EIS Crevice-Free Test Cell for Coated Thin Sheet Specimens

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Abstract

An innovative test cell arrangement is proposed for Electrochemical Impedance Spectroscopy (EIS) testing of coated thin sheet. This arrangement uses a multilayer medical cotton cloth as a medium for containing and transporting 5% sodium chloride solution. The novel cell avoids the intervention of edge effects into EIS measurements on painted metals. It also avoids the frequent formation of crevice attack experienced at the edges of conventional sample holders or under the edge of masking materials. This arrangement is simple and flexible and can suit different sizes and shapes of flat samples. It can also be used effectively for specimens other than thin sheets. The test cell is also suitable for all types of electrochemical test as well as impedance measurements. The EIS results reported here demonstrate the viability of the novel test cell to be used as an accelerated non-destructive, repetitive measurement on coated thin metal substrates.

Keywords: Electrochemical Impedance Spectroscopy, Coatings, Crevice Cell

Introduction

Electrochemical Impedance Spectroscopy (EIS) is by now a well known technique in the field of corrosion and is frequently used for evaluation of coating degradation for corrosion protection. The study of electrode interfaces by means of impedance and admittance techniques has been the subject of considerable research during the past 20 years [1,2,3]. In this work, EIS has been used for the evaluation of three different coating systems painted on two different types of thin metal sheets; Al and tin plated Cu. These substrates are used as friction fitted fins in air conditioner units.

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A thin sheet specimen cannot be used in conventional electrochemical testing cells as proposed by Deflorian [4] and others [5]; two main problems being apparent. Firstly, there is the possibility of the specimen edge dominating the impedance as the surface condition at the edges of flat sheet is different from that of the sheet surface. Secondly, the specimen is very thin and difficult to mount in conventional test cell arrangements. In the case of painted or coated thin sheets, the edges as supplied by the manufacturer of the air conditioning system, were not coated even after installation on the units. Specifically any metal sample has greater tendency toward corrosion at the edges than the body of the matrix because of the effect of geometrical and other considerations. In the case of thin flat coated specimens, this edge effect is much more significant because cut edges may not be effectively coated, or the coating may be thinner at the edges. In the latter case, this would result in lower impedance at the edges and would detrimentally influence the measured impedance values significantly. Therefore, it becomes essential to avoid this edge effect in order to obtain a true representation of the EIS spectrum in such specimens.

Generally, with conventional EIS test cell a sealed specimen holder is used for thin sheet samples as shown in Figure 1.
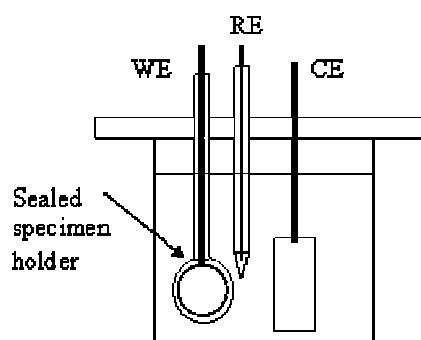

Figure 1: Conventional electrochemical cell with sealed specimen holder.

This arrangement avoids the edge effect but gives rise to the formation of a new problem of crevice near the edge of sample holder gasket. The same problem appears sometimes when the edges of sheets are masked with some types of non-conducting material.

There are different approaches to solve this problem. Others [6] have developed a crevice free test cell, which works on the principle of a flooded gasket seal; i.e. the working electrode area is confined by the gradually increasing concentration of contact electrolyte from the cell electrolyte solution to deionised water. However, the drawback in this design

of test cell is the limitation on the dimensions of the specimen to be tested to approximately (50 mm x 50 mm). This is not applicable for large thin sheet panels.

The main objective of our work was to develop test cells that can accommodate actual corrosion coupons of size 150 x 100 mm. These are similar samples as those used for atmospheric exposure as well as in the laboratory accelerated weathering corrosion cabinet test. The emphasis also has been directed to find a non destructive test sample that can provide real time repetitive measurements using the above-mentioned environments under the newly proposed design of EIS testing cell.

