# FINAL

# Peer Review Summary Report for the External Peer Review of

# Final TAP 766 Report:

***TAP 766: Characterizing the Behavior of Inconel Clad A387 Steel (ASTM A387 Grade F22, Class 2) in High Pressure-High Temperature, Corrosive Environments — Material Models for Fatigue and Fracture Properties of Inconel 625 Cladding–Final Report (April 30, 2018)***

## VOLUME 2: Summary of Peer Review Panel Meeting

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EnDyna, Inc.

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## Contents

# 1. PEER REVIEW PANEL MEETING OVERVIEW

**Volume 2: Summary of Peer Review Panel Meeting** represents the second of three volumes for the peer review summary report documenting the external panel peer review of the final report for the BSEE study entitled, *TAP 766: Characterizing the Behavior of Inconel Clad A387 Steel (ASTM A387 Grade F22, Class 2) in High Pressure-High Temperature, Corrosive Environments — Material Models for Fatigue and Fracture Properties of Inconel 625 Cladding–Final Report* (April 30, 2018).

**Volume 2** of the peer review summary report provides information about the March 5-6, 2019 peer review panel meeting held at EnDyna's office in McLean, Virginia. For additional information about the peer review:

- Refer to **Volume 1** of the peer review summary report for the final written peer review comments from the selected expert peer reviewers.
- Refer to **Volume 3** of the peer review summary report for the public comments on the final TAP 766 report. Volume 3 also summarizes information about the April 24, 2019 conference call scheduled as an addendum to the March 5-6, 2019 panel meeting to discuss the compiled public comments on the final TAP 766 report.

Section 1 of **Volume 2: Summary of Peer Review Panel Meeting** provides an overview of the panel meeting. Section 1.1 includes an introduction to the peer review panel. Section 1.2 provides the objective and scope of this peer review, as outlined in the peer review charge document provided to the peer reviewers. Section 1.3 summarizes the process for requesting BSEE's written responses to the peer reviewer questions. Section 1.4 outlines the "ground rules" for the peer review panel meeting and Section 1.5 briefly describes the requirements to provide an impartial peer review. Finally, Section 1.6 outlines the organization of **Volume 2** of the peer review summary report.

## 1.1 Introduction

EnDyna selected a peer review panel of four senior scientists (see Table 1) with expertise in:

1) Material science and engineering, corrosion background, metallurgy engineering background.
2) Practical experience and knowledge of corrosion behavior including stress corrosion cracking of nickel-based alloys, metallic material fatigue, and fracture behavior.
3) Practical experience with design of offshore equipment in high-pressure and high-temperature environments.
4) Practical experience with metallic material testing and evaluation, metallography, material properties testing, microscopy, mechanical testing, corrosion testing, environmental testing, and analytical chemistry.

Each peer reviewer prepared an initial written review of the final report of the BSEE study entitled, *TAP 766: Characterizing the Behavior of Inconel Clad A387 Steel (ASTM A387 Grade F22, Class 2) in High Pressure-High Temperature, Corrosive Environments — Material Models for Fatigue and Fracture Properties of Inconel 625 Cladding–Final Report* (April 30, 2018). The peer reviewers submitted their initial written review to EnDyna prior to the March 5-6, 2019 peer review panel meeting. EnDyna compiled the initial written comments and distributed those compiled initial written comments to all peer reviewers on February 6, 2019 for their review prior to the peer review panel meeting.

The peer review panel meeting was held on March 5-6, 2019 at EnDyna's office in McLean, Virginia. Each peer reviewer attended and participated fully in the 2-day peer review panel meeting. The EnDyna Peer Review Lead and the EnDyna Facilitator managed the peer review panel meeting with the four peer reviewers. The purpose of this meeting was to encourage discussion among the peer reviewers to result in a robust and insightful review that was centered on BSEE's Charge Questions. BSEE did not participate in the deliberations of the peer review panel meeting, but the BSEE COR and other BSEE staff were available, if needed, to provide information on the history and background of this TAP 766 study.

As part of **Volume 2** of the final peer review summary report, **Section 2** presents the meeting summary developed by EnDyna from the discussion at the peer review panel meeting. **Section 3** presents the agenda for the peer review panel meeting.

**Table 1. Peer Reviewers for Final TAP 766 Report**

| Name | Affiliation | Credentials / Advanced Degrees |
|---|---|---|
| **Robert (Bob) Badrak, PE, FNACE, FASM** | **Director of Engineering Materials**, Weatherford International, Houston, TX | • FNACE: Fellow of NACE International (The Corrosion Society), elected 2019<br>• FASM: Fellow of ASM International (The Materials Information Society), elected 2010<br>• MSE, Metallurgical Engineering, University of Michigan, 1977 |
| **Jeffrey (Jeff) Hawk, PhD, FASM (Alternate)** | **Materials Research Engineer**, National Energy Technology Laboratory (NETL), U.S. Department of Energy (DOE), Albany, OR | • FASM: Fellow of ASM International (The Materials Information Society), elected 2001<br>• PhD, Materials Science, University of Virginia, 1986<br>• MS, Materials Science, University of Virginia, 1983 |
| **Raúl Rebak, PhD, FNACE, FASM** | **Senior Corrosion Scientist**, GE Global Research Center, Schenectady, NY | • FASM: Fellow of ASM International (The Materials Information Society), elected 2019<br>• FNACE: Fellow of NACE International (The Corrosion Society), elected 2014<br>• PhD, Corrosion and Metallurgy, The Ohio State University, 1993<br>• MS, Chemical Engineering, National University of Misiones, Argentina, 1982 |
| **Elizabeth Trillo, PhD** | **Principal Engineer**, Environmental Performance of Materials Section, Materials Engineering Department, Mechanical Engineering Division, Southwest Research Institute (SWRI), San Antonio, TX | • PhD, Materials Science and Engineering, The University of Texas at El Paso, 1997<br>• MS, Metallurgical and Materials Engineering, The University of Texas at El Paso, 1994 |

## 1.2 Peer Review Objective

The final TAP 766 report meets the criteria for "Highly Influential Scientific Assessment" under the Office of Management and Budget (OMB) "Final Information Quality Bulletin for Peer

Review" (OMB M-05-03, December 2004). Therefore, BSEE determined that this report contains new scientific information that shall be subjected to peer review. This study's findings may have a direct bearing on the methods, industry standards, best practices, and material selection for equipment utilized for HPHT offshore oil and gas operations. This study's results may suggest the need for revisions of respective industry standards and could affect how BSEE and industry interpret those standards. The results from this study are important for reviewing BSEE's proposals for new technologies for offshore oil and gas operations under HPHT corrosive environments.

The objective of this external panel peer review was for BSEE to receive comments from individual experts on the final report of the BSEE study entitled, *TAP 766: Characterizing the Behavior of Inconel Clad A387 Steel (ASTM A387 Grade F22, Class 2) in High Pressure-High Temperature, Corrosive Environments — Material Models for Fatigue and Fracture Properties of Inconel 625 Cladding–Final Report* (April 30, 2018). This panel peer review was scientific and technical in nature, reviewing the methods, assumptions, data quality, the strengths of any inferences made, and the overall strengths and limitations of the study.

## 1.3 BSEE Charge for Scope of Peer Review

To focus the peer review process effectively on BSEE's Charge Questions, BSEE carefully defined the scope of this peer review for the final report of the BSEE study entitled, *TAP 766: Characterizing the Behavior of Inconel Clad A387 Steel (ASTM A387 Grade F22, Class 2) in High Pressure-High Temperature, Corrosive Environments — Material Models for Fatigue and Fracture Properties of Inconel 625 Cladding–Final Report* (April 30, 2018).

EnDyna instructed the peer reviewers that their written peer review comments should stay within the BSEE Scope defined below. The peer reviewers were also informed that it was important to remember that this panel peer review was scientific and technical in nature, reviewing the methods, assumptions, data quality, the strengths of any inferences made, and the overall strengths and limitations of the study.

**BSEE Charge for the Scope of this Peer Review**
**The scope of this peer review is focused only on the <u>scientific and technical merit</u> of the assumptions, inputs, methodologies, modeling, and results for the BSEE study entitled, *TAP 766: Characterizing the Behavior of Inconel Clad A387 Steel (ASTM A387 Grade F22, Class 2) in High Pressure-High Temperature, Corrosive Environments — Material Models for Fatigue and Fracture Properties of Inconel 625 Cladding–Final Report* (April 30, 2018). This peer review is scientific and technical in nature and includes reviewing the methods, assumptions, data quality, the strengths of any inferences made, and the overall strengths and limitations of the study. The scope of the peer review includes the material, fabrication, computations, testing, engineering factors, modeling, results, and final recommendations generated from the TAP 766 study. As such, the peer reviewers should focus on providing comments on the <u>scientific and technical merit</u> of the TAP 766 study. Because this peer review is scientific and technical in nature, BSEE is not interested in comments focusing on editorial style.**

The following are considered **Out-Of-Scope** for this peer review; any and all **Out-Of-Scope** comments will not be considered by BSEE during this peer review process:

- BSEE is not interested in general comments related to high-pressure high-temperature (HPHT) equipment or environments, because: 1) this peer review is focused only on the methods and approach for testing in a sour-gas environment under HPHT conditions that were used in the TAP 766 study referenced above, and 2) this peer review is focused on the standards that were used in the TAP 766 study referenced above (see Tables 3 and 4 in final TAP 766 report).
- BSEE is not interested in comments on, or suggestions for, alternative fatigue and fracture testing methods, except for comments on any omissions or errors identified in the specific material testing methods used for testing in a sour-gas environment under HPHT conditions in the TAP 766 study referenced above, because this peer review is focused on the research already completed for this TAP 766 study.
- BSEE is not interested in comments about API RP 17TR8 because BSEE has already completed a peer review for a previous BSEE study evaluating methods recommended by API RP 17TR8. Comments about API RP 17TR8 will not be considered during this peer review.
- This peer review is scientific and technical in nature, and does not extend to BSEE policies or BSEE regulations. Comments related to BSEE policies and decisions or to current or proposed BSEE regulations will not be considered.

## 1.4 BSEE's Written Responses to Peer Reviewer Questions

To facilitate obtaining as much information as possible prior to the panel meeting, EnDyna's Peer Review Lead listed/paraphrased EnDyna's and the peer reviewer's initial questions about the final TAP 766 report. EnDyna provided BSEE a list of the peer reviewer's questions on December 5, 2018. EnDyna requested that BSEE provide responses to these peer reviewer questions in writing so that EnDyna could distribute written responses to all four peer reviewers in advance of the peer review panel meeting. EnDyna received BSEE's written responses on February 4, 2019 and reformatted to improve readability.

**Section 4** presents BSEE's written responses to the peer reviewer questions. EnDyna distributed BSEE's written responses to the peer reviewer questions to all four peer reviewers on February 14, 2019.

## 1.5 Peer Review Panel Meeting "Ground Rules"

The "ground rules" provided to the peer reviewers both prior to and during the peer review panel meeting are listed below:

- An external peer review is intended to solicit individual reviewer feedback, to increase the independence of the peer review process.
- The panel is not asked to, and should not attempt to, form consensus or collective recommendations, ratings, or opinions, and panel reviewers must understand that they should provide individual feedback on the research product.

- Any BSEE staff that may attend the panel meeting can only provide background information on the research product to the peer reviewers, which can occur only during the panel meeting run by EnDyna, and at EnDyna's request.
- The panel meeting will not include discussion related to BSEE policies and decisions or current or proposed BSEE regulations.

## 1.6 Conflict of Interest – Impartiality

Each peer reviewer's signature on their Conflict of Interest (COI) Form and their signed Non-Disclosure/Confidentiality Agreement (NDA) certified that each peer reviewer would provide an impartial, technically sound, objective, and independent scientific and technical review, or in other words, not provide a biased opinion in responding to BSEE's Charge Questions and in providing general impressions.

## 1.7 Organization of Report

**Volume 2** of this peer review summary report is comprised of seven sections, as listed below:

- **Section 1** provides an introduction to the subject matter experts selected by EnDyna for the peer review panel.
- **Section 2** presents the meeting summary developed by EnDyna from the discussion at the peer review panel meeting.
- **Section 3** provides the agenda developed by EnDyna for the peer review panel meeting.
- **Section 4** provides BSEE's written responses to the peer reviewer questions.
- **Section 5** provides EnDyna's TAP 766 study background presentation.
- **Section 6** presents a handout prepared by Mr. Badrak that identified the parts of ANSI/NACE MR0175 / ISO 15156 that Mr. Badrak considered relevant to the TAP 766 study.
- **Section 7** provides Reference [14] or the "project interim report" cited on page 9 of the final TAP 766 report.

# 2. PEER REVIEW PANEL MEETING SUMMARY

The peer review panel meeting was held on March 5-6, 2019 at EnDyna's office in McLean, Virginia. This section presents the meeting summary developed by EnDyna from the discussion at the peer review panel meeting.

Attendees:

- Ms. Amy Doll, EnDyna, Peer Review Lead
- Mr. Ken Rock, EnDyna, Facilitator
- Robert (Bob) Badrak, PE, FNACE, FASM, Expert Peer Reviewer
- Jeffrey (Jeff) Hawk, PhD, FASM, Expert Peer Reviewer (Alternate)
- Raúl Rebak, PhD, FNACE, FASM, Expert Peer Reviewer
- Elizabeth Trillo, PhD, Expert Peer Reviewer

## 2.1 Day-1: March 5, 2019

EnDyna's Facilitator, Mr. Ken Rock, opened Day-1 of the panel meeting at 9:00am by introducing himself. Mr. Rock asked EnDyna's Peer Review Lead, Ms. Amy Doll, to introduce herself. Each of the peer reviewers introduced themselves and provided some brief background on their expertise and experience.

Mr. Rock reviewed the "ground rules" for the peer review panel meeting (see Section 1.4). Ms. Doll briefly reminded the peer reviewers about the peer review process and the schedule for submitting final written peer review comments after the panel meeting.

**Background on TAP 766 Study**

EnDyna's Peer Review Lead, Ms. Doll, presented background information about the TAP 766 study. Prior to the panel meeting, the BSEE COR (Mr. Mark Kozak) had reviewed and approved EnDyna's background presentation. EnDyna sent the background presentation to the peer reviewers on February 28, 2019 for their review in advance of the panel meeting. **Section 5** provides EnDyna's TAP 766 study background presentation.

The peer reviewers noted that the background information about BSEE's TAP Program and the TAP 766 study was helpful, particularly the information about the $618,475 award value. Each of the peer reviewers acknowledged that the TAP 766 project team conducted a considerable amount of material testing given the available budget.

Ms. Doll also provided a brief overview of the federal government guidelines for a **Highly Influential Scientific Assessment** or **Highly Influential Scientific Information** peer review, which include requirements for a more rigorous peer review and greater transparency through public participation. This overview included a brief summary about the Office of Management and Budget (OMB) "Final Information Quality Bulletin for Peer Review" (OMB M-05-03, December 2004), and the BSEE "Peer Review Process Handbook" (dated May 2017), both of which were referenced in the peer review charge document provided to the peer reviewers.

Ms. Doll also explained that BSEE's public comment period should have started in mid-November 2018. As noted in the peer review charge document provided to the peer reviewers, EnDyna had expected to provide the relevant scientific and technical public comments to the peer reviewers for their review prior to the panel meeting. BSEE first experienced internal delays in making the announcement, and then the federal government shutdown caused further delays in BSEE's announcement of the public comment period. The public comment period for the final TAP 766 report began on February 18, 2019 and was open for six weeks until April 1, 2019. Ms. Doll mentioned that one option to accommodate for this unexpected delay would be to schedule a conference call later in April 2019 when the peer reviewers could discuss any relevant scientific and technical public comments received about the final TAP 766 report.[1]

**General Impressions: *Overall impressions addressing the accuracy of information presented, clarity of presentation, and soundness of conclusions*.**

Mr. Rock asked each peer reviewer to use around five minutes to provide a high-level summary of their general impressions for the BSEE study entitled, *TAP 766: Characterizing the Behavior of Inconel Clad A387 Steel (ASTM A387 Grade F22, Class 2) in High Pressure-High Temperature, Corrosive Environments — Material Models for Fatigue and Fracture Properties of Inconel 625 Cladding–Final Report* (April 30, 2018).

Dr. Trillo commented that the material tests in the final TAP 766 report represented a diverse and thorough evaluation of fatigue and fracture for the Inconel 625 clad material. Although acknowledging a wide range of material tests were conducted, Dr. Trillo stated that more detail could have been included in the report about the material testing methods. Dr. Trillo also stated that the report should have provided a more explicit and systematic description of the variables for each material test method. This additional detail was important, as Dr. Trillo believed this was a "toe in the water" study, which was intended for others to follow to increase understanding of cracking/fracture susceptibility of the Inconel 625 clad material and the additional test details would be important to accurately replicate test conditions in future work.

Mr. Badrak noted that overall the direction of the TAP 766 study was good; however, there were areas needing improvement. In particular, Mr. Badrak commented that the temperature of 350°F used in all elevated temperature tests for this project was too benign, and noted that 350°F was the lower boundary for the definition of HPHT environments. Mr. Badrak emphasized that temperature is most important (versus pressure) as an environmental component of the cracking/fatigue mechanisms. Mr. Badrak expressed concerns about project execution because material tests were conducted using only a single plate upon which two layers of Inconel 625 was weld applied. Moreover, Mr. Badrak noted that the weld overlay process was not described in detail in the report.

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[1] Volume 3 of the final peer review summary report summarizes information about the April 24, 2019 conference call scheduled as addendum to the March 5-6, 2019 panel meeting to discuss the compiled public comments on the final TAP 766 report.

Dr. Hawk commented that the TAP 766 project was well thought out and executed. Dr. Hawk observed that the report was a good start in establishing a baseline of information. Dr. Hawk stated that the report needed more documentation of the methodology, including the testing laboratory equipment used for each material test method. To support reproducibility by other researchers, Dr. Hawk emphasized that more specific information on how the material test methods followed the standards in Table 3 and Table 4 was necessary. It would be important for other researchers to know how closely the TAP 766 study followed the standards and, if there actually were any differences from the standards, to document specific information about any such differences from those standards.

Dr. Hawk also acknowledged making "a lot of assumptions" during initial review of the final TAP 766 report. More specifically, Dr. Hawk mentioned making assumptions that the testing laboratory had used generally accepted material testing protocols for the TAP 766 experiments. Dr. Hawk now observed that not much technical detail about the TAP 766 material testing protocol was actually documented in the report.

Dr. Trillo also acknowledged making assumptions during initial review of the final TAP 766 report that the testing laboratory had used generally accepted material testing protocols. Upon further evaluation, Dr. Trillo observed that the report lacked the necessary technical details to document completely the approach used for material testing in the TAP 766 study.

Dr. Rebak had no issues with the material tests that were selected, but stated that the data and results provided in the report were not put in the proper context. Dr. Rebak emphasized that the report lacked sufficient detail for anyone to reproduce or perform complimentary tests to increase confidence in the results reported by the TAP 766 study.

There was more discussion among the peer reviewers about the need to provide sufficient detail to support reproducibility by other researchers. Dr. Hawk suggested that more detailed technical information should have been provided in appendices to the report. Dr. Trillo and Mr. Badrak also commented that it was necessary to describe more systematically what was and was not included with respect to each standard (Table 3 and Table 4) for each of the material testing methods.

**Charge Question 1: Were the objectives of the study clearly defined (Section 1)? If not, what are your recommendations for improving the description of this study's objectives?**

Dr. Hawk stated that the objectives were clearly defined. After observing that this was a "scout" study given the available resources, Dr. Hawk commented that the results from the TAP 766 study do not provide a wide variation of data for alloy/clad combinations or sufficient information to verify general environment/property trends for such alloy/clad systems. Dr. Hawk noted that the TAP 766 study used only one heat (sample set) and commented that eventually it would be good to obtain material testing data for more heats (sample sets) from maybe 5-6 different cladding manufacturers. Dr. Hawk suggested that the report should include an explicit caveat stating clearly that because the material testing was based on only one heat (sample set), no conclusions can be drawn yet.

Mr. Badrak added that by using only one temperature (350°F) in the material tests for the TAP 766 study, the methodology did not evaluate environments "closer to the edge" of potential environment/property trends for alloy/clad systems. By not evaluating temperatures greater than 350°F, Mr. Badrak suggested that it would be difficult to draw conclusions about achieving an adequate margin of safety.

Dr. Rebak commented that the objectives should describe why the TAP 766 study wanted to perform those material tests. Dr. Rebak suggested that it was necessary to have better context for the TAP 766 study and emphasized that this is not the first time such tests were performed. In addition, Dr. Rebak noted that better context could be achieved by explaining what is currently known, and what and why new information was necessary from the material tests in the TAP 766 study.

Mr. Badrak stated that the objectives were clear; however, the report should have described the scope of the project better. Mr. Badrak observed that describing how limited the scope actually was for the TAP 766 study would help to understand the methodology. Although the scope was limited, Mr. Badrak noted that the TAP 766 study provided a good "springboard" for future studies.

Dr. Trillo commented that overall the objectives were good to identify the cracking/fracture behavior of the Inconel 625 clad material. Dr. Trillo stated that from a technical perspective, the report covered all the cracking mechanisms to adequately identify any cracking/fracture susceptibility of the Inconel 625 clad material. Dr. Trillo noted that the report should have stated that the material test results represented only one data set and thus it was a limited study and any use of data would be limited to this specific case.

**Group Discussion:  Background on ANSI/NACE MR0175 / ISO 15156**

Mr. Badrak provided background information about ANSI/NACE MR0175 / ISO 15156, followed by a group discussion. **Section 6** presents a handout prepared by Mr. Badrak that identified the parts of ANSI/NACE MR0175 / ISO 15156 that Mr. Badrak considered relevant to the TAP 766 study.

**Charge Question 2:  Were the analyses used for the pre-tested metallurgical analysis (Section 3) appropriately designed, clearly described, and adequately characterized? Were there any apparent strengths, weaknesses, omissions, or errors? Provide an explanation for your answers.**

Dr. Rebak commented that the metallurgical analysis of the fabricated slab of material was adequate; however, also observed that the report had gaps in characterization data. With respect to chemical composition, for example, Dr. Rebak stated that the spatial distribution of iron in the layers of the Inconel 625 cladding was provided in Figure 6, but the report did not include similar data on chromium and molybdenum. In addition, Dr. Rebak pointed out there was no image for nickel in Figure 5, which was intended to confirm the chemical elements in the Inconel 625 cladding.

Dr. Rebak and Mr. Badrak noted that chromium and molybdenum are the two elements that provide corrosion resistance, and actually make Inconel 625 a corrosion resistant alloy (CRA).

Mr. Badrak commented that the properties of the Inconel 625 weld overlay were not adequately characterized. In particular, Mr. Badrak expressed concerns that the iron content data reported for the two layers of the overlay were different from what would be expected for iron dilution. Specifically, Mr. Badrak would have expected the second layer (away from substrate interface) to have an iron content less than 5% given that the iron content in the first layer was relatively constant at about 8% at distances from 2000-4800 μm from the steel interface.

Mr. Badrak observed that a cladding weld overlay with two layers is the typical practice for industry. Mr. Badrak liked the approach of characterizing each of the two cladding layers because the top layer is important for corrosion resistance.

After observing that the report did not include thickness hardness traverses, Mr. Badrak emphasized that hardness traverses are important to adequately characterize the properties of a deposited weld overlay. Mr. Badrak explained that ANSI/NACE MR0175 / ISO 15156-2, referring specifically to Clause 7.3.3 and Figure 6, requires hardness traverses through the overlay and into the base metal. Mr. Badrak referred to Clause A.13 of ANSI/NACE MR0175 / ISO 15156-3 as the relevant reference section for how to define weld overlay properties completely.

Dr. Trillo commented that the optical micrographs in the report were good. Dr. Trillo stated that the second clad layer (away from substrate interface) was not characterized properly. Dr. Trillo also commented that hardness traverse measurements for the Inconel 625 weld overlay should have been included in the report.

Dr. Hawk commented that the chemistry of the weld overlay was not clearly described in the report. After looking more thoroughly at iron dilution in Figure 6 of the report, Dr. Hawk expressed concerns that the iron content values were not reasonable. Dr. Hawk stated that the metallurgical analysis of the material was not clearly described and expressed concerns that the report had more ambiguity on iron content than necessary.

Mr. Badrak emphasized again that there was something peculiar about the iron content measurements in the report. Mr. Badrak pointed to the last bullet on page 5 of the report. Dr. Hawk asked Mr. Badrak how important iron content was for the metallurgical analysis in this report. Mr. Badrak stated that iron content is important when it gets above 5% and iron content becomes important for corrosion when it gets up to 20%.

Dr. Hawk also commented that it was not clear from the report if the TAP 766 study included hardness measurements. There was discussion among the peer reviewers about the importance of hardness measurements and how data for hardness traverses would complement the iron content measurements. The peer reviewers looked over the various certifications that were provided in BSEE's written responses (see Section 4, Questions #1 and 2, and related Section 4 appendices).

Based on experience conducting many weld overlays, Mr. Badrak observed that some fundamental information for metallurgical analyses was missing from the report. In addition, Mr.

Badrak commented that taking more measurements (beyond iron content) for metallurgical analysis of the Inconel 625 weld overlay would be important.

Dr. Hawk observed that the ability to reproduce the work for construction of the welded specimen directly influences the usefulness of the TAP 766 study results. Mr. Badrak commented that the report does not clearly describe or adequately characterize metallurgical analyses that would ordinarily be expected in such reports. Dr. Hawk added that more clarity was needed to put into context the various certifications that were provided in BSEE's written responses (see Section 4, Question #1 and 2, and related Section 4 appendices).

**Charge Question 3: Were the analytical methods used for the Engineering Stress-Strain Tensile Testing (Sections 4.1, 5, and 6.1) appropriately designed, clearly described, and adequately characterized? Were there any apparent strengths, weaknesses, omissions, or errors? Provide an explanation for your answers.**

- **Were the test objects selected for analysis valid test objects to evaluate the material properties?**
- **Were the assessments of engineering safety factors for the cited methods/standards (Table 4) valid for the expected applications in HPHT corrosive (sour gas) environments?**
- **Were the computational methods and research design clearly defined, accurate, and appropriate for this material testing method?**
- **Were the material testing methodology and underlying assumptions clearly defined, accurate, and appropriate?**

Mr. Badrak noted that only a single tensile test was conducted and used as the basis upon which stress levels were selected for the other material tests. Mr. Badrak commented that at least two tensile tests are typically performed to determine the mechanical properties. Mr. Badrak stated that at least two all weld metal tensile specimens must be run in each weld layer to determine the mechanical properties of each weld layer.

Mr. Badrak expressed concerns about the lack of hardness traverses because it should have been possible to use hardness traverses to help identify the weakest local regions of the test specimen or those local regions with lower tensile strength. Mr. Badrak noted again that compliance with ANSI/NACE MR0175 / ISO 15156-2 requires hardness traverses. Mr. Badrak also noted that the hardness traverse in the overlay would provide information regarding variability in tensile strength and changes in strength with respect to location.

Mr. Badrak commented that NACE TM0198 – Slow Strain Rate Test Method for Screening Corrosion Resistant Alloys for Stress Corrosion Cracking in Sour Oilfield Environments, should have been referenced in the final TAP 766 report, instead of using ANSI/NACE TM0284 (more appropriate for products being made from plate such as pipelines).

Mr. Badrak suggested that further work on material testing should address all three potential cracking mechanisms for solid-solution nickel-based alloys (e.g., Inconel 625): stress corrosion cracking, sulfide stress cracking, and galvanically induced hydrogen stress cracking. Mr. Badrak added that there is an issue now in low-temperature environments about hydrogen embrittlement of nickel-based alloys.

Dr. Rebak commented that this test was well designed for the TAP 766 study. Dr. Rebak emphasized; however, that this section of the report was inadequate because it never compared the TAP 766 study findings on the clad mechanical properties with values from vendors or from the literature for wrought or cast alloy 625. Dr. Rebak commented that such comparison between wrought and clad cast materials would have added a greater degree of certainty to the results reported from the TAP 766 study.

Dr. Hawk commented that this section of the report was clearly described and that this test was designed appropriately and conducted correctly. Dr. Hawk stated that more than one tensile test was needed.

Dr. Trillo commented that the methods used for this test were appropriately designed and characterized. Dr. Trillo acknowledged assuming that the TAP 766 study ran two tensile tests, because at least two tensile tests would be the typical approach at testing laboratories. Dr. Trillo also acknowledged that the final TAP 766 report actually provided results for only one tensile test.

There was discussion among the peer reviewers about whether NACE TM0198[2] was actually the correct standard that should have been referenced in the report, as Mr. Badrak had noted earlier, instead of ANSI/NACE TM0284[3] (see Table 4 in final TAP 766 report). Dr. Trillo commented that referencing the wrong NACE standard (TM0284) in Table 4 of the report instead of the correct NACE TM0198 standard “had to be a typo.” [4]

**Group Discussion: Background on uses of screening tools/techniques (e.g., SSRT)**

Dr. Rebak provided background information about the uses of screening techniques, such as the slow-strain-rate tensile test, followed by a group discussion about how screening techniques can help determine which material testing method is the best approach.

**Charge Question 4: Were the analytical methods used for the Slow-Strain-Rate Tensile Testing (Sections 4.2, 5, and 6.2) appropriately designed, clearly described, and adequately characterized? Were there any apparent strengths, weaknesses, omissions, or errors? Provide an explanation for your answers.**

- **Were the test objects selected for analysis valid test objects to evaluate the material properties?**
- **Were the assessments of engineering safety factors for the cited methods/standards (Table 4) valid for the expected applications in HPHT corrosive (sour gas) environments?**
- **Were the computational methods and research design clearly defined, accurate, and appropriate for this material testing method?**

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[2] NACE TM0198 "Slow Strain Rate Test Method for Screening Corrosion-Resistant Alloys for Stress Corrosion Cracking in Sour Oilfield Service" is not cited or referenced in the final TAP 766 report.
[3] ANSI/NACE TM 0284 was listed in Table 4 and as Reference [11] in Section 10 of the final TAP 766 report. ANSI/NACE TM0284 is “Evaluation of Pipeline and Pressure Vessel Steels for Resistance to Hydrogen-Induced Cracking.”
[4] After the peer review panel meeting, BSEE confirmed directly with DNV-GL (Ramgopal Thodla) that the TAP 766 study used NACE TM0198 for the Slow-Strain-Rate Tensile Testing and BSEE clarified that referencing ANSI/NACE TM0284 in Table 4 and as Reference [11] in Section 10 of the final TAP 766 report was “a typo.”

- **Were the material testing methodology and underlying assumptions clearly defined, accurate, and appropriate?**

Dr. Trillo commented that the strain rate of 4 x $10^{-6}$/sec was too fast. Dr. Trillo stated it would have been more appropriate to test at a slower strain rate of 1 x $10^{-6}$/sec for this material. Dr. Trillo explained that a slower strain rate would be more sensitive to environmental effects and could have better captured the HPHT sour-gas environmental effects.

Dr. Trillo also stated that the correct NACE standard was NACE TM0198 – Slow Strain Rate Test Method for Screening Corrosion Resistant Alloys for Stress Corrosion Cracking in Sour Oilfield Environments.[5] Dr. Trillo explained that this slow-strain-rate test method was developed for screening of CRA material resistance to stress corrosion cracking in simulated oilfield production environments at elevated temperatures. In addition, Dr. Trillo stated that the report needed to provide all of the slow-strain-rate test data in a table as required by the NACE TM0198 standard.

Mr. Badrak also explained that conducting this material test at a slower strain rate would have been a better approach for the Inconel 625 weld overlay. Mr. Badrak also noted that the TAP 766 study did not comply with the NACE TM0198 standard for the slow-strain-rate tensile testing.

Dr. Hawk noted that the strain rate was too fast, but in particular, emphasized that all of the material testing data should have been presented in table format. Dr. Hawk expressed concerns that overall there was insufficient technical detail in the report and emphasized that graphs are not a substitute for fully reporting all the material testing data.

Dr. Hawk pointed to Reference [14] or the "project interim report" cited on page 9 of the final TAP 766 report and questioned why Reference [14] had the same publication date as the final report and whether Reference [14] may have more data. Dr. Hawk requested that Ms. Doll try to obtain a copy of Reference [14] for the peer reviewers.

Ms. Doll contacted the BSEE COR (Mr. Mark Kozak) who immediately sent a copy, and Ms. Doll emailed a PDF version to each peer reviewer and provided a printed copy to review during the panel meeting. **Section 7** provides Reference [14] or the "project interim report" cited on page 9 of the final TAP 766 report.

Dr. Rebak noted that slow-strain-rate tensile testing is a good and fast first screening technique to determine susceptibility of alloys to stress corrosion cracking in an HPHT sour-gas environment; however, emphasized that selecting an appropriate strain rate is important for this test. Dr. Rebak expressed concerns that the report did not explain why the strain rate of 4 x $10^{-6}$ $s^{-1}$ was selected for the TAP 766 study. Dr. Rebak stated that the chosen strain rate would not allow for discovery of slow forming cracks and did not give the material the opportunity to react with the environment. Dr. Rebak commented that the strain rate could be lower, such as 1 x $10^{-6}$ $s^{-1}$ or

[5] NACE TM0198 "Slow Strain Rate Test Method for Screening Corrosion-Resistant Alloys for Stress Corrosion Cracking in Sour Oilfield Service" is not cited or referenced in the final TAP 766 report.

even 5 x $10^{-7}$ $s^{-1}$. Dr. Rebak also commented that presenting tabular data and including more context through comparison with literature data for other CRAs would strengthen the report.

