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Evaluation of the K_{IEC} of UNS N07718 (Alloy 718) with and without Delta Phase in Simulated Seawater with Cathodic Protection using Double Cantilever Beam Tests

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ABSTRACT

Most of the recent work done to characterize the fracture toughness of age-hardened nickel-based alloys in simulated seawater with cathodic protection (SW+CP) environments has used rising displacement techniques and compact tension specimens. The results of these tests are sometimes used in fracture mechanics calculations for design verification, though there is some uncertainty about the appropriate method for determining environmental fracture toughness from the results. There has not to date been much work performed to characterize threshold stress intensities in SW+CP environments using double cantilever beam (DCB) tests.

In this work, NACE TM0177 Method D (DCB) tests were performed on UNS N07718 (Alloy 718) in a simulated seawater environment with cathodic polarization. The tests were performed in a deaerated 3.5% NaCl solution at 4 °C (40 °F) with pH adjusted to 8.2 and an applied potential of -1050 mV versus a saturated calomel electrode (SCE). For comparison purposes, tests were performed on Alloy 718 with and without delta (δ) phase in the microstructure. Material and specimen examinations included microstructural characterization and scanning electron microscopy. The test data was compared to published data from several sources. The results support the current understanding that δ phase can be detrimental to the hydrogen embrittlement resistance of Alloy 718.

Key words: UNS N07718, Alloy 718, Hydrogen Embrittlement, DCB, Seawater, Cathodic Protection

INTRODUCTION

UNS N07718 (Alloy 718) is an attractive material for use in subsea and downhole environments due to its combination of corrosion resistance and high strength relative to carbon and low-alloy steels. However, use of improperly solution-annealed Alloy 718 and aerospace grades (i.e. ASTM⁽¹⁾ B637⁽¹⁾) in subsea and downhole environments have resulted in several high-profile failures.^{2,3,4,5} Early work convincingly showed a relationship between grain boundary delta (δ) phase precipitates and the susceptibility of the material to hydrogen stress cracking / hydrogen embrittlement.⁶ These findings and others led the industry to draft API⁽²⁾ Standard 6A718⁷, later superseded by API Standard 6ACRA⁸, which specifies grades of Alloy 718 that are largely free of grain boundary δ phase and other deleterious microstructural constituents. The prevention of δ phase formation is achieved by specifying a solution annealing temperature above the δ phase solvus temperature.

The resistance of the oil and gas grades of Alloy 718 to sulfide stress cracking, a form of hydrogen stress cracking, have been reasonably well demonstrated through NACE⁽³⁾ MR0175⁹ balloting initiatives, which require three heats of material to pass constant stress tensile, bend, or C-ring tests. Some Alloy 718 materials have been further qualified through galvanic coupling and slow strain rate testing. However, these qualification tests in H₂S-bearing environments do not necessarily qualify the materials for use in seawater environments with cathodic protection (SW+CP). Such environments can charge materials with hydrogen even in the absence of corrosion. Over the last several years, more work has been done to characterize the critical stress intensities of several oil and gas grades of Alloy 718 in these SW+CP environments. Several of these studies have included rising displacement and rising step load fracture mechanics-based tests.^{10,11,12}

Double cantilever beam (DCB) tests are constant displacement crack arrest tests, so it is a reasonable hypothesis that they may be good candidates to screen alloys for hydrogen embrittlement resistance in applications where dynamic loading is not expected. DCB tests may also better account for the material response to long term exposure in hydrogen-charging environments due to their test duration. A literature review of published studies revealed only one example of DCB tests being used to assess the performance of Alloy 718 in a simulated seawater environment with cathodic protection.¹³ The tests performed by Esaklul and Martin showed little to no cracking during the tests, so it was not clear from that work whether DCBs would be an effective technique for screening out questionable candidate materials.

The purpose of this study was to evaluate whether DCB tests are a feasible test method by which to assess the performance of different grades of Alloy 718 in a simulated seawater environment with cathodic protection. This study performed tests on two different Alloy 718 materials: one with a high temperature solution annealing heat treatment to prevent δ phase formation and one with a lower temperature solution annealing heat treatment to promote the formation of δ phase along grain boundaries. Both materials were removed from the same raw material in order to minimize the impact of heat to heat compositional variations.

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MATERIALS

The raw material used for this study was a 76-mm (3-inch) diameter fastener made of UNS N07718, previously heat treated in accordance with API Standard 6A718. The original chemical composition of the raw material is shown in Table 1.

