



Mustafa Elsherkisi

Advancing the Synergy Between Models and Experiments to
Investigate Environmentally and Mechanically Driven Crack
Propagation

School of Aerospace, Transport and Manufacturing

PhD

Academic Year: 2020–2023

Supervisors: Dr G. M. Castelluccio, Dr S. Gray
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Abstract

Aero-gas turbine running temperatures are rapidly increasing in order to improve their efficiency, and as a consequence components are subjected to more extreme environments. With higher operational temperatures and improved reliability, there is an increased chance of both corrosion and mechanical degradation. In addition to operational temperatures, the environment in which an aircraft flies has a significant effect on the material life. Many contaminants are ingested by the engine and deposited on the turbine blades, which often leads to surface degradation. Depending on the ingested contaminants, temperature, and applied stresses, cracking can be initiated and propagated rapidly. This is particularly evident in the lower-shank regions of single-crystal nickel-based superalloy blades, which have recently experienced significant cracking.

This study aims to understand the mechanisms behind crack propagation in single-crystal nickel alloys exposed to intermediate temperatures, and when this propagation is either mechanically or chemically driven. This research started by assessing crack interaction mechanisms that were hypothesised to be both stagnating and accelerating crack growth, depending on specific length scales and crack formations. This was performed by integrating available experimental data to calibrate a phase field model that could predict the extension of cracks for different crack separations and layouts. The modelling results clearly characterised the length scales needed to encourage crack shielding, and which crack formations would see a stress intensification and consequently crack coalescence.

These results informed the decision to revisit the experimental setup to optimise which experiments were performed. Using this newly developed methodology, the salt deposition method was amended with the aim of isolating the deposition sites to minimise crack interaction mechanisms. The hypothesis was that significantly longer cracks would be ob-

served if this could be achieved. This was performed for both the C-ring (at 550°C), and corrosion-fatigue (at 700°C) tests. In the case of CMSX-4, the results were striking, with the C-ring seeing cracks as much as ten times the size of those previously seen. CMSX-10 however, did not show a significant difference, as such, a microstructural characterisation analysis was conducted, whereby the γ/γ' structure for the two alloys was replicated from microscopy data and further phase field models were run. The results showed that a more regular structure was more resistant to crack propagation owing to the misalignment of γ' , which caused stress relaxation in the γ channel and at the interface.

Finally, this thesis shows how the model, once calibrated for one material and species, can be used to approximate the response expected for another single-crystal nickel alloy or a change in the embrittling species, while accounting for a degree of uncertainty. This is not to say that modelling can or should replace experiments but rather to highlight that preliminary modelling results can be used to build a test matrix that can reduce the number of experiments that are run. It should be noted that this thesis does not focus on the chemical/corrosive aspects in much detail, but rather investigates the importance of stress. This thesis summarises the importance of integrating modelling, microscopy, and experiments to set and answer hypotheses more efficiently.

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List of Abbreviations

SATM	School of Aerospace, Technology and Manufacturing
GT	Gas Turbine
FEA	Finite Element Analysis
FEM	Finite Element Modelling
XFEM	Extended Finite Element Modelling
CZM	Cohesive Zone Model
PFM	Phase field Modelling
PF	Phase Field
UEL	User ELement
UMAT	User MATerial
CMSX-4	Canon Muskegon single-crystal 4
CMSX-10	Canon Muskegon single-crystal 10
CGR	Crack Growth Rate
SIF	Stress Intensity Factor
TBC	Thermal Barrier Coating
UTS	Ultimate Tensile Strength
LEFM	Linear Elastic Fracture Mechanics
DS	Directionally Solidified
SCC	Stress Corrosion Cracking
LTHC	Low Temperature Hot Corrosion
HC	Hot Corrosion
SEM	Scanning Electron Microscopy
XRM	X-ray Microscopy
CTOD	Crack Tip Opening Displacement

CTOA Crack Tip Opening Angle

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Chapter 1

Introduction

1.1 Background

Gas turbine (GT) technology has become the backbone of propulsion systems and is a key driver of energy production, particularly in the UK. They are thermodynamically more efficient than other power plants [1] (e.g. coal-powered plants), and natural gases produce fewer pollutants (such as NO_x and SO_x) than oil [2]. Manufacturers are constantly working to improve all aspects of their designs to increase efficiency. Fundamental and key principles such as aerodynamics, fluid mechanics, and material science should be considered in detail if manufacturers want to design safe and efficient products.

Extensive material degradation within GTs can threaten the overall efficiency of these systems [3] and are often caused by environmentally driven degradation mechanisms. These harsh environments are a result of high temperatures [4, 5], high stresses (including high centrifugal and vibratory stresses [6]), and the intake of corrosive species. These contaminants include sulphur [7], which can be produced as a by-product of the combustion of fuel sources [8], and from ingesting exhaust fumes from other aircraft during landing/take-off. This combination often results in the degradation of key components, and ultimately system performance through mechanisms such as hot corrosion (HC) [9], stress corrosion cracking (SCC), corrosion-fatigue [10], and creep [11]. As such, emphasis is placed on manufacturers developing technologies that can prevent such mechanisms, which include, but are not limited to, thermal barrier coatings [12]; adjusting specific cast-

ing techniques and heat treatments; or designing internal cooling systems [13, 14].

This thesis looks at a common issue within the industry, which is the cracking, and eventually complete failure, of components within the lower shank region of intermediate-pressure blades [15]. These materials are often single-crystal nickel-based superalloys that are exposed to temperatures between 450 and 750°C. These components are also subjected to both cyclic and static loading conditions [16], and owing to the nature of the systems, they introduce complicated dwell times and loading waveforms [17]. Literature shows that little work has been done so far, both experimentally and computationally, to understand the failure mechanisms of materials in this region of the GT, where component temperatures are lower in comparison to other areas. This damage leads to increased maintenance costs [18] for manufacturers and airlines that use their products. As such, emphasis is placed on pushing the boundary of knowledge to single out mechanisms and understand how to prevent them, thereby reducing the maintenance costs.

Given the complexity of both operational environments and loading conditions, this thesis assesses both corrosion and corrosion-fatigue; varying dwell times and frequencies; and operating temperatures, including both 550 and 700°C, to understand the role of mechanically and chemically driven crack propagation mechanisms.

1.2 Aims and Objectives

This thesis aims to understand the crack propagation mechanisms that occur in single-crystal nickel alloys exposed to corrosion, fatigue, and corrosion-fatigue conditions at 550 and 700°C. This thesis uses computational techniques to simulate GT environments to understand the synergy of corrosion and fracture mechanics principles on crack growth. As such, the following objectives were established.

1. To develop a single model that can account for both corrosion and fatigue mech-

anisms in crack growth in single-crystal nickel alloys at 550 and 700°C, and use experimental data to validate.

2. To validate set hypotheses by using modelling results to redefine how experiments are run, and justify the potential for test matrices, based on preliminary modelling results.
3. To predict the corrosion-fatigue life of components and understand how factors such as frequency, dwell time, and stress intensity factors all have an influence on what damage mechanisms dominate.

1.3 Thesis Structure

The remainder of this thesis is organised as follows.

1. Background, Theory, and Literature Review

The background includes a brief introduction, objectives, and highlights where the work has already been published and presented. A general summary and relevant background theory for the topics discussed in this thesis are highlighted in the literature review. The literature review begins by generalising the aero-GT environment and relevant materials before focusing on the different types of nickel-based superalloy structures. This section concludes with single-crystal nickel alloys, particularly CMSX-4 and CMSX-10. The focus is then placed on several high-temperature corrosion and corrosion-fatigue damage mechanisms that are known to occur in aero-GT environments (at intermediate to high temperatures), and which experimental setups are used to test for these conditions. Finally, the literature review covers the key fracture mechanics principles used throughout this thesis and models that embody these principles to determine crack propagation predictions.

2. Methodology and Results

Chapter 3 covers the methodology, implementation, and validation of phase field

modelling (PFM) and why it was selected as the most appropriate model to simulate crack predictions for the environments of interest. Chapter 4 focuses on early PFM results that integrated C-ring experimental data to determine the critical length scales and distributions of cracks required to observe crack-interaction mechanisms. Chapter 5 further assesses crack interaction mechanisms by first using modelling know-how to redesign the experimental setup to prove the extensive role of crack shielding in propagation, and by integrating microscopy data with models to further validate these assumptions. Chapter 6 summarises the roles of dwell time, frequency, and stress intensity factor (SIF), on which the damage mechanism is dominant. Chapter 7 summarises these key chapters and highlights how the boundary of knowledge is pushed, given what is now known.

3. Discussion, Conclusions, and Future Work

This thesis closes with a discussion and conclusions of the mentioned work before proposing future studies that can be done to continue this work.

1.4 Presentations and Publications

This work has been presented by the author of this thesis in the following conferences.

- M. Elsherkisi, oral presentation, "The Integration of Models and Experiments for Understanding Stress Corrosion Cracking at High Temperature" - Henry Royce Student Summit, September 2022
- M. Elsherkisi, oral presentation, "Integration of XRM and FEA into a digital twin" and "Phase field Modelling of Stress Corrosion Cracking in Superalloys at High Temperature" - TMS Superalloy, March 2023
- M. Elsherkisi, poster presentation, "Dominant Effect of Crack Shielding in Superalloy at High Temperature" - ECCC Conference – Creep & Fracture in High

Temperature Components, Design & Life Assessment, May 2023

- M.Elsherkisi, oral presentation, "Role of Salt Deposition Pattern on SCC in Superalloys at Intermediate Temperatures Informed by Modelling" - High Temperature Corrosion Gordon Research Seminar, July 2023
- M.Elsherkisi, poster presentation, "Role of Salt Deposition Pattern on SCC in Superalloys at Intermediate Temperatures Informed by Modelling" - High Temperature Corrosion Gordon Research Conference, July 2023
- M.Elsherkisi, oral presentation, "Integration of X-Ray Microscopy and Phase Field Modelling to Understand Stress Corrosion Cracking Mechanisms in Nickel Superalloys" - International Conference on Fracture, Damage and Structural Health Monitoring, September 2023

The work was also presented in the following journal articles.

- M. Elsherkisi, F. Duarte-Martinez, J. Mason-Flucke, S. Gray, G.M. Castelluccio
"Interaction of Stress Corrosion Cracks in Ni-Base Superalloys single-crystals"
(Has been accepted by Engineering Fracture Mechanics) - This paper makes up chapter 4
- M. Elsherkisi, F. Duarte-Martinez, J. Mason-Flucke, S. Gray, G.M. Castelluccio
"Role of Salt Deposition Pattern on Stress Corrosion Cracking in Superalloys at Intermediate Temperatures - Informed by Modelling" (in progress of writing) - This paper makes up part of chapter 5
- M. Elsherkisi, F. Duarte-Martinez, J. Mason-Flucke, S. Gray, G.M. Castelluccio
"Using Modelling Techniques to Understand the Role of Microstructure on Crack Propagation" (in progress of writing) - This paper makes up part of Chapters 5 and 6

Chapter 2

Literature Review

2.1 Aero Gas Turbine Environments and Materials

Both aero and industrial GT machinery require the detailed integration of multiple subsystems to function safely and efficiently as a singular machine. A small deviation from the norm (e.g., material degradation [19]) can cause the system to collapse. Material degradation is often associated with individual mechanisms, such as corrosion, creep, oxidation, fatigue, or corrosion-fatigue; however, owing to the complexity of these systems, it can often be a combination of them [20, 21], which leads to the failure of components. In the case of corrosion, factors such as temperature and impurities [22] (which may come from combustion [23]) can result in catastrophic defects such as cracking, meaning that manufacturers must remove these components from service to ensure passenger safety. The literature has shown that in the case of many superalloys (specifically nickel-based), the combination of sulphur (generally as a byproduct from the combustion of jet fuel) and NaCl (a major constituent of sea salt) ingested from the environment can cause detrimental damage to components through corrosion processes such as hot corrosion [24, 25, 26, 27]. Furthermore, the combination of corrosion-fatigue conditions is often a cause of concern [28, 9], because of its ability to rapidly induce early fracture, which can often result in unplanned maintenance and financial loss.

As such, the appropriate selection of materials for each section of the GT is essential to prolong its life and minimise any unplanned maintenance. A wide variety of superal-

loys are used in GT systems depending on their strengths and weaknesses in relation to the environment at that given point. For example, titanium- and nickel-based superalloys are among the most popular alloy options. Titanium has a well-established strength-to-weight ratio and excellent resistance to both corrosion and creep [29], but it is limited to temperatures below 700°C owing to its poor resistance to oxidation [30]. Therefore, it is limited to the compressor and fan sections. The combustion chamber can reach temperatures as high as 2000°C; therefore, cobalt-based superalloys are often selected for these components owing to their excellent heat [31] and creep [32] resistance properties. The same applies to nickel-based superalloys.

This thesis focuses primarily on corrosion and corrosion-fatigue conditions for temperatures ranging between 550-700°C, which aligns with that of intermediate pressure (IP) turbine blades, specifically the under-platform regions of the blades (highlighted in Figure 2.1). Thus, nickel-based superalloys were selected as the primary focus of this investigation.

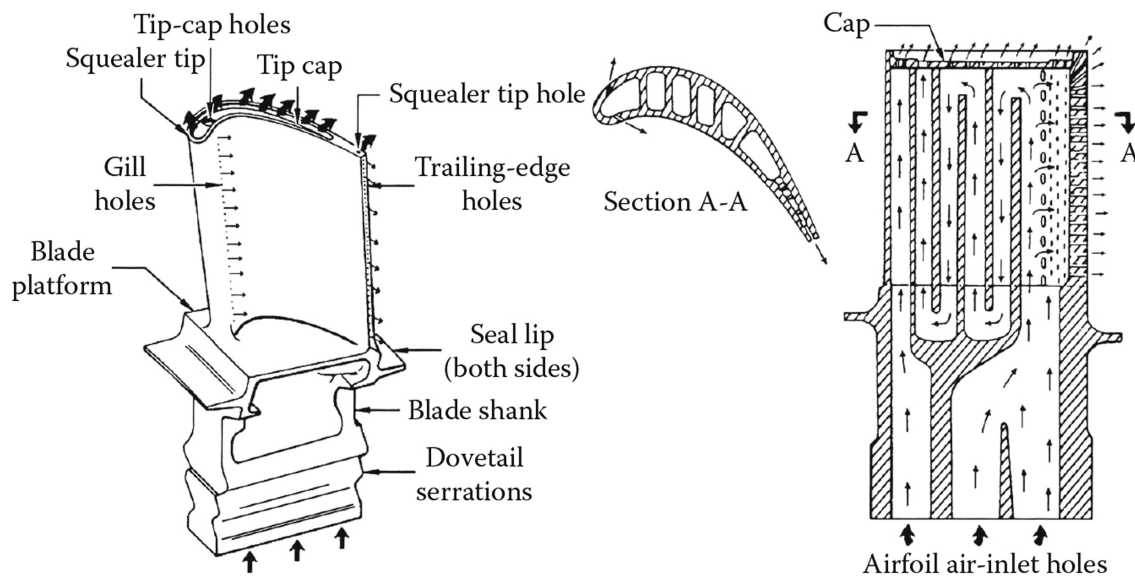


Figure 2.1: A schematic representing a gas turbine blade highlighting the under platform region of the blades [33]

2.1.1 Nickel-based Superalloys - Microstructure and Composition

Nickel superalloys can be formed in one of two ways, comprising either polycrystalline or single-crystal microstructures. Polycrystal materials are composed of multiple grains and tend to be used as disk materials [34], whereas single-crystals are composed of a singular grain [35]. Directionally solidified (DS) structures branch under polycrystals and have longitudinal grains oriented parallel to the vertical direction of the blade.

This thesis looks at single-crystal alloys in general, but is implemented for two nickel alloys in particular, CMSX-4 [36] and CMSX-10 [37], as they are both popular alloys in the industrial and aero GT industries and therefore, require a good understanding of their responses under corrosion and fatigue conditions. Both alloys are high Re-containing alloys with CMSX-4 and CMSX-10 being second- and third-generation single-crystal nickel-based superalloys, respectively. Both have a coherent γ' volume fraction of approximately 70% [38, 39]; their compositions are listed in Tables 2.1 and 2.2. Single-crystal nickel alloys are favoured over polycrystalline blades because the absence of grains provides greater creep and thermal fatigue resistance as well as resistance to grain boundary sliding [32]. Secondary elements such as boron and zirconium are also used to strengthen grain boundaries; however, their absence in single crystals results in a higher melting temperature [40]. Single crystals are also anisotropic in nature, meaning that they have different elastic properties in each direction, such that the material's fatigue life can be improved by using a crystallographic direction that follows that of the lowest Young's modulus, as the lower stresses follow this direction. It is also known that for many single crystals, there is an increase in both tensile and yield strengths as the temperature increases [41, 42], but this is only to a certain point. CMSX-4 is a perfect example, which shows an increase in both the yield and tensile stress up to 800°C [43].

Nickel-based superalloys are often manufactured to achieve two phases: the γ matrix,

which is a solid-solution phase comprising a face-centred cubic (FCC) structure, where atoms are randomly distributed. The γ' precipitate has an $L1_2$ structure that generally has a chemical formula of Ni_3Ti , Ni_3Al , or a combination of the two $\text{Ni}_3(\text{Al}, \text{Ti})$, where Ni is located at the centre of the faces and the aluminium/titanium make up the corners of the cubes [44], as shown in Figure 2.2.

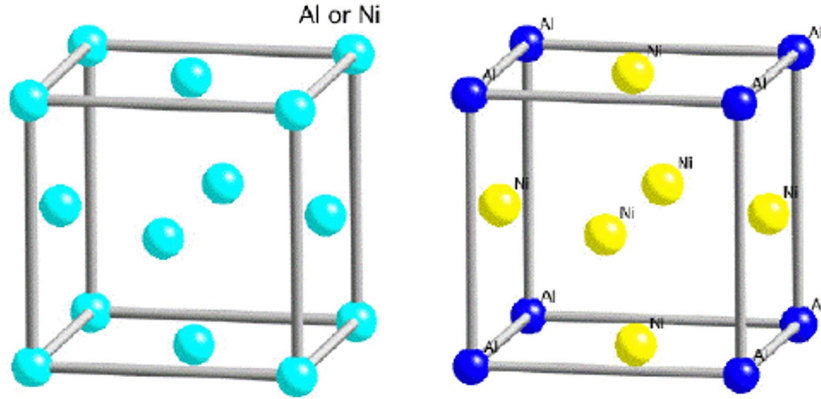


Figure 2.2: The arrangements that make up the structures of both the gamma phase (left) and the gamma prime precipitate (right) [45]

The γ' structure provides strength to the material by creating barriers that dislocations must overcome to continue moving through the material [46]; the properties of the bulk material can be modified by adjusting the size, shape, and orientation of these precipitates [47, 48]. The coherency (or lattice misfit) of these two phases affects the properties of bulk materials. If the material has high coherence between γ and γ' , it leads to a small lattice misfit value and vice versa. A higher misfit leads to γ' precipitates that are cubical in nature, whereas lower values lead to more spherical shapes.

Table 2.1: Composition of CMSX-4 (wt.percentage) [49]

Ni	Cr	Al	Co	Ta	W	Mo	Ti	Re	Hf
Balance	6.5	5.6	9.0	6.5	6.0	0.6	1.0	3.0	0.1

Table 2.2: Composition of CMSX-10 (wt.percentage) [50]

Ni	Cr	Co	Mo	Ti	Al	Ta	Hf	W	Nb	Re
Balance	2.0	3.0	0.4	0.2	5.7	8.0	0.03	5.0	0.1	6.0

2.2 Damage Mechanisms

The combination of harsh operating environments and extreme loading conditions results in extensive material degradation, meaning that early maintenance is required to remove components before they reach catastrophic failure. Therefore, it is crucial that action is taken to either design new materials that can withstand these environments or measures are taken to protect alloys currently in circulation. Typically, failure modes such as oxidation, corrosion, and fatigue are among the common mechanisms that can cause catastrophic failure [51, 52].

2.2.1 Hot Corrosion Mechanisms

Owing to both the high operating temperatures and deposition of salts on turbine blades, hot corrosion (HC) is among the most damaging mechanisms observed in aero-GTs [53]. For HC to occur, the salt contaminants must be either in a liquid phase or in an environment that encourages a reaction to see a transition from a solid to a liquid. Potential salts include Na_2SO_4 and NaCl (a major constituent of sea salt) [54], which degrade the protective oxide layers of materials. HC can be split into two further mechanistic regimes depending on many environmental parameters, specifically operational temperatures. These mechanisms are Type I HC (high-temperature hot corrosion) and Type II (low-temperature hot corrosion) and are summarised in Figure 2.3[55].

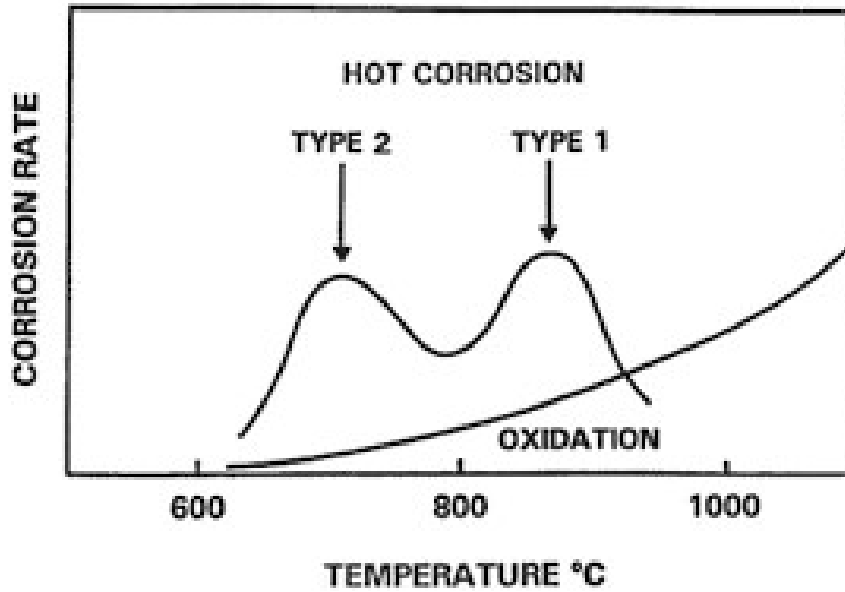
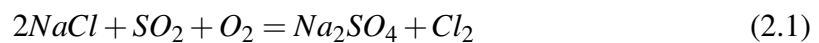


Figure 2.3: Temperature ranges expected to see both type I and II HC [55]

Generally, type I HC is seen between temperatures of 850°C and 950°C [56], where the salt deposit, is already in a molten phase. An example of this molten salt would be Na_2SO_4 , which is formed by a chemical reaction between the ingested NaCl (a major constituent of sea salt) and the presence of sulphur, either from exhaust gases from other aircraft or produced as a by-product from the fuel, and is highlighted by Equation 2.1[57]. This reaction between the salt and sulphur compounds degrades the protective oxide of the material and allows for inward diffusion of sulphur, leading to internal sulfidation.



Conversely, type II HC occurs at temperatures as low as 550°C [58] (or even lower when stress-assisted [27]) which is below the melting temperature of Na_2SO_4 such that the mechanism is driven by the formation of a liquid eutectic in the form of NiSO_4 - Na_2SO_4 or CoSO_4 - Na_2SO_4 . This is followed by the inward diffusion of SO_3 (which requires the correct partial pressures) and outwards diffusion of nickel and cobalt oxides. Under the right conditions, type II HC can be extremely detrimental to the components.

It should be noted that despite both HC mechanisms being responsible for degradation in aero GTs, it is likely that crack initiation and propagation are accelerated by an accumulation of mechanisms [20, 21].

2.2.2 Stress Corrosion Cracking Mechanisms

Stress corrosion cracking (SCC) degrades a material owing to the combination of tensile stresses, a susceptible material microstructure, and a corrosive (or oxidation-driven) environment [59], as summarised in Figure 2.4. SCC often occurs below the typical stress and temperatures associated with cracking but can result in unexpected, rapid, and catastrophic failure of components. Because the mechanism is both material- and environment-specific, it is difficult to understand and predict [60]. SCC is extensive in materials used in aerospace components, chemical and power plants, piping, and wind turbines.

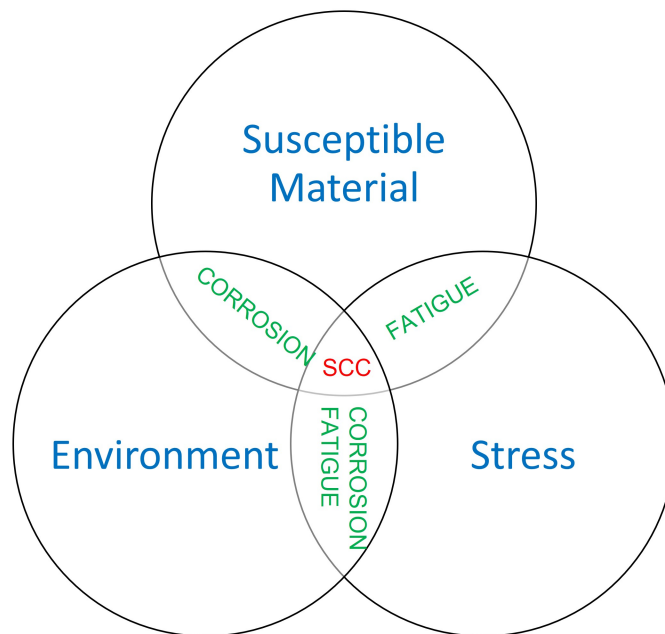


Figure 2.4: A Venn diagram to highlight the necessary conditions needed to initiate SCC, and the resultant mechanism if one of these variables was to be removed [61]

SCC can be classified into different mechanisms depending on the combination of materials and environments. For example, chloride-assisted SCC occurs in many steels and

alloys in the presence of both chlorine and oxygen ions at high temperatures and under tensile stress. Caustic embrittlement is similar to the above mechanism but requires the presence of hydrogen as opposed to oxygen [62]. In the oil and gas industry, SCC occurs in steels in the presence of hydrogen sulphide [63]. Finally, seasonal cracking occurs in brass in the presence of ammonia [64].

Nickel alloys tend to be more resistant to corrosion than those previously mentioned [65]; however, they still occur. As with other alloys, nickel alloys suffer from either uniform or localised SCC [66]. Uniform SCC occurs under a reducing effect, whereby hydrogen is released from acids, such as HCl or H₂SO₄. Localised SCC is observed in the form of pitting or crevice corrosion, in which these local structures act as stress magnifiers [67] and are considered precursors to cracking. The transition from pit-to-crack is generally considered when two factors have been met: the first is when the pit now exceeds a minimum threshold value which corresponds to a minimum SIF and when the crack growth rate (CGR) exceeds that of the pit [68, 69].

Several measures can be taken to minimise the likelihood of SCC. For example, because tensile stress is the driving force for SCC, reducing the overall stress levels that components are exposed to minimises the chance of it occurring. Treatments such as shot-peening, create compressive residual stresses on the surface of the components, and as such, are recommended to prevent SCC [70]. Thermal barrier coatings (TBCs) and other protective coatings are viable protective measures employed by many industries, including aero-and industrial GT. Specific heat treatments have also been adopted to produce less-susceptible microstructures.

2.2.3 Role of Fatigue Mechanisms

Fatigue failure of materials is observed when components are exposed to cyclic loading, where the applied stresses are considerably lower than the ultimate tensile strength

(UTS), over a prolonged period of time [71]. The fatigue life of a component is defined as the number of loading cycles a material can withstand before it reaches complete failure [72]. Factors that contribute to increasing/decreasing the fatigue life include material properties, material defects, whether it is a single or polycrystalline [73] material (and therefore, grain size has an effect), temperature [74], and the presence of corrosion species [72]. The fatigue life can be equated to the life under crack initiation and crack propagation, as summarised below.

$$\frac{da}{dN_{Total}} = \frac{da}{dN_{Initiation}} + \frac{da}{dN_{Propagation}} \quad (2.2)$$

Extensive work has been conducted to understand the role of fatigue on the CGR of single-crystal nickel alloys. Sengupta and Putanda [75] assessed the CMSX-4 compact tension (CT) samples at 25, 650, and 800°C. At 25°C, the cracks propagated along the $\{111\}$ plane in a zigzag fashion. However, when elevated to 650 and 800°C, there was a shift to the $\{001\}$ plane accompanied by smooth and flat cracked surfaces. Joyce et al. [76] evaluated CMSX-4 fatigue behaviour in air and vacuum at 650°C, and saw generally higher propagation rates in air compared to vacuum. They attributed this to the effects of both oxidation-induced closure and increased slip homogenisation, which suppressed the faster stage I CGR. In addition, on a macroscopic scale, crack growth occurred along alternating $\{111\}$ planes at low to mid- ΔK to coarser slip-band cracking along the planes for ΔK values above a critical threshold. They also tested two different secondary crystal orientations and reported that a change in both the orientation and environment affected the final CGR. Palmert et al. [77] also assessed a single-crystal nickel alloy in the form of a disked CT specimen, at 20, 500, and 750°C and with different crystal orientations. The dwell times were evaluated at 750°C. They observed that the CGR generally increased with increasing temperature, and that non-crystallographic crack growth was dominant at 20°C. In contrast, the tendency for crystallographic crack growth was shown to be highest at 500°C, but more tests are required to understand why. In the case of the highest tem-

perature test, da/dN was shown to increase with higher dwell times at the maximum load; however, this dominant effect of the hold time decreased with decreasing ΔK .

Evangelou et al. [78] assessed the combined effects of oxidation and fatigue on CGR in CMSX-4 at 550°C. Different frequencies, dwell times, and environments were tested to further understand the driving force of fatigue CGR. They concluded that it is indeed a complex issue to consider at this temperature and that it is likely that multiple mechanisms compete at the crack tip. They reported both crystallographic slip and γ' shearing as well as cracking in the γ channels, which are likely to be thermally activated. Further work also showed that oxidation did not have an embrittling effect on CGR at these temperatures, but finger-like protrusions that extended past the crack tip were observed, which was attributed to the presence of plastic strain at the crack tip.

While the life of a component with a pure fatigue-driven crack is determined by the number of cycles (and notably the frequency effect), it is well known that the presence of corrosive and embrittling environments can reduce the number of cycles to failure. The work from Evangelou [78] highlighted the role of the environment on fatigue crack growth and emphasised the importance of further investigating corrosion-fatigue mechanisms.

2.2.4 HC, SCC, and Fatigue Single-Crystal Nickel-Based Superalloys

Owing to the complex loading conditions in GTs [22], varying temperatures (not only between different sections of the engine, but also the cyclical heating and cooling of components when turned on/off [79]), and extremely corrosive environments, it is likely that the tortuous combination of HC, SCC, and fatigue all contribute to material failure [20, 21].

Extensive work has already been conducted to show that HC-SCC mechanisms oc-

cur at temperatures as low as 550°C. Brooking [24] investigated CMSX-4 in corrosion environments (at intermediate temperatures) for monotonic and cyclic loading to assess the HC-SCC and corrosion-fatigue mechanisms, respectively. Initial studies [80] used C-ring tests to understand the role of static loads under corrosion conditions at 550°C, and several conclusions were drawn. SEM/EDX characterisation techniques concluded that the chemical composition of the products found post-exposure aligned with what is expected from SCC, with evidence of pitting morphology. They observed a shift in the degradation mechanisms when combined with static loads (as low as 500 MPa), resulting in an SC-LTHC-type mechanism. Using FEA to calculate the stress intensity of the crack tips, it was also concluded that the presence of HC-type mechanisms can reduce the stress intensity threshold (K_{Th}) needed to see cracks by as much as 75%.

Further work by Chapman et al. [81] was performed to investigate the detrimental effects of corrosive environments on the fatigue life of CMSX-4 samples. They found that by decreasing the ramp rate, the fatigue life decreased. It was also noted that the presence of corrosive species resulted in a shift in the crack propagation plane from the $\{111\}$ to the $\{001\}$ plane with an increase in the corrosion attack.

Cockings et al. [58] conducted a study on the effects of corrosion and fatigue on the life of two nickel superalloys (U720Li and RR1000) used for disk components. The aim was to replicate the damage features observed within the service but to combine a varying salt flux and cyclic loading to understand the morphology of corrosive products and the fatigue life of the material. There was shown to be a noticeable relationship between the loading conditions (peak stress levels, cyclic/monotonic loads, and salt flux) and the expected life of the component. A significant reduction in the fatigue life was observed.

These are amongst the many examples that highlight how, under differing conditions, the degradation mechanism changes at both the macro- and micro-scales. This empha-

sises the complexity of GT systems and how factors such as varying temperatures, which can change rapidly between neighbouring sections, can be sensitive to accelerating or reducing the crack propagation rate [82].

2.3 Experimental Evaluation of Damage Mechanisms

To replicate the harsh conditions mentioned above, an appropriate experimental setup is required. This thesis demonstrates how modelling results can be used to better inform the experimental setup to reduce overall costs and to set and prove hypotheses. Therefore, it is necessary to cover the experimental setup, including that of the C-ring and fatigue tests.

2.3.1 C-ring Setup

C-ring tests are a simple means of determining a material's susceptibility to stress-induced corrosion mechanisms, and their small size makes it easier to run many tests simultaneously in a single furnace [83], making them a financially sustainable technique. C-ring tests were used to generate a high multi-axial stress gradient [27] and understand the effects under different experimental conditions.

The C-ring tests used in this study follow the same guidelines as those in [24, 25]. The tests were performed using specimens machined from CMSX-4 bars which were solutioned and heat-treated according to the specifications of Rolls-Royce Plc. The samples had a crystal orientation in which the [001] crystallographic orientation was aligned with the cylindrical axis. The C-ring tests follow the ISO 7539-5 guidelines [84, 85], and the C-ring dimensions are highlighted in Figure 2.5. The figure shows that the C-ring has two holes (approximately 6.5 ± 0.5 mm) which allows for a bolt, nut, and two washers to be passed, which when tightened, creates this tensile stress along the apex of the C-ring. Commercial standard waspalloy are used for nuts, washers, and bolts because they have

been shown to minimise the stress relaxation effects on the C-ring [86].

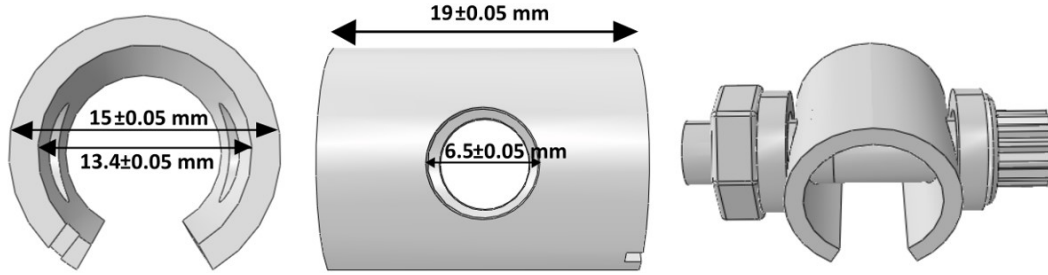


Figure 2.5: C-ring geometry without and with the bolt that is used to induce tensile stress and accompanying dimensions [86]

The methodology of Duarte et al. [86] displaced the C-ring to a predetermined value, which was calculated using FEA to reach the desired peak stress values. The samples were then cleaned with isopropyl alcohol (IPA) before being heated on a hot plate to approximately 200°C. A spraying method was used to deposit a salt + deionised water solution onto the top surface of the C-ring, which had a flux of approximately 1.2 $\mu\text{g}/\text{cm}^2/\text{hr}$ before being heated again on the hot plate to evaporate any excess water. Subsequently, the loaded samples, now layered with salt contaminants, were placed in a furnace where they were exposed to high temperatures and different environmental gases. Where appropriate, the samples were resalted every 50 hr until the test was completed. The time, temperature, and specific gaseous environment change depending on the degradation mechanism of interest.

2.3.2 Corrosion-Fatigue Setup

The purpose of the corrosion-fatigue test is to evaluate the role of corrosion mechanisms, such as those mentioned above, on the number of cycles a material can experience before reaching failure [87]. The results of a series of these fatigue tests can be used to produce S-N curves for a given material (i.e. the number of cycles, $N(S)$, a material

will endure before failure, for a given stress range, S) and to understand how factors such as temperature, exposure environment, and loading parameters can affect the expected life. An S-N curve is made up of three regions (as seen in Figure 2.6), where region I shows tests being carried out at or above the yield strength of the material (and hence is in the elastoplastic regime); region II the material is loaded to a stress level that is below the yield (and so is in the elastic regime); and region III essentially defines a material to experience unlimited life [88].

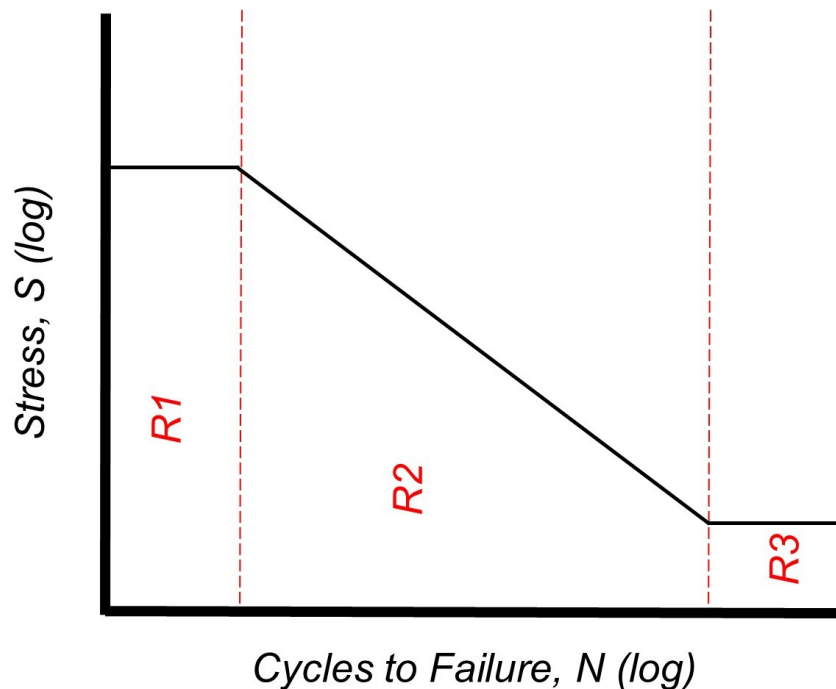


Figure 2.6: A generic SN curve highlighting the three separate regions that a material will experience (reconstructed from [89])

The work by Brooking et al. [27] followed the methodology in which samples, prior to testing, were cleaned with IPA and an ultrasonic cleaning tank and then placed on a hot plate to heat the material. A salt flux + deionised water solution was used to the notches before being placed on the hot plate to remove excess water. The test piece was then loaded into a fatigue rig, where several test parameters were adjusted to simulate a given environment.