Electrochemical Test Cell Setup

The proposed novel setup of the test cell is as shown in Plate 1 along with the schematic drawing in Figures 2 and 3. The EIS cell is composed of a rectangular mounting stand of Perspex sheets. The thin flat sheet specimen to be tested is mounted on the stand, where the testing side or working electrode surface faces upwards. A clean cotton cloth is laid down and stretched over the specimen or the working electrode surface. A graphite plate of the same size as that of the specimen is placed on the cloth, which acts as a counter electrode. Electrical contacts between the working electrode and the graphite plate are made with the help of screwed nuts and wires ending with crocodile clips. One end of the wet cloth is immersed in a beaker filled with the electrolyte solution (e.g. 5% NaCl in de-ionized water). The other end of the cloth rests freely in another empty beaker.
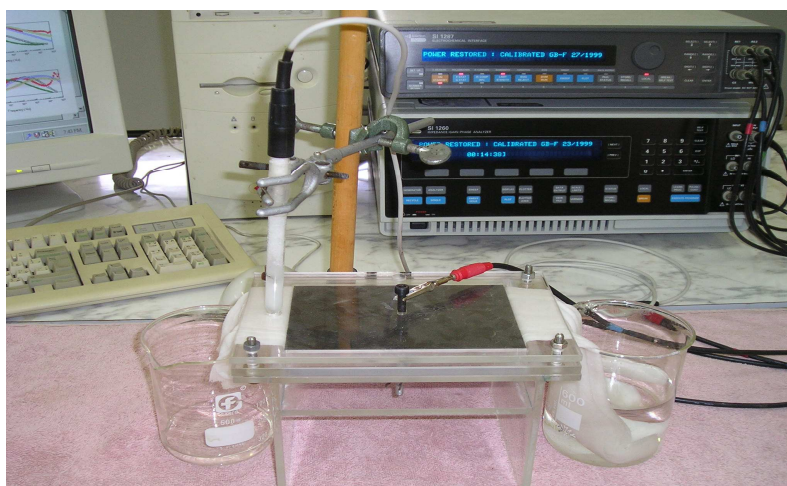

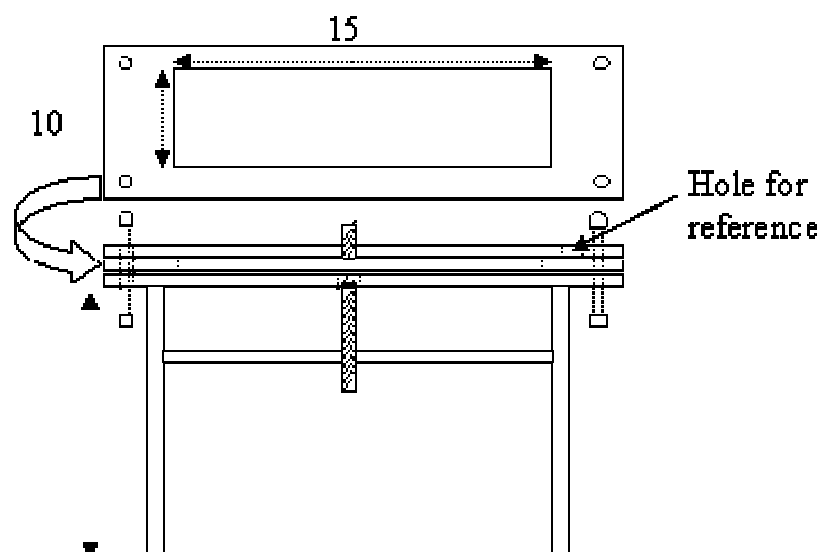Plate 1: Photograph of the new cell arrangement

