CHARACTERIZATION OF STRESS CORROSION CRACKING (SCC) IN LIQUID NITROGEN PROCESSED AERMET® 100 ULTRA HIGH STRENGTH STEEL (UHSS)

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Abstract
The objective of this work is to test the hypothesized beneficial effect of cryogenic processing on stress corrosion cracking (SCC) of a modern ultra-high strength steel, Aermet® 100. The mechanistic justification is elimination of thin-film austenite at martensite lath interfaces. Thin film austenite becomes hydrogen saturated due to environmental exposure during service; and transforms to embrittled martensite under crack tip strain. Experiments yielded two dramatically different classes of SCC behavior for fatigue precracked single edge notch (SEN) specimens stressed in 3.5% NaCl solution near the free corrosion potential. One-half of eight replicate experiments with the LN2 chilled condition evidenced immunity to SCC, while four experiments yielded significant transgranular SCC identical to that observed for commercially processed Aermet® 100 UHSS. While the deleterious role of residual austenite is logical and justified theoretically, there is no direct evidence for LN2-dependent change in the typically low amounts of this austenite. An explanation for the bimodal SCC response based on heterogeneous CrS dissolution is supported by measured-high Cr enrichment in the corrosion product present only on fast SCC surfaces; however, CrS is not evidenced in the microstructure. The technological impact of near-immune SCC response for ultra-high strength steel justifies continued research on the role of residual austenite.

Introduction
Aermet® 100 is used in applications such as aircraft landing gear and fasteners due to its extraordinary combination of plane strain fracture toughness (130 MPa\(\sqrt{m}\)) and tensile yield strength (1725 MPa) when commercially processed to the mildly overaged condition [1,2]. The microstructure consists of stable TiC dispersoids and nano-scale (Cr,Mo)\(_2\)C carbides in a fine lath/plate martensite matrix with a small amount of retained/precipitated austenite [2,3]. This steel is typically treated with rare earth addition to getter tramp elements [2]; eliminating the intergranular cracking associated with hydrogen embrittlement and SCC [4,5]. Critically, however, Aermet® 100 is susceptible to embrittlement due to atomic hydrogen (H) from both internal and external environmental sources resulting in severe transgranular cracking as shown in Figure 1 [6,7].

Figure 1 Crack surface morphology for internal hydrogen embrittlement of commercially processed Aermet® 100 [7].

Transgranular cracking from internal hydrogen embrittlement was shown to occur along martensite lath interfaces in tempered
5.5%Ni steel [8][9]. Such transgranular cracking propagated along \{110\} quasi-cleavage planes correlating with the favored \{110\} martensite lath interfaces. Morris also showed that austenite was present at lath interfaces and may participate in SCC and H-cracking [8]. For commercially heat treated AerMet® 100 steel, a dry ice (-73°C) chill is used after austenization to reduce the amount of retained austenite in the microstructure, optimizing the strength and fracture toughness [2]. This treatment may not transform all retained austenite. Moreover, austenite will precipitate at lath interfaces during aging at temperatures used for commercial heat treatment [3]. Critically, austenite forms in a thin-film morphology; coupled with fine-scale martensite-lath morphology, this austenite should provide an interconnected path through the AerMet® 100 microstructure.

Following Morris and coworkers [8], we expect that this austenite will contain an abnormally high level of H, dissolved during stress corrosion exposure. This metastable austenite will transform to H-supersaturated martensite at 23°C in the presence of high crack tip stress and plastic strain [10]. Transformed martensite will provide a connected crack path for transgranular SCC with respect to the prior austenite grain structure. The fractograph in Figure 1 shows a morphology that is consistent with this speculative cracking scenario. We hypothesize that addition of multiple liquid nitrogen chills (-196°C) to the standard heat treatment schedule will reduce the amount of residual austenite present in the microstructure of AerMet® 100, thus improving the H-damage and SCC resistance of this steel. The objective of this research is to test this hypothesis.

### Experimental Procedure

A 15 cm diameter forged cylinder of AerMet® 100 steel, of the composition shown in Table 1[6, 7], was obtained from Carpenter Technology and sectioned into 1.6 cm thick discs for heat treatment.

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Table 1 Composition of Aermet®100 (weight %) [7].

