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Optimizing Ni-base Superalloys for Environmental Fracture Resistance and Preventing Catastrophic Failures

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EXECUTIVE SUMMARY

High strength structural materials are used throughout the U.S. Navy in subsea applications where there is a substantial tradeoff between strength and environmental susceptibility to fracture. Ni-base superalloys are commonly chosen for having high strength while maintaining high resistance to both environmental fracture and corrosion. However, Ni-base alloys *failures have occurred in service at loads and environmental conditions less severe than their predicted immunity thresholds*, even at loads well below 130 ksi yield strength, a common ill-conceived threshold for material immunity, irrespective of material type. While failures are uncommon, they are unpredictable and very often occur with no clear root cause. Instead, failure is often ascribed to a perfect storm of conditions. The correlation between microstructure, environment factors, and environmental fracture mechanisms for Ni-base alloys is not well understood.

Advancements in environmentally assisted cracking (EAC) resistance has been obtained for precipitation hardened Ni-Cr-Mo alloys by controlling heat treatment temperatures and times, with specific heat treatment profiles developed from observations of detrimental microstructural features present in the alloy in recognized susceptible forms. Despite the advancing experimental knowledge of these alloy systems, improvements are still only possible using a path dictated by trial-and-error experiments, involving production of experimental material lots, EAC testing, and viability for scaled production. As an initial step towards developing materials by engineering their microstructure to improve their EAC behavior, we developed microstructurally representative models to identify the role of precipitates in localized deformations near an advancing crack front and identify the driving forces responsible for hydrogen transport at the fracture process zone.

We developed an algorithm for the creation of representative microstructures of Ni-base alloys to allow the de-coupling of the effects of the different phases within the microstructure and their role in localized deformation near an advancing crack. The algorithm allows control of the number of grains and their size, the dimension and distribution of the δ -phase, the dimension and number of the γ' and γ'' phases, and the orientations of all features within the microstructure.

We demonstrated that shear slip accumulation and high interfacial stresses can occur near grain boundary precipitates, particularly δ -phase, with precipitate distribution affecting their extent. We additionally observed that specific slip systems are activated, and that dislocation-density interactions and partial dislocation activity are enhanced at the interface of matrix and precipitates. By varying their size and distribution we observed increased dislocation activity for larger γ'' within metal grains and δ precipitates at the grain boundary. At the grain boundary, we hypothesized this represents the driving force for a stronger hydrogen transport path, weakening the resistance to fracture in this area of the microstructure. Conversely localization of dislocation activity at the grain interior represents a path for increased driving force in transgranular fracture. We leveraged these results to explicitly simulate the driving force for hydrogen transport towards a fracture process zone within the material microstructure ahead of an advancing crack by including dislocation transport, trapping, and diffusion driven by stress gradients within the microstructure. The results show that trapping by immobile dislocations can also strongly influence the location of the highest hydrogen concentration, moving it towards the interior of the material.

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Optimizing Ni-base Superalloys for Environmental Fracture Resistance and Preventing Catastrophic Failures

INTRODUCTION

High strength structural materials are used throughout the U.S. Navy in subsea applications where there is a substantial tradeoff between strength and environmental susceptibility to fracture. Mission requirements are necessitating a move towards higher strength materials, less redundancy in design and lower safety factors. To meet these strength requirements, Ni-base superalloys are commonly chosen for their high strength while maintaining high resistance to both environmental fracture and corrosion. The seawater exposure environment consists of either cathodic protection where the risk of hydrogen embrittlement is increased or, in the absence of corrosion protection where stress-corrosion cracking can occur.

These high strength materials are often used in single point of failure engineering designs with potentially catastrophic consequences. Ni-base alloys *failures have occurred in service at loads and environmental conditions less severe than their predicted immunity thresholds*, even at loads well below 130 ksi yield strength, a common ill-conceived threshold for material immunity, irrespective of material type. While failure occurs with low probability, failures are unpredictable and very often fail with no clear root cause. Instead, failure is often ascribed to perfect storm of conditions.

Parallels can be drawn to the Oil and Gas industry where the exposure environment is similar, and the risks of failure are comparably high. A recent study by the National Academy of Engineering investigated low-probability, high-consequence fastener failures in the O&G industry to provide recommendations for strategies for improving fastener reliability [1]. The central R&D finding of the Academy report is that risk cannot be solved by just reducing the strength of the fastener materials alone. The report goes on to call for proactive efforts to establish a clear understanding of the correlation between material chemistry, properties, and exposure environment, which is clearly lacking in the existing body of literature. The report emphasizes that the in-service outcomes provide critical information to support R&D effort. Many of these same lessons apply to material failures and use in the US Navy. The correlation between microstructure, environment factors, and environmental fracture mechanisms for Ni-base alloys is not well understood.

Technical Objective

This project combines advanced microstructural characterization tools, environmental fracture laboratory testing methods that aim to replicate in-service loading conditions, and computational modeling that accounts for the influence of hydrogen on crack propagation to demonstrate how microstructure dictates local mechanical response with the overarching goal of defining critical microstructural features in Ni-base superalloys which promote accelerated crack growth rates.

Naval and Marine Corps Needs

Understanding root causes of failure of high strength Ni-base superalloys is a critical Naval need, particularly as higher strength requirements cause increased applications of these alloys. Insight into how

microstructure and precipitation hardening contribute to environmental fracture susceptibility can then be used to optimize alloy composition and alloy processing to improve performance, and it can guide inspection and replacement criteria to reduce risk in service.

Outline

The first section investigates the effect of precipitates and precipitate volume on dislocation activity using a Dislocation-Density Crystalline Plasticity Model. The representative microstructures were derived from characterization of different grades of Inconel 718 (UNS N00718) and were used as prototypical microstructure containing secondary phases, precipitation hardening phases, and carbides present in many Ni-base superalloys.

The second section presents a study on how delta phase precipitates influence localized deformation and compares experimental environmentally assisted crack growth rates to a meso-scale microstructural model based on a dislocation-density based crystal plasticity formulation. The model predicts increased dislocation activity for larger γ'' and δ precipitates at the grain boundaries, which is likely enhanced by hydrogen transport paths through weak areas of the microstructure.

The third section presents the role of precipitation hardening on hydrogen diffusion using a meso-scale model that accounted for grain size, the grain orientation, and the major precipitates along both the grain boundaries and within the grains. The model was then subjected to the plastic strain conditions identified at the notch to simulate the metal grain aggregate response from a mechanical standpoint when a crack initiates. Then, the hydrogen concentration was modeled using a stress-assisted diffusion formulation to account for the role of hydrostatic stress as a contributor to the chemical driving force. Hydrogen trapping by immobile dislocations, simulated as a decrease in diffusivity corresponding to locations within the microstructure with the highest calculated density of dislocations, was found to strongly influence the location of the highest hydrogen concentration, moving it towards the interior of the material.

The final two sections present environmentally assisted cracking behavior of different grades of Inconel 718 and other high strength alloys measured in seawater at open circuit, cathodic protection or during alternate immersion. Crack growth rates are reported for each condition tested. The final section highlights a novel technique for environmentally assisted cracking testing of high strength structural materials. This technique was used to simultaneously test many samples for up to 15,000 hours while monitoring the growth of cracks with accuracy comparable to laboratory testing techniques.

MODELING OF UNS 07718 MICROSTRUCTURES

In this section a microstructure analysis of Ni-Cr-Mo alloy UNS N07718 is presented for a form of the material produced to achieve very high strength. Based on experimental measurement of this material behavior under HE conditions, the alloy is considered highly susceptible to environmentally assisted cracking. Its microstructure shows elongated δ -phase precipitates forming along the grain boundaries of the material. An analysis of the influence on micromechanical behavior of large δ -phase precipitates distributed along grain boundaries is presented by using a dislocation-based crystal-plasticity formulation.

Microstructure and Deformation Behavior of UNS 07718 Material

Point-, line-, and plane-defects, precipitates, twins, and grain-boundaries (GBs) all play a major role in the mechanical behavior of precipitation hardened alloys, such as 718. Point defects, such as substitutional or interstitial atoms, affect the material ductility by pinning dislocations and requiring additional energy to deform the lattice; for example, in martensitic steels carbon atoms serve as point defects, distorting the lattice and increasing the strain energy needed for shear deformations [2]. Line defects, such as dislocations, play an essential role in inelastic deformations as they directly affect the required stress for ductile deformations. Dislocation population interaction and multiplication can harden and strengthen alloys [3]. Planar defects, such as GBs, typically impede dislocation motion, affecting texture and strengthening the microstructure. Dislocation accumulation at GBs can result in twins or crack nucleation due to high angle misorientations [4]. Precipitates and inclusions are the main strengthening mechanisms in precipitation hardened (PH) alloys, and control over their density, size, and coherence giving the desired mechanical behavior. For example, pearlite laminate carbides within ferrite α steels with heat treatments below eutectic points can form colonies that provide phase interfaces inside the grain interiors. These phase interfaces reduce the dislocation-density mobility, which can harden crystalline materials [5, 6]. For nonferrous alloys, such as Ni-based superalloys precipitation hardening can be used to achieve high performance for high temperature applications.

Inconel alloys represent a broad class of super nickel crystalline alloys that are generally high strength, corrosion resistant, thermally stable, and formable. Hence, Inconel alloys, such as Inconel 718, are widely used in harsh conditions, such as exposure to high temperatures and corrosive environments. Inconel 718 (IN718) can have a microstructure of austenite γ and a combination of stable and metastable precipitates, such as γ' , γ'' , δ [7, 8], and MC or M₆C or M₂₃C₆ carbides, where 'M' is a metal such as eutectic niobium in niobium carbide NbC [9, 10]. Solid solution strengthening in the γ matrix can also benefit the overall behavior of the alloy [11]. The age hardening in IN718 is mainly achieved by the precipitation of the metastable γ'' phase that have coherent disc shaped morphology with habit plane and additional strengthening is also obtained by the precipitation of coherent γ' particles [12, 13]. The metastable γ'' Ni₃Nb phase in IN 718 is generally considered the source of its high-temperature strength.

As a transformation product of the metastable γ'' phase, the δ phase forms at austenite grain boundaries as well as coherent and incoherent twin boundaries [9, 12, 14]. The δ phase precipitates can increase the hardness of the alloy by pinning dislocations, however, as the volume fraction of the δ phase increases, Nb atoms are rapidly consumed, resulting in a decrease in the volume fraction of the γ'' phase, which adversely affects the strength. Intragranular precipitation can occur at higher temperatures, with needle and plate like morphology occurring [8, 14]. Controlled precipitation of δ phase has some beneficial effects like grain stabilization and improved stress rupture properties. Different precipitate morphologies, specifically for the δ phase, have been observed in various metallic and manufacturing processes, and these morphologies can also have a significant effect on the thermo-mechanical behavior.

A typical solution annealing (SA) and heat-treating process for 718 involves solution treatment at 954 °C (1750 °F) followed by a two-step ageing treatment near 720 °C (1328 °F) and 620 °C (11480 °F) in the first and second step, respectively, for different periods of time. Initial heat treatment temperatures produces blocky δ particles, mainly at grain boundaries, to stabilize the grains, while heat treatments at latter temperatures are given to precipitate γ' and γ'' particles in the matrix to strengthen the alloy [12].

Higher SA temperatures, above the δ phase solvus, are used in certain specifications of IN 718 to minimize or eliminate the precipitation of intergranular δ phase, shown to have detrimental properties in relation to stress corrosion cracking and hydrogen embrittlement [15-19]. Heat treatments at temperatures lower than 954 °C(1750 °F) has been employed to generate a microstructure (consisting of high volume fraction of uniformly dispersed fine δ particles) that is used during forging operations to produce fine grain grades of Alloy 718 [20, 21].

The δ phase precipitates can be sources of dislocation-density nucleation and accumulation, which can result in significant ductility [22, 23]. However, incompatibilities associated with matrix-precipitate interfaces can lead to stress and inelastic strain gradients that can lead to crack nucleation and propagation and ultimately fatigue failure at these precipitate-matrix interfaces [20]. The localized stresses at the precipitate-matrix system will interact with dislocation motion and significantly affect local and global behavior. For precipitation hardened Ni-base superalloys, strengthening is achieved by impeding the motion of dislocations by the coherent precipitates, but there is also a competition associated with shear slip or plasticity accumulation between the preferentially oriented slip systems of the precipitates and the matrix.

A detailed understanding of the microstructural mechanisms pertaining to the interactions of the precipitates and the matrix is needed to optimize high strength and ductile behavior. Additionally, the environmentally assisted cracking (EAC) behavior of PH Ni-based alloys is of great interest in the oil and gas and naval industry as more demanding applications require materials with improved resistance to sour gaseous and hydrogen environments. As EAC behavior depends strongly on microstructure, an understanding of the effects of various parts of the material microstructure is important.

In this investigation, a detailed and integrated experimental and DCP approach is used to gain a fundamental understanding of how defects, such as total and partial dislocation densities, affect local behavior at matrix-precipitates interfaces and how this is related to overall mechanical behavior. We investigate how different volume fractions of precipitates affect the behavior of a commercial (UNS N07718) specification, AMS 5962, which specifies a thermo-mechanical treatment tailored to give the highest strengths attainable in this alloy. Total and partial dislocation densities interactions and evolution can lead to the competing effects of strengthening and ductility. Hence, in combination with an experimental investigation, we utilize a recently developed dislocation-density crystalline plasticity (DCP) model that incorporates both total and partial dislocation-densities, such that we can develop a new understanding of how matrix-precipitate interfaces affects microstructural behavior.

This paper is organized as follows: the experimental methodology is discussed in Section II; the DCP model is outlined in Section III(A); the immobile dislocation-density interaction and partial dislocation-densities are briefly presented in Section III(B); the computational approach is given in Section III(C); the experimental and FE predictive results are discussed in Section IV, and a summary of the findings is given in Section V.

Table 1 – Material properties of UNS N07718 AMS 5962.

Material	Specification	Yield Strength, 0.2% offset	Tensile Strength	Elongation	Hardness
UNS N07718	AMS 5962	1,482 MPa (215 ksi)	1,560 MPa (226 ksi)	10.6%	47.8 HRC

In this work we characterize the microstructure of UNS N07718 procured according to the AMS 5962 specification to achieve a very high level of strength. This specification is used in the aircraft industry for the procurement of high strength fastener with good resistance to atmospheric exposure conditions [24]. The material was prepared by solution anneal at 1800 °F, then water quenched and cold drawn to reduce its cross section to a 2-inch round bar. The material is then aged at 1325 °F for 8 hours, it is furnace cooled to 1150 °F in two hours, then held at 1150 °F for an aging time of approximately 18 hours. The properties

of the material are shown in Table 1. For mechanical testing, the specimens were designed and tested based on ASTM E8 guidelines [25].

We used SEM microscopy to obtain images at different resolution and identify precipitate locations and distributions. At approximately 1625 times magnification, we can identify precipitates that are mostly present along grain boundaries (Figure 1(a)). At 5000 times magnification, a more detailed distribution and morphology can be obtained (Figure 1(b)). The δ phase precipitates (white) are observed with short-rod shapes at the GBs and the material shows an average grain size of $6.67\mu\text{m}$. This corresponds to an ASTM grain number of 11.5, making this a very-fine-grained microstructure.

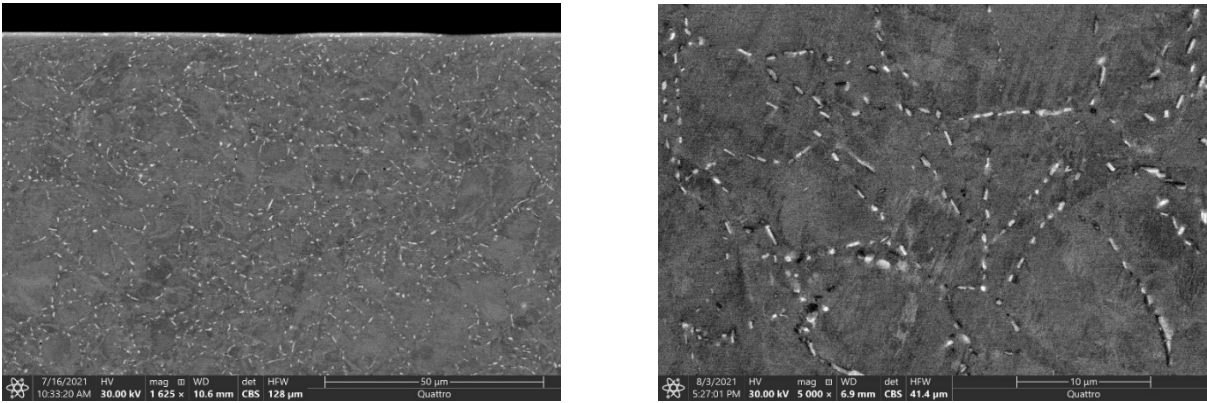


Figure 1 – SEM image of (a) the AMS 5962 specimen at 1625 times magnification and (b) at 5000 times magnification for the undeformed specimens.

We selected this material for our microstructural simulation work to better understand the role of the delta phase precipitates in a very-fine-grained microstructure with additional strength contributions coming from delta and γ'' precipitation. While the role of the acicular δ phase in EAC of PH nickel alloys is documented, the role of discrete δ phase presence on the grain boundary is not fully understood. Acicular δ was shown to be detrimental to EAC leading to a significantly lower ratio of strain to failure for 718 compared to a microstructure that did not show any acicular δ phase [26-30].

We showed that significant differences exist between AMS 5664 and API6ACRA materials in terms of EAC behavior in natural seawater, for alternate immersion OCP conditions and under CP [16, 31]. Both specifications have a heat treatment designed to minimize the precipitation of δ phase by SA above the δ solvus., the main differences between the two specifications are in the HT: a single vs double step following SA for API and 5664 respectively. The presence of some regularly shaped δ phase along grain boundary was shown for 718 API6A specification 120K by Thodla et al. and Obasi et al. [32, 33], despite this specification generally being the best performing in terms of EAC.

The main questions that we are focusing are: a) what are the interactions between the developed dislocation densities and δ phase during ductile deformation? b) what are the implications in terms of activated slip at critical locations such as grain boundaries and precipitate-matrix interface locations? The latter specifically is intended to give an initial indication of the mechanistic reasons behind the demonstrated lack of resistance of this high strength grade of alloy 718 to stress corrosion cracking and hydrogen embrittlement [31]. The material modeled in this work has a clear and regular distribution of δ precipitates along the grain boundaries as shown in Figure 2 b, making it an ideal candidate material for this investigation.

Dislocation-Density Crystalline Plasticity (DCP) Modeling Approach

The DCP formulation and the nonlinear FE computational framework [34, 35] are based on a rate dependent constitutive law given by

$$\dot{\gamma}^{(\alpha)} = \dot{\gamma}_{ref}^{(\alpha)} \left[\frac{\tau^{(\alpha)}}{\tau_{ref}^{(\alpha)}} \right] \left[\left[\frac{\tau^{(\alpha)}}{\tau_{ref}^{(\alpha)}} \right] \right]^{\frac{1}{m}-1}, \quad (1)$$

where the shear strain rate $\dot{\gamma}^{(\alpha)}$ is a function of the resolved stress $\tau^{(\alpha)}$ with m as the rate sensitivity parameter, $\dot{\gamma}_{ref}^{(\alpha)}$ is the reference shear slip rate, and $\tau_{ref}^{(\alpha)}$ is the reference shear stress. The reference shear stress of the α^{th} system is given by

$$\tau_{ref}^{(\alpha)} = \left(\tau_y^{(\alpha)} + G \sum_{\beta=1}^{nss} b^{(\beta)} \sqrt{a_{\alpha\beta} \rho_{im}^{(\beta)}} \right) \left(\frac{T}{T_0} \right)^{-\xi}, \quad (2)$$

where $\tau_y^{(\alpha)}$ is the static yield stress; G is the shear modulus; nss is the total number of the slip system; $b^{(\beta)}$ is the Burger's vector on the β^{th} slip system; T_0 is the reference temperature; ξ is the thermal softening exponent, and $a_{\alpha\beta}$ is the Taylor coefficient, which is based on the partial dislocation-density interaction between the slip systems.

The dislocation density evolution equations assumes that the immobile dislocation density can be decomposed into mobile and immobile part as ρ_m and ρ_{im} [35]. As the deformation evolves, the two dislocation densities are coupled as

$$\begin{aligned} \frac{d\rho_m^\alpha}{dt} &= |\dot{\gamma}^\alpha| \left(\frac{g_{source}^\alpha}{b^2} - g_{minter-\rho_m}^\alpha - \frac{g_{immob-}^\alpha}{b} \sqrt{\rho_{im}^\alpha} \right), \\ \frac{d\rho_{im}^\alpha}{dt} &= |\dot{\gamma}^\alpha| \left(g_{minter+\rho_m}^\alpha + \frac{g_{immob+}^\alpha}{b} \sqrt{\rho_{im}^\alpha} - g_{recov}^\alpha \rho_{im}^\alpha \right), \end{aligned} \quad (3)$$

where the different coefficients correspond to different mobile and immobile dislocation interactions and mechanisms as outlined in Table 2. The coefficients, which are assumed as unknown are updated at each increment, and the updating scheme is detailed in [35].

Table 2 – Coefficients for the evolution equations.

g	Expression	General Coefficient Description
g_{sour}^α	$b^\alpha \varphi \sum_\beta \sqrt{\rho_{im}^\beta}$	Dislocation-density source term
$g_{minter-}^\alpha$	$l_c f_o \sum_\beta \sqrt{a_{\alpha\beta}} \left(\frac{\rho_m^\beta}{\rho_m^\alpha b^\alpha} + \frac{\dot{\gamma}_m^\beta}{\dot{\gamma}_m^\alpha b^\beta} \right)$	Mobile and immobile dislocation density interactions
g_{immob-}^α	$\frac{l_c f_o}{\sqrt{\rho_{im}^\alpha}} \sum_\beta \sqrt{a_{\alpha\beta}} \rho_{im}^\beta$	
$g_{minter+}^\alpha$	$\frac{l_c f_o}{\dot{\gamma}^\alpha \rho_m^\alpha} \sum_{\beta,\gamma} n_\alpha^{\beta\gamma} \sqrt{a_{\beta\gamma}} \left(\frac{\rho_m^\gamma \dot{\gamma}^\beta}{b^\beta} + \frac{\rho_m^\beta \dot{\gamma}^\gamma}{b^\gamma} \right)$	
g_{immob+}^α	$\frac{l_c f_o}{\dot{\gamma}^\alpha \sqrt{\rho_{im}^\alpha}} \sum_\beta n_\alpha^{\beta\gamma} \sqrt{a_{\beta\gamma}} \rho_{im}^\gamma \dot{\gamma}^\beta$	
g_{recov}^α	$\frac{l_c f_o}{\dot{\gamma}^\alpha \sqrt{\rho_{im}^\alpha}} \left(\sum_\beta \sqrt{a_{\beta\gamma}} \frac{\dot{\gamma}^\beta}{b^\beta} \right) \exp \left(\frac{-H_0 + H_0 \sqrt{\rho_{im}^\alpha / \rho_s}}{kT} \right)$	Recovery for annihilation of dislocation-densities

Partial dislocation-densities can occur as in f.c.c. aggregates. Partial dislocation-densities, such as Shockley and Frank partials, occur due to the dissociation and the interaction of total densities. A Shockley partial dislocation has its Burgers vector in the plane of the fault, and hence it is glissile. Some dislocations, however, have their Burgers vector not in the plane of the fault which are incapable of gliding. In our approach, since we are dealing with large inelastic behavior, we focus on partial dislocation densities.

We recently developed a formulation for partial dislocation-densities incorporating within the DCP [36, 37] that are briefly discussed in this section. We define, a distribution tensor $q_{\alpha\beta} \in R^{12 \times 12}$, such that the perfect immobile dislocation densities can be allocated due to the interactions formed by the slip systems α and β . This distribution tensor was based on the paired total dislocation densities ρ_{im}^α and ρ_{im}^β and the summation of immobile dislocation density, $\sum_i^{12} \rho_{im}^i$, as

$$q_{\alpha\beta} = \frac{\rho_{im}^\alpha \otimes \rho_{im}^\beta}{\sum_i^{12} \rho_{im}^i}. \quad (4)$$

In addition to this distribution tensor, a mapping tensor $J_{\alpha\beta p} \in R^{12 \times 12 \times 21}$ assigns $q_{\alpha\beta}$ to the p^{th} of the 21 resultant partial dislocation densities as

$$\rho_p^s = q_{\alpha\beta} J_{\alpha\beta p}. \quad (5)$$

The resultant partials include 12 Shockley, six Lomer, and three Hirth partial dislocation densities. The mapping tensor contains the existence information of the p^{th} partials in each interaction with Boolean values that true is existence, and false is absence. With the tensor product in (10), the summation of $q_{\alpha\beta}$ over α and β index with the mapping tensor $J_{\alpha\beta p}$ for the p^{th} partials determined the partial dislocation-density, ρ_p^s , where the superscript s represents the sum of the partial dislocation densities.

Computational Approach

The total deformation rate tensor D_{ij} and the plastic deformation rate tensor D_{ij}^p , are needed to update the material stress state. The method used here is that developed in [34, 35] for rate-dependent crystalline plasticity formulations, and only a brief outline will be presented here. For quasi-static deformations, an implicit FE method with Broyden–Fletcher–Goldfarb–Shannon algorithm (BFGS) iteration is used to obtain the total deformation rate tensor D_{ij} . To overcome numerical instabilities associated with stiffness a hybrid explicit-implicit method is used to obtain the plastic deformation rate tensor D_{ij}^p . This hybrid numerical pattern is also used to update the evolutionary equations for the mobile and immobile densities. The hybrid approach is based on using explicit Runge-Kutta, and an implicit Euler method, when numerical stiffness is encountered. Numerical stiffness can be encountered when due to different rate changes along slip systems, slip-rates, resolved shear-stresses, and dislocation-densities can vary widely.

Results and Discussion

Based on the SEM observation of AMS5962 (Figure 1), Voronoi tessellation was used to create the grain morphology with an average grain size as 6.32 micrometers while short rod like δ precipitates were distributed at the GBs, which was consistent with the experimental observations (Figure 2(a)). Three volume fractions of δ precipitates were investigated: no precipitates, 5.12% and 9.32% precipitates. Random low angle misorientations with a maximum misorientation of 5° as denoted on the stereographic projections (Figure 2(b)).

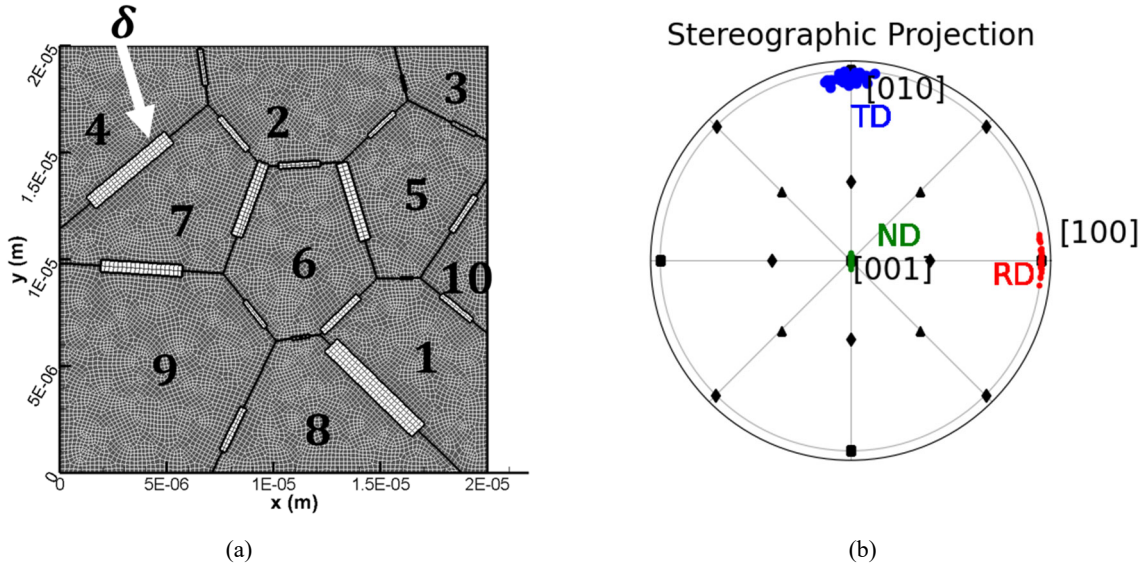


Figure 2 – (a) Grain morphology and (b) stereographic projections of random low angle misorientation that are less than 5 degrees.

A plane strain model subjected to a quasi-static tensile displacement load with a constant strain-rate of $\dot{\epsilon} = 7.5 \times 10^{-3} \text{ s}^{-1}$ was used. The convergent mesh is comprised of 12,000 elements (Figure 2(a)). The relative properties of the FCC crystal are chosen for both matrices and precipitates based on literature data for 718 [38-40]. Density functional theory (DFT) calculations by Xiong et al. [40] show a higher Young's modulus for the γ'' phase with respect to the gamma matrix and γ' phases, estimated respectively 204 GPa for the gamma matrix, 213 GPa for γ' and 243 GPa for the γ'' . There is no direct calculation that could be found in the literature that estimates the elastic properties of the δ phase, we could assume the γ'' properties hold. This could hold for relatively small size δ , however as the δ phase size increases, it tends to become only semi-coherent with the gamma matrix [37]. In our model, the change in coherency is introduced as a reduction of the “effective” properties of the precipitate, treating the δ phase as a softer

precipitate. This is convenient from a computational perspective and also implicitly represents the expected reduction in the strengthening capabilities of the δ phase.

The properties are summarized in Table 3 for both the matrix and the precipitates, where E is Young's modulus, ν is Poisson ratio, τ_y is static yield stress, m is sensitivity parameter; ρ_{mo} is the initial mobile dislocation density, ρ_{imo} is the initial immobile dislocation density; and ρ_{sat} the saturation dislocation density.

Table 3 – Material properties.

Matrix		Precipitate δ	
Properties	Values	Properties	Values
E	440 GPa	E	180 GPa
ν	0.3	ν	0.3
τ_y	420 MPa	τ_y	300 MPa
m	20	m	20
ρ_{mo}	$10^5 m^{-2}$	ρ_{mo}	$10^5 m^{-2}$
ρ_{imo}	$10^6 m^{-2}$	ρ_{imo}	$10^6 m^{-2}$
ρ_{sat}	$10^{13} m^{-2}$	ρ_{sat}	$10^{13} m^{-2}$

The stress-strain behavior for the AMS 5962 material procured in this work is shown in Figure 4, where the true stress and strains are reported. The stress-strain behavior reproduced using our modeling approach by simulating a tensile test with our grain aggregate shown in Figure 3 is also shown in Figure 3. The stresses are normalized by the static yield stress of 420 MPa. The predictions for the aggregate with the highest volume of precipitates has the lowest stress response, which can indicate that as the precipitates volume fraction increases, strength is lowered, and ductility can be enhanced. This is the expected behavior for 718 alloy, with increasing δ precipitation giving lower strength as γ'' precipitates are consumed by the growth of δ . However, the reduction of γ'' phase volume fraction is not explicitly modeled in our formulation. The results from the model indicate that the interaction of dislocation densities and precipitates is an additional factor influencing strength, albeit having a secondary role giving the relatively small change in strength. The experimental results, denoted as AMS5962, are bounded by the predicted results of the zero and 9.4% precipitates. Based on the global response, the aggregate with the 5.12% volume fraction of precipitates is chosen to further understand and physically represent the local deformations behavior within the material microstructure.

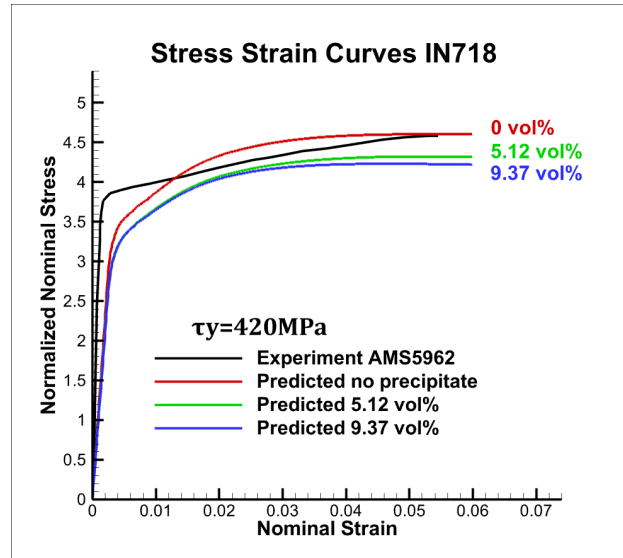


Figure 3 – Normalized global stress-strain response of experiments and modeling for the different δ -precipitate volume fractions.

The local contours of stresses normalized by the static yield of 420 MPa at 6 percent nominal strain are shown in Figure 4. The contour plots show that the matrix phase has higher strength than the δ -precipitates, as a result of the lower critical stress input used for modeling them (Table 2). More significantly, the softer precipitates result in stress accumulation areas at the precipitate-matrix interfaces. The stress accumulates to a normalized value of 8.5 at the edge of the bottom right precipitate while the surrounding grain interior have much lower normalized values of 5.5. The highest slip accumulation occurs along a grain boundary with the presence of one of the largest precipitates, as shown in Figure 4 b).