Figure 2: Schematic testing cell arrangement for EIS electrochemical testing of thin flat sheets
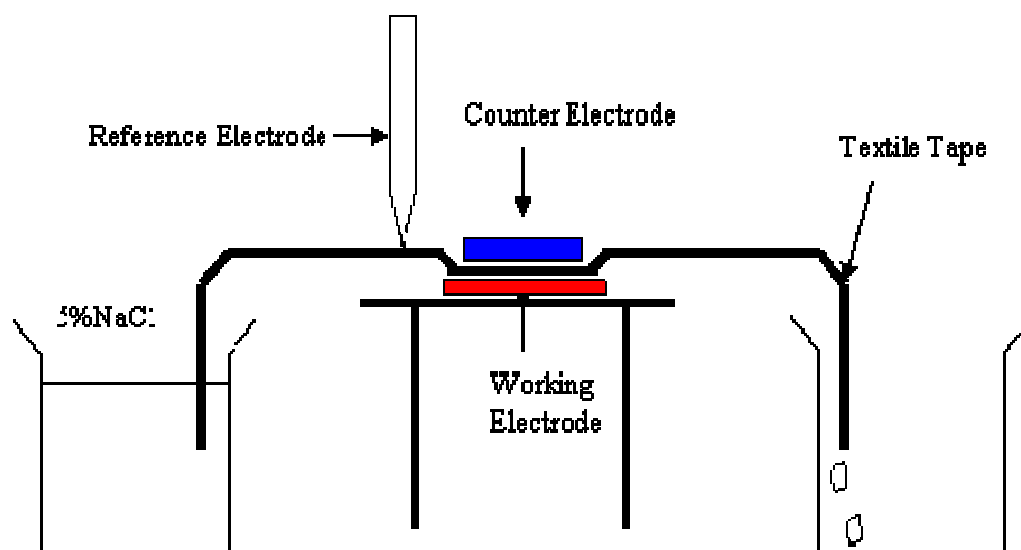

Figure 3: Side view of EIS test cell showing specimen mounting arrangement

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This whole test arrangement helps a continuous flow of electrolytic solution to be established from one beaker to another diffusing through the cotton cloth and passing over the specimen while wetting the surface via capillary action. A small hole is made on the upper cover Perspex plate to accommodate the reference electrode tip, which is placed touching the wetted cloth.

Materials and methods

Triplicate thin sheet specimens were prepared for EIS testing. Two types of metal sheets were used they were mill finished aluminium (MF) and tin–plated copper (TC) of approximate dimensions 10 cm x 15cm. The average thickness of the aluminium mill finished substrate was 100 μm while the average thickness of the tin plated copper sheet was 200 μm. Both types of thin sheets were spray coated in the laboratory with "Protecta" as an example of a poor quality coating. The dry film thickness of the coated aluminium mill finish (MP) samples was 7.5 μm and the thickness of the coated tin copper (TP) was approximately 4.4 μm. The Protecta coating is a commercial quality coating system based on silicon–modified polyester and used for the protection of aluminium fins and copper coils in air condition systems. Another set of thin aluminium test sheets were factory applied and pre–coated with a blue hydrophilic coating of approximately 2–3 μm. These pre–coated panels are designated as (PA). The as–received uncoated flat sheet samples were also tested using the novel EIS test cell.

