Fatigue Crack Growth Behaviour in Pipeline Steels under Mean Load Pressure Fluctuation in Near-Neutral pH Environment

by

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Abstract

This research aimed to understand the effect of mean load pressure fluctuations on crack growth in near-neutral pH (NNpH) environment. This work contributes to a larger effort to develop a crack growth predictive model based on variable amplitude loading. The motivation for this study is that although pressure fluctuations have been recognized as an important factor in crack propagation in NNpH environments, current predictive models for crack growth are still based on constant amplitude loading. The observed discrepancies between the predicted life of cracks based on such models and the fatigue life observed in the operating pipelines call for a review of such models. It is believed that a predictive model based on realistic pressure fluctuations will provide a more accurate representation of the fatigue lives of cracks in pipelines.

Pressure fluctuations can be divided into underload, mean load and overload cycles. The effect of mean load pressure fluctuation on crack growth in NNpH environment has not been studied previously. Although there is agreement that mean load is less severe compared to underload cycles in terms of crack propagation, it appears that there is disagreement on the effect of load sequence in the mean load on crack propagation. Literature review shows that while some researchers observed retardation of crack growth when Type II mean load cycles (underload+overload+minor cycles, UL+OL+MC) was applied, some other researchers disagree and reported that retardation of crack growth was observed under Type I mean load (overload+undeload+minor cycles, OL+UL+MC).

This research focuses on mean load pressure fluctuation and its effect on crack propagation in the NNpH environment. By studying crack growth behaviour in both Type I and Type II mean load pressure fluctuations, the effect of load sequence in the mean load is highlighted. This study also considers the effect of sensitivity of both Type I and Type II mean loads to increase in the magnitude of overload. The effect of increase in the number of minor cycles was also studied in both mean load types. Crack growth behaviour under mean load pressure fluctuations was compared to constant amplitude cyclic loading as well as underload+minor cycles. The results showed that in the NNpH environment, crack growth is higher under Type II loading than Type I. Type II loading crack growth rate is very sensitive to increase in the magnitude of overload in the mean load compared to Type I. This suggests that Type II mean load could be detrimental to pipeline integrity, similar to underload pressure fluctuations. This is interesting because many cracks and failures have been reported in areas close to the discharge part of the pipe where underload and mean load pressure fluctuation are observed. Overall, the best retardation of crack growth rate was observed in both Type I and Type II mean load with 5% overload. Based on these findings, suggestions were made on actions that can mitigate crack propagation during pipeline operations due to planned or unplanned shutdowns.

Preface

This thesis is an original work by Olayinka Tehinse. Initial findings in Chapter 4 were published in a peer-reviewed conference paper "Tehinse, O., Chen, W., Been, J., Chevil, K., Keane, S., and Van Boven, G. "Application of Load Sequence to Control Crack Growth in Steel Pipelines Under Near Neutral pH SCC." *Proceedings of the 2016 11th International Pipeline Conference*. *Volume 1: Pipelines and Facilities Integrity*. Calgary, Alberta, Canada. September 26–30, 2016.". Chapters 3 and 4 have been combined as a full paper and accepted for publication as "O. Tehinse, L. Lamborn, K. Chevil, E. Gamboa, W. Chen, Influence of load interaction and hydrogen on fatigue crack growth behaviour in steel pipelines under mean load pressure fluctuations. *Fatigue Fract Eng Mater Struct*. 2021; 44: 1073– 1084. O. Tehinse and W. Chen designed and conducted the experiments. L. Lamborn, K. Chevil, and E. Gamboa con- tributed to the data interpretation. O. Tehinse prepared the manuscript, and all authors contributed to the review of the manuscript.

Chapter 5 has been published in a peer-reviewed conference paper as "Tehinse, O., Chen, W., Chevil, K., Gamboa, E., & Lamborn, L. "Influence of Mean Load Pressure Fluctuations on Crack Growth Behaviour in Steel Pipelines." *Proceedings of the 2018 12th International Pipeline Conference*. *Volume 1: Pipeline and Facilities Integrity*. Calgary, Alberta, Canada. September 24–28, 2018".

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Chapter 1: Introduction

1.1 Introduction

The history of pipelines for oil and gas applications dates to the 1860s. In those early years, labour disputes and high transportation costs were the main drivers for improving pipeline technology. From barrels to wooden boards and later steel pipelines, there has been a lot of improvement in the materials, design and construction of pipelines. [1] Today, pipelines provide a safe, reliable, economic and energy-efficient means of transporting oil and gas products to consumers worldwide. Pipeline application for oil and gas products is important to meet the increasing energy demands for everyday life. At present, about 100 million barrels of oil is consumed daily worldwide. Although there is an increase in demand for alternative energy sources such as renewable energy, oil and gas products are still important to meet the increasing energy demands due to population increase and technological development worldwide. [2, 3]

The manufacturing, installation, operation and maintenance of pipelines are regulated by standards; and regulatory bodies ensure that pipeline companies follow the standards. This is because failure of pipelines poses a threat to pipeline applications. Although they are rare occurrences, pipeline failures can have a huge impact, sometimes causing loss of lives, endangering the ecosystem and environment. Pipeline failures can also have a negative impact on the economy and the company image. Hence in recent times, there has been an increase in concern for the safety of pipelines. Among other causes, stress corrosion cracking (SCC) is a common threat to pipeline operation. Near-neutral pH (NNpH) SCC as a form of pipe degradation is initiated by the breakdown of protective coatings on the pipeline surface, thereby allowing the pipeline steel to be in contact with the soil solution. The disbonded coating and soil resistivity may cause resistance to cathodic protection. This creates a localized anaerobic region that favours crack propagation where there is insufficient or no cathodic protection. [4]

The life of a crack under NNpH SCC can be classified into three stages. Stage I is the crack initiation and early crack growth stage. The crack growth behaviour of a crack in stage I is highly influenced by environmental factors. In stage II, the conditions for crack propagation are available and crack growth is influenced by the mechanical loading condition and the influence of hydrogen. Stage II crack growth is very important for engineering applications and life predictions to ensure the structural integrity of pipelines. In stage III of crack growth, crack growth occurs as a fast fracture causing rupture of the pipe. This stage has little or no engineering applications except that a clean-up will be needed. [4]

Crack growth in stage II is influenced by mechanical loading conditions. Therefore, understanding the role of pressure fluctuations is vital to understand crack growth behaviour in NNpH environment. This will be useful in calculating and predicting the remaining life of crack features in pipeline steel. There has been a lot of research effort on understanding the effect of pressure fluctuations on pipeline steel. However, many of them have focused on constant amplitude cyclic loading. Experience has shown that it is difficult to correlate the fatigue life based on such predictive models to the remaining life of pipes in the field. Hence there is a need to apply realistic loading conditions that simulate pressure fluctuations in the field to understand the effect of pressure fluctuations on crack propagation.

In most structural applications such as aircraft and bridges, realistic loading conditions are applied to understand crack growth behaviour and predict crack growth rates. Some of such loading conditions might be similar to pipelines, but the environment and low frequency of pressure fluctuations in steel pipelines create a unique situation that should be investigated and treated. Realistic loading conditions can be divided into three variable amplitude loading waveforms. These are underload, mean load and overload pressure fluctuations. In structural materials, underload is identified for having an acceleration effect on crack growth. Overload is well known for having a retardation effect on crack growth. Mean load is a combination of underload and overload and it is believed to achieve some retardation of crack growth compared with underload.

Recent work on the effect of variable amplitude loading on crack propagation in pipeline steel was carried out by Yu *et al*. based on underload pressure fluctuation. The main finding in that research work confirmed the acceleration effect of underload on crack growth in pipeline steels under NNpH environment. Yu's research also analyzed the role of minor cycles on crack growth behaviour in NNpH environment. Minor cycles, also known as ripple loads, were previously regarded as loading cycles not contributing to crack propagation and hence not considered in crack growth analysis for pipeline steels. There is a need to develop an understanding of other pressure fluctuations such as mean load and overload cycles. Generally, overload pressure fluctuations are observed at the suction part of the pipeline and studies have shown that most pipeline failures are observed in the discharge part of the pipeline. Although underload has been related to accelerated crack growth and failures recorded in the discharge part of the pipe, it is important to know that mean load pressure fluctuations can also be observed at the discharge part of the pipe. Hence it is important to understand crack growth behaviour under underload and mean load pressure fluctuations. [5, 6]

Given that lots of research have been carried out on constant amplitude loading, some experimental studies were conducted to establish crack growth behaviour under constant amplitude loading considering the loading conditions applied in this study. For comparison, a test was conducted using the underload loading condition. The effect of mean load pressure fluctuation was divided into two. The first part was focused on underload+overload+minor cycles (UL+OL+MC, Type II mean load). The other part of the effect of mean load pressure fluctuation on crack growth behaviour was focused on overload+underload+minor cycles (OL+UL+MC, Type I mean load). Since minor cycles have been suspected to influence crack growth behaviour in variable amplitude loading, the effect of minor cycles on crack growth in both Type I and Type II mean load have also been considered in this research. To highlight the effect of environment on crack growth behaviour, selected tests were also conducted in air. Understanding crack growth behaviour under mean load pressure fluctuations[1] can guide integrity decisions such as shutdowns or depressurization.

1.2 Thesis Outline

This thesis is based on understanding of the effect of mean load on crack growth in NNpH environment.

Chapter 2 provides background information on NNpH crack growth as a threat to pipeline integrity. The chapter will also provide a literature review to show that NNpH cracking can be related to the corrosion fatigue mechanism.

Chapter 3 of the thesis shows the effect of constant amplitude cyclic loading on crack growth behaviour in NNpH environment and air. Also, the mechanisms driving crack propagation in minor cycles are discussed.

Chapter 4 of the thesis discusses the effect of Type II mean load on crack growth behaviour in NNpH solutions. This involves applying different magnitudes of overload in the mean load to determine the sensitivity of Type II mean load to crack propagation. Also, the effect of the number of minor cycles on crack growth in Type II mean load are discussed.

[1] In this study, mean load refers to the pressure fluctuation that involves both pressure drops and pressure increases without a predominate occurrence. It does not correspond to the average of maximum and minimum stress.

Chapter 5 of the thesis discusses the effect of Type I mean load on crack growth behaviour in NNpH. This involves applying different magnitudes of overload in the mean load to determine the sensitivity of Type I mean load to crack propagation. Also, the effect of the number of minor cycles on crack growth in Type I mean the load is discussed.

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Chapter 2: Literature Review

2.1 Stress Corrosion Cracking -A Threat to Pipeline Integrity

Pipeline failure occurs when the integrity of the system is compromised. This is caused by several factors. Figure 2-1 shows the statistics of pipeline failure collected by the Canadian Energy Pipeline Association (CEPA) between 2014-2018. [1] Figure 2-1 also shows that geotechnical factors such as earthquakes and landslides can cause pipeline failures. Metal loss occurs when internal and external corrosion causes reduction of the pipe wall thickness. External interference such as digging, ground works and piling can influence pipeline failure. Material, manufacturing and construction processes can also cause pipeline failure through defects developed during the manufacturing and construction. As shown in Figure 2-1, cracking is one of the leading causes of pipeline failures. A unique type of cracking is often referred to as Stress Corrosion Cracking. Stress Corrosion Cracking (SCC) is an environmentally assisted cracking that is usually accompanied by a slow propagation of crack on an engineering component due to the synergistic effect of tensile stress, corrosive environment on a susceptible material.

An example of pipeline failure caused by SCC is the failure of Line 100-2 operated by TransCanada Pipeline Limited (now TC Energy) on February 19, 2011. [2] The failed pipeline was laid in 1972. It had a wall thickness of 9.13 mm, an outside diameter of 914.4 mm and double-submerged arc-welding (DSAW). At the time of failure, Line 100-2 was transporting sweet gas. Also, Line 100-1 and Line 100-3 were parallel lines connected to Line 100-2. The pipeline's failure caused gas to escape and the ignition of the gas caused an explosion immediately. Three pipe sections broke and were ejected 100m from the rupture site. Figure 2-2 shows the site of failure of the pipeline.

Failure analysis of the failed pipe showed no evidence of internal corrosion, pitting or general corrosion on the external surface close to the rupture location. However, several colonies of SCC were distributed randomly over the surface of the upstream joint. The failure was attributed to longitudinal stress corrosion cracks. The event described here shows the severe impact of SCC and pipeline failure on life, environment and property. Hence, there has been a lot of effort to understand the mechanism of SCC in pipeline steels.

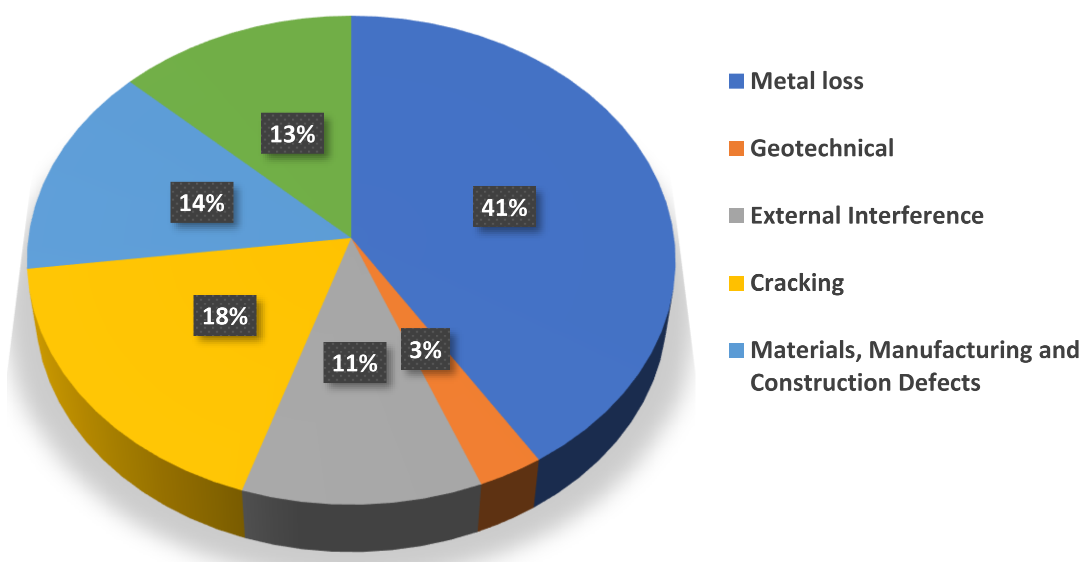

Figure 2 - 1 Causes of pipeline failure based on statistics of pipeline failure collected by CEPA between 2014-2018. [1]

Figure 2 - 2 Failed section of Line 100-2 showing the catastrophic effect of SCC. [2] Reproduced with permission from Transport Safety Board of Canada.

2.2 SCC in Pipeline Steels

SCC is characterized as a form of environmentally-assisted-cracking (EAC) that involves the synergistic effect of a corrosive environment and applied tensile stress on a susceptible material. For SCC to occur, mechanical loading and chemical reactions that favour cracking must be combined simultaneously on a susceptible material. [3] This implies that for a material to be susceptible to SCC, the composition, the microstructure of the material, and pipeline steel processing play an important role in SCC initiation. The tensile stress required for SCC can be either applied stress or residual stress due to manufacturing processes such as welding. Usually, this stress is below the macroscopic yield strength of the material but it must be above the SCC threshold. [3] The environment required for SCC is usually aqueous available due to the service or operating condition. Usually, SCC cracks show little or no evidence of external corrosion but have a drastic effect on the integrity and pressure carrying capacity of the pipeline system. As shown in Figure 2-3, the cracks are usually in colonies and under favourable conditions, the small cracks propagate and coalesce to form large cracks that can cause rupture incidents, such as described above in the case of Line 100.

Figure 2 - 3 Stress corrosion crack colony on buried pipeline steel.

Depending on the type of environment at the location of the crack on the pipe surface, SCC in pipeline steel can be classified into High pH SCC or Near Neutral pH SCC. [4]

2.2.1 High pH SCC

High pH SCC was first discovered in the 1960s in the United States. High pH SCC also known as classical SCC, was identified as the cause of failure of a gas transmission pipeline in Louisiana in 1965. Failure analysis of the failed pipe section showed that the rupture was initiated at crack colonies observed on the pipe surface. These findings facilitated research into SCC and motivated the United States government to enforce pipeline safety regulations. Studies showed that high pH SCC cracks are usually intergranular and the orientation of cracks is usually perpendicular to the stress direction. Early field investigations of SCC crack colonies in failed pipes showed that the environment under the disbonded pipe coating in the crack area had concentrated carbonate-bicarbonate solution with traces of nitrates. The chemistry of the trapped solution can be attributed to carbon dioxide gas (formed from the decay of organic matter) in the soil and the application of cathodic protection to the pipe. [5] Application of cathodic protection current influences increase in the pH under disbonded coating such that the localized region favours the dissolution of CO_2 and create a high concentration solution in a pH range 9-10. [6]

2.2.2 Near Neutral pH SCC

Near-neutral pH (NNpH) SCC was first documented in Canada as a unique form of external pipeline stress corrosion cracking in 1986. Since then, NNpH SCC has been identified as an integrity threat to pipeline operations in many countries. [3] Near-neutral pH SCC is characterised by transgranular cracks that are perpendicular to the stress direction. Also, NNpH SCC is associated with soils with alternating wet-dry seasons and conditions that promote disbondment or damage to coatings. The environment under the disbonded coating at the crack location usually contains a dilute

HCO_3^- solution in the pH range of 5.5-7.5. The chemistry of the trapped solution in near-neutral pH can be attributed to CO_2 dissolved from decayed organic matter in the soil. A near-neutral pH environment is favoured by lack of or inadequate supply of cathodic protection due to defective cathodic protection current supply to the localized region, high soil resistivity or shielding from the disbonded coating. [5] The cracking occurs at -760 and -790 mV (Cu/CuSO4). Although similar in physical appearance, there are significant differences between Near neutral and High pH SCC. [5] Crack morphology in near-neutral pH SCC shows wide transgranular cracks and the crack walls are usually corroded because there is no passivation in the near-neutral pH range, as shown in Figure 2-4. High pH SCC usually shows narrow intergranular cracks with a black oxide layer due to passivation. [5-8]

It has been identified that in NNpH SCC, cracks or failure distribution varies across the length of the pipeline as follows. [5]

- 65% of failures: compressor to the first downstream valve
- 12% of failure: first to the second valve
- 5% of failure: second to the third valve
- 18% of failure: downstream of the third valve

This suggests that cracking in pipeline steel is influenced by pressure fluctuations that are peculiar to the discharge regions of the pipe.

Figure 2 - 4 Transgranular crack growth in pipeline steel under NNpH SCC. [9]

2.3 Pipeline Integrity Management

Pipeline Integrity Management is a proactive approach to manage the lifecycle of a pipe from cradle to grave to understand and operate pipelines efficiently and safely. Pipeline integrity management prevents and avoids unexpected catastrophic failures by assessing, mitigating and monitoring the propagation of threats such as cracks, dents and corrosion features. This enables the pipeline operator to verify the fitness for service capabilities of the pipe. As stated earlier, pipelines are constructed, installed and operated to promote integrity during operation. This requires operations such as hydrostatic testing, in-line inspection and direct assessment. [10]

The first line of defence or protection for pipelines is coating and cathodic protection. Pipeline coatings are applied before installation to protect the pipe from corrosive species in the soil environment. The performance of a coating is dependent on being an electrical insulator, low water permeability, good adhesion to the pipe, good cohesive strength, good damage resistance and non-shielding to cathodic protection if disbonded from the pipe. [11-13] Previously, polyethylene tape coatings were used for pipelines, but studies have shown that this coating type provides conditions that favour SCC initiation. Today, Fusion Bonded Epoxy (FBE) is more commonly used due to its strong adhesive properties and better SCC resistance. [14]

Cathodic Protection (CP) is applied to alter the electrochemical reactions associated with corrosion of pipeline in case of contact with the soil environment due to disbonded coating. The supply of negative potential to the pipeline makes it cathodic and prevents corrosion on the pipe surface. In cases where the disbonded coating shields the pipe from CP current or due to high soil resistivity, there will be an absence of CP under the disbonded coating. This situation creates a suitable environment for NNpH SCC. [15-17]

2.4 Factors affecting Near Neutral pH SCC in Steel Pipelines

2.4.1 Effect of pipe materials

Material properties are influenced by factors like composition, microstructure and processing. Steel pipes of different grades have shown susceptibility to NNpH SCC. [18] Non-uniformity of microstructure has been suggested as an influencing factor for NNpH susceptibility. [18-19] Notched samples with homogenous bainite or bainitic-ferrite microstructure under cyclic loading in NS4 solution showed better resistance to SCC compared to those with a non-homogenous/non-uniform ferrite-pearlite microstructure. [19] According to Chu *et al.*, X-65 steel in NNpH environment showed preferential dissolution along planes on banded structures. [20]. It was suggested that galvanic coupling between banded structures and the steel matrix caused the selected dissolution. This implies that non-homogenous microstructure and metallurgical discontinuities can set up a galvanic cell and favour crack initiation and propagation due to selective dissolution at the interface between the discontinuity and the adjacent matrix.

Production processes such as rolling and welding can also influence the susceptibility of steel pipes to NNpH SCC. The rolling of flat steel plates involves about 70% strain at the final stage. Such high strain generates high residual stress in the steel pipe. Beavers *et al.* showed that SCC colonies were observed on steel pipes in regions with higher residual stress. [21] An increase in crack growth rate or susceptibility to SCC has been observed in the Heat Affected Zones (HAZ) of steel pipes compared to the base metal. [22, 23] High residual stress and metallurgical discontinuities in the HAZ increases susceptibility to NNpH SCC when there is formation of a holiday due to the breakdown of the coating at the long seam weld toe. For instance, in Double Submerged Arc Welded (DSAW) seam pipes, the weld cap geometry creates high stress concentration at the weld toe and this can be related to the high number of reported cracks at the seam weld toe. [24] Also, Electric Resistance Welded (ERW) pipes have crack features such as lack of fusion which can affect pipe integrity. [25, 26]

2.4.2 Effect of environment

The environmental conditions at the localised vicinity of the crack surface affect its susceptibility to NNpH SCC. Most NNpH cracks are found in dilute solutions with bicarbonate, carbonate, sulphate and chloride ions. [27] This requires the presence of CO_2 dissolved in dilute ground water. About 4-23% CO_2 have been found in soils in SCC vicinity [14]. The solution is maintained at the NNpH range (pH = 5.5-7.5) due to a generous supply of CO_2. [9] The lack of cathodic protection at the localised region under the disbonded coating keeps the environment at -760 to -790mV ($Cu/CuSO_4$) in open circuit potential. [14] These conditions can also be promoted by varying soil moisture levels, anaerobic conditions and high soil resistivity.

The following reactions explain the effect of CO_2 on crack propagation in NNpH environment:

$$\mathrm{CO_{2(g)}} \leftrightarrow \mathrm{CO_{2(aq)}} \tag{1}$$

$$\mathrm{CO_{2(aq)}} + \mathrm{H_2O_{(l)}} \leftrightarrow \mathrm{H_2CO_{3(aq)}} \tag{2}$$

$$\mathrm{H_2CO_{3(aq)}} \leftrightarrow H^+{}_{\mathrm{(aq)}} + \mathrm{HCO_3}^-{}_{\mathrm{(aq)}} \tag{3}$$

$$Fe_{(s)} \rightarrow Fe^{2+}{}_{(aq)} + 2\mathrm{e}^- \tag{4}$$

$$\mathrm{HCO_3}^-{}_{\mathrm{(aq)}} + Fe^{2+} \rightarrow FeCO_3 + H^+ \tag{5}$$

$$2\mathrm{H}^+ + 2\mathrm{e}^- \rightarrow 2\mathrm{H_{(ads)}} \tag{6}$$

As shown in the equations above, the dissolution of CO_2 in water enhances crack propagation in an anaerobic environment by creating a corrosive environment and generating hydrogen atoms that can diffuse into the steel and cause hydrogen embrittlement.

2.4.3 Effect of stress

The pressure-bearing capabilities of a pipeline are strongly dependent on the stress due to the internal pressure of the product passing through the pipe. Figure 2-5 shows that internal pressure in steel pipes generates circumferential (hoop) stress and axial (longitudinal) stress. Circumferential stress in the pipe generates axial cracks, while longitudinal stress generates circumferential cracks. Hoop stress is a more significant source of stress (twice) in pipelines compared to longitudinal stress. [25] The significance of hoop stress is supported by findings that 73% of Canada's pipeline failures are related to axial cracks. [14] Apart from internal pressure in the steel pipe, other hoop stress sources are thermal stress, residual stress, localized stress at stress raisers, secondary stress from land slides and earth movements. [14]

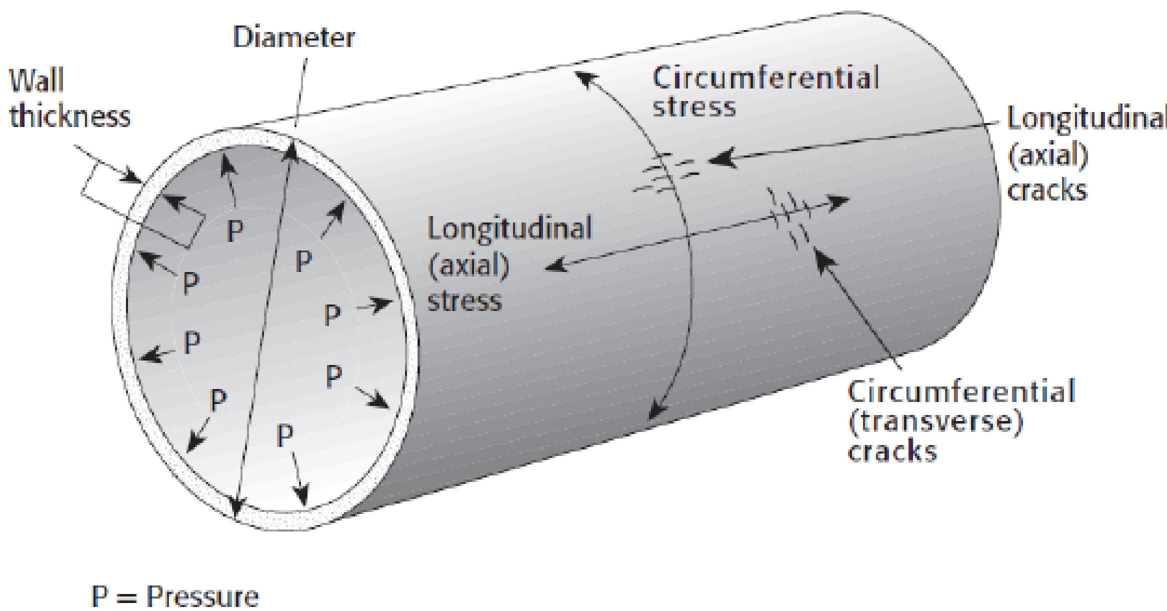

Figure 2 - 5 Stresses in pipeline steel causing axial and circumferential crack growth. [14] Reproduced with permission from Canada Energy Regulator.

2.5 Fatigue crack growth mechanism

Plastic deformation of materials requires dislocation motion causing the breakage of atomic bonds. A high level of force is required to break atomic bonds in a crystal plane but at lower stress, dislocation motion allows slipping of atoms in crystal planes. Such slipping motion is favoured on atomic planes with a high density of atoms within the grain. Hence slip will occur along parallel planes in the grain of the material. A group of parallel closely spaced slip planes are referred to as slip bands. Generally, the initiation of fatigue cracks occurs at the material surface. Under cyclic loading, dislocation motion at stress concentrations on the material surface creates Persistent Slip Bands (PSB). PSBs have extrusion areas (where materials rise above the surface) and regions of intrusion (where materials fall below the surface). At the initiation stage (stage I) of the fatigue crack, the steps created by intrusion and extrusion in PSB become a stress raiser and nucleates microcracks along slip plane with high shear stress (usually 45º to the loading direction). [28-30] The crack size of the initiated microcracks is usually measured as grain size.

Propagation of fatigue crack involves macroscopic crack growth in a direction perpendicular to the applied stress. Formation of striations on the crack surface in the direction perpendicular to applied stress is a major characteristic of stage II crack growth. In stage III, continuous application of cyclic loading will cause some cracks to coalesce and the crack propagates until the remaining material does not have the load-bearing capacity of the applied stress. At this stage, the material will fail due to rapid crack growth. [3] Figure 2-6 shows the stages in crack propagation.

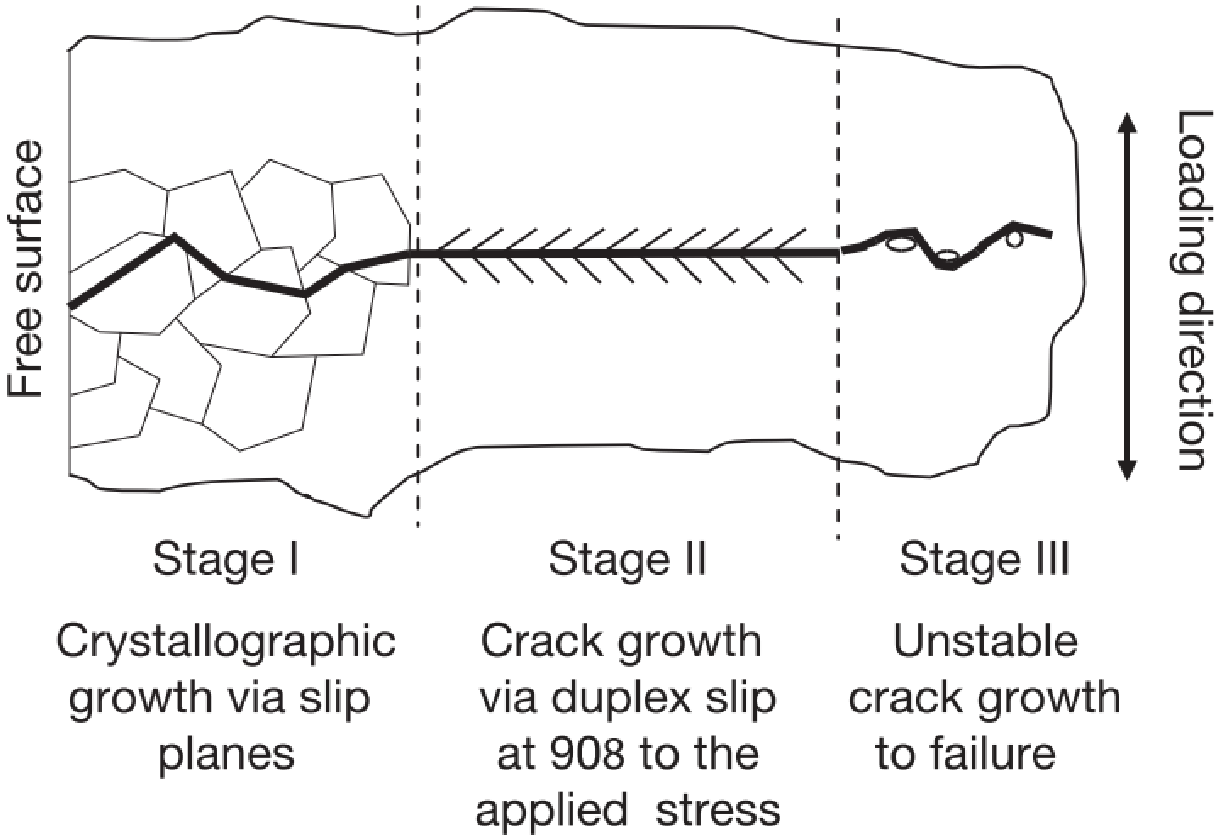

Figure 2 - 6 Initiation, propagation and unstable crack growth in a non-corrosive environment. [30]

2.6 Corrosion Fatigue

Corrosion fatigue is a damage mechanism that involves the synergistic effects of a corrosive environment and cyclic or dynamic loading. The presence of a corrosive environment reduces the fatigue life of a material. Corrosion fatigue involves corrosion damage, mechanical fatigue, anodic dissolution and cathodic reactions. [31] The additive effect of fatigue and corrosion damage is lesser than the effect of corrosion fatigue. The corrosive environment's contribution influences stage I and stage II of fatigue crack life, as shown in Figure 2-7. This implies that failure of material under corrosion fatigue is enhanced compared to fatigue in a non-corrosive environment. As mentioned above, in a non-corrosive environment, crack initiation could occur at PSBs due to localized plastic deformation.

Corrosion Fatigue Initiation: The presence of a corrosive environment introduces more crack initiation mechanisms, thereby reducing the time required for crack initiation. Corrosion fatigue involves crack initiation mechanisms such as pitting or local galvanic effect between different phases. Corrosion fatigue cracks may also initiate at surface defects like non-metallic inclusions. Inclusions are stress raisers and localized plastic deformation is favoured at inclusion sites. Under cyclic loading, micropits are formed at inclusion sites in a corrosive environment, allowing the initiation of fatigue cracks. Also, corrosion fatigue cracks can initiate from pits formed from localized microgalvanic cells. The pit continues to grow until there is a transition from pit to crack. In corrosion fatigue, the pit to crack transition is the most important stage.

Corrosion Fatigue Crack Propagation: The presence of a corrosive environment increases crack growth rate under stage II. This is made possible by providing a favourable environment and loading condition for the short crack to transition to a long crack. At this stage, the Linear Elastic Fracture Mechanics (LEFM) approach applies once the plastic zone size ahead of the crack tip is less than 1/50th of the crack length. [32]

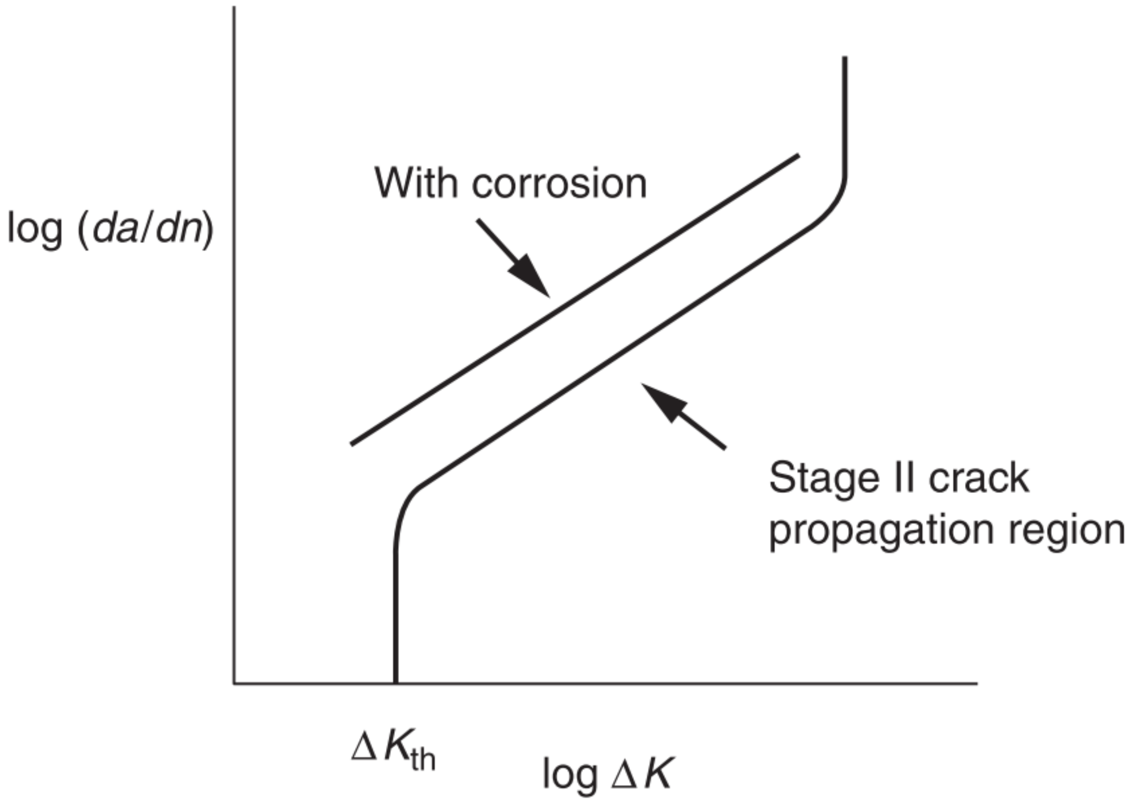

Figure 2 - 7 Effect of corrosive environment on fatigue crack growth compared to non-corrosive environment. [3]

2.7 Cracking in Steel Pipelines under Near Neutral pH Environment – A Case of Corrosion Fatigue

Generally, cracks are initiated as microstructurally short cracks formed by several mechanisms such as crack initiation at stress raisers like corrosion pits, dissolution at defects like inclusions and grain boundaries and PSBs. [3, 33-39] In NNpH environment, initiation and growth of cracks in steel pipeline are promoted by conditions like the disintegration of coating on the pipe surface, presence of near-neutral pH environment and susceptible steel metallurgy. These conditions diminish or cut off the supply of protective cathodic current and allow the bare pipe surface access to the corrosive NNpH environment. [3] The presence of tensile residual stress on the pipe surface and the applied stress can influence the formation of crack colonies at favourable sites on the pipe surface. In the initiation stage (stage I), crack propagation is highly controlled by the dissolution mechanism. [40] It has been observed that the dissolution rate decreases as the crack depth increases due to a gradient in dissolved CO_2 at the crack tip. [41,42] At a depth of approximately 1 mm, dormancy has been observed in 95% of cracks in steel pipelines. [31] Crack propagation in stage II is influenced by a crack-activating mechanism that involves the mechanical loading condition and hydrogen. These conditions favour an increase in crack growth rate as the depth increases to develop a stage III crack driven mainly by mechanical loading conditions that eventually cause the pipeline to rupture. Figure 2-8 shows the stages in the development of a crack in a steel pipeline under a near-neutral pH environment. As shown in Figure 2-8, the dormancy phenomenon observed in 95% of cracks creates a transition from a dissolution-controlled mechanism to a mechanical-driven and hydrogen-controlled mechanism. [43] Figure 2-8 shows that once cracks are initiated, Stage II is critical to control crack propagation and extend the fatigue life.