Control specimens were heat treated according to the commercial approach for AerMet® 100 steel: austenitize in vacuum at 885°C for 30 minutes, quench to 23°C using pressurized N₂, chill to -73°C and hold for 1 h, temper to peak age (482°C) for 5 h, and air cool [2]. The effect of variable chill temperature on SCC resistance was examined in two experimental groups: no chill and liquid nitrogen chill (LN₂). No chill specimen blanks were processed in an identical fashion as commercial AerMet®100 without refrigeration. Identical LN₂ blanks were cut from the original forged bar, austenitized in vacuum at 885°C for 1 h, quenched to 23°C in oil, refrigerated in liquid nitrogen (-196°C) for 5 h, tempered at 482°C for 5 h, water quenched to room temperature and refrigerated for an additional 5 h in LN₂. Rockwell hardness testing showed little difference between the 3 experimental groups, implying that variable chill temperature did not significantly alter the mechanical properties of AerMet® 100, from the commercial HT, shown in Table 2 [6, 7].

<table>
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<th>σYS (MPa)</th>
<th>UTS (MPa)</th>
<th>R.A. (%)</th>
<th>E (GPa)</th>
<th>KIC (MPa√m)</th>
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<td>65</td>
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Table 2 Mechanical Properties of Aermet®100
Heat treated blanks were machined into single edge notch fracture mechanics (SEN) specimens with a flat gauge section nominally 35 mm long, 10.2 mm wide, and 2.54 mm thick. During SCC testing, mode I loading was applied in the circumferential (C) direction of the original forging with crack growth in the radial (R) or length (L) dimensions. Due to the isotropic microstructure of AerMet® 100, the two orientations (C-R, C-L) are considered to be identical. Edge notches were machined into each sample via EDM machining to a depth of 110 ± 10 µm. To ensure a consistent-sharp crack front during SCC testing, all specimens were fatigue precracked in air, under decreasing ΔK (constant $K_{\text{min}}/K_{\text{max}} = 0.10$), yielding a total crack plus notch depth of ~500 µm at a final $K_{\text{max}}$ level of 8 MPa√m.

**SCC Testing**

A servo-electric, feedback controlled testing machine (89 kN force capacity) operating in grip displacement control, was used for SCC testing of the fatigue precracked SEN specimens. Samples were loaded in mode I via constant slow displacement (0.0005 mm/min) while encapsulated in an acrylic environmental cell filled with circulating non-deaerated 0.6M NaCl solution. An external potentiostat maintained the sample at a constant potential throughout the test. Specimen load, actuator displacement, crack length, potentiostat current, and test time were computer-logged at 60s intervals. Crack length was calculated via the direct current potential difference (dcPD) method. Alumel wires were spot welded ~0.5 mm above and below the edge notch, allowing for the measurement of the potential difference when 4.000 +/-0.004 A of direct current is passed through the grips/sample from an external current source. This potential is converted to crack length via Johnson’s equation [11]. Thermal voltage fluctuations were minimized through current reversal: 1120 dcPD readings were recorded at 10 A and then 1120 dcPD readings were recorded at -10 A with a 0.5 s delay between the measurements. These dcPD readings were averaged to a single-true dcPD value that was recorded with the other test parameters at set intervals. Following HEAC testing, samples were removed from the cell and fractured in air using high ΔK fatigue at 20 Hz, then ultrasonically cleaned in acetone and methanol, and stored under vacuum.

![Figure 2](image-url)  
See Figure 2: Effect of chill temperature on SCC growth rates for AerMet® 100 stressed in 3.5% NaCl solution at -550 mV_{SCF}. [6]

Scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDS) were employed to investigate differences in the fracture surfaces of the LN$_2$ SCC samples. Fractographic analysis was conducted on a JSM 840 SEM at 15kV accelerating voltage using secondary electron imaging (SEI). Fracture surface morphology was analyzed over the length of the crack path for evidence of hydrogen assisted fracture mechanisms. Corrosion by-product surface composition
was measured via quantitative EDS against a sample of non-corroded AerMet® 100 of a known composition. The progression of corrosion by-product composition along the crack path was captured in 3 EDS spectra for each sample; a 50 µm x 80 µm area (500x) was scanned each time.

Results and Discussion

The addition of cryogenic processing to the commercial heat treatment of AerMet® 100 initially appears to have a dramatic effect on SCC resistance for slow-rising K loading in 3.5% NaCl at a fixed potential of -550 mV_{SCE}, which is about 40 mV cathodic of a the typical open circuit potential (OCP) of this steel. Similar and significant SCC is observed (Figure 2) for the commercial and chill-free conditions; a low value of threshold stress intensity for the onset of subcritical crack growth, as well as similar values of Stage II crack growth rates in the range of 10 to 100 nm/s for K levels between 15 and 50 MPa√m.