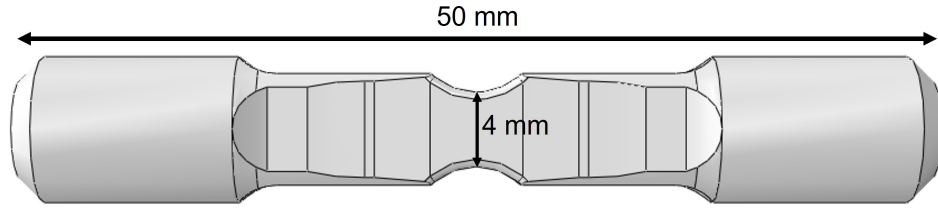


Figure 2.7: Geometry of the fatigue specimen that was used in all corrosion-fatigue experiments and simulations

Trapezoidal waveforms were used (which is summarised in Figure 2.8), where T_1 is the time to increase to the maximum load; T_2 is the dwell time for which the maximum applied load is held; T_3 is the time to decrease back to the minimum load; and finally, T_4 is the time for which the minimum load is maintained. The tests were displacement controlled and run until failure, with some tests being resalted at regular intervals.

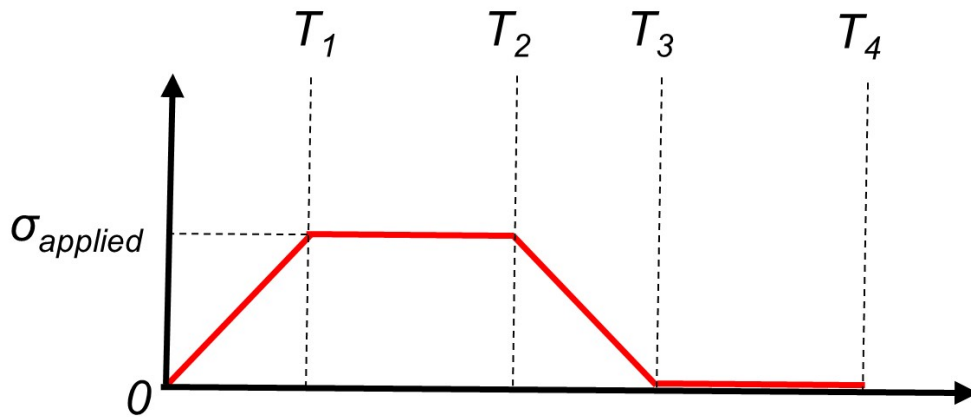


Figure 2.8: A schematic to represent the trapezoidal loading waveform used in all corrosion-fatigue experiments referred to in this thesis. T_1 is the ramp up, T_2 is the hold time, T_3 is the ramp down, and T_4 is the hold time without applied load. Reconstructed from [27]

2.4 Elastic and Plastic Material Response

A key aspect of fracture mechanics and materials science is the deformation of a material which is considered to be either elastic or plastic. Elasticity refers to the ability of a material to resist permanent damage and return to its original size and shape when force is removed [90]. This differs from plasticity, in which a material experiences permanent deformation when a sufficient load is applied to the system, and will not return to its typical size and shape when unloaded [91]. At the atomic scale, elastic deformation occurs when atomic bonds in a material are stretched or distorted [92]. However, with plastic deformation, the layers of atomic bonds shear as they pass over each other [93]. Figure 2.9 shows both forms of deformation at the atomic scale.

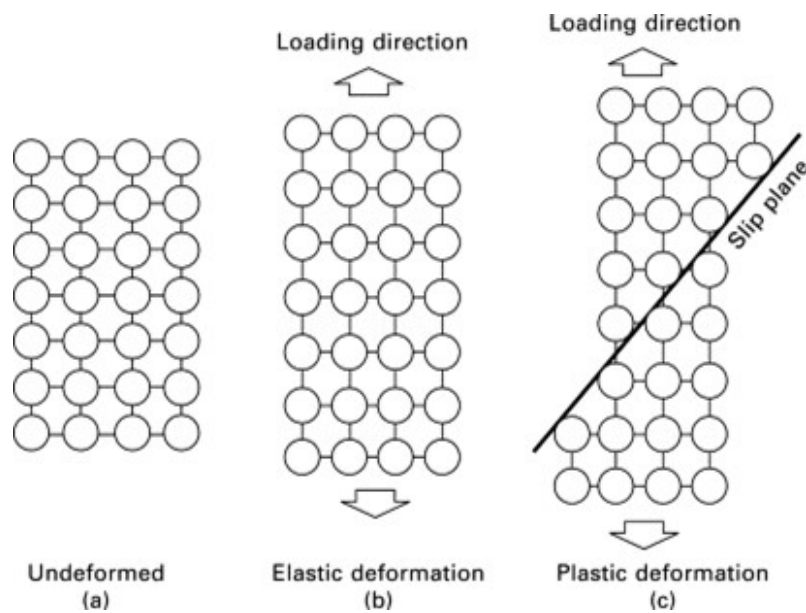


Figure 2.9: Visualising the atomic structure for a material that is undeformed, undergoing elastic deformation, and finally undergoing plastic deformation [93]

To understand the material response, the stress–strain relationship must first be determined. Figure 2.10 shows a typical stress versus strain plot for a material that has reached a critical fracture and highlights the two distinct regions, namely the plastic and elastic strain regimes [94]. When combined, the elastic and plastic strains totalled the total strain

of the system. Stage 1 refers to the linear elastic regime, where the stress and strain are proportional, that is, they obey Hooke's law, and the slope of the line is equal to Young's modulus [95]. This stage only considers the elastic deformation. Stage 2 introduces the plastic regime, where the stress is now equal to the yield of the material; this is called the strain-hardening region [96]. The stress continued to increase until it reached the ultimate tensile strength (or UTS). The third stage is referred to as the necking region, in which the cross-sectional area decreases significantly below the average [97]. This stage ends with complete fracture of the material [98]. This indicates the start of the fourth and final stages, which is the unloading of the material owing to failure [99].

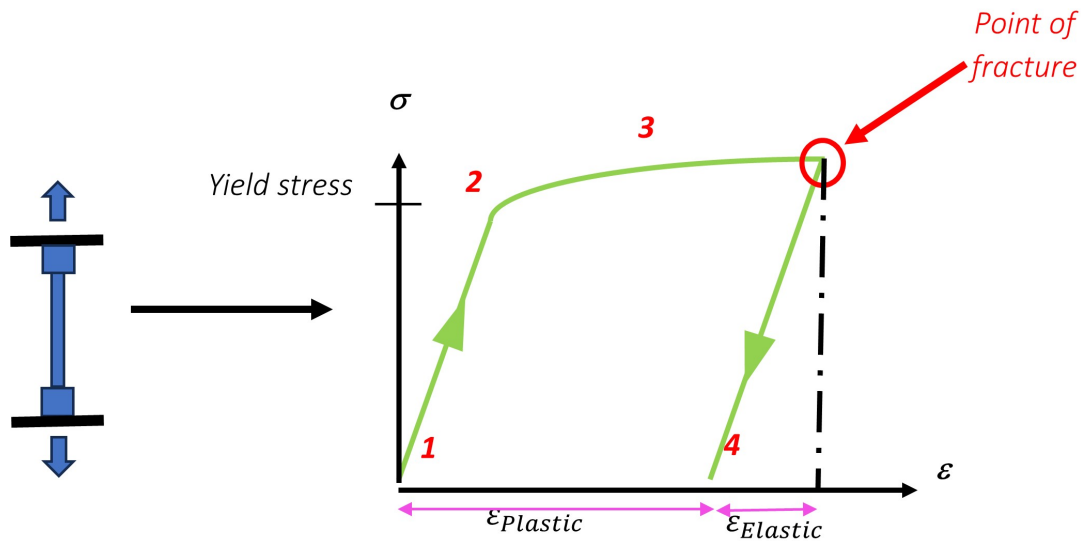


Figure 2.10: A generic stress-strain curve highlighting the elastic and plastic regimes experienced by a component as it reaches critical failure [95]

There are two methods to solve the mathematical equations involved in a material response: analytical and numerical [100]. Analytical solutions provide an analytical or exact solution to governing equations [101] and can be adopted to understand simplified problems. On the other hand, numerical solutions, such as FEA, provide an approximation to the issue and are used for more complex problems [71]. Numerical solutions can come in many forms, and depending on the application, they can account for an elastic or plastic

response. Elastic solutions can assume a linear or nonlinear elastic response, where both return to their original state once unloaded [102] but have different relationships between stress and strain. A linear elastic response assumes that the relationship is linear and obeys Hooke's law, whereas a non-linear response does not and is more complex [103]. Plasticity models, however, can be broken down into two forms, one which accounts for isotropic hardening and the other that assumes kinematic hardening, both of which have their ideal applications [104]. Plasticity modelling is used to describe the material response when the applied stresses exceed the material yield [105]. Examples of plasticity models include, but are not limited to, the Tresca, von Mises, Drucker–Prager, and Crystal Plasticity models.

2.5 Computational Evaluation of Damage Mechanisms

Computational modelling can provide data that cannot easily be measured experimentally, including the driving force for crack extension (such as SIF [106]), CGR, and understanding of the crack growth path and mechanism. Modelling techniques can reveal different aspects of the crack propagation process [107], and as such, it is important to consider which model is most appropriate to the problem at hand. This is best accomplished by understanding the available inputs (e.g. experimental data for validation/calibration [108]) and the desired outputs. This section of the thesis will provide some examples of different models used to predict crack growth and explain why PFM was used for this

2.5.1 Alternative Modelling Techniques

Extended Finite Element Methods

Recently, the use of extended finite element modelling (XFEM)[109] has become increasingly popular for predicting crack extension because it minimises the need to re-mesh [110]. The term "extended" refers to the improvement in the methods used to extend

the methods already used in conventional FEA to predict crack propagation. This is because, as cracks propagate, discontinuities form when the displacement is approximated in the FEA [111]. Discontinuities can be divided into two forms: strong discontinuities which occur when solving solution variables (e.g. displacements); and so includes displacement jumps such as cracks and holes [112]. The second form includes weak discontinuities and occur within the derivations of these solution variables and involve variations in displacements (e.g. spikes in strain values)[113].

XFEM was first developed in 1999 by Moes et al. [114], who modelled fatigue crack propagation in modes I and II loading with a 2D plate with two holes. Other uses of this technique include the work of Sukumar et al. [115] who conducted the first XFEM test to simulate the fatigue crack extension through a 3D solid body (Mode I cracking). The “level set method” was used to track the location of the crack front and the “fast marching” technique to extend the crack front. They predicted the process of converting from what was initially an elliptical crack to a penny-shaped crack, but did not compare it to any experimental data. Zhan et al. [116] proposed a modelling framework to predict the fatigue life of metallic components, where they used FEM techniques, informed by continuum damage mechanics, to predict the initiation life and used XFEM to predict crack extension. However, their life prediction was 28% shorter than the experimental results, indicating that this technique requires further work to improve its predictive capabilities.

Where XFEM prevails other FEM techniques fail, specifically in relation to meshing techniques [117]. In most cases, the crack needs to be explicitly meshed, and thus a high density of nodes needs to be placed along the crack, at the crack tips, and in the regions of predicted crack extension to accurately represent the asymptotic fields in the vicinity of the crack tips [118]. As cracks propagate, re-meshing techniques are required to maintain accuracy, resulting in high computational costs, especially in 3D examples. However, XFEM rarely requires re-meshing [119]. Another advantage of XFEM is that

it is merely a technique for representing crack extension in a body, but there is no fixed method that defines how they propagate. As such, it gives the user the flexibility to set different propagation criteria [120], as different material microstructures will fracture and fail in different ways.

However, there are some disadvantages to using XFEM. XFEM is a difficult process to implement, especially when complex geometries and 3D problems are considered [121]. Implementation often requires complex integration routines and process stability of the simulation, especially when incremental cutbacks are very small. Another major drawback of XFEM is that it can only be used for LEFM [122] and this thesis is primarily concerned with crack propagation under SCC and corrosion-fatigue conditions. Although XFEM has been used to assess SCC issues, the integration of SCC degradation mechanisms and XFEM is a complex process that often yields unreliable predictions. For example, Bashir et al. [123] assessed the mesh density effect of XFEM on SIF, strain gradient, and SCC growth rates and found that the predicted values of SCC CGR were incompatible with the values obtained from experimental results. Therefore, XFEM was not selected as an appropriate and efficient tool for this study.

Cohesive Zone Modelling

Barenblatt [124] was the first to propose a theory that fracture is the process of separating material surfaces, and this theory constitutes the backbone of the cohesive zone model (CZM). The cohesive zone propagation follows a theory similar to Griffith's theory [125]. It assumes that the area ahead of a crack tip is a cohesive zone comprised of an upper and lower surface which is held by cohesive traction, which can be related back to the displacement needed to separate the two layers [126]. This is often referred to as either a cohesive law or a traction-cohesive law [127]. When a force is applied to the body, the two surfaces begin to separate, and when a critical separation displacement is met, crack growth occurs. However, the cohesive traction law must be a nonzero integer to prevent

stress singularity at the crack tips. CZM is constructed in such a way that traction between surfaces gradually increases until a critical ceiling is met, and then returns to zero as the distance between separated surfaces increases [128].

The popularity of CZM means that it is readily available [129] in most FEM packages such as ABAQUS, making it an easy option for many researchers. The CZM can also be used to simulate the response for uncracked components (e.g. smooth-notched specimens such as the double-notched fatigue sample), which is a significant advantage over other crack prediction techniques that require starting with a pre-cracked body [130] (like those driven by LEFM).

However, there are a few drawbacks to CZM; for example, for more complex geometries and scenarios, predicting material behaviour often comes with complex solutions. CZM also requires the user to already know the crack propagation path [131], which makes it difficult to model the initiation and propagation of cracks in environments that encourage SCC because of the random nature of degradation. Therefore, the CZM was not selected as an appropriate and efficient tool for this study.

2.5.2 Why Phase Field Modelling?

The use of PFM for predicting brittle fractures can be traced back to as early as the 1990s [132] and was first introduced by Langer [133] and Fix [134]. It is often used to understand mechanisms such as phase transformation, grain growth, and more recently, crack extension.

PFM was selected as the most effective computational method for modelling corrosion, fatigue, and corrosion-fatigue conditions owing to its relatively straightforward approach of incorporating multiple physics-based driving forces [135]. PFM can solve the diffusion equation for a corrosive species in a particular material and simultaneously

degrade the bulk material based on this result [136]. This allows a predefined crack to extend when the material's fracture toughness drops below a critical point. The tensile stress experienced in both the C-ring and fatigue tests could also be accounted for when solving the diffusion equation. The transition between phases is highlighted using the damage parameter Φ which is a diffusive interface and is driven by the driving force [132]. This allows the user to track the transition from undamaged to cracked material. The methodology section 3.1 assesses PFM in greater detail and breaks down the mathematical structure of the model.

However, there are some disadvantages, the most obvious of which is the lack of physics behind the length scale parameter [137], which will be discussed later in this thesis, and a potential improvement is suggested. The PF approach also requires a high mesh density in the area of propagation [137], meaning that the user must already know the expected crack extension path. The high mesh density also equates to a higher computational cost [138], particularly for complex geometries such as the fatigue specimen used in this methodology.

Despite this, PFM has proven to be a powerful tool for further understanding the crack propagation mechanisms in single-crystal nickel superalloys exposed to GT environments and has provided a fresh perspective on how we approach the experiments that are run.

2.6 Stress Analysis of Cracked Specimens

Fracture mechanics is used to assess materials with one (or more) cracks to understand when catastrophic failure occurs, and is used to improve the mechanical performance of components. Griffith [125] introduced the notion in the early 1920s that all material components contained some kind of flaw, including cracks, and that these flaws are a

contributing factor to the reduction of a material's ability to prevent catastrophic failure. Fracture mechanics states that crack propagation is driven by an appropriate driving force that is measured using the SIF (K) or the J-integral [139], which can be used to describe the dominant stress fields surrounding the crack tips. Hence, fracture mechanics principles can be used to increase the fracture toughness of materials to minimise the likelihood of crack propagation.

2.6.1 Stress Intensity Factor

The three modes of stressing are opening (tensile), in-plane shear (sliding), and out-of-plane shear (tearing), also known as modes I, II, and III [140], respectively, as shown in Figure 2.11. Mode I is often the most common of the three, and is observed after a tensile stress is applied to the body, resulting in the separation of two faces [141]. For modes II and III, the key difference is that for mode II, the shearing is normal to the crack front, whereas for mode III, it is parallel to the crack front [142]. Any one of these modes or a combination of modes results in cracking.

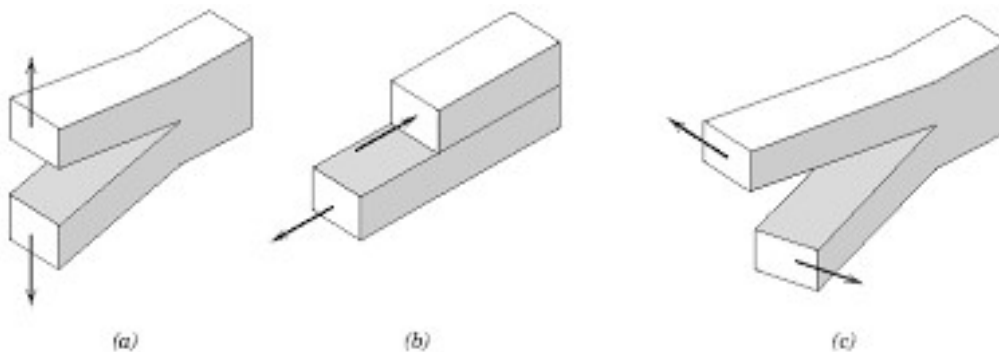


Figure 2.11: The modes of a crack extension where a, b, and c correspond to opening, in-plane shear, and out-of-plane shear respectively [143]

Plastic deformation is experienced by a material just before it reaches the point of brittle failure [144, 145]; as such, the traditional Griffith theory does not apply in this instance. However, a contribution by Irwin introduced the idea that crack-tip stresses are a function of both applied stress and crack size, better known as the SIF. Fracture mechanics describes the SIF (K) as a parameter that measures the stress state in the vicinity of crack

tips, as a result of either an applied load or residual stresses [146]. It is worth noting that SIF assumes a crack to be in a linear elastic body and is mathematically sharp [140]. The magnitude of the SIF ($\text{MPa}\sqrt{m}$) can be analytically described using Equations 2.3 and 2.4 and depends on the applied stress (σ), crack depth (a), and crack geometry (Y) [147, 148].

$$K = \sigma\sqrt{\pi aY} \quad (2.3)$$

whereby, Y , is dependent on both the crack depth, a , and the width, w , of the specimen and is denoted by,

$$Y = f\left(\frac{a}{W}\right) \quad (2.4)$$

According to LEFM, the stress distribution at the crack tip (σ_{ij}), using polar coordinates as seen in Figure 2.12, can be given using the following formula,

$$\sigma_{ij}(r, \theta) = \frac{K}{\sqrt{2\pi r}} f_{ij}(\theta) + \text{higher order terms} \quad (2.5)$$

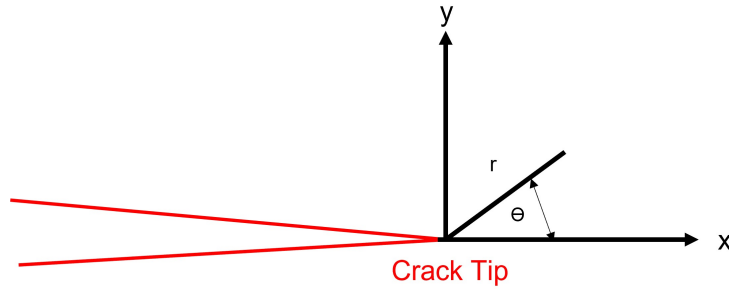


Figure 2.12: A schematic that represents the polar coordinates at the crack tip [149]

where $f_{ij}(\theta)$ varies with both the applied load and geometry. As r moves towards 0, theoretically, σ_{ij} moves towards ∞ , causing a stress singularity [150]. However, in reality, this relationship does not work with low values of r (or close to the crack tip), as plasticity is likely to occur when stresses exceed the yield strength, and as such, LEFM can no longer be considered.

2.6.2 Fracture Toughness

The fracture toughness of a component is an important aspect of material science, and is defined as the ability of a brittle material to resist the propagation of a flaw under applied stress [151]. Such flaws include voids, inclusions, weld defects, cracks, or their accumulation [152]. The critical stress intensity factor, K_C , is used to demonstrate the fracture toughness and can be defined as the SIF needed to observe instant crack extension under certain conditions, such that crack growth will be catastrophic [32]. A flaw with a sufficiently long depth (a) and an accompanying SIF that exceeds K_C with sufficient loading, will cause component failure.

Several environmental factors affect the fracture toughness of a material, such as the exposure temperature [153], gaseous environment, presence of damaging impurities [154], and loading conditions. For example, Huang et al. [155] observed a significant reduction in the fracture toughness of ferritic steel in the presence of hydrogen. However, several mechanical aspects alter the expected values, such as the component thickness [156]. The thickness may alter the crack-tip constraint conditions, where thick components encounter plane strain conditions and thin components encounter plane stress conditions. Plane strain is defined as the condition in which the displacements of points in a body are parallel to the given plane such that the displacement is not dependent on the perpendicular distance to the plane [157]. The plane stress is such that two of the principal stresses are parallel to the given plane, and are constant in the normal direction [158]. It is known that the fracture toughness decreases with increasing material thickness until a point is reached where the thickness is high enough to reach plane-strain conditions [159]. Beyond this point, the fracture toughness reaches a constant value called the plane-strain fracture toughness [160], and mode I loading is referred to as K_{IC} . The fracture toughness for a specific thickness can be calculated using,

$$K_C = K_{IC}[1 + B_k e^{-(A_k t/t_0)^2}] \quad (2.6)$$

where t is thickness, A_k and B_k are material constants, and t_0 represents the plane-strain thickness at the point of critical loading and can be calculated as follows,

$$t_0 = 2.5 \left(\frac{K_{IC}}{\sigma_{ys}} \right) \quad (2.7)$$

where σ_{ys} denotes tensile yield strength. Other aspects, such as the strength of the material, must be considered, as the fracture toughness decreases with increasing strength [158]. For example, if a component had work done to increase its strength, such as heat treatment methods, then a reduction in the fracture toughness is typically observed [161]. As such, it is critical to keep these factors in mind when fracture toughness is selected for components during the design and analysis process.

Testing the fracture toughness of materials is an important step when designing new components because the probability of avoiding flaws is highly unlikely. Fracture toughness testing quantifies a material's resistance to further fracture and involves testing the conditions necessary to propagate an existing crack. There are a variety of ways to do so, including Charpy impact tests [162], where there is an impact on a notched specimen (for example, U- or V-notched samples), or displacement-based methods, such as a three-point bend test with pre-cracked specimens. ASTM standards (E1820) specifically recommend the use of single-edge bends and compact and disk-shaped compact specimens [163], where all samples contain sharp cracks.

2.6.3 Critical Energy Release Rate - Relation to SIF

Fracture mechanics states that as a crack grows, newly formed surfaces move relative to one another, and as such, stresses, or energy, are released [125], otherwise known as the release of elastic strain energy by the body as a crack extends. This release of energy for

new fracture surfaces is called the 'energy release rate', G , and is given in units of MPa m. This can often be difficult to measure, however in a simple case where a 2D crack propagates (as seen in Figure 2.12 where it extends in the X direction), the energy release rate is given using Irwin's [164] formulation below

$$G = \lim_{\Delta a \rightarrow 0} \frac{1}{\Delta a} \int_{\Delta a}^0 \sigma_{i2}(x_i, 0) u_i(\Delta a - x_1) \pi dx_1 \quad (2.8)$$

where Δa is the change in the crack length per increment and the stress and displacement parameters are in polar coordinates. u_i refers to the displacement of the crack opening as a result of mode I, II, or III loading. Based on these assumptions, the energy release rate of a material can be related to the SIF (of the three modes of loading previously mentioned) using the following equation:

$$G = \frac{K_I^2}{E'} + \frac{K_{II}^2}{E'} + \frac{K_{III}^2}{2\mu} \quad (2.9)$$

Where the Young's modulus used is dependent on whether a plane strain or plane stress condition is assumed [165], highlighted by Equation 2.10.

$$E' = \begin{cases} E & \text{plane - stress} \\ \frac{E}{1-\nu^2} & \text{plane - strain} \end{cases} \quad (2.10)$$

The critical energy release rate, G_c , can be related to the failure stress, σ_f , using Equation 2.11, where the numerator is the square of the critical SIF, K_c , and as such becomes Equation 2.12

$$G_c = \frac{\sigma_f^2 \pi a}{E} \quad (2.11)$$

$$G_c = \sigma_f \sqrt{\pi a} \quad (2.12)$$

Thus, K_c can be related to G_c giving the following when σ is greater than σ_f :

$$G_c = \frac{K_c^2}{E} \quad (2.13)$$

Crack growth occurs in the body when G exceeds the value of G_c , which is a mechanical property.

2.7 Crack Interaction Mechanisms

For a crack to propagate, two factors are required: the first is a relative driving force (for example, SIF), and the second is a lack of resistance from the material microstructure. As cracks propagate through a solid body, the local stress fields are altered, which often leads to catastrophic failure of components. Fracture mechanics principles make it known that the stress fields surrounding a propagating crack can be affected if an existing crack is encountered, and as such can either increase or decrease the rate of catastrophic failure [166, 167, 168]. Many studies have already been conducted on this topic, and work by Kranz et al. [169] and Horii et al. [170] showed that the growth and interaction between microcracks play a critical role in controlling the dominant mechanisms involved in macroscopic material failure. Crack interaction mechanisms, such as stress shielding and crack coalescence, occur naturally in many engineering environments, indicating that a fundamental understanding of such mechanisms is crucial for designing new materials.

2.7.1 Crack Shielding

Crack-tip shielding is a phenomenon that causes an effective and drastic reduction in the local stresses, often seeing a singular crack dominate and effectively reducing the driving force for neighbouring cracks [167]. This is of great importance when factors such as exposure environments and loading conditions lead to mechanisms (for example, SCC or corrosion-fatigue) that reduce the fracture toughness of a material [171]. This often leads to a high density of crack embryo formation, which is more likely to interact [172], particularly when inherent material flaws are also considered [166].

Crack shielding is not a novel idea and goes back to fracture mechanics principles; hence, much research has been conducted in this area, particularly for static loads. For example, several studies by Kamaya [173, 174] concluded a relationship between crack positioning and the magnitude of the interaction effects when using models to simulate semi-circular and semi-elliptical cracks under tensile loading. Jiang et al. [175] wished to understand how two cracks, that were unequal in length, may react when a body with finite width was subjected to a tensile displacement. They found that, as a result of strong crack-shielding mechanisms, the SIF at both tips drastically decreased. They concluded that when the spacing between the two cracks was minimised, the shortest crack became inactive and its effect could be neglected. Ma et al. [176] assessed the potential interaction mechanisms between two predefined cracks, one as an edge crack and the other as an established embedded crack, and found that both the separation distance and total crack lengths had an effect on the SIF.

Understanding the crack-tip shielding mechanisms under cyclic loading has become increasingly popular. Further work by Kamaya [177, 178, 179] involved fatigue testing (both experimentally and using FEA) to assess how the two separated cracks may interact under cyclic conditions. They found that in both cases, the inner crack tips changed the propagation path and shifted the planes to meet each other, and the outer tips continued growing parallel to the loading direction. This was further validated by Jiang et al. [180], who found that, in the case of two parallel edge cracks in a finite plate, the cracks deviated from their crack path, and this effect became more evident when the crack lengths increased and the separation decreased. Han et al. [181] assessed the crack interaction mechanisms in 304 stainless steel specimens under fatigue conditions using two parallel cracks, both experimentally and numerically. From a numerical standpoint, they found that, depending on the positioning and spacing of these cracks, they either saw an enhancement or shielding effect on the SIF.

This interaction effect can be accounted for when predicting the CGR by comparing one and multiple cracks. This is evident from the work of Hui et al. [182] who observed that the CGR for a singular crack was different from that of multiple cracks, and thus introduced a new parameter to account for this by considering the net stress range for a given section and calculating a more accurate ΔK as the driving force for crack extension.

2.7.2 Crack Coalescence

Crack coalescence involves two flaws (e.g. cracks or voids) connecting to form a singular crack, and can occur between just two flaws or multiple flaws [183]. When coalescence occurs between multiple secondary cracks, the newly formed crack will likely experience stress intensification, and as it increases in length and depth, it can lead to unstable crack growth and critical failure [184].

Crack coalescence is known to occur during all aspects of crack extension once embryos have initiated and occurs in many engineering applications, especially SCC, where a high density of crack embryos is known to initiate. For example, Parkins [185] sectioned components from high-pressure gas transmission pipelines exposed to SCC conditions and found that many crack embryos initiated from different points but later coalesced to form a singular long crack that propagated along the length of the specimen. Qiao [186] found that in the case of single-crystal stainless steel, an interaction between the main crack and surrounding micro-cracks saw an increase in the SIF to as much as 17 times the original value, resulting in the coalescence of these cracks. Shen et al. [187] carried out uniaxial compression tests to investigate coalescence effects on open and closed flaws in a material with pre-existing cracks. They observed that for given configurations and contact conditions (i.e. flaws open/closed), crack coalescence was encouraged under tensile loading conditions. Coalescence starting from pre-existing crack tips and from the centre of the bridges between these cracks were observed.

A final example from Jiang et al. [188] assessed the crack propagation behaviour in single-crystal nickel superalloys during in-situ SEM tensile tests at high temperatures (1000°C). They showed that the coalescence between a primary crack and a secondary microcrack at the γ/γ' interface resulted in an increase in the crack propagation rates from 0.3 $\mu\text{m/s}$ to 0.4 $\mu\text{m/s}$.

2.8 Summary of Literature Review

In this chapter, a brief overview of the GT system is provided, and emphasis is placed on how these individual processes need to be integrated to improve the safety and efficiency of the overall system. Single crystal microstructures are discussed, and their strengths and weaknesses determine where they may be found in the GT system to tackle harsh environments at different stages. Single crystals are often favoured for turbine blades over polycrystals because of their increased melting temperatures and yield strength. An overview of the two-phase structure is provided, along with the importance of the γ' precipitate for providing material strength.

Different material degradation mechanisms associated with the intermediate-temperature regime are discussed. Emphasis is placed on how complex environments and loading conditions often lead to the synergy of multiple degradation mechanisms and how this can be detrimental to components, such as HC-SCC-based degradation. Both the C-ring and corrosion-fatigue experimental setups are reviewed based on previous work and how they can be used to better understand the different degradation mechanisms. Based on the literature, C-ring tests have proven beneficial in understanding the importance of stress in SCC, as they highlight both the stress and time-dependent nature of the mechanism. Corrosion-fatigue testing has shown how detrimental corrosive mechanisms can be used to determine the number of cycles a component can reach before failure. Both elastic and plastic material behaviours are discussed, and methods for solving different material

responses are discussed. Several computational techniques often used to predict crack extension are discussed, and the justification for using PFM is highlighted. Both physical and computational testing techniques are often discussed independently; however, this thesis aims to emphasise the importance of integrating the two and using the results from one to feed into the thought process of the other.

In addition to corrosive mechanisms, this thesis also uses fracture mechanics principles to understand the crack extension. As such, different techniques for conducting stress analysis of cracked components (such as SIF) are discussed and related to the principles of fracture mechanics. Emphasis is also placed on crack interaction mechanisms because of their relevance to the results chapters of this thesis.

Chapter 3

Modelling - Methodology and Implementation

3.1 Introduction

This chapter introduces an informed formulation for PFM. The extension to both corrosion and fatigue is then considered, and how experimental results are used to parameterise and validate the model. A brief introduction to its implementation within Abaqus is also discussed, including an extension to account for the highly anisotropic nature of the materials being considered. This chapter concludes with two sensitivity analyses. The first considers a simple mathematical test to narrow down which diffusion coefficients should be considered, and the second is a mesh sensitivity study that was used to further understand the role of the length scale parameter.

3.2 Phase Field Modelling Formulation

PFM [189, 190] represents a smooth transition between two phases as opposed to a more traditional sharp change. This is represented using the PFM damage parameter Φ , which varies on a scale from 0 (undamaged material) to 1 (completely damaged material) [191] as highlighted in Figure 3.1.

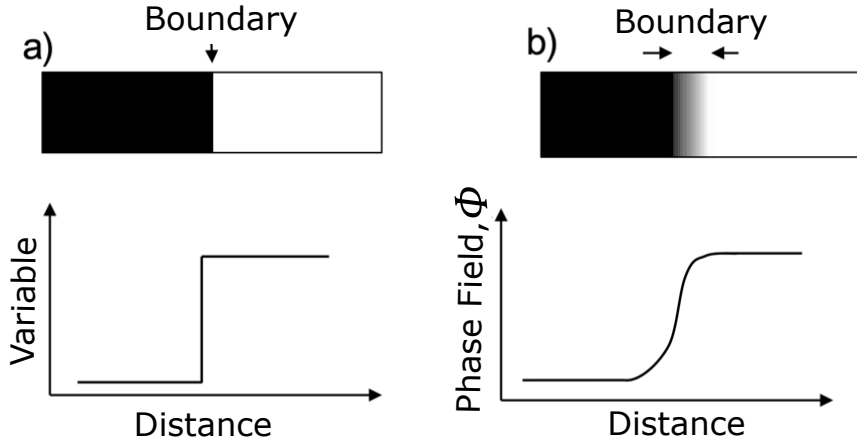


Figure 3.1: A Schematic representing the boundary seen between phases for both a) a sharp interface and b) a diffusive interface seen in PFM [192]

The phase field implementation of corrosion-driven crack propagation follows that of Martínez-Pañeda [193], which was designed as a way to explore hydrogen-induced degradation, and as such the PF parameter, Φ , is calculated as such [194]

$$\Phi(x) = e^{\frac{|x|}{l}} \quad (3.1)$$

where l represents the length scale parameter which manages the width of the smear crack, and its effect as shown in Figure 3.2. Φ is then used to calculate the crack functional variable, Γ_L (highlighted in Equation 3.2), which informs Equation 3.3 to calculate the fracture energy needed to form new surfaces, that is, to crack the material [194]. Here, $G_c(\theta)$ refers to the degraded critical energy release rate based on the diffusion of embrittling species.

$$\Gamma_l(\Phi) = \int_{\Omega} \left(\frac{\Phi^2}{2l} + \frac{l}{2} |\nabla \Phi|^2 \right) dV \quad (3.2)$$

$$\int_{\Gamma} G_c(\theta) ds = \int_{\Omega} G_c(\theta) \left(\frac{\Phi^2}{2l} + \frac{1}{2} l |\nabla \Phi|^2 \right) dV \quad (3.3)$$

A degradation function, that accounts for the reduction in the stiffness of the material is introduced with Equation 3.4, and Equation 3.5 introduces the elastic strain energy density formulation where ε and C_0 are the strain and stiffness tensors respectively. To prevent the regression of Φ as the simulation converges, a history variable, ψ_H , is introduced using Equation 3.6, where ψ_T is the calculated value from the previous time increment.

$$D_g = (1 - \Phi)^2 \quad (3.4)$$

$$\psi_0(\varepsilon) = \frac{1}{2} \varepsilon^T : C_0 : \varepsilon \quad (3.5)$$

$$\psi_H = \begin{cases} \psi_0(\varepsilon) & \text{if } \psi_0(\varepsilon) > \psi_H \\ \psi_T & \text{otherwise} \end{cases} \quad (3.6)$$

Equations 3.4 and 3.6 are then used to inform Equation 3.7 and integrated over the total volume of the body to calculate the total potential energy function of the system.

$$\psi = \int_{\Omega} (D_g \psi_H + G_c(\theta)) \left(\frac{\Phi^2}{2l} + \frac{l}{2} |\nabla \Phi|^2 \right) dV \quad (3.7)$$

And when considered with respect to $\delta \Phi$ the weak form of PF formulation is achieved below.

$$\int_{\Omega} (-2(1 - \Phi) \psi_H \delta \Phi + G_c(\theta) \left(\frac{1}{l} \Phi \delta \Phi + l \nabla \Phi \cdot \nabla \delta \Phi \right)) dV = 0 \quad (3.8)$$

As such, the final PF equation for the given solid body, Ω , becomes,

$$G_c(\theta) \left(\frac{1}{l} - l \Delta \Phi \right) - 2(1 - \Phi) \psi_H = 0 \quad (3.9)$$

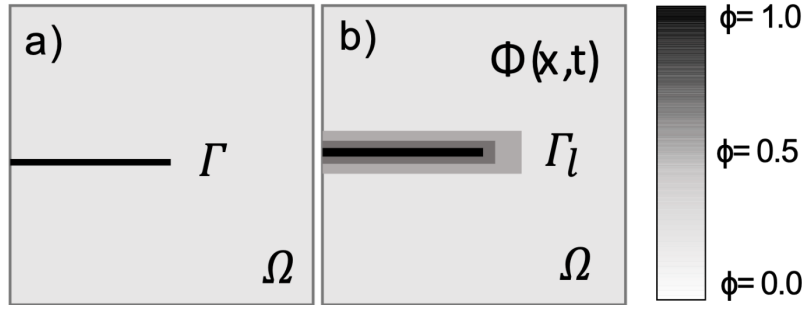


Figure 3.2: A schematic representing a crack in a body, Ω , for both a) a sharp interface and b) a diffusive crack interface whose width is controlled by l

3.2.1 Adaptation to Corrosion

The PFM follows the work of Griffith [125] and uses the critical energy release rate, G_c , that he first proposed. This implementation of PFM degrades the energy release rate based on the applied tensile stress and diffusion of corrosive impurities, which is driven by the total surface coverage, θ , of a given impurity (formally referred to as the Langmuir-McLean isotherm equation [195]). This is represented by Equation 3.10 where $\Delta g_b \theta$ is the Gibbs free energy difference, c is the concentration, R is the molar gas constant, T is the temperature, and finally ‘ a ’ is a parameter introduced to control the rate of θ .

$$\theta = \frac{c * a}{(c * a) + \exp(\frac{-\Delta g_b \theta}{RT})} \quad (3.10)$$

Equation 3.10 is then linked with Equation 3.11 to calculate an updated and degraded G_c value, where $G_c(0)$ and $G_c(\theta)$ are the critical energy release rate of the raw material before and after impurity degradation respectively. χ is the material degradation function and accounts for the effect of the corrosive product on the reduction in the material’s fracture toughness.

$$G_c(\theta) = G_c(0)(1 - \chi\theta) \quad (3.11)$$

The diffusion problem was solved using an extension of Fick's law of diffusion [196], where a modified flux equation is shown in Equation 3.12. It comprises the diffusion coefficient (D), contaminant concentration (c), molar gas constant (R), operational temperature (T), and chemical potential gradient ($\nabla\mu$) which drives the diffusion process. Equation 3.13 equates the chemical potential and includes μ_0 , the chemical potential at given concentration C_0 , partial molar volume of impurity x ($\bar{V}_x$), and hydrostatic stress (σ_H). The final form is obtained when these two equations are combined and the stress-driven diffusion process is given by Equation 3.14 [197].

$$J = -\frac{DC}{RT}\nabla\mu \quad (3.12)$$

$$\mu = \mu_0 + RT\ln\left(\frac{C}{C_0}\right) - \bar{V}_x\sigma_H \quad (3.13)$$

$$J = -D\nabla C + \frac{DC}{RT}\bar{V}_x\nabla\sigma_H \quad (3.14)$$

3.2.2 Adaptation to Corrosion-Fatigue

In addition to corrosion, corrosion-fatigue environments are also considered for parts of this thesis, and as such, the above formulation needs to be extended following the implementation of Carrara et al. and Kristensen et al. [198, 199]. For this extension, the general premise of the model is as mentioned above; however, fatigue is accounted for by incorporating a fatigue accumulation variable $\bar{\alpha}$, where the energy release rate of the material is reduced when this value reaches a value above the critical threshold. $\bar{\alpha}$ can be calculated following the approach of [198] as seen in Equation 3.16, where $\bar{\Theta}(\alpha\dot{\alpha})$ is a heavy side function, α refers to the reduction of elastic strain energy density by the degradation function previously mentioned, and τ is simply the pseudo-time.

$$\alpha = D_g\psi_0 \quad (3.15)$$

$$\bar{\alpha}(t) = \int_0^t \bar{\Theta}(\alpha \dot{\alpha}) |\dot{\alpha}| d\tau \quad (3.16)$$

The threshold for fatigue, α_{Th} is simply given the value of one-third of the yield. When the accumulation of fatigue exceeds this value, a fatigue degradation function is introduced as represented below:

$$\alpha_{Th} = \frac{\sigma_y}{3} \quad (3.17)$$

$$f(\bar{\alpha}(t)) = \begin{cases} 1 & \text{if } \bar{\alpha}(t) \leq \alpha_{Th} \\ (\frac{2\alpha_{Th}}{\bar{\alpha}_t + \alpha_{Th}}) & \text{if } \bar{\alpha}(t) \geq \alpha_{Th} \end{cases} \quad (3.18)$$

If the accumulation of fatigue remains below the threshold value, the degradation function becomes 1, and no effect is exerted on the energy release rate of the material.

