

**DIRECT ASSESSMENT OF LOCALIZED CORROSION USING LOCALIZED CORROSION  
MONITORING TECHNIQUE**

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**ABSTRACT**

The control of internal corrosion of carbon steel structures is a critical element of asset integrity management program in oil and gas production facilities. The selection of a corrosion inhibitor for field deployment is typically based on laboratory screening tests, followed by field trials. This paper describes some of the experience gained in using new localized corrosion monitoring (LCM) technique in laboratory beaker and flow loop tests to differentiate the capability of corrosion inhibitors in controlling localized corrosion. A new data analysis program was employed to identify the occurrence of localized events in terms of magnitude, duration and distribution. This information is used to assess severity and time and spatial distribution of localized corrosion activities. Good correlation with post exposure metallographic examination of the test electrodes was obtained. Examples of monitoring localized corrosion processes including mild steel and stainless steel in various environments are also presented.

**Key Words:** localized corrosion, monitoring, corrosion inhibitors

## INTRODUCTION

Corrosion can manifest in the form of general or localized corrosion. The direct cost of corrosion in the US was reported to be \$276 billion per annum in a recent study published in July 2002<sup>1</sup>. An earlier study also suggested that localized corrosion accounted for 80% of process plant failures<sup>2</sup>. However, it is recognized that corrosion can be controlled if appropriate measures are implemented. For example, it is common industrial practice to deploy corrosion inhibitors to control internal corrosion of carbon steel structures in oil and gas production facilities. One critical component of any corrosion management program is the measurement and verification of the effectiveness of the corrosion control strategy. Such measurements may be by means of direct or indirect methods, e.g. weight loss coupons, electrical resistance measurements, electrochemical monitoring, non-destructive inspection and potential measurements, etc. Selection of the most appropriate techniques is dependent upon the service environment as well as the type of information required.

From a practical perspective, general corrosion is relatively easier to monitor and to predict; whereas due to the random nature of localized corrosion, it is more difficult to monitor. The conventional approach in corrosion monitoring is mainly based on retrospective analyses, typically weight loss measurements and inspection, etc. Although the information may appear to be reliable and the data may be used to trend the corrosion behavior over time in the case of general corrosion, such information may not be relied upon to provide longer term representative behavior of localized corrosion. This is because localized corrosion events, such as pitting, do not corrode at a constant rate. The localized corrosion activity (e.g. pitting) can occur in a recurring process of initiation, propagation and repassivation.

In recent years, electrochemical corrosion monitoring techniques are gaining wider applications in evaluating localized corrosion. The techniques employed include potentiodynamic polarization scans<sup>3</sup> and, in particular, electrochemical noise<sup>4</sup> (EN) measurement, which is based on the monitoring of corrosion potential and current fluctuations. Analysis of the data can provide information on pit initiation, propagation and repassivation behavior<sup>5</sup>. A number of analytical methods have been developed to assist the identification of the likelihood of occurrence of the localized corrosion events, which are either based on statistical analysis of the EN data<sup>6-10</sup>, or digital signal processing/transformation techniques to provide corrosion information<sup>11-15</sup>. The statistical parameters obtained from the digital signal processing techniques include localization index, skew, kurtosis, coefficient of variations, characteristic charge/frequency and pitting factor. There were reports of some degree of success with these types of analyses in identifying localized corrosion behavior<sup>16, 17</sup>. The merits and limitations of the parameters were also discussed in literature<sup>18</sup>.

In localized corrosion it is important to determine the occurrence of localized events in-situ and to provide information on the severity and spatial distribution of these events. In the present study of localized corrosion behavior of mild steel in oxygen (O<sub>2</sub>) and carbon dioxide (CO<sub>2</sub>) environments and stainless steel Alloy 304 (UNS S30400) in sodium chloride (NaCl) solution, a novel monitoring system, Localized Corrosion Monitoring (LCM<sup>(1)</sup>), was employed. The LCM is based on a recently developed corrosion monitoring technique, potentiostatic electrochemical noise<sup>19-21</sup>, and the incorporation of data analysis software<sup>22</sup> to provide in-situ quantitative information of the localized corrosion events, which include the magnitude, distribution and relationship between these events. The monitoring technique is based on the measurements of potential and current data on one working electrode at/near open circuit potential. It operates in an alternating mode of open circuit measurements and potentiostatic control. With the aid of the data analysis, the severity and the occurrence of potential and current transients associated with various types of localized corrosion can be identified. This paper presents three studies undertaken to demonstrate unique capabilities of the LCM to identify and quantify localized corrosion.

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<sup>(1)</sup> LCM<sup>TM</sup> is a trademark of Baker Petrolite

## EXPERIMENTAL PROCEDURE

Three corrosion systems were investigated. The first 2 sets of tests were designed to promote pitting corrosion and to validate the corrosion information obtained with LCM system. Another set of tests was conducted to evaluate the performance of candidate corrosion inhibitors in simulated oil production environment. Details of the tests are as follows:

### **Carbon Steel/NaCl, O<sub>2</sub>**

The first set of tests was the exposure of carbon steel grade C1018 (UNS G10180) in 0.1% sodium chloride (NaCl) solution containing 100 ppm nitrite (in the form of sodium nitrite), at a constant temperature of 50°C. Alloy 276 (UNS N10276) material was used as reference and counter electrodes. Prior to immersion into the test solution, the carbon steel electrode was polished to 1200 grit surface finish, degreased, rinsed with water followed by acetone and dried with air. The carbon steel test electrode was then immersed into the test solution and the corrosion behavior was monitored throughout the duration of the exposure test. The surface morphology of the carbon steel test electrode after the test was then examined using an optical microscope to determine the extent and type of corrosion damage.

### **Stainless Steel/NaCl, O<sub>2</sub>**

The set-up of the test was similar to the pitting corrosion test of carbon steel, but the pitting corrosion behavior of Alloy 304 was investigated. The stainless steel test electrode was prepared using the same procedures as described above prior to being exposed to 1% NaCl solution at 50°C. On completion of the test, the surface appearance of the stainless steel test electrode was also examined using an optical microscope.

### **Carbon Steel/Brine, CO<sub>2</sub>**

The performance of corrosion inhibitors to control general and localized corrosion was evaluated using a laboratory high shear flow loop<sup>2</sup> to simulate turbulent operating conditions of a flow line with mixed brine/oil phases. The main components of the high shear flow loop are a closed tube system with a reservoir, a flow cell incorporating a test probe assembly, a circulating pump, a make-up pump with a make-up reservoir and a gas supply system. The circulating pump passes the liquids through the flow cell, which consists of a rectangular flow channel (3 cm wide x 3 cm high) containing a flat, flush mounted, carbon steel test probe assembly. The shear stress applied can be derived from the pressure drop in the flow cell.

