

# **CRACK INITIATION: STRESS-CORROSION CRACK IN X-52 PIPELINE STEEL IN NEAR-NEUTRAL pH ENVIRONMENT**

**Mimoun Elboujdaini<sup>1,\*</sup>; Bingyan Fang<sup>2</sup>; and Rrg Eadie<sup>3</sup>**

1- CANMET Materials, Natural Resources Canada, 3303 - 33rd Street NW, Calgary, AB, Canada T2L 2A7

2- G.E. Oil & Gas PII Pipeline Solution, 4908 – 52<sup>nd</sup> Street SE, Calgary, AB, Canada, T2B-3R2

3- Dept. of Chemical and Materials Engineering, University of Alberta, Edmonton, Alberta, Canada, T6G 2G6

\* {Corresponding author: [melboujd@nrcan.gc.ca](mailto:melboujd@nrcan.gc.ca)}

## **ABSTRACT**

Specimens from a failure X-52 pipeline that had been in-service for 34 years were pitted using passivation/immersion method developed by the authors to simulate pitted pipelines observed in service. The resulting pitted samples were then cyclically loaded in near-neutral pH environment sparged with 5% CO<sub>2</sub> / balance N<sub>2</sub> gas mixture at high stress ratios (minimum stress/maximum stress), low strain rates and low frequencies which are close to those experienced in active flowing service. It was found that the majority of cracks initiated from the corrosion pits and were less than 0.5 to 0.6 mm deep, which were generally quite blunt. These cracks were transgranular in nature and designated as Stage I cracks and were typical of cracks found in most crack colonies. However, the further growth of these short blunt cracks was significantly influenced by the distribution of the nearby non-metallic inclusions. Inclusions enhanced the stress-facilitated dissolution crack growth, which is the crack growth method proposed by the authors in a related paper. When the orientation of the inclusions was at an acute angle to the orientation of the pits or cracks, and the inclusions were in the same plane as crack initiation or advance, these inclusions would enhance crack growth, or even trap hydrogen which further resulted in the formation of clusters of tiny cracks, which appeared to be caused by hydrogen. The hydrogen produced cracks could be eaten away later by the stress-facilitated further dissolution of the blunt cracks. If these cracks can grow sufficiently however they pose an integrity risk as they can initiate long cracks (near-neutral pH SCC). These hydrogen caused cracks in Stage I were rare. It was nevertheless suggested that cracks deeper than 0.5 to 0.6 mm in the field should be removed to reduce or avoid the threat of rupture. If active corrosion can be prevented such as by cathodic protection then smaller cracks are innocuous.

## **1. INTRODUCTION**

Near-neutral pH stress corrosion cracking (NNpHSCC) has been a significant integrity issue for gas and oil pipelines for over two decades since it was first documented in 1985 [1], and it has been investigated extensively. More than 18,000 colonies of NNpH SCC had been reported on Canadian pipelines up to August 2000 [2], and more than 95% of these colonies became dormant. A small percentage, however, which might grow until failure if not detected and removed, would cause a considerable risk to pipeline integrity management. Pipeline failures have been reported to decrease recently, possibly because more rigorous pipeline integrity management programs have been in place. However, there still have been one or two ruptures a year in Canada since 1995 and numerous cracking discovered through ILI (in-line inspection) or hydrotesting. Although soil models and simulations have been developed to help assess risk [3], these are rather imprecise, and

can only predict the occurrence of minor SCC rather than significant SCC. The mechanisms for NNpHSCC are not yet understood, and further research in this area is needed.

Generally, the main part of pipeline life is consumed in the crack initiation process. Evidence from field failures suggests that corrosion pits might be a common site for crack initiation [3]. In laboratory investigation, it has been found that the earliest cracks appeared to initiate at corrosion pits [4-7] that formed around non-metallic inclusions and later cracks grew from corrosion pits that formed randomly on the surface [8-11], and some cracks induced by corrosion pits were related to stress cells caused by the difference of residual stress level over a much large area [12]. These pits may act as stress raisers to initiate cracks. Cracks can also be nucleated around other types of pits associated with metallurgical discontinuities. Wang *et. al.*[13] indicated that some corrosion pits can be formed preferentially along the heavily deformed metal in scratches on the surface. It was also reported that preferential corrosion occurs at the boundaries of pearlite colonies, and transgranular crack-like features can grow from such surface attack [14]. So NNpHSCC most commonly originates from corrosion pits.

For pipelines in the field, it generally takes years for pits to grow and initiate cracks, and the pit growth may proceed under intermittent exposure conditions. Thus, it would be difficult to study NNpHSCC processes under the conditions close to those in the field. So an accelerating technique to generate pits was employed in this study, the details of which have been reported in another paper [15]. Basically it consists of an acid-immersion treatment to passivate the surface and then a second immersion in dilute hydrochloric acid, which leads to rapid pitting growth at sites where the passive layer is either removed with a needle or at innate weaknesses in the film where pits grow spontaneously.