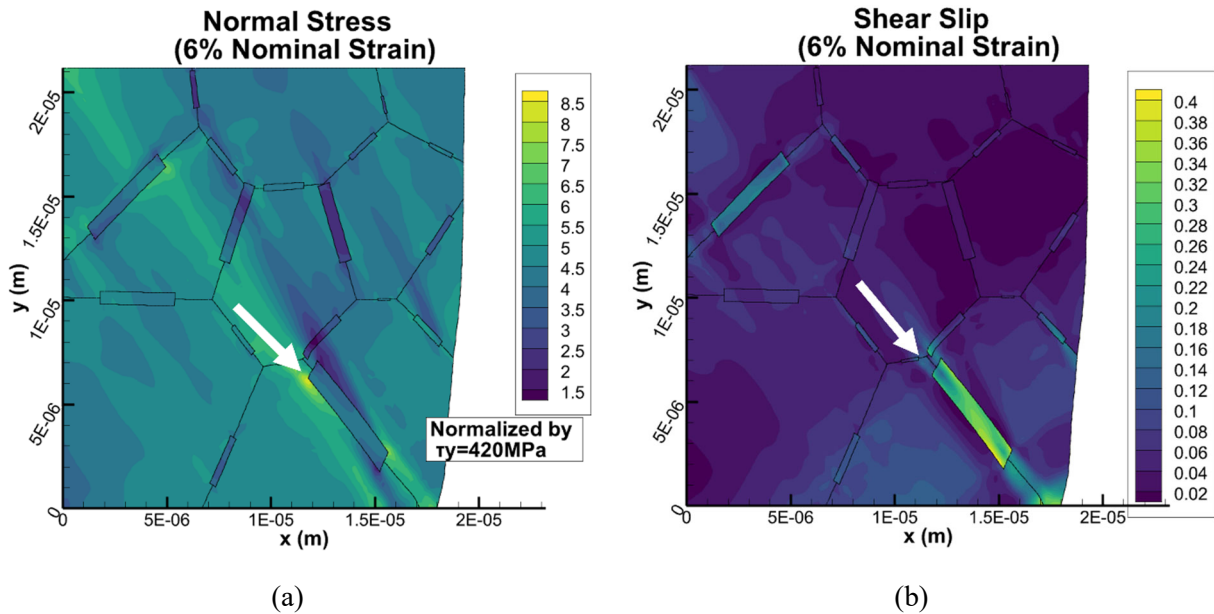


Figure 4 – Normal stresses at 6% nominal strain for the 5.12% volume fraction case

To gain further understanding of the development of total and partial dislocation-density effects at a local level, the modeling results are interrogated along a line path nearing the precipitate edges, as shown

in Figure 5(a), at the nominal strain of 6%. All dislocation-densities were normalized by the initial immobile dislocation density ($1 \times 10^6 m^{-2}$). The line plots in Figure 5(b) indicate extensive dislocation density activity adjacent to the precipitates. This implies that the softer precipitates activate slip systems, which results in total and partial dislocation density and shear slip accumulations in a neighboring area around their perimeter. In Figure 5(b), slip systems $(111)[\bar{1}\bar{1}0]$ and $(1\bar{1}\bar{1})[011]$ are activated at the precipitate edges to attain normalized immobile total dislocation-densities of 7×10^6 times (the total immobile densities are normalized by the initial immobile densities) for the $(111)[\bar{1}\bar{1}0]$ system and 5×10^6 for the $(111)[\bar{1}\bar{1}0]$, while they are generally inactive in grain interiors. These stress gradients near the softer δ phases result in slip system activity and higher reference shear stresses (Eqn. 2) due to the higher total immobile dislocation-densities. Hence, the precipitates result in higher ductility due to this slip system activity, and this is consistent with experimental observation of the higher ductility associated with δ precipitates in IN 718 [22]. Furthermore, this higher ductility is substantiated by the predicted shear strains at 6% nominal strain values, which attain values as high as 0.36.

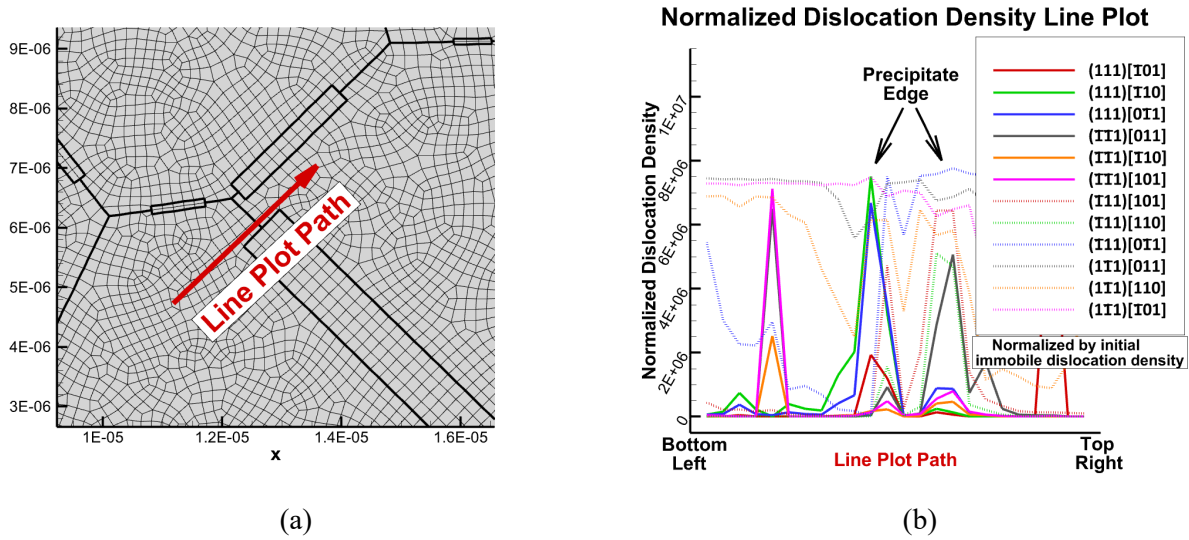


Figure 5 – (a) The line plot path nears the precipitate edge. (b) Normalized total dislocation densities (normalized by the initial immobile dislocation density, $10^6 m^{-2}$) at 6% nominal strain for the 5.12% precipitate volume fraction.

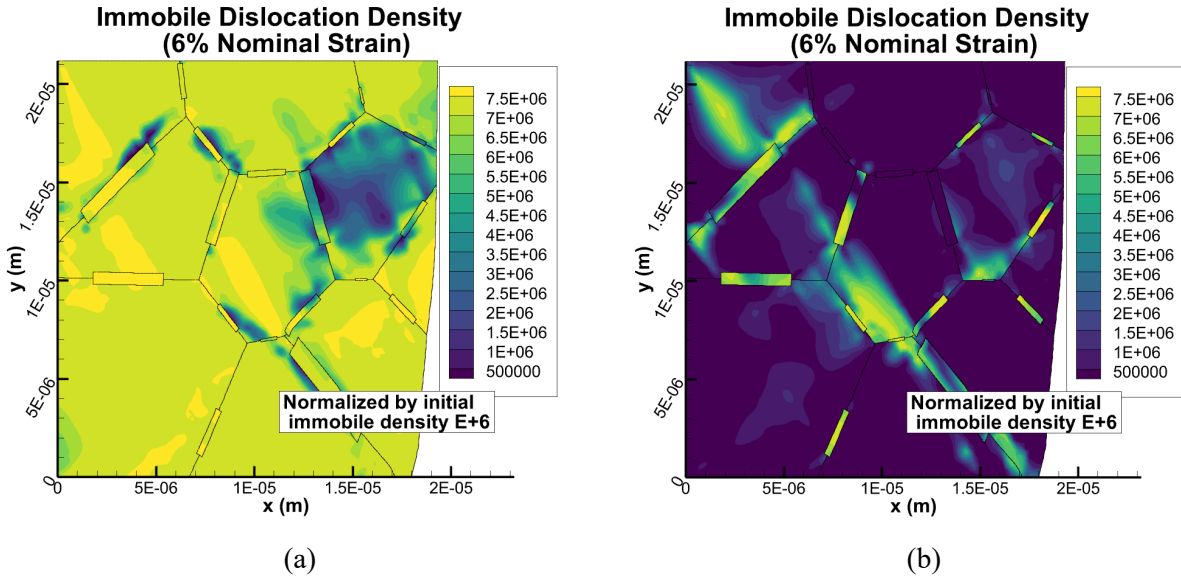


Figure 6 – Normalized immobile dislocation densities of (a) the most significant slip system $(11\bar{1})[011]$, and (b) the inactive slip system, $(111)[\bar{1}10]$ at 6% nominal strain for the 5.12% precipitate volume fraction.

Further understanding of this precipitate-matrix interaction can be obtained by analyzing the collective behavior of the immobile density evolution. The normalized immobile dislocation densities (Figure 6(a)) for slip system $(11\bar{1})[011]$ and for slip system $(111)[\bar{1}10]$ at six percent nominal strain (Figure 6(b)) are shown. The most active slip system, $(11\bar{1})[011]$, is dominant in the interior of the grain, and attains a maximum normalized immobile density value of 7.5×10^6 (Figure 6(a)). In contrast, slip system, $(111)[\bar{1}10]$, is activated only near the precipitate edges. This slip system has a normalized immobile density value of 7.5×10^6 times (Figure 6(b)), which is approximately equal to the density accumulation in the grain interiors. This indicates that along the precipitate edge, initially inactive slip systems can be activated due to higher resolved shear stresses.

The partial dislocation densities can also provide mechanistic insights for dislocation-density interactions at precipitate grain interfaces. The most active slip system, $[011](1\bar{1}1)/2$, and the slip system, $[\bar{1}10](111)/2$, interact to result in a stair-rod, $[101]/6$, and a Lomer partial, $[101]/3$, as

$$[011](1\bar{1}1)/2 + [\bar{1}10](111)/2 \rightarrow [101]/6 + [101]/3. \quad (6)$$

The partial dislocation-density were, the Lomer partials $[101]/3$, with a normalized value 6.5×10^6 times of initial immobile density, as shown in Figure 7(a), and in Figure 7(b) where a zoomed-in view of the detail highlighted in Figure 7(a) is shown. Note that the partial dislocation densities were also normalized by the initial immobile density. The Lomer partials have the highest values at the precipitate edges. Because of the activated slip-systems near the precipitate-matrix interfaces, these Lomer and stair-rod partials occur, and combined with the total immobile and mobile dislocation densities, collectively affects the shear strain and stress accumulation.

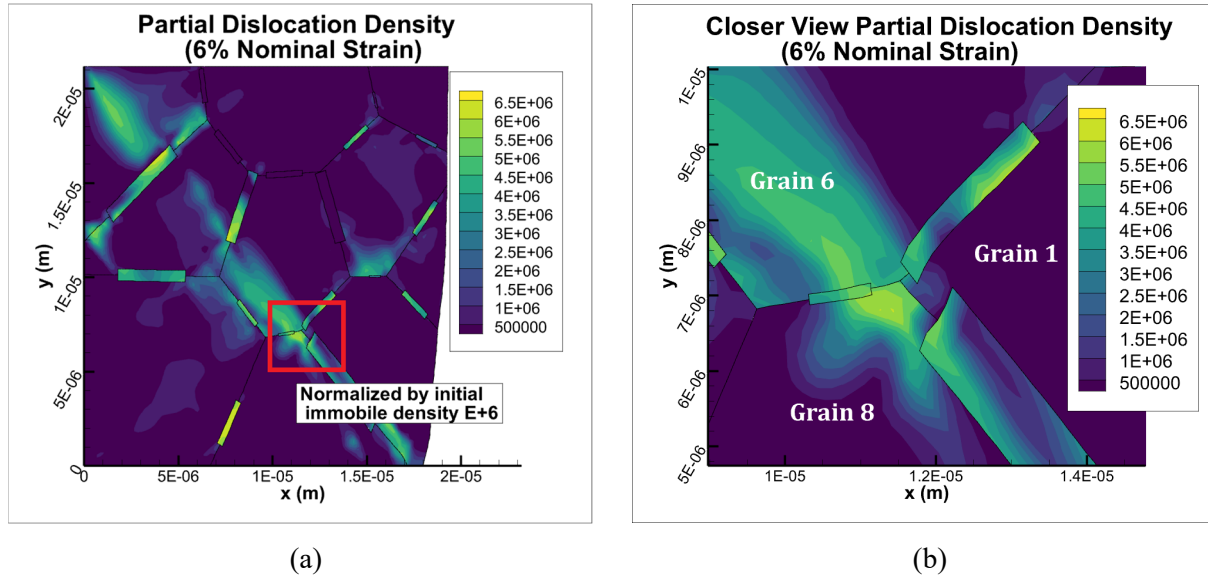


Figure 7 – (a) Normalized partial dislocation density of Lomer partial, $[101]/3$, normalized by the initial immobile dislocation density 10^6 m^{-2} with (b) a zoomed in view around the precipitate for three grains.

Our assumptions were mechanistic in nature and the results are mainly driven by the properties in Table 2, along with the deformation mechanisms modeled: crystalline plasticity to include 12 FCC systems, elastic and plastic interaction between matrix and precipitates, which are assumed to be fully coherent with the matrix. Within this set of assumptions, the mechanical interactions between gamma matrix and δ phase are a significant driver of the elastic-plastic deformations. Specifically, our analysis shows that activity at the precipitate-matrix interface mostly includes:

1. Shear localization, Figure 4 b.
2. Activation of specific slip systems and dislocation-density interactions, Figure 6 b.
3. Increased partial dislocation activity at the δ -gamma interface, Figure 7 b.

Mechanistically, shear localization is considered a strong driving force for hydrogen assisted cracking [32, 33]. Trapped hydrogen is more easily transported by dislocations and accumulated at these sites, possibly causing the progressive degradation of the grain boundary strength and leading to decohesion. Additionally, our results also imply that different slip systems are activated at the precipitate-matrix interface with respect to the main activated slip systems at the grain boundary interior. The interaction of dislocations on different slip systems near the interface of the precipitate with the matrix is another potential driving force for increased hydrogen uptake in the vicinity of the precipitates. A localized decrease in stacking fault energy (SFE), as observed for PH Ni alloys systems in the presence of hydrogen would reduce the gliding of dislocations on crossing slip planes leading to increased slip planarity and the formation of areas around the precipitates where slip intersections would act as sites for accumulation of hydrogen. Accumulation of partial dislocations near the precipitate-matrix interface in our model is another indication of this additional driving force. The modeled driving force for partial dislocation generation is not directly related to a change in SFE, however their accumulation based on the mechanical interaction of matrix and precipitates would further exacerbate the effects of lower SFE.

The three main activities that are outlined by our model point to a strong driving force for hydrogen in the presence of discrete δ phase precipitated at the grain boundary. The size and shape of γ'' are also shown to affect the EAC behavior of 718 alloys and a mechanistic understanding of the driving forces for hydrogen is still lacking. The interacting of γ'' of different size and shape and the precipitated δ phase along grain boundary was shown by Obasi to be a clear discerning factor in HE behavior.

Summary and Conclusions

An integrated experimental and DCP based modeling approach was used to investigate Inconel specimens with different precipitate volumes. The microstructural modeling was based on SEM observation of AMS5962 grain and precipitates morphologies and crystalline orientations. The AMS5962 specimen contained short rod shape δ phase precipitates that formed along the GBs due to the high GB interfacial energy. The DCP modeling indicated that the short rod shape δ -precipitates result in significant total and partial dislocation activity that leads to both ductility and increased strength with the Lomer partials as the dominant partial defect. The excess dislocation activities of the most active perfect and partial dislocation are $[\bar{1}10](111)/2$ and $[101]/3$, respectively.

The shear slip accumulation near the GB precipitates leads to an increase in local and global ductility. In addition to this ductile behavior, there were increases in strength due to the interfacial stress incompatibilities between the matrix and the precipitates. For the 5.12% precipitate volume fraction, the maximum shear strain accumulation was approximately 40% and the maximum normal stress was approximately seven times the static yield. These high values indicate both increases in strength and ductility for Inconel with different precipitate distributions are due to the different total and partial dislocation density interactions and evolving normal stresses at precipitate interfaces. Hence, based on this analysis, the proposed approach can be a roadmap for optimizing desired Inconel alloy ductile and high strength behavior. Based on our validated predictions and experimental approach, in the following sections, we will focus on IN 718 microstructures and the role of the changes in microstructure with respect to EAC by directly modeling fracture within this microstructure as determined first by the mechanical deformation behavior of the alloy and second by hydrogen diffusion.

THE ROLE OF DELTA PHASE ON LOCALIZED DEFORMATION

In this section we present experimental measurements for a version of UNS 07718 that is generally recognized to have acceptable EAC resistance. Results show that despite a tighter control in SA temperature and heat treatment the behavior of UNS N07718 AMS 5664 material shows significantly lower threshold and higher growth rates than UNS N07718 produced according to the API 6A CRA specification. We recognize the influence of the δ -phase in micromechanical behavior of UNS N07718 material and systematically study the changes in response as a function of its size and distribution. The algorithm presented in Appendix I is used to create the representative microstructures. The relative size and density of the δ -phase in the reproduced microstructure is more closely related to the two materials specifications in this work than the previous high-strength version from the previous Section. While previous work highlighted the increased activity at the precipitate-matrix interface, this work shows how the rest of the microstructure also responds to the localization of deformations.

Introduction and Motivation

Nickel-based alloy Inconel 718 is a high strength alloy extensively used in marine environments due to its combination of high general corrosion resistance, good strength, and high resistance to chloride and sulfide stress corrosion cracking [41, 42]. The most commonly used condition in marine applications, particularly in the Oil and Gas sector, is produced to meet the American Petroleum Institute (API) standard 6A 718 specification, which has a minimum yield strength of 120 ksi [21, 43]. Inconel 718 API 6A has a unique chemistry, heat treatment, and microstructure to enhance its resistance to hydrogen embrittlement (HE) and stress corrosion cracking (SCC). The API specification requires compliance with NACE MR0175 for all cases involving exposure to H₂S, which limits the maximum hardness to Rockwell Hardness (HRC) 40. Variations in materials microstructure are a function of the manufacturing process [38] and there is considerable interest in understanding the role of the microstructure in the HE performance of the material.

The annealed condition (AMS 5662) is commonly sold for general use in the aerospace and oil and gas industries, which can then be heat treated to meet various other conditions: aged per AMS 5663 to meet aerospace requirements or aged to meet API 6A and/or NACE MR0175 requirements for use in the oil and gas industry for marine applications. The AMS 5663 condition requires a minimum of 150 ksi yield strength and 180 ksi strength. Typical 718 bar for Navy use are procured per AMS 5664, or if that is not available, per AMS 5662 or 5663 by applying an additional heat treatment to effectively yield a 5664 material. The requirement is similar to AMS 5663 in terms of strength, but with a higher solution annealing temperature and longer first and second heat treatment steps.

The use of Inconel 718 (API 6A) in naval applications is increasing, particularly as a fastener material, due its resistance to SCC and HE under cathodic protection systems that maintain an applied potential equal to or more electronegative than -850 mV vs Ag/AgCl [15, 44]. This work compares the behavior of these two commonly used forms of 718, API 6A and AMS 5664, in two typical marine environments: natural seawater under alternating wet and dry conditions and under cathodic protection at -850 mV vs Ag/AgCl. Modeling of the material microstructure to study the effects of precipitated phases within the metal grains is also presented.

Materials and Experimental Methods

The AMS 5664 material was in the form of a round bar of 4.25 in of diameter and the minimum yield strength was 150 ksi. The API 6A material was in the form of a round bar of 3.75 in of diameter and with minimum yield strength of 120 ksi. The samples were both CT sample 1.25 in thickness, and 2.5 in width and loaded using a bolt, as shown in Figure 8. The samples were tested in natural unfiltered seawater environment at open circuit potential under alternate immersion conditions and under cathodic protection. The latter was achieved by connecting the samples to a low voltage aluminum anode (LVAA) also immersed in seawater as to maintain the nominal potential of -850 mV vs Ag/AgCl.

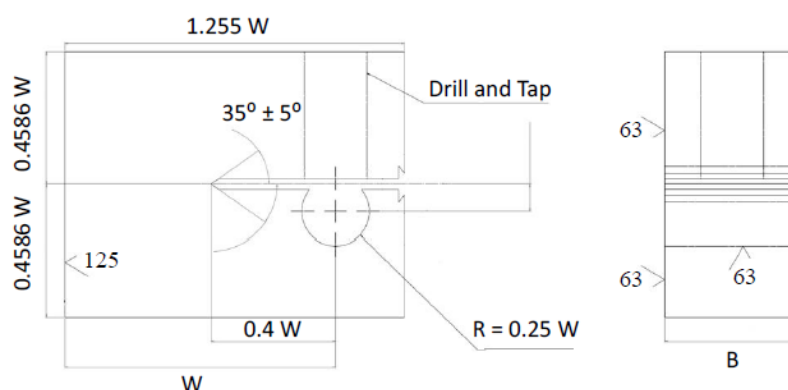


Figure 8 – Bolt loaded Compact Tension (CT) geometry.

We subjected fatigue pre-cracked samples to a constant crack mouth opening displacement (CMOD) while exposed to the environment, and monitored the progression of the crack over time by measuring the sample compliance with the use of a strain gauge attached to the back face of the sample [45]. This technique allows to study the environmental assisted cracking (EAC) behavior of the material when the driving force progressively reduces over time, with the advancement of the crack. All samples were fatigue pre-cracked according to ASTM E647 guidelines and using the load shedding method [46]. The fatigue pre-crack length was approximately $0.5 W$ for all samples. The final maximum of the Stress Intensity Factor (SIF), K_{max} , during fatigue cycling was lower than the applied stress intensity factor K at the beginning of the test per ASTM standard guidelines.

A clip gauge on the front face of the sample was used to monitor the applied bolt pre-load before exposure to the testing environment. The SIF was calculated by knowing the initial crack length and the applied CMOD. Immediately after loading, the clip gauge was removed, the sample was deployed in the test environment, and then monitored continuously using a strain measurement on the back face of the sample [45]. The strain was monitored using a quarter Wheatstone bridge configuration with completion resistors within the data recording system. The temperature of one of the samples was monitored to allow temperature correction of the strain recording. The resistance of the cables from the location of testing to the location of data recording (~30 feet) was included in the gauge factor as additional correction. Previous work showed that the resolution of this technique is $25\text{--}50\text{ }\mu\text{m/day}$. The strain gauge was protected using a polyurethane rubber compound for OCP conditions. An additional polyurethane layer (3M 5200 marine adhesive compound) was used for exposures under cathodic protection under cathodic protection.

Results

For alloy 718 AMS 5664 a total of 3 samples were tested under OCP conditions and 5 under CP conditions. Table 4 shows for each sample: the environment of testing, open circuit potential under alternate immersion, abbreviated OCP-AIm, or cathodic protection under full immersion conditions, abbreviated CP-850, the initial K , the average crack growth rate measured for the sample, the final recorded value of K , and the total test time.

The results for Sample 1, 4, 5, 6, and 7 of AMS 5664 are shown in Figure 9. All these samples showed measurable crack growth by the end of the test. The general trend for crack progression was of a very low incubation time upon exposure to the environment, and of high crack growth rates in the early phases of the test. The growth rates progressively reduced, and crack arrest occurred between 800 and 1,800 hours of testing. The samples were left exposed for over 10,000 hours in the case of OCP testing and over 5,000 hours for the tests under CP and did not show any further crack progression. Sample 4 showed a shorter incubation time and generally faster growth rates than the corresponding Sample 5, tested at nominally the same conditions. Figure 2 (b) shows a direct comparison of these two tests for the initial phases of cracking.

We do not currently know the reason for this behavior and can only assume it represents the scatter in EAC behavior of this material. Analysis of incubation times and stress intensity among all tests did not show a clear trend or correlation, suggesting that there is significant variability in the EAC behavior of this material, and a much larger test matrix is needed.

Alloy 718 API(6A) was not susceptible to EAC during under OCP alternate immersion testing conditions. The initial stress intensity for the three samples tested was near 92 ksi $\sqrt{\text{in}}$ and only Sample 1 showed some initial indication of cracking. This was recorded during the initial few hours of exposure, followed by crack arrest for the remaining test duration. An average crack growth rate is calculated for comparative purposes only. Sample 2 and 3 did not show any significant crack progression. The data are summarized in Table 5. The data under OCP alternate immersion condition are compared to literature results for CP.

Table 4 – Test conditions for nickel alloy 718 AMS 5664 and average measured crack growth rates. The two exposure environments are OCP-A_{Im}, Open Circuit Potential – Alternate Immersion, and CP, Cathodic Protection with reference electrode potential of -850 mV vs Ag/AgCl.

	Environment	Initial K (ksi $\sqrt{\text{in}}$)	Average da/dt	Final K (ksi $\sqrt{\text{in}}$)	Total Test Time (hr)
Sample 1	OCP – A _{Im}	106.0	$1.015 \cdot 10^{-5}$	94.75	>10,000
Sample 2	OCP – A _{Im}	104.9	No cracking	*	>10,000
Sample 3	OCP – A _{Im}	106.1	No cracking	*	>10,000
Sample 4	CP ₋₈₅₀	103.7	$1.792 \cdot 10^{-4}$	82	1,800
Sample 5	CP ₋₈₅₀	103.0	$3.560 \cdot 10^{-5}$	94.5	>5,000
Sample 6	CP ₋₈₅₀	82.4	$4.672 \cdot 10^{-5}$	78	>5,000
Sample 7	CP ₋₈₅₀	82.0	$1.891 \cdot 10^{-5}$	75	>5,000
Sample 8	CP ₋₈₅₀	81.3	No cracking	*	>5,000

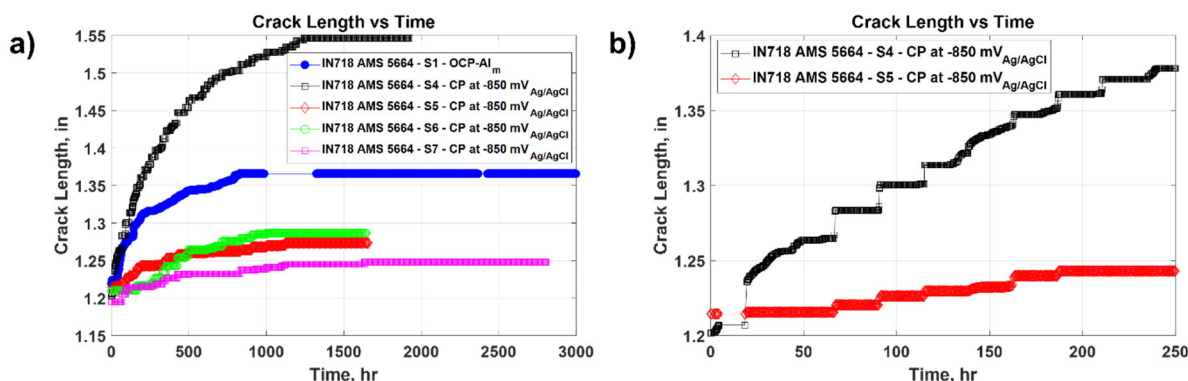


Figure 9 – Resulting EAC progression for samples that showed susceptibility during long term exposure testing.

Table 5 – Test conditions for nickel alloy 718 API6A and average measured crack growth rates.

	Environment	Initial K (ksi $\sqrt{\text{in}}$)	Measurable da/dt	Final K (ksi $\sqrt{\text{in}}$)	Total Test Time (hr)
Sample 1	OCP – A _{Im}	92.9	$4.472 \cdot 10^{-7}$	89.0	>10,000
Sample 2	OCP – A _{Im}	91.4	No	*	>10,000
Sample 3	OCP – A _{Im}	92.1	No	*	>10,000

Figure 10 shows a summary of all the results regressed as SIF vs crack growth rate expressed in inches per hour (in/hr). The downward arrows indicate the lowest SIF that showed cracking for the material and experimental condition tested. The AMS 5664 variant showed significant cracking under OCP alternate

immersion conditions. The crack growth rates progressively reduced until crack arrest was observed at a SIF of 94.75 ksi $\sqrt{\text{in}}$, here identified as the observed threshold. The same material under CP conditions showed measurable crack growth at significantly lower K. As shown, Samples 4, 6, and 7 all showed crack growth rates above 10^{-5} in/hr for K higher than 80 ksi $\sqrt{\text{in}}$. The behavior of Sample 4 and Sample 5 however were not in good agreement, with the first showing significant crack progression and a decrease in K of almost 20 ksi $\sqrt{\text{in}}$, while the second showed crack growth rates decreasing below 10^{-5} in/hr at a K = 94.5 ksi $\sqrt{\text{in}}$, followed by crack arrest. Two out of the three materials tested at an initial K of 80 ksi $\sqrt{\text{in}}$ showed measurable crack progression. The initial fatigue crack growth rates were in agreement with the measurements from Sample 4 at the beginning of the test and then progressively reduce below 10^{-5} in/hr when K reduced below 75 ksi $\sqrt{\text{in}}$.

Under OCP alternate immersion conditions 718 API6A did not show significant crack progression and an average crack growth rate is calculated and shown in Figure 10 for reference only. Data from [33] show that the crack growth rate for 718 API6A in constant immersion conditions is near 10^{-6} in/hr for an applied stress intensity of 100 ksi $\sqrt{\text{in}}$, and near 10^{-7} in/hr for an applied stress intensity of 80 ksi $\sqrt{\text{in}}$. The data from literature are for a constant-K type test rather than a K-shedding test and represent a conservative upper bound.

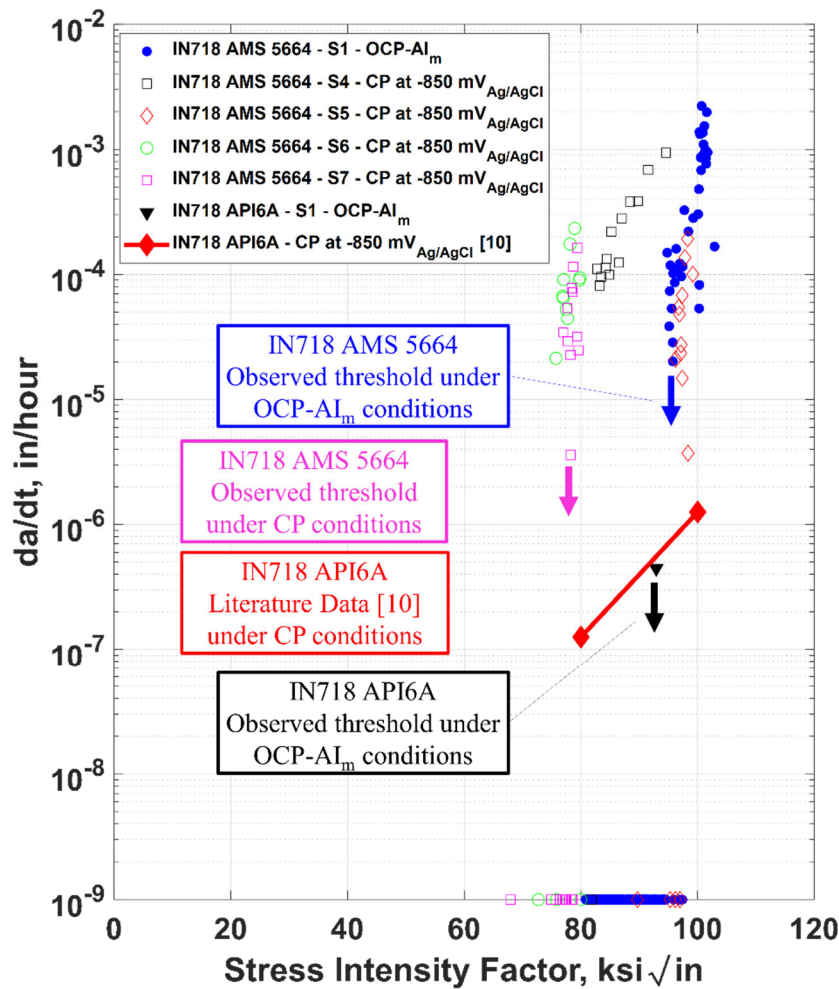


Figure 10 – Summary of results from long term exposure testing of alloy 718 (IN718) produced according to AMS 5664 and API6A.

While the microstructure of alloy 718 has been extensively studied and specific compositional limits and thermal processing steps are standardized to reduce HE susceptibility, the role of precipitates in deformation and hydrogen mechanism behavior is not well understood. A microstructural meso-scale model was developed to study the mechanical response of the elements of the microstructure for a constant applied displacement. Scanning Electron Microscopy (SEM) was performed to identify the parts of the microstructure to be modeled. A portion of the cross section of the 718 AMS 5664 round bar where the samples were extracted from was encapsulated in conductive epoxy, ground and polished to progressively finer grit. A final step of vibratory polishing in colloidal silica allowed the visualization of the grain boundaries and of the δ phase. The microstructure showed metal grains with average dimensions close to 10 microns (ASTM E114 number near 8) and the presence of δ phase at the grain boundary. The δ phase is generally considered to have an orthorhombic structure, with stoichiometry Ni_3Nb . Other precipitates, specifically γ' and γ'' , are smaller and mostly coherent with the FCC matrix and are not visible under SEM. Direct characterization using transmission electron microscopy (TEM) is planned in future work.

The grain structure was reproduced by using Voronoi tessellation: a set of 16 grains in a $16\text{ }\mu\text{m} \times 16\text{ }\mu\text{m}$ microstructure in a 2-dimensional plane-strain finite element analysis. The number of grains was defined based on a compromise between good representation of the variations in the material microstructure and a manageable computational time. As our initial goal is to understand the roles of the different parts of

the microstructure, computational time is minimized to allow an expansion of modeling parameters. The grain size, overall similar to the observed grain size from polished samples of AMS 5664, was also a compromise between reflecting the expected size in the materials studied and the computational burden given by the modeling of the phases within each grain. The larger the grain, the higher the number of precipitated phases inside the grains and therefore the computational size of the model. The δ phase was modeled at the grain boundaries as a circular phase with diameter ranging from 0.1 μm to 0.2 μm . The model allows for different options for δ phase shape, size, and distribution on the grain boundary. The option for the results presented in this work was of a fixed number of two δ phases on each grain boundary, for grain boundaries that were long enough to accommodate them. Their number on different grain boundaries also varied as to avoid intersections with other δ phases or parts of the microstructure. The δ phase was modeled as an ordered tetragonal crystal with elastic constants $C_{11} = 264$ GPa, $C_{12} = 175$ GPa, $C_{13} = 155$ GPa, $C_{33} = 261$ GPa, $C_{44} = 142$ GPa, $C_{66} = 150$ GPa [40]. The material orientation of the δ phase was simplified as the average of the Euler angles of the two adjacent grains.

