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Electrochemical Noise for Detection of Stress Corrosion Cracking of Low Carbon Steel Exposed to Synthetic Soil Solution[1]

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In the last years, electrochemical noise (EN) has demonstrate that is capable of detect the initiation of cracks during the slow strain rate tests (SSRT). In this research, EN has been used as a tool to detect stress corrosion cracking (SCC) in low carbon steel (X52) exposed to a synthetic soil solution called NS4 at room temperature with pH of 5, 8 and 10. The electrochemical potential and current noise were measured simultaneously during SSRT. Relation between EN and SCC process was analyzed. EN readings of current consisted of transients with high intensity and frequency during all SSRT tests. EN readings of potential at the maximum strength (UTS) and before fracture, increase intensity and amplitude of the transients, attributed to beginning of cracking. Localized index (LI) values indicate a mix corrosion type (general and localized corrosion) during SSRT tests. According to SCC index obtained from mechanical properties, it is clear that X52 steel has low SCC susceptibility. Scanning electron microscopy (SEM) observations were carried out in the fracture surface and longitudinal sections. The specimens tested in NS4 solution with pH 5 were the most likely to present SCC, additionally in this condition a brittle fracture with transgranular appearance was observed.

Keywords: *Corrosion, Stress corrosion cracking (SCC), electrochemical noise (EN), slow strain rate tests (SSRT), X52 steel.*

1. Introduction

Underground pipeline steels of oil and gas industry are mainly low alloy steels of API specifications, such as X52, which has a ferritic-pearlitic structure. These materials are susceptible to transgranular stress corrosion cracking (TGSCC) in dilute solutions of near neutral pH when the protective coating is damaged and where the ground water is in direct contact with the pipe surface[1-6].

Different electrochemical techniques have been used to estimate the risk of the SCC. The potentiodynamic polarization curve (PC) is one of these techniques; it has been used to predict the SCC process and to establish a susceptibility potential range in pipeline steel[7].

Another technique is the electrochemical noise (EN). This technique study the fluctuations of the potential and current generated by the corrosion process when the metal does not has any perturbation (at open circuit)[8].

The application of the EN to detect and monitoring SCC process in laboratory is widely accepted by many researchers. Cottis et al.[9] were the first researchers that reported the study of SCC by EN. They measured and analyzed the noise of the current and potential generated by three different electrolyte-metal systems; they found an increment in the noise level generated by the sample under stress at the beginning of the cracks and a decrement of this noise level, as the crack propagates. González et al.[10] used the EN in order to detect the beginning and propagation of the cracks in SCC, in a martensitic stainless steel sample immersed in acid and deaerated NaCl brine. In this work, the researcher correlated the potential transients with the nucleation and propagation of the cracks. In addition, Gonzales et al.[11] carried out some investigation about the differences between general and localized corrosion in steel immersed in thiosulfate solution. The analysis of EN in the time domain showed current transients with high intensity and low frequency during the SCC process. On the other hand, when the steel was passivated and under uniform corrosion, current transients with low intensity and high frequency were found.

Electrochemical noise techniques have been used for the detection of spontaneous changes in corrosion process. The current noise can provide real-time evaluation of stress corrosion cracking (SCC) behavior in austenitic stainless steel[12,13], nickel-based alloy[14,15], martensitic stainless steel[11,16]

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and aluminum alloys[17,18]. The changes in the amplitude and frequency of the potential and the current transient are the results of the breakdown of the passive film followed by the initiation and propagation of SCC. By combining electrochemical noise and acoustic emission techniques is possible to distinguish between damage stages with a predominant electrochemical contribution and damage stages with a predominant mechanical contribution to crack propagation, therefore with these two techniques is possible obtaining a reliable detection of SCC[13,16]. However, there is few studies in low carbon steels[19,20]. Therefore, in this work, EN has been used as a tool to detect SCC in low carbon steel (X52) exposed to a synthetic soil solution called NS4.

2. Experimental Procedure

2.1 Material

The material used in the present study was an API 5L X52 low carbon steel with the chemical composition showed in Table 1. The samples used in SSRT were machined according to NACE TM 198 standard[21].

The surface in the gage section was polished with 400 to 1200 SiC grit paper in an orientation parallel to the subsequent loading direction of the SSRT in order to assure similar surface conditions for all tests. All the SSR tests were carried out by triplicate to ensure repeatability.

2.2 Test solution

A simulated ground water solution (called NS4), with pH of 5, 8 and 10, as the corrosive environment in this study was used. NS4 synthetic solution has been widely used to simulate the soil solution in the study of near neutral pH-SCC behavior. However, another synthetic soil solution called NS1, NS2, NS3, NS4, NOVA and C1 has been used in similar studies[22-25]. Table 2 shows the chemical composition of the NS4 solution used. The NS4 pH solution was around 8.0. The pH solution was adjusted with HCl and sodium hydroxide. All SSRT were carried out at room temperature and atmospheric pressure.

