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THE APPLICATION OF ELECTROCHEMICAL NOISE BASED CORROSION MONITORING TO NUCLEAR WASTE TANK VAPOR SPACE ENVIRONMENTS AT THE HANFORD SITE

Prepared for the U.S. Department of Energy
Assistant Secretary for Environmental Management

Contractor for the U.S. Department of Energy
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ARES Corporation

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THE APPLICATION OF ELECTROCHEMICAL NOISE BASED CORROSION MONITORING TO NUCLEAR WASTE TANK VAPOR SPACE ENVIRONMENTS AT THE HANFORD SITE

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ABSTRACT

Vapor space corrosion data collected by electrochemical noise (EN) based corrosion probes installed in double shell tanks (DSTs) at the Department of Energy's Hanford Site in Richland, Washington have historically been characterized by surprisingly high levels in current. In late 2003, a program was established to assess the significance of archived Hanford DST vapor space EN data. This program showed that the high vapor space current levels are likely the result of crevice corrosion on the vapor space electrodes. The design of DST vapor space electrodes provides tight metal-to-metal and gasket-to-metal interfaces necessary for this type of localized corrosion to occur. In-tank activities (splashing, etc.), or more likely condensation of water vapor in the vapor space, provide the necessary moisture. Because crevice corrosion appears to be active on the vapor space EN electrodes, data collected from these electrodes are *not* likely to be applicable to the large flat metal surfaces that make up the bulk of the DST domes and upper walls. The data do, however, indicate that conditions in the DST vapor spaces are conducive to accelerated crevice corrosion at creviced areas in the tank vapor space (overlapping joints, riser interfaces, equipment penetrations, etc.) under high humidity conditions.

Keywords: electrochemical noise, corrosion monitoring, nuclear waste tank, vapor space corrosion

INTRODUCTION

Corrosion monitoring and control are currently provided at the Hanford Site through a waste chemistry sampling and analysis program. In this process, tank waste is sampled, analyzed and compared to a selection of laboratory exposures of coupons in simulated waste. Tank wall corrosion is inferred by matching measured tank chemistries to the results of the laboratory simulant testing. This method is expensive, time consuming, and does not yield real-time data.

In early FY 1996 the Hanford Site, with support from the Department of Energy Tanks Focus Area, launched an effort to develop the EN technique for application to corrosion monitoring in high-level nuclear waste tanks. EN was selected for application to waste tanks because of the technique's ability to detect and discriminate between localized forms of corrosion. Localized corrosion has been identified as the primary threat to DST integrity at Hanford.[1]

In its simplest form, the EN technique in aqueous environments involves the immersion of electrodes made of a material of interest in an environment of interest. A typical channel on an EN monitoring system requires three nominally identical electrodes: one working electrode, one counter electrode, and one pseudo-reference electrode. If the measurement environment is conductive enough, fluctuations in current and voltage caused by the corrosion of these electrodes can be passively measured by the EN equipment.

EN monitoring in gasseous or vapor space environments is more complicated than EN monitoring in aqueous environments. If the solution resistance between the electrodes installed in a gasseous environment is infinite, as in a perfectly dry environment, it is impossible to measure any current fluctuations since this would imply an infinitely large change in the potential (i.e.: voltage) difference between the working electrodes. Similarly, there would be no potential fluctuations to measure, since no current can pass between the working and pseudo-reference electrodes. In a more realistic case where some electrical conduction through surface films is possible, measurement of EN is also theoretically possible. In this case, the key question becomes whether the resistance between the electrodes is low enough to allow something sensible to be measured. If it is, then EN can be measured. However, *what* is measured will depend on the details of the probe configuration and the instrumentation. More specifically, it must be recognized that all measurements must account for the electrical conduction through films over the probe housing, rather than directly between the electrodes, and this demands careful analysis on a system-by-system basis.[2]

Unlike electrical resistance (ER), in linear polarization resistance (LPR), and other more traditional uniform corrosion monitoring techniques, no external voltage or current is artificially applied to the electrodes during EN measurements. Data analysis techniques vary widely and are beyond the scope of discussion in this work. However, they are all dependent on the fact that different forms of corrosion create different fluctuation patterns in current and voltage. Thus, analysis of EN data can reveal information about the type of corrosion occurring, and specifically for Hanford, if pitting or stress corrosion cracking has initiated. Traditional ER and LPR corrosion monitoring techniques do not offer this capability.

EN proof-of-principle tests in aqueous simulated waste solutions were conducted at Oak Ridge National Lab (ORNL) and Pacific Northwest National Lab in 1995 and 1996.[3] No gaseous environments were studied at this time. Based on this study, a 3-channel prototype system was constructed and deployed in DST 241-AZ-101 in August 1996.[4] Following the successful demonstration of the prototype for approximately a year, a longer more advanced 8-channel system was designed and installed in DST 241-AN-107 in September 1997.[5] Figure 1 shows the installation of this system. Unlike the prototype, the in-tank probe on this system reached from tank top to tank bottom and immersed two channels of EN electrodes in the sludge at tank bottom, four channels in the tank supernate, and two channels in the tank vapor space. Figure 2 shows the two types of electrode channels on the probe. Bullet-shaped electrodes expose a surface area of approximately 25 cm^2. C-ring electrodes expose a surface area of approximately 47 cm^2. Both types of electrodes are made from archived American Society for Testing and Materials (ASTM) A537 CL 1 (Unified Numbering System (UNS) K02400) mild steel leftover from the construction of the 241-AP tank farm. This probe was

removed and replaced in early 2001. Over the last several years, four additional systems of similar design have been installed into other DSTs in the 241-AN tank farm.[6-9] Three of these systems are currently in operation in 241-AN-104, 241-AN-105 and 241-AN-107. These systems are remarkably robust and have returned a great quantity of corrosion information from the supernate and sludge layers in the monitored waste tanks.

The same cannot be said, however, for the vapor space channels of these probes. Shortly after installation, EN electrodes suspended in DST vapor spaces began producing unexpected data, most often with surprisingly high current readings. High EN currents are associated with accelerated corrosion - not an expected problem in the vapor spaces of DST. EN current levels reached levels high enough to corrupt data on adjacent channels on the probes by early 2002. On March 5, 2002 the vapor space channels were disconnected from system instrumentation to preserve supernate and sludge channel data. After data collection on vapor space electrodes was stopped, previously collected high-current data were archived for future analysis.

Although the EN technique has been successfully applied by others to corrosion monitoring in industrial vapor space environments,[10-27] the technique has never been applied to nuclear waste tank vapor space environments. In addition to reviewing historical work by others and the analysis of vapor space electrodes removed from the original 241-AN-107 probe, a simple DST vapor space simulant test program was established in late 2003. The results from this work and their applicability to current and future corrosion monitoring at Hanford are presented herein.

TEST CONDITIONS AND EQUIPMENT

On September 2, 2003 test equipment was unpacked and set up at ORNL. A 40-liter High Density Polyethylene (HDPE) drum was partially immersed in a large stainless steel constant temperature bath held at 23°C. Approximately six liters of waste simulant prepared from reagent grade chemicals and demineralized water was added to the bottom of the drum. The composition of the waste simulant is shown in Table 1. A plant compressed air line was routed through a pressure regulator, oil removal filter and flow meter and into the drum to provide ventilation at a rate of approximately 14 cm^3/min. This flow rate produced an air exchange rate in the drum vapor space above the simulated waste that is approximately the same as the turnover rate in a 241-AN tank farm DST. The temperature of the forced air in the test cell and the temperature of the bath were not varied during the test. At the conclusion of test cell setup, two channels of 25 cm^2 UNS K02400 bullet-shaped electrodes identical to those used on the Hanford corrosion probes were suspended in the top of the drum above the waste simulant.