The data in Figure 2 show that the -73°C chill after austenitization is not necessary from the SCC perspective. However, this chill step is important for yield strength-toughness optimization [2,3]. Critically, note the beneficial effect of cryogenic processing on SCC resistance. For K levels up to 46 MPa√m, crack growth rates calculated from the dcPD measurements were less than 1 nm/s and just above the operational resolution limit of the dcPD measurement method (0.25nm/s). More recent experiments have revealed an extraordinary but reproducible bimodal diversity in the SCC resistance of LN2 processed AerMet® 100. This behavior is represented in Figure 3 which is a plot of the effect of applied potential on da/dt for AerMet® stressed at a K of 40 MPa√m and loading rate of ~1 MPa√m/h. The strong potential dependence of da/dt for commercially processed steel is given by (●), showing near-immunity to SCC at -600 to -700 mV_{SCE} and severe cracking for both more cathodic and anodic polarization as well as more anodic polarization [6]. The electrochemical basis for this behavior was established previously and is of engineering importance given the ability of various metallic coatings such as Cd or Al to galvanically polarize ultra-high strength low alloy steels such as AerMet® 100 [6]. The LN2 chilled specimens (●) exhibit two different SCC behaviors when tested in NaCl solution at -550 mV_{SCE}. Four replicate tests show a departure from the commercial SCC behavior, exhibiting crack growth rates which are 2 orders of magnitude below that of commercially processed AerMet® 100, following the LN2 behavior detailed in Figure 2. In contrast, four identical LN2 specimens stressed at -550mV_{SCE} showed the similar-severe SCC behavior documented for multiple specimens of commercially processed
AerMet® 100. The LN₂ processed specimens exhibited near immunity for 4 of 8 experiments, but severe-similar SCC for the remaining 4 of these 8 experiments. In all experiments, SCC growth was in a mode I orientation without significant crack path branching previously interpreted as caused by alloy element (Cr, Ni and Mo) segregation that favored intergranular SCC in AerMet® 100 [12]. This extreme variation in SCC behavior persists across laboratories and experimenters. No testing or specimen preparation artifact was found to explain this bimodal transgranular SCC behavior for LN₂ processed specimens, indicating that the mechanism for this divergent cracking must be intrinsic to the alloy.

An SEM fractographic analysis of the fast and slow growth LN₂ samples confirmed the dramatic difference in SCC resistance suggested in Figures 2 and 3. As illustrated in Figure 4, a fast growth sample exhibited severe transgranular H cracking (upper image), while the slow growth specimen exhibited no SCC (lower image). The slow growth sample has a distinct featureless zone, ~10 µm in width, present along the boundary between the fatigue precrack and post-test fatigue regions for the majority of the boundary. The morphology and location suggest that this is a “stretch zone” produced by crack tip blunting. This implies that the minimal amount of crack growth seen during the HEAC (Hydrogen Environment Assisted Cracking) testing is actually caused by electrical resistivity increase due to crack tip plasticity. The slow growth LN₂ processed samples represented in Figures 2 and 3 are in fact immune to SCC in the specified lab conditions.

**Figure 4** Fractographic comparison of the fatigue precrack-to SCC transitions for: (upper) fast growth LN₂ chilled sample, and (lower) slow growth LN2 chilled sample. Crack growth is from top to bottom and the fatigue precrack is imaged in the top half of each SEM fractograph.

**Mechanistic Interpretations**

Given the technological importance of resistance to SCC and hydrogen embrittlement in ultra-high strength steels; it is important to understand the cause(s) of the behavioral variations represented in Figures 2 and 4. The dramatic improvement in the SCC resistance of LN₂ processed AerMet® 100 appears to validate the hypothesis that a low volume fraction of thin film austenite, formed on martensite lath interfaces from commercial aging practices, is the primary cause of the SCC of this steel [3][10]. Strain induced
transformation of H-saturated austenite to brittle thin-film martensite provides a logical scenario for transgranular (with regard to prior austenite gain boundaries) cracking during SCC. However, the bimodal results shown in Figure 3 demonstrate that SCC in UHSS is not fully understood.

Austenite Measurements
We have assumed that LN$_2$ processing removes essentially all retained and precipitated austenite that is otherwise present in low amounts (1 to 4 vol pct [3]) in commercially processed AerMet®100. However, no direct experimental evidence has been gathered to substantiate this hypothesis. X-ray diffraction (XRD) measurements of coupons of commercially and LN$_2$ processed AerMet®100 yielded identical-average total austenite contents of 1.92 vol pct (-73°C chill) and 1.99 vol pct (LN$_2$ chill). These values are consistent with those reported by Ayer and Machmeier [3]. It is possible that the presence of austenite in an amount less than that below the reproducible detection limit of XRD controls transgranular SCC by the proposed H cracking mechanism [8,10]. Therefore, future transmission electron microscopy is required to establish the impact of LN$_2$ processing on residual austenite, both its amount and distribution in the complex lath martensite microstructure.

