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The Influence of Stress Concentration and Plastic Strain on the Resistance of Precipitation-Hardened Nickel Alloys to Hydrogen Embrittlement

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ABSTRACT

Precipitation-hardened nickel alloys (PHNAs), such as UNS N07718, are commonly used in oil and gas subsea service for components such as bolts, fasteners and gaskets, which require high strength and corrosion resistance. Numerous subsea failures of these materials have been attributed to hydrogen embrittlement (HE) caused by hydrogen evolved by cathodic protection (CP) systems, and in the presence of geometrical design features, i.e. grooves, notches, threads etc. acting as stress raisers, and sometimes a compromised microstructure associated with inappropriately controlled alloy manufacturing processes. Although the coexistence of a susceptible microstructure, sufficiently high stress and hydrogen content is deemed necessary for cracking to occur, the significance of those parameters and their complex interrelationship is not understood. The lack of such understanding calls into question the relevance of the widely-used test methodologies for designing and assessing with PHNAs, as it is not established what combination of parameters is of highest significance to be monitored and measured.

In previous work, the co-authors and other researchers have extensively used slow strain rate tensile (SSRT) testing of plain-sided (un-notched) specimens, primarily as a qualitative screening method, to rank the HE resistance of various alloys in different microstructural conditions. In some cases, attempts have been made to use the test and its results to obtain a quantitative insight into the cracking sequences and stress/strain thresholds. Whilst the majority of the test outputs have inherently been qualitative, observations of slip bands on the brittle fracture surfaces of SSRT test specimens suggest the mechanism of HE is associated with a degree of plastic strain and strain localization prior to crack initiation. Given the significance of such a strain threshold, and the observation that most failures occur at stress concentrators, this study explores the role of stress raisers on the resistance of UNS N07718 to HE, using notches with different stress concentration factors (SCFs) introduced into the test specimens. SSRT testing and incremental step load (ISL) testing were combined with finite element (FE) modelling to investigate the usefulness of each approach in terms of providing a robust, quantitative assessment and design criterion for HE resistance, based on a hypothesized threshold of critical strain localization to activate HE crack initiation in PHNAs.

Key words: Hydrogen Embrittlement, Precipitation hardened (PH) Nickel Alloys, SSRT Testing, ISL Testing, Incremental Step Load (ISL) Testing, Notch, and Strain Localization.

INTRODUCTION

PHNAs are commonly used in a variety of downhole, wellhead, and Christmas tree applications¹, because of their high strength and corrosion resistance. These austenitic materials achieve their high strength as a result of distributions of γ' ($\text{Ni}_3\text{Al/Ti}$) and γ'' (Ni_3Nb) precipitates developed during solution and ageing heat treatments. API 6ACRA:2015² defines heat treatments and acceptance criteria for a range of PHNAs intended to maximize the resistance of these materials to HE, when used in subsea service. Since the introduction of this Standard, the number of failures associated with HE in these materials has fallen significantly. TWI is unaware of any failures of API 6A CRA compliant material to date, however, failures of PHNAs due to HE have not been eliminated entirely and the cracking mechanisms of PHNAs in subsea service continues to be the subject of intense research^{3,4,5,6,7}.

Most studies on this subject have used SSRT testing to rank the resistance to HE of PHNAs with different microstructural conditions in different environments⁷. The application of CP to PHNAs in SSRT tests successfully reproduces the brittle fractures observed in service failures and shows the significantly reduced ductility and UTS of these materials when CP is active. Recent studies have also suggested that strain localization plays a critical part in the HE failure mechanism and the authors and others have highlighted the presence of significant plasticity, as manifested by dislocation slip bands on the 'brittle' fracture surfaces of SSRT test specimens, when CP is applied^{7,8,9}. The role of plasticity in the HE failure mechanism suggests that geometric features of components, which concentrate stress and therefore strain, could render those materials susceptible to failure by HE.

Stress localization is clearly a factor in the majority of service failures, which typically occur at pre-existing grooves and notches, such as threaded sections of fasteners and bolts. Environmental SSRT tests of notched specimens have shown that PHNAs are notch sensitive in that the knockdown of UTS, due to the application of CP, is more significant in notched specimens than in un-notched, plain-sided specimens⁷. However the extent, to which the susceptibility of a material to HE is dependent on the acuity of notches, is currently unknown.