Table 1
Chemical Composition of Raw Material

Element	Concentration (wt. %)
Nickel	54.2
Chromium	18.2
Iron	17.9
Niobium	5.02
Molybdenum	2.89
Titanium	0.87
Aluminum	0.35
Carbon	0.03
Cobalt	0.22
Manganese	0.11
Silicon	0.11
Phosphorus	0.005
Sulfur	<0.002
Boron	0.003
Copper	0.05

The fastener was longitudinally cut in half, and the halves were labeled “A” and “B”. Sample A was solution annealed at 954 °C (1750 °F) for 2 hours to promote the formation of δ phase in the microstructure. Sample B was solution annealed at 1038 °C (1900 °F) for 2 hours to avoid δ phase formation. Both samples were water-quenched and then aged simultaneously in the same furnace. A two-step aging heat treatment was selected to target grade 150K material: age at 718 °C (1325 °F) for 9 hours, furnace cool to 621 °C (1150 °F) and hold for 9 hours, remove and air cool. The heat treatment for Sample A was consistent with ASTM B637 for N07718, and the heat treatment for Sample B was consistent with API Standard 6ACRA for 150K N07718. The thermal experience for each sample was recorded with a contact thermocouple, and the temperature plots are presented in Figure 1.

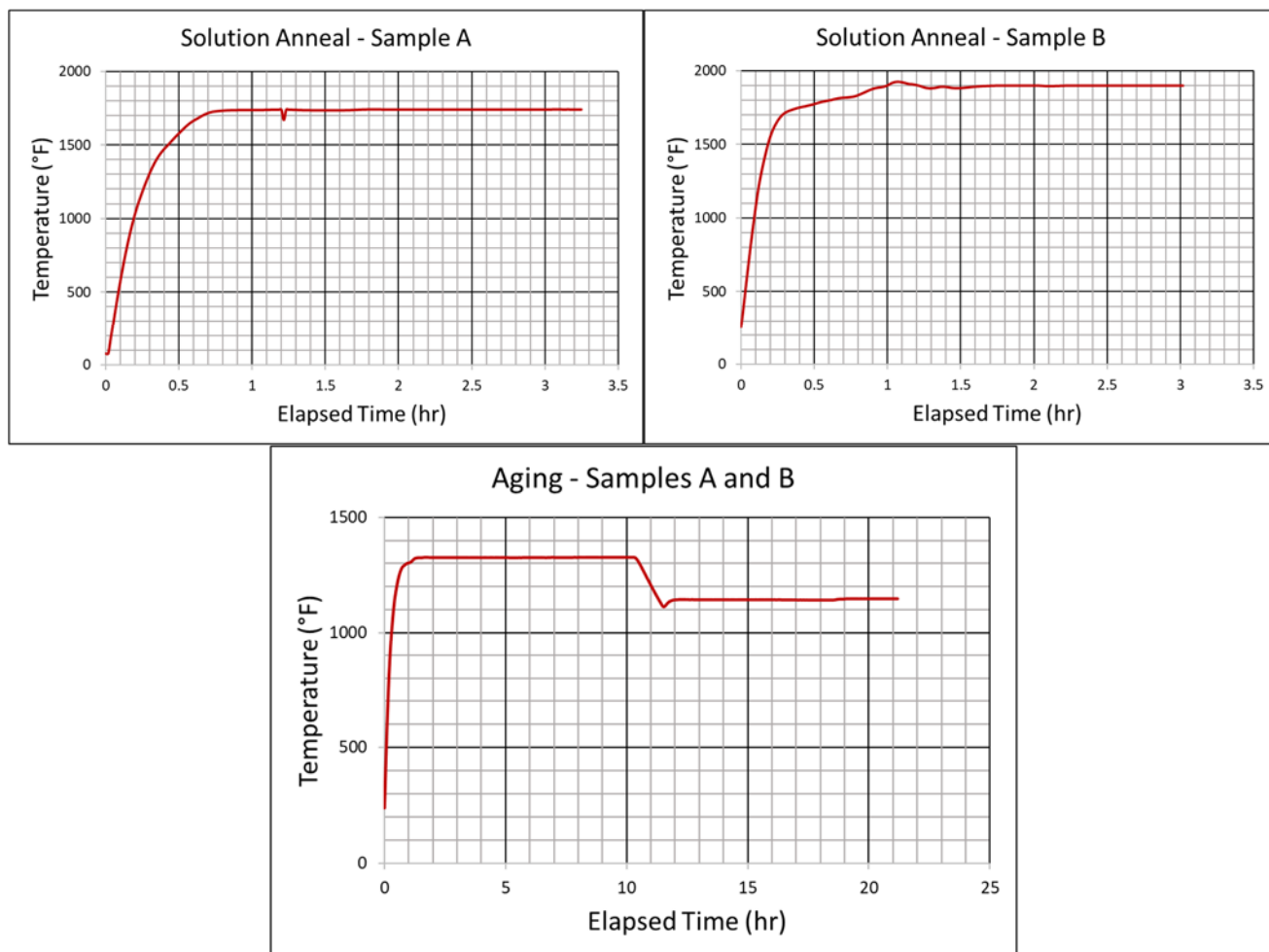


Figure 1: Heat treatment charts