The γ'' phase particles were modeled at the interior of the grain boundary as ellipses with major to minor axis ratio of 2. The major axis dimension was varied between 0.1 μm to 0.2 μm . The former value is representative of an upper bound limit for the expected size of this phase for materials with comparable thermal history to the material tested in this work (AMS 5664 and API 6A) [32, 38, 47]. The larger size is used for comparative purposes only and it is not meant to be a representative microstructure. It is a useful reference microstructure to study the trends in the model. The γ'' phase particles were randomly distributed at the interior of each grain with constraints dictated by distance from the grain boundary, to avoid intersection with δ phase or grain boundaries, and by distance to the other γ'' phase particles. Although a characteristic set of orientations was observed for this phase within the γ microstructure [48], this model randomizes the orientation of the major axis with respect to the orientation of the metal grain. Future work will include the expected misorientations of the γ'' phase. Constant volume fractions of γ'' phase were compared by varying their size and number within the microstructure. The γ'' phase was also modeled as an elastic crystalline material with the same constants as the δ phase. The material orientation of the γ'' phase was the same as the grains that includes it.

A crystal plasticity formulation was implemented into a User Material subroutine (UMAT) in Abaqus based on the work by Huang [49] to model the mechanical response of the metal grains. Before plastic deformations occur, the FCC γ -matrix of the material is modeled as an elastic crystal with constants: $C_{11} = 233$ GPa, $C_{12} = 147$ GPa, $C_{44} = 112$ GPa [50]. The formulation accounts for shear strain deformation on all 12 slip planes when plastic deformation occurs. The constitutive equations were modified to account for the distribution of mobile and immobile dislocations within the microstructure. Following the work by Zikry [51, 52], the strength of each slip system was modeled as a function of the mobile and immobile dislocation densities developed upon plastic deformations. The main step of the implementation of the theory is in the following evolution equations and their coupling to the shear strain rate and strength equations:

$$\begin{aligned} \dot{\gamma}^{(\alpha)} &= \dot{\gamma}_{ref}^{(\alpha)} \left(\frac{\tau^{(\alpha)}}{\tau_{ref}^{(\alpha)}} \right) \left(\frac{|\tau^{(\alpha)}|}{\tau_{ref}^{(\alpha)}} \right)^{(1/m-1)} \\ \tau_{ref}^{(\alpha)} &= \left(\tau_y^{(\alpha)} + G \sum_{\beta=1}^{nss} b^{(\beta)} \sqrt{a_{\alpha\beta} \rho_{im}^{(\beta)}} \right) \end{aligned} \quad (7)$$

Where $\dot{\gamma}^{(\alpha)}$ is the shear strain rate and $\dot{\gamma}_{ref}^{(\alpha)}$ is a reference shear strain rate, $\tau^{(\alpha)}$ is the resolved shear stress, $\tau_y^{(\alpha)}$ is the initial strength, G is the shear modulus, $b^{(\beta)}$ is the Burger's vector, and all refer to the slip system α . The variable nss is the number of slip systems, $a_{\alpha\beta}$ represents the influence of dislocation

accumulation along a β slip system, 0.2 for cross-coupling and 1.0 for self-coupling, $\rho_{im}^{(\beta)}$ is the immobile dislocation density for the β slip system. The evolutions of the mobile and immobile dislocation densities are coupled through a separate set of differential equations [51, 52]. The material properties for the crystal plasticity model were developed by simulating the tensile response of a grain aggregate and comparing with the stress-strain response of the material.

The model also included a sharp crack-like defect at one side, and it was loaded in tension by applying a constant displacement on the top boundary with the bottom boundary being fixed. The area near the crack is modeled as an elastic crystal. The mesh was refined around all the δ and γ'' phases, for a minimum element size of 0.0125 μm . The largest model, named model C1, included 16 grains, 41 δ phase particles, and 6100 γ'' phase particles; the number of nodes was 1,132,375. The model was developed as parametric, allowing all the inputs to be changed systematically and to observe the variations in mechanical response. Results for three variations of the largest model developed are presented here. A summary of the input variations of model C1 is shown in Figure 11 each is given a name for ease of comparison in Table 6.

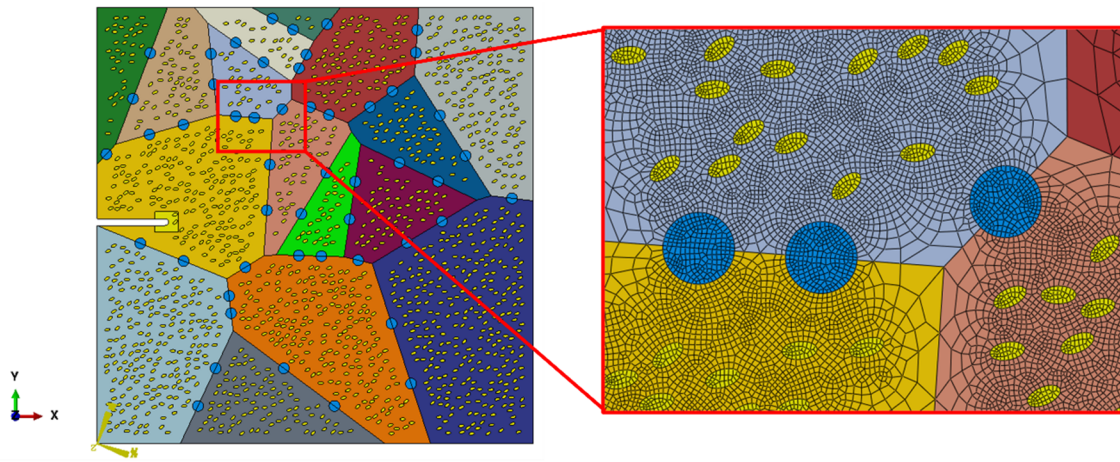


Figure 11 – Representative microstructure 2D plane strain model for alloy 718; detail view of the phases in the finite element model: grain boundary δ phase and intergranular γ'' phase.

Table 6 – Summary of the differences in δ and γ'' phase modeling between the three models presented in this work.

Model Name	γ'' number of particles	γ'' major diameter, μm	γ'' total area, μm^2	δ number of particles	δ diameter, μm	δ total area, μm^2
C1a	6100	0.1	23.9	41	0.4	5.15
C1b	1500	0.2	23.5	41	0.4	5.15
C1c	1500	0.2	23.5	41	0.2	1.29

The first set of results compares the effects of size of γ'' phase particles at the same volume fraction. The volume fractions translate into an area ratio in the 2D model developed. The total area of the γ'' phase particles is 23.9 μm^2 for the first model, named C1a, and 23.5 μm^2 for the second model, named C1b. The difference between the two models is in the size of the γ'' phase: the major diameter is 0.1 μm for model C1a and 0.2 μm for model C1b, all γ'' phase particles have the same major to minor diameter ratio of 2.0. Their distribution is different, and it is determined by randomly positioning particles within the interior of the grain, leaving a distance equal to the diameter of the δ phase from the grain boundary. The models also have the same number of grains, grain orientation, δ phase position, size and orientation.

The resulting total accumulated shear strains for model C1a and C1b are shown in Figure 12. The maximum in the contour plot refers to 0.5% shear strain, while the minimum refers to no accumulated shear. The accumulated shear strain is slightly higher for model C1a with the smaller γ'' major diameter. Some

differences are visible near a grain boundary towards the bottom of the microstructure. A higher accumulated shear strain occurs there because the two adjacent grains have high misorientation. The contours for model C1a are less fragmented and more homogenous throughout the model. Near the grain boundary this results in a higher accumulated shear strain for model C1a.

Some differences are also observed in the distribution of immobile dislocation densities, with dislocation accumulation on a (111) plane in the $[-110]$ direction, shown in Figure 13. A small increase in dislocation density for the model with the larger size of γ'' particles can be appreciated in several parts of the microstructure. Higher level of dislocation activity is shown at grain boundaries, particularly at locations within δ phase particles. A simple quantitative metric can be obtained from the figures to have a better sense of their visual qualitative difference. The images are translated into an 8-bit RGB representation and the pixels with red component higher than 200/255, and with blue and green component equal or lower than 1/255 are counted. Results show that the number of red pixels for model C1a is lower than for model C1b.

The third model, named C1c, uses the same size, distribution, and orientation of the γ'' phase particles in model C1b and compares the effects of the δ phase volume fraction by reducing the size of the δ phase in the model to 0.1 μm in diameter. The results of accumulated slip, in Figure 14 identify the same critical grain boundary visualized previously as the location of highest accumulated slip. The maximum in the contour plot refers to 0.5% shear strain, while the minimum refers to no accumulated shear. For both models the accumulated shear strain contour plots are similarly fragmented by the network of γ'' phase particles. The comparison also shows a trend of increasing shear strain with decreasing δ phase diameter. Figure 15 **Error! Reference source not found.** shows the immobile dislocation densities along the (111) plane in the $[-110]$ direction as shown previously. There is a very significant impact of the δ phase size along the grain boundaries, with significantly decreased activity throughout the microstructure. Particularly significant is the reduction in dislocation density at the critical grain boundary earlier identified. The same metric calculated previously confirms these qualitative observations and the red pixel number in Figure 15 is reduced with respect to both model C1a and C1b.

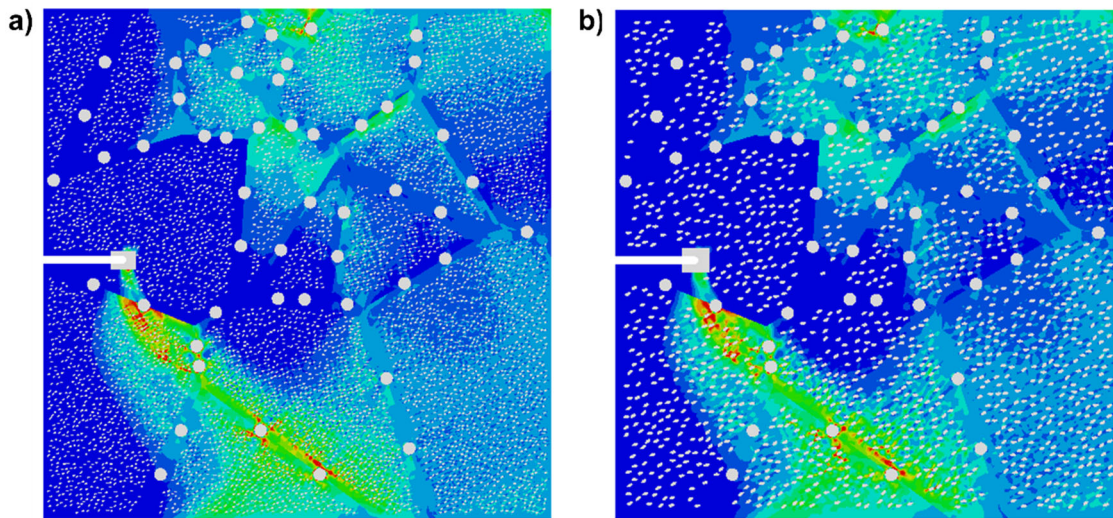


Figure 12 – Total accumulated shear strain for model C1a and C1b. A higher accumulation of shear strain for model C1b occurs at a grain boundary towards the bottom of the microstructure, while higher accumulated shear occurs within the grains of model C1a.

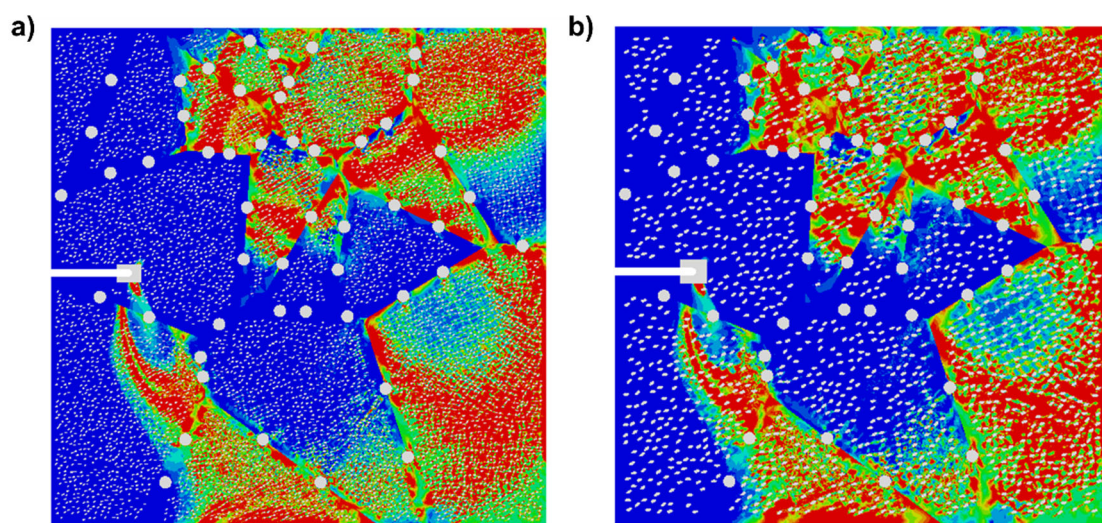


Figure 13 – Dislocation density along a (111) plane in the [-110] direction, for model C1a and model C1b.

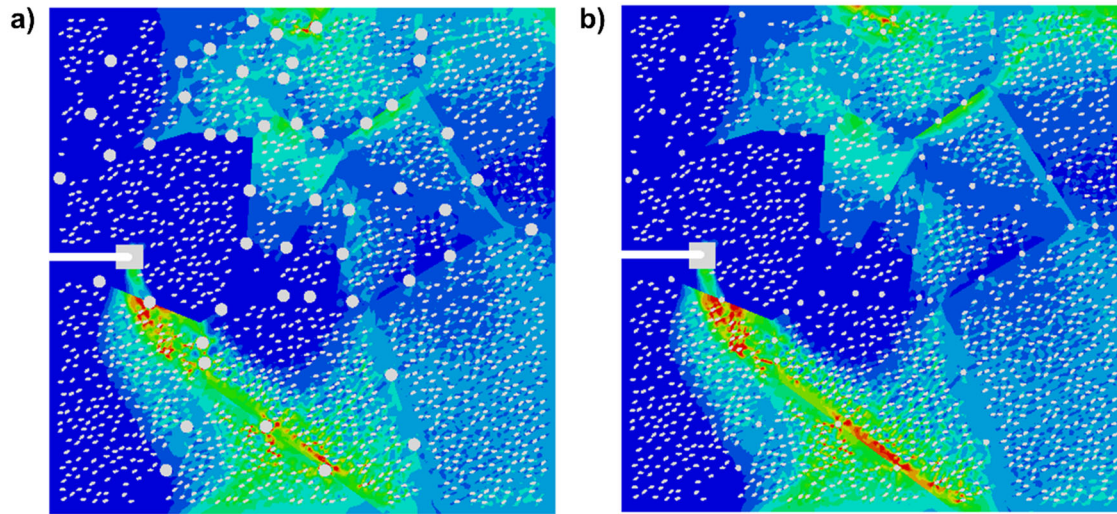


Figure 14 – Total accumulated shear strain for model C1a and C1c. A higher accumulation of shear strain for model C1c occurs at a grain boundary towards the bottom of the microstructure.

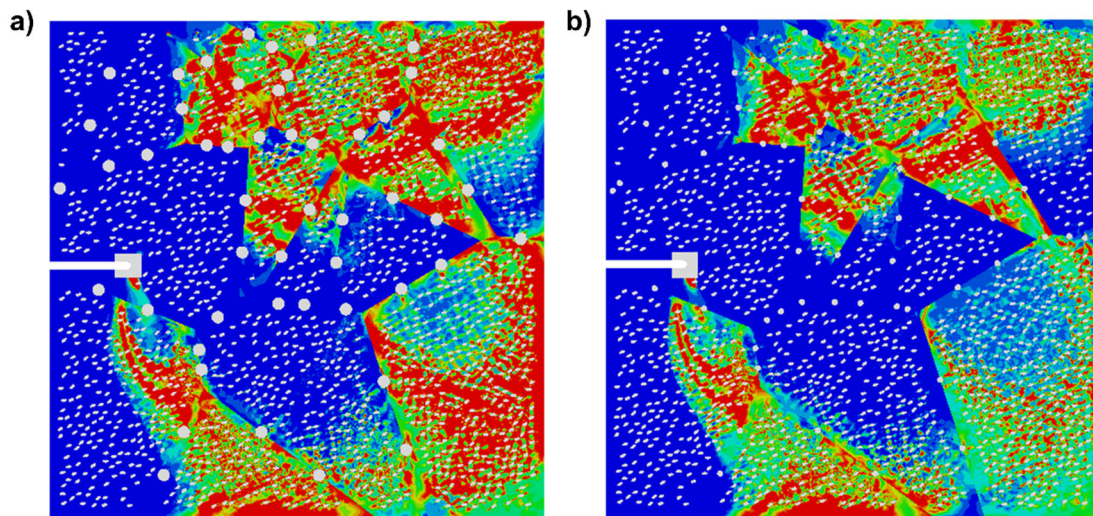


Figure 15 – Dislocation density along a (111) plane in the [-110] direction, for model C1b and model C1c.

Discussion

Experimental

The experimental results reflect a trend of higher growth rates and lower threshold for higher hydrogen-producing environments. Both materials show a degree of susceptibility to SCC and HE, with the 5664 variant being the most affected. All the results presented, with the exception of the results from [15], were obtained for K-shedding conditions. These results are noteworthy, as shedding-K testing conditions are considered less aggressive than constant- or rising-K. Fasteners in stiff joints are more likely to experience a decreasing load when the stiffness of the fastener reduces while the joint displacement remains constant or in redundant structures if the load redistributes to other parts of the bolted connection. Additionally, because of the constant applied bolt displacement in the tests, the K decreases faster over time for the samples showing higher crack growth rates. The higher the initial rate, the higher the reduction in

dK/da experienced by the sample per unit time, which is known to affect the response of the materials to SCC and HE [15, 53-56].

It is interesting to note that despite the higher rate of reduction of dK/da for Sample 4 when compared to Sample 5, the latter shows crack arrest at higher K. We can infer that the sensitivity to a reduction in K is not as strong in natural seawater under the application of cathodic protection at this potential. Trends for nickel alloy 718 and other precipitation hardened nickel alloys in comparable environmental conditions [15] show lower crack growth rates for K-shedding conditions. Constant- or rising-K tests are therefore expected to show higher growth rates and lower thresholds. It is possible that the long exposure time in unfiltered natural seawater causes local crack tip conditions to be significantly more aggressive and reduce the sensitivity of the material to a decreasing K condition. This practically means that crack arrest may not readily occur, and cracks may continue to progress for AMS 5664 material even after a quick reduction in K. The observed differences may also be related to the intrinsic material variabilities. Additional experiments are needed to clarify which factor affects the results more strongly.

Modeling

The models developed represent a microstructure with coarse γ'' precipitates and grain boundaries decorated with δ precipitates of regular size. The coarse size of the γ'' precipitates is an upper bound limit of observed distributions for the materials modeled [32]. The δ phase distribution is representative of the occasional precipitation of regularly shaped δ phase within the microstructure, which meets the acceptance criteria of the API 6A specification [43]. The C1a model more closely represent the microstructure for a material that underwent a two-phase heat treatment, like AMS 5664, with coarser and less coherent precipitates present within the matrix, discrete δ phase, and a precipitate free zone near the grain boundary [32, 57]. The microstructure for 718 API6A is tailored to minimize the precipitation of δ and the coarsening of the γ'' phase. Recent work showed that the API6A could still result in precipitation of δ phase at the grain boundary, albeit in less regular, more elongated shape, and γ'' precipitates with a length near and above 50 nm within the microstructure [15]. As small differences in microstructures of precipitation hardened alloy 718 were shown to determine significant differences in EAC behavior, two models that better represent the materials tested in this work will be developed based on SEM and TEM examinations in future work.

The model developed in this work also simplifies the deformation behavior of the microstructure of the material, in particular it simplifies the interaction of the matrix and the precipitates without considering the shearing of precipitates [58]. Because of the choice in constitutive equations the precipitates are considered fully coherent with the matrix during elastic deformations, while they act like obstacles to deformations when the matrix plastically deforms. The intent is to idealize the behavior of the interaction to reduce the computational burden for a numerical simulation of a larger portion of the microstructure ahead of the crack tip. The results from the study of mechanical response are being currently used to develop a model for hydrogen transport through the microstructure, coupled with the deformation and dislocation density results presented.

Improvements to the model are underway to represent a more realistic distribution and orientation of the metal grains, and of the γ'' precipitates, as well as to include γ' precipitates, along with more realistic shapes of δ phase at the grain boundary. A tailored two-step heat treatment process able to yield a 150 ksi API6A-compliant material was shown to decrease the size of the γ'' precipitates, increase the co-precipitation of γ' , and obtain precipitate-free grain boundaries [15, 18, 33]. The modeling presented will focus next on these relatively small changes in microstructure to study their impact on mechanical behavior.

Despite its shortcomings, the model is able to capture the main trends that have been identified by experimental work on nickel alloy 718 microstructures. The modeling results in this work indicate that coarsening of the γ'' phase results in increased dislocation density accumulation at the critical areas of the microstructure in the simulated deformation. Larger γ'' phase particles in the model likely act by enhancing defined paths of shear deformation in the model, while obstructing shear deformations on other paths. Increased dislocation density for other slip planes modeled at the same location (not shown) supports the

theory that intersection of dislocation slip bands and interaction of dislocation motion may result in increased strain localization and increased susceptibility to EAC. An increased dislocation activity is also observed in between the two δ precipitates modeled on one of the critical grain boundaries in Figure 5 and Figure 7.

The reduction in δ phase size also translates into a decreased activity comparing model C1b and C1c in Figure 8. This seems consistent with observations of increased occurrence of intergranular cracking in the presence of δ phase at the boundaries, particularly for grain boundaries with higher coverage of δ precipitates. The change in dislocation activity is significant in all of the areas of the microstructure, where the δ phase acts as a magnet for increased dislocation activity. The results also indicate increased strain localization in the precipitate free zone.

Conclusions

This study compared the EAC behavior of two commonly used variants of alloy 718. Two different exposure conditions, OCP under alternate immersion conditions and CP at -850 mV vs Ag/AgCl highlight the susceptibility to hydrogen producing environments of AMS 5664 variant. A significant degree of variability in the results at high applied SIF was also observed. The API 6A variant was significantly less susceptible to EAC in OCP alternate immersion conditions. A meso-scale microstructural model was developed to study the role of precipitates within the materials microstructure. An initial set of models was developed to represent the susceptible microstructure of the AMS 5664 material studied. The model uses a dislocation-density based crystal plasticity formulation and simplifies the matrix-precipitate interaction to represent partial coherence with the matrix. Results of the model show trends in line with literature observations on HE sensitivity of material microstructures. Increased dislocation activity for larger γ'' and δ precipitates at the grain boundary are interpreted as increased hydrogen transport paths to weak areas of the microstructure.

THE ROLE OF Γ' AND Γ'' PRECIPITATES ON HYDROGEN DIFFUSION

In this work, we directly study the local deformation conditions for a representative material element at the notch of a fracture mechanics sample. While a general condition of yielding is recognized, the specific strain levels as dictated by the applied stress intensity are determined. This strain condition is used as boundary condition for a micromechanical model to simulate the deformation behavior of a representative grain aggregate and an incipient microstructurally small crack. The input from the mechanical analysis is used to define a driving force for hydrogen within the microstructure and study the distribution of hydrogen ahead of the incipient crack.

Introduction and Motivation

Nickel-based alloy UNS N07718 is a high strength, precipitation hardened alloy extensively used in marine environments due to its combination of high general corrosion resistance, good strength, and high resistance to chloride and sulfide stress corrosion cracking [41, 42]. The strength of the material is given primarily by precipitation hardening of three intermetallic phases, namely γ' , $Ni_3(Al, Ti)$, with L12 structure, γ'' , a metastable Ni_3Nb phase with DO₂₂ structure, and δ phase, the stable counterpart of the γ'' phase with orthorhombic structure. Because of the material heterogeneous microstructure and the role of the precipitates in both deformation and environmental assisted cracking behavior, controlled chemistry, solution annealing, and heat treatments are required to obtain a material resistant to hydrogen producing marine environment. Variations in material microstructure are a function of the manufacturing process [38] and minimizing the presence of δ phase in the microstructure is shown to be important in reducing the susceptibility to intergranular cracking. The most commonly used condition in marine applications, particularly in the Oil and Gas sector, is produced to meet the American Petroleum Institute (API) standard 6A 718 specification which, requires a solution annealing temperature between 1870 and 1925 °F, above the δ solvus, and includes a single heat treatment step at a temperature between 1425 and 1475 °F for 6 to 8 hours to achieve a minimum yield strength of 120 ksi [21, 43]. The API specification also requires compliance with NACE MR0175 for all cases involving exposure to H₂S, which limits the maximum hardness to Rockwell Hardness (HRC) 40. The resulting microstructure was shown to have minimal δ precipitation at the grain boundaries and the environmentally assisted cracking (EAC) behavior to be among the best of similar PH Ni-based alloys [15, 18].

The use of UNS N07718 (API 6A) in naval applications is increasing, particularly as a fastener material, due its resistance to EAC and hydrogen embrittlement (HE) under cathodic protection systems that maintain an applied potential equal to or more electronegative than -850 mV vs Ag/AgCl [15, 44]. The API specification includes two additional grades for 718 with increased yield strength up to a maximum of 150 ksi. EAC data on the latter grades are being investigated with interest as the potential impact of an increase in yield by 25% is significant [33]. There is considerable interest in understanding the role of microstructure in the EAC performance of the material, with the potential of further refining the heat treatment to achieve even better performance at higher strengths and because of the significant use of 718 in additive manufacturing [59]. We have shown in previous investigation that the matrix-precipitate interactions are important in determining the material elastic-plastic response causing increased shear localization and partial dislocation activity at the δ -phase and matrix interface, and activation of specific slip systems and dislocation-density interactions [60]. Mechanistically, shear strain localization is considered a strong driving force for hydrogen assisted cracking [15, 32]. Trapped hydrogen can be more easily transported by dislocations and therefore might accumulate at locations with highly localized strain and at grain boundaries causing progressive degradation of the interfacial strength, leading to decohesion. Additionally, previous microstructural modeling for 718 showed that different slip systems are activated at the precipitate-matrix interface with respect to the main activated slip systems at the grain boundary interior [60]. The interaction of dislocations on different slip systems near the interface of the precipitate with the matrix is another potential driving force for increased hydrogen uptake in the vicinity of the precipitates.

In this study we developed a microstructural model representative of UNS N07718 alloy and define the local stress-strain conditions at the tip of a notch of a fracture mechanics sample to calculate the driving

forces for hydrogen diffusion within the material microstructure. The driving forces coming from hydrostatic stress and mobile dislocation densities were combined to provide an assessment of hydrogen concentration ahead of an initiated crack at the root of the notch. The role of immobile dislocations as irreversible traps and the changes in hydrogen concentration trends was also identified.

Modeling Methods and Boundary Conditions

A global model representative of a typical fracture mechanics geometry was developed using the Finite Element (FE) method to best approximate the boundary conditions of a detailed microstructural model at the crack tip. The model is a 2D plane-strain simulation of a wedge-opening load sample with a sharp notch with a root radius, $\rho = 0.5$ mm. The load is applied as a surface pressure at the bottom of the sample as indicated in Figure 16 a) and at a normalized distance of $0.25W$ from the edge, where W is the width of the sample. The notch length is $0.52 \times a/W$, where a is crack depth, the total length of the sample is $1.25W$ and the height of the sample is $1.2W$. The geometry is consistent with a typical compact tension C(T) sample, and used for environmental assisted cracking (EAC) assessment of structural materials [61]. The model is subjected to an applied load such that the stress intensity factor (SIF) at the notch root K is $88 \text{ MPa} \sqrt{\text{m}}$, assuming the sharp notch has a microstructurally small crack at its root.

The model uses a Ramberg-Osgood formulation to describe the material stress-strain behavior:

$$E\varepsilon = \sigma + \alpha \left(\frac{|\sigma|}{\sigma_y} \right)^{n-1} \sigma \quad (8)$$

where σ is Cauchy stress, ε is mechanical strain, E is Young's modulus, α is the “yield” offset defined as: $\frac{\varepsilon_y E}{\sigma_y} - 1$, at the condition of yielding, σ_y is yield strength, and n is the hardening exponent, fitted to the material stress-strain behavior. The API specification gives different minimum strengths depending on the grade, but in this work we refer to the specification with the minimum yield of 120 ksi. Commercial forms produced according to this specification generally have higher yield strength than the minimum. In this work $\sigma_y = 135$ ksi, $\alpha = 0.5$, and $n = 9.0$, obtained from fitting experimental stress-strain data from the literature [18]. The material response according to the Ramberg-Osgood formulation with the linear elastic portion and the 0.2% offset is shown in Figure 16 b).

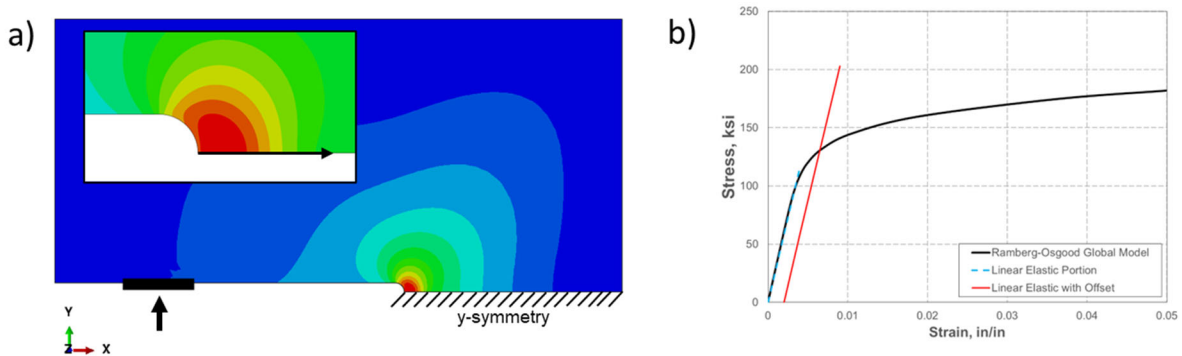


Figure 16 – Finite Element model of fracture mechanics sample and resulting maximum principal stress.

The contour of the maximum principal stress is shown in Figure 16 a). The plastic zone defined as the area with stress above $2\sigma_y$, reflecting the plain strain constraints, is shown in red. Figure 17 a) shows the resulting normalized stress gradient σ_{yy}/σ_y as a function of distance from the sample notch. The peak is at approximately 0.5 mm from the notch tip and corresponds to 2.25 times the yield strength for the material.

The stress rapidly decreases as a function of distance from the notch root. Figure 17 b) shows the distribution of strain, ϵ_{yy} , as a function of distance from the notch tip. Strain exceeds 4% at the notch tip and rapidly decreases to less than 0.2%.

The results of the analysis are considered the initial mechanical condition for the sample before a crack initiates from the notch [62]. This condition also holds when the crack is short when compared to the notch root radius [63]. As the crack grows beyond the influence zone of the notch, its mechanical response tends to the ideal sharp crack solution. The results are used as boundary conditions for a local model, representing the microstructure of the material.

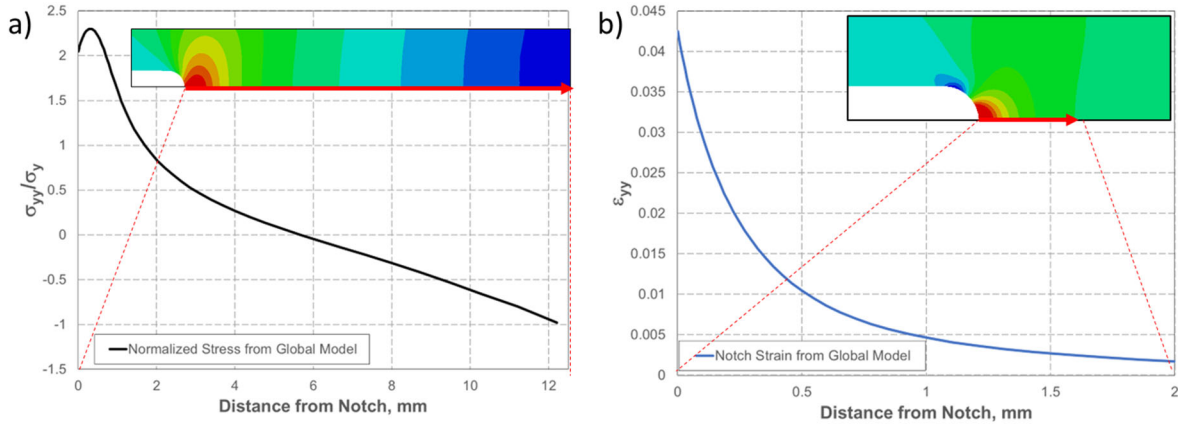


Figure 17 – Stress, σ_{yy} , and strain, ϵ_{yy} , as a function of distance from the notch tip.

Methods

Local Model and Assumptions

A local model was developed to better represent the stress-strain condition at the notch when a crack initiates and then propagates as driven by environmental driving forces. The model developed in this work is intended to provide a more in-depth understanding of the effects of local microstructure in promoting the diffusion of hydrogen towards a fracture process zone (FPZ). The mechanical driving force distribution within the material microstructure is used as direct input for a simulation of diffusion of hydrogen. In this work we focus on hydrogen assisted cracking in a regime where the crack growth rates are higher for increasingly electronegative potentials, such as -1.0 V vs Ag/AgCl [18, 64]. In this regime, the hydrogen at the wake and tip of an initiated crack diffuses towards the fracture process zone, leading to crack propagation. While there is still significant debate over the role of hydrogen in EAC for this material, experimental data for UNS N07718-API alloy points towards the significance of the diffusion of hydrogen through the material microstructure as the rate limiting step for crack growth progression [65]. More recent studies looked at the role of the material microstructure in hydrogen assisted cracking of 718 alloy, identifying the importance of grain size, grain boundary characteristics, and dislocation-precipitates interactions [66-68].

The grain structure was reproduced by using Voronoi tessellation: a set of 18 grains in a 100 μm x 100 μm microstructure in a 2-dimensional plane-strain finite element analysis. The number of grains in the representative area was defined based on an ASTM grain size finer than 2.0, as required by the API specification and literature on the material [21]. The major precipitates are explicitly modeled within the material microstructure. The δ phase was modeled at the grain boundaries as an ellipsoidal phase with a major axis of 1.0 μm , and an aspect ratio of 2.0. The number of δ phases on each grain boundary is maximized at 2.0, for grain boundaries that were long enough to accommodate them. Their number on

different grain boundaries also varied as to avoid intersections with other δ phases or parts of the microstructure. A visual representation of the microstructure is shown in Figure 18.