CrS Hypothesis
It is equally important to understand the origin of the bimodal -550 mV$_{SCE}$ cracking behavior shown in Figure 3 if cryogenic processing is to be transitioned to processing of AerMet®100 and other SCC-susceptible UHSS which are invariably going to contain residual austenite. Our working hypothesis for the

systematic variation in behavior is focused on a secondary competing mechanism which degrades the intrinsically beneficial LN$_2$ heat treatment. Stage II crack growth rates are controlled by the local concentration of hydrogen (C$_{H-TIP}$) delivered from the surface in equilibrium with the local crack solution to the crack tip fracture process zone [5, 6]. For applied potentials near open circuit, C$_{H-TIP}$ depends on the occluded crack tip electrochemistry: pH and over-potential for H production. This local crack tip pH at OCP will be dominated by metal cation hydrolysis from crack tip anodic dissolution. For commercially processed AerMet® 100, Fe$^{+3}$ hydrolysis is believed to dominate over Cr$^{+2}$ and other metal cations, resulting in a crack tip pH of ~4 and a sufficient amount of hydrogen production to embrittle the microstructure; producing the characteristic transgranular HEAC susceptibility. If we assume the austenite content of the alloy is reduced via LN$_2$ processing; an intrinsic resistance to SCC would result by limiting this amount of crack tip H. However, if an alternate-alloyed specie, such as Cr, can dissolve to produce ions in the crack solution (Cr$^{+3}$), then Cr$^{+3}$ dominated hydrolysis will lower crack tip pH to ~2.5 and C$_{H-TIP}$ will increase. In this scenario, it is possible that increased H will overwhelm the intrinsic benefit of reduced austenite content provided by LN$_2$ processing.

The question then centers on the mechanism for specimen to specimen variation and the source of alternate metal specie cations, such as Cr$^{+3}$. CrS inclusions, formed rather than Ce or La based oxi-sulfides, due to inadequate rare earth gettering, are a logical chromium ion source [13-15]. Such inclusions can form in the microstructure if rare earth additions are
inadequate to combine with all impurity sulfur, or if such additions are heterogeneous in the ingot. CrS is readily soluble in acidic chloride solutions typical of Fe-cation hydrolysis dominated crack tips [16].

Quantitative EDS was performed on SCC surfaces, which were cleaned ultrasonically in acetone and methanol after specimen fracture. Elemental compositions were calibrated using a polished-bulk sample of AerMet® 100. The results are presented in Table 3 as the element content of corrosion product and underlying alloy, normalized to the standard alloy composition, for different parts of the cracked specimen surface. The deviation in elemental content of the fracture surface, from the standard, correlates with SCC growth rate variation. Notably, chromium enrichment supports the hypothesis of CrS dissolution. Cr is enriched substantially for the commercially heat treated specimen which was susceptible to SCC. Significant, but less intense Cr enrichment is measured for the fast-SCC growth LN2 specimen surface, implying a modest intrinsic benefit of LN2 processing. The Mo enrichment is notable but complicated to interpret since EDS peaks associated with Mo L\(_{\alpha1}\) (2.293 KeV) and S K\(_{\alpha1}\) (2.037 KeV) overlap. Mo levels can still be reported to reasonable accuracy due to Mo K\(_{\alpha1}\) peak analysis. Mo-S peak overlap, coupled with intrinsically low bulk-S content (0.0008 wt pct) of AerMet® 100, precludes any definitive-quantitative analysis of S enrichment. The measured Cr enrichment follows the hypothesis that LN2 processing provides an intrinsic, albeit inadequate, beneficial effect on SCC resistance in the presence of CrS inclusions. Quantitative elemental EDS mapping was also employed to analyze the distribution of elements on each part of the

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<tr>
<td>Cr</td>
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Table 3 Quantitative EDS results for crack surface areas in AerMet® 100, processed with commercial or LN\(_2\) chilling, reported against a standard EDS spectra obtained from a metallographically polished, non-corroded AerMet® 100 surface. Crack surface. At 500x (50 µm x 80 µm area), no elemental localization could be detected; consistent with the hypothesized mechanism that Cr dissolves to participate in a hydrolysis reaction that raises crack H\(^+\) concentration and forms a uniformly precipitating chromium hydroxide phase crack wake. Similar

### Table 3

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\[\text{wt}\%_{\text{sample}}/\text{wt}\%_{\text{standard}}\]
environmental exposure times preclude crack wake electrochemistry artifacts in the measurements. That is, if the fatigue precrack tip intersected CrS inclusions, then Cr\textsuperscript{+3} dissolution, lowered pH, and corrosion product precipitation should have progressed as proposed in each sample. Following our CrS hypothesis, the only explanation for the absence of Cr in the corrosion product is insufficient CrS particles were present in the fatigue crack.