The inhibitor tests were conducted using a mixture of 80% synthetic brine and 20% crude oil. The composition of the test brine is tabulated in Table 1. The test temperature was 80 °C, the CO<sub>2</sub> partial pressure was 1.4 bars and the shear stress acting on the specimen was 150 Pa. The brine/oil mixture was initially deaerated using carbon dioxide. A probe assembly incorporating a carbon steel test electrode polished to 320 grit surface finish was mounted into the test section. There was no period of uninhibited corrosion (pre-corrosion), the inhibitor was added to the flow loop content from the onset of the test.

Two corrosion inhibitors were evaluated in this series of tests. Each test was carried out over a 7-days period. In both sets of tests, the concentration of corrosion inhibitor was 100 ppm/v. The test fluid was allowed to circulate in the flow loop, and electrochemical measurements commenced when the system conditions became stabilized. LCM measurements were made to evaluate the corrosion behavior. In

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<sup>2</sup> Test equipment operated by Shell Global Solutions.

addition, periodic measurements by using linear polarization resistance (LPR) and AC impedance, which are the standard techniques adopted in this type of evaluation tests, were also made to complement the LCM data.

## RESULTS AND DISCUSSION

### LCM Potential/Current Transient Analysis

In a typical LCM experiment, both potential and current are alternatively recorded with time using 30 seconds on (current) and 30 seconds off (potential) potentiostatic control/open circuit potential sequence. LCM relies on the measurements of time of occurrence, magnitude, duration, frequency and distribution of distinct potential (negative) and current (positive) transients as a result of initiation and/or propagation/repassivation of localized corrosion events (e.g. pitting, crevice). Based on the magnitude, duration and relative rate of decrease/increase of potential and current signals, four different types of transients can be observed in the LCM time records: (i) initiation/propagation (IP), (ii) initiation/partial repassivation (IPR), (iii) initiation/repassivation (IR) and (iv) initiation/repassivation/propagation (IRP) transients are observed. This transient analysis of the potential/current time dependence will be used in quantifying localized corrosion activity on the carbon steel and stainless steel tests.

**Type I:** Potential IP transients are characterized by a sudden decrease in open-circuit potential, i.e. pit initiation (1-3 sec), followed by a slow increase in potential (> 30 sec), i.e. pit propagation, close to or lower than its original value. The typical decrease in potential is < 3 mV. The corresponding current transients, whether preceding or following the potential transients can vary significantly depending on the localized corrosion activity (0.1 – 100  $\mu$ A). The lower the ratio of the magnitudes of potential and current transients ( $R_t$ ), the more active the pit and greater the area affected. A typical potential/current IP transient is presented in Figure 1a showing sharp decrease (pit initiation) and subsequent slow increase (pit propagation) in potential accompanied with the current peak. Pits that grow by this mechanism are generally most active, non-uniformly distributed, large and deep (see discussion on corrosion inhibitor).

**Type II:** Potential IPR transients can be described in terms of sudden, decrease in open-circuit potential (< 3 mV) followed by a slow increase in potential to, higher or lower than the initial open-circuit potential. These transients can extend over much larger time periods (> 1000 sec) compared to the potential IP transients. The corresponding current transients show both larger current initiation (increase) and lower current partial repassivation (decrease) signals. The typical current increases during these transients are < 10  $\mu$ A. Figure 1b depicts one of the IPR potential/current transients showing pit initiation/partial repassivation. Pits formed by the IPR mechanism are generally active, more uniformly distributed, smaller and more shallow (see discussion on corrosion inhibitors).

**Type III:** Potential IR transients can be characterized by a rapid and generally larger decrease in potential (2-100 mV) associated with an equally fast increase in potential to its original value within few free potential/potential hold cycles. The corresponding current transients (<1-2  $\mu$ A) show equally strong positive (initiation) and negative (passivation) signals (Figure 1c). Typical IR transients are associated with passive, numerous, uniformly distributed extremely small pits (see discussion on stainless steel in NaCl).

**Type IV:** Potential IRP transients can be described in terms of a steady and large decrease in potential (10-50mV) followed by a slow increase in potential to a level that is significantly lower than the initial open-circuit potential. These potential transients extend over much larger time periods 1-10 cycles) compared to the potential IP, IPR and IR transients. The IRP current transients show generally successive repassivation and propagation associated with multiple localized corrosion events. Figure 1d depicts one of the IRP transients showing pits initiation/repassivation and continuous propagation of certain number of them. Pits formed by the IRP mechanism are generally in very large number, more

or less active, uniformly distributed, smaller and more shallow than the IP and IPR (see discussion on corrosion inhibitors).

### **Pitting Corrosion of Carbon Steel**

In this system, partial passivation of the carbon steel electrode was expected because of insufficient amount of nitrite used (100 ppm) in the test solution. Detail examination of the time records of both potential and current showed periods of high localized corrosion activities as indicated by the presence of number of transients (Figure 2). The data revealed three periods of significant pitting activities (< 2.2 hours, 6.4 – 7.5 hours, 13 – 14.2 hours). During the first two time periods numerous distinct potential IPR (active) and IR (passive) transients were observed (Figure 3), while only two IPR transients were recorded in the latter part of the test. Figure 4 shows 7 potential IPR and 13 IR transients with 3 IPR transients preceding 7 IR transients and 2 other IPR transients preceding 6 remaining IR transients. Two last IPR transients (Figure 4a) occurred towards the end of the test to be indicative of any significant localized activity.

These data are compared with the detailed microscopic examination of the surface of the test specimen with regard to localized corrosion, i.e. presence and nature of pits as shown in Figure 5. There were altogether 5 larger pits surrounded by greater number of smaller secondary pits and possibly 5 smaller incipient pits. 3 of the large pits were in close proximity of each other. The pit depths were within 4 – 6  $\mu\text{m}$ . The surface morphology showing 5 distinct pits with unspecified number of small pits are in good agreement with the number of potential IPR transients (5) and IR transients (10) recorded. The additional IPR (3) and IR (2) transients that occurred much latter in the test are probably 5 small pits observed under the microscope.

### **Pitting Corrosion of Stainless Steel**

Another set of pitting test conducted was the immersion of stainless steel Alloy 304 in 1% NaCl solution. The potential and current test data recorded suggested extensive activities throughout the duration of the test, Figure 6. Typical potential and current transients observed are shown in Figure 7. All potential transients are IR type, i.e. characteristic of small, shallow and uniformly distributed passivated pits (Figure 1c). With the aid of LCM analysis, the results showed that this material did not exhibit the same behavior as that shown in the carbon steel/nitrite test discussed above. The analyzed data indicated that there were numerous initiation/passivation events ( $\sim 200$ ), while neither initiation/propagation nor initiation/partial repassivation events were observed (Figure 8). In comparison to the carbon steel/nitrite system discussed above, the time distribution of the localized events indicated that they were relatively more evenly spread.