Understanding the nature and influence of mechanical loading conditions on stage II crack growth has developed over the years. Previously, cracking in steel pipelines has been attributed to the phenomenon of Stress Corrosion Cracking (SCC). As discussed previously, SCC involves applying static stress to a susceptible material in a corrosive environment. In an NNpH environment, it is plausible to conclude that SCC will promote crack blunting due to low-temperature creep.

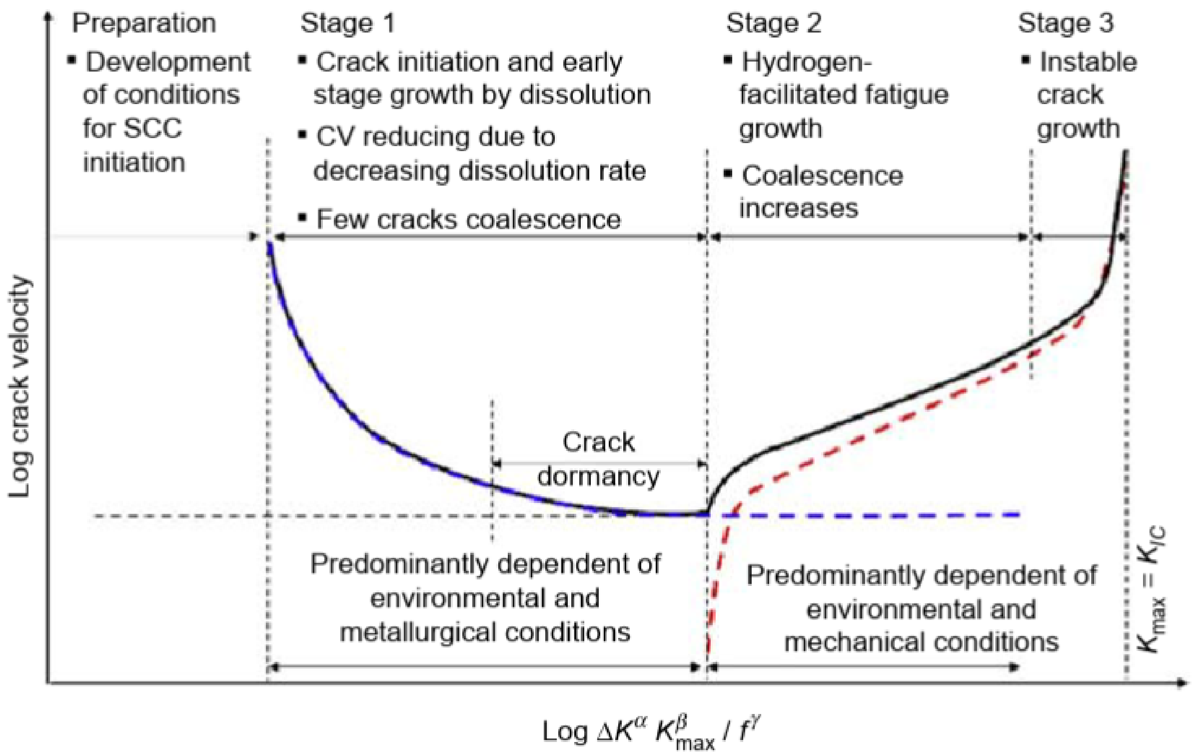

Figure 2 - 8 A modified bathtub model showing stages of crack growth in NNpH environment. [40]

This is due to near static loading at the crack tip and corrosion dissolution at the crack wall and crack tip since the NNpH environment is non-passivating. While this could explain 95% dormant cracks, it is difficult to explain why 5% of the crack can grow to a critical size that could cause failure of the pipe in the NNpH environment. [31]

In fact, the study of the effect of loading conditions on crack growth in steel pipelines under NNpH conditions showed that while cracks may initiate in an NNpH environment under static loading, it is impossible to propagate a crack in this environment. Chen *et al.* showed that a propagating crack becomes dormant when the loading condition was changed to static hold, as shown in Figure 2-9.

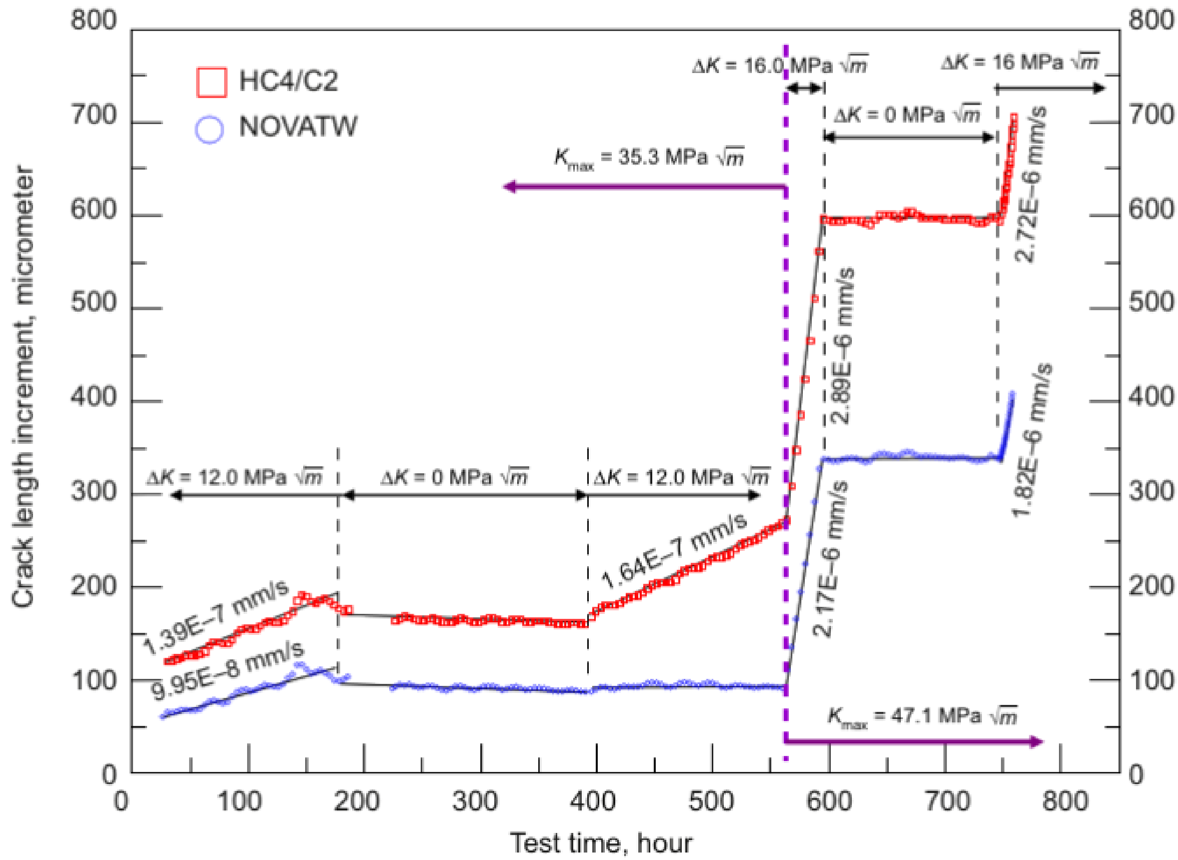

Figure 2 - 9 Effect of static hold vs dynamic cyclic loading on crack growth. [31]

This was observed even under a high stress intensity. This observation was attributed to crack tip blunting and a decrease in any mechanism that favours sharpening of the crack tip. [31] It was also observed that a pre-existing axial crack became dormant in an NNpH environment under static loading.

As stated above, mechanisms that promote sharpening of the crack tip or re-sharpening of a blunt crack tip are responsible for the growth observed in the 5% crack growth. This shows that rather than SCC, crack propagation in steel pipelines under NNpH environment can be described as corrosion fatigue. Therefore, it is important to consider the effect of dynamic loading conditions on crack propagation in stage II.

2.7.1 Effect of hydrogen on corrosion fatigue crack propagation in NNpH environment

Another mechanism that influences crack growth in Stage II is diffusible hydrogen in the NNpH environment. Hydrogen is produced during the corrosion of steel pipes in the NNpH environment. It has been observed that an increase in CO_2 concentration can be related to a decrease in the ductility of steel. [44-46] The presence of diffusible hydrogen at the weakest links ahead of the crack tip can promote microcracks initiation at the crack tip and favour sharpening or re-sharpening of a blunt crack tip. The coalescence of such small cracks with the main crack's tip is responsible for re-sharpening of the blunt crack tip. [31] The crack-sharpening process is not effective under static loading in NNpH SCC since the concentration of hydrogen under NNpH environment is about 1/10th of the hydrogen concentration that can cause hydrogen-induced cracking in steel pipe. [47, 48] Chen *et al.* showed that although hydrogen is generated at the crack tip, the diffusion of hydrogen generated on the pipe surface to the crack tip is the main contribution of hydrogen-induced cracking at the crack tip. The influence of hydrogen can also affirm that crack propagation under NNpH environment is by corrosion fatigue mechanism. [31] Studies have shown that under static loading, hydrogen can influence dissolution. However, there was no evidence of cracking under static loading and an increase in crack growth rate was observed at higher pH. This shows that the interaction between diffusible hydrogen and slip bands enhances dislocation motion along the slip bands, thereby promoting hydrogen diffusion and enabling slip localization under cyclic loading. This further proves that the corrosion fatigue mechanism is best used to interpret crack growth under NNpH environments. [31]

2.7.2 Corrosion fatigue under variable amplitude loading

Similar to most structural components, pipelines are subjected to variable amplitude loading during operation. Figure 2-10 shows the three different types of pressure fluctuations common in oil and gas pipelines. [49] As shown in Figure 2-10, compared to oil pipelines, gas pipelines experience fewer variations in pressure during operation due to the compressibility of gasses. Underload pressure fluctuations are identified with pressure drops, while mean load pressure fluctuations are identified by increase and decrease in pressure values. Overload is identified with occasional increase in pressure values. It is important to consider the effect of these variable amplitude loading conditions on crack growth in steel pipelines as they present a realistic picture of field operations. Some of the recent findings on the effect of NNpH environment on crack growth have been based on underload pressure fluctuations. Yu *et al* studied the effect of underload pressure fluctuations on crack growth in NNpH environment. The work showed that crack growth in underload pressure fluctuation was about five times higher than constant amplitude loading, as shown in Figure 2-11. [43, 50] This result also showed that it is not accurate to use constant amplitude loading for predicting crack growth under variable amplitude loading due to the effect of minor cycles on crack growth.

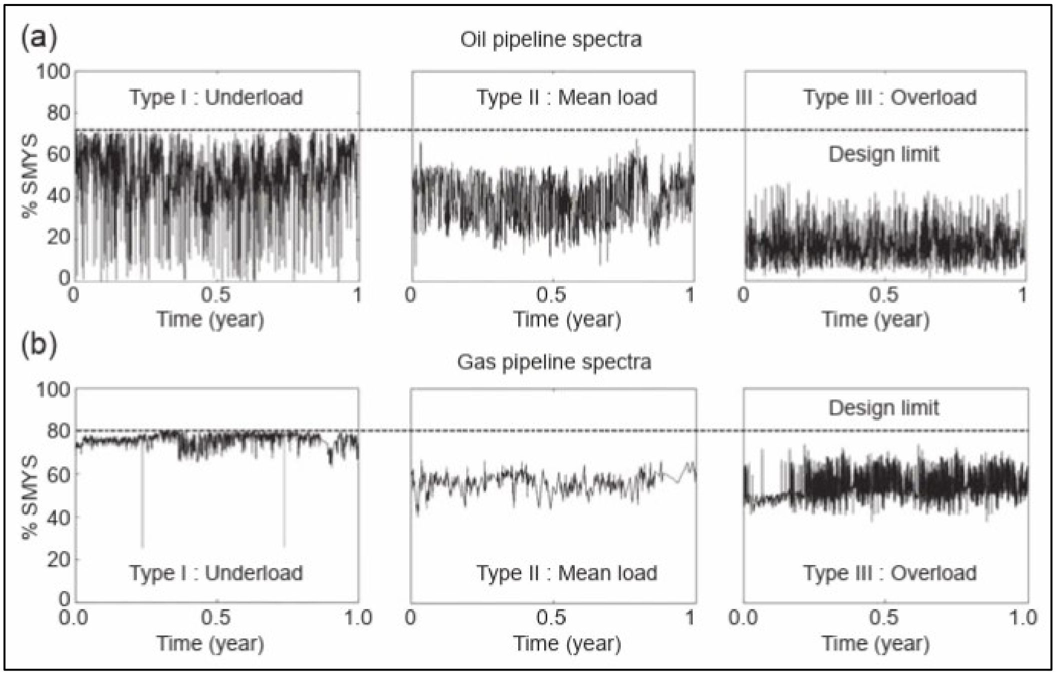

Figure 2 - 10 Typical pressure fluctuations in (a) oil and (b) gas pipelines showing underload, mean load and overload cycles. [49] Reproduced with permission from the American Society of Civil Engineers (ASCE).

Previously, minor cycles were referred to as non-propagating and hence not considered in crack growth predictions. [49] The inability to correlate the predicted remaining lives based on such predictive models compared to the life of pipes in service calls for a review of crack growth modelling under variable amplitude loading. This observation correlates with findings in other structural materials that underload causes acceleration effect on crack growth. It was also observed that crack growth responds to loading frequency, as shown in Figure 2-12. It appears that there is a transition in crack growth at 10^{-3} Hz. Above this critical frequency, crack growth was observed to increase with a decrease in loading frequency in both constant amplitude and underload conditions under NNpH conditions.

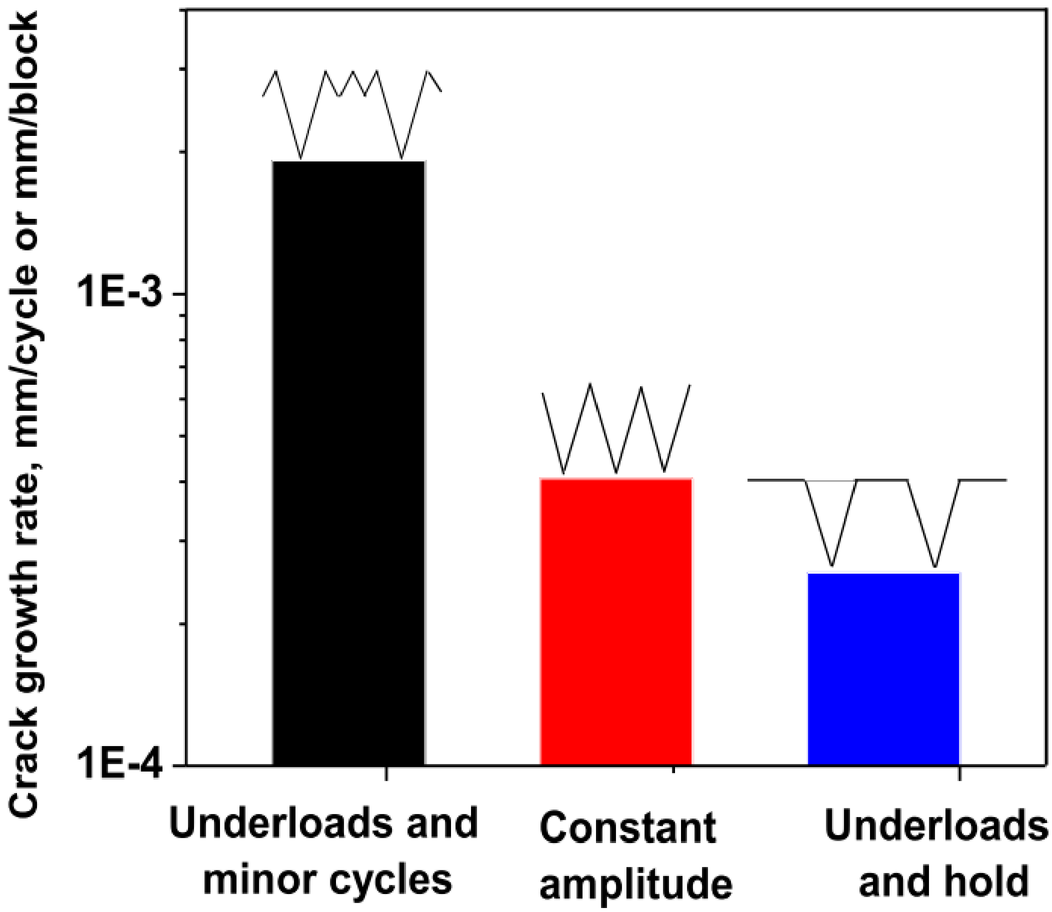

Figure 2 - 11 Acceleration effect of underload compared to constant amplitude loading in pipeline steel under NNpH conditions. [43, 50]

At frequency below the critical frequency, crack growth was observed to decrease with decreasing frequency under constant amplitude loading, while there was no significant change in crack growth as frequency decreased in underload variable amplitude loading conditions. The acceleration effect of the underload was higher below the critical frequency. [43, 50]

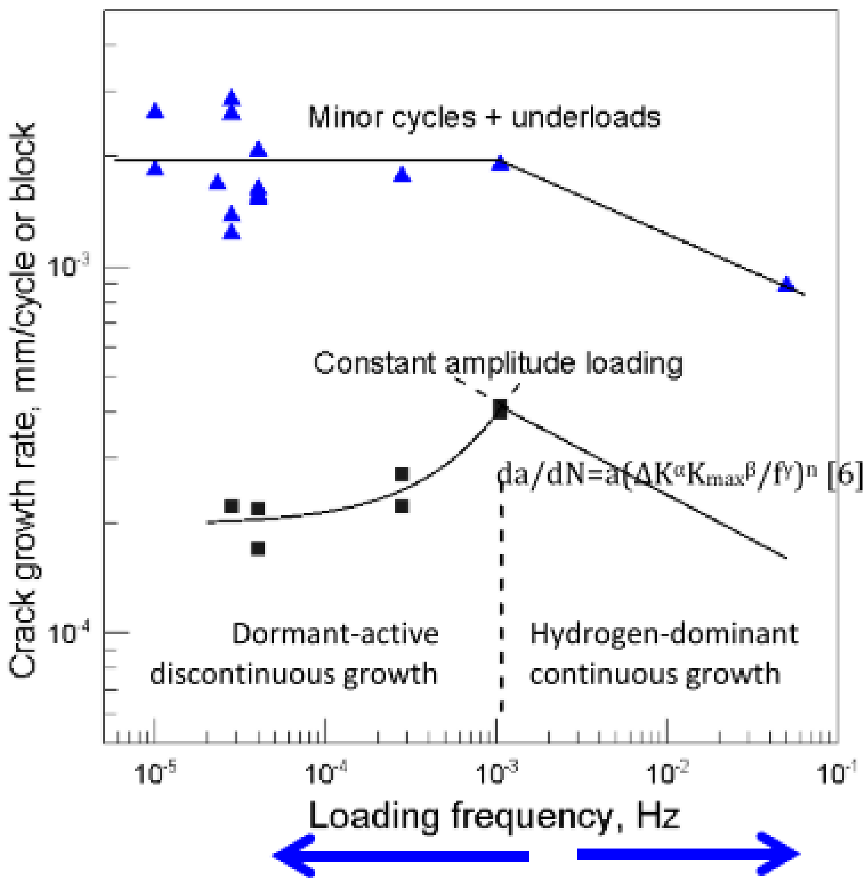

Figure 2 - 12 Effect of loading frequency on crack growth rate in underload + minor cycles compared to constant amplitude loading. [43, 50]

The effect of maximum stress intensity factor (K_{max}) on crack propagation in underload variable amplitude loading is shown in Figure 2-13. The result showed that while crack growth in air is insensitive to increase in K_{max} in underload variable amplitude loading, increase in K_{max} causes an increase in crack growth rate in NNpH environments. This trend in crack growth was explained by the effect of hydrogen embrittlement in the NNpH environment.

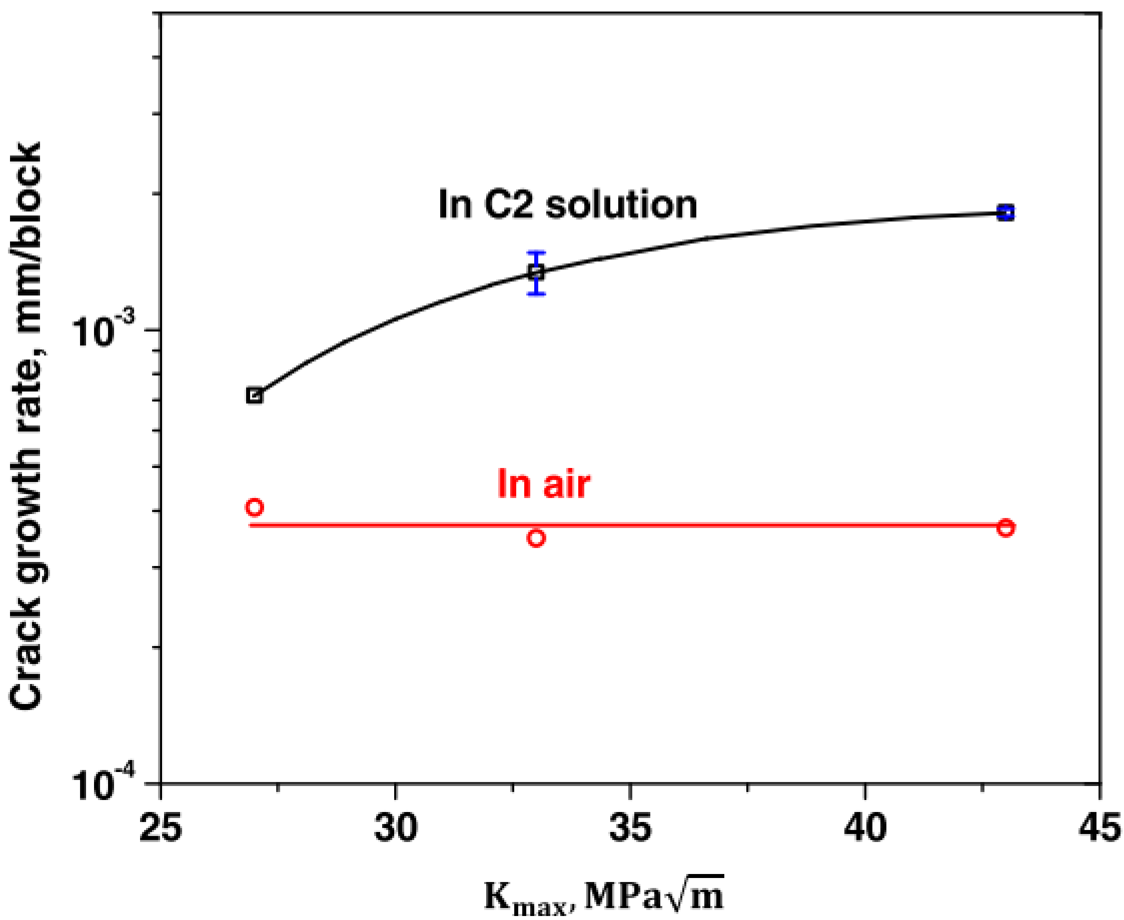

Figure 2 - 13 Effect of K_{max} on crack growth rate in pipeline steel under near neutral pH conditions. [43, 51]

Previously, minor cycles were regarded as loading cycles not contributing to crack propagation. Yu showed that minor cycles, combined with underload cycles as a variable amplitude loading, can cause crack growth acceleration. This trend was observed both in air and in NNpH environment. The contribution of minor cycles to crack growth was supported by fracture surface observation showing mini-striations. [43]

Based on the need to simulate cracks in the field as close as possible, some recent works have been conducted using samples with surface cracks. Engel studied the effect of underload + minor cycles (UL+MC), constant amplitude cyclic loading (CA) and underload + hold (UL + Hold) on surface crack growth. [52] The results showed that surface cracks could propagate at a threshold below the compact tension (CT) growth threshold under constant amplitude loading. This reduction in the crack growth threshold in surface cracks was related to the active nature of the environmentally short-cracks.

Engel's research also showed that overall, the crack growth rate of a surface crack is lower than that of a through-thickness crack for all the loading conditions. Based on the low crack growth rate recorded for the CA test in surface crack, it was also suggested that the contribution of minor cycles to crack growth in surface cracks might be significantly higher than in through-thickness samples. When the UL + Hold waveform is compared to CA in surface cracks, the trend is opposite from that observed in through-thickness samples. Engel attributed the high crack growth rate observed in the surface cracks to the increased accumulation of hydrogen. [52]

Li studied the effect of changing the sequencing of cycles in underload fundamental blocks on surface cracks. [53] The work showed that altering the sequence of cycle can affect the growth rate in an underload spectrum. A higher crack growth rate was observed when the agglomeration of minor cycles is high. This confirms that minor cycles contribute to crack growth rate and a short agglomeration with an underload cycle can reduce the contribution of minor cycles to crack growth rate.

2.7.3 The role of hydrogen on crack growth under variable amplitude loading

It is generally accepted that hydrogen assisted cracking is a failure mechanism that can degrade material strength and reduce the service life of an engineering component. Among other factors, corrosive electrolytes, cathodic potential and gaseous environment can contribute to hydrogen-induced cracking in materials. Although it appears that there is no single mechanism that can explain crack growth observed under the influence of hydrogen, hydrogen enhanced decohesion (HEDE) and hydrogen enhanced localized plasticity (HELP) mechanism are widely accepted atomistic mechanisms of hydrogen assisted cracking. The hydrogen enhanced localized plasticity theory shows that the trapping of hydrogen in the plastic zone causes a decrease in the flow stress. This enhances dislocation motion and results in localized deformation. Hydrogen enhanced decohesion theory shows that diffusion of hydrogen into the material will cause a reduction in the bond strength of the parent material and could cause a brittle fracture. [54] In an NNpH environment, hydrogen may be generated through corrosion. In an effort to understand the crack growth mechanism in NNpH environment, Yu studied the crack growth rate in coated and bare samples exposed to NNpH environment. [43]

The influence of hydrogen on crack growth in NNpH conditions is significant compared to crack growth in air. It was observed that cracks were wider on the surface and crack width reduced in the mid-section of the samples tested in NNpH environment. The reduction in the crack width towards the midsection of the sample suggests that the influence of corrosion on crack advancement is negligible, so the effect can be related to HELP mechanism.

In terms of macro-mechanism of hydrogen assisted cracking, Gangloff showed that hydrogen embrittlement can be classified in terms of the source of hydrogen causing the damage. This was classified into hydrogen environment assisted cracking (HEAC) and internal hydrogen assisted cracking (IHAC). [55] In HEAC, the electrochemical reaction in localized regions close to the crack tip favours hydrogen atom production. Due to mechanical loading, hydrogen produced from corrosion diffuses through the crack tip into the fracture process zone, as shown in Figure 2-14. [55-57] The high concentration of hydrogen in this region will enhance crack advance. In IHAC, hydrogen can diffuse into the bulk material due to exposure to a corrosive environment such as NNpH. The load application will enhance the redistribution of hydrogen to the fracture process zone to promote crack advance.

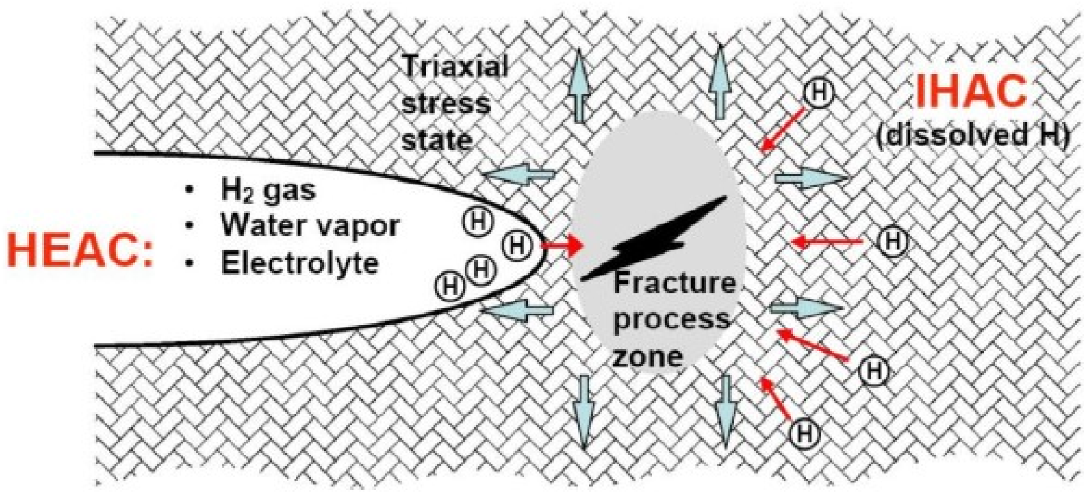

Figure 2 - 14 Hydrogen damage by HEAC and IHAC in pipeline steel under NNpH environment. [55-57]

Experiments in reference [31] confirmed that IHAC contributes significantly to crack propagation in NNpH environments. As shown in Figure 2-15, samples were designed to show three conditions.

The first condition was totally coated to eliminate the effect of corrosion and hydrogen on crack growth.

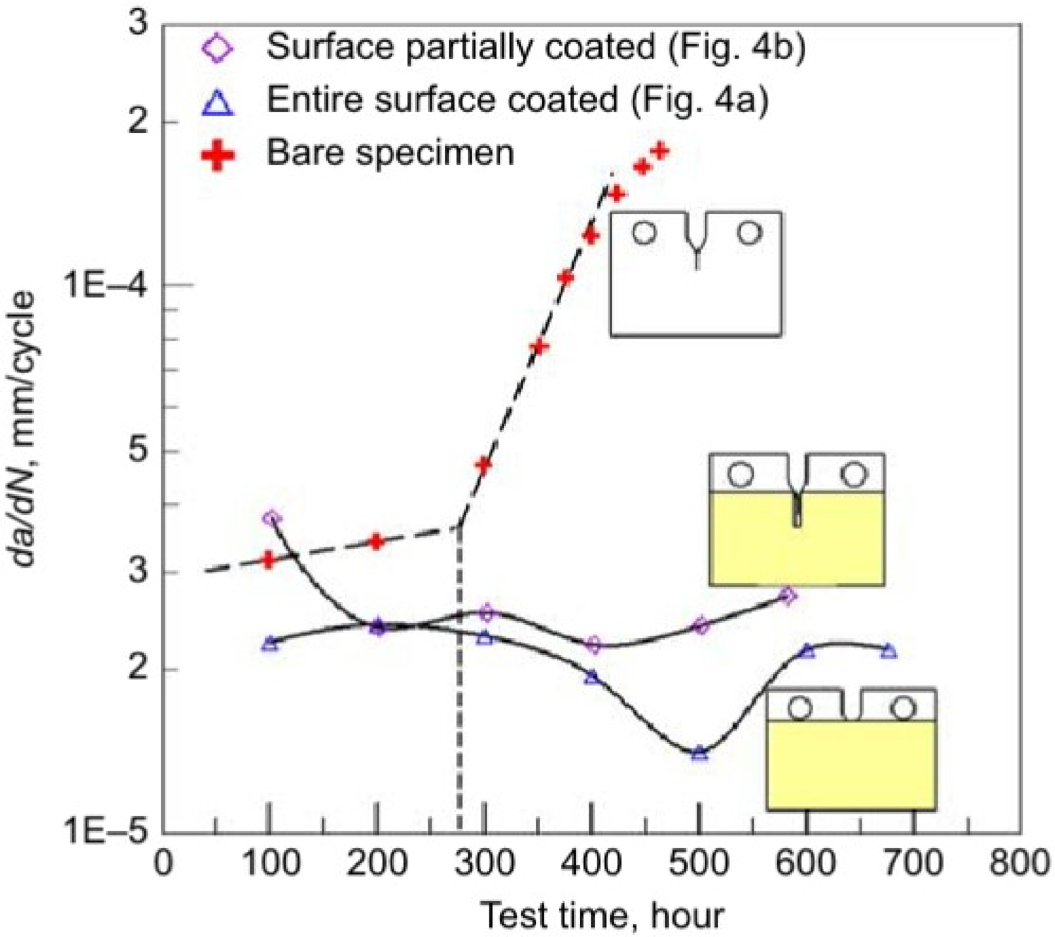

Figure 2 - 15 Effect of hydrogen from HEAC (coated sample) vs IHAC (bare sample) on crack growth in NNpH. [31]

The second sample was partially coated to allow only HEAC by not coating the crack tip region. The third sample was tested without any coating to allow the effect of corrosion and hydrogen from the crack tip (HEAC) and hydrogen from the bulk material (IHAC). The results shown in Figure 2-15 shows that hydrogen embrittlement through HEAC does not have a significant effect on crack growth rate. However, the IHAC condition promoted an increase in crack growth rate.

2.7.4 Crack growth models for constant amplitude loading in NNpH environment

Paris crack growth model

The relationship between crack growth rate and ΔK is shown in Figure 2-16 below. The three stages in crack growth are represented as stages I, II and III, respectively. [58] Stage I is the crack initiation stage at which the ΔK must have reached a threshold value below which crack propagation does not occur. In stage II, crack propagation occurs and crack growth rate can be represented by the Paris law. In stage III, the crack growth rate is higher and a fast fracture of the material occurs. Most of the engineering application of the material considers stage II crack growth. Paris law presents a simple fracture mechanics approach to analyse fatigue crack growth behaviour in structural materials, as shown in the equation below.