All electrodes used in the laboratory work were fabricated from archived UNS K02400 steel leftover from the fabrication of the 241-AP tank farm at Hanford. This archived material is approximately same age as most of the Hanford DSTs and was suspended in DST 241-AP-101 during the stress-relief heat treatment to which the tank was subjected following fabrication.

All test electrodes were suspended on custom built stainless steel electrode holders similar to the electrode holders on the Hanford corrosion probes. The electrodes were electrically isolated from the holders with low-conductivity, peroxide-cured Ethylene Propylene Diene Monomer (EPDM) O-ring type gaskets and glass-lined feed-throughs identical to those used on the Hanford corrosion probes. A stainless steel bar was welded across each channel of electrode holders to provide continuity between electrode holders similar to that provided by the corrosion probe body in the field. Electrodes were spaced ~5 cm apart on centers after placement in the HDPE drums. The working (or common) electrode

on each channel was common to the measurement of corrosion current and corrosion potential. Fluctuations in corrosion current were measured between working and counter electrodes on each channel. Likewise, fluctuations in corrosion potential (ie: voltage difference) were measured between working/counter electrode assemblies and pseudo-reference electrodes of each channel. As in the field, the lab tests did not use standard reference electrodes. The electrode arrangement prior to insertion in test cell is shown in Figure 3. A detailed view of one channel of electrodes is shown in Figure 4. The assembled test cell is shown in Figure 5.

As in the field installations at Hanford, laboratory EN data were collected with a four-channel CIS 400 instrument from CAPCIS Ltd.[1] EN data were collected at a rate of one simultaneous measurement of current and potential every two seconds on each channel. The sign convention on the equipment caused voltage values to move in the negative direction when the counter/working electrode assembly became more anodic (corroding) than the pseudo-reference electrode (i.e.: where V_{ref} equals the potential of the pseudo-reference electrode and V_{work} equals the potential of the working/counter electrode assembly, the equipment records the measurement of $V_{ref} - V_{work}$). Data were stored, processed, and analyzed with Amulet software manufactured by Corrosion Condition and Control Ltd.[2] Approximately three meters of Belden 8162 cable were used for each channel to collect the analog corrosion signals from the test cell electrodes and transmit them to the corrosion monitoring instrumentation.[3] As in the field, all conductors used in the laboratory passed through MTL 755-AC intrinsic safety barriers.[4]

Midway through the testing program a new CIS 5K instrument and two new probe channels were added to the simulated waste tank test apparatus.[1] Each channel was formed from three small EN electrodes isolated in ceramic and mounted in a stainless steel probe head. Electrodes on the new probes were fabricated from archived UNS K02400 material leftover from the construction of the 241-AP tank farm at Hanford. Each electrode exposed a surface area of approximately 0.7 cm^2. Figure 6 shows the face of one of the new probe heads. The addition of the new equipment was made without changing the configuration or operation of the existing test equipment. Other test and measurement parameters on the new instrument were set to match the original instrument settings. Data collection was also continued on the original test equipment.

RESULTS AND DISCUSSION

The majority of data from the laboratory tests show little if any corrosion activity on the vapor space electrodes monitored by either instrument. Figure 7 shows a week of data from the 25 cm^2 bullet-shaped vapor space electrodes shortly after the start of testing. Figure 8 shows another week of data from the 25 cm^2 bullet-shaped vapor space electrodes near the conclusion of testing. Data collected during the balance of testing are very similar to that in Figures 7 and 8. Figures 9 and 10 show the 25 cm^2 bullet-shaped vapor space electrodes at the conclusion of testing. As the data predict, no significant corrosion damage is visible on the exposed electrode surfaces (Figure 9) or at the electrode/gasket interfaces (Figure 10). All of these results obviously point to little or no corrosion activity on the electrodes.

[1] CIS 400 and CIS 5K are registered trade names of Capcis Systems, a UMITEK Company, Sycamore Court, North Leigh Business Park, Woodstock Road, Witney, Oxford, OX29 6SW
[2] Amulet is a registered trade name of Corrosion & Condition Control Ltd., Dingwall, Ross-Shire, England
[3] 8162 is a registered trade name of Belden Inc., St. Louis, Missouri
[4] MTL 755-AC is a registered trade name of Measurement Technology, Ltd., Luton, Bedfordshire, England

As with the 25 cm^2 bullet-shaped vapor space electrodes, the majority of laboratory data from the 0.7 cm^2 vapor space electrodes show little if any corrosion activity. Figure 11 shows a week of vapor space data from the 0.7 cm^2 vapor space electrodes shortly after the start of testing. Figure 12 shows another week of data near the end of testing. Data collected during the balance of testing on these electrodes were very similar to that in Figures 11 and 12. Electrodes were inspected for corrosion damage at the conclusion of testing to verify the EN data. Figure 13 shows the vapor space electrodes at the conclusion of testing. As the data predict, no significant corrosion damage is visible on the exposed electrode surfaces.

Laboratory data from both instruments are strikingly similar to field data produced by the vapor space electrodes on the corrosion probes in the 241-AN tank farm shortly after installation in the DSTs. Figures 14 and 15 respectively show data collected from 25 cm^2 bullet-shaped electrodes and 47 cm^2 C-ring shaped electrodes shortly after probe installation in the vapor spaces of DSTs 241-AN-104 and 241-AN-107. Data shown in Figures 14 and 15 are typical of other probe vapor space data collected shortly after installation. Both laboratory and field data indicate little or no corrosion activity in the vapor space immediately following installation.

The good correlation between laboratory and field data all but dissappears however at longer exposure times. Surprisingly, the level of current EN (ie: corrosion activity) on most of the field vapor space electrodes rises, most often sharply, within a few months of installation. Figures 16 and 17 show the sharp changes in vapor space electrode behavior in DSTs 241-AN-105 and 241-AN-107. Other tank vapor space electrodes displayed similar behavior. None of the laboratory vapor space electrodes displayed the sharp increase in current.

The increase in current on vapor space electrodes in the field has never been visibly correlated with corrosion damage on vapor space electrodes removed from service. No currently monitored vapor space electrodes (showing high current levels) have ever been removed from service and analyzed for corrosion damage. No correlation was found between the sharp changes in current seen in the field vapor space electrodes and weather related changes or tank farm activities. No sharp changes in the behavior of other probe supernate electrodes immersed in the tank coincided with the sharp changes in vapor space current.