The behavior observed in this test suggested that the alloy was susceptible to pitting corrosion in the test environment. This was confirmed by microscopic examination of the surface of the electrode following completion of the test. There were much higher numbers of small pits randomly distributed across the surface than in the case of carbon steel/nitrite test, Figure 9. The diameters of these corroded sites were generally only a few  $\mu\text{m}$ , though a few relatively larger diameter pits were also present, but those were still very small, <10 $\mu\text{m}$ . The pit depths were very low, below the sensitivity of the optical microscope, to be measured with accuracy.

### **Corrosion Inhibitor Evaluation**

The first set of test was conducted on carbon steel using Inhibitor A. LCM measurements were made in periods between LPR and AC impedance measurements. The potential and current time records exhibited minor and random fluctuations at the start of the test, which were typical of general corrosion. The occurrence of localized activities was observed a few hours into the tests. However, the overall duration of these episodes of increased activities was relative short, generally less than 20 seconds. The short potential transients with slow recovery are typical for pit initiation/propagation

events as shown in Figure 1a. An example of current and potential transients observed is shown in Figure 10.

The appearance of transients became more frequent on day 2. Generally, the amplitude of the potential transients was typically between 2 to 5 mV, and the majority was >2 mV (Figure 11a). The occurrence of these characteristic IP transients increased with time and reached a maximum on day 3 of the test. The frequency then showed a decreasing trend and was at the minimum on day 5. A reverse back to an upward trend was then observed at the end of the test. The time transient plot suggested that the transients tended to occur in clusters. This feature was similar to that observed in the carbon steel/nitrite tests, and it is indicative of a more localized nature of the attack suggesting that the individual pits are occurring in close proximity to each other within the cluster.

The calculated corrosion rate for the first 3 days of the test suggested that it was between 0.3 to 0.6 mm/year, (calculations based on noise resistance method and the total surface area of the test electrode), which was in good agreement with the data obtained with LPR/AC impedance measurements. However, the actual active area would be expected to be much smaller, hence the actual pitting rate could be many times higher than that estimated.

Examination of the test electrode after the test suggested that corrosion damage was mainly in the form of deep cut grooves of length 5 - 37 mm, width 190 - 380  $\mu\text{m}$  and depth 60 -80  $\mu\text{m}$  (Figure 12). In addition to 4-5 such deep groove-like pits along the polishing marks, there were also a number of isolated pits with lower extent of penetration. These large groove-like pits show large number of smaller pits within the inner part of their periphery suggesting that the mechanism of growth is the propagation of many smaller overlapping pits. This is consistent with the occurrence of potential and current IP transients in close proximity to each other within the clusters (4) and long periods without any additional new localized activity. It should be noted that no other type of transients were observed during the test. In this cases, the actual active surface area is expected to be very small that the corrosion rate can be many times higher than the rates obtained using conventional measurements, e.g. LPR and AC impedance monitoring techniques.

A second set of evaluation test was undertaken using Inhibitor B. The occurrences of potential and current transients, indicating localized activities, were evident at the start of the test. The LCM data also exhibited significant potential swings or fluctuations throughout the duration of the tests, characteristic of potential IPR and IR transients (Figure 13) which was not observed in the earlier test on inhibitor A. The swing of the potential data is considered to be indicative of occurrence of both types of transient intermittently on the surface of the test electrode, leading to two types of pitting. No attempt was made to differentiate between them.

In this set of test, a similar frequency profile to that of Inhibitor A was obtained (Figure 14a). The profile showed an initial trend of increasing frequency, followed by gradual decrease and then returning to higher frequency at the end of the test. Overall, the number of transients obtained was lower than with Inhibitor A. The amplitude of the IPR potential transients was much higher in this test, generally between 6 – 13 mV, but associated with much smaller current transients (2  $\mu\text{A}$  versus 10  $\mu\text{A}$  with Inhibitor A), indicating relatively less intense localized activities. Overall, when both the distribution of frequency and the amplitude of these transients were taken into consideration, the data indicated more uniformly distributed localized corrosion with larger number of more shallow pits (Figure 15).

Spot corrosion rate calculations were again made based on the whole surface area of the test electrode. The corrosion rate was in the range 0.04 – 0.2 mm/year. The variation in corrosion rate over time is presented in Figure 16. The test data with inhibitor B suggested that the “general” corrosion rate was significantly lower than that with inhibitor A. The corrosion rate profile of AC impedance was overlaid onto Figure 16 showing the same trend. The LCM data were again in good general agreement with the microscopic observation. Examination of the test electrode on completion of the test revealed pitting occurred more uniformly than the previous tests, and the pit depth was about 10 times lower.

## CONCLUSIONS

A new LCM system and transient analysis were developed to characterize localized corrosion behavior. The following conclusions can be drawn from the results presented in this paper:

- The results of localized corrosion tests with carbon steel in inhibitor containing solution and stainless steel in sodium chloride solution demonstrated the effectiveness of the LCM to identify and characterize localized corrosion events with respect to number, magnitude, time of occurrence and spatial distribution.
- Quantitative analysis of the transients was employed to evaluate the localized corrosion, which allowed a better assessment of the pitting corrosion to be made. The analysis provided a more direct assessment of the localized events including number of pitting events, pit distribution and type of pitting when compared to statistical parameter analysis (e.g. localization index) used.
- The results showed good correlation between the analysis of the number, type and distribution of individual transients and the microscopic evaluation of the metal surface.
- The calculated “general” corrosion rate derived with the LCM data, (calculations based on the full surfaced area), were in good agreement with the convention electrochemical techniques, such as LPR and AC impedance.