The resulting microstructures were characterized by preparing cross-sections from each sample near mid-radius. The cross-sections were polished and etched with Kalling's No. 2 etchant to reveal the microstructures. Figure 2 and Figure 3 show the microstructures at 100X and 500X, respectively. The microstructure of Sample A clearly shows the presence of grain boundary and acicular δ phase. The microstructure of Sample B shows that the microstructure was largely free of δ phase at a magnification of 500X. Figure 4 shows secondary electron images of grain boundary triple points for each sample, which confirmed the presence of δ phase in Sample A and the absence of δ phase in Sample B.

The tensile strength of each sample was determined by a single round bar tensile specimen prepared from near mid-radius and tested in accordance with ASTM A370¹⁴. The hardness was measured using Rockwell C scale tests. Results are shown in Table 2. The yield strength at 0.2% offset for Sample A was 1111 MPa (161.2 ksi), and the yield strength at 0.2% offset for Sample B was 1122 MPa (162.7 ksi), both of which satisfied the target minimum yield strength of 1034 MPa (150 ksi). Hardness and tensile strengths were similar for both samples. The most notable difference between the two samples was that Sample B exhibited a substantially higher reduction of area. There was insufficient material to evaluate the impact toughness of either sample.

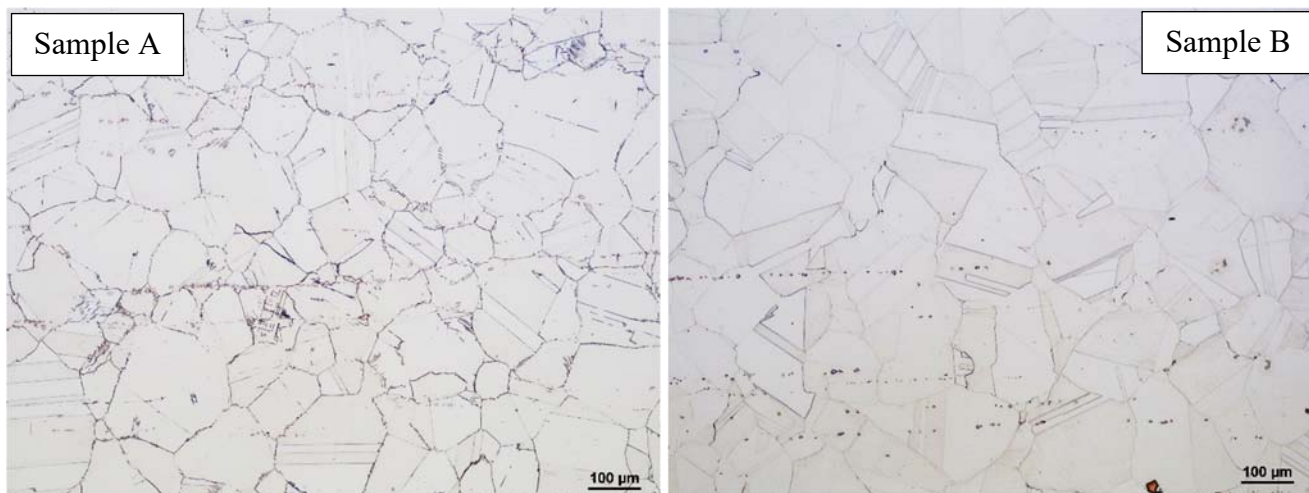


Figure 2: Microstructures at 100X original magnification. Kalling's No. 2 etchant.