The γ' and γ'' phases were modeled at the interior of the grain as circular precipitates with a diameter of $0.1 \mu\text{m}$ and as elliptical precipitates with major axis of $0.2 \mu\text{m}$ and aspect ratio of 2. These values are representative of an upper bound limit for the expected size of these phases for materials with comparable thermal history [32, 38, 47]. The γ' and γ'' phases were randomly distributed at the interior of each grain with constraints dictated by distance from the grain boundary, to avoid intersection with δ phase or grain boundaries, and by distance to the other γ' and γ'' phase particles.

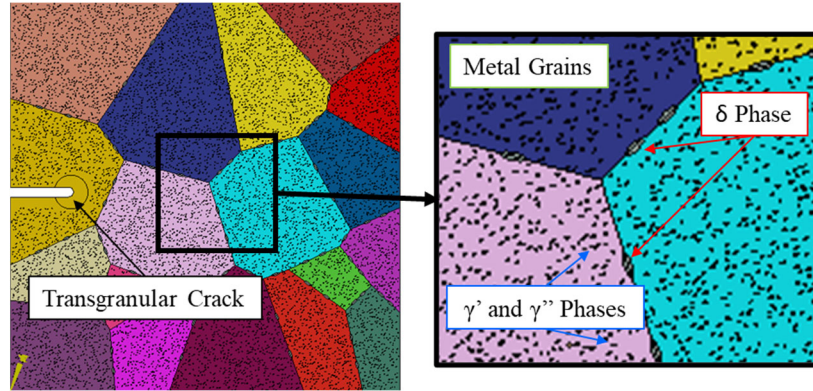


Figure 18 – Local model with representative microstructure for 718-API material.

A crystal plasticity formulation was implemented into a User Material subroutine (UMAT) in Abaqus based on the work by Huang [49] to model the mechanical response of the metal grains. Before plastic deformations occur, the FCC γ -matrix of the material is modeled as an elastic crystal with constants: $C11 = 233 \text{ GPa}$, $C12 = 147 \text{ GPa}$, $C44 = 112 \text{ GPa}$ [50]. The formulation accounts for shear strain deformation on all 12 slip systems when plastic deformation occurs. The constitutive equations were modified to account for the distribution of mobile and immobile dislocations within the microstructure. Following the work by Zikry [51, 52], the strength of each slip system was modeled as a function of the mobile and immobile dislocation densities developed upon plastic deformations. The main step of the implementation of the theory is in the following evolution equations and their coupling to the shear strain rate and strength equations as:

$$\dot{\gamma}^{(\alpha)} = \dot{\gamma}_{ref}^{(\alpha)} \left(\frac{\tau^{(\alpha)}}{\tau_{ref}^{(\alpha)}} \right) \left(\frac{|\tau^{(\alpha)}|}{\tau_{ref}^{(\alpha)}} \right)^{(1/m-1)} \quad (9)$$

where

$$\tau_{ref}^{(\alpha)} = \left(\tau_y^{(\alpha)} + G \sum_{\beta=1}^{nss} b^{(\beta)} \sqrt{a_{\alpha\beta} \rho_{im}^{(\beta)}} \right) \quad (10)$$

and $\dot{\gamma}^{(\alpha)}$ is the shear strain rate and $\dot{\gamma}_{ref}^{(\alpha)}$ is a reference shear strain rate, $\tau^{(\alpha)}$ is the resolved shear stress, $\tau_y^{(\alpha)}$ is the initial strength, m is a rate sensitivity parameter, G is the shear modulus, $b^{(\beta)}$ is the Burgers vector, and all refer to the slip system α . A value of $\tau_y^{(\alpha)} = 330 \text{ MPa}$ was used based on previous work with

this alloy system [60], and the $\dot{\gamma}_{ref}^{(\alpha)}$ and m values were determined based on an optimization approach to give the best match with the material yield strength. The selected values were respectively $\dot{\gamma}_{ref}^{(\alpha)} = 0.01$ and $m=10$. The variable nss is the number of slip systems, $a_{\alpha\beta}$ represents the influence of dislocation accumulation along a β slip system, 0.2 for cross-coupling and 1.0 for self-coupling, $\rho_{im}^{(\beta)}$ is the immobile dislocation density for the β slip system. The evolutions of the mobile and immobile dislocation densities are coupled through a separate set of differential equations [51, 52].

$$\frac{d\rho_m^\alpha}{dt} = |\dot{\gamma}^\alpha| \left(\frac{g_{source}^\alpha}{b^2} - g_{minter}^\alpha \rho_m^\alpha - \frac{g_{immob}^\alpha}{b} \sqrt{\rho_{im}^\alpha} \right) \quad (11)$$

$$\frac{d\rho_{im}^\alpha}{dt} = |\dot{\gamma}^\alpha| \left(g_{minter}^\alpha \rho_m^\alpha + \frac{g_{immob}^\alpha}{b} \sqrt{\rho_{im}^\alpha} - g_{recov}^\alpha \rho_{im}^\alpha \right) \quad (12)$$

where g_{source}^α is a coefficient pertaining to the increase in mobile dislocation density due to dislocation sources, g_{minter}^α is related to the trapping of mobile dislocations due to forest intersections or cross-slip around obstacles or dislocation interactions, g_{immob}^α is related to the immobilization of mobile dislocations, g_{recov}^α is related to the rearrangement and annihilation of immobile dislocations. The complete formulation of all of the coefficients was developed by Zikry et al. [51, 52]. The dislocation-based crystal plasticity parameters controlling the evolution of mobile and immobile dislocation densities for Nickel alloy were assumed to be consistent with the ones obtained for a 718 material model [60]. The calibration of the several model parameters is a significant challenge for the development of a crystal plasticity formulation that can best represent the stress-strain material behavior. In this work we implemented an optimization methodology to identify the $\tau_y^{(\alpha)}$, $\dot{\gamma}_{ref}^{(\alpha)}$, and m parameters that maximize the correlation with the yield strength of the material. The simulation is performed for a model of grain aggregates subjected to plane stress conditions.

The δ phase was modeled as an ordered tetragonal crystal with elastic constants $C11 = 264$ GPa, $C12 = 175$ GPa, $C13 = 155$ GPa, $C33 = 261$ GPa, $C44 = 142$ GPa, $C66 = 160$ GPa [40]. The material orientation of the δ phase was simplified as the average of the Euler angles of the two adjacent grains. The γ' phase was modeled as an elastic crystalline material with elastic constants $C11 = 224$ GPa, $C12 = 147$ GPa, $C13 = 147$ GPa, $C33 = 224$ GPa, $C44 = 124$ GPa, $C66 = 124$ GPa [40], with orientation inherited from the grain it resides in. The γ'' phase was also modeled as an elastic crystalline material with the same constants as the δ phase. A characteristic set of orientations was observed for the γ'' phase within the γ microstructure [48], this model randomizes instead the orientation with respect to the orientation of the metal grain as a simplification.

The material properties for the crystal plasticity model were developed by simulating the tensile response of a grain aggregate and comparing with the stress-strain response of the material. The mesh was refined around all the δ and γ'' phases, for a minimum element size of $0.0125 \mu\text{m}$. The model includes 18 grains, 68 δ phase particles, 5000 γ' and 5000 γ'' phase particles; the number of nodes was 1,262,155. The model was developed as parametric, allowing all the inputs to be changed systematically and to observe the variations in mechanical response.

The microstructural model is loaded in tension by applying a constant displacement on the top boundary with symmetry conditions at the bottom. The displacement boundary condition for the microstructural model was set as to represent the condition shown in Figure 19 with the model being subjected to the applied strain calculated from the global model. The condition of strain, ε_{yy} , is simplified

as an effective strain of 3.5%, which corresponds in Figure 17 to an average value of strain obtained withing a distance of 100 μm from the notch tip. The model also included an initiated crack at one side of the grain aggregate corresponding to the notch root of the sample. The crack is modeled as a microstructurally sharp notch with root radius of 1.25 μm .

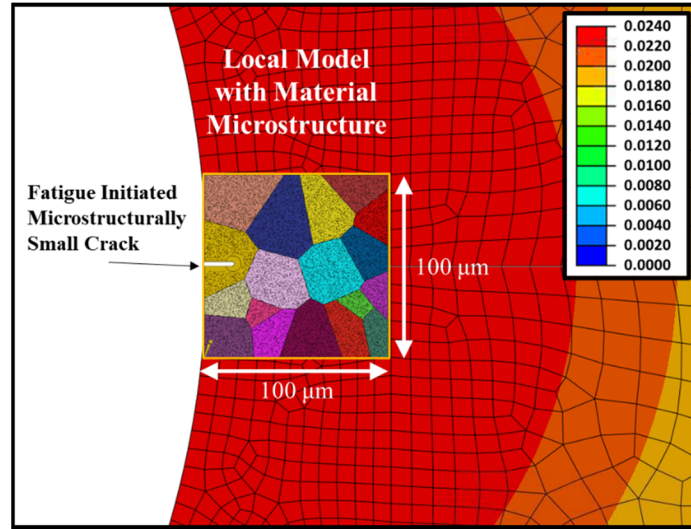


Figure 19 – Representation of local model in reference to the global model and the resulting plastic strain at the notch.

Formulation for Hydrogen Diffusion

The diffusion of hydrogen through the microstructure was modeled by using Fick's law of diffusion [69]. The assumption is that hydrogen is generated at the mouth and surface of the initiated crack and then diffuses within the material microstructure. The model uses a stress-assisted diffusion formulation to account for the role of hydrostatic stress as a contributor to the chemical driving force given by the concentration gradients throughout the microstructure. The diffusion simulation is only weakly coupled to the mechanical analysis as it is performed with input from it, but not at the same time as the mechanical simulation and it does not influence the mechanical response.

The flux of diffusible hydrogen is given by the chemical and hydrostatic stress gradients within the material. The model implementation accounts for the effects of chemical and hydrostatic stress gradients directly. The additional driving force is given by the velocity of mobile dislocations, obtained from the mobile dislocation densities calculated from the mechanical loading of the sample [70]. The formulation for hydrogen transport can be summarized as:

$$\frac{\partial}{\partial t}(C_L + C_T) + \nabla \cdot (\mathbf{J}_T + \mathbf{J}) = 0 \quad (13)$$

$$\mathbf{J}_T = \alpha \theta_T N_T^m V_{\rho_m} \quad (14)$$

$$\mathbf{J} = -D \nabla C_L + \frac{DV_H}{3RT} C_L \nabla \sigma_{kk} \quad (15)$$

where C_L and C_T are the trapped and diffusible concentrations of hydrogen in the microstructure, $\mathbf{J}_T$ and $\mathbf{J}$ are the corresponding fluxes, with α being the number of sites per trap, and θ_T the number of traps per solute volume, N_T^m is the dislocation trap density, V_H is partial molar volume of hydrogen in solid

solution, σ_{kk} is hydrostatic stress. The hydrogen diffusivity, D , and solubility are $2.9 \cdot 10^{-11} \text{ cm}^2/\text{s}$ and $20.85 \text{ molH}_2/\text{m}^3\sqrt{\text{MPa}}$ respectively based on literature data [71]. Hydrogen trapped by mobile dislocations is transported with a velocity V_{ρ_m} calculated as:

$$V_{\rho_m} = \frac{\dot{\epsilon}_p}{\rho_m b} \quad (16)$$

where $\dot{\epsilon}_p$ is obtained by assuming a final plastic strain of 3.5%, a total loading time for the sample of 1800 s, Burgers vector, b , is 0.2 nm, and ρ_m is obtained from the mechanical analysis. The flux of trapped hydrogen is determined by the number of available trap sites per volume of solute, $\alpha\theta_T$, and by the dislocation trap density, expressed as:

$$N_T^m = \lambda \rho_m / a \quad (17)$$

where ρ_m is mobile dislocation density calculated by the model, a is lattice parameter, and $\rho = \sqrt{3}$ for FCC materials. The model does not calculate separate concentrations for diffusible, C_L , and trapped, C_T , hydrogen and uses the above formulation to calculate a single concentration of hydrogen at steady state. The concentration is the summation of C_L and C_T .

The model accounts indirectly for the effects of immobile dislocations as irreversible traps for hydrogen by including a dependence of hydrogen diffusivity on immobile dislocation densities. The diffusivity is modified as a function of immobile dislocation density, changing linearly and becoming $1/2$ of its initial value when the immobile dislocation density is $10^5 \text{ 1}/\mu\text{m}^2$. The latter value does not have at this stage a supporting experimental basis, and it is only used as variable to estimate the effects of trapping by immobile dislocations. The results will be shown for the case of no trapping by immobile dislocations, considering the effects of the driving forces in Eq. 14 and 15 alone, and for the case of medium trapping and strong trapping effects, arbitrarily defining a diffusivity reduced to $1/2$, and to $1/10$ respectively of its initial value when the immobile dislocation density is $10^5 \text{ 1}/\mu\text{m}^2$.

The solution domain for the diffusion analysis is the grain aggregate shown in Figure 18. The hydrogen flux is constant at the beginning of the analysis for the “external surfaces” of the representative microstructure, i.e., the surfaces on the left side of the domain in Figure 18 and for the surfaces forming the crack faces and crack tip. These areas correspond to the notch root of the global model, shown in Figure 19; note that the solution is not obtained for the entire global model domain and only limited to the local representative microstructure domain in Figure 18. The rest of the domain is set to have a concentration of 0.0 ppm at the beginning of the simulation, and the diffusion analysis proceed in two steps. In the first step the flux is constant at the boundary until the concentration of hydrogen at the boundary reaches a level of 5.13 ppm, corresponding to the total hydrogen concentration, including trapped hydrogen, for the material as obtained from the literature when the material is polarized to -1 VSCE [72]. In the second step of the analysis, the concentration at the boundary remains at 5.13 ppm and the diffusion equations are continuously solved throughout the domain.

The boundary condition change is justified, based on electrochemical hydrogen charging experiments at different cathodic potentials were performed for 718 by Lillard, followed by measurements of hydrogen concentration by inert gas fusion analysis. A relation between cathodic current and hydrogen concentration showed in Lillard’s work is used in this work along with the polarization curve for 718 from [18] to find the current density at -1.0 V. The flux is set to yield a concentration of 5.13 ppm at the external boundary in 1 hour of exposure time. The analysis is continued with a constant concentration boundary condition at the external surfaces for a total of 10^7 seconds or approximately 2,700 hours.

Microstructural Model Results

Polycrystalline Stress-strain response

This section describes the results of the stress-strain simulation performed with the model of the microstructure without the initiated crack to validate the choice of material parameters in the crystal plasticity model described previously. The polycrystal is subjected to a prescribed strain and the average stress in the direction of straining calculated by the model is used as a metric for comparison with the stress-strain behavior of the material. The analysis assumes plane stress constraints to represent a typical tensile test standard condition. A constant strain is applied as boundary condition at the top portion of the model with the bottom being symmetric. An average measure of stress is obtained from the model as a representation of the engineering stress value.

Figure 20 shows the resulting stress-strain response of the grain aggregate. The stereographic projections representing the orientations of the grains within the microstructure are shown on the right [60]; this is representative of a uniformly oriented grain structure as it would be obtained for example after forming into a solid bar. The Euler angles are not from direct measurements, and randomly generated for this set of grains. The stress-strain results are consistent with the elastic portion of the curve and for the initial phase of yielding of the material. The simulation results are shown to deviate for large strain values, with fluctuating stresses for strains higher than 1%. This is due to the low number of grains simulated and the texture of the grain aggregate, showing the progressive activation of more slip directions as the material is strained beyond yielding [73]. The engineering stress-strain curve is an upper limit for the analysis for strains within 1.5%, while being lower than the stress predicted by the crystal plasticity model. The further increase in stress as a function of strain, rather than a progressively lower strain hardening is a function of the directionality of the metal matrix.

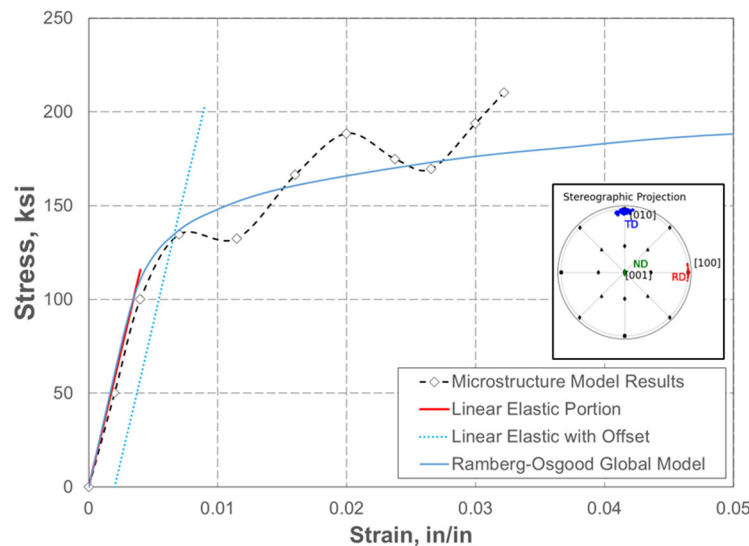


Figure 20 – Stress-strain response of the metal grain aggregate subjected to tension (no notch present).

Mechanical Response in the Presence of a Crack

The results from the mechanical loading of the microstructural model with an initiated crack are presented in this section. Boundary conditions representative of the strain state at a sharp notch of a fracture mechanics samples, as presented in Figure 2, are applied at the top surface (see orientation in Figure 16 and Figure 19). A condition of symmetry is specified at the bottom boundary. A state of plane strain is imposed as solution condition within the model to simulate the constraint effects at the center of a fracture mechanics sample.

Figure 21 a) shows the total cumulative shear strain at the end of the deformation step, with 5% being the maximum value shown by the scale. The total cumulative shear strain is defined as the sum of the absolute values of shear strains in all slip systems, as $\gamma = \sum_{(\alpha)} \int_0^t |\dot{\gamma}^{(\alpha)}| dt$. Shear strain is highly localized at the initiated crack, as expected, but it is also significantly localized at grain boundaries. near the crack is significantly higher than 5%, exceeding 20% at the tip of the crack. The slip gradient across some grain boundaries is high, mostly driven by the relative grain misorientation, and specifically for grain boundaries with a 45° orientation with respect to the loading direction.

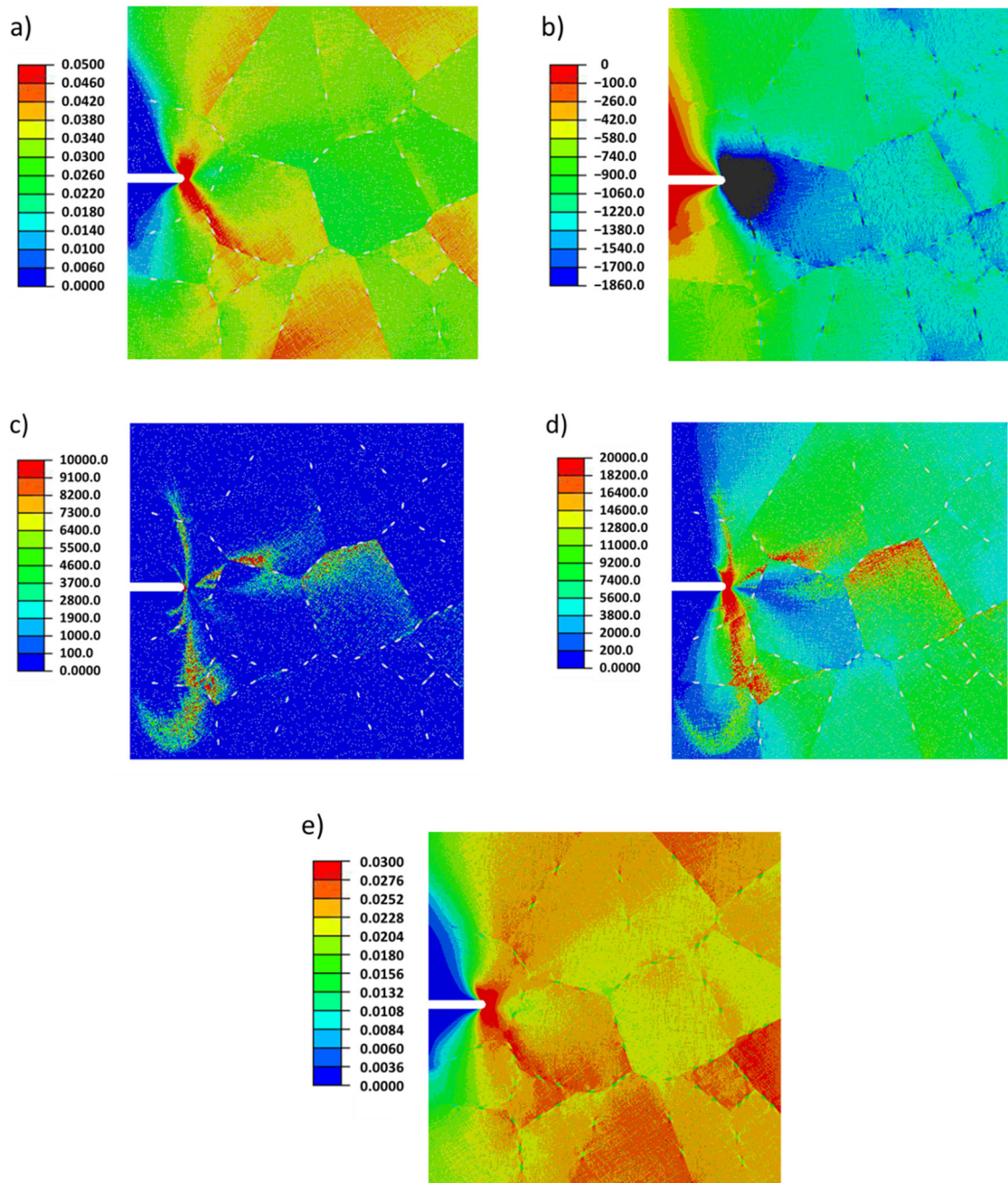


Figure 21 – Resulting total slip accumulation a), hydrostatic stress b), mobile dislocation density c), and immobile dislocation density d), and calculated plastic strain e), from the application of a constant displacement resulting in 4% strain of the metal aggregate representing the state of deformation at the notch tip.

Figure 21 b) shows the distribution of hydrostatic stress within the microstructure. A large area of high hydrostatic stress ahead of the crack tip is determined by the model. Note that the finite element solver used for this problem defines pressure stress as $-\frac{1}{3}\sigma_{ii}$, hence the results in Figure 6 b) indicate positive hydrostatic pressure. The average value of hydrostatic stress in this area is above two times the yield

strength of the material. The locations throughout the microstructure with increased hydrostatic stress are mostly in the vicinity of grain boundaries, forming a path in the center of the microstructure. The interface between matrix and precipitates also shows increased hydrostatic pressure. This results from the difference in the material properties between the matrix and precipitates, also increased by a different modeling strategy used in this work: the matrix is modeled as an elastic-plastic material, while the particles as elastic, leading to local increase in hydrostatic stress to accommodate the difference in lateral constraints at the interface during inelastic deformations.

Figure 21 c) shows the mobile dislocation density obtained by the model for the applied 3.5% strain. The maximum value shown in the image is $10,000 \text{ } 1/\mu\text{m}^2$, which corresponds to a density of $10^{16} \text{ } 1/\text{m}^2$. Mobile dislocation densities are highest at the root of the crack; however areas of increased dislocation activity are visible, specifically along grain boundaries towards the interior of the microstructure. The mobile dislocation density path along the grain boundaries results from the higher resistance to slip as determined by the different relative grain orientation and the grain boundary orientation with respect to the applied load. This causes a significant gradient in both mobile dislocation densities across the grain boundary.

Figure 21 d) shows the immobile dislocation density obtained by the model for the applied 3.5% strain. The maximum value shown in the image is $20,000 \text{ } 1/\mu\text{m}^2$, which corresponds to a density of $2 \times 10^{16} \text{ } 1/\text{m}^2$. The immobile dislocation densities calculated by the model are on average higher than the mobile densities. The highest values of immobile dislocation densities are shown around the crack and along grain boundaries leading towards the interior of the microstructure. A significantly large area ahead of the crack tip shows a higher density of immobile dislocations, followed by an area of low density immediately adjacent to it. The immobile dislocation density gradients are less severe than the gradients shown for mobile dislocation densities. This is particularly evident by looking at the immobile dislocation density distribution at or near grain boundaries in comparison with the distribution at the grain interior.

Figure 21 e) shows the plastic strain calculated by the model for the domain. The plastic strain value at the crack tip is higher than the upper bound value indicated in the figure, with strain exceeding 10% at the tip of the crack. The limit values of the legend only represent useful bounds to appreciate the location of higher calculated plastic deformations. The areas where highest shear strain was identified earlier clearly show higher plastic deformations in the model.

Microstructural Model Diffusion Results

The results of hydrogen diffusion from the surface of material to the fracture process zone of an initiated microstructurally small crack are presented in this section. The results are obtained first for a model including the effects of hydrostatic stress, mobile dislocation velocity, and trapping by immobile dislocations. For this case the trapping by immobile dislocations is simulated by decreasing the diffusivity of hydrogen linearly with increasing immobile dislocation density, becoming $1/2$, its initial value when the immobile dislocation density is $10^5 \text{ } 1/\mu\text{m}^2$.

Figure 22 shows the resulting hydrogen concentration after a simulation time of 10^7 seconds. The maximum simulation time is identified as the time that minimizes the maximum change in concentration as a function of time calculated in the model. The final state is considered achieved when the maximum concentration change in the model is 0.01 ppm per 86400 s (1 day). Figure 22 a) shows the concentration of hydrogen is highest at a small distance ahead of the crack tip. The concentration is higher than the surface concentration by a factor of 1.2X. The normalized profile of hydrogen concentration as a function of distance from the crack tip is shown in Figure 22 b). The data are extracted from the path shown in Figure 7 a), and the normalized profiles of hydrostatic stress, mobile and immobile dislocation densities, and accumulated slip are also shown for reference. The hydrogen concentration is normalized by 5.13 ppm, the boundary condition at the sample surface at steady state. The area of highest hydrogen concentration generally corresponds to the area with the highest hydrostatic stress calculated by the model. The location

of the highest concentration does not however correspond the location of maximum stress, as it is affected by the presence of a high density of mobile and immobile dislocations. The high immobile dislocation density right at the tip of the crack causes a reduction in the flux and a lower accumulation of hydrogen, resulting in a reduction of hydrogen concentration with respect to the condition at the boundary, followed by a significant increase.

Other areas of higher concentration of hydrogen can be identified throughout the microstructure: precipitates, such as the δ phase on some grain boundaries show higher concentration than average, γ' phases on the other end show lower concentration than the surrounding matrix, while γ'' does not seem to play a strong role in either increasing or decreasing the nearby hydrogen concentration.

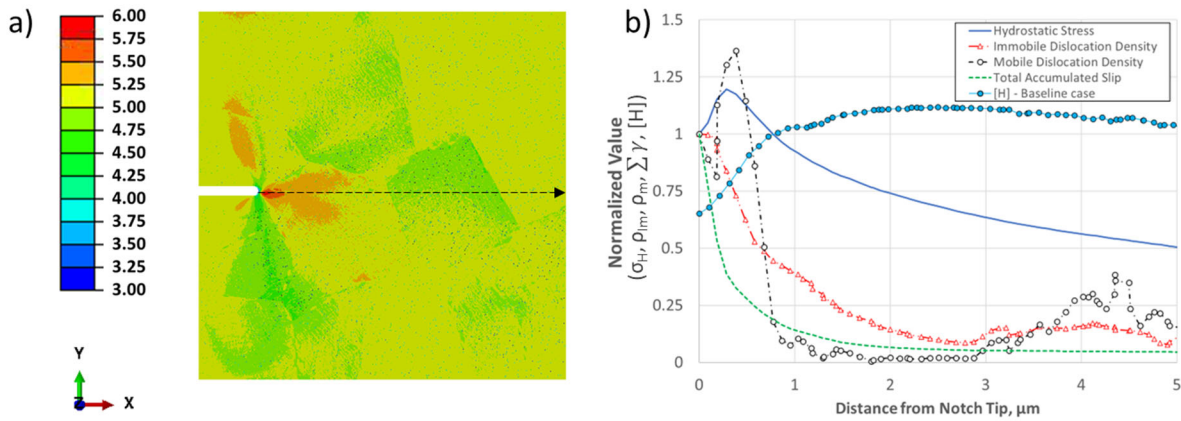


Figure 22 – Diffusion results for microstructural-representative model of UNS 07718 material. Distribution of hydrogen through the material microstructure a), and normalized concentration as a function of distance from the crack tip b), along the path shown in a).

To assess the contribution of irreversible trapping of hydrogen by immobile dislocations, two additional models are developed, and the results compared to Figure 22. In the first model the trapping by immobile dislocations is not included in the analysis. This is simulated by removing the dependency of diffusivity on immobile dislocation densities. In the second model a more severe change in hydrogen diffusivity as a function of immobile dislocation densities is implemented. This is achieved by modeling a sharper decrease in diffusivity with increasing immobile dislocation densities: the diffusivity decreases to $1/10$, of its original value when the immobile dislocation density reaches $10^5 \text{ 1}/\mu\text{m}^2$.

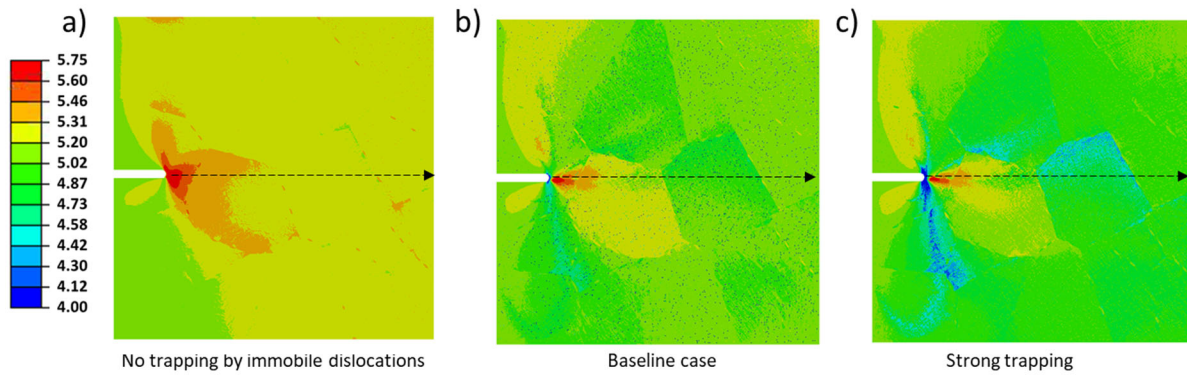


Figure 23 – Distribution of hydrogen within the material microstructure based on the diffusion modeling analysis for three cases of influence of trapping by immobile dislocations.

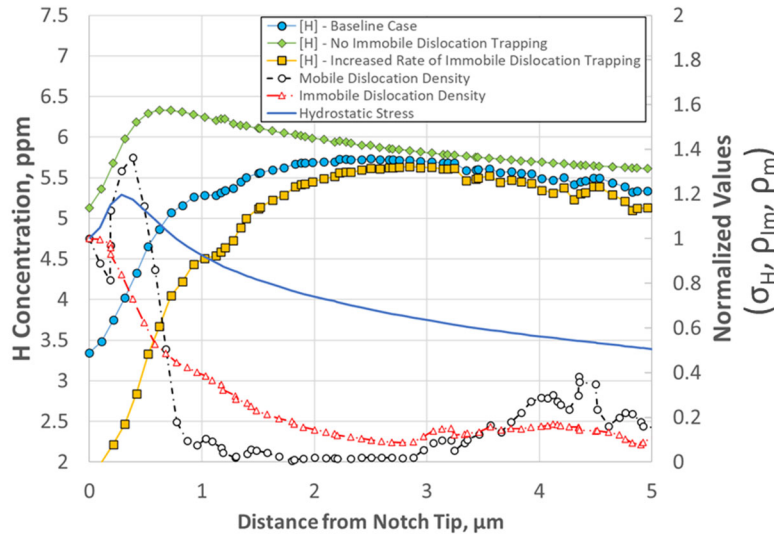


Figure 24 – Profile of hydrogen concentration along the paths shown in Figure 23 as a function of distance from the tip of the simulated crack. The normalized profiles of hydrostatic stress, σ_H , immobile dislocation density, ρ_{Im} , mobile dislocation density, ρ_m , and accumulated slip, $\sum \gamma$, are shown for reference.

The map of hydrogen concentration for all three cases is shown in Figure 23. The scale is changed with respect to the results in Figure 22 to better appreciate the relative differences. Figure 24 shows the distribution of hydrogen as a function of distance from the crack tip and compares the results for the case of diffusivity changed by a factor of $\frac{1}{2}$ (baseline case), and by a factor of $\frac{1}{10}$ and when trapping by immobile dislocations is suppressed. The normalized profiles of hydrostatic stress, σ_H , immobile dislocation density, ρ_{Im} , mobile dislocation density, ρ_m , and accumulated slip, $\sum \gamma$, are shown for reference. Trapping by immobile dislocations changes both the peak hydrogen concentration and the distribution of hydrogen near the crack tip. The concentration is highest for the analysis case where no trapping by immobile dislocations. The peak of hydrogen concentration moves closer to the tip of the crack when trapping is not present. An increased rate of trapping on the other hand causes the peak to shift down and towards the interior of the microstructure. To better identify the role of mobile dislocation velocity, its contribution is removed from the analysis. The results are shown in Figure 25 where the normalized

concentration of hydrogen as a function of distance from the crack tip is shown. The effect of transport by mobile dislocation is small, with the peak changing by less than 1%.

