ThinCorr: a miniaturized tool for localized electrochemical measurements

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Abstract

This work concerns the design, realization and testing of a miniaturized electrochemical cell, called ThinCorr, devised for localized measurements. Sometimes it is necessary to carry out localized measurements on large components and, in many cases, it is not possible to extract small samples from them to make an analysis in laboratory. In such situations measurements on site become necessary. The miniaturized cell that we present here is designed in such a way that the electrolyte can flow in proximity of the working electrode, avoiding concentration gradients and the corresponding polarizations. Within the body of the EC Minicell there are channels that allow the flow of the electrolyte and that contain a counter electrode and a reference electrode. At the nozzle of the cell there is a gasket that delimits the area of the working electrode, when the EC Minicell is pressed against a metallic surface. Examples of applications are reported in the paper.

Introduction

Microcells for localized electrochemical investigations have been proposed and their limitations have been described [1, 2, 3]. Special theta-glass capillary are used to define working electrode surface areas ranging from units to thousands of μm^2, in order to investigate the behavior of individual grains or particles.. Other types of miniaturized cells are used for electrochemical testing in industrial applications. In one of these cells the working electrode surface area, of the order of mm^2, is made by a porous polymer body impregnated with a stagnant electrolytic solution [4].
In this work we present a miniaturized cell (EC Minicell) devised to perform localized electrochemical investigations on surface areas of the order of mm^2. The EC Minicell has been designed in order to be used also for in-field electrochemical tests, in combination with a portable potentiostat.(ThinCorr system)

Experimentals

The miniaturized EC Minicell consists of a robust hollow cylindrical body which allows the flow of the electrolytic solution in proximity of the working electrode area by means of an external miniaturized pump. The working area is isolated by means of a circular gasket with a diameter of

the order of millimeter. The body of the cell incorporates the reference electrode (Ag/AgCl) and the counter-electrode (Pt), and it is manufactured by laser stereo-litography using a photo-polymerizable resin, even though other materials are possible, such as metals or composites and ceramics. The electrochemical tests are carried out by using an AMEL potentiostat 2556.

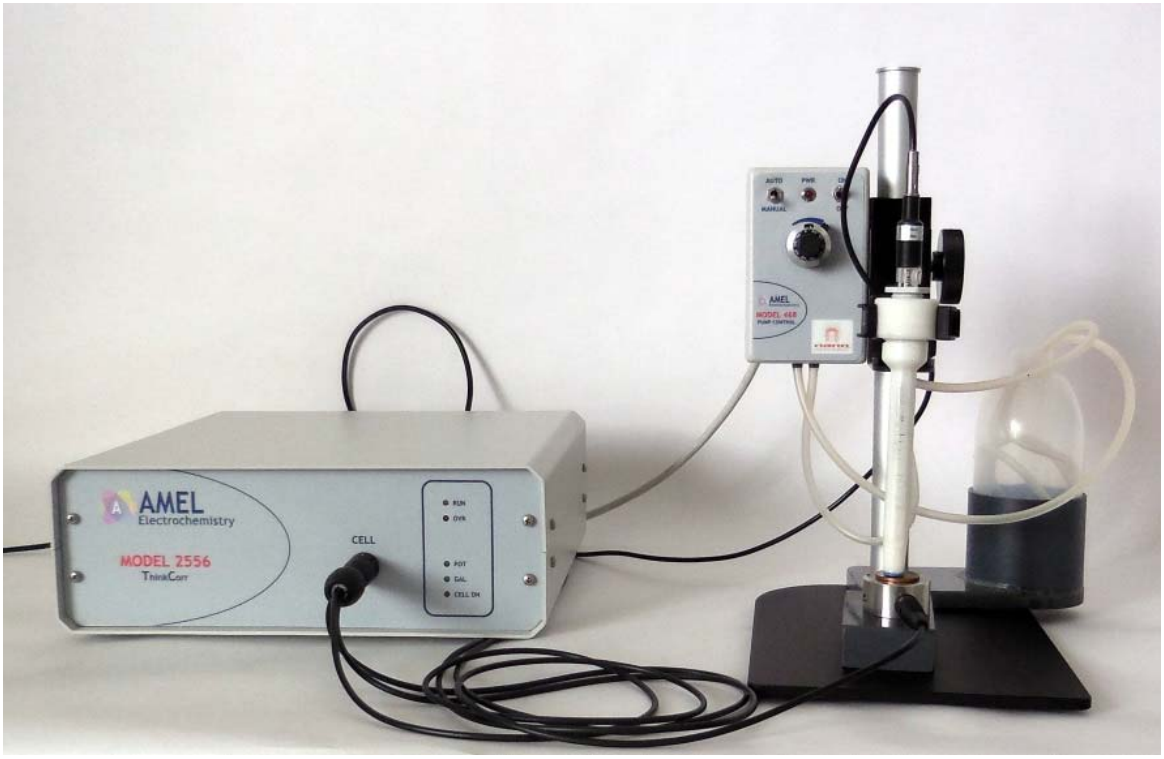

Figure 1. ThinCorr system.