2.3 Slow strain rate tests (SSRT)

SSRT technique has been used in the last years with smooth cylindrical specimens, machined from the pipe steel, and strained to failure in presence of a corrosive environment. This technique is very effective to identify probable cracking environment of underground pipelines. Figure 1 shows the experimental set up used to carry out the SSRT. SSRT were carried on smooth cylindrical tensile samples machined according to NACE TM 198 using a MCERT machine (Mobile Constant Extension Rate Tests) at strain rate of 1×10^{-6} s^{-1} in air and in a synthetic soil solution (NS4 solution) with pH 5, 8 and 10. Most SSRT are conducted in the range of extension rates from 10^{-5} to 10^{-7} in/s (2.54×10^{-4} and 2.54×10^{-6} mm/s). According to ISO 7539-7 for ferritic steels a strain rate of 1×10^{-6} s^{-1} is recommended. The length direction of the sample was parallel to the circumferential direction of the pipeline steel in order to ensure that the subsequent crack growth was located in the longitudinal direction of the pipe as is typically observed in the field. Cylindrical samples with a reduced length of 1 inch and 0.150 inches in diameter were machined with a total exposed area of 3.26 cm^2. After the test was completed, the fractured sample was immediately removed, cleaned using inhibit acid and acetone for SEM examination.

Table 2. Chemical composition of NS4 solution (g/L).

$NaHCO_3$	$CaCl_2 . 2H_2O$	$MgSO_4 .7H_2O$	KCl
0.483	0.181	0.131	0.122

2.4 Stress corrosion cracking assessment

After carried out the SSRT, SCC susceptibility was evaluated according to NACE TM-0198 and ASTM G-129[21,26]. The degree of susceptibility to SCC is generally assessed through observation of the differences in the behavior of the mechanical properties such as reduction in area (RA), time to failure (TF), plastic elongation (PE), elongation (EL) and strain (S) of the material in tests conducted in a specific environment (in this case NS4 solution) from that obtained from tests conducted in the controlled environment (air).

Complementary metallographic examination was performed through SEM to establish whether or not there is cracks on the samples. The presence of cracks was evaluated on the longitudinal section of the gage. This observation through the SEM is recommended especially when the ratios (I_{scc}) obtained from the mechanical properties are lower than 0.8.

2.5 Electrochemical noise tests

Electrochemical noise measurements were carried out in a Potentiostat using a typical electrochemical cell with a three electrodes array as is shown in figure 2. Cylindrical tensile samples of X52 steel were used as working electrode (WE), the second working electrode is a platinum (Pt) microelectrode; while a saturated calomel electrode (SCE) was used as reference electrode (RE). With the purpose of analyzing EN transients vs time, the original NS4 solution with pH 8 (without adjust the pH) was selected in EN evaluations. The goal of uses this NS4 solution was to analyze the SCC process in near neutral pH solution by EN monitoring where a high EN response was expected.

Table 1. Chemical composition of the API X52 steel (wt.%).

C	Mn	Si	P	S	Mo	Nb	Cu	Cr	Ni	V	Ti	Fe
0.24	1.40	0.45	0.025	0.015	0.15	0.05	0.50	0.30	0.30	0.10	0.04	Bal.

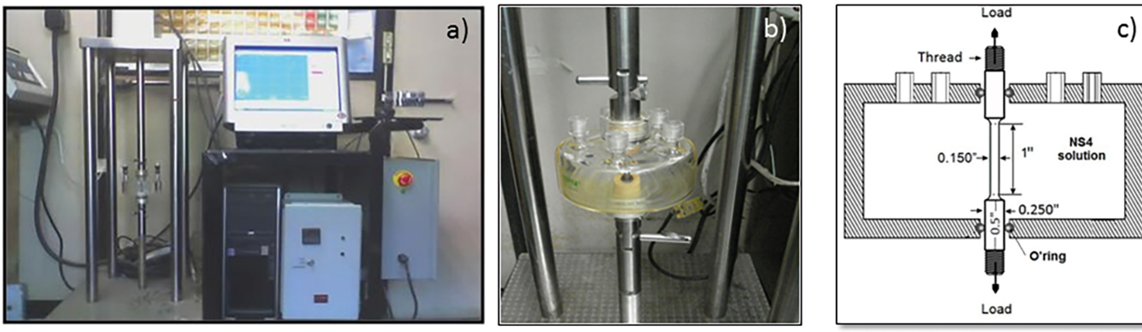

Figure 1. Experimental set up used to perform the SSRT, a) MCERT machine b) glass autoclave and c) specimen dimensions.