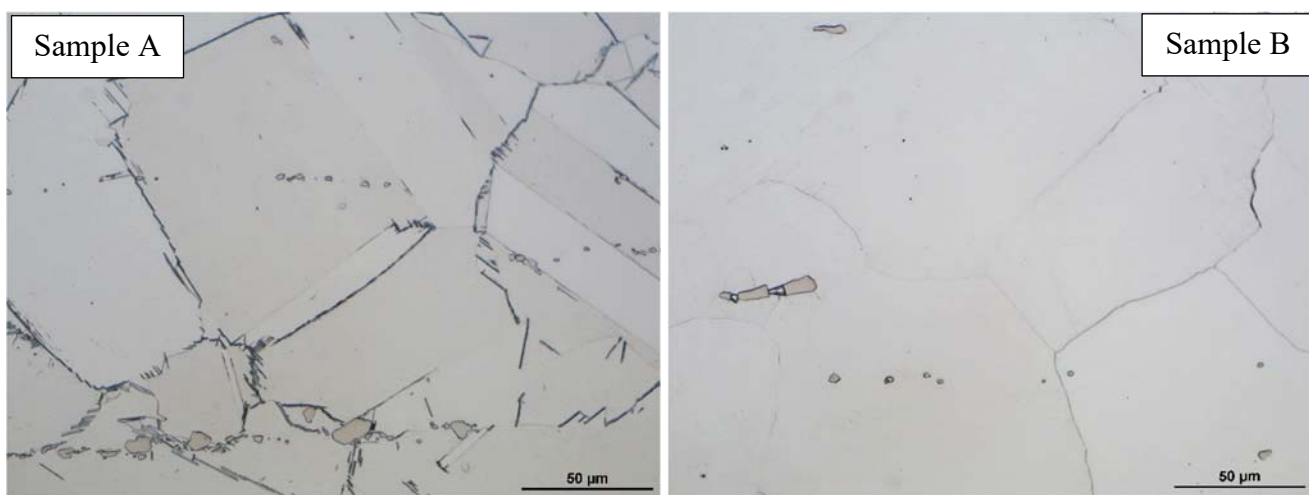


Figure 3: Microstructures at 500X original magnification. Kalling's No. 2 etchant.

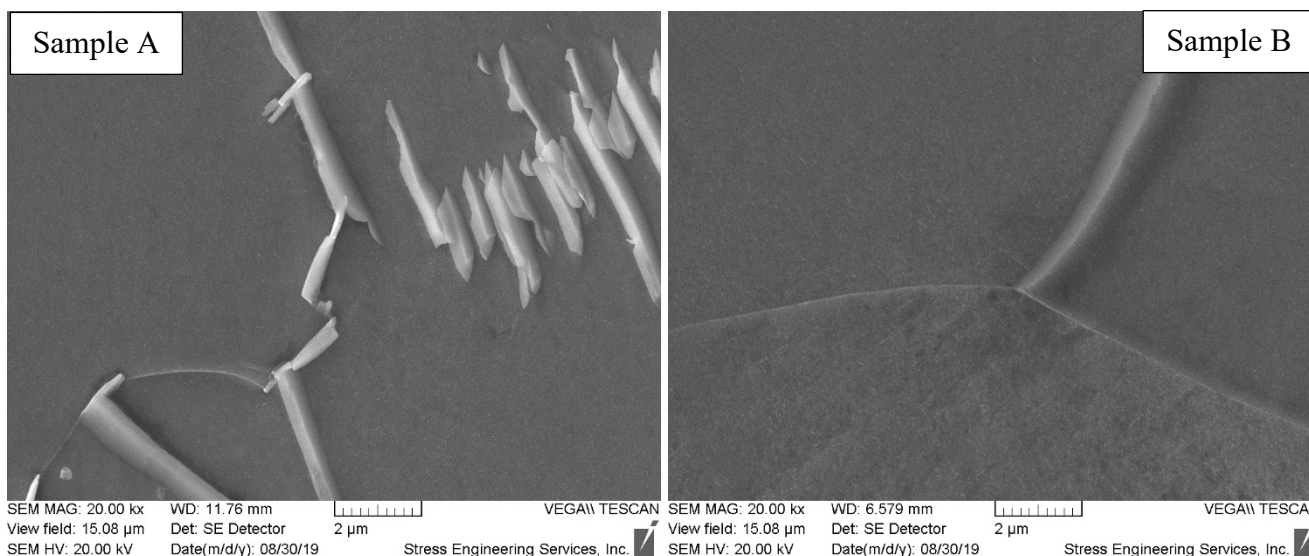


Figure 4: Secondary electron images showing difference in δ phase precipitation. 20,000X original magnification.