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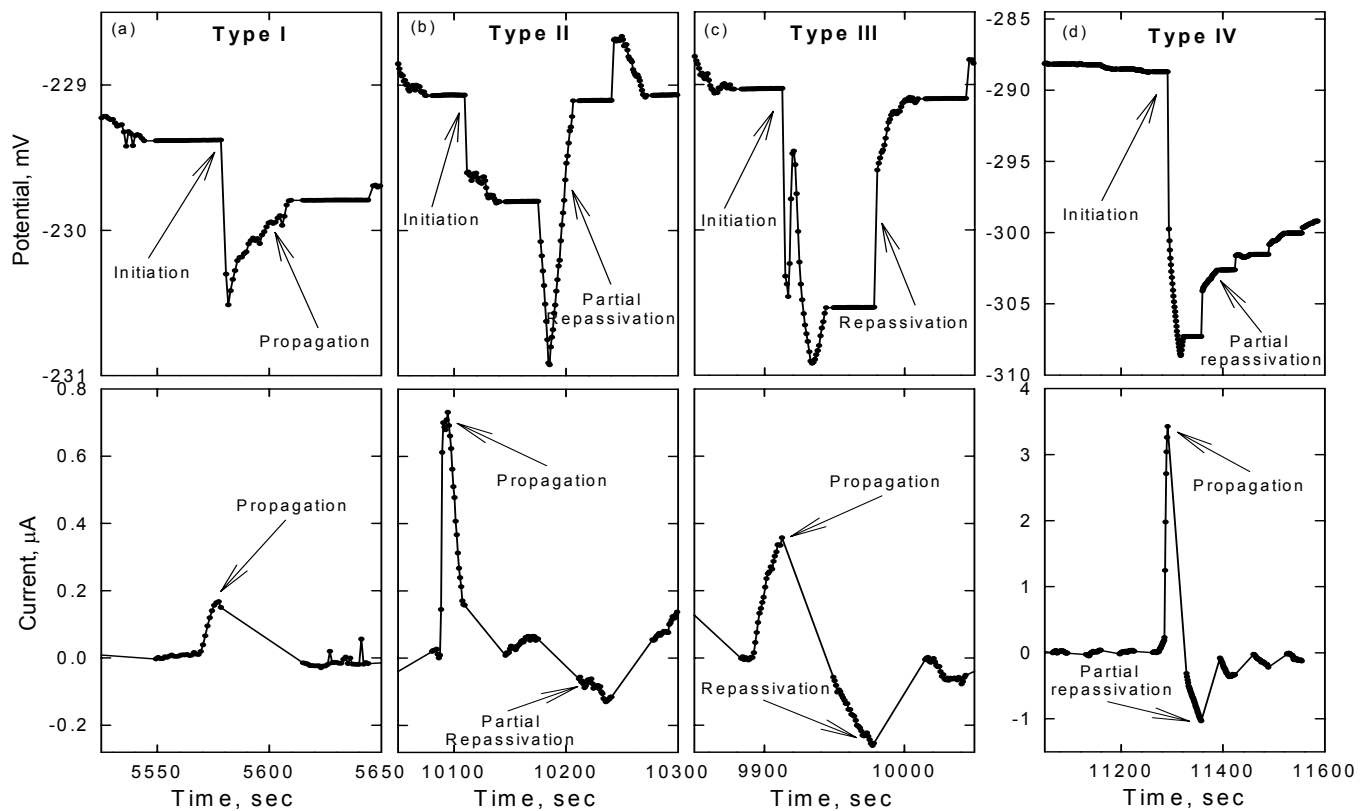
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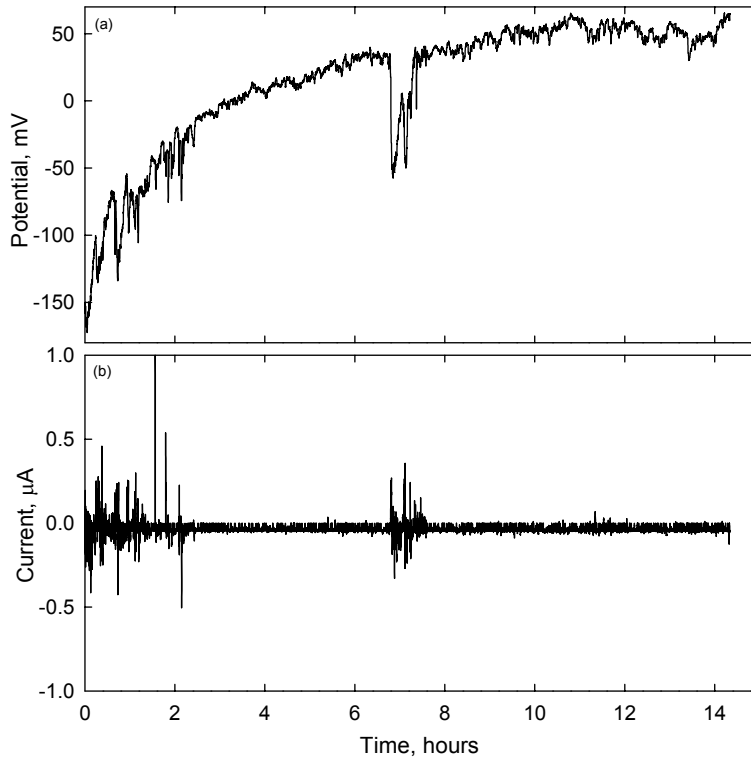


**TABLE 1**  
**Water Chemistry**

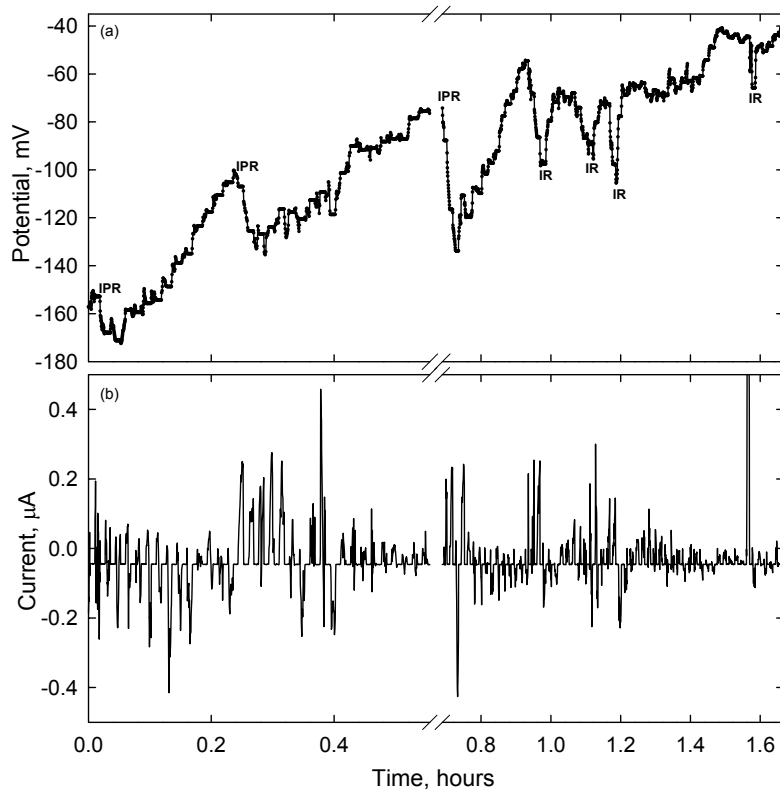
Chemical	g/litre
Chloride	52
Calcium	2.1
Magnesium	0.54
Acetate	0.45
Sodium	30.3
Potassium	0.32