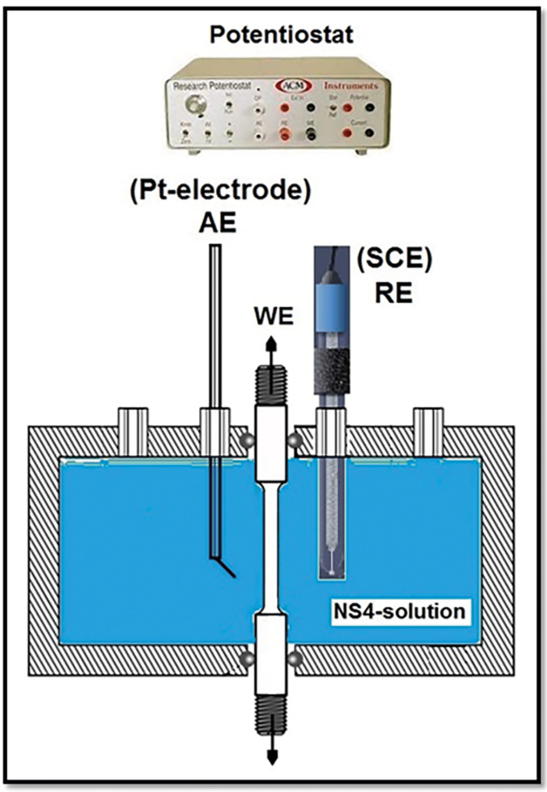

Figure 2. Experimental set up for electrochemical noise tests.

In order to stabilize the redox reactions, the corrosion potential was measured during 30 minutes. Sampling frequency in EN used was 1 Hz. Each EN measurement consisted of 1200 data points and these were carried out at different exposure time. EN data were analyzed in time with the localized index (LI), noise resistance (Rn) and transients of current and potential.

3. Results and Discussion

3.1 Microstructural characterization

Figure 3 shows the typical microstructure for API X52 pipeline steel, which are composed of ferrite (light phase) and perlite (dark phase). Perlite is formed by layers of ferrite and cementite. The average grain size of the microstructure is around 10-20 µm. Low carbon steels generally have a ferrite-pearlite structure containing little pearlite. Additionally, is common that these steels contain sulphur forming manganese sulphide inclusions (MnS).

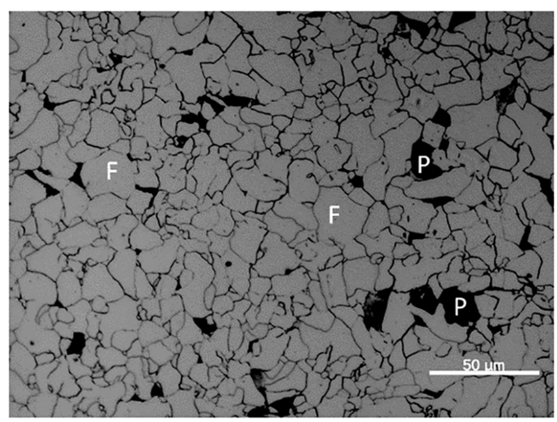

Figure 3. Microstructure of API X52 steel obtained by optical microscopy.

3.2 Slow strain rate test (SSRT)

These specimens were evaluated through SSRT at $1x10^{-6}$ s^{-1} strain rate in NS4 solution with different pH. Figure 4 shows the stress-elongation and stress-strain curves obtained from the SSRT carried out in air and in NS4 solution with different pH at room temperature. It is clearly observed that elongation and strain is affected by the solution. The profile corresponding to X52 steel immersed in NS4 solution shows deterioration of mechanical properties such as elongation (EL) and strain in comparison with the values of the profile of X52 in air. It was observed that solution with pH 5 has a more noticeable effect in decrease the mechanical properties of X52 steel. This effect can be attributed to aggressiveness of solution with acid pH. Decrease in yielding strength (YS), ultimate tensile strength (UTS) and elongation can be attributed to diffusion of hydrogen atoms into the steel provoking embrittlement.