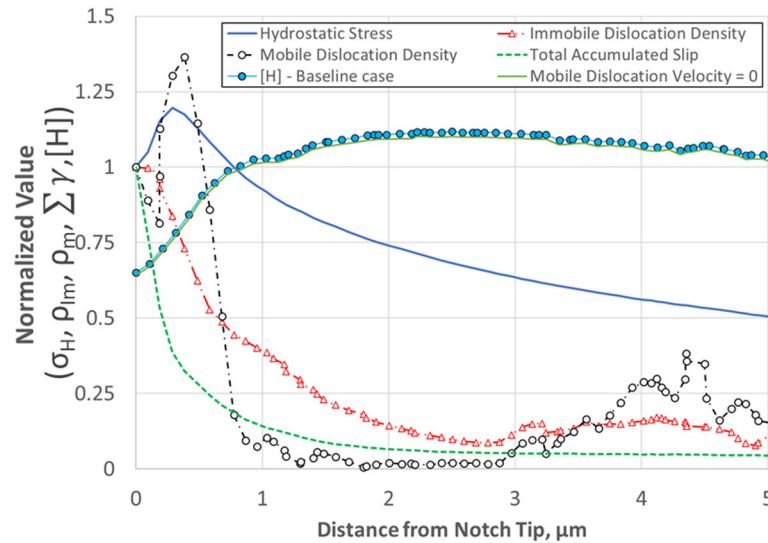


Figure 25 – Comparative results of hydrogen concentration for the analysis with mobile dislocation velocity set equal to 0, as compared to the baseline case; normalized concentration as a function of distance from the crack tip, along the path shown in Figure 22 a).

Discussion

In this work we identified the main characteristics of a PH nickel alloy microstructure that play a significant role in the material susceptibility to hydrogen embrittlement and developed a model to represent the response of the material to the applied loads. The microstructure of UNS 07718 is known to be driving the response to hydrogen producing environments, however the role of different constituents in the microstructure is not understood from a perspective of combined mechanical-chemical driving force.

From a mechanical standpoint the local conditions at a critical location, such as a notch, are needed. Despite the condition at the notch tip being nominally of yielding, it is important to advance the qualification of this state with the help of numerical tools currently available. Crystal plasticity formulations are suitable for this purpose and within the limits of the modeling assumptions can help qualify the differences between dissimilar microstructures. We hypothesized that the mechanical response of the material at the level of grains, precipitates, and dislocation densities would inform our understanding of hydrogen diffusion from the tip of an initiated crack to the fracture process zone, determining its advancing rate.

The results show that within an area of the microstructure subjected to significant plastic deformations mechanical driving forces vary significantly, both in terms of localized deformations (slip), and in terms of accumulated dislocations. Our formulation implements a hardening law as a function of generated dislocations within the microstructure. The nucleation, emission, and annihilation of dislocations are empirically accounted for by the evolution equations (Eq. 11 and 12) and associated coefficients [52, 60]. Within these assumptions, grain orientation and grain boundary characteristics are observed to influence the spatial gradients of hydrostatic stress, mobile and immobile dislocations.

All of these are considered important in determining the driving force for hydrogen diffusion ahead of an initiated crack, as described by Eq. 14 through 15, and discussed in the literature [74]. A high localization of slip, mobile and immobile dislocations at the tip of the initiated crack is expected, however the balance of the three main driving forces ahead of the crack tip considered in this work is important. The relative comparisons of hydrogen diffusion within the microstructure show that the hydrostatic pressure gradient

ahead of the advancing crack is the strongest driving force for hydrogen accumulation. Hydrogen is drawn towards the fracture process area; however it is not the area with the highest calculated hydrostatic stress, but an area adjacent to it to be the area with the highest concentration. This is due to the combination of high hydrostatic stress and the presence of immobile dislocation density, acting as traps for hydrogen in the material. As shown by comparing the results of Figure 23 and Figure 24, the effects of immobile dislocation density in modifying the location of the highest concentration of hydrogen is significant. Interestingly, the area with the highest concentration of hydrogen is an area that includes part of the microstructure with the lowest values of accumulated slip, within the highest hydrostatic stress region, corresponding to the regions of the microstructure with the lowest density of traps. Diffusion by mobile dislocation velocity is not, in this formulation, as strong of a driving force as hydrostatic stress; results obtained by excluding the effects of mobile dislocation velocity do not differ significantly. The work from Dadfarnia et al. [70] showed the contribution of dislocation velocity and transport on hydrogen diffusion ahead of the tip of the crack was significant, which is contrary to what we found in our analysis. Qualitatively both models agree that the peak of hydrogen concentration is ahead of the crack tip with the distribution further into the material being a function of hydrogen diffusivity through the lattice. Our work indicates that the local microstructure can influence the contribution of hydrogen transport by dislocations within the material, reducing its effectiveness in some cases. Our work however did not account for a full coupling at this stage, and the mobility of dislocations is not being affected by the increasing concentration of hydrogen, which could increase the transport of hydrogen and balance the trapping by immobile dislocations.

Areas at the interfaces of matrix and precipitates are locations where hydrogen accumulation is influenced by the driving forces in this model. As expressed above, while it is good to note that these areas correspond to the precipitates within the microstructure, this only implies a tendency of hydrogen to redistribute at sites of gradients and discontinuities in deformation response of the materials. In this model matrix and precipitates have been modeled with significantly different behavior, elastic-plastic and only elastic respectively, to exacerbate the inherent differences in mechanical response. The results certainly stress the importance of the combination of the driving forces modeled in this work for hydrogen diffusion and the consideration of including their effects into a modeling strategy. The model developed in this work also simplifies the deformation behavior of the microstructure of the material, in particular simplifies the interaction of the matrix and the precipitates without considering the shearing of precipitates [58]. Because of the choice in constitutive equations modeled the precipitates are considered fully coherent with the matrix during elastic deformations, while they act like obstacles to deformations when the matrix plastically deforms. The intent was to idealize the behavior of the interaction to reduce the computational burden for a numerical simulation of a larger portion of the microstructure ahead of the crack tip. At the level of volume fractions modeled in this work, the assumption seems reasonable.

The use of a computational model is advantageous as separate effects can be quantified independently. While a validation data set would be difficult to obtain for this type of results, making a quantitative evaluation of material behavior based on the results of modeling difficult, relative comparisons can be beneficial to inform our understanding of the relation among different driving forces, such as hydrostatic stresses, mobile and immobile dislocations, and hydrogen diffusion within the material microstructure. Specifically, the local material texture, grain size and orientation, along with grain boundary characteristics could be systematically varied to understand what presents the weakest link in the progression of an initiated crack.

Experimental Validation Efforts

Experimental characterization of the material microstructure is currently underway to inform the microstructural model presented in this work. The goal is to characterize the precipitates within the metal grains, their sizes and characteristics to develop a unique model for variants of 718 material and

representing their composition, thermal-mechanical processing and resulting microstructure. Measurements were made using Atom Probe Tomography (APT) to identify at the atomic scale the composition, shape, and conformation of the precipitates for UNS N07718-API alloy produced following the API specification and a thermal-mechanical processing to yield a minimum yield strength of 150 ksi. APT samples were prepared by standard liftout and milling techniques in a ThermoFisher Nova 600 Dual Beam focused ion beam-scanning electron microscope (FIB-SEM), and APT was conducted using a CAMECA 4000X Si local electron atom-probe (LEAP). A sequence of sample preparation steps to yield a sample that can be analyzed with APT are shown in Figure 26 a) and b). A protective Pt strap was deposited on the 718 sample before lifting out material with a micromanipulator needle, Figure 26 a). The liftout material is a triangular prism that's been shaped using the FIB and then deposited onto a grid of posts on a microtip array. The array is made of pre-shaped posts for placing the materials of interest, which is then sharpened using a series of annular mills with an ion beam into a very sharp needle shape, Figure 26 b), so that ions can be field evaporated from it during the APT measurement. The results in Figure 26 c) show the microstructure of the material visualized by iso-surface contours with a threshold is an APT reconstruction of one of these tips. The interface surfaces indicated correspond to 57% Ni concentration, and highlight the interfaces between the matrix, colored in pink, and Ni-, Nb- rich precipitates, shown in green. Figure 26 d) shows a proximity histogram, which gives the atomic concentration measured in the APT sample as a function of distance from the precipitate-matrix surfaces shown in Figure 26 c). The data show the quantitative partitioning of elements to the matrix and the precipitates.

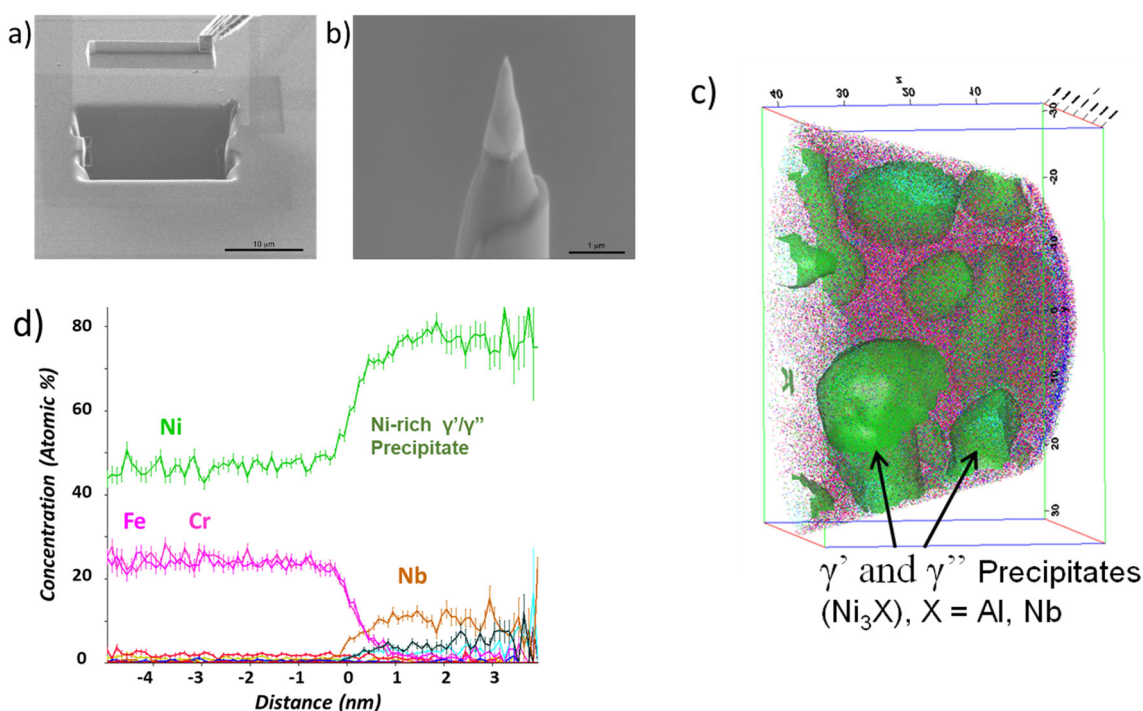


Figure 26 – Ongoing experimental validation effort by atom probe tomography; a) and b) showing sample preparation, c) showing the iso-surface reconstruction of precipitates with limiting surface being 57% Ni in content, and d) showing a proximity histogram collected during measurement and highlighting the precipitates boundaries and compositions.

The experimental technique is planned to be adapted for post-charging hydrogen measurements to identify at the atomic level the distribution of hydrogen within the material microstructure. This would provide a unique validation data set for the modeling approach in this work. The goal is to electrochemically charge a needle-shaped sample, such as the one shown in Figure 26 b), in a polarization test to allow hydrogen to diffuse through the material microstructure. The APT measurement post-charging will enable mapping the distribution of hydrogen within the material, providing a reference window and validation of

the microstructural material modeling. Extending the model shown in this work to a 3D representative volume element to represent the material microstructure such as the one in Figure 26 c), would also allow a more effective validation methodology. This technique combined with microstructural characterization by Electron Backscatter Diffraction (EBSD) and transmission electron microscopy (TEM) will enable characterization across multiple length scales and of larger volumes, and distributions of microstructural descriptors could be used as inputs for developing a microstructural model of the local conditions at the crack tip [75].

Conclusions

In this study, we modeled the stress-strain behavior at the notch of a standard fracture mechanics sample by modeling. The material was PH nickel-based superalloy 718, and the relevant material properties were used to identify the local deformations at a sharp notch for a CT sample. A meso-scale microstructural model was developed to study the role of the microstructure in the deformation behavior of a small representative volume of material ahead of the notch. The representative model of the material microstructure included the metal grains, the material texture, and the major precipitates along the grain boundaries and within the grains. The model was subjected to the plastic strain conditions identified at the notch to simulate the metal grain aggregate response from a mechanical standpoint when an initiated crack is introduced. The modeling results were used as input for the diffusion of hydrogen from the surface of the notch, assuming the material had been introduced to a hydrogen producing environment. The results show that hydrostatic stress is a strong driving force for hydrogen, increasing its concentration near the crack tip. Trapping by immobile dislocations, simulated as a decrease in diffusivity corresponding to locations within the microstructure with the highest calculated density of dislocations, can strongly influence the location of the highest hydrogen concentration, moving it towards the interior of the material.

HIGH-FIDELITY MEASUREMENTS OF ENVIRONMENTALLY ASSISTED CRACKING BEHAVIOR

In this work the EAC behavior of high strength UNS N07718 material is measured by using a constant stress intensity technique. Two versions of this material with strengths above 180 ksi are tested. Despite having the highest strength, one of the two versions of the material shows lower crack growth rates when tested in the same conditions.

Full Immersion under Cathodic Polarization UNS N07718

High strength alloys in marine service applications experience a wide range of environmental conditions including cathodic protection, galvanic coupling, ennoblement of freely corroding surfaces, and alternate immersion cycles. Environmentally assisted cracking (EAC) is a critical design assessment criterion and is highly dependent on microstructure, loading condition, and the exposure environment. An established approach to assessing EAC risk and environmental compatibility is through a fracture toughness evaluation utilizing crack growth rate behavior measured in a relevant environment.

Precipitation hardened (PH) alloy UNS N07718 is employed in many applications exposed to marine environments, and it is often in direct contact with other materials. Some of these materials, like structural steels, require cathodic protection, leading to prolonged exposure in hydrogen producing environments. The use of UNS N07718 API 6ACRA in marine applications is increasing as a fastener material due to its resistance to SCC in immersion and alternate immersion conditions [45] and hydrogen embrittlement (HE) under cathodic protection systems that maintain an applied potential equal to or more electronegative than $-850 \text{ mV}_{\text{Ag|AgCl}}$ [44].

UNS N07718 produced according to the API 6ACRA [43] specification is chemically and microstructurally controlled to address resistance to hydrogen producing environments. The API 6ACRA heat treatment utilizes single step aging, which is designed to minimize the precipitation of the delta phase (δ) along grain boundaries and avoid its growth within the grains as acicular phase. Two variants for UNS N07718 within this specification are available, with minimum yield strengths of 120 and 150 ksi. The 120 ksi version is the most commonly used specification for the most aggressive conditions in marine applications [15].

Higher strength versions of UNS N07718 can be achieved by heat treatment and cold working, with the highest tensile strength values exceeding 200 ksi. These materials have good resistance to EAC during atmospheric exposure [76]. They are commonly used in aerospace applications where the combination of good high and cryogenic temperature performance, and corrosion resistance in atmospheric conditions, make them well suited for joints of matching UNS N07718 components and, with additional mitigation strategies, for joining different materials [77]. Manufacturing of these fasteners involves the introduction of a critical amount of cold work into the material either during bar/wire production and a precipitation hardening operation, without prior solution heat-treatment⁶. However, the high strength and thermal processing of these materials makes them known suspects for EAC in full immersion marine environments under cathodic protection [77, 78].

In this work, we compare the EAC susceptibility for two UNS N07718 materials with different thermo-mechanical histories. We heat treated an AMS 5662 [79] metal bar to meet the AMS 5663 [80] specification, which requires a minimum of 150 ksi yield strength and 185 ksi tensile strength. For the second thermo-mechanical condition, UNS N07718 material was procured according to AMS 5962 [81]. This material is stronger than AMS 5663, where the minimum tensile strength for AMS 5962 is 220 ksi. The material is cold worked after solution annealing and subsequently heat treated to achieve the high tensile strength.

The focus of this testing program is to evaluate the EAC properties of high strength UNS N07718 material typically used as fastener materials. The material behavior was compared by measuring the crack growth rates of fatigue-initiated cracks under constant-K conditions during exposure to varying levels of polarization. The data were obtained for a range of potentials, from $-1050 \text{ mV}_{\text{SCE}}$ to $+300 \text{ mV}_{\text{SCE}}$. The latter

value represents the estimated potential when the nickel alloy is coupled to titanium and exposed to natural seawater without cathodic protection. The crack length of samples was constantly monitored during testing by direct current potential drop (DCPD) methods. The constant-K conditions were determined based on the estimated stress intensity for a thumbnail crack in a fastener. The variations of K were based on the relative material comparison and within a range of the initial K to assess the existence of a KIEAC, a threshold stress intensity.

The two materials tested in this study are shown in Table 7, along with the measured yield and tensile strengths, ductility, and hardness, where available. The AMS 5962 material is processed to obtain a very high yield strength, with reduced ductility and strain hardening.

Table 7 – List of materials tested and mechanical properties.

Material	Specification	Yield Strength, 0.2% offset	Tensile Strength	Elongation	Hardness
UNS N07718	AMS 5663	1,138 MPa (165 ksi)	1,393 MPa (202 ksi)	21.0%	
UNS N07718	AMS 5962	1,482 MPa (215 ksi)	1,560 MPa (226 ksi)	10.6%	47.8 HRC

Both AMS 5663 and 5962 specify a two-step heat treatment. The bars in this work were vacuum aged at 1,325 °F for 8 hours, controlled cooled to a temperature of 1,150 °F, and held at this temperature for up to a total heat treatment time of 18 hours. The difference between the two material was that the AMS 5663 bar was solution annealed and hot rolled to its final dimensions of 2.5 inches in diameter before heat treatment, while the ASM 5962 bar was solution annealed and cold drawn to it final 2.0 inches in diameter.

The microstructure of the AMS 5962 material is shown in Figure 27. A sample from the cross section of the bar was encapsulated in conductive epoxy, ground, and polished to progressively finer grit size. A final step of vibratory polishing in colloidal silica allowed the visualization of the grain boundaries and of the δ phase. Precipitation of δ phase is evident at the grain boundaries in the form of elliptical/rod shaped particles. Significant precipitation is evident along the grain boundaries particularly from Figure 1 b), but the delta phase does not appear to be forming a continuous path along the grain boundaries. The grain size is estimated from the images to be between 10-20 μm .

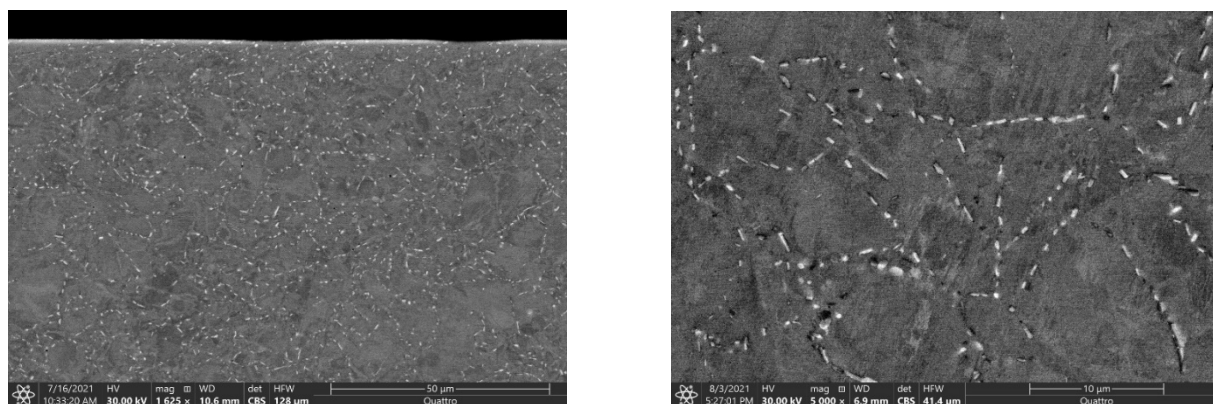


Figure 27 – Microstructure of UNS N07718 heat treated to AMS 5962.

The sample geometry is a standard compact tension C(T) specimen, as shown in Figure 28. All specimens were fatigue precracked according to ASTM E647 guidelines using the load shedding method [82]. The fatigue precrack length was approximately 0.4 times the width, W, for all samples. Per the ASTM E647 guidelines, the final Kmax reached during precracking was lower than any of the applied K during

subsequent testing. The specimens are side-grooved after precracking, with a side grooved thickness being 80% of the original specimen thickness.

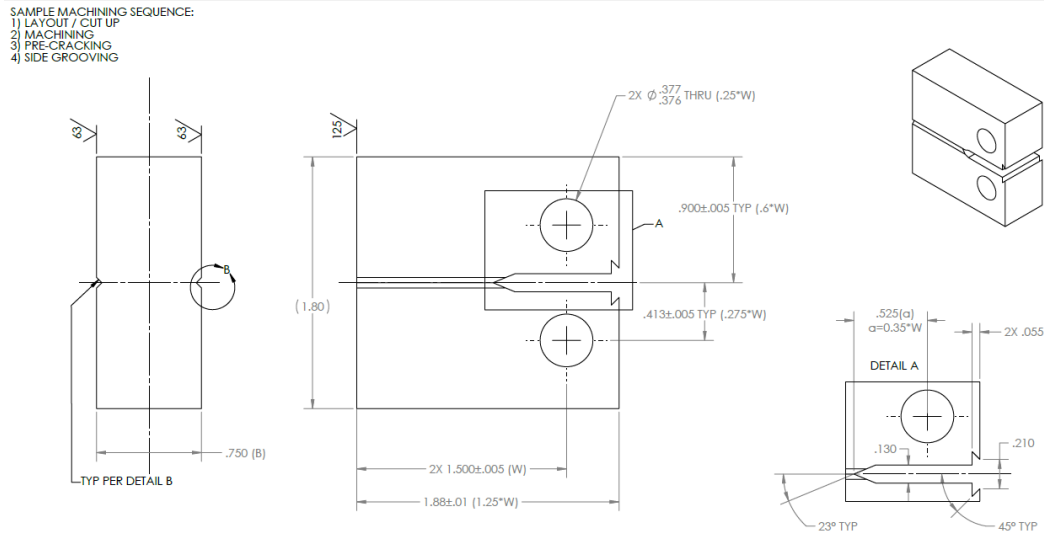


Figure 28 – Sample geometry tested in this work.

The tests are performed in a closed environmental cell, as shown in Figure 29; the sample is loaded in a servo electric frame with 10,000 lbs capacity. The test solution is refreshed periodically to ensure consistent environmental conditions. The sample is connected to a potentiostat through an electrochemical circuit with a reference and counter electrode. The crack length is monitored by the DCPD method: 5 platinum wires are connected to the sample, encapsulated so as to be isolated from the test solution, and protected by a layer of epoxy at the contact point with the sample. The platinum wires are spot welded to the sample: 2 wires for constant current input through the sample, 2 wires for voltage measurement, and 1 wire for electrochemical potential application.

An expected constant-K condition for a crack in a fastener was determined assuming a preload of 2/3 the yield strength. The yield strength used here was the average of the two materials tested: 1310 MPa (190 ksi). The range of possible fastener diameters were assumed to be between 12.7 and 57.1 mm (0.5 and 2.25 in). An initial thumbnail crack at the thread root of the with a depth of 1.6 mm (1/16 inches) can be considered a conservative estimate of the minimum detectable flaw size that can be obtained by using dye penetrant inspection (Type 1, Method C or D, Level 3 or 4, Form a or d) [80]. Mettu et al. published a general Stress Intensity Factor (SIF) solution for cracks in threaded fasteners with specific SIF solutions for standard aerospace bolt sizes [83]. Based on this SIF solution, an initial assessment of SIF conditions reveals a range of K between 47-58 MPa√m (43 and 53 ksi√in).

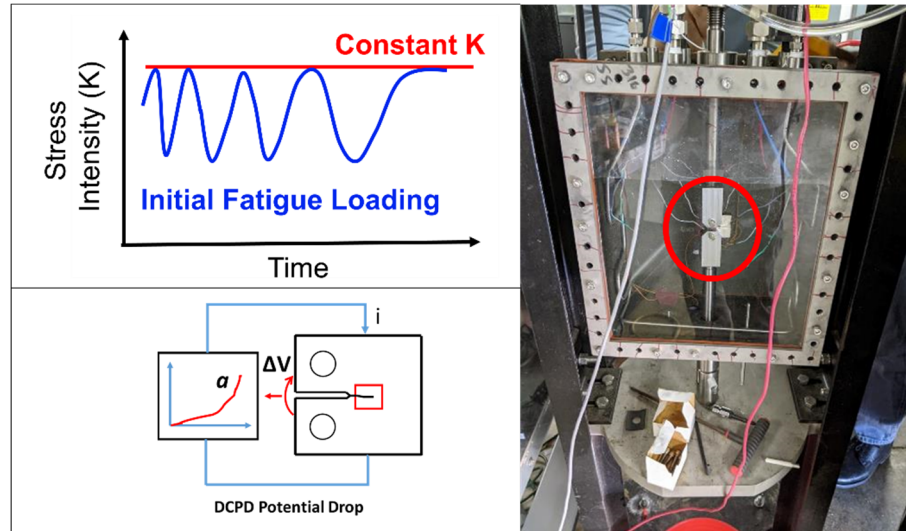


Figure 29 – Experimental setup and testing methodology with schematic representation of the loading and crack monitoring method.

For the materials in this investigation, no data were available in the literature to assess the extent of EAC, or whether a KIEAC was expected at the K-levels considered. The initial K conditions for all tests were set to $60.5 \text{ MPa}\sqrt{\text{m}}$ ($55 \text{ ksi}\sqrt{\text{in}}$) to ensure the initial measurements were within the resolution limit of the experimental technique. The tests are typically performed by starting a fatigue cycle with K_{max} equal to $60.5 \text{ MPa}\sqrt{\text{m}}$ and with K_{min} equal to $\frac{1}{2} K_{\text{max}}$ to initiate crack propagation. The tests start at a frequency of 0.1 Hz, and the frequency is gradually reduced to 0.001 Hz. The rising portion of the cycle is always 9/10 of the entire cycle time, while the falling portion is 1/10 of the total time. The last fatigue cycle includes a hold for 9,000 s at the K_{max} , for an effective frequency of 10^{-4} Hz. The test is finally transitioned to a constant-K condition, and the load is adjusted to maintain the same K during exposure. The potential in the fatigue portion of the tests is $-1050 \text{ mV}_{\text{SCE}}$. Once the constant-K test is started, the potential is decreased to other target levels in small steps. The testing matrix of K levels and applied potential conditions are shown in Table 8, where small variations in K and potential were applied to minimize the effects of prior history. Table 8 reports whether the testing condition refers to a constant-K or fatigue test, and for fatigue tests the frequency, in Hz, and the load ratio, R, defined as $K_{\text{min}}/K_{\text{max}}$.

The test starts by applying fatigue loading in the initial environmental condition (Table 8) and then progressively reducing the frequency of the cycle until a transition to a constant-K condition is achieved. A schematic view of the loading mode and measurement methodologies is shown in Figure 29. A resolution of $\sim 10^{-9} \text{ mm/s}$ can be achieved by using this experimental setup [18].

Table 8 – List of testing parameters for the materials in this work.

Material	K or K_{max}	Potential	Constant-K test/Fatigue Test	R and Frequency Range (Hz)
	MPa $\sqrt{m}$ (ksi $\sqrt{in}$)	mV $_{SCE}$		
UNS N07718 AMS 5663	60.5 (55.0)	From -1050 to -850	Constant-K test	-
	24.7 (22.5)	From -1050 to -850	Constant-K test	-
	60.5 (55.0)	-1050	Fatigue Test	R=0.5 10^{-4} – 0.1 Hz
UNS N07718 AMS 5962	60.5 (55.0)	From -1050 to +300	Constant-K test	-
	60.5 (55.0)	From -1050 to +300	Constant-K test	-
	60.5 (55.0)	-1050	Fatigue Test	R=0.5 10^{-4} – 0.1 Hz

Results

The results for the two materials are shown in this section, first the constant-K conditions for each alloy with crack growth rates as a function of K and applied potential are presented. The results are regressed to obtain crack growth rates, da/dt , by linear fitting of the crack length, a , as a function of time, t . The crack growth rates are plotted as a function of K and potential. The fatigue data are summarized next for both alloys.

AMS 5663 – High Strength UNS N07718 without cold working

The crack length, a , for AMS 5663 is shown in Figure 30 a) as a function of time for an applied potential varying between -1.0 VSCE and -850 mVSCE and for an applied K of 60.5 MPa $\sqrt{m}$. Temperature during testing was 23 °C. The results show an initially visibly constant crack growth rate, reducing when the potential is reduced to -900 mVSCE, and further reducing when the potential is reduced further to -850 mVSCE. The crack growth rates calculated from the a vs t chart are significant for all applied potentials at this K level. Figure 30 b) shows the crack growth rates obtained: all values are near 10^{-5} mm/s. Figure 4 b) also reports replicate results for this material at a slightly lower temperature of 18 °C. No significant trend is evident with this small change in temperature, and the crack growth rates remain high. Figure 4 b) also presents the results for a lower level of applied K, $K = 24.7$ MPa $\sqrt{m}$ (22.7 ksi $\sqrt{in}$). Despite more than halving the value of K with respect to the higher K test, the crack growth rates remain significant, in the 10^{-6} mm/s ($3.9 \cdot 10^{-8}$ in/s) range.

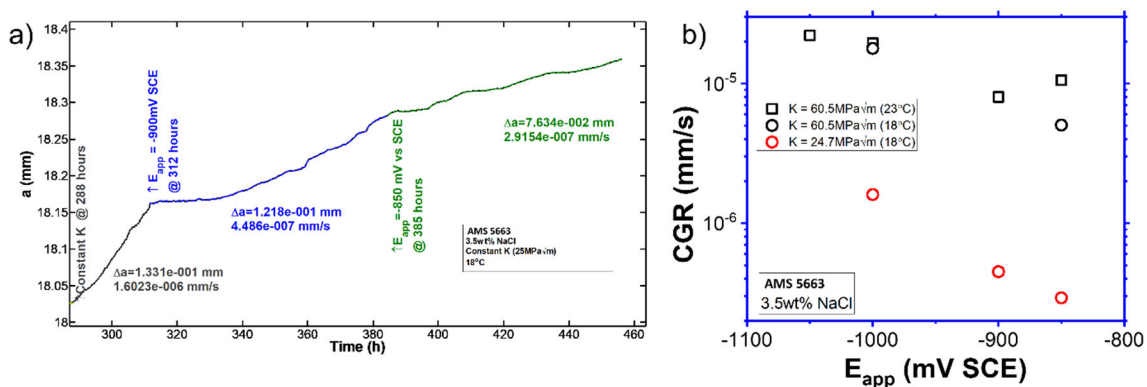


Figure 30 – (a) Resulting crack length, a , in mm, vs time, t , in hours, and (b) calculated crack growth rates, in mm/s, for AMS 5663 material under constant-K conditions.

A 10^{-5} mm/s ($3.9 \cdot 10^{-7}$ in/s) crack growth rate is considered significant because it corresponds to an unsustainable crack growth rate of $3.6 \cdot 10^{-2}$ mm/hour ($1.4 \cdot 10^{-3}$ in/hour), which would clearly cause fastener

failure quickly for fasteners with an outer diameter in the range 12.7 and 57.1 mm. This only represents an initial assessment as the applied K in fastened joint is expected to vary depending on the joint characteristics.

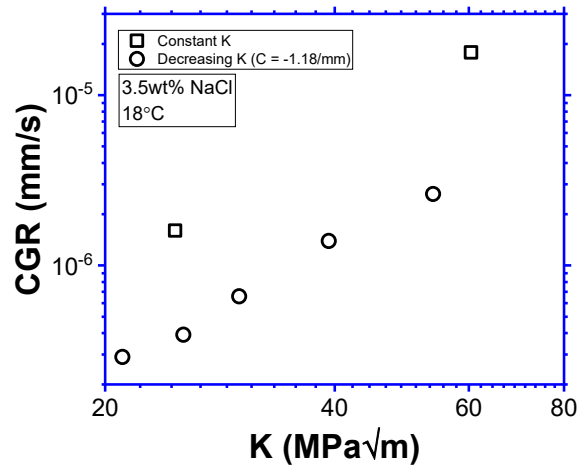


Figure 31 – Crack growth rates, in mm/s, as a function of K for constant-K testing and for decreasing-K.

Figure 31 shows additional results for AMS 5663 material when the value of applied stress intensity is slowly decreased to assess the crack growth rates for a growing crack in a fastener that is progressively shedding load. The applied potential is -1.0 VSCE. The crack growth rates are significantly reduced, on average by a factor of 3X-4X, but remain higher than 10⁻⁶ mm/s.

Material with Cold Working

Figure 32 a) shows the crack length, *a*, as a function of time for the AMS 5962 material for an applied potential starting at +300 mVSCE, the potential was changed in the next phases of the test, and Figure 6 b) shows the results for negative potentials varying between -1050 mVSCE and -850 mVSCE. The applied K is 60.5 MPa√m for all potentials shown, and a change in K is applied at +300 mVSCE. The crack growth rate measured at 60.5 MPa√m and at +300 mVSCE is significant, above 10⁻⁶ mm/s, and remains nearly constant even when the applied K is reduced continuously down to a value of 39.6 MPa√m, indicating the crack growth rates measured at this potential are nearly independent of applied K. Figure 6 b) shows the crack length vs time recording for the case of applied negative potentials. The crack growth rates measured are above 10⁻⁶ mm/s in all cases and a dependence on applied potential is evident starting at -900 mV when the measured rates start reducing significantly.

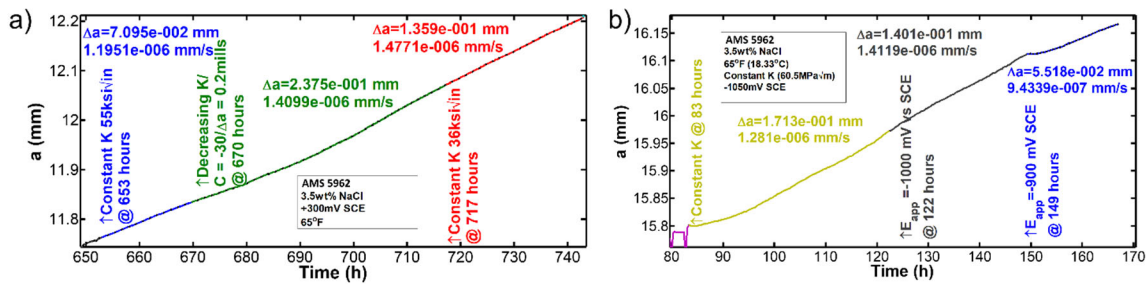


Figure 32 – Resulting crack length, a , in mm, vs time, t , in hours, for AMS 5962 material under constant-K conditions for (a) an applied potential of +300 mVSCE, and (b) for a range of potentials between -1050 mVSCE and -850 mVSCE.