The total potential energy of the system, governed by Equation 3.19 is amended to include the contribution by fatigue to become,

$$\int_{\Omega} (-2(1 - \Phi)\psi_H \delta\Phi + f(\bar{\alpha})G_c(\theta)(\frac{1}{l}\Phi\delta\Phi + l\nabla\Phi \cdot \nabla\delta\Phi))dV = 0 \quad (3.19)$$

This implementation of the model can account for the corrosion, fatigue, and corrosion-fatigue environments. As such, it includes what is referred to as switch variables, which are, in essence, parameters that allow the user to switch on and off the role of the corrosion and fatigue degradation mechanisms, depending on the environment in which they

are concerned.

3.3 Elastic Properties and Anisotropic Crystal Orientation

The single-crystal nickel alloys used in this study, such as CMSX-4 and CMSX-10, are highly anisotropic; hence, the correct crystal orientation needs to be implemented in the model to reflect the true extent of SCC. To replicate the material's elastic response the stiffness matrix [200] was constructed using values for C_{11} , C_{12} , and C_{44} (as seen in equation 3.20) where their derivations can be seen in Equations 3.21, 3.22, and 3.23 respectively [201]. E (Young's modulus), ν (Poisson's ratio), and G (shear modulus) are material-dependent data, and the values at specific temperatures are used.

$$C = \begin{pmatrix} C_{11} & C_{12} & C_{12} & 0 & 0 & 0 \\ C_{12} & C_{11} & C_{12} & 0 & 0 & 0 \\ C_{12} & C_{12} & C_{11} & 0 & 0 & 0 \\ 0 & 0 & 0 & C_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & C_{44} & 0 \\ 0 & 0 & 0 & 0 & 0 & C_{44} \end{pmatrix} \quad (3.20)$$

$$C_{11} = \frac{E(1 - \nu)}{(1 + \nu)(1 - 2\nu)} \quad (3.21)$$

$$C_{12} = C_{11} \frac{\nu}{1 - \nu} \quad (3.22)$$

$$C_{44} = G \quad (3.23)$$

To maintain comparability with the experimental results, the model was amended to match the correct crystal orientation of the experimental C-rings. The experimental spec-

imens follow Rolls-Royce's material coordinate system, where the material orientation is defined using the three angles δ, γ and κ [25]. δ denotes a rotation around the X-axis with Equation 3.24; γ denotes a rotation around the Y-axis with Equation 3.25; and κ denotes a rotation around the Z-axis with Equation 3.26 [202]. Thus, the local (material) orientation can be calculated by first calculating the 3×3 rotation matrix R_{Local} .

$$R_x(\delta) = \begin{pmatrix} 1 & 0 & 0 \\ 0 & \cos(\delta) & \sin(\delta) \\ 0 & -\sin(\delta) & \cos(\delta) \end{pmatrix} \quad (3.24)$$

$$R_y(\gamma) = \begin{pmatrix} \cos(\gamma) & 0 & \sin(\gamma) \\ 0 & 1 & 0 \\ \sin(\gamma) & 0 & \cos(\gamma) \end{pmatrix} \quad (3.25)$$

$$R_z(\kappa) = \begin{pmatrix} \cos(\kappa) & \sin(\kappa) & 0 \\ -\sin(\kappa) & \cos(\kappa) & 0 \\ 0 & 0 & 1 \end{pmatrix} \quad (3.26)$$

$$R_{local} = R_z(\kappa) \cdot R_x(\delta) \cdot R_y(\gamma) = \begin{pmatrix} R_{xx} & R_{xy} & R_{xz} \\ R_{yx} & R_{yy} & R_{yz} \\ R_{zx} & R_{zy} & R_{zz} \end{pmatrix} \quad (3.27)$$

However, in its current form, it cannot be directly used; therefore, it is converted into a 6×6 rotation matrix, as shown in Equation 3.28. This new rotation matrix was multiplied by the material stiffness matrix and the transposed rotation matrix to formulate the rotated material stiffness, as represented in Equation 3.29 [203]. This calculates the updated value and distribution of the peak stresses, which has a significant role in predicting not only the extent of cracking, but also the location of possible cracking.

$$M = \begin{pmatrix} R_{xx}^2 & R_{xy}^2 & R_{xz}^2 & 2R_{xy}R_{xz} & 2R_{xx}R_{xz} & 2R_{xx}R_{xy} \\ R_{yx}^2 & R_{yy}^2 & R_{yz}^2 & 2R_{yy}R_{yz} & 2R_{yx}R_{yz} & 2R_{yx}R_{yy} \\ R_{zx}^2 & R_{zy}^2 & R_{zz}^2 & 2R_{zy}R_{zz} & 2R_{zx}R_{zz} & 2R_{zx}R_{zy} \\ R_{yx}R_{zx} & R_{yy}R_{zy} & R_{yz}R_{zz} & R_{yy}R_{zz} + R_{yz}R_{zy} & R_{yx}R_{zz} + R_{yz}R_{zx} & R_{yx}R_{zy} + R_{yy}R_{zx} \\ R_{xx}R_{zx} & R_{xy}R_{zy} & R_{xz}R_{zz} & R_{xy}R_{zz} + R_{xz}R_{zy} & R_{xx}R_{zz} + R_{xz}R_{zx} & R_{xx}R_{zy} + R_{xy}R_{zx} \\ R_{xx}R_{yx} & R_{xy}R_{yy} & R_{xz}R_{yz} & R_{xy}R_{yz} + R_{xz}R_{yy} & R_{xx}R_{yz} + R_{xz}R_{yx} & R_{xx}R_{yy} + R_{xy}R_{yx} \end{pmatrix} \quad (3.28)$$

$$C' = MCM^T \quad (3.29)$$

Figure 4.1 shows an example of stress distribution before and after a small rotation in the second crystal orientation.

3.4 Implementation in Abaqus CAE

The PFM mentioned above, which includes anisotropic elastic variation, was implemented within a user-element subroutine (UEL) and run in the Finite Element software Abaqus 2021 [204]. A UEL allows the production of user-defined elements using what is referred to as a 'dummy mesh'. Once a geometry is designed, meshed, and has boundary conditions applied within Abaqus' user interface, the input file is post-processed using a Python script to introduce the dummy mesh and user-defined elements. The UEL is a very robust subroutine but does have its pitfalls, which are discussed in further detail in the future work section (Section 9).

3.4.1 Boundary Conditions

To replicate the displacement, and therefore the predicted peak stress distribution, boundary conditions were applied to the C-ring CAD geometry (as shown in Figure 3.3)

by pinning (i.e. $U_X = U_Y = U_Z = 0$) all nodes along the bottom ridge on one side and displacing the nodes along the opposite ridge in the Z direction (that is, $U_X=U_Y=0$) until the desired peak stress (approximately 700-750 MPa depending on the crystal orientation) was reached.

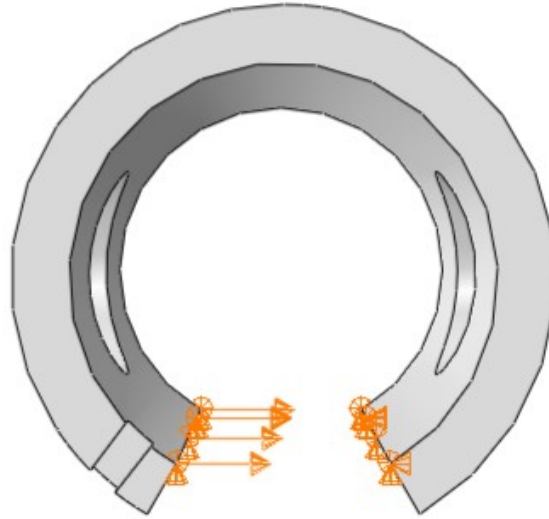


Figure 3.3: A schematic to represent where the boundary conditions are applied to replicate the desired stress levels throughout the C-ring geometry

Similarly, for the fatigue specimen, the outermost surface of the bottom grip was pinned in all directions ($U_X=U_Y=U_Z=0$), while the outermost surface of the top grip was displaced in the X-direction. As fatigue testing accounts for nominal stress rather than the peak stress seen at the notch, it was necessary to manually calculate an approximation for the applied force on the specimen using the known surface area at the notch and Hooke's law. The displacement was determined using this method.

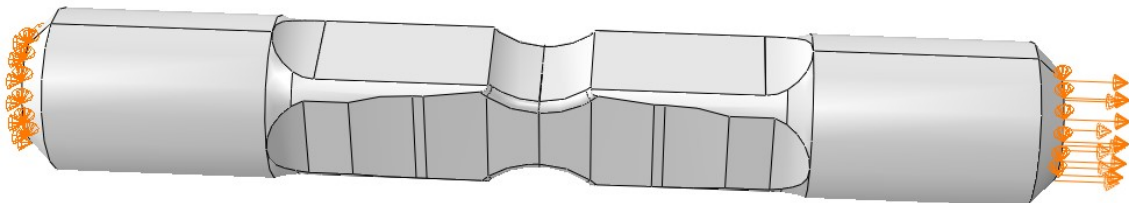


Figure 3.4: A schematic to represent where the boundary conditions are applied to replicate the desired stress levels along the notch of the fatigue specimen

Where necessary for both tests, temperature boundary conditions (which adopt the temperature degrees of freedom within Abaqus) were used to allocate a concentration of corrosive impurities, and a diffusion coefficient was supplied as an input to the model. The focus of this thesis was on crack propagation mechanisms, and as such, a predefined crack was assumed, and initiation was neglected. These predefined cracks are first drawn as a 2D approximation of a typical ‘thumbnail’ shape crack (which was assumed based on experimental observations [25]), before allocating a seam crack to this region in the Abaqus user interface. When a seam crack is applied, Abaqus doubles the number of nodes along the edge, and when a load is applied, a crack opening is produced [204]. Seam cracks will not extend along a given boundary and can only be placed on a 2D face. This will allow for local crack tip stresses and strains to be approximated

3.4.2 Input Parameters

All the input parameters used for both the CMSX-4 and CMSX-10 simulations are summarised in Tables 3.2 and 3.3. Arguably, one of the most important input parameters for the PFM is the critical energy release rate of the material, which can be calculated using Equation 3.30. This equation accounts for Irwin’s contribution [164] to the original equation, first proposed by [125], resulting in Equation 3.30 [146].

$$G_c(0) = \frac{K_{IC}^2}{E'} \quad (3.30)$$

Where,

$$E' = \begin{cases} E & \text{plane – stress} \\ \frac{E}{1-\nu^2} & \text{plane – strain} \end{cases} \quad (3.31)$$

In the case of both materials K_{IC} is estimated to be $75 \text{ MPa}\sqrt{m}$ [205].

The exact corrosive impurity (or combination of impurities) that encourages SCC is not yet known, which makes it difficult to determine the initial diffusion coefficient.

Therefore, 1D diffusion at 600°C was simulated using Fick's second law [196] of diffusion to narrow down the potential species. Fick's Second law assesses the role of diffusion on the change of concentration over time, and when the partial differential equation is used in a 1D state it becomes,

$$\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial x^2} \quad (3.32)$$

Where C is the concentration, t is the time, x is the distance travelled, and D is the diffusion coefficient. When the boundary conditions were set, the following equation was obtained:

$$\frac{C}{C_0} = \operatorname{erfc} \frac{x}{2\sqrt{Dt}} \quad (3.33)$$

Where C_0 is the initial concentration and $\operatorname{erfc}(x)$ is the complementary error function. The 1D simulation assessed the change in concentration of hydrogen, oxygen, sulphur, aluminium, and tungsten across 10 μm and 10 nm (with intervals of 5×10^{-8}) and for experimental times of 1 and 900 s. The diffusion coefficients used represent the rate of diffusion of the above-mentioned elements in pure nickel (at 600°C) and are listed in Table 3.1.

Table 3.1: List of known diffusivity coefficients for the mentioned elements in pure Ni at 600°C

Element	D (mm^2/s)	Reference
Hydrogen	3.57×10^{-3}	[206]
Oxygen	9.36×10^{-6}	[206]
Sulphur	1.40×10^{-10}	[206]
Aluminium	2.58×10^{-14}	[207]
Tungsten	1.32×10^{-26}	[207]

Very early stage propagation was assessed over 10 μm (as seen in Figure 3.5), and the 1D simulation showed that hydrogen and oxygen diffused too quickly, whereas aluminium

and tungsten were too slow to potentially play a role in this particular SCC mechanism. The only element that is viable under the current conditions is sulphur, or an element with a similar diffusion coefficient; hence, this is considered the most appropriate value for all simulations. This is not to say that sulphur is solely responsible for SCC in this particular environment; however, for now, it is a good approximation.

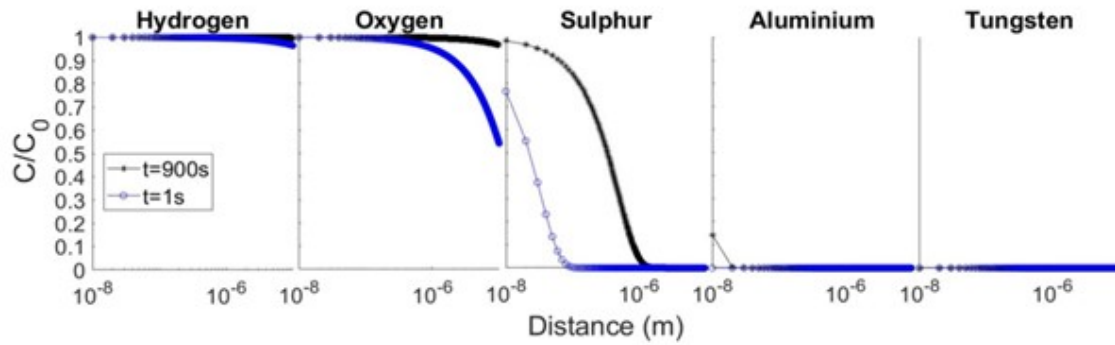


Figure 3.5: 1D diffusion simulations using Fick's second law for a distance of 10 μm and time of 900 and 1 s

Furthermore, a diffusion distance of 10 nm is also considered as Dawson et al. [208] had found tungsten disulphide structures within the first 10 nm of the SCC cracks in the C-ring. The simulation showed that, again, oxygen and hydrogen diffused too quickly (meaning that such species exceed the length scales associated with experimental data [25] and can therefore be ruled out), but sulphur showed a similar trend. Tungsten is predicted to diffuse too slowly, and it is hypothesised that it could be pulled down the crack by aluminium as well as sulphur.

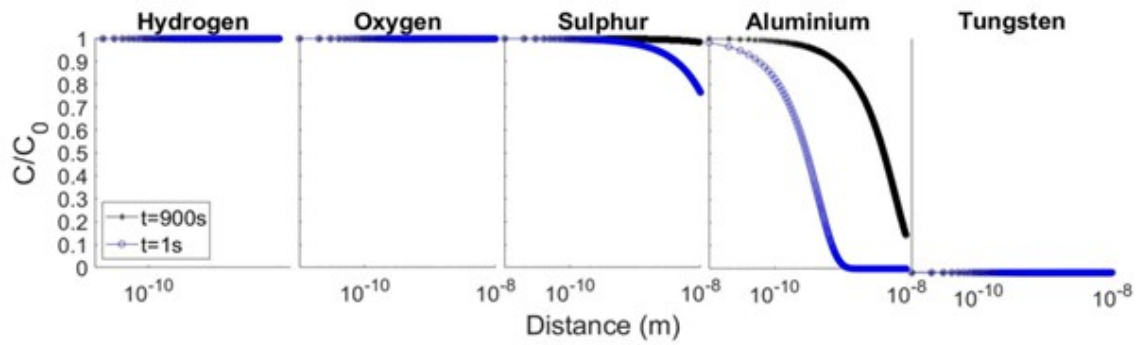


Figure 3.6: 1D diffusion simulations using Fick's second law for a distance of 10 nm and time of 900 and 1s

The elastic properties of both materials are described below. It should be noted that in the case of CMSX-4, these constants are at 540°C, which differs by 10°C from the experimental conditions, but will have a minimal effect on the results. The addition of plasticity was not considered, as despite the fact that elastic models are approximations, they can still be useful even in complex material behaviours such as those mentioned in this thesis. In these cases, the loading conditions are nominally below the yield, and the plasticity will only be on a small scale.

Table 3.2: Values of the elastic constants used for both CMSX-4 and CMSX-10 simulations

Parameter	CMSX-4 Model	CMSX-10 Model
Young's Modulus (GPa)	110.460	112.937
Poisson's Ratio (ND)	0.3858	0.386
Shear Modulus (GPa)	114.600	116.924
$G_c(0)$	40	40
Temperature (°C/K)	540/813	550/823

Several parameters used in the model were based on the diffusion of a particular species through a metal substrate. Both sulphur and oxygen-containing environments are of interest to this study as such the parameters and values used can be found below.

Table 3.3: Values of the input parameters used to account for the diffusion and degradation by sulphur and oxygen at 550°C

Parameter	Sulphur	Oxygen
Initial Concentration (wppm)	0.5	5
Molar Gas Constant (N mm/(mol K))	8314.5	8314.5
Gibbs Free Energy Difference (kJ/mol)	-30	-160
χ	0.998	0.9
Diffusion Coefficient (mm ² /s)	1x10 ⁻¹⁰	9x10 ⁻⁶
Steepness Parameter, 'a'	1.25	1x10 ⁻¹⁰⁰

The thermodynamic database software FactSage was used to estimate the values of Δg_b^0 for the following simple reaction. Owing to the abundance of hydrogen in the atmosphere, it is assumed that H₂ is present in the reaction [209]. For an environmental temperature of 550°C (823 K), a value of -30 kJ/mol was predicted.



This value is further corroborated by the work of Jo et al.[210] who also estimated a value of approximately -30 kJ/mol. For simulations involving oxygen, a value of -160 kJ/mol was calculated for the simple reaction between Ni and O.



For χ , the data highlighted the role of degradation in nickel by illustrating intergranular fracture (percentage) with increasing concentrations of the respective species. The results for both sulphur [211] and oxygen [212] are highlighted in Figure 3.7, and in the case of sulphur, it shows almost 100% intergranular fracture for concentrations between 10-20%, as opposed to oxygen which sees an order of magnitude difference. Hence, when running a simulation with sulphur diffusion coefficients, it is essential to degrade the ma-

material such that the critical energy release rate is reduced to almost zero, as shown by the red line in Figure 3.8. For oxygen (purple line), both the steepness of the line and the minimum threshold need to be amended to replicate the relationship shown in the previous figure. This is achieved by adjusting both the parameters χ and 'a' in Equations 3.11 and 3.10. It is worth noting that despite the materials of interest being single crystals, data for intergranular failure was the closest that could be found in the literature to relate to the fracture toughness of the material and was hence used to roughly estimate the constants needed.

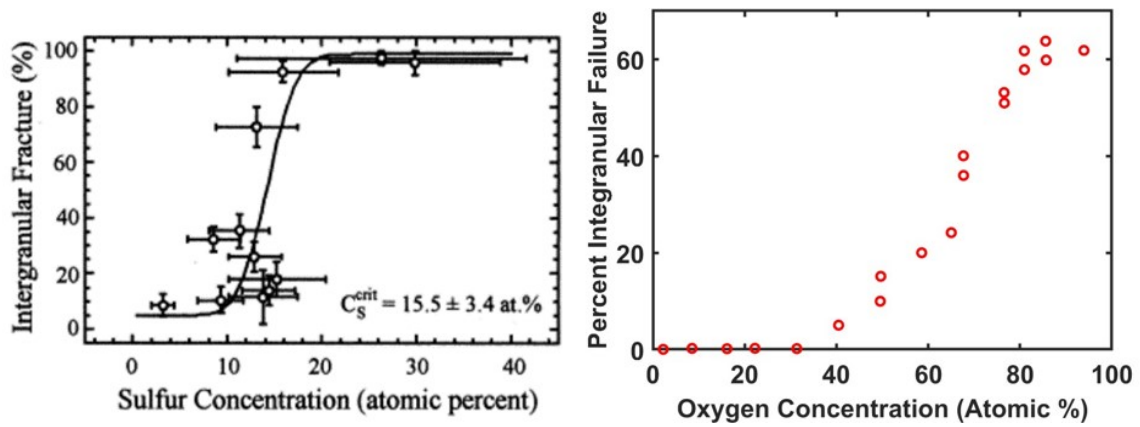


Figure 3.7: Percentage of intergranular failure of nickel with increasing concentration of sulphur (left [211]) and oxygen (right [212]) taken from the literature

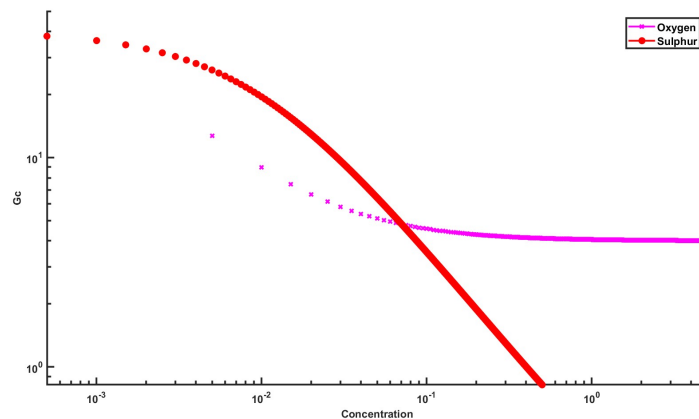


Figure 3.8: MATLAB plot used to illustrate Equations 3.11 and 3.10 with varying parameters used for oxygen and sulphur degradation of G_c in nickel

3.5 Mesh Sensitivity and Length Scale Parameter

PFM has a level of mesh sensitivity that often requires a high mesh density in the region of the expected crack growth to aid in the convergence of Φ . Many studies assume the length scale parameter (l) to be a function of the mesh size [193, 213, 199, 214], which is generally between two and ten times the mesh size used. To understand the physical meaning of l , a simple study was conducted simulating a single-edge specimen, as shown in Figure 3.9. A predefined crack spanning 25% of the total width (i.e. $a/w=1/4$) was used, and the block was loaded by pinning the bottom nodes ($UX=UY=UZ=0$) and applying a perpendicular displacement of 0.005 mm along the top nodes. Material properties representing those of CMSX-4 at intermediate temperatures were used and run for a total experimental time of 100s. The test was used to assess the variation in crack extension predictions, where a threshold of $\Phi \geq 0.95$ was used to distinguish between cracked and uncracked materials. Figures 3.10 and 3.11 indicate the positions for both the threshold and predefined crack used.

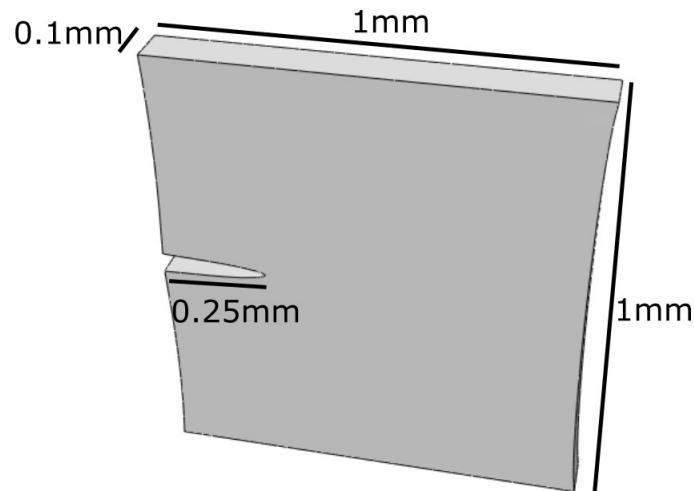


Figure 3.9: Schematic representing a 1x1mm geometry with a single edge predefined notch which was used for the mesh and length scale parameter study

The first saw all aspects of the simulation (boundary conditions, material properties, temperature, etc.) to be identical, with the exception of the mesh size and its relative l (eight times this size across all cases). The results, highlighted in Figure 3.10, show a

large variability in lengths for predicted crack extension ranging between 0.33 and 0.62 mm, despite all conditions (both environmental and mechanical) being the same. For the next set of simulations, once again, all mechanical and environmental aspects were kept identical; however, l was given a fixed value of 0.0125 mm, while a varying mesh size was adopted. Figure 3.11 shows that there is now far less variability in the prediction of the total crack extension in comparison to the previously run test, highlighting the potential physical meaning behind l , as opposed to a numerical interpretation adopted in certain studies [191, 215, 216, 217]. These analyses suggest that the assumption of constant l could be associated with a relatively constant value, such as the Crack Tip Opening Angle (CTOA) or Crack Tip Opening Displacement (CTOD) upon cracking [218]. It is known that l controls the width of the extension zone, and by considering the similarity of the fracture processes, it naturally leads to imposing a maximum mesh size (and, in turn, l) for PFM. This can in turn improve the validity of the model by correlating the width of said process zone to a physical constant like CTOD/CTOA, which can be measured using experimental data (in the form of microscopy) as opposed to a numerical assumption.

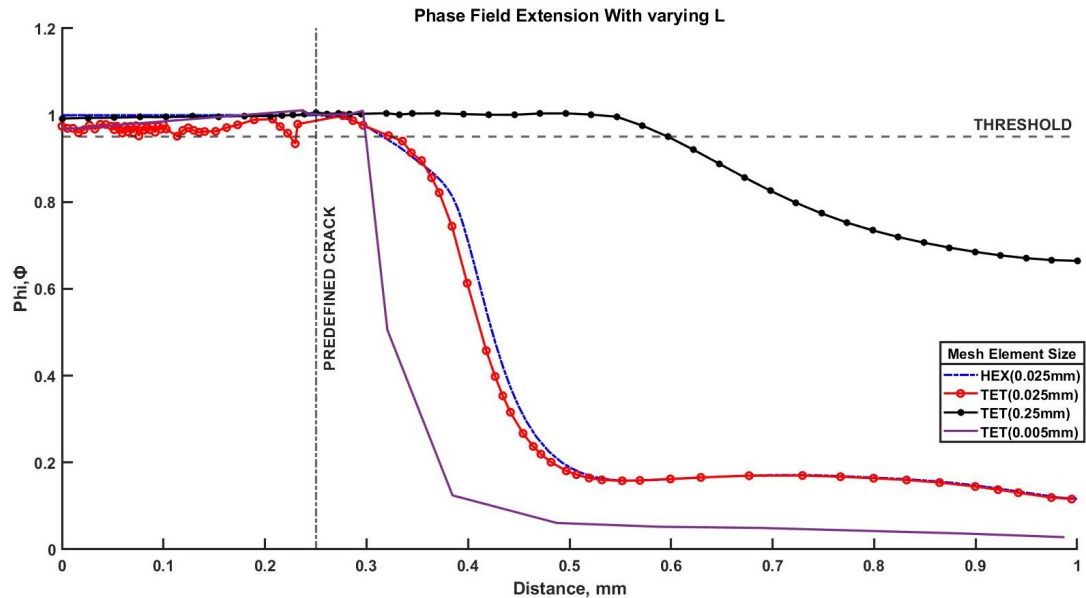


Figure 3.10: Crack propagation prediction based on numerous mesh sizes, each time having l , 8 times this value

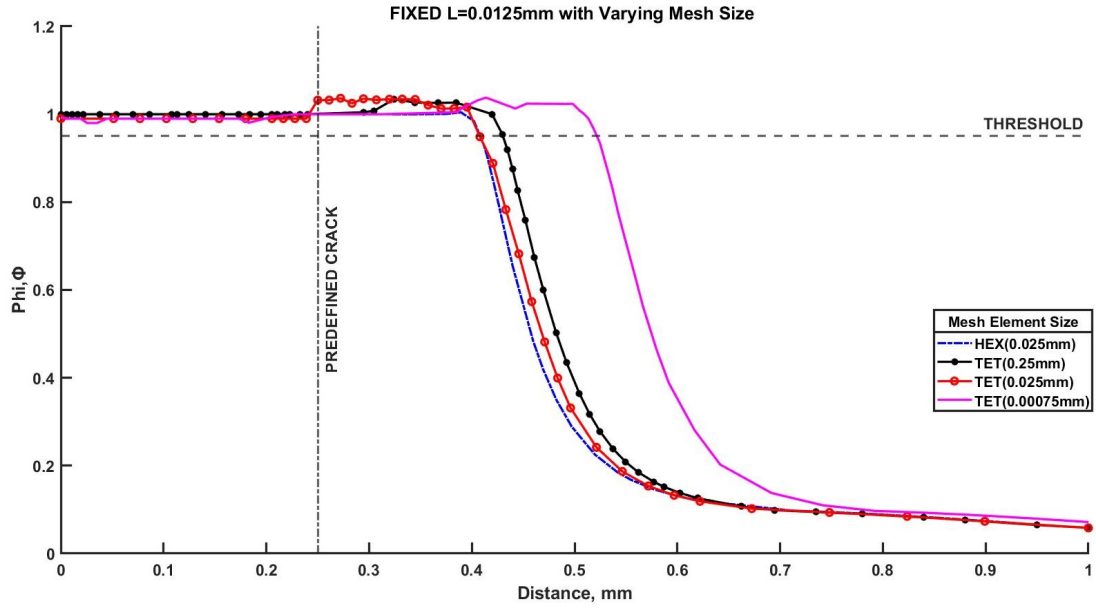


Figure 3.11: Crack propagation prediction based on a fixed l but varying the mesh size

A further length-scale parameter test was conducted on a C-ring and again found little change when l was fixed and the mesh size was changed. This is again highlighting the sensitivity of the length scale parameter and how variability based on mesh size and not a more physical meaning can introduce further uncertainties. These results can be viewed in Appendix A.

3.6 Accounting for Plasticity

Several simulations throughout the thesis were run using a plasticity-based model provided by Rolls-Royce Plc. which enables nonlinear creep and plasticity analysis to be carried out. The model is captured within a User MATerial (UMAT) subroutine and is solved using the FE solver ABAQUS. The intermediate temperature range is too low to observe the effects of creep; therefore, it is outside the scope of this study. As such, the model was run without creep contribution. The yield of the material is defined such that plasticity is only considered once the load point surpasses this threshold, below which the behaviour is entirely elastic. It is a multi-layer kinematic model, meaning it attempts to take the

best approaches from both isotropic (the more general stress-strain curve description) and kinematic (yield threshold translation) models and combine them. The calculation of the yield surface is highlighted in Equations 3.36 and 3.37 for the isotropic and kinematic hardening, respectively.

$$\sigma_y = K \epsilon_{pl}^n \quad (3.36)$$

$$\sigma_y = K \epsilon_{pl} \quad (3.37)$$

However, due to the nature of the current PFM and plasticity model being incorporated within a UEL and UMAT respectively, it was not possible to couple the two. As such, this plasticity model was not considered as part of the PF formulation. Any plasticity-based modelling results were run independently of the PFM and were used to produce stress maps, and the justification for not using plasticity is discussed later.

Chapter 4

Understanding Crack Interaction Mechanisms Under SCC Conditions

4.1 Introduction

Crack interaction mechanisms are well documented, but they are often difficult to assess if the correct techniques are not implemented [219]. The crack spacing and configuration associated with a high density of crack initiation embryos often enhance the probability of accelerating or decelerating the crack growth rate (CGR) [181]. Studies such as those carried out by Qiao [186] observed a significant increase in the stress intensity factor of cracks as a result of coalescence, which could lead to unstable crack growth. In contrast, the literature has countless examples of a local stress relaxation effect at crack tips, ultimately arresting them because of long cracks shielding neighbouring ones [220].

Much consideration of these mechanisms needs to be placed on the components exposed to high-temperature corrosive environments. Materials are subjected to extensive changes in surface chemistry, so much so that it often results in the initiation and propagation of multiple crack embryos [221], which have the potential for extensive interaction. A prime example of this type of environment is GTs which operate at extremely high temperatures. Between the by-products of the fuels and the environments in which they fly, corrosive impurities are introduced into the local air flux. When stress is introduced into the system (in this example, the centrifugal forces applied by the rotating blade), mech-

anisms such as SCC play a significant role in the degradation of a material. Combined, these conditions can cause a high density of crack initiations on the component surfaces, and their interaction can drastically reduce their service lives [222]. A previous study [223] showed that the injection of SO_x (introduced to the environment by the fuel) and the deposition of salt (NaCl) particles on the surface of single-crystal nickel alloys such as CMSX-4 can cause widespread SCC along the surface of the material in intermediate temperature ranges (450-650°C).

Phase field modelling has already been used to investigate crack interactions in several materials and environments. For example, Xu et al. [224] assessed the crack growth and interactions in sandstone and concluded that three modes of crack coalescence occurred. Zhao et al. [33] investigated the interactions between double cracks in Zircaloy-4, a cladding material, and found that there was a critical distance below which hoop stresses of the material reached minimum values. Carlsson and Isaksson [166] used PFM to investigate the role of porosity on crack dynamics and shielding, and found that as the porosity increased, the chances of shielding also increased. However, the novelty of this work lies not within using similar techniques to highlight crack interactions in materials, a concept not new to fracture mechanics, but rather in its application. Aero GTs are often associated with high operational temperatures, but the intermediate temperature ranges (450-700 °C) [24] found in the lower shank region of turbine blades are often neglected. Despite the lower temperatures, catastrophic cracking was still recorded, making this research extremely relevant [25].

This chapter explores the interaction mechanisms between stress corrosion cracks at intermediate temperature ranges in single-crystal nickel alloys, such as CMSX-4.

4.2 Preliminary Phase Field Models

4.2.1 Stress Dependent Nature of the C-ring Test

The study began by implementing a simple elastic model using a C-ring which used five equally spaced cracks (80 μm in length and 40 μm in depth) in different regions along the apex (making sure to have cracks covering the entire stress gradient along the C-ring surface), as shown in Figure 4.1; the first was on the apex of the C-ring and consecutive cracks separated by a distance of 1500 μm . The experimental C-rings incorporated a 36° rotation of the crystal orientation around the Z-axis, which was measured using X-ray diffraction techniques and accounted for in the model to achieve the same value and position of the peak stress. The figure shows the crack positioning in relation to the stress distribution, in which cracks A and B are exposed to stresses between 650-700 MPa, C is around the 500 MPa mark, and D and E fall into regions of much lower values (350 and 50 MPa, respectively). The figure also clearly depicts the asymmetric nature of the stress distribution owing to the deviation of the [001] crystal direction from the C-ring axis, as previously mentioned.