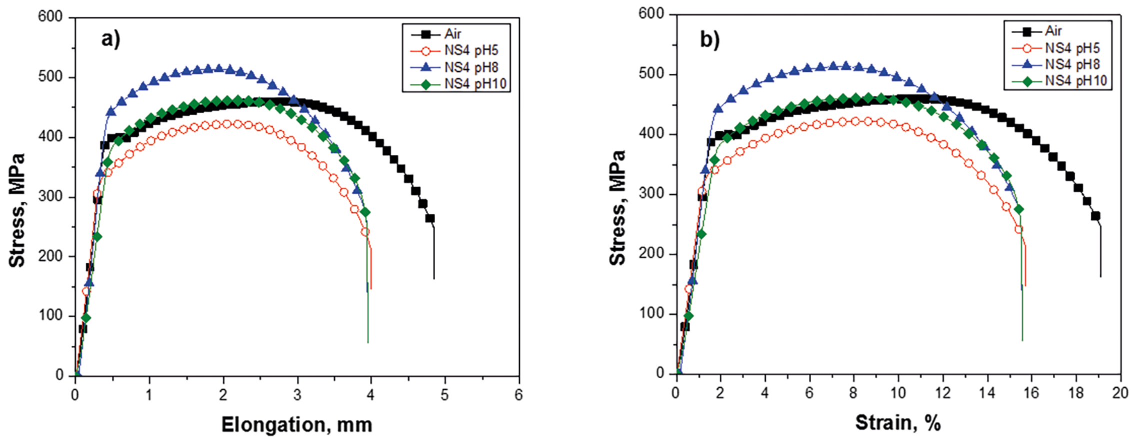

Figure 4. a) Stress-elongation and b) stress-strain profiles obtained from the SSRT.

3.3 Assessment of the SCC susceptibility

The degree of susceptibility to SCC is generally assessed through observation of the differences in the behavior of the mechanical properties of the material in tests conducted in a specific environment (in this case the NS4 solution) from the properties obtained from tests conducted in the controlled environment (air). Table 3 shows a summary of the mechanical properties obtained from SSRT. Ratios of mechanical properties were obtained and results are showed in Table 4. For each specimen change in RAR, TFR, RAR, PER, ELR and SR, in addition to observation by SEM for visual indications of cracks, or sometimes a combination of these methods were used to determining susceptibility to SCC according to NACE TM 198[21] and ASTM G129[26].

The index values obtained from mechanical properties from 0.8 to 1 indicate that the steel has high resistance to SCC and values lower than 0.5 indicate that the steel has high susceptibility to SCC[27]. According to the values of the susceptibility index by SCC presented in Table 4, the X52 pipeline steel has low susceptibility to SCC in NS4 solution. A clear effect of pH solution on the SCC index was not observed. However, a decreases in YS, UTS, EL and deformation was observed, which can be attributed to diffusion of hydrogen atoms into the steel generating hydrogen embrittlement.

3.4 Surface fracture analysis

Figure 5 shows the SEM images of the fracture surfaces of the X52 pipeline steel after carried out the SSRT in air and in the synthetic soil solution (NS4 solution). Measurements of final diameter of fracture were carried out in order to calculate the reduction area (RA). The SSRT of the X52 steel in air presented a ductile fracture, and it is characterized by extensive plastic (permanent) deformation of the material (figure 5a). One mechanism of ductile fracture is known as microvoid coalescence[28]. These voids grow until adjacent voids connect; coalescing into larger voids until final failure of the material occurs[29].

For the samples tested in air the neck formation (reduction of diameter) prior to steel failed was observed, whereas in tests carried out in NS4 solution it can be observed a minor formation of neck in the surface fracture. This feature was

Table 3. Summary of the mechanical properties obtained from SSRT of X52 steel.

Environment	YS (MPa)	RS (MPa)	UTS (MPa)	E (GPa)	Strain (%)	EL (mm)	TF (h)	RA(%)	PE (%)
Air	368	243	450	245	19.1	4.85	52.31	78.95	17
NS4 pH 5	330	212	422	259	15.7	3.99	45.28	72.72	15
NS4 pH 8	463	273	545	279	15.5	3.94	45.24	67.90	14
NS4 pH 10	372	153	462	209	15.6	3.95	45.44	68.16	15

Table 4. SCC index obtained from mechanical properties of SSRT.

Environment	RA (%)	RAR	TF (h)	TFR	PE (%)	PER	ELR (mm)	ELR	Strain (%)	SR
Air	78.95	-	52.31	-	17	-	4.85	-	19.1	-
NS4 pH 5	72.72	0.92	45.28	0.87	15	0.88	3.99	0.82	15.7	0.82
NS4 pH 8	67.90	0.86	45.24	0.86	14	0.82	3.94	0.81	15.5	0.81
NS4 pH 10	68.16	0.86	45.44	0.87	15	0.88	3.95	0.81	15.6	0.82