Figure 33 a) compares the crack growth rates of the AMS 5663 and AMS 5962 specifications of UNS N07718. A reference point for material produced according to the API 6ACRA specification is also provided. The crack growth rates for the AMS 5962 specification are almost two orders of magnitude lower than the AMS 5663 specification at the same level of K and potential. While both AMS 5663 and AMS 5962 material exhibit significant crack growth rates that are several orders of magnitude higher than the growth rates for API 6ACRA material, it is interesting to note that the application of cold work can impact the EAC response under the same nominal conditions. Figure 33 b) compares the fatigue crack growth rates of both materials at -1050 mVSCE for an applied $K_{max} = 60.5$ MPa√m and $R = 0.5$. The fatigue crack growth rates confirm the significant difference between the two materials; however they also show that their difference progressively increases as the frequency is reduced. At 10^{-4} Hz the fatigue crack growth rate for AMS 5663 is more than 1 order of magnitude higher.

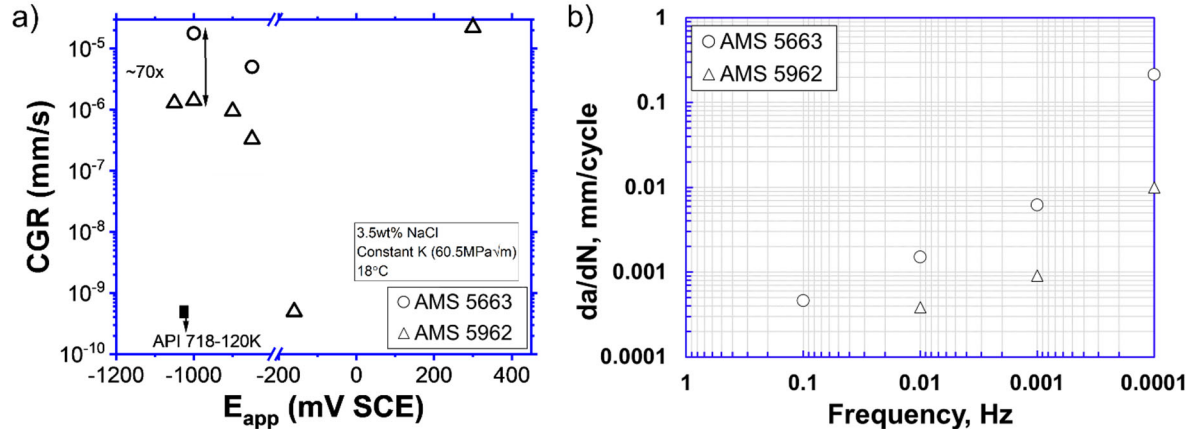


Figure 33 – a) Crack growth rate vs. applied potential for AMS 5663 and AMS 5962 from constant-K testing at 60.5 MPa√m, and b) fatigue crack growth rates as a function of frequency for both materials at -1050 mVSCE, $K_{max}=60.5$ MPa√m and $R=0.5$.

The results in this work highlight the sensitivity of PH nickel alloys to thermo-mechanical treatment in relation to EAC behavior. The two materials tested in this work are produced according to the same specification, AMS 5662, and have chemistry conforming to it. We heat treated the two materials to follow the same two-step heat treatment and investigated the differences related to their production method, where the very high strength AMS 5962 material was cold drawn, and the lower strength AMS 5663 material was hot rolled.

The results highlight a counterintuitive trend: the highest strength material outperforms the lower strength one, with crack growth rates under the same driving force being almost 2 orders of magnitude

lower for AMS 5962 compared to AMS 5663. Testing on a different material batch indicates that a high level of scatter is possible, however the scatter is lower than the systematic $\sim 70X$ difference obtained for crack growth rates at the same nominal conditions of AMS 5663 and 5962.

Figure 34 shows a comparison of a number of the results that illustrate the differences among UNS N07718 specifications [43, 80, 81]. All of the crack growth rates obtained for AMS 5663 and 5962 are measured in this work, and refer to stress intensity levels of $60.5 \text{ MPa}\sqrt{\text{m}}$. All other data points indicate the API 6ACRA specification grade: 120K or 150K, followed by the applied K, ranging between $66 \text{ MPa}\sqrt{\text{m}}$ and $100 \text{ MPa}\sqrt{\text{m}}$. The API 6ACRA material is typically tested at K levels higher than the K tested in this work for AMS 5663 and AMS 5962 to show measurable crack growth rates with the experimental technique in this work. The crack growth rates for the API 6ACRA material are $\sim 225X$ lower than the crack growth rates measured for the AMS 5962 specification at -1050 mVSCE when comparing an applied K of $70 \text{ MPa}\sqrt{\text{m}}$ for API 6ACRA and $60.5 \text{ MPa}\sqrt{\text{m}}$ for AMS 5962. At the same applied K of $60.5 \text{ MPa}\sqrt{\text{m}}$, the difference between the API 6ACRA and AMS 5962 specifications is expected to be even greater. The measured crack growth rates for both the AMS 5962 and AMS 5663 materials are significantly higher when compared to the specifically designed specification for marine environments, API 6ACRA, for all potentials and K conditions in Figure 34.

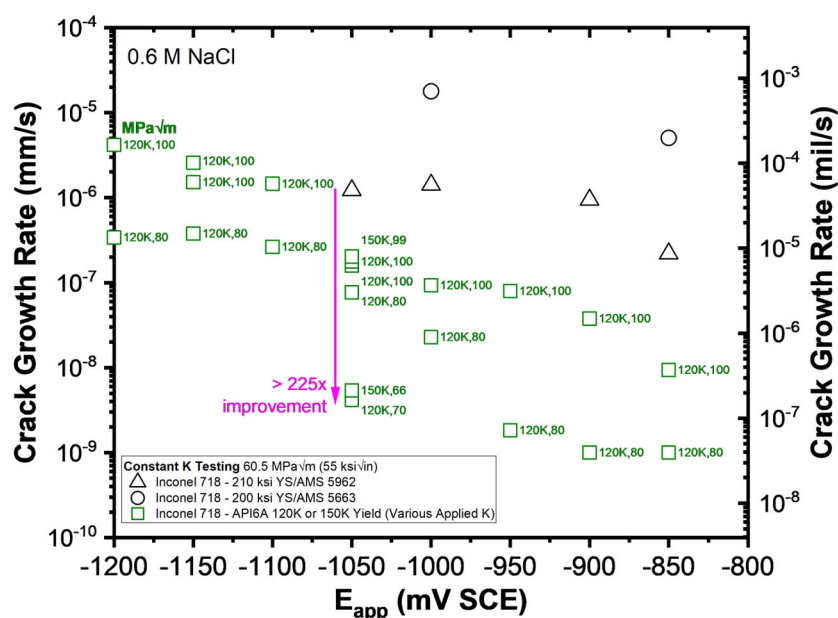


Figure 34 – Summary of constant-K testing for UNS N07718 material produced according to API 6ACRA, AMS 5663, and AMS 5962 specifications.

Conclusions

We compared two different high strength specifications of UNS N07718 material and evaluated their relative EAC behavior under varying levels of applied stress intensity factor K and applied potentials. The results highlight that a cold working step prior to the heat treatment of the material to be of significant importance in reducing the measured crack growth rates under polarization. Measured rates for both materials are significantly higher than specifically designed specifications for marine environments, like API 6ACRA.

ENVIRONMENTALLY ASSISTED CRACKING ASSESSMENT OF NI-CR-MO ALLOYS AND NI-CR-MO-CO ALLOY MP35N UNDER CATHODIC POLARIZATION

In this section we present the experimental data collected for Ni-Cr-Mo alloys to compare their relative resistance to environmental assisted cracking. The experimental data are a collection of results with sometimes nominally different conditions, however relative trends are still observed. Most of the data are fatigue crack growth rates at low frequencies and some data are available for fixed maximum stress intensity, K_{max} .

UNS N07725 and UNS N09945

The same technique presented in the previous section of this report is used to collect the experimental data. The tests are performed in a closed environmental cell, as shown in Figure 29; the sample is loaded in a servo electric frame with 10,000 lbs capacity. The test solution is refreshed periodically to ensure consistent environmental conditions. The sample is connected to a potentiostat through an electrochemical circuit with a reference and counter electrode. The crack length is monitored by the DCPD method, as previously described. The tests are typically performed by starting a fatigue cycle with K_{min} equal to $\frac{1}{2} K_{max}$ to initiate crack propagation. The tests start at a frequency of 0.1 Hz, and the frequency is gradually reduced to 0.001 Hz. The rising portion of the cycle is always 9/10 of the entire cycle time, while the falling portion is 1/10 of the total time. The last fatigue cycle includes a hold for 9,000 s at the K_{max} , for an effective frequency of 10^{-4} Hz.

The composition and mechanical properties of each material tested is shown in Table 9.

Table 9 – Range of compositions (wt%) of Ni-Cr-Mo materials tested under EAC conditions.

	Ni	Cr	Fe	Nb	Mo	Ti	Al	Cu	UTS (ksi)	0.2% YS (ksi)	Elong (%)
UNS N07718 120 API	52.9	18.4	18.7	4.90	2.89	0.97	0.44		177	136	27
UNS N07718 150 API	53.1	18.5	18.2	5.08	3.04	0.95	0.55		186	161	27
UNS N07718 5663	53.84	17.78	18.54	5.11	2.90	0.90	0.60	0.03	202	165	21
UNS N07718 5962	53.35	18.63	18.16	5.03	2.84	0.98	0.55	0.06	229	217	10
UNS N07725	58.02	20.65	7.35	3.62	8.01	1.62	0.20	0.03	180	137	31
UNS N09945	53.6	20.6	14.74	3.98	3.18	1.47	0.12	1.96	181.5	157.3	24.7

The results for UNS N09945 material, named “945X”, are shown Figure 35. The testing was performed for two different K_{max} levels, the results show generally small differences with respect to applied K . There is considerable scatter, particularly at the highest frequency of 0.1 Hz, while the results at the other frequencies are within the same order of magnitude. There is no significant increase in crack growth rates for decreasing frequency.

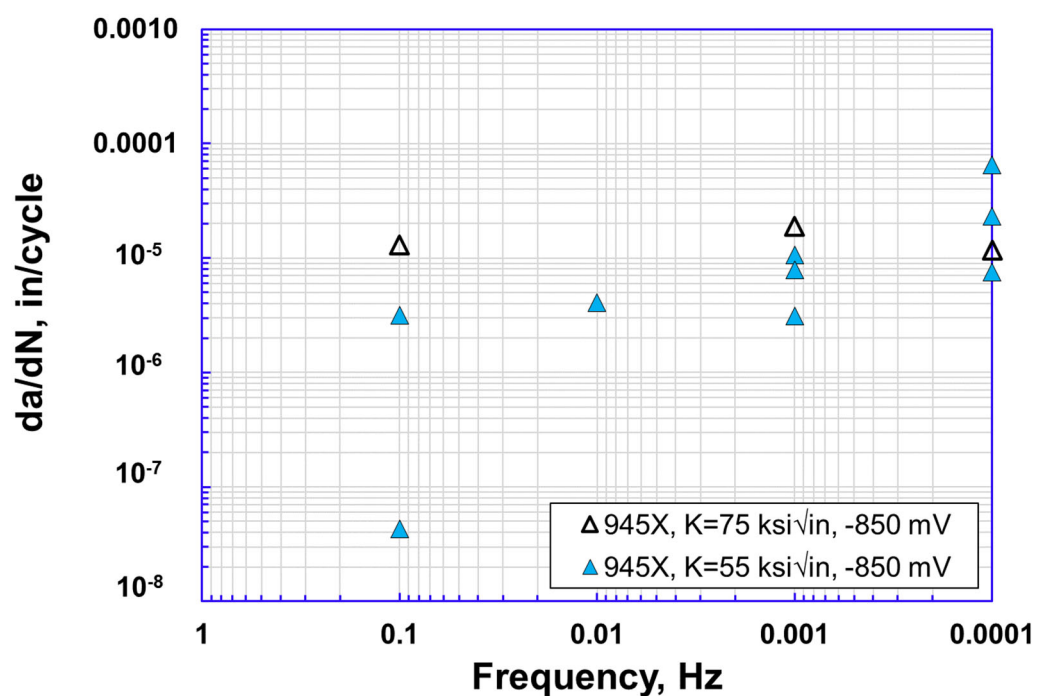


Figure 35 – Summary of fatigue test results for Ni-Cr-Mo alloy 945X for an applied potential of -850 mV vs Ag/AgCl.

The results for UNS N07725, named “725”, are shown in Figure 36 and directly compared to the results for UNS N09945. The applied potential for this material is -1.0 V vs Ag/AgCl and the crack growth rates are higher than the results for UNS N09945 at -850 mV vs Ag/AgCl. Alloy N07725 only shows a modest increase in crack growth rates for decreasing frequency, possibly within experimental scatter.

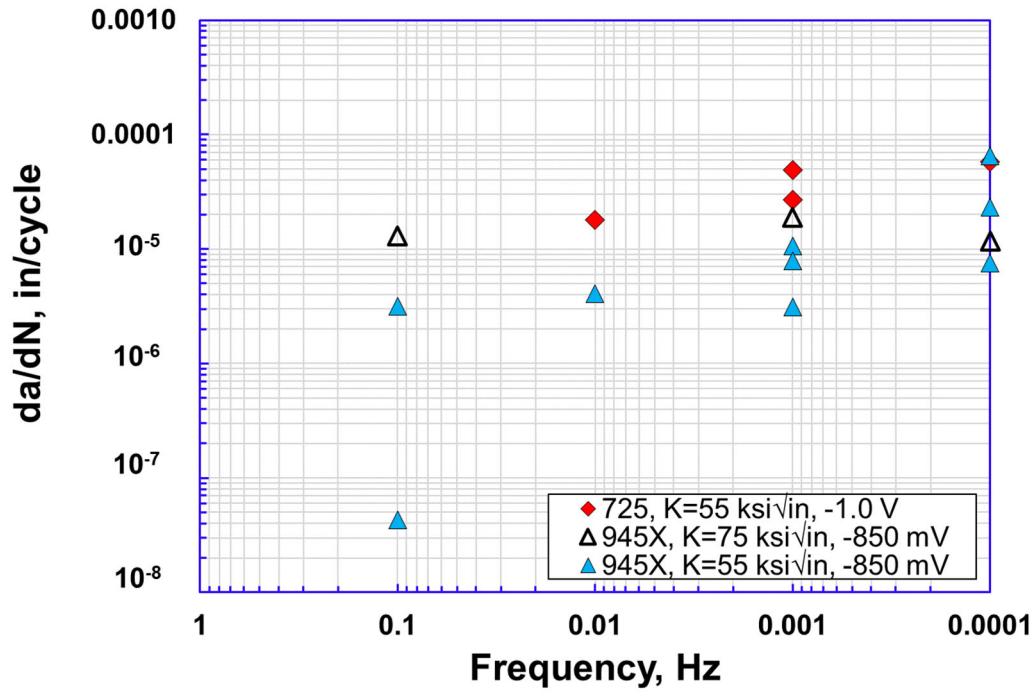


Figure 36 – Summary of fatigue test results for Ni-Cr-Mo alloy 725 and compared to 945X for an applied potential of -1.0 V vs Ag/AgCl.

A comparison with data from the literature for UNS N07718 in the API 6A CRA condition, for a minimum yield of 120 ksi [84], and for the AMS 5663 and AMS 5962 experimental data presented in the previous section, is shown in Figure 37. All data for UNS N07718 are obtained for an applied potential of -1.0 V vs Ag/AgCl. The relative comparison shows that AMS 5663 material shows the highest crack growth rates, the AMS 5962 and UNS N07725 materials show comparable behavior under the same conditions. The UNS N07718 material produced according to the API specification shows among the lowest growth rates despite the higher applied K_{max} . There is a trend of increasing crack growth rates as a function of decreasing frequency. The most robust trend is for AMS 5663 and for 5962, while generally a modest increase is shown by N07718 API 6A material, and N07725.

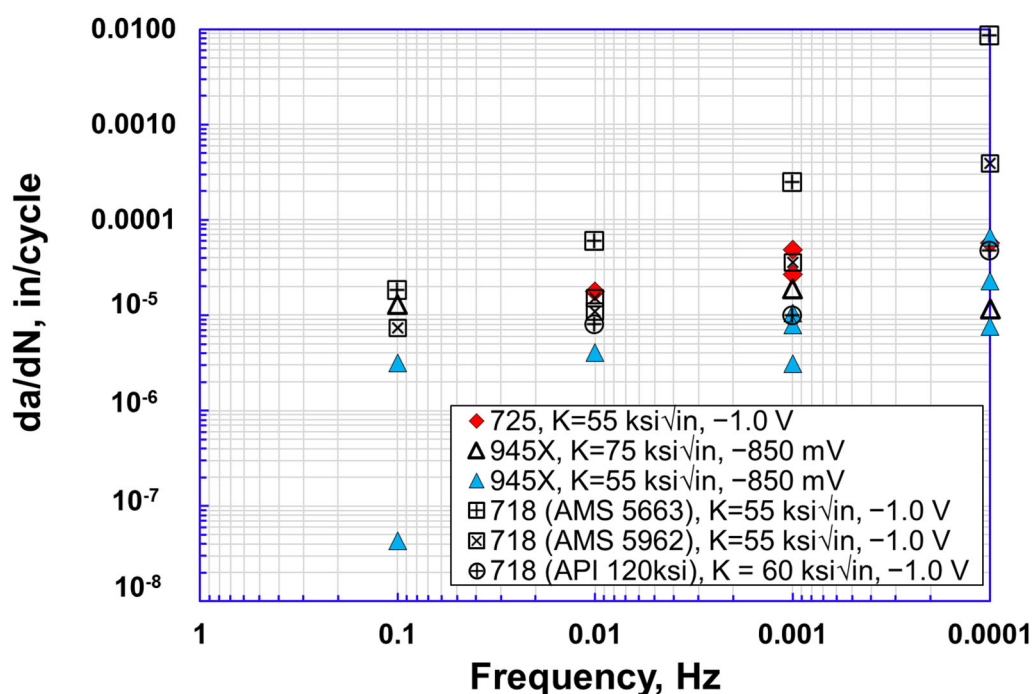


Figure 37 – Comparison of fatigue test results for Ni-Cr-Mo alloy 725 and 945X to 718 material procured according to different specifications.

UNS R30035

The Ni-Cr-Mo-Co alloy UNS R30035 was also tested under similar conditions to compare the fatigue crack growth behavior with the previous set of alloys. Its composition is shown in Table 10 for reference.

Table 10 – Range of compositions (wt%) of Ni-Cr-Mo-Co alloy MP35N tested under EAC conditions.

	Ni	Cr	Fe	Mo	Ti	Co	UTS (ksi)	0.2 YS (ksi)	Elong. (%)
MP35N	35.7	20.2	0.09	9.69	0.74	BAL (30.55)	213	195	18

* Remainder

The testing was performed at a significantly higher $K_{\max}$ than all previous testing as the material was known to be highly resistant to EAC. No test was performed as a direct comparison with the previous set of Ni-Cr-Mo alloys. The fatigue crack growth rates at -850 mV vs Ag/AgCl and under open circuit potential (OCP) are similar. There is no significant increase in crack growth rates for decreasing frequency after 0.01 Hz.

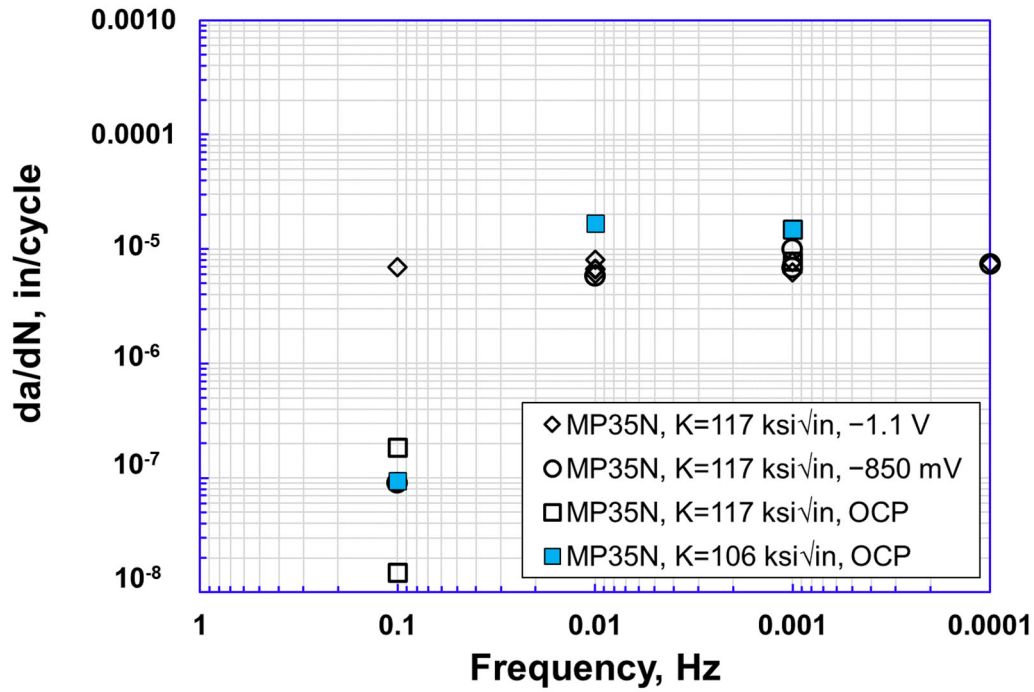


Figure 38 – Summary of fatigue test results for Ni-Cr-Co-Mo alloy MP35N for an applied potential of -850 mV vs Ag/AgCl and for Open Circuit Potential (OCP).

Summary

Figure 39 – Summary of constant-K testing vs potential for MP35N (UNS R30035), 945X (UNS N09945), and 718 (UNS N07718) material produced according to API 6ACRA, AMS 5663, and AMS 5962 specifications a) at cathodic protection potentials and at open circuit (MP35N) or applied potential of 300 mV_{SCE} (Inconel 718 AMS5962). Figure 39 summarizes the constant K crack growth rate of each alloy tested. MP35N was also tested at 117 ksi√in and -1 V_{SCE} for 1200 hours and did not exhibit detectable crack growth. MP35N exhibited relatively high crack growth at OCP (approximately 200 mV_{SCE}) suggesting susceptibility to stress corrosion cracking mechanisms during long term seawater exposure.

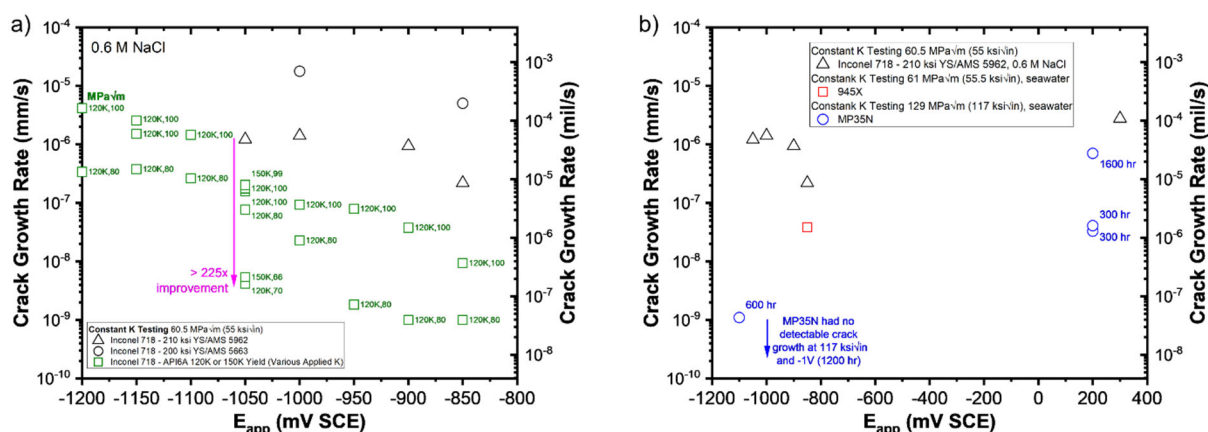


Figure 39 – Summary of constant-K testing vs potential for MP35N (UNS R30035), 945X (UNS N09945), and 718 (UNS N07718) material produced according to API 6ACRA, AMS 5663, and AMS 5962 specifications a) at cathodic protection potentials and at open circuit (MP35N) or applied potential of 300 mV_{SCE} (Inconel 718 AMS5962).

STRESS CORROSION CRACKING IN ALTERNATE IMMERSION AND OPEN CIRCUIT POTENTIAL

In this section a set of structural materials for fastener applications is tested under alternate immersion conditions to verify and rank their EAC behavior. A novel measurement technique is presented and validated.

Introduction and Motivation

Stress Corrosion Cracking is most commonly evaluated by simulated testing in the laboratory or by direct environmental exposure of precracked and pre-loaded samples (e.g., ASTM E1681 [61]). Laboratory testing allows the use of crack monitoring techniques such as sample compliance or potential drop to measure crack growth rates. The latter method typically requires a dedicated testing machine and data acquisition system for each test, limiting the sample throughput and the ability to characterize large sample sets, e.g., sample to sample and batch to batch variability. Testing of precracked and pre-loaded samples allows for high throughput testing to identify SCC thresholds, but typically without the ability to monitor the progression of the crack at the same fidelity as laboratory testing.

The main focus of this testing program is the SCC behavior of high strength materials, particularly steel and nickel alloys, typically used as fastener materials in marine applications under alternating immersion conditions to evaluate their KIEAC. This paper discusses a new testing methodology that uses bolt loaded compact test geometries with a strain gauge positioned at the back-face to allow a large sample throughput and sensitive monitoring of crack progression suitable for a wide array of environmental exposure conditions, such as atmospheric exposure, simulated sea spray, and alternate or full immersion conditions. The crack length was constantly monitored while the samples were in test and statically loaded by a tightened bolt of the same material to reduce galvanic effects. As the SCC crack progresses, the Stress Intensity Factor (SIF), K , decreases, yielding lower growth rates until a KIEAC condition is achieved. The importance of direct exposure with continuous monitoring also lies in the improved understanding of SCC behavior, with information on thresholds, incubation periods and growth rates.

The list of materials tested in this study is shown in Table 11. The list encompasses a set of materials typically used in marine applications: Ni-based alloys UNS N05500 (Monel K-500) and UNS N07718, both 718 API 6A and 718 AMS 5664 specifications, Ni-Co alloy UNS R30035 (MP-35N), Fe-Ni-Cr alloy UNS S66286 (A286), a high-strength and high-toughness low alloy steel UNS K91973 (Ferrium M54), and a

high-strength low alloy steel A722, for pre-stressed concrete applications. The yield strengths range between 90 and 250 ksi (621 and 1724 MPa).

Table 11 – List of materials tested.

Material	Specification	Yield Strength, 0.2%
UNS K91973	NA: Equivalent to AMS 6516 alloys	250 ksi (1724 MPa)
UNS R30035	AMS 5844	170 ksi (1172 MPa)
A722	ASTM A722	120 ksi (827 MPa)
UNS N05500	QQ-N-286	90 ksi (621 MPa)
UNS N07718 – 5664	Navy Drawing 7329673, AMS 5664	150 ksi (1034 MPa)
UNS N07718 – API6A	API 6A	140 ksi (965 MPa)
UNS S66286	AMS 5737	95 ksi (655 MPa)

The sample geometry is based on a typical fracture mechanics specimen, with a straight crack front progressing along the mid-plane of the sample: the geometry is similar to a compact tension C(T) specimen, with longer width and different H/W ratio [85]. This geometry is referred in some literature as a wedge opening load (WOL) specimen, as earlier work used wedges between the crack faces to apply a desired crack opening displacement (COD) [86]. The geometry is named bolt-loaded CT specimen per ASTM E1681. The sample geometry is shown in Figure 40. Considering the differences in strengths, three different values of width, W, and thickness, B, are used, depending on the material, to comply with ASTM standard and ensure proper constraints at the crack tip for all levels of K tested.

All samples are extracted from bar stock form, as it is more representative of the typical form used for fasteners. All bolts and pins are made of the same material and with their longitudinal axis oriented along the longitudinal bar direction. The samples are oriented in the L-R direction. Because of its very high strength, the dimensions of the sample for UNS K91973 are a width of 1.255 inches (31.9 mm) and a thickness of 0.5 inches (12.7 mm); the sample is extracted from a 2.40 inches (61 mm) diameter bar. A 1/4-20 bolt is used with this geometry. A 3.5 inches (89 mm) diameter bar of UNS R30035 material is used to excise samples 2.51 inches (63.8 mm) wide and 1 inch (25.4 mm) thick; a bar stock of 3.0 inches (76.2 mm) in diameter is used for A722 steel for the same final geometry. A 1/2-20 bolt is used for this geometry. Bar stock of 3.75 inches (95.2 mm), 4.25 inches (108 mm), and 4.00 inches (101.6 mm) in diameter are respectively used for, UNS N05500, UNS N07718 API6A, UNS N07718 AMS 5664, and UNS S66286; the samples are 3.1375 inches (79.7 mm) wide and 1.25 inches (31.8 mm) thick. A 5/8-18 bolt is used for this geometry.

All specimens were fatigue precracked according to ASTM E647 guidelines and using the load shedding method [82]. The fatigue precrack length was approximately 0.5 W for all samples. A final K_{max} lower than any of the applied K at the beginning of the test was reached during precracking per ASTM standard guidelines. All materials were tested in replicates of 3 samples at the same level of K. Some materials that showed cracking were tested at different K conditions to augment the da/dt vs K data collected. The final precracking levels and the initially applied K levels are shown in Table 12.

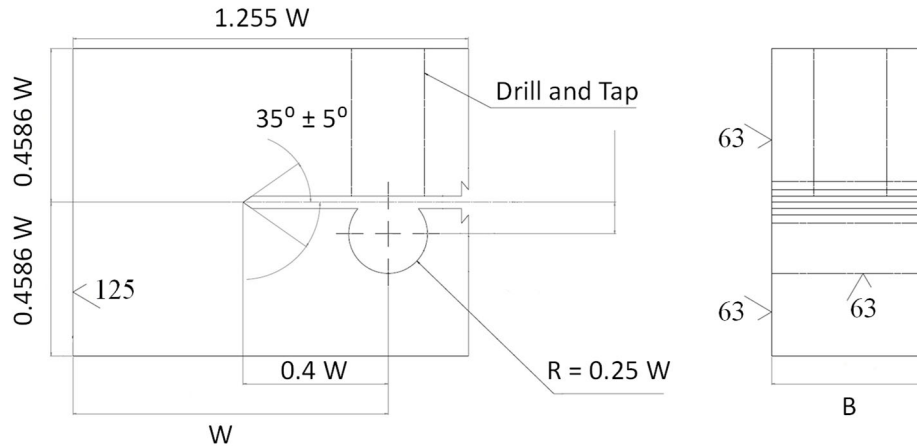


Figure 40 – Bolt-loaded SCC sample geometry.

The expected K for a crack in a fastener in service is estimated by using the SIF solution for a fastener with major diameter between 0.5 inches and 2.25 inches (12.7 and 57.1 mm). We assume that a thumbnail crack forms at the thread root of the fastener and that $2/3$ of the yield strength is the applied preload. The crack depth is conservatively assumed to be $1/16$ inches (1.6 mm). Mettu et al. published a general SIF solution for cracks in threaded fasteners with specific SIF solutions for standard aerospace bolt sizes [83]. The equations also allow for sizes that are applicable to other industrial applications and for the sizes in this work. Based on this SIF solution, an initial assessment of SIF conditions reveals a range of K between 35 and 74 $\text{ksi}\sqrt{\text{in}}$ (38.5-81.3 $\text{MPa}\sqrt{\text{m}}$) depending on material and strength of the fastener. For all materials in this investigation, no data are available to verify whether SCC is expected within this range of K . Specifically, no K_{Isc} data exist for alternate immersion in natural seawater. Based on these calculations and the yield strengths of the materials tested in this effort, the initial conditions for testing are shown in Table 12. The data are obtained directly from the initial measurements of K , as will be described in the following sections.

Table 12 – List of starting stress intensity factors for all samples tested in phase 1 and 2.

Material	Sample Name	Final K _{max} Fatigue Precracking	Initial K	Final K _{max} Fatigue Precracking	Initial K
		(ksi $\sqrt{\text{in}}$)	(ksi $\sqrt{\text{in}}$)	(MPa $\sqrt{\text{m}}$)	(MPa $\sqrt{\text{m}}$)
UNS K91973	S4	36.0	60.9	39.6	66.9
	S5	36.0	60.4	39.6	66.4
	S6	36.0	64.2	39.6	70.5
	S7	39.0	80.1	42.9	88.0
	S8	39.0	80.2	42.9	88.1
	S9	39.0	80.4	42.9	88.3
UNS R30035	S1	25.0	123.5	27.5	135.7
	S2	25.0	123.5	27.5	135.7
	S3	25.0	123.9	27.5	136.1
A722	S6	33.0	57.1	36.3	62.7
	S7	39.0	66.1	42.9	72.6
	S8	39.0	65.2	42.9	71.6
	S9	39.0	65.4	42.9	71.9
UNS N05500	S1	17.0	68.1	18.7	74.8
	S2	17.0	68.0	18.7	74.7
	S3	17.0	67.5	18.7	74.2
UNS N07718 718 – API6A	S1	23.0	92.9	25.3	102.1
	S2	23.0	91.4	25.3	100.4
	S3	23.0	92.1	25.3	101.2
UNS N07718 718 – 5664	S1	27.0	106.0	29.7	116.5
	S2	27.0	104.9	29.7	115.3
	S3	27.0	106.1	29.7	116.6
UNS S66286	S3	21.0	84.2	23.1	92.5
	S5	21.0	85.3	23.1	93.7
	S6	21.0	84.9	23.1	93.3
	S7	21.0	85.5	23.1	94.0

The initial load is applied by tightening of the bolt. The more the bolt is tightened the more the crack mouth is opened, increasing the SIF applied to the fatigue crack. A clip gauge is used to monitor the applied bolt pre-load allowing a real-time calculation of applied K, as shown in Figure 41. A sample of UNS R30035 material assembled with its machined bolt and flattened pin is also shown prior to being instrumented with the back-face strain gauge.