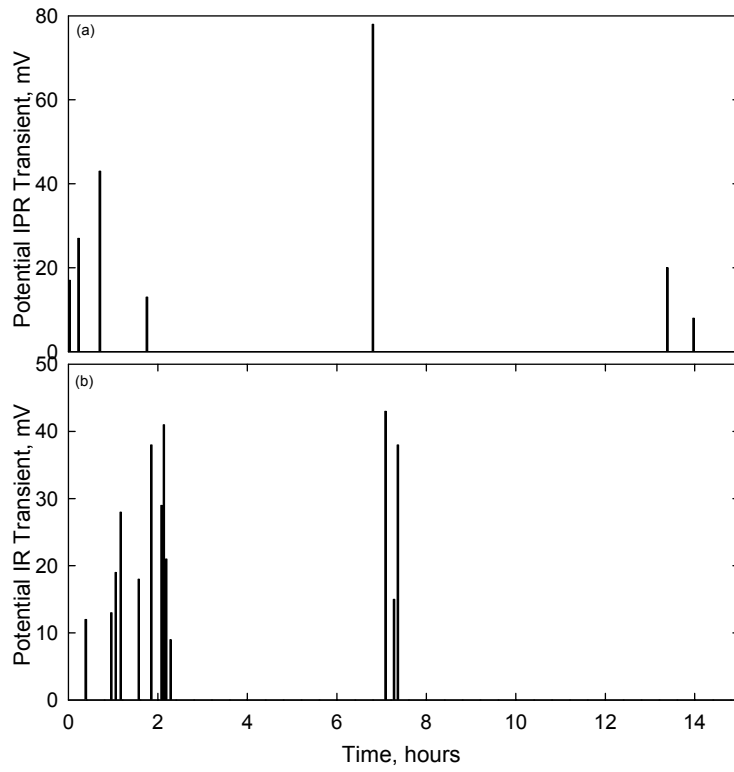
**Figure 1. Four characteristic potential/current transients for pit (a) initiation/propagation (IP transients), (b) initiation/partial repassivation (IPR transients), (c) initiation/repassivation (IR transients) and (d) initiation/repassivation/propagation (IRP transients).**



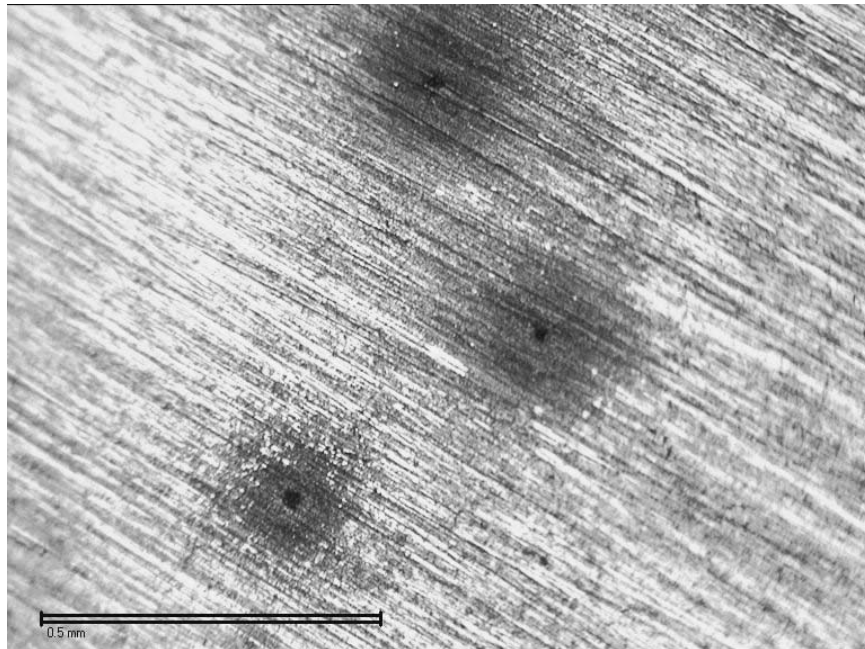
**Figure 2. Potential and current time records for carbon steel in 0.1% NaCl/100 ppm  $\text{NO}_2^-$  test.**



**Figure 3. Potential and current time records for carbon steel in 0.1% NaCl/100 ppm  $\text{NO}_2^-$  test (1.7 hours) showing 3 potential IPR and 4 IR transients.**



**Figure 4. Time profile of potential IPR and IR transients for carbon steel in 0.1% NaCl / 100 ppm NO<sub>2</sub><sup>-</sup> test.**



**Figure 5. Micrograph of carbon steel test electrode, showing pitting damage.**

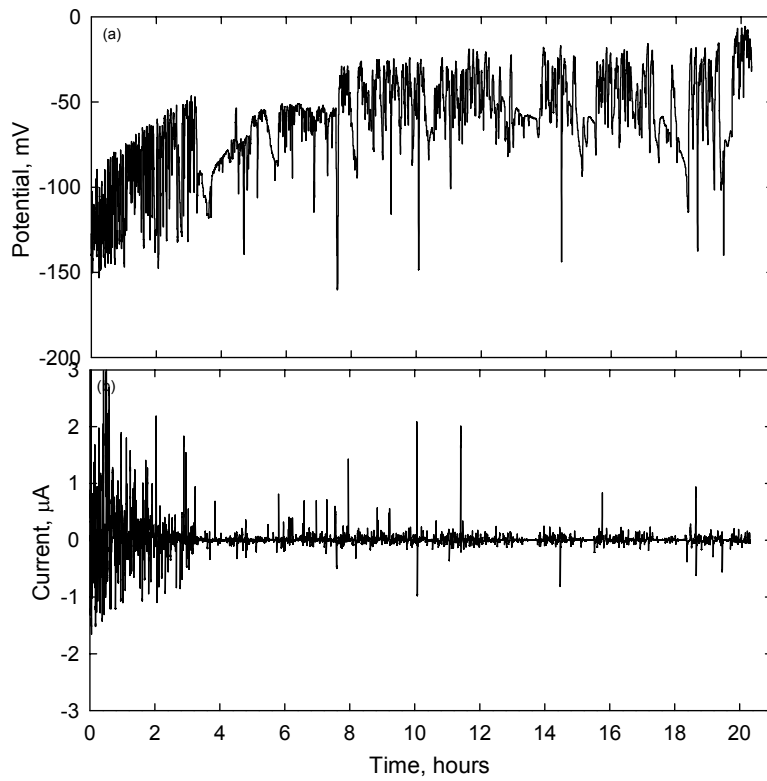


Figure 6. Potential and current time records for Alloy 304 in 1% NaCl solution.