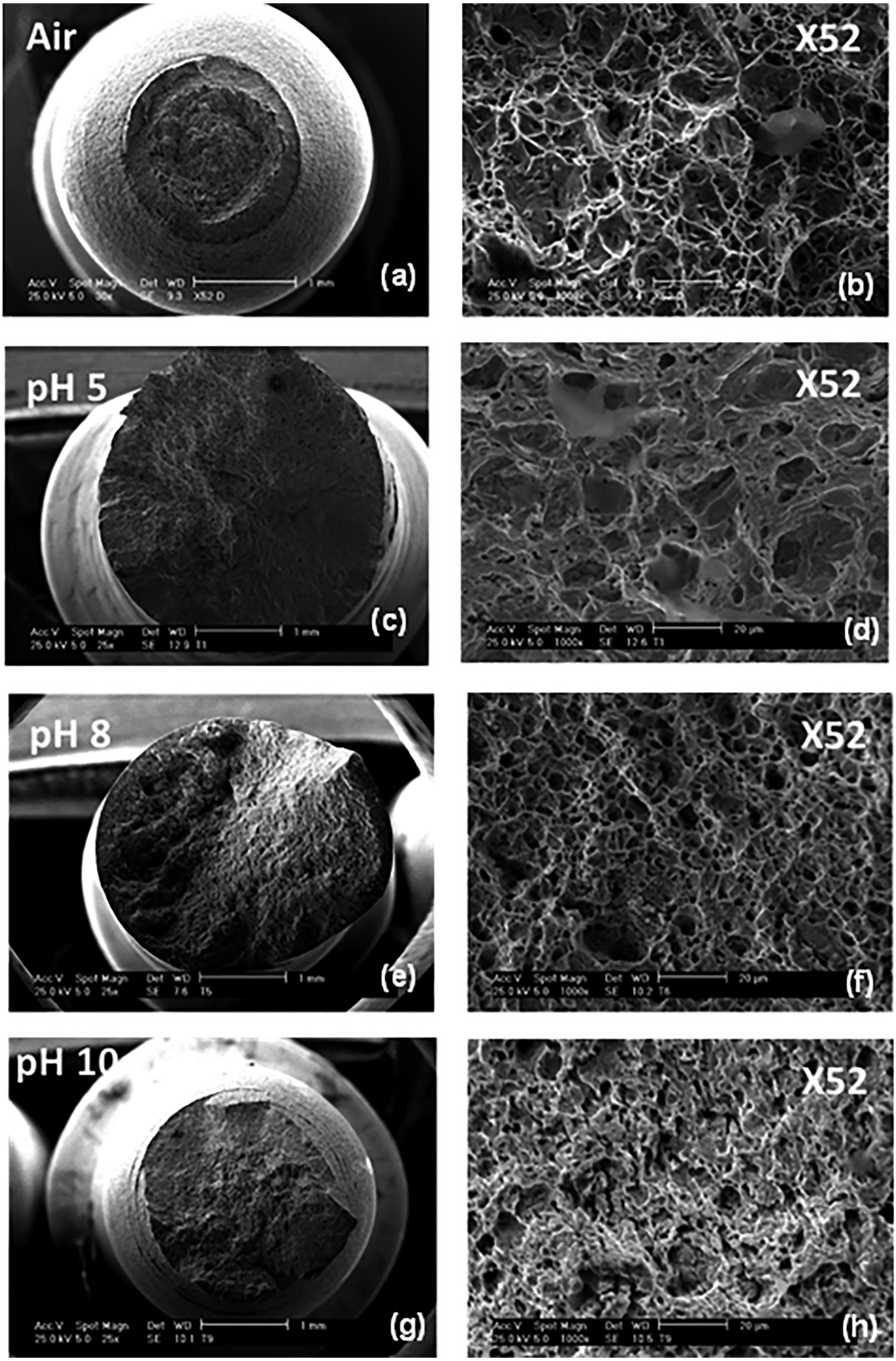

Figure 5. SEM images of the fracture surfaces from X52 pipeline steel, (a-b) samples tested in air and NS4 solution with pH of 5 (c-d), pH 8 (e-f) and pH 10 (g-h).

observed overall in tests with pH 5; this characteristic is indicative of brittle fracture, which can be attributed to diffusion of hydrogen atoms into the steel provoking a brittle fracture type with transgranular appearance as is shown in images of figure 6. Figure 6a shows the main crack surface fracture showing a brittle fracture with faceted characteristic and some cracks close to surface fracture in the deformation region. A selected area observed a higher magnification revealed that hydrogen atoms produce internal microcracks as is shown in figure 6a1. Figure 6a2 shows a faceted region typical of transgranular fracture.

For most brittle fractures, crack propagation corresponds to the successive and repeated breaking of atomic bonds along specific crystallographic planes; such a process is termed cleavage. This type of fracture is called transgranular, because the fracture cracks pass through the grains. Macroscopically, the fracture surface may have a faceted texture (Figure 6a), as a result of changes in orientation of the cleavage planes from grain to grain.