Mr. Badrak commented that the slow-strain-rate tensile testing conditions were too benign for evaluation of stress corrosion cracking for the 9% Mo nickel-based alloy weld overlay and agreed with the other reviewers that the strain rate was too fast. Mr. Badrak stated that more confidence in the results of the report could be achieved by including scanning electron microscopy (SEM) figures presenting a fracture surface for each test sample at low magnification and the most suspect area identified and presented in a high magnification photograph. Mr. Badrak liked Dr. Rebak's comment about the importance of discovering slow forming cracks.

Dr. Trillo and Dr. Rebak added that the report should have provided many more SEM figures.

**Charge Question 5: Were the analytical methods used for the Bent Beam Stress Corrosion Cracking (SCC) Testing (Sections 4.3, 5, and 6.3) appropriately designed, clearly described, and adequately characterized? Were there any apparent strengths, weaknesses, omissions, or errors? Provide an explanation for your answers.**

- **Were the test objects selected for analysis valid test objects to evaluate the material properties?**
- **Were the assessments of engineering safety factors for the cited methods/standards (Table 4) valid for the expected applications in HPHT corrosive (sour gas) environments?**
- **Were the computational methods and research design clearly defined, accurate, and appropriate for this material testing method?**
- **Were the material testing methodology and underlying assumptions clearly defined, accurate, and appropriate?**

Dr. Rebak noted that this test applies a sideways force and is a good test, but requires the right test environment and test laboratory equipment for an effective test. Dr. Rebak stated that perhaps the testing conditions used for this test in the TAP 766 study were too benign. Dr. Rebak commented that the results of this test showed nothing useful, but that was not surprising because the slow-strain-rate test also found nothing useful. Dr. Rebak explained that because the Alloy 625 cladding did not undergo cracking, neither of the tests (bent beam test or slow-strain-rate test) could determine the limit of susceptibility of the Alloy 625 cladding to environmentally assisted cracking.

Mr. Badrak commented that this test was the least discriminating test for cracking resistance due to the load dropping off rapidly from the surface, but it is a good test for welds. Mr. Badrak stated that the test conditions were too benign for the Inconel 625 alloy weld material and also that the 30-day test duration was inadequate for nickel-based alloys and overlays. Mr. Badrak commented that a 90-day test duration was the minimum. Mr. Badrak expressed concerns that the report did not indicate whether this test used strain gages, which is a common practice for this test.

Dr. Trillo had assumed the TAP 766 study must have used strain gages to calibrate for this test, and stated that the report should have explained about strain gages but did not. Dr. Trillo explained that it is industry practice to test nickel-based alloys for longer test durations, such as using a 90-day exposure, not 30 days. Dr. Trillo mentioned that ISO 15156 Part 3 will in the future include a revision for testing of nickel-based alloys for 90 days under constant load.

Dr. Hawk asked for clarification about whether the 90-day revision that might occur soon was specifically for nickel-based alloys. Mr. Badrak stated yes.

Dr. Hawk commented that there was a big need for more material testing data from the TAP 766 study to conduct an effective peer review. There was discussion among all the peer reviewers about the need for more detailed tabular data and results from the material testing.

Ms. Doll noted that the agenda included an opportunity to review the Fatigue and Fracture Database, which was discussed in Section 7 of the final TAP 766 report. Using a projector, Ms. Doll walked the peer reviewers through the MS Access database to show the peer reviewers the material testing data included in the Fatigue and Fracture Database. Ms. Doll distributed a handout with the database instructions. Given the significant interest among the peer reviewers in the material testing data, later during Day-1, Ms. Doll provided this MS Access Fatigue and Fracture Database and the database instructions to each peer reviewer on CD and via email.

Also using a projector, Ms. Doll demonstrated a portion of the CTC Metadata files that were provided to BSEE with the final TAP 766 report as supplementary information. Given the significant interest among the peer reviewers in evaluating this detailed material testing data, photographs, and other data, Ms. Doll arranged to obtain the full CTC Metadata files from BSEE.[6]

**Charge Question 6: Were the analytical methods used for the Fracture Toughness Testing (Sections 4.4, 5, and 6.4) appropriately designed, clearly described, and adequately characterized? Were there any apparent strengths, weaknesses, omissions, or errors? Provide an explanation for your answers.**

- **Were the test objects selected for analysis valid test objects to evaluate the material properties?**
- **Were the assessments of engineering safety factors for the cited methods/standards (Table 4) valid for the expected applications in HPHT corrosive (sour gas) environments?**
- **Were the computational methods and research design clearly defined, accurate, and appropriate for this material testing method?**
- **Were the material testing methodology and underlying assumptions clearly defined, accurate, and appropriate?**

Dr. Hawk noted that this test is one of the hardest to run to achieve a valid result. Given the known difficulty of achieving a valid result from this test, Dr. Hawk suggested that additional testing could be performed in the future, if possible, especially given the nature of the clad-base material. Dr. Hawk explained that this test shows how much energy the specimen absorbs before

[6] In the evening after Day-1 of the panel meeting, Ms. Doll emailed the partial CTC Metadata files that EnDyna then had available to all four peer reviewers. On Day-2 of the panel meeting, Ms. Doll provided a flash drive to each peer reviewer with the full CTC Metadata files for the TAP 766 study. The BSEE COR (Mr. Mark Kozak) delivered the full CTC Metadata files to EnDyna in the morning of Day-2 of the panel meeting.

it cracks. Dr. Hawk also commented that typically a very large specimen is necessary for a good test.

Dr. Hawk commended the TAP 766 researchers for the unique approach used to design the compact tension specimen to test only the Inconel 625 cladding. The TAP 766 researchers built up a portion of the compact tension specimen using additive manufacturing to add alloy (Inconel 625) to the clad surface to obtain acceptable specimen dimensions for a valid test specimen geometry and to conform to the ASTM standard.

Dr. Hawk also commented that the conditions used for this test in the TAP 766 study were benign. Mr. Badrak added that it would have been surprising if any failure had occurred because those test conditions were benign for Inconel 625 alloy.

Dr. Trillo commented that the TAP 766 study should have done a hardness profile between the additive manufacturing and the cladding to confirm there were no major differences. Dr. Trillo also commended the TAP 766 researchers for using additive manufacturing to resolve the sample geometry needed to obtain a full test sample. Dr. Trillo stated this innovative approach was a great way to use additive manufacturing technology to complete the sample configurations and also noted that microstructure was not impacted as was demonstrated by the optical microscopy.

Mr. Badrak liked the additive manufacturing technique, but expressed concerns that there was some uncertainty about test data validity because of the residual stresses that accompany additive manufacturing. Mr. Badrak commented that the report did not present information about whether the magnitude and distribution of those residual stresses could have affected the test results. Mr. Badrak also stated that it would be necessary to take into account changes in the clad overlay strength because of the additive manufacturing approach.

Mr. Badrak expressed concerns that the final TAP 766 report had stated this test relied on using a proprietary DNV method, making it difficult to evaluate the test process used. Mr. Badrak noted that electrochemical measurements may have provided information that could document the effectiveness of the proprietary DNV method. Dr. Trillo added that it was surprising that a proprietary method that could not be disclosed was used for the TAP 766 study.

Mr. Badrak asked whether the impressed current from the voltage drop method (DCPD) would have an effect on the corrosion at the crack tip that could skew results. Mr. Badrak noted that this issue has been debated among experts over time. Dr. Hawk added that indeed this issue had been subject to much debate among experts. There was discussion among the peer reviewers about how researchers know if the current passed through the compact tension specimen does not interfere with the corrosion processes at the crack tip. Dr. Rebak offered to look into this issue, and believed it had been settled, but was not sure about the evidence that there is no effect of the current on the crack tip behavior.

Dr. Rebak noted that fracture testing of the weld overlay is an important way of testing the integrity of the weld overlay. Dr. Rebak commented that the TAP 766 study never made a valid conclusion from this test. Dr. Rebak stated that the test was done under benign conditions and it was not surprising that there were no measurable deleterious results for the Inconel 625 alloy.

Dr. Rebak commented that because everything passed in this test, it was not clear how this test data can be used.

**Charge Question 7: Were the analytical methods used for the Fatigue Testing (Sections 4.5, 5, and 6.5) appropriately designed, clearly described, and adequately characterized? Were there any apparent strengths, weaknesses, omissions, or errors? Provide an explanation for your answers.**

- **Were the test objects selected for analysis valid test objects to evaluate the material properties?**
- **Were the assessments of engineering safety factors for the cited methods/standards (Table 4) valid for the expected applications in HPHT corrosive (sour gas) environments?**
- **Were the computational methods and research design clearly defined, accurate, and appropriate for this material testing method?**
- **Were the material testing methodology and underlying assumptions clearly defined, accurate, and appropriate?**

Mr. Badrak commented that the fatigue test data exhibited a lot of scatter; however, that was not unusual given the variations typically observed with weld material. Mr. Badrak expressed concerns that the number of tests was insufficient so the resulting test data was not of much use. In addition, Mr. Badrak commented that the test conditions represented high cycle fatigue instead of achieving the TAP 766 study's objective to characterize low-cycle fatigue behavior at high stresses/strains. Mr. Badrak also commented that the test environment was not aggressive enough to assess corrosion fatigue behavior for the Inconel 625 clad overlay in HPHT conditions. Mr. Badrak stated that overall there were serious concerns about the predictive nature of these relatively benign fatigue tests that would make it impossible to develop a model from this fatigue testing data.

Dr. Rebak stated that because the Inconel 625 material is resistant to fatigue, it was not clear what was accomplished by this benign test. Dr. Rebak expressed concerns that because there was no comparison with what is known about Inconel 625 alloy in the literature, it was not clear if testing conditions for the TAP 766 study were close to the edge or far away. Dr. Rebak expressed concerns about how this fatigue testing data could be used and how reassuring the TAP 766 study's fatigue test results were. Dr. Rebak commented that the fatigue testing methodology was valid, but questioned if the data from the fatigue testing as performed for the TAP 766 study would be useful to evaluate failure in service in an HPHT environment.

Dr. Trillo commented that the intent was good; however, the TAP 766 researchers had to change their approach as the fatigue testing proceeded in order to get low-cycle fatigue. Dr. Trillo observed that initially it was not practical because the fatigue testing took too long. Dr. Trillo stated that changes in test sample configuration were appropriate, although it was still necessary to increase the test peak cycle stresses to reach the desired number of cycles to failure. Based on experience in laboratory testing, Dr. Trillo commented that such calibration step changes were good practice in order to meet the fatigue testing needs. Dr. Trillo stated that some of the fatigue test data may be useful when applied to subsequent testing.

Dr. Hawk reviewed Table 11 in the final TAP 766 report with the fatigue test results. Dr. Hawk concurred with the other reviewers about questioning the usefulness of this fatigue test data. Dr.

Hawk observed that with Inconel 625, if loaded too much, “it fails right away,” and if loaded a bit less, “it can go for a million cycles.” Dr. Hawk emphasized that it would be important to understand what is known about Inconel 625 in order to design appropriate fatigue test methods.

Dr. Hawk also commented that changes in stress concentration were necessary in the fatigue testing plan (shown in Table 8 in the report) to adjust the length of the fatigue test to something reasonable for the scope of this project. Dr. Hawk emphasized that additional testing would be necessary to establish a comprehensive fatigue curve. Dr. Hawk observed that the TAP 766 researchers figured out how best to do the fatigue testing by running the tests. Dr. Hawk stated that typically the most difficult part is not testing in air, but testing in the environment. Dr. Hawk commented it was not clear how useful the TAP 766 study’s fatigue test data was and that clearly more testing would be better, especially concerning the stress ratio and stress concentration factor.

There was discussion among the peer reviewers about fatigue testing and how to address challenges related to the time required for fatigue tests for evaluating low-cycle fatigue for materials such as Inconel 625 alloy.

## 2.2 Day-2: March 6, 2019

Mr. Rock opened Day-2 of the panel meeting at 8:30am.

Ms. Doll asked the peer reviewers if there was any information that should be discussed further from the charge questions covered in the afternoon the previous day. Ms. Doll also asked whether any of the peer reviewers were able to look over any of the partial CTC Metadata files emailed the previous evening. Ms. Doll explained that the BSEE COR would deliver the full CTC Metadata files to EnDyna later that morning, and then EnDyna would provide a flash drive to each peer reviewer with the full CTC Metadata files for the TAP 766 study.

Prior to starting the panel discussion, Ms. Doll provided a summary of the peer review process for a **Highly Influential Scientific Assessment** or **Highly Influential Scientific Information** peer review to answer several questions from Mr. Badrak the previous day. The following is a summary of key points provided in a handout and discussed with the peer reviewers. Generally the additional processes for peer review of Highly Influential Scientific Information might include:

- **Use external peer review**, in order to ensure independence from the Agency. This might also include the contractor that is conducting the external peer review using additional scrutiny in selecting peer reviewers to ensure more documentation of the peer reviewer’s independence from the Agency.
- **More rigorous peer review**. For example, peer reviewers might be provided the underlying data and/or models in addition to the report.
- **More transparent peer review process**, which usually involves public participation.
- **Consider both BALANCE and EXPERTISE in selection of reviewers**, for which “balance” means ensuring that different scientific/technical perspectives (if they exist) for the topic(s) are represented (“balance” might have more relevance for controversial topics).

- **Disclaimer included on each page of report**. With Highly Influential Scientific Information, the OMB's required disclaimer should be included on each page of the report provided for peer review.

Ms. Doll explained that more information about the peer review process can be found on BSEE's website, including a PDF of the BSEE "Peer Review Process Handbook" (dated May 2017). Ms. Doll placed a printed copy of this BSEE "Peer Review Process Handbook" on the conference room table if any peer reviewers wanted to consult it during Day-2.

In addition, Ms. Doll explained that after EnDyna has completed this external panel peer review, the OMB Bulletin on peer review (OMB M-05-03) and BSEE "Peer Review Process Handbook" require that BSEE prepare a Comment-Response Document for a Highly Influential Scientific Assessment or Highly Influential Scientific Information peer review. Ms. Doll mentioned that BSEE could chose to have the TAP 766 study contractor help prepare BSEE's Comment-Response Document. Ms. Doll noted that BSEE will eventually post on the BSEE website both EnDyna's Peer Review Summary Report and BSEE's Comment-Response Document for this peer review.

Ms. Doll and Mr. Rock opened up the discussion to address whether there was any information that should be discussed further from the charge questions covered in the afternoon during Day-1 of the panel meeting.

Dr. Hawk noted that hydrogen embrittlement is a common issue, especially with nickel-based alloys, and could be included in future research. Dr. Hawk commented that the TAP 766 study was a good effort to begin identifying key factors related to failure that should be studied under environmental conditions.

Dr. Hawk asked again whether there was any additional technical documentation available about the laboratory testing equipment that was used in the TAP 766 study. Dr. Hawk and Dr. Trillo commented that information about the laboratory testing equipment should have been included in the report or provided as appendices to the report.

Dr. Rebak provided a summary, after looking into it the previous night, about evidence that there is no effect of the impressed current from the voltage drop method (DCPD) on the corrosion behavior at the crack tip. There was more discussion among the peer reviewers about this issue related to the fracture toughness testing (Charge Question #6).

There was discussion among the peer reviewers again about the need for a more systematic description of how each of the material testing analytical methods incorporated the selected NACE standards in Table 4 in the final TAP 766 report. The peer reviewers agreed this would be important to provide more clarity and completeness about how those NACE standards applied to the material testing conditions, for each of the TAP 766 study's material testing methods.

Mr. Badrak asked if more explanation could be provided about expected applications for the Inconel 625 cladded material for equipment in offshore HPHT environments. Mr. Badrak stated that it would be helpful in developing final written comments on the TAP 766 study to have a better understanding of what types of equipment would be using this Inconel 625 cladded material in offshore HPHT environments. Ms. Doll stated she would ask the BSEE COR about obtaining that information for the peer reviewers.

The peer reviewers noted that the partial CTC Metadata files that Ms. Doll had emailed the previous night included some of the needed material testing data that was omitted from the final TAP 766 report. Dr. Trillo mentioned that it was helpful to review the tables of calibration data. Dr. Hawk mentioned that some of the photographs were missing from the partial CTC Metadata files, and Ms. Doll noted that the full CTC Metadata files should include all the photographs and material testing data provided to BSEE from the TAP 766 study.

Ms. Doll stated that the Fatigue and Fracture Database, which is discussed in Section 7 of the final TAP 766 report, could be projected again during Day-2, if needed. Ms. Doll also mentioned that after the full CTC Metadata files were delivered by BSEE, any of that material testing data could also be projected during Day-2, if needed, for review during the panel discussion.

**Group Discussion: Background on scientific/technical challenges related to FCGR testing**

Dr. Hawk provided background information about the challenges related to fatigue crack growth rate (FCGR) testing, followed by a group discussion. Generally, both high cycle and low-cycle fatigue testing are used to provide an indication of when failure would occur. Although cracks may initiate early on, the most important information is the FCGR.

**Charge Question 8: Were the analytical methods used for the Fatigue Crack Growth Rate (FCGR) Testing (Sections 4.6, 5, and 6.6) appropriately designed, clearly described, and adequately characterized? Were there any apparent strengths, weaknesses, omissions, or errors? Provide an explanation for your answers.**

- **Were the test objects selected for analysis valid test objects to evaluate the material properties?**
- **Were the assessments of engineering safety factors for the cited methods/standards (Table 4) valid for the expected applications in HPHT corrosive (sour gas) environments?**
- **Were the computational methods and research design clearly defined, accurate, and appropriate for this material testing method?**
- **Were the material testing methodology and underlying assumptions clearly defined, accurate, and appropriate?**

Dr. Trillo commented that the FCGR testing was appropriately designed. Dr. Trillo stated that the sample fabrication had allowed for measurement in both the upper and lower clad layers, which would allow a distinction between the two clad layers. Dr. Trillo noted that the test laboratory obtained calibration frequency scans, which would be a normal test protocol.

Dr. Hawk noted that although the TAP 766 study provided good information for FCGR testing, it was only a start. Dr. Hawk emphasized that the report needed more photographs for the FCGR test results. Dr. Hawk stated that typically besides obtaining the FCGR, it is important to know if a failure would be catastrophic.

Dr. Hawk observed that not many laboratories are set up to perform FCGR testing. Dr. Hawk mentioned that hydrogen sulfide destroys electronic equipment, so good secondary containment is necessary. Dr. Hawk stated it is difficult to perform an FCGR test in a pressure barrel.

Dr. Trillo commented that the test results showed an increase in FCGR with decreasing test frequency. In addition, the FCGR was an order of magnitude higher on the lower clad layer. Dr. Trillo commented that the conclusion would be that cracking would accelerate in the inner clad layer once it started from the outer clad layer.

Dr. Trillo stated that the TAP 766 researchers did not get as much FCGR data as they would have liked. Dr. Trillo noted the additional FCGR testing conducted was based on a frequency that would allow testing in a realistic time frame, but still showed a trend.

Dr. Trillo also commented that other researchers will follow the TAP 766 study to obtain more FCGR data. Dr. Trillo emphasized that it will be important to make comparisons of the TAP 766 study results for FCGR testing after similar research is conducted by others.

Dr. Hawk commented that it was a good approach to perform FCGR testing in both of the cladding layers and that this approach was able to initiate cracks in both cladding layers. Dr. Hawk stated that the FCGR results were a good start in providing information about cladding on steel plates, and anticipated that the TAP 766 study results can be supplemented later to expand the Fatigue and Fracture Database.

Dr. Hawk expressed concerns that the conclusions that can be drawn from the FCGR testing results did not come through clearly in the report; however, Dr. Hawk observed those conclusions were more apparent by reviewing the MS Excel tables. Dr. Hawk questioned why the narrative report did not include that additional FCGR testing data, at least some of which was available. Dr. Hawk suggested that including this additional FCGR testing data would increase the utility of the report.

Dr. Rebak had no additional comments about the FCGR testing.

Mr. Badrak commented that the test design for determining FCGR in each weld overlay layer was good, and experimentally the FCGR testing was good. Mr. Badrak expressed concerns; however, that the TAP 766 study overlooked some factors. Mr. Badrak commented that the report did not discuss other factors that influence FCGR, such as welding differences, specimen geometry, and notch geometry. Mr. Badrak also expressed concerns about the unknown effects of the residual stress from additive manufacturing used to construct the test specimen.

**Group Discussion: Background on uses of FCGR mathematical models for independent laboratory testing (e.g., design evaluation, life assessment, failure analysis)**

Dr. Trillo provided background information about uses of FCGR mathematical models for independent laboratory testing, followed by a group discussion. All reviewers agreed that fatigue testing experts are familiar with the Paris Law and Walker equation. Dr. Trillo noted that most FCGR data are for ambient air; there is not much FCGR data for HPHT environments. Mr. Badrak commented that the Walker equation allows for prediction of fatigue, but only if enough data exists. Dr. Trillo stated that the final TAP 766 report had specifically noted that data collected during FCGR testing was only a first step. Dr. Trillo explained that FCGR models can allow experts to say, for example, when inspections are needed.

**Charge Question 9: Do the FCGR material modeling results (Section 6.6) describe with reasonable accuracy the basis for decisions in the two mathematical models used:**

- **Were the assumptions clearly defined, accurate, and appropriate for the methods of modeling used for this study?**
- **Were the limitations and uncertainties clearly identified and adequately characterized for the methods of modeling selected?**
- **Did the report identify and adequately address the strengths or weakness of the analytical methods used for modeling?**

**Provide an explanation for your answers for each model used:**

1) **Paris Law FCGR Material Models**
2) **Walker Equation FCGR Material Model**

Dr. Hawk commented that there was enough information to use the Paris Law equation, but not enough information for the Walker equation. Dr. Hawk observed that this effort to use the Walker equation in the TAP 766 study might be considered “a screening operation.” Dr. Hawk pointed out that the report clearly stated that $da/dN$ versus $\Delta K$ must be available at multiple values of R to use the Walker equation and that this condition was not met for either of the clad layers.

There was discussion among the peer reviewers about FCGR testing and the values needed for the Walker equation. Mr. Badrak added that fitting the Walker equation requires data from at least two sets of R values. Mr. Badrak also stated that the Walker equation is only valid in either the tension or compression regions, not both.

Mr. Badrak commented that although this effort was “a step in the right direction,” there was not enough data to use the Walker equation. Mr. Badrak emphasized that the Walker equation relies upon material dependent variables that were not determined in the TAP 766 study’s FCGR test program. Mr. Badrak commented that the TAP 766 researchers made assumptions from literature data for Inconel 718 alloy that were not necessarily correct; however, the report clearly stated that using an assumption for the p value of Inconel 625 cladding in the Walker equation would allow a <u>first approximation</u> [emphasis from report] to the Walker equation for the Inconel 625 cladding. Mr. Badrak commented that the Walker equation was appropriate, but because insufficient data was available to use the Walker equation it was necessary to pull other data from a reference source. Mr. Badrak acknowledged making such assumptions from a reference source was “all they could do” and a reasonable approach for making an initial approximation.

Dr. Trillo observed that the TAP 766 researchers used an approach to start with the simplest FCGR model and then add on. Dr. Trillo agreed with that incremental approach, but emphasized that the TAP 766 study did not have enough FCGR data. Dr. Trillo commented that the FCGR material modeling results were presented well and clearly described the basis for how the FCGR models were used in the TAP 766 study.

Dr. Trillo explained that the Paris Law is a well utilized equation to relate the stress intensity factor range to sub-critical crack growth. Dr. Trillo stated that using the Paris Law is appropriate to obtain a well understood and quick relationship regarding the FCGR behavior of materials. Dr. Trillo commented that the Paris Law is the most appropriate first-pass model to describe cracking behavior. Dr. Trillo commended the TAP 766 researchers for following a statistical method by Schneider and Maddox, which Dr. Trillo stated is well utilized in the literature and thus was appropriate to use with the TAP 766 study results.

Dr. Trillo noted that the Walker equation allows for including material dependent values. Dr. Trillo explained that the Walker equation adds complexity and allows for FCGR modeling of the material in a more definitive way. Dr. Trillo commented that the TAP 766 study used values from the literature for the Walker equation that fit well with the TAP 766 study's FCGR data.

Mr. Badrak added that conducting FCGR testing in an HPHT environment is expensive. Because the environment is probably the single most important variable affecting FCGR, Mr. Badrak was unsure about the predictive validity of FCGR tests conducted for HPHT environments unless the test environment conditions were sufficiently aggressive for the overlay.

Dr. Rebak had no additional comments, but observed that it seemed the approach was a good start.

Ms. Doll, who had talked with the BSEE COR earlier on Day-2 regarding Mr. Badrak's request for more information about expected applications for Inconel 625 cladded material for equipment in offshore HPHT environments, put Mr. Russell Hoshman on the speakerphone to provide this additional background information. Mr. Hoshman is Technical Advisor for Regional Field Operations at BSEE's Gulf of Mexico Regional Office (New Orleans). Mr. Hoshman explained that in 2012 there were issues with equipment failures in an HPHT sour-gas environment after relatively few cycles. Industry practice is to clad metal when placing equipment in service in a sour-gas environment. BSEE practice is to determine that when a crack appears, "you're done" with that equipment. Mr. Hoshman emphasized that BSEE considered any type of pressure-containing or pressure-controlling equipment, especially such equipment that has a complex geometry, as the expected applications for Inconel 625 cladded material for equipment in HPHT sour-gas environments. In addition, Mr. Hoshman mentioned that weld cladding was one method to prevent stress corrosion cracking and explained that the TAP 766 study was designed to evaluate fatigue and fracture behavior for weld cladded CRAs in an HPHT sour-gas environment. Mr. Hoshman explained that examples of relevant applications include subsea trees, valves, manifolds, blowout preventers (BOPs), intervention well control equipment, and possibly subsea wellheads.

**Charge Question 10: Were the conclusions based on the TAP 766 study findings in the report (Section 8) logical and appropriate based on the material testing and FCGR material modeling results? Were the other conclusions related to the material testing appropriate? Provide an explanation for your answers.**

Dr. Rebak commented that the conclusions in Section 8 mostly reflected the findings in the report, but expressed concerns that those conclusions were presented in a disorganized manner. Dr. Rebak commended the TAP 766 researchers for conducting a lot of work for the available budget and schedule. Dr. Rebak mentioned that although the TAP 766 researchers claimed that the data in this report was only a start, there was no discussion about whether similar materials in upstream oil and gas have been evaluated using the material testing techniques used in the TAP 766 study. As an example, Dr. Rebak commented that Conclusion #4 about the results of slow-strain-rate tensile tests could have been correlated to MR0175 / ISO 15156.

Dr. Rebak expressed concerns about whether the chemical composition for the cladding layers was reported correctly. Dr. Rebak stated that Conclusion #6 suggested that the higher crack propagation in the first layer was due to higher iron dilution; however, Dr. Rebak noted that Figure 6 showed little difference in iron content between the two layers of the cladding.

Dr. Hawk commented that the conclusions in Section 8 were reasonable and supported by the material testing results; however, also noted that more research would be better. Dr. Hawk commented that research for the TAP 766 study introduced a different nature of cladded test specimen to previous materials research focused on steel and nickel superalloy. Dr. Hawk noted that the complicating nature of a clad surface and its performance in sour-gas conditions revealed interesting results that need further investigation. Different cladding materials and cladding processes, if not manufactured properly, could potentially lead to catastrophic and unpredictable failures. Dr. Hawk noted that service in sour-gas conditions is not usually evaluated for these composite systems. Dr. Hawk again emphasized that the TAP 766 study evaluated only one specific “alloy/cladding” heat (sample set) through conducting material testing only for Inconel 625.

Dr. Hawk also commented that Conclusion #6 was puzzling with respect to iron dilution. Dr. Hawk stated that more data points and/or additional information were needed to explain why the iron content was so high even in the second layer. Dr. Hawk observed that the explanation of high iron content values was not necessarily supported in the report. Dr. Hawk also observed that Conclusion #3 was not necessarily supported with respect to iron dilution.

Mr. Badrak had no problems overall with the conclusions in Section 8, but expressed concerns that those conclusions had limited applicability because of the benign conditions selected for the material testing and the limited data sets. Mr. Badrak liked that the material testing approach involved evaluating each cladding layer separately. Mr. Badrak commented that the TAP 766 researchers should have done additional measurement for residual stresses typically associated with additive manufacturing and also should have used more severe test conditions and conducted more validation of material test results.

Dr. Trillo commented that the conclusions in Section 8 were logical based on the material testing results and evaluations that were presented in the results sections of the final TAP 766 report. Dr.

Trillo stated that the conclusions mostly followed the data and related evidence for each of the separate material tests performed in the TAP 766 study. Dr. Trillo stated that more iron measurements and more material test data would be necessary to support Conclusion #6. Dr. Trillo expressed concerns that Conclusion #8, which described the reduction in area and time to failure scatter, was not presented in the slow-strain-rate results section (Section 6.2). Dr. Trillo noted that it would be useful to add the scatter data to the report.

**Charge Question 11: Were the recommendations (Section 9, Appendix A) logical, appropriate, and supported by the conclusions of the material testing results, empirical analysis, and FCGR material modeling results? The scope of the recommendations pertains to all recommendations, not just those derived from the FCGR material modeling results. Provide an explanation for your answers.**

Mr. Badrak supported the first four recommendations in Section 9 and the need for additional research. Mr. Badrak commented that Recommendation #5 was insufficiently supported by the TAP 766 study results. More specifically, Mr. Badrak stated that: 1) for the Inconel 625 cladding with process and test conditions used in the TAP 766 study, the statement in Recommendation #5 was correct in that the peak cyclic stress must be near the yield strength; and 2) the necessary peak stress levels for other materials, weld processes, and environmental conditions was not proven. In addition, Mr. Badrak mentioned that research on cathodic charging as well as galvanically induced hydrogen stress cracking will also eventually be needed.

With respect to Appendix A, Mr. Badrak commented about the importance of conducting replicate tests for each varied test condition.

Dr. Trillo agreed with the recommendations in Section 9 and commented that there are good ways to follow up on the material tests conducted in the TAP 766 study. Dr. Trillo noted that one good way would be to conduct additional testing from other cladding material vendors to ensure that the Inconel 625 clad microstructures that were tested in the TAP 766 study actually represented the industry methods for Inconel 625 cladding. Dr. Trillo stated that such additional testing would increase understanding of the impact of variability among industry cladding practices on microstructure, which may impact the stress corrosion cracking, fracture and fatigue results. Dr. Trillo also supported the recommendation to research the cracking behavior of new cladding materials. Because it was clear that additional FCGR data would be necessary to use the more involved NASGRO equation, Dr. Trillo stated that was an appropriate recommendation.

With respect to Appendix A, Dr. Trillo stated that conducting replicate tests was a good recommendation.

Dr. Hawk supported Recommendations #1, #2, and #3 in Section 9, and emphasized again that more data and information is always better. Dr. Hawk suggested adding scanning electron microscopy (SEM) work on fracture surfaces for all failure samples.

Dr. Hawk commented that Recommendations #4 and #5 were valid and should involve obtaining information from industry for the research design. Dr. Hawk suggested that Recommendation #4 would need some direct industry guidance as to ranking appropriate cladding systems and that

Recommendation #5 would need industry input on stress levels for cladding material systems as well as other component information to establish a testing variable range.

Dr. Rebak commented that the recommendations in Section 9 were valid, and emphasized again that providing more context through comparison with literature data for other CRAs is important to identify data and information gaps for research design. Dr. Rebak also wanted to see more research about lower temperature or near ambient temperature conditions where most hydrogen effects on materials occur (e.g., hydrogen embrittlement).

**Charge Question 12: Are there any additional study findings or conclusions that could be drawn from the material testing and FCGR material modeling results? Provide an explanation for your answers.**

Dr. Trillo stated that with this research focused on cladding materials, testing at room temperature and hydrogen testing was the next logical step. Dr. Trillo also suggested conducting material testing for 90 days on static testing and testing at a slower strain rate (1 x $10^{-6}$/sec) for slow-strain-rate tests. Dr. Trillo commented that the TAP 766 researchers could have also made a comparison between the slow-strain-rate and fatigue testing, which used the same sample configuration. Dr. Trillo noted that dynamic stresses were used to evaluate fracture behavior in both of those test methods.

Dr. Trillo also commented that another potential evaluation that could be conducted from the TAP 766 study is the effect that stressing has on fracture toughness and FCGR that allowed for observing an HPHT sour-gas environmental effect, but not observing that environmental effect on the slow-strain-rate testing. Dr. Trillo noted it was possible that the slow-strain-rate testing was not performed at a slow enough rate to observe an environmental effect, even though both the inner and outer clad layers were represented in the slow-strain-rate specimen geometry.

Dr. Rebak concurred with Dr. Trillo about testing at room temperature and hydrogen testing as the next logical step. Dr. Rebak suggested using a larger temperature range, from $350^{0}$F and maybe higher down to ambient temperature. Dr. Rebak also suggested evaluating different weld overlay deposition methods and expanding the research to evaluate hydrogen effects. Dr. Rebak commented that each test must be replicated at least two times and also suggested perhaps engaging another testing laboratory for replicating tests.

Dr. Hawk concurred with the previous suggestions by Dr. Trillo and Dr. Rebak, but also suggested using more R values (e.g., at least 2-3) to expand FCGR modeling options, using a different notch configuration for the test specimen, and considering a test environment using a higher temperature (e.g., $500^{0}$F). Dr. Hawk noted that usually when performing research of this nature, the key findings are not information learned from the research, but the gaps in information discovered when trying to apply the information developed during the course of the research project.

