ANL/ES/CP-98177

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YuPo J. Lin, James R. Frank, and Edward J. St.Martin
Argonne National Laboratory
9700 S. Cass Avenue
Argonne, IL 60439

and

Daniel H. Pope
Bioindustrial Technologies, Inc.
40105 Industrial Park Circle
Georgetown, TX 78626

submitted to

Corrosion/99
Annual Conference and Exposition

April 25-30, 1999
San Antonio, Texas

Paper No. 99198

Sponsored by

NACE International
Houston, Texas

January 1999

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ABSTRACT

Because of the chaotic nature of the corrosion process and the complexity of the electrochemical noise signals that are generated, there is no generally accepted method of measuring and interpreting these signals that allows the consistent detection and identification of sustained localized pitting (SLP) as compared to general corrosion. We have reexamined electrochemical noise analysis (ENA) of localized corrosion using different hardware, signal collection, and signal processing designs than those used in conventional ENA techniques. The new data acquisition system was designed to identify and monitor the progress of SLP by analyzing the power spectral density (PSD) of the trend of the corrosion current noise level (CNL) and potential noise level (PNL). Each CNL and PNL data point was calculated from the root-mean-square value of the ac components of current and potential fluctuation signals, which were measured simultaneously during a short time period. The PSD analysis results consistently demonstrated that the trends of PNL and CNL contain information that can be used to differentiate between SLP and general corrosion mechanisms. The degree of linear slope in the low-frequency portion of the PSD analysis was correlated with the SLP process. Laboratory metal coupons as well as commercial corrosion probes were tested to ensure the reproducibility and consistency of the results. The on-line monitoring capability of this new ENA method was evaluated in a bench-scale flow-loop system, which simulated microbially influenced corrosion (MIC) activity. The conditions in the test flow-loop system were controlled by the addition of microbes and different substrates to favor accelerated corrosion. The ENA results demonstrated that this in-situ corrosion monitoring system could effectively identify SLP corrosion associated with MIC, compared to a more uniform general corrosion mechanism. A reduction in SLP activity could be clearly detected by the ENA monitoring system when a corrosion inhibitor was added into one of the test loops during the corrosion testing.

Keywords: electrochemical noise, corrosion, microbially influenced corrosion (MIC), sensor, sustained localized pitting (SLP).

INTRODUCTION

Corrosion of pipelines and storage facilities is a significant problem for the oil and gas industry. The most destructive form of corrosion is sustained localized pitting (SLP), in which the metal or alloy is perforated rapidly. Although there are several methods to measure general corrosion rates, no reliable on-line methods are available to predict when and where SLP will occur. A preliminary evaluation indicated that electrochemical noise analysis (ENA) could be an effective method to analyze SLP[1].

ENA is a non-destructive, in-situ method of monitoring natural corrosion processes. Using ENA, researchers measure the endogenous electrochemical corrosion current and potential fluctuations simultaneously. Because of the chaotic nature of the natural corrosion processes, signal processing of the recorded current and potential noise is very critical in interpreting the data collected. In order to characterize the corrosion mechanism, different signal processing algorithms were used to interpret the ENA data. Several characteristic evaluations of the time-series noise signal, such as potential and current noise levels, noise resistance[2,3,4,5], and pitting index[6] were proposed to describe the corrosion mechanism. These data and methods alone did not allow researchers to effectively identify the different corrosion mechanisms[7]. However, when spectral analysis of the chaotic electrochemical noise was also applied, researchers found that it was a powerful signal processing technique that could be used to characterize the noise data in a frequency domain[8,9]. In this technique, the slope of the power spectral density (PSD) vs. frequency of the transformed signal is believed to relate to the corrosion mechanism[10,11,12,13]. Most of the results presented in the references demonstrated a correlation between the corrosion process and the slope of the PSD under controlled, constant potential or current, laboratory studies. However, very few literature citations discussed the application of this technique in a natural corrosion process where the potential or current is not controlled. Coupling the PSD analysis technique with ENA would be an important step in developing a practical tool for monitoring SLP for industrial applications.

The main challenge is to differentiate SLP from uniform corrosion. The uniform corrosion process discussed here includes general corrosion as well as the development of a very large number of uniformly distributed small corrosion pits. SLP, as its name implies, refers to the few pits or group of pits that dominate the corrosion process and force a very fast metal loss on a small portion of the surface area. As discussed in reference 13, uniform corrosion appears to be a stochastic process; localized pitting corrosion appears to be a deterministic process. In most natural corrosion systems, SLP occurs together with uniform corrosion. They can be seen in the potential noise PSD analysis as a plateau in the high-frequency portion of the spectrum for the stochastic process and as a slope in the low-frequency portion of the spectrum of the spectrum for the deterministic process[13]. Therefore, the low-frequency portion of the potential noise PSD could be used to characterize the pitting corrosion process. Conventional ENA techniques have been used with a certain degree of success in detecting the pitting corrosion process. However, it was not clear from these studies whether conventional ENA could effectively differentiate the rapid growth of corroded pits in a small area (i.e., SLP) from the uniformly distributed pitting over the entire area. It appeared to be necessary to inspect a much lower frequency range of the potential noise PSD spectrum (e.g., beyond 10^{-3} Hz) to differentiate dominating SLP from uniformly distributed pitting. However, in this low-frequency range, some artifacts, such as flicker noise, could limit the application of conventional ENA.