The EDS evidence of Cr enrichment on SCC-prone crack surfaces (Table 3) qualitatively and logically supports the hypothesized role of heterogeneous CrS precipitates in governing the bimodal cracking behavior (Figure 3). An SEM analysis was conducted on the specimens represented in Figure 3, particularly the fast and slow growth LN\textsubscript{2} processed specimens tested at -550 mV\textsubscript{SCE}. High magnification imaging of metallographically prepared sections has not revealed the presence of any CrS precipitates in the present lot of AerMet\textsuperscript{®} 100; on a size scale expected based on prior research with similar steels [10, 13].

**Time to Failure Modeling**

Research continues to definitively understand the strong-bimodal SCC behavior of LN\textsubscript{2} processed AerMet\textsuperscript{®} 100, and the general role of residual austenite in SCC of ultra-high strength steels, including the hypothesized benefit associated with austenite-reduced H diffusivity in the complex AerMet\textsuperscript{®} 100 microstructure [10, 18]. The interaction between a strong potential dependence of da/dt near free corrosion levels and reduction of thin film austenite by LN\textsubscript{2} processing are of particular interest. The importance of the potential benefit of LN\textsubscript{2} processing is illustrated by the time to failure predictions in Figure 5. Here, the da/dt versus K kinetic data shown in Figure 2 was used as a SCC growth rate law which inputs to a fracture mechanics model to predict the SCC life. The predicted lives for the No Chill and -70°C chill experimental groups agree with published experimental time-to-failure results for this steel in full immersion chloride solution at near free-corrosion conditions [17]. Critically, the projected SCC lives based on the slow da/dt values for the LN\textsubscript{2} processed specimens are in excess of two orders of magnitude longer than those predicted for the commercially chilled case. Since SCC growth reported in Figures 2 and 3 for the slow growth LN\textsubscript{2} specimens is a dcPD artifact of crack tip plasticity (see Figure 4), then the SCC lifetimes shown in Figure 5 are substantial underestimates of the benefits that LN\textsubscript{2} processing can provide.

**Conclusions**

- Liquid nitrogen chilling of AerMet\textsuperscript{®} 100 steel, after both austenitization and
aging, results in immunity to transgranular SCC, under slow-rising stress intensity loading of precracked specimens immersed in 0.6M NaCl solution at -550 mV_{SCE}, for ~50% of specimens tested to date.

- The extreme SCC resistance of LN$_2$ processed AerMet® 100 is only observed in half of the replicate experiments conducted; the cause of this bimodal behavior is not yet understood. Bimodal behavior persists across laboratories and experimenters, with no observable trend.

- The proposed-deleterious role of residual austenite in SCC by the hydrogen environment assisted cracking mechanism is reasonable, but not supported by direct observations of chill-dependent low-level austenite content variation.

- The role of CrS inclusions and occluded crack chemistry change, as the source of the bimodal variation of SCC growth rate, is supported by Cr enrichment of fast-growth SCC surfaces, but not by direct observations of this undesirable phase.

- The technological impact of near-immune SCC response for modern ultra-high strength steels justifies continued research on the role of residual austenite interacting with crack electrochemistry.

Acknowledgements

The author gratefully acknowledges the contributions and collaborations of Yongwon Lee, Dr. Warren M. Garrison, and Dr. Richard P. Gangloff (advisor). This research was sponsored by an STTR on Development of Corrosion-Hydrogen Crack Resistant Aircraft Alloys from Mechanistic Understanding (STTR Topic # N08-T010, Phase I, and Contract #N68335-09-C-0378) with Dr. Jeffrey Waldman as Principal Investigator at Navmar Applied Sciences Corporation and Dr. William E. Frazier as Program Officer at NAVAIR (Patuxent River, MD). Financial support for G.L.P. was provided by the DoD Office of Corrosion Policy and Oversight, lead by Mr. Daniel J. Dunnire, through a funding agreement administered by the United States Air Force Academy

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