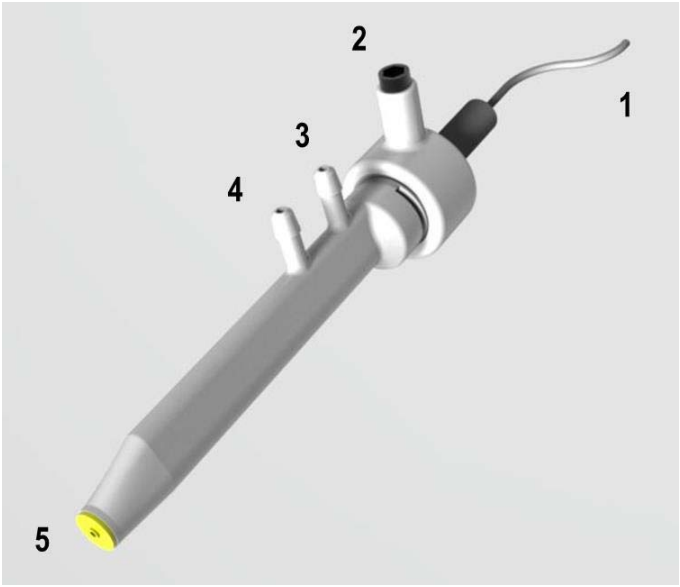

Figure 2. Rendering of EC Minicell (1: reference and counter electrode cables; 2: auxiliary port; 3: electrolyte outlet; 4: electrolyte inlet, 5: nozzle with gasket).

The performances of the ThinCorr were tested by cyclic voltammetry (10 $mV.s^{-1}$) on AISI 316, in 1M H_2SO_4 aqueous solutions at different flow rates (0, 18, 30 and 42 $ml.s^{-1}$). The results are in agreement with literature data [5].

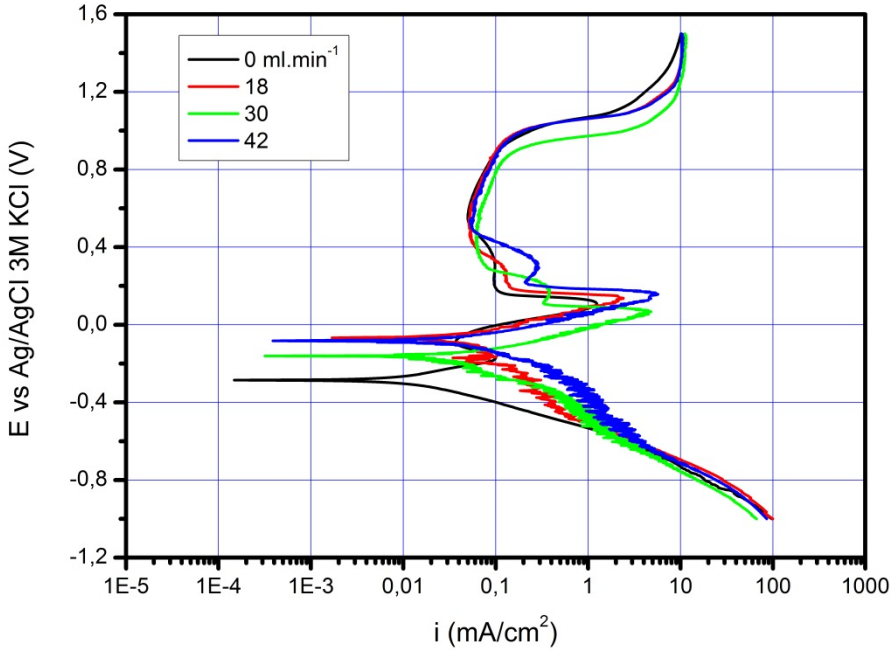

Figure 3. Potentiodynamic polarization curves on AISI 316 in 1M H_2SO_4 at 10 mV s^{-1} and room temperature. WE area 1.772 m^2, flowrate: (0, 18, 30 and 42 $ml.s^{-1}$)

In another experiment the thickness measurement of an electrogalvanized Zn coating on iron was carried out according the coulometric method (ISO 2177). The aqueous solution used for the test contained 100 $g.l^{-1}$ of KCl. The surface area was 1.54 mm^2 and the current flowing in the cell was 3 mA. The flow rate of the pump was adjusted to 10 $ml.s^{-1}$. The coulometric test provided us a value of t_{Zn} = 9.2 µm compared with 9.5 µm obtained by XRF (figure 4). A similar value (9.3 µm) was obtained by using a current of 2 mA (151 s for the voltage change).