We have developed a new approach to signal collection for electrochemical noise measurements. The new method can effectively detect the SLP process and avoid the artifacts. Instead of collecting the original corrosion current and potential noise signal, we collect the current and potential noise level (CNL and PNL). The CNL and PNL values are collected by calculating (in-situ) the root-mean-square (RMS) of the ac components of a few hundred noise-signal data points. These data points are continuously recorded during one electrochemical noise measurement. Under the new method, the data collected reflect the trend of amplitude change of the current and potential noise caused by corrosion during any test period. Therefore, in theory, the new form of signal data can be collected at much lower frequencies (e.g., 10^{-6} or lower), allowing researchers to avoid the signal drift (or any interference from other sources) caused by using a very low recording (i.e., sampling) rate. We also found no artifacts, such as flicker noise effect, from the instruments or environment during the CNL and PNL measurements. This new approach, in principle, should improve the resolution of PSD analysis in the low-frequency range and allow analysis of any deterministic process recorded by the noise signal. The effectiveness of this new technique in detecting SLP was demonstrated by laboratory experiments using electrochemical cells under natural pitting corrosion conditions. The method was also evaluated using laboratory flow-loop systems that were built to simulate corrosion in a gas pipeline. The flow-loop system also allowed us to design reproducible experiments under controlled conditions to examine various corrosion mechanisms and effects of chemical treatments.

EXPERIMENTAL METHODS

Electrodes

Electrochemical noise measurements were carried out by simultaneously recording the current noise and potential noise with a three-electrode probe. The working and counter electrodes were shorted together and connected through a zero-resistance-amperometer (ZRA) to monitor the corrosion current flow. The third electrode was used as a reference electrode to measure the corrosion potential of the shorted electrode pair through a high-impedance voltameter. A personal computer with plug-in-potentiostat (PC-3)(1) served as the ZRA and voltameter.

Corrosion Cell

The results were obtained from the study of galvanic corrosion in an aqueous system. A glass H-cell was used to perform the experiments. The solution in the cell could be purged with oxygen or nitrogen gas in two different chambers of the cell. Two types of electrochemical probes were used. Type I probe included a standard calomel electrode (SCE) as the reference electrode and a carbon steel coupon (C1018)(2) as the working electrode. The counter electrode was either the same material as the working electrode or a stainless steel (S.S. 316) electrode. The stainless steel counter electrode is nobler than the carbon steel coupon. Therefore, the working electrode (i.e., the carbon steel coupon) was forced into a galvanic corrosion. The type II probe was a commercial probe(3) that consisted of three round-end cylindrical electrodes. Again, carbon steel (C1018) was used both in the working and in the counter electrodes. A stainless steel (S.S. 304) electrode was used as the reference.

Before each experiment, the new specimens were cleaned in an acid cleaning solution and rinsed with deionized water. After rinsing, the specimen was cleaned by sonication in water. The cleaned specimen was dipped into acetone then dried in an oven to remove the water. Electrochemical noise and weight loss measurements were made in each experiment.

Flow-Loop System

Four independent flow loops (A, B, C, and D) with separate simulated produced water solutions, fluid reservoirs, and pumps were used. Each flow loop was 4 in. in diameter and 6 ft long with separate flow control and gas purging system. The simulated produced-water fluid was circulated from 5-gal reservoirs through each loop. Two commercial ENA probes (i.e., type II) were inserted into each of the four loops, for a total of eight probes.

In order to evaluate the performance of the new ENA technique in detecting SLP caused by MIC, several nutrients and salts (such as chloride and sulfate) were added to accelerate the MIC in the flow-loop system. Ethanol was injected into each test loop to trigger pitting corrosion due to the fact that ethanol was rapidly converted by sulfate-reducing bacteria to acetic acid and hydrogen sulfide.

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(1) Trademark of Gamry Inatruments, Inc.
(2) Obtained from Metal Samples, Inc.
(3) Rohrback Cosasco, Inc.

Data Acquisition

The current and potential fluctuations of corroding samples were read during a short period of time (e.g., 4 to 30 s) with consecutive 400 to 600 measurements (i.e., sampling rate of 0.01 s to 0.05 s). The RMS of the ac components of these consecutive data points was calculated and the results were recorded in a data file. The measurement/calculation was repeated at every time interval (e.g., at 10 to 120 s) for a long period of time (e.g., 20 to 120 h). Thus, the data recorded represent the potential and current noise levels. These results were analyzed by fast Fourier transform to obtain the PSD of potential and current noise levels. The linear slope of the low-frequency portion of the PSD (called the α value) was calculated by linear fitting using the least-square method.

RESULTS AND DISCUSSION

Corrosion Process Evaluation in a Laboratory Electrochemical Cell

Researchers have demonstrated some success in using the conventional ENA technique to detect the initiation of pitting corrosion, but the technique is subject to interference in the analysis of the PSD in low frequency ranges. Although detecting the initiation of pitting corrosion in a process environment is important, the ultimate goal is to monitor the sustained growth of pits in a localized area. Pits caused by corrosion can cover the entire or a large portion of the area of the material or it can happen only in a very limited area. On the basis of total weight loss from a metal surface, the former is no different than the latter. However, SLP is obviously of more concern because of its tendency to cause a hole in the material. The uniform corrosion rate can be measured using many different methods. The challenge is to detect the severity of SLP in a background of uniform corrosion. The new ENA technique is capable of extracting the sustained corrosion signal from the mixture of uniform corrosion and pitting corrosion processes.