After the transition from pits to cracks has occurred in the field, tiny, elongated blunt cracks, frequently in crack colonies, are often seen in very large numbers [16]. The vast majority of these small cracks are found to become dormant and hence tend to be innocuous. However, if the small cracks can surpass a threshold depth, around 0.5 to 0.6 mm [17], these cracks can be activated and begin to propagate and may eventually lead to pipeline rupture if not detected and removed. So studies concerning the growth of these small cracks and how potential growth can be identified and avoided will contribute significantly to an understanding of NNpHSCC initiation and help in pipeline integrity management.

## **2. EXPERIMENTAL**

### **2.1. SPECIMENTS**

X-52 line pipe steel from Enbridge Pipelines Inc. that had exhibited NNpHSCC crack colonies after thirty-four years of service was used in this project. The pipe was 34" (864 mm) in external diameter and 7.9 mm in measured wall thickness. The chemical composition can be seen in Table 1. The microstructure consisted of pearlite and ferrite. It was found that there were very few, if any, inclusions on the axial transverse (A-T) surface, while there were a lot of inclusions on the radial transverse (R-T) and the axial radial (A-R) surfaces [18]. In addition, the inclusions on the R-T surface were relatively smaller than those on the A-R surface. These non-metallic inclusions (MnS) were more abundant on the axial-radial section at mid-wall [18]. Hence, it was likely that the inclusions were elongated along the axial direction during the rolling processes.

Table 1. Chemical composition of X-52 pipeline steel (wt. %)

Elements	X-52	Elements	X-52
C (Carbon)	0.261	Cr (Chromium)	0.027
Mn (Manganese)	1.150	Mo (Molybdenum)	0.013
P (Phosphorus)	0.008	Al (Aluminum)	0.002
S (Sulfur)	0.019	V (Vanadium)	0.003
Si (Silicon)	0.036	Nb (Niobium)	0.003
Cu (Copper)	0.033	Ti (Titanium)	0.002
Sn (Tin)	0.002	Co (Cobalt)	0.006
Ni (Nickel)	0.032	Fe (Iron)	Balance

Dog-bone specimens with dimension of 36×7×3.5 mm in reduced section were machined from pipe with the length of the specimens along the transverse direction of the pipe so that the crack-advance plane (radial-axial) was the same as for NNpHSCC in the field (see Figure 1 for a schematic of the orientations). After the sample surfaces had been ground to 1200 grit in accordance with the standard metallographic procedure, they were degreased and cleaned using acetone and ethanol.

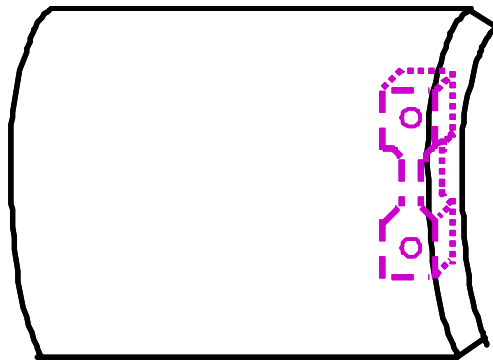


Figure 1. Schematic of the sample with orientations in the pipe.

## 2.2. PITTING THE SURFACES

After the surfaces were masked using PTFE tape leaving the gauge section exposed, the samples were put in concentrated H<sub>2</sub>SO<sub>4</sub> solution to passivate the surfaces and then exposed to dilute HCl solution (the ratio of HCl to distilled water was 1:10) to grow pits, as reported by *Fang et al. [15]*. After 135 hours, the pitted samples were taken out and washed using tap water. Then they were cleaned using EDTA and then ethanol in an ultrasonic cleaner to ensure that the passive films on the surfaces had been almost removed.

## 2.3. SCC TESTS

The pitted specimens were cyclically loaded under a balanced triangular waveform as shown in Table 2 in a soil solution (ratio of soil to water is 1:5) and a NNpH inorganic test solution identified as C2 solution, both sparged with 5% CO<sub>2</sub> /balance N<sub>2</sub> gas mixture. C2 solution is made with the following ingredients 0.0274 g/L MgSO<sub>4</sub>•7H<sub>2</sub>O, 0.0255 g/L CaCl<sub>2</sub>, 0.0035 g/L KCl, 0.0195 g/L NaHCO<sub>3</sub>, and 0.0606 g/L CaCO<sub>3</sub> dissolved in high purity water. The pH after sparging was close to 6.2 for the C2 solution and near 7.0 in the soil solution.