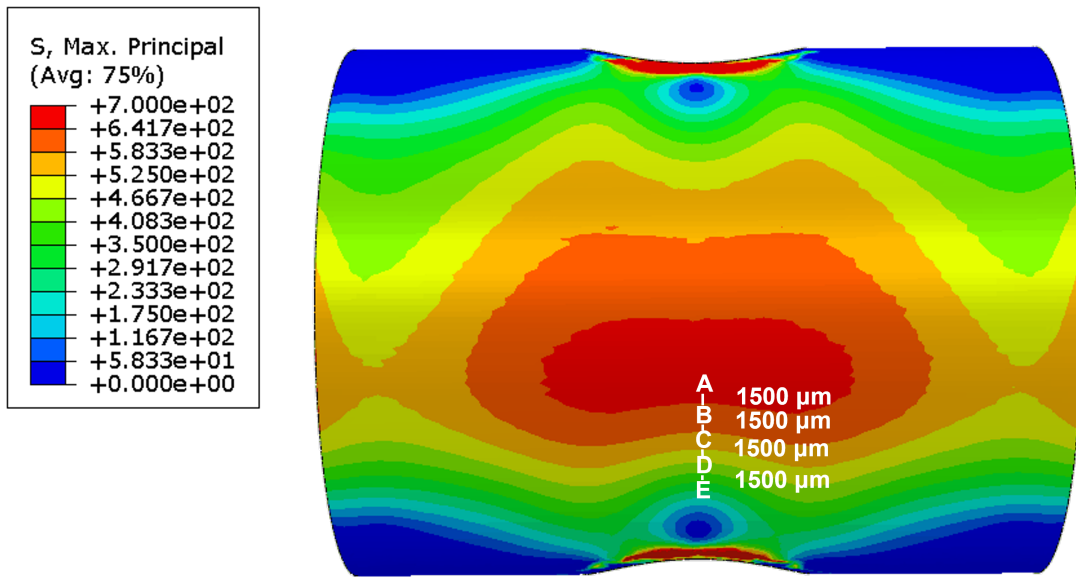


Figure 4.1: The distribution of stress (MPa) as a result of the deviation of crystal orientation

A PFM was run using this format for a 15-min experimental time, and experimental results [223] have shown that a 600s (10 min) incubation period is needed to initiate cracks; as a predefined crack embryo is used, this time was neglected and the simulations were run for a total experimental time of 300 seconds (or 5 min). The experimental work also clearly highlighted the stress-dependent mechanism that plays a role in the propagation, for which cracks located beyond a certain distance from the apex could not physically grow, which was used as a way to parameterise the model. In C-ring experiments, cracks are seen to initiate under salt deposition halos because of the surface reactions occurring between the materials, salt deposition, and the environment. Therefore, the predefined cracks are assumed to be under a salt deposition site and therefore a species concentration is given to the crack (in this case ‘sulphur’, or something with similar diffusion kinetics). Thus, as the diffusion problem is solved, and sulphur travels ahead of the crack tips, a reduction in G_c occurs until it is sufficiently low for crack extension to occur. This is highlighted in Figure 4.2 which shows both Φ and concentration maps at the start, middle, and end of the simulation. As such, a diffusion coefficient matching that of sulphur in nickel is assumed, and a criterion is set so that nodes with a $\Phi > 0.95$, was considered to be cracked. This accounts for uncertainties in the prediction of the model. As expected, slight variations in crystal orientation did indeed have an effect on the peak stress levels experienced and the position of the peak stress.

In summary, cracks B and C resulted in the longest cracks, reaching total lengths of 790 and 125 μm respectively, owing to the asymmetric nature of the peak stress induced by the rotation of the crystal orientation. Crack A then followed with a significantly lower crack length of 81 μm , with cracks D and E not showing any propagation in this experimental time, as a result of the stress-dependent nature of this issue. The results are presented in Table 4.1 and Figure 4.3.

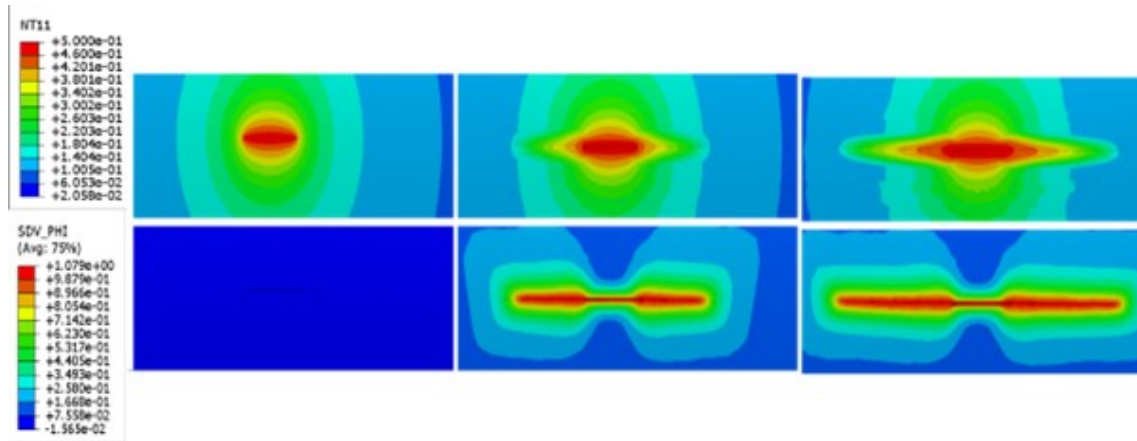


Figure 4.2: Highlighting the concentration (top) and Phi (bottom) change for the first, middle, and last time step. This shows how Phi is converging to 1 for areas in which sufficient diffusion of embrittling species has occurred

Table 4.1: Crack morphology details for initial test

CRACK	TOTAL LENGTH (μm)	INITIAL LENGTH (μm)	FINAL LENGTH μm)
A	81	80	1
B	790	80	710
C	125	80	45
D	80	80	0
E	80	80	0

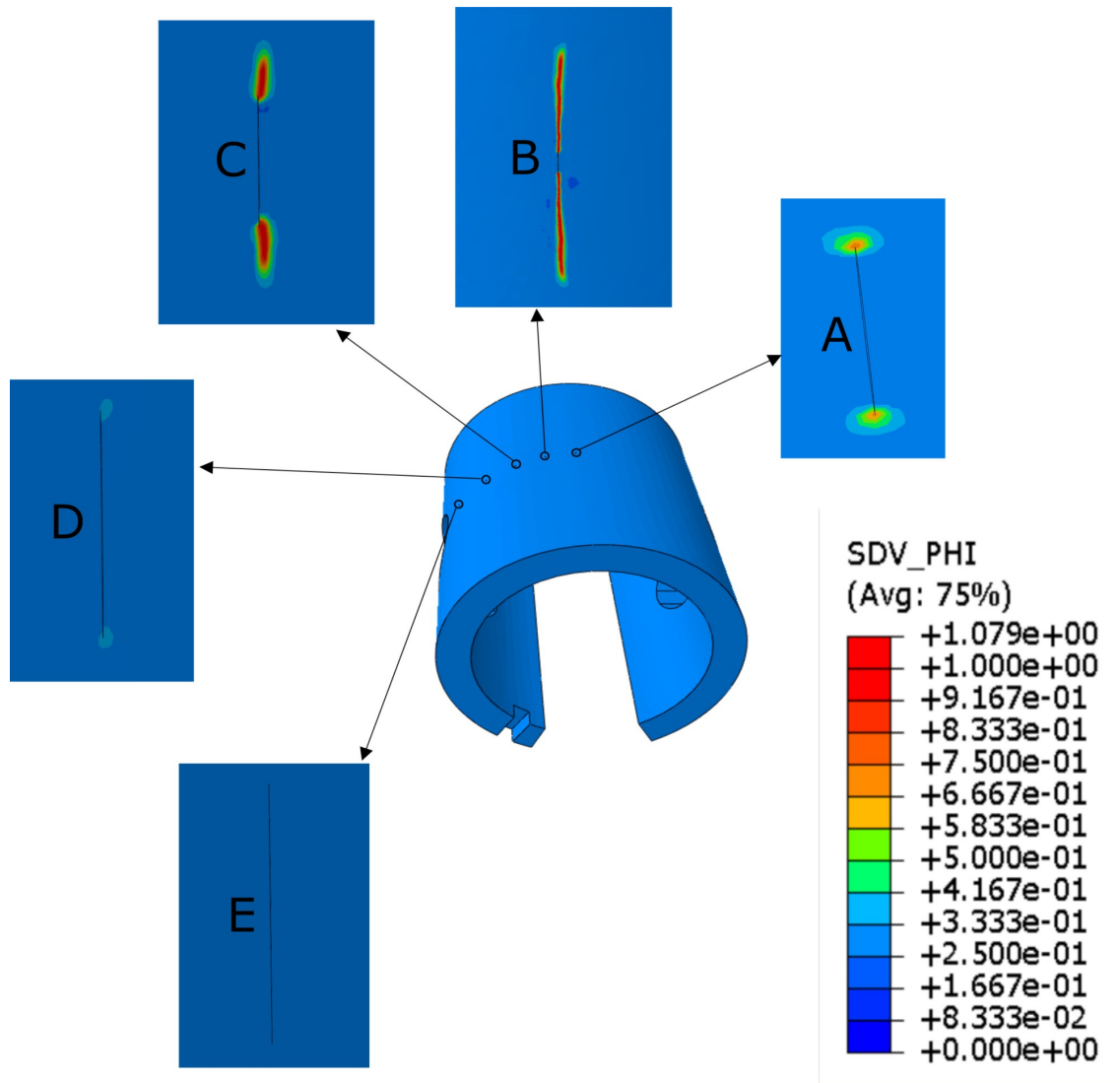


Figure 4.3: Results from the initial PFM calibrated against C-ring results that represent the time and stress-dependent nature of the mechanism. This model shows that as you deviate away from regions of peak stress, expected crack growth decreases.

This is further corroborated by optical images (as shown in Figure 4.4, taken from [25]) from experimental specimens, which exemplify the anisotropic-dependent nature of this SCC mechanism for these materials. They showed that cracks do not nucleate beyond a certain distance from the apex of the C-ring, resulting in asymmetric crack propagation behaviour.

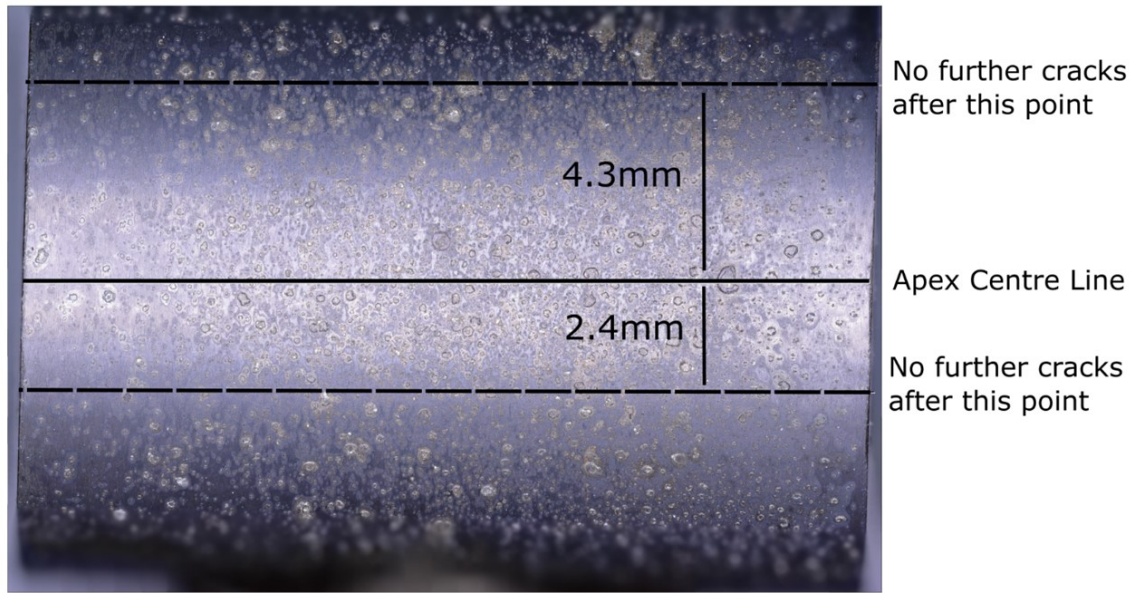


Figure 4.4: Microscopy from Experimental samples [25] which when assessed captured the anisotropic-dependent nature of cracking in this material, and highlighted that beyond a certain distance from the apex further crack initiation is not observed. The dotted lines distinguish where cracks were and were not seen.

4.2.2 Time Dependent Nature of the C-ring Test

A time-dependent mechanism also contributed to the propagation of these cracks, with experimental results (from the study mentioned above) showing not only longer cracks as time progressed, but also crack embryos initiating in areas of the apex with lower levels of stress. To assess the response of the model to such a mechanism, a simulation was run matching the experimental setup (displacement, temperature, and crystal orientation) and a total time of 7200s (2 hr). Note that the 600s initiation time was also neglected, leaving a final experimental time of 6600s. A crack embryo (40 μm in length by 20 μm in depth) was initiated at 4500 μm from the apex (as shown in Figure 4.5, corresponding to a stress level between 450-550 MPa. Again, these predefined cracks were associated with the sulphur concentration through a specific boundary condition. After the first initiation period of 300s, the damage parameter Φ at the crack tip increased significantly, but this time did not allow it to crack ($\Phi < 0.95$), as highlighted in Figure 4.6. On the contrary, the figure also shows that with sufficient time, Φ eventually reaches 1, and the crack propagates

approximately 650 s (11 min, or 21 min if the 10-min initiation time is considered) after initiation. This study demonstrates that PFM can account for the time-dependent nature of this C-ring test, and the need to consider this as crack embryos initiate further and further from the apex. This also aligns with experimental C-ring data [25], which showed that the distance from the apex that we would expect cracks is limited for the 15 minute tests.

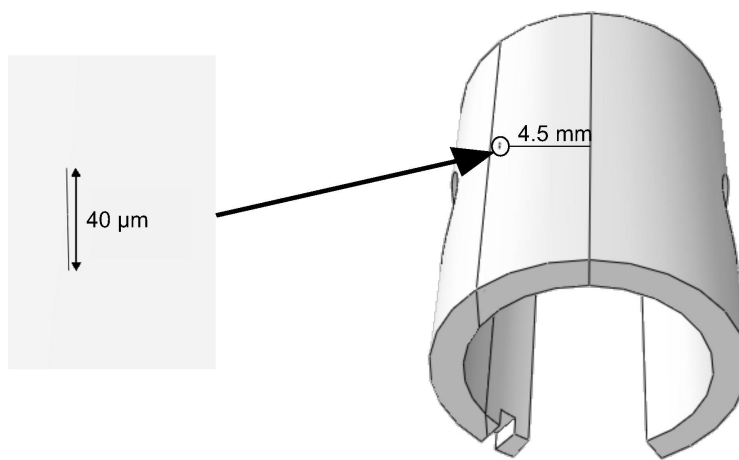


Figure 4.5: A simulation setup with the predefined crack shifted 4.5mm from the C-ring's apex, to be run in a region of lower bulk stresses

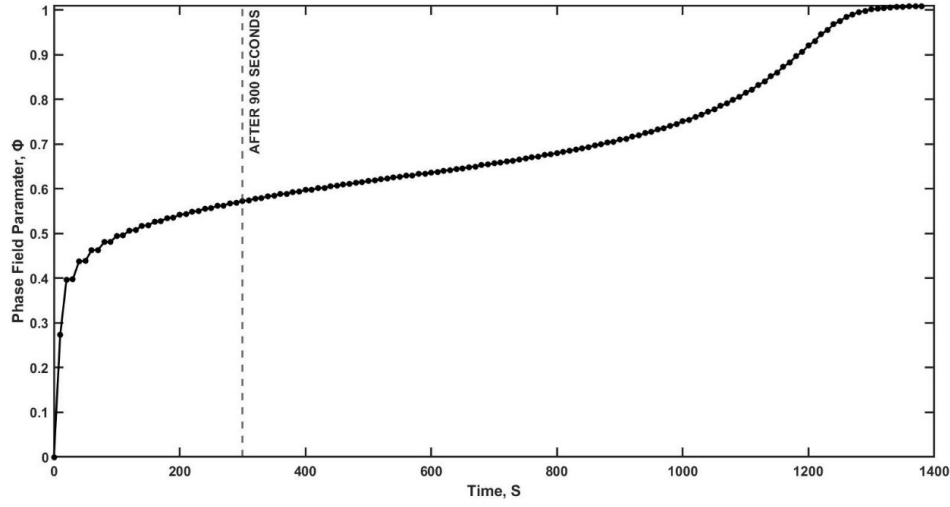


Figure 4.6: Results from initial PFM that represents the time-dependent nature of the C-ring experiment

4.3 Predicting Interaction Length Scales Using SEM Images

4.3.1 Crack Shielding

Figure 4.3 shows that the well-spaced cracks could grow unbound owing to the lack of interaction. Thus, a study was conducted to assess the effects of crack interaction mechanisms on propagation, particularly crack shielding and coalescence, and the distances required to encourage it. Two parallel cracks ($80 \times 40 \mu\text{m}$ and $40 \times 20 \mu\text{m}$) were separated by distance L (50, 100, 150, and $200 \mu\text{m}$), as shown in Figure 4.7.

Both the hydrostatic stress (MPa, the driving force for the diffusion process) and the damage parameter, Φ , were extracted from the first three nodes ahead of each crack (as shown in Figure 4.7b). As mentioned in Chapter 3, hydrostatic stress is degraded as the crack propagates (as a function of Φ); however, it is clear that when crack shielding occurs, it begins to stagnate for the non-dominant crack (as seen in Figure 4.8a, b, and

c). The crack shielding effect was most noticeable for a 50 μm (Figure 4.8a) separation distance, which saw Φ for the longer crack reach and surpass a value of 1 (full cracked material) but for the shorter crack it halts at 0.45 (Figure 4.8a).

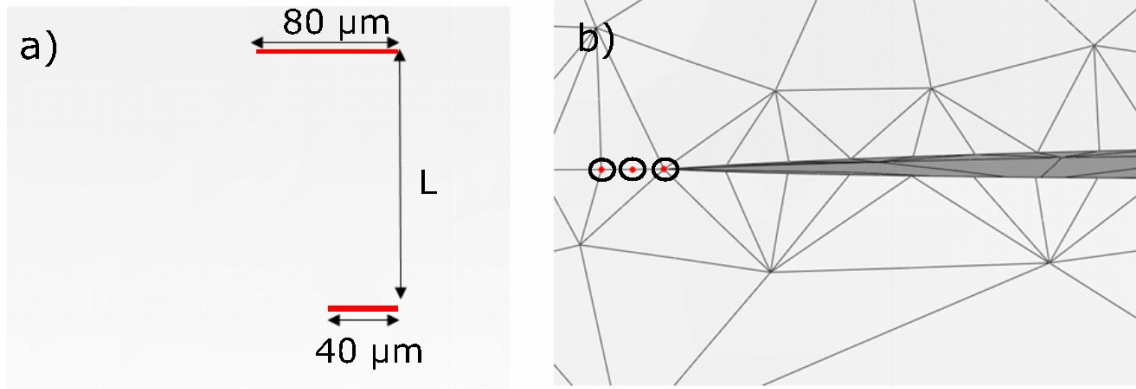


Figure 4.7: a) Predefined crack lengths and the spacing between them and b) nodes from which results were extracted

Separation distances of 100 and 150 μm (Figure 4.8b and c respectively) developed very similar results, with both significantly progressing until approximately $\Phi = 0.8$ before stagnating, with the main difference being that a 150 μm separation resulted in slightly higher hydrostatic stress. Further separation to 200 μm (Figure 4.8d) showed that both cracks reached $\Phi = 1$ unaffected and grew independently without any interaction.

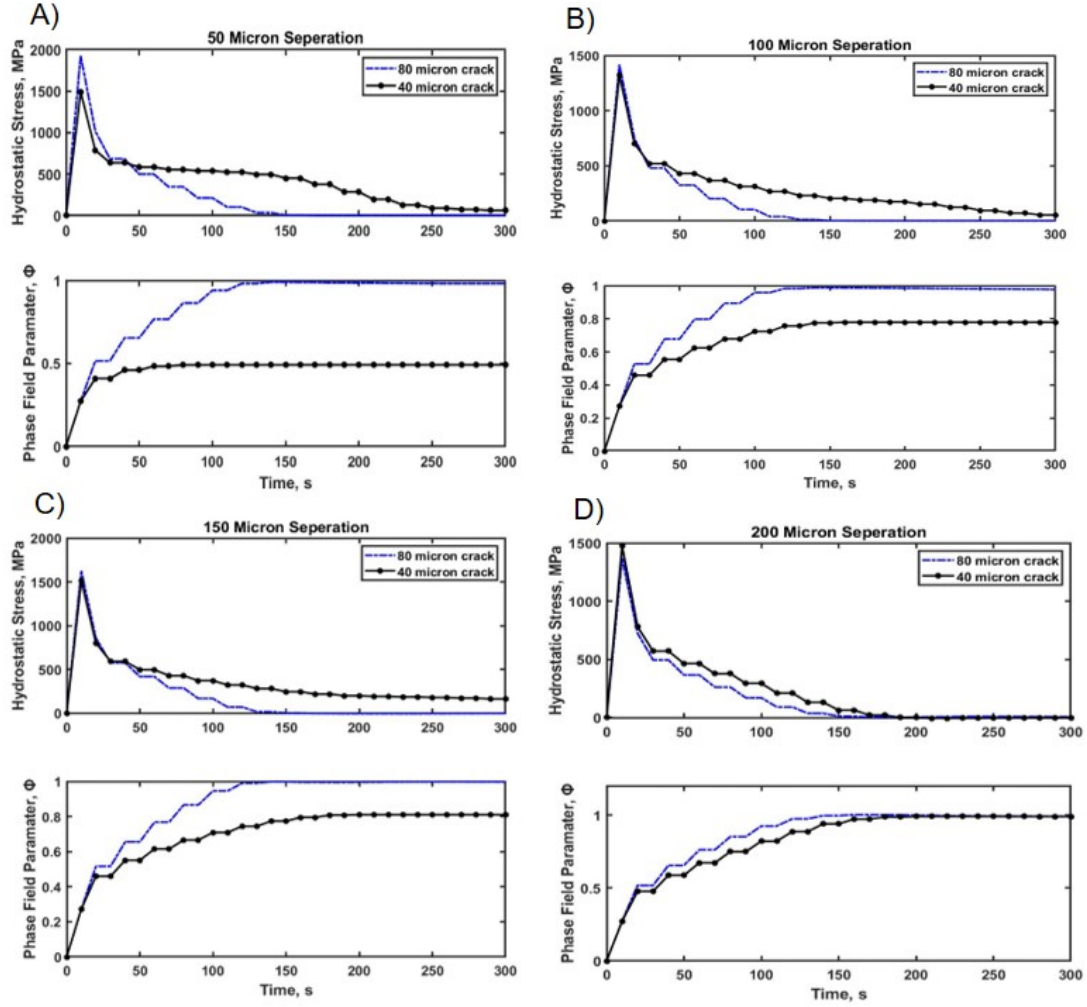


Figure 4.8: Plots representing the development of both Φ and Hydrostatic stress (MPa) for separation distances of 50, 100, 150, and 200 μm . The figure shows that as the separation distance increases from 50 to 200 μm , the chances of shielding occurring is reduced

Using this knowledge of crack interaction and critical spacing, SEM images from SCC C-ring experiments [86] were analysed to find cases of potential interaction. Figure 4.9a shows a prime example of this, where there are three visible cracks, with cracks #1 and #2 separated by a distance of 200 μm , and #3 separated by a little less than 100 μm . A simulation was created to replicate this crack formation ($50 \times 20 \mu\text{m}$ crack embryos were used as initiation sites), as shown in Figure 4.9b, and assess whether crack interaction mechanisms could explain the stagnation of crack #3 any further to the left. The results showed that cracks #1 and #2 could grow without interaction because of sufficient spacing (as shown in Figure 4.10a). Similarly, crack #3 grew to the right without issue, but as it

began progressing to the left, a crack-shielding mechanism was initiated and the crack was arrested. This is highlighted in Figure 4.11 which represents the development of Φ at three locations ahead of the crack tip #3 (P1, P2, and P3 in Figure 4.10b), with P1 and P2 reaching $\Phi = 1$ relatively quickly. However, P3 exhibited a shielding effect at 20 s and did not go any further than $\Phi = 0.8$. It is also worth pointing out the accuracy of the prediction for the minimum distance needed to observe a crack interaction, with the SEM image below doing well to match the predicted value.

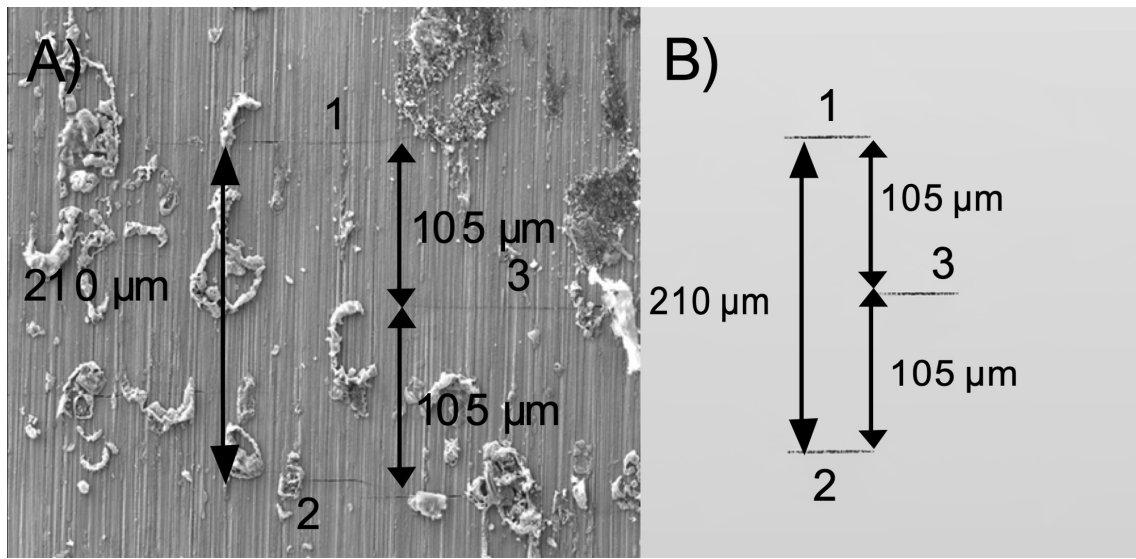


Figure 4.9: a) An example of a crack shielding mechanism using an SEM image and b) Crack formation to represent the SEM image used for a simulation

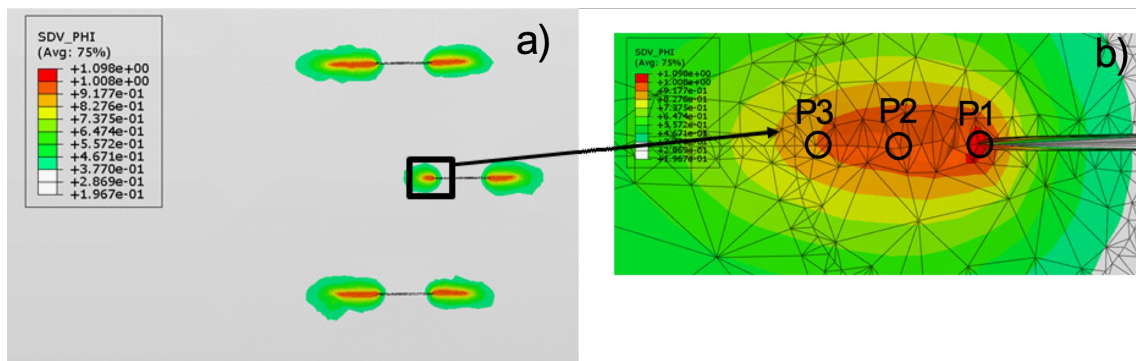


Figure 4.10: a) end result for the above layout showing the final crack formation and b) points from which the data was extracted for crack #3

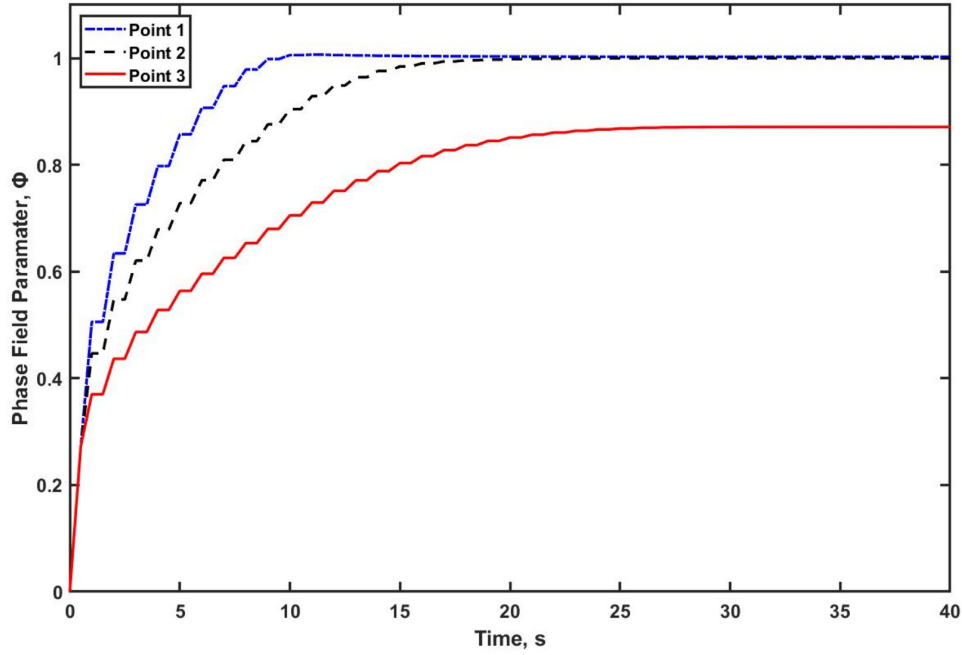


Figure 4.11: A plot representing the convergence of Φ to 1 for P1 and P2 but stagnation of P3 ahead of crack tip #3

4.3.2 Crack Coalescence

Further analysis of the SEM images also showed cases of closely packed cracks merging, resulting in newly formed longer cracks, realistically with a stress intensity sufficiently high to sustain rapid propagation. A great example of this scenario is shown in Figure 4.12b, in which cracks #2 and #3 merge, while #1 begins changing planes and converges with the tip of crack #3. It is now clear that the normal distance between cracks is critical for understanding whether cracks shield, coalesce, or grow unaffected, and the results from this study have shown that 10 μm or less between off-parallel cracks resulted in crack coalescence. To further analyse this scenario, a simulation was run to replicate Figure 4.12b and the results are presented in Figure 4.13.

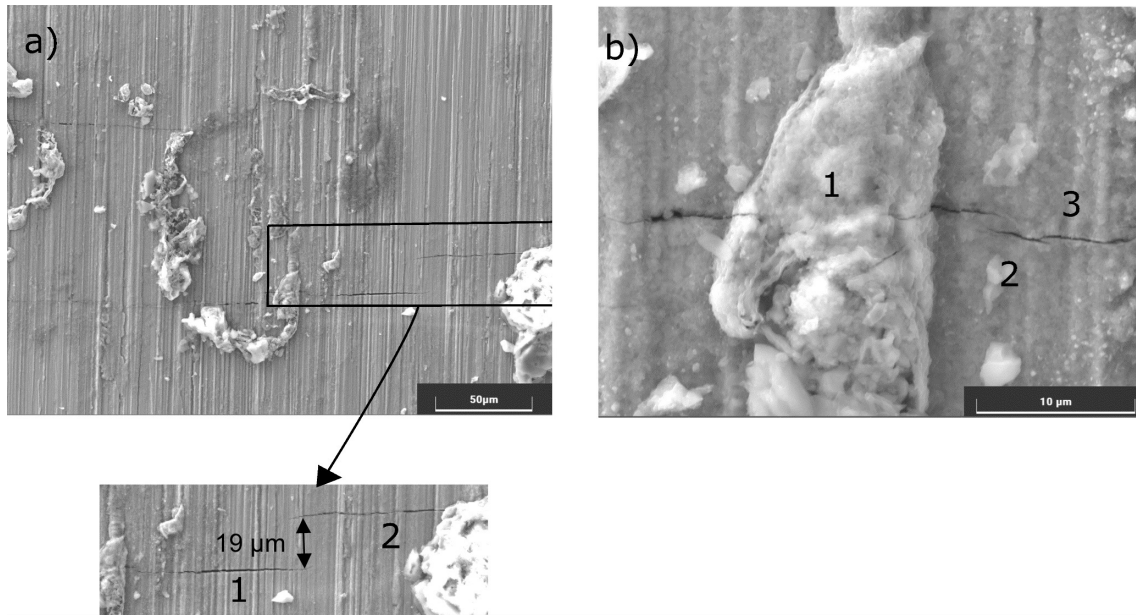


Figure 4.12: Magnified SEM images that represent examples of crack tip shielding and coalescence

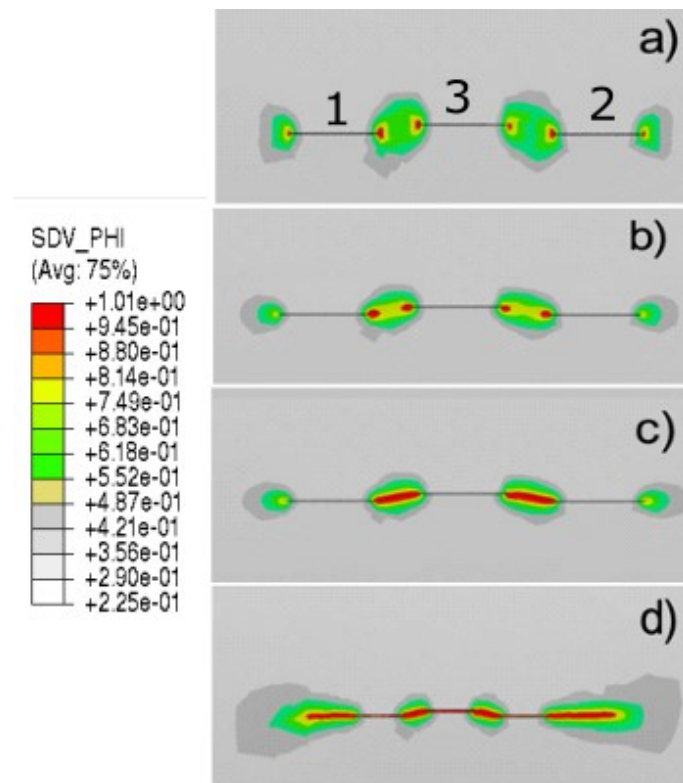


Figure 4.13: Simulation results that were run to further understand crack tip coalescence effects between off parallel cracks with a separation distance of 10 μm or less. Results show that under this critical separation distance crack coalescence is encouraged due to a stress intensification

Three cracks were located on the apex of the C-ring (with dimensions of $50 \times 20 \mu\text{m}$), with the central crack separated by a parallel distance of $10 \mu\text{m}$. The results indicated that for $10 \mu\text{m}$, there was a clear stress intensification and an increase in Φ for the area between the cracks. This is clear in Figure 4.13 which shows the steady merging of the three cracks to form one crack. The plots in Figure 4.14b and c show that the hydrostatic stress ahead of cracks #1 and #2 degrades to 0, indicating that $\Phi = 1$, further highlighting the coalescence effect. This scenario was then run twice, but with increased separation distances of 50 and $100 \mu\text{m}$, to find that crack shielding becomes the dominant mechanism. Figures 4.14c and d demonstrate this mechanism by highlighting the stagnation in both Φ and the hydrostatic stress of crack #3, leaving crack #1 to grow without interaction. No recalibration of the model was needed to capture the coalescence effect shown in Figures 4.10 and 4.13, and this further emphasises the importance of the role of stress in these tests, which is controlled by the critical spacing and distances between cracks.

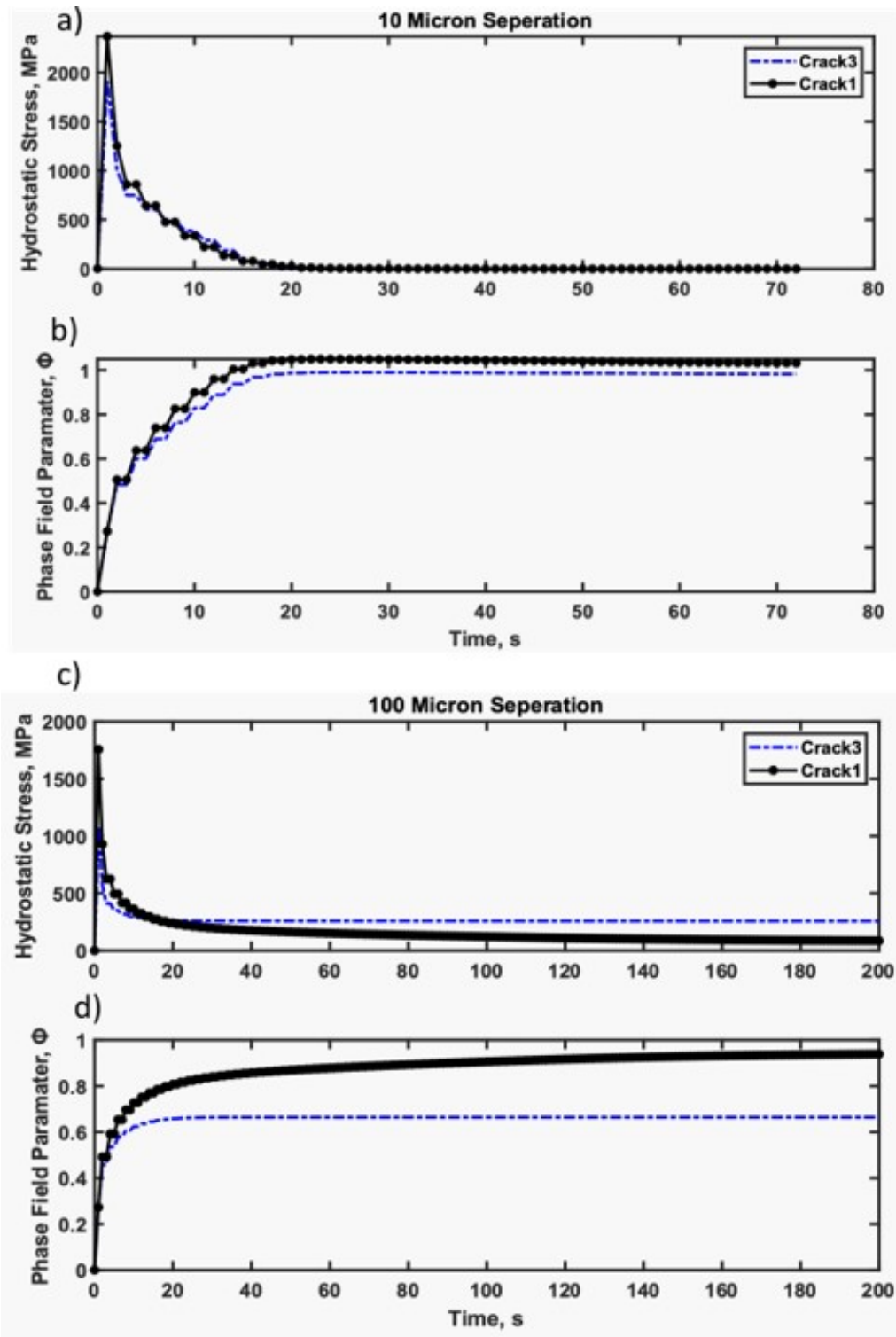


Figure 4.14: Results from the above simulation that shows stress intensification as a result of coalescence effects (a and b) and the stagnation of both stress and Φ as a result of shielding (c and d)

4.4 Understanding Role of Salt Deposition Site Using PFM

4.4.1 2D Salt Deposition Simulation

The above results have highlighted the role of separation distances needed to initiate interaction mechanisms, which saw the CGR either accelerating or halting, depending on the spacing and configuration of the cracks. However, previous research [86, 225] has clearly shown that cracks initiate under the halo structure of the salt deposition site. The combination of these results naturally leads to questioning the role of the spatial distribution of salt deposition sites; hence, PFM was implemented in a simple 2D model that was designed after assessing SEM images (such as those in Figure 4.15)[223].