Figures 1 (a) and (b) show the PSD of conventional ECN measurements of SLP and uniform corrosion on metal coupons. The potential PSD in both cases shows a very similar value of slope (i.e., $1/f^{\alpha}$, $\alpha = 37$) with the linear portion of each line, both bending at a frequency of around 0.1 Hz. On the basis of Figure 1, it appears that the conventional PSD of potential (PSDP) cannot be used to distinguish between SLP and uniform corrosion. This result was expected because of the difficulties caused by the interference generated in the low-frequency range of the PSD analysis. However, using the new ENA data acquisition procedure, Figures 1 (c) and (d) show a clear difference in the PSDs of measured potential noise level (PSDPNL). The slope (i.e., the $-\alpha$) of the PSDPNL of the coupon with uniform corrosion is -11, while that of the coupon with SLP is -39. The α value thus shows a significant difference between uniform corrosion and SLP.

The correlation of SLP with the new PSD of PNL is demonstrated for metal coupons in Figures 2 and 3. When SLP is in progress, the PSDPNL changes dramatically. Unlike uniform corrosion, which is represented by the stochastic process, once SLP occurs, the PSD of PNL reveals a deterministic process. This process can be seen in Figure 2(a), which shows a carbon steel coupon with deep pits. Because the coupon was immersed in pure water with air purge only, a passivation film of iron hydroxide covered most of the coupon surface area. However, traces of chloride ion might exist in the water and aid in the formation of pits. The PSDPNL (Figure 2[b]) shows a α value of 40 in the low-frequency portion, which indicates a deterministic process. Depending on the total number and depth of the pits that are forming, the α value for the PSDPNL could vary from 0 to 40, which reflects the domination of the SLP formation compared with uniform corrosion. When the α value is between 10 and 30, the metal surface is under an intermediate corrosion process attack. In this regime, the formation of pits was either relatively shallow or many shallow pits were merging to form a group of pits that extend over a large portion of the total surface area. An example of uniform corrosion caused by shallow and uniformly distributed pits is shown in Figure 3. In this case, the coupon was immersed in deionized water for 68 h. The final α value of the PSDPNL is 15; the surface morphology shows that shallow pits have formed on the coupon surface. This apparently indicates a less dominating deterministic process of pit formation compared to the stochastic process of uniform corrosion. A comparison between the coupon in Figure 2 and the coupon in Figure 3 supports the hypothesis of a competition between a deterministic and a stochastic process more clearly. The operating conditions for coupons in Figure 2 and Figure 3 were the same. However, the coupon shown in Figure 2 has much deeper pits than the coupon in Figure 3. The total number of pits on the coupon in Figure 3 is greater than on the coupon in Figure 2 (the total pit number ratio $\approx$ 50/5). The aggressiveness of SLP (i.e., the domination of the deterministic process over the stochastic process) shown in Figure 2 make its α value near 40. The overall corrosion rate for the coupon in Figure 2 is one order-of-magnitude smaller than that for the coupon in Figure 3 (i.e., 1.08×10^{-2} mg/h vs. 1.78×10^{-1} mg/h from the weight loss measurement). These results are also evidence for the domination of SLP over uniform corrosion.

Corrosion Process Evaluation in the Flow-Loop System

The new ENA system was used to monitor MIC using simulated produced-water fluid in a flow-loop system.

In order to evaluate the performance of the new ENA technique in detecting SLP, several chemicals were added to accelerate MIC pitting. Ethanol was injected into each loop to trigger pitting corrosion during the test period. Figure 4 shows the slope profile (4a) of PSDPNL and the morphology on the tip of the probe (4b). As discussed above, the slope indicates that uniform corrosion was dominating the corrosion process. This finding was confirmed by the morphology on the tip shown in Figure 4(b).

Figure 5(a) shows the change in slopes in PSDPNL during the test period from a separate probe in a different loop. From the change in profile of the PSDPNL slopes, it is clear that uniform corrosion and pitting corrosion can alternate and dominate the corrosion process. The injection of ethanol was also able to trigger SLP, which is indicated by a decrease in the slope. Analysis of the change in profile of slope in the tested probes revealed that six out of the eight probes developed SLP. These results were confirmed by weight loss and morphology analysis of the probes after the test period. Figure 5(b) shows the morphology of the localized pitting corrosion attack on an electrode surface. Similar statistical correlation between maximum pitting rate or the total corrosion rate and several other parameters derived from the signal processing of the prescreened ENA data were attempted. None of them appeared to have a significant linear correlation constant.

Another experiment was performed in the flow loops to monitor in-situ the effectiveness of corrosion inhibitors in the test system. During the test period, corrosion inhibitor was added to loop D while ethanol was added to both loops D and B. Figure 6 shows the change in the profile of the slope of PSDPNL in loops B and D. As expected, the PSDPNL slope of the probe in loop D changed immediately upon addition of the treatment chemical from a SLP signal to a uniform corrosion signal (i.e., larger than -10). On the other hand, the probe in loop B continued to show SLP signal. Therefore, it appears that the new ENA method can be used as an in-situ process monitor for corrosion control.