Table 2 Loading Conditions in NNpH Solutions (All were sparged with 5% CO<sub>2</sub>)

Condition	Peak Stress	Stress ratio	Frequency	No. of cycles
A	100% SMYS	0.8	$1 \times 10^{-4}$ Hz	1,345
B-1	100% SMYS	0.63	$7.6 \times 10^{-5}$ Hz	1,394
B-2	106% SMYS	0.63	$7.6 \times 10^{-5}$ Hz	1,394

After testing in the solution for the desired number of cycles, the tests were stopped and the samples were taken out. After cleaning using EDTA and ethanol in the ultrasonic cleaner, the samples were rinsed and dried with warm air and the sample surface observations were made using optical microscopy (OM) and scanning electron microscope (SEM). Thereafter, the sample surfaces were sectioned step by step to examine crack and pit depths, and crack and pit shapes. The sectioning was perpendicular to the cracked and pitted surfaces, and the sectioning progressed perpendicular to the loading direction. After each grinding and polishing step, measurements were made using OM and SEM to reveal the crack and pit dimensions.

### 3. RESULTS AND DISCUSSION

#### 3.1. BLUNT CRACK INITIATION AND CRACKS DISTRIBUTION

When pitted specimens were cyclically loaded in NNpH solutions, many blunt cracks were seen to initiate from the bottom of the pits (Figure 2). These pits acted as stress concentrators (micro-notches) and were the principal sites for crack initiation. The pits would locally raise the stresses near the pits. Thus, plastic deformation would occur locally, and there would be plastic zones near the corrosion pits. The iron atoms in the plastically deformed zones would have enhanced energy because of the increased dislocation density and increased stress, and would behave as anodes in the solutions relative to the less deformed and lower stressed nearby regions. Thus, stress cells would form leading to localized corrosion. So a preferential electrochemical attack occurred in the regions where plastic deformation was localized. Thus, dissolution in the plastic zones around the corrosion pits was accelerated. This, in turn, would further enhance the stress concentrations so that each cycle would create more deformed material to be removed by further corrosion. In the end, blunt cracks were initiated. These cracks were very wide, especially at the crack mouth, as shown in Figure 2. Hence, they were designated as blunt cracks or Stage I cracks. At the same time, in contrast to the fatigue cracks in air, the crack advance direction was perpendicular to the applied load direction.

After cyclic loading in NNpH environment, it was clearly seen that most of cracks were distributed between 50 and 450  $\mu\text{m}$  in depth, as shown in Figure 3a, and the most probable crack depth was around 150 to 250  $\mu\text{m}$ . Figure 3b shows that the median of crack depth was at 221  $\mu\text{m}$ , and the lower and upper box values (quartiles) were 156 and 351  $\mu\text{m}$ , respectively. The whisker was from 61 to 633  $\mu\text{m}$  with a few outliers shown with asterisks. All the evidence once again reflected that the majority of crack depths were below approximately 0.5 to 0.6mm, consistent with the field observations.

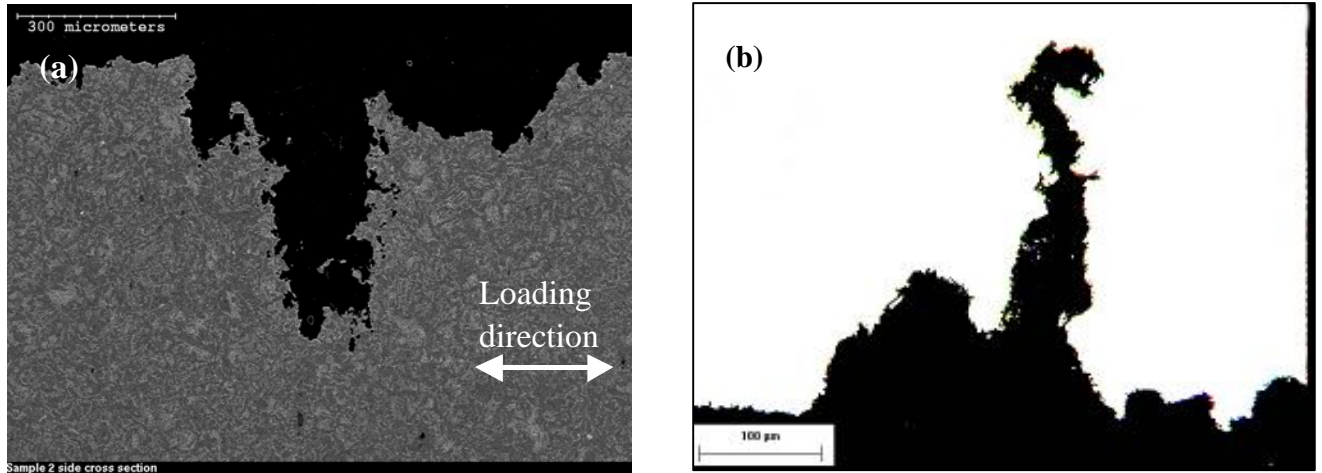


Figure 2. Photomicrographs of NNpHSCC crack cross-section features on the R-T surface cycled in NNpH solution sparged with 5% CO<sub>2</sub>/ balance N<sub>2</sub> gas mixture (a) in soil solution under load condition A [19]; (b) in C2 solution under load condition A [20].