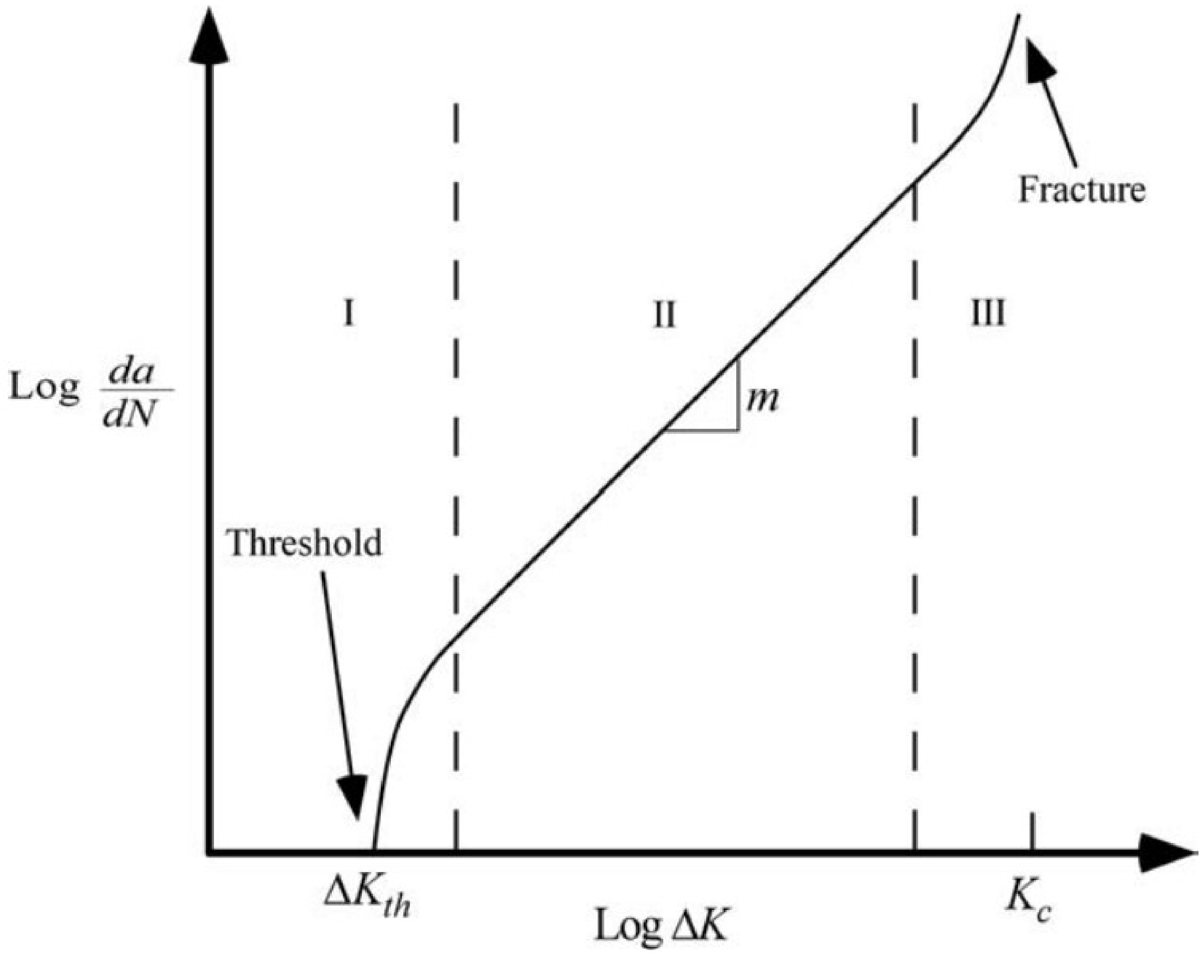

Figure 2 - 16 Fatigue crack growth rate under Paris law. [58]

$$\frac{da}{dN} = C\Delta K^m \quad (7)$$

Where da/dN = crack growth rate

ΔK = stress intensity factor range

C and m = material constants describing geometry effect

However, as discussed earlier, Paris law does not consider the effect of K_{max} and frequency on crack growth behaviour. While Paris law might be suitable for crack propagation in a non-corrosive environment, it is not applicable to NNpH environment, as explained by Chen *et al*. [59]

Superposition model

The superposition model is one of the early models applied to analyse crack growth rate in NNpH environment. As shown in the equation below, the superposition model is based on a combination of crack growth under fatigue and crack growth due to SCC. Based on this equation and the low operating frequency common in pipeline operations, the superposition model tends to overestimate the contribution of SCC to crack propagation. [59] This is inconsistent with the observation that crack growth in NNpH environment does not occur under static or monotonic loading, which is a requirement for SCC.

$$\left(\frac{da}{dN}\right)_{total} = \left(\frac{da}{dN}\right)_{fatigue} + \frac{1}{f}\left(\frac{da}{dt}\right)_{SCC} \tag{8}$$

Crack tip stain rate model

The crack tip strain rate model shows that there is a relationship between the strain rate generated at the crack tip and the loading condition represented as the R ratio. As shown in the equation, at a low R ratio, the strain rate will be high and this corresponds to an increase in dissolution rate and hence an increase in crack growth rate. It is worth noting that this analysis is not applicable to NNpH environment where there is no passivation of oxide films at the crack tip. [59]

$$Crack\ tip\ strain\ rate = 4f(1 - R) \tag{9}$$

Given that f is the loading frequency, and R is the ratio of minimum stress to maximum stress.

Combined Factor Model

The combined factor model was developed to specifically analyze crack growth behaviour in NNpH environment as a true corrosion fatigue crack growth behaviour with consideration of mechanical loading conditions like ΔK, K_{max} and loading frequency. [59] The combined factor model provides a good fit for crack growth rate in NNpH environment, as shown in Figure 2-17. It also shows that there is a threshold value below which crack propagation is negligible and crack dormancy will occur. The combined factor model showed that under constant amplitude loading, the crack growth rate increases with ΔK and K_{max}. Also, using frequency in the range of 0.1 to 0.00125 Hz, Chen *et al.* showed that crack growth rate increases with a decrease in loading frequency. [59] The result also shows that a threshold combined factor value (~8500 $(MPa\sqrt{m})^3/Hz^{0.1}$) is required for crack propagation to occur in C2 solution. Based on these results, Chen *et al.* showed that there is a region separating crack dormancy from crack growth and frequency plays an important role in the transition from dormancy to crack growth. Chen's result confirmed that crack growth at 10^{-5} Hz as observed in reference [60] is below the combined factor threshold for crack propagation and hence the dormancy was observed.

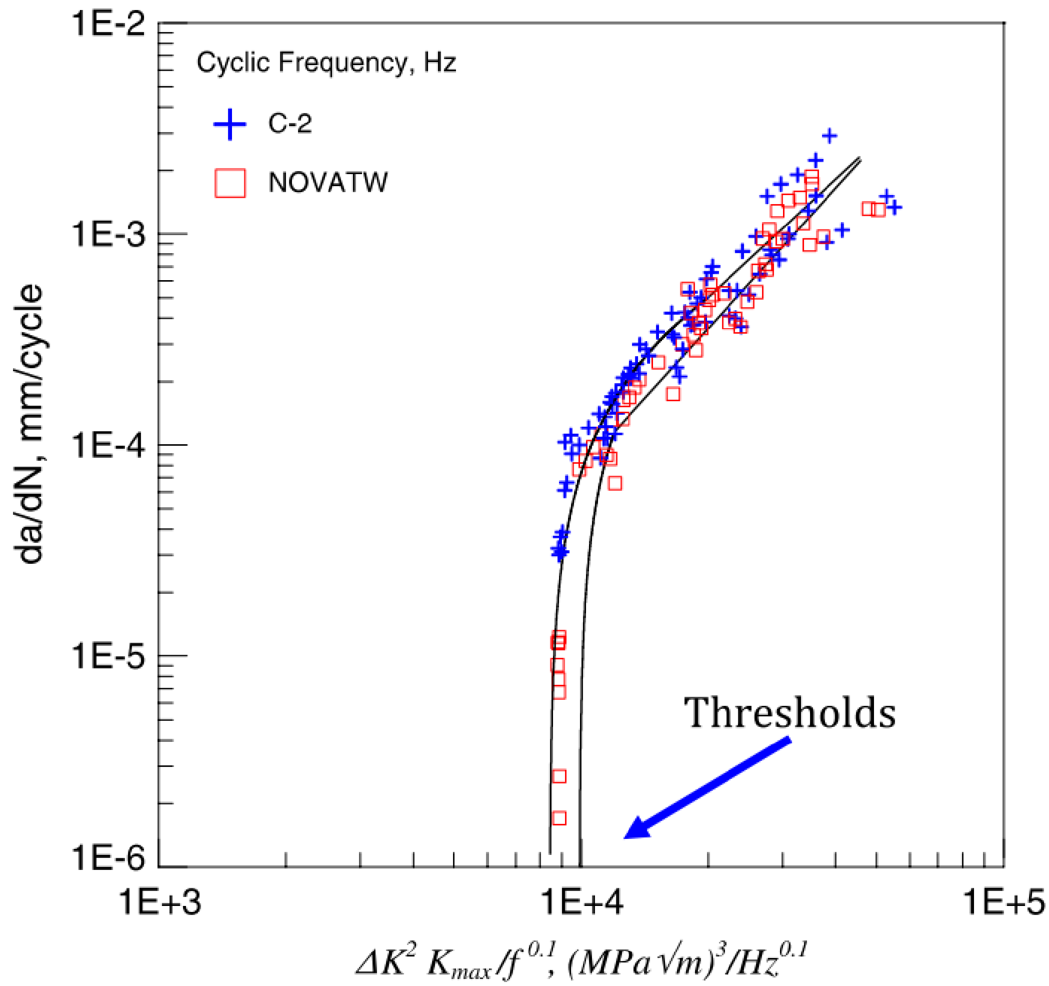

Figure 2 - 17 Crack growth rate in NNpH environment correlation combined factor model. [59]

2.7.5 Load interaction in variable amplitude loading

The phenomenon of load interaction shows that fatigue crack growth rate is affected by previous load history. This implies that crack growth under constant amplitude loading will differ from variable amplitude loading even when the maximum and minimum stress intensity factors are the same. [6] Accurate prediction of fatigue life must consider the effect of load interaction on fatigue crack growth. This requires that load-time history in service should be converted to variable amplitude load sequences that can simulate the load that the component experienced in service. [6, 61]

2.7.5.1 Load sequences with overloads

Variable amplitude loading with overload can be a single overload, blocks of overload or periodic overload. Generally, it has been established that load interaction effects cause crack growth retardation in overloads, as shown in Figure 2-18.

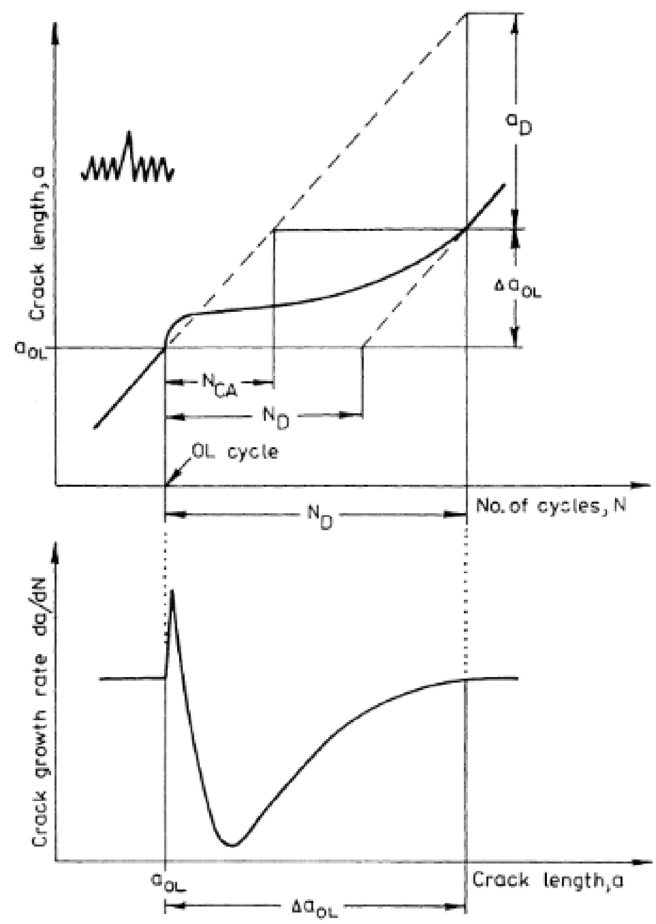

Figure 2 - 18 Retardation effect of overload on crack growth rate. [69]

The retardation effect is measured by the number of delay cycles (N_D), delay distance (a_D) and overload affected crack growth increment (Δa_{OL}). An increase in the retardation effect has been observed with an increase in the overload ratio. [62-69]

2.7.5.2 Load sequence with underload

Underload variable amplitude loading has been related to the acceleration of crack growth. [70] The acceleration effect of underload is well documented but a few works have also found negligible load interaction effect and underload induced retardation. [71-73] The acceleration factor usually defines the detrimental effect of the underload spectrum. The acceleration factor is the ratio of crack growth rate under variable amplitude loading to crack growth under constant amplitude loading.

2.7.5.3 Load sequence with underload and overload

Most research papers available on load combinations of underload and overload in a variable amplitude loading suggest that when overload is followed by an underload, crack growth rate is higher than when an underload is followed by an overload, as shown in Figure 2-19. [69, 70, 74-77] However, another study on the effect of load combination of underload and overload on crack growth in stainless steel showed that underload followed by overload increased crack advance compared to overload followed by underload in stainless steel. [69] A similar trend was reported in reference [77], as shown in Figure 2-20. These results suggest that there is no agreement on the effect of the combination of load sequence of underload and overload.

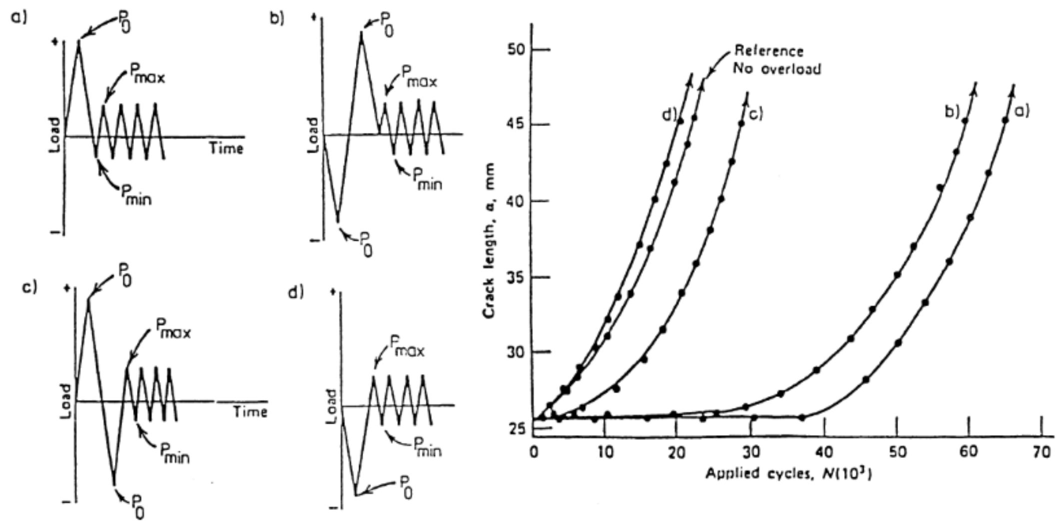

Figure 2 - 19 Effect of load interaction on crack growth in aluminium alloys. [76]

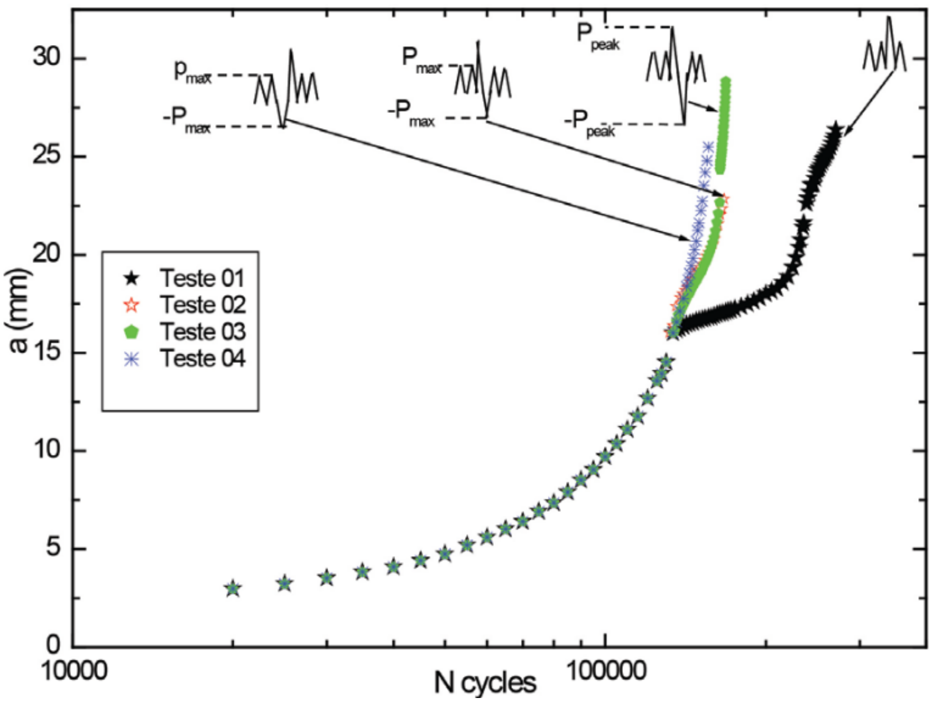

Figure 2 - 20 Effect of load sequence on crack growth showing that UL+OL+MC could achieve a higher crack growth rate compared to OL+UL+MC in steel. [77]

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Chapter 3: Crack Growth Behaviour under Constant Amplitude Loading in NNpH Environment

3.1 Introduction

Propagation of cracks under near-neutral pH (NNpH) condition remains a significant threat to pipeline integrity. It has been established that the conditions required for these cracks to propagate are stress, susceptible material and a corrosive environment. While there is consensus that applied stress influences the propagation of cracks in NNpH environment for pipeline steels, there are conflicting ideas on the effect of the nature of the applied stress. Some research works have confirmed that pressure fluctuations during the pipeline operation are the driving force for crack propagation in NNpH SCC. [1-4] However, some other research works suggest that crack initiation and growth can occur under constant or static loading. [5, 6]

Pipeline operators take proactive steps to meet regulatory requirements and ensure that pipelines run efficiently and safely. This requires understanding the behaviour of threats such as corrosion, dents and cracks. This understanding also helps to achieve accurate prediction of fatigue life and guide integrity decisions such as repair or replacement of the pipe section. Most predictive models for crack growth in pipeline steels are based on constant amplitude cyclic loading. Constant amplitude cyclic loading is a form of cyclic fatigue loading in which mean load and load amplitude are constant. [7] Constant amplitude cyclic loading consists of simplified and clearly defined cycles of loading. Although engineering structures are subjected to random or variable amplitude loading, constant amplitude cyclic loading is often considered to determine fatigue damage experienced in service. This is partly due to the simplicity of constant amplitude cyclic loading and due to the short-term nature of the tests. This makes sense, especially given the low loading frequency of operations of pipeline steels which would require extended long-term tests to replicate. However, real-life pipeline operations can be described as variable amplitude loading, as shown in Figure 3-1. [8, 9]

It has been observed that it is often difficult to correlate crack growth behaviour observed on pipelines in service to fatigue life predictions based on constant amplitude predictive models. [10-15] In an effort to achieve accurate prediction of the fatigue life of cracks, it is important to consider real-life pressure fluctuations and their effect on crack growth behaviour. Research on the application of constant amplitude cyclic loading has been well reported. [14]

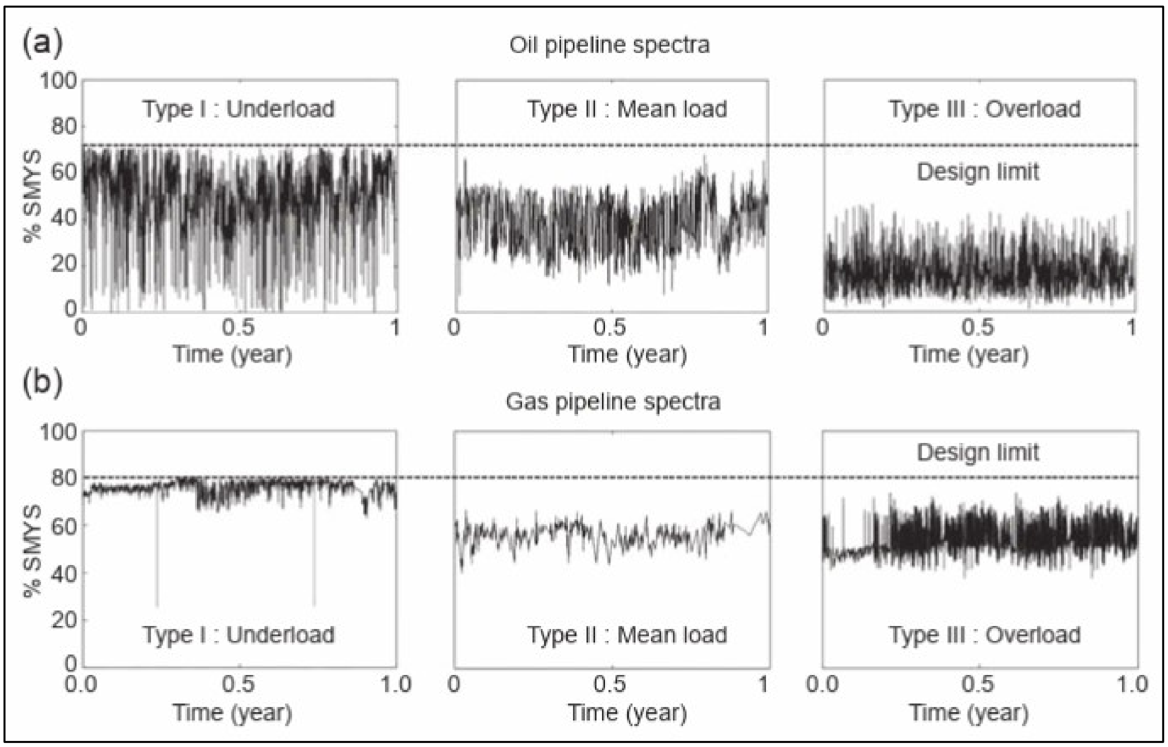


Figure 3- 1 Typical pressure fluctuation in (a) oil and (b) gas pipelines showing variable amplitude loading cycles. [9] Reprinted with permission from the American Society of Civil Engineers (ASCE).

One such approach is given in reference [16], where Chen *et al.* showed that crack growth under constant amplitude cyclic loading could be simulated by the combined factor equation. The study showed that factors such as ΔK, K_{max} and frequency can affect crack propagation in near-neutral pH conditions. In a more recent study, Yu and Chen have investigated the effect of constant amplitude cyclic loading at frequencies that are more feasible regarding pipeline operations. [14] The result showed that there is a threshold frequency below which crack growth rate does not increase with a decrease in loading frequency.

The effect of constant amplitude cyclic loading will be the subject of this chapter. The objective of this study is to apply constant amplitude cyclic loading conditions with parameters that simulate mean load pressure fluctuations. This will provide an understanding of crack growth behaviour under such loading conditions and provide information on crack growth expectations based on constant amplitude cyclic loadings. Also, the effect of minor cycles (high R ratio cycles also known as ripple load) will be considered. Such high R ratio (R ratio=ratio of minimum stress to maximum stress) cycles were previously considered as non-propagating. It is important to understand the mechanism of crack growth under minor cycles to determine in what cases every load cycle contributes to crack growth. The effect of the environment on crack growth in pipeline steel will also be considered by conducting some constant amplitude tests in air. Fracture surface morphology will be presented to analyse crack growth behaviour under constant amplitude cyclic loading.

3.2 Experimental Procedure

3.2.1 Material

The material used in this study was the API 5L X-65 steel pipeline that was known to be susceptible to NNpH SCC in service. Table 3-1 lists the chemical composition of the X-65 steel. The microstructure of typical X65 steel is shown in Figure 3-2. Compact Tension (CT) specimens were machined from a section of the pipe. Figure 3-3 shows the dimension of CT specimens used. To simulate axial cracks found on pipes, the notch direction was oriented parallel to the longitudinal direction of the pipe. The samples were ground on both surfaces with 120, 240, 400 and 600 grades of grinding papers. Fatigue pre-cracking was carried out to initiate a crack of 2.5 mm length based on ASTM standard E647. It was ensured that the difference in crack length on the sides did not exceed 0.2 mm. The crack tip was marked using an indentation. A Scanning Electron Microscope (SEM) was used to determine the position of the indentation before the test.

3.2.2 Test Environment

An NNpH environment referred to as C2 solution was used in this research. Table 3-2 lists the composition of the C2 solution. The prepared C2 solution was bubbled with 5% CO_2 + 95% N_2 prior to the test until the pH stabilized and then continuously for the test duration.

3.2.3 Loading Condition for Constant Amplitude Tests

Figure 3-4 shows the waveform for constant amplitude cyclic loading. Details of the loading conditions are listed in Table 3-3. The maximum and minimum stress intensity factor applied in the constant amplitude cyclic loading simulates mean load pressure fluctuation. Minor cycles have been considered as a case of high R ratio of constant amplitude cyclic loading in Test 6 to understand the contribution of minor cycles to crack growth.

Table 3- 1 Chemical composition of API 5L X65 pipeline steel

Element	**C**	**Mn**	**P**	**S**	**Si**	**V**	**Nb**	**Cr**
Composition (wt. %)	0.12	1.5	0.017	0.0046	0.26	<0.01	0.049	<0.005

Table 3- 2 Chemical composition of C2 solution

Salt	**KCl**	**$NaHCO_3$**	**$CaCl_2.2H_2O$**	**$MgSO4.7H_2O$**	**$CaCO_3$**
Concentration (g/L)	0.0035	0.0195	0.0255	0.0274	0.0606

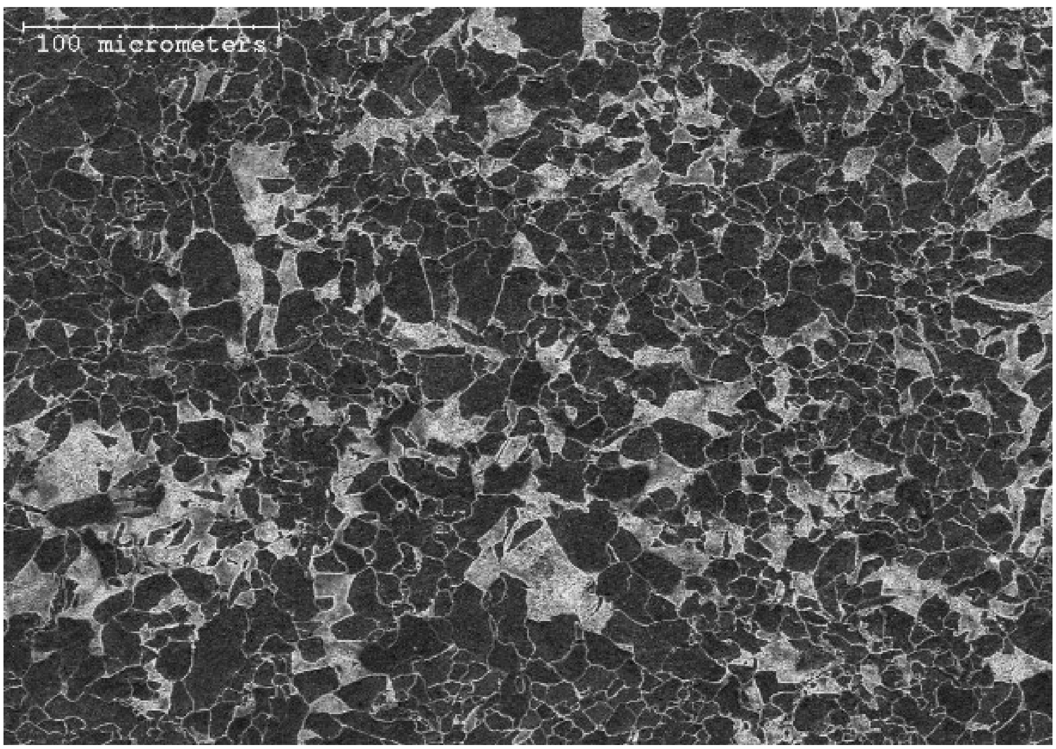


Figure 3- 2 Typical microstructure of X65 pipeline steel showing a ferrite-pearlite microstructure.

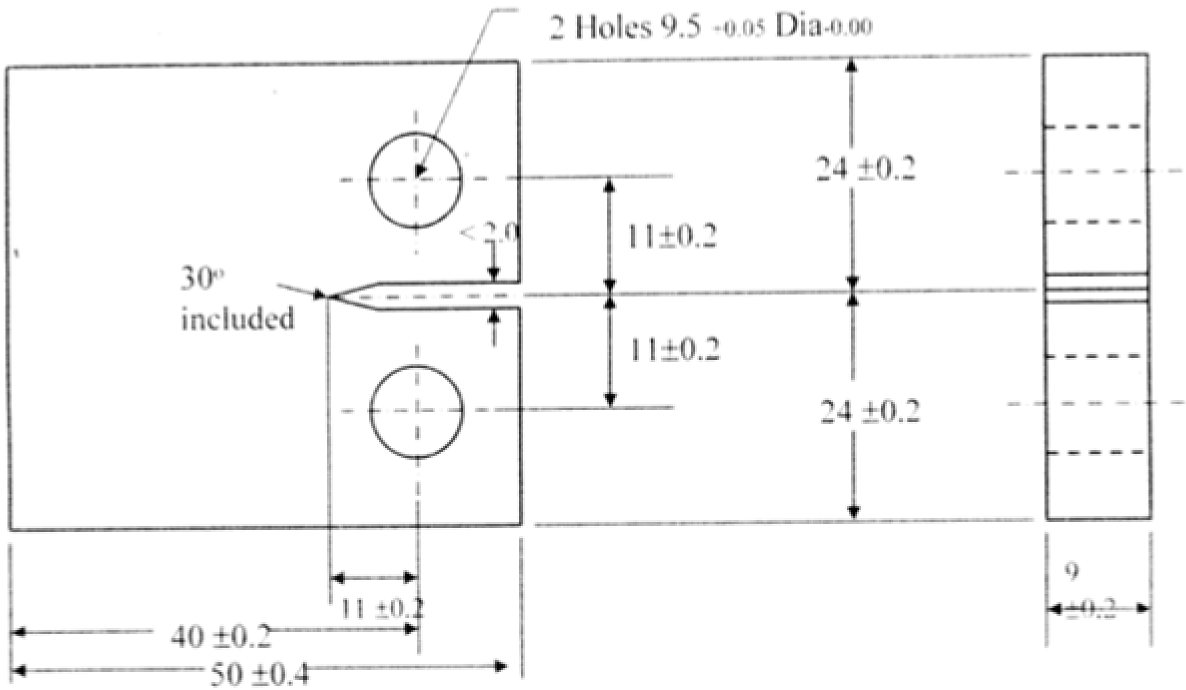


Figure 3- 3 Dimensions of CT specimen.

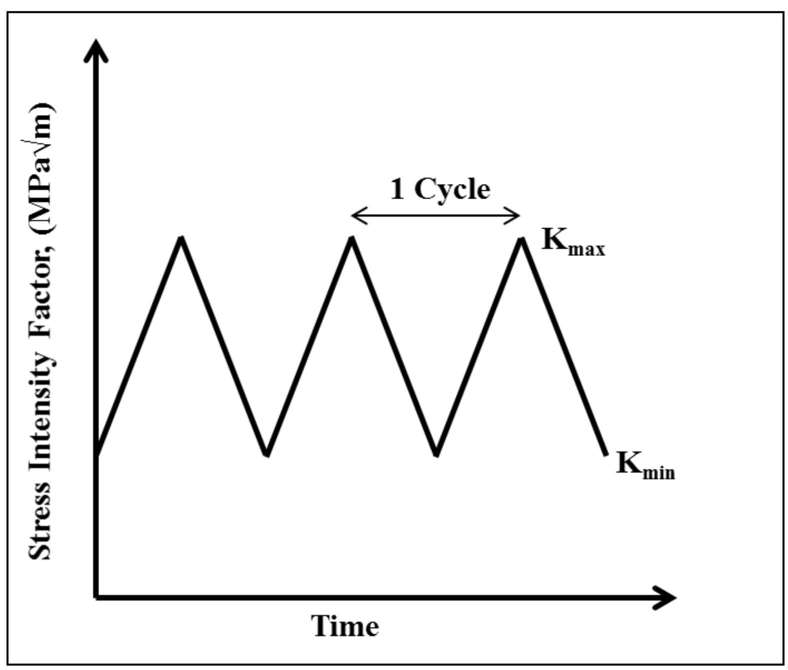


Figure 3- 4 Loading waveform for constant amplitude cyclic loading. [17]

Table 3- 3 Loading parameters for constant amplitude cyclic loading.

Test	K_{max} (MPa√m)	K_{min} (MPa√m)
1	33	16.5
2	34.65	16.5
3	36.3	16.5
4	39.6	16.5
5	42.9	16.5
6	33	29.7

At the end of the test, the samples were preserved and cleaned in ethanol. Scanning Electron Microscope (SEM) images of the fracture surface were taken to measure crack growth. The crack growth rate for each condition was determined as mm/cycle. This was obtained by dividing the measured crack growth by the number of cycles.

3.3 Results and Discussion

3.3.1 Constant amplitude loading

Figure 3-5 shows crack growth rate under constant amplitude cyclic loading with respect to the combined factor. As shown in Figure 3-5, the crack growth rate increased as the magnitude of ΔK and K_{max} increase under constant amplitude cyclic loading condition. [15] This trend correlates with the combined factor model (Eq. 1). The equation for the combined factor model is given as: [16]

$$\frac{da}{dN} = A\left[\frac{\Delta K^{\alpha} Kmax^{\beta}}{f^{\gamma}}\right]^{n} + b \quad (1)$$

Where: da/dN is the crack growth rate and A, α, β, n, γ are constants and α+β=1. ΔK is the stress intensity factor range at the crack tip, K_{max} is the maximum stress intensity factor at the crack tip, f is the loading frequency, α and β represent the respective relative contribution of ΔK and K_{max} to crack growth. The influence of the corrosion environment on the crack growth rate is represented as γ and b is the contribution of stress corrosion crack tip dissolution.

Crack growth under minor cycles loading has been considered as constant amplitude cyclic loading with a high R ratio. Previously, minor cycles were considered as non-propagating because they were below the crack propagation threshold. [18] Hence the contribution of minor cycles to crack propagation was mostly ignored in crack growth predictive models previously. Many pipeline operators now assume every load cycle causes growth and a no-growth threshold is not considered. To clarify the contribution of minor cycles to crack propagation, a long-term test that involved minor cycles was conducted. Figure 3-6 compares crack propagation under minor cycles acting alone (Test 6) to other constant amplitude cyclic loading both measured (test) and predicted (combined factor equation). The result for minor cycles acting alone shows a crack growth rate that is about three orders of magnitude less than the predicted value based on Eq. 1. Also, in Figure 3-6 crack growth rate obtained under minor cycles is compared to the dissolution rate in NNpH environment. The result in Figure 3-6 shows that crack growth rate under minor cycles with no previous underloads can be related to dissolution rate due to corrosion in NNpH environment. [17] Hence it can be concluded that crack growth under minor cycles acting alone is driven mainly by dissolution at the crack tip.