In brittle fractures, it can be assumed that all the energy of fracture is spent on the joining of cracks (figure 6a1) and the plastic deformation associated with it. Consequently, deformation in brittle fractures is localized at the boundaries of the cleavage steps. According to this behavior is possible to point out that SCC susceptibility of the X52 steel exposed to NS4 solution with pH 5 is really low. This was corroborated with SEM observations of gage sections of SSRT specimens. Only few microcracks were observed in the gage section of X52 samples tested in NS4 solution with pH5. Sometimes the cracks initiate from pitting, the conditions of stress and geometry of the pits, permit to evolved cracks from the bottom of the pits[30].

3.5. Electrochemical noise (EN)

EN measurements during the SSRT in NS4 solution with pH 8 were carried out in five different conditions (figure 7) on the stress-strain profile: a) at beginning of the test, b) elastic zone, c) yield strength (YS), d) ultimate tensile strength (UTS) and e) before rupture (RS).

The EN measurements were analyzed in time and frequency domain. In time domain the analysis of E and I transients, localized index (LI) and noise resistance (Rn) were the statistical methods used to study EN.

3.5.1 E and I transients

With the purpose of analyzing EN transients vs time, NS4 solution with pH 8 (without adjust the pH) was selected in EN evaluations. Figure 8 shows record of the current and potential transients of X52 pipeline steel immersed in NS4 solution with pH 8 at room temperature and atmospheric pressure during the SSRT tests. All EN measurements were carried out in five different points on the stress-strain profiles showed in figure 7. In all EN analysis the trend was not removed. Figure 8a-c shows characteristic current transients corresponding to localized corrosion, although it is possible to mention that as increased exposure time of the sample under stress, increased the number of transients. In figure 8a, EN measurements of E showed fluctuations with low amplitude (0.05mV) and a decay with time. Records of I showed a very low occurrence of transients (0.06μA) followed by an exponential decay. In figure 8b can be observed a similar behavior to figure 8a. Finally, in figure 8c, at yield point, it was a higher occurrence of I transients, however the magnitude of the transient decreased (0.02μA). Figures 8d-e corresponding to the plastic zone and prior to the fracture point respectively. In the records of E, the amplitude of fluctuations increased (0.5mV) and the I records showed fluctuations of low amplitude, which can be attributed to an intense localized corrosion process on the metal surface, or to a uniform corrosion process due to was difficult to find transients[18].

According to Bertocci et al.[31] some SCC mechanisms indicate that the cracks can propagate in continuous way with a constant speed of active surface generated in the crack tip, so that the continuous propagation of this crack can provide information about electrochemical noise specifically, transients with really low amplitude and low frequency corresponding to the potential and current signals, such is the information

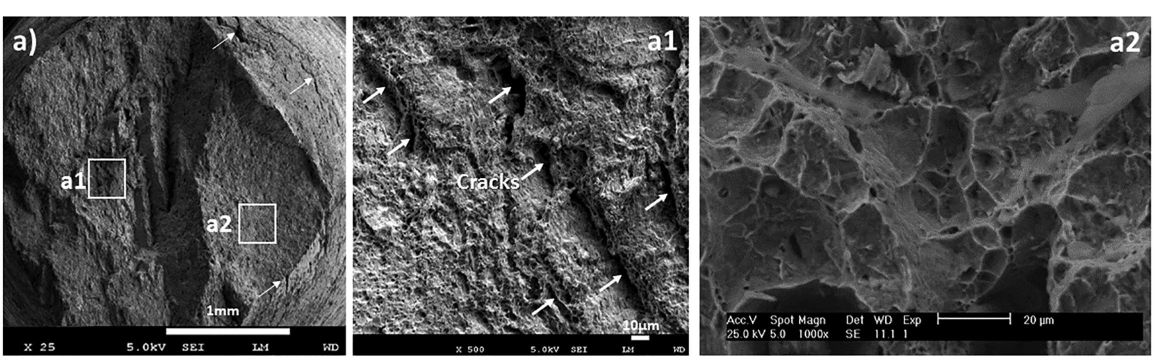

Figure 6. SEM images of the fracture surfaces of X52 pipeline steel tested in NS4 solution with pH of 5 showing the brittle fracture with transgranular appearance.

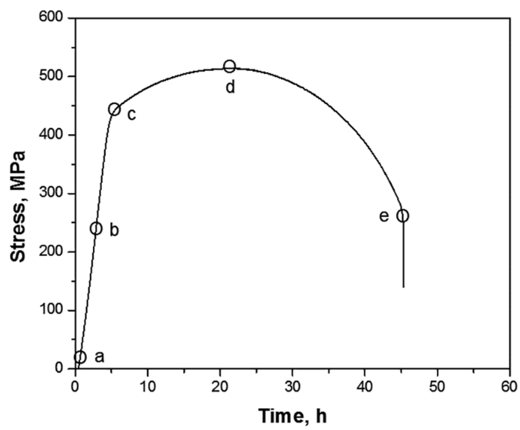

Figure 7. Points of EN measurements, a) at beginning of the test, b) elastic zone, c) yield strength, d) ultimate tensile strength and e) before fracture.