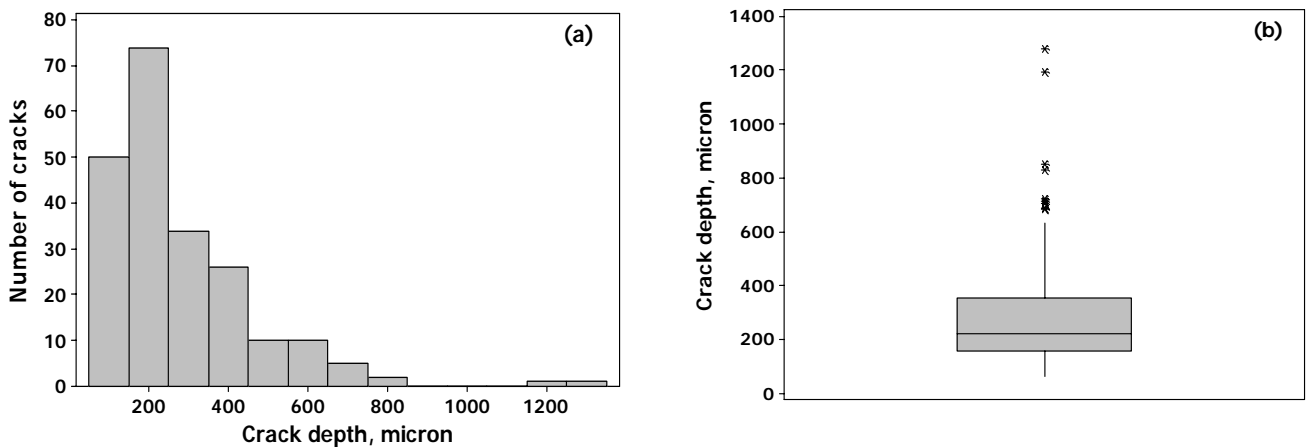


Figure 3. Distribution of crack depth on the sample cyclically loaded at condition B1 in C2 solution; (a) number of cracks versus crack depth; (b) box plot of crack depth.

### 3.2. BLUNT CRACK GROWTH

After blunt cracks had initiated, they might grow under benign conditions. However, the majority of the cracks would not grow to the critical depth size of 0.5 to 0.6 mm. Once the blunt crack growth rate was higher than the pit growth rate, the stress facilitated corrosion dissolution rate would be dominant and thus, dissolution would be localized and localized corrosion around the corrosion pits would be accelerated and blunt cracks would grow. In the end, dissolution formed blunt crack would survive and might grow under intermittent conditions.

It was sometimes seen that non-metallic inclusions (mainly MnS as revealed by EDS) were located ahead of the cracks on the crack advancement plane, as shown in Figure 4. When the blunt cracks grew and reached these non-metallic inclusions, the solution in the blunt crack enclaves could get to these inclusions and electrochemically react with the inclusions [5-11]. The inclusions would then

be corroded and eaten away since the non-metallic inclusions (MnS) acted as an anode and the base steel nearby acted a cathode. The relative area ratio of the base steel to the non-metallic inclusion was huge, thus, galvanic corrosion would be accelerated and corrosion attack would be localized on the inclusions, which, in return, increased stress concentrations and plastic deformation. Thus, blunt crack growth because of inclusions enhancing stress facilitated dissolution would be maintained, as reported previously [18]. When the inclusions were completely eaten away, the blunt crack would advance as stress facilitated dissolution reported previous. In addition, at this time, the stress concentration would be much higher, which might further enhance the stress riser and plastic deformation. All of these, in turn, would contribute to the blunt crack growth.

Still in Figure 4, there were few non-metallic inclusions ahead of the blunt crack tip and these aligned inclusions could contribute to significant crack growth.

Considering the microstructure differences on the different surfaces as reported in a previous paper [18], the nonmetallic inclusions were elongated along the longitudinal direction, and some inclusions were about 0.5 mm long, which would have facilitated pit nucleation, if the R-T surface was exposed to the NNpH solution, and promoted pit-to-crack transition and blunt crack growth. As the crack growth direction in depth was the same as the elongated inclusions, the latter would also have assisted crack growth, as clearly shown in Figure 5. So it was seen that the blunt cracks had grown easily when they encountered non-metallic inclusions.

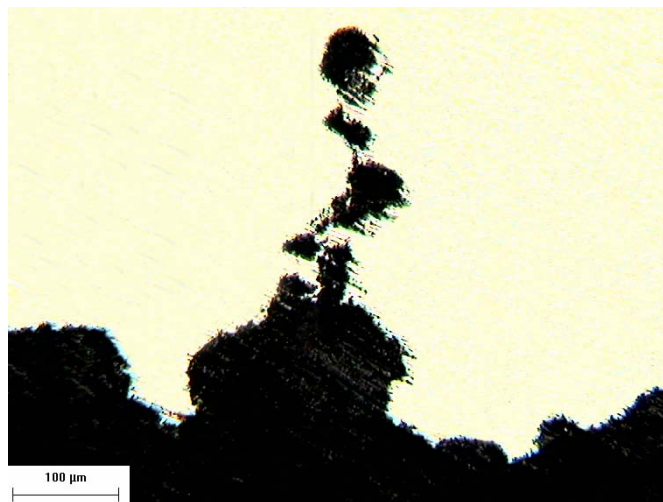


Figure 4. Photograph of a crack initiation from a corrosion pit and non-metallic inclusions located ahead of the crack tip.