CONCLUSION

A new data-collection and analysis approach for ENA to monitor SLP was developed. The new PSD analysis technique with an appropriate ENA sensor was used to distinguish SLP from generalized corrosion mechanisms on a metal surface in an aqueous system. When the metal was under a SLP attack, the PSDPNL curve appeared as a linear decline in the low-frequency portion with a slope equal to -40 dB/decrease ($\alpha = 40$). The linear decline in the low-frequency range is believed to be caused by the dominating process of localized pitting corrosion on the entire surface. Under the hypothesis of stochastic/deterministic competition during the SLP process,[13] the corrosion mechanism changed to a deterministic process from a more general stochastic process. When only uniform corrosion occurs, the PSDPNL was maintained at a near-constant level throughout the entire frequency range (i.e., the α value is close to zero). This was due, again (according to the hypothesis), to the fact that the general corrosion mechanism is a stochastic process. Therefore, its PSDPNL is independent of the frequency. If the uniform and SLP equally dominate on the metal surface, a slope between -10 and -30 (db/decade) is observed. The departure of α value from the 40 db/decade level may occur for two reasons. First, the large increase in the number of pitting sites on the coupon surface would diminish the deterministic signal created by the SLP process as it started to shift into a more random signal. The second reason is opposite to the first; if the localized pitting sites were very few and small, their weak potential noise signal would be influenced more strongly by the random signal from the background general corrosion signal that predominates.

A flow-loop system was used to test the ENA corrosion monitoring system. The ENA probes were evaluated using simulated produced water in a flow-loop system with nutrients added to activate the MIC processes. As demonstrated previously using small corrosion cells under stagnant conditions, the linear slope of PSDPNL is also capable of detecting the SLP in a flow-loop system. The ENA probes were able to detect the onset of pitting corrosion and record the change in corrosion when treatment chemicals were added.

ACKNOWLEDGMENTS

This work was supported by the U.S. Department of Energy, Energy Research, Laboratory Technology Research Program, under Contract W-31-109-Eng-38 and under a Cooperative Research and Development Agreement (CRADA) with the Southern California Gas Company and the Gas Research Institute.

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LIST OF FIGURE CAPTIONS

FIGURE 1: Power spectrum of electrochemical noise data acquired by zero-resistance measurement of conventional as well as ANL new data acquisition system:
(a) Coupon sample with pitting corrosion, by conventional data acquisition system.
(b) Coupon sample with uniform corrosion, by conventional data acquisition system.
(c) Coupon sample with pitting corrosion, by ANL data acquisition system.
(d) Coupon sample with uniform corrosion, by ANL data acquisition system.

FIGURE 2: PSD of potential noise level and surface morphology of type I probe with S.S. 316 as counter electrode. The probe was immersed in water with traces of NaCl and purged with air, pH=6.12.

FIGURE 3: PSD of potential noise level and surface morphology of type I probe with S.S. 316 as counter electrode. The probe was immersed in water and purged with air, pH=6.97.

FIGURE 4: Change profile of PSDPNL and morphology of probe 2 from flow loop A.

FIGURE 5: Change profile of PSDPNL and morphology of probe 6 from flow loop C.

FIGURE 6: Change profile of PSDPNL's linear slope in different corrosion environments.

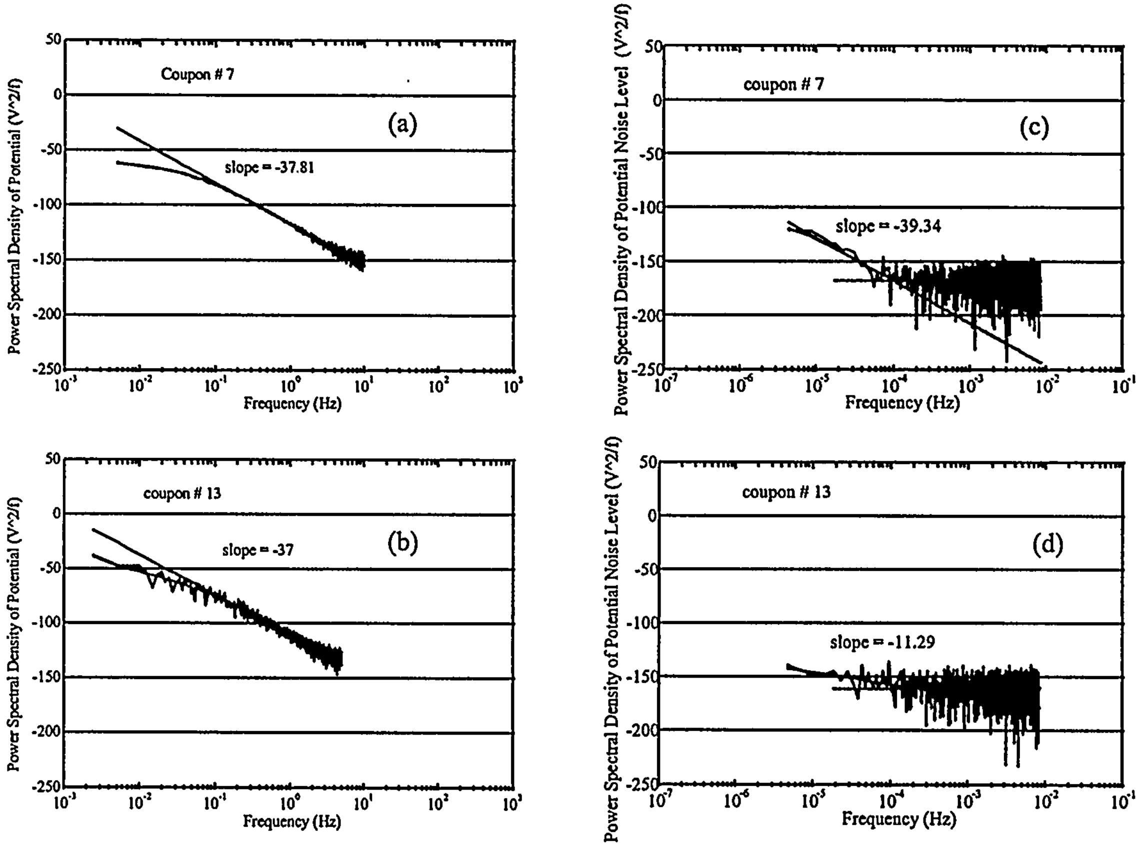
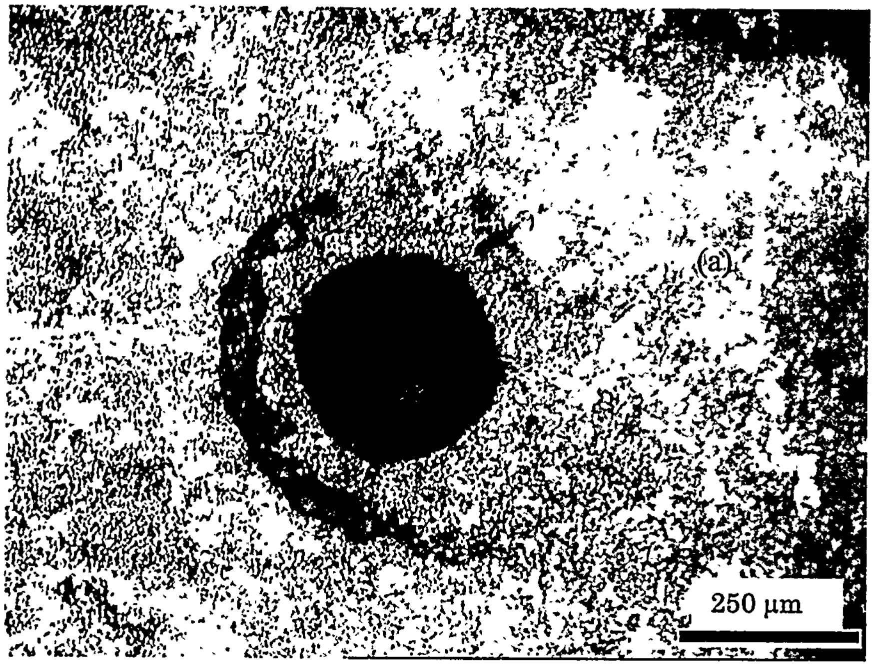