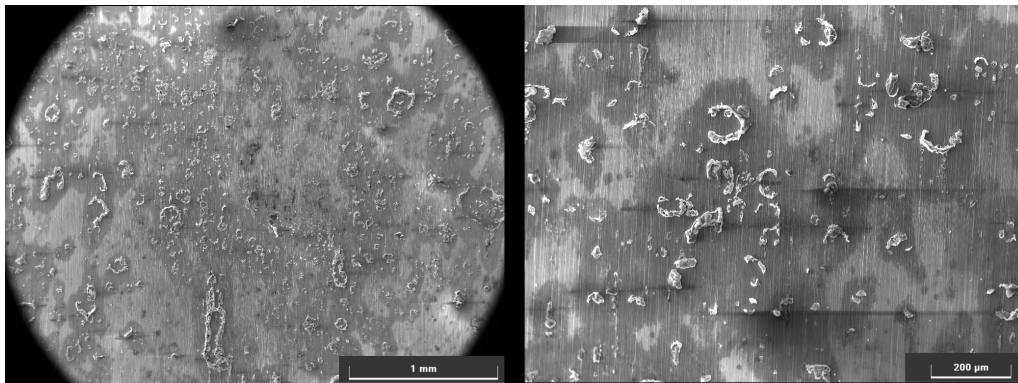


Figure 4.15: SEM images highlighting the random nature of salt deposition sites as a result of the traditional spray deposition methods

This simple 2D model is summarised in Figure 4.16 which comprises a 1500×1500 μm block, within which an 800×800 μm area is defined. This area progressively saw an increase in the density of salt deposition sites (of constant concentration) across the four models, with the first using just a single deposition site and the second, third, and fourth using 4, 9, and 16, respectively. Here, $\Phi = 1$ was defined for the deposition sites to represent the corroded material, and the nature of the propagation pattern was assessed. The outer perimeter was used to apply the boundary conditions, where the bottom of the structure was pinned ($U_X=U_Y=U_Z=0$), the top nodes were displaced by 0.2625 mm, and the sides were restricted in the x-direction ($U_X=0$), which in turn produced a uniformly

distributed stress of 700 MPa. Each deposition site was assigned a concentration of 0.5 wppm and associated with a diffusion coefficient which matches that of Sulphur in nickel at 600°C [207]. Finally, elastic properties matching those of CMSX-4 in intermediate temperature ranges were employed. The aim of this test was to analyse the effect of different densities of salt deposition sites on the crack interaction mechanisms for a 15-min experimental time.

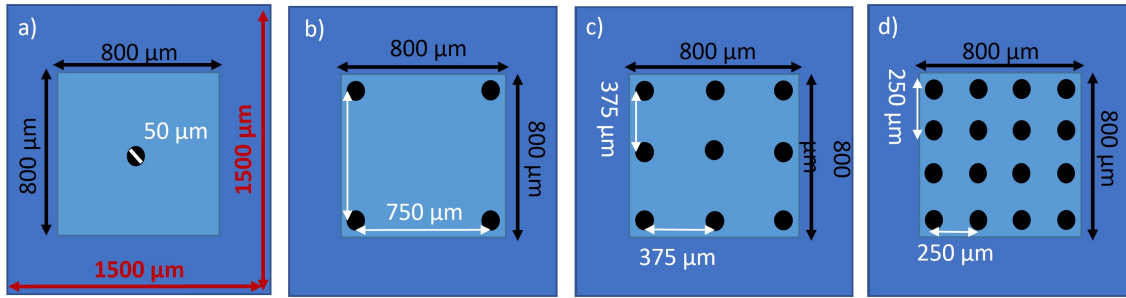


Figure 4.16: A picture to represent the simple 2D simulation used to analyse the salt deposition densities on the CGR. 1, 4, 9, and 16 deposition sites respectively were used

The results are highlighted in Figure 4.17 with the PF damage parameter Φ plotted to the left to illustrate its transition from 0 (undamaged) to 1 (fully damaged) material. The first plot, as isolated, sees no interactive behaviours and, as such, is allowed to grow freely and likely unbound. As the number of sites increased from one to four, a coalescence effect was observed between the perpendicular cracks (for both the top and bottom sets), which we can now assume as two newly formed longer cracks. However, as there was sufficient spacing between these two long cracks, no interactive mechanisms were observed, and a symmetrical effect occurred. A very similar situation is seen when increased to 9 and 16 deposition sites, whereby a coalescence occurs between the outermost set of cracks to form two new long cracks. However, a strong stress shielding effect occurs at the innermost sites owing to a reduction in the separation distances, allowing for one crack to dominate another and reduce local crack tip stresses. The final two plots of the figure show that the crack embryos start extending before the propagation is halted.

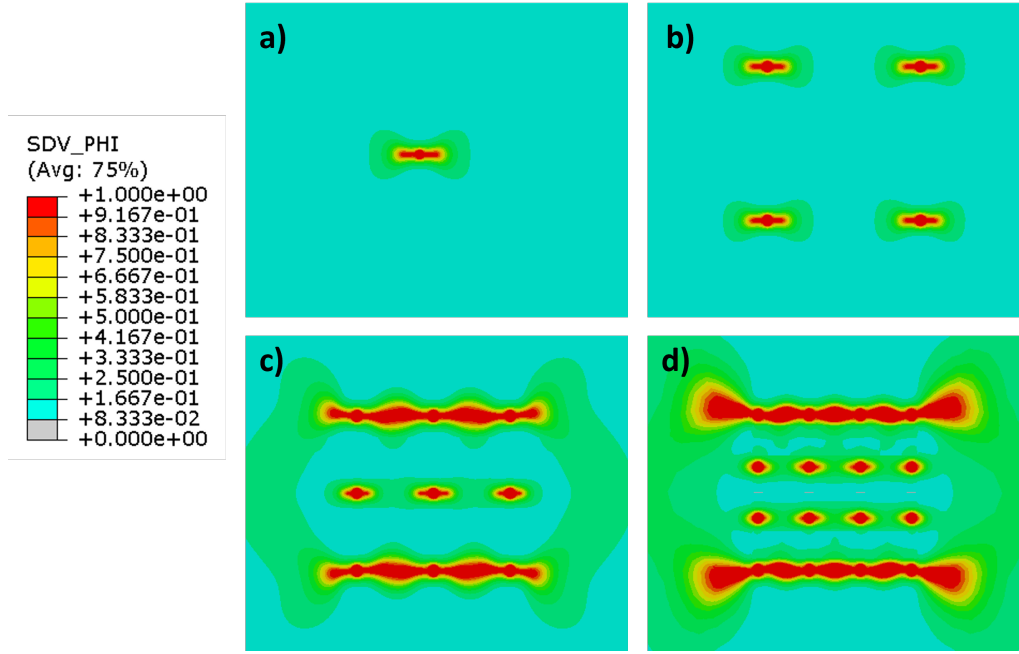


Figure 4.17: Final configuration of cracks for varying densities of salt depositions

The stagnation of the inner deposition sites in Figure 4.17c and d shows that the salt deposition density has the potential to stunt crack growth in the early stages. Several factors, including the deposition method, can affect the deposition density. The random nature associated with the traditional method of spray deposition (e.g. like that used in the case of the above SEM images) ultimately encourages clustering of salt particles or, in contrast, their isolation. Therefore, it is crucial to understand salt deposition patterns to better predict its role. Factors such as salt flux may also play a significant role; for example, if the volume of salt solution remains constant but the salt flux is increased, it is probable that a higher density of salt depositions for a given area would be observed, and an associated spike in the number of embryos initiated, hence greater stress shielding would occur. However, if the salt flux is reduced, fewer salt depositions would probably be seen for the same area, and as such, the chances of crack shielding are lower, resulting in fewer but longer cracks. This suggests that a low salt flux could be more detrimental than a high flux. However, a threshold could be considered above which a high flux would now become more detrimental, as it may encourage a coalescence effect (i.e. the merging of multiple neighbouring cracks), which could result in a rapid increase in the crack tip

stress intensity [226]. A similar study corroborated this hypothesis [227], however, further investigation is required to confirm this hypothesis.

4.4.2 3D Salt Deposition Simulation

This deposition pattern effect was then tested using a 3D simulation, where the C-ring geometry was modelled using a single deposition site. An established predefined crack (400 μm in length $\times$ 250 μm in depth) was initiated in the centre of the apex, and an environment that matched that of the furnace was simulated to assess how long the crack would grow if a singular deposition site was used. A total experimental time of 50 hr was simulated using a crystal orientation that matched that of a previously used sample, and the results are summarised in Figure 4.18.

It is shown that a singular deposition site is predicted to have a total crack extension of approximately 1750 μm (or 1.75 mm) owing to a lack of interaction. In contrast, when compared to optical and SEM images from C-ring tests (as seen in Figure 4.15), the predicted crack length seems somewhat unrealistic, with results generally showing high crack density regions with crack lengths within a 20-300 μm range. The PFM results in this section have already provided an explanation for this by showing a deceleration of the CGR owing to a local stress relaxation effect induced by bordering cracks. As such, in order to validate this hypothesis and the accuracy of the modelling results, the experimental setup needs to be redesigned in such a way that it replicates a scenario similar to that in Figure 4.18 and hence minimises crack interaction mechanisms.

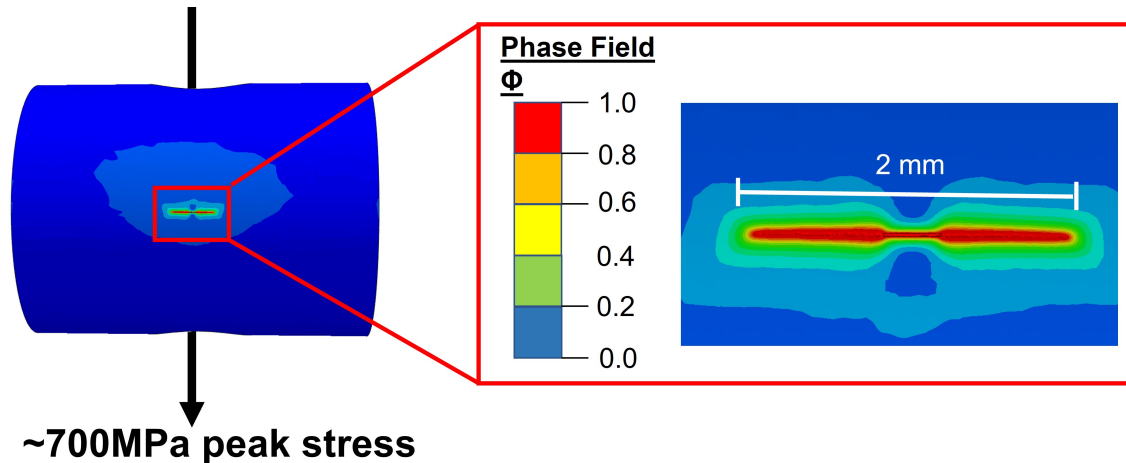


Figure 4.18: A schematic diagram of the PFM results that were used to predict crack extension when a singular salt deposition site is used

4.5 Discussion

The implementation of experimental results from other studies [25] within PFM has highlighted potential benefits for further understanding how and why the crack growth rate stagnates, enhances, or continues to grow without issue. The models were initially parameterised to match both the time- and stress-dependent mechanisms observed to play a role in the SCC C-ring experiments, thereby increasing the reliability of the model for predicting potential crack interaction mechanisms. In summary, PFM has proven to be a useful tool, when parameterised against experimental data, to understand the true extent of crack interaction mechanisms regardless of corrosive mechanisms.

This chapter highlights the importance of stress in these SCC mechanisms, and how critical separation distances and crack formations have the potential to drastically alter the CGR, either causing peak stresses to plummet or intensify. However, further to this point, the distribution and density of the initial salt deposition could potentially be more important, as it is known that cracks initiate under these halos [25]. Numerous factors may affect this salt deposition density, which may encourage both the clustering and isolation of deposition sites, such as the salt flux for a given concentration of a solution. If

the salt flux for a given concentration increases, it is likely that a higher density of salt depositions would be observed, and as such, more initiation embryos would be observed for a given area. This would naturally lead to a higher probability of crack interaction mechanisms occurring and a reduction in CGR [227], whereas if a lower salt flux is used, fewer initiations would potentially occur, leading to the isolation of cracks. This isolation will minimise crack interactions, and if given the driving force it requires, represents the chance of growing unbound. The experimental temperature and salt deposition techniques may also have an effect. However, this all requires further physical testing, some of which will be covered in the next chapter

This chapter has discussed the potential outcomes of a high density of salt depositions and crack embryos; however, the results naturally lead to the question of what would happen if salt depositions were isolated. Statistically, the probability of having a singular salt deposition on the surface of a turbine blade is probable, and this scenario should be considered and tested for. Based on the results in this chapter, it is believed that an isolated crack has the potential to be far more catastrophic for a component in comparison to a high density of cracks and suggests that the way in which the salt is deposited could dictate the end result. The next chapter uses these results to prove that.

4.6 Final Remarks

This chapter highlights initial PFM results that were validated against experimental results (and accompanying SEM images) to better understand stress corrosion-driven crack propagation in nickel alloys at moderate temperatures, which will later be used to inform physical testing. Hence, it can be concluded that,

1. Previous studies [25] have shown that there is a stress-dependent mechanism occurring that is highly dependent on both the total C-ring displacement and the crystal orientation of the material. Beyond certain points on the surface of the C-ring,

crack extension either becomes a function of a time-dependent mechanism or does not propagate. The PFM has reproduced these results and highlighted the stress gradient across the C-ring.

2. PFM results have predicted that crack distribution, crack lengths, and separation distances could all play a significant role in determining just how critical a given configuration can be. Modelling results showed that interaction mechanisms can either rapidly accelerate or decelerate the CGR through crack coalescence and shielding mechanisms, respectively.
3. Simple 2D simulations have highlighted that the density of deposition sites is arguably more critical to the CGR, given that cracks initiate underneath these deposition halos. This PFM prediction has will be further tested in the next chapter.
4. Finally, this chapter has suggested potential changes with the C-ring tests, specifically with the salt deposition method, that will be further tested in the next session.

In summary, this study has highlighted the role of interaction mechanisms (from both salt boundary initiations and crack propagation) in understanding SCC mechanisms; hence, this leads to questioning the salt deposition method itself. The modelling know-how can then be used to better define the experimental setup.

Chapter 5

Integration of FEA, Experiments, and Microscopy

5.1 Introduction

The integration of multiple testing and characterisation techniques often comes with difficulties such as how to correctly display data. However, when done right can prove beneficial, as not all data can be obtained experimentally, but can be modelled. The efficient integration of modelling and experiments has many advantages, including bringing about a more informed understanding of mechanisms, reducing the number of experiments, reducing the overall costs of a project, and is often more time efficient. This chapter highlights the potential of combining the best aspects of modelling, microscopy, and experiments to set and answer hypotheses, and why it is necessary to feed the outcome back into the loop of techniques. Computational work was further enhanced by integration with microscopy, where a combined group effort converted real cracks from XRM data to form 3D CAD geometries that could be modelled. At the micro-scale, enhanced SEM and XRM images were also used to further understand how the microstructure might aid or impede crack propagation mechanisms by using PFM to predict the crack extension depending on the size, shape, and volume fraction of the γ' precipitates and γ channels.

5.2 Revisiting C-ring Experimental Setup

Early modelling results highlighted that crack interaction mechanisms played a greater role in C-ring cracks than initially thought. Consequently, computational results were used to reinforce the experiments (i.e. C-ring and corrosion-fatigue tests), and a new experimental setup was proposed. This novel protocol was designed with the goal of minimising the chance of crack interaction and hence accelerating CGR by adjusting the salt distribution. This was done across two iterations to encourage what was referred to as the ‘worst case’ scenario in which shielding is restricted.

5.2.1 ‘Zebra’ Specimen

The first iteration of the specific experiment was titled the ‘Zebra specimen’. Results such as those in Figure 4.3 highlight that established cracks with a spacing of over 150-200 μm lack any crack shielding and, as such, grow unbound. The design process for the Zebra specimen involved covering sections of the C-ring surface before coating it with a salt solution, as shown in Figure 5.1. Both the salt flux value ($1.2 \mu\text{g}/\text{cm}^2/\text{hr}$) and the spraying deposition method used for this test were the same as all previously run C-ring tests [25], but tape was now used. Removal of the tape resulted in distinct regions of salted and unsalted material. It was hypothesised that the cracks that initiate next to the salt/no salt boundaries would continue to grow without surrounding interaction, and as such, reach lengths that have not been observed before in experiments. Figure 5.2a and b represent the optical images taken before and after furnace exposure respectively, and clearly match the initial design in Figure 5.1. The C-ring was then exposed to the same environment (i.e. air + 50 PPM of SO_2) for 50 hr, with the end result highlighted by the optical image in Figure 5.2b.

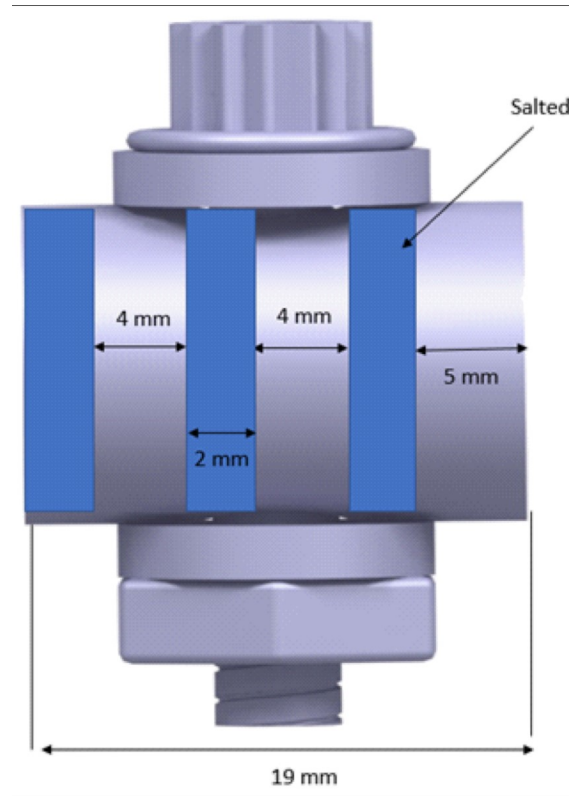


Figure 5.1: Zebra C-ring Test - Note the taped-off regions done before salting as a way to minimise the likelihood of crack interaction mechanisms

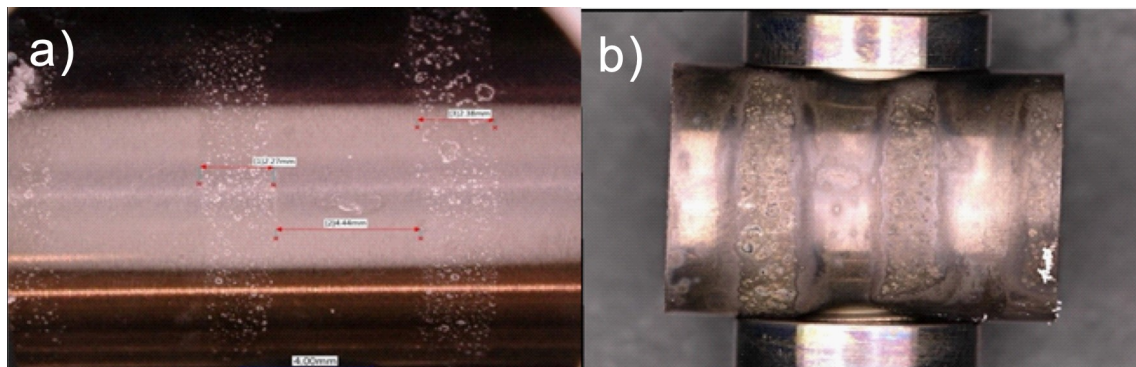


Figure 5.2: Optical images representing the distribution of salt for the Zebra specimen with a) pre-exposure and b) post-exposure to the furnace

SEM analysis of the specimen surfaces was conducted, as shown in Figure 5.3, which shows crack propagation lengths between 500 and 1000 μm . This indicates that cracks are now as much as three times the length of those previously observed (which were at most 300 μm) in multiple C-ring tests [223]. The results align well with those of the initial models and clearly highlight that the placement and pattern of salt deposition have

a critical effect on the expected crack propagation. This highlights the predictive power of the PFM which showed crack extensions of over 850 μm , and shows that if sufficient spacing was maintained, cracks would continue to grow. Similarly, the maximum crack depths also showed an extension, with previous C-ring tests showing a maximum depth of 240 μm , whereas the Zebra test reached 370 μm .

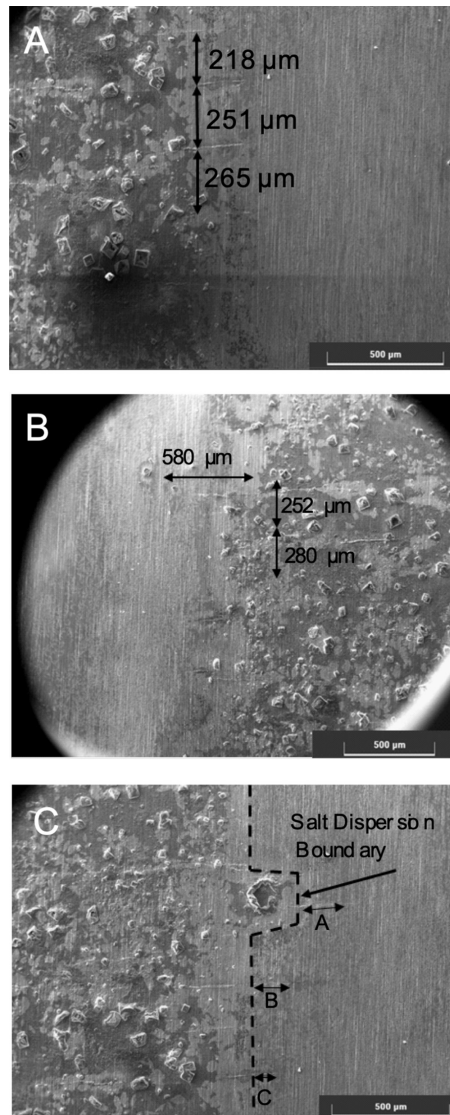


Figure 5.3: SEM images highlighting a) standardised spacing between long cracks which agree with simulation results b) example of long cracks and c) positioning of the salt dispersion boundary and its role on total crack lengths. These samples were exposed for 50 hours in an air + 50 PPM of SO_2 containing environment with a salt flux of 1.2 $\mu\text{g}/\text{cm}^2/\text{hr}$.

Section 4.3.1 narrowed down the critical spacing needed to observe an interaction be-

tween these C-ring cracks to roughly 10-200 μm . Between these values, crack shielding mechanisms dominated, and resulted in the stagnation of any further propagation, above which cracks propagated without any interaction. Figure 5.3a shows a strikingly consistent and regular spacing over this 200 μm value predicted to avoid shielding by the model in Chapter 4. Examples of this critical spacing are presented in Figure 5.3b. This Zebra test highlights the role of salt boundary diffusion on the extent of crack propagation (or at least the early stage), showing that cracks rapidly halt once they pass through the boundary from salted to unsalted material. Indeed, this mechanism of crack arrest immediately after the boundary, indicates another length scale that affects cracking, as illustrated in Figures 5.3c and 5.4. Factors such as the temperature, salt deposition method, and salt solution concentration could all be responsible for the effects of this newly determined length scale.

Crack interactions were not considered a dominant mechanism to explain why cracks stopped propagating until the model predicted it. This highlights how epistemic uncertainties involved in experiments are often overlooked and how even simple modelling can be used to overcome them. The ‘Zebra’ test was the result of the effective integration of models, microscopy, and experiments.

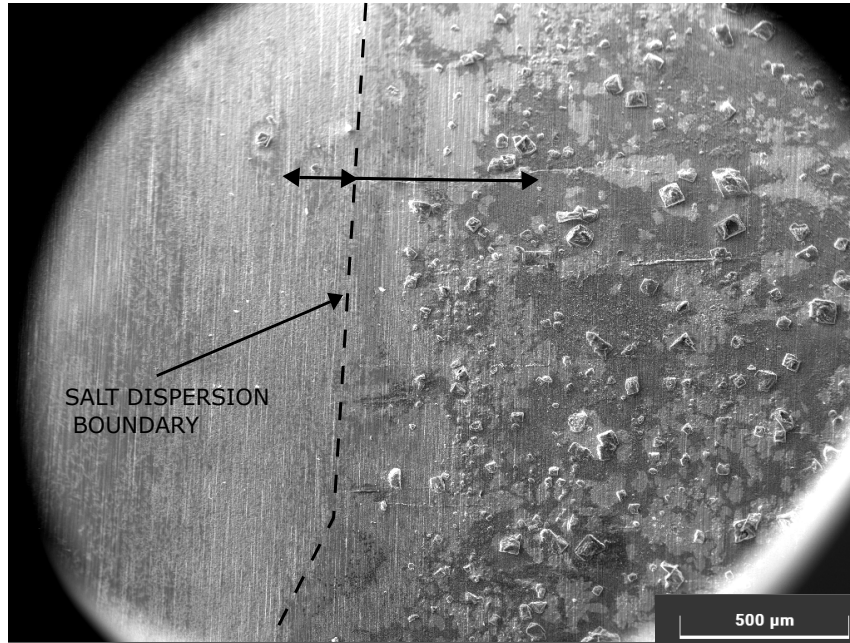


Figure 5.4: An example of an SEM image that shows crack propagation quickly stagnating after passing the salt dispersion boundary

5.2.2 ‘Dot’ Specimen

Using the ‘Zebra’ model and experiment, a further iteration was developed to prove that a singular salt deposition can be far more detrimental to a turbine blade when compared to a high density of depositions. This test is termed the ‘Dot’ test. The rationale behind this test was to further reduce the likelihood of crack interactions and achieve even longer cracks than the traditional and ‘Zebra’ C-ring tests.

The aim of this test was to minimise the salt deposition area on the C-ring surface. Three individual droplets were placed using a micropipette to ensure a sufficient spacing between them. However, unlike previous tests, where the salt flux remained constant, the concentration was increased to observe the effects of varying the salt flux on the total crack extension. This introduces another potential length scale, that is, the salt boundary dispersion. The initial droplet had a flux of $1.2 \mu\text{g}/\text{cm}^2/\text{hr}$, whereas the second and third droplets had fluxes two and four times this value, respectively. All other aspects of the experimental setup (i.e. temperature and displacement) remained the same, and were run for

a total duration of 50 hr at 550°C. Figure 5.5 and 5.6 show the C-ring surface before and after furnace exposure, respectively, and highlight that the higher the salt concentration, the smaller the area of the deposition site. This could be a result of the surface tension effect or reduced concentration of water.

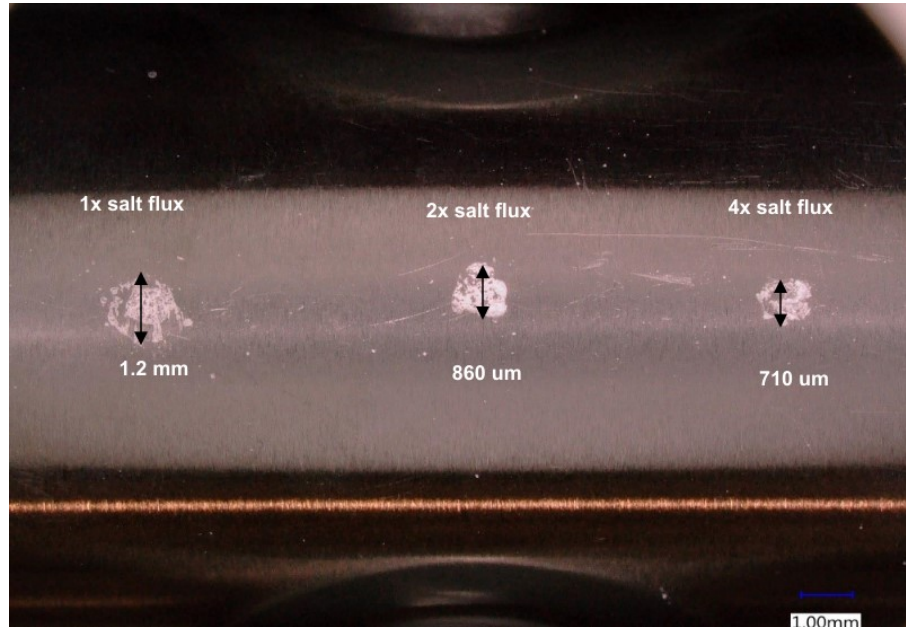


Figure 5.5: An optical image showing the distribution of salt solution droplets before exposure for 1x, 2x, and 4x salt flux

The primary purpose of this experiment was to isolate a salt deposition site. The final results are depicted by the optical images in Figure 5.6 taken after the test was run for 50 hr. Even if we just consider Figure 5.6b, which has the same salt flux as all previously run C-ring tests, it reached a final length of approximately 1.6 mm, a value more than five times longer than the most prolonged cracks previously seen (approximately 300 μm) and as much as twice those seen in some of the 'Zebra' cracks. The results showed that a singular salt deposition has the potential to be far more critical to turbine blades by drastically reducing their life in comparison to a high density of sites.

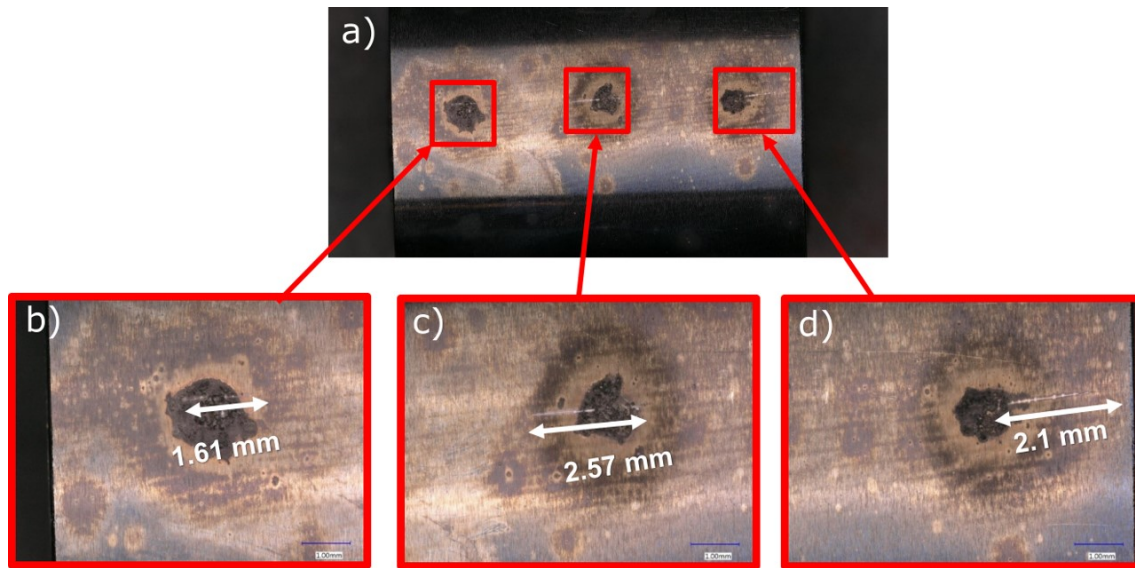


Figure 5.6: Optical images depicting the extent of cracking for each site of varying salt concentration with a) being an overview and b), c), and d) representing 1x, 2x, and 4x salt flux respectively

In this test, it was evident that as the salt flux increased, the crack lengths seemed to increase and even reached lengths of up to 2.5 mm. This effect is a result of the salt boundary dispersion length scales, which state that cracks will cease further extension shortly after passing the boundary from salted to unsalted material. In this test, as the salt flux increased, further diffusion of the salt boundary was observed. The dimensions of these dispersion boundaries are summarised in Figure 5.7 and Table 5.1 and were measured using the software ImageJ. There was an increase of over 7 mm^2 between the deposition sites of the 1x and 4x flux droplets, which generally explains why longer cracks are observed with higher salt concentrations when the deposition sites are isolated. In summary, this highlights that both the pattern of deposition and the amount of salt in a given solution need to be considered when investigating how critical the SCC can be on a material's life.

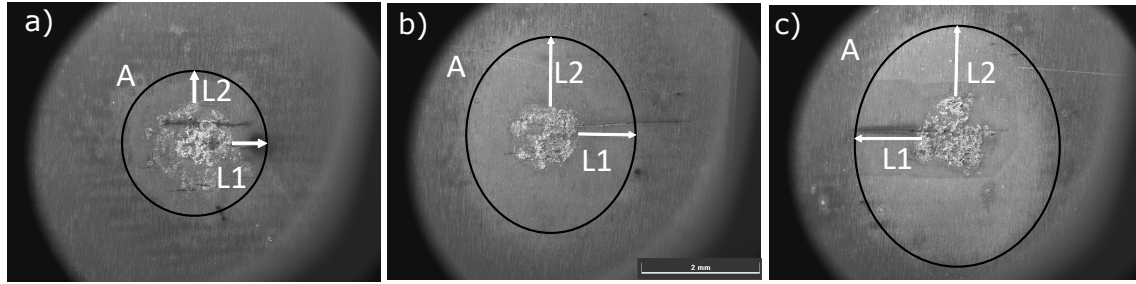


Figure 5.7: A schematic depicting an SEM images of varying salt flux ($1.2 \mu\text{g}/\text{cm}^2/\text{hr}$, twice, and four times as much respectively) showing that as salt flux increases so does the area of salt dispersion

Table 5.1: Final salt dispersion site dimensions (post-experiment) for the increasing salt flux values, highlighting this increase in total area as the flux increases

Measurement	1x Flux	2x Flux	4x Flux
L1 (mm)	0.44	1.45	1.53
L2 (mm)	0.34	1.32	1.59
Area (mm²)	4.15	10.68	11.28

XRM work was conducted on a part of the third sample, highlighted in Figure 5.8, and showed a single long crack as it propagated through the dendrite structure. Figure 5.9 highlights three TIFF slices from this dataset and shows that the crack deviated from the original path as it passed through a dendrite structure, before transitioning again as it passed through another. This indicates a microstructural effect that can alter crack paths. The dendrite might impede and resist further propagation, causing the crack to find a path of lower resistance and hence force a change in the crack path. However, further investigation is needed before justification can be provided. This could be achieved by reconstructing an approximation of the crack in relation to the dendrites and running an FEA model which could highlight potential justifications.

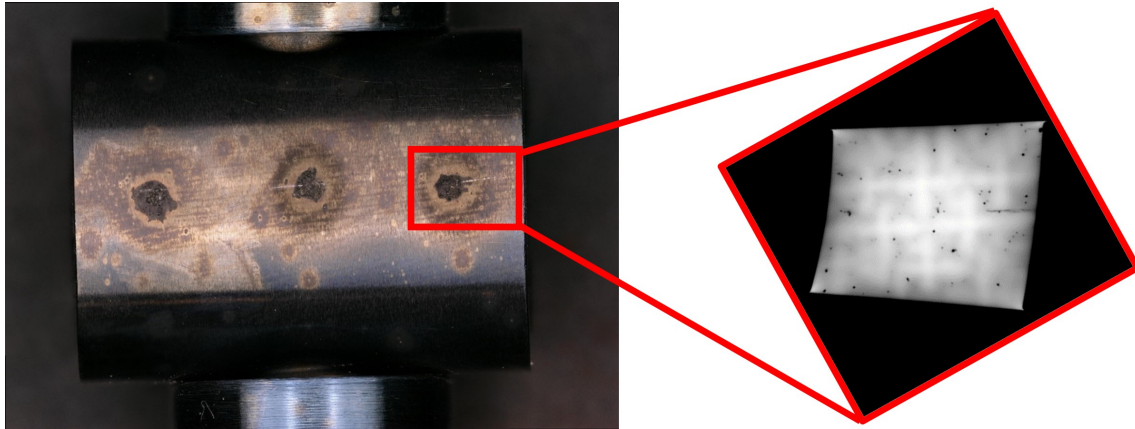


Figure 5.8: XRM data taken from the third sample which shows a single distinct crack as it propagates through the material

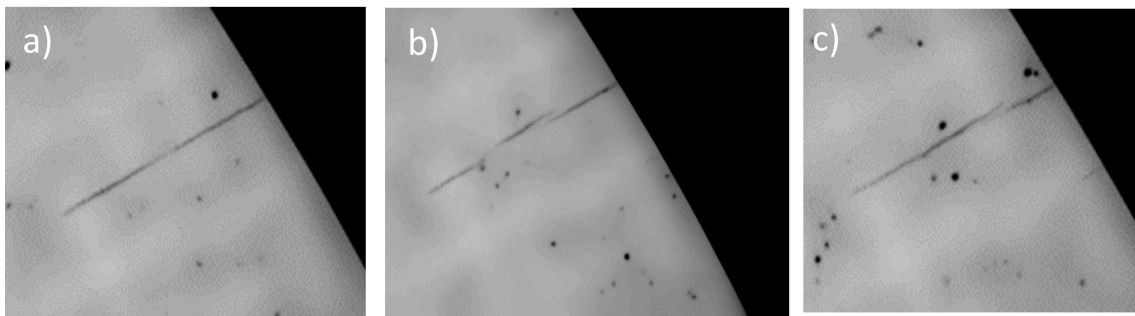


Figure 5.9: Three TIFF slices taken through the material which visualises the crack changing its path as it propagates through dendritic structures, highlighting a potential microstructural effect on determining the crack path

It is also observed in Figure 5.8 that all three cracks consistently grew parallel to the dendritic structure and, in some cases, along the interface between the dendritic and interdendritic regions. This is illustrated in Figure 5.10. It is hypothesised that this could be a mechanical property effect, where a mismatch in material properties (e.g., strength or fracture toughness of the different phases) may induce a stress gradient and therefore pull the cracks along these regions. Further studies are required to validate this hypothesis.