In late 2001, vapor space electrodes were analyzed upon removal of the orignal 241-AN-107 probe. Pictures of a C-ring and pin-type electrode removed from this probe are shown in Figures 18 and 19. Electrodes removed from this probe show little uniform corrosion, but significant corrosion at metal/metal mating surfaces on C-ring electrodes and near the electrode gasket seating surface on other vapor space electrodes.[28] No significant corrosion product buildup was observed on electrodes immersed in the supernate or sludge in the tank. Heavy corrosion product buildup around joints and other shared surfaces is a classic sign of crevice corrosion. Corrosion damage shown in Figures 18 and 19 could not be correlated to EN data from this probe because long analog data cables used on this system substantially corrupted all data collected from this probe (newer corrosion monitoring systems in the 241-AN farm do not use long analog data cables as a result of the data collection problems they created on the original 241-AN-107 system).[29]

Crevice corrosion demands a tight space (such as a gasket/metal or metal/metal interface) and moisture to initiate and proceed. In-tank equipment operation, waste transfers, gas release events, or

waste disturbances could have caused splashing and provided the moisture necessary to induce crevice corrosion in the vapor space electrodes on the original 241-AN-107 probe (and any of the newer

probes). However, condensation is a far more likely source for this moisture since the probe body is likely to be slightly cooler than the tank vapor space in which it is suspended. Once active, the accelerated corrosion at the base of a crevice is capable of producing the high current levels seen on the current probes.

No crevice corrosion (or any other form of corrosion) was observed on vapor space electrodes in the controlled laboratory environment during the recent testing at ORNL. Likewise, no laboratory vapor space electrodes produced high current levels like those seen in the 241-AN DST probes. Although the gasket seating surfaces differ in design between the original 241-AN-107 probe and the more recently installed probes, it seems likely that vapor space electrodes in tank farm service are suffering from condensation induced crevice corrosion at tight metal-to-metal or gasket-to-metal interfaces.

SUMMARY AND CONCLUSIONS

Field data collected from EN electrodes suspended in 241-AN farm DSTs have historically been characterized by abnormally high levels of current. High EN currents are typically associated with high rates of corrosion. Unmonitored vapor space electrodes removed from tank farm service have shown relatively severe corrosion damage at metal/metal and gasket/metal interfaces. Crevice corrosion is the most likely cause for both the high current levels on monitored electrodes and the corrosion damage on unmonitored electrodes removed from service. Condensation of vapors in the vapor space is the most likely source for the moisture necessary to induce this type of corrosion.

Hanford DST domes and upper wall surfaces are primarily formed from welded steel plates. The bulk of the steel plate surfaces are inherently smooth and crevice-free. Because crevice corrosion appears to be active in crevices on the vapor space EN electrodes, data collected from these electrodes are *not* likely to be applicable to the large flat metal surfaces that make up the bulk of the DST domes and upper walls (by definition crevice corrosion cannot exist on large bare flat metal surfaces). The data do however indicate that conditions in the DST vapor spaces are conducive to accelerated crevice corrosion at creviced areas in the tank vapor space (overlapping joints, riser interfaces, equipment penetrations, etc.) under high humidity conditions. For verification, one of the new EN probes that has produced high vapor space elctrode currents should be removed or temporarily lifted out of the tank to verify the existence of crevice corrosion on the vapor space electrodes.

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TABLE 1
WASTE SIMULANT COMPOSITION