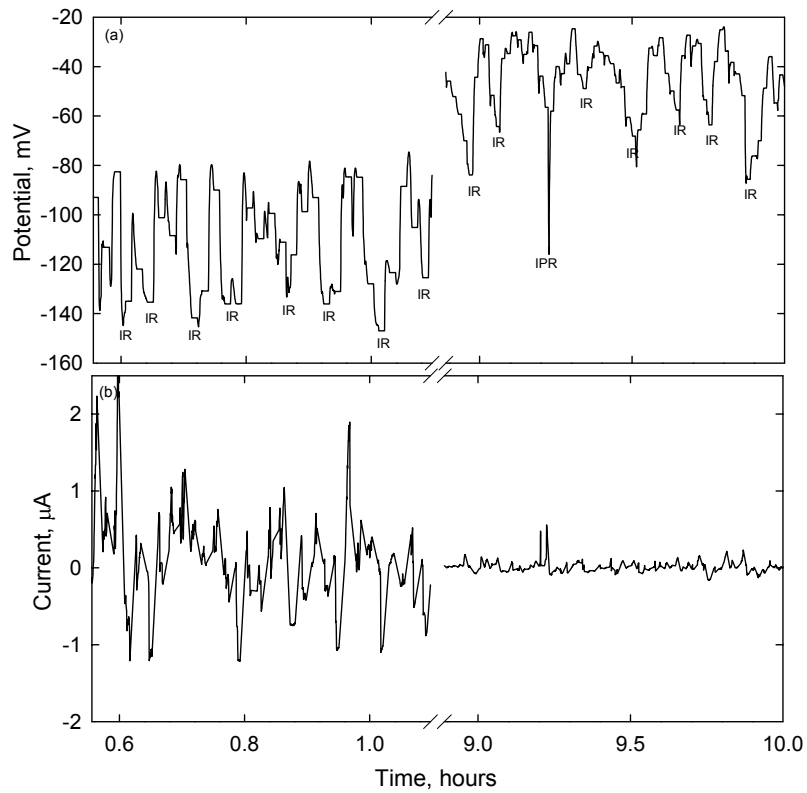
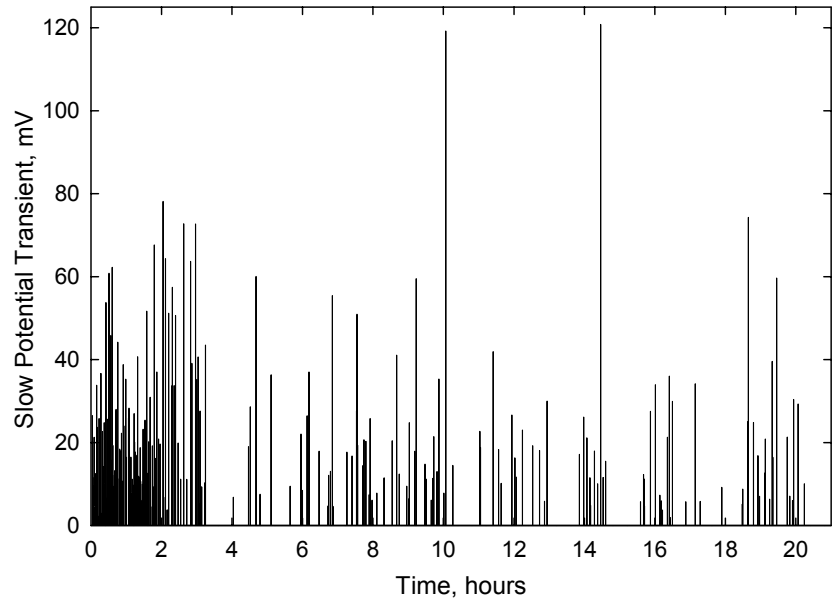
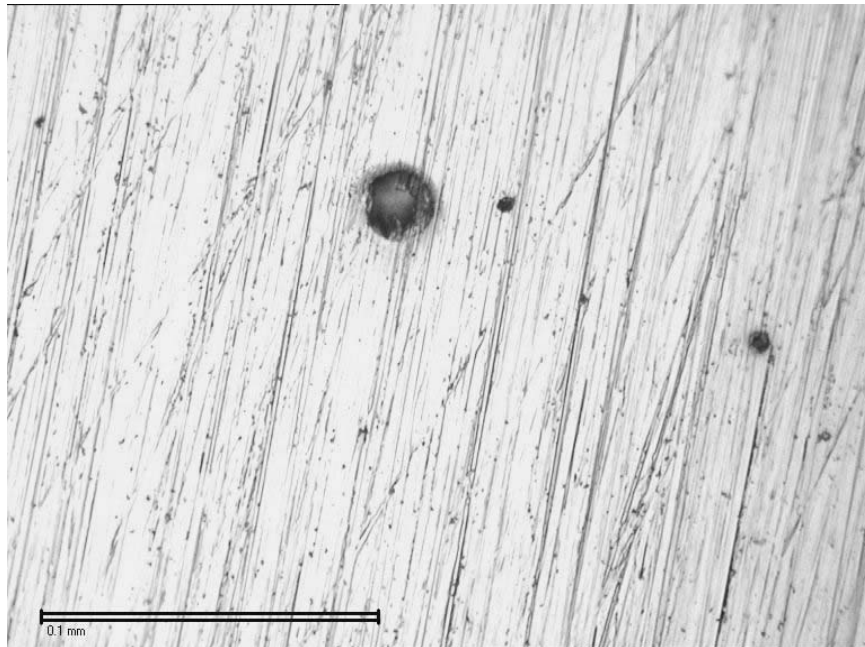