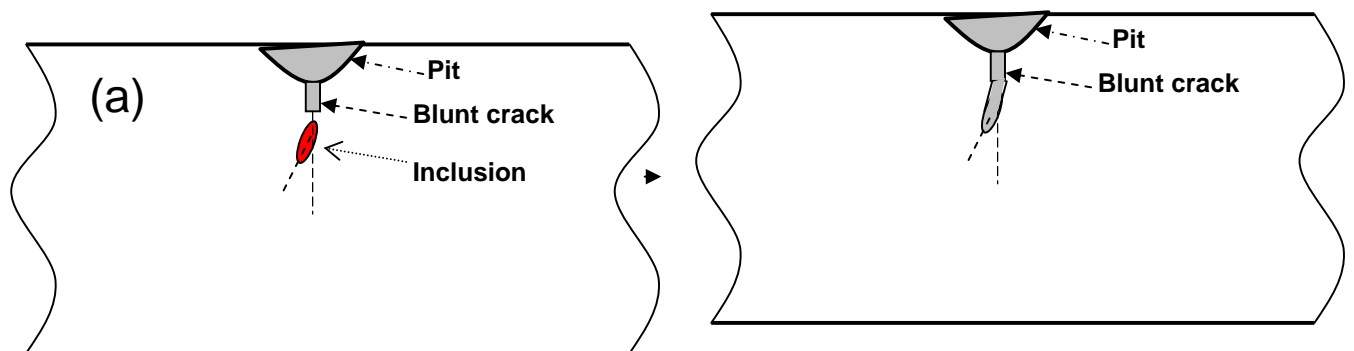
(a)



Figure 5. Photographs of; (a) Stage I crack emanating from a corrosion pit and blunt crack growth associated with inclusions for the sample in NNpH environment under Condition

### 3.3. ORIENTATION OF INCLUSIONS AND BLUNT CRACKS

As discussed in previous section, non-metallic inclusions significantly influenced blunt crack growth. However, how they affected blunt crack growth would need to be addressed further, especially the orientation of inclusions with regard to blunt cracks.



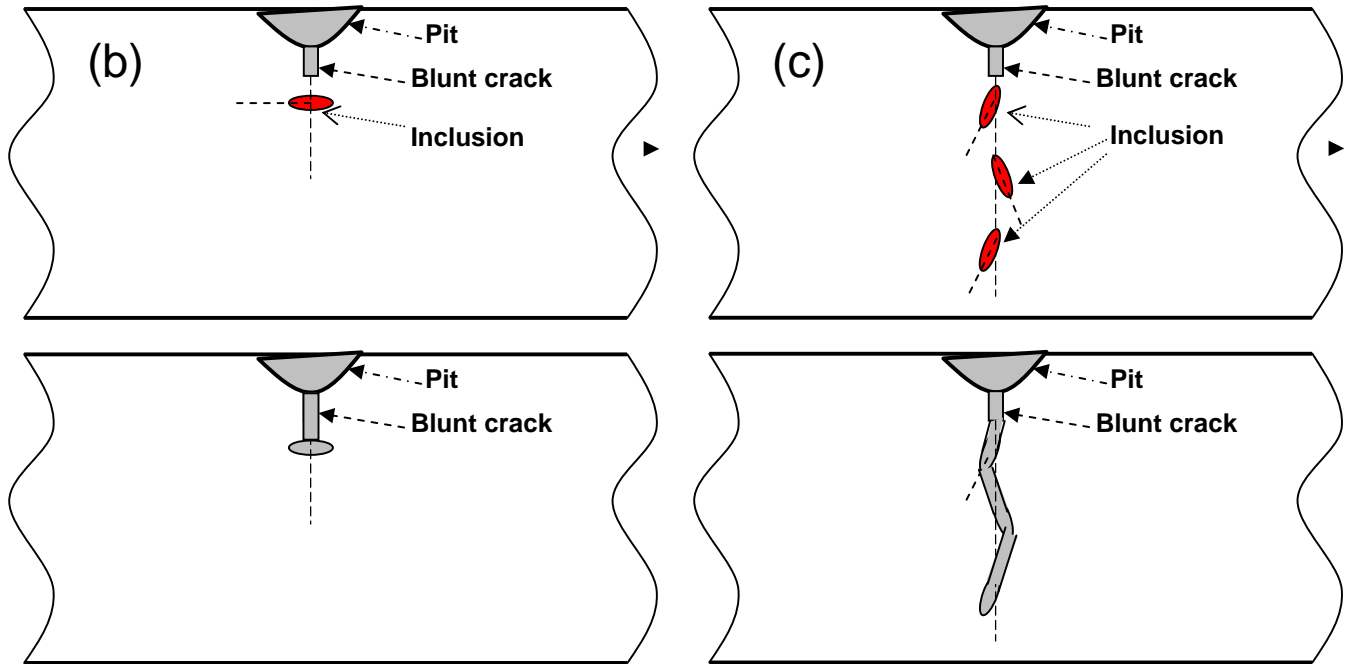


Figure 6. Schematic illustration on orientation of inclusions and blunt cracks.