FIGURE 1 Power spectrum of electrochemical noise data acquired by zero-resistance measurement of conventional as well as ANL's new data acquisition system.

(a) Coupon sample with pitting corrosion, by conventional data acquisition system.
(b) Coupon sample with uniform corrosion, by conventional data acquisition system.
(c) Coupon sample with pitting corrosion, by ANL's data acquisition system.
(d) Coupon sample with uniform corrosion, by ANL's data acquisition system.



diameter : 275 µm, depth : 88 µm

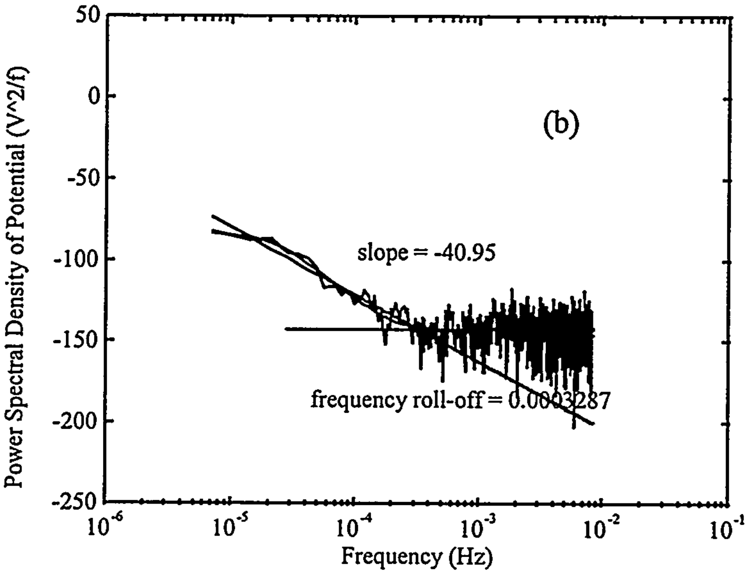
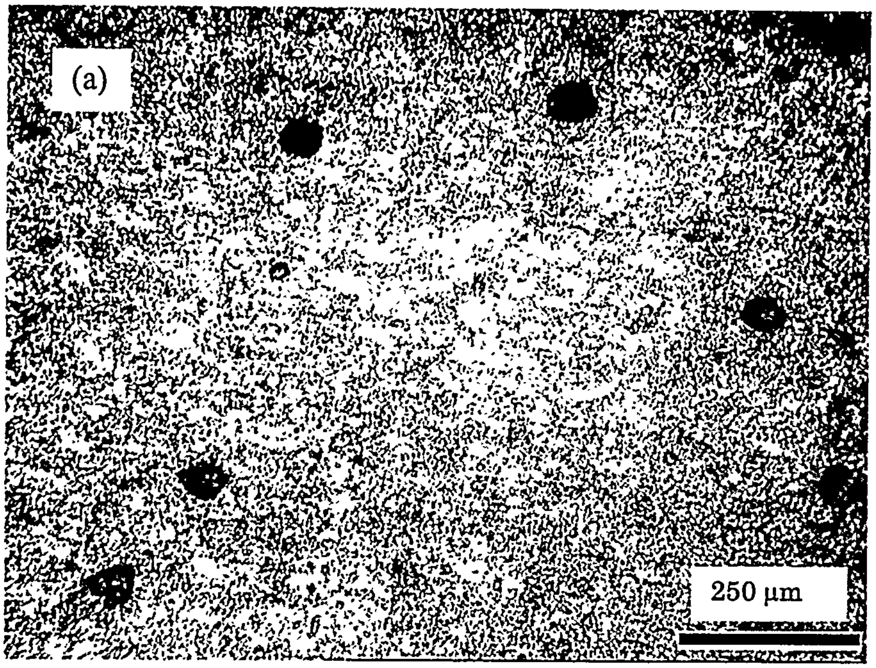