The correlation between measured CMOD and back-face strain is calculated at the beginning of the test by a developed code that records both the clip gauge and the back-face strain signals. Once the target K is reached the clip-gauge is removed and the sample is deployed in the desired environment. The strain is continuously monitored throughout the test by a quarter bridge configuration and a National Instrument (NI) acquisition board and chassis. Data is collected periodically every 10 minutes and at the beginning and end of each immersion cycle. The change in back-face strain as a function of crack length is obtained using numerical analysis (as shown later). During exposure, if a change in crack length occurs, the applied load decreases as a function of crack length. By knowing the initial conditions of the sample, namely the initial crack mouth opening displacement (CMOD), initial K, and crack length, an updated measure of crack length can be obtained from the value of back-face strain.

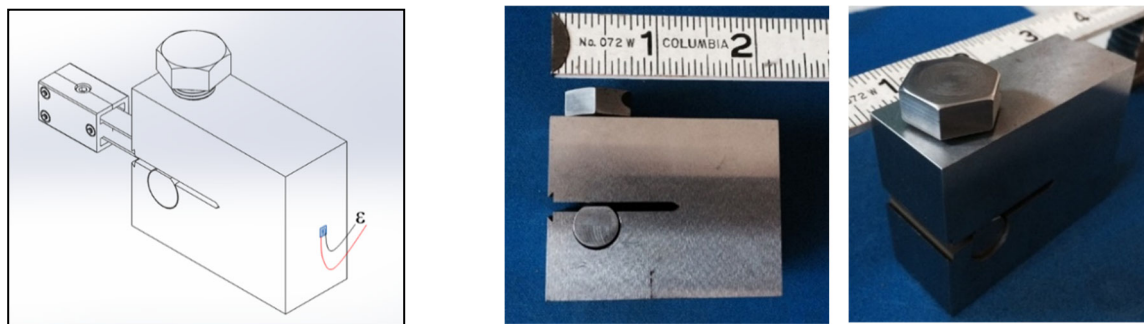


Figure 41 – Schematic of geometry and loading methods with monitoring of back-face strain and SCC sample assembled with bolt and flattened pin before being instrumented with back-face strain gauge.

The loading curve is recorded using a custom-developed program that serves as monitor during loading to achieve the desired K level. The CMOD information is collected and regressed in real time to allow the operator, tightening the bolt with a torque wrench, to see the progress in loading the sample and stop at the desired maximum K. The recorded CMOD vs time data for a UNS K91973 sample tested at an initial K of 80 ksi $\sqrt{\text{in}}$ (87.9 MPa $\sqrt{\text{m}}$) is shown in Figure 42. The final value of CMOD is used for all further calculations of crack length and estimated applied load, as it is kept constant by the tightened bolt.

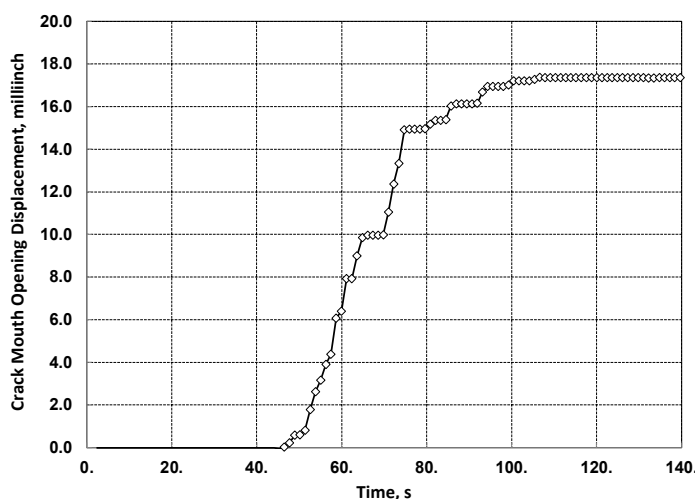


Figure 42 – Recorded data with clip gauge applied at the mouth of the crack from loading of the sample before exposure to alternate immersion.

The instrumented samples are prepared according to a validated procedure for exposure protection of strain gauges to extreme environments: the back face of the samples is sanded with 320 grit silicon-carbide paper in horizontal, vertical and 45 degree directions, the surface is then wetted with a phosphoric-acid compound that acts as a mild etchant and accelerates the cleaning process, followed by neutralization step that is used to stop any chemical reaction introduced by the conditioner and to produce an optimum surface for the strain gauge adhesives. The surface is then prepared with tape to allow only a small area, just larger than the size of the strain gauge to bond gauge and metal. A two-part epoxy bonding agent was used to bond the strain gauge to the back-face of the sample. After curing, the tape is removed, and a layer of nitrile-rubber coating is applied to both the gauge and approximately to about 1 inch length of the vinyl-insulated lead wires to improve bonding with the protective polysulfide coating layer. This last protective layer is applied to the entire back-face of the sample, including approximately 1 inch length of the insulated lead wires, to form approximately a 0.5-inch-thick layer of flexible rubbery coating. Figure 43 (left) shows the

final phase of the preparation of the samples with the polysulfide coating applied to the samples, and the strain gauge wire spools. Additionally, each sample geometry is equipped with a self-adhesive K-type thermocouple, to allow for temperature correction of the strain signal. A fourth thermocouple is used for environmental monitoring of trough temperature during the test. This coating system proved successful for UNS K91973 and A722 steel for 3,000 hours of exposure, and for all other alloys for over 10,000 hours.

Alternate Immersion Exposure to Unfiltered Natural Seawater

The exposure to unfiltered natural seawater in alternate immersion is performed at the Naval Research Laboratory in Key West, FL testing site. Exposure troughs are constantly supplied with a flow of seawater in an open loop configuration. The wet and dry cycles alternate all day, 6 times a day, and the wet cycle is approximately 15 minutes long. Figure 43 (center) shows the exposure location: the trough is shown on the left and all strain gauge wires are connected to a NEMA box with electric terminals on the side of the trough. The exposure trough is equipped with two PVC water outlet pipes that function as water level adjusters for the tank. The first sets the lowest level of water 10 inches (254 mm) below the samples, Figure 43 (center), and is connected to a valve that automatically opens and closes to allow the water level to rise towards the second outlet. The second level is high enough that all samples are immersed in seawater up to and not touching the bolt, therefore exposing the fatigue precrack and the SCC crack to brief periods of seawater immersion, as shown in Figure 43 (right).



Figure 43 – Instrumented samples showing polysulfide coating protecting the strain gauge for long term exposure. Example photographs of the dry and wet cycle showing the maximum water reached the precrack.

Once the sample is loaded and exposed to the environment, a custom developed program monitors all samples and records any variations of back-face strain during exposure. This information is then used to calculate the state of the sample in terms of crack length and applied load, therefore making use of detected changes in compliance.

The C(T) specimen is one of the most widely used geometries to determine fracture properties and measure fatigue-crack-growth rates in metallic materials. The earliest BFS relation was obtained using a two-dimensional finite-element method [87]. The expected negative elastic strain per unit load obtained at the back-face of the C(T) specimen is correlated with the crack-length-to-width (a/W) ratio. The normalized factor is typically $|E(\epsilon W)B/P|$, where the applied load, P , the width and thickness of the sample, W and B , and elastic modulus of the material, E , are included. Several other studies progressively improved upon the developed solution and extended its applicability to a larger range of a/W ; the latest development extended its use to an a/W ratio of 0.95 [88-90].

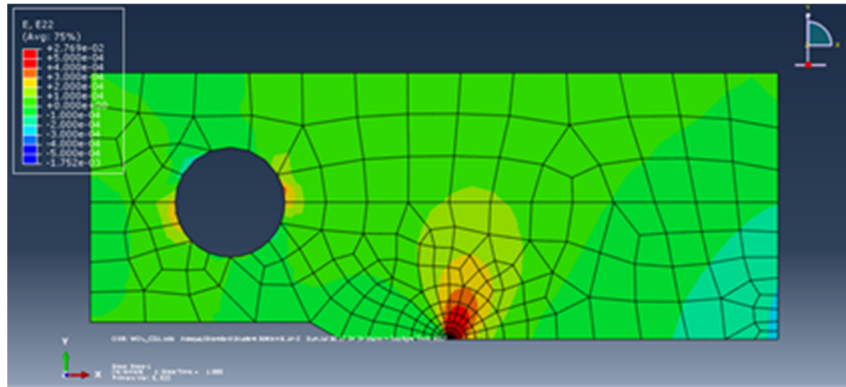


Figure 44 – FEM model of WOL sample: strain in the y-direction is shown.

Despite the extensive literature available for C(T) geometries, a solution for the WOL geometry is not available; although the two geometries are similar, they are not identical with the main differences in the planar dimensions being a height of $1.2W$ for the C(T) sample and a $0.972W$ for the WOL geometry, and a full sample width of $1.25W$ for C(T) and 1.255 for WOL. A solution for the WOL sample is developed in this work to allow the monitoring of compliance using a BFS approach. The Finite Element Method (FEM) is used to model the geometry, loads, and boundary conditions and to calculate the strain.

The geometry is modeled with 2D plane stress elements, and it is symmetric with respect to the height of the sample i.e., the y-direction in Figure 44. The loading is applied as an evenly distributed bearing load at the location where the bolt applies the opening pressure. The crack tip is modeled using special crack tip elements. The $1/\sqrt{r}$ singularity can be obtained in a finite element mesh with a ring of collapsed quadrilateral elements by using second order elements. Three collapsed nodes at one edge of the element form a wedge, and with the mid-side nodes positioned at $1/4$ point along the two edges that lead to the crack tip, the singularity is properly approximated [91]. The mesh is structured so that a spider-web type conformation of elements around the discontinuity is formed, as shown in Figure 44. The BC consist of symmetry in the y-direction and the crack tip is fixed in both x- and y- directions.

The model is first verified using available solutions for CMOD and SIF from Saxena and Hudak [12]; the results of the validation are shown in Figure 45, with the a/W going from 0.35 to 0.9. Figure 45 (left) shows the normalized CMOD, BEV/P , where B is thickness, E is elastic modulus, v is CMOD, and P is applied load, in log-scale as a function of a/W . The current FEM analysis is within 1% for all cases modeled. Figure 45 (right) shows the SIF expressed in $\text{ksi}\sqrt{\text{in}}$ for the material, geometry, and all the applied loads: the SIF is within 0.5% for all the cases modeled. Note that the SIF is calculated using the contour integral method [92].

Finally, a solution for the back-face strain is developed using numerical results, with the normalized strain in the y-direction shown in Figure 46 (left); note that the strain is compressive or negative and the absolute value of the normalized parameter is shown. The results are presented in terms of $|E(\epsilon W)B/P|$, where the width, W , thickness, B , elastic modulus, E , applied load, P , and back-face strain, ϵ , are regressed as a normalized parameter, as a function of normalized crack length, a/W . The results are once more regressed into the factor U in Equation (18) where the normalized factor $|E(\epsilon W)B/P|$ is used.

$$U = \frac{1}{\sqrt{|E\epsilon WB/P| + 1}} \quad (18)$$

This method allows the U vs a/W relation to be more easily regressed into a polynomial form. The latter is performed using a least square method and the resulting fit, correlation coefficient, and polynomial coefficients are shown in Figure 46 (right).

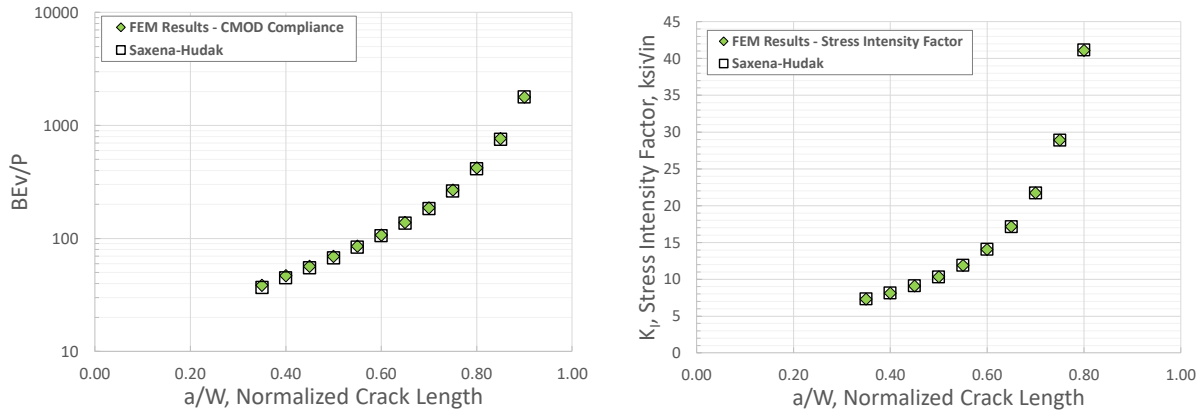


Figure 45 – Verification of current model results for CMOD and SIF with numerical form by Saxena-Hudak.

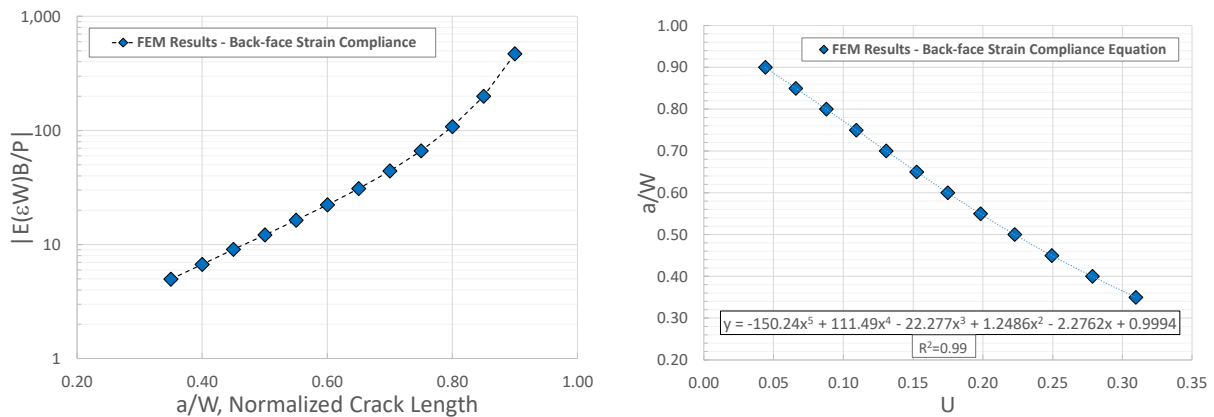


Figure 46 – (left) Numerical results to develop a back-face strain solution for WOL geometry: normalized factor is plotted as a function of normalized crack length and (right) regression to define a polynomial form as conventionally found in literature as a function of the factor U .

The equations developed are implemented into a LabView VI program to regress the information from the loading phase of the test, and from the monitoring of the strain gauge data. During the loading phase of the test, the developed CMOD equations are used in a monitoring program to allow the real-time visualization of the applied K during the tightening of the bolt. The strain signal is used to obtain an estimate of crack length during loading with applied load information obtained from the CMOD data being collected,

and to establish a calibration for the strain-gauge signal. During the monitoring phase of the test, the back-face strain equations are used to regress the strain-gauge information to obtain a measure of crack length and applied load. The solution is obtained by numerical implementation of the system of non-linear equations, formed by the two equations:

$$\frac{BEv}{P} = \left(\frac{1+\alpha}{1-\alpha} \right)^2 (b_0 + b_1\alpha + b_2\alpha^2 + \dots) \quad (19)$$

$$\frac{E\epsilon WB}{P} = \frac{1}{(1-\alpha)^2} (a_0 + a_1\alpha + a_2\alpha^2 + \dots) \quad (20)$$

where α is normalized crack length, a/W , E is elastic modulus, P is applied load, ϵ is back-face strain, v is initial CMOD, W is width, B is thickness, $a_0, a_1, \dots, a_n$ are fitting parameters from data in Figure 45 $b_0, b_1, \dots, b_n$ are fitting parameters from data in Figure 46. The two unknowns, α and P , can be found at every instant in time when ϵ is collected.

Results

Three alloys did not show any SCC: UNS R30035, UNS N05500 and UNS N07718 API6A. All remaining materials showed varying levels of SCC. The data for UNS K91973 steel are presented in a more detailed form, with crack length, a , as a function of time for all samples, to illustrate the capabilities of this experimental technique. Additionally, a detailed tracing of the crack length and K vs time for the first 500 hours of testing is also shown for one of the UNS K91973 samples. The results for all other materials are regressed to obtain crack growth rates, da/dt , as a function of K and are presented in the following section.

Materials Showing Resistance to SCC in Alternate Immersion

UNS R30035 did not show any SCC during the test and the highest applied K that did not cause any fracture for UNS R30035 material was $123.9 \text{ ksi}\sqrt{\text{in}}$ ($136.2 \text{ MPa}\sqrt{\text{m}}$). Similarly, UNS 05500 did not show any SCC and the highest applied K that did not cause any fracture was $68.1 \text{ ksi}\sqrt{\text{in}}$ ($74.8 \text{ MPa}\sqrt{\text{m}}$). UNS 07718 API6A material only showed very early indications of cracking and no additional growth for the remaining exposure. The highest applied K was $88.5 \text{ ksi}\sqrt{\text{in}}$ ($97.3 \text{ MPa}\sqrt{\text{m}}$).

Materials Showing SCC

UNS K91973 steel showed significant stress corrosion cracking in the conditions studied. The normalized crack length vs time data are presented in Figure 47. All tests show exposure periods of different durations prior to crack initiation, and then alternate between phases of quick and slow/no crack growth. Figure 48 shows a detailed view of the progression of the SCC crack for sample S9. The first 500 hours of testing show an initial period of incubation of approximately 150 hours, followed by a phase of rapid growth. Another incubation period of 100 hours is then followed by progressively decreasing rate of growth. The crack growth behavior between 100 and 150 hours is shown in Figure 48 b in further detail. The alternation of wet and dry cycles is also shown, but no correlation between cycles and the incubation times or crack growth rates is directly evident.

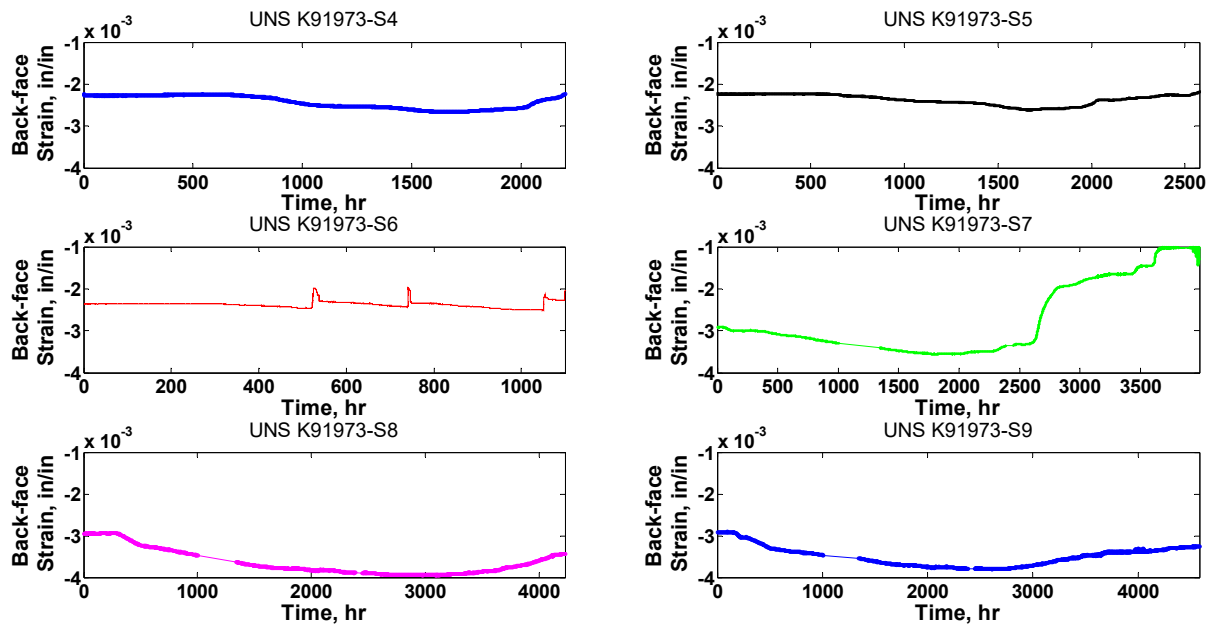


Figure 47 – Normalized crack length vs time for UNS K91973.

The data are regressed using a secant method into a da/dt vs K curve to define the SCC behavior. An average crack growth rate for a 12 hours-time block is calculated by regressing the crack vs time data and retaining the data point if the correlation coefficient of the linear regression is higher than 0.5. The results of the analysis are shown in Figure 49 a). Samples UNS K91973-S4, S5, and S6 started at an applied K of ~ 60 ksi $\sqrt{\text{in}}$ (66 MPa $\sqrt{\text{m}}$), samples S7, S8, and S9 started at an applied K of ~ 80 ksi $\sqrt{\text{in}}$ (88 MPa $\sqrt{\text{m}}$). The crack growth rates are high for the highest K level testing, reaching 10^{-3} in/hr (0.025 mm/hr). For K lower than 70 ksi $\sqrt{\text{in}}$ (77 MPa $\sqrt{\text{m}}$), crack growth rates stabilize around 10^{-4} in/hr (0.0025 mm/hr). The data below 50 ksi $\sqrt{\text{in}}$ (55 MPa $\sqrt{\text{m}}$) is excluded. At this level, the sample continued to crack under the shedding K , however the test was no longer valid. The KIEAC is expected to be lower than 50.5 ksi $\sqrt{\text{in}}$ (55.5 MPa $\sqrt{\text{m}}$).

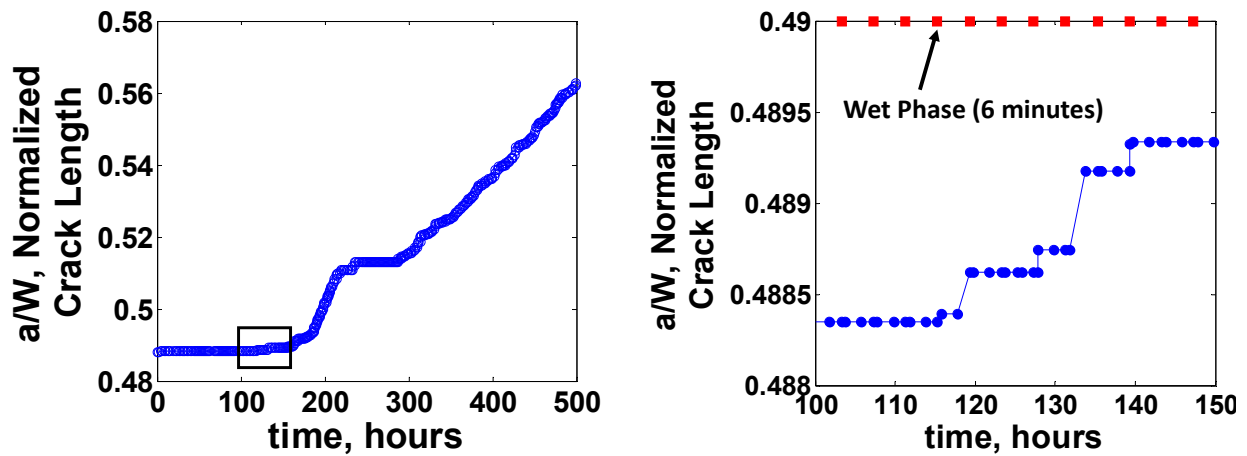


Figure 48 – Crack length vs. time after regression of back-face strain data for UNS K91973 Sample S9 shows progressive growth of SCC crack after incubation periods of no crack growth and no correlation with wet and dry phases (fully wet periods indicated by red boxes).

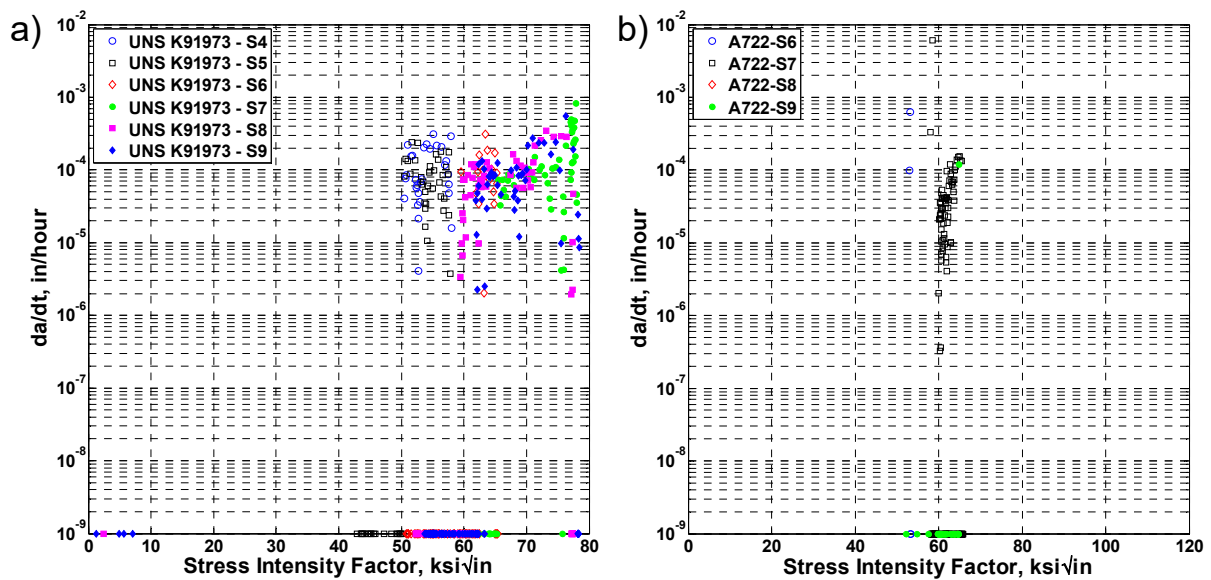


Figure 49 – Crack growth rate vs. stress intensity factor after regression of back-face strain data for a) UNS K91973, and b) A722.

The results for A722 material are presented in Figure 49 b). Samples S7 showed a pronounced increase in crack length at 5635 hours, with a progressive decrease in crack growth rate and final crack arrest at 9000 hours. The crack growth rates for this sample started at high values, above 10^{-4} in/hr (0.0025 mm/hr) and then progressively decreased to 2×10^{-7} in/hr (5×10^{-6} mm/hr), with stress intensity K starting at 65.5 ksi $\sqrt{\text{in}}$ and reducing to 60 ksi $\sqrt{\text{in}}$ at the end of the test (70 to 65.9 MPa $\sqrt{\text{m}}$). Samples S6 and S9 only showed an initial brief increase in crack length, which corresponds to the data points shown in Figure 10 b). Crack arrest occurred after this initial growth event, with no further sign of SCC growth for over 2,000 and 3,000 hours for sample S6 and S9, respectively. Sample S8 did not show any SCC. The highest K level where no fracture was observed for A722 material was 53.0 ksi $\sqrt{\text{in}}$ (58.2 MPa $\sqrt{\text{m}}$).

The results for UNS N07718 AMS 5664 materials are presented in Figure 50 a. Only the results for sample S1 are showing strain recordings consistent with crack growth, while the remaining two samples did not show any indication of SCC. Sample S1 showed SCC for the first 840 hours of testing and then the crack stabilized around an a/W of 0.5463 or a length of 1.366 inches (34.7 mm). The initial K is approximately 106 ksi $\sqrt{\text{in}}$ (116.5 MPa $\sqrt{\text{m}}$) and the initial crack growth rates are higher than 2.0×10^{-3} in/hr (0.05 mm/hr). The crack growth rates progressively decreased to 2×10^{-5} in/hr (5×10^{-4} mm/hr), with stress intensity reducing to 94.7 ksi $\sqrt{\text{in}}$ (104.1 MPa $\sqrt{\text{m}}$) at the end of the test. The decrease in K and growth rates forms a sigmoidal pattern, indicating the reaching of a threshold at this K level.

The results for A286 material presented in Figure 50 b. Sample S3 shows significant SCC growth. The crack length vs time data for sample S3 (not presented) showed long incubation times followed by bursts of crack growth, resulting in a step-wise crack progression. Only a few data points are retained from the regression of crack length vs time data because of the low correlation coefficients obtained in calculating da/dt . The crack growth rates are scattered and dispersed going from 4.0×10^{-3} to 5.0×10^{-5} in/hr between 80 and 65 ksi $\sqrt{\text{in}}$ (from 0.1 to 0.001 mm/hr between 87.9 and 71.4 MPa $\sqrt{\text{m}}$). Samples S6 and S7 showed an initial brief increase in crack length, which corresponds to the da/dt data points shown in Figure 50. Crack arrest occurred after this initial growth event, with no further sign of SCC growth for over 6,000 and 10,000 hours respectively. The K level for A286 material where no fracture was observed was 66.3 ksi $\sqrt{\text{in}}$ (72.9 MPa $\sqrt{\text{m}}$).

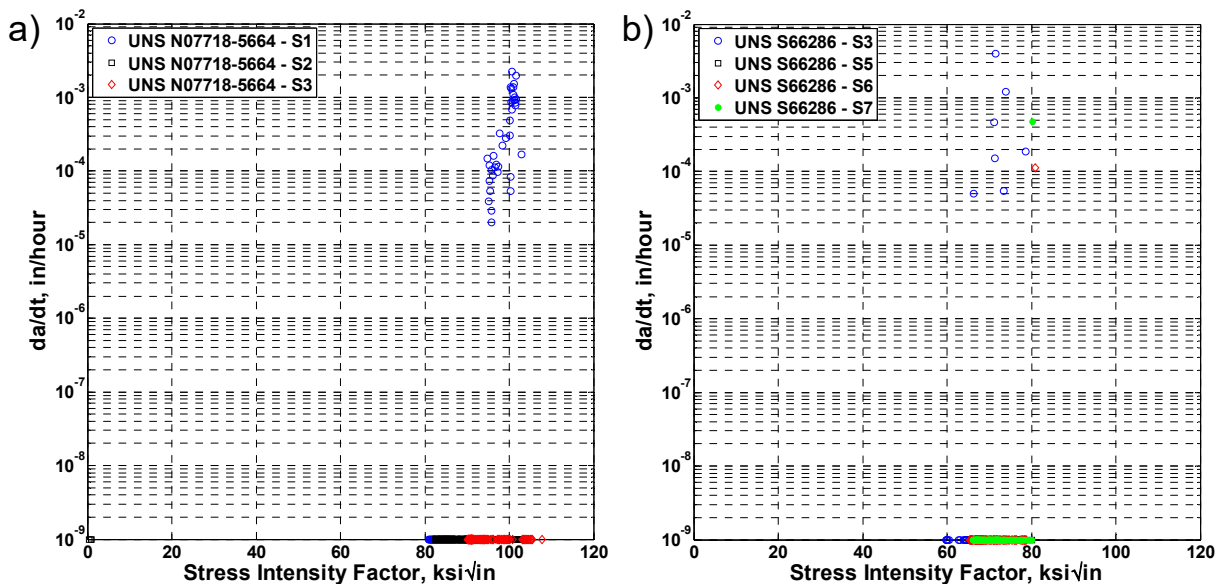


Figure 50 – Crack growth rate vs. stress intensity factor after regression of back-face strain data for a) UNS N07718 – 5664 and b) UNS S66286.

Summary of SCC under Alternate Immersion Conditions

SCC initiation times varied significantly among different materials. UNS K91973 showed initiation on average after 425 hours of exposure. The initiation time was shorter for the K91973 samples tested at higher initial K . UNS N07718-5664 showed an incubation time of 30 hours for one of the three samples tested. UNS S66286 showed initiation after 162 hours of exposure for one out of four samples, and A722 showed initiation only past 5640 hours of exposure for one of the four samples tested. The remaining materials did not show SCC in this environment. For these materials, the reported KIEAC is the highest K tested.

Table 13 – Resulting K_{IEAC} for the materials in this study.

Material	SCC Initiation	K_{IEAC}		Yield Strength (0.2%)		YS/ K_{IEAC} Ratio
	(hr)	(ksi $\sqrt{in}$)	(MPa $\sqrt{m}$)	(ksi)	(MPa)	
UNS N05500	No Initiation	$\geq 67.9^\dagger$	$\geq 74.6^\dagger$	90	621	0.75
UNS N07718 - 5664	30 (1/3 samples)	94.7 †	104.1 †	150	1034	0.63
UNS N07718 - 6A	No Initiation	$\geq 88.5^\dagger$	$\geq 97.2^\dagger$	140	965	0.63
UNS R30035	No Initiation	$\geq 123.6^\dagger$	$\geq 135.8^\dagger$	170	1172	0.73
UNS S66286	162 (1/4 samples)	66.4 †	73.1 †	95	655	0.75
UNS K91973	425 (all samples avg)	$< 50.5^\dagger$	$< 55.5^\dagger$	250	1724	< 0.20
A722	5640 (1/4 samples)	53.0 †	58.2 †	120	827	0.44

A summary of the SCC observations for all materials is shown in Table 13. Most materials show a ratio of yield strength and K_{IEAC} threshold between 0.6 and 0.75. UNS K91973 shows a ratio of 0.2, however this ratio is expected to be lower because the samples did not stop cracking at this K level, and the tests became invalid. A722 steel shows a ratio of 0.44.