Anion	Concentration (M)
Nitrite	2
Nitrate	4
Chloride	0.02
Hydroxide	3

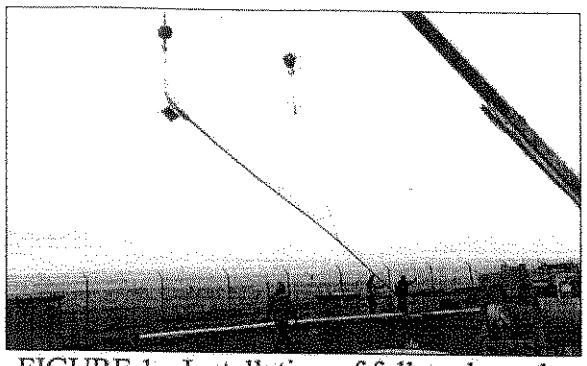

FIGURE 1 - Installation of full-scale probe

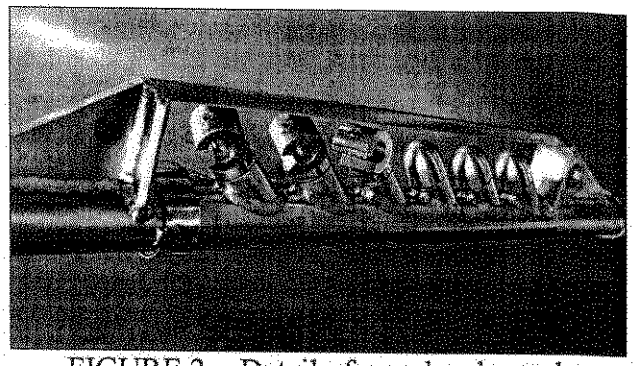

FIGURE 2 - Detail of a probe channels

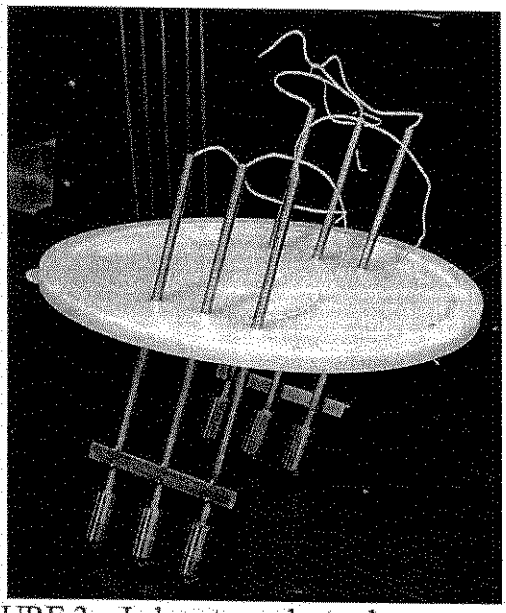

FIGURE 3 – Laboratory electrode arrangement

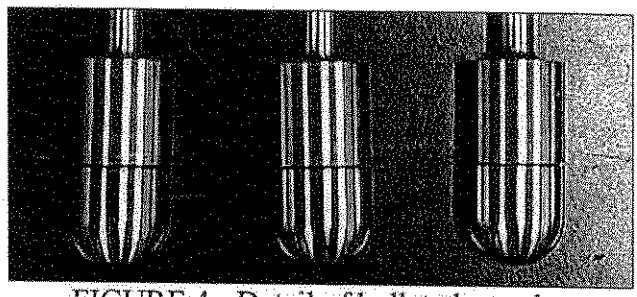

FIGURE 4 - Detail of bullet electrodes

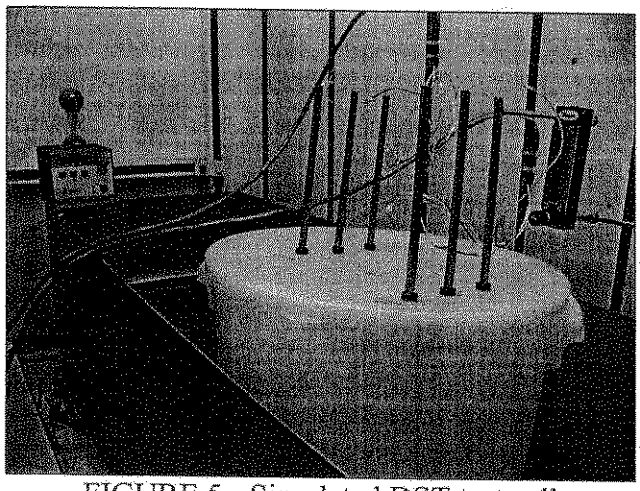

FIGURE 5 - Simulated DST test cell

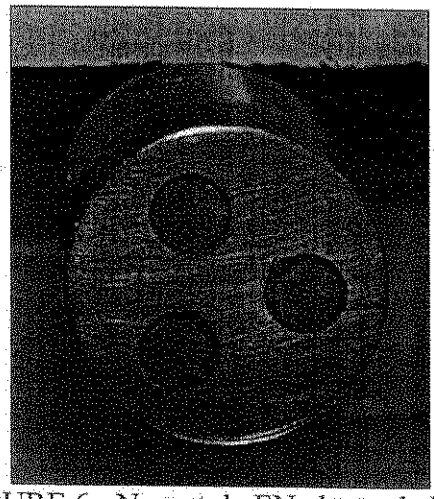

FIGURE 6 - New style EN electrode head

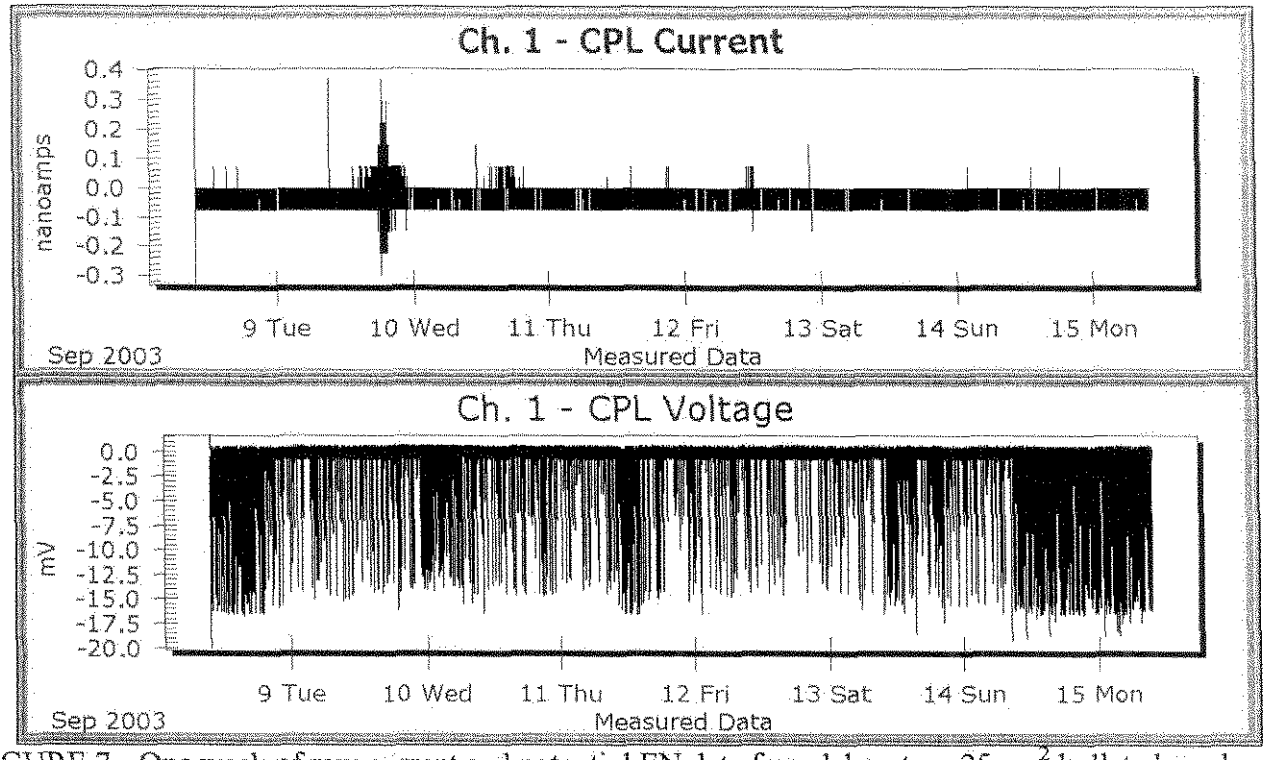

FIGURE 7 - One week of raw current and potential EN data from laboratory 25 cm^2 bullet-shaped vapor space electrodes showing very low corrosion activity at start of testing

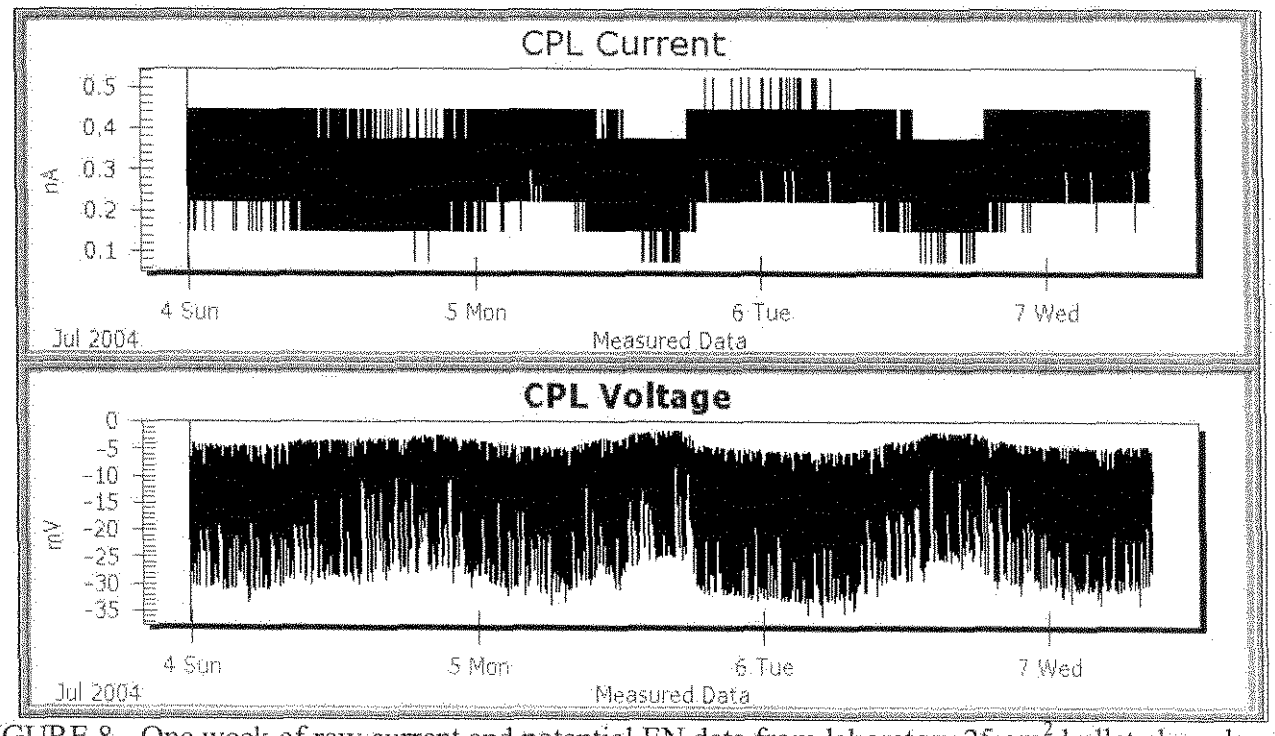

FIGURE 8 - One week of raw current and potential EN data from laboratory 25 cm^2 bullet-shaped vapor space electrodes showing very low corrosion activity near the end of testing