When the inclusions were in the same plane as crack initiation and growth, generally, there were three types of the orientation of inclusions and blunt cracks, as shown in Figure 6.

1. The orientation of the inclusions was at a small acute angle to the orientation of the pit growth direction or blunt cracks (Figure 6a). The pits or blunt cracks would grow and encounter the inclusions, after which the blunt crack growth would be localized along the inclusions until the inclusions were eaten away completely due to accelerated galvanic corrosion. Thereafter, blunt cracks might keep growing, depending on driving forces (stress concentrations and stress intensity factors) and local environment at the crack tip.

2. The orientation of the inclusions was approximately perpendicular to the pit growth direction or blunt cracks, as shown in Figure 6b. In this case, the blunt cracks might grow and touch the inclusions under appropriate conditions. Also once the blunt cracks encountered the inclusions, the dissolution and growth would be localized on the inclusions. However, since the inclusions were approximately perpendicular to the blunt cracks, when the inclusions were corroded completely, the blunt crack tips would become much blunter, which therefore reduced stress concentrations and stress intensity factors, and hence lowered the crack growth driving forces. Thus, the cracks might stop growing and became dormant. This type of crack was also observed in this report, as shown in Figure 7 where a balloon-like morphology was seen. The cracks observed in this case might not threaten pipeline integrity from a management point of view.

3. A bunch of inclusions were aligned with the blunt cracks at small acute angles, as presented in Figure 6c. This case could be considered as repeated steps of the case 1. When the first inclusion was eaten away, the blunt crack growth might slow down and grow under intermittent conditions. However, once this blunt crack grew to the second inclusion, the latter would be dissolved by



accelerated galvanic corrosion. Thereafter, the accelerated growth and slow growth might be repeated again and again until a critical depth was exceeded when Stage II (sharp) cracks would have initiated and grew [20]. This case was also seen in this project, as shown in Figures 4 and 5, where the blunt cracks grew along the inclusions.

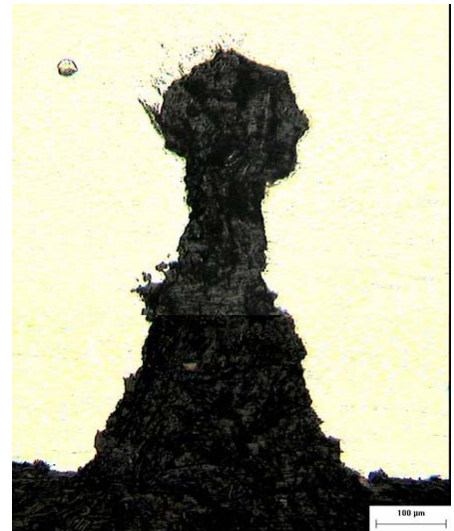


Figure 7. Photograph of a blunt crack and the blunt crack tip on R-T surface for a specimen cyclically loaded in C2 solution under condition B-1.

Since inclusions were elongated in the longitudinal direction, especially in the mid-wall, the crack growth in the longitudinal direction would be significantly influenced by the coalescence with inclusions. These cracks, if not detected and removed, might put a pipeline at a risk to cracking or failure. The case in Figure 6c was much more dangerous for pipeline integrity management. It would be helpful and important if some tools could be employed to monitor the existence of this type of crack and inclusion, and some measures could be taken to prevent the cracks from growing.

### 3.4. HYDROGEN EFFECT

In near-neutral pH environment, the metal in the crack tip is dissolved, assisted by stress concentration associated galvanic corrosion cells. Some of the generated hydrogen atoms outside the blunt crack enclaves, *i.e.*, specimen surfaces, and the crack walls by water reduction could enter and diffuse into the steel. The lattice (interstitial) hydrogen solubility was elevated ahead of stressed blunt cracks or pits, and these sites could accumulate higher hydrogen concentrations. The same hydrostatic stresses that increased the hydrogen solubility in the lattice also enhanced the amounts of hydrogen trapped in deep traps, such as large defects (inclusions and phase boundaries) ahead of the crack tips. When some nearby non-metallic inclusions ahead of the crack tips that were perpendicular to the applied stress had the orientation of a small acute angle to the orientation of the pits or the blunt cracks, and the nearby non-metallic inclusions ahead of the blunt cracks or pits were on the same plane as the pits or the cracks, the trapped hydrogen on these nearby non-metallic inclusions might weaken the bond strength. The interactions between the trapped hydrogen and the inclusions might occur at some regions on the inclusions in advance of the crack tips where the stress and / or strain conditions were particularly appropriate for the nucleation of clusters of tiny cracks, as

shown in Figure 8. The elongated inclusions not only influenced pit nucleation and pit growth [18], but also affected pit-to-crack