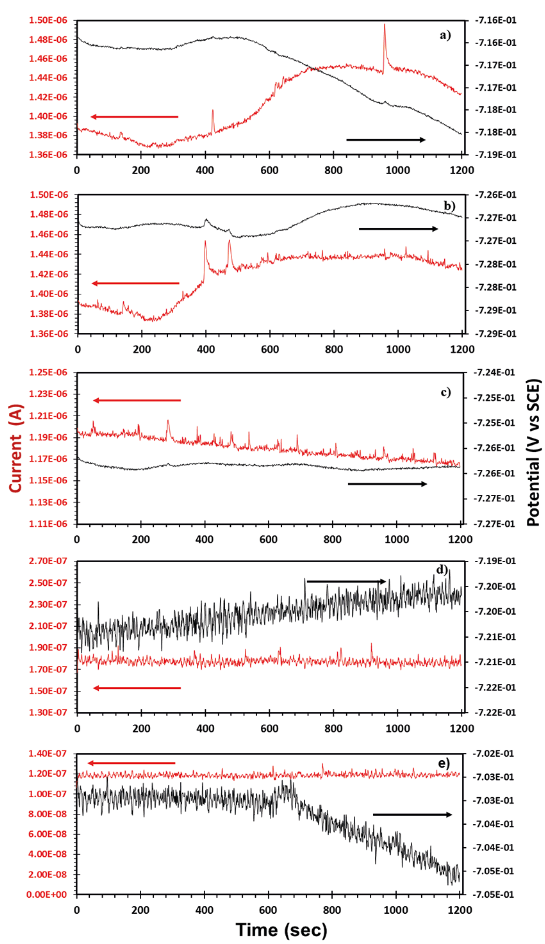

Figure 8. Currents and potential transients of EN for X52 pipeline steel in NS4 solution with pH 8 obtained during SSRT at: a) beginning of the tests, b) elastic zone, c) yield strength, d) UTS and e) before fracture.

showed in the present research and for that reason is possible to say that in spite of that EN data have low amplitude and frequency, the microcracks generated in the SSRT indicate a localized corrosion process.

3.5.2 Localized index (LI)

LI is the distribution measure data around of the root mean square of current. Equation 1 shows the parameters used in the calculation of LI.

$$LI = \sigma_I / RMS_I \qquad (1)$$

Where σ_I and RMS_I is the standard deviation and root mean square of the current.

LI has values from 0 to 1. Values close to 1 indicate localized corrosion, whereas, values close to 0 indicate general corrosion[8,22,32]. Table 5 shows the values range for LI and its relation with the corrosion type according with the morphology of the attack. Table 6 shows the LI values calculate by EN data obtained by X52 pipeline steel corrosion in NS4 solution. These LI are presented as a function of the conditions on the stress-strain profile.

The LI values showed in Table 6 indicate that a mix process dominates the total corrosion process. So it is, the steel sample in this two conditions is attacked by general and localized corrosion. The general corrosion is attributed to the corrosion products film that it was adsorbed on surface of the steel and the localized corrosion is attributed to the cracking induced by SCC. It is important to point out that the mix corrosion process begin in UTS conditions for that reason is possible to note that the nucleation (it should be by pitting) and formation of the crack were in the plastic zone of the stress-strain profile.

3.5.3 Electrochemical noise resistance (Rn)

Noise resistance was calculated at selected conditions of the stress-strain profile. Rn was estimated from the potential and current time records[8,33,34]. Rn was calculated according to equation 2.

$$Rn = \sigma_E / \sigma_I \qquad (2)$$

Where σ_E is the standard deviation of the potential, and σ_I is the standard deviation of the current. Then, corrosion rate (CR) was calculated by dividing the theoretical Stern-Geary constant (B = 0.120 V) by the estimated Rn values[35,36] and finally, the current obtained was divide by the total exposure area in order to get the current density. Figure 9 shows the CR obtained from electrochemical noise resistance (Rn), and the stress profile versus time. This figure shows that in the first corrosion values (Beginning of the test, elastic zone and Yield Strength) the CR decreased, then, in the

Table 5. Correlation between localized index (LI) and corrosion type.

LI values range	Corrosion type
0.001 < LI < 0.01	General corrosion
0.01 < LI < 0.1	Mix corrosion
0.1 < LI < 1.0	Localized corrosion

Table 6. Statistical parameters obtained from EN.