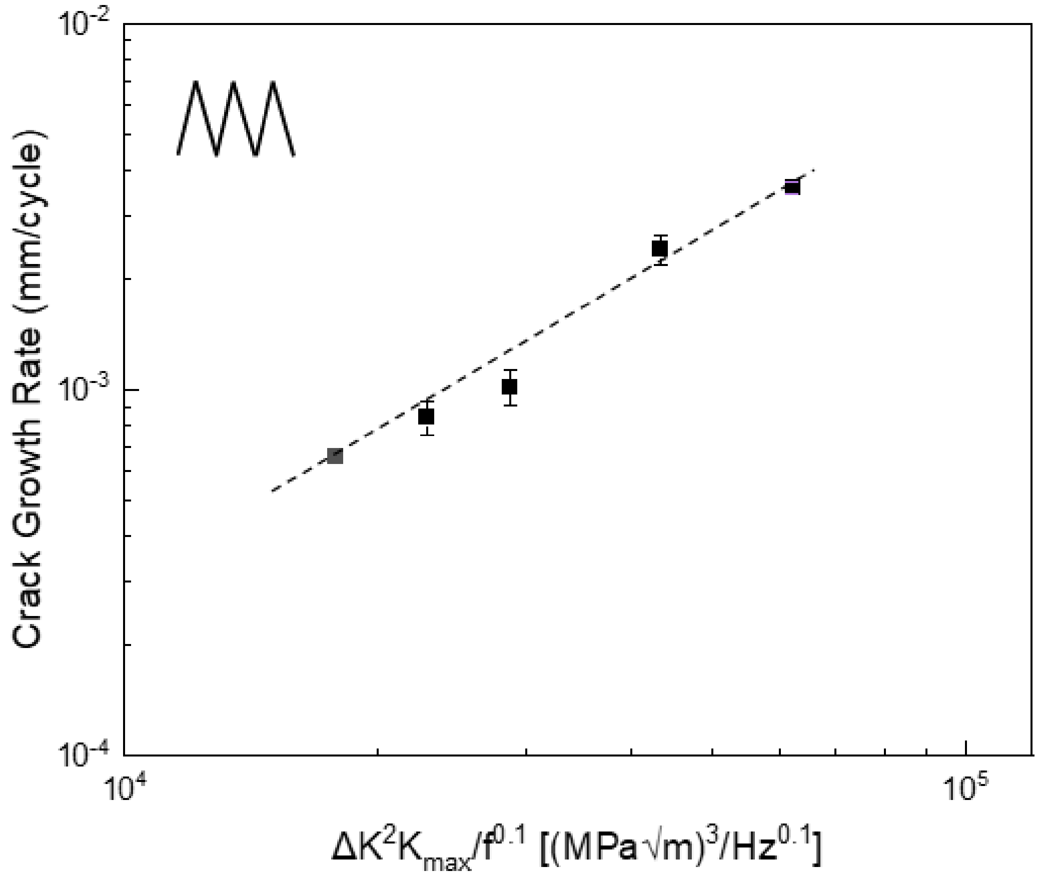


Figure 3- 5 Crack growth rate in NNpH environment under constant amplitude cyclic loading schemes showing increase in crack growth rate with increase in K_{max} and ΔK. [17]

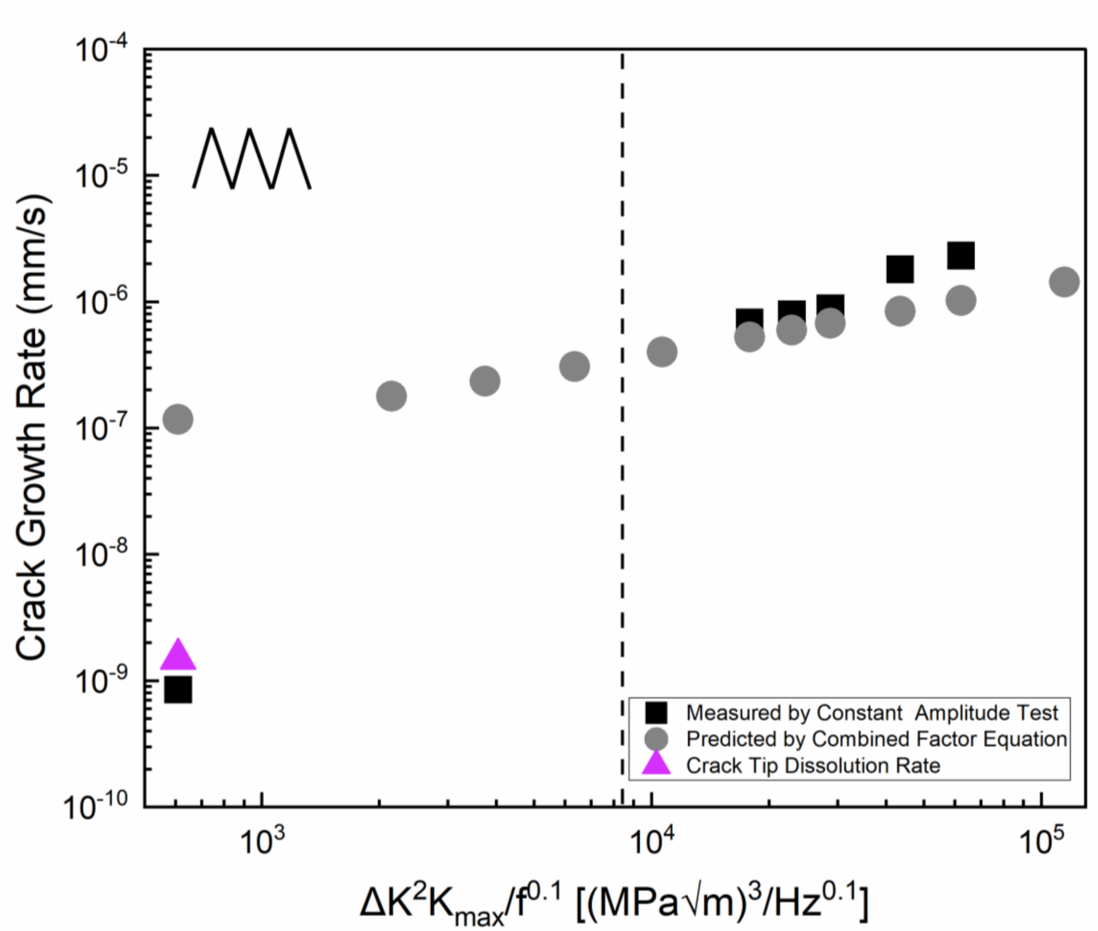


Figure 3-6 Crack growth rate under constant amplitude showing predicted (grey) and measured crack growth rate (black) under constant amplitude cyclic loading in NNpH environment. Measured crack growth rate under minor cycles acting alone (black data point below the threshold indicated by the dashed line) loading can be related to dissolution rate (purple) in NNpH environment. [17]

3.3.2 Effect of environment on crack growth under constant amplitude cyclic loading

Figure 3-7 shows crack growth in air compared to crack growth rate in NNpH solution under constant cyclic amplitude loading. Figure 3-7 shows that increase in K_{max} in air did not significantly increase the driving force for crack propagation and the reverse is the case in the NNpH environment.

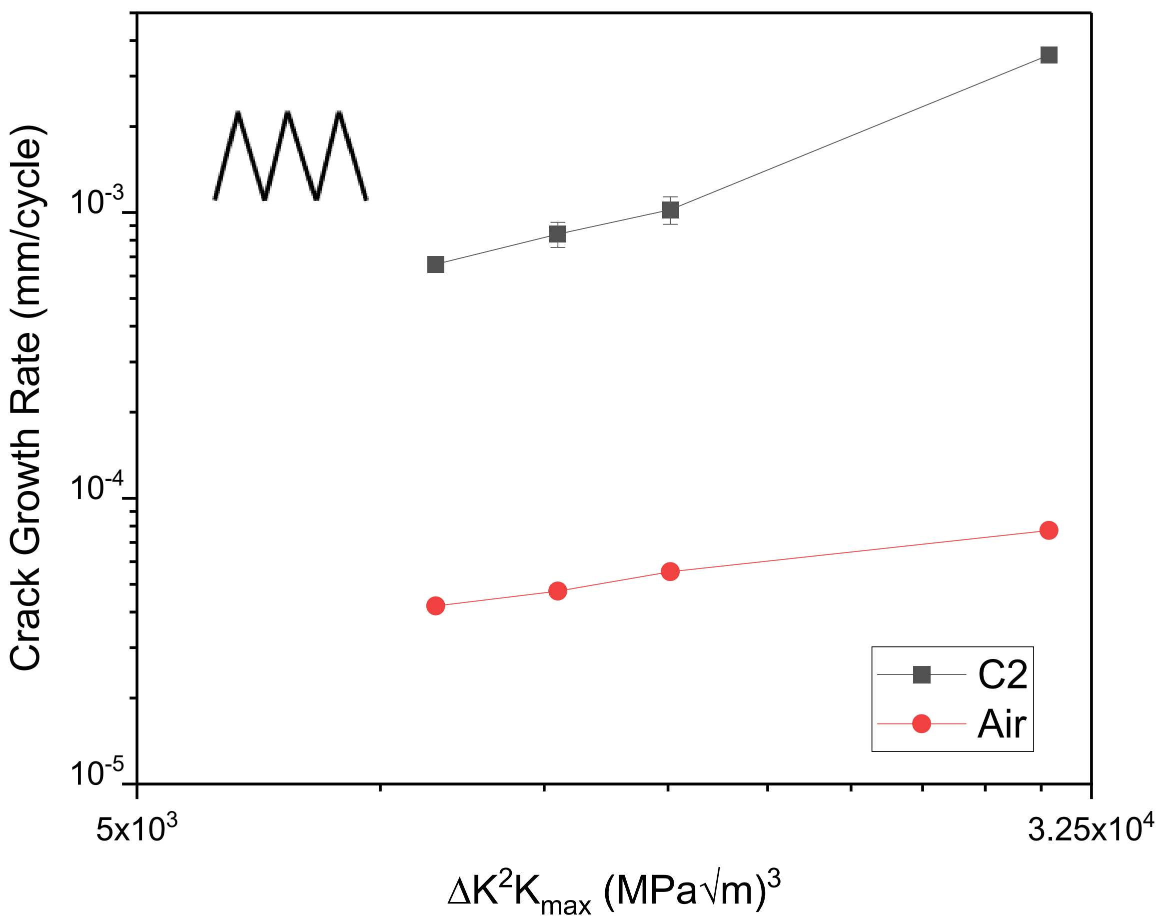


Figure 3- 7 Crack growth rate in air compared to crack growth rate in NNpH solution under constant cyclic amplitude loading. [17]

Therefore, the high crack growth rate in the NNpH environment can be related to the influence of hydrogen on crack growth. [17, 19-20]

3.3.3 Fracture surface analysis

The effect of loading cycles and environment on crack growth is shown on the fracture surface morphology, as shown in Figure 3-8. Region 1 is the initial pre-crack growth. Region 2 shows the crack growth region during the constant amplitude test. Region 3 shows the cleavage fracture in liquid nitrogen. Figure 3-9 (crack growth direction is from left to right) shows the fracture surface for constant amplitude cyclic loading with K_{max} = 33 MPa√m (Test 1) in NNpH environment. The fracture surface observed showed quasi cleavage growth with an abundance of distinct and large striation features, as shown in Figures 3-9a and b. Figure 3-9c shows the crack front at the end of the test. It appears that the large striations are still observed in this region.

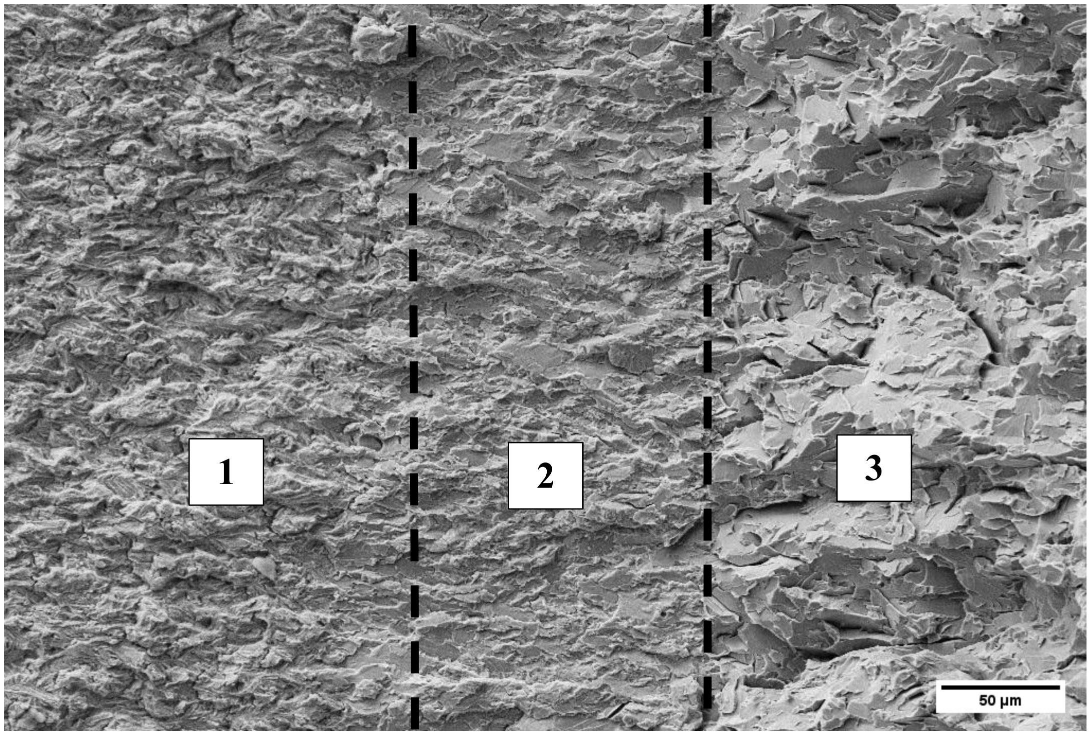
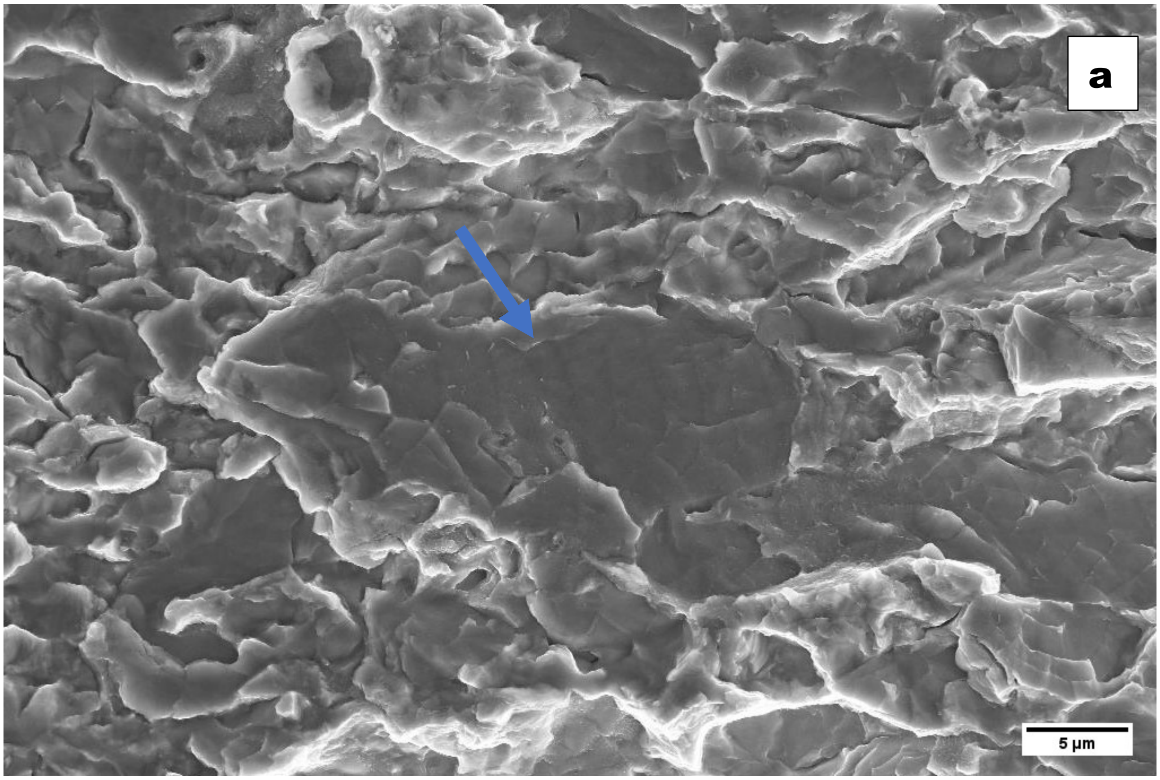
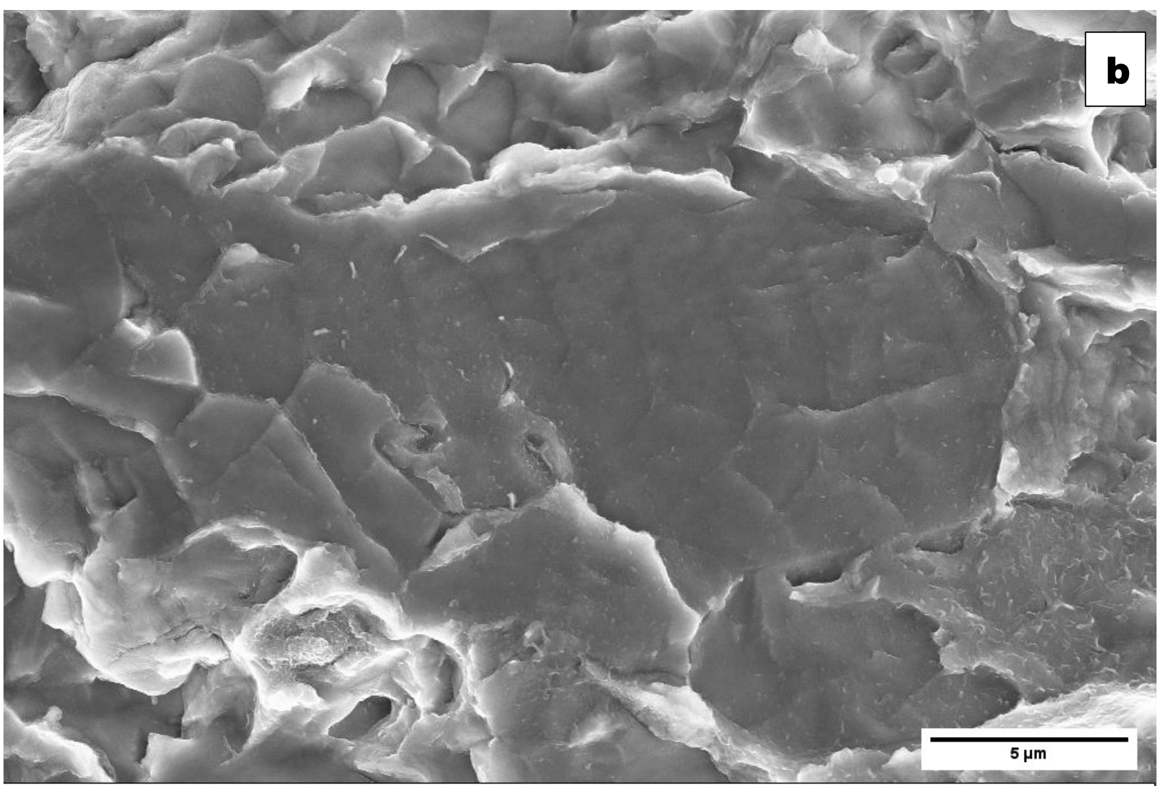


Figure 3- 8 Fracture surface morphology. Region 2 shows the crack growth during the test. Crack direction is left to right.

In Figure 3-10 (crack growth direction is from left to right), the fracture surface morphology of constant amplitude cyclic loading with K_{max} = 34.65 MPa√m (Test 2) can be observed. Similar to Figure 3-9, the fracture surface in Figure 3-10 can be described as quasi-cleavage in nature with large distinct brittle striations on the fracture surface. Figure 3-11 (crack growth direction is from left to right) shows the fracture surface morphology of constant amplitude cyclic loading with K_{max} = 36.3 MPa√m (Test 3). As shown in Figure 3-11a, the fracture surface shows quasi-cleavage fracture with an abundance of striations. Figure 3-11b shows that there is evidence of cracking under this loading condition. In Figure 3-11c, the fracture surface morphology at the crack front at the end of the crack shows some brittle striations.


a
5 µm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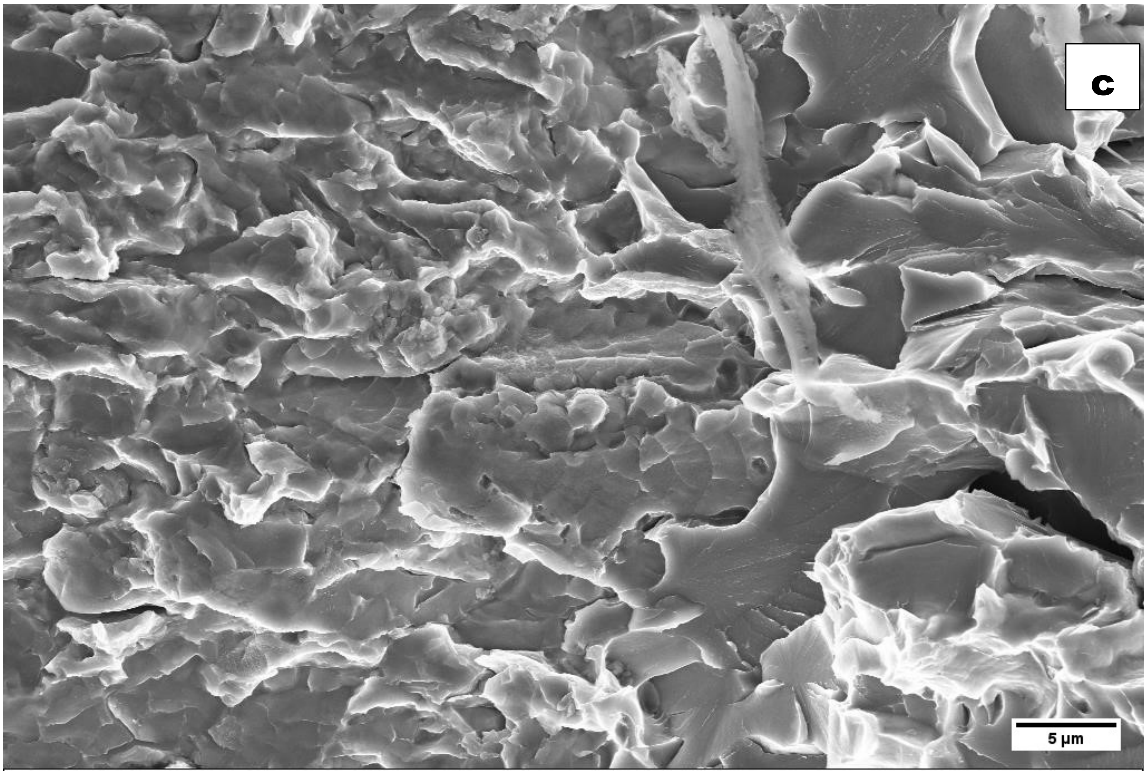


Figure 3- 9 Fracture surface analysis for Test 1 (K_{max} = 33 MPa√m) in NNpH environment. Large striations were observed on the fracture surface. Fracture direction is left to right. [17]

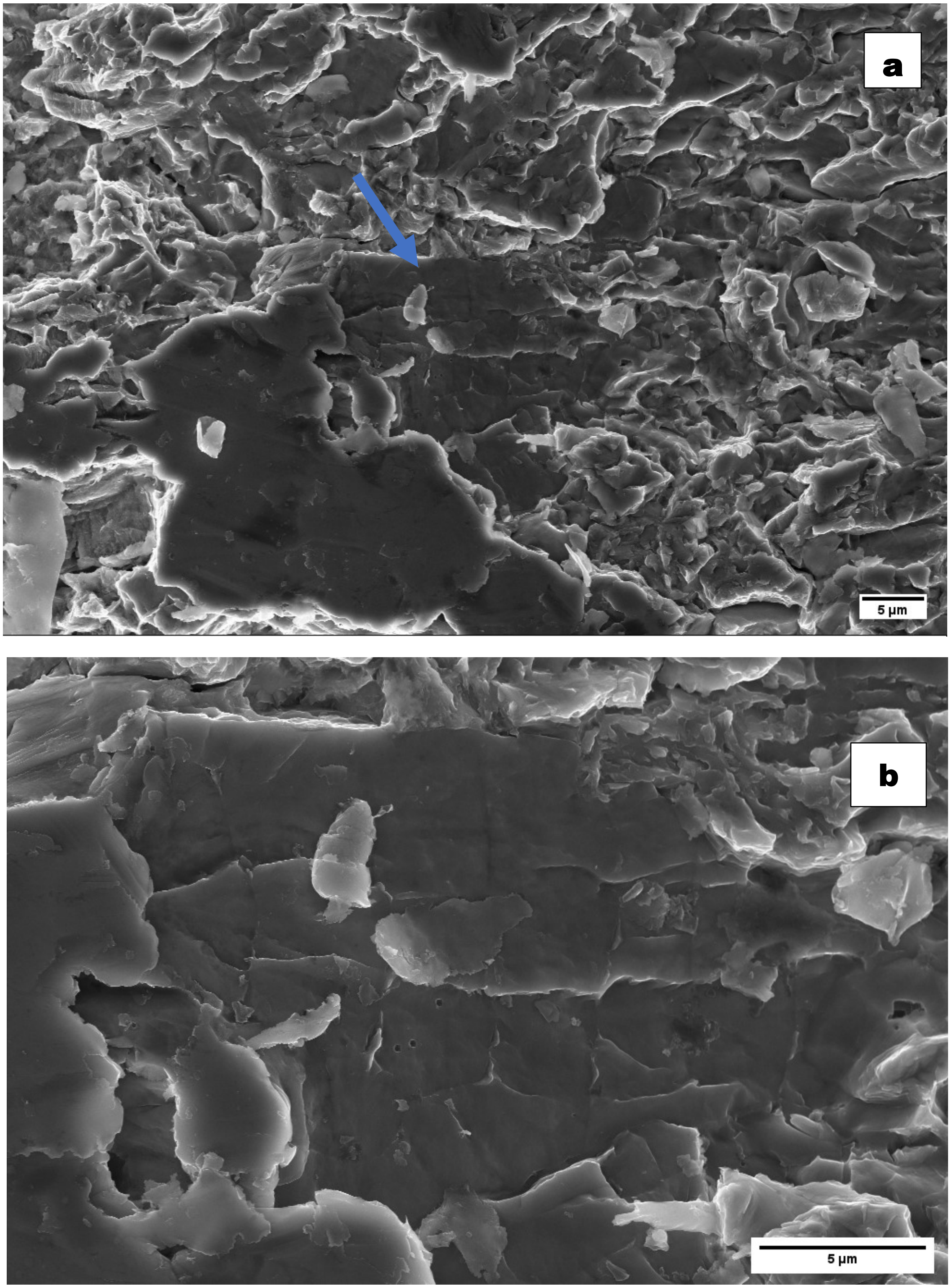
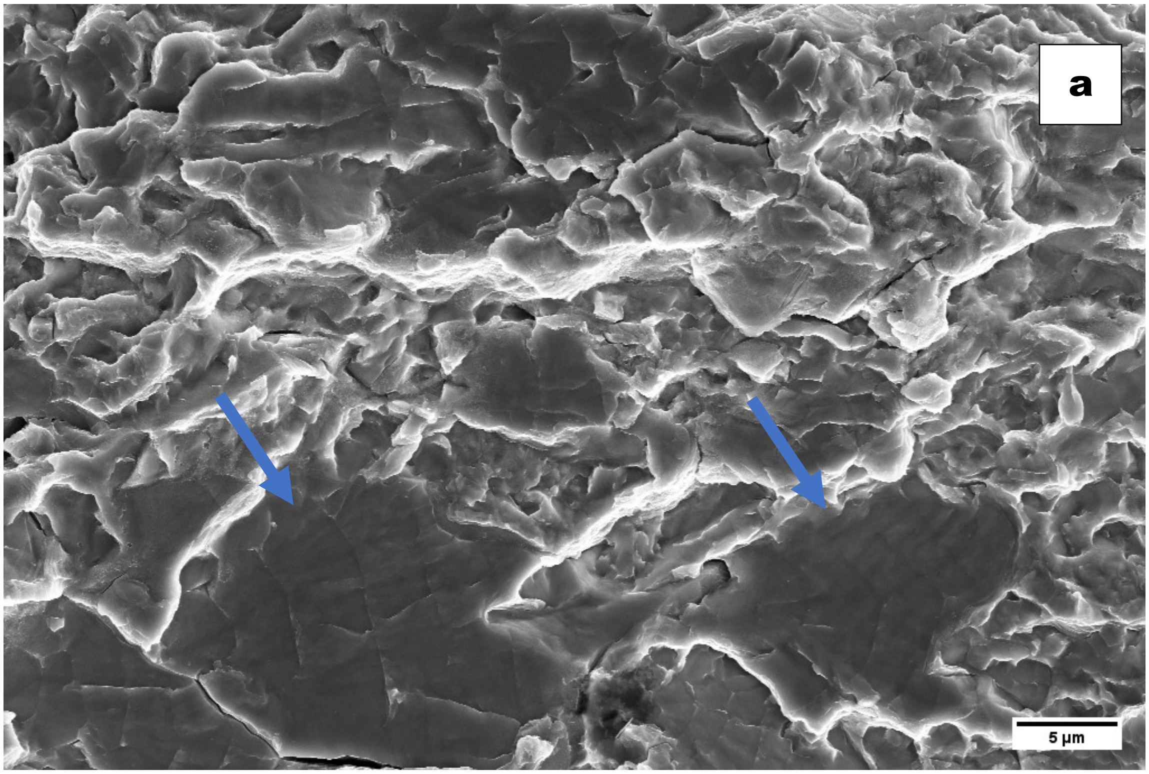


Figure 3- 10 Fracture surface analysis for Test 2 (K_{max} = 34.65 MPa√m) in NNpH environment. Large striations were observed on the fracture surface. Crack direction is left to right. [17]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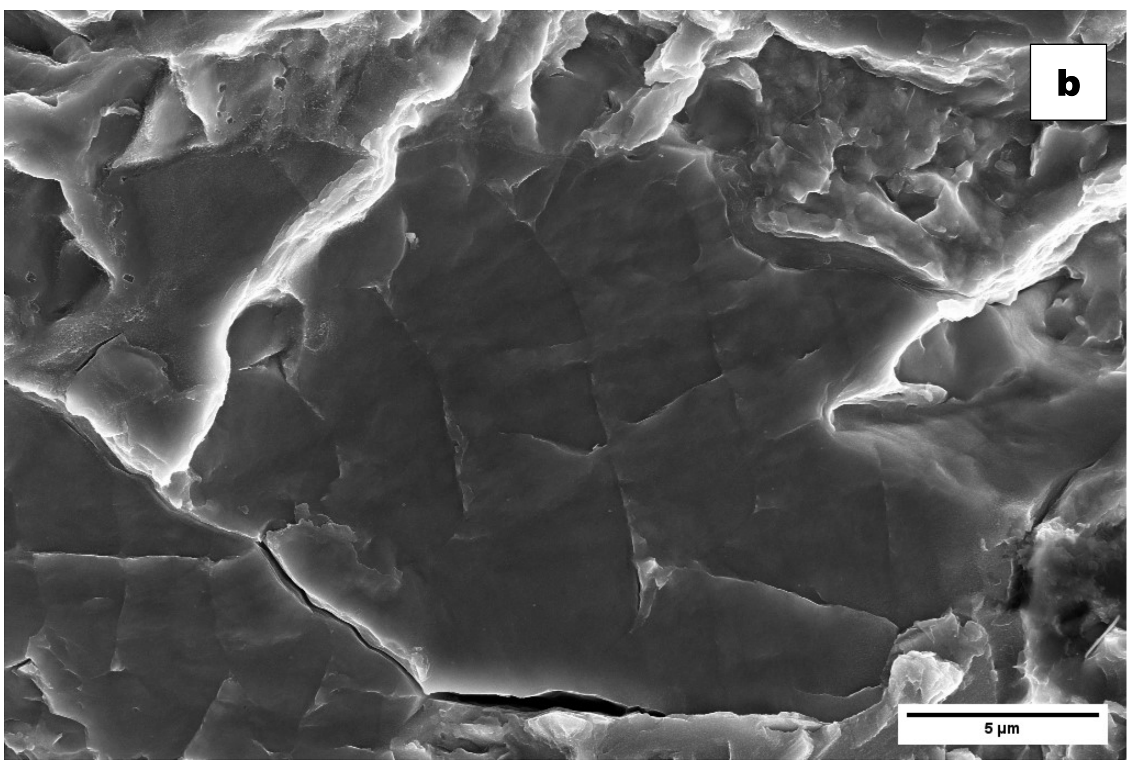
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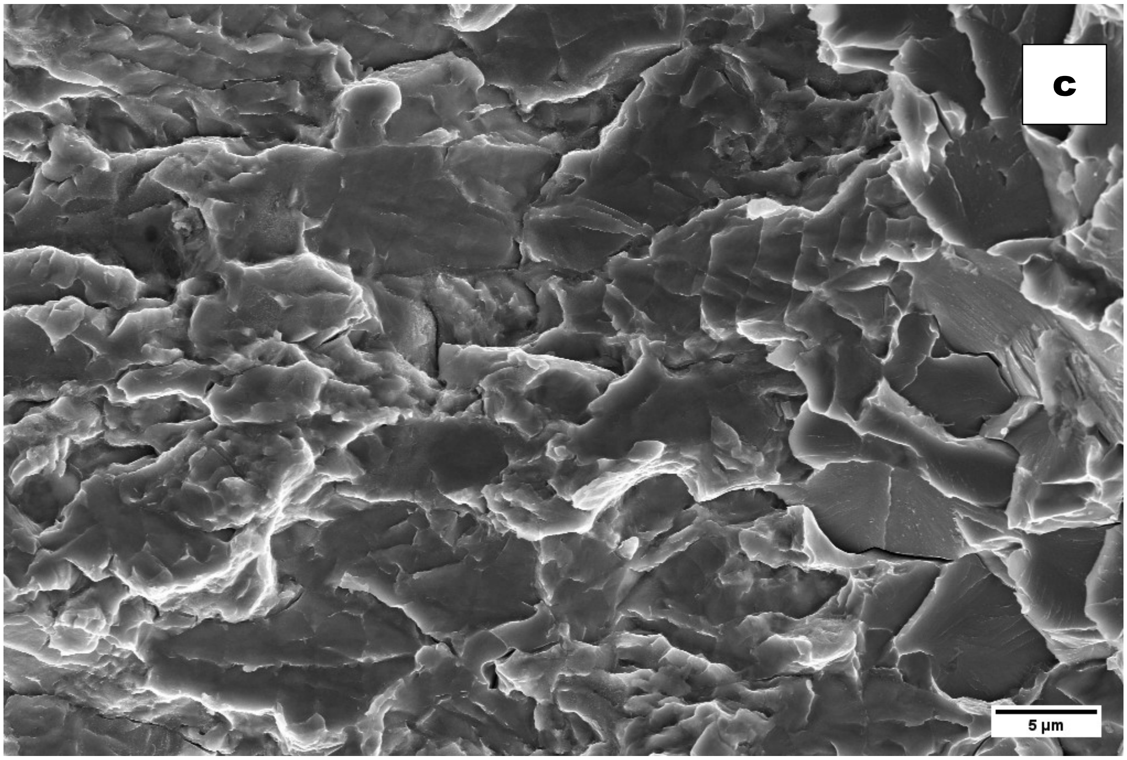

Figure 3- 11: Fracture surface analysis of samples tested under Test 3 (K_{max} = 36.3 MPa√m) in NNpH environment. Large striations were observed on the fracture surface. Crack direction is left to right. [17]

In all the fracture surfaces analyzed for constant amplitude cyclic loading, it appears that an increase in the magnitude of K_{max} can be related to a slight increase in the striation spacing. However, it is difficult to correlate the measured crack growth rate to the striations spacings. In reference [11], it was observed that one loading cycle does not equate to one striation.