Figure 5.10: An optical image of the 2X salt flux shown above which highlights the crack growing parallel to the dendrite structure

5.3 Further Validation with Corrosion-Fatigue Experimental Setup

5.3.1 ‘Dot’ Corrosion-Fatigue Test

The work done in Chapters 5.2.1 and 5.2.2 conclusively demonstrated that well-spaced salt deposition sites could significantly reduce the life of CMSX-4; however, these tests only considered one material under one experimental and environmental condition. To further validate the role of crack-interaction mechanisms, it is essential to test different materials and degradation mechanisms. As such, the principles of the ‘Dot’ test were applied to cyclic loading tests at increased temperatures.

Using a similar experimental setup as that of the ‘Dot’ test, the fatigue specimen was initially warmed on a hot plate before a micropipette was used to place a small salt solution droplet (with a flux of $1.2 \mu\text{g}/\text{cm}^2/\text{hr}$) as close to the centre of the notch as possible (note that it was only placed on one of the two notches), as shown in Figure 5.11a. The remaining moisture surrounding the salt deposition site was removed by placing the component on the hot plate for a few seconds. The specimen was then loaded into the test rig, where it was exposed to an air + 300 vpm SO_2 -containing environment at 700°C , 150°C above that used in the C-ring tests. This increase would be sufficient to observe a shift in the primary SCC mechanism and determine whether crack interaction mechanisms still occurred. This fatigue test was strain-controlled, and a displacement was applied to maintain a nominal load of 680 MPa under cyclic loading (where $R=0$) using a 1-120-1-1s trapezoidal waveform (i.e., a 1s up, 120s hold, 1s down and 1s hold under no tension). This specific setup was run for CMSX-4 and CMSX-10 fatigue samples, and both tests were left to run until failure.

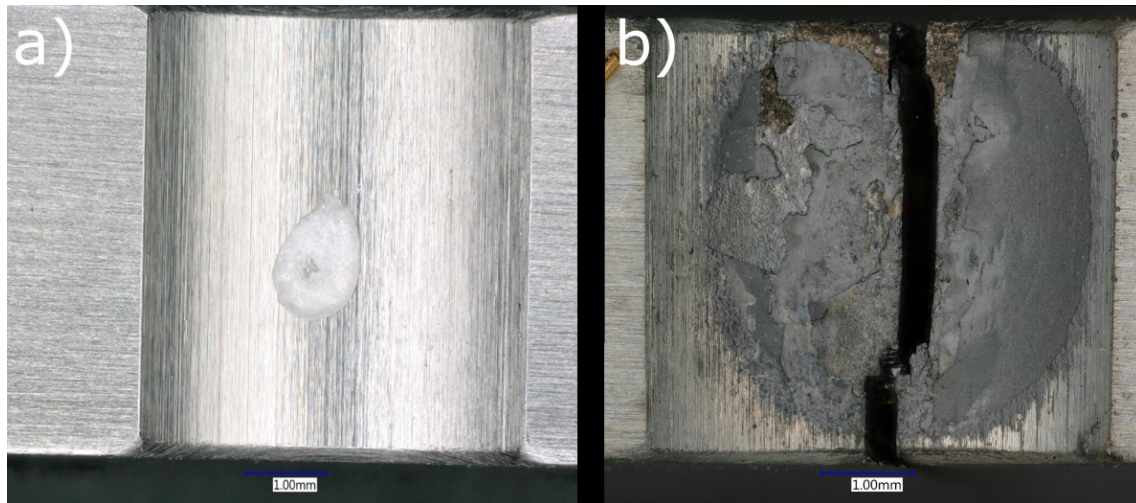


Figure 5.11: An optical image of the CMSX-4 sample showing a) the singular droplet of salt solution on a central point along the notch of the specimen and b) the notch after the failure of specimen

In the case of CMSX-4, the results showed that even at higher temperatures and under cyclic loading conditions, the crack interaction mechanisms had a significant effect on the expected life of the component. Figure 5.12 emphasises how quickly aerospace

materials, such as CMSX-4, can reach critical cracking should initiation embryos be isolated, and further validates the results from the specifically designed C-ring tests. The specimen (highlighted by the solid red line) failed within 1505 cycles (without the need to re-salt) and before doing so reached a total crack depth of 1.93 mm, shown in Figure 5.13. This data was then reviewed against three other random datasets from CMSX-4 fatigue specimens that were exposed to the same environmental and loading conditions. The only significant difference was observed in the salt deposition method. In these tests, the same salt flux was deposited on one of the two notches using a spray method. They were re-slated every 2000 cycles (previously thought to be essential for cracking) until the specimen failed to prevent the stagnation of crack growth, as indicated by the orange circles in Figure 5.12. This ‘Dot’ CMSX-4 fatigue test saw a 50% decrease in life compared to the next most damaging of the four tests. However, this reduction in material life was as high as 83% for the slowest tests, showing unequivocally that when the number of initiation embryos is minimised, it can be far more detrimental to the life of the component, and as such increases the probability of catastrophic failure. As this ‘Dot’ CMSX-4 test uses only a singular location for potential embryos to initiate, it almost eliminates the probability of any interaction mechanisms occurring entirely.

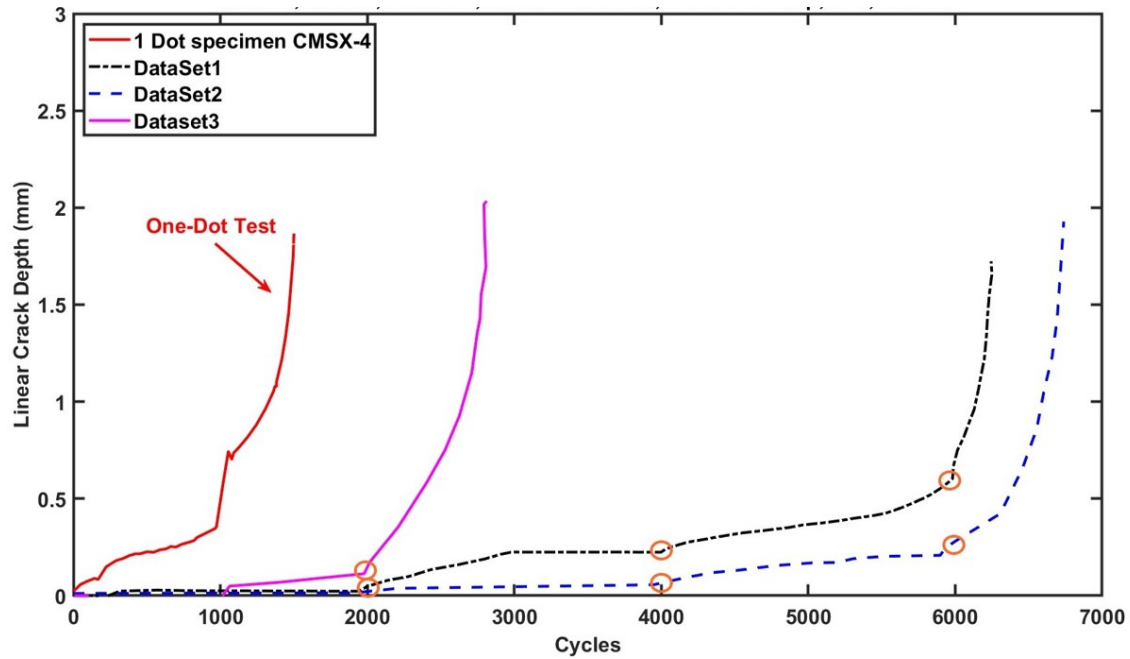


Figure 5.12: Plot highlighting the number of cycles to failure for the CMSX-4 ‘Dot’ corrosion-fatigue test (indicated by the red line), and three random CMSX-4 corrosion-fatigue tests. Please note that the ‘Dot’ test was not re-salted every 2000 cycles unlike the others. The All samples were exposed to 700°C in an air + 300 PPM of SO₂ containing environment.

With comparable CMSX-4 data, there were clear regions of stagnation in the linear crack depth. It is postulated that this could simply be a crack shielding effect, where there is a sufficiently high density of cracks such that the critical separation distances are low enough to see shielding. After re-salting, the datasets saw a spike in the CGR, which could be explained by the process of initiating fresh crack embryos within the critical separation distance needed to encourage crack coalescence. This merging effect would likely result in local stress intensification at the crack tips and as such, create a new crack with a sufficiently high stress intensity to cause a spike in the CGR.

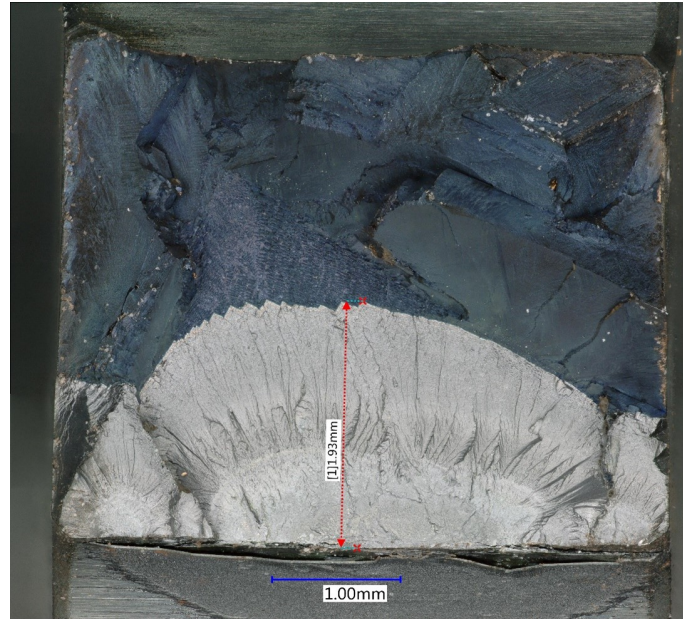


Figure 5.13: An optical image showing the crack surface morphology after the failure of the specimen, which occurred at approximately 1505 cycles

This test was also run for a CMSX-10 fatigue specimen, with most aspects of the setup remaining the same; the only difference was that the specimen was not heated before the salt droplet was placed. This means that the surface tension effect was reduced, and the dispersion of the salt before the test began was minimised. However, it was heated after the deposition to remove any unnecessary moisture. The pre- and post-exposed notches are shown in Figure 5.14a and b, respectively, again showing a single central salt deposition. This test did not reproduce the effect to the same extent as both the CMSX-4 C-ring, and fatigue tests. Several key points were obtained by assessing the crack surface, as shown in Figures 5.15 and 5.14b, such that it can be assumed that it failed along a different plane when compared to the previous test, and that the main crack initiated and extended away from the deposition site (as shown in Figure 5.14b).

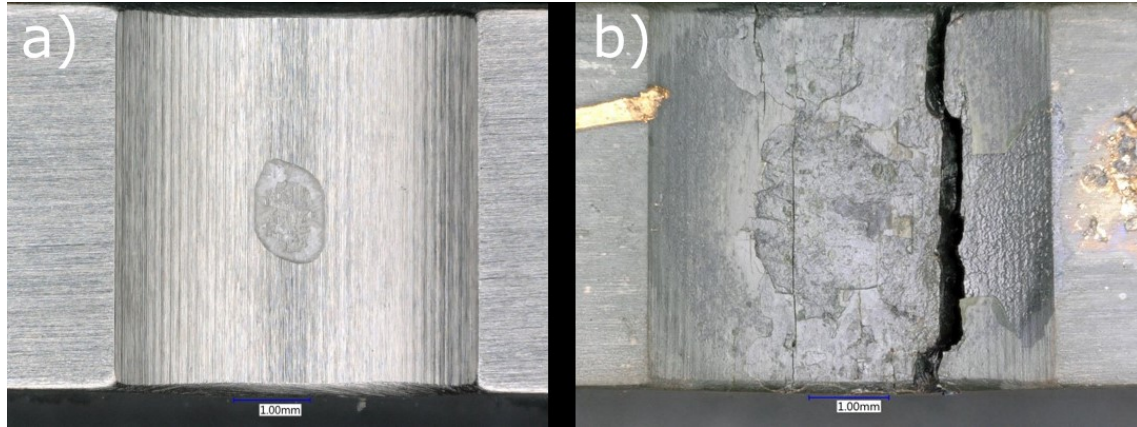


Figure 5.14: An optical image of the CMSX-10 sample showing a) the singular droplet of salt solution on a central point along the notch of the specimen and b) the notch after the failure of specimen

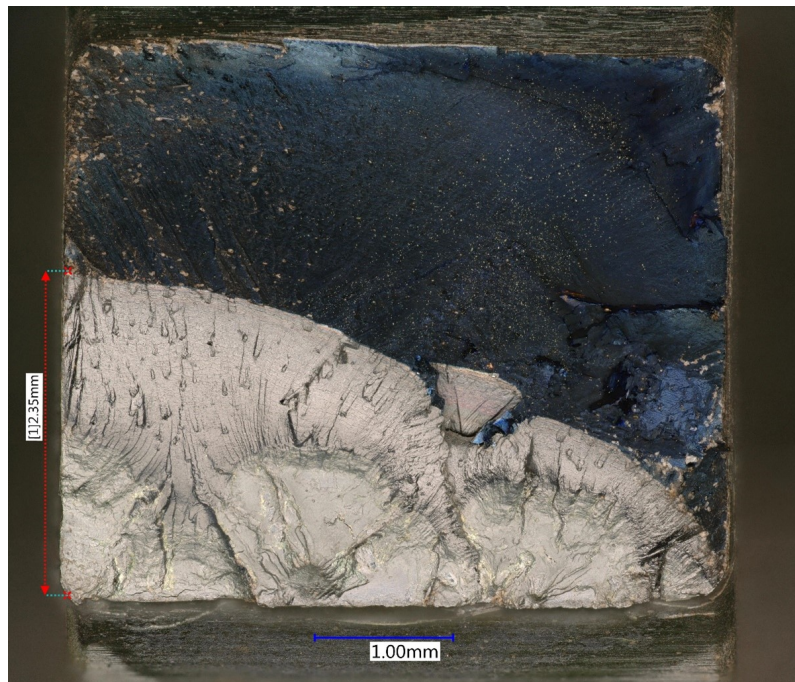


Figure 5.15: An optical image showing the crack surface morphology after the failure of the specimen, which occurred at approximately 8000 cycles

The red line depicted in Figure 5.16 represents the 'Dot' test and the remaining plots are the randomly selected data sets, which were re-salted every 2000 cycles. In contrast to the CMSX-4 test, the CMSX-10 specimen did not exhibit a significant reduction in the number of cycles required to achieve failure. However, it is worth noting that this test eventually failed at approximately 8000 cycles, without the need to re-salt. Hence, it

would be worthwhile re-running data sets 1-4 without re-salting to see if there is a difference between them and the 'Dot' test.

CMSX-10 has previously been shown to initiate more cracks in a given time frame compared to CMSX-4. However, even with more embryos initiating far fewer cracks in CMSX-10 propagate long enough to cause significant damage, especially in comparison with CMSX-4 (which could be due to several reasons for example different mechanical properties, different casting or treatment techniques etc.), where cracks exceed $100\text{ }\mu\text{m}$ in just 15 min [25]. It is postulated that, because of the microstructural differences between the two alloys, CMSX-10 struggles far more to propagate cracks, and as such, it is less likely to observe the effects of crack interactions. This could explain the difference between the results of the two 'Dot' tests.

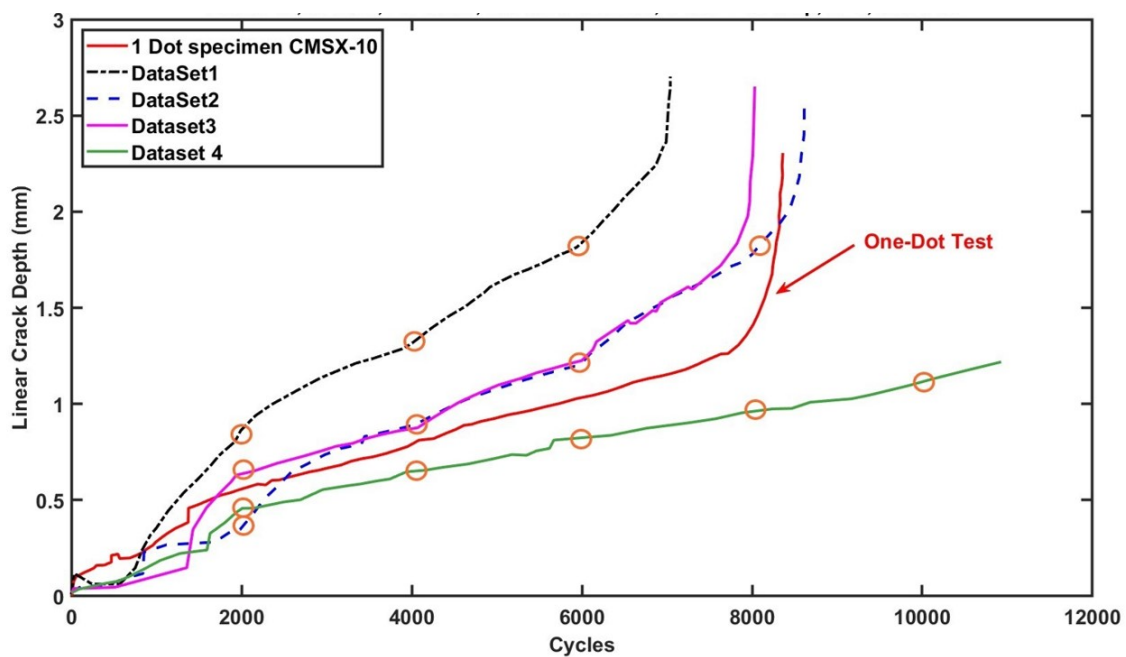


Figure 5.16: Plot highlighting the number of cycles to failure for the CMSX-10 Dot corrosion-fatigue test (indicated by the red line), and four random CMSX-10 corrosion-fatigue tests. Please note that the Dot test was not re-salted every 2000 cycles unlike the others, which is highlighted by the orange circles

5.4 Modelling Microstructural Variations

To assess the potential microstructural effect on the above results, a simplistic approach was adopted using 2D simulations to compare parameters that cannot be tested easily but rather modelled. Emphasis was placed on the ‘regular’ and ‘irregular’ microstructures, where CMSX-4 had a more regular structure and showed similar γ' precipitates as opposed to CMSX-10 which showed far more variation in γ' size, shape, and orientation. This is highlighted in Figure 5.17, where the disparity between the two sets is clear and the experiments show strikingly different crack growth responses. Differentiating the properties of the two microstructures is difficult to perform experimentally, but simple modelling approaches can provide important details.

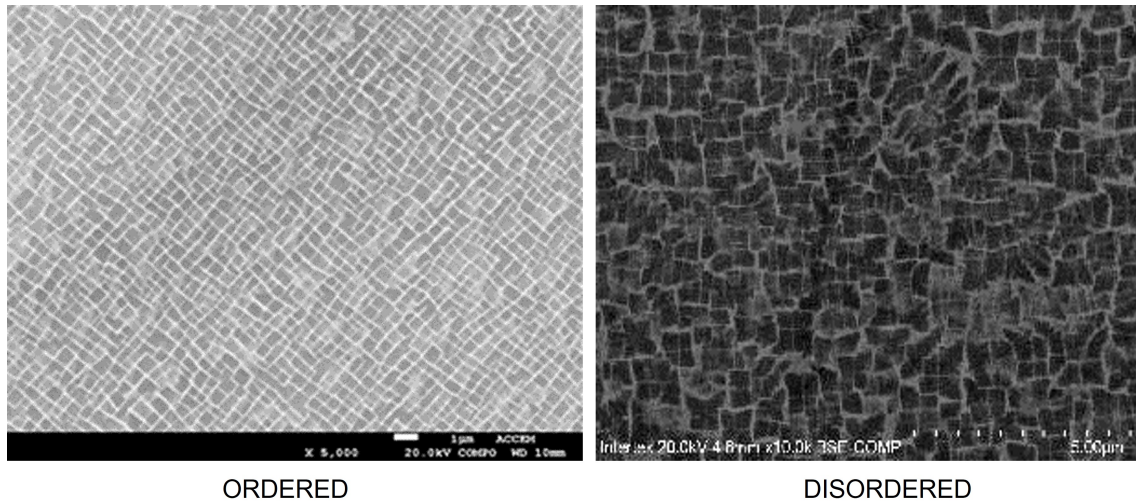


Figure 5.17: TEM images visualising the γ/γ' structures for CMSX-4 (left) and CMSX-10 (right) that were exposed to the same conditions. Note the more structured layout for CMSX-4 as opposed to CMSX-10

This study was inspired by the work of Karamitros et al.[228] and therefore followed a similar method, in which different elastic properties were assigned to the γ and γ' materials; these values are summarised in Table 5.2. This simulation was performed using a predefined field-variable boundary condition that allows the PFM to distinguish between the two structures, as highlighted in Figure 5.20. The regular γ/γ' system representing CMSX-4 uses $0.45\ \mu\text{m}$ precipitates separated by a $0.1\ \mu\text{m}$ γ channel, whereas the irreg-

ular system is based on a small section of the CMSX-10 SEM image which shows four precipitates, as shown in Figure 5.18. ImageJ was used to measure the γ' precipitates and γ channels, and the final formation for both simulations is shown in Figure 5.19. It should be noted that the lower-left precipitate for the irregular structure was given the same dimensions as those in the regular formation, and the predefined cracks (0.1 μm in length) were placed 0.015 μm to the right of the lower-left precipitate in both cases. This was performed to maintain compatibility between the two crack extension predictions.

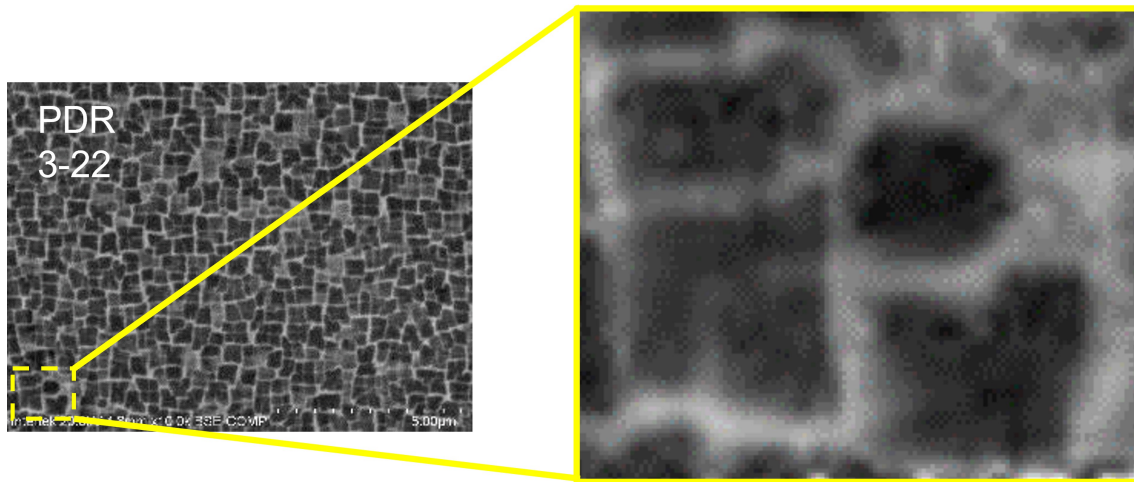


Figure 5.18: An example of the irregular microstructure that was used to design the γ/γ' formation

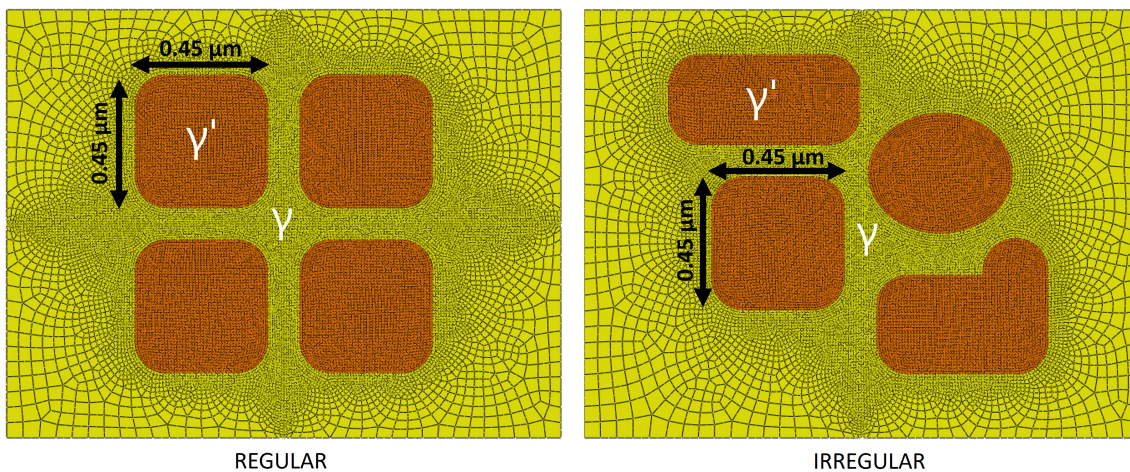


Figure 5.19: Simple 2D layout used to model the microstructure effect for CMSX-4 (left) and CMSX-10 (right)

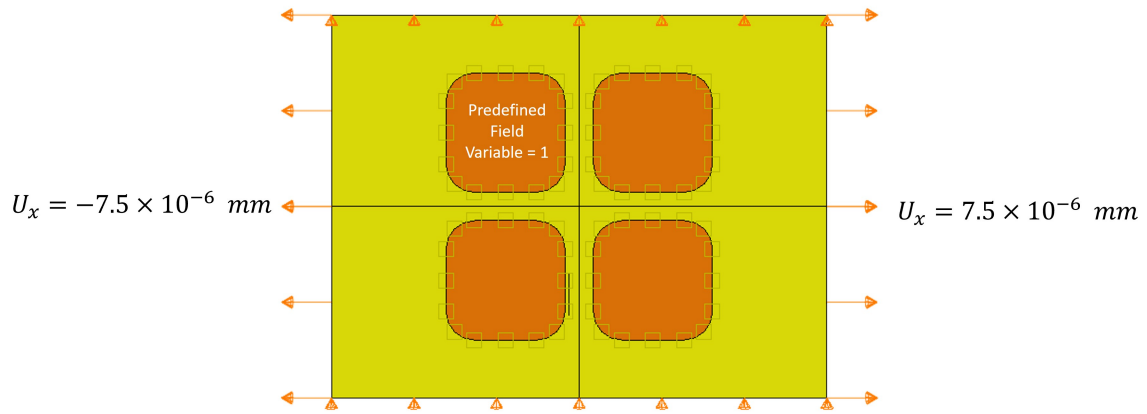


Figure 5.20: Boundary conditions used within Abaqus CAE to apply load to the block and the predefined fields used to distinguish between γ and γ' . The top and bottom nodes are pinned in the y-direction, whilst a displacement in the x-direction is applied to each side

Table 5.2: Elastic properties used for both γ and γ' for the microstructural assessment [228]

Elastic Property	γ	γ'
Young's Modulus (GPa)	92	95
Poisson's Ratio (ND)	0.415	0.427
Shear Modulus (GPa)	105	112

The 2D block was equally displaced by 7.5×10^{-6} mm on either side in the X direction to apply a distributed load across the structure. Table 5.3 highlights the parameters used in PFM, where the sulphur concentration, and accompanying properties, are associated with the predefined crack. An experimental temperature of 700°C is assumed to align with the 'Dot' corrosion-fatigue tests. It is worth noting that a lower $G_C(0)$ value is used for the microstructural tests when compared to the bulk models, as it is assumed that the fracture toughness at the interface between the channel and precipitate is lower than that at the bulk level.

Table 5.3: Parameters used for the PFM

Parameter	Value
Sulphur Concentration (wppm)	0.5
$G_c(0)$	5
Temperature ($^{\circ}\text{C}/\text{K}$)	700/973
Molar Gas Constant ($\text{N mm}/(\text{mol K})$)	8314.5
Gibbs Free Energy Difference (kJ/mol)	-30
χ	0.998
Diffusion Coefficient (mm^2/s)	1×10^{-10}
Steepness Parameter, 'a'	1.25

Figure 5.21 shows a von Mises stress plot for both the regular and irregular microstructures and clearly indicates a stress differential between the two formations. The regular spacing between the precipitates allows for stress intensification in the γ channel, and the stress gradient can act as a driving force to pull cracks through the lower stress regions (i.e. from one set of parallel precipitates to the next). In comparison, the irregular microstructure led to stress relaxation in the γ channel, with regions of this channel seeing sufficiently low stresses to prevent crack propagation. This essentially leads to precipitates that act as barriers to crack extension. This postulation is further emphasised by the PFM results in Figure 5.22, which show that in a 100-second experimental time, the regular structure exhibited significant crack propagation with no propagation seen in the irregular structure. However, despite what the PFM has predicted, further testing (both physically and computationally) needs conducting to validate the preliminary hypothesis set. Several sensitivity analyses also need conducting to better understand the above results as factors such as boundary conditions and PFM parameters (such as the length scale parameter) may significantly alter the results.

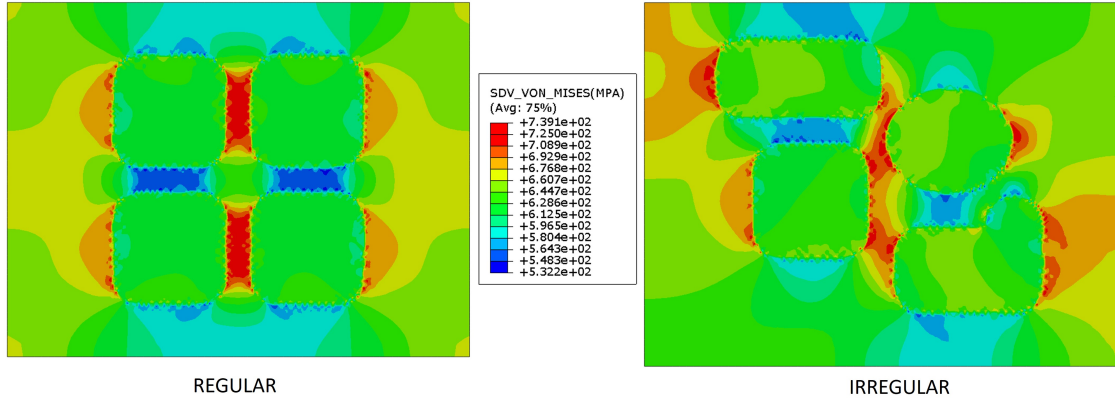


Figure 5.21: von Mises stress (MPa) maps for the regular (left) and irregular (right) microstructures

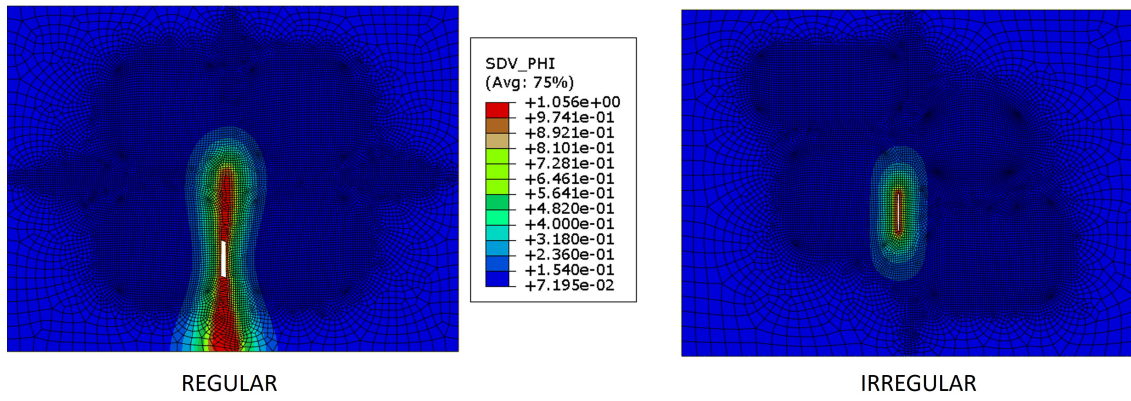


Figure 5.22: Final predicted crack extension after 100 seconds for the regular (left) and irregular (right) microstructures

5.5 Highlighting the Stress Effect on CGR and Salt Dispersion

The variation between CMSX-4 and CMSX-10 corrosion-fatigue tests prompted a desire to apply the same experimental setup but to a CMSX-10 C-ring. The C-ring was displaced to the same value as that of the CMSX-4 test (0.44 mm), and the same NaCl salt solution (with a flux of $1.2 \mu\text{g}/\text{cm}^2/\text{hr}$) was used. However, unlike the CMSX-4 test, where the salt fluxes used were 1x, 2x, and 4x the initial value, this time 1x and 0.5x the initial flux was used. A micropipette was again adopted to deposit solution droplets onto the surface of the C-ring, with the right and left droplets in Figure 5.23 being the 1x and 0.5x salt fluxes, respectively. The test was run for the same 50 hr exposure time.

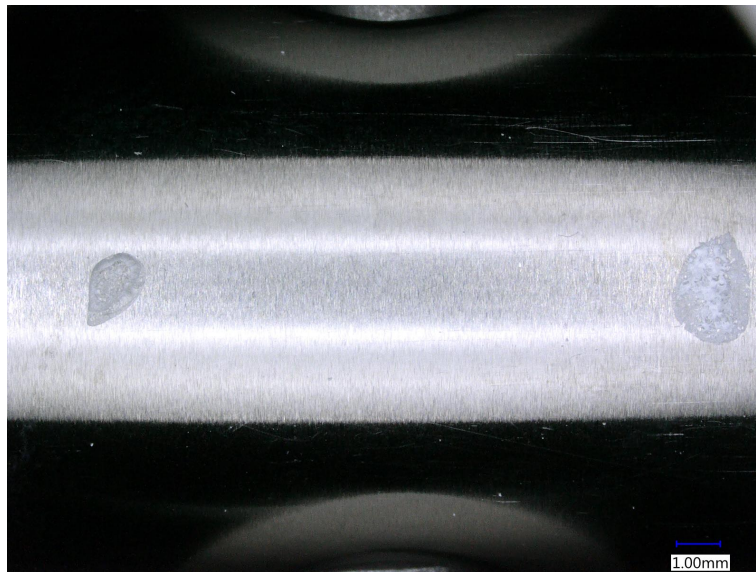


Figure 5.23: Pre-exposed CMSX-10 C-ring with salt 1x (left) and 0.5x (right) salt flux values

The results in Figure 5.24 once again highlight that this ‘Dot’ test produced significantly longer cracks, with SEM analysis (Figure 5.25) measuring 3.068 and 1.578 mm for the 1x and 0.5x salt fluxes, respectively. However, contrary to what was expected, the lower salt flux exhibited a greater dispersion of the salt boundary, which could be potentially explained by the positioning of the salt deposition in relation to the C-ring

edges, where the stress was highest. Additionally, this C-ring had a significantly lower κ angle of only 2° , meaning that the peak stress was expected to be not only more central to the apex of the C-ring but also at a higher value. ImageJ was used to approximately measure the area of the salt depositions and the distance from the edge, and it was found that the 0.5x and 1x salt flux droplets were approximately 1.361 mm and 2.491 mm from the edges, respectively, before exposure. A simple simulation was performed to account for the correct bolt displacement and crystal orientation of the test specimen. The resulting stress distribution and positioning relative to the salt deposition sites are shown in Figure 5.26. It was evident that the 0.5x salt flux deposition was in an area of greater stress than the other droplets, which could potentially explain why the boundary of the lower salt flux was dispersed the furthest. It is clear that the right side of the C-ring has a consistently higher peak stress which is more continuous than that on the left side, indicating an area of stress relaxation between the two regions of peak stress. First, it was assumed that this was a mesh effect; thus, a mesh sensitivity analysis was conducted by increasing the mesh density. Both meshes highlight the same response, indicating that this is merely a crystal orientation effect. The inconsistent deposition method is also an uncertainty that may explain why the lower flux resulted in greater dispersion. As shown in Figure 5.23, the initial droplet was smaller and far more controlled for the 1x salt flux compared to the 0.5x flux, which had a greater contact area with the C-ring surface upon placement. This, combined with the hot C-ring causing a surface tension effect, could explain the difference in dispersion.