Point	Condition	Rn (kW.cm²)	LI
a	Beginning of the tests	98.84	0.02
b	Elastic zone	134.93	0.02
c	Yield strength	300	0.01
d	Ultimate Tensile Strength	120.36	0.02
e	Before fracture	45.28	0.02

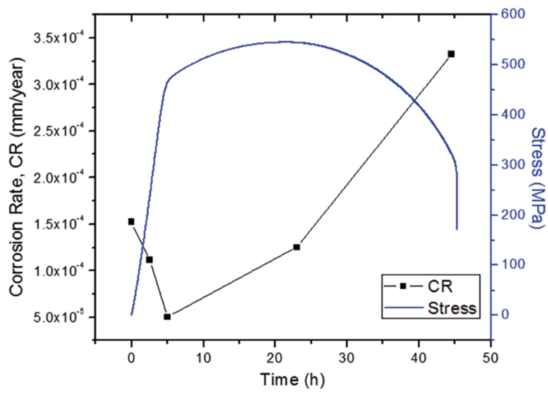

Figure 9. Stress-time profile and CR as a function of the time. CR was obtained by Rn in NS4 solution with pH 8. Five conditions where used: beginning of the test, elastic zone, YS, UTS and before of the fracture.

UTS conditions, the CR increased in a considerable form, to finally, the CR incremented to obtaining the highest CR.

In addition, where the steel sample is under stress in the elastic zone, some crystal defects, like dislocations, oscillate around its equilibrium positions, but they do not move provoking with this behavior that the interface electrolyte-metal do not be affected and the corrosion products film can formed on surface of the steel generating low signal of electrochemical noise current. Since UTS and just after the fracture, the electrochemical noise currents incremented, this behavior could be attributed to the nucleation and subsequently formation of crevice corrosion. It is important to point out that when the stress, in the SSRT, exceeds the elastic limit of the steel sample, a significant movement of the dislocations occur and active places are generated on surface of the steel[37] provoking the rupture of the corrosion products film adsorbed on surface of the steel and mainly it collaborate actively in the formations of the crevice.

3.5.4 Electrochemical noise impedance (Zn)

In frequency domain, the electrochemical noise analysis was carried out transforming the domain time data to power spectrum. Spectral estimation is the process of estimating the amplitude of the various frequencies present in a signal (power spectrum). There are many ways in which a power spectrum can be estimate, but two are commonly used with electrochemical noise signals, the Fourier transform in the form of fast Fourier transform (FFT) and maximum entropy method (MEM). As the methods are commonly used, FFT produces noisy spectra, whereas the MEM produces smoother spectra. According to equation 3 is possible to estimate the Zn[8,31].

$$Zn = \sqrt{\frac{PSD_E}{PSD_I}} \quad (3)$$

Where PSD_E and PSD_I are the power spectrum density of the potential and current respectively.

Figure 9 also shows the CR, obtained from electrochemical noise impedance (Zn), and the stress profile versus time. The behavior of CR values obtained by Zn is similar to the behavior of the CR values obtained by Rn. It is, the CR at the beginning of the test, elastic zone and Yield Strength trended to decreased, but in the UTS condition, the CR increased in a considerable form, to finally, the CR incremented to obtaining the highest CR. These behavior indicate that low electrochemical noise current signals were obtained at the first conditions of the SSRT and these are attributed to the microcracks formation. Since UTS and just after the fracture, the electrochemical noise currents incremented, these should be attributed to the nucleation and subsequently formation of the crack. It is important to note that when the stress, in the SSRT, exceeds the elastic limit of the steel sample the movement of the dislocations can produce cracks that they accelerate the corrosion process.

In general is possible to say that the behavior of CR of the steel sample obtained in the time domain (Rn) and frequency domain (Zn) are similar and they are agree with the analysis of the potential and current transients and LI results. So it is, the current transients and LI results indicating that low electrochemical noise current signals were obtained at the first three conditions of the SSRT and since UTS to after the fracture, the electrochemical noise current signals were more high and these were attributed to the nucleation and formation of the crack.

4. Conclusions

Electrochemical current and potential noise measurements were performed on API X52 pipeline steel samples during SSRT to monitoring the SCC process. The electrochemical noise technique was capable of detecting transient activationpassivation, which may correspond to the cracking phenomenon. According to SCC index obtained from mechanical properties, it is clear that X52 steel has low SCC susceptibility. The specimens tested in NS4 solution with pH 5 were the most likely to present SCC, additionally in this condition a brittle fracture with transgranular appearance was observed. However, only few secondary cracks were observed in the gage section of the samples. Fractures for air and pH 8 and 10 showed a ductile type fracture. According to the analysis of EN, is possible to observe many typical transient corresponding to a localized and mix corrosion process. This fact was verified with the LI values.

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