Figure 3-12 shows the fracture surface for constant amplitude cyclic loading with K_{max} = 34.65 MPa√m (Test 2) in air. The fracture surface morphology here appears to have ductile striations with relatively equal spacing across the surface. When compared to the fracture surface morphology in Figure 3-10, the drastic difference in the morphology and spacing and distribution of striations can be observed. This observation confirms that there is a significant difference in the mechanism of crack propagation in the two environments. It can also be confirmed that the crack propagation in the NNpH environment is more aggressive, as shown in the crack growth rate in Figure 3-7. The trend observed in Figures 3-7, 10 and 12 confirms the effect of hydrogen in the NNpH environment on crack growth.

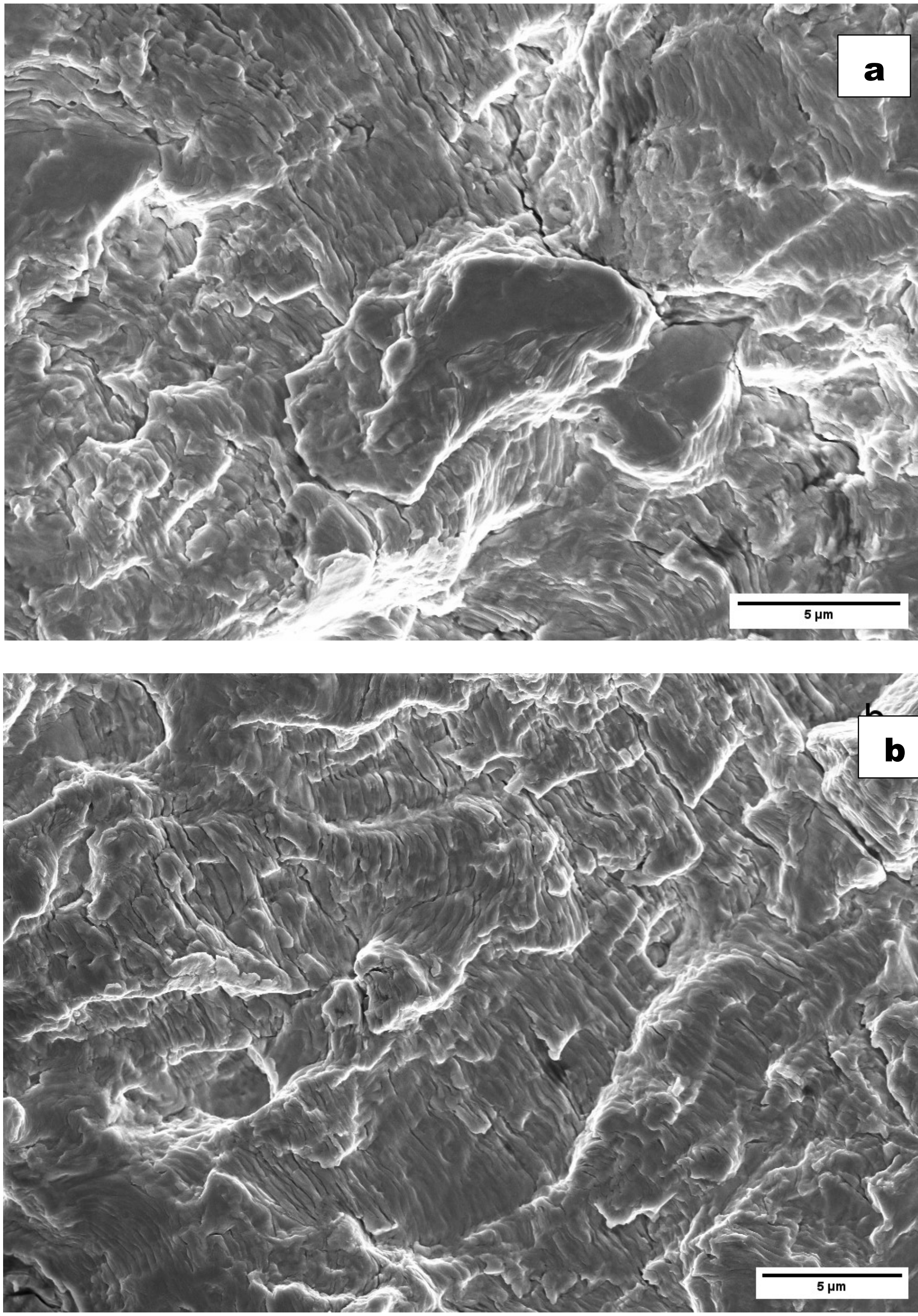

Figure 3- 12 Fracture surface analysis of samples tested under Test 2 (K_{max} = 34.65 MPa√m) in air. Striation spacings appears to be smaller than tests in NNpH environment. Fracture direction is left to right. [17]

3.4 Conclusion

The effect of constant amplitude cyclic loading on crack growth in an NNpH environment has been studied in X65 pipeline steel. Using ΔK and K_{max} that represents an increase in the magnitude of overload in the mean load under frequency that simulates pipeline operation. The following conclusion can be made:

- Crack growth rate under constant amplitude cyclic loading in the NNpH environment is influenced by mechanical factors such as ΔK and K_{max}. Increase in ΔK and K_{max} causes increase in the driving force for crack propagation.
- The effect of constant amplitude cyclic loading in the case of minor cycles shows that the primary mechanism driving crack propagation in such a high R ratio cycle acting alone is dissolution at the crack tip.
- The differences in the crack growth mechanism in air compared to the NNpH environment are confirmed by fracture surface morphology. In the NNpH environment, the fracture surface appears to have large brittle striations, while the fracture surface morphology in air showed small striations that appear to be ductile.
- The effect of constant amplitude cyclic loading in air shows that crack growth is not significantly sensitive to increase in ΔK and K_{max}. This shows the importance of considering the environment in predictive models.

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Chapter 4: Influence of Load Interaction and Hydrogen on Fatigue Crack Growth Behaviour in Pipeline Steels under Type II Mean Load Pressure Fluctuations

4.1 Introduction

Pipelines serve as an energy-efficient and low-cost means of transporting hydrocarbon products. Despite the increase in energy demands, there has been significant increase in debates and concerns about the safety of operation and the effect of pipelines operations on the environment. In fact, discussions on pipelines are a significant topic in social and political debates. [1, 2] This is due to the catastrophic nature of pipeline failure and its effect on the environment, economy and ecosystem. This calls for accurate prediction of crack life to improve pipeline safety and prevent catastrophic failures. Most structural components fail by fatigue in service. [3] Similarly, pipelines are subjected to the fluctuation of internal pressure during operation, causing dynamic loading. The synergistic action of the dynamic loading from the internal pressure fluctuation on susceptible material such as pipeline steel in a corrosive environment has been related to the formation and propagation of cracks. This occurs when the protective coating on the pipe is compromised and the pipe comes in contact with the corrosive environment. It has been observed that cracks in pipeline steel are found under disbonded coating containing solution with pH in the range of 5.5 - 7.5 for near-neutral pH or 9-10 for high pH. Cracking under NNpH environment is a significant threat to pipeline integrity. [4]

Recent findings on cracks in NNpH environment suggest that contrary to the previous belief that these cracks were stress corrosion cracks, it is better to relate the source of stress that drives crack propagation in this system to dynamic loading due to internal pressure fluctuations during operation. [5] Chen *et al.* observed that a propagating crack experienced crack arrest when the loading condition was changed from cyclic to static loading. The crack propagation resumed only when the loading condition was changed back to cyclic from static. This trend observed both in low and high maximum stress intensity suggests that corrosion fatigue is the mechanism driving crack propagation in NNpH environment. [6, 7, 8] As discussed in the previous chapter, most predictive models and studies on crack propagation in pipeline steels are based on constant amplitude cyclic loading. [9] The main challenge with such models is the unrealistic predicted fatigue life for steel pipes in service.

Realistic pipeline operations involve upsets, outages, pressure drops, start/shutdown, change in density of product, and hydrostatic testing. These pressure fluctuations create dynamic loading conditions that can be best explained as variable amplitude loading. [6, 9, 10] This calls for understanding the effect of variable amplitude loading on crack propagation in pipeline steels under NNpH environment. Variable amplitude loading usually involves large and small stress amplitude cycles. The large amplitude cycles can be underload and or overload, while the small amplitude cycles are referred to as minor cycles (also known as ripple load). Typical pressure fluctuations common to oil and gas operations are classified as underload, mean load and overload. [11]

In variable amplitude loading, the sequence of underload and overload, as well as the interaction between all the loading cycles, can accelerate or decelerate crack propagation. [12] Figure 4-1 shows the typical response expected for growth in variable and constant amplitude loading as suggested in references based on various tests. [13, 14] It has been established that underload variable amplitude causes acceleration of crack growth. One factor causing increase in crack growth in underload variable amplitude loading is related to load interaction between the large underload and minor cycles. [15, 16] In overload variable amplitude loading, the load interaction between the large overload cycle and the minor cycles causes retardation. In references [17-19], overload caused retardation of crack growth and it was observed that the retardation effect of overload increased with increase in the magnitude of overload. These effects need quantitative evaluation for NNpH SCC.

Mean load pressure fluctuation presents a more complex variable amplitude loading condition by combining underload, overload and minor cycles. By applying load sequence, mean load can be expressed as a Type I or Type II as shown in Figure 4-1. Type I is mean load with overload followed by an underload and Type II is underload followed by an overload. It is expected (as shown in Figure 4-1) that mean load can achieve retardation of crack growth. [20] Figure 4-1 suggests that these load sequences can affect the extent or measure of retardation obtained in mean load.

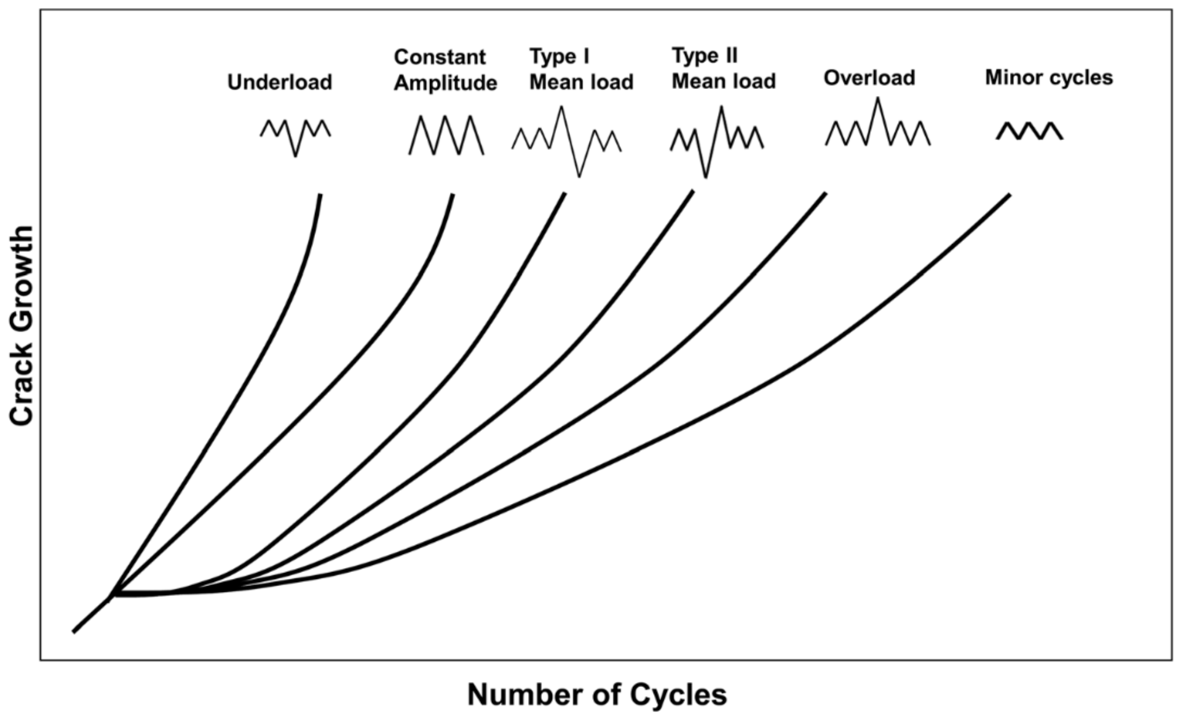


Figure 4- 1 Effect of constant and variable amplitude loading on crack growth in some structural materials. [21]

It should be noted that Figure 4-1 is based on crack growth in aluminium alloys with the underload in compression. [22] Currently, there is no agreement on the effect of mean load on crack growth particularly in steel. Some researchers have observed a similar trend as shown in Figure 4-1, where retardation was observed when Type II mean load was applied [13,14,23-25] while others observed that Type I mean load achieved more retardation. [26, 27] Hence, there is a need for further studies on the effect of mean load on crack growth. For the following reasons:

- To understand the effect of Type I and Type II mean load on crack propagation under conditions that simulate pipeline operation.
- To understand the effect of the magnitude of overload in mean load on crack propagation.
- To be able to leverage mean load effects in reducing crack growth caused by pipeline operations that causes crack acceleration, such as depressurization.

Generally, there is a need for further study on variable amplitude loading and its effect on crack growth in steel pipelines regarding unique conditions for pipeline operations such as NNpH environment and low frequency. [21] Recent works by Yu *et al.* studied the effect of underload pressure fluctuation on crack growth in steel pipelines. [9, 10] Those studies showed that underload pressure fluctuations caused an increase in crack propagation (about five times) compared to constant amplitude loading in steel pipelines under NNpH environment. [6, 9, 10] This suggests that underload is a severe and aggressive form of variable amplitude in pipeline steels.

In this work, the focus will be on Type II mean load pressure fluctuations (UL+OL+MC) and its effect on crack growth in pipeline steel under NNpH environment. The study aimed to understand the effects to be able to attenuate the acceleration effect of pipeline operations (such as depressurization) on crack growth by applying a subsequent overload. Overall, by applying realistic loading conditions for pipeline operations, a better understanding of crack growth behaviour in mean load pressure fluctuations can be achieved and this will ensure accurate life prediction of cracks and improve pipeline integrity.

4.2 Experimental Procedure

4.2.1 Material

The material used in this study was an X-65 steel pipeline that was known to be susceptible to NNpH SCC in service. The chemical composition (wt.%) of the X-65 steel is as follows: C: 0.12, Mn: 1.5, P: 0.017, S: 0.0046, Si: 0.26, V: 0.01, Cr: 0.005, Nb:0.049 Fe: Balance. Compact Tension (CT) specimens were machined from a section of the pipe. To simulate axial/longitudinal cracks on pipes, the notch was cut in the longitudinal direction of the pipe. Sample preparation involved grinding of each face up to the 600 grade of grinding papers. The samples were cleaned in ethanol and fatigue pre-cracking was carried out to initiate a crack of 2.5 mm length based on ASTM standard E647. It was ensured that the maximum difference in crack length was 0.2 mm. The crack tip was marked with an indentation. A Scanning Electron Microscope (SEM) was used to determine the initial crack length indicated by the position of the indentation.

4.2.2 Test Environment

The composition of the NNpH environment used in this study (also known as C2 solution) contains (g/L): KCl:0.0035, $NaHCO_3$:0.0195, $CaCl_2.H_2O$:0.0255, $MgSO_4.7H_2O$:0.0274, $CaCO_3$:0.0606. The solution was prepared and bubbled with 5% CO_2 + 95% N_2 continuously for the test duration.

4.2.3 Loading condition for variable amplitude tests

The waveforms applied for Type II mean load (UL+OL+MC) and underload test (UL+MC) are shown in Figures 4-2a and b, respectively. To study the retardation effect of overload in mean load, different magnitudes of overload from 3-50% overload were applied. Table 4-1 lists the Type

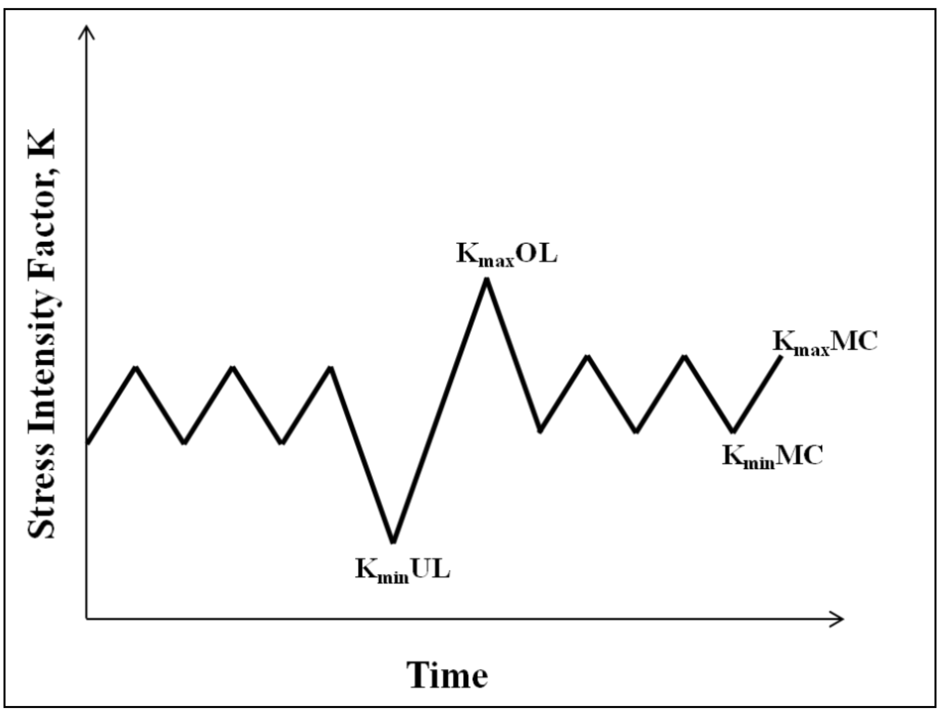
II mean load tests as Test 1-6. In all the tests maximum stress intensity factor of underload and minor cycle (K_{maxUL} and K_{maxMC}) were 33 MPa√m. This stress intensity factor corresponded to typical long but shallow cracks observed in pipes under NNpH environment with crack depth ≈25% of the wall thickness, crack length was ten times the depth and corresponding load about 75% of Specified Minimum Yield Strength (SYMS).

The minimum stress intensity factor for underload and minor cycle (K_{minUL} and K_{minMC}) were 16.5 and 29.7 MPa√m, respectively. This achieved R ratios (K_{min}/K_{max}) of 0.5 for underload and 0.9 for minor cycles. For the minor cycles, loading a frequency of 5×10^{-3} Hz was applied. The loading rate of underload + overload cycle is 0.01kN/s. In each load sequence, underload followed by an overload corresponding to 3%, 5%, 10%, 20%, 30% or 50% increase in K_{max} was applied. This corresponded to overload with stress intensity factor (K_{maxOL}) of 33.9, 34.6, 36.3, 39.6, 42.9 and 49.5 MPa√m, respectively. In each block of the mean load waveform, 300 minor cycles were applied (n=300). The duration of test for 70 blocks was between 45-47 days for each test. For mean load test with UL+50%OL+MC, the test was conducted for 35 blocks. Figure 4-2b shows the waveform for UL+MC. The loading parameters are listed in Table 4-1 as Test 7. Similar variable amplitude loading tests were conducted in air to understand the effect of the NNpH environment on crack growth.

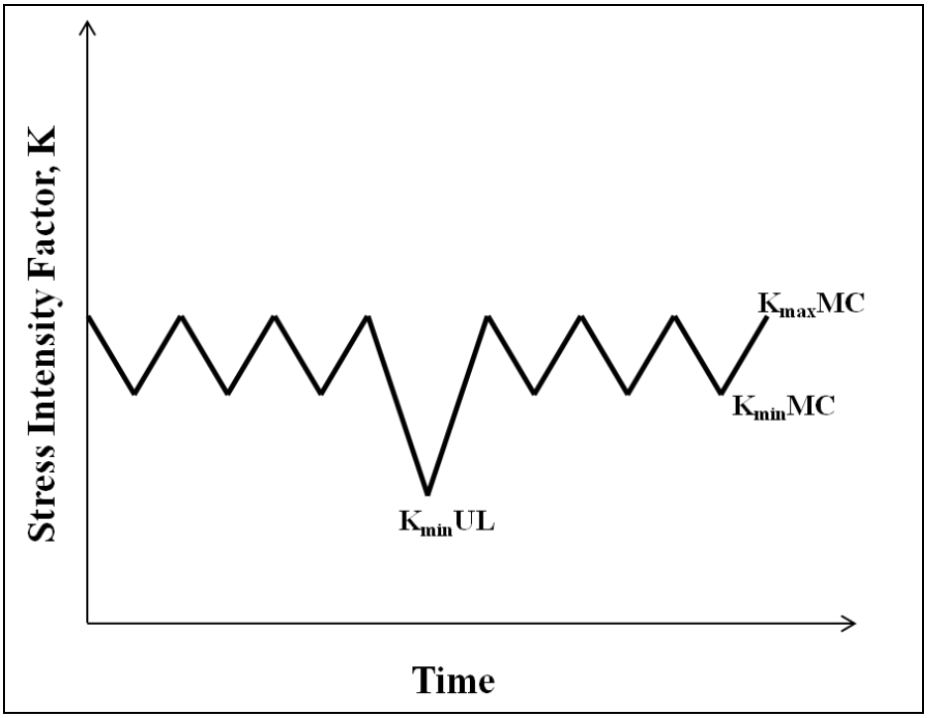
At the end of the test, the samples were cleaned in ethanol and preserved in a dry environment. SEM images were taken using the indentation mark as a reference to measure crack growth. The crack growth rate for the variable amplitude loading was measured as mm/block. This was obtained by dividing the measured crack growth by the number of blocks applied.

Table 4- 1 Loading parameters for variable amplitude loading.

Test	Loading	K_{maxOL} (MPa√m)	K_{maxUL} (MPa√m)	K_{minUL} (MPa√m)	K_{maxMC} (MPa√m)	K_{minMC} (MPa√m)
1	UL+3%OL+MC	33.99	33	16.5	33	29.7
2	UL+5%OL+MC	34.65	33	16.5	33	29.7
3	UL+10%OL+MC	36.3	33	16.5	33	29.7
4	UL+20%OL+MC	39.6	33	16.5	33	29.7
5	UL+30%OL+MC	42.9	33	16.5	33	29.7
6	UL+50%OL+MC	49.6	33	16.5	33	29.7
7	UL+MC	-	33	16.5	33	29.7



(a)



(b)

Figure 4- 2 Waveforms showing variable amplitude loading for (a) mean load (b) underload. [21]

4.3 Results

4.3.1 Variable amplitude loading – Underload + Overload + Minor cycles

Figure 4-3 shows the effect of Type II mean load on crack growth in pipeline steel under NNpH environment. As shown in Figure 4-3, the crack growth rate is expressed with respect to the combined factor. The result shows the effect of Type II mean load with various magnitudes of overload (ranging 3-50% OL) on the crack growth rate, thereby reflecting the effect of load sequence and load interaction on crack growth rate under Type II mean load. The results in Figure 4-3 shows that increasing the magnitude of overload greater than 5% in Type II mean load will increase the crack growth rate.

The results in Figure 4-3 also shows Type II mean load crack growth rate relative to UL+MC. The results in Figure 4-3 indicate that Type II mean load with magnitude of OL <10% can achieve retardation of crack growth compared to UL+MC.

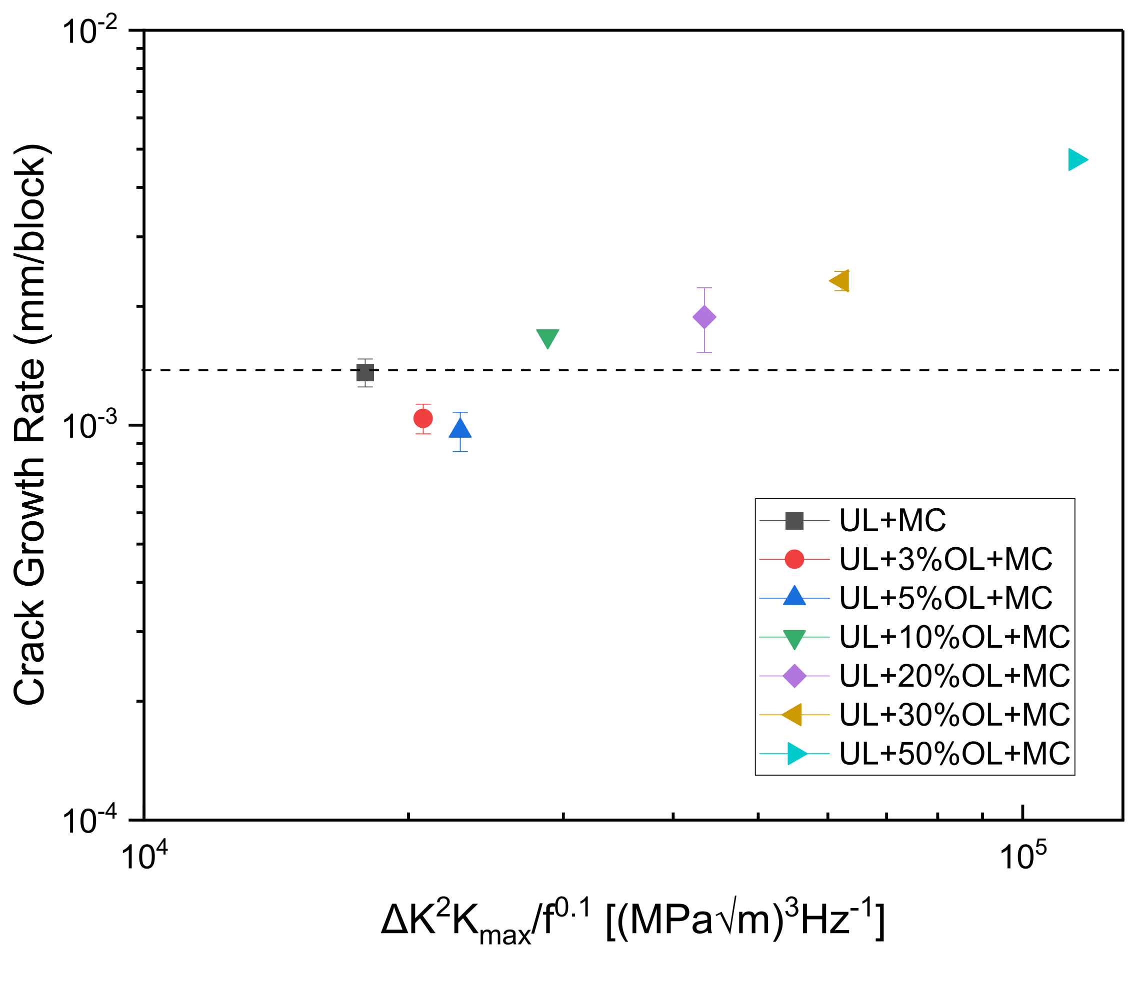


Figure 4- 3 Effect of overload cycle on crack growth rate in UL+OL+MC loading schemes. The dashed line indicates crack growth in UL+MC. [21]

Increase in the magnitude of OL >10% caused a significant increase in crack growth rate. This result implies that a higher magnitude of overload favours crack growth in NNpH environment. Optimum retardation of crack growth under Type II mean load was observed at UL+5%OL+MC.

In Figure 4-4, the response (in terms of crack growth rate) to an increase in the number of minor cycles in Type II mean load is shown. Type II mean load with UL+5%OL+MC was investigated with increase in the number of minor cycles (n varied from 10 to 697).

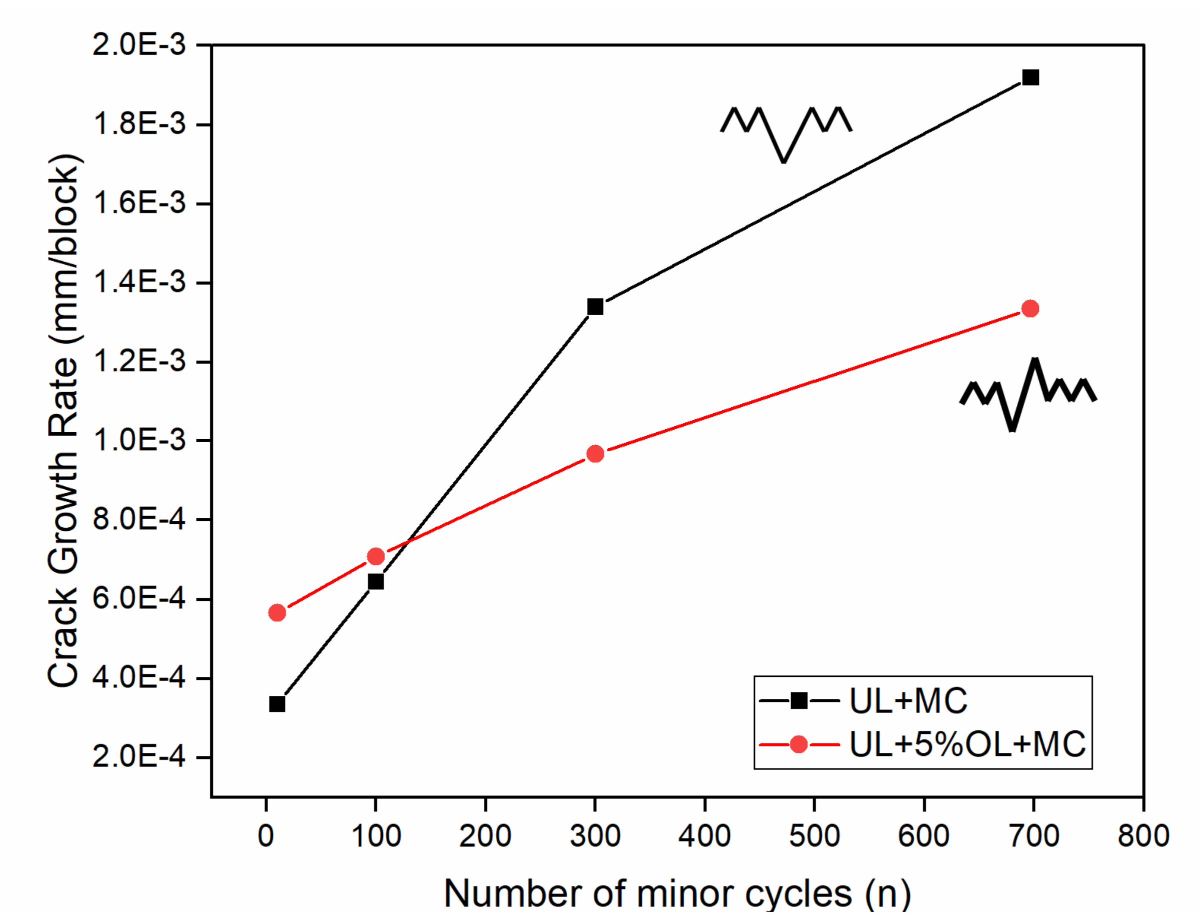


Figure 4- 4 Effect of the number of minor cycles on crack growth rate in mean load (UL+5%OL+MC) compared to UL+MC. [21]

As shown in Figure 4-4, the crack growth rate increases with increase in the number of minor cycles. However, compared to UL+MC, crack growth rate in UL+5%OL+MC is lower if the number of minor cycles is greater than 150.

4.3.2 Effect of environment

Figure 4-5 shows crack growth behaviour under UL+OL+MC in air. As shown in Figure 4-5, crack propagation in air was not sensitive to an increase in the magnitude of overload in mean load. As discussed in section 4.3.1, Figure 4-3 shows that the reverse is the case in NNpH environment. Interestingly, the optimum reduction in crack growth rate was observed at 5%OL in both air and NNpH environment from Figure 4-3 and Figure 4-5. This suggests that the influence of hydrogen on crack growth in mean load is not significant at UL+5%OL+MC.