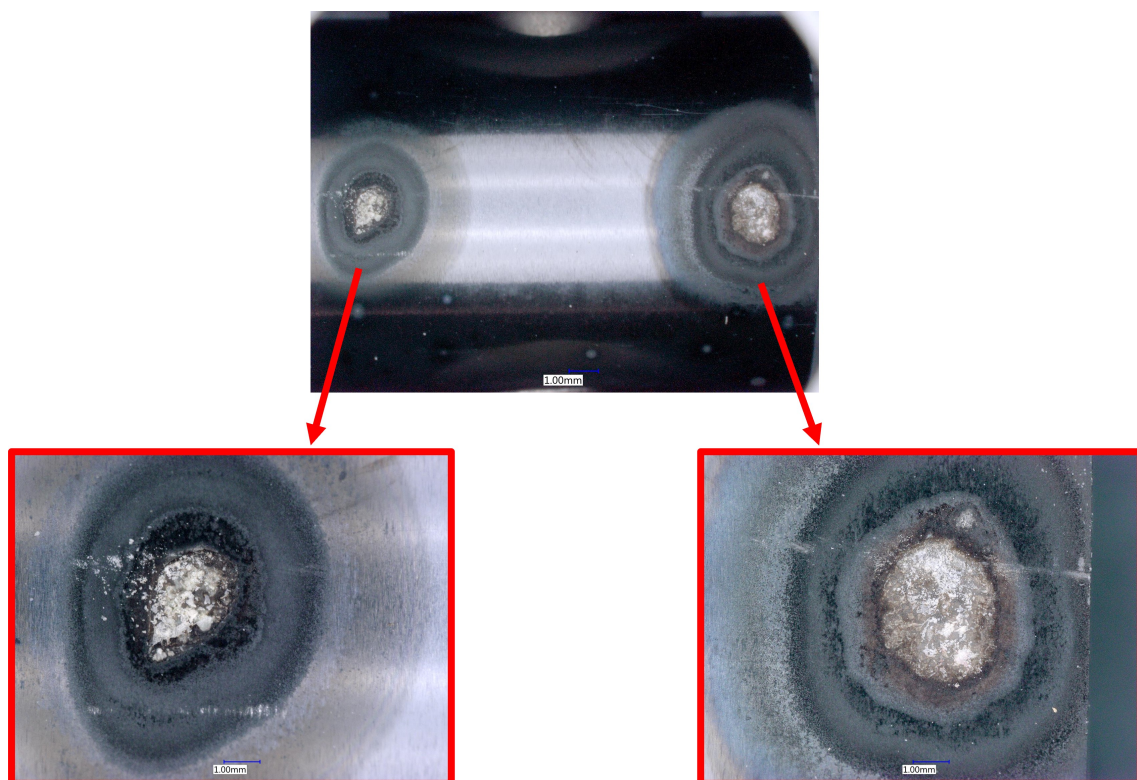


Figure 5.24: Post-exposed CMSX-10 C-ring with 1x (left) and 0.5x (right) salt flux values

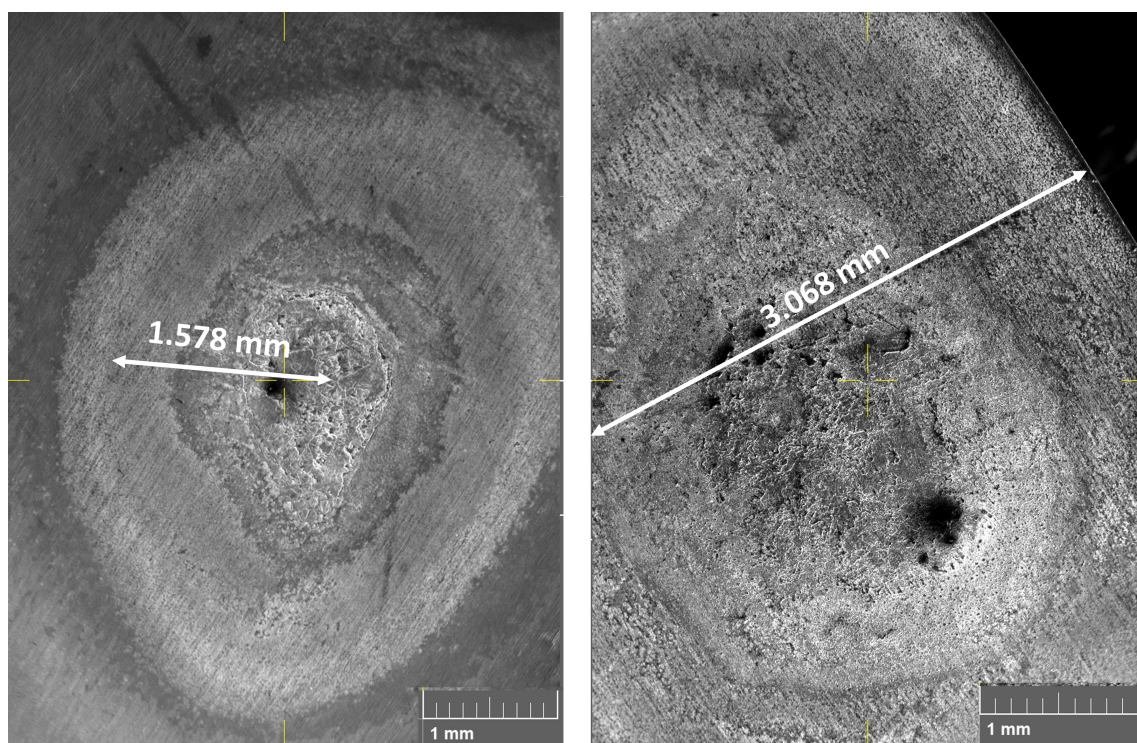


Figure 5.25: SEM analysis of the 1x (left) and 0.5x (right) salt flux values highlighting the longest cracks seen in both cases

However, this test somewhat contradicts the varying results observed between the two corrosion-fatigue ‘Dot’ specimens, which showed that CMSX-4 had a faster CGR than CMSX-10 when the deposition sites were isolated. This suggests that CMSX-10 was less sensitive to isolated cracks. In contrast, the CMSX-10 C-ring had longer cracks in the same 50 hr period than CMSX-4. As such, a test to compare the expected stress distribution for the two C-rings, given their correct crystal orientation and applied load, is shown in Figure 5.27. In both cases, the salt droplets were placed as close to the apex as possible. Despite CMSX-10 being postulated to be more resistant to crack propagation, the droplets were in a region with significantly higher stresses compared to CMSX-4, which had a higher κ angle, and thus, peak stresses deviated further from the apex. There is a difference of approximately 200 MPa along the apex of the two C-rings, highlighting how crucial stress is in determining crack extension under these conditions. This indicates further uncertainty in the current experimental setup, and how preliminary modelling can inform how and where depositions are placed, depending on the predicted stress distribution. If this test were to be run again, a comparison like that seen below would be conducted before determining the positioning of the droplets, as it could be said that the cracks from the two C-ring tests are incomparable owing to such a significant stress difference. It should also be noted that at the time of writing, this C-ring was not sectioned, and as such, it is not known whether this sample was of the ‘regular’ or ‘irregular’ microstructural variation. This is a prime example of how the redefined experiment is passed back to modelling for further validation and reassessment.

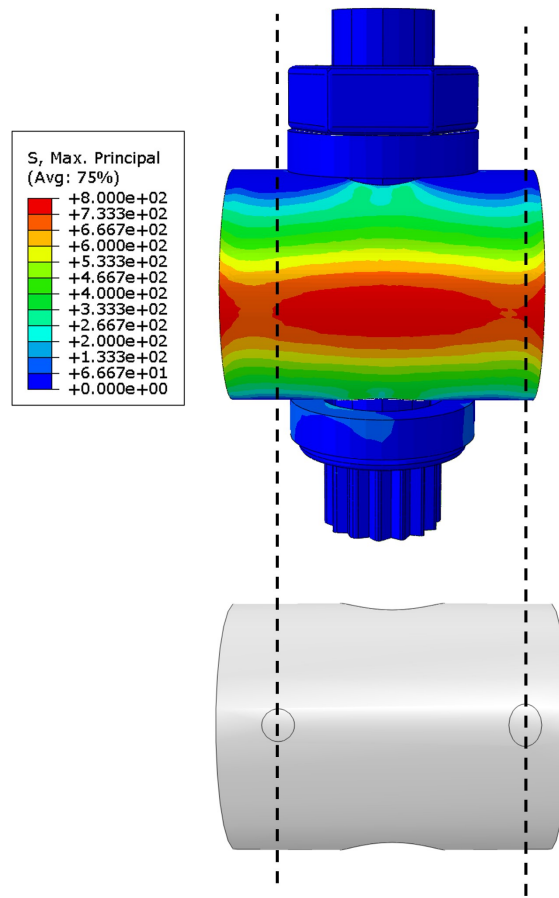


Figure 5.26: Predicted stress distribution for the CMSX-10 C-ring accounting for the slight shift in crystal orientation and its relation to the droplet sites

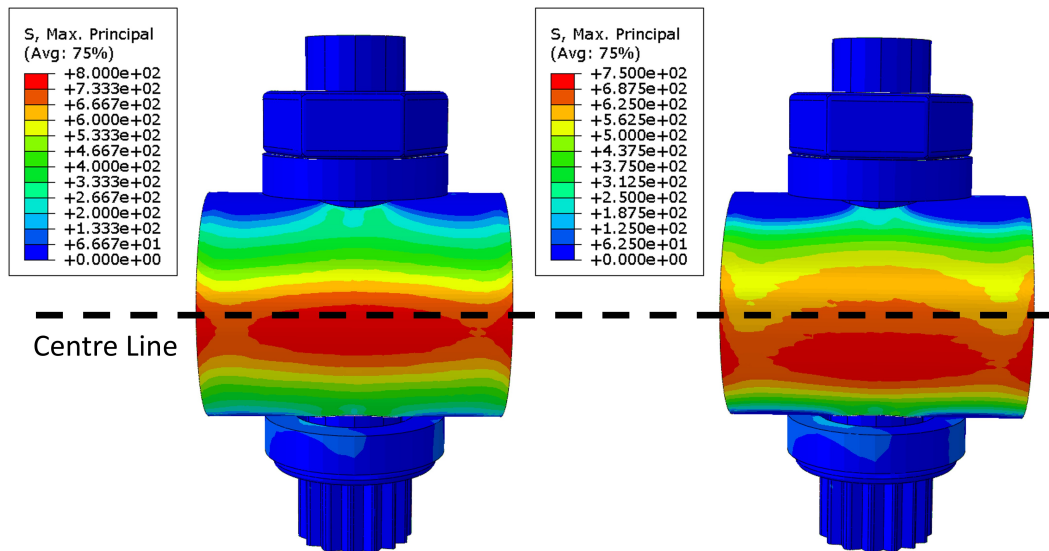


Figure 5.27: Predicted stress distribution for the CMSX-10 (left) and CMSX-4 (right) C-rings used for these 'Dot' tests. The correct crystal orientation is accounted for in both cases

5.6 Crack Interaction Modelling Using XRM Data

A methodology, following the work of [229, 230], was used to digitise XRM data to create 3D CAD geometries of cracks. These solid body cracks could then be intersected with the C-ring geometry and meshed ready for FEA, essentially providing the user with data that cannot be recreated in experiments, but rather modelled. As a result, this means that data such as the J-integral assessment could be taken from the outermost ridge of the crack and could help understand how and why these values might change as the user moves through the peaks and troughs of the outer ridge. The benefit of the J-integral is that the elastic component can be related back to the SIF; thus understanding where the driving force, or conversely lack thereof, is along the crack. The development of the algorithm itself is of no interest to this thesis; however, its novelty lies in its use. The key details and development of the workflow can be found in the following thesis' [229, 230].

A C-ring test from previous work at Cranfield [25] was initially used in the algorithm, and FEA was performed to test for potential crack interactions. This C-ring was covered with a layer of NaCl solution on its surface before exposure to an air + SO₂-containing furnace at 550°C for 400 hr. The XRM data is presented in Figure 5.28a, from which three cracks were initially selected, digitalised, and intersected in the same position from which they were observed in the experimental C-ring. The newly formed CAD geometry (i.e. cracked component) was meshed, and a production plasticity-based model, provided by Rolls-Royce in the form of a User Material subroutine (UMAT), was run to test for potential interactions. Boundary conditions were applied to the C-ring geometry to replicate the displacement (0.44 mm) used in the experiments, and were loaded until it reached the maximum displacement at a time step of 1 s. In this case, only the stress distribution was considered; hence, no impurities or chemical properties were considered. The results are presented in Figure 5.28b and show that for this formation, both forms of crack interactions were present. The regions surrounding cracks #1 and #2 exhibited a stress shielding

effect, as highlighted by the blue-contoured area ahead of the crack. Conversely, cracks #2 and #3 exhibited a significant stress intensification effect, which could potentially result in a crack merging phenomenon and is depicted by the red contour regions. From this, cracks were added to determine how the global bulk stresses may vary, and the three cracks became 25.

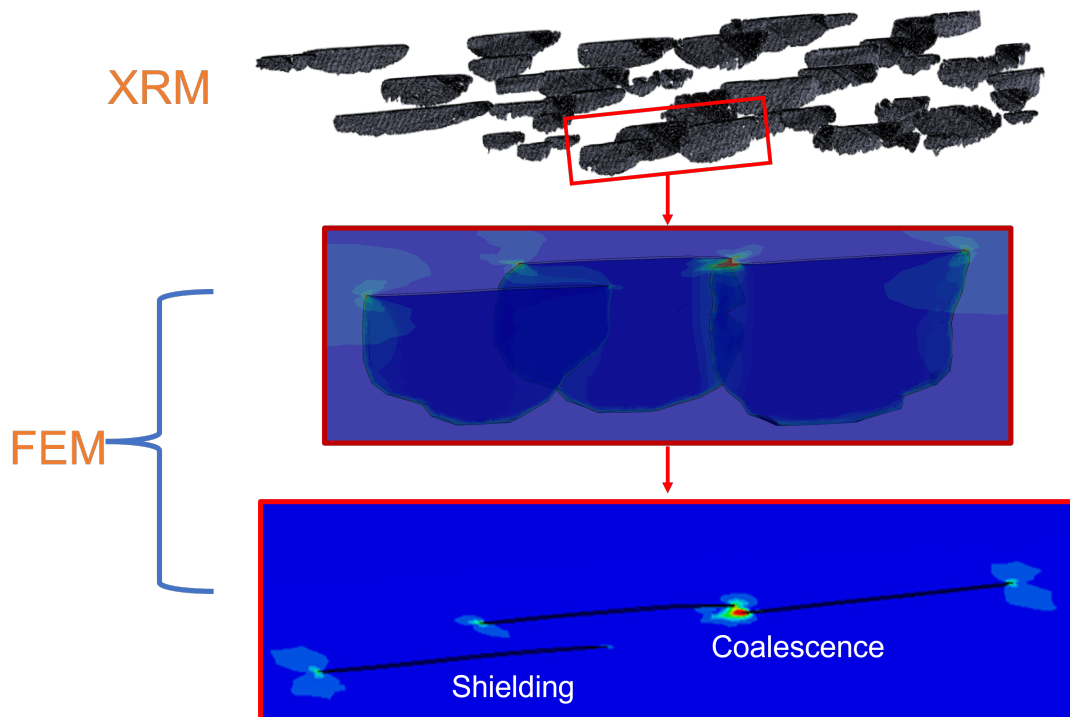


Figure 5.28: Data from the 400 hr C-ring represented in both segmented XRM data of all cracks for the section, and three of these cracks being digitalised and intersected with the C-ring for FEA testing

Two independent models were run using 25 of the total cracks to further validate both the mechanisms and use of this algorithm. The first run was the Rolls-Royce plasticity UMAT to produce a stress map that highlighted potential areas of stress intensification and shielding. This was followed by a PFM to predict the crack extension for the given crack configuration. These are shown in Figures 5.29a and b, respectively. The plasticity model predicted a significant stress shielding effect for small cracks, which can be accounted for if their positions with respect to the longest cracks are considered, as they

were consistently separated by a critical distance roughly matching those predicted in Section 4.3.1. The model also calculated the areas of significant stress intensification between off-parallel cracks, again indicating the areas of potential crack coalescence mechanisms. This hypothesis was further supported by the independently run PFM in Figure 5.29b, which anticipated that these areas of stress intensification saw a crack coalescence effect, as shown by the red contoured regions between the long and off-parallel cracks. Both models were run independently but yielded a very similar response, meaning that if the C-ring was placed back in the furnace with the given crack formation, it is highly probable that crack merging would be observed.

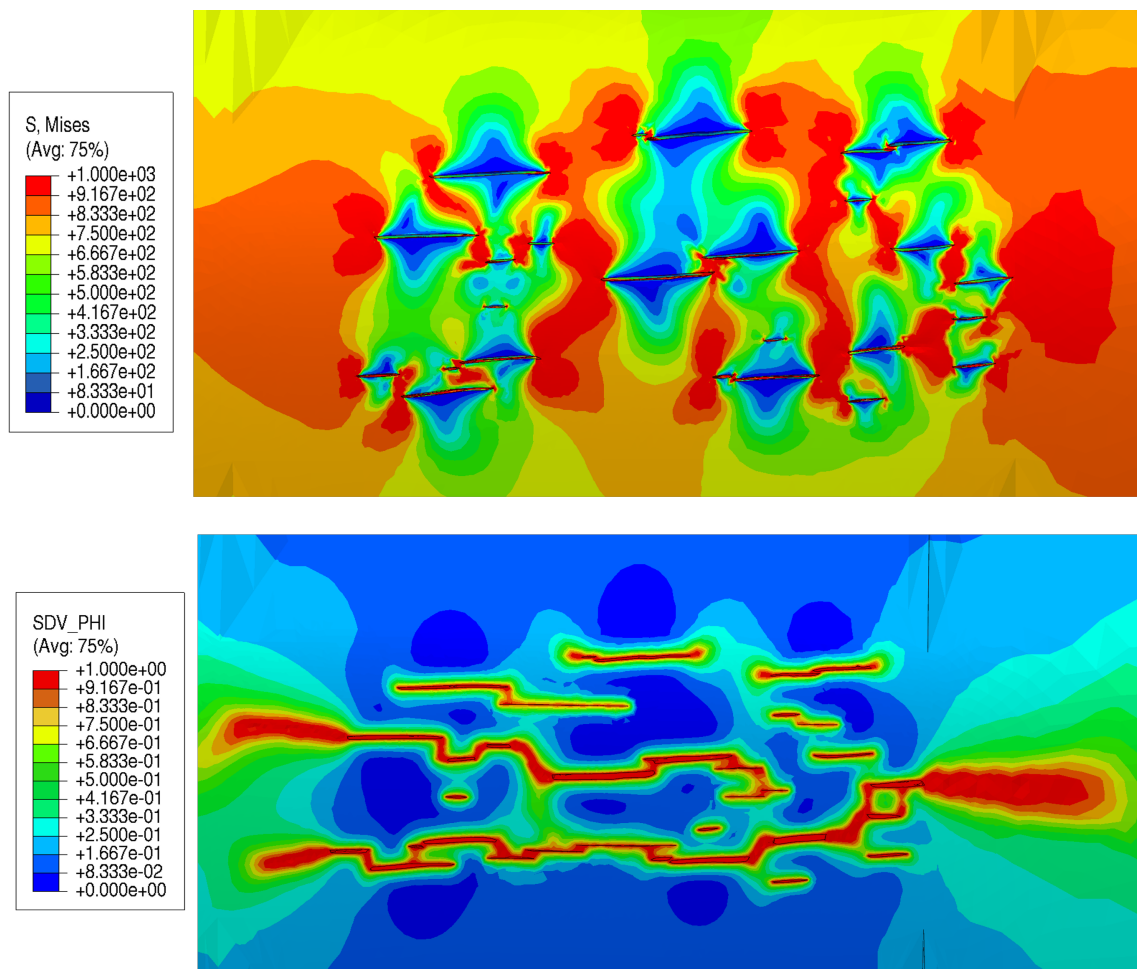


Figure 5.29: Digitised XRM data from the 400 hr C-ring represented using two independently ran models a) a plasticity-based UMAT to determine areas of stress shielding and intensification and b) a PFM to predict crack extension under these conditions

The idea that after a certain threshold of surface cracks is reached, it can globally be

considered a singular crack must be considered based on these results. Figure 5.30 shows a side view of Figure 5.29b and the 25 cracks used in the simulation. The model predicts that these cracks extend into areas of the undamaged material as a singular crack. This result highlights the importance of crack interactions in SCC tests.

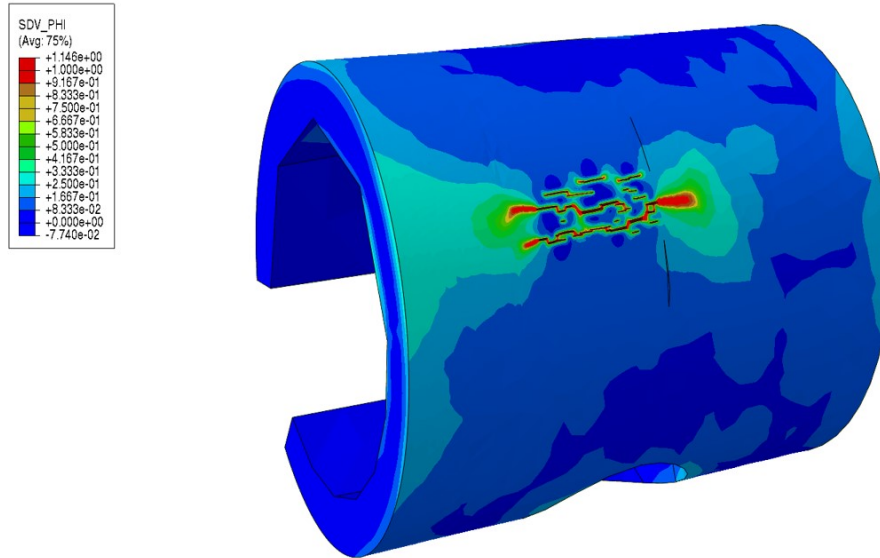


Figure 5.30: Digitised XRM data from the 400 hr C-ring represented using a PFM to predict crack extension under these conditions. This is a side view from the previous figure to appreciate how this given density of cracks could globally be considered as a singular crack

5.7 Discussion

The results in Ref. [223] showed that the presence of NaCl in the SO_x -containing environment is crucial for initiating cracks at intermediate temperatures. Surface chemistry causes the initiation of embryo materialisation under the halos of salt particles. This chapter was able to further develop this knowledge by suggesting that the density and pattern of these salt depositions play a role in determining the detrimental effects of these cracks on material life. Both the modelling and experimental data show that a singular salt deposition has the potential to be far more catastrophic, and without any interaction from surrounding cracks, it could grow as far as the dispersion of the salt boundary allows it. In addition to explaining failure mechanisms, simulations have been instrumental in the

development of novel experiments. By design, both the C-ring and fatigue tests reduced the likelihood of crack interactions, resulting in significantly longer cracks. These tests limited crack interaction and promoted crack growth by depositing salt in a manner that allowed sufficient space for cracks to grow. Compared to traditional C-ring tests, both novel specimens resulted in cracks 3-8X longer, demonstrating that the initiation sites will continue to grow when left alone. This hypothesis was further validated by running a CMSX-4 fatigue specimen which was exposed to the same environmental conditions (i.e. salt composition and deposition method and an air + SO₂-containing environment), but at a higher temperature and under cyclic loading conditions. Minimising crack interaction mechanisms caused the fatigue component to fail up to 83% quicker, further proving the influence of crack interactions on the extent of total crack expansion. In contrast, running the same test with a CMSX-10 fatigue sample had little effect on the number of cycles required to reach failure and, in fact, followed a crack growth rate similar to that of the four other fatigue tests using the same material and standard salt deposition.

A potential explanation to describe the difference in CGR between the two alloys could be due to the irregular structure causing a stress-relief effect, which may impede crack propagation, and forcing to grow along a different path. This may result in needing more energy to further fracture the material, which could explain why cracks struggle to propagate despite CMSX-10 being an easier material to initiate. By contrast, regular precipitates encourage higher and more consistent stresses in the γ channels, and potentially create a stress gradient in the channel that is sufficiently high enough to pull cracks through low-stress regions. This is related to the extent of crack interactions seen in both materials, as CMSX-10 seems to have a significantly lower driving force for SCC within its substructure, and fewer cracks were observed to propagate, such that little crack interaction was observed. This means that it is not expected to experience much of an effect when the singular cracks are isolated. In contrast, the model (with the limited assumptions made and does not account for many other complex effects that maybe occurring such as

environmental resistance) predicted CMSX-4 to have a higher and more consistent stress level in the γ channel, where an isolated crack has a greater driving force to encourage crack propagation. This is related to the coherency between the two phases, otherwise known as lattice misfit, and how variations in this can lead to different responses in the bulk material. If manufacturers produce materials with high coherency between the two phases, small lattice misfit values are observed, resulting in γ' precipitates that are more spherical in nature, whereas higher misfit values result in more cuboidal precipitates. It is worth noting that these assumptions and hypothesis have been made based on the modelling results at the time but further physical testing needs conducting before any definitive assumptions can be made. Despite this irregular microstructure being more suitable for minimising crack extension in CMSX-10, it is less resistant to creep, meaning that a compromise would need to be made in certain regions of the engine if this microstructure is to be deployed. This highlights the need to integrate models and experiments at all material scales to understand the responses of components to different environments.

It is believed that under these SCC conditions, many closely packed initiation sites are less critical for aero and industrial GT blades than for individual cracks, which have the potential to grow unbound. This hypothesis could have a significant impact in the industrial environment, as it can be used to improve the capability of predictive methods and allow engineers to understand which patterns of salt deposition could cause catastrophic cracking. In the long term, this could reduce the maintenance costs or even be used to design materials that can better withstand crack initiation and propagation.

The nature of these proposed experiments, which were specifically designed in comparison to the previously run C-ring experiments, proved beneficial and helped validate both the PFM and the understanding of crack propagation experiments. Crack interaction has been heavily emphasised in previous chapters, and these results were used to rethink how the experiments were set up. However, modelling can be used for more than just

that and can be implemented to reduce the epistemic uncertainty of experiments. For example, before running a 'Dot' test a simple simulation can be used, accounting for the correct crystal orientation, to determine the value and positioning of peak stresses, informing where the salt depositions should be placed. Simulations can also be used in such a way that they build an overall test matrix (which is made up of both physical and computational methods), whereby a proportion of the experimental results are determined by modelling, narrowing down the number of physical tests, and reducing the overall test costs. This is further discussed in the next section.

5.8 Final Remarks

This chapter highlights how powerful the combination of experiments, microscopy, and modelling can extend the boundary of knowledge and the need to take the best of each to gain a mechanistic understanding. This chapter concludes the following:

1. Salt contamination pattern played a critical role in the life expectancy of materials, as highlighted by both the 'Zebra' and 'Dot' experiments, which in the case of CMSX-4, is regardless of the loading conditions and experimental temperature.
2. The extent to which a material experiences crack interaction mechanisms varies significantly even between different single-crystal nickel superalloys, and it is hypothesised that this could be due to variations in microstructure. Even a slight misorientation of γ' precipitates can induce a reduction in peak stress at the precipitate interface, which may reduce the driving force for CGR, and provide a potential explanation for why CMSX-10 propagates significantly slower. It is crucial to model such responses when designing new materials to understand how they may affect a given material's response to cracking to maximise a component's life.
3. All specific experiments also highlighted the effect of several length scales and the role they play alongside deposition patterns. These length scales include, but are not

limited to, the area of salt boundary dispersion and the spacing between deposition sites.

Chapter 6

Corrosion - Fatigue: Can Preliminary Modelling be used for Blind Testing?

6.1 Introduction

Fatigue degradation can cause catastrophic failure of components at loads significantly lower than their yield; thus, it is crucial that the role of cyclic loading, both in combination with and without corrosive species, on crack propagation is considered. Arguably, one of the most difficult aspects to consider is how these processes interact at the crack tip and the effect on the path and rate at which cracks grow, especially when multiple temperature ranges are considered. As such, understanding which mechanism will dominate and when, based on factors such as frequency, or if certain mechanisms are cycle/time dependent, would provide useful insights.

This chapter aims to first assess the PFM prediction of da/dN and da/dt for varying frequencies in an oxygen-containing environment and how it compares to the established data in the literature. Once calibrated, the model can be used to predict (accounting for a given uncertainty) similar responses but for different species, and even different materials of the same family (i.e. single-crystal nickel-based superalloys). This chapter postulates that the dominant mechanisms are cycle- and time-dependent, in which either corrosion or fatigue mechanisms dominate at the crack tip. The purpose of this is not to provide accurate predictions for the CGR, but rather to show how preliminary modelling results

can be used to assess initial correlations and results before deciding on which experiments actually need running. This would reduce the overall costs and highlights the importance of synergy between the techniques.

Following the formulation, boundary conditions, and model parameters laid out in Section 3.1, a PFM model that accounts for both stress-corrosion and fatigue degradation was used to predict the crack propagation. An investigation into both CMSX-4 and CMSX-10 was conducted, assuming environmental exposure to sulphur and oxygen-containing environments at 550°C. This study does not consider crack initiation (due to the complexity of crack initiation often being difficult to model using FEA) and assumes that an established thumbnail shaped crack ($300 \times 150 \mu\text{m}$ as shown in Figure 6.1). Further investigation into other crack geometries would be useful to understand if it has any effect on the end results, but is it not covered in this thesis due to time restriction.

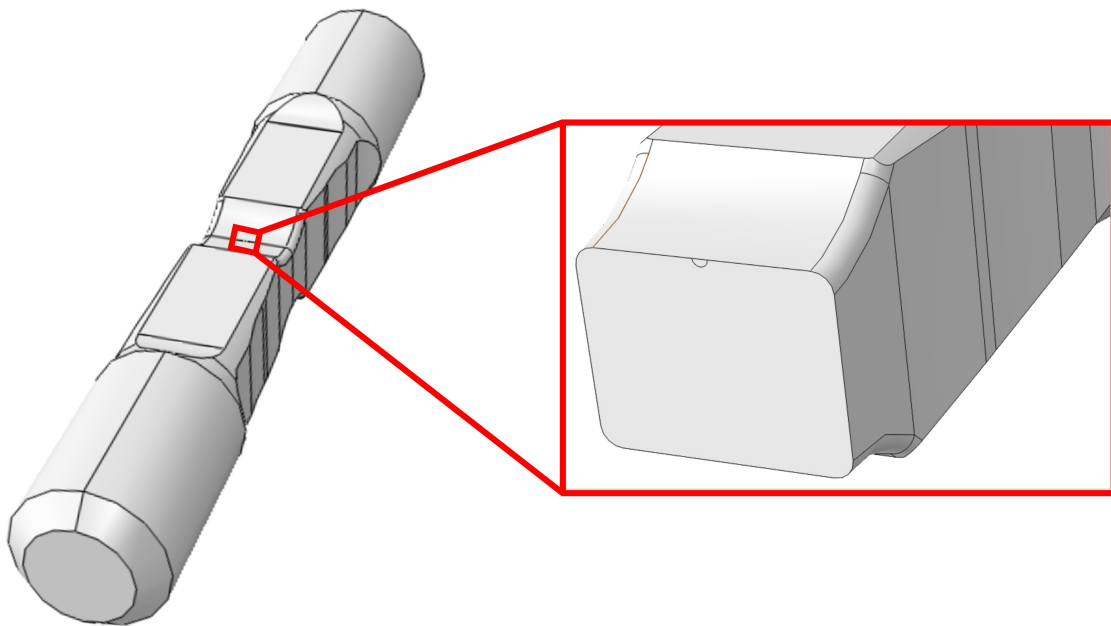


Figure 6.1: A $300 \times 150 \mu\text{m}$ predefined crack used in all fatigue simulations in this thesis

6.2 Effect of SIF on da/dN in Fatigue

To assess the ability of the model to capture the fatigue mechanisms, simulations were run across a number of initial SIF values. These were calculated using Equation 2.3, where a crack depth of 150 μm was assumed, and the stress at the centre of the notch was calculated using FEA (accounting for crystal orientation effects). Simulations were run using frequencies of 0.142 and 5 Hz, which correspond to the 1-1-1-1s and 0.05-0.05-0.05-0.05s trapezoidal waveforms, respectively. These simulations assumed only fatigue (based on the formulations and assumptions set in Section 3.2.2), and as such, the stress-corrosion flag variable was switched to zero, signifying the removal of stress-corrosion degradation from the PF formulation. All simulations were run for 100 cycles using the modelling parameters listed in Table 6.1, including the assumed fatigue threshold parameter based on Equation 3.17. Based on this, da/dt is calculated for each initial SIF value, as shown in Figure 6.2.

Table 6.1: Values of the elastic constants used for all CMSX-4 simulations

Parameter	Value
Young's Modulus (GPa)	110.460
Poisson's Ratio (ND)	0.3858
Shear Modulus (GPa)	114.600
$G_c(0)$	40
Temperature ($^{\circ}\text{C}/\text{K}$)	540/813
Fatigue Threshold (GPa)	0.25

Two key results are shown in Figure 6.2; firstly, the model recovered a slope with a shape similar to that of the textbook Paris law curves, providing some confidence in the formulation used. It was expected that the lower SIF values of 3 and 5 $\text{MPa}\sqrt{m}$ would

show a steeper drop; however, this could be because the fatigue threshold was too high. Regardless, it is believed that increasing this value would still capture the same overall shape (as the physics of the model is not changing, just the threshold value), and future work (e.g., further sensitivity analyses which tests a number of different threshold values) can be conducted to better replicate these lower SIF values more accurately. Secondly, both frequencies recovered identical results, which further supports the hypothesis that these higher frequencies (or lower cycle times) are dominated by fatigue mechanisms at the crack tip. It should be noted that no amendments were made to the model to capture both of these results and this highlights how, once calibrated, such physics-based FEA models can provide possible explanations that cannot always be gathered experimentally.

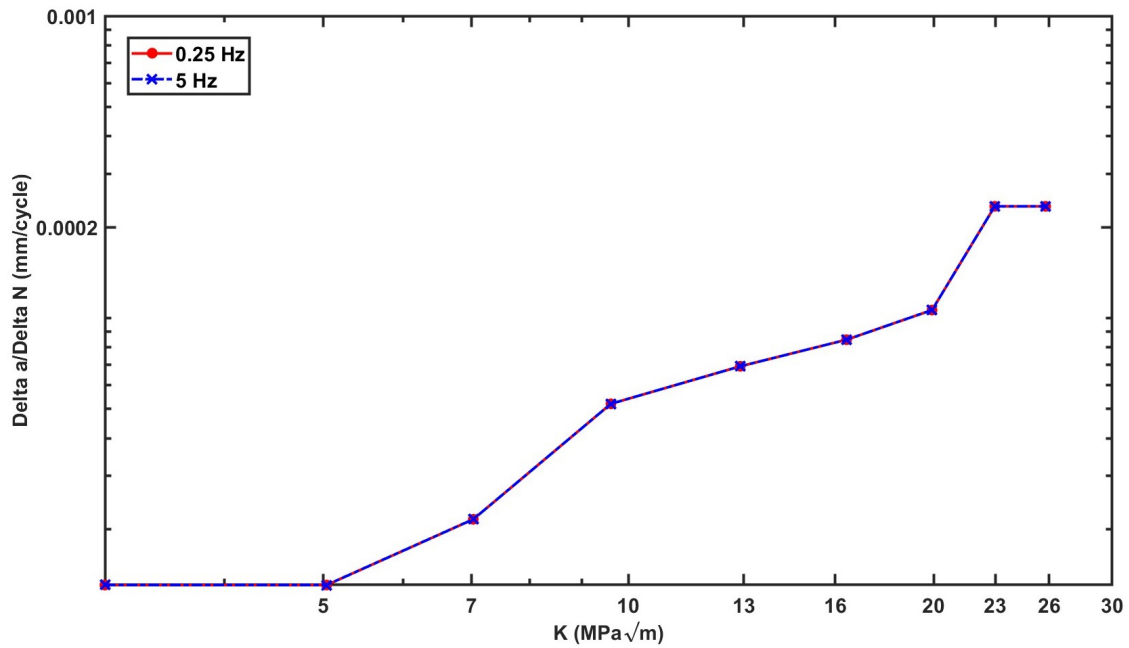


Figure 6.2: CGR data per cycle for different initial SIF values for frequencies of 0.25 and 5 Hz

6.3 Effects of Frequency and Dwell in an O₂ Containing Environment

The next step was to run the model and match it against some experimental data in the literature, where experimental data from the University of Southampton [78] seemed the most appropriate. This data looked at the same material and temperature regime, CMSX-4 (material parameters mentioned in Table 6.1), at 550°C, making the work relevant to this study. It investigated oxidation-fatigue damage mechanisms under a constant ΔK (of 20 MPa $\sqrt{m}$), and also suggested a time- and cycle-dependent nature for the tests, as shown by the red data points in Figure 6.3. PFM was used to perform two models. The first used an initial SIF value of 20 MPa $\sqrt{m}$, and the second used 16 MPa $\sqrt{m}$ for 50 and 100 cycles, respectively. The latter simulations showed little propagation across 50 cycles, so the decision was made to extend to 100 cycles. The same predefined crack mentioned above was used for both simulations, and several frequencies were considered. As in the C-ring tests, the predefined crack is associated with concentration using the temperature degrees of freedom. Owing to the abundance of oxygen in the atmosphere, especially in comparison with sulphur, a higher initial concentration of 5 wppm was assumed. Table 6.2 lists the frequencies and dwell times that were used.

Table 6.2: Frequencies and respective loading waveforms used for these simulations

Frequency (Hz)	Loading Waveform (s)
5	0.05-0.05-0.05-0.05
1	0.25-0.25-0.25-0.25
0.25	1-1-1-1
0.036	1-25-1-1
0.008	1-120-1-1

Figure 6.3 highlights the initial results from the $20 \text{ MPa}\sqrt{m}$ simulation and is compared to the experimental data [78], which clearly predicts the same trend. Both independent results resulted in a similar outcome, where there is a plateau in da/dN for higher frequencies and a similar plateau in da/dt for lower frequencies. This indicates the potential competition between the mechanisms at the crack tip. These results indicate a cycle and time-dependent nature, whereby when there is sufficient dwell, it allows for greater diffusion of degrading species and, as such, a chemical degradation mechanism dominates. In contrast, for more rapid cycle fatigue, mechanical degradation dominates the extension.

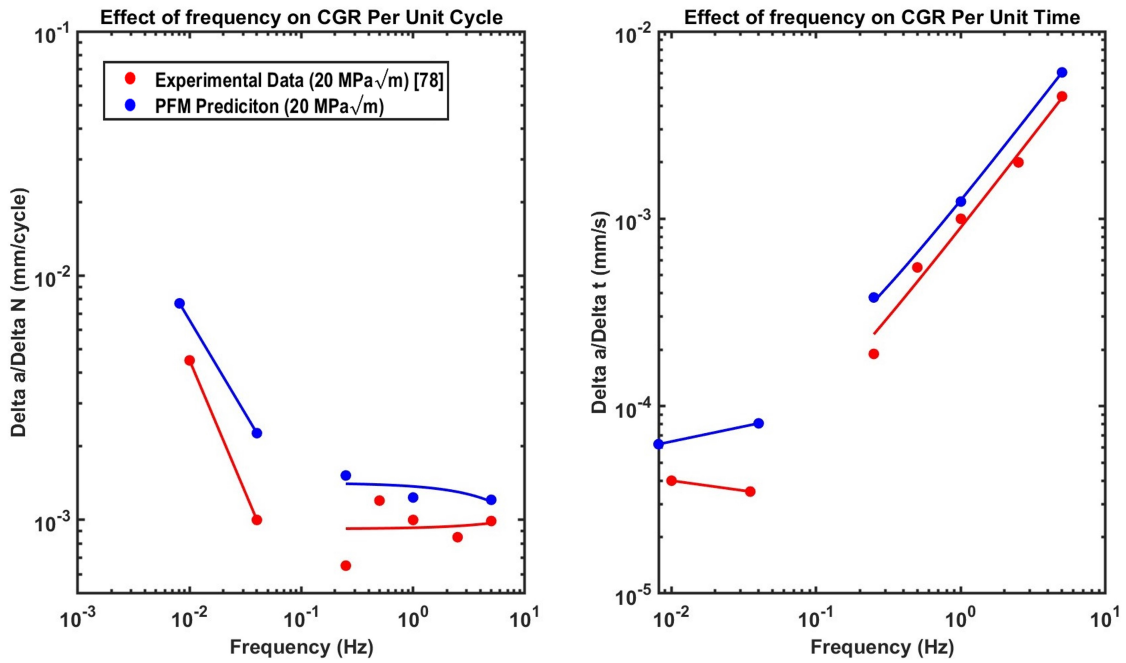


Figure 6.3: Initial results comparing data from Evangelou et al. [78] (in red) with the PFM approximation for an initial SIF of $20 \text{ MPa}\sqrt{m}$ (blue)

However, it is important to note that the oxidation-fatigue experiment was run using a constant ΔK , which is not impossible to perform for the simulation but is definitely time-consuming. The simulations would need stopping after every step to readjust the applied load to maintain a constant ΔK , and with some simulations taking over a week to run, it was not feasible. Instead, the load was reduced to apply an initial SIF of $16 \text{ MPa}\sqrt{m}$, and the results are shown in Figure 6.4 using the lower bound of the blue data points, with 20

$\text{MPa}\sqrt{m}$ being the upper bound. The red data points (experimental data) are now situated between the two sets of PF predictions, essentially creating this candlestick plot, where the upper and lower bounds can be counted for any uncertainty. This candle-stick plot is assumed to account for both modelling and experimental uncertainty. These results show the predictive ability of the PFM and how it can be used to reproduce the experimental data, taking into account the degree of uncertainty quantified by the upper and lower limits.