Mr. Badrak commented about a range of additional points regarding the research in the TAP 776 study:

- Researching hydrogen embrittlement is important because sacrificial anodes generate nascent (or atomic) hydrogen; hydrogen sulfide is not always necessary to have a source of hydrogen. Atomic hydrogen is so small that it can penetrate directly into the metal causing hydrogen embrittlement.

- Testing the variability among cladding materials is important, although industry uses only a few types of materials for cladding.
- Conducting the slow-strain-rate test at a slower rate – specifically using $1 \times 10^{-6}$ (four times slower than $4 \times 10^{-6}$).
- Conducting material testing at higher temperatures is also important if researchers want to realistically test the Alloy 625 overlay and make predictions on performance.
- Obtaining more data will be necessary from at least two (2) and preferably three (3) R values. More R values will be necessary to develop any FCGR models.

Mr. Badrak noted that it seemed there was not much disagreement among the peer reviewers about the final TAP 766 report. Mr. Badrak suggested that BSEE might find it useful to get subject matter experts involved prior to conducting a research study to help ensure more useful results.

## 3. AGENDA FOR PANEL MEETING

The agenda developed by EnDyna for the peer review panel meeting is presented below.

**AGENDA: PEER REVIEW PANEL MEETING**

*TAP 766: Characterizing the Behavior of Inconel Clad A387 Steel in High Pressure-High Temperature, Corrosive Environments — Material Models for Fatigue and Fracture Properties of Inconel 625 Cladding–Final Report*

**Tuesday, March 5, 2019**

| | |
|---|---|
| 8:30am | **Arrive at EnDyna office** |
| 8:45-9:15am<br>(30 mins) | **Welcome and Introductions; Review of Agenda/Process for 2-day Panel Meeting**<br>Amy Doll and Ken Rock, EnDyna |
| 9:15-9:30am<br>(15 mins) | **Background on TAP 766 Study:** Amy Doll, EnDyna, Peer Review Lead |
| 9:30-10:00am<br>(30 mins) | **General Impressions:** Provide overall impressions addressing the accuracy of information presented, clarity of presentation, and soundness of conclusions.<br>(***each reviewer will present a high-level summary using around 5 minutes***) |
| 10:00-10:15am | BREAK |
| 10:15-10:45am<br>(30 mins) | **Charge Question 1:** Were the objectives of the study clearly defined (Section 1)? If not, what are your recommendations for improving the description of this study's objectives? |
| 10:45-11:00am<br>(15 mins) | **Group Discussion: Background on ANSI/NACE MR0175 / ISO 15156**<br>Discussion Leaders: Bob Badrak and Ken Rock |
| 11:00-11:45pm<br>(45 mins) | **Charge Question 2:** Were the analyses used for the pre-tested metallurgical analysis (Section 3) appropriately designed, clearly described, and adequately characterized? Were there any apparent strengths, weaknesses, omissions, or errors? Provide an explanation for your answers. |
| 11:45-1:00pm | LUNCH (on your own) |
| 1:00-1:45pm<br>(45 mins) | **Charge Question 3:** Were the analytical methods used for the **Engineering Stress-Strain Tensile Testing** (Sections 4.1, 5, and 6.1) appropriately designed, clearly described, and adequately characterized? Were there any apparent strengths, weaknesses, omissions, or errors? Provide an explanation for your answers.<br>• Were the test objects selected for analysis valid test objects to evaluate the material properties?<br>• Were the assessments of engineering safety factors for the cited methods/standards (Table 4) valid for the expected applications in HPHT corrosive (sour gas) environments?<br>• Were the computational methods and research design clearly defined, accurate, and appropriate for this material testing method?<br>• Were the material testing methodology and underlying assumptions clearly defined, accurate, and appropriate? |
| 1:45-2:00pm<br>(15 mins) | **Group Discussion: Background on uses of screening tools/techniques (e.g., SSRT)**<br>Discussion Leaders: Raúl Rebak and Ken Rock |

**AGENDA: PEER REVIEW PANEL MEETING**

*TAP 766: Characterizing the Behavior of Inconel Clad A387 Steel in High Pressure-High Temperature, Corrosive Environments — Material Models for Fatigue and Fracture Properties of Inconel 625 Cladding–Final Report*

**Tuesday, March 5, 2019**

| | |
|---|---|
| 2:00-2:45pm<br>(45 mins) | **Charge Question 4:** Were the analytical methods used for the **Slow-Strain-Rate Tensile Testing** (Sections 4.2, 5, and 6.2) appropriately designed, clearly described, and adequately characterized? Were there any apparent strengths, weaknesses, omissions, or errors? Provide an explanation for your answers.<br>• Were the test objects selected for analysis valid test objects to evaluate the material properties?<br>• Were the assessments of engineering safety factors for the cited methods/standards (Table 4) valid for the expected applications in HPHT corrosive (sour gas) environments?<br>• Were the computational methods and research design clearly defined, accurate, and appropriate for this material testing method?<br>• Were the material testing methodology and underlying assumptions clearly defined, accurate, and appropriate? |
| 2:45-3:00pm | BREAK |
| 3:00-3:45pm<br>(45 mins) | **Charge Question 5:** Were the analytical methods used for the **Bent Beam Stress Corrosion Cracking (SCC) Testing** (Sections 4.3, 5, and 6.3) appropriately designed, clearly described, and adequately characterized? Were there any apparent strengths, weaknesses, omissions, or errors? Provide an explanation for your answers.<br>• Were the test objects selected for analysis valid test objects to evaluate the material properties?<br>• Were the assessments of engineering safety factors for the cited methods/standards (Table 4) valid for the expected applications in HPHT corrosive (sour gas) environments?<br>• Were the computational methods and research design clearly defined, accurate, and appropriate for this material testing method?<br>• Were the material testing methodology and underlying assumptions clearly defined, accurate, and appropriate? |
| 3:45-4:00pm | BREAK (also opportunity for introduction to Fatigue and Fracture Database; Section 7) |
| 4:00-4:45pm<br>(45 mins) | **Charge Question 6:** Were the analytical methods used for the **Fracture Toughness Testing** (Sections 4.4, 5, and 6.4) appropriately designed, clearly described, and adequately characterized? Were there any apparent strengths, weaknesses, omissions, or errors? Provide an explanation for your answers.<br>• Were the test objects selected for analysis valid test objects to evaluate the material properties?<br>• Were the assessments of engineering safety factors for the cited methods/standards (Table 4) valid for the expected applications in HPHT corrosive (sour gas) environments?<br>• Were the computational methods and research design clearly defined, accurate, and appropriate for this material testing method?<br>• Were the material testing methodology and underlying assumptions clearly defined, accurate, and appropriate? |
| 4:45-5:00pm | **Conclusion and Preparation for Day-2:** Amy Doll and Ken Rock, EnDyna |

**AGENDA: PEER REVIEW PANEL MEETING**

*TAP 766: Characterizing the Behavior of Inconel Clad A387 Steel in High Pressure-High Temperature, Corrosive Environments — Material Models for Fatigue and Fracture Properties of Inconel 625 Cladding–Final Report*

**Wednesday, March 6, 2019**

| | |
|---|---|
| 8:30am | **Arrive at EnDyna office** |
| 8:30-8:45am | **Review of Agenda for Day-2:** Amy Doll and Ken Rock, EnDyna |
| 8:45-9:30am<br>(45 mins) | **Charge Question 7:** Were the analytical methods used for the **Fatigue Testing** (Sections 4.5, 5, and 6.5) appropriately designed, clearly described, and adequately characterized? Were there any apparent strengths, weaknesses, omissions, or errors? Provide an explanation for your answers.<br>• Were the test objects selected for analysis valid test objects to evaluate the material properties?<br>• Were the assessments of engineering safety factors for the cited methods/standards (Table 4) valid for the expected applications in HPHT corrosive (sour gas) environments?<br>• Were the computational methods and research design clearly defined, accurate, and appropriate for this material testing method?<br>• Were the material testing methodology and underlying assumptions clearly defined, accurate, and appropriate? |
| 9:30-9:45am<br>(15 mins) | **Group Discussion: Background on scientific/technical challenges related to FCGR testing**<br>Discussion Leaders: Jeff Hawk and Ken Rock |
| 9:45-10:30am<br>(45 mins) | **Charge Question 8:** Were the analytical methods used for the **Fatigue Crack Growth Rate (FCGR) Testing** (Sections 4.6, 5, and 6.6) appropriately designed, clearly described, and adequately characterized? Were there any apparent strengths, weaknesses, omissions, or errors? Provide an explanation for your answers.<br>• Were the test objects selected for analysis valid test objects to evaluate the material properties?<br>• Were the assessments of engineering safety factors for the cited methods/standards (Table 4) valid for the expected applications in HPHT corrosive (sour gas) environments?<br>• Were the computational methods and research design clearly defined, accurate, and appropriate for this material testing method?<br>• Were the material testing methodology and underlying assumptions clearly defined, accurate, and appropriate? |
| 10:30-10:45am | BREAK (also opportunity to review Fatigue and Fracture Database; Section 7) |
| 10:45-11:00am<br>(15 mins) | **Group Discussion: Background on uses of FCGR mathematical models for independent laboratory testing (e.g., design evaluation, life assessment, failure analysis)**<br>Discussion Leaders: Elizabeth Trillo and Ken Rock |
| 11:00-11:45pm<br>(45 mins) | **Charge Question 9:** Do the **FCGR material modeling results** (Section 6.6) describe with reasonable accuracy the basis for decisions in the two mathematical models used:<br>• Were the assumptions clearly defined, accurate, and appropriate for the methods of modeling used for this study?<br>• Were the limitations and uncertainties clearly identified and adequately characterized for the methods of modeling selected?<br>• Did the report identify and adequately address the strengths or weakness of the analytical methods used for modeling?<br>Provide an explanation for your answers for each model used:<br>1) Paris Law FCGR Material Models |

**AGENDA: PEER REVIEW PANEL MEETING**

*TAP 766: Characterizing the Behavior of Inconel Clad A387 Steel in High Pressure-High Temperature, Corrosive Environments — Material Models for Fatigue and Fracture Properties of Inconel 625 Cladding–Final Report*

| Wednesday, March 6, 2019 | |
|---|---|
| | 2) Walker Equation FCGR Material Model |
| 11:45-1:00pm | LUNCH (on your own) |
| 1:00-1:45pm (45 mins) | **Charge Question 10:** Were the conclusions based on the TAP 766 study findings in the report (Section 8) logical and appropriate based on the material testing and FCGR material modeling results? Were the other conclusions related to the material testing appropriate? Provide an explanation for your answers. |
| 1:45-2:00pm | BREAK (also opportunity to review Fatigue and Fracture Database; Section 7) |
| 2:00-2:45pm (45 mins) | **Charge Question 11:** Were the recommendations (Section 9, Appendix A) logical, appropriate, and supported by the conclusions of the material testing results, empirical analysis, and FCGR material modeling results? The scope of the recommendations pertains to all recommendations, not just those derived from the FCGR material modeling results. Provide an explanation for your answers. |
| 2:45-3:00pm | BREAK (also opportunity to review Fatigue and Fracture Database; Section 7) |
| 3:00-3:45pm (45 mins) | **Charge Question 12:** Are there any additional study findings or conclusions that could be drawn from the material testing and FCGR material modeling results? Provide an explanation for your answers. |
| 3:45-4:00pm | **Conclusion:** Amy Doll and Ken Rock, EnDyna |

**Attendees:**

- Ms. Amy Doll, EnDyna, Peer Review Lead
- Mr. Ken Rock, EnDyna, Facilitator
- Robert (Bob) Badrak, PE, FNACE, FASM, Expert Peer Reviewer
- Jeffrey (Jeff) Hawk, PhD, FASM, Expert Peer Reviewer
- Raúl Rebak, PhD, FNACE, FASM, Expert Peer Reviewer
- Elizabeth Trillo, PhD, Expert Peer Reviewer

**Peer Review Panel Meeting "Ground Rules"**

- An external peer review is intended to solicit individual reviewer feedback, to increase the independence of the peer review process.
- The panel is not asked to, and should not attempt to, form consensus or collective recommendations, ratings, or opinions, and panel reviewers must understand that they should provide individual feedback on the research product.
- Any BSEE staff that may attend the panel meeting can only provide background information on the research product to the peer reviewers, which can occur only during the panel meeting run by EnDyna, and at EnDyna's request.

- The panel meeting will not include discussion related to BSEE policies and decisions or current or proposed BSEE regulations.

**Peer Review Objective and Scope**
**[Excerpts from BSEE TO#12 Charge Document]**

The objective of this panel-style peer review is for BSEE to receive comments from individual experts on the final report of the BSEE study entitled, *TAP 766: Characterizing the Behavior of Inconel Clad A387 Steel (ASTM A387 Grade F22, Class 2) in High Pressure-High Temperature, Corrosive Environments — Material Models for Fatigue and Fracture Properties of Inconel 625 Cladding–Final Report* (April 30, 2018). This panel-style peer review is scientific and technical in nature, reviewing the methods, assumptions, data quality, the strengths of any inferences made, and the overall strengths and limitations of the study.

**BSEE Charge for the Scope of this Peer Review**
BSEE has carefully defined the scope of this peer review for the final report of the BSEE study entitled, TAP 766: Characterizing the Behavior of Inconel Clad A387 Steel (ASTM A387 Grade F22, Class 2) in High Pressure-High Temperature, Corrosive Environments — Material Models for Fatigue and Fracture Properties of Inconel 625 Cladding–Final Report (April 30, 2018), in order to focus the peer review process effectively on BSEE's Charge Questions. Your written comments should stay within the BSEE Scope defined below. It is important to remember that this panel-style peer review is scientific and technical in nature, reviewing the methods, assumptions, data quality, the strengths of any inferences made, and the overall strengths and limitations of the study.

**The scope of this peer review is focused only on the <u>scientific and technical merit</u> of the assumptions, inputs, methodologies, modeling, and results for the BSEE study entitled, *TAP 766: Characterizing the Behavior of Inconel Clad A387 Steel (ASTM A387 Grade F22, Class 2) in High Pressure-High Temperature, Corrosive Environments — Material Models for Fatigue and Fracture Properties of Inconel 625 Cladding–Final Report* (April 30, 2018). This peer review is scientific and technical in nature and includes reviewing the methods, assumptions, data quality, the strengths of any inferences made, and the overall strengths and limitations of the study. The scope of the peer review includes the material, fabrication, computations, testing, engineering factors, modeling, results, and final recommendations generated from the TAP 766 study. As such, the peer reviewers should focus on providing comments on the <u>scientific and technical merit</u> of the TAP 766 study. Because this peer review is scientific and technical in nature, BSEE is not interested in comments focusing on editorial style.**

The following are considered **<u>Out-Of-Scope</u>** for this peer review; any and all **<u>Out-Of-Scope</u>** comments will not be considered by BSEE during this peer review process:

- BSEE is not interested in general comments related to high-pressure high-temperature (HPHT) equipment or environments, because: 1) this peer review is focused only on the methods and approach for testing in a sour-gas environment under HPHT conditions that were used in the TAP 766 study referenced above, and 2) this peer review is focused on the standards that were used in the TAP 766 study referenced above (see Tables 3 and 4 in final TAP 766 report).
- BSEE is not interested in comments on, or suggestions for, alternative fatigue and fracture testing methods, except for comments on any omissions or errors identified in the specific

material testing methods used for testing in a sour-gas environment under HPHT conditions in the TAP 766 study referenced above, because this peer review is focused on the research already completed for this TAP 766 study.

- BSEE is not interested in comments about API RP 17TR8 because BSEE has already completed a peer review for a previous BSEE study evaluating methods recommended by API RP 17TR8. Comments about API RP 17TR8 will not be considered during this peer review.
- This peer review is scientific and technical in nature, and does not extend to BSEE policies or BSEE regulations. Comments related to BSEE policies and decisions or to current or proposed BSEE regulations will not be considered.

# 4. BSEE's WRITTEN RESPONSES TO PEER REVIEWER QUESTIONS

To facilitate obtaining as much information as possible prior to the peer review panel meeting, EnDyna's Peer Review Lead compiled/paraphrased EnDyna's and the peer reviewers' initial questions about the final TAP 766 report. EnDyna provided BSEE a list of the peer reviewer questions on December 5, 2018. EnDyna requested that BSEE provide responses to these peer reviewer questions in writing so that EnDyna could distribute those written responses to the three peer reviewers and one alternate peer reviewer in advance of the peer review panel meeting. EnDyna received BSEE's written responses on February 4, 2019 and reformatted to improve readability. EnDyna distributed BSEE's written responses to the peer reviewer questions to the three peer reviewers and one alternate peer reviewer on February 14, 2019.

BSEE's written responses to the peer reviewer questions are provided below.

**TO#12: BSEE Responses to Peer Reviewer Questions**
**February 14, 2019**

1) Could BSEE clarify the form (e.g., plate, rod) of the Inconel® Alloy 625 used for the weld overlay in the TAP 766 study and also clarify the condition of that source material:

  1) As-Rolled condition,
  2) Annealed condition, or
  3) Solution-treated condition?

This is not clarified in the final TAP 766 report; however, the Reference [1] outlines in its Table 5 different Nominal Room-Temperature Mechanical Properties for Inconel® Alloy 625 based on form and condition (see Table 5 in:
http://www.specialmetals.com/assets/smc/documents/alloys/inconel/inconel-alloy-625.pdf).

2) Could BSEE provide additional technical details about the weld cladding process for the Inconel® 625 cladding used in the TAP 766 study?

**BSEE RESPONSE TO #1 and #2** (Please see appendices below for detailed information):

Weld overlay
Inconel® 625 alloy
0.045-inch diameter wire (assume wire drawn.)
Required two passes to achieve desired minimum overlay thickness of 0.25 inch
Gas metal arc welded
Minimum preheat temperature = 400 °F
Maximum interpass temperature = 550 °F
Welded in flat position
Stringer beads

190–210 amps
22–25 volts
28 in/min travel speed
350–450 in/min wire feed speed
Shielding gas: 100% argon
Post weld heat treatment: 1250 °F at 1.0 hour per inch of thickness; minimum of 0.25 hours.
As a welded product, the properties are expected to differ from those listed in Table 5 of the referenced Special Metals Website.

3) Could BSEE provide more information about the decision to conduct all the TAP 766 material testing experiments at 350°F (see Table 5 on page 4 in final TAP 766 report)?

- Could BSEE clarify the rationale for the decision to conduct material testing at 350°F, although HPHT was defined as ≥ 350°F? Could BSEE clarify whether or not this might mean that BSEE did not intend TAP 766 study results to be applicable to the realm of HPHT above 350°F?
- If the rationale for conducting material testing experiments at 350°F was related to BSEE data on current or proposed HPHT projects in deep water, could BSEE provide a brief high-level summary of any such relevant HPHT project information to provide context for the decision to conduct material testing experiments at 350°F?
- Could BSEE clarify the rationale for the decision (see pages 3-4 in final TAP 766 report) about: *With the acknowledgement of BSEE, testing at HPHT conditions was completed under Level VI conditions as defined in ANSI/NACE MR0175/ISO 15156; the test conditions are highlighted in Table 5: Physical Experimental Environmental Conditions*.?

**BSEE Response to #3:**

The intent of the program was to evaluate the range of conditions on the edge of temperature limits of interest and provide a framework with which to evaluate the materials. It is not the intent of the program to generate data for every single possible situation but to highlight the methodology that would be needed.

Currently there are no projects in deep water that are exposed to both HP and HT conditions. All projects greater than 15,000 psia currently under review have temperatures less than 350°F, and one project greater than 350°F but less than 15,000 psia. Generally the most current deep water HP projects require equipment designed for 16,500 to 17, 500 psia. Temperature ranges for these projects are generally from 250°F to 350°F. This would be the shut-in tubing pressures at the sea floor and flowing temperature at the sea floor. Maximum temperature and pressure are not in phase except at the moment of shut-in.

The decision to conduct testing at Level VI of ANSI/NACE MR0175/ISO 15156 was based on selecting conditions that were aggressive in terms of the partial pressure of acid gases /pH and Chloride.

4) Could BSEE clarify whether there was a formal hypothesis or an informal working hypothesis related to selecting the mechanical property test limits for the following material testing experiments, and, if so, provide a summary of any such related hypotheses:

- Slow-Strain-Rate Tensile Testing (Sections 4.2, 5, and 6.2)
- Bent Beam Stress Corrosion Cracking (SCC) Testing (Sections 4.3, 5, and 6.3)
- Fracture Toughness Testing (Sections 4.4, 5, and 6.4)
- Fatigue Testing (Sections 4.5, 5, and 6.5)
- Fatigue Crack Growth Rate (FCGR) Testing (Sections 4.6, 5, and 6.6)

**BSEE Response to #4:**

The intent of the test conditions for SSR was based on the NACE TM0298.[7] The bent beam tests were tested at 100% and higher similar to what is typically performed for CRAs in sour service. Fracture toughness testing was performed at various slow K-rates after which limited testing was performed at the appropriate K-rate. FCGR was performed at a choice of Kmax that was expected with stress being close to YS and a defect size that would be 50% of the thickness of the clad. This was assumed to be conservative for the test program. The rationale for the SN data generated is discussed in the text, the intent was to identify conditions where the cycle life was on the order of a few thousands of cycles typical of the design life of HPHT components.

In the design of complex geometry HP equipment, there are areas on the inner surface of the vessel where the stress exceeds yield for each HP cycle. API Technical Report 17TR8 is used as guidance for HPHT equipment for oil field service. Generally, equipment is designed using ASME Section VIII Div 2 or Div 3. Per API 17TR8, if yielding within the inner wall exceeds 5% of the thickness of the vessel, Elastic-Plastic Analysis will be required. All designs must undergo fatigue screening per ASME Section VIII Div 2. If it is determined that a fatigue assessment is required, this design analysis must be performed. Very little public data is available that shows what effect a crack in the CRA cladding has on the base metal; a crack that penetrates the cladding to the base metal.

[7] BSEE intends to clarify the accuracy of Section 6.2 that cites Reference [21]. In Section 10 of the final TAP 766 report Reference [21] is: NACE Standard TM0298, "Evaluating the Compatibility of FRP Pipe and Tubulars with Oilfield Environments."

# SECTION 4: APPENDICES

# Appendix A: Certification for Substrate Plate

02/16/2016 WINGATE ALLOYS, INC.
PL#: F15283

Item: 1 (1 PC) 1-1/4" X 22" X 48"

ARCELORMITTAL PLATE LLC

TEST CERTIFICATE

PAGE NO: 01 OF 02
FILE NO: 0284-01-05
MILL ORDER NO: 26331-001
MELT NO: U0099
SLAB NO: 9A
DATE: 01/31/14

01-C

STEEL PLATE DIMENSIONS / DESCRIPTION

| TOTAL QTY | GAUGE | WIDTH | LENGTH | DESCRIPTION | PIECE WEIGHT |
|---|---|---|---|---|---|
| 1 | 1.25" | 120" | 480" | RECTANGLE | 20419# |

CUSTOMER INFORMATION

CUSTOMER PO: 97797-NY

PART NO. 3

SPECIFICATION(S)

THIS MATERIAL HAS BEEN MANUFACTURED AND TESTED IN ACCORDANCE WITH PURCHASE ORDER REQUIREMENTS AND SPECIFICATION(S).

ASTM A387 YR 10 GR 22 CL 2
ASME SA387 EDITION 13 GRADE 22 CLASS 2
THE MANAGEMENT SYSTEMS FOR MANUFACTURING OF THIS PRODUCT ARE CERTIFIED TO ISO 9001:2008 (CERTIFICATE NO. 30130) AND ISO 14001 (CERTIFICATE NO. 009496).

CHEMICAL COMPOSITION

| | C | MN | P | S | CU | SI | NI | CR | MO |
|---|---|---|---|---|---|---|---|---|---|
| MELT:U0099 | .15 | .52 | .009 | .005 | .21 | .20 | .16 | 2.26 | .94 |

| | V | TI | B | AL | CB | SB | AS | SN |
|---|---|---|---|---|---|---|---|---|
| MELT:U0099 | .004 | .001 | .0001 | .025 | .001 | .0020 | .0030 | .008 |

MANUFACTURE

ELECTRIC FURNACE QUALITY - FINE GRAIN PRACTICE

HEAT TREAT CONDITION

| MATL OR TEST | HEAT TREAT DESCRIPTION | NOM TEMP | HOLD MINS | COOL MTHD |
|---|---|---|---|---|
| PL/TEST | NORMALIZE | 1675F | 34 | AIR COOL |
| PL/TEST | TEMPER | 1280F | 85 | AIR COOL |

Certified [illegible] the [illegible]

3/6/14

WE HEREBY CERTIFY THE ABOVE INFORMATION IS CORRECT:

ARCELORMITTAL PLATE LLC
QUALITY ASSURANCE LABORATORY
139 MODENA ROAD
COATESVILLE, PA 19320

SUPERVISOR – TEST REPORTING
ELINORE ZAPLITNY

02/16/2016

WINGATE ALLOYS, INC.

PL#: F15283

Item: 1 (1 PC) 1-1/4" X 22" X 48"

TEST CERTIFICATE

PAGE NO: 02 OF 02
FILE NO: 0284-01-05
MILL ORDER NO: 26331-001
MELT NO: D0059
SLAB NO: 9A
DATE: 01/31/14

TENSILE PROPERTIES

| SLAB NO. | LOC | DIR | YIELD STRENGTH PSI X 100 | TENSILE STRENGTH PSI X 100 | ELONGATION GAGE LGTH | % | %R.A. |
|---|---|---|---|---|---|---|---|
| 9A | BOT. | TRANS. | 820 | 1000 | 2.00" | 22.0 | 72.0 |

GENERAL INFORMATION

ALL STEEL HAS BEEN MELTED AND MANUFACTURED IN THE U.S.A.
TEST CERTS. ARE PREPARED IN ACCORD. WITH PROCEDURES
OUTLINED IN EN 10204:2004 TYPE 3.1.
MERCURY OR MERCURY COMPOUNDS ARE NOT USED IN THE
MANUFACTURE OF ARCELORMITTAL PLATE LLC PRODUCTS.
ACID SOLUBLE ALUMINUM
FOR MORE INFORMATION AND PROCESSING GUIDELINES, REFER TO
WWW.ARCELORMITTAL.COM/PLATEINFORMATION

B/L #41843 TTPX 805483

WE HEREBY CERTIFY THE ABOVE
INFORMATION IS CORRECT:

ARCELORMITTAL PLATE LLC
QUALITY ASSURANCE LABORATORY
139 MODENA ROAD
COATESVILLE, PA 19320

Elinore Zaplitny

SUPERVISOR – TEST REPORTING
ELINORE ZAPLITNY

## Appendix B: Cladding Statement of Work

Concurrent Technologies Corporation (CTC) will supply a steel plate (ASTM A387 Grade 22) with approximate dimensions of 48 in x 22 in x 1.25 in. The vendor shall clad one face of the steel plate with Inconel 625 alloy using the weld overlay process. The following scope of work shall be performed to produce the weld overlay.

1. Fabrication welding and welders/welding operations should be qualified in accordance with applicable internationally recognized standards such as ASME Section IX, ANSI/NACE MR0175/ISO 15156, or equivalents.
2. Prepare and clean one (1) face of the steel plate for weld overlay deposition.
3. The overlay weld shall be made using Alloy 625 weld wire (ERNiCrMo-3).
4. Apply weld overlay over an area of 48 in x 20 in on the face of the steel plate.
5. The weld overlay must run in the longest direction of the steel plate.
6. The final weld overlay shall be near flat and must have a minimum thickness of 0.25 inches. The cladded plate shall not be machined.
7. Surface inspection of the weld overlay shall be conducted to detect surface imperfections via penetrant inspection in accordance with applicable ASME or ASTM standards for non-destructive inspection (NDI).
8. The steel plate with the un-machined weld overlay surface shall be sent to Juan J. Valencia, CTC, 128 Industrial Park Road, Johnstown, PA 15904-1942. CTC will complete final stress relief.
9. The process parameters used to create the weld overlay shall be provided with the cladded plate.

### SCHEDULE AND DELIVERABLES FOR CLAD PLATE

The manufacturing facility shall provide a sound un-machined weld overlay steel plate. Also, both an electronic and hardcopy of the welding procedures, wire weld material certificates and NDI results in accordance with the reporting requirements of the applicable specifications shall be provided to CTC.

# Appendix C: Weld Overlay Certification

Hi-Tech Weld Overlay Group, LLC
1695 SE Decker St.
Lee's Summit, MO 64081

**Welding Procedure Specification (WPS)**

WPS No.: **CRO F43-P5A H** Date: **10/4/2010** Rev. No.: **0** Page 1 of 2
Supporting PQR(s): **625-P5A 2G**
Welding Process(es) / Type(s): **(1) GMAW / Semiautomatic and Machine**

**Joint Design (QW-402)**
Weld Type: Corrosion resistant overlays

WELD JOINT DESCRIPTIONS SHOWN ARE NOT INCLUSIVE OF ALL THOSE FOUND ON A JOB. WELD JOINT DESIGN REFERENCE IN AN ENGINEERING SPECIFICATION OR A DESIGN DRAWING SHALL TAKE PRECEDENCE OVER WELD JOINTS SHOWN IN THIS WPS.

**Base Metals (QW-403)**
P-No. 5A Thickness Range: 0.25 in. minimum

**Preheat (QW-406)**
Minimum Preheat Temperature: 400 °F
Maximum Interpass Temperature: 550 °F
Preheat Maintenance: Temperature sensing crayons
Heat pads or rosebud torch

**Postweld Heat Treatment (QW-407)**
PWHT Type: PWHT below lower transformation temperature
PWHT Temperature : 1250 °F
PWHT Holding Time: 1.0 hr./in., 0.25 hr. minimum

Initial and Interpass Cleaning: With stainless steel brush clean area to be overlayed.
Method of Back Gouging: Remove overlay defects by grinding only.

We certify that the statements in this record are correct and that the test welds were prepared, welded, and tested in accordance with the requirements of Section IX of the ASME Code.

Accepted By: Jeff Burkitt 10/4/2010 QC Manager
Jeff Burkitt Date

Hi-Tech Weld Overlay Group, LLC

**Welding Procedure Specification (WPS)**

WPS No.: **CRO F43-P5A H** Rev. No.: **0** Page 2 of 2

**First Process:** GMAW **Type:** Semiautomatic and Machine

**Filler Metal (QW-404)**
Weld Deposit Limits: 0.1875 in. minimum
AWS Classification: ERNiCrMo-3 SFA Specification: 5.14 F-No.: 43
A-No. or Chemical Composition: N/A
Filler Metal Product Form: Bare (Solid)
Supplemental Filler Metal: None

**Position (QW-405)**
Position of Joint: Flat & Horizontal
Weld Progression: N/A

**Gas (QW-408)**
Shielding: 100% Argon / 41-54 CFH
Trailing: None / - CFH

**Electrical Characteristics (QW-409)**
Current Type and Polarity: DCEP (reverse)
Transfer Mode: Pulsating arc
Max. Heat Input, 1st Layer (J/in): 11315
Energy/Power Thermal Arc Power Master

**Technique (QW-410)**
Stringer or Weave Bead: Stringer bead
Nozzle / Gas Cup Size: 3/8" to 5/8"
Oscillation: None
Peening: None
Contact Tube to Work Distance: 3/4" - 1"
Number of Electrodes: 1
Multiple or Single Layer(s): Multiple layer

**First Process Welding Parameters**

| Layer(s) and/or Pass(es) | Filler Metal AWS Classification | Filler Metal Size (in.) | Current Type and Polarity | Current Amperage Range | Wire Feed Speed (in/min) | Voltage Range | Travel Speed Range (in/min) |
|---|---|---|---|---|---|---|---|
| 1-n | ERNiCrMo-3 | 0.045 | DCEP (reverse) | 190-210 | 350-450 | 22-25 | 28 |
| 2-n | ERNiCrMo-3 | .045 | DCEP (reverse) | 190-210 | 350-450 | 22-25 | 28 |

HI TECH

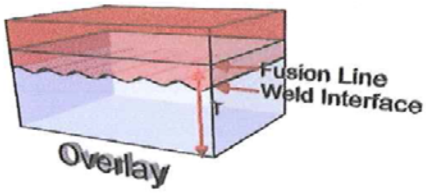
**Hi-Tech Industrial Services, Inc.**
1695 SE Decker St.
Lee's Summit, Mo. 64081

**Procedure Qualification Record (PQR)**

PQR No.: **625-P5A 2G** Date: **10/1/2010** WPS No.: **CRO F43-P5A H** Page 1 of 2

Welding Process(es) / Type(s): **(1) GMAW / Machine**

**Joint Design (QW-402)**
Weld Type: Corrosion resistant overlay




**Base Metals (QW-403)**
Specification Type and Grade: SA-387, Grade 21, Cl. 2
P-No. 5A Group No. 1
Thickness (in.): 0.25

**Preheat (QW-406)**
Minimum Preheat Temperature: 400 °F
Preheat Maintenance: Temperature crayons
Maximum Interpass Temperature: 600 °F
Maintained preheat using heating pads.

**Postweld Heat Treatment (QW-407)**
Type: PWHT performed below lower transformation temp.
PWHT Temperature: 1250 -1350 °F
PWHT Holding Time: 0.25 hr.
Ambient to 200°F at a moderate rate, 200°F to 1250°F - 1350°F at a rate not to exceed 133°F/hr. Hold 1250°F - 1350°F for .25 hours minimum 1250°F - 1350°F to 400°F at a rate not to exceed 133°F/hr 400°F to ambient cool in still air.