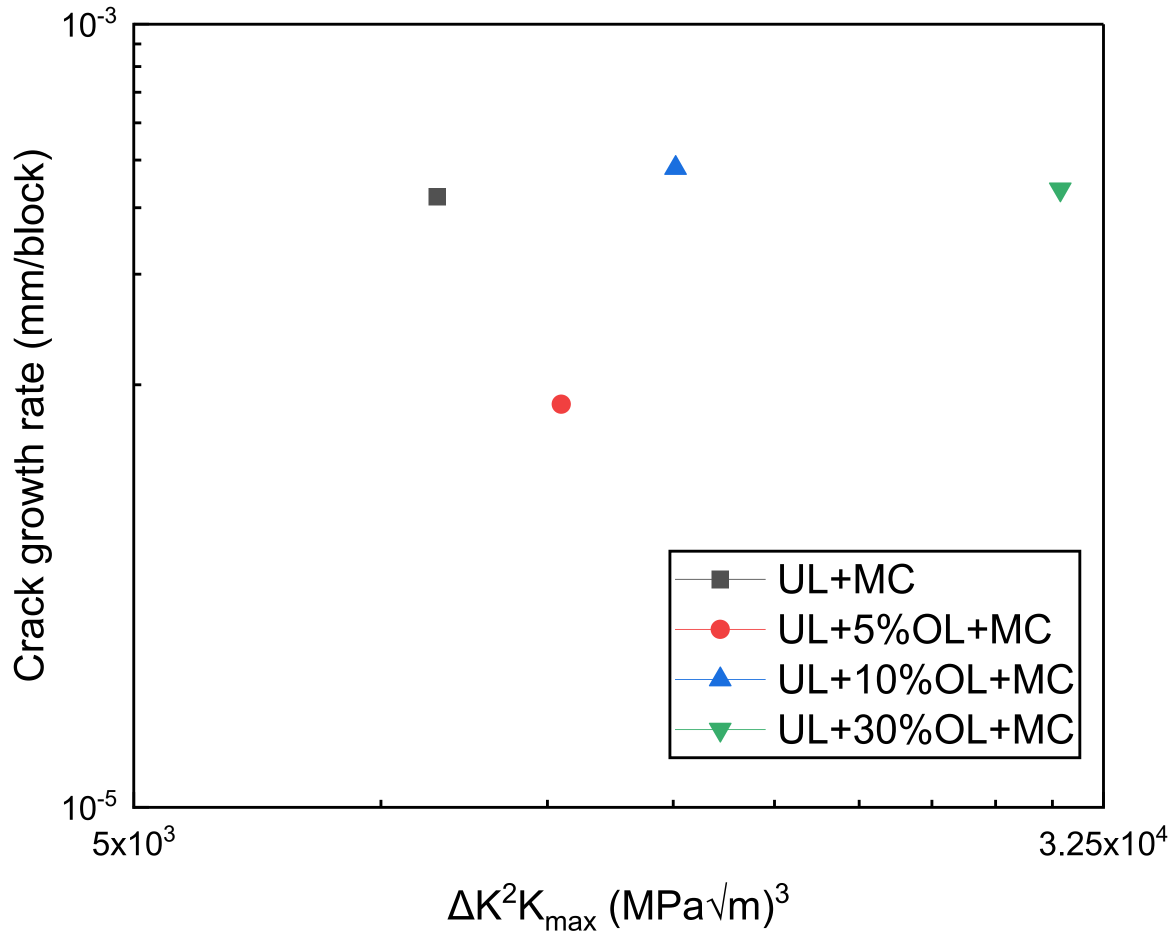
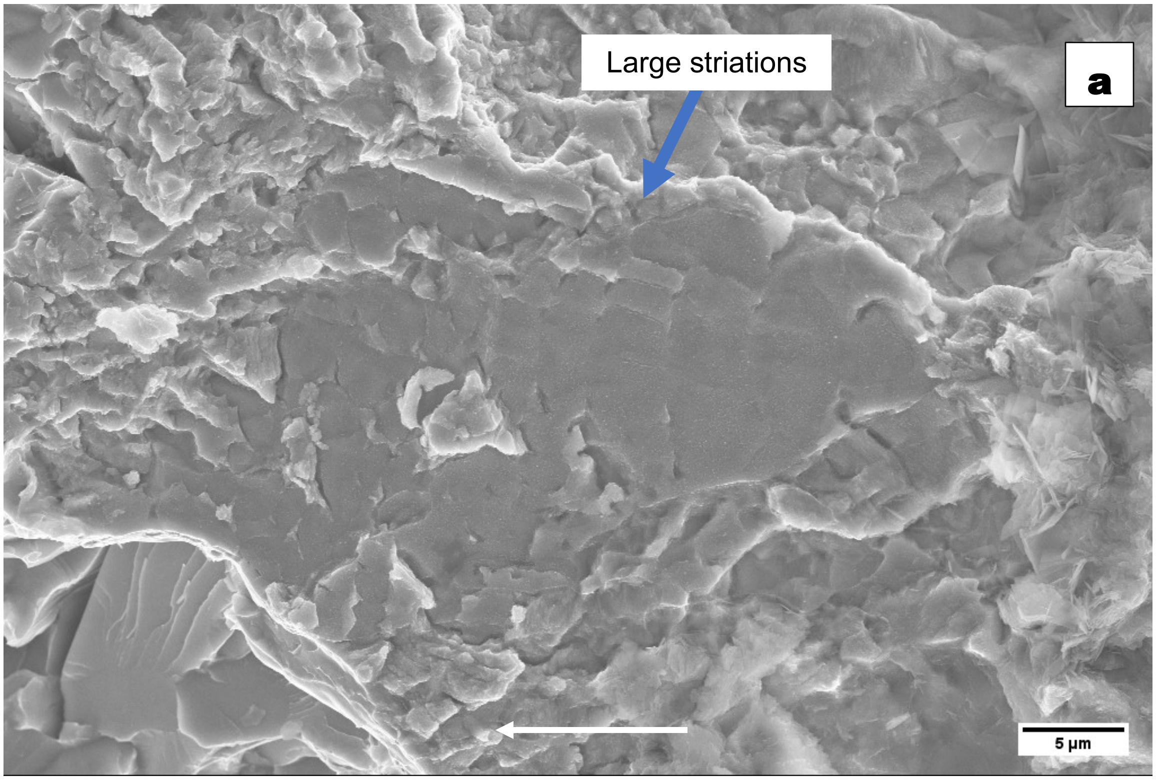


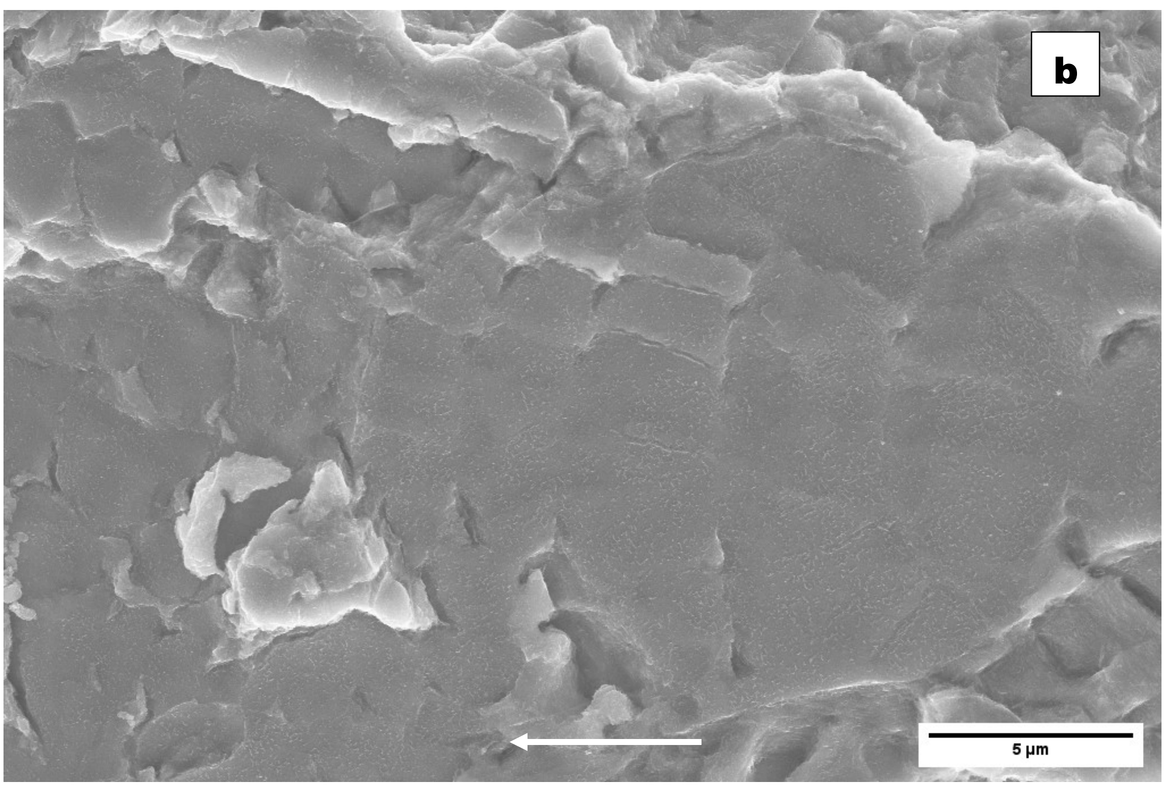
Figure 4- 5 Crack growth rate in air under Type I mean load (UL+OL+MC). [21]

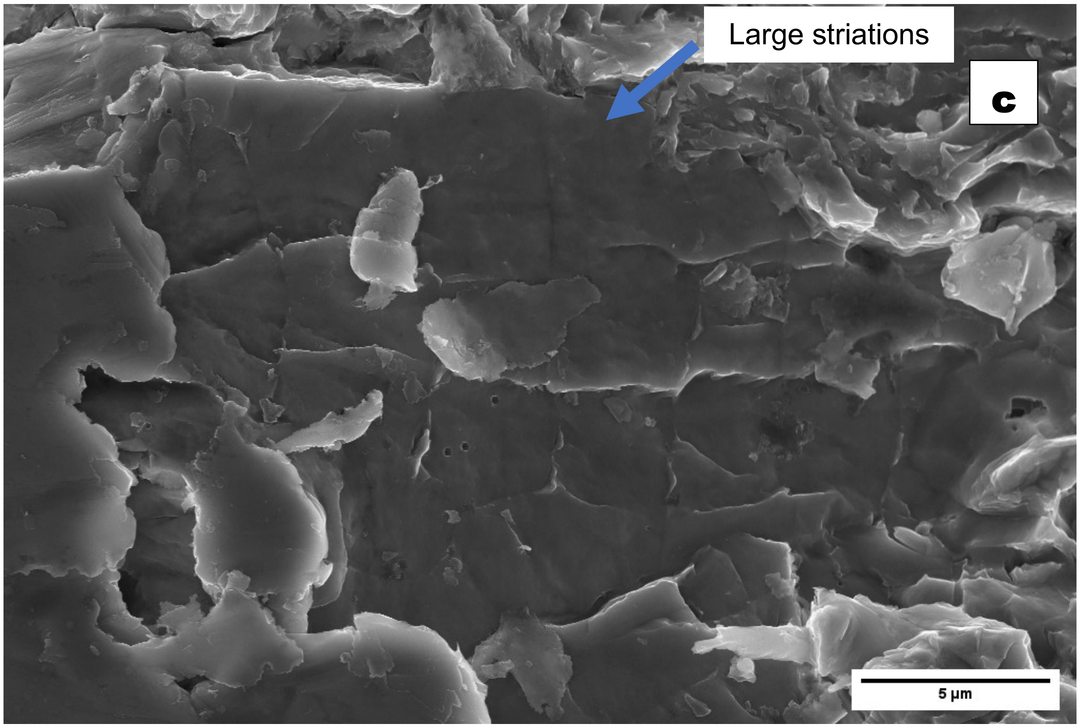
4.3.3 Fracture Surface Analysis

Figure 4-6 shows the fracture surface analysis for UL+5%OL+MC in NNpH environment (white arrow indicates crack growth direction). As shown in Figures 4-6a and b, the fracture surface appears to be quasi-cleavage with some striations. It can be observed that the striations on the fracture surface are different in morphology compared to constant amplitude loading (shown here as Figure

4-6c for comparison) as reported in chapter 3. The large striations in Figures 4-6a and b can be related to large loading cycles and it can be observed that there appear to be two regions in the large striation. Figure 4-6d shows the crack front region at the end of the crack. There are mini-striations at these regions, which was not observed in the constant amplitude loading fracture surface. The existence of mini striations was also reported by Yu *et al.* in the case of UL+MC. [7, 9] These fractographic features have been related to the influence of minor cycles on crack growth in variable amplitude loading. Another possible explanation for these mini striations could be based on their appearance. They could be described as ductile striations marking the transition of crack growth from brittle to ductile. Figure 4-7 shows the fracture surface morphology of UL+5%OL+MC in air (white arrow indicates crack growth direction). Figures 4-7a and b show a similar trend of large striations observed in Figures 4-6a and b. Also, mini-striations were observed, as shown in Figure 4-7c. Figure 4-8 shows the fracture surface analysis for UL+10%OL+MC in air (black arrow indicates crack growth direction). The morphology seems similar to Figure 4-7 but it appears that the mini striations are more widely distributed across the fracture surface. The existence of mini striation on the fracture surface in air suggests that they can be related to the mechanical loading conditions.


Large striations
a
5 µm


b
5 µm



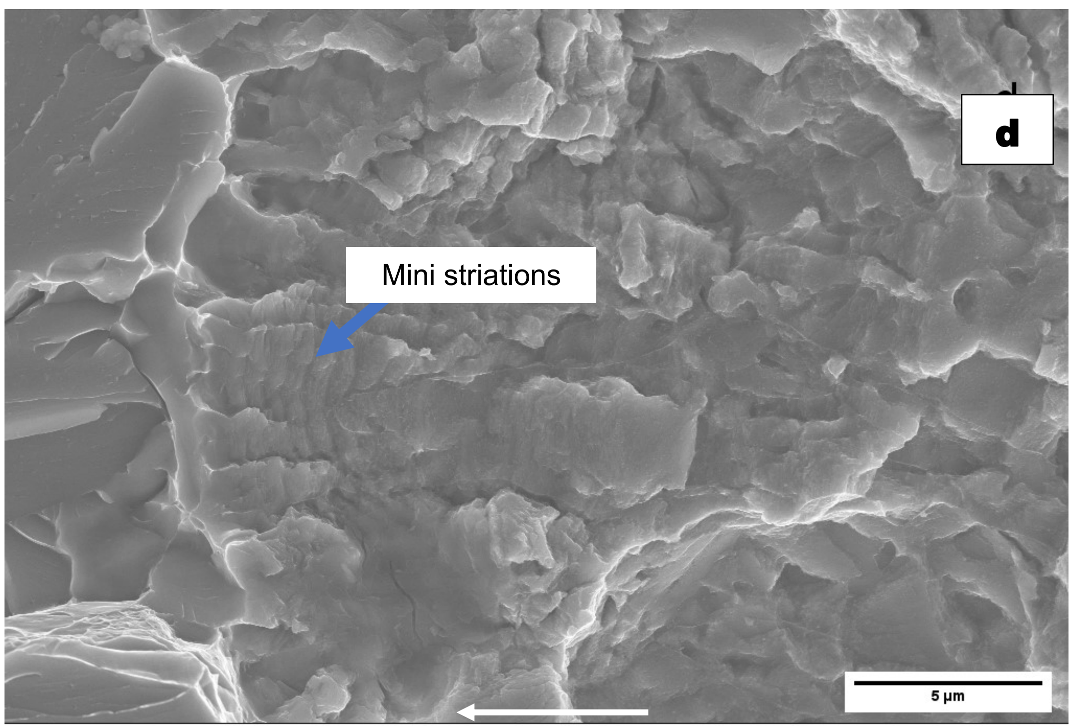
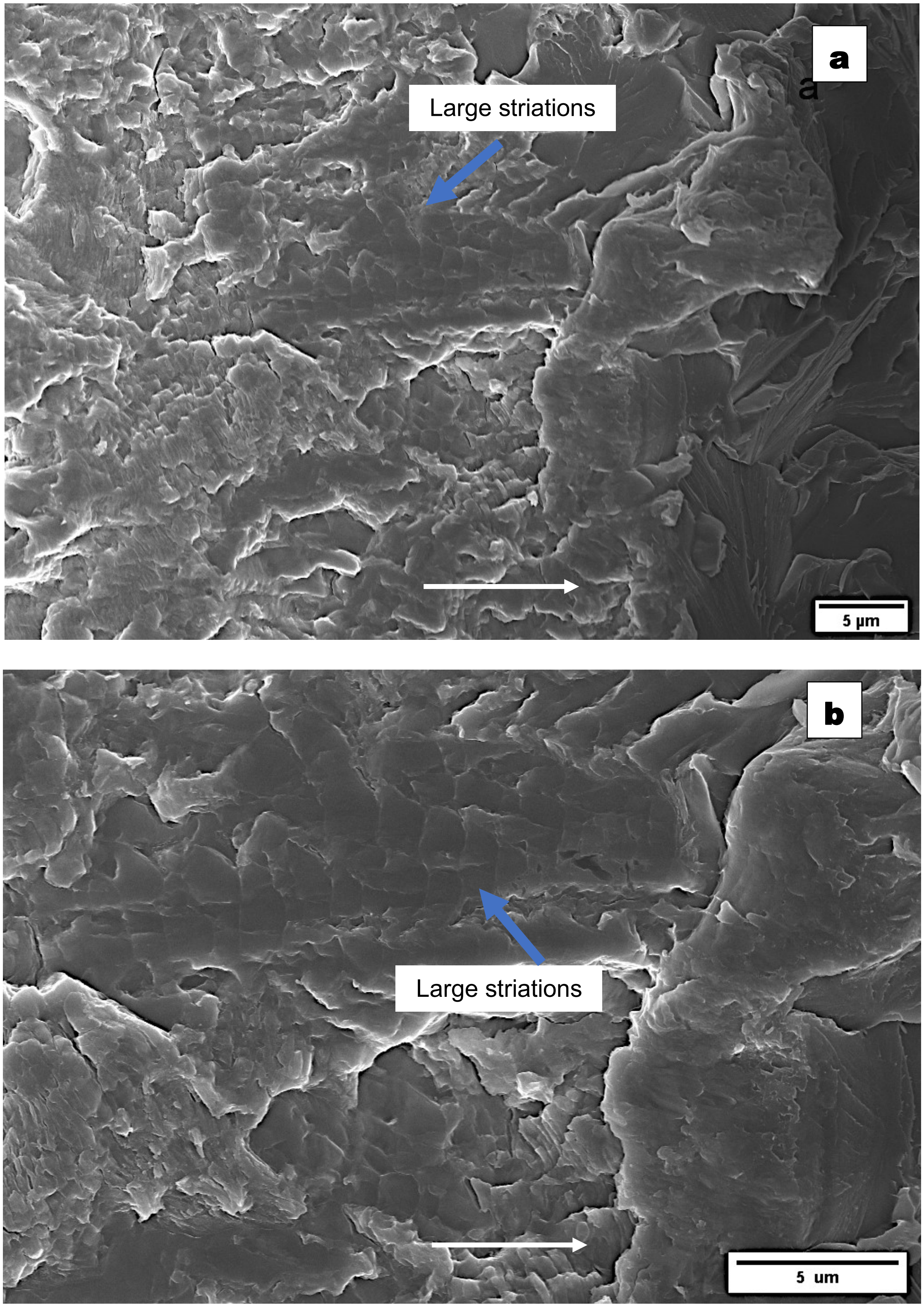


Figure 4- 6 Fracture surface analysis for mean load UL+5%OL+MC in NNpH. Large striations as well as mini-striations were observed on the fracture surface. White arrow shows crack growth direction. [21]


a
Large striations
a
5 μm
b
Large striations
5 um

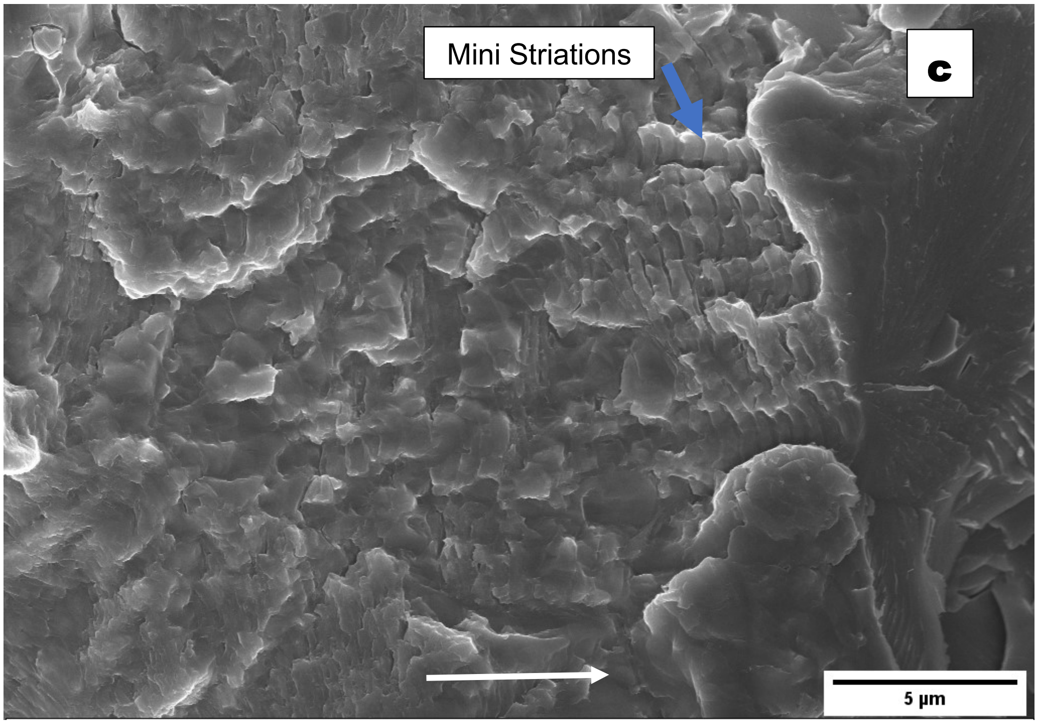


Figure 4-7	Fracture surface analysis of samples tested under UL+5%OL+MC in air. Large striations as well as mini-striations were observed on the fracture surface. White arrows show crack growth direction. [21]

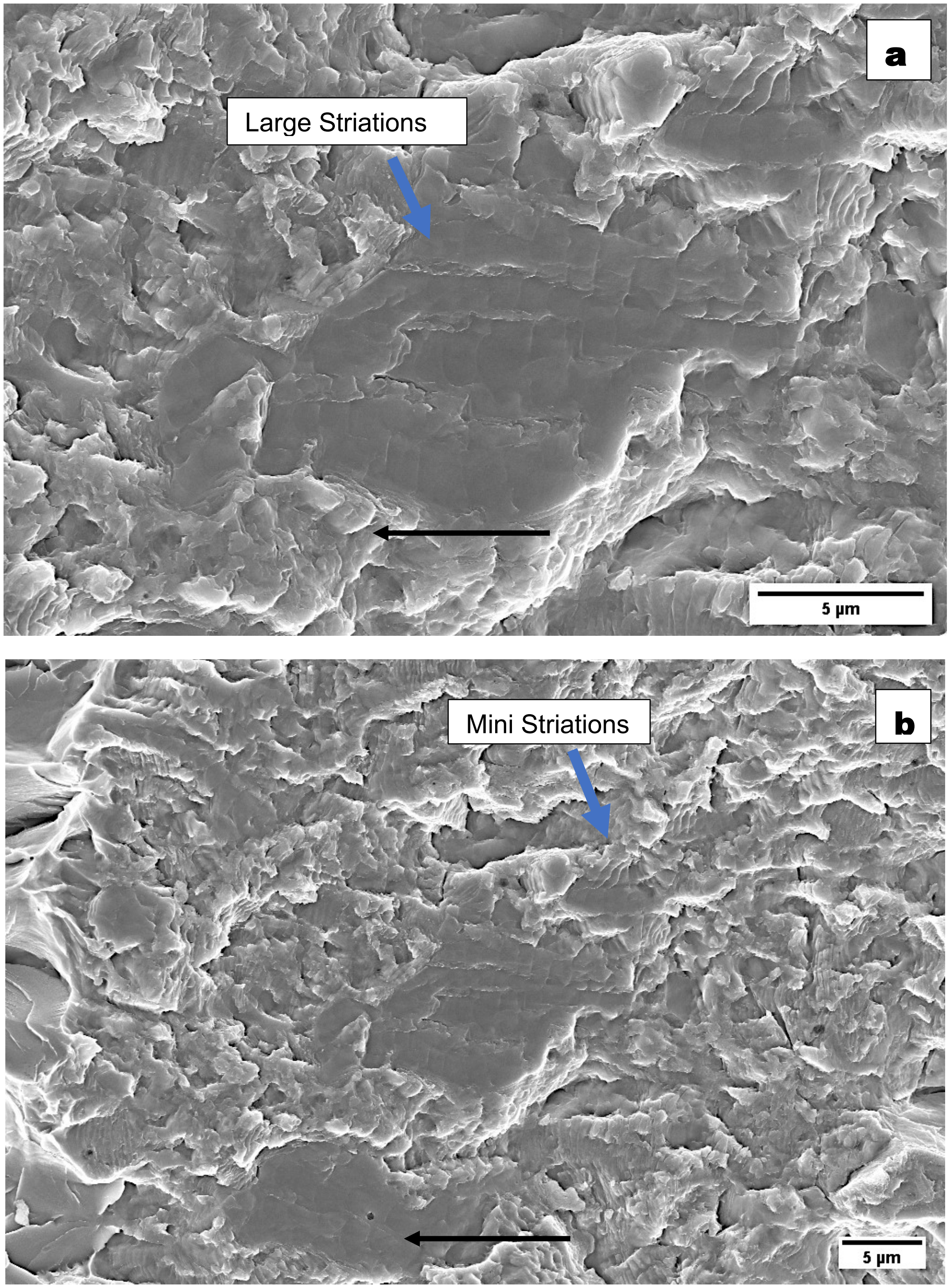


Figure 4- 8 Fracture surface analysis of samples tested under UL+10%OL+MC in air. Large striations as well as mini-striations were observed on the fracture surface. Black arrows show cracking direction. [21]

4.4 Discussion of Results

4.4.1 Effect of load interaction on crack propagation

Although most predictive models for crack growth are based on constant amplitude loading, the results obtained so far have shown the importance of applying variable amplitude considerations to crack growth predictions. This study has shown that crack growth behaviour in mean load is more complex and cannot be simply defined by constant amplitude crack growth. To confirm this, the predicted crack growth rate can be compared with the measured crack growth rate for the mean load. The predicted crack growth rate in mean load can be obtained from the sum of measured crack growth of UL+OL (constant amplitude) cycle and crack growth for n number of MC as given in Equation 1 below. Prior to this research, the actual crack growth rate for minor cycle loading in NNpH environment has not been considered in the predicted crack growth rate. [10] As discussed in Chapter 3, the test result shows a crack growth rate of 1.7×10^{-7} mm/cycle for minor cycles.

$$\left(\frac{da}{dN}\right)_{\boldsymbol{UL+OL+MC\ predicted}} = \left(\frac{da}{dN}\right)_{\boldsymbol{UL+OL}} + n\left(\frac{da}{dN}\right)_{\boldsymbol{MC}} \tag{1}$$

Figure 4-9 compares predicted crack growth rate for Type II mean load and UL+MC to measured crack growth rate. Figure 4-9 shows that there is significant agreement between predicted and measured crack growth rate for UL +5%OL+MC in crack growth rate. Figure 4-9 shows that optimum reduction was observed in both measured and predicted growth rates at UL+5%OL+MC. For UL+10%OL+MC, the measured crack growth rate is slightly higher. However, as %OL in Type II mean load increased above 10%OL, the measured crack growth rate was reduced compared to the predicted rate. It appears that the retardation observed at >10%OL is unrealistic since the crack growth rate is still higher than in UL+MC. These results show that an increase in the magnitude of overload in Type II mean load can be deleterious to fatigue life of cracks in NNpH conditions. This also suggests that the fatigue life of cracks under Type II mean load variable amplitude loading cannot be predicted accurately by constant amplitude models. Therefore, it is important to consider the effect of load interaction in predictive models.

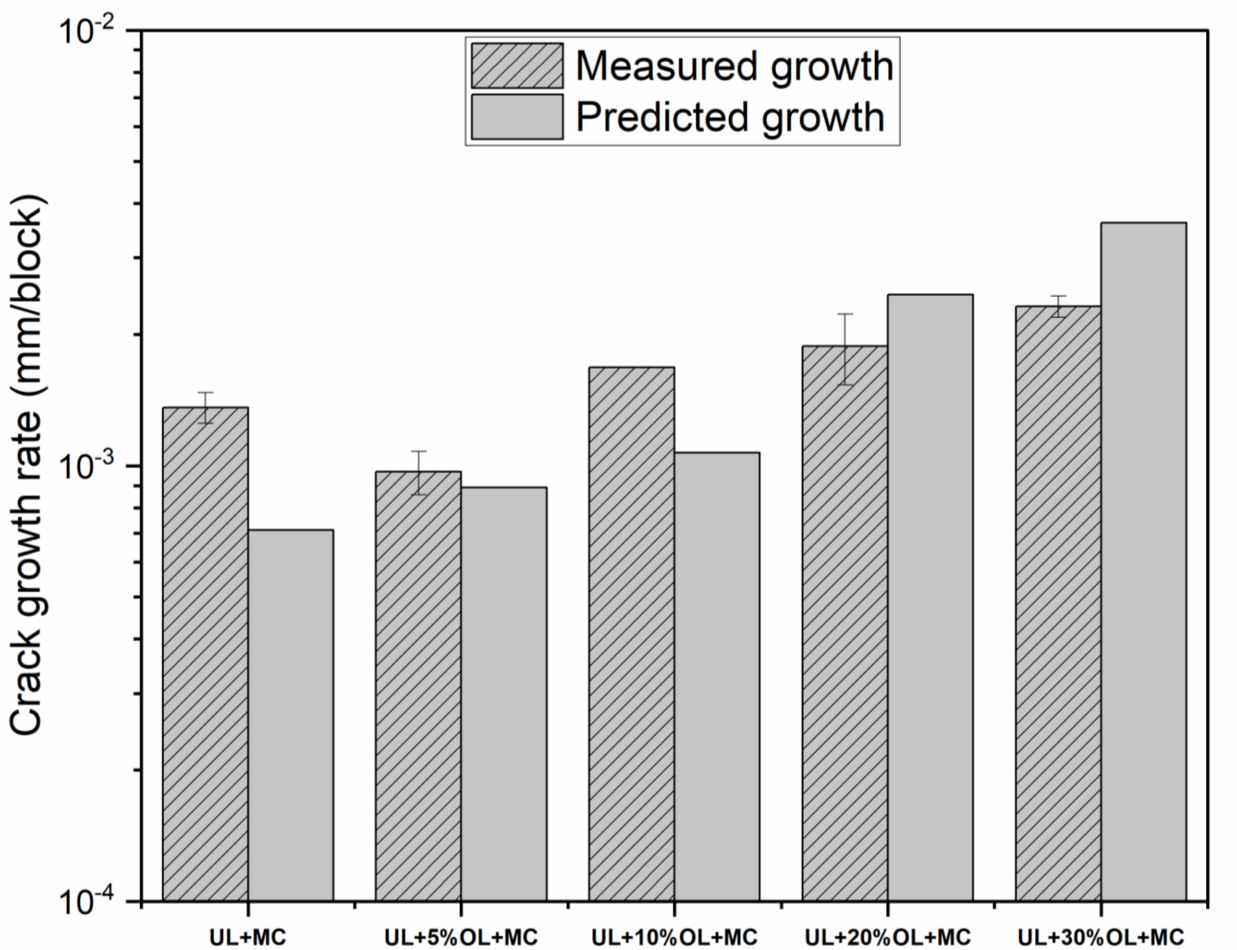


Figure 4- 9 Measured crack growth rate compared with predicted crack growth rate.

4.4.2 Mechanism of crack growth in Type II mean load (UL + OL + MC)

Overload is usually related to the retardation effect in most structural materials. It has also been observed that in a single overload, the retardation effect of an overload increases with increase in the magnitude of the overload.[19] The retardation phenomenon observed when overload is applied is usually related to residual stress ahead of crack tip and crack closure.

The crack closure concept shows that even under tensile loading, the crack tip is opened for just some part of the tensile loading cycle and that fracture surfaces could be in contact. This implies that there is an effective stress intensity factor that can be related to the applied stress intensity factor as $\Delta K_{eff} = K_{max} - K_{op}$. Where K_{max} is the applied maximum stress intensity factor and K_{op} is the opening stress intensity factor. Oxide induced crack closure can be related to the formation of oxides or corrosion products on the fracture surface which forms a wedge and increases the K_{op}, thus enhancing retardation of crack growth. This type of crack closure has been used to explain the retardation effect observed in corrosion fatigue such as in high pH environment. [28] However, there is no passivation or formation of oxide film in NNpH environment therefore, retardation by means of oxide-induced crack closure is unrealistic.

Residual stress at the crack tip is also used to explain the retardation of crack growth. An overload creates a compressive stress region ahead of the crack tip. As the crack propagates under subsequent loading the growth is impeded by the compressive residual stress in this area. This causes crack growth retardation which continues until the crack growth leaves this region. [29, 30]. In view of this, it is expected that some retardation of crack growth can be achieved in the case of an underload followed by an overload as indicated in the case of Type II mean load. [20] The results in Figure 4-3 shows that this mechanism can be applied to the retardation effect observed in UL+5%OL+MC. Under UL+5%OL+MC, the observed crack growth retardation is significant because it is below the crack growth rate in UL + MC. This retardation was observed even at a high number of minor cycles in UL+5%OL+MC as shown in Figure 4-4. This implies that in Type II mean load with a low magnitude of overload, the contribution of minor cycles to crack growth can be suppressed to achieve retardation of crack growth. It appears that further increase in the magnitude of overload in Type II mean load diminishes the retardation effect of overload. This observation can be related to the effect of hydrogen embrittlement in NNpH environment.

4.4.3 Effect of hydrogen on crack growth in Type II mean load (UL+OL+MC)

Corrosion fatigue crack growth in stage II is influenced by hydrogen in NNpH environments. Hydrogen at the crack tip enhances the sharpening and re-sharpening process, thereby facilitating crack growth. Hydrogen is a corrosion product in the NNpH environment and the diffusion to the plastic zone due to high hydrostatic stress in the regions can accelerate crack propagation. [31, 32]. There is the possibility that the presence of hydrogen in the plastic zone can affect the retardation ability in NNpH environment. In Kelestemur and Chaki's work, it was observed that there was some reduction in the retardation when overload was applied to hydrogenated 304 stainless steel. [33]

The Hydrogen Enhanced Localised Plasticity (HELP) micro-mechanism can be related to crack propagation in NNpH environments. Crack propagation in an NNpH environment can be enhanced by the accumulation of hydrogen in the plastic zone, thereby causing an increase in crack growth due to high dislocation mobility. Also, the accumulation of hydrogen into the plastic zone and other susceptible sites causes the formation of microcracks that can accelerate crack growth by sharpening/ re-sharpening processes. In an effort to model crack growth under variable amplitude loading in NNpH environment, Xing *et al.* showed that the rate of accumulation of hydrogen during loading is higher compared to the diffusion rate during unloading. The research work showed that in UL+OL+MC, there is an increase in hydrogen accumulation into the plastic zone during loading as the K_{maxOL} increases. [32] Accumulation of hydrogen atoms in the plastic zone can also increase due to the effect of minor cycles. [32]

In Type II mean load, increase in stress gradient with increase in ΔK and K_{maxOL} increases the driving force for hydrogen diffusion into the plastic zone. In the absence of a subsequent under-load, the diffusion of hydrogen away from the plastic zone will be very slow and hydrogen stays longer. The observed increase in crack growth as the K_{maxOL} increase is due to the damaging effect of hydrogen.

Therefore, retardation of crack growth can be achieved in Type II mean load (UL + OL + MC) by ensuring that the magnitude of overload is sufficient to reduce the contribution of underload and minor cycles to crack growth and also reduce the acceleration effect due to diffusion and segregation of hydrogen ahead of the crack tip. Such a balance between these two mechanisms can achieve significant retardation of crack propagation as observed in UL+5%OL+MC.

4.5 Conclusion

The effect of Type II mean load (UL+OL+MC) pressure fluctuation on crack growth behaviour in steel pipelines under NNpH environment has been studied. The following conclusions can be made based on the findings:

- Type II mean load sequence can achieve retardation of crack growth. The optimum reduction in crack growth was observed at UL+5% OL+MC.
- Cracks are subjected to hydrogen assisted cracking in NNpH environment. In addition to the mechanical driving force, the accumulation of hydrogen atoms into the plastic zone

during loading can reduce the retardation capabilities of overload cycles in Type II mean load.

- High magnitude of overload in Type II mean load can be deleterious to pipeline integrity. As the magnitude of overload increases, there will be increase in the ΔK and K_{maxOL}. This will increase the driving force for crack propagation. Increase in the magnitude of overload can also be related to higher accumulation of hydrogen to the plastic zone during loading. A combination of load interaction and the damaging effect of hydrogen accumulation during loading cycles can be used to explain the loss of retardation as K_{maxOL} increases.
- Dissolution at the crack tip is the primary mechanism driving crack growth under minor cycles. However, when minor cycles are accompanied by large cycles such as UL+OL in the case of Type II mean load, load interaction can influence acceleration of crack growth. The overall contribution of minor cycles in the case of Type II mean load is lower than in UL+MC. This indicates the retardation effect of overload.
- This work supports that hydrogen produced in NNpH environment can influence crack growth behaviour. Hence, crack growth predictive models for crack growth in pipelines should consider the role of hydrogen.
- It appears that a combination of mechanical factors, load interaction and hydrogen effects can be related to the trend observed in the crack growth rate under Type II mean load in NNpH environment.
- In case of an unplanned shutdown or pressure drop, intentionally applying a low magnitude post-overload cycle after the shutdown can mitigate the expected acceleration effect caused by depressurization.

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Chapter 5: Influence of Type I Mean Load Pressure Fluctuations on Crack Growth Behaviour in Steel Pipelines

5.1 Introduction

Pipeline operators have identified cracking of steel pipelines under near-neutral pH (NNpH) environment as a significant threat to pipeline integrity. Thus, understanding NNpH SCC is important to be able to prevent product release caused by pipeline failure from leaks and ruptures. Some research has been conducted to understand the crack propagation mechanism in an NNpH environment. Some of the significant findings have shown that [1, 2]:

- The initiation and propagation of cracks under the NNpH environment occur under disbonded coating, where the effect of cathodic protection is diminished.
- Crack growth occurs under the synergistic influence of environment, stress, and susceptible material.
- Crack propagation will not occur unless the applied stress intensity is higher than the threshold stress intensity.