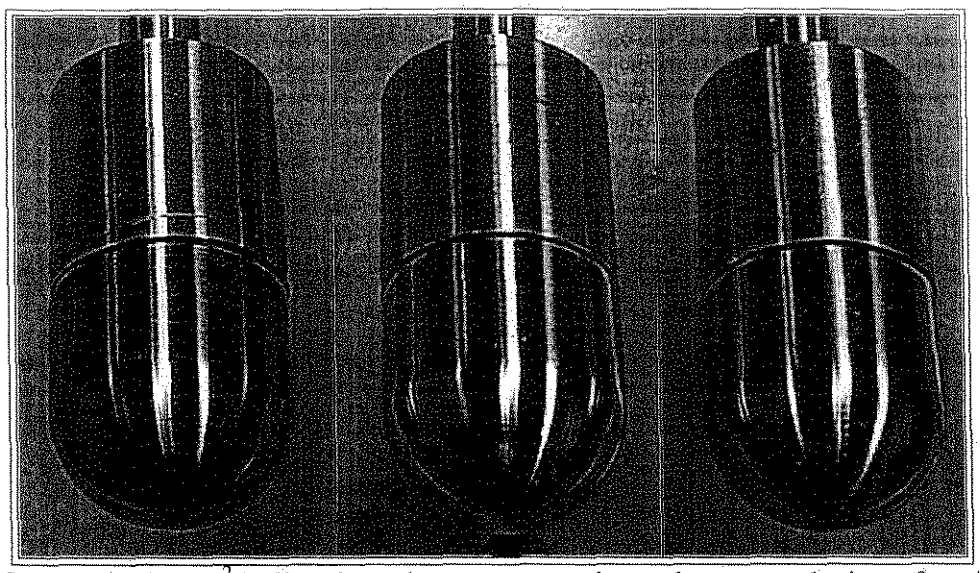

FIGURE 9 – 25 cm^2 bullet-shaped vapor space electrodes at conclusion of testing

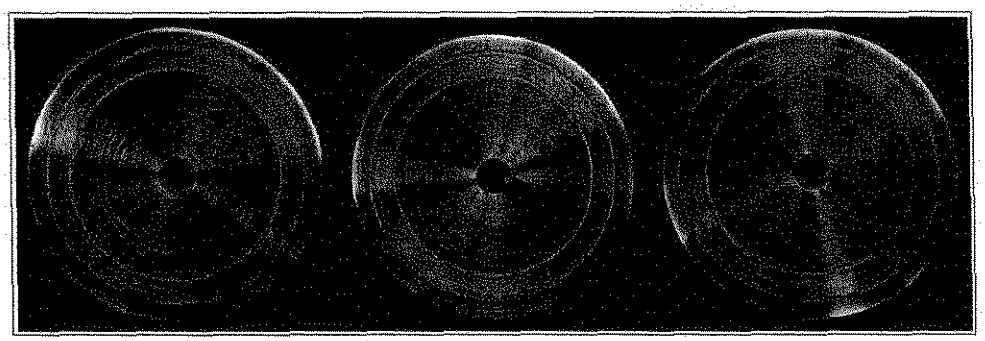

FIGURE 10 - Gasket/electrode interfaces on bullet-shaped electrodes at conclusion of testing

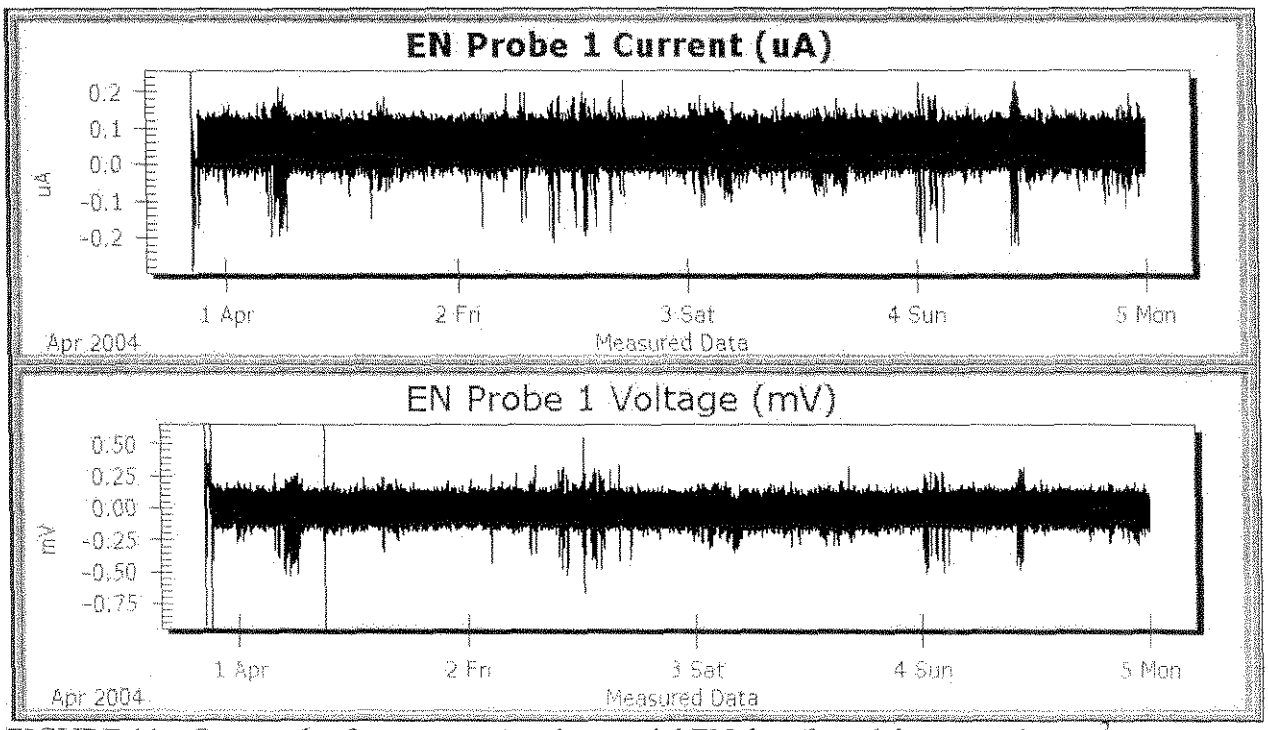

FIGURE 11 - One week of raw current and potential EN data from laboratory 0.7 cm^2 vapor space electrodes showing very low corrosion activity at start of testing