A Solartron electrochemical Interface (Potentiostat/Galvanostat model 1287), together with a Solartron Frequency Response Analyzer (FRA 1260) were used for the electrochemical impedance measurements. AC impedance was measured over a frequency domain ranging from 100 kHz to 10 mHz. The amplitude of the AC signal was 10 mV and was applied at the open circuit potential.
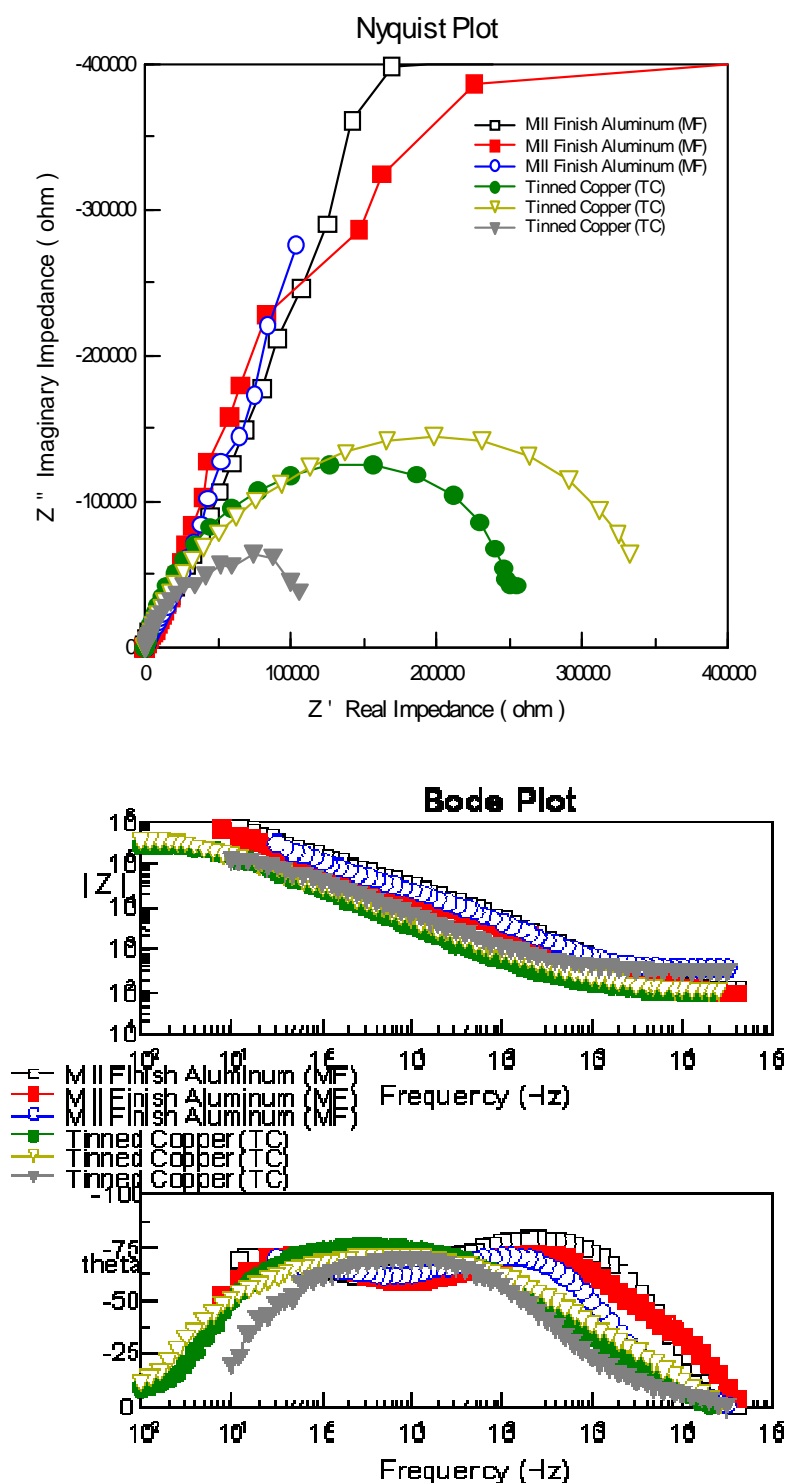
Figure 4: EIS Nyquist and Bode plots for Al and Cu fins for the
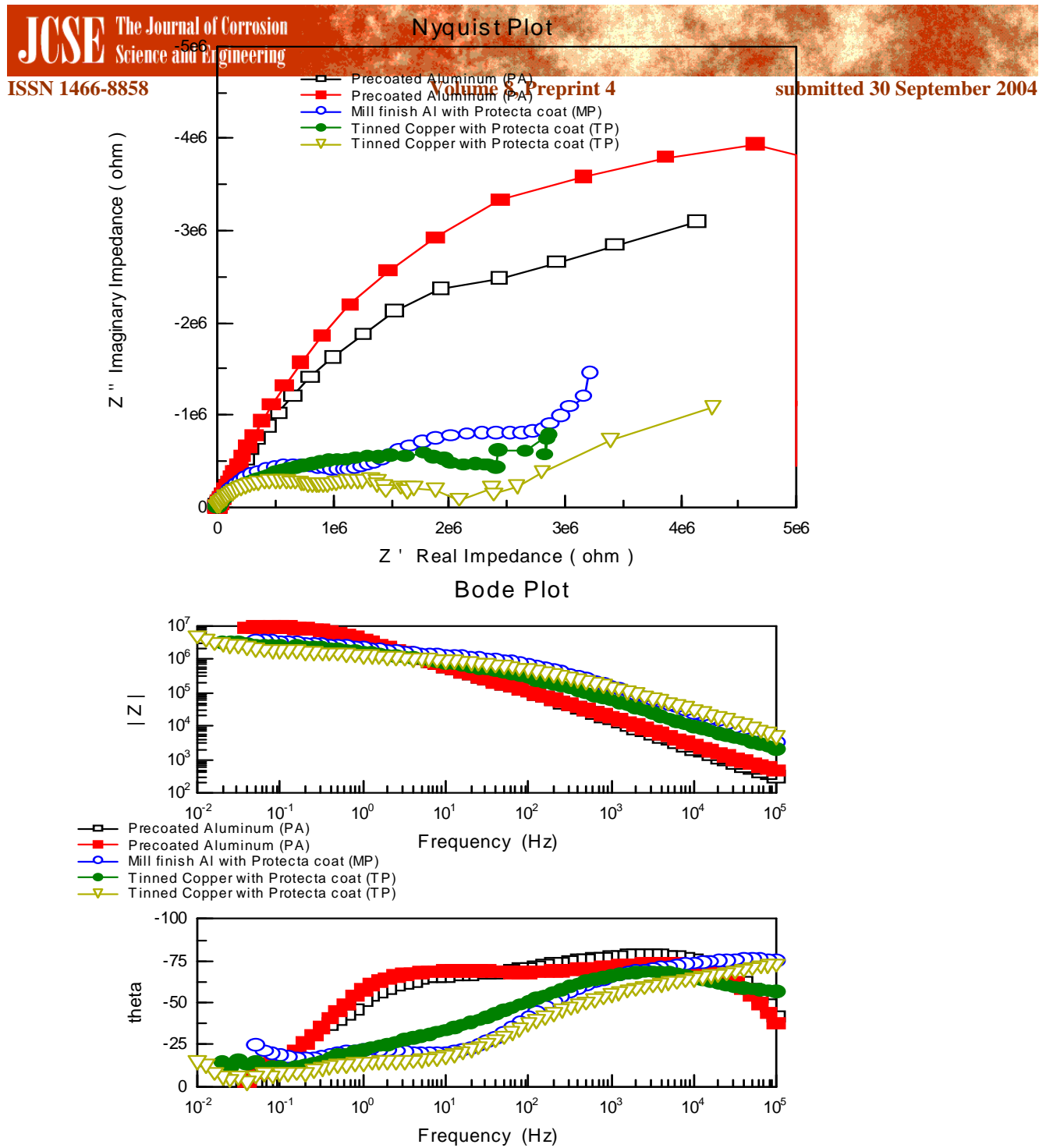as-received un-coated specimens