Discussion

Advantages and disadvantages of the developed testing method

The back-faced strain gauge testing method has the clear advantage of automating the data collection process for a large number of samples. In the laboratory, this is often limited by machine and instrumentation availability. This method allows direct exposure to operational environment with great survivability of instrumentation for up to 2 years of alternate immersion exposure. Minor issues were observed with the protection of the strain gauge for materials undergoing a significant level of corrosion. For corrosion susceptible materials, corrosion attack may compromise the bonding between polysulfide and metal at the interface, eventually compromising the measurement. No issues were observed for materials reasonably resistant to the exposure conditions. The resolution of the experimental technique is well within the range of compliance techniques in laboratory environment. The technique is able to resolve at least 0.001 in (25 μm) and often less on average. A generalized resolution of 25-50 $\mu\text{m/day}$ is within the capabilities of this method.

A possible technical challenge with strain recordings for long exposure times at the back face comes from a relaxation of the strain response over time. The relaxation is more clearly visible for materials showing resistance to SCC, an example is shown for UNS N05500 in Figure 51. The observed change in back-face strain does not correlate to the growth of the crack, and in fact it is in the opposite direction with respect to the expected change in strain if the crack were to progress. The average relaxation rate for all alloys is 2.5 10⁻⁸ in/in/hr or 0.025 $\mu\text{ε/hr}$. The strain gauge bonding material is considered to be the source of the relaxation, however dedicated tests were not performed. The use of appropriate creep constants based on the mechanical behavior of the gauge bonding material is a possible solution to correct the strain signals for long exposure times.

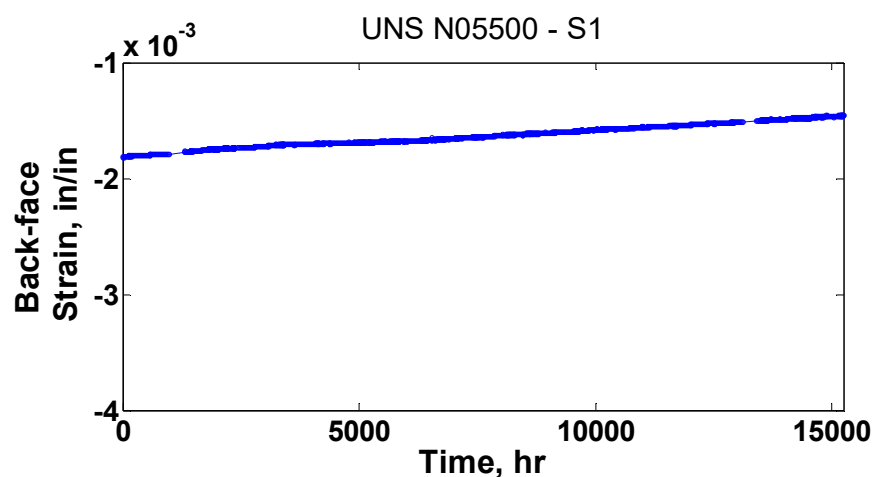


Figure 51 – Relaxation of back-face strain as a function of time for a UNS N05500 sample.

Assuming this average rate of change over time, the apparent change (increase) in a/W , the normalized crack growth rate can be calculated. This change also depends on the value of strain that was reached at the end of the loading phase of testing and on the a/W at the instant in time where the apparent change is calculated. A summary of the expected %error as a function of time because of the relaxation of strain is shown in Table 14. The highest and lowest back-face strain conditions observed during testing are shown for the two extremes of $a/W=0.5$ and $a/W=0.8$

Table 14 – Effect of strain recovery rate in determining crack length.

Initial Strain (in/in)	Normalized Crack Length a/W	% Error in a/W after		
		1,000 hr	5,000 hr	10,000 hr
-0.0015	0.5	0.7	3.9	9.7
	0.8	0.3	1.6	4.0
-0.0030	0.5	0.3	1.9	5.0
	0.8	0.1	0.8	2.1

The error remains within 1% and 4% for all cases when the exposure is less than 5,000 hours. The error is higher than 5% only in cases of low initial compressive strain and long exposure times. Future testing with this method will choose the levels of initial strain and exposure times to remain within 5% error due to relaxation.

In this study, the lowest strains were obtained for A722 steel. The data presented earlier were for exposure times within 5,000 hours and they are potentially affected by the strain relaxation observed, up to 3.9%. No data for A722 steel were reported beyond 8,000 hours of testing, where the same calculation as in Table 14 gives a maximum expected error of 8.5% in a/W . These errors translate into a change in the calculated K of 5.6% and 11.8%, respectively. The initial back face strains for all other materials were lower (more compressive than) -0.002 and the data are considered valid up to 10,000 hours with a maximum expected error in a/W of 7.4%, corresponding to a K reduction of 10.4%. For high-risk applications, the lower, more conservative, threshold values should be used.

Conclusions

We successfully demonstrated the application of a novel technique for SCC testing of high strength structural materials in alternate immersion conditions conducted at the NRL-Key West exposure site with flowing natural seawater. This technique was used to simultaneously test a large number of samples for up to 15,000 hours while monitoring the growth of cracks with accuracy comparable to laboratory testing

techniques. Some limitations on the use of strain gauges for long term exposure were observed. Corrosion may interfere with the strain gauge attachments over time. Relaxation of back-face strain over time was measured and its effects on the calculation of crack length at different points in time is estimated. Correlation of fractography measured crack length to the in situ measurements is underway to fully validate the test method.

Ni-Cu alloy UNS N05500, Ni-Co alloy UNS R30035, and Ni alloy UNS N07718 - 6A did not show susceptibility to SCC in alternate immersion at the K levels tested. All other alloys showed some level of SCC susceptibility, however minimal SCC was observed for UNS N07718 AMS 5664. SCC of high strength Fe-based alloys UNS S66286, A722, and UNS K91973 was observed. Notably, UNS K91973 showed significant SCC susceptibility and a threshold of less than $46 \text{ ksi}\sqrt{\text{in}}$ ($50 \text{ MPa}\sqrt{\text{m}}$), accounting for relaxation error.

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80. AMS5663, *ALLOY BARS, FORGINGS, AND RINGS, CORROSION AND HEAT RESISTANT Nickel Base - 19Cr - 3.1Mo - 5.1(Cb + Ta) - 0.90Ti - 0.50Al Solution and Precipitation Heat Treated Consumable Electrode or Vacuum Induction Melted*.
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APPENDIX I – AN AUTOMATED 2D MICROSTRUCTURE GENATOR FOR UNS N07718

Microstructure Generation

A MATLAB-based program was developed to automate the generation of material microstructures representative of Ni-Cr-Mo alloys, such as UNS N0718 material. The program has a User Interface (UI) to help the visualization of the details that are included in the material microstructure. The UI generates a python script that can be read by the finite element program Abaqus to create a 2D model of the material microstructure. The list of inputs is:

- 1) Number of grains and size of the microstructure
- 2) Presence of δ -phase, dimension, and distribution
- 3) Presence of γ' and γ'' phase, dimension, number within the microstructure of each
- 4) Grain orientation (Euler angles) and rules for δ , γ' and γ'' phase orientation

The UI is shown in Figure 52 along with a typical input.



Figure 52 – UI developed to create 2D representative microstructures of Ni-Cr-Mo alloy systems.

The program uses the visual interface to create a python script that is then input into Abaqus to automatically generate the microstructure. This is done to avoid the tedious task of creating the input needed for all the microstructural constituents in the analysis. Abaqus requires each particle domain to be defined by a geometry, partitioned from the rest of the model, selected and assigned material properties. With the number of γ' and γ'' phases present within the microstructure (typically between 1,000 and 6,000), a manual input is not feasible, or would require a compromise between developing a representative material

microstructure and amount of user input time. The python script developed by the program automates the creation, the partition, the property assignment, including orientations (Euler angles), of all domains, along with the application of boundary conditions and loads, and the creation of specific seeds to facilitate the meshing of the microstructure.

Abaqus Script and Model Creation

```

# -*- coding: mbcs -*-
from part import *
from material import *
from section import *
from assembly import *
from step import *
from interaction import *
from load import *
from mesh import *
from optimization import *
from job import *
from sketch import *
from visualization import *
from connectorBehavior import *

mdb.models['Model-1'].ConstrainedSketch(name='__profile__', sheetSize=100.0)
mdb.models['Model-1'].sketches['__profile__'].rectangle(point1=(0.0, 0.0), point2=(12,12))
mdb.models['Model-1'].Part(dimensionality=TWO_D_PLANAR, name='Part-1',
type=DEFORMABLE_BODY)
mdb.models['Model-1'].parts['Part-1'].BaseShell(sketch=mdb.models['Model-1'].sketches['__profile__'])

% creation of Voronoi structure and  $\delta$  domains
s1=mdb.models['Model-1'].ConstrainedSketch(name='_Voronoi_',sheetSize=144)
s1.Line(point1=([6.7474,5.8244]),point2=([7.1078,5.8225]))
s1.Line(point1=([3.6321,4.6289]),point2=([3.5945,4.4982]))
...
s1.Line(point1=(-2.0873,9.2319),point2=(-24.7934,13.1851))
s1.Line(point1=([8.89,2.3396]),point2=([18.7175,-14.9403]))
s1.CircleByCenterPerimeter(center=([1.8125,6.125]),point1=([1.8125,6.5625]))

% creation of  $\gamma''$  domains
s1.EllipseByCenterPerimeter(center=([2.7525,9.9305]),axisPoint1=([2.9214,9.8234]),axisPoint2=([2.7882,
9.9868]))
...
s1.EllipseByCenterPerimeter(center=([11.7839,6.1628]),axisPoint1=([11.8301,6.1819]),axisPoint2=([11.7
775,6.1781]))

% creation of  $\gamma'$  domains
s1.CircleByCenterPerimeter(center=([10.9136,0.69423]),point1=([10.9336,0.69423]))
...
s1.CircleByCenterPerimeter(center=([0.66661,4.6086]),point1=([0.68661,4.6086]))

```

```

p=mdb.models['Model-1'].parts['Part-1']
f=p.faces
pickedFaces = f.getSequenceFromMask(mask=('[#1 ]', ), )
e1, d2 = p.edges, p.datums
p.PartitionFaceBySketch(faces=pickedFaces, sketch=s1)
grainnumb=0
deltanumb=0
deltataken=[]
graintaken=[]
graintakensize=[]
gammataken=[]
deltaidcs=[]
gammaprimetaken=[]
% Identification and grouping of all created domains by area
% and creation of model "sets"
for x in range(len(f)):
    fcb=f[x]
    if fcb.getSize() >0.60192:
        grainnumb=grainnumb+1
        p.Set(name=str('setGrain'+str(grainnumb-1)),faces=f[x:x+1])
        graintaken.append(fcb.index)
        graintakensize.append([grainnumb-1,fcb.getSize()])
    else:
        adjf=fcb.getAdjacentFaces()
        for y in range(len(adjf)):
            fcdj=adjf[y]
            if fcdj.getSize() <0.12566:
                alreadytaken=False
                for z in range(len(deltataken)):
                    if adjf[y].index==deltataken[z] :
                        alreadytaken=True
                if alreadytaken!=True :
                    getstupidseq=f.findAt(adjf[y].getCentroid(),fcb.getCentroid())
                    deltanumb=deltanumb+ 1
                    deltataken.append(fcb.index)
                    deltataken.append(adjf[y].index)
                    deltaidcs.append([fcb.index,adjf[y].index])

```

```

        p.Set(name=str('setDelta'+str(deltanumb-1)),faces=getstupidseq)
    gammanumb=0
    gammaprimenumb=0
    gammagrain=[]
    gammaprimegrain=[]
    for x in range(len(f)):
        foundFF=False
        for z in range(len(deltatoken)):
            if f[x].index==deltatoken[z] :
                foundFF=True
        for z in range(len(graintoken)):
            if f[x].index==graintoken[z] :
                foundFF=True
        if foundFF==False :
            if f[x].getSize()<0.00357:
                gammaprimenumb=gammaprimenumb+1
                adjf=f[x].getAdjacentFaces()
                gammaprimegrain.append([f[x].index,adjf[0].index])
                gammaprimetaken.append(f[x].index)
            else:
                gammanumb=gammanumb+1
                adjf=f[x].getAdjacentFaces()
                gammagrain.append([f[x].index,adjf[0].index])
                gammataken.append(f[x].index)
    for x in range(grainnumb):
        if 'pickedfacesGGP' in locals() :
            del pickedfacesGGP
        for ggp in range(gammaprimenumb):
            if gammaprimegrain[ggp][1]==graintaken[x] :
                if 'pickedfacesGGP' in locals() :
                    pickedfacesGGP=pickedfacesGGP+f.findAt(f[gammaprimegrain[ggp][0]].getCentroid())
                else :
                    pickedfacesGGP=f.findAt(f[gammaprimegrain[ggp][0]].getCentroid())
        p.Set(name=str('AllGammaPGrain'+str(x)),faces=pickedfacesGGP)
    for x in range(grainnumb):
        if 'pickedfacesGG' in locals() :
            del pickedfacesGG

```

```

for ggp in range(gammanumb):
    if gammagrain[ggp][1]==graintaken[x] :
        if 'pickedfacesGG' in locals() :
            pickedfacesGG=pickedfacesGG+f.findAt(f[gammagrain[ggp][0]].getCentroid())
        else :
            pickedfacesGG=f.findAt(f[gammagrain[ggp][0]].getCentroid())
    p.Set(name=str('AllGammaGrain'+str(x)),faces=pickedfacesGG)
for x in range(len(f)):
    foundFF=False
    for z in range(len(deltatoken)):
        if f[x].index==deltatoken[z] :
            foundFF=True
    for z in range(len(gammataken)):
        if f[x].index==gammataken[z] :
            foundFF=True
    for z in range(len(gammaprimetaken)):
        if f[x].index==gammaprimetaken[z] :
            foundFF=True
    if foundFF==False :
        if 'pickedfacesG' in locals() :
            pickedfacesG=pickedfacesG+f.findAt(f[x].getCentroid())
        else :
            pickedfacesG=f.findAt(f[x].getCentroid())
    p.Set(name='AllGrains',faces=pickedfacesG)
for z in range(len(deltatoken)):
    if 'pickedfacesD' in locals() :
        pickedfacesD=pickedfacesD+f.findAt(f[deltatoken[z]].getCentroid())
    else :
        pickedfacesD=f.findAt(f[deltatoken[z]].getCentroid())
    p.Set(name='AllDelta',faces=pickedfacesD)
for z in range(len(gammataken)):
    if 'pickedfacesA' in locals() :
        pickedfacesA=pickedfacesA+f.findAt(f[gammataken[z]].getCentroid())
    else :
        pickedfacesA=f.findAt(f[gammataken[z]].getCentroid())
    p.Set(name='AllGamma',faces=pickedfacesA)
for z in range(len(gammaprimetaken)):

```

```

if 'pickedfacesAP' in locals() :
    pickedfacesAP=pickedfacesAP+f.findAt(f[gammaPrimetaken[z]].getCentroid())
else :
    pickedfacesAP=f.findAt(f[gammaPrimetaken[z]].getCentroid())
p.Set(name='AllGammaPrime',faces=pickedfacesAP)
grainteta=[]
graintetavalues=[]
def takeSecond(elem):
    return elem[1]
graintakensize.sort(key=takeSecond)

% Creation of reference axes and assignment orientation for all grains
graintetavalues=[-0.27824,-0.41417,-0.27692,-0.32596,-0.41931,-0.38773,-0.34088,-0.26922,-0.26793,-
0.40882,-0.26693,-0.26928,-0.35162,-0.29666,-0.41157,-0.36272,-0.27651,-0.29807]
for x in range(len(graintakensize)):
    teta=graintetavalues[x]
    point1x=cos(teta)
    point1y=sin(teta)
    grainteta.append([graintaken[graintakensize[x][0]],teta])
    gamma=teta+30./180.*pi
    point2x=cos(gamma)
    point2y=sin(gamma)
    mdb.models['Model-1'].parts['Part-1'].DatumCsysByThreePoints(coordSysType=
        CARTESIAN, name=str('DatumCSYS_'+str(x)), origin=(0.0,0.0,0.0),
    point1=(point1x,point1y,0.0),point2=(point2x,point2y,0.0))
    mdb.models['Model-1'].parts['Part-1'].MaterialOrientation(
        additionalRotationField="", additionalRotationType=ROTATION_NONE, angle=0.0
        , axis=AXIS_3, fieldName="", localCsys=
        mdb.models['Model-1'].parts['Part-1'].datums[mdb.models['Model-1'].parts['Part-1'].datums.keys()[x]],
    orientationType=SYSTEM,
        region=mdb.models['Model-1'].parts['Part-1'].sets[str('setGrain'+str(graintakensize[x][0]))],
    stackDirection=
        STACK_3)

% Creation of material properties
from material import*
mdb.models['Model-1'].Material(name='CP0')
mdb.models['Model-1'].materials['CP0'].UserMaterial(type=MECHANICAL,mechanicalConstants=[168])
mdb.models['Model-1'].Material(name='CP1')
mdb.models['Model-1'].materials['CP1'].UserMaterial(type=MECHANICAL,mechanicalConstants=[168])

```

```

mdb.models['Model-1'].Material(name='CP2')
mdb.models['Model-1'].materials['CP2'].UserMaterial(type=MECHANICAL,mechanicalConstants=[168])
mdb.models['Model-1'].Material(name='CP3')
mdb.models['Model-1'].materials['CP3'].UserMaterial(type=MECHANICAL,mechanicalConstants=[168])
mdb.models['Model-1'].Material(name='CP4')
mdb.models['Model-1'].materials['CP4'].UserMaterial(type=MECHANICAL,mechanicalConstants=[168])
mdb.models['Model-1'].Material(name='CP5')
mdb.models['Model-1'].materials['CP5'].UserMaterial(type=MECHANICAL,mechanicalConstants=[168])
mdb.models['Model-1'].Material(name='CP6')
mdb.models['Model-1'].materials['CP6'].UserMaterial(type=MECHANICAL,mechanicalConstants=[168])
mdb.models['Model-1'].Material(name='CP7')
mdb.models['Model-1'].materials['CP7'].UserMaterial(type=MECHANICAL,mechanicalConstants=[168])
mdb.models['Model-1'].Material(name='CP8')
mdb.models['Model-1'].materials['CP8'].UserMaterial(type=MECHANICAL,mechanicalConstants=[168])
mdb.models['Model-1'].Material(name='CP9')
mdb.models['Model-1'].materials['CP9'].UserMaterial(type=MECHANICAL,mechanicalConstants=[168])
mdb.models['Model-1'].Material(name='CP10')
mdb.models['Model-1'].materials['CP10'].UserMaterial(type=MECHANICAL,mechanicalConstants=[168])
mdb.models['Model-1'].Material(name='CP11')
mdb.models['Model-1'].materials['CP11'].UserMaterial(type=MECHANICAL,mechanicalConstants=[168])
mdb.models['Model-1'].Material(name='Delta')
mdb.models['Model-1'].materials['Delta'].Elastic(type=ORTHOTROPIC,table=((168400.,121400.,168400.,121400.,121400.,168400.,75400.,75400.0,75400.0),))
mdb.models['Model-1'].Material(name='Gamma')
mdb.models['Model-1'].materials['Gamma'].Elastic(type=ORTHOTROPIC,table=((168400.,121400.,168400.,121400.,121400.,168400.,75400.,75400.0,75400.0),))
mdb.models['Model-1'].Material(name='GammaPrime')
mdb.models['Model-1'].materials['GammaPrime'].Elastic(type=ORTHOTROPIC,table=((168400.,121400.,168400.,121400.,121400.,168400.,75400.,75400.0,75400.0),))

```

% Creation of reference axes and assignment orientation for all remaining domains

```

import random
for x in range(deltanumb):
    adjf=f[deltaidcs[x][0]].getAdjacentFaces()
    for y in range(len(adjf)):
        fcadj=adjf[y]
        if fcadj.index!=deltaidcs[x][1] :

```

```

        for xy in range(len(grainteta)):
            if fcadj.index==grainteta[xy][0] :
                angolo1=grainteta[xy][1]
adjf=f[deltaidcs[x][1]].getAdjacentFaces()
for y in range(len(adjf)):
    fcadj=adjf[y]
    if fcadj.index!=deltaidcs[x][0] :
        for xy in range(len(grainteta)):
            if fcadj.index==grainteta[xy][0] :
                angolo2=grainteta[xy][1]
teta=(angolo1+angolo2)/2.
point1x=cos(teta)
point1y=sin(teta)
gamma=teta+30./180.*pi
point2x=cos(gamma)
point2y=sin(gamma)
mdb.models['Model-1'].parts['Part-1'].DatumCsysByThreePoints(coordSysType=
    CARTESIAN, name=str('DatumCSYS_'+str(grainnumb+x)), origin=(0.0,0.0,0.0),
point1=(point1x,point1y,0.0),point2=(point2x,point2y,0.0))
mdb.models['Model-1'].parts['Part-1'].MaterialOrientation(
    additionalRotationField="", additionalRotationType=ROTATION_NONE, angle=0.0
    , axis=AXIS_3, fieldName="", localCsys=
    mdb.models['Model-1'].parts['Part-1'].datums[mdb.models['Model-1'].parts['Part-
1'].datums.keys()[grainnumb+x]], orientationType=SYSTEM,
    region=mdb.models['Model-1'].parts['Part-1'].sets[str('setDelta'+str(x))], stackDirection=
    STACK_3)
donesetsgamma=[]
import random
for x in range(len(gammataken)):
    indcorgrain=0
    adjf=f[gammataken[x]].getAdjacentFaces()
    for y in range(len(adjf)):
        fcadj=adjf[y]
        for xy in range(len(grainteta)):
            if fcadj.index==grainteta[xy][0] :
                angolo1=grainteta[xy][1]
                indcorgrain=xy
teta=angolo1

```

```

if indcorgrain in donesetsgamma:
    mabastaconstaro=0
else:
    mdb.models['Model-1'].parts['Part-1'].MaterialOrientation(
        additionalRotationField="", additionalRotationType=ROTATION_NONE, angle=0.0
        , axis=AXIS_3, fieldName="", localCsys=
        mdb.models['Model-1'].parts['Part-1'].datums[mdb.models['Model-1'].parts['Part-
1'].datums.keys()[indcorgrain]], orientationType=SYSTEM,
        region=mdb.models['Model-1'].parts['Part-1'].sets[str('AllGammaGrain'+str(indcorgrain))],
stackDirection=
        STACK_3)
    donesetsgamma.append(indcorgrain)
import random
donesetsgammaprime=[]
for x in range(len(gammaprimetaken)):
    indcorgrain=0
    adjf=f[gammaprimetaken[x]].getAdjacentFaces()
    for y in range(len(adjf)):
        fcadj=adjf[y]
        for xy in range(len(grainteta)):
            if fcadj.index==grainteta[xy][0] :
                angolo1=grainteta[xy][1]
                indcorgrain=xy
    teta=angolo1
    if indcorgrain in donesetsgammaprime:
        mabastaconstaro=0
    else:
        mdb.models['Model-1'].parts['Part-1'].MaterialOrientation(
            additionalRotationField="", additionalRotationType=ROTATION_NONE, angle=0.0
            , axis=AXIS_3, fieldName="", localCsys=
            mdb.models['Model-1'].parts['Part-1'].datums[mdb.models['Model-1'].parts['Part-
1'].datums.keys()[indcorgrain]], orientationType=SYSTEM,
            region=mdb.models['Model-1'].parts['Part-1'].sets[str('AllGammaPGrain'+str(indcorgrain))],
stackDirection=
            STACK_3)
        donesetsgammaprime.append(indcorgrain)

% Creation of Abaqus Sections for all domains and Section Assignment
mdb.models['Model-1'].HomogeneousSolidSection(material='CP0',
name='SectionCPG0',thickness=None)

```

```

mdb.models['Model-1'].HomogeneousSolidSection(material='CP1',
name='SectionCPG1',thickness=None)

mdb.models['Model-1'].HomogeneousSolidSection(material='CP2',
name='SectionCPG2',thickness=None)

mdb.models['Model-1'].HomogeneousSolidSection(material='CP3',
name='SectionCPG3',thickness=None)

mdb.models['Model-1'].HomogeneousSolidSection(material='CP4',
name='SectionCPG4',thickness=None)

mdb.models['Model-1'].HomogeneousSolidSection(material='CP5',
name='SectionCPG5',thickness=None)

mdb.models['Model-1'].HomogeneousSolidSection(material='CP6',
name='SectionCPG6',thickness=None)

mdb.models['Model-1'].HomogeneousSolidSection(material='CP7',
name='SectionCPG7',thickness=None)

mdb.models['Model-1'].HomogeneousSolidSection(material='CP8',
name='SectionCPG8',thickness=None)

mdb.models['Model-1'].HomogeneousSolidSection(material='CP9',
name='SectionCPG9',thickness=None)

mdb.models['Model-1'].HomogeneousSolidSection(material='CP10',
name='SectionCPG10',thickness=None)

mdb.models['Model-1'].HomogeneousSolidSection(material='CP11',
name='SectionCPG11',thickness=None)

mdb.models['Model-1'].HomogeneousSolidSection(material='Delta',
name='SectionDelta',
thickness=None)

mdb.models['Model-1'].HomogeneousSolidSection(material='Gamma',
name='SectionGamma',
thickness=None)

mdb.models['Model-1'].HomogeneousSolidSection(material='GammaPrime',
name='SectionGammaPrime', thickness=None)

mdb.models['Model-1'].parts['Part-1'].SectionAssignment(offset=0.0,
offsetField="", offsetType=MIDDLE_SURFACE, region=mdb.models['Model-1'].parts['Part-
1'].sets['setGrain0'], sectionName='SectionCPG0', thicknessAssignment=FROM_SECTION)

mdb.models['Model-1'].parts['Part-1'].SectionAssignment(offset=0.0,
offsetField="", offsetType=MIDDLE_SURFACE, region=mdb.models['Model-1'].parts['Part-
1'].sets['setGrain1'], sectionName='SectionCPG1', thicknessAssignment=FROM_SECTION)

mdb.models['Model-1'].parts['Part-1'].SectionAssignment(offset=0.0,
offsetField="", offsetType=MIDDLE_SURFACE, region=mdb.models['Model-1'].parts['Part-
1'].sets['setGrain2'], sectionName='SectionCPG2', thicknessAssignment=FROM_SECTION)

mdb.models['Model-1'].parts['Part-1'].SectionAssignment(offset=0.0,
offsetField="", offsetType=MIDDLE_SURFACE, region=mdb.models['Model-1'].parts['Part-
1'].sets['setGrain3'], sectionName='SectionCPG3', thicknessAssignment=FROM_SECTION)

mdb.models['Model-1'].parts['Part-1'].SectionAssignment(offset=0.0,
offsetField="", offsetType=MIDDLE_SURFACE, region=mdb.models['Model-1'].parts['Part-
1'].sets['setGrain4'], sectionName='SectionCPG4', thicknessAssignment=FROM_SECTION)

```

```

mdb.models['Model-1'].parts['Part-1'].SectionAssignment(offset=0.0,
    offsetField="", offsetType=MIDDLE_SURFACE, region=mdb.models['Model-1'].parts['Part-
1'].sets['setGrain5'], sectionName='SectionCPG5', thicknessAssignment=FROM_SECTION)
mdb.models['Model-1'].parts['Part-1'].SectionAssignment(offset=0.0,
    offsetField="", offsetType=MIDDLE_SURFACE, region=mdb.models['Model-1'].parts['Part-
1'].sets['setGrain6'], sectionName='SectionCPG6', thicknessAssignment=FROM_SECTION)
mdb.models['Model-1'].parts['Part-1'].SectionAssignment(offset=0.0,
    offsetField="", offsetType=MIDDLE_SURFACE, region=mdb.models['Model-1'].parts['Part-
1'].sets['setGrain7'], sectionName='SectionCPG7', thicknessAssignment=FROM_SECTION)
mdb.models['Model-1'].parts['Part-1'].SectionAssignment(offset=0.0,
    offsetField="", offsetType=MIDDLE_SURFACE, region=mdb.models['Model-1'].parts['Part-
1'].sets['setGrain8'], sectionName='SectionCPG8', thicknessAssignment=FROM_SECTION)
mdb.models['Model-1'].parts['Part-1'].SectionAssignment(offset=0.0,
    offsetField="", offsetType=MIDDLE_SURFACE, region=mdb.models['Model-1'].parts['Part-
1'].sets['setGrain9'], sectionName='SectionCPG9', thicknessAssignment=FROM_SECTION)
mdb.models['Model-1'].parts['Part-1'].SectionAssignment(offset=0.0,
    offsetField="", offsetType=MIDDLE_SURFACE, region=mdb.models['Model-1'].parts['Part-
1'].sets['setGrain10'], sectionName='SectionCPG10', thicknessAssignment=FROM_SECTION)
mdb.models['Model-1'].parts['Part-1'].SectionAssignment(offset=0.0,
    offsetField="", offsetType=MIDDLE_SURFACE, region=mdb.models['Model-1'].parts['Part-
1'].sets['setGrain11'], sectionName='SectionCPG11', thicknessAssignment=FROM_SECTION)
mdb.models['Model-1'].parts['Part-1'].SectionAssignment(offset=0.0,
    offsetField="", offsetType=MIDDLE_SURFACE, region=mdb.models['Model-1'].parts['Part-
1'].sets['AllDelta'], sectionName='SectionDelta', thicknessAssignment=FROM_SECTION)
mdb.models['Model-1'].parts['Part-1'].SectionAssignment(offset=0.0,
    offsetField="", offsetType=MIDDLE_SURFACE, region=mdb.models['Model-1'].parts['Part-
1'].sets['AllGamma'], sectionName='SectionGamma', thicknessAssignment=FROM_SECTION)
mdb.models['Model-1'].parts['Part-1'].SectionAssignment(offset=0.0,
    offsetField="", offsetType=MIDDLE_SURFACE, region=mdb.models['Model-1'].parts['Part-
1'].sets['AllGammaPrime'],
    sectionName='SectionGammaPrime',
    thicknessAssignment=FROM_SECTION)
edgs=p.edges
uplist=[]

```

% Creation of Boundary Conditions and Loads

```

for x in range(len(edgs)):
    if edgs[x].pointOn[0][1]==12:
        uplist.append(x)
for x in range(len(uplist)):
    if 'upedgspicked' in locals() :
        upedgspicked=upedgspicked+edgs.findAt(edgs[uplist[x]].pointOn)
    else :

```

```

        upedgspicked=edgs.findAt(edgs[uplist[x]].pointOn)
p.Set(name='TopBC',edges=upedgspicked)
lwlist=[]
for x in range(len(edgs)):
    if edgs[x].pointOn[0][1]==0.:
        lwlist.append(x)
for x in range(len(lwlist)):
    if 'lwedgspicked' in locals() :
        lwedgspicked=lwedgspicked+edgs.findAt(edgs[lwlist[x]].pointOn)
    else :
        lwedgspicked=edgs.findAt(edgs[lwlist[x]].pointOn)
p.Set(name='BBC',edges=lwedgspicked)
vts=p.vertices
indexorigin=vts.findAt((0.,0.,0.)).index
p.Set(name='Corner',vertices=vts.findAt(vts[indexorigin].pointOn))
mdb.models['Model-1'].setValues(noPartsInputFile=True)
mdb.models['Model-1'].StaticStep(initialInc=0.1, name='UpwardD', previous='Initial')
mdb.models['Model-1'].rootAssembly.Instance(dependent=ON, name='Part-1-1',
    part=mdb.models['Model-1'].parts['Part-1'])
mdb.models['Model-1'].DisplacementBC(amplitude=UNSET, createStepName='UpwardD',
    distributionType=UNIFORM, fieldName="", fixed=OFF, localCsys=None, name=
    'TopDisp', region=
    mdb.models['Model-1'].rootAssembly.instances['Part-1-1'].sets['TopBC'], u1=
    UNSET, u2=0.012, ur3=UNSET)
mdb.models['Model-1'].YsymmBC(name='BottomSymm',createStepName='UpwardD',region=
    mdb.models['Model-1'].rootAssembly.instances['Part-1-1'].sets['BBC'])
mdb.models['Model-1'].DisplacementBC(amplitude=UNSET, createStepName='UpwardD',
    distributionType=UNIFORM, fieldName="", fixed=OFF, localCsys=None, name=
    'CornerFixed', region=
    mdb.models['Model-1'].rootAssembly.instances['Part-1-1'].sets['Corner'], u1=
    0., u2=0.0, ur3=UNSET)

% Creation of notch/crack in the created domain
s2=mdb.models['Model-1'].ConstrainedSketch(name='_Notch_',sheetSize=144)
s2.Line(point1=([0.0,6]),point2=([1.75,6]))
s2.Line(point1=([0.0,6.25]),point2=([1.75,6.25]))
s2.Line(point1=([0.0,6.25]),point2=([0.0,6]))
s2.ArcByStartEndTangent(point1=([1.75,6.25]),point2=([1.75,6]),vector=[1.0,0.0])

```

```

myedge=edgs.findAt((0.0,6,0.0))
p.CutExtrude(sketchPlane=f[0],sketchPlaneSide=SIDE1,sketchUpEdge=myedge,sketchOrientation=LEFT,sketch=s2)
s3=mdb.models['Model-1'].ConstrainedSketch(name='_AreaNotch_',sheetSize=144)
s3.Line(point1=([0.75,6.75]),point2=([2.75,6.75]))
s3.Line(point1=([0.75,5.5]),point2=([2.75,5.5]))
s3.Line(point1=([0.75,5.5]),point2=([0.75,6.75]))
s3.Line(point1=([2.75,5.5]),point2=([2.75,6.75]))
% Creation of mesh seeds
p.setMeshControls(regions=f[0:len(f)],elemShape=QUAD,technique=FREE)
miotype=ElemType(elemCode=CPE8R, elemLibrary=STANDARD)
p.setElementType(elemTypes=(miotype,),regions=p.sets['AllGrains'])
p.setElementType(elemTypes=(miotype,),regions=p.sets['AllDelta'])
p.setElementType(elemTypes=(miotype,),regions=p.sets['AllGamma'])
p.setElementType(elemTypes=(miotype,),regions=p.sets['AllGammaPrime'])
edgs=p.edges
f=p.faces
for x in range(len(f)):
    if f[x].getSize()<0.12566:
        edfgs=f[x].getEdges()
        for z in range(len(edfgs)):
            p.seedEdgeBySize(edges=(edgs[edfgs[z]],),size=0.0125)
myedgectrl=edgs.findAt((1.875,6.125,0.0))
p.seedEdgeBySize(edges=(edgs[myedgectrl.index],),size=0.0125)
p.seedPart(size=0.2)

```