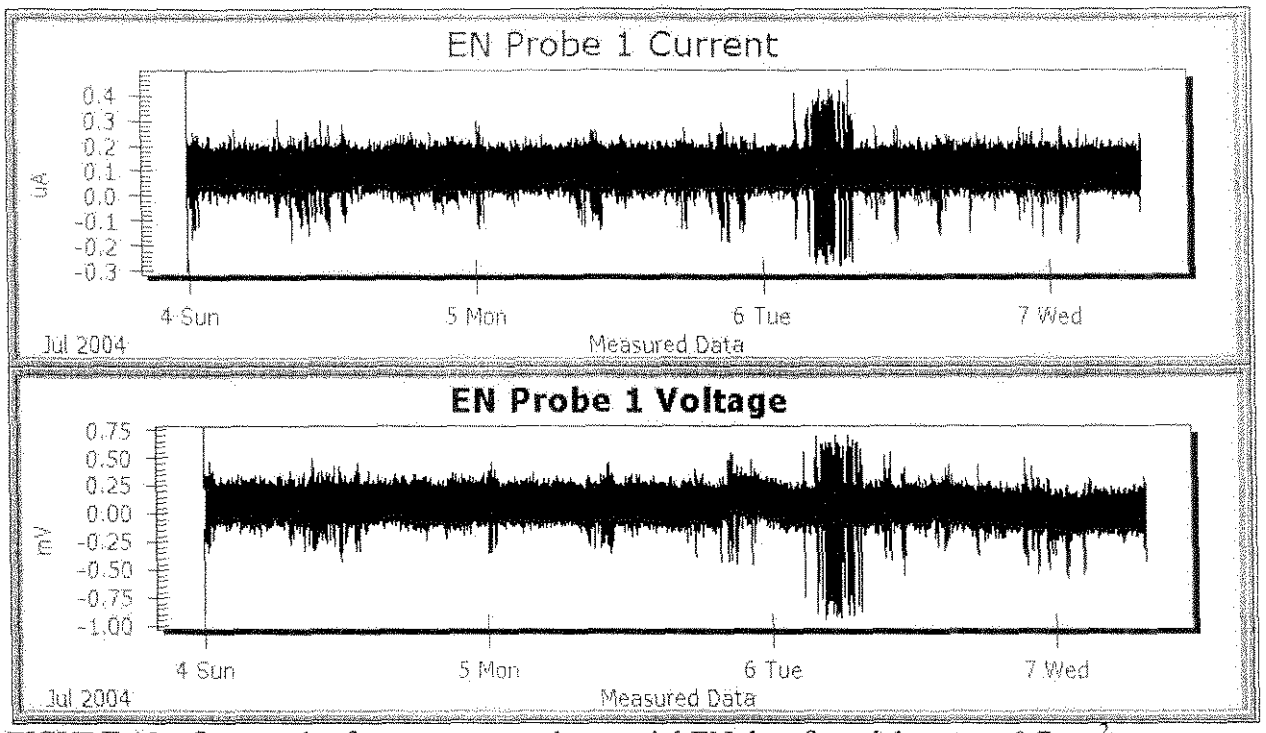

FIGURE 12 - One week of raw current and potential EN data from laboratory 0.7 cm^2 vapor space electrodes showing very low corrosion activity near end of testing

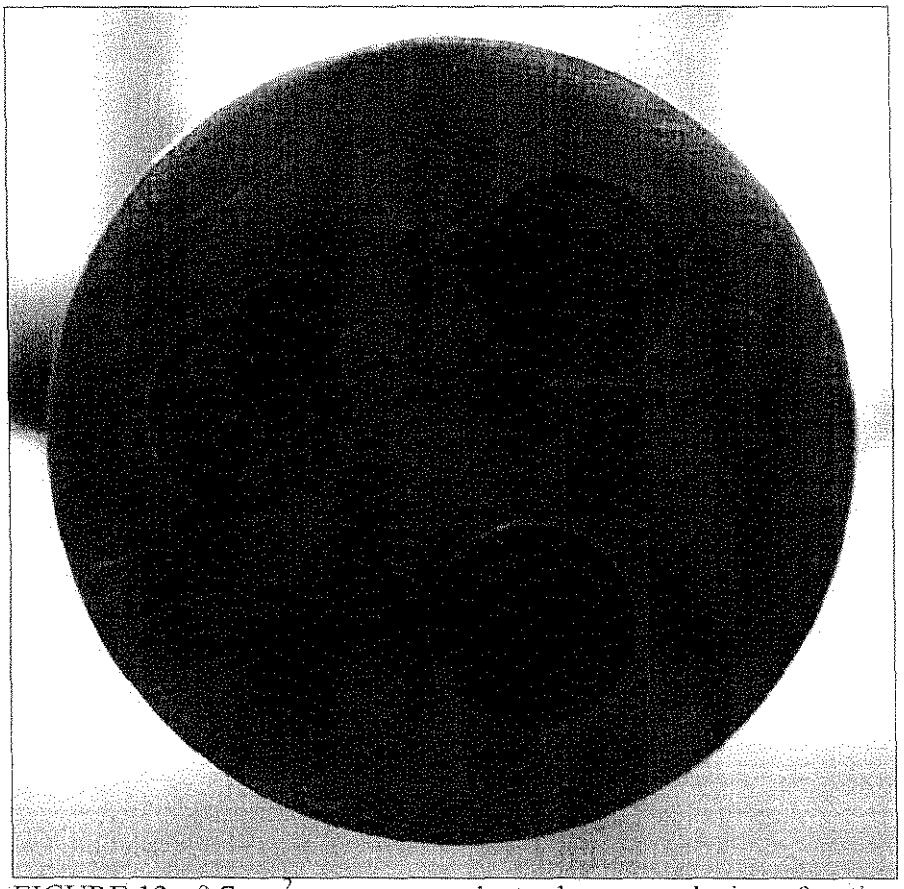

FIGURE 13 - 0.7 cm^2 vapor space electrodes at conclusion of testing

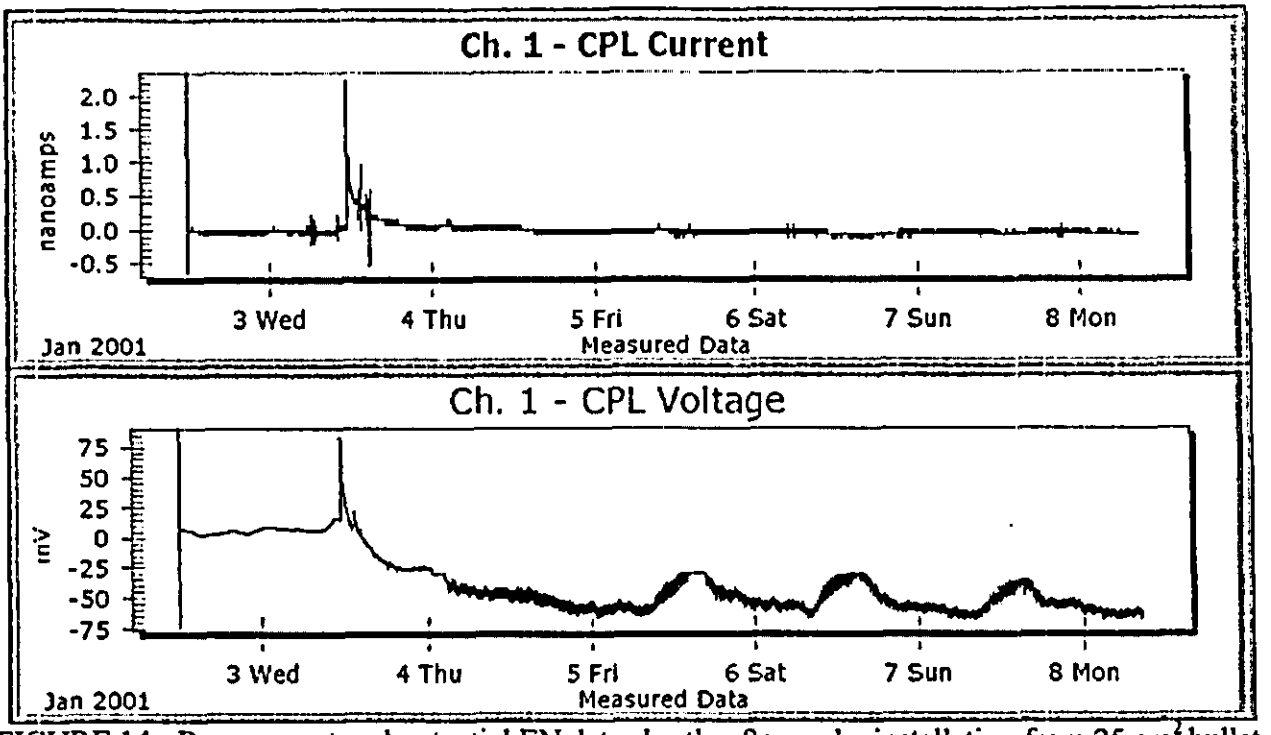

FIGURE 14 - Raw current and potential EN data shortly after probe installation from 25 cm^2 bullet-shaped vapor space electrodes on the 241-AN-104 corrosion probe showing low corrosion activity (sharp change in data at ~01/03/01 11:00 from cable connection during installation).