In this study, we seek to identify the significance of notch acuity on the resistance of PHNAs to HE. The performances of two notch geometries, one representative of threaded fasteners and a second, sharper notch with a higher SCF, were compared in SSRT and ISL testing, under CP. FE modelling of each notch type was used to predict the local strains developed at the notch root, in order to identify if cracking by HE were associated with a critical level of local plastic strain, which could be developed into a quantitative design criterion. The study was carried out using UNS N07718 PHNA, in the solution-treated and aged condition. UNS N07718, meeting API 6A CRA, is workhorse PHNA not typically associated with a high susceptibility to HE, making this a useful material for comparing notch sensitivity in SSRT and ISL tests.

EXPERIMENTAL PROCEDURE

Material

UNS N07718 120ksi material was supplied as 12.7 mm (0.5") diameter round bar in the solution-treated and aged condition, in accordance with API 6ACRA:2015. The microstructure of the material was examined using light microscopy of a metallographic cross-section containing the axis of the bar, at approximately mid-thickness.

Tensile specimen design and FE modelling

Round tensile specimens for SSRT and ISL testing were machined parallel to the axis of the bar. The distance between the shoulders was 22.5 mm and the gripped sections were approximately 12.5 mm in length. The diameter of the reduced section was 6.35 mm, prior to notching with 1 mm deep notches.

Notched tensile specimens were prepared using wire electro-discharge machining (EDM). Two notch geometries were designated 'N1' and 'N2': the sharper N1 geometry was based on a design previously used by the authors⁷ and the less-sharp N2 geometry was designed to have an equivalent stress concentration factor (SCF) to a fastener known to be associated with a failure due to HE in service. The SCF at the root of the fastener thread was determined by preparing a metallographic cross-section through the threaded section and using light microscopy and digital image analysis of ten threads to determine the average root radius. This geometry was modelled using FE to determine the SCF at the root of the thread and define an equivalent notched tensile geometry to reproduce the same SCF, while preserving the same gauge thickness as the N1 specimen. The SCF's of the N1 and N2 notch geometries were 20 and 12, respectively, and the details of each notch type are given in Table 1.

Table 1: Details of the test specimens used in this study.

Notch	SCF	Notch radius (mm)	Notch depth (mm)
Plain-sided	1	-	-
N1	20	0.075	1.0
N2	12	0.22	1.0

FE modelling was also used to predict the plastic strain at the notch root as a function of applied load in uniaxial tension for each notch type.

Standard tensile testing

Standard tensile tests were carried out, in accordance with BS EN ISO 6892-1, on the as-received material, to establish the tensile properties. Tensile specimens were extracted parallel to the axis of the bar and tested in the plain-sided and notched (N2) conditions.

Slow strain rate testing

After being placed in the load frame and with no load applied, the specimens were pre-charged in a 3.5%NaCl solution with hydrogen at -1100 mV vs a standard calomel reference electrode (SCE), for 48 hours. Pre-charging was performed to ensure a residual layer of hydrogen was present at the outer-surface of the specimens, prior to testing, consistent with SSRT tests previously carried out by the authors. Hydrogen pre-charging was conducted at room temperature for 48 hours, which was sufficient for hydrogen to reach approximately 50 μm deep into the specimen according to calculations previously performed^{7,8}. Upon completion of the pre-charging period, a uniaxial load was applied to the specimens. Specimens were tested at an initial strain rate of approximately $5 \times 10^{-6} \text{s}^{-1}$, with CP continuously applied at the pre-charge potential.

To investigate the cracking mechanism in more detail (and particularly, in attempt to capture crack initiation), several of the SSRT tests were stopped, prior to failure, in order to examine cracking. Tests were stopped at gross cross-section loads of 883MPa and 1514MPa, corresponding to local plastic strains of ~1.8 and 10.0% at the notch root as described below. These test specimens were visually examined for cracking and metallographic specimens, containing any observed cracks, were prepared for examination using light and scanning electron microscopy (SEM), including electron backscatter diffraction (EBSD), as further detailed below.