There is a need to understand the nature and characteristics of the stress that drives crack growth in the NNpH environment. For example, although these cracks have been referred to as NNpH SCC, in reality recent studies have shown that there is no crack propagation under static loading NNpH environment. [3] This finding implies that the term SCC which connotes a static loading condition does not fit the actual loading conditions that drive crack propagation in NNpH environment. Chen *et al.* show that a propagating crack under cyclic loading experienced a crack arrest when the loading condition was changed to static loading. It was observed that the crack resumed propagation after the loading was changed to cyclic. This trend was observed under low and high magnitude of stress. [3] Based on this finding, the nature or characteristic of stress required for crack propagation is dynamic or cyclic in nature. [3] Hence the term corrosion fatigue is more suitable to refer to the mechanism that governs crack propagation in NNpH environment. While this thesis focuses on corrosion fatigue cracks under NNpH conditions, the results and conclusions can be applied to the propagation of cracks in structural systems where NNpH conditions prevail. This thesis considers the synergistic effect of the realistic dynamic loading condition and corrosive NNpH environment. Dynamic loading affects crack propagation significantly in that it can reduce the threshold stress intensity factor required for crack propagation. [4] One of the challenges in the pipeline industry at present is to achieve accurate prediction of fatigue life. It appears that it is challenging to correlate crack growth predicted by current models to crack growth behaviour observed in pipeline steel in service [5]. This discrepancy could be partly due to pressure fluctuations which create variable stress amplitudes in service rather than constant amplitude. The variable amplitude loading comprises large amplitude cycles (underload or overload) and small amplitude cycles (minor cycles). In variable amplitude loading, interaction and sequence of these stress cycles can accelerate or retard crack growth. [6] This implies that the accuracy of fatigue life predictions can be improved by understanding the effect of load sequence and interaction on crack propagation in steel pipelines.

The stages of crack life can be divided into initiation (often occurring at manufacturing flaw or by forming cracks at corrosion attack sites), stable growth driven by dynamic loading, and finally unstable growth to fracture. [7] It appears that that one of the factors that enable the transition from initiation to propagation is pressure cycling. Pipeline operators have observed dormancy of crack in stage II under light pressure cycling. However, under aggressive pressure cycling, active crack growth has been observed. This implies that the nature (in terms of type and amplitude) of pressure fluctuations that are applied to the pipe in service can influence crack propagation and eventual failure of the pipe if not mitigated. Based on the pressure history of oil and gas pipelines, pressure fluctuations can be broadly classified into underload, mean load, and overload. [8] Changes in operational circumstances along a pipeline are the main cause of these pressure fluctuations.

The acceleration effect of underload on crack propagation has been observed in structural components. [9-20] A recent study analysed the effect of underload pressure fluctuation in NNpH environment. The work showed that under NNpH conditions, underload pressure fluctuations accelerate crack propagation in steel pipes. [21-24] In most structural materials, it is a common belief that overload causes retardation of crack growth due to load interaction. [25-33] This retardation phenomenon is usually explained by a residual stress mechanism. Application of overload causes plastic deformation and create a large compressive residual stress at the crack tip. In this region, crack propagation by subsequent load cycles is impeded. This slows down crack growth and causes retardation of crack growth until the plastic zone grows beyond the region of compressive residual stress. [34]

Mean load pressure fluctuation is the most complex because it combines both large cycles of underload and overload and minor cycles. The large cycles have $0<R<1$ and minor (small) loading cycles have $R\approx1$. There are discrepancies in the understanding of the effect of mean load on crack propagation in structural materials. While some studies found that application of overload before the underload in the mean load can increase crack growth rate [35-37], an opposite trend has been seen in other studies. [38, 39] Also, it is important to consider the effect of the unique service environment of steel pipelines in terms of near-neutral pH environment and loading frequency. Therefore, the effect of mean load pressure fluctuation on crack propagation in steel pipelines under NNpH conditions merits additional study. The previous chapter discussed the effect of mean load with load sequence of underload+overload+minor cycles (also referred to as Type II mean load, UL+OL+MC) on crack growth in steel pipes under NNpH environment. [40] The study showed that while Type II mean load can achieve a reduction in crack growth at low magnitude of overload, increasing the magnitude of overload could cause an increase in crack growth rate.

The aim of the present study is to understand the contribution of Type I mean load pressure fluctuation to crack growth in steel pipelines under a near-neutral pH environment. In this study, the waveform sequence has been modified to simulate overload+underload+minor cycle (Type I mean load, OL+UL+MC). The effect of the load sequence interaction on the retardation effect of overload will be studied by applying varying magnitudes of overload. It is important to explore the retardation effect of overload and understand how it can be leveraged to mitigate the acceleration effect of underload in NNpH environment. For instance, an overload cycle prior to depressurisation of pipeline steel in operation could retard crack growth and reinforce pipeline integrity management methods. The results obtained in this study will be analysed and compared with constant amplitude loading, UL+MC and Type II mean load.

5.2 Experimental Procedure

5.2.1 Material

An API 5L Grade X-65 pipeline steel was used in this research. The material selection was based on its known susceptibility to NNpH SCC in service. The chemical composition (wt.%) of the X-65 steel used in this research is as follows: C: 0.12, Mn: 1.5, P: 0.017, S: 0.0046, Si: 0.26, V: 0.01, Cr: 0.005, Nb:0.049 Fe: Balance. Compact Tension (CT) specimens were machined from a section of the pipe. The notch direction in the CT samples was cut to simulate axial cracks on the pipe. The surface preparation involved grinding up to 600 grit abrasive, after which the samples were cleaned in ethanol. Crack initiation was carried out using an Instron machine to initiate a crack 2.5 mm deep.

5.2.2 Test Environment

To simulate a near-neutral pH service environment of steel pipelines, C2 solution was used in this research. The salt composition in this NNpH solution is (g/L): KCl:0.0035, $NaHCO_3$:0.0195, $CaCl_2.H_2O$:0.0255, $MgSO_4.7H_2O$:0.0274, $CaCO_3$:0.0606. The solution was prepared and bubbled with 5% CO_2 + 95% N_2 continuously for the test duration.

The sample was placed in a test cell filled with C2 and sealed from oxygen. To maintain the NNpH environment, the solution was bubbled with 5% CO_2+95% N_2 for the duration of the test. The temperature was maintained at 30°C.

5.2.3 Loading Parameters for Type I mean load

Figure 5-1a shows the waveforms for Type I mean load-OL+UL+MC used in this study. To study the effect of overload in Type I mean load, varying magnitudes of overload were analysed from 5-30%OL in OL+UL+MC tests. Per cent OL is defined as:

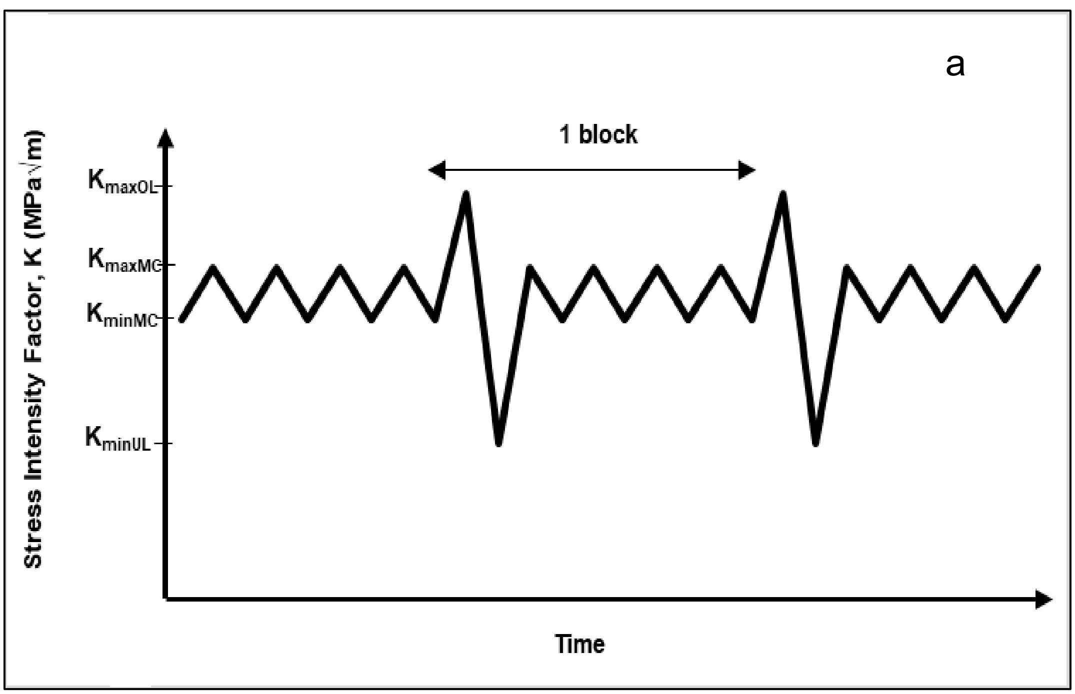
$$\%OL = \frac{K_{maxOL} - K_{maxMC}}{K_{maxMC}} \times 100 \quad (1)$$

Where K_{maxOL} is the maximum stress intensity of overload and the value varies with the magnitude of overload. K_{maxMC} represents the maximum stress intensity factor of underload.

The per cent OL represents pressure increase or overshoot an operator would impose over the peak minor cycle pressure. The test loading rate in the underload and overload cycles was 0.01kN/s. In each mean load test, 300 minor cycles were applied per block and the loading frequency was 0.005Hz similar to other research [21, 41, 42]. K_{minMC} and K_{maxMC} represent the minimum and maximum stress intensity factor of minor cycles respectively and K_{maxUL} is the maximum stress intensity factor of underload. The duration of test was 45-47 days for 70 blocks of each mean load waveform. In addition, a test was conducted using underload+minor cycle (UL+MC) alone without an overload as shown in Figure 5-1b.

5.2.4 Fracture surface analysis

To analyze the fracture surface after the test, the sample was sliced into a smaller piece and placed in liquid nitrogen for 3-5 hours. The sample was then fractured and the two fracture surfaces were placed in acetone immediately to prevent oxidation. Fracture surface analysis was carried out using a Zeiss Sigma 300-VP FESEM to identify and analyze features that correlate with the fatigue loading process during the tests.



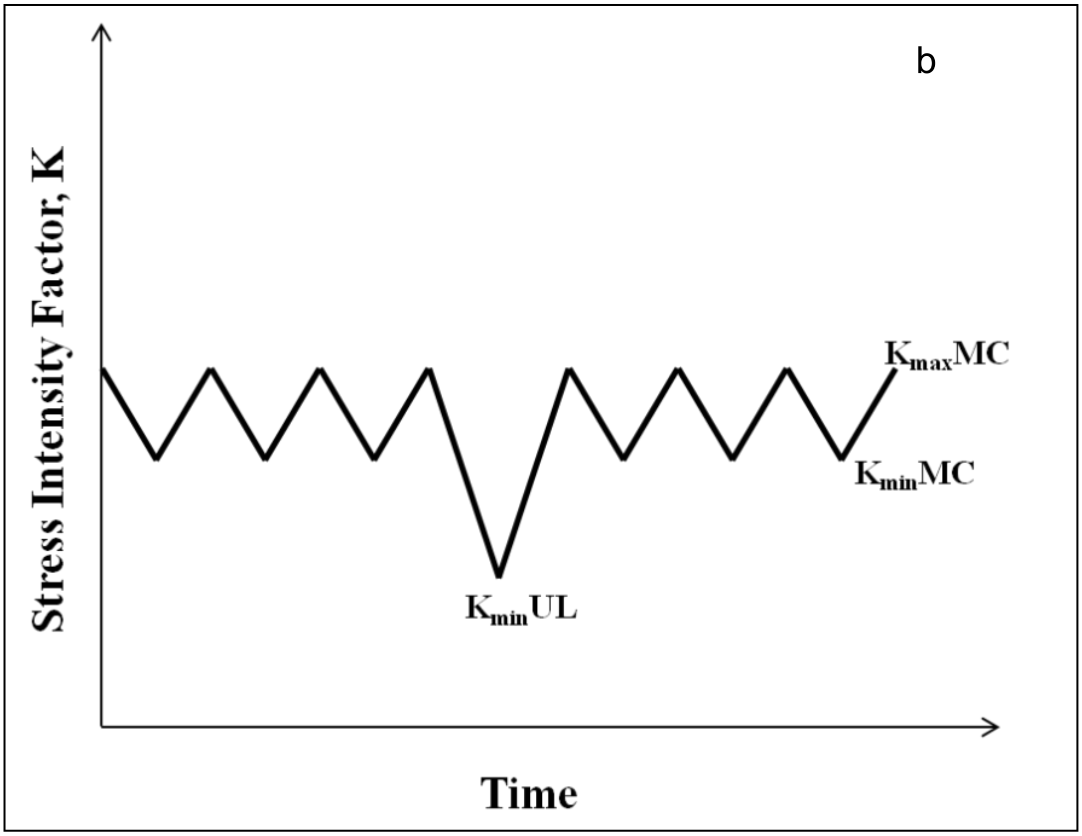


Figure 5- 1 Loading waveforms for (a) Type I mean load-OL+UL+MC (b) UL+MC.

Table 5- 1 Loading parameters for Type I mean load-OL+UL+MC.

Test	Loading	K_{maxOL}	K_{maxUL}	K_{minUL}	K_{maxMC}	K_{minMC}
1	5%OL+UL+MC	34.65	33	16.5	33	29.7
2	10%OL+UL+MC	36.3	33	16.5	33	29.7
3	20%OL+UL+MC	39.6	33	16.5	33	29.7
4	30%OL+UL+MC	42.9	33	16.5	33	29.7
5	UL+MC	-	33	16.5	33	29.7

5.3 Results

5.3.1 Sensitivity of Crack Growth Rate to %OL in OL+UL+MC

The effect of OL+UL+MC (Type I mean load) on crack growth rate is shown in Figure 5-2. The result expressed crack growth rates of several overload amplitudes preceding the underload cycle as a function of combined factor. [43] The results show that applying an overload cycle prior to underload can achieve a reduction that was highest near 5%OL. As shown in Figure 5-2, it is interesting to find that the largest retardation effect corresponds to the 5% overload (similar to Type II mean load), making it the easiest to integrate on an operating pipeline.

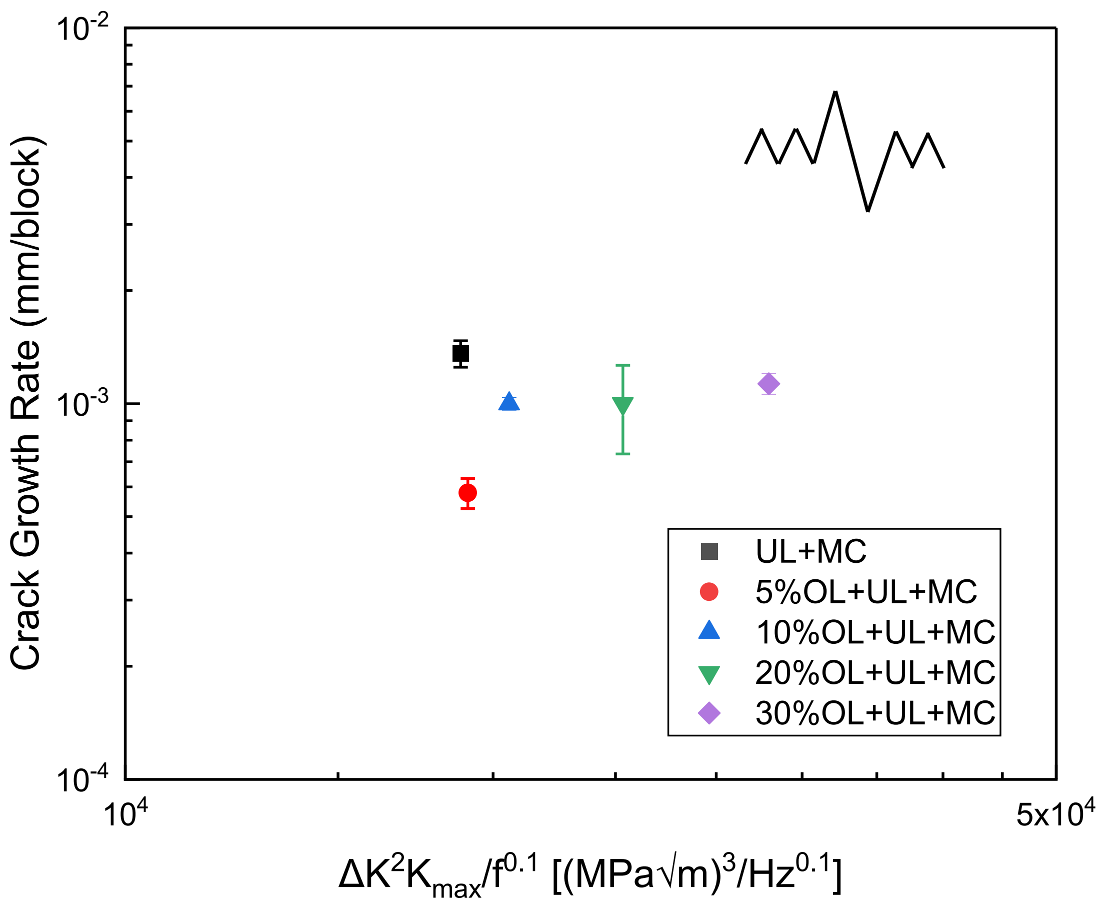


Figure 5- 2 Crack growth rate showing the effect of OL+UL+MC with varying OL.

Figure 5-2 also shows that the large magnitude of overload still corresponds to a decrease in crack growth rate below UL+MC. The result shows that overall crack propagation is relatively insensitive to increase in the magnitude of overload in Type I mean load. From this result, it appears that applying an overload cycle prior to underload in mean load variable amplitude reduces the acceleration effect caused by load interaction between underload and minor cycles. The ability of Type I mean load to achieve some retardation of crack growth has been confirmed by some other researchers. [38, 39].

Figure 5-3 compares crack growth rates obtained from Type I mean load to predicted crack growth based on constant amplitude results obtained from Equation 1.

$$\left(\frac{da}{dN}\right)_{\boldsymbol{OL+UL+MC\ predicted}} = \left(\frac{da}{dN}\right)_{\boldsymbol{OL+UL}} + n\left(\frac{da}{dN}\right)_{\boldsymbol{MC}} \tag{1}$$

Equation 1 was introduced in the previous chapter to acknowledge the contribution of minor cycles to crack growth and to show the significance of load interaction on crack growth. [40] In reference [40], a detailed experimental analysis was conducted to show that cracks may propagate at a relatively slow rate under simple minor cycle loading due mainly to dissolution at the crack tip. The increase in crack growth rate when minor cycles are applied in variable amplitude loading confirms that these high R ratio cycles can contribute to crack propagation due to load interaction. The result in Figure 5-3 shows that load interaction can influence crack propagation under Type I mean load. As shown in the result, predicting crack growth rate under high magnitude of overload tend to overestimate crack growth rate which therefore might be too conservative. This finding highlights the fact that load sequencing can achieve retardation of crack growth and reduce the interaction between underload and minor cycles. Figure 5-4a shows a summary of the effect of different loading conditions on crack growth rate in NNpH environment. Crack growth rate under Type I mean load (represented by blue data points) is compared with constant amplitude loading (represented by the dashed line) and Type II mean load (represented by the red data points). The %OL considered here are 5, 10, 20 and 30%OL, respectively in Figure 5-4. The research work in the previous chapters shows details of the constant amplitude and Type II mean load. The result of crack growth rate under UL+MC is also shown in Figure 5-4.

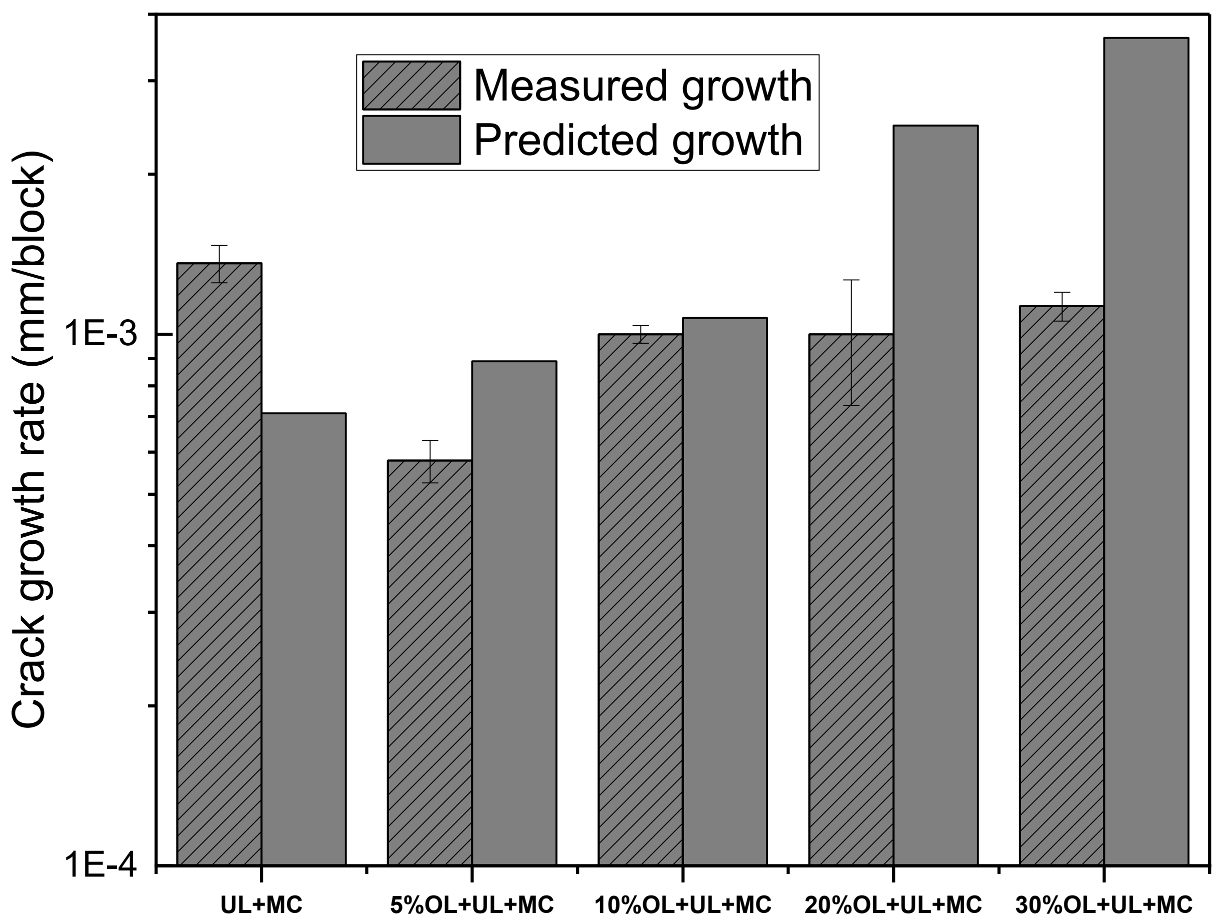


Figure 5- 3 Crack growth rate showing the effect of load interaction on crack growth by comparing measured growth to predicted growth.

It is important to note that for the case of Type I mean load the combined factor was obtained based on the loading part of the cycle only. It is assumed that crack will not propagate during the unloading. Figure 5-4 compares crack growth under Type I and Type II mean load to constant amplitude loading. The result shows that under constant amplitude cyclic loading, crack growth rate increases with increase in ΔK and K_{max}. The result also shows that the overall effect of Type I mean load is less severe compared to Type II mean load. It is interesting to note that in both Type I and Type II mean load, the best retardation effect of overload was achieved at 5% overload.

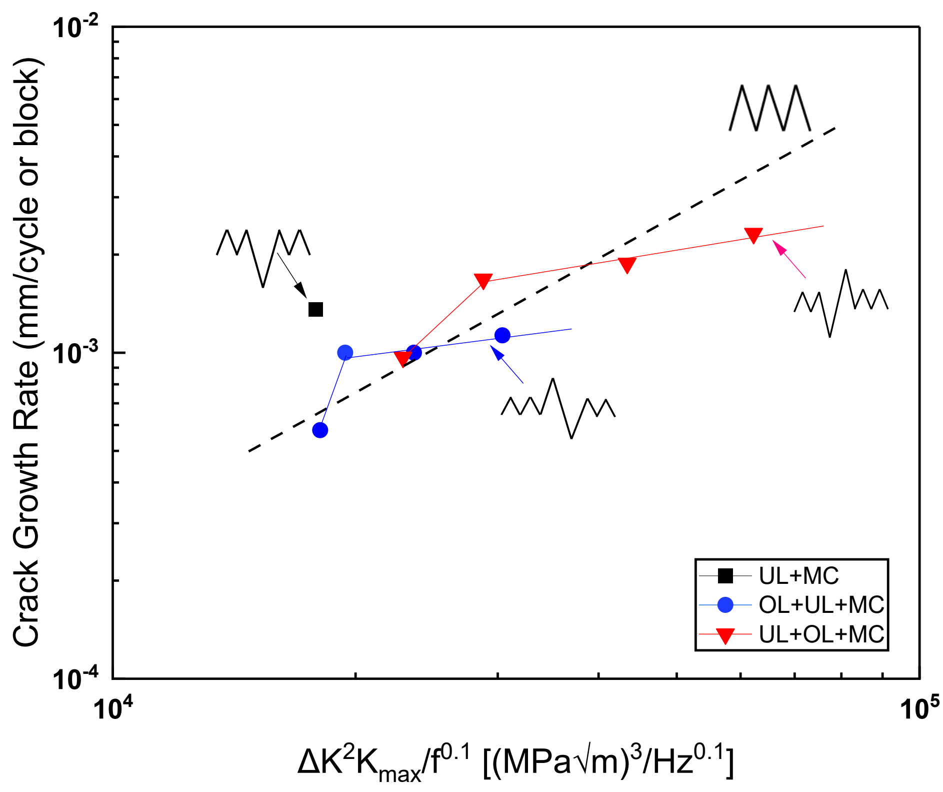


Figure 5- 4 Crack growth rate in constant amplitude cyclic loading compared to variable amplitude loading. The overloads are 5, 10, 20 and 30 for the points respectively.

However, Type I mean load achieved the best retardation effect. The response of Type I and Type II mean load to increase in %OL shows that it will be inaccurate to determine fatigue life based on constant amplitude only.

Figure 5-4 shows that ignoring the effect of load interaction and the effect of contribution of minor cycles to crack growth even in predictive crack growth calculations as in Eq. 1 will not achieve accurate results. [44] The results for Type II mean load with ≥10% overload shows that this type of pressure fluctuation can be very detrimental to pipeline integrity as much or even more than UL+MC. This supports the fact that higher crack growth and pipeline failure have been reported close to the discharge part of the pipe, where both UL+MC and mean load have been observed. Figure 5-5 shows crack growth rate in both Type I and Type II mean load expressed as crack growth ratio with the respective overload ratio. Crack growth ratio is the measure of the sensitivity of mean load crack growth to increase in the magnitude of overload, and it is given as:

$$\text{Crack growth ratio} = \frac{\text{Crack growth rate under mean load}}{\text{Crack growth rate under UL+MC}} \qquad (2)$$

The overload ratio (OLR) is given as:

$$\text{OLR} = \frac{K_{maxOL} - K_{minMC}}{K_{maxMC} - K_{minMC}} \qquad (3)$$

Where: K_{maxOL} is maximum stress intensity factor of overload, K_{maxMC} is maximum stress intensity factor of minor cycle and K_{minMC} is the minimum stress intensity factor of minor cycle.

As shown in Figure 5-5, crack growth retardation can be achieved in Type II mean load when the OLR is less than 1.75. The result in Figure 5-5 shows that in Type II mean load, further increase in OLR will cause increase in crack growth rate. It appears that in Type I mean load, retardation of crack growth can be achieved even with increase in the OLR. In all the OLR conditions considered, crack growth rate was below UL+MC.

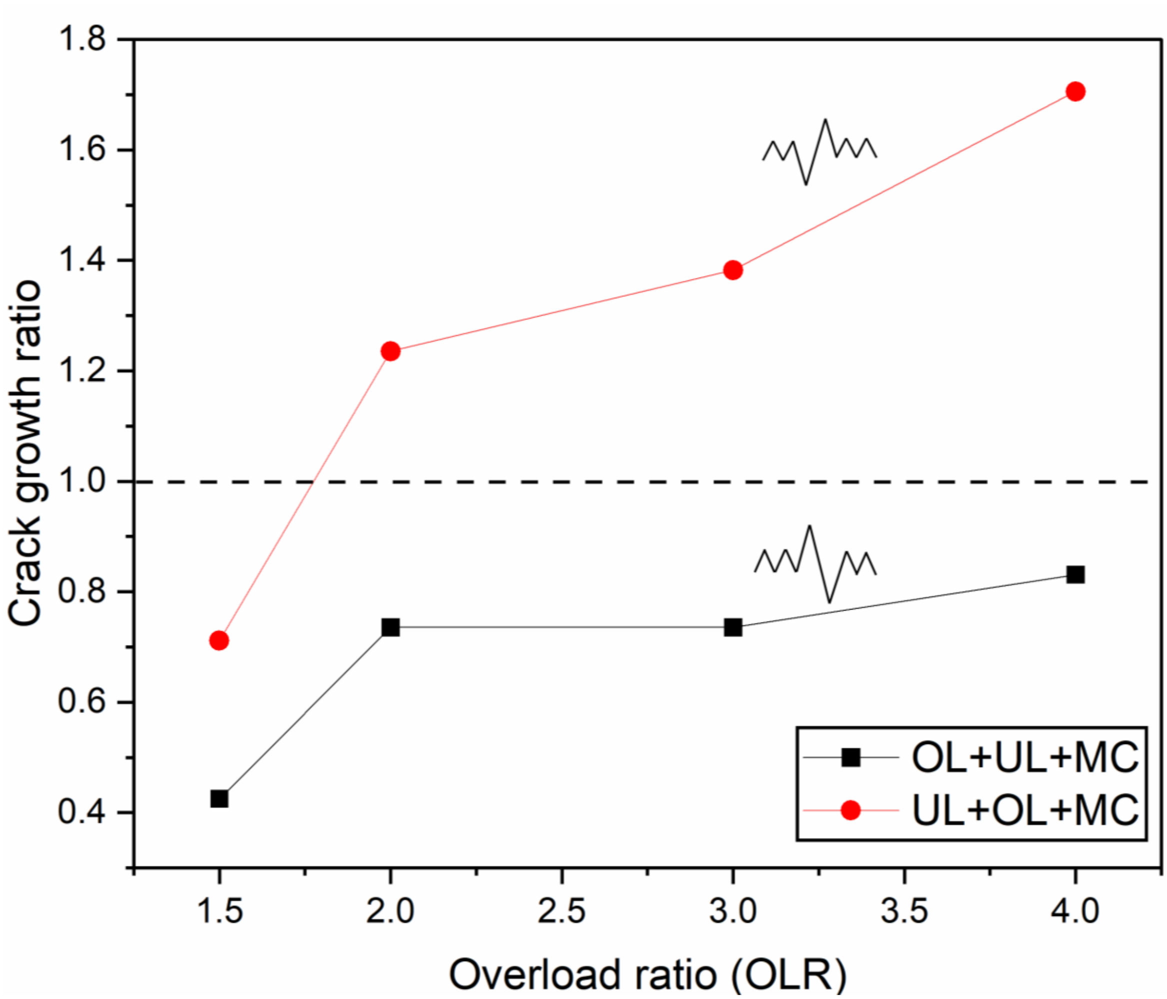


Figure 5- 5 Crack growth ratio in Type I (black) and Type II (red) mean load.

Consider mean load with 5%OL, Figure 5-6 summarizes crack growth rate in mean load, UL+MC and constant amplitude cyclic loading. The results show that the effect of mean load pressure fluctuation on crack growth in steel pipelines can not be accurately achieved by simply assuming constant amplitude loading.

Under similar ΔK, it is obvious from Figure 5-6 that both Type I and Type II mean load pressure fluctuation can achieve retardation in crack growth rate compared to UL+MC. The observed subsequent reduction in crack growth can be related to the reduced drive for crack growth due to the initial compressive residual stress region formed during the overload cycle. [45]

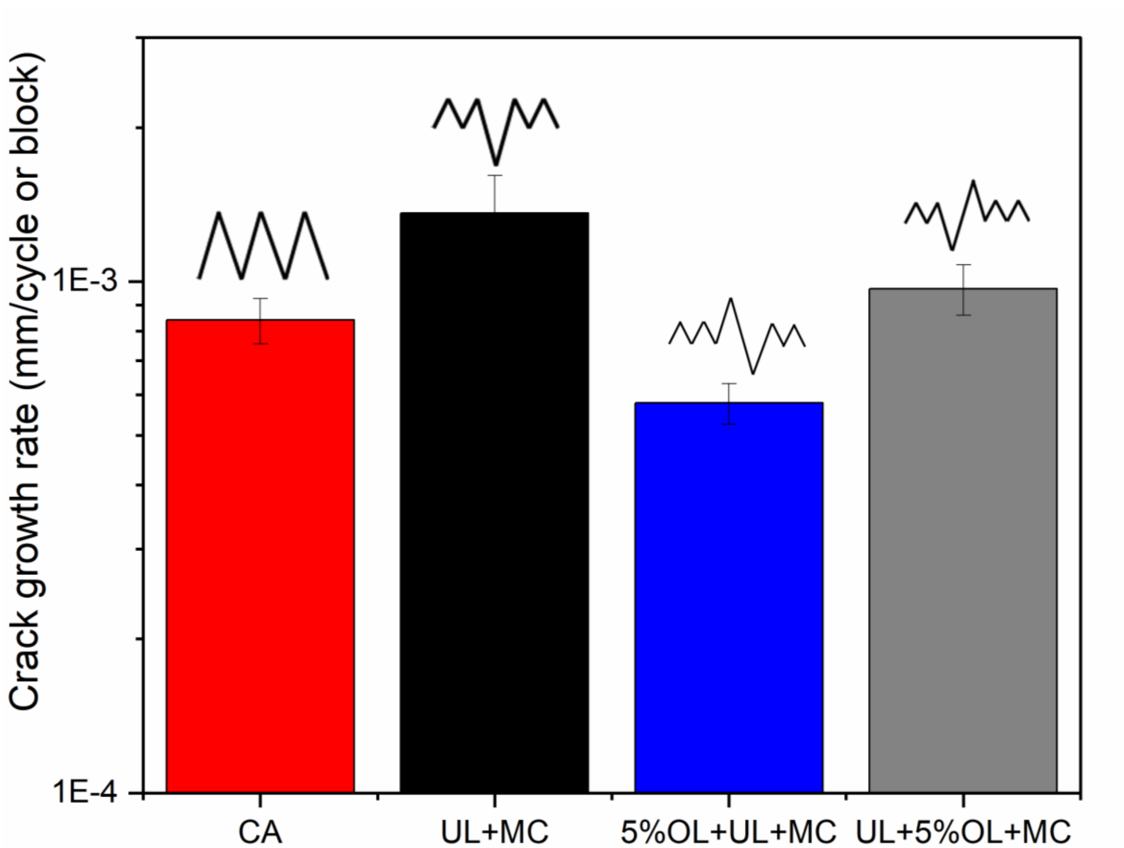


Figure 5- 6 Crack growth results showing the effect of different loading conditions.