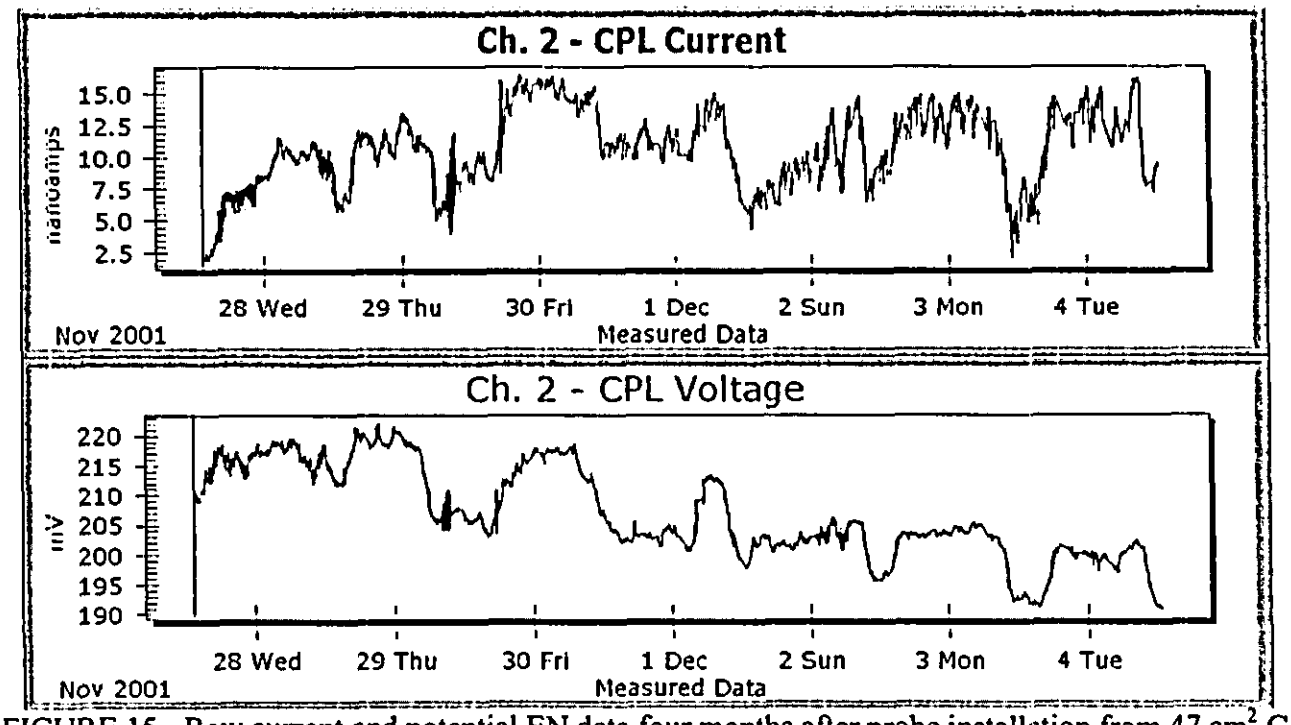

FIGURE 15 - Raw current and potential EN data four months after probe installation from 47 cm^2 C-ring shaped vapor space electrodes on the most recent 241-AN-107 probe showing low corrosion activity.

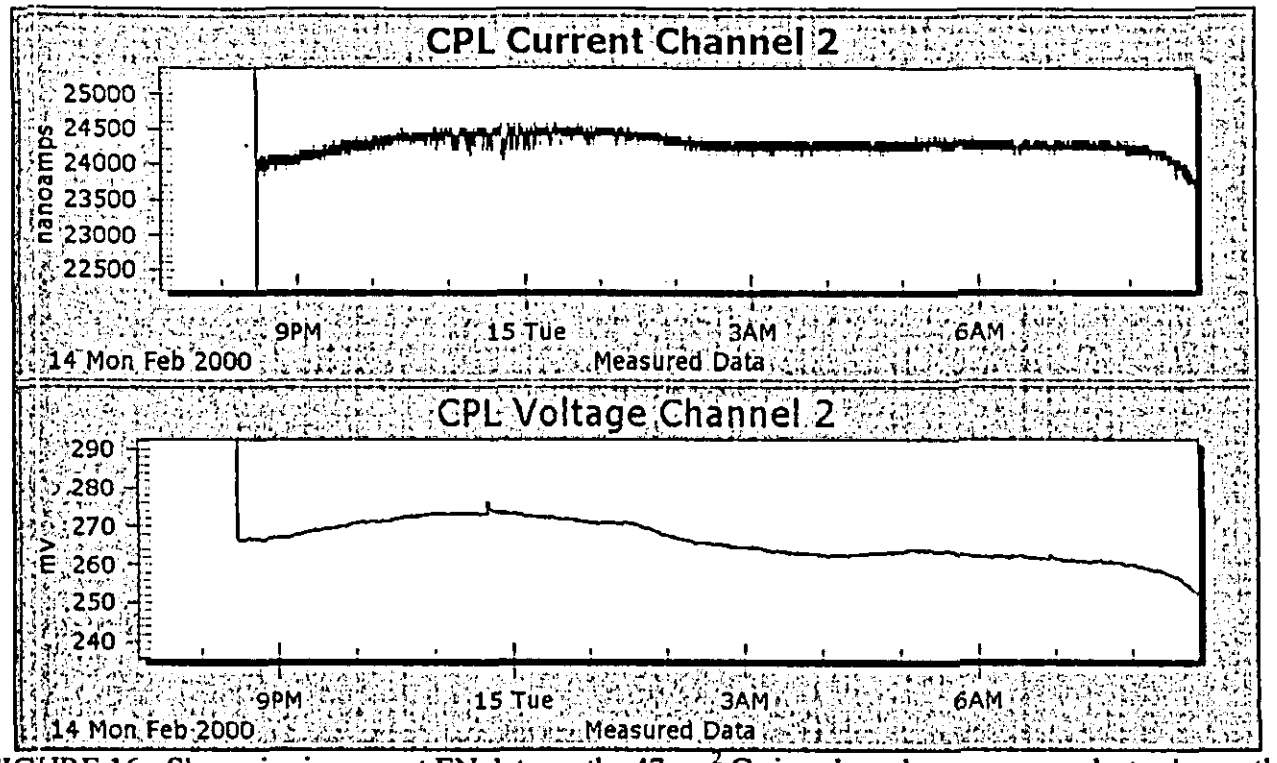

FIGURE 16 - Sharp rise in current EN data on the 47 cm^2 C-ring shaped vapor space electrodes on the 241-AN-105 probe approximately 1 month after installation