FIGURE 2 PSD of potential noise level and surface morphology of type I probe with S.S. 316 as counter electrode. The probe was immersed in water with traces of NaCl and purged with air, pH=6.12.



small pits uniformly distributed on the front surface
diameter : 50 µm, depth : 11µm

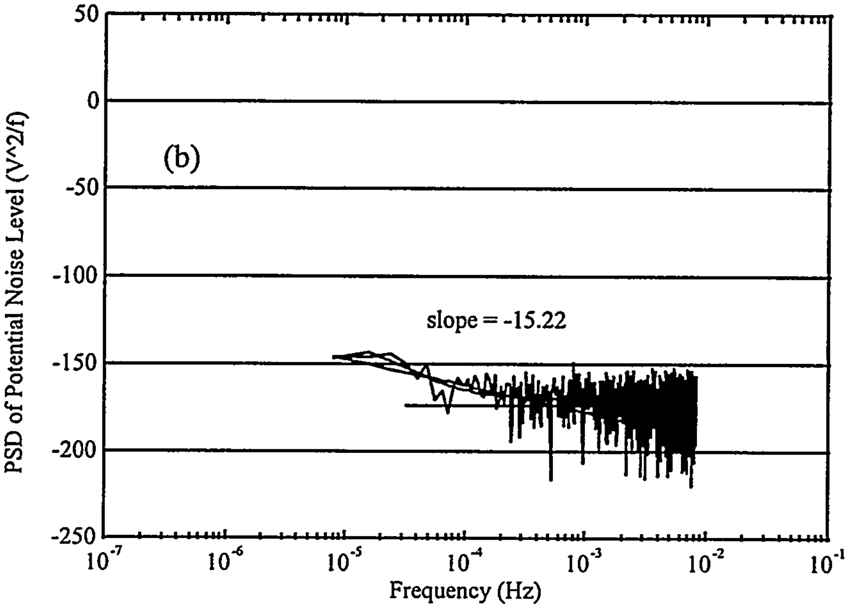
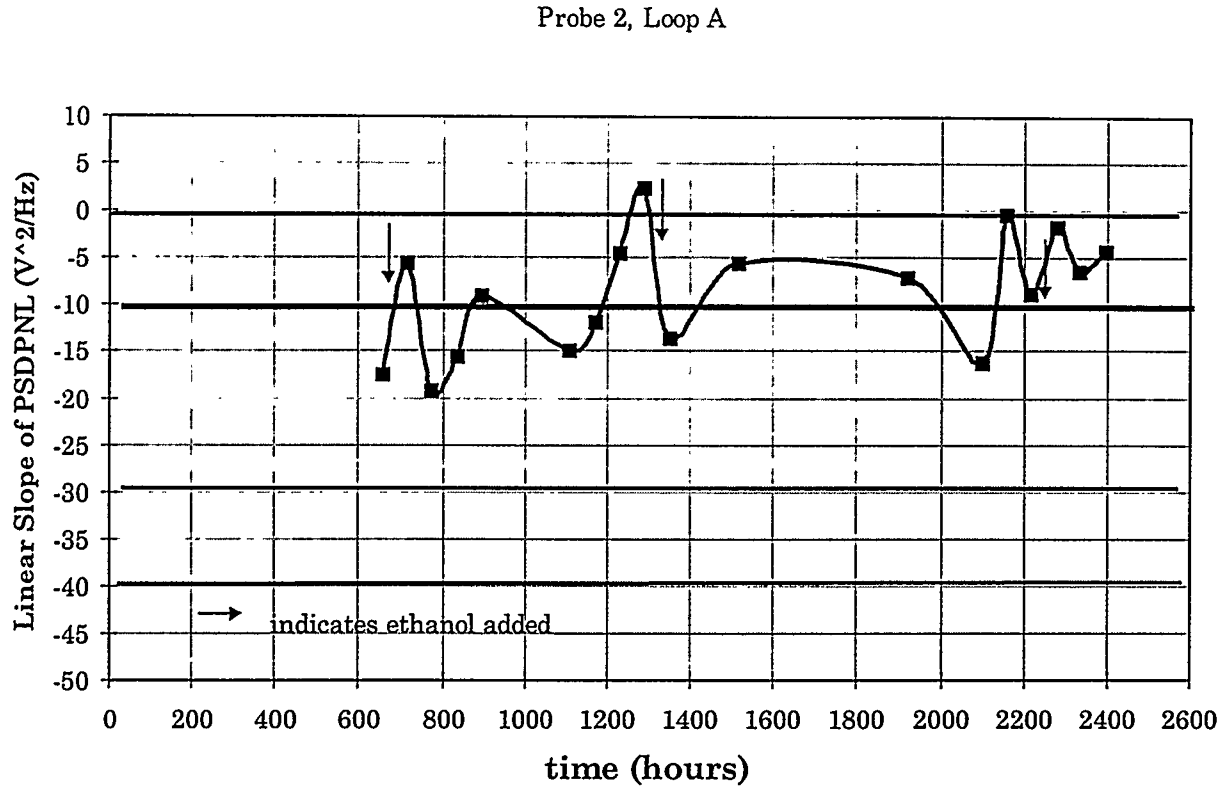
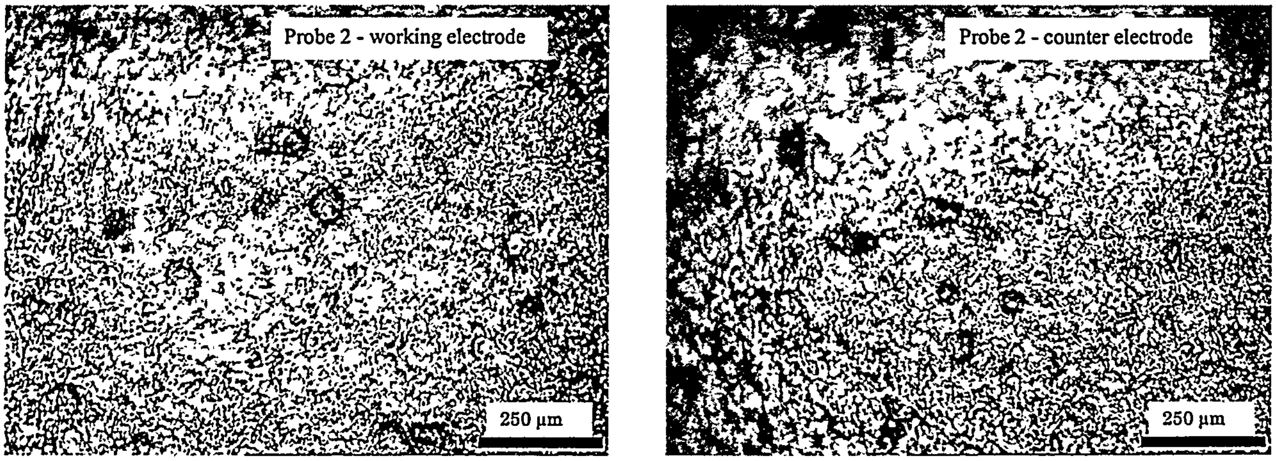