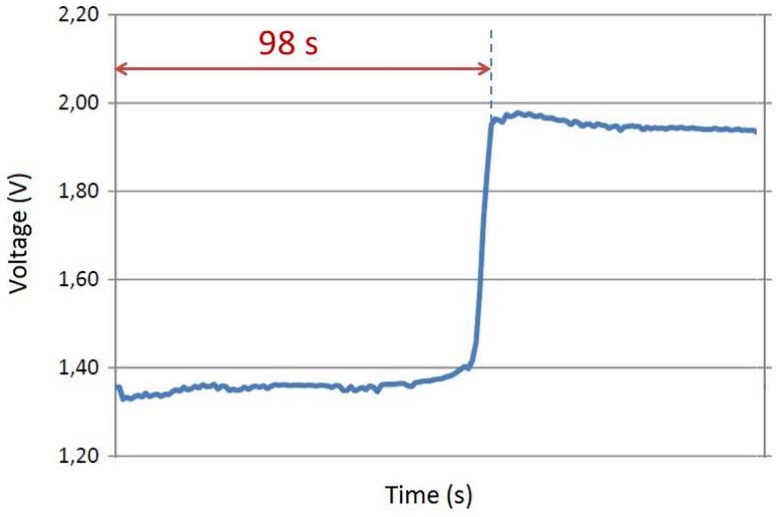

Figure 4. Thickness measurement by coulometric method.

Finally, figure 5 shows the application of the EC Minicell to a complex shape. It is displayed a microwave cavity made of an aluminum alloy undergoing a test for OCP measurement in order to detect porosities in the external silver coating.

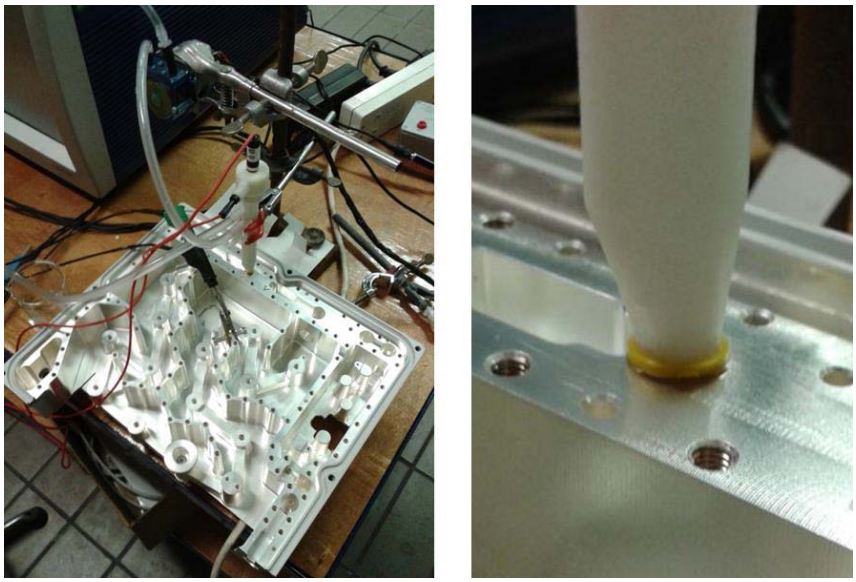

Figure 5. Testing the porosity of silver coating deposited on microwave cavity.

Conclusions

1. EC Minicell is a miniaturized, resistant, handy, and complete electrochemical cell suitable for localized electrochemical measurements.
2. Measurements are carried out with the electrolytic solution flowing from the reservoir to the WE and then back to the reservoir in a closed circuit loop. Fluid flow favors the removal of reaction products from the WE region.
3. There are not specific constraints on the types of electrochemical tests that can be performed, *e.g.* cyclic voltammetry, coulometry, and localized electrodeposition.
4. EC Minicell with a portable AMEL Potentiostat (ThinCorr) is a cheap and versatile instrument for a variety of electrochemical measurements.

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