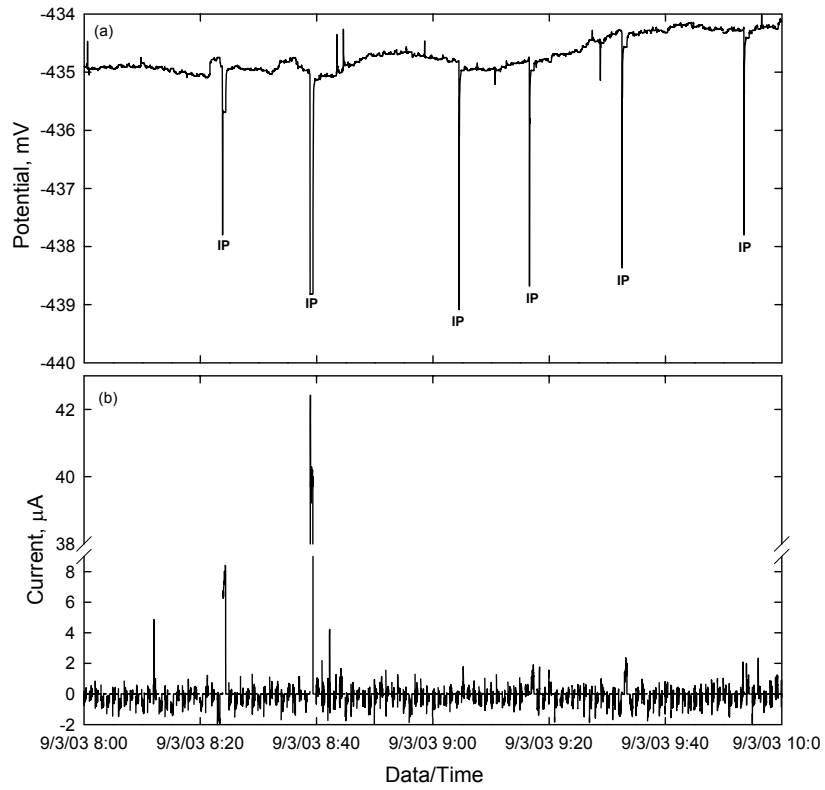
Figure 7. Typical IR transients recorded during the Alloy 304 immersion test.



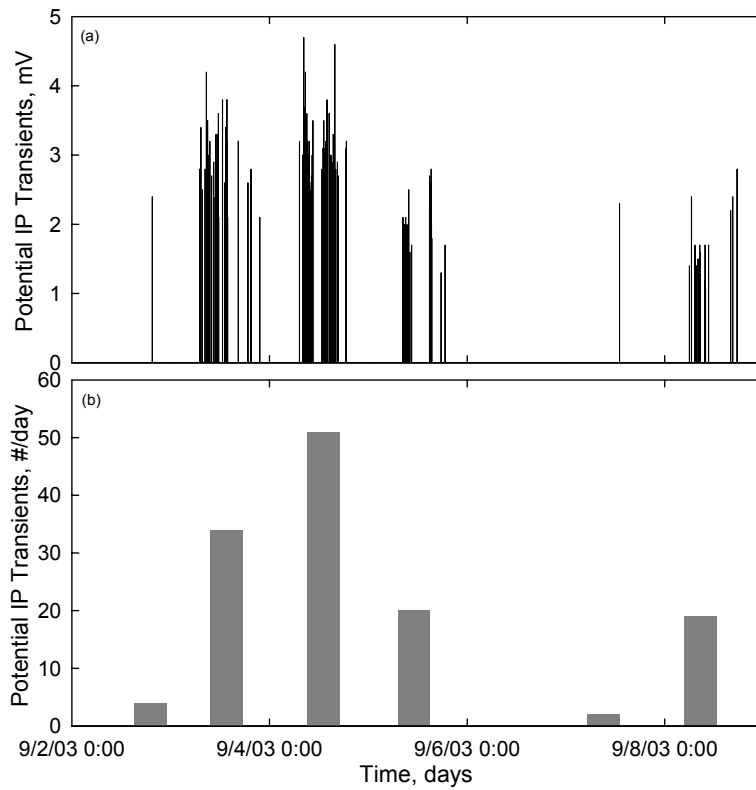
**Figure 8. Time profile of potential IR transients for Alloy 304 in 1% NaCl solution.**



**Figure 9. Micrograph of Alloy 304 test electrode, showing pitting damage.**



**Figure 10. Typical potential IP and corresponding current transients for Inhibitor A.**



**Figure 11. Time and frequency profiles of potential transients for Inhibitor A.**

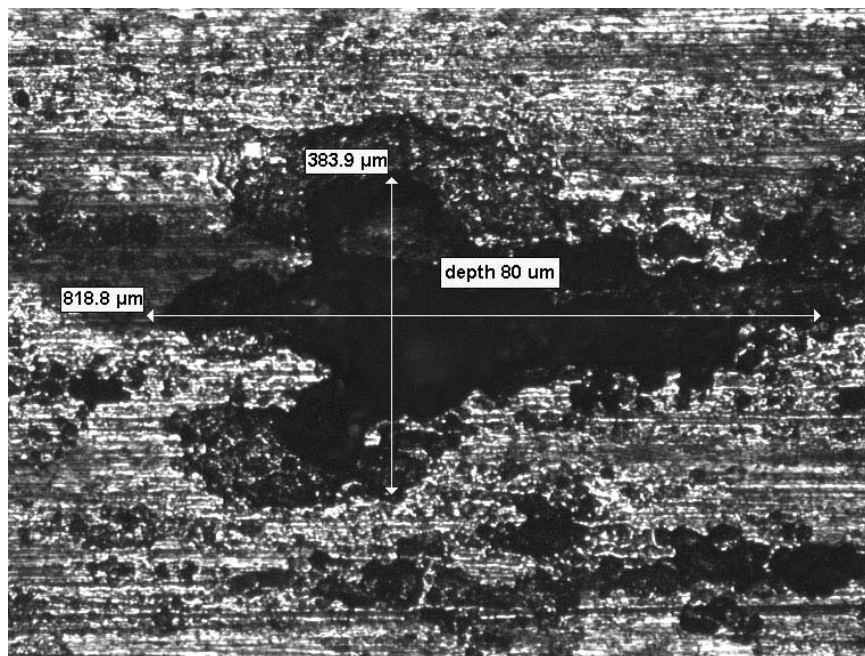


Figure 12. Micrograph of carbon steel electrode with inhibitor A showing large pit.

