control the convection of the scaling solution in the vicinity of the electrode surface. A potential corresponding to the current of oxygen reduction was applied allowing the interfacial pH between the solution and the electrode surface to be increased. The pH enhancement promoted the scale deposition from a carbonically pure water (3). The QCM led the current of oxygen reduction and the mass of the electrodeposit to be measured *versus* time. Information about the scaling power and the density of the deposit were obtained by using this technique (4).

As shown in figure 2, the original transparent electrode allowed the $CaCO_3$ vaterite and calcite crystal growth to be observed *versus* time. The image processing software allowed the nuclei to be counted over time from 1 µm minimum size. The individual area of vaterite and calcite crystals was measured *versus* time as well (2). It was noticed that the surface of a vaterite crystal grew about four times faster than that of a calcite crystal. Thus, the nucleation growth equations were developed taking into account both scaling rates of vaterite and calcite. Figure 3 shows the experimental covered surface by the insulating layer of $CaCO_3$ measured *versus* time by means of the microscope lens for different Ca^{2+} concentrations. According to the expectations, the scaling rate increased with the hardness of the solution. A good accordance was found between the experimental values and the calculated values from the nucleation growth equations before overlapping occurred (5).

As the kinetic of coverage was perfectly defined, the electrochemical impedance and the microscope observation through the transparent electrode were measured simultaneously *versus* time. We propose to show the correlation between the percentage of covered surface and the double layer capacitance measured at the same time of the experiment.

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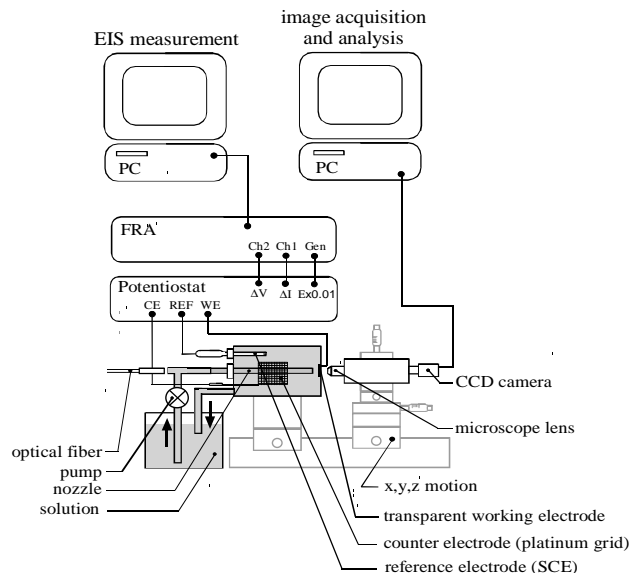5. O. Devos, C. Gabrielli, M. Tlili and B. Tribollet, *J. Electrochem. Soc.*, **submitted**.
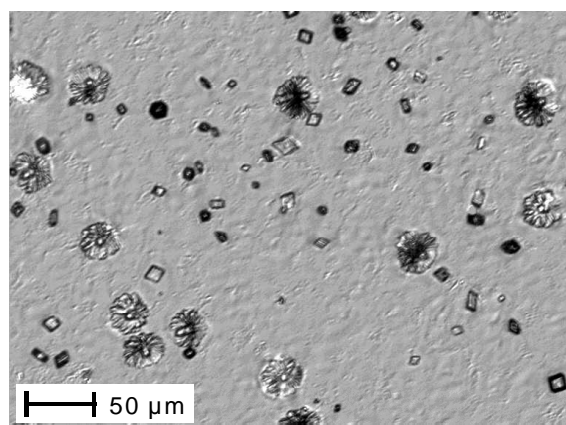
Fig.1. Experimental setup

Fig.2. Microscope image observed through the transparent working electrode after 15 min of scale deposition. $[Ca^{2+}]$ = 160 mgL^{-1}, V = -1 V/SCE, flow rate = 400 mLmin^{-1}.
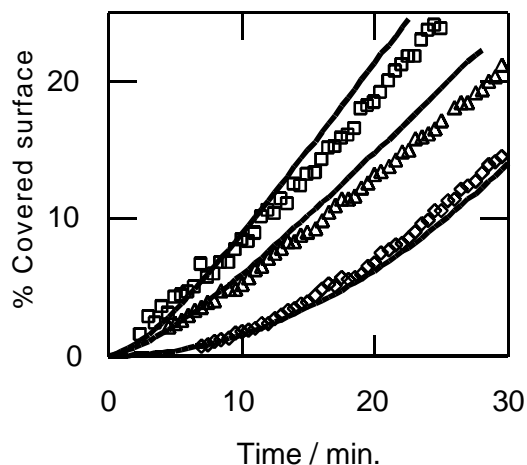

Fig.3. Experimental and calculated (full line) percentage of covered surface by the $CaCO_3$ deposit *versus* time. (◊) $[Ca^{2+}]$ = 120 mgL^{-1}, (Δ) $[Ca^{2+}]$ = 160 mgL^{-1}, (□) $[Ca^{2+}]$ = 200 mgL^{-1}.