FIGURE 3 PSD of potential noise level and surface morphology of type I probe with S.S. 316 as counter electrode. The probe was immersed in water and purged with air, pH=6.97.

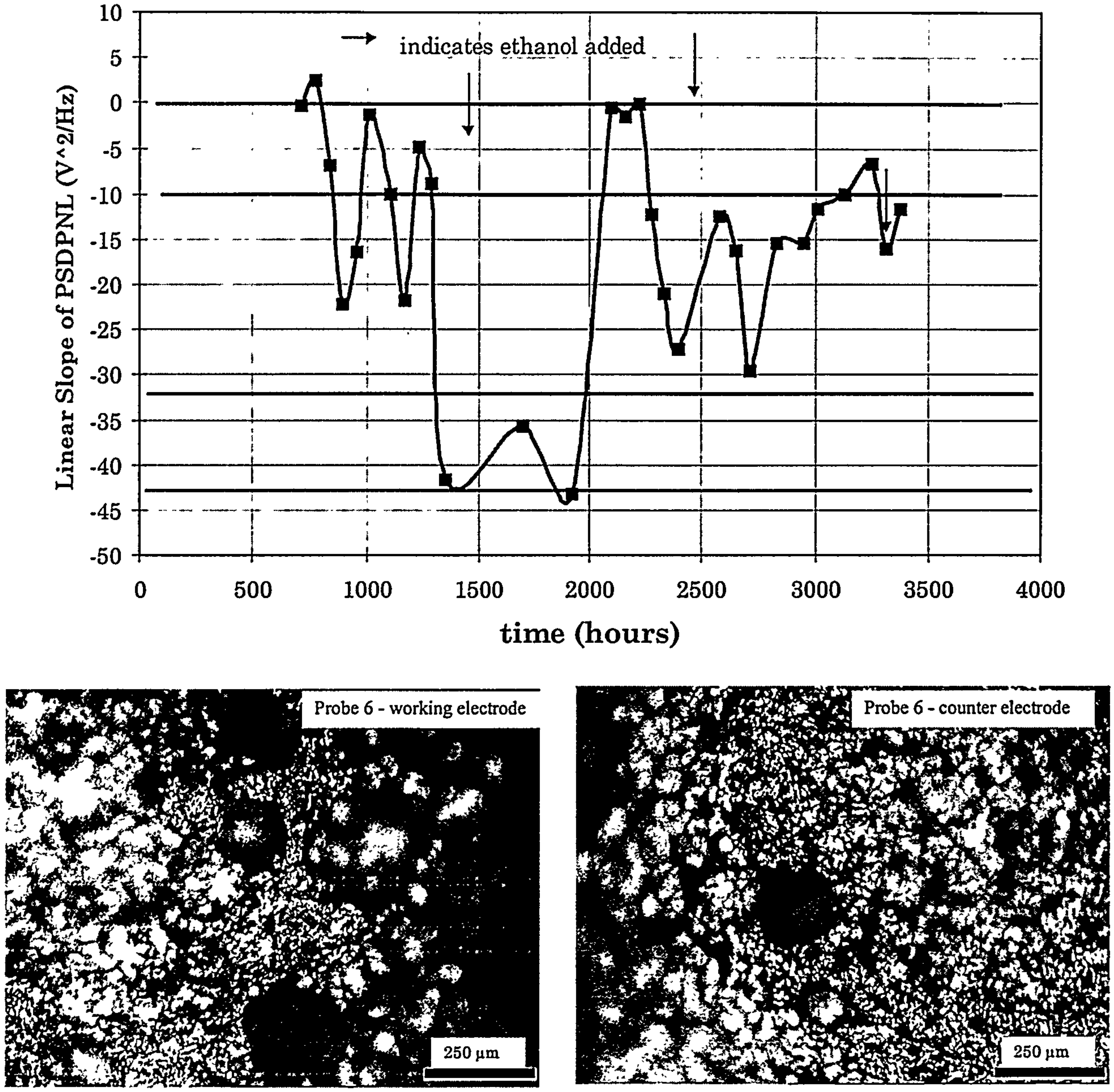




diameter ≈ 30-50 µm; depth ≈ 10-12µm; surface roughness ≈ 3 µm

diameter ≈ 40 µm; depth ≈ 10 µm; surface roughness ≈ 3 µm

FIGURE 4 Change profile of PSDPNL and morphology of probe 2 from flow loop A.



diameter ≈ 125 µm; depth ≈ 80 µm; surface roughness ≈ 3 µm