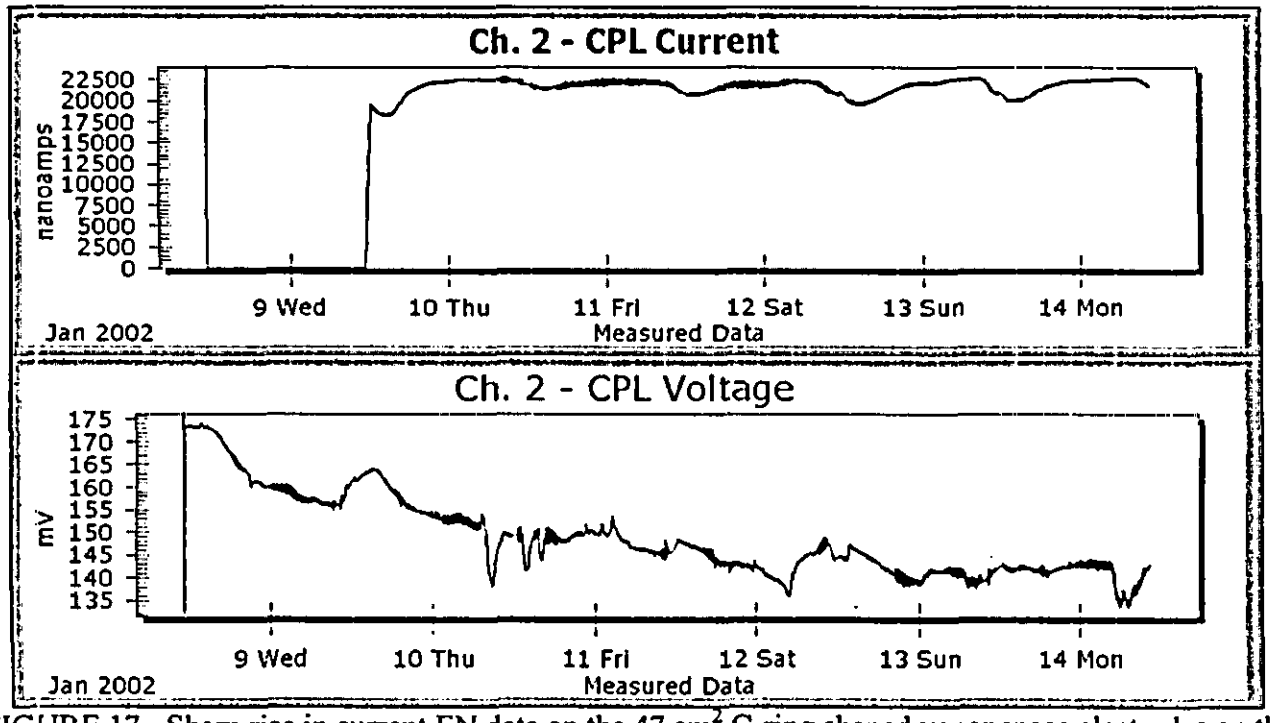

FIGURE 17 - Sharp rise in current EN data on the 47 cm^2 C-ring shaped vapor space electrodes on the most recent 241-AN-107 probe approximately 5 months after installation

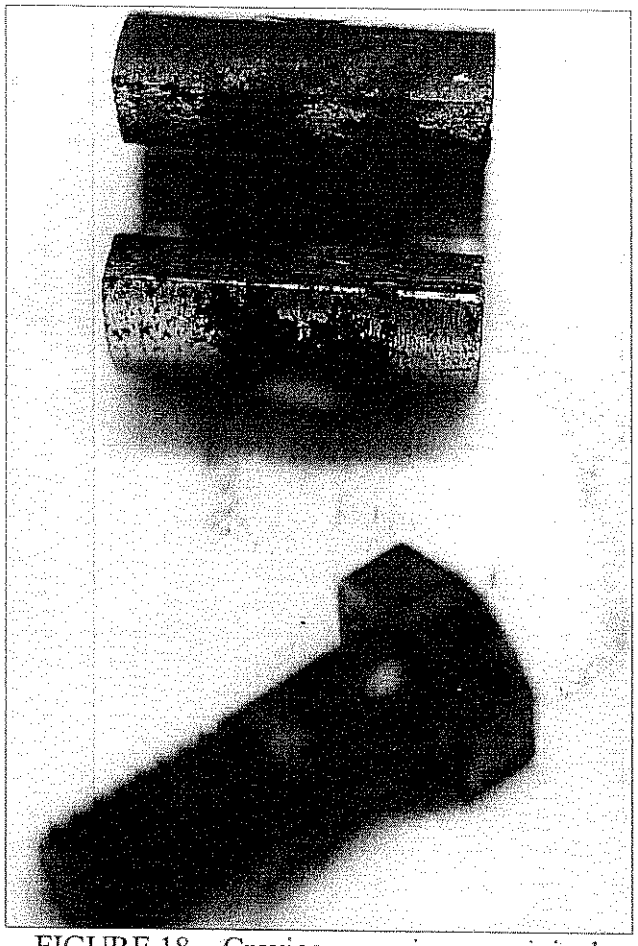

FIGURE 18 – Crevice corrosion on original 241-AN-107 probe C-ring electrode after exposure to DST vapor space

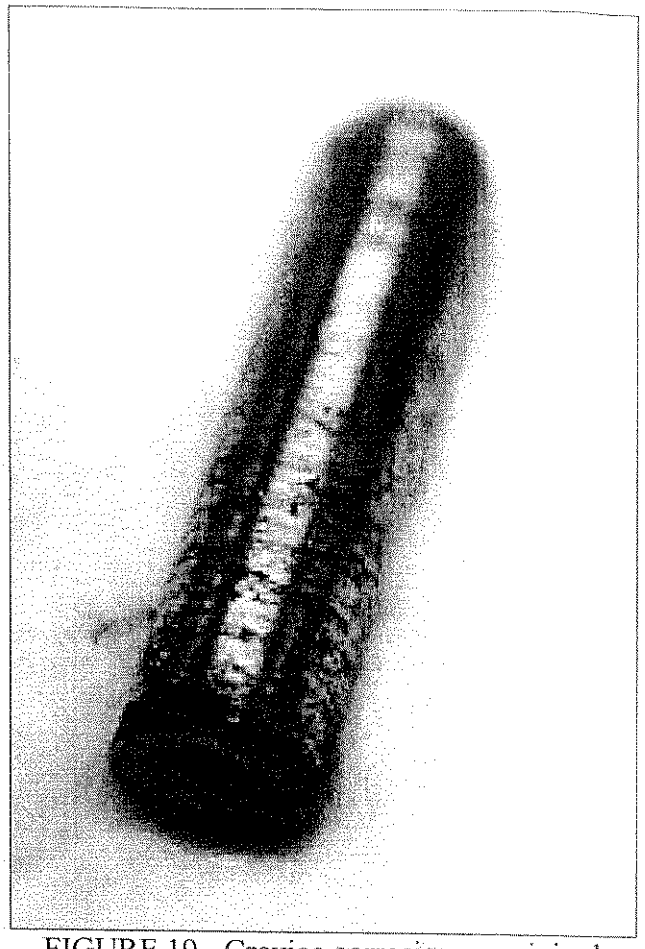

FIGURE 19 - Crevice corrosion on original 241-AN-107 probe pin-type electrode after exposure to DST vapor space