5.3.2 Effect of Minor Cycles

Previously minor cycles (also referred to as ripple load) were neglected and considered as non-propagating. However, recent findings in the previous chapter and this current work have shown that these high R ratio cycles contribute to crack propagation in variable amplitude loading as shown for UL+MC, OL+UL+MC and UL+OL+MC, respectively. Figure 5-7 shows the effect of the number of minor cycles to crack growth rate in UL+MC, 5%OL+UL+MC and UL+5%OL+MC. This result shows that in all these three variable-amplitude loading conditions, an increase in the number of minor cycles causes an increase in the crack growth rate. The highest sensitivity to crack growth was observed in UL+MC. As shown in Figure 5-7, mean load can achieve a reduced crack growth with the number of minor cycles due to the presence of overload. The most significant growth reduction with minor cycles is OL+UL+MC. This is an indication of the ability of a preceding overload cycle to reduce or slow down the contribution of subsequent minor cycles to crack growth. The contribution of minor cycles to crack propagation in NNpH environment is partly due to load interaction and due to the effect of hydrogen.

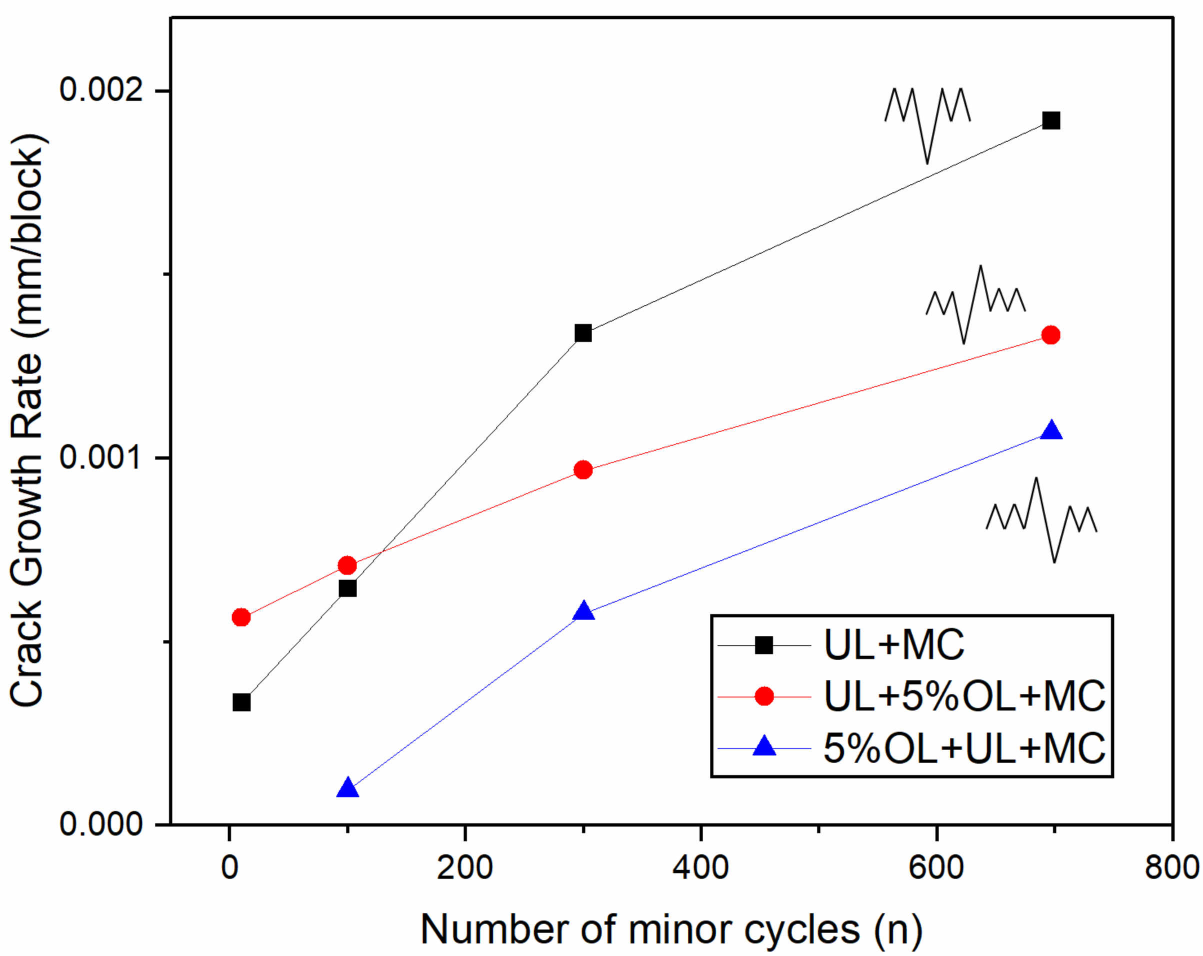
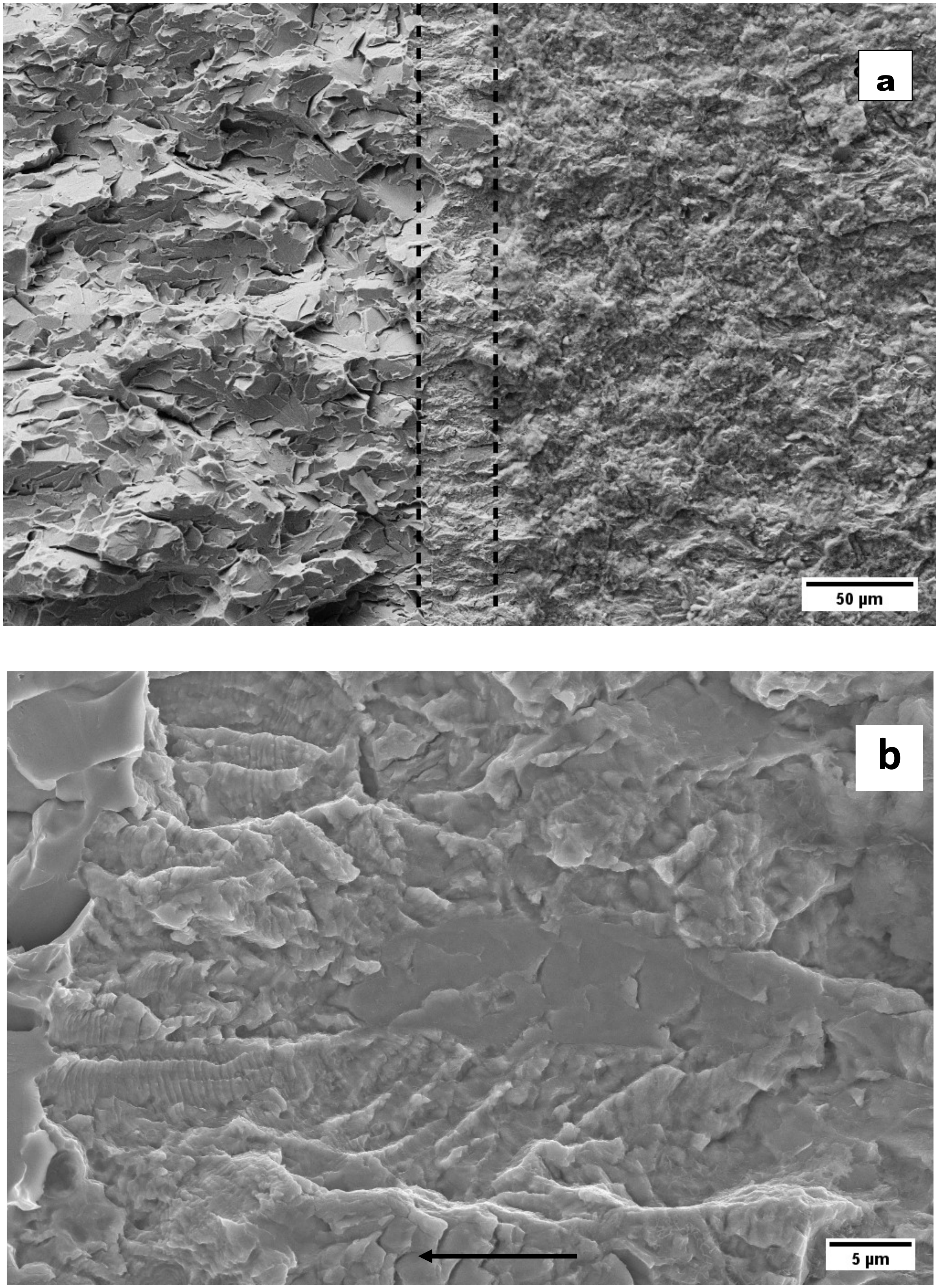
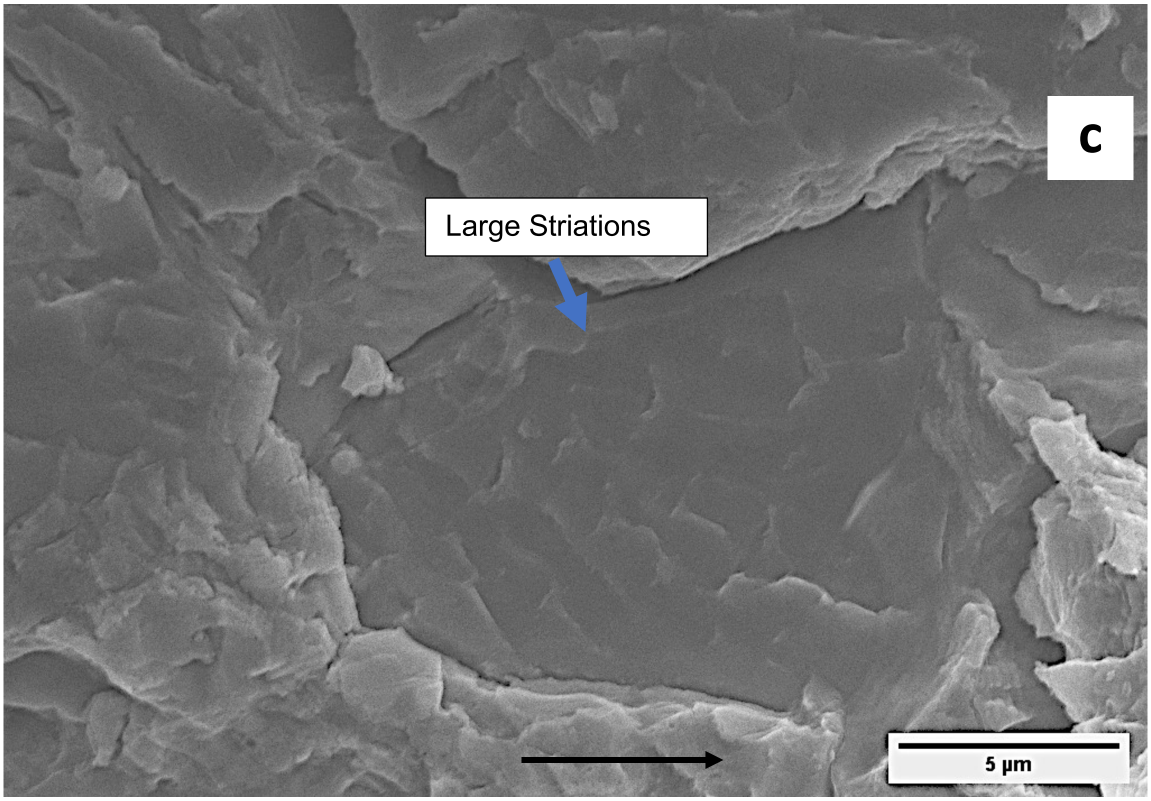


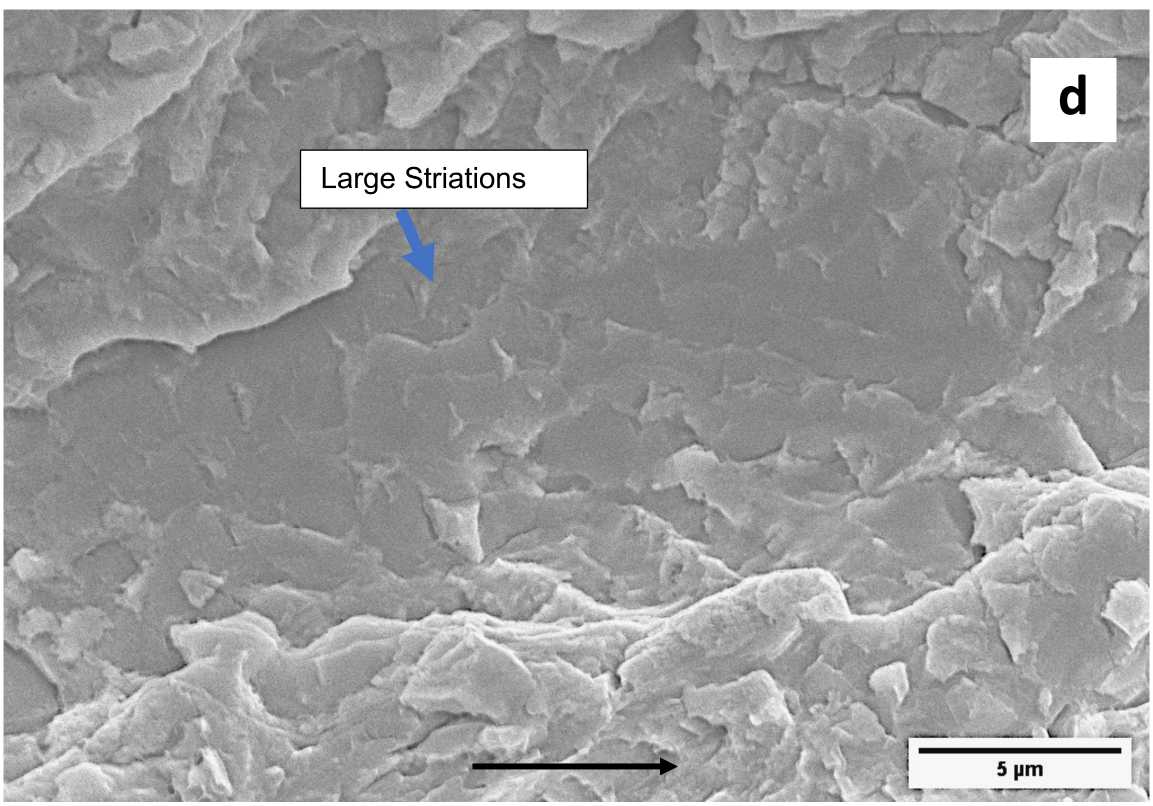
Figure 5- 7 Crack growth rate showing the effect of minor cycles on crack growth under mean load compared to UL+MC.

5.3.3 Fracture Surface Analysis

Striations are microscopic features that are considered as evidence of fatigue crack growth. It is difficult to analogize the striation spacing on a fracture surface to crack growth rate since each loading cycle does not correspond to a striation. However, analyzing these microscopic features can explain crack growth behaviour based on the loading waveforms applied. Usually, steels are characterized by non-distinct striations compared to some other alloys like aluminium. Another challenge with characterizing the fracture surface in this work is the fact that the NNpH environment, being corrosive, may destroy or reduce striations from the fracture surface. [46] It is also difficult to preserve the sample from corrosion due to the long-term exposure to a corrosive environment. Figure 5-8a shows the crack surface morphology after fracture, with the crack propagating to the left. The region studied (the area between the dashed line) is crack growth during the corrosion fatigue test. Figure 5-8b-f shows the fracture surface from OL+UL+MC (n=100). As shown in Figure 5-8b-d, there are large striations on the fracture surface that have been attributed to large loading cycle. As shown in Figures 5-8e and f, there are also mini striations on the fracture surface (with count > 70) as well. Interestingly, the mini striations are observed at the end of the crack growth region. This observation corresponds to the minor cycles at the end of the loading waveform. Hence the mini striations could be attributed to minor cycles to confirm their contribution to crack propagation. It is also interesting to see, as shown in Figure 5-8f, some mini striations away from the end of the crack growth region.


a
50 µm
b
5 µm


c
Large Striations
5 µm


d
Large Striations
5 µm

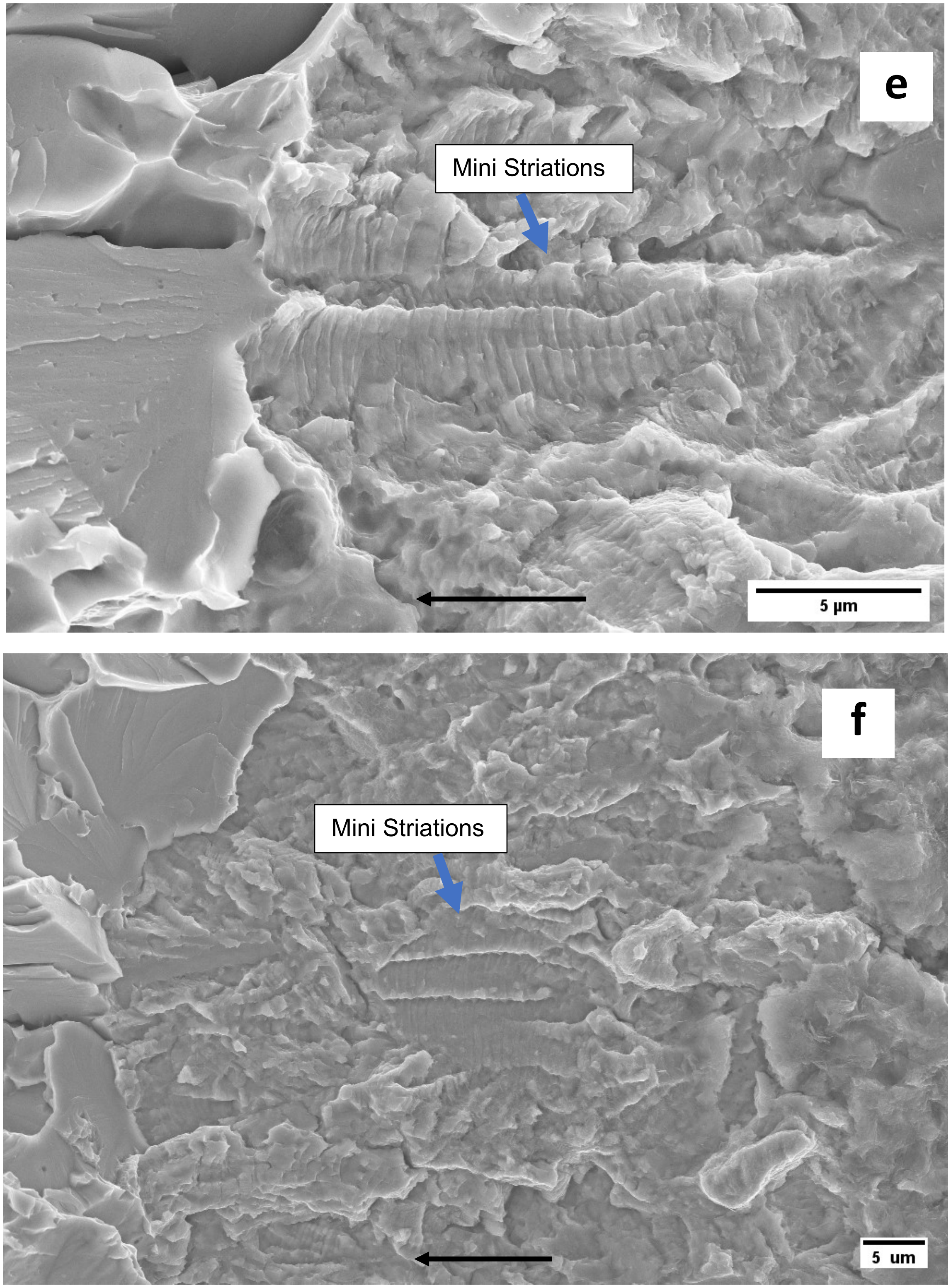


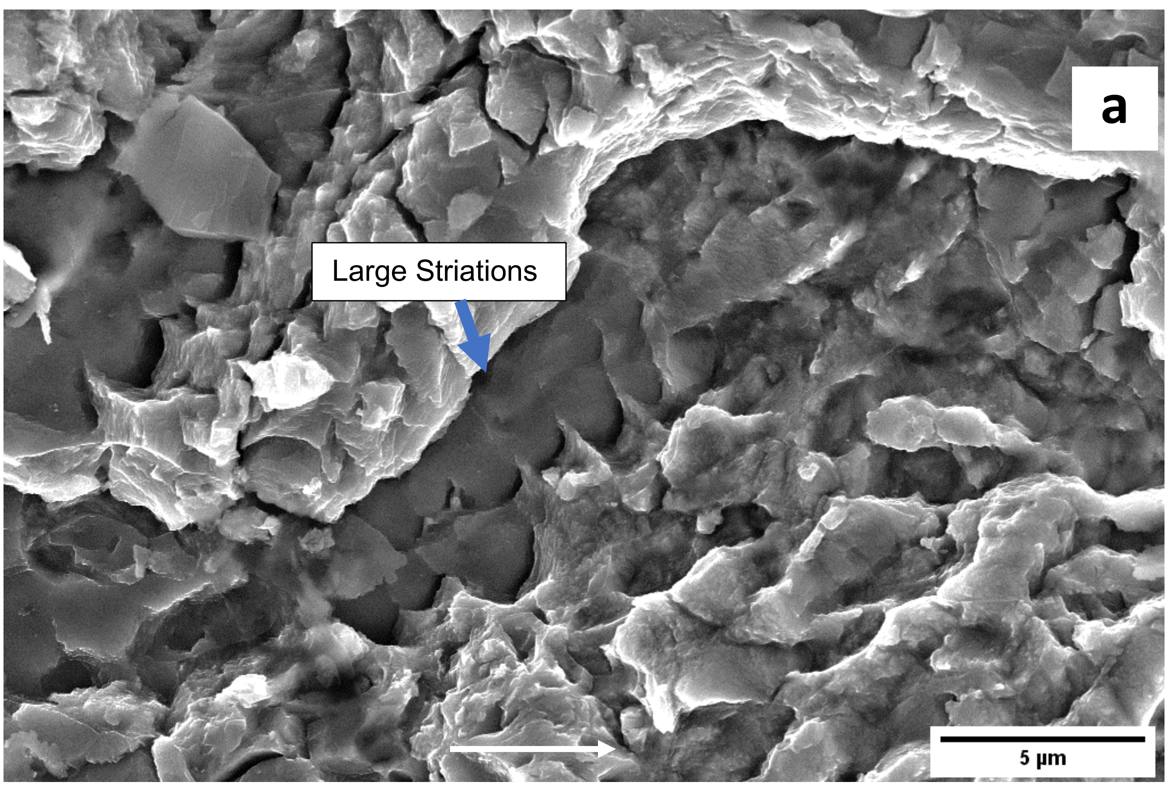
Figure 5- 8 Fracture surface morphology mean load- OL+UL+MC (n=100). Black arrows show cracking direction.

5.3.4 Effect of load history on fracture surface morphology

Fracture surface morphology can provide some information on the effect of load history on crack growth behaviour. Some works have correlated crack growth measured from fracture surface to visual crack growth. [47-49] For example, Schijve observed macrobands in large cracks in aluminium alloys. [47] These bands correspond to overload blocks and a faint line was observed for a single overload cycle. In Yu *et al.'s* work, it was reported that striation morphology observed on the fracture surface were similar when UL+MC with the number of minor cycles ranging from 0-10 was applied (n=0 being constant amplitude loading). [21] An increase in the number of minor cycles showed mini-striations between large striations. The mini-striations were attributed to the contribution of minor cycles to crack growth. It should be noted that unlike what Yu *et al.* reported, it appears that the mini striations were predominantly found at the end of the crack in all the mean load cases that were observed in this study. Additionally, the following observations could be made based on the fractography in Figure 5-8:

- Overall fracture surfaces have a quasi-cleavage appearance.
- The large striations in OL+UL+MC appear to be erratic brittle striations (Figure 5-8 b-d). Hence this could be related to the low crack growth in Type I mean load.
- The mini-striations could also be identified as ductile striations and that could mean that there was some sort of changes from brittle to ductile behaviour along the crack front (Figure 5-8 e-f).

It will be interesting to understand how fracture surface morphology might provide information on the retardation of crack growth observed in Type I mean load (OL+UL+MC) compared to Type II mean load (UL+OL+MC) in NNpH environment. However, it must be noted that unlike in constant amplitude loading where striations can be observed clearly, under variable amplitude loading, striations are not usually clearly defined. For instance, Figure 5-9 below shows the surface morphology of UL+5%OL+MC with n=100 (black arrow indicates crack growth direction). Figures 5-9a and b show the presence of large striations on the fracture surface. Figure 5-9c shows the mini striations on the fracture surface. It can be noticed from comparing Figure 5-9 (which corresponds to UL+5%OL+MC) to Figure 5-8 (which corresponds to 5%OL+UL+MC) shows that the large striations in Figure 5-9a and b appear to be more brittle. Due to the limitation in viable fracture surfaces in this study, further study will be required to explore fractographic analysis to support crack growth behaviour observed in the mean load.



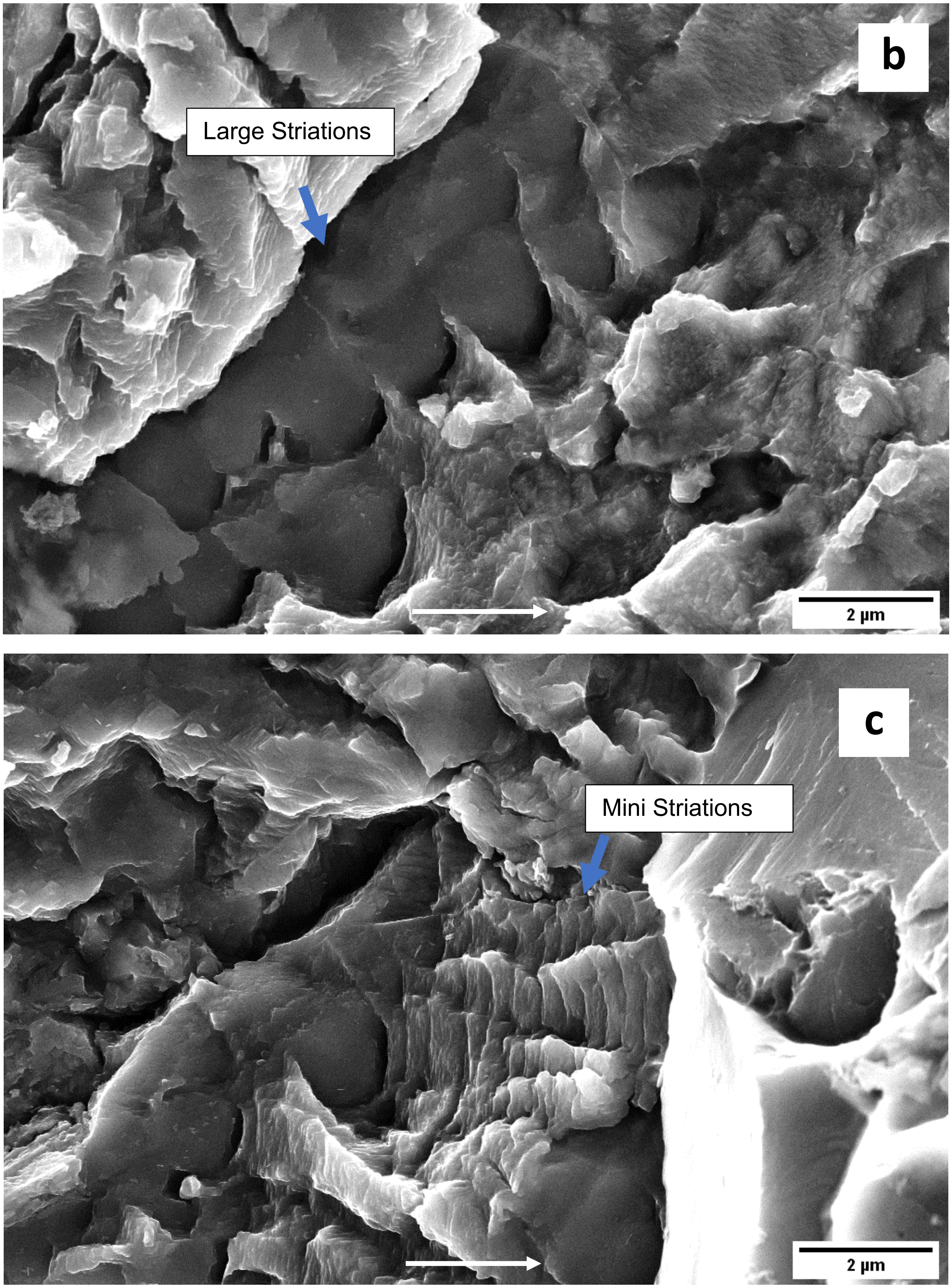


Figure 5- 9 Fracture surface morphology mean load- UL+OL+MC (n=100). Large striations as well as mini striations was observed. White arrows show cracking direction.

5.3.5 Effect of hydrogen on Type I mean load

The overall reduction in crack growth observed in OL+UL+MC can be related to load interaction. The retardation effect of overload reduces the contribution of minor cycles to crack growth and the effect of hydrogen. Hydrogen as a corrosion product can be adsorbed at the crack tip and cause environmental assisted cracking through IHAC or HEAC mechanism. The diffusion of hydrogen results in embrittlement ahead of the crack tip. Hydrogen enhanced localised plasticity (HELP) and hydrogen enhanced decohesion (HEDE) are widely accepted mechanisms that explain the damage or embrittlement due to hydrogen diffusion. [50] In Type I mean load, crack propagation and hydrogen accumulation are mainly influenced by the loading cycles. The load sequence in Type I (OL+UL+MC) creates a stress gradient that allows the diffusion of hydrogen away from the plastic zone due to the underload. Hence the damaging effect of hydrogen is reduced and crack growth is retarded in Type I mean load. However, in Type II mean load (UL+OL+MC), stress gradient to higher peak stress at K_{maxOL} causes accumulation of hydrogen. Due to the load sequence in Type II mean load (UL+OL+MC), there is no high driving force (following the overload) to remove hydrogen so hydrogen stays longer. The residual damaging effect of hydrogen is responsible for the increase in crack growth observed. This explains the differences observed in the crack growth behaviour in Type I and Type II mean load. Other research works have shown that the presence and distribution of hydrogen are significant to the formation of brittle striation. [51, 52] The erratic appearance of the brittle striations observed in OL+UL+MC could be an indication of the reduced contribution of hydrogen.

5.4 Conclusions

The effect of Type I mean load (OL+UL+MC) on crack growth in NNpH environment has been studied in this research. The following conclusions can be reached based on the findings:

- OL+UL+MC load sequence can maximize the retardation effect of overload. This can be explored to reduce the acceleration effect caused by load interaction between minor cycles and underload.
- Crack growth rate in Type I mean load can achieve retardation of crack growth even with increase in overload. This implies that OL+UL+MC provides a wide tolerance band for pipeline operators to apply the pre-overload operating method (assuming the maximum pressure does not exceed design limits).
- Pipeline operators can leverage pressure fluctuations to minimize crack growth in NNpH conditions. Applying 5% overload prior to initiating minor cycles (pumping operations) and before underload (shutdown) can reduce crack growth rates by more than a factor of 2.
- It appears that a combination of mechanical factors, load interaction and hydrogen effects can be related to the trend observed in the crack growth rate under Type I mean load in NNpH environment.
- In the case of a planned shutdown, intentionally applying a pre-overload before the shutdown can mitigate the expected acceleration effect caused by depressurization.

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Chapter 6: Conclusions, Research Impact and Recommendations

6.1 Conclusion

The discussions in this thesis have shown that it is crucial to consider mean load variable amplitude loading due to pressure fluctuations as an influence on crack growth behaviour in pipeline steel under an NNpH environment.

The following conclusions were made based on the effect of constant amplitude loading on crack growth in X65 pipeline steel:

- Mechanical factors such as ΔK and Kmax affect the driving force for crack propagation, especially in the NNpH environment compared to air.
- Crack growth under constant amplitude cyclic loading can be affected by the loading severity and environment. This was confirmed by the fracture surface morphology. Hence it is essential to consider the effect of the environment in any predictive model.
- Crack growth under minor cycle loading only is mainly driven by dissolution at the crack tip. This is not true when the MC are immediately preceded by an underload in a propagating crack.

The following conclusions were made based on the effect of Type II mean load pressure fluctuation on crack growth in X65 pipeline steel:

- Type II mean load sequence can achieve retardation of crack growth provided that the magnitude of the overload applied is 5%. The largest reduction in crack growth was observed at UL+5% OL+MC.
- High magnitude of overload in Type II mean load can be damaging to pipeline integrity. The load sequence of underload followed by overload allows hydrogen accumulation in

the plastic zone. Without a subsequent underload cycle after the overload, hydrogen stays much longer because the diffusion will be slow. The damaging effect of hydrogen causes an increase in crack growth observed at higher magnitude of overload.

- In mean load variable amplitude loading, minor cycles contribute to crack propagation in the NNpH environment. Therefore, they should not be considered as non-propagating. In a low magnitude overload (UL+5%OL+MC), the overall growth rate is lower than UL+MC (even with increasing minor cycles) showing some influence of the retardation effect of overload.
- Crack growth under mean load pressure fluctuation is influenced by hydrogen produced in the NNpH environment. Hence, crack predictive growth models for crack growth in pipe-lines should consider the role of hydrogen.
- It appears that a combination of mechanical factors, load interaction and hydrogen effect can be related to the trend observed in the crack growth rate under Type II mean load in NNpH environment.

The following conclusions were made based on the effect of Type I mean load pressure fluctuation on crack growth in X65 pipeline steel:

- Type I mean load sequence can maximize the retardation effect of an overload even at high magnitude of overload. This should be explored to reduce the acceleration effect caused by load interaction between minor cycles and underload.
- Type I mean load presents the least crack growth rate compared to UL+MC and UL+OL+MC.

- It appears that a combination of mechanical factors, load interaction and hydrogen effect can be related to the trend observed in the crack growth rate under Type I mean load in NNpH environment. Pipeline operators can leverage pressure fluctuations to minimize crack growth in NNpH conditions. Applying 5% overload prior to initiating minor cycles (pumping operations) and before underload (shutdown) can reduce crack growth rates by more than a factor of 2.

6.2 Research Impact

- This research has confirmed that the primary mechanism driving crack growth under minor cycles is dissolution at the crack tip. This finding has improved calculating predicted life under constant amplitude cyclic loading.

- The effect of mean load pressure fluctuation on crack growth in pipeline steels under NNpH conditions provides information that can guide pipeline operators on using mean load sequence to mitigate crack growth due to depressurization.

- The results in this research justify using applicable mean load pressure cycles to achieve accurate prediction of crack growth.

6.3 Recommendations for further studies

The following areas should be considered for future studies:

- Further analysis of the morphology of fracture surface under mean load pressure fluctuations should be conducted. This will provide more understanding of the crack propagation mechanisms. This might require designing tests with short-term corrosion exposure.

- The effect of both Type I and Type II mean load on crack propagation should be studied using surface cracks. This might also provide trends related to field observations since axial cracks observed in the field are surface cracks.
- The effect of a NNpH environment on the retardation effect of overload pressure fluctuations should be considered. This will provide a realistic idea of the effect of the environment on load interaction in structural materials.
- The effect of other parameters/ factors such as the R ratio of minor cycles, minimum stress intensity factor of underload, cathodic protection on crack growth under mean load should be studied. This will provide a deeper understanding of the growth trends observed in mean load.
- The results obtained on Type I and Type II mean loads should be validated using full-scale tests to simulate pipeline operations in the field better.
- It is recommended that Finite Element Analysis of the stress ahead of the crack tip should be explored to further investigate the mechanisms driving crack growth in NNpH environment under mean load pressure fluctuations.

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