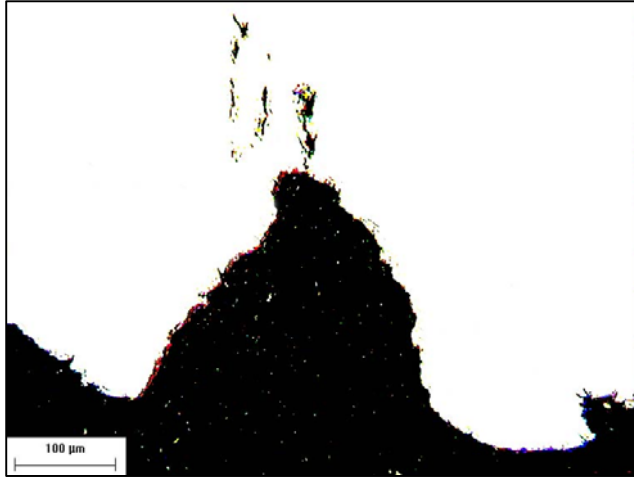


Figure 8. Tiny cracks in the inclusion ahead of the blunt crack.

transition and crack propagation, if the elongated inclusions were parallel to crack growth direction. However, if the inclusions were almost hemispherical, they would only affect the pit formation and growth, even tiny hydrogen induced cracks but these were very rare. If input fugacity of hydrogen was high enough, recombination at internal sites such as non-metallic inclusions would be prevailing, causing blistering or cracking (hydrogen-induced cracking), but this case could not happen in near-neutral pH condition. The hydrogen that diffused into the steel could also be trapped on some phase interfaces ahead of the blunt cracks, such as Figure 9, and caused tiny hydrogen related cracks to initiate. These hydrogen trapped phase interfaces were also perpendicular to the external stress applied. These hydrogen induced cracks were also rare, but they contributed to the blunt crack growth.

Under appropriate conditions, the blunt cracks or pits would continue to grow and link to the nucleated hydrogen induced tiny cracks. In the end, these hydrogen produced clusters of tiny cracks would have been eaten away by the further growth of the mother blunt cracks or pits by further strain deformation facilitated dissolution. Therefore, the overall apparent crack growth process seemed that there was only stress assisted dissolution growth, engendered by galvanic stress corrosion cells, which concealed hydrogen related tiny crack initiation, although the latter was rare and only could occur under very stringent conditions.

It could be considered that the blunt crack growth was associated with the bursts of dissolution of aligned non-metallic inclusions with the same orientation as the blunt cracks, and rare hydrogen induced tiny cracks while the rate-controlling periods seemed “non-propagation” which were concerned with the stress facilitated dissolution to form blunt cracks again that established the conditions for further crack bursts, probably because of dissolution of further non-metallic inclusions or even more rare hydrogen caused clusters of tiny cracks, or severe stress facilitated dissolution with higher stress concentrations. The crucial role of stress or strain was continued, resulting in much further dissolution, and when the total blunt cracks reached a critical size, approximately around 0.5 to 0.6 mm deep, the stress integrity factors could be sufficient, and the plastic zones ahead of the blunt

cracks tips were large enough to trap more hydrogen, leading to hydrogen related growth. At this time, the crack shape became sharp [18], and was significantly different from Stage I cracks. Thus, the cracks in this period were named Stage II cracks. Stage II cracks would grow intermittently, and in the end, they could surpass the critical threshold for long cracks, after which the cracks would grow in a typical corrosion fatigue model. This implied that for pipeline steel in near-neutral pH environment, these different mechanisms of stress corrosion cracking could have occurred within a continuous spectrum, with a gradual transition from one to another as the dominance of corrosive processes was replaced by stress or strain, led readily to the notion that steel microstructure, electrochemistry and stress or strain might interact in a variety of ways, and that the transformation from one mechanism to another might result from complex relation between stress risers, dissolution driven by stress concentrations, inclusions, and trapped hydrogen.

This is the first time that different crack types and mechanisms have been observed and reproduced in the laboratory. These different mechanisms are necessary for NNpHSCC crack initiation and early growth. These are quite consistent with the observations in the field. The sharp cracks in Stage II can create a significant risk to pipeline integrity. If they can't be detected and removed, they will likely keep growing and eventually once the long crack corrosion fatigue threshold is exceeded, cracks will grow much faster, even leading to pipeline rupture or leakage. Compared to the long crack growth stage, the blunt crack growth in Stage I and the sharp crack growth in Stage II consume a majority of pipeline life. So Stages I and II cracks are very important for pipeline integrity management and should be addressed intensively. Unfortunately, so far no research has touched Stage I blunt cracks and little has paid attention to Stage II sharp cracks, while most has focused on long crack growth.