Incremental step load testing

The ISL test method was based on ASTM F1624-12b, but tests were conducted under load control (rather than displacement control), allowing the application of certain load targets and the determination of the corresponding stress/strain thresholds. The load profile consisted of 10 steps of 5% UTS increments, held for 2 hours per step, followed by 10 steps at 5% UTS increments, held for 4 hours per step. The UTS was determined from conventional tensile tests of equivalent plain-sided and N2-notched

specimens, respectively. ISL tests were carried out using both plain-sided and the N1 and N2-notched specimens, to enable direct comparisons between each type. Both N1 and N2 notches were loaded using the same load profile, based on UTS of the N2 notch. Plane sided and N1 notched specimens were tested at room temperature in air and in a 3.5% NaCl solution with $-1100 \text{ mV}_{\text{SCE}}$ CP applied, the N1 notched specimen was tested in the 3.5% NaCl solution with CP applied, a single specimen of each type was tested. The specimens tested in 3.5%NaCl under CP were pre-charged with hydrogen at $-1100 \text{ mV}_{\text{SCE}}$ for 48 hours before the application of load. The same test methodology has also been used by the authors in a parallel paper on a similar research topic, studying the mutual, interrelated effects of CP potential and stress concentration on HE¹⁰.

Microstructural characterisation

Fractographic analysis of all specimens was performed by visual examination, stereoscopic light microscopy and SEM. Metallographic cross-sections of each test specimen fracture surface, sampling the full specimen diameter, were prepared using standard techniques and examined using both light microscopy and SEM, including EBSD. EBSD orientation maps are presented, relative to the axis of the bar. EBSD data are also presented as kernel average misorientation (KAM) maps, which show the magnitude of misorientation between neighboring pixels, as an indication of localized strain: higher KAM values indicating higher strain.

RESULTS

Material characterisation

Light micrographs of the as-received UNS N07718 bar material are shown in Figure 1. An equiaxed grain structure is apparent, containing a significant number of annealing twins. The material also contains stringers of particles parallel to the extrusion direction of the bar. No continuous network of grain boundary precipitates or delta phase are visible, showing the material is compliant with the microstructural requirements of API 6ACRA:2015.

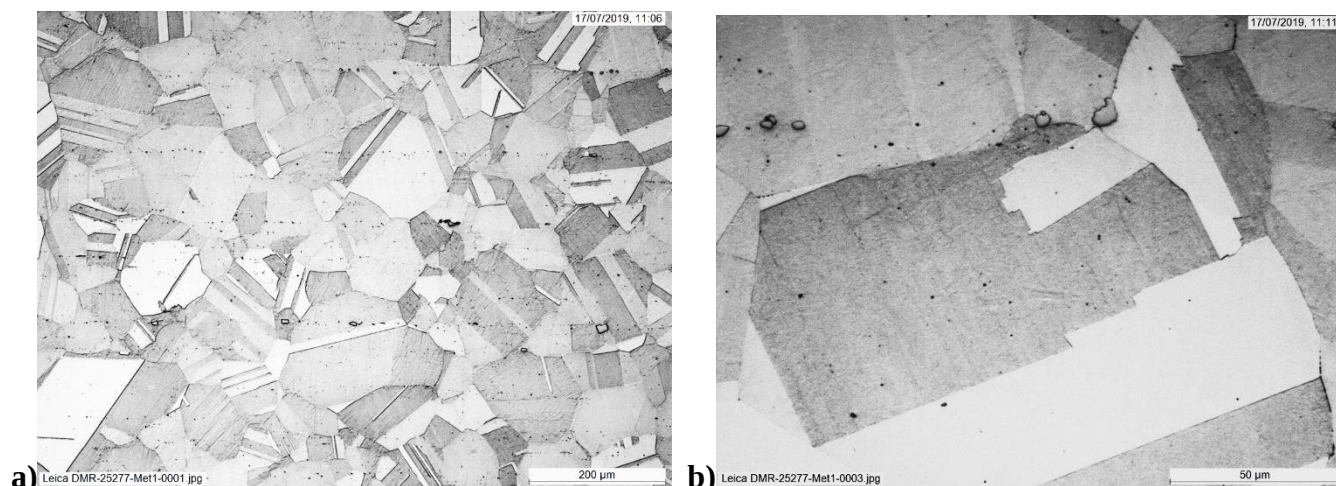


Figure 1: Light micrographs of the UNS N07718 parent material.

Standard tensile testing

The results of tensile tests, conducted at room temperature, are shown in Table 2. The plain-sided test specimen shows the material meets the requirements of API 6ACRA:2015 for UNS N07718 120ksi material, in terms of proof strength, UTS, ductility and reduction in area. The N2 notched specimen has a lower max load than the plain-sided specimen but higher UTS and 0.2% proof stress. The lower max