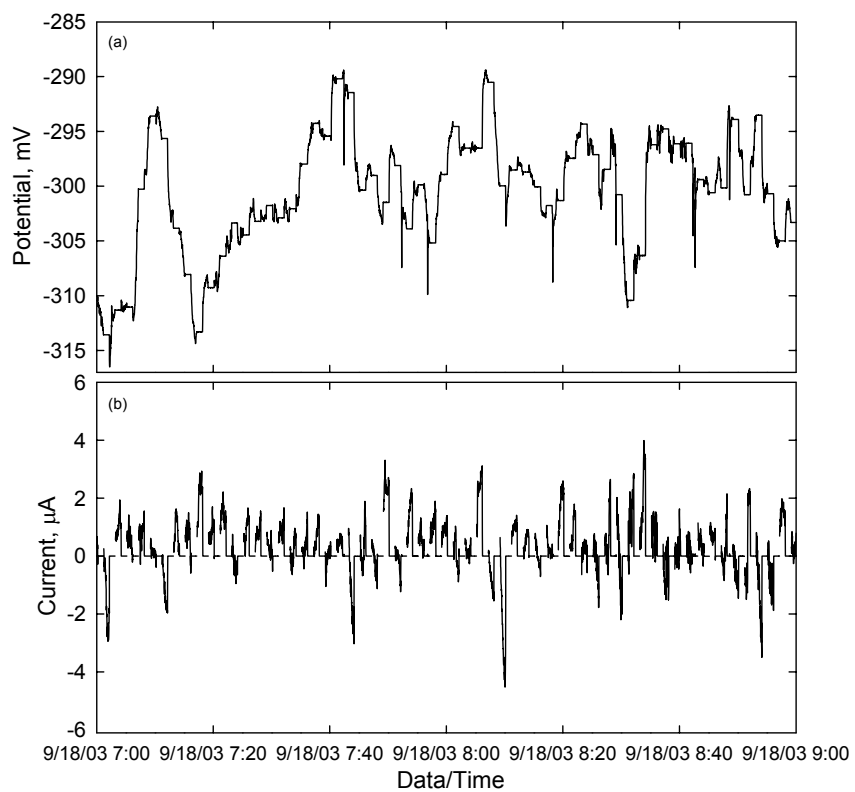
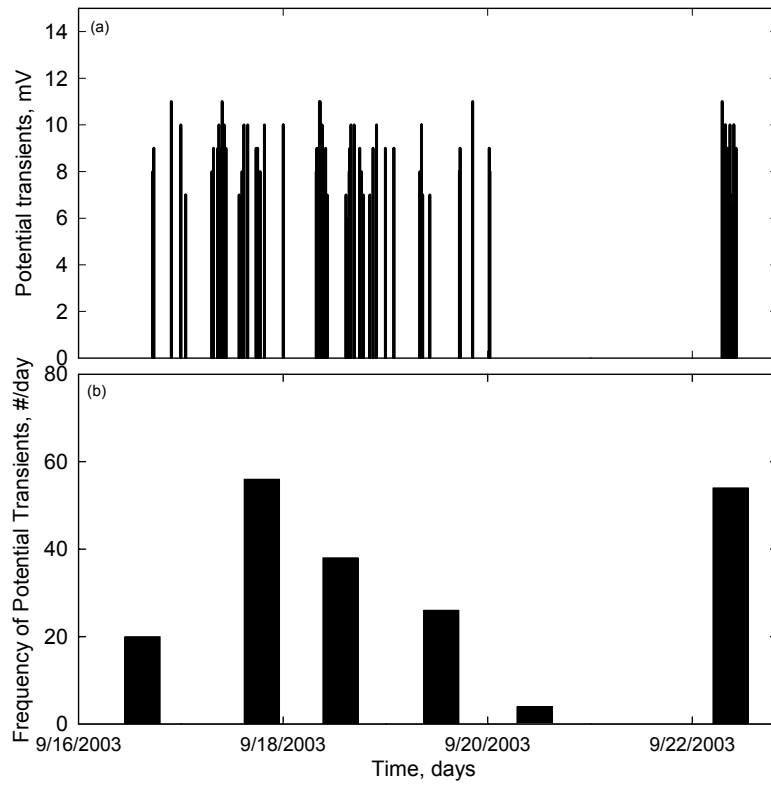
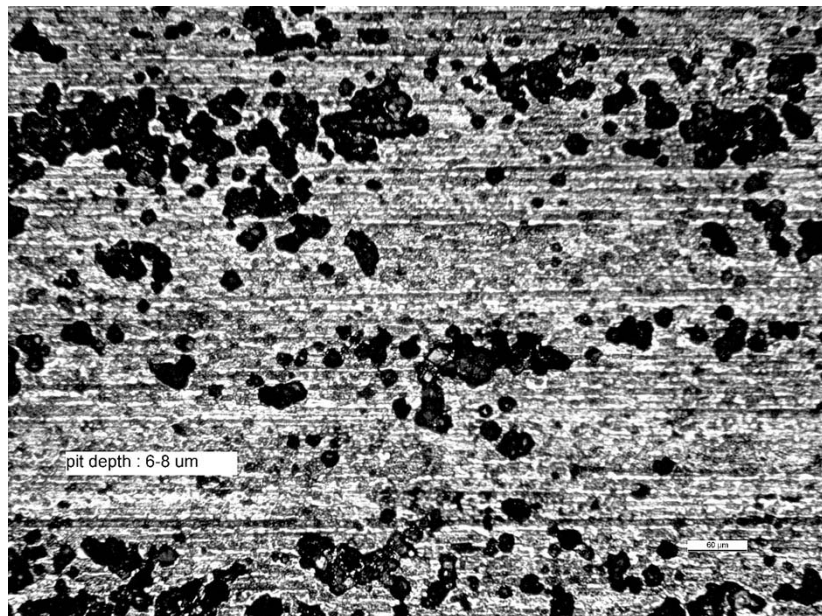


Figure 13. Typical potential/current transient for Inhibitor B.

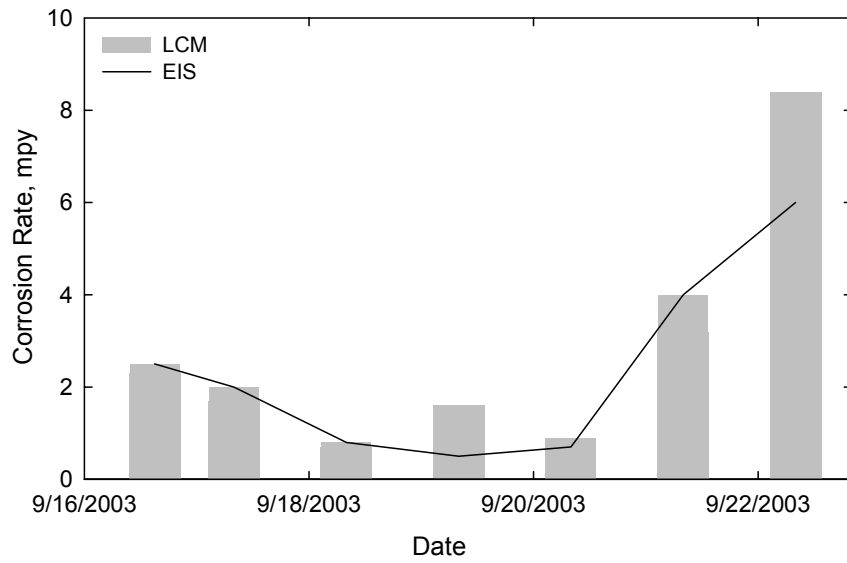


**Figure 14. Time and frequency profiles of potential transients for Inhibitor B.**



**Figure 15. Micrograph of carbon steel electrode with inhibitor B showing larger number of more shallow pits.**





**Figure 16. Corrosion rates based on LCM and EIS measurements.**