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Nyquist Plot

Bode Plot

Figure 5: EIS Nyquist and Bode plots for the two different coating systems.

Sheet Material	Mill finish Al, (MF)	"Protecta" coated Al, (MP)	Factory coated Al, (PA)	Tinned Copper, (TC)	"Protecta" coated copper, (TP)
R_{coat} (MΩ.cm^2)	–	430	980	–	120
C_{coat} (pF/cm^2)	–	50	280	–	60
R_p (MΩ.cm^2)	350	–	–	40	
C_{dl} (nF/cm^2)	15	–	–	40	
icorr (µA/cm^2)	0.07	–	–	0.67	

Table 1: Capacitance and Impedance parameters obtained from the EIS Nyquist plot.

Results and Discussion

The results of the AC impedance testing are presented in Figures 4 and 5. The corrosion rates obtained from R_p values for uncoated samples and coating parameters for coated samples are given in Table 1.

The results for coated samples show that the factory pre-coated (PA) aluminium samples have relatively high impedance values. This demonstrates the presence of uniform and good quality coating system. In contrast samples with a spray applied "Protecta" coat show low impedance values and thus generally inferior quality coatings.

In the case of the uncoated samples, EIS impedance tests show that the mill finish (MF) aluminium has a lower corrosion rate than that of tinned copper (TC). Subsequent laboratory accelerated corrosion test (ASTM B 117) has also shown that tinned Cu is much more vulnerable to corrosion than Al. This is apparently unexpected where the difference in corrosion behaviour of this material is attributed to the tinned plated surface layer being anodic to the copper substrate, where it protects the copper in the earlier stages of corrosion. However, the tinplate layer contains an appreciable amount of discontinuities that may be reflected in the observed impedance response, enhancing. Therefore, the phenomenon may be attributed to the nature and corrosion mechanism of the tin plated layer; that is, it corrodes at faster rate at the later stage. Unlike the tinned copper specimen, the aluminium flat sheet specimen showed the presence of intact air formed oxide film

characteristic of Al alloys having amphoteric properties which provide excellent surface protection in 5% NaCl solution.

A good correlation is found between the EIS test results and the subsequent laboratory accelerated corrosion test (ASTM B 117) results for coated specimens.

Conclusions

1) The electrochemical EIS test cell arrangement suggested in this study is simple, flexible and provide effective solution for avoiding edge effect and crevice formation.

2) The same test cell can be used as non destructive measurement of impedance with actual sample dimension as well as on samples retrieved from field exposure and laboratory salt spray cabinet tests.

3) EIS test results show good agreement with laboratory accelerated corrosion test (ASTM B117) for all specimens.

4) The EIS test results on the factory pre-coated Al (PA) samples show the best coating protection performance among the rest of the coating systems studied.

References

1) "The application of AC impedance to study the performance of lacquered aluminium in acetic acid solutions", J.D. Scantlebury, K. Galic, *Progress in Organic Coatings*, **31**. pp. 201-207 (1997).

2) "Interpretation of low frequency AC impedance data for organic coatings on mild steel", B.J. Hepburn, K.R. Gowers, J.D. Scantlebury, *British Corrosion. Journal,* **21**, pp.104-108 (1986).

3) "Basic aspects of electrochemical impedance application for life prediction of organic coatings on metals", M.W. Kendig, J. Scully, *Corrosion Science*, **46**, 1, pp. 22-28 (1990).

4) "Evaluation of Organic Coatings by Electrochemical Impedance Measurements", Application Note: AC-2, Electrochemical Instruments Group, EG&G Princeton Applied Research, Princeton, New Jersey, (1984).

5) "Corrosion behaviour of epoxy coated galvanized steel", F. DeFlorian, L. Fedrizzi, P.L. Bonora, in "Progress in the Understanding and Prevention of Corrosion", Edited by J.M. Costa and A.D. Mercer, Vol.1, (1993).

6) "Flex Cell Critical Pitting Test Cell Kit", Operator's Manual, Gamry Instruments Inc. PA, USA, (1999).