**First Process:** **GMAW**

**Filler Metals (QW-404)**
AWS Classification: ERNiCrMo-3
SFA Specification: 5.14 F-No.: 43
A-No. or Chemical Composition: N/A
Filler Metal Trade Name: Inconel 625
Filler Metal Product Form: Bare (Solid)
Supplemental Filler Metal: None
Min Qualified 't' (in.): 0.1875

**Positions (QW-405)**
Position of Joint: 2G - Horizontal
Weld Progression: N/A

**Gas (QW-408)**
Shielding: 100% Argon / 45 CFH
Trailing: None / - CFH

**Type:** **Machine**

**Electrical Characteristics (QW-409)**
Current Type and Polarity: DCEP (reverse)
Transfer Mode: Pulsating arc

**Welding Details**

| | | | |
|---|---|---|---|
| Filler Metal Size (in.): | 0.045 | 0.045 | - |
| Amperage Used: | 200 | 205 | - |
| Wire Feed Speed (in/min): | 400 | 400 | - |
| Voltage Used: | 24 | 25 | - |
| Travel Speed (in/min): | 28 | 28 | - |

Max. Heat Input, 1st Layer (J/in): 10286

**Technique (QW-410)**
Thermal Processes: No
Stringer or Weave Bead: Stringer bead
Nozzle / Gas Cup Size: 5/8"
Contact Tube to Work Distance: 3/4" - 1"
Oscillation: None
Number of Electrodes: 1
Multiple or Single Layer(s): Multiple layer

**Hi-Tech Industrial Services, Inc.**

**Procedure Qualification Record (PQR)**

PQR No.: **625-P5A 2G** Page 2 of 2

**Guided Bend Tests (QW-160)**

| Type and Figure No. | Result | Type and Figure No. | Result |
|---|---|---|---|
| Perpend. per QW-453 | Acceptable | Perpend. per QW-453 | Acceptable |
| Perpend. per QW-453 | Acceptable | Perpend. per QW-453 | Acceptable |

Visual Examination: No Indications

Liquid Penetrant Test: Satisfactory per QW 195.2

Macro-Examination Test: None

Chemical Analysis: C=0.03%, Cr=21.20%, Mo=8.71%, Ni=61.64%, Mn=0.05%, Si=0.04%, P<0.008%, S<0.005%, Fe=3.57%, AL=0.32%, Tl=0.31%, Nb=3.53%, Cu<0.01%, Co<0.01%

Welder's Name: Hubbard, Jason I.D.: Q7 Stamp No.: Q7

PQR was done and welding of coupon was witnessed by: Hi-Tech Industrial Services, Inc.

Test conducted by: Sherry labs Lab Test No.: 10100059-002-v1

We certify that the statements in this record are correct and that the test welds were prepared, welded, and tested in accordance with the requirements of Section IX of the ASME Code.

Accepted By: Jeff Burkitt 10/1/2010 QC Manager
Jeff Burkitt Date

N.D.E. V.T. REPORT

H-Tech Industrial Services Inc.
1695 SE Decker St.
Lee's Summit, Mo. 64081

| | |
|---|---|
| Customer Name: Concurrent Technologies Corporation<br>128 Industrial Park Road Johnstown PA, 15904 | Job Number: 4018 |
| | Unit Number: |
| Unit ID: Test Coupon | |
| Examination Technique: | Light Intensity Measurement: (1000 lux min.) |
| Procedure Number: **VT-1, Rev 1** | **500 Watt Quartz Light** |
| | **> 2500 lux at 24"** |

Identification of area examined:

**48"x 20" overlayed plate after 1st pass of Inconel 625**

Examination results ( location of rejected indications or area free from indications)

**2 rejectable indications found and repaired after which no indications found**

| | | |
|---|---|---|
| | 3/10/2016 | 0409-016 |
| Operator Signature | Date | Cert. # |

N.D.E. D.P.T. REPORT

Hi-Tech Industrial Services, Inc.
1695 Decker Street
Lees Summit, MO 64081

| | |
|---|---|
| Customer Name: Concurrent Technologies Corp | Job Number: 4018 |
| | Unit Number: |
| Penetrate Type: Color Contrast | Procedure Number: **P.T.- DPT-1 Rev. 2** |
| Brand Name: Spot Check | Examination Technique: Direct |
| Manufacturer : Magnaflux | |
| Batch No.: | Light Intensity Measurement: (100 fc min.) >100 fc |

**Identification of area examined:**
48"x20" plate with a double pass of 625 inconel overlay

**Examination results (location of rejected indications or area free from indications):**
No rejectable indications noted at time of examination.

**Comments:**

| | | | |
|---|---|---|---|
| [signature] | | 3/14/2016 | 0409-016 |
| Operator Signature | Level | Date | CERT. # |

Hi-TECH

POSITIVE MATERIAL IDENTIFICATION REPORT

| | |
|---|---|
| Report Number: | 1 |
| Job Number: | 4018 |
| Calibration Date: | 03/23/15 |

| | | | | | |
|---|---|---|---|---|---|
| Client: | CTC | Procedure Number: | Hi-Tech PMI | Revision: | 0 |
| Project: | Double Pass Inco 625 on P5A | Equipment Type: | Niton XL3t 980 | Serial Number: | 83196 |

| Test | Description | Material | Heat | Cr % | Ni % | Mo % | Mn % | Cu % | Fe % | Time/Sec | Accept | Reject |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | * | * | * | | | |
| 1 | Pass #1 Inconel 625 on P5A | 625 Inconel | QQ756 | 21.10 | 54.78 | 7.80 | | | 12.27 | | | |
| 2 | Pass #2 Inconel 625 on P5A | 625 Inconel | QQ756 | 23.13 | 61.77 | 8.61 | | | 1.0 | | | |
| 3 | | | | | | | | | | | | |
| 4 | | | | | | | | | | | | |
| 5 | | | | | | | | | | | | |
| 6 | | | | | | | | | | | | |
| 7 | | | | | | | | | | | | |
| 8 | | | | | | | | | | | | |
| 9 | | | | | | | | | | | | |
| 10 | | | | | | | | | | | | |
| 11 | | | | | | | | | | | | |
| 12 | | | | | | | | | | | | |
| 13 | | | | | | | | | | | | |
| 14 | | | | | | | | | | | | |
| 15 | | | | | | | | | | | | |
| 16 | | | | | | | | | | | | |
| 17 | | | | | | | | | | | | |
| 18 | | | | | | | | | | | | |

Comments: (Operator shall check Analyzer for Calibration prior to each use with "Certified Reference Material" provided with the Analyzer.)

| | | | |
|---|---|---|---|
| Operator Name: | Greg Richards | Client Representative: | |
| Date: | 3/11/2016 | Date: | |

Note: (Next Analyzer Calibration Due on: 3/23/16)

| | | | |
|---|---|---|---|
| Quality Control: | Don Jones | Hi-Tech Representative: | |
| Date: | 3/11/2016 | Date: | |

721864-1

**HAYNES**
**Wire Company**

158 N. Egerton Rd
P.O. Box 677
Mountain Home, NC 28758
828-692-5791
828-697-9038 FAX

## PRODUCT CERTIFICATION

WORK ORDER
W02286

HEAT NUMBER
QQ756

SALES ORDER / RLS
010563 / 4

CERT ID / REV
00006947 / 01

SOLD TO

Haynes International, Inc.
158 North Egerton Road
Mountain Home, NC 28758
USA

ISO 9001:2008 Certified
AS:9100 C Certified

| CUSTOMER P.O. | CUSTOMER PART | QUANTITY | COILS | LADING NO | SHIPMENT DATE |
|---|---|---|---|---|---|
| BF WAREHOUSE | | 5,241 Lbs | 169 | 00011319 | 06/19/2014 |

SPECIFICATION 625-0450-12BP-I500
HAYNES® 625 RTW™ WIRE, 0.0450, 12" DIN 300 Spool, 30.0 lbs, Bright,

CERTIFICATION REQUIREMENTS
UNS N06625
AMS 5837G
AWS A5.14:2009 & 2011 ERNiCrMo-3
ASME SFA-5.14:2004 & 2013 ERNiCrMo-3
BS EN10204:2004 3.1

### Chemical

| | | | | |
|---|---|---|---|---|
| C | Mn | Fe | P | S |
| 0.01 | 0.03 | 0.22 | <0.003 | <0.001 |
| Si | Cu | Ni | Co | Al |
| 0.05 | <0.01 | 64.5 | 0.02 | 0.11 |
| Ti | Cr | Cb/Nb | Nb/Ta | Mo |
| 0.18 | 22.36 | 3.62 | 3.623 | 8.75 |
| TOE | | | | |
| <0.50 | | | | |

### Mechanical by Lot

| TEST | UNITS | HIGH | LOW | AVERAGE |
|---|---|---|---|---|
| Diameter | In | 0.0438 | 0.0435 | 0.0436 |

| TEST | RESULT |
|---|---|
| Surface Check | Pass |

HI-TECH INDUSTRIAL SERVICES, INC.
DATE 10/2/14
APPROVED BY QA/QC

This is to certify that all required samplings, inspections and tests have been performed in accordance with the specification requirements. The test report represents the actual attributes of the material furnished and the values shown are correct and true. The material described by this certificate is in full compliance with all order and inspection requirements. We hereby certify that the above data are in accordance with the specification requirements.

Alton Batson
Quality Manager
ABatson

Page 1

Date Printed 06/20/2014

## Appendix D: Heat Treatment Requirements

### Stress Relief of Cladded Inconel 625 on ASTM A387 Grade 22 Steel Plate
### Statement of Work
### 2016-03-14

### SCOPE OF WORK

Concurrent Technologies Corporation (CTC) is requesting to conduct stress relief treatment of a steel plate cladded with Inconel 625 alloy. The steel plate (ASTM A387 Grade 22) has approximate dimensions of 48 in. x 22 in. x 1.25 in. and one face has the cladded a layer. The Inconel 625 cladded layer has a thickness of approximately 0.3 in and it was produced by the weld overlay process. The stress relief shall be conducted using the following parameters.

1. The stress relief of the plate shall be conducted in vacuum or in an inert atmosphere.
2. The furnace atmosphere shall be completely free of sulfur, sulfur compounds and other contaminants such as carbon, phosphorous, lead, zinc and carbon containing compounds.
3. Prior to stress relieving the plate shall be free of oil, grease and other contaminants.
4. The plate shall be in a horizontal position with the cladding face up in the furnace.
5. No paint or ink markings shall be made on the cladded plate
6. The cladded plate shall be stress relieved at 1075 °F ± 25 °F and held for 4 (four) hours at temperature.
7. The heating ramp up shall be at 100 °F per hour.
8. The cooling rate shall be equivalent to air cool to below 400 °F.
9. The stress relieved plate shall be properly crated and shipped to CTC at the following address.

   128 Industrial Park Road
   Johnstown, PA 15904-1942
   Attention: Juan Valencia

### SCHEDULE AND DELIVERABLES

The heat treating facility shall provide the cladded steel plate clean and damage free. Also, an electronic and/or hardcopy of the thermal history during the stress relief process shall be provided to CTC.

### POINTS OF CONTACT

CTC technical point of contact information is provided below. The test facility shall identify their technical POC upon award of purchase order.

| | |
|---|---|
| Juan J. Valencia<br>Concurrent Technologies Corp.<br>100 CTC Drive<br>Johnstown, PA 15904-1935<br>Phone/Fax: 814-269-2552<br>valencia@ctc.com | Michael Tims<br>Concurrent Technologies Corp.<br>100 CTC Drive<br>Johnstown, PA 15904-1935<br>Phone/Fax: 814-269-2515<br>tims@ctc.com |

# Appendix E: Heat Treatment Certification

SOLAR ATMOSPHERES INC.

Solar Atmospheres of Western PA

**Certification**

**Order No.:** 109330
**Date:** 04/04/2016
**Entry Date:** 03/30/2016
**Page:** 1 of 1

**To:**
CONCURRENT TECH. CORP.
100 CTC DRIVE
JOHNSTOWN, PA 15904-1935

**Purchase Order No.:** 160300129
**Packing List No.:**
1 **Material:** INCONEL 625

All work performed subject to Solar Atmospheres Terms Of Sale as presented on form TOS-SAWPI.

| Quantity | Part Number / Part Name / Part Description | Pounds |
|---|---|---|
| 1 | MATERIAL INCONEL 625<br>CLADDED STEEL PLATE<br>REQ: VAL3211403 / 48" X 1.5" X 22' | |

| Insp. Type | Scale | Minimum | Maximum | Number | Other |
|---|---|---|---|---|---|
| **Customer Requirements:** | | | | | |
| Visual | | . | . | . | |

THIS IS TO CERTIFY THAT THE ABOVE LISTED PARTS WERE PROCESSED IN ACCORDANCE WITH YOUR PURCHASE ORDER REQUIREMENTS AND SCOPE OF WORK 1.0 DATED 03/14/2016.

FURNACE RUN#: 70-9098-6004

RAMPED AT 100°F (MAX.) TO 1075°F ±25°F
HELD AT 1075°F ±25°F FOR 4 HOURS +10 / -0 MINUTES
ARGON COOLED

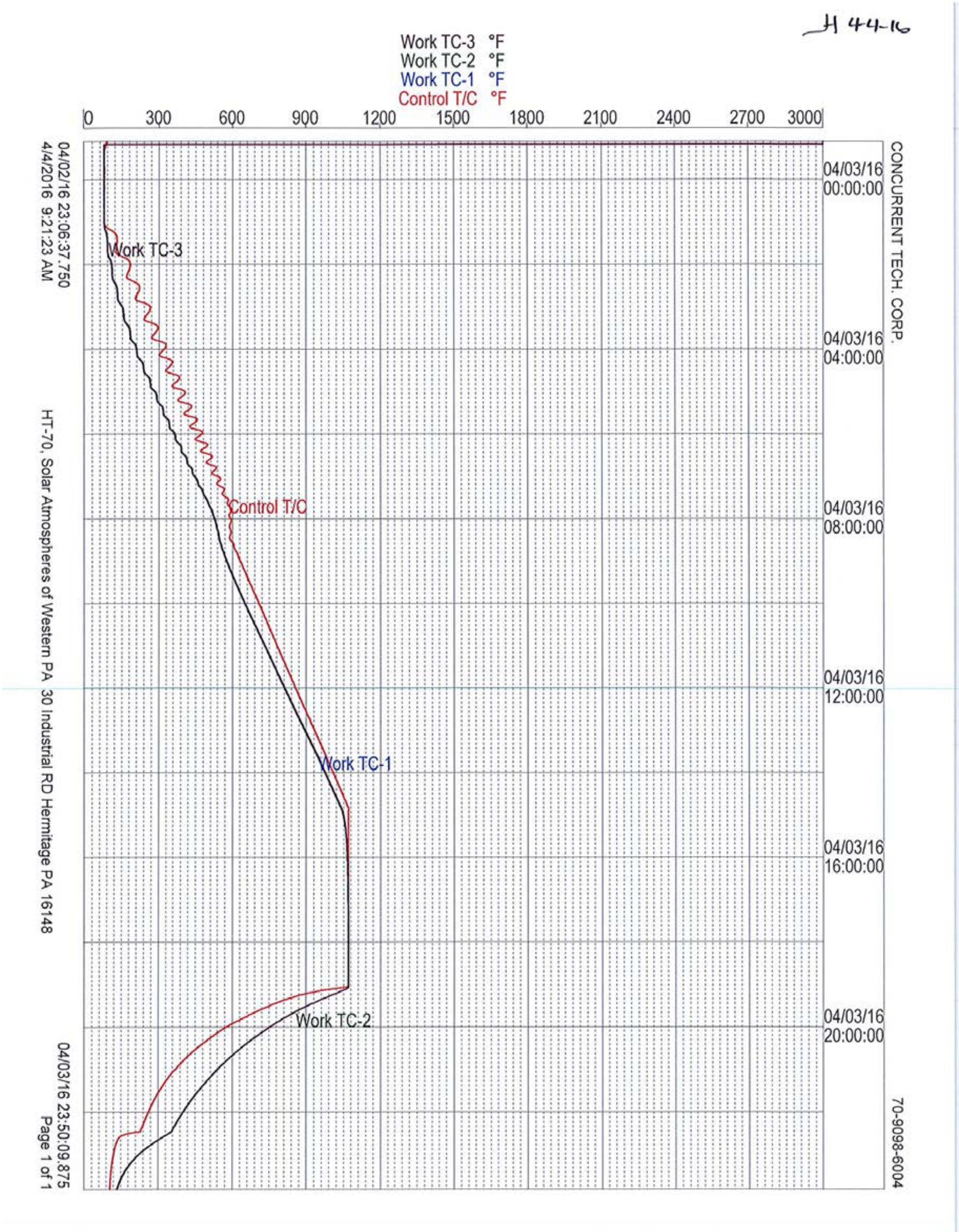
Jack Hardesty

This certification is no guarantee of material performance, properties, or microstructure. Mechanical, physical, and/or metallurgical testing is not performed unless specifically itemized on your purchase order to Solar Atmospheres.

Jack Hardesty
Special Projects Coordinator /
SOLAR ATMOSPHERES INC.

The recording of False, Fictitious, or Fraudulent statements or entries on this document may be punished as a felony under federal statutes including FEDERAL LAW. TITLE 18, CHAPTER 47.

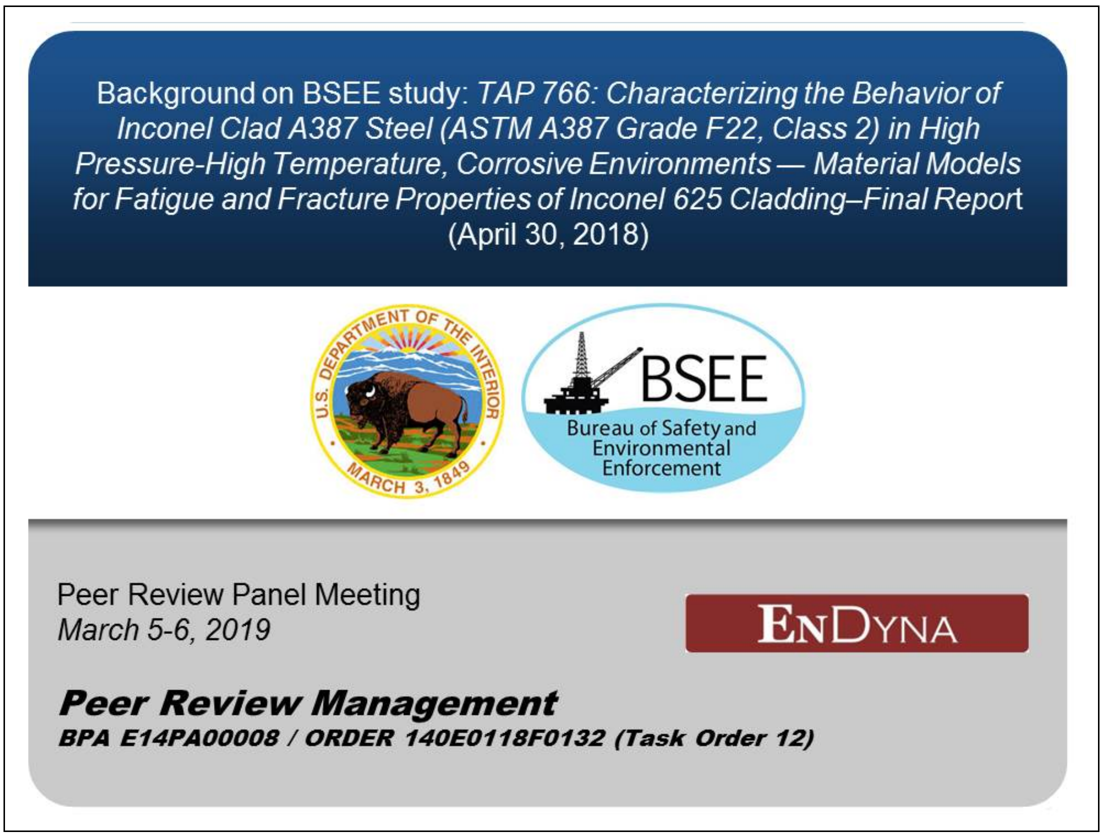
30 Industrial Road Hermitage PA 16148 **Phone:** 724-982-0660 **Fax:** 724-982-0593


H 4-4-16
Work TC-3 °F
Work TC-2 °F
Work TC-1 °F
Control T/C °F
0
300
600
900
1200
1500
1800
2100
2400
2700
3000
04/03/16 00:00:00
04/03/16 04:00:00
04/03/16 08:00:00
04/03/16 12:00:00
04/03/16 16:00:00
04/03/16 20:00:00
Work TC-3
Control T/C
Work TC-1
Work TC-2
CONCURRENT TECH. CORP.
70-9098-6004
04/02/16 23:06:37.750
4/4/2016 9:21:23 AM
HT-70, Solar Atmospheres of Western PA 30 Industrial RD Hermitage PA 16148
04/03/16 23:50:09.875
Page 1 of 1

# 5. BACKGROUND PRESENTATION

This section provides EnDyna's background presentation on the TAP 766 study, which was discussed at the beginning of Day-1 of the peer review panel meeting.

Prior to the panel meeting, the BSEE COR (Mr. Mark Kozak) had reviewed and approved EnDyna's background presentation. EnDyna sent the background presentation to the peer reviewers on February 28, 2019 for their review in advance of the panel meeting.



## Technology Assessment Program (TAP)

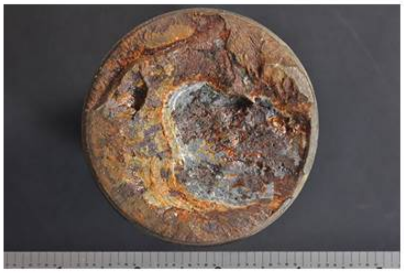
- BSEE develops standards and regulations to enhance operational safety and environmental protection in connection with the exploration, development, and production of offshore oil and natural gas on the U.S. Outer Continental Shelf (OCS)
- BSEE's responsibilities include permitting, inspections, development of offshore regulatory policy, and development of training courses for agency personnel
- BSEE's **Technology Assessment Program (TAP)** supports research regarding operational safety and environmental protection related to offshore oil and natural gas exploration and development, and is an important part of BSEE's safety program

2 Presented to March 5-6 2019 Peer Review Panel Meeting ENDYNA

## TAP Program (continued)

**BSEE's TAP Program:**

- Operates through contracts with universities, private firms, and government laboratories to evaluate safety-related technologies
- Nearly 900 research and development projects, since its inception



TAP was previously known as the Technology Assessment & Research Program

3 Presented to March 5-6 2019 Peer Review Panel Meeting ENDYNA

## TAP Program Objectives

### BSEE's TAP Program has four primary objectives:

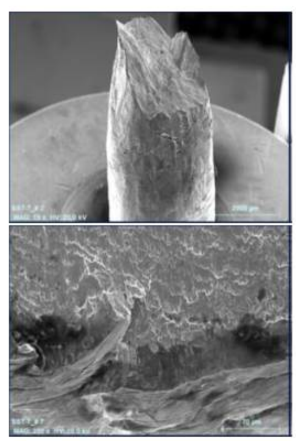
- **Technical Support** - Provides BSEE's decision makers with technical engineering support as they evaluate operational proposals from industry on a variety of topics and related technical issues. The Technical Support staff ensure these proposals comply with applicable regulations, rules, operational guidelines, and standards.
- **Technology Assessment** - Investigates and assesses industry applications of technological innovations and promotes the use of Best Available and Safest Technologies (BAST) in BSEE regulations, rules, and operational guidelines.
- **Research Catalyst** - Promotes operational safety, pollution prevention, and supports leadership in the fields of operational safety and environmental protection associated with offshore energy development activities.
- **International Regulatory** - Provides international cooperation for Research and Development initiatives, enhances the safety of offshore energy extraction activities and the development of appropriate regulatory program elements worldwide.

These objectives are met through TAP's functional research activities, which focus on the development of new concepts, operational procedures, and technologies to meet the physical and economic challenges imposed by the operating environments associated with OCS oil and gas work.

4 *Presented to March 5-6 2019 Peer Review Panel Meeting* ENDYNA

## Overview of TAP 766 Study

- Initial Project Title:
  Determination of Fracture/Fatigue-Fracture Behavior of Equipment Constructed with Cladded Weld Materials
  - Awarded in 2015 to Concurrent Technologies Corporation (CTC)
  - Award value: $618,475
- Description:
  - Define a general approach to, and begin to advance development of, a useful modeling tool for analyzing failure modes of cladded weld materials used in oil and gas operations in high pressure (≥15,000 psi) and high temperature (≥350°F) (HPHT), corrosive environments ($H_2S$, Cl, S, and $CO_2$)
  - Apply physical material testing with modeling to predict material performance in a specified high pressure-high temperature (HPHT), corrosive environment



From TAP 766 Study

5 *Presented to March 5-6 2019 Peer Review Panel Meeting* ENDYNA

## TAP 766 Study Objectives

- Offshore oil and gas drilling and production operations are occasionally conducted in high-pressure (≥15,000 psi) high-temperature (≥350˚F) (HPHT), and highly corrosive (H2S, Cl, S, and CO2) conditions. These harsh environments pose operational challenges for equipment currently used by the oil and gas industry.
- The purpose of TAP 766 was to conduct physical material testing and develop a modeling tool to predict the component failure modes and define fatigue and fracture behavior of a weld-overlay clad material in HPHT, sour-gas environments.
- In addition, the TAP 766 study developed fatigue and fatigue crack growth rate (FCGR) models (i.e., mathematical equations) suitable for numerical simulations. Such material models are often required for accurate hand calculations or numerical simulations of equipment to predict response during fatigue or fracture events.


From TAP 766 Study

6 *Presented to March 5-6 2019 Peer Review Panel Meeting* ENDYNA

## TAP 766 Project Team

- TAP 766 awarded to CTC, with Michael Tims as Principal Investigator
- CTC selected DNV GL of Dublin, Ohio, as the test vendor (based on an open solicitation) to complete material tests under HPHT sour-gas conditions
  - DNV GL had preexisting facility and experience in conducting similar tests in desired test environment
  - All HPHT sour-gas material testing was conducted at DNV GL
- CTC machined the test specimens
- DNV GL and CTC completed selected metallurgical analyses
- CTC completed development of material models and a database of results

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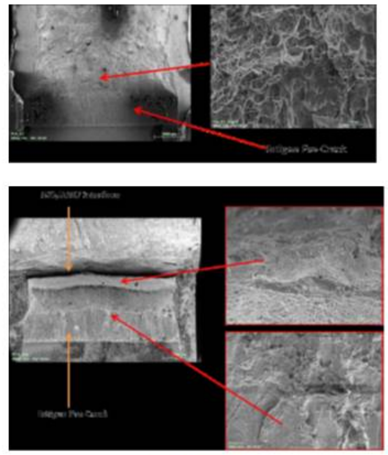
## TAP 766 Material Testing

The objective of the experimental work was to measure the following fatigue and fracture properties of nickel-based Inconel® 625, which had been cladded to a steel alloy ASTM International (ASTM) A387 Grade F22, Class 2 (A387) substrate plate:

- Stress corrosion cracking,
- Fracture toughness,
- Cyclic fatigue, and
- Fatigue crack growth rate (FCGR).



From TAP 766 Study

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## TAP 766: Mechanical Property Tests

**Table 3: Completed Mechanical Property Tests**

| Mechanical Property | ASTM Method | Rationale | Number of Test Specimens | Comments |
|---|---|---|---|---|
| Engineering Stress-Strain | E21 [4] | Determine yield and ultimate tensile stresses | 1 | Required to determine stress levels for cyclic fatigue and other testing |
| Cyclic Fatigue | E466 [5] | Generate $S$-$N_f$ curves to evaluate fatigue performance | 20 | Establish complete specimen failure by cyclic fatigue; $S$ = peak cyclic stress; $N_f$ = number of cycles to failure |
| Fracture Toughness | E1820 [6] | Determine $J_{Ic}$ fracture toughness values | 7 | Establishes dynamic fracture behavior; $J_{Ic}$ = plastic-elastic fracture toughness |
| Fatigue Crack Growth Rate | E647 [7] | Determine crack growth rates | 10 | Establishes crack growth rate resulting from loading on material with a given flaw |
| Slow Strain Rate Tensile | G129 [8] | Evaluate the effects of HPHT sour-gas environment relative to testing in air | 6 in target HPHT sour-gas environment; 4 in air | Qualitatively measure rate of attack on cladding subjected to HPHT sour-gas environment |
| Stress Corrosion Cracking | G39 [9] | Qualitatively evaluate the effects of environment on crack propensity | 9 | Three replicates of each of three apparent yield load levels |

*(see Table 3 in Final TAP 766 Report)*

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## TAP 766: Sour Test Environment/Corrosion Specifications

**ANSI** (American National Standards Institute) and **NACE** (NACE International, formerly known as National Association of Corrosion Engineers) Standards related to testing in HPHT sour-gas environments:

- **ANSI/NACE MR0175/ISO 15156**, Petroleum and natural gas industries - Materials for use in H2S-containing environments in oil and gas production – 2015 (includes parts 1, 2, and 3). Third Edition (Replaced 2009 Second Edition)
- **ANSI/NACE TM0284-2016**, Evaluation of Pipeline and Pressure Vessel Steels for Resistance to Hydrogen-Induced Cracking (Revises NACE TM0284-2011)
- **NACE TM0177-2016**, Laboratory Testing of Metals for Resistance to Sulfide Stress Cracking and Stress Corrosion Cracking in H2S Environments (Revises NACE Standard TM0177-2005)

*(see Table 4 in Final TAP 766 Report)*

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## TAP 766: Selected by BSEE for Peer Review

- Current BSEE regulations require that offshore oil and gas operators submit detailed information that demonstrates equipment are capable of performing in the applicable HPHT environment as specified in submitted Applications for Permit to Drill (APD), Applications for Permit to Modify (APM), and Deep Water Operations Plans (DWOP) (30 CFR 250.804).
- BSEE has a need to determine whether or not operators' submissions are acceptable and equipment is fit for service based upon proper modeling, safety factors, appropriate applicable standards, and whether appropriate design basis were applied.

BSEE considers TAP 766 Study to be a **highly influential scientific assessment**

This peer review will evaluate and assess the TAP 766 project results

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# 6. HANDOUT

This section provides a handout prepared by Mr. Badrak that identified the parts of ANSI/NACE MR0175 / ISO 15156 that Mr. Badrak considered relevant to the TAP 766 study.

## Background on ANSI/NACE MR0175 / ISO 15156

"ANSI/NACE MR0175/ISO 15156 gives requirements and recommendations for the selection and qualification of CRAs (corrosion-resistant alloys) and other alloys for service in equipment used in oil and natural gas production and natural gas treatment plants in $H_2S$-containing environments whose failure can pose a risk to the health and safety of the public and personnel or to the environment. It supplements, but does not replace, the materials requirements of the appropriate design codes, standards, or regulations" (SCOPE part 3)

This part of ANSI/NACE MR0175/ISO 15156 applies to the qualification and selection of materials for equipment designed and constructed using load-controlled design methods. For design utilizing strain-based design methods, see ANSI/NACE MR0175/ISO 15156-1:2015, Clause 5.

### 15156-1 Clause 5

Users of the ANSI/NACE MR0175/ISO 15156 series shall first assess the conditions to which the materials they wish to select can be exposed. These conditions shall be evaluated, defined, and documented in accordance with this part of ANSI/NACE MR0175/ISO 15156.

The equipment user shall determine whether or not the service conditions are such that the ANSI/NACE MR0175/ISO 15156 series applies.

Materials selection shall be made following the requirements and recommendations of ANSI/NACE MR0175/ISO 15156-2 or ANSI/NACE MR0175/ISO 15156-3, as appropriate.

The use of ANSI/NACE MR0175/ISO 15156-2 or ANSI/NACE MR0175/ISO 15156-3 can require an exchange of information (for example, concerning required or suitable service conditions) between the equipment user and the equipment or materials supplier. If necessary, the equipment user should advise other parties of the service conditions.

NOTE It can be necessary for the equipment supplier to exchange information with the equipment manufacturer, the materials supplier, and/or the materials manufacturer.

Qualification, with respect to a particular mode of failure, for use in defined service conditions also qualifies a material for use under other service conditions that are equal to or less severe in all respects than the conditions for which qualification was carried out.

It is the equipment user's responsibility to ensure that any material specified for use in their equipment is satisfactory in the service environment.

It is the equipment or materials supplier's responsibility to meet the metallurgical and manufacturing requirements and, when necessary, any additional testing requirements of the ANSI/NACE MR0175/ISO 15156 series for the material selected in the condition in which it enters into service.

It is the equipment or materials supplier's responsibility to comply with the requirements for the marking/documentation of materials in accordance with ANSI/NACE MR0175/ISO 15156-2:2015, Clause 9 or ANSI/NACE MR0175/ISO 15156-3:2015, 7.2, as appropriate.

This part of ANSI/NACE MR0175/ISO 15156 applies to the qualification and selection of materials for equipment designed and constructed using load controlled design methods. For designs utilizing strain-based design methods, use of this part of ANSI/NACE MR0175/ISO 15156 might not be appropriate and other test methods, not addressed in ANSI/NACE MR0175/ISO 15156, might be required. The equipment/material supplier, in conjunction with the equipment user, shall define and agree on other testing requirements and acceptance criteria.

**15156-3 Clause 6**

**Qualification and selection of CRAs and other alloys with respect to SSC, SCC, and GHSC in H2S-containing environments**

**General**

CRAs and other alloys shall be selected for their resistance to SSC, SCC, and/or GHSC as required by the intended service.