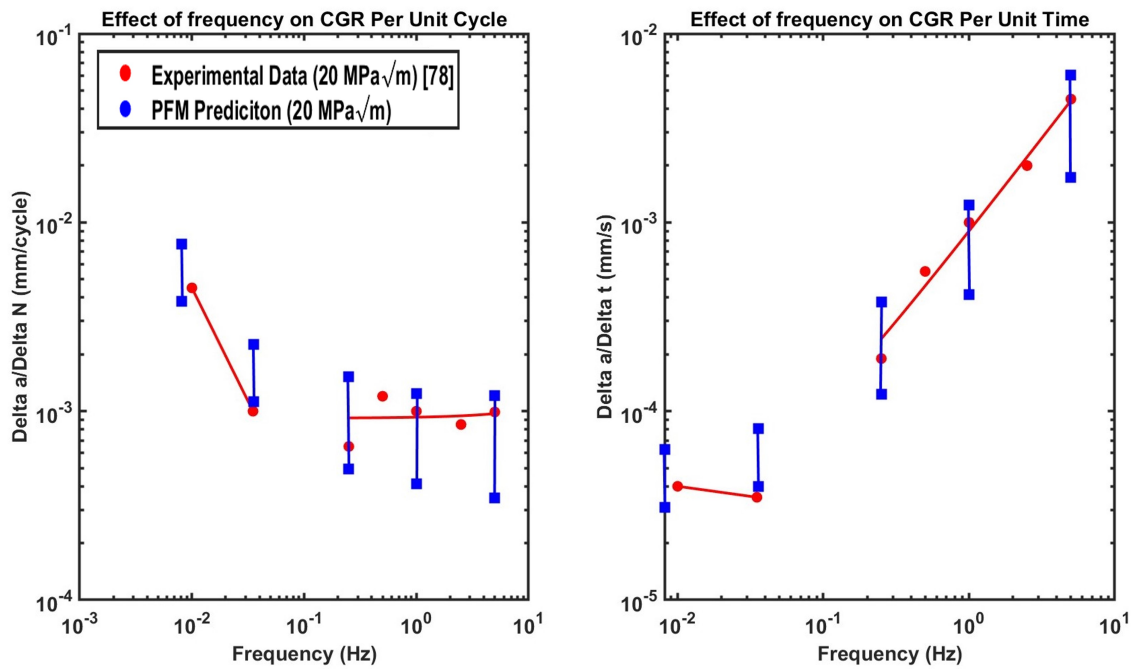


Figure 6.4: Results comparing data from Evangelou et al. [78] (red) with the PFM approximation for initial SIF of 16 and 20 $\text{MPa}\sqrt{m}$

Both bounds show the same sharp increase as longer dwell times are introduced, and indicate a potential for oxygen to play a detrimental role on CGR (at this temperature), and how longer dwell times may allow for a greater diffusion of species ahead of the crack tip.

6.4 Predicting the Response of Another Material

Now that the initial model has been tested against experimental data, it builds confidence in the model's ability to analyse different materials (of the same family) under the same conditions. A model was run, again accounting for the same predefined crack and loading conditions, but now for CMSX-10 (for which the material parameters are found in Table 3.2). All assumed testing conditions and modelling parameters were kept the same as those mentioned above, with the elastic constants being the only variable to change (as a variation in elastic constants may see a variation in peak stress levels). The results are highlighted in Figure 6.5, and as expected, respond in a similar fashion but with a smaller degree of uncertainty between the upper and lower bounds, particularly for the higher frequency regimes.

It should be noted that experimental data are required for CMSX-10 in order to gain a more reliable understanding. However, the value of this blind prediction comes from providing a free approximation to gauge a material response before performing any physical testing. The results show that the lower the frequency, the closer the prediction between the two materials, and for the lowest frequency (1-120-1-1s loading waveform), the da/dN and da/dt results are almost identical. This could mean that the stress-corrosion mechanisms dominate in a similar fashion across both materials, but the fatigue response is different, with CMSX-10 being less sensitive to SIF at higher frequencies. This might not be observed in physical testing, and to reiterate, experimental data would need to be obtained to validate this. However, using preliminary modelling results has potentially provided a way to characterise the difference between the two materials by providing insights that may have been missed in the experiments.

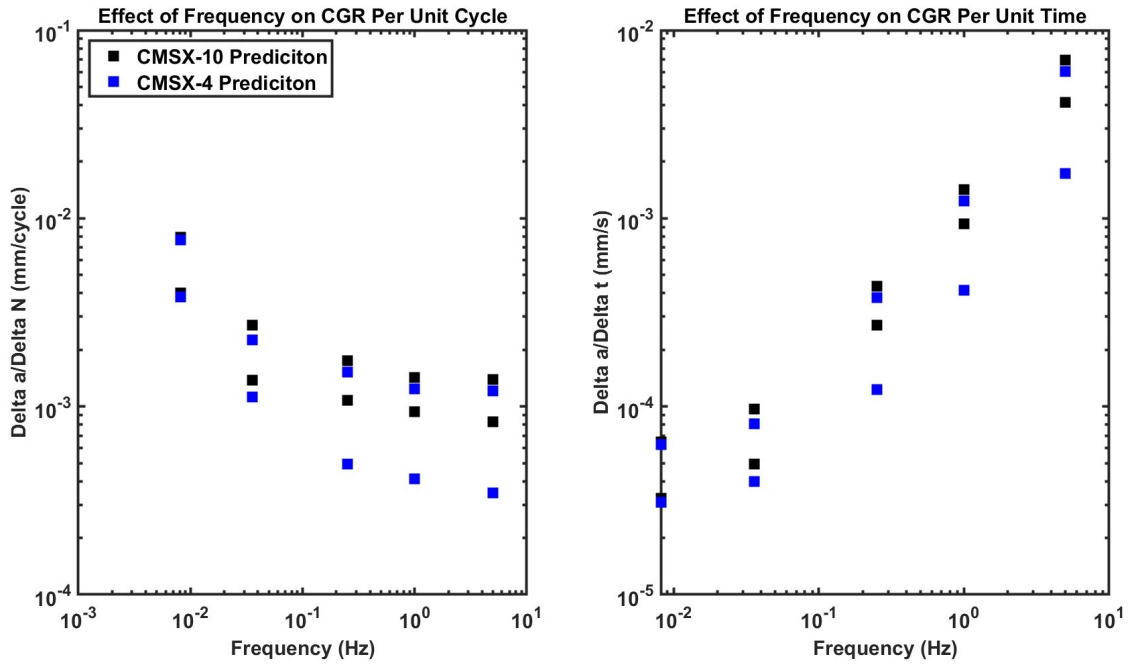


Figure 6.5: A blind prediction for CMSX-10 (black) response in an oxygen containing environment and its comparison against calibrated CMSX-4 (blue) data

6.5 Effects of Frequency and Dwell in an S Containing Environment

By changing the degrading species from oxygen to sulphur, the model can gauge the response of CMSX-4 to an environment that is more aligned with corrosion-fatigue. The model now accounts for the diffusion coefficient matching that of sulphur in nickel. Following the calibration from Chapter 4 (and the corresponding parameters used in Tables 3.2 and 3.3), CMSX-4 was modelled following the same frequencies as mentioned above, but with initial SIF values of 13 and 16 $\text{MPa}\sqrt{m}$ across 100 and 50 cycles, respectively.

The results are highlighted in Figure 6.6 which shows two key results. As expected, the initial SIF values do affect the CGR in a sulfur-containing environment, with 16 $\text{MPa}\sqrt{m}$ predicting higher da/dN and da/dt trends than 13 $\text{MPa}\sqrt{m}$. The predictions also showed the same plateau in da/dN for higher frequencies, followed by a sharp increase at

lower frequencies. This further indicates that longer dwell times provided sufficient time for the diffusion of sulphur ahead of the crack tip, and a stress-corrosion driven mechanism dominated. On the contrary, it is clear that the same plateau in da/dt for decreasing frequency is not observed, and it is postulated that given that the diffusion coefficient for sulphur is now significantly slower than that of oxygen in nickel, longer dwell times would be needed to see this more consistent da/dt response. Further assessments are required.

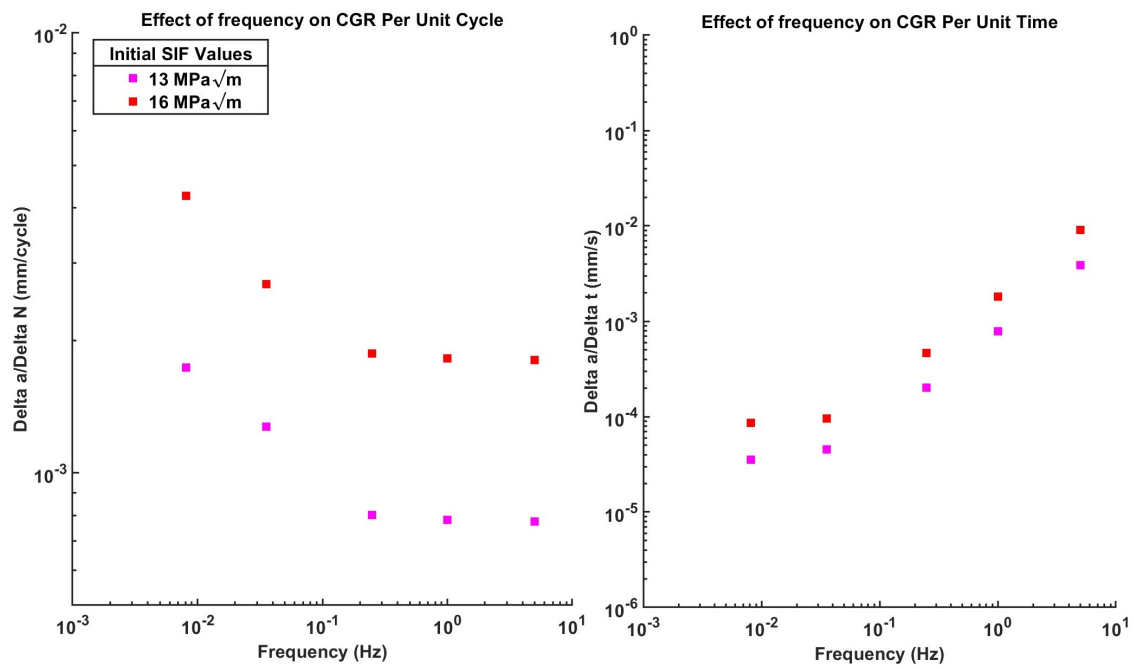


Figure 6.6: PFM prediction for CGR in CMSX-4 when exposed to a sulphur containing environment with initial SIF values of 13 and 16 MPa $\sqrt{m}$

6.6 Discussion

This chapter highlights a PFM that can account for both stress-corrosion and fatigue degradation mechanisms, which are independently calibrated. The initial sulphur degradation calibration for C-ring testing, outlined in previous chapters, was carried forward and extended to account for oxygen, using experimental data for an oxidation-fatigue environment. Once calibrated, this chapter presents a method for which blind predictions can be made for different environmental compositions and similar materials, prior to physical testing.

This chapter shows the predictive power of the PFM for fatigue-based degradation mechanisms by showing that when da/dN is plotted for different initial SIF, it can recover a slope similar to that of the textbook Paris Law for a purely fatigue environment. It is worth reiterating that the lower initial SIF values do need to be repeated, as a steeper drop in this range is expected, and this could be fixed by simply increasing the fatigue threshold. Further work needs to be done to understand why the model behaved this way in the lowest SIF regions. The size of the initial predefined crack can also be increased or decreased to alter the initial SIF value. Finally, plasticity needs to be introduced to account for initial SIF values higher than those used; at that point, the boundary conditions would enforce a peak stress greater than that of the material yield. The model was then tested against experimental data for an oxidation-fatigue environment and showed that when two initial SIF values were considered, they aligned either side of the experimental data, acting as an upper and lower bound. The upper and lower bounds used in Figure 6.4 correspond to an error bar that accounts for both modelling and experimental errors. The percentage difference between these upper and lower bounds was of interest (shown in Figure 6.7), as it showed that a general increase in frequency resulted in an increase in the percentage difference between the two bounds. This signifies the sensitivity of stress in stress-corrosion cracking. This is a postulation and further testing, both physically and

computationally, is required to validate this hypothesis.

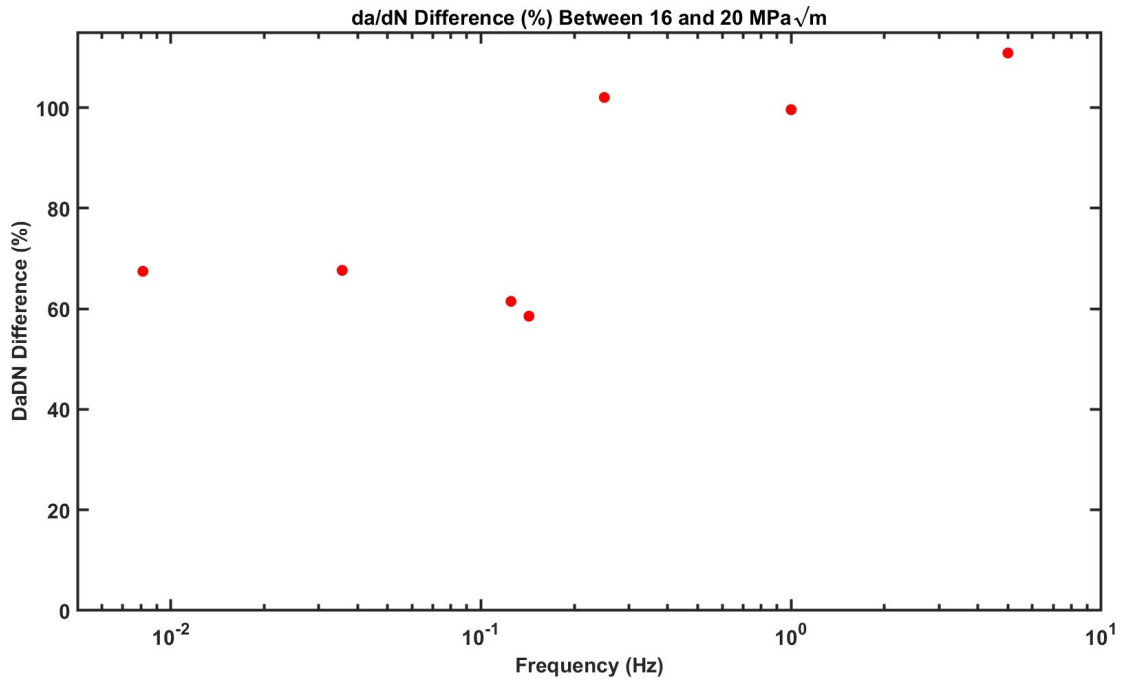


Figure 6.7: The percentage difference between the da/dN values for the initial SIF values of 16 and 20 $\text{MPa}\sqrt{m}$ for varying frequency values

Further blind testing was performed for CMSX-10; however, it should be noted that these results need further validation, but are a good example of how preliminary data can inform decision-making. The purpose of these simulations was to predict the response of CMSX-10, whether it would insinuate the same mechanisms postulated to occur in CMSX-4, and whether it might highlight any relationship between the two materials. The model predicted a lower variation between the two bounds for high frequency, and showed that the results were almost identical for the longest dwell times. This highlights how preliminary modelling can provide valuable insights before physical testing. It would be useful to run a 120s dwell test for both CMSX-4 and CMSX-10 to assess whether they show similar CGR per unit time and cycle, as a way to validate the model prediction.

Based on all the modelling data and its comparison with the available experimental data, it is clear that a potential competition between the dominating mechanisms at the

crack tip occurs. It is postulated that the deciding factor for the dominant mechanism is controlled by the test frequency (or equally the dwell time). This is such that stress-corrosion mechanisms are enhanced when longer dwell times occur, as they provide sufficient time for the diffusion of embrittling species ahead of the crack tip. However, fatigue dominated when more rapid cycles occurred. The essence of preliminary modelling would be to suggest correlations such as this before testing is done, to think of the best possible experimental solution. An example of how this thinking can be used to test for this time- and cycle-dependent nature would be to conduct several very high and very low frequency ranges and inspect the fracture surfaces, which can indicate whether the propagation is dominated by chemically or mechanically driven mechanisms. Using this methodology, assessing the effect of hydrogen under these conditions is the next most obvious case to investigate, and using preliminary modelling to dictate that round of testing would prove useful. It is also important that any future modelling work, that any crack path effects are also assessed, especially for crack growth in single crystals and at these lower temperature ranges.

Using preliminary modelling results to inform how and when physical testing is conducted, potentially by developing a test matrix, results in a more environmentally and financially sustainable method of testing. The more variables that need testing, the more time- and energy-consuming, a phase of testing may be, leading to increased costs. For example, aspects such as dwell time have a significant effect on life of corrosion fatigue samples, and as such, many dwells need testing to paint an accurate picture. Modelling may be useful to run simulations between these high- and low-frequency regimes and to reduce the number of physical tests performed. Modelling essentially allows the user to play God and adjust variables in the model, where the only cost is computational, in order to understand different conditions before physical testing. Creating a test matrix based on initial modelling is a PhD in itself, and as such far more work needs doing, but this chapter at least sets the path for this kind of thinking.

6.7 Final Remarks

In summary, this chapter emphasises how computational techniques can be used to gain an initial understanding of material responses and even to find potential correlations and relationships between data that may not have been previously seen. The following conclusions were drawn.

1. The PFM results agree well with experimental data [78], highlighting a clear competition between mechanisms occurring at the crack tips, whereby either stress-corrosion- or fatigue-driven mechanisms dominate.
2. By having both stress-corrosion and fatigue mechanisms simultaneously enabled in the model, then the dominating mechanism is seen to be controlled by the dwell times and frequency. Corrosion-driven crack propagation is time-dependent, and when sufficient time is given, the corrosion species can diffuse ahead of the crack tip and lead to material degradation. In contrast, fatigue degradation is cycle-dependent and dominates when insufficient time is given for diffusion.
3. Once calibrated, preliminary modelling results can be used to understand a material's response to different environments, before physical testing is conducted. It also allows relationships and mechanisms to be hypothesised, which could affect how the experiments are run.
4. Finally, this chapter briefly identifies the place for using preliminary results to produce a test matrix, to better inform how and when physical testing is done. This results in both sustainability and financial advantages.

Chapter 7

Discussion and Summary

The synergy between modelling and experiments has shown that the interaction of several key length scales plays a detrimental role in determining whether cracks continue to grow under a steady state, rapidly accelerate, or halt. PFM was used to capture this phenomenon and proved useful, independent of the corrosion mechanism.

The modelling results highlighted that both the positioning, and separation distances, are critical for predicting propagation, and that 150 μm or less between parallel cracks is required to initiate crack shielding. Under these conditions, a longer crack dominates and drastically reduces the local crack-tip stresses of neighbouring cracks, and reduces the driving force for SCC. These results align with those found in the literature, where studies assessed the role of plasticity [231] in such assumptions, as well as investigated shielding effects on SIF values [175, 232]. This mechanism is extremely relevant to SCC observed in aero GTs, where a large density of crack embryos are seen to initiate. It is postulated that these embryos could interact with, and prematurely shield, cracks, and prevent catastrophic cracking, meaning that a high density of small cracks could be less detrimental to components than a singular crack left to grow unbound. However, both modelling and experiments have shown the opposite, whereby off-parallel cracks separated by 10 μm or less, experienced stress intensification in the areas surrounding the respective crack tips, leading to the coalescence of several shorter cracks to a newly formed long crack.

Furthermore, experimental data [25] have shown that both the presence of NaCl and

SO_x are necessary to initiate cracks at these temperatures, and embryos form as quickly as 10 min. Surface chemistry reactions show that crack embryos initiate under the halo structure of salt particles, naturally leading to the idea that the initial distribution and density of salt depositions control where cracks initiate and therefore crack spacing. As such, further assessments with both modelling and experiments have shown that a singular salt deposition is far more detrimental to the life of a component than a cluster of depositions. This could have a significant impact on the industry, as it allows engineers to account for such mechanisms in engineering-level predictive tools, and understand which deposition patterns and distributions are more likely to result in catastrophic failure. Salt deposition patterns and placement is not something that can be controlled in service, but by understanding the two extreme ends (i.e., densely and sparsely populated salt depositions), it allows engineers to understand the upper and lower safety bounds. In the long term, it has the potential to reduce the overall maintenance, and improve the design of new materials. The nature of these specifically designed C-ring and corrosion-fatigue experiments proved extremely beneficial and useful in validating the PFM. It also provided a fresh perspective on experimental uncertainty and encouraged an interpolated way of thinking outside of traditional means.

These ‘Dot’ corrosion-fatigue tests highlighted that two different single-crystal nickel-based superalloys resulted in different responses, and PFM was used to provide a hypothesis to explain the difference. Modelling results showed that an irregular microstructure (which is representative of CMSX-4) presents a reduction in the peak stresses by way of γ' impeding the crack propagation path. These simulations demonstrated the potential synergy between models and experiments, and highlights how simple 2D simulations can provide useful insights and understanding. However, it may result in decreased creep resistance (which was not tested in this thesis) which is a disadvantage given the components high operational temperatures. Thus, the same technique can be used to design new alloys that can potentially provide resistance to both. This can be, and has been, achieved

by varying γ' shape; size; and orientation; adjusting the volume fraction; adjusting the mechanical properties of the precipitates and channels; etc.. A potential solution to account for both creep and crack propagation could be to alter the γ' shape and size of the first few layers of precipitates, which would reduce (but not completely eliminate) the chances for crack propagation to pass through the first few layers, but also maintain the material creep resistance. This process has the potential to decrease the time required to design new alloys as well as reduce the overall costs. A potential method of physically testing the CGR differences between regular and irregular microstructures could include running a fatigue test without a corrosion environment. If a purely mechanical test setup can be produced and all aspects of corrosion are removed, it could show whether the difference in the CGR is driven by mechanical/microstructural effects or not.

The corrosion-fatigue model was run using two initial values for SIF, which acted as upper and lower bounds for comparison with the experimental oxidation-fatigue data, which was operated using a constant ΔK . Both the experimental [78] and modelling results showed a plateau in da/dN for high frequencies and da/dt for low frequencies. This indicates a potential competition for the dominant mechanism at the crack tip. It is postulated that for low frequencies (or long dwells), there is sufficient time for the diffusion of potentially embrittling species ahead of the crack tip, where corrosion degradation dominates and is driven by local crack tip stresses. At high frequencies (or short dwells), the rapid accumulation of fatigue degradation occurs at the crack tip, which dominates. This made it possible to perform two blind tests, where the material was changed to CMSX-10, whilst the second assessed sulphur degradation in CMSX-4 at 550°C. The aim of this study is to highlight how preliminary modelling can be performed to make assumptions and further develop a testing matrix.

The results in this thesis demonstrate that modelling plays a more important role than just matching experimental data, and it has provided prime examples of where predictive

modelling can actually be used to inform how experiments are run. This thesis lays the groundwork for future projects which use preliminary modelling results to not only develop new experimental techniques, but also build test matrices to instruct experimental teams which tests need running, and which can be neglected. This is certainly industry-dependent; some can take the risk of reducing their physical testing more than others. For example, an aerospace manufacturer, which is responsible for the lives of passengers, may not cut their physical testing as drastically compared to other manufacturers. However, the benefits of doing so are definitely appealing to all, especially at a time when reducing energy consumption is encouraged. There also comes a financial gain; on average a corrosion-fatigue test may cost £1500, and even higher depending on the length of the test (which varies from a few hours to a few weeks), and the more variables that are being tested, the more samples need running. For example, if a test period is set to test the dwell effect and ten different dwells are tested, a test matrix can be set up to physically test five tests, which can be used to calibrate a model which can predict the remaining five. This would result in a £7500 average decrease for one round of testing and these savings would in turn accumulate over the course of the year.

Another example of this synergy is trying to understand which species in the testing atmosphere are responsible for the degradation. The experiment provides data, such as the species present, CGR, and loading conditions, which can all be used as inputs to a computational technique that does not require FEA. Atomistic methods, such as molecular dynamics, and density functional theory (DFT), are valid options. The selected techniques could individually test how a particular species diffuses and degrades the substrate and understand how they may see differences under stress-corrosion conditions. Thermodynamic tools, such as FactSage, can also be used to provide potential reactions and useful input parameters to the PFM. Such predictions can then be used to build a test matrix to determine the species, or a combination of species, responsible for degrading the material under stress-corrosion conditions. However, more options are possible when

models, microscopy, and experiments are correctly integrated to benefit from their individual strengths.

The epistemic uncertainty involved in both the modelling and experiments used in this thesis must be addressed. For example, there is uncertainty on how much salt was applied to the ‘Zebra’ and ‘Dot’ specimens, especially where salt flux values were increased or decreased to assess the effect of the flux. However, to account for this uncertainty in the future, the specimens need weighing before and after salting to determine how much salt was actually added. Without doing so, it cannot be certain how much is actually at these deposition sites, especially when the flux values are varied. For example, in the CMSX-10 ‘Dot’ C-ring test, the salt solution was diluted until it reached approximately half the concentration, however, unless weighed before and after each deposition it is impossible to tell how much variability there is between the different sites. This could explain why the 0.5x salt flux deposition dispersed further than the 1x deposition, and further emphasises the need to make sure all modelling assumptions (such as crystal orientation) is correct.

There was also a lack of consistency with how the salt solution was dropped onto the surfaces of the specimens. It is difficult to control the size and shape of a droplet; therefore, a more consistent method is required. A potential solution could include designing a mask using CAD software and 3D printing it to fit the C-ring surface and fatigue specimen notch. These masks would have holes to account for how many deposition sites are needed, as well as their size and shape. A salt solution spraying method can be adopted to distribute salt deposits across these desired areas more evenly. However, it is also worth investigating which spray deposition method aligns more with service conditions.

As seen in Section 5.5, the positioning of the salt depositions is crucial to understanding the outcome. Evident from both ‘Dot’ C-ring tests, where all deposition sites were along the apex of the C-ring, the different crystal orientations made for an inaccurate comparison, and highlight the orientation sensitivity in single crystals. There was an almost 200 MPa difference in the peak stress along the apex, which highlighted the discrepancy

in the results. Potential solutions include running a simple simulation in advance (accounting for both the known crystal orientation and the desired bolt displacement) and allowing the results to dictate the location of the droplets. Another solution would be to adopt the ‘Zebra’ methodology, which would cover the entire stress range of the C-ring. But as a way to minimise the number of initiations and initial salt boundary dispersion, the ‘Zebra’ strip would need to be thinner than that previously used. This will again minimise the likelihood of stress shielding occurring.

There are also uncertainties with the model; for example, the boundary conditions applied to the nodes of the crack to account for the species concentration results in an infinite supply of concentration to those nodes. This is evidently not representative of the physical testing. Furthermore, the PFM used throughout this thesis does not account for plasticity, and should the model be used to predict CGR under corrosion-fatigue conditions at higher loads, plasticity would need to be introduced to alleviate any uncertainties once loading has surpassed the material yield. The reasoning for doing so is as the displacement surpasses the current values, then the peak notch stresses will then exceed the yield, and emphasises the need for plasticity solutions. There are numerous ways in which plasticity can be added, including integrating it with the plasticity model provided by Rolls-Royce, but such additions need thoroughly investigating to prevent significantly long computational times. The addition of plasticity will not change any of the underlying modelling assumptions, but will have an effect on CGR these higher stress levels. The operational temperature affects which corrosion mechanisms dominate (for example, it can dictate the transition from a Type I to a Type II HC mechanism), and the PFM does well to replicate SCC at intermediate temperature ranges. However, uncertainty may lie in predicting the crack propagation at very high or very low temperatures. As such, changes to the degradation functions within the model, may need to be changed to account for these different corrosion mechanisms.

Finally, it should be noted that the underlying physics of the model developed in this thesis can be improved to better capture the stress-corrosion and fatigue-corrosion processes; however, this is difficult to do while it is still unclear from the experiments what is occurring. Extensive characterisation of the chemical mechanisms has been (and continues to be) carried out at Cranfield, and this thesis was able to re-emphasise the importance of stress in stress-corrosion mechanism, through highlighting the role of crack interaction mechanisms or lack thereof.

Chapter 8

Conclusions

The successful integration of modelling, microscopy, and experiments has further investigated the stress-dependent nature of stress-corrosion and fatigue-corrosion mechanisms in superalloys at intermediate temperature ranges. From the analysis, the following conclusions can be made.

1. The positioning, size, and separation lengths between cracks are crucial in determining whether cracks will accelerate, halt, or grow at a steady state. In the case of C-ring cracking from the mentioned experimental work [25], it is approximated that 10-150 μm is needed to initiate a crack shielding mechanism. A vertical separation distance of 10 μm or less is needed between off-parallel cracks to see stress intensification and crack coalescence. However, these are not one case fits all values, and will be a function of crack lengths and spacing, which may change from one test to the next.
2. Preliminary modelling results can be used to rethink how experiments are approached and allow engineers to alter experimental setups to best answer hypotheses. This was evident from the iterations of specifically designed C-ring experiments, which all highlighted significantly longer cracks in CMSX-4 when salt depositions were isolated. This would not have been proposed without the input of modelling.
3. As much as an 83% reduction in the number of cycles needed to reach failure for CMSX-4 components was seen using the 'Dot' methodology. However, this was not the case for CMSX-10, which saw a minimum effect in comparison.

4. PFM provided a potential explanation for such a drastic change in final CGR and showed that a irregular microstructure resulted in a reduction in the peak stress in the γ channels, as such reducing the SCC driving force. At this point, this is merely a hypothesis and further work needs doing to validate it. However, it does highlight how simple 2D simulations maybe useful in the future.
5. The PFM was setup in such a way that both stress-corrosion and fatigue degradation mechanisms are simultaneously switched on, and allows for the competition of mechanisms to occur. Modelling results have shown that competition for the dominant mechanism at the crack tip is controlled by frequency and dwell times. Longer and shorter dwell times were more likely to be stress-corrosion and fatigue-corrosion driven mechanisms, respectively.
6. Once calibrated, multi-scale based modelling (that accounts for both physics and chemistry based mechanisms), such as PFM, can be used to predict potential mechanisms and outcomes before physical testing, meaning how and when experiments are run can be redefined. Modelling can also be used to reduce the number of experiments that need to run in a test phase, by using simulations to fill in gaps in a trend line. One way of doing so could include developing a test matrix that is built around preliminary modelling results.

Chapter 9

Suggested Future Work

This project provides good insight into the benefits of integrating models and experiments. However, there are several areas that require further improvement to increase the predictive power and efficiency of this integration. Some suggestions for future work are as follows:

1. First and foremost, the physics and chemistry of the model can be improved. They met the purpose of this project which was to have a model that accounted for both stress-corrosion and fatigue-corrosion degradation mechanisms and integrate these findings into how the experiments were conducted. However, further improvements are essential. In particular, when solving the diffusion problem, further physics can be added to account for the specific diffusion mechanisms. For example, how can it be modified to account for different cases in which vacancy or interstitial diffusion might be more applicable? Improvements can also be made to account for how the material is degraded by the diffusion of damaging species. Finally, the fatigue degradation parameter at this point is numerical, and more physical meaning is required. Time will need to be spent investigating other fatigue based crack propagation models, to see how others incorporate a threshold parameter, and determine ways which will suit the PFM best.
2. The model in its current form is solved within a User ELement (UEL) subroutine. However, a UEL requires a convoluted process to prepare an input file (with what is referred to as a dummy mesh) ready for use. A UEL also prevents the user from using many of the features within the ABAQUS user interface, and as such, a

transition to a User MATERIAL (UMAT) subroutine is suggested as future work. A UMAT will open up many of the features that ABAQUS has to offer (for example, J-integral assessment and integration with XFEM) and allow for an easier experience with the model. This also makes it more appealing for use by the industry.

3. Results have shown that the irregular microstructure is more resistant to crack propagation compared to the regular structures. However, Rolls-Royce have confirmed that these structures are far less creep resistant, meaning that a balance needs to be made to account for both mechanisms, and as such, further microstructural characterisation is needed. The results from the CMSX-4 'Dot' C-ring showed crack extension in relation to the dendritic structure, highlighting that they consistently grew parallel to the dendrites. It is postulated that this could be driven by mechanical features such as a stress gradient (caused due to the difference in mechanical properties between the two structures), but further computational assessment is required. A suggestion would be to follow the methodology of [229, 230] to digitalise the dendrite structure and conduct a crack extension prediction study using FEA, to assess if a difference in mechanical properties is enough to cause a shift in crack propagation path.
4. This thesis has made clear that crack interaction mechanisms do have a strong effect on CGR. Therefore, physics and chemistry-based FEA, such as PFM, can be used to inform predictive and lifting tools specifically for the industry. For example, an adaptation to lifting tools such that it can account for the varying CGR for one versus multiple cracks is important, as current tools may overpredict the CGR if they do not account for shielding mechanisms.
5. More specifically designed experiments, such as the 'Zebra' C-ring and 'Dot' variations, need conducting to further validate the results. Different materials outside the world of nickel-based superalloys need to be considered to understand the different material responses; for example, will a copper- or aluminum-based alloy behave

in the same way? It would be interesting to observe how different microstructures respond (e.g. poly versus single crystals). Finally, the salt flux effect must be considered. It is believed that this phenomenon of isolating a crack is not salt flux-dependent, and regardless of the level used, cracks will still be observed to be significantly longer than a high density of cracks. However, to prove this, many 'Dot' tests need to be run with varying salt flux levels so that statistical analysis can be performed. A potential way of doing so would be to run as many as ten C-rings all with different flux values, where the C-rings are weighed before and after to obtain a more accurate idea of how much salt is deposited. By doing so, a clear correlation can be made between whether increasing or decreasing salt flux has a significant effect on the CGR. Further assessment of the chemistry also needs accounting for.

6. A final suggestion would be to use the synergy between models and experiments mentioned throughout this thesis to design a test matrix for future work. This would mean using models to initially collect data and build a test matrix to determine which experiments still need to be run. The aim of this is to drastically reduce the number of experiments that need to be run using modelling knowledge. This is similar to how a machine learning model is trained with initial datasets to increase its predictive power before being deployed for prediction to reduce what physically needs to be done. The implications of this would be massive to industry and academia, as experimental costs far outweigh the computational costs of the proposed integrations.

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Appendix A

C-ring Mesh Sensitivity test

To further validate the length scale parameter study, two C-ring simulations were performed to strengthen this hypothesis. In the case of crack propagation in metals, more physical meaning can be given to l .

In both cases, all the loading conditions and material parameters (including l with a value of 0.04 mm) were kept the same; the only thing to change was the mesh. In case #1, the mesh size was eight times smaller (0.005 mm) than the length scale (just as with the simple 3D block in the original manuscript), and for case #2 a coarser mesh (0.08 mm, now twice the size of l) was used.

The plot below shows that even in this case, by fixing the parameter l , minor deviations in the mesh size had little effect on the result, and highlights that the physical meaning behind l will provide a more accurate representation when compared to a numerical justification. In some cases, numerical justification may be sufficient; however, in the case of crack propagation, it is believed that CTOA/CTOD is a great example of a more physical meaning. CTOA/CTOD is known to remain constant during steady crack propagation in metals, and as such, would make sense to relate it to the width of a smear crack for modelling purposes.

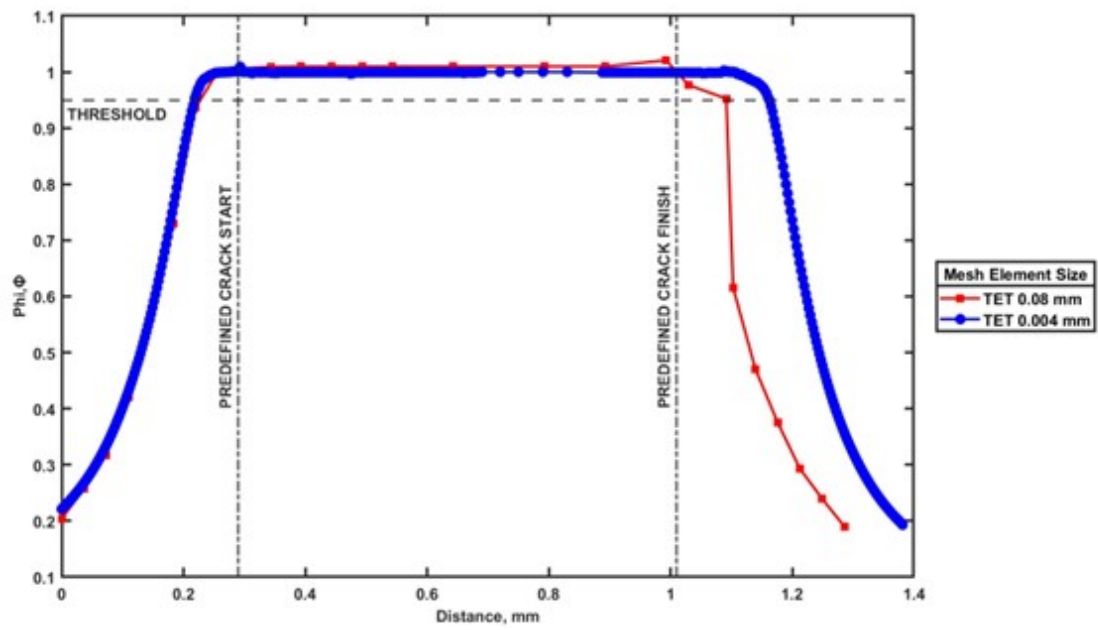


Figure A.1: Crack extension prediction for a C-ring test with varying mesh sizes for a fixed length scale parameter