For in-service pipelines, removing Stage II cracks that are deeper than 0.5 to 0.6 mm would benefit the pipeline integrity and enhance the remaining life. If Stage I crack growth can be impaired, that would significantly extend the remaining life. If active corrosion can be prevented such as by cathodic protection then smaller cracks are innocuous.

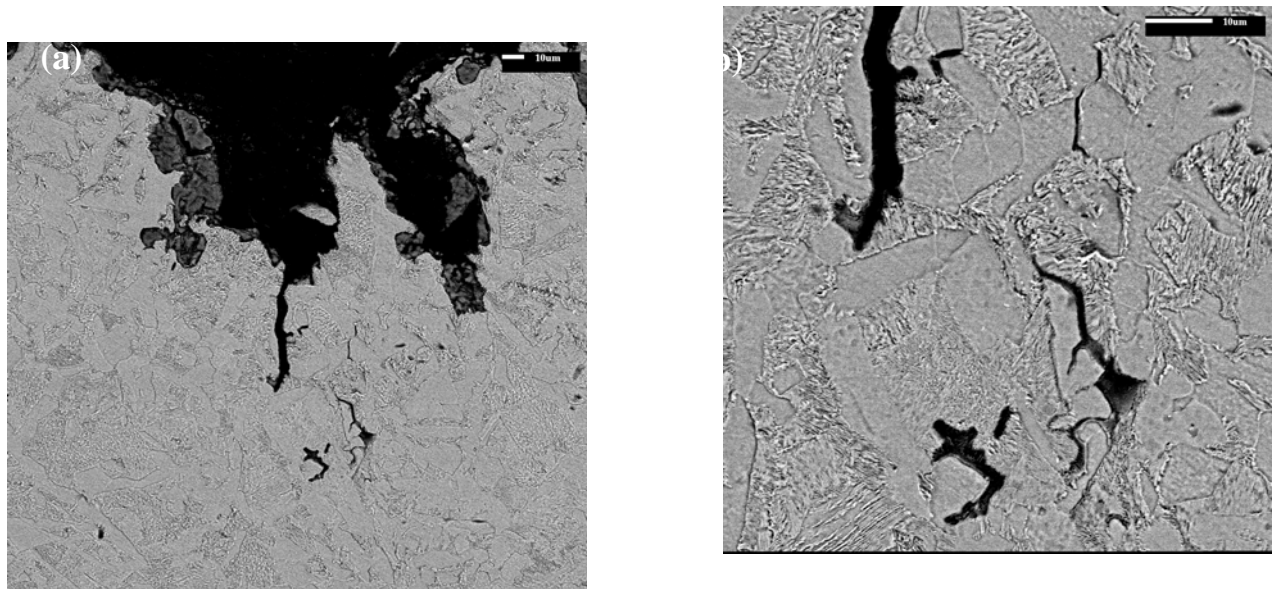


Figure 9. Tiny hydrogen produced cracks along the phase boundary between pearlite and ferrite ahead of the main crack.

## 4. CONCLUSIONS

Based on the results and discussion above, the following conclusions were made.

1. Blunt cracks were seen to have initiated from the corrosion pits on the pitted samples cyclically loaded in near-neutral pH environment and these blunt cracks were generally less than 0.5 to 0.6 mm deep, consistent with the observations from the field. These blunt cracks were transgranular and believed not to put pipelines at a risk from integrity management point of view.
2. The distribution of the nearby non-metallic inclusions significantly influenced the blunt crack growth by enhancing the stress-facilitated dissolution of the blunt cracks.
3. The orientation of the nearby non-metallic inclusions with regard to the blunt cracks was crucial for the blunt cracks to continue growing or keep dormant. Only when the orientation of the nearby inclusions was at a small acute angle to that of the pits or cracks, and the inclusions were in the same plane as crack initiation or extension, these inclusions would contribute to further crack growth. Thereafter, the inclusions would be eaten away by further growth of the blunt cracks.
4. When the inclusions were approximately perpendicular to the blunt crack growth, the cracks would become blunter and dormant.
5. The hydrogen trapped in the nearby inclusions and phase interface would cause clusters of tiny cracks to form, which appeared to be produced by hydrogen. These hydrogen generated cracks could be eaten away later by the stress-facilitated further dissolution of the blunt cracks.
6. When the blunt cracks reached a critical size, *i.e.*, 0.5 to 0.6 mm deep, sharp cracks would be formed, engendered by hydrogen.
7. Different mechanisms could occur in pipeline steel in near-neutral pH environment. The transformation from one mechanism to another might result from complex relation between stress risers, dissolution driven by stress concentrations, inclusions, and trapped hydrogen

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