Compliance of a CRA or other alloy with this part of ANSI/NACE MR0175/ISO 15156 implies cracking resistance within defined environmental service limits. These limits are dependent on the material type or the individual alloy.

To enable qualification and/or selection of CRAs and other alloys, the equipment purchaser can be required to provide information on the proposed conditions of exposure to the equipment supplier.

In defining the severity of $H_2S$-containing environments, exposures that can occur during system upsets or shutdowns, etc. shall also be considered. Such exposures can include unbuffered, low pH condensed water. The limits given in the tables in Annex A are for production environments and do not cover conditions occurring during injection or flowback of chemicals that can reduce the *in situ* pH.

CRAs and other alloys shall be selected using Annex A or following qualification by successful laboratory testing in accordance with Annex B. Qualification based on satisfactory field experience is also acceptable. Such qualification shall comply with ANSI/NACE MR0175/ISO 15156-1.

In Annex A, materials are identified by materials groups. Within each group, alloys are identified by materials type (within compositional limits) or as individual alloys. Acceptable metallurgical conditions and environmental limits are given for which alloys are expected to resist cracking. Environmental limits are given for $H_2S$ partial pressure, temperature, chloride concentration, and elemental sulfur.

A CRA or other alloy can be qualified by testing for use under operating conditions that are more severe than the environmental limits given in Annex A. Similarly, a CRA or other alloy can be qualified for use in different metallurgical conditions (higher strength, alternative heat treatment, etc.) to those given in Annex A.

The documentation of qualifications performed in accordance with Annex B shall meet the requirements in ANSI/NACE MR0175/ISO 15156-1:2015, Clause 9.

The equipment user shall verify qualifications (see B.2.2) and retain documentation supporting the materials selections made.

**Cracking-resistance properties of welds**

**General**

The metallurgical changes that occur when welding CRAs and other alloys can affect their susceptibility to SSC, SCC, and/or GHSC. Welded joints can have a greater susceptibility to cracking than the parent material(s) joined.

The equipment user may allow the cracking susceptibility of weldments to govern the limits of safe service conditions for a fabricated system.

Processes and consumables used in welding should be selected in accordance with good practice and to achieve the required corrosion and cracking resistances.

Welding shall be carried out in compliance with appropriate codes and standards as agreed between the supplier and the purchaser. Welding procedure specifications (WPSs) and procedure qualification records (PQRs) shall be available for inspection by the equipment user.

Welding PQRs shall include documented evidence demonstrating satisfactory cracking resistance under conditions at least as severe as those of the proposed application. Such evidence shall be based upon one or more of the following:

- compliance with the requirements and recommendations for the specific materials group of Annex A (see also 6.2.2.2 and 6.2.2.3);
- weld cracking-resistance qualification testing in accordance with Annex B;
- documented field experience modelled upon that specified for parent materials in ANSI/NACE MR0175/ISO 15156-1.

The requirements and recommendations given in Annex A might not be appropriate for all combinations of parent and weld metals used in the fabrication of equipment and components.

The equipment user may require evidence of successful cracking-resistance testing as part of the welding procedure qualification to ensure the weldment produced provides adequate resistance to SSC, SCC, and GHSC for the application.

Hardness testing is an integral component of weld qualification. Hardness Vickers 5kg or 10kg traverses are essential.

| **Materials type** | **Cr mass fraction** | **Ni + Co mass fraction** | **Mo mass fraction** | **Mo + W mass fraction** | **Metallurgical condition** |
|---|---|---|---|---|---|
| | min<br>% | min<br>% | min<br>% | min<br>% | |
| Type 4a | 19.0 | 29.5 | 2.5 | — | Solution-annealed or annealed |
| Type 4b | 14.5 | 52 | 12 | — | Solution-annealed or annealed |
| Type 4c | 19.5 | 29.5 | 2.5 | — | Solution-annealed or annealed and cold-worked |
| Type 4d | 19.0 | 45 | — | 6 | Solution-annealed or annealed and cold-worked |
| Type 4e | 14.5 | 52 | 12 | — | Solution-annealed or annealed and cold-worked |
| Type 4f[a] | 20.0 | 58 | 15.5 | — | a) Solution-annealed or annealed and cold-worked condition<br>b) Solution-annealed or annealed and cold-worked and aged condition |
| Table D.3 lists the chemical compositions of some alloys that can, but do not necessarily, meet the restrictions of one or more of these types. In some cases, more restrictive compositions than those shown in Table D.3 may be needed.<br>[a] The type 4f family is currently limited to only UNS N07022. | | | | | |

| **Materials type** | **Temperature** | **Partial pressure $H_2S$ $pH_2S$** | **Chloride conc.** | **pH** | **Sulfur-resistant?** | **Remarks** |
|---|---|---|---|---|---|---|
| | max<br>°C (°F) | max<br>kPa (psi) | max<br>mg/l | | | |
| Cold-worked alloys of types 4c, 4d and 4e | 232 (450) | 200 (30) | See "Remarks" column | See "Remarks" column | No | Any combination of chloride concentration and *in situ* pH occurring in production environments is acceptable. |
| | 218 (425) | 700 (100) | See "Remarks" column | See "Remarks" column | No | |
| | 204 (400) | 1 000 (150) | See "Remarks" column | See "Remarks" column | No | |

| Materials type | Temperature max °C (°F) | Partial pressure $H_2S$ $pH_2S$ max kPa (psi) | Chloride conc. max mg/l | pH | Sulfur-resistant? | Remarks |
|---|---|---|---|---|---|---|
| | 177 (350) | 1 400 (200) | See "Remarks" column | See "Remarks" column | No | |
| | 132 (270) | See "Remarks" column | See "Remarks" column | See "Remarks" column | Yes | Any combination of hydrogen sulfide, chloride concentration, and *in situ* pH in production environments is acceptable. |
| Cold-worked alloys of types 4d and 4e | 218 (425) | 2 000 (300) | See "Remarks" column | See "Remarks" column | No | Any combination of chloride concentration and *in situ* pH occurring in production environments is acceptable. |
| | 149 (300) | See "Remarks" column | See "Remarks" column | See "Remarks" column | Yes | Any combinations of hydrogen sulfide, chloride concentration, and *in situ* pH in production environments are acceptable. |

### A.4.3 Welding solid-solution nickel-based alloys of this materials group

The requirements for the cracking-resistance properties of welds shall apply (see 6.2.2).

The hardness of the HAZ after welding shall not exceed the maximum hardness allowed for the base metal and the hardness of the weld metal shall not exceed the maximum hardness limit of the respective alloy used for the welding consumable.

There are no hardness requirements for welding solid-solution nickel-based alloys with solid-solution nickel-based weld metal.

### A.13 Cladding, overlays, and wear-resistant alloys

### A.13.1 Corrosion-resistant claddings, linings and overlays

The materials listed and defined in A.2 to A.11 can be used as corrosion-resistant claddings, linings, or as weld overlay materials.

Unless the user can demonstrate and document the likely long-term in-service integrity of the cladding or overlay as a protective layer, the base material, after application of the cladding or overlay, shall comply with ANSI/NACE MR0175/ISO 15156-2 or this part of ANSI/NACE MR0175/ISO 15156, as applicable.

This may involve the application of heat or stress-relief treatments that can affect the cladding, lining, or overlay properties.

Factors that can affect the long-term in-service integrity of a cladding, lining, or overlay include environmental cracking under the intended service conditions, the effects of other corrosion mechanisms, and mechanical damage.

Dilution of an overlay during application that can impact on its corrosion resistance or mechanical properties should be considered.

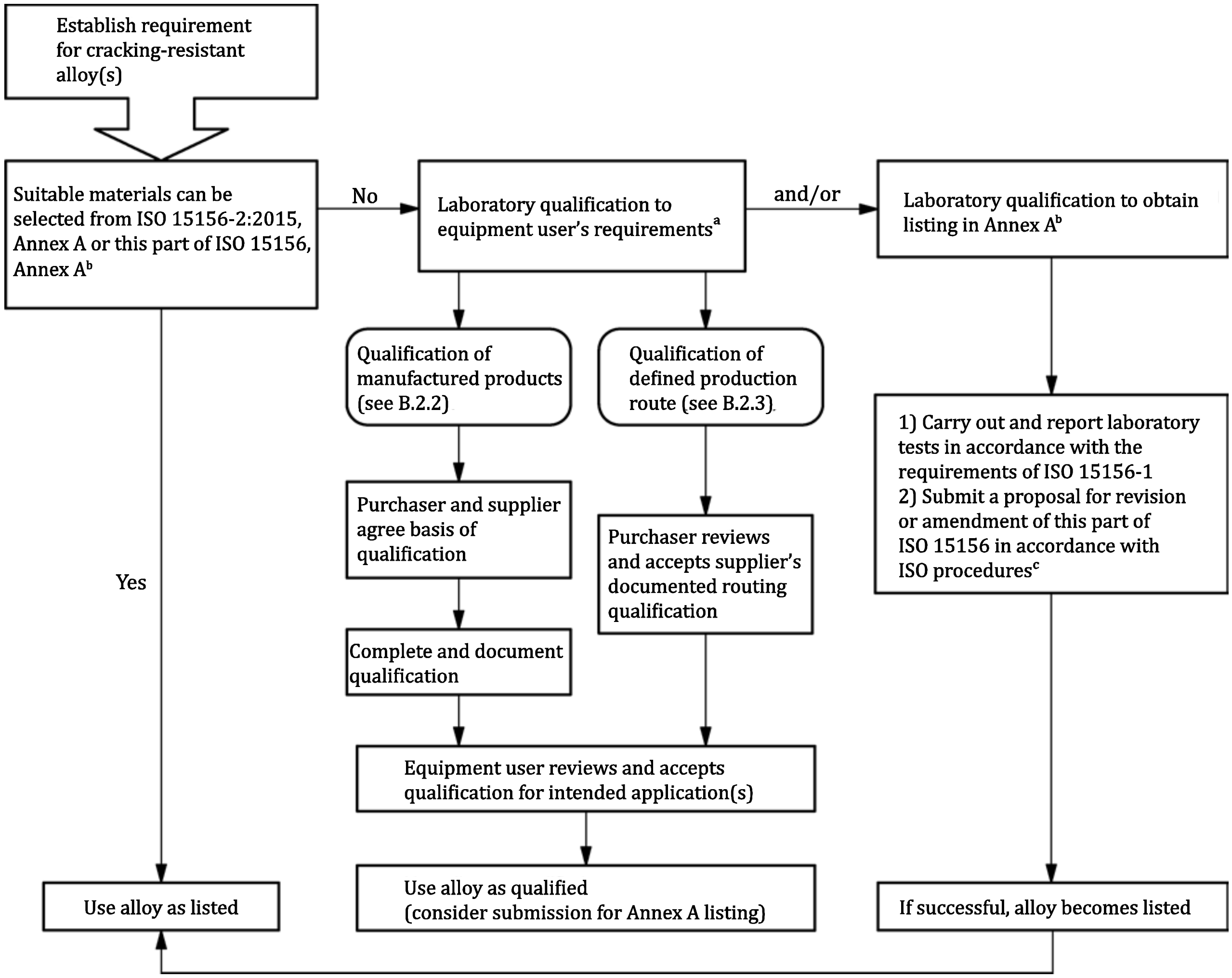
**Table B.1 — Cracking mechanisms that shall be considered for CRA and other alloy groups**

| Materials groups of Annex A | Potential cracking mechanisms in $H_2S$ service[a, b] | | | Remarks |
|---|---|---|---|---|
| | SSC | SCC | GHSC | |
| Solid-solution nickel-based alloys (see A.4) | S | P | S | Some Ni-based alloys in the cold-worked condition and/or aged conditions contain secondary phases and can be susceptible to HSC when galvanically coupled to steel.<br>In the heavily cold-worked and well-aged condition coupled to steel, these alloys can experience HSC. |

[a] P indicates primary cracking mechanism.
[b] S indicates secondary, possible, cracking mechanism.

## General

An overview of the uses of laboratory qualifications is given in Figure B.1




Key

a This part of ANSI/NACE MR0175/ISO 15156 addresses SSC, SCC, and GHSC of CRAs and other alloys. ANSI/NACE MR0175/ISO 15156-2 addresses SSC, HIC, SOHIC, and SZC of carbon and low alloy steels.

b Annex A addresses SSC, SCC, and GHSC of CRAs and other alloys. ANSI/NACE MR0175/ISO 15156-2:2015, Annex A addresses SSC of carbon and low alloy steels.

### B.2.2 Qualification of manufactured products

The user of this part of ANSI/NACE MR0175/ISO 15156 shall define the qualification requirements for the material in accordance with ANSI/NACE MR0175/ISO 15156-1 and Annex B.

This definition shall include the application of the following:

a) general requirements (see ANSI/NACE MR0175/ISO 15156-1:2015, Clause 5);
b) evaluation and definition of service conditions (see ANSI/NACE MR0175/ISO 15156-1:2015, Clause 6);
c) material description and documentation (see ANSI/NACE MR0175/ISO 15156-1:2015, 8.1);
d) requirements for qualification based upon laboratory testing (see ANSI/NACE MR0175/ISO 15156-1:2015, 8.3);

e) report of the method of qualification (see ANSI/NACE MR0175/ISO 15156-1:2015, Clause 9).

Appropriate “test batches” and sampling requirements shall be defined having regard to the nature of the product, the method of manufacture, testing required by the manufacturing specification, and the required qualification(s) (see Table B.1).

Samples shall be tested in accordance with Annex B for each cracking mechanism to be qualified. A minimum of three specimens shall be tested per test batch. The test batch shall be qualified if all specimens satisfy the test acceptance criteria.

Retesting is permitted in accordance with the following. If a single specimen fails to meet the acceptance criteria, the cause shall be investigated. If the source material conforms to the manufacturing specification, two further specimens may be tested. These shall be taken from the same source as the failed specimen. If both satisfy the acceptance criteria, the test batch shall be considered qualified. Further retests shall require the purchaser’s agreement.

Testing of manufactured products may be carried out at any time after manufacture and before exposure to $H_2S$ service.

Before the products are placed in $H_2S$ service, the equipment user shall review the qualification and verify that it satisfies the defined qualification requirements. Products with a qualification that has been verified by the equipment user may be placed into $H^2S$ service.

### B.2.3 Qualification of a defined production route

A defined production route may be qualified for the production of qualified material.

A qualified production route may be followed to avoid order release testing for $H_2S$ cracking resistance.

A materials supplier may propose to a materials purchaser that a qualified production route be used to produce qualified materials. The qualified production route may be used if the materials supplier and materials purchaser agree to its use.

A qualified production route may be used to produce qualified material for more than one materials user.

To qualify a production route, the material supplier shall demonstrate that a defined production route is capable of consistently manufacturing material that satisfies the applicable qualification test requirements of Annex B.

The qualification of a production route requires all of the following:

a) definition of the production route in a written quality plan that identifies the manufacturing location(s), all manufacturing operations, and the manufacturing controls required to maintain the qualification;

b) initial testing of products produced on the defined production route in accordance with B.2.2 and verifying they satisfy the acceptance criteria;
c) periodic testing to confirm that the product continues to have the required resistance to cracking in $H_2S$ service. The frequency of "periodic" testing shall also be defined in the quality plan and shall be acceptable to the purchaser. A record of such tests shall be available to the purchaser;
d) retaining and collating the reports of these tests and making them available to material purchasers and/or equipment users.

A material purchaser may agree additional quality control requirements with the manufacturer.

The accuracy of the quality plan may be verified by site inspection by an interested party.

Changes to a production route that fall outside the limits of its written quality plan require qualification of a new route in accordance with a), b), c), and d) above.

### B.3.2 Materials

The materials tested shall be selected in accordance with the requirements found in ANSI/NACE MR0175/ISO 15156-1:2015, 8.3.2.

In addition, consideration shall be given to the following:
a) the cracking mechanism for which testing is required (see Table B.1);
b) the testing of appropriately aged samples of alloys that can age in service, particularly HSC testing of downhole materials that can be subject to ageing in service ("well ageing");
c) the directional properties of alloys because cold-worked alloys may be anisotropic with respect to yield strength and for some alloys and products, the susceptibility to cracking varies with the direction of the applied tensile stress and consequent orientation of the crack plane.

### B.3.3 Test methods and specimens

Primary test methods use constant load, sustained load (proof-ring), or constant total strain (constant displacement) loading of smooth test specimens.

Uniaxial tensile (UT) tests, four-point bend (FPB) tests, and C-ring (CR) tests may be performed with the above loading arrangements.

Generally, constant load tests using UT specimens are the preferred method of testing homogeneous materials.

Test specimens shall be selected to suit the product form being tested and the required direction of the applied stress. A minimum of three specimens shall be taken from each component tested.

UT specimens may be taken from welded joints in accordance with EFC Publication Number 17, Figure 8.1. Other specimens taken from welded joints may be tested with weld profiles as intended for service.

When double (back-to-back) FPB specimens are used (in accordance with EFC Publication Number 17, Figure 8.2a, or similar), uncracked specimens shall be disqualified as invalid if the opposing specimen cracks.

Alternative test methods or specimens may be used when appropriate. The basis and use of such tests shall be documented and agreed with the equipment user.

Examples of test methods that may be considered are as follows.

- Fracture mechanics tests, e.g. double cantilever beam (DCB) tests, may be used if cracks are unaffected by branching and remain in the required plane. This normally limits DCB tests to SSC and HSC tests.
- Tests involving the application of a slow strain rate, e.g. SSRT in accordance with NACE TM0198, interrupted SSRT in accordance with ISO 7539-7 or RSRT in accordance with the method published as NACE CORROSION/97 Paper 58.

Tests may utilize testing of full-size or simulated components when appropriate.

### B.3.4 Applied test stresses/loads for smooth specimens

The yield strengths of CRAs used to derive test stresses shall be determined at the test temperature in accordance with the applicable manufacturing specification. In the absence of an appropriate definition of yield strength in the manufacturing specification, the yield strength shall be taken to mean the 0.2 % proof stress of non-proportional elongation ($R_{p0,2}$ as defined in ISO 6892-1) determined at the test temperature.

Directional properties shall be considered when selecting test specimens and defining test stresses.

For welded specimens, the parent metal yield strength shall normally be used to determine test stresses. For dissimilar joints, the lower parent metal yield strength shall normally be used. When design stresses are based on the yield strength of a weld zone that is lower than the yield strength of either adjoining parent metals, the yield strength of the weld zone may be used to determine test stresses.

For constant-load tests and sustained-load (proof-ring) tests, specimens shall be loaded to 90 % of the AYS of the test material at the test temperature.

For constant total strain (deflection) tests, specimens shall be loaded to 100 % of the AYS of the test material at the test temperature.

NOTE Constant total strain (deflection) tests might not be suitable for materials that can relax by creep when under load.

Lower applied stresses can be appropriate for qualifying materials for specific applications. The use and basis of such tests shall be agreed with the purchaser and documented.

# 7. REFERENCE #14 FROM FINAL TAP 766 REPORT

During the peer review panel meeting, as noted in Section 2, Dr. Hawk pointed to Reference [14] or the "project interim report" cited on page 9 of the final TAP 766 report and questioned why Reference [14] had the same publication date as the final report and whether Reference [14] may have more data. Dr. Hawk requested that Ms. Amy Doll, EnDyna's Peer Review Lead, try to obtain a copy of Reference [14] for the peer reviewers.

During the panel meeting, Ms. Doll contacted the BSEE COR (Mr. Mark Kozak) who immediately sent a PDF version of Reference [14] or the "project interim report" cited on page 9 of the final TAP 766 report. Ms. Doll emailed the PDF version to each peer reviewer and provided a printed copy for the peer reviewers to examine during the panel meeting.

Because the peer reviewers frequently examined Reference [14] throughout the remainder of the peer review panel meeting, this section provides Reference [14] or the "project interim report" as part of EnDyna's documentation for the peer review panel meeting.

**Characterizing the Behavior of Inconel Clad A387 Steel in High-Pressure High-Temperature, Corrosive Environment**
**Bureau of Safety and Environmental Enforcement (BSEE)**

**Contract No. E15PC00010**

# Fatigue and Fracture Properties of Inconel 625 Cladding under High-Pressure High-Temperature Sour-Gas Conditions: Revision 1

**Prepared by**

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**Report Date**
**April 30, 2018**

**Submitted by**

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## EXECUTIVE SUMMARY

The environment in which deep-water oil and gas exploration and extraction occurs is often both high pressure (15,000 psi or more) and high temperature (350 °F or higher) (HPHT). These conditions are often exacerbated by highly corrosive sour gas (with high concentrations of $H_2S$ and $CO_2$) and high concentrations of chloride ($Cl^-$). Components made of high-strength ferrous alloys are susceptible to hydrogen embrittlement under these conditions. To combat this problem, the industry uses corrosion-resistant alloys (including nickel-based Inconel[1] alloys) weld cladded to the surfaces of ferrous components that come into contact with HPHT sour-gas conditions. While providing resistance to these conditions, the impact to fatigue and fracture of these cladding materials has not been well documented in the open literature. Therefore, the Bureau of Safety and Environmental Enforcement (BSEE) awarded a contract to Concurrent Technologies Corporation (CTC) to generate common fatigue and fracture data. Using specialized test equipment, DNV GL measured the following properties: fracture toughness, fatigue crack growth rate (FCGR) and cyclic fatigue under HPHT sour-gas conditions. Specifically, this team evaluated Inconel 625 cladding, which was weld cladded to an ASTM A387 Grade 22, Class 2 steel substrate plate. As the clad test plate required two clad passes to achieve the minimum 0.25-inch clad thickness, and due to differences in dilution from the substrate plate in each of the clad layers, fracture toughness and FCGR were measured separately for both clad layers. Supporting these tests, slow-strain-rate (SSR) tensile, engineering stress-strain and bent-beam stress corrosion cracking (SCC) tests were also completed.

Salient conclusions from the work include the following.

- The data provided in this report are a good start towards having a broad collection of publically available fatigue and fracture data for use by designers, failure analysts and regulatory bodies within the oil and gas exploration and extraction industry for clad components subjected to HPHT sour-gas conditions.
- Fatigue and fracture differences were noted between the inner and outer layers of the two-layer weld cladding evaluated in the present project. Treating each clad layer as a “separate” material in fatigue and fracture assessments is justified.
- No observable cracking or pitting was observed in any of the SCC specimens, which were subjected to the HPHT sour-gas environment for 30 days. A total of nine SCC specimens were tested: three replicates each tested at 95%, 110% and 120% of apparent yield load.
- Slow strain rate tensile tests performed in the HPHT sour-gas environment did not show any evidence of environmentally assisted cracking. The fracture surface exhibited a ductile failure mode.
- Fracture toughness tests performed in air and sour-gas environments in both the upper (low iron dilution) and lower (high iron dilution) Inconel 625 clad layers indicated the fracture toughness of both clad layers is high. The fracture surfaces exhibited ductile features, suggesting that neither clad layers were susceptible to environmentally assisted fracture. The specimen from the lower clad layer (i.e., the one with greater dilution from the steel substrate) generally had higher initiation fracture toughness (threshold value of $J$ > 240 N/mm, where $J$ is fracture toughness) than did the specimens from the upper clad layer (threshold value of $J$ ~190 N/mm). Plane-strain plastic-elastic fracture toughness ($J_{Ic}$, defined as the $J$ value at a crack

[1] Inconel is a registered trademark of Special Metals Corporation, Huntington, WV.

mouth opening displacement of 0.2 mm) averaged 257 N/mm for the upper clad layer; the singular $J_{Ic}$ value for the lower clad layer was 344 N/mm.

- FCGR frequency scan tests on both the upper (low iron [Fe] dilution) and lower (high Fe dilution) Inconel 625 clad layers did not exhibit a strong frequency dependence between 1 Hz and 3 mHz. However, between 1 mHz and 0.1 mHz, FCGR increased by about 100×. Although this suggests that chemical attack occurs at the crack tip, thereby making the material more susceptible to crack <u>growth</u> over time, effects of static growth rate, especially at the lowest test frequencies, may also have played a role in the increased FCGR at low test frequencies. During frequency scans, the lower layer (i.e., the one more highly diluted with substrate material) was found to have a higher FCGR by about an order of magnitude (i.e., 10×) over the upper layer. When the material was tested in the Paris law[2] region, the FCGR of the lower layer was about twice that of the upper layer. These results suggest any crack that starts from the exterior of a cladded component may accelerate its growth rate once the outer clad layer has been completely penetrated and the crack grows into the lower clad layer.
- To achieve failure within a few hundred to a few thousand cycles, cyclic fatigue specimens must be notched with a stress concentration factor of about 4.0 and subjected to nominal stresses that exceed yield. (For the Inconel 625 cladding evaluated here, the measured 0.2% offset yield strength at 350 °F was 65.9 ksi.) Fatigue failures occurred between 4000 to 10,000 cycles in the peak cyclic stress range of 60 to 88 ksi. The log-log relationship between the number of cycles to failure and peak cyclic stress followed a linear relationship with minimal scatter around the best-fit curve, which included peak cyclic stresses both below and above the Inconel 625 cladding yield strength.
- While the HPHT sour-gas environment may lead to greater scatter (~5%) in tensile elongation, reduction of area and time to failure during slow-strain-rate testing, the <u>mean</u> values of these tensile properties were not significantly altered (< 1%) from values measured in air at 350 °F.
- Additive manufacturing methods were useful for providing material to the top of cladding without impairing its original microstructure/mechanical properties and enable physical completion of fracture and FCGR specimens.

The following recommendations are offered based upon the work.

- Given the typical variability of weld cladding properties, additional testing is recommended to supplement those discussed here. Specimens from additional Inconel-625-clad test plates and additional clad vendors would help to further define the variability that could be expected among potential clad vendors and the normal variability expected from the weld cladding process itself.
- Similarly, while cyclic fatigue testing was completed at a single stress ratio ($R$ = 0.13), completing additional cyclic fatigue tests at other stress ratios (and possibly with other than sinusoidal stress versus time cycles) would provide the industry with additional valuable data.
- Since other cladding alloys are either being used or are being considered for use by the oil and gas exploration and extraction industry, complementing the present work by assessing the fatigue and fracture behavior of these other materials would also benefit the industry.

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[2] The Paris law region of a FCGR curve is the linear region of the log-log curve of crack growth rate versus the range of the stress intensity factor.

- To determine low-cycle stress-based fatigue curves for common cladding materials, the test should start with nominal peak cyclic stresses just above and just below the yield strength of the cladding.

The measured values are useful for numerical simulations of fatigue and fracture. Specifically, designers can use the information to estimate the lifetime of components. Accident investigators can use the information to determine critical issues that led to failures. Regulatory bodies can use the information to evaluate the value of designs proposed for use in HPHT sour-gas conditions. Armed with these data, designers can use the information to estimate the lifetime of components. Accident investigators can use the information to determine critical issues that led to failures. Regulatory bodies can use the information to assess the value of designs proposed for use in HPHT sour-gas conditions.

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# LIST OF ACRONYMS, ABBREVIATIONS AND SYMBOLS

| | |
|---|---|
| *a* | Crack Length |
| Al | Aluminum |
| AM | Additive Manufacturing |
| ANSI | American National Standards Institute |
| API | American Petroleum Institute |
| ASTM | ASTM International |
| AYL | Apparent Yield Load |
| A387 | ASTM A387 Grade 22, Class 2 |
| *a*/*W* | Crack Length to Specimen Width Ratio |
| B | Boron |
| *B* | Specimen Thickness |
| BSEE | Bureau of Safety and Environmental Enforcement |
| C | Carbon |
| CD | Compact Disc |
| CGR | Crack Growth Rate |
| $Cl^-$ | Chloride |
| CMOD | Crack-Mouth Opening Displacement |
| $CO_2$ | Carbon Dioxide |
| Cr | Chromium |
| CRA | Corrosion Resistant Alloy |
| CTC | Concurrent Technologies Corporation |
| Cu | Copper |
| C(T) | Compact Tension |
| d*a*/d*N* | Instantaneous Crack Growth Rate per Cycle |
| d*a*/d*t* | Time Rate of Crack Growth |
| DCPD | Direct Current Potential Drop |
| Dia | Diameter |
| DMM | Digital Multimeter |
| DNV | DNV GL |
| EDM | Electrical Discharge Machining |
| EDS | Energy Dispersion Spectroscopy |
| EL | Elongation |
| Env | Environment, i.e., HPHT sour-gas conditions |
| etc. | Et Cetera, And So on |
| e.g. | Exempli gratia; for example |
| *f* | Frequency |
| FAD | Failure Assessment Diagram |
| FAT | Fatigue Test |
| FCGR | Fatigue Crack Growth Rate |
| FE | Finite Element |
| Fe | Iron |
| FT | Fracture Toughness |
| g | Gram |

| | |
|---|---|
| h | Hour |
| Hi-Tech | Hi-Tech Weld Overlay Group, LLC |
| HPHT | High Press High Temperature |
| hr | Hour |
| Hz | Hertz |
| $H_2O$ | Water |
| $H_2S$ | Hydrogen Sulfide |
| ID | Identification |
| in | Inch |
| Inc. | Incorporated |
| IN625 | Inconel 625 |
| ISO | The International Organization for Standardization |
| i.e. | Id Est; That Is |
| $J$ | Fracture Toughness |
| $J_{Ic}$ | Plastic-Elastic Fracture Toughness |
| $J_{MaxLoad}$ | Value of $J$ at Maximum Load |
| $J_q$ | Provisional Estimate for $J_{Ic}$ |
| $J_{th}$ | Threshold Fracture Toughness |
| $J_{0.2\ mm}$ | Value of $J$ at 0.2 mm CMOD |
| $J_{1.0\ mm}$ | Value of $J$ at 1.0 mm CMOD |
| $K$ | Stress Intensity Factor |
| K | Thousands |
| $K_J$ | Crack Initiation Toughness |
| $K_{max}$ | Maximum Stress Intensity Factor |
| ksi | Thousands of Pounds per Square Inch |
| $K_t$ | Stress Concentration Factor |
| l | Liter |
| lbs | Pounds |
| LLC | Limited Liability Company |
| log | Logarithm – Base 10 |
| LOM | Light Optical Microscopy |
| m | Meter |
| Max | Maximum |
| mg | Milligram |
| mHz | Millihertz |
| Min | Minimum |
| min | Minute |
| mm | Millimeter |
| MMPDS | Metallic Materials Properties Development and Standardization |
| Mn | Manganese |
| MO | Missouri |
| Mo | Molybdenum |
| MPa | Millions of Pascals |
| MSE | Mixed Solvent Electrolyte |
| Msi | Millions of Pounds per Square Inch |
| mV | Millivolt |

| | |
|---|---|
| N | Newton |
| $N$ | Number of Cycles |
| N | No |
| NACE | NACE International |
| NaCl | Sodium Chloride |
| $NaHCO_3$ | Sodium Bicarbonate |
| Nb | Niobium |
| NDI | Nondestructive Inspection |
| NETL | National Energy Technology Laboratory |
| $N_f$ | Number of Cycles to Failure |
| $N_{f,97.5\%}$ | One-Sided Statistical Estimate of $N_f$ for the Lower Bound of the Population at 97.5% Confidence |
| $N_{f,99\%}$ | One-Sided Statistical Estimate of $N_f$ for the Lower Bound of the Population at 99% Confidence |
| Ni | Nickel |
| NJ | New Jersey |
| No. | Number |
| $N_2$ | Nitrogen |
| N/A | Not Applicable |
| OH | Ohio |
| P | Phosphorus |
| PA | Pennsylvania |
| $p_{CO2}$ | Partial Pressure of $CO_2$ |
| PH | Precipitation Hardened |
| pH | Potential Hydrogen |
| $p_{H2S}$ | Partial Pressure of $H_2S$ |
| ppm | Parts per Million |
| psi | Pounds per Square Inch |
| psia | Pounds per Square Inch – Absolute |
| PTFE | Polytetrafluoroethylene |
| R | Radius |
| $R$ | Stress Ratio |
| $R$ | Crack Growth Resistance |
| RA | Reduction of Area |
| Ra | Roughness Average of a Surface |
| RT | Room Temperature |
| $S$ | Peak Cyclic Stress |
| S | Sulphur |
| s | Second |
| $\hat{s}$ | Root Mean Square Error |
| SCC | Stress Corrosion Cracking |
| sec | Second |
| SEM | Scanning Electron Microscopy |
| Si | Silicon |
| SSR | Slow-Strain Rate |
| $t$ | Student-t Value |

| | |
|---|---|
| $t$ | Time |
| Ti | Titanium |
| TX | Texas |
| Typ | Typical |
| UNC | Unified National Course Threads |
| USA | United States of America |
| UTS | Ultimate Tensile Strength |
| V | Vanadium |
| $W$ | Specimen Width |
| wt% | Weight Percent |
| WV | West Virginia |
| w/ | With |
| Y | Yes |
| YS | Yield Strength |
| °C | Degrees Celsius |
| °F | Degrees Fahrenheit |
| % | Percent |
| ″ | Inch |
| $\Delta a$ | Change in Crack Length |
| $\Delta K$ | Change in Stress Intensity Factor |
| $\Delta\sigma$ | Difference between Maximum and Minimum Stresses during Cyclic Fatigue Testing |
| $\sigma_{max}$ | Maximum Stress during Cyclic Fatigue Testing |
| $\sigma_{min}$ | Minimum Stress during Cyclic Fatigue Testing |
| μin | Microinch |
| μm | Micron |
| √ | Square Root |
| ~ | Approximately |
| < | Less Than |
| × | Multiplied by |
| ± | Plus or Minus |

# 1.0 INTRODUCTION

The environment in which deep-water oil and gas exploration and extraction occurs is often both high pressure (15,000 psi or more) and high temperature (350 °F or higher) (HPHT). These conditions are often exacerbated by highly corrosive sour (or sweet) gas (with high concentrations of $H_2S$ and $CO_2$) and high concentrations of chloride ($Cl^-$). Components made of high-strength ferrous alloys are susceptible to hydrogen embrittlement and stress corrosion cracking under these conditions. To combat this problem, the industry uses corrosion-resistant alloys (including nickel-based Inconel[3] alloys) weld cladded to the surfaces of ferrous components that come into contact with HPHT sour-gas conditions. While providing resistance to these conditions, the impact to fatigue and fracture of these cladding materials has not been well documented in the open literature. Therefore, the Bureau of Safety and Environmental Enforcement (BSEE) awarded a contract to Concurrent Technologies Corporation (CTC) to generate fatigue and fracture data for a common cladding used in deep-water oil and gas exploration and extraction equipment.

The objective of the current work was to experimentally measure the following fatigue and fracture properties of nickel-based Inconel® 625 [1], which has been cladded to steel alloy ASTM International (ASTM) A387 Grade 22, Class 2 (A387) [2][4]:

- Stress corrosion cracking
- Fracture toughness
- Cyclic fatigue
- Fatigue crack growth rate.

In addition, fatigue and fracture material models (i.e., mathematical equations) suitable for numerical simulations were also desired. Such material models are often required for accurate hand calculations or numerical simulations of equipment to predict response during fatigue or fracture events.

Many failures in the oil and gas exploration and extraction industry occur at a relatively low number of fatigue cycles (several hundreds to a few thousands). In addition, the industry, when asked by the authors, indicated a greater interest in using fatigue data under stress conditions since a majority of components are designed based on stress rather than stain-based fatigue response [3]. Therefore, the present project focused on stress-based fatigue rather than strain-based fatigue measures even at the desired low-cycle count.

In the present work, Inconel 625 cladding was added to a 1-1/4-inch-thick ASTM A387 plate. The required minimum cladding thickness was 0.25 inch. To achieve the needed clad thickness, two separate clad layers were required. In this case, the application direction for both clad layers was identical. Figure 1 illustrates the clad plate from which specimens were taken. With the different level of dilution in each of the two clad layers, CTC, with concurrence from BSEE, agreed to treat each of the two layers as separate materials. Therefore, separate material properties were measured when specimen geometries permitted separate property measurements in each of the clad layers. After application of the cladding, the plate was heat treated to relieve stress. The selected heat treatment is also commonly used for deep-water Inconel-625-cladded-steel exploration and extraction equipment.

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[3] Inconel is a registered trademark of Special Metals Corporation, Huntington, WV.
[4] This combination of materials is often used in the oil and gas exploration and extraction industry.

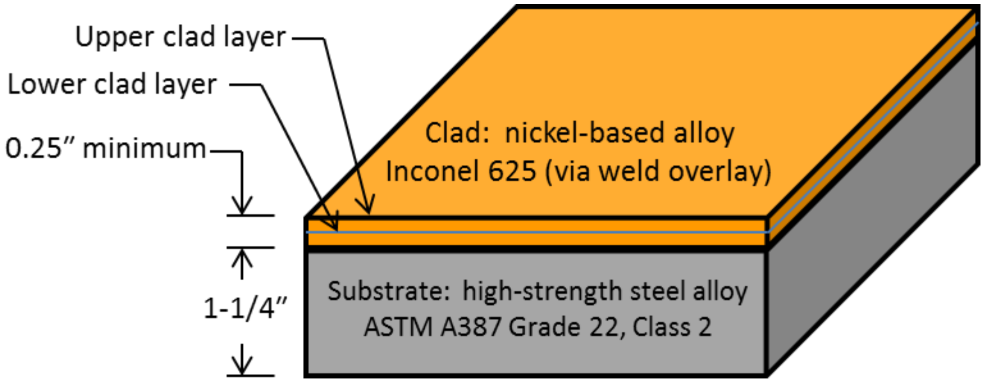



**Figure 1: Cladded test plate used in current investigation**

The chemical composition and strength of the substrate plate are listed in Table 1, while the chemical composition of Inconel 625 is shown in Table 2. However, during application of a cladding, dilution of the substrate material into the cladding occurs due to melting of the top surface of the substrate, which mixes with the melted cladding materials prior to solidification. Dilution of the iron, and to a significantly lesser extent for other elements in the steel substrate, is highest in the first clad layer to be added. The amount of dilution in each additional clad layer is successively reduced. Since the fatigue and fracture of a metallic material is dependent upon its alloy composition, each of the first several clad layers may have different fatigue and fracture properties. Therefore, as much as the test sample geometries allow, separate properties were measured for each of the clad layers.

**Table 1: Chemistry and Strength of Substrate Materials**

| High-Strength Steel Alloy | C | Mn | P | S | Si | Cr | Mo | Yield Strength (MPa) | Ultimate Tensile Strength (MPa) | Elongation (%) | Reduction of Area (%) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| ASTM A387 | 0.05–0.15 | 0.30–0.60 | 0.025 Max | 0.025 Max | 0.50 Max | 2.00–2.50 | 0.90–1.10 | 310 Min | 515–690 | 18 Min | 40 Min |
| ASTM A387 plate in present analysis* | 0.15 | 0.52 | 0.009 | 0.005 | 0.20 | 2.26 | 0.94 | 560 | 690 | 22.0 | 72.0 |

*Properties from plate certification – see Appendix A.
C = carbon; Mn = manganese; P = phosphorus; S = sulfur; Si = silicon; Cr = chromium; Mo = molybdenum; MPa = megapascal; Max = maximum; Min = minimum

**Table 2: Chemical Composition of Inconel 625 [1]**

| Element | Ni | Cr | Fe | Mo | Nb + Ta | C | Mn | Si | P | S | Al | Ti | Co |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Weight percent | 58.0 Min | 20.0–23.0 | 5.0 Max | 8.0–10.0 | 3.15–4.15 | 0.10 Max | 0.50 Max | 0.50 Max | 0.015 Max | 0.015 Max | 0.40 Max | 0.40 Max | 1.0 Max |

Nb = niobium; Ta = tantalum; Al = aluminum; Co = cobalt

In the present work, cladding was added to a 1-1/4-inch-thick ASTM A387 plate according to the requirements defined in Appendix B. The plate cladding certifications is provided in Appendix C. The required minimum cladding thickness was 0.25 inch. To achieve the needed clad thickness, two separate clad layers were required. In this case, the application direction for both clad layers was identical. Figure 1 illustrates the clad plate from which specimens were taken. With the different level of dilution in each of the two clad layers, CTC, with concurrence from BSEE, agreed to treat each of the two layers as separate materials. Therefore, separate material properties were measured when specimen geometries permitted property measurements in each of the clad layers. After application of the cladding, the plate was heat treated to relieve stress – see Appendix D for heat-treatment specifications. Appendix E is a copy of the heat-treatment certification. The selected heat treatment is also commonly used for deep-water Inconel-625-cladded-steel exploration and extraction equipment. The cladded plate as delivered to CTC is shown in Figure 2, which also shows the layout of the specimens used for property measurements. The following properties were measured: stress-strain at a slow strain rate, cyclic fatigue, fracture toughness (FT), stress corrosion cracking (SCC) and fatigue crack growth rate (FCGR). To support these measurements, engineering stress-strain of the cladding was also measured in air at 350 °F. Several of the specimens were required to calibrate the equipment and/or to establish the test range used to measure many of the properties. Given their preexisting facility and experience in completing similar tests under the desired test environment, all HPHT sour-gas testing was completed at DNV GL (DNV) in Dublin, OH.

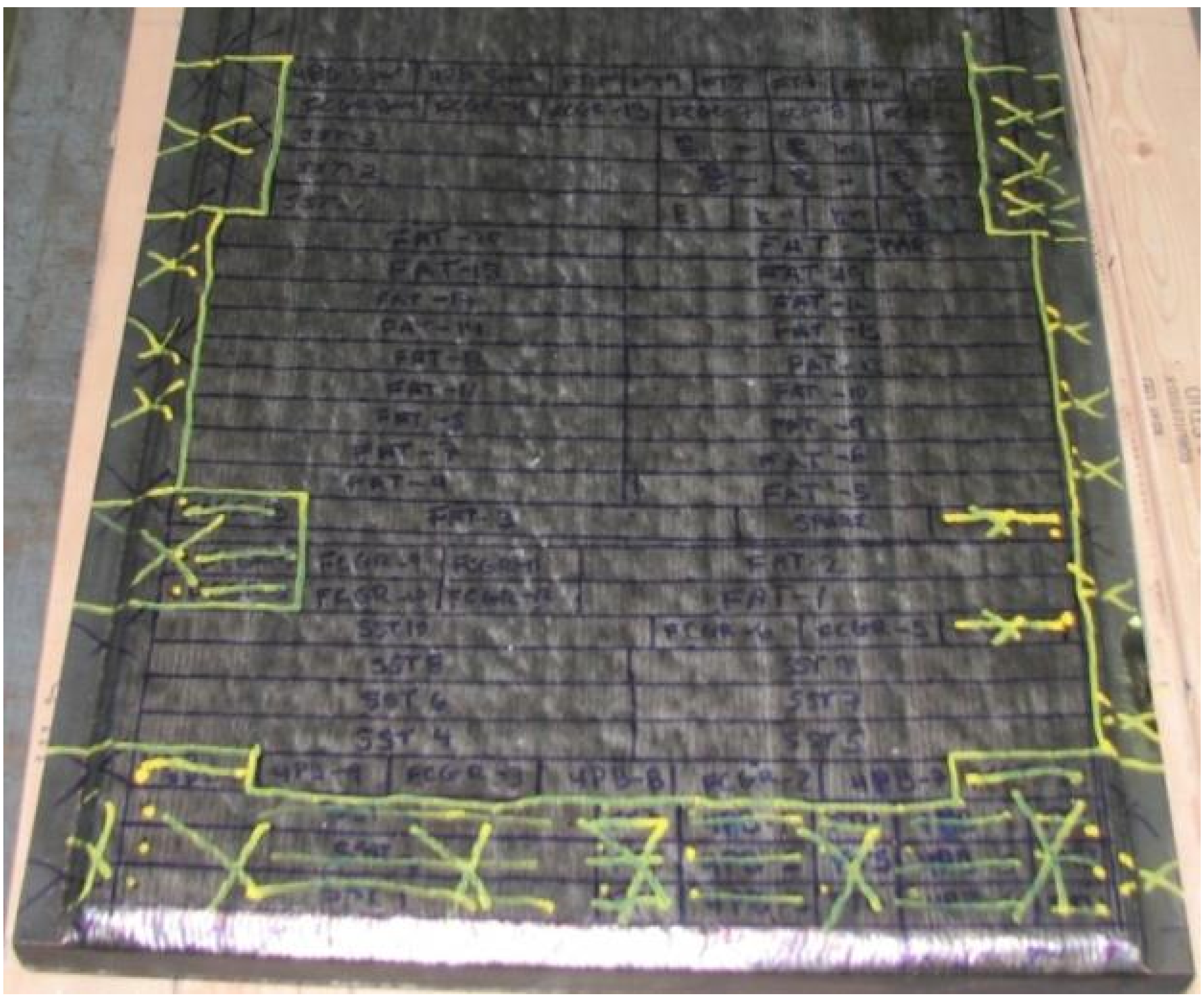


**Figure 2: Photo of Inconel-625-clad steel plate used to extract test specimens**

Table 3 shows the type and number of tests completed to determine the desired properties. The totals include calibration tests as well as tests from which fatigue and fracture data were measured. Table 3 also shows the ASTM standards associated with each of these property measurements.

**Table 3: Mechanical Property Testing Completed**

| **Mechanical Property** | **ASTM Method** | **Rationale** | **Number of Test Specimens** | **Comments** |
|---|---|---|---|---|
| Engineering Stress-Strain | E21 [4] | Determine yield and ultimate tensile stresses | 1 | Required to determine stress levels for cyclic fatigue and other testing |
| Cyclic Fatigue | E466 [5] | Generate $S$-$N_f$ curves to evaluate fatigue performance | 20 | Establish complete specimen failure by cyclic fatigue; $S$ = peak cyclic stress; $N_f$ = number of cycles to failure |
| Fracture Toughness | E1820 [6] | Determine $J_{Ic}$ fracture toughness values | 7 | Establishes dynamic fracture behavior; $J_{Ic}$ = plastic-elastic fracture toughness |
| Fatigue Crack Growth Rate | E647 [7] | Determine crack growth rates | 10 | Establishes crack growth rate resulting from loading on material with a given flaw |
| Slow Strain Rate Tensile | G129 [8] | Evaluate the effects of HPHT sour-gas environment relative to testing in air | 6 in target HPHT environment; 4 in air | Qualitatively measure rate of attack on cladding subjected to HPHT sour-gas environment |
| Stress Corrosion Cracking | G39 [9] | Qualitatively evaluate the effects of environment on crack propensity | 9 | Three replicates of each of three apparent stress levels |

Table 4 highlights several test conditions required by American National Standards Institute (ANSI) and NACE International (NACE, formerly known as the National Association of Corrosion Engineers) standards related to testing in HPHT sour-gas environments. With the acknowledgement of BSEE, testing at HPHT conditions was completed under Level VI conditions as defined in ANSI/NACE MR0175/ISO 15156 [10]; the test conditions are highlighted in Table 5. At DNV's recommendation and with BSEE's agreement, the potential hydrogen (pH) of the sour gas condition was 4–5; therefore, the aim pH was 4.5.

**Table 4: Sour Test Environment/Corrosion Specifications**

| ANSI/NACE Method | Title | Rationale for Use |
|---|---|---|
| ANSI/NACE MR0175/ISO 15156 | Petroleum and Natural Gas Industries-Materials for Use in $H_2S$ Containing Environments in Oil and Gas Production; Part 1 General Principles for Selection of Cracking-Resistant Materials | Used in conjunction with the standard ASTM test methods as guidelines for testing in a sour ($H_2S$) environment under HPHT conditions |
| ANSI/NACE TM0284 [11] | Evaluation of Pipeline and Pressure Vessel Steels for Resistance to Hydrogen-Induced Cracking | |
| ANSI/NACE TM0177 [12] | Laboratory Testing of Metals for Resistance to Sulfide Stress Cracking and Stress Corrosion Cracking in $H_2S$ Environments | |

**Table 5: Physical Experimental Environmental Conditions**

| Condition | Value |
|---|---|
| Temperature (°F/°C) | 350/177 |
| Pressure (psi/bar) | 1150/78 |
| Hydrogen Sulfide, $H_2S$ (partial pressure, psi) | 500 |
| Chloride, $Cl^-$ (mg/l [minimum]) | 150,000 |
| Carbon Dioxide, $CO_2$ (partial pressure, psi) | 500 |
| pH | 4–5 |

## 2.0 TEST SOLUTION

Using version 8 of the mixed solvent electrolyte (MSE) model [13] available from OLI Systems, Inc. of Cedar Knolls, NJ, DNV determined the equivalent thermodynamic corrosion potential at the test pressure (1150 psia) to that of the desired oil and gas exploration and extraction pressure (i.e., the service pressure, 15 ksi). This calculation was performed to identify the sour-gas solution chemistry. Table 6 shows the resulting chemistry.

**Table 6: Solution Chemistry of the Test Environment for Equivalent Sour-Gas Service Conditions**

| Chemical Compound | Chemical Formula | Mass per Unit Volume (g/l) | Mass Fraction (%) |
|---|---|---|---|
| Deionized Water | $H_2O$ | 915.10 | 78.69 |
| Sodium Chloride | NaCl | 247.30 | 21.27 |
| Sodium Bicarbonate | $NaHCO_3$ | 0.5065 | 0.0436 |

## 3.0 LITERATURE REVIEW

Offshore oil and gas exploration and production has increased in recent years. A significant number of the recently found fields have been in deep water and the wells operate under high pressure and high temperature. The increased water depths combined with extreme HPHT conditions present a significant challenge for various materials. For example, the increased water depth places a significant demand on the strength of the material. The use of conventional material for subsea applications with yield strength (YS) in the range of 70–100 ksi makes it impractical to transport and install the equipment in deep-water applications. This has led the industry to explore high-strength steels for these applications. A range of steels can be heat treated to obtain strengths in the range of 120–160 ksi. However, most of these materials have poor resistance to hydrogen embrittlement. It is also expected that the internal environment of the subsea equipment will experience temperatures in excess of 350 °F, with partial pressures of $H_2S$ in the range of 1–2 psia coupled with low pH. These environments are extremely aggressive and typically lead to high corrosion rates of steels and potentially also lead to cracking due to hydrogen embrittlement. This has led to the development of clad systems, where low-alloy steels are cladded with high-nickel, corrosion-resistant alloys (CRAs) such as Inconel 625, which allows for a cost-effective solution by using low-alloy steels to be protected by CRAs. However, only a limited amount of work has been performed to understand the environmentally assisted cracking behavior of clad materials in sour-gas environments under HPHT conditions.

SCC resistance has been investigated primarily by performing static SCC tests such as four-point bend and C-rings in order to determine the long-term performance of CRAs in specific environments of interest. There has also been development of the NACE TM0298 [14] SSR test method, which involved dynamic straining of CRAs to determine their SCC resistance. However, these techniques are designed to provide pass/fail criteria with no quantitative information available from these tests for use in design. While these tests have been successful in evaluating martensitic (e.g., 13 chromium [Cr]) type of material, the use of static techniques has been less successful in evaluating nickel-based alloys. An example of this was evident in the recent work [15] on precipitation hardened (PH) nickel alloys on creviced four-point bend specimens, which showed a range of scatter on the occurrence of cracking after different exposure times. It is clear that in some cases tests in 30/90-day exposures showed evidence of cracking, but longer-term exposures of 183 and 365 days did not exhibit any evidence of cracking under the same environmental conditions tested. The specimens that were not creviced, did not exhibit any evidence of cracking, while the creviced specimens in certain cases did exhibit cracking. Cross sectional analysis of the specimens suggested that the cracks initiated from the creviced area.

The above observations suggest that cracking in CRAs is related to localized corrosion initiation/growth. Crack growth rate measurements on 13Cr material has suggested similar behavior where the crack growth rate (CGR) is a strong function of applied potential, and increases sharply as the applied potential is above the repassivation potential [16].

Localized corrosion sites like pits/crevices can act as initiation sites for subsequent cracking. The pits/crevices cannot only act as stress concentrators but also serve to provide the local electrochemical conditions for sustaining/transitioning to cracks. The local environments at the bottom of pits/crevices are acidic due to metal ion hydrolysis, and concentrated in chloride to maintain charge neutrality. This would suggest that the micro processes that control pit growth and crack growth are similar. A schematic illustration of the pit growth process and crack growth process and the relationship between them is shown in Figure 3.

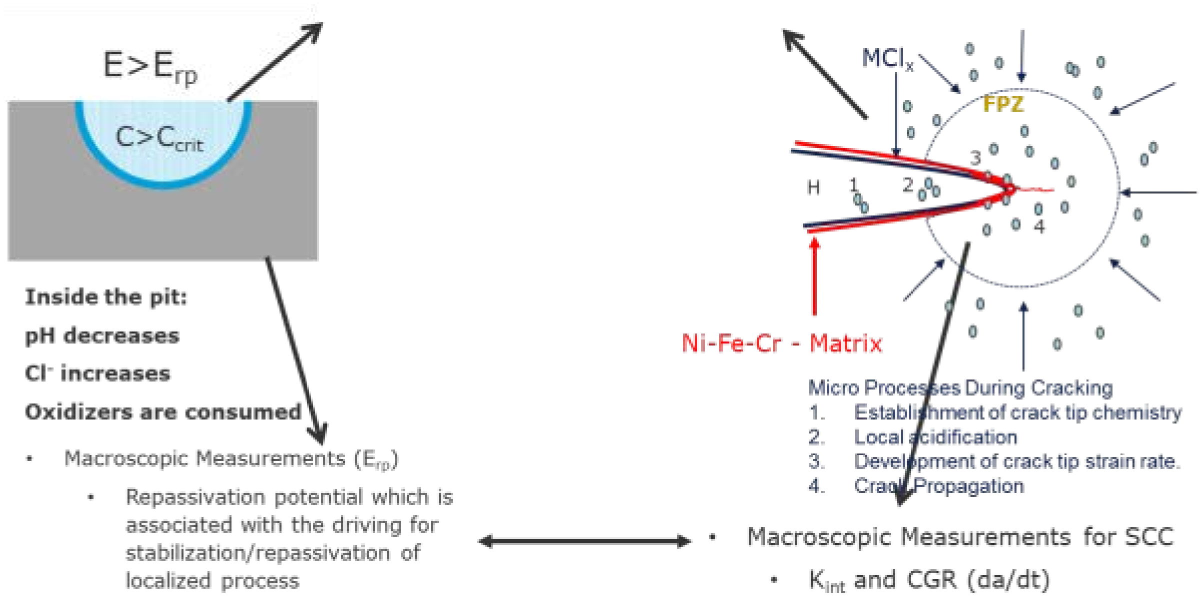



**Figure 3: Schematic illustration of the relationship between the micro processes for localized corrosion and SCC in CRAs**

There has been limited work performed in the area of fatigue and fracture applications in HPHT sour-gas environments to understand the behavior of nickel-based alloys. Fracture toughness tests on Inconel 625+ (a participation hardened version of Inconel 625) in sour-gas environments in a range of sour environments are shown in Figure 4 [17]. The results indicate that increasing chloride concentration leads to decreasing initiation toughness ($K_J$) and increasing CGR. The $K_J$ and CGR are related to the difference between repassivation potential and corrosion potential, as the difference in repassivation and corrosion potential increases as $K_J$ increases and CGR decreases.

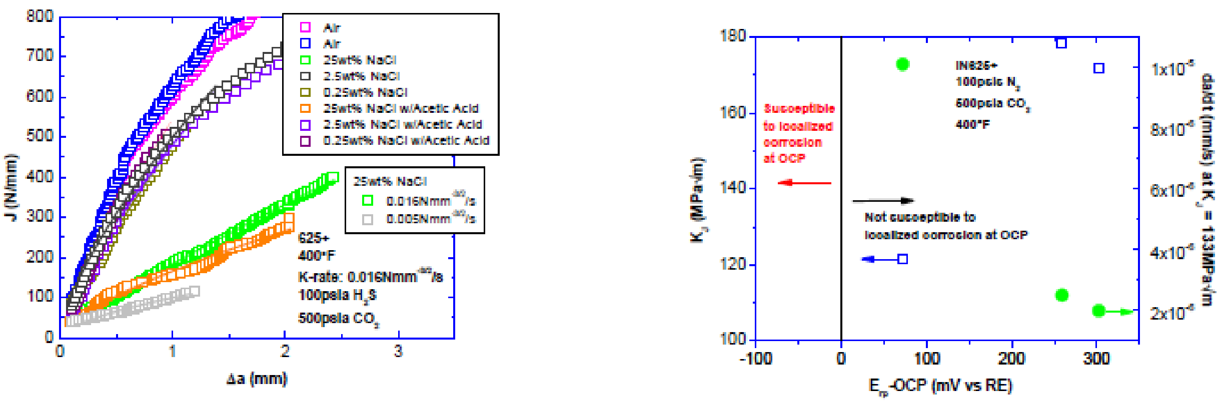



**Figure 4: Fracture toughness curves in a range of sour environments at 400 °F, and relationship between initiation toughness and crack growth rate to the repassivation potential [17]**

The above data suggests that developing a correlation between environmental cracking parameters and localized corrosion data will enable development of an analytical framework with which predictive models can be developed to address not only a range of environmental conditions but also a range of material chemistry (e.g., Inconel 625 vs Inconel 825). It is also important to note that the microstructure of the clad components will be a welded/as-cast microstructure, which could have a significant impact on the environmental fatigue and fracture resistance in sour-gas environments and needs to be captured in order to validate the use of cladding as a viable option for HPHT sour-gas conditions.

Further complicating the prediction of fatigue and/or fracture performance of cladded structures is the microstructure of the multi-material components, especially near the interface between the substrate metal and cladding. The microstructure (including the atomic arrangement of the material and the spatial distribution of alloying elements) plays a significant role in the local strength, fatigue and fracture properties of the cladding. It also has a significant role in crack initiation and propagation, as well as corrosion rate. In addition, chemical dilution of substrate material into the fusion weld clad layers is important, especially given that the amount of dilution varies significantly over the first several clad layers. As an example, on another project, CTC had multiple layers of nickel-based alloy Inconel 625 clad onto a high-strength steel substrate [18]. Not until the third clad layer did the dilution effects of the substrate essentially vanish – see Figure 5.[5] Furthermore, the strength, fatigue and fracture properties of the individual clad layers are expected to vary as a result of the significant differences in dilution between each clad layer. Given the higher dilution of iron into the bottom clad layer, it is likely to have higher corrosion and stress-corrosion rates than the less-iron-diluted outer clad layer of the two-layer cladded specimen made for experimentally measuring strength, fatigue and fracture properties in this report. To correctly define the fatigue and fracture behavior of multi-layer cladding systems, this difference must be quantified, thereby requiring separate property measurements in each of the clad layers.

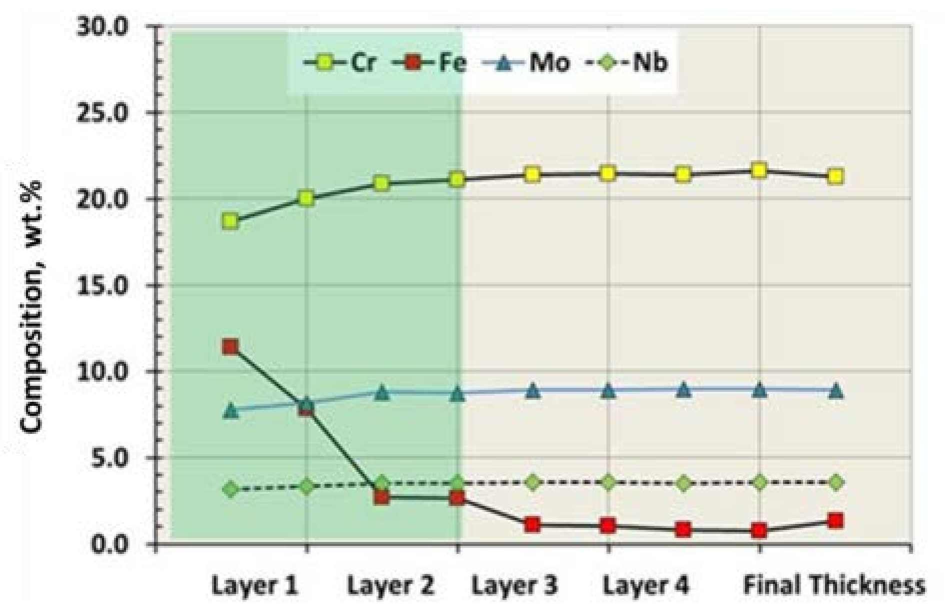



**Figure 5: Dilution of elements from steel substrate to Inconel 625 weld cladding [18]**

[5] The amount of and rate of change of dilution effects across the clad layers is dependent upon many factors including the power level, torch speed and power source. The dilution effects discussed in Reference 18 represent a common, but not comprehensive dilution distribution in cladded structures.

### 3.1 Application of Fracture Mechanics Approaches

There is a growing trend in the industry to use a fracture-mechanics-based approach for design as opposed to a conventional stress-based design approach. A fracture-mechanics-based design approach can optimize the design process and take advantage of the improved properties of advanced materials like clad nickel-based alloys. It is essential to have crack growth and fracture toughness data of the nickel-based alloys for this new design approach.

There is a need to develop data on the material performance in the context of the new fracture-mechanics-design-basis in sour-gas environments. While there is a need to develop data to address environmental effects in HPHT sour-gas conditions, there is also a need to clearly identify how fracture toughness and FCGR data will be applied to clad systems. It is currently unclear if standard failure assessment diagram (FAD)-based fracture mechanics assessment methods, such as those found in BS 7910 [19] and API/ASME 579 [20], are suitable for HPHT sour-gas subsea applications, or if finite element (FE) -based methods (i.e., crack mesh) need to be developed.

## 4.0 PROJECT PARTICIPANTS

The project was funded and overseen by BSEE, who offered excellent technical direction and accountability. Based upon an open solicitation, DNV of Dublin, OH was selected as the test vendor to complete tests under HPHT sour-gas conditions. CTC machined the test specimens. DNV and CTC completed selected metallurgical analyses. CTC completed development of material models and a database of results.

## 5.0 PRE-TESTED METALLURGICAL ANALYSIS

CTC completed several metallurgical analyses of pre-tested Inconel 625 clad on the ASTM A387 steel substrate. Metallographic prepared samples taken from the cladded steel were analyzed using light optical microscopy (LOM) and scanning electron microscopy coupled with energy dispersion spectroscopy (SEM-EDS). SEM-EDS microscopy was primarily used to evaluate the effects of iron dilution throughout the Inconel 625 clad layers. The analyses demonstrated (as shown in Figures 6 through 8, which were obtained from scanning electron microscopy [SEM]):

- A good bond existed between the cladding and the steel substrate.
- Epitaxial crystal growth of the weld layers was observed.
- Neither coarse porosity, inclusions nor cracks were observed in the Inconel 625 cladding.
- Relatively coarse and directionally aligned dendrites were observed at the steel/overlay weld and overlay/overlay welds interfaces.
- Most of the dendritic microstructure away from the interface had a small arm spacing.
- The microstructural characteristics of the outer clad layer were found to be very similar to those of the inner clad layer: primary coarse dendritic columnar crystals that grew from the interface of preexisting material and much finer dendrites above the coarser ones.
- Microsegregation was more pronounced at the coarser dendrites; the interdendritic segregation was presumably very fine delta and Laves phases common to nickel-based alloys.

- Iron dilution fades as the distance from the steel interface increases; presumably iron was carried away from the interface by convection currents produced by the welding process.
- The elemental map showed high concentration of niobium (Nb) and molybdenum (Mo) at the interface with the steel substrate, which indicates there is more microsegregation towards this region of the weld; this also indicates that the presence of Laves phases is more prominent in this region of the weld; chromium (Cr) is uniformly distributed in the microstructure.
- The iron, as determined by SEM-EDS elemental iron analysis, content decreases from approximately 26 weight percent (wt%) at the steel interface to approximately 8.5 wt%, at approximately 2500 microns (0.0985 inch) into the first weld layer – see Figure 9. The iron content practically remained constant into the second layer, but decreased towards the interface with the additively manufactured material. The additively manufactured material layer is discussed below.

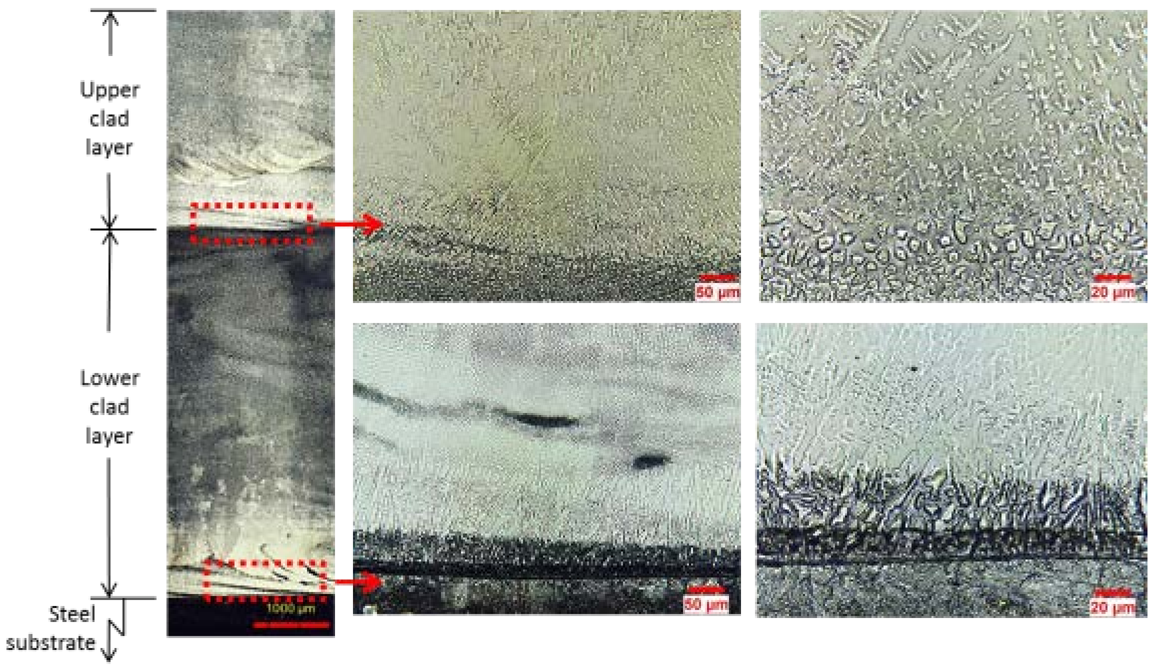



**Figure 6: Typical LOM microstructure at interface of clad layers**

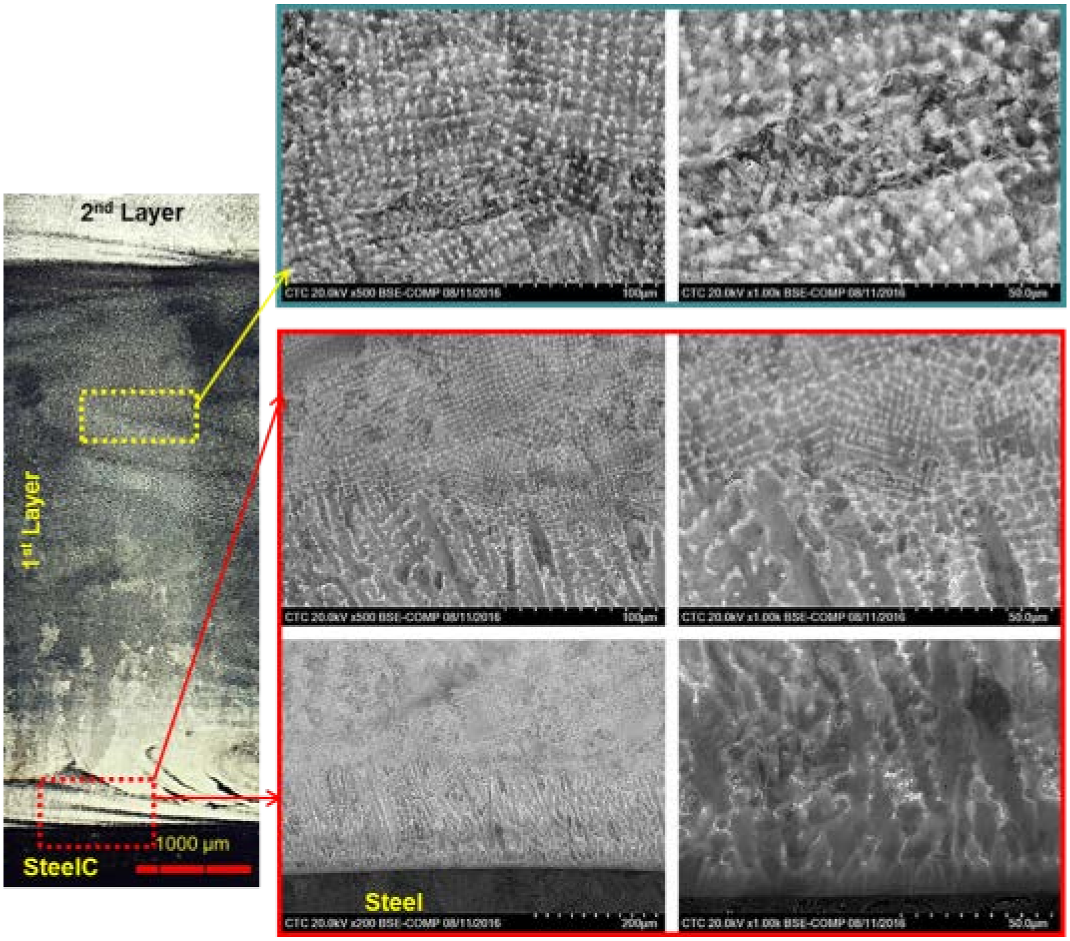



**Figure 7: Typical SEM microstructure of the lower clad layer**

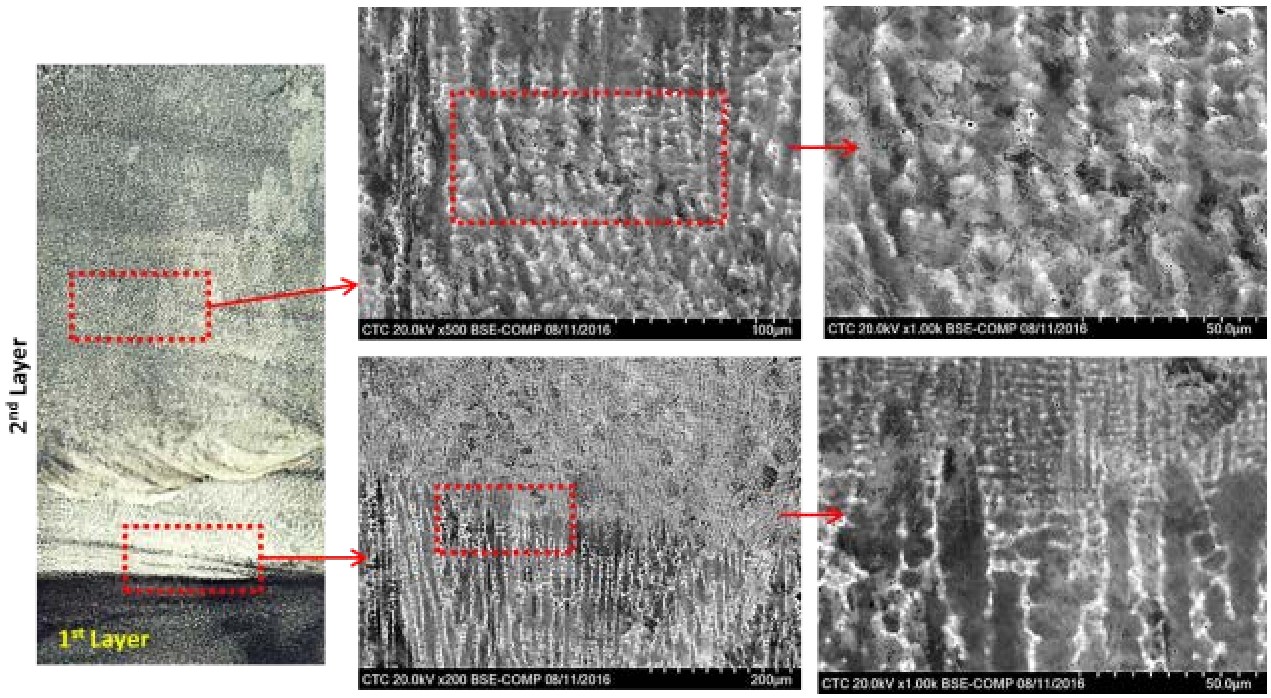



**Figure 8: Typical SEM microstructure of the upper clad layer**

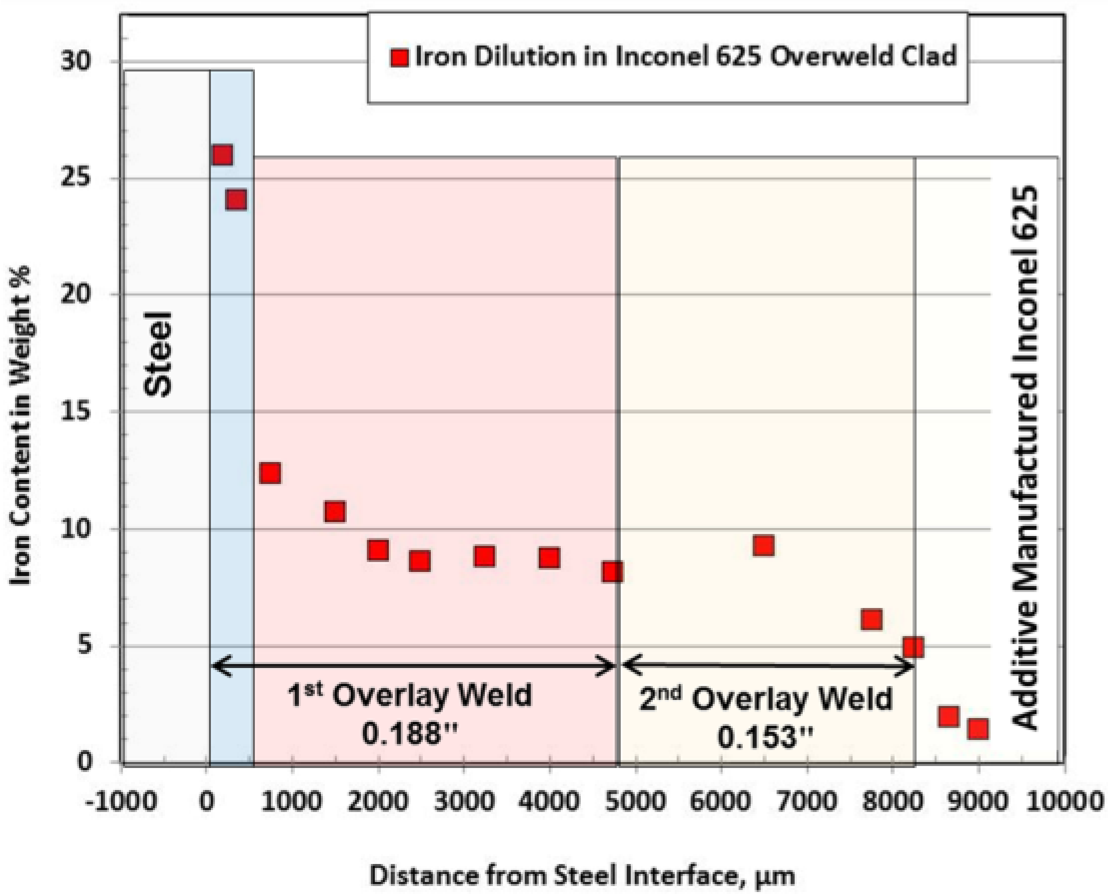



**Figure 9: Iron dilution in the Inconel 625 cladding**

# 6.0 EXPLANATION OF TESTING PERFORMED

## 6.1 Engineering Stress-Strain Tensile Testing

Stress levels used in many of the subsequent fatigue and fracture tests relied on measured yield strength (YS) and ultimate tensile strength (UTS) at 350 °F. Accordingly, one specimen conforming to ASTM E21 [4], as depicted in Figure 10 was tested in air. The specimen was taken so that its axis was perpendicular to the direction of individual clad passes (i.e., longitudinally). This orientation ensured the specimen would include the mixed microstructure associated with several weld beads. This orientation is typically the weakest direction in a weldment, including weld cladding. For these specimens, material in the gage length was a mixture of both upper and lower clad layers. The center gage area consisted of clad material, while substrate material was permitted in the threaded section, if needed to achieve a complete specimen.

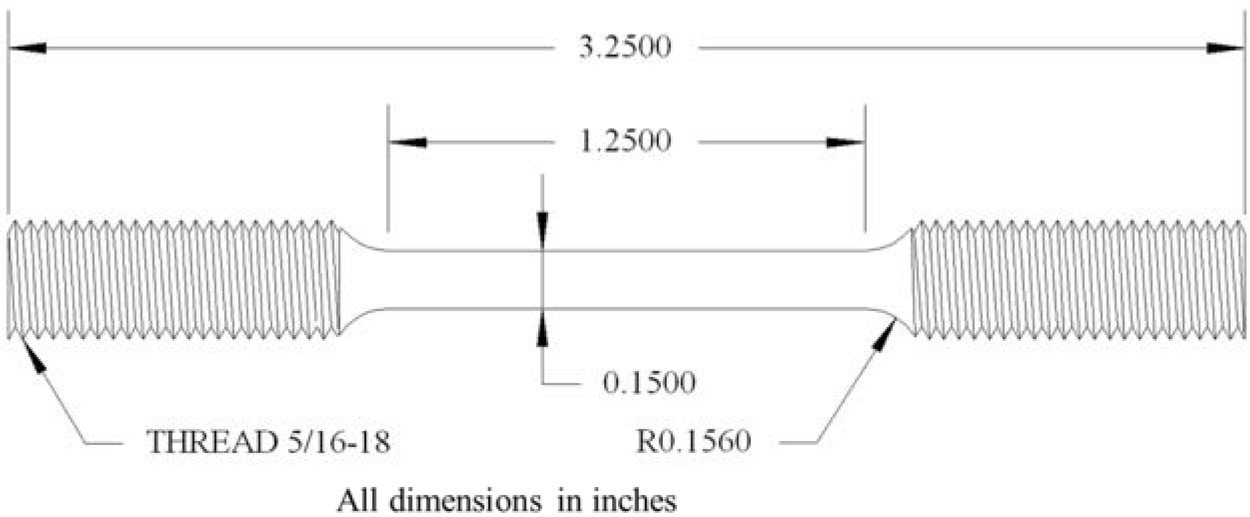



**Figure 10: Engineering stress-strain specimen geometry**

## 6.2 Slow-Strain-Rate Tensile Testing

SCC susceptibility performance of the material was evaluated using two methodologies: slow-strain-rate tensile testing and bent-beam SCC, as described below. The SCC susceptibility performance of the material was evaluated in accordance with ASTM G129 [8]. Slow-strain-rate (SSR) tensile testing is a standard material test method in which specimens are subjected to elongation at a constant engineering strain rate of $4\times10^{-6}$/sec. The load was varied to maintain the constant engineering strain rate. This test qualitatively gauges the effects of local environmental conditions on SCC behavior, material fracture susceptibility or SCC susceptibility. Standard tensile type specimens per ASTM G129 (round 0.150-inch diameter, 1.0-inch gage length, Figure 11) were utilized for testing and sectioned from the cladded plate as illustrated in Figure 12. Specimens only included clad metal. The gage length of the specimens included material from both clad layers. Testing was performed at both ambient conditions and the temperature, pressure and environment defined in Table 5. The change in tensile properties between in-air and tests under HPHT sour-gas environment was used to qualitatively determine environmental effects. Table 7 shows the test conditions under which slow-strain-rate tests were completed.

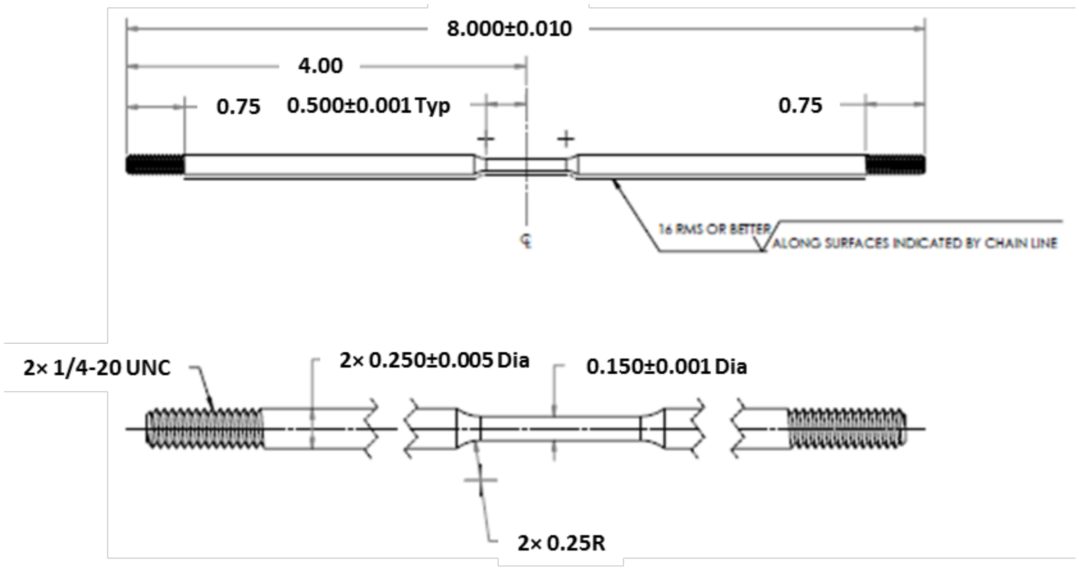



**Figure 11: Drawing of slow-strain-rate test specimen**

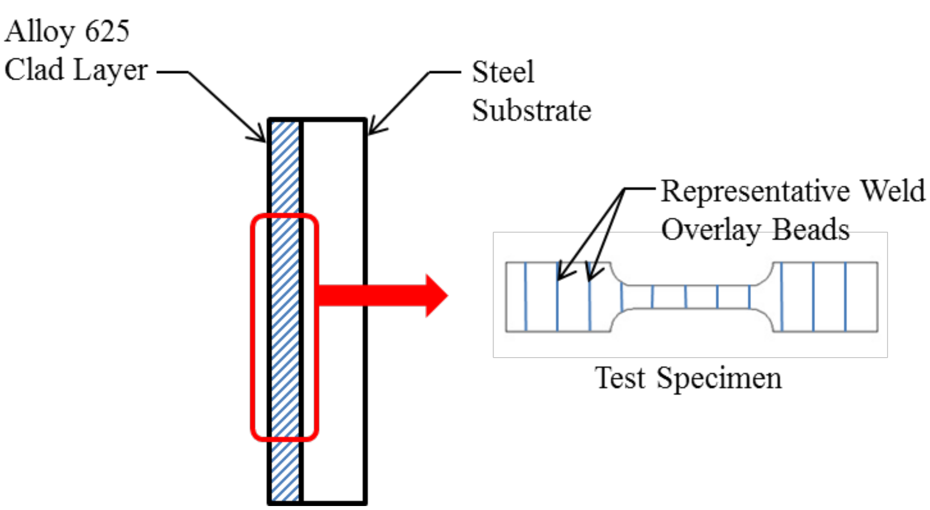



**Figure 12: Slow-strain-rate test specimen**

**Table 7: Test Details for SSR Tensile Tests**

| Test Conditions | Number of Specimens |
|---|---|
| Air environment; room temperature | 2 |
| Air environment; 350 °F | 2 |
| Sour-gas environment; room temperature; high pressure | 3 |
| Sour-gas environment; 350 °F; high pressure | 3 |

### 6.3 Bent-Beam Stress Corrosion Cracking (SCC) Testing

Additionally, a semi-qualitative evaluation of the cladding's ability to resist SCC was evaluated using a bent-beam test specimen as illustrated in Figure 13. The SCC susceptibility performance of the material was evaluated in accordance with ASTM G39 [9]. Specimens were ground to a surface roughness not exceeding 30 µin Ra. Standard four-point, all-clad metal bend specimens, 2 inches × 0.400 inch × 0.125 inch were tested concurrently using stress values based on the slow-strain-rate test results discussed above. Stress levels were based on a percentage of the apparent yield load during bending in the test clamp. All SCC tests were completed in HPHT sour-gas conditions defined in Table 5. The peak load applied to the specimens was 95%, 110% or 120% of apparent yield load. Three replicates were tested under each of the applied load values; specimens were held for 30 days in the HPHT environment.

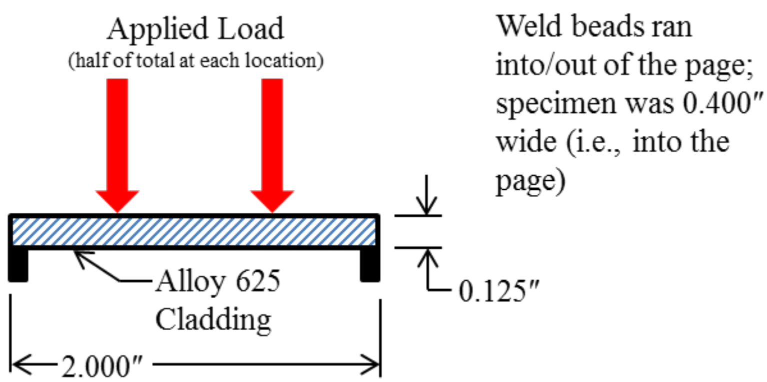
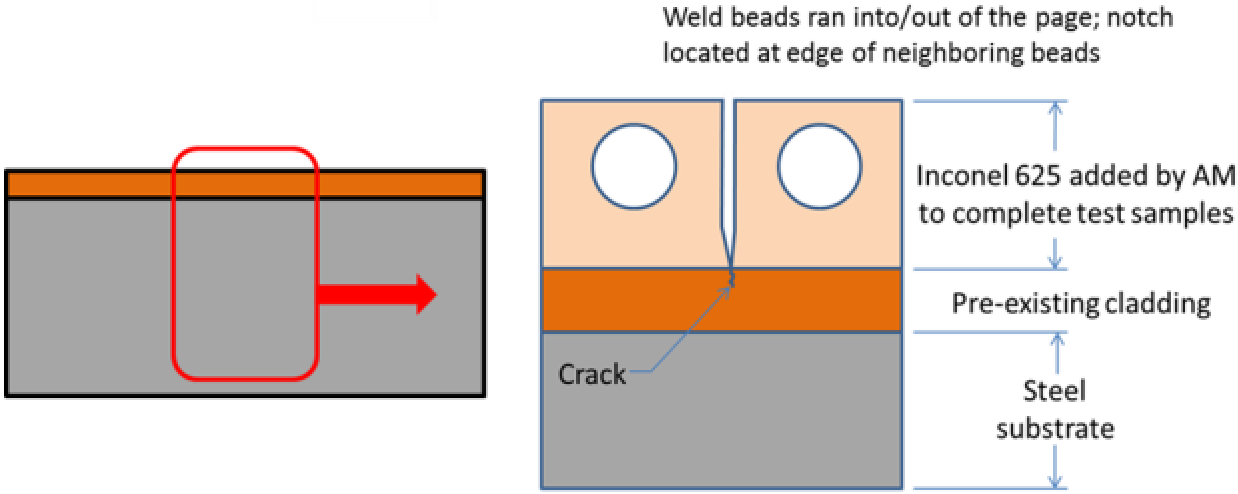



**Figure 13: Four-point bent-beam SCC test specimen**

### 6.4 Fracture Toughness

The fracture toughness (FT) performance of the material was evaluated in accordance with ASTM E1820 [6]. Fracture toughness describes the ability of a material containing a crack to continue to absorb and dissipate energy by crack growth, but resist fracture. In this case, the plastic-elastic fracture toughness, denoted by $J_{Ic}$, was measured and represents the energy required to grow a thin crack. Fracture toughness is a quantitative way of conveying a material's resistance to fracture when a crack is present. If a material has high fracture toughness, it will probably undergo ductile fracture. Standard compact tension specimens, C(T) [specimen thickness, $B$ = 0.5 inch; specimen width, $W$ = 1.0 inch], were utilized for testing. Test specimens were sectioned from the cladded plate as illustrated in Figure 14. Typically, the specimens for fracture toughness are removed from the parent material such that the entire specimen is of the parent material. However, two factors did not allow single-material specimens to be used for testing of the cladding. First, the two-layer cladding, where the fracture toughness was measured, was only 0.25 inch thick. The crack, illustrated at the base of the machined notch in Figure 14, must remain in the desired clad layer to test material in that layer. Steel from the substrate was needed to complete the physical test specimens below the substrate. Secondly, no material was initially available to complete the physical test specimens above the crack, i.e., the portion of the specimen that includes the pin loading holes used to apply the load. Inconel 625 was added to the top of the cladding by additive manufacturing (AM) using an SLM 280$^{HL}$ laser-powder bed fusion AM machine at CTC. After adding the material, the resulting multi-material specimen was machined to dimensions consistent with ASTM E1820 as indicated in Figure 15. During testing in the HPHT sour-gas environment, the steel substrate was protected from reacting with the environment using a proprietary method developed by DNV.




(a) Cladded Steel Test Blank (b) Completed Test Specimen

**Figure 14: Fracture toughness and FCGR test specimens**

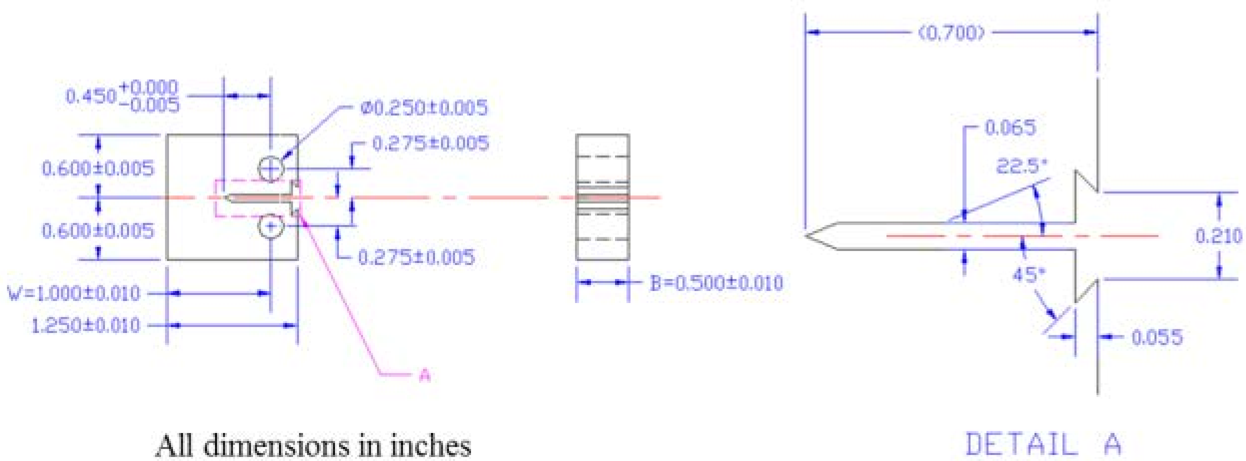



**Figure 15: Fracture toughness test specimen drawing**

The weakest material was assumed to lie at the boundaries between the individual clad passes. Therefore, specimens were machined, ground and slightly macro-etched to reveal the boundaries of the clad passes. This allowed the machinist to accurately align the specimen notches with the edge of a clad pass as illustrated in Figure 16. Fracture toughness testing was performed either at 350 °F in air (to calibrate the test method) or at the temperature, pressure and environment defined in Table 5. Table 8 lists the final test conditions tested.

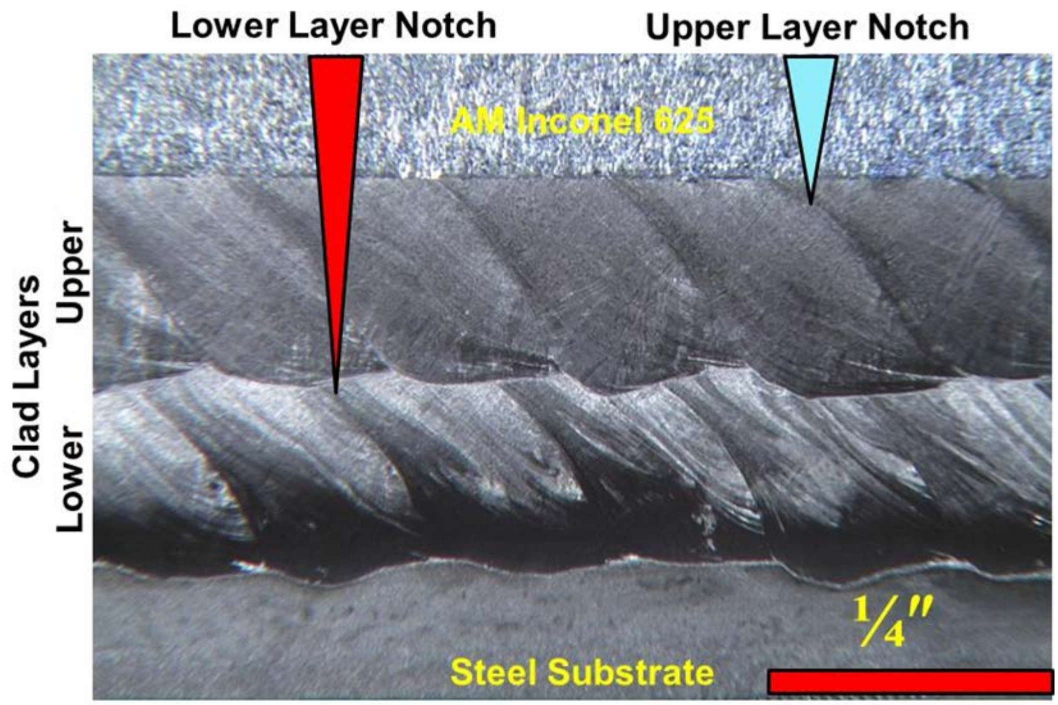



**Figure 16: Schematic representation of notch locations for FT and FCGR specimens**

**Table 8: Details of Fracture Toughness Testing**

| Test Method | Notch Location (Layer) | $K$-Rate* ($N \cdot mm^{-3/2}$/sec) | Number of Tests | Notes |
|---|---|---|---|---|
| Direct Current Potential Drop (Calibration) | Lower | N/A | 1 | Tested at 350 °F in air |
| | Upper | | 1 | |
| Slow Rising Displacement | Upper | 0.085 | 1 | Tested at HPHT sour-gas conditions |
| | Upper | 0.016 | 1 | |
| | Upper | 0.0037 | 1 | |
| Slow Rising Displacement | Lower | 0.005 | 1 | |
| | Upper | 0.005 | 1 | |

*Values were selected after completion of first round of slow-rising displacement tests. *K*-rate values were the highest (among 0.085, 0.016 and 0.0037 $N \cdot mm^{-3/2}$/sec) leading to consistent behavior with lower *K*-rate values. Notch locations for the last two slow-rising displacement tests were used to supplement those from the first set of three slow-rising displacement tests. They were tested at the layer having the lowest fracture toughness from the initial slow-rising displacement tests.
*K*-rate is the time rate of change of applying the stress intensity factor.

In order to characterize the entire *J*-*R*-curve (i.e., the curve of crack growth resistance, *R*, relative to *J*, the material's fracture toughness) it was important to be able to measure the crack length in-situ using direct current potential drop (DCPD). However, in order to accurately characterize the crack length using this method in a multilayer system as illustrated in Figure 14, it was essential that a calibration curve be developed prior to making fracture toughness measurements. The calibration curve was developed using two tests in-air at 350 °F, one in each of the clad layers to develop a co-relation between the potential drop signals and the crack length (*a*). The pre-cracks for these calibration tests were located at a crack length to specimen width ratio (*a*/*W*) of 0.5, which was similar to the *a*/*W* value of 0.5 proposed for the environmental tests.

Fatigue and fracture toughness measurements were performed on servo electric frames, and the crack growth was measured using the DCPD technique. A constant current of 4.0 amps was applied across the crack mouth and the voltage drop across the crack mouth was measured using a high resolution digital multimeter (DMM). Platinum wires of 40-mil diameter were used for voltage and current probes. The platinum wires were heat shrunk in polytetrafluoroethylene (PTFE) to prevent contact with the cell and the solution. The spot weld locations of the probes on the samples were protected with a coating from Epoxy Systems™ Product 641 to prevent corrosion around the probes. The measured voltage drop was converted into crack length using the Johnson equation [21]. The crack mouth opening displacement (CMOD) measurements were performed using a load line correction.

## 6.5 Fatigue Testing

Fatigue is defined as the weakening of the test material caused by cyclically applied stress, typically below the yield strength of the test material. However, to achieve the desired number of cycles to failure, many fatigue tests were completed with a peak cyclic stress above the yield strength, but below the UTS, of the cladding.

The fatigue performance of the cladded material was characterized by $S$-$N_f$ ($S$ = peak cyclic stress; $N_f$ = number of cycles to failure) fatigue curves. In high-cycle fatigue conditions, material performance is commonly characterized by an $S$-$N_f$ curve. Given the desire of BSEE to include low-cycle fatigue (of the order of several hundred to a few thousand cycles), and with the overwhelming stress-based fatigue design criterion used by the oil and gas industry (as opposed to strain-related design criterion) [3], all fatigue testing was completed in stress-controlled conditions. Therefore, significant scatter in the fatigue data was anticipated for the cladding, which is a welded material. Much of the anticipated scatter is due to the mixed microstructures common to welded material and discussed in Section 3.0 Pre-Tested Metallurgical Analysis.

Fatigue results are typically graphed as the logarithm (log) of cyclic stress against the logarithm of cycles to failure. Sinusoidal stress loading was applied during fatigue testing. Testing was performed in accordance with ASTM E466 [5] using an axially loaded test specimen in stress control at a test frequency ($f$) not greater than 0.3 Hz.[6] Standard axial fatigue coupons (round 0.150-inch diameter, 1.0-inch gage length, Figure 17) were utilized for testing and were excised from the cladded plate as illustrated in Figure 18.

[6] The maximum frequency of 0.3 Hz is a specified condition defined in American Petroleum Institute (API) 17TR8 [22].

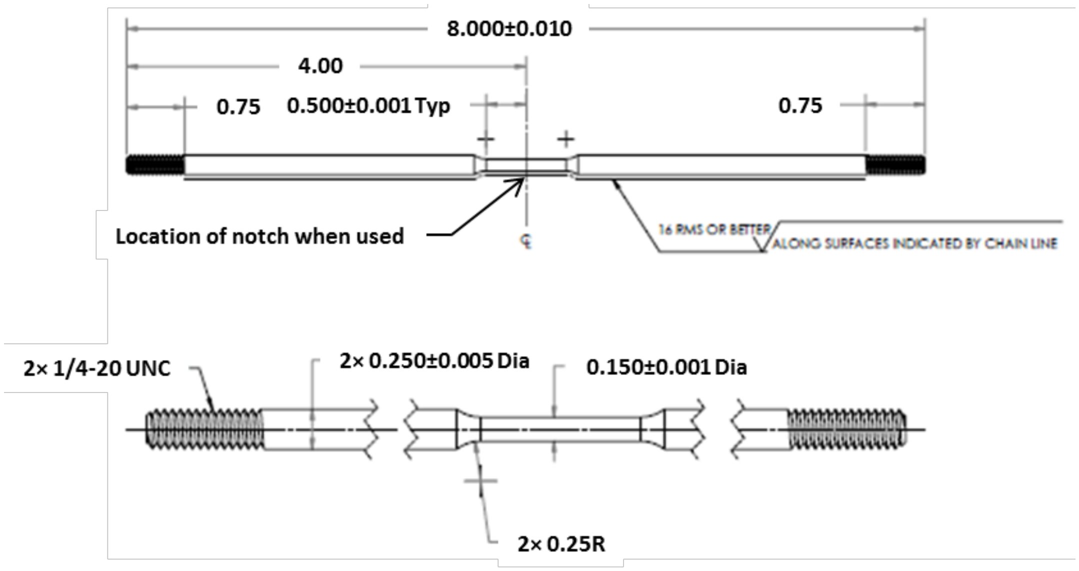



**Figure 17: Drawing of axial fatigue test specimens**

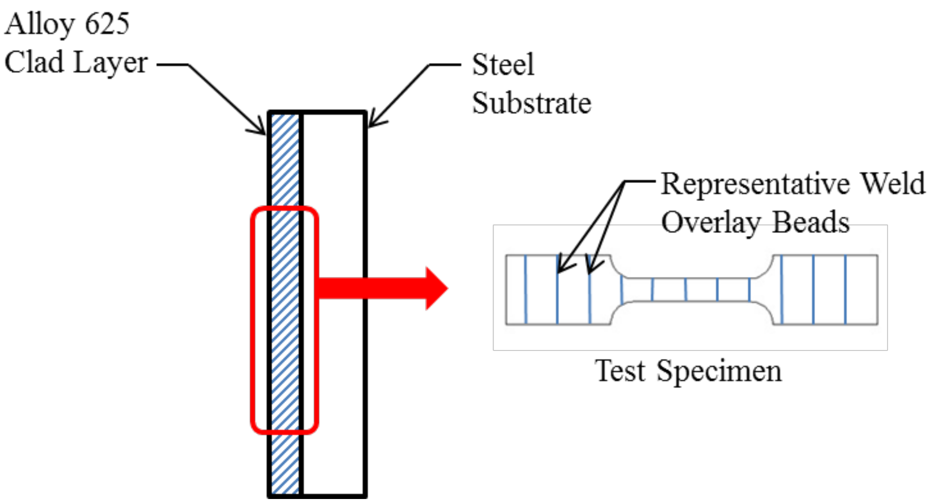



**Figure 18: Axial fatigue test specimen of clad material**

Testing was performed at the temperature, pressure and environment defined in Table 5. Cyclic loading was completed at a stress ratio ($R$, minimum stress [$\sigma_{min}$] divided by the maximum stress [$\sigma_{max}$]) of 0.13. A single stress ratio was selected due to the limited number of fatigue specimens tested and the wide scatter expected in the results.[7] As a result of achieving significantly greater number of cycles to failure in early fatigue tests, the subsequent specimens were notched via machining on a lathe. The associated stress concentration factor ($K_t$) for this notch design was originally 3.0, but to increase the local stress concentration and thereby reduce the number of cycles to failure, it was changed to a geometry yielding a $K_t$ value of 4.0 for the remaining specimens. Figure 19 shows the dimensions of the notch, which was centered along the length of the gage area as noted in Figure 17. Therefore, for any nominal stress (defined as the tensile load divided by the cross-sectional area of the specimen at the base of the notch) greater than 25% of

[7] More measurements under a given stress ratio increases the reliability of the resulting data trends and the associated mathematical material models.

yield strength, the material at the base of the notch was stressed beyond the yield point of the pre-tested material.

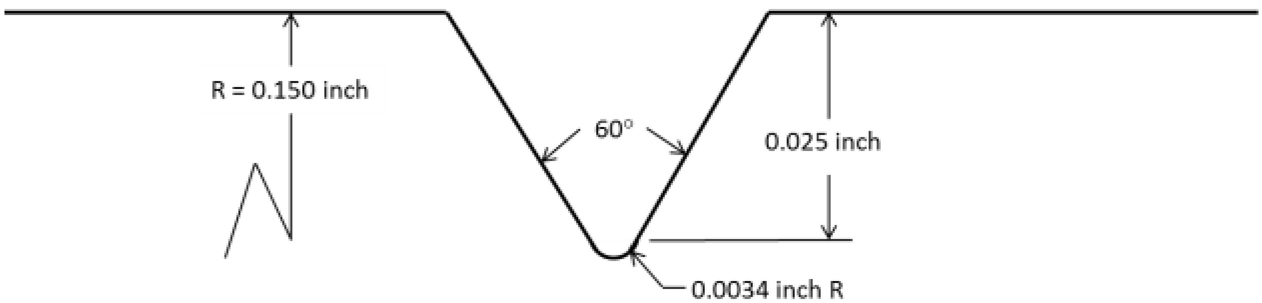



**Figure 19: Fatigue specimen notch resulting in a notch stress concentration factor of 4.0**

The actual stress levels of the specimens in the HPHT sour-gas environment evolved as the fatigue data were being generated. This evolution was aided through low-cost (relative to testing in HPHT sour-gas conditions) testing in air at 2 Hz. From those early in-air fatigue tests, it became clear that notched specimens were required to meet the desired cycle count of several hundreds to a few thousands. To achieve the desired cycle count, it became apparent that nominal stresses approaching the yield strength (YS) of the Inconel 625 cladding were needed. These first several specimens tested at HPHT sour-gas conditions, however, did not fail until after several tens of thousands or even several hundred thousand cycles. Therefore, the peak cyclic stress for subsequent specimens was increased. Eventually, fatigue tests were conducted with peak stresses above YS, which is not unprecedented for Inconel 625 [23, 24]. The peak stress condition applied during successive fatigue tests continued to increase until the total number of desired fatigue tests was completed. The resulting test conditions shown in Table 9 were thereby established.

**Table 9: Fatigue Test Plan**

| Environment | Temperature (˚F) | Specimen ID | Notched | *R* | $\sigma_{max}$ (ksi) | *f* (Hz) |
|---|---|---|---|---|---|---|
| Air | 350 | FAT-30 | Y | 0.13 | 60 | 2 |
| | | FAT-9 | N | 0.13 | 60 | |
| | | FAT-32 | N/Y | 0.13 | 60 | |
| | | FAT-35 | Y | 0.13 | 60 | |
| | | FAT-13 | Y | 0.13 | 52 | |
| | | | | 0.13 | 52 | |
| | | | | 0.13 | 52 | |
| | | FAT-16 | Y | 0.13 | 63.7 | |
| | | | | 0.13 | 63.7 | |
| | | | | 0.13 | 63.7 | |
| | | FAT-3 | Y | 0.30 | 60 | |
| | | FAT-33 | Y | 0.30 | 60 | |
| | | FAT-31 | Y | 0.75 | 60 | |
| HPHT Sour-Gas 500-psia $H_2S$ 500-psia $CO_2$ | 350 | FAT-7 | Y | 0.13 | 52 | 0.3 |
| | | FAT-7 | Y | 0.13 | 72 | 0.3 |
| | | FAT-11 | Y | 0.13 | 60 | 0.3 |
| | | FAT-8 | Y | 0.13 | 60 | 0.1 |
| | | FAT-10 | Y | 0.13 | 60 | 0.1 |
| | | FAT-2 | Y | 0.13 | 63.7 | 0.3 |
| | | FAT-15 | Y | 0.13 | 63.7 | 0.3 |
| | | FAT-15 | Y | 0.13 | 68 | 0.3 |
| | | FAT-14 | Y | 0.13 | 70 | 0.3 |
| | | FAT-36 | Y | 0.13 | 75 | 0.3 |
| | | FAT-17 | Y | 0.13 | 85 | 0.3 |
| | | FAT-4 | Y | 0.13 | 85 | 0.3 |
| | | FAT-34 | Y | 0.13 | 88 | 0.3 |

Specimens FAT-7 and FAT-15 were initially tested at the lower of the two stress conditions without failure after a large number of cycles. The stress level was then increased and fatigue testing was restarted.
Y = yes; N = no

## 6.6 Fatigue Crack Growth Rate (FCGR)

The FCGR performance of the material was evaluated in accordance with ASTM E647 [7]. FCGR testing, also known as d$a$/d$N$ testing, is a method of evaluating the ability of a material to grow a crack and then quantifying the rate of the crack growth, where $a$ = crack length, $N$ = number of cycles and d$a$/d$N$ = the instantaneous crack growth rate. Unlike fatigue testing where the specimens are initially crack free, FCGR evaluates the safety and reliability of materials by subjecting the specimen to repeated loading and unloading in the presence of a preexisting crack. The FCGR test reports the resistance to stabilized crack extension under cyclic loading. The

Paris law[8] regime was examined in this evaluation.  Standard compact tension, C(T), [$B$ = 0.5 inch; $W$ = 1.75 inches] specimens – see Figure 20 – were utilized for testing and sectioned from the cladded plate as illustrated in Figure 14.  Test details are defined in Table 10.  Calibration testing was completed in air while the bulk of testing was completed in the HPHT sour-gas environment defined in Table 5.

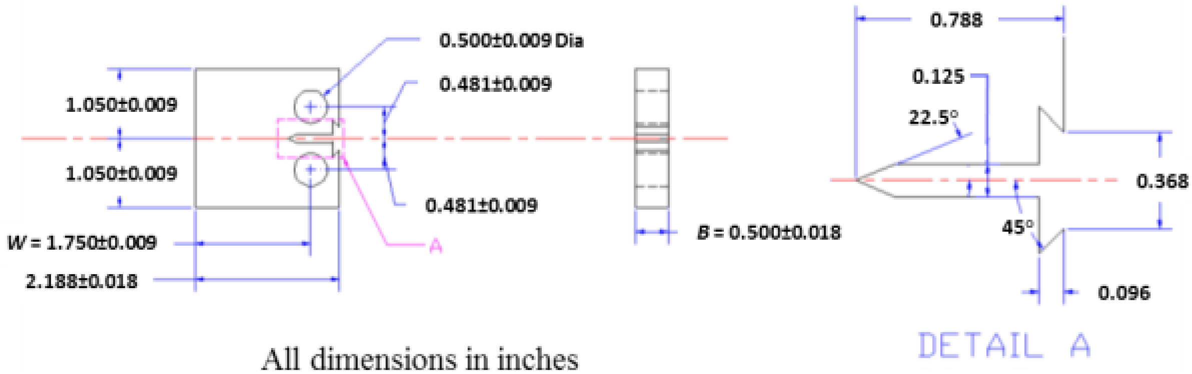



**Figure 20:  Drawing of FCGR specimen**

**Table 10:  Details of Fatigue Crack Growth Rate Testing**

| Test Type | Notch Location (Layer) | $\Delta K$ (ksi·in$^{1/2}$) | Frequency (Hz) | Number of Tests |
|---|---|---|---|---|
| Direct Current Potential Drop (Calibration)* | Lower | Over range | Over range | 1 |
| | Upper | | | 1 |
| Frequency Scan (Calibration)** | Lower | Aim:  15–18 | 0.001–1.0 | 1 |
| | Upper | | | 1 |
| Paris Curves** | Lower*** | Increasing $\Delta K$ | 0.1 | 3 |
| | Upper*** | | | 3 |
| | Lower | | | 1 |

*Testing was completed in air at 350 °F.
**Specimens were tested under HPHT sour-gas conditions.
***One of each of these specimens yielded no useful data since no crack growth was observed under the tested conditions.
$\Delta K$ is the change in stress intensity factor.

The FCGR behavior of nickel-based alloys in sour-gas environments was thought to be a strong function of the frequency at which the tests are performed.  Therefore, several tests were performed at a constant $\Delta K$ and varying frequency.  The purpose of these tests was to characterize the frequency response of the material/environment combination.  It was expected that with decreasing frequency, the FCGR (i.e., the rate of crack extension per cycle) would increase because of the increased per cycle exposure time creating a potentially thicker passive film and subsequent dissolution of the fresh metal, which can enhance the FCGR.  It was proposed to perform this test at a high $R$-ratio of about 0.7 and an intermediate $\Delta K$ in the range of about 15–18 ksi•in$^{1/2}$.

[8] The Paris law region of a FCGR curve is the linear region of the log-log curve of crack growth rate versus the range of the stress intensity factor.

In order to characterize the FCGR behavior it was important to measure the crack length in-situ using DCPD. In order to accurately characterize the crack length using this method in a multilayer system as illustrated in Figure 14, a calibration curve was developed using two tests (one for each of the two clad layers) in-air at 350 °F to develop a co-relation between the potential drop signals and the crack length. The pre-cracks for these calibration tests were located at an $a/W$ value of 0.35, which was similar to the $a/W$ value of approximately 0.23 proposed for the environmental tests.

## 7.0 SPECIMEN PREPARATION

Dilution of iron from the substrate plate into the cladding is significant enough to alter the fatigue and fracture properties of the cladding from those of its reported pure-clad-alloy values [25]. This effect is also related to the amount of dilution, which changes within each layer of a multi-layer cladding. Actual dilution effects for material used in the current project were measured by SEM coupled with energy dispersion spectroscopy (SEM-EDS). A plot of iron content through the clad layers is presented in Figure 9.

A 48-inch × 20-inch × 1-1/4-inch plate of steel alloy ASTM A387 Grade 22, Class 2 was procured from Wingate Alloys, Inc. (steel plate manufactured by ArcelorMittal USA) for use in preparing specimens – see Appendix A for material certification. Inconel 625 cladding was then applied by Hi-Tech Weld Overlay Group, LLC (Hi-Tech) of Lee's Summit, MO – an experienced cladding producer for the oil and gas industry – see Appendix B for the cladding statement of work. Appendix C is a copy of the clad application certification. Two clad layers were required to achieve the minimum desired clad-layer thickness of 0.25 inch. After application of the cladding, the plate was stress relieved by Solar Atmospheres, Inc. in Hermitage, PA as follows: heat to 1075 °F ± 25 °F at a heating ramp up rate of 100 °F per hour; hold at temperature for 4 hours; and cool at a rate equivalent to air cool to below 400 °F, resulting in a mean cooling rate of 3.5 °F per minute.

Test specimens for SSR tensile, bent-beam SCC and fatigue testing were extracted with their axes aligned perpendicular to the application direction of individual clad weld beads (see Figure 21). This direction was selected since it crosses multiple weld beads and is therefore likely the weakest direction. Test specimens were taken so the test region included 100% clad material. Individual SSR tensile, bent-beam SCC and fatigue test specimens included material from both clad layers. Therefore, the measured SSR tensile, bent-beam SCC and fatigue properties were a composite of the two clad layers.

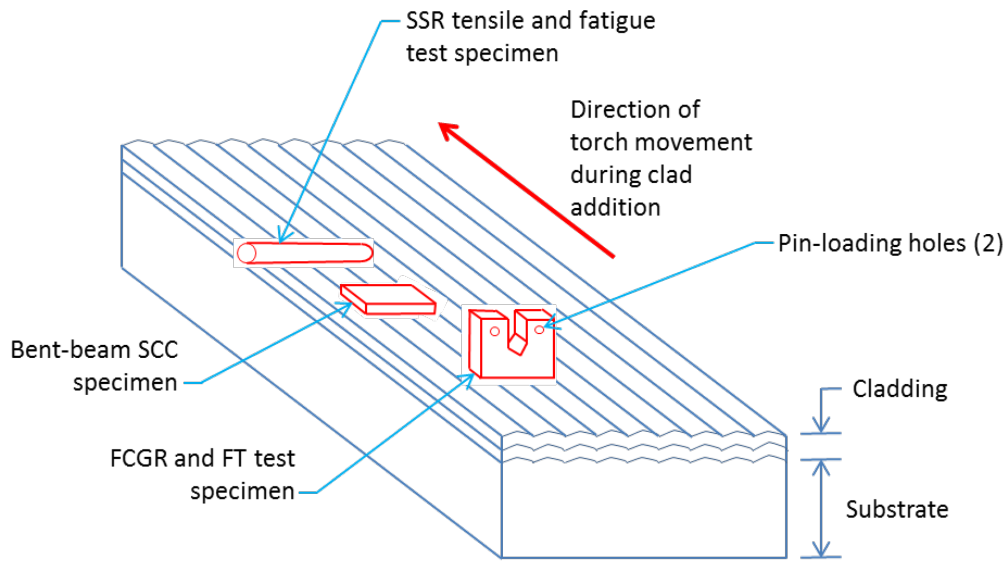



**Figure 21: Illustration of test specimen orientations as extracted from clad plate**

Test specimens for FCGR and FT were oriented as illustrated in Figure 21 so the cracks would grow from the top of a given clad layer downward towards the steel substrate. Test specimens were also oriented so their crack faces would be parallel to the bead application direction as illustrated in Figure 21. FCGR and FT test specimens required additional material be present above the cladding. This additional material is needed for the pin-loading holes (see Figures 15 and 20) used to apply the load to the specimen during testing. Initially, using minimum heat input, CTC attempted to fusion weld wrought Inconel 625 extensions onto the top of a cladded test piece whose top surface was machined flat in preparation for fusion welding. However, upon metallurgical examination, the test piece was found to have a significantly different microstructure (and thereby likely also differing mechanical properties) than the unaltered cladding. To alleviate the undesirable heat-related metallurgical impact to the cladding, CTC successfully added the needed material via metal additive manufacturing (AM) on the machined top surface of the cladding – see illustration of finished specimen in Figure 14. The resulting clad microstructure on the test specimen was not changed below a thin layer (< 200 microns = 0.008 inch) at the clad/AM material interface. FCGR and FT measurements were made on material at least 0.050 inch below this interface; therefore, the addition of metal by AM was noted as a significant success as it allowed testing of metallurgically unaltered clad material in the desired test orientation. After chemical etching (to reveal locations of individual weld beads), notch locations were determined. Notches were located at the root of neighboring weld beads as illustrated in Figure 16. Overall test specimen dimensions were obtained by machining (i.e., milling). The front and back faces of the specimens were then ground smooth and flat to reduce surface roughness on the potentially notch-sensitive Inconel 625 cladding to avoid any stress concentration effects from the earlier machining operations. The notch and pin-loading holes were then machined via wire electrical discharge machining (EDM). Finally, the FT specimens were side grooved to the standard 10% of specimen thickness ($B$) and pre-cracked according to ASTM Standard E1820 [6]. The grooves helped to keep the crack straight during testing, thus improving the likelihood of achieving a valid $J_{Ic}$ value instead of just a $J_q$ (provisional estimate for $J_{Ic}$) value.

## 8.0 SUMMARY AND DISCUSSION OF RESULTS

Detailed metadata for each of the tests is provided on a compact disc (CD) accompanying this report. Summarized here are the resulting material properties measured for each of the properties described above.

### 8.1 Engineering Stress-Strain Tensile Testing

Material was extracted from the cladding so that it axis was perpendicular to the direction of clad addition. The specimen was tested according to ASTM E21 [4]. To serve as a needed reference for the HPHT testing to be completed for other properties, a tensile specimen was tested in air at 350 °F. The specimen was strained at constant rate of 0.005/min to 6–7% strain and then continued at 0.05/min to failure. This resulted in a 0.2% offset yield strength of 65.9 ksi and an ultimate tensile strength of 105.6 ksi as noted in Figure 22. These data were used as the basis for many of the mechanical property test limits selected in the fatigue and fracture testing described below.

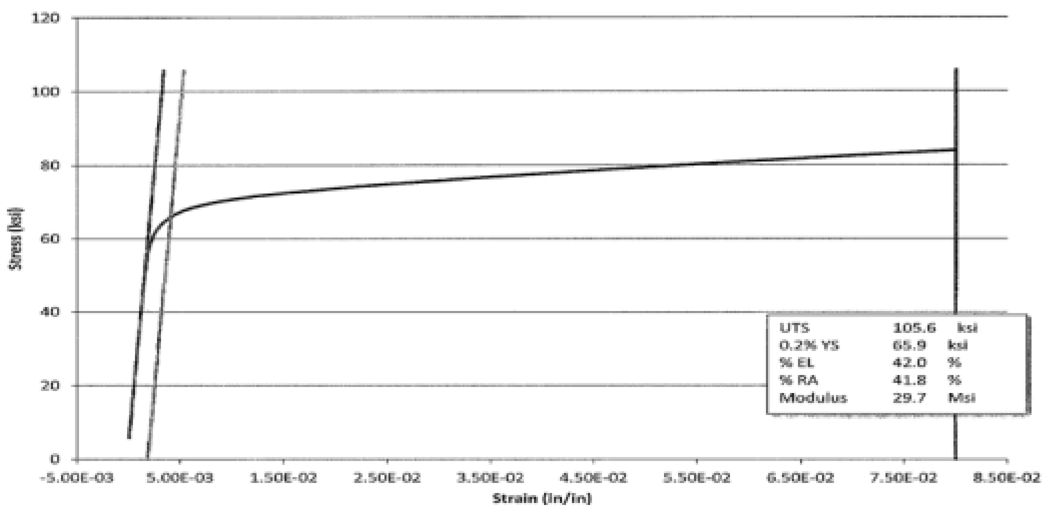



**Figure 22: Engineering stress-strain curve of Inconel 625 cladding at 350 °F**

### 8.2 Slow-Strain-Rate Tensile Testing

Typically SSR tests are performed according to NACE TM0298 [14], which involves performing tests at a strain rate of $4\times10^{-6}$/sec, which is the rate used in the present SSR tests. Specimens were taken to failure in tension. The resulting stress-strain curves are summarized in Figures 23 and 24. The material yielded at approximately 80 ksi and 70 ksi at room temperature and 350 °F, respectively. Ultimate tensile strength was approximately 118 ksi and 105 ksi at room temperature and 350 °F, respectively. Higher elongations were consistent with lower flow stress values. Elongation was reduced from a strain of approximately 45% at room temperature to about 40% at 350 °F. Little scatter (< 1% around the mean of the test results) was observed for the in-air tests. However, some scatter in tensile properties (~5% around the mean of the test results) was observed in the HPHT sour-gas results. For each of the two sets of curves (i.e., room temperature and 350 °F), the sour-gas environment did not appear to significantly impact the mean tensile properties of the Inconel 625 cladding. However, the sour-gas environment appeared to have increased the scatter in the resulting measurements.

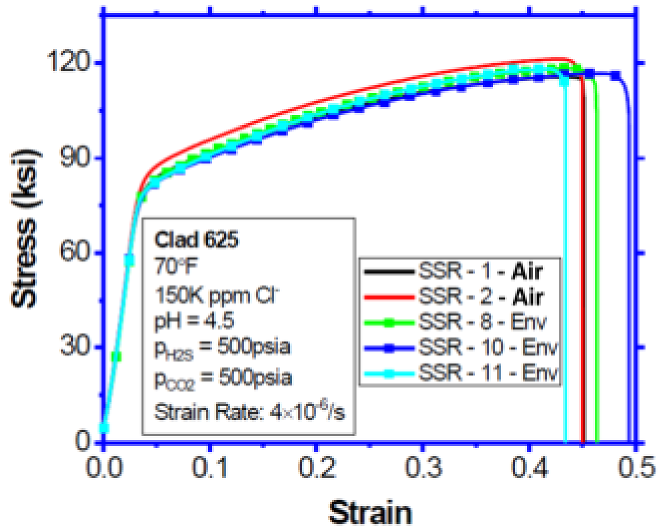



**Figure 23: SSR curves for Inconel 625 cladding at room temperature**

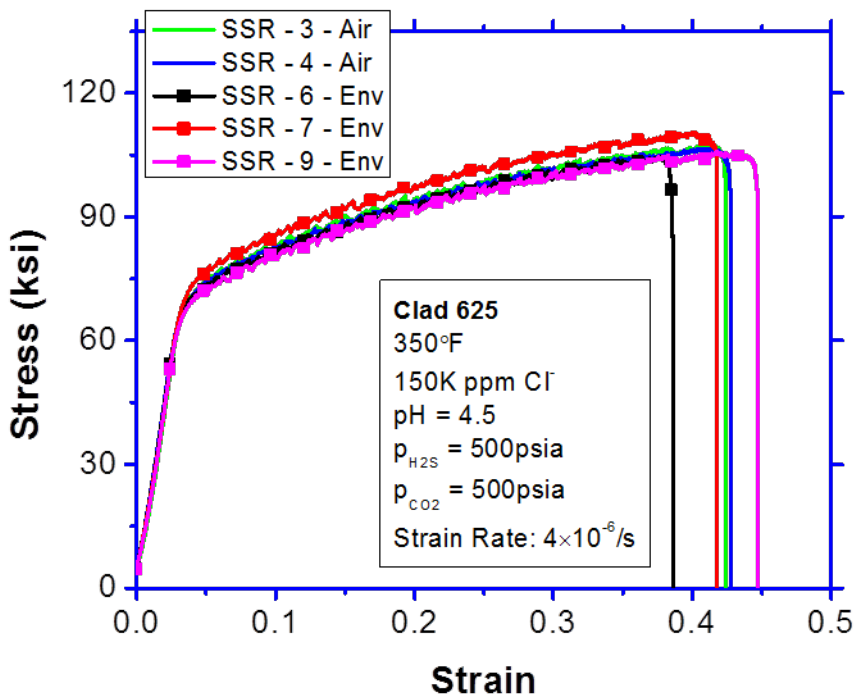



**Figure 24: SSR curves for Inconel 625 cladding at 350 °F**

Tensile elongation, reduction of area and time to failure results are graphically summarized in Figures 25 and 26 for room temperature and 350 °F, respectively. This method of displaying the results focuses on the scatter observed at each temperature/environment condition. All values are normalized around a value of 1.0. Very consistent values can be seen in air, while the values under sour-gas conditions demonstrate some scatter, as noted above.

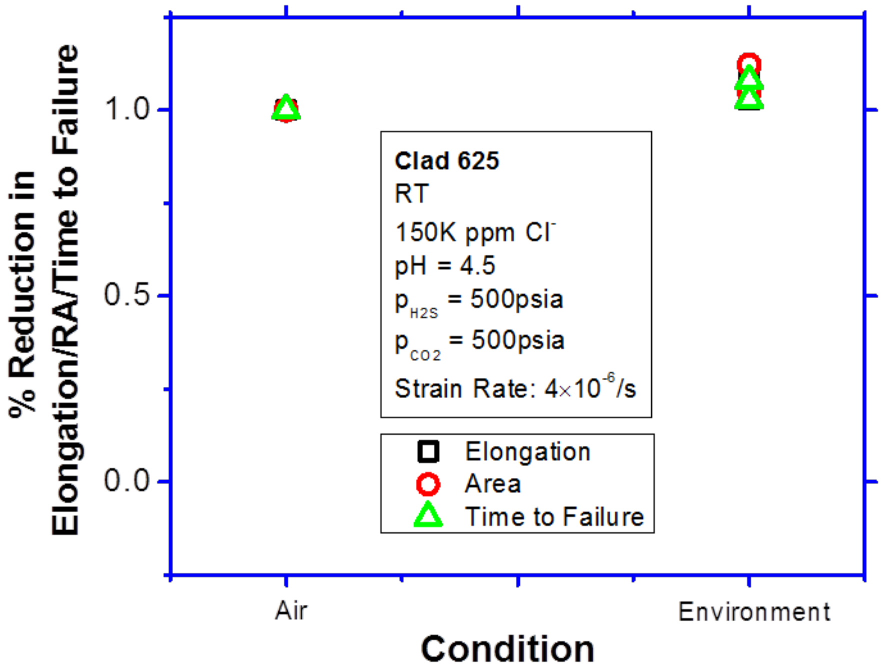



**Figure 25: Normalized elongation, reduction of area and time to failure of Inconel 625 cladding at room temperature**

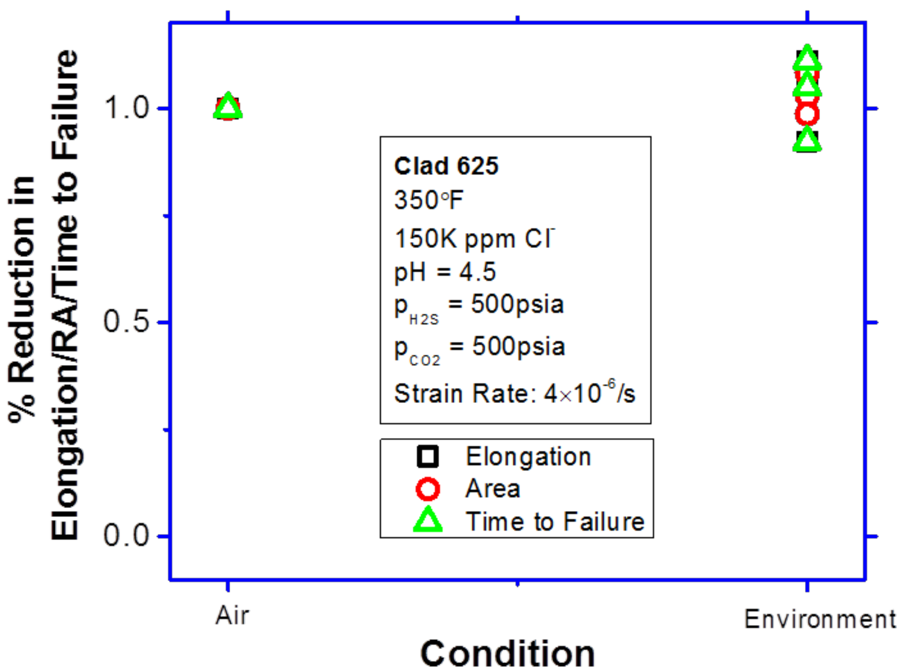
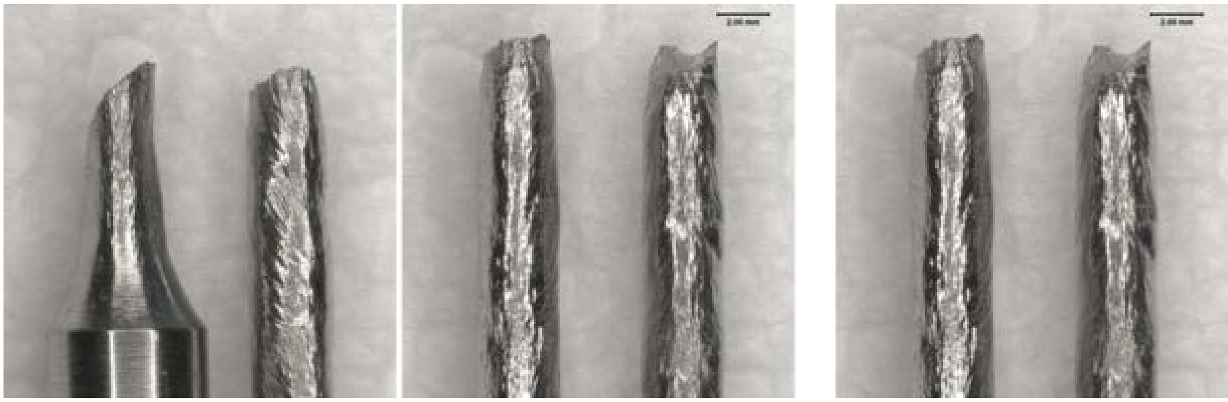


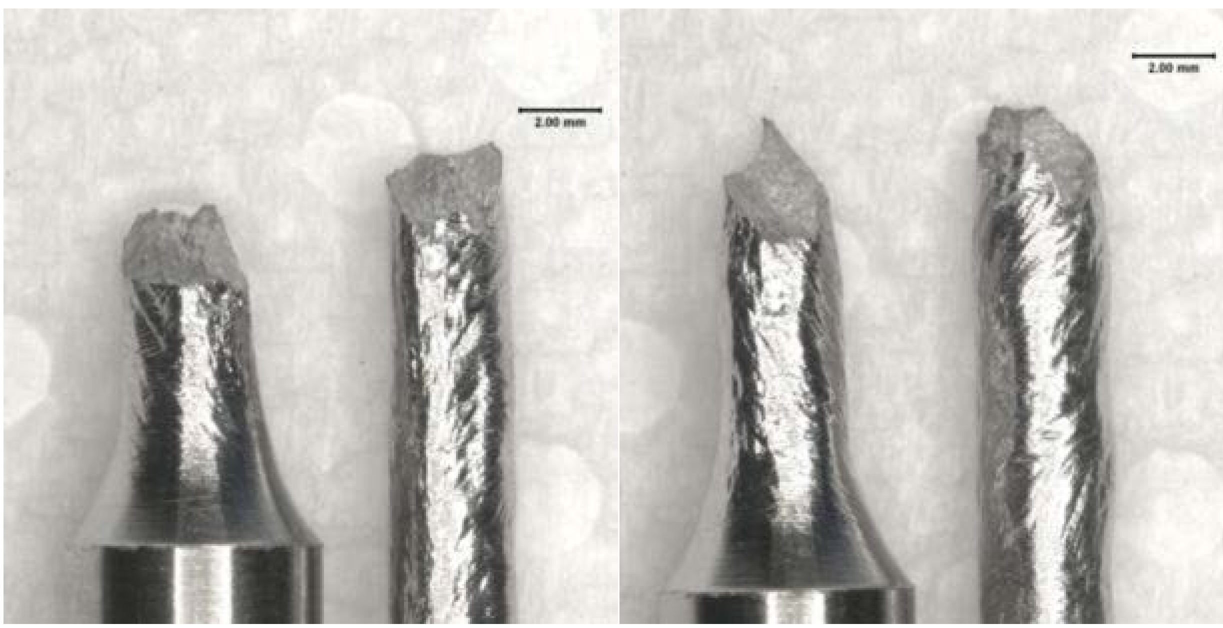

**Figure 26: Normalized elongation, reduction of area and time to failure of Inconel 625 cladding at 350 °F**

Macrographs of selected tensile tests are shown in Figures 27 through 30. Other than the obvious failure location, no evidence of additional cracking was observed on the surface of these specimens. SEM images for both a room-temperature and a 350 °F specimen are shown in Figures 31 and 32, respectively. The fracture surfaces showed no evidence of any secondary cracking; the fracture surfaces exhibited evidence of ductile fracture with no evidence of brittle fracture. The samples showed an extensive orange-peel-like effect due to the crystallographic texture of clad alloy, which is induced by the significant plasticity consistent with the high strain-to-failure values.