diameter ≈ 120 µm; depth ≈ 37 µm; surface roughness ≈ 3 µm

FIGURE 5 Change profile of PSDPNL and morphology of probe 6 from flow loop C.

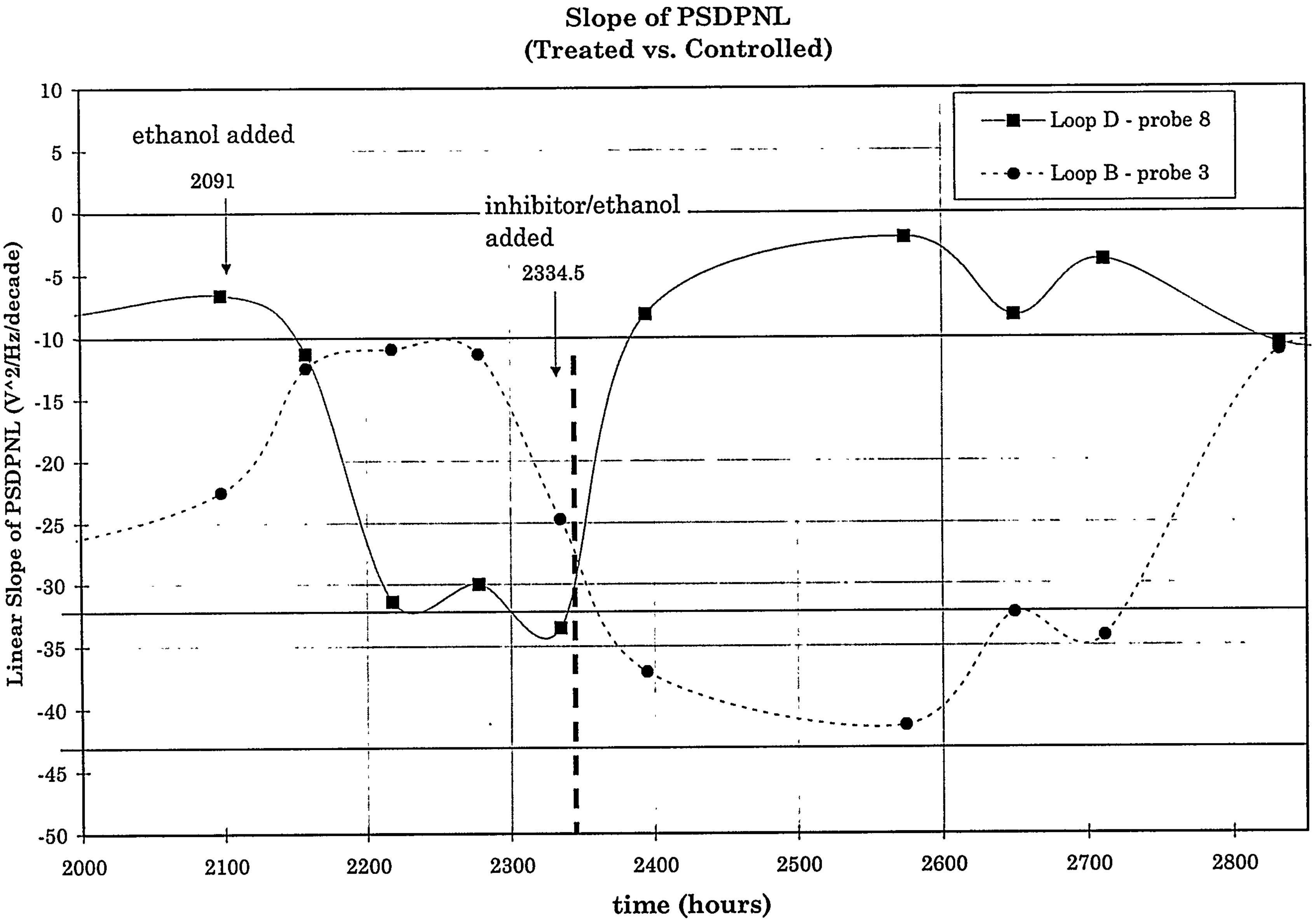


FIGURE 6 Change profile of PSDPNL's linear slope in different corrosion environments.

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Author(s) YuPo J Lin, James R Frank, E J St Martin, Daniel H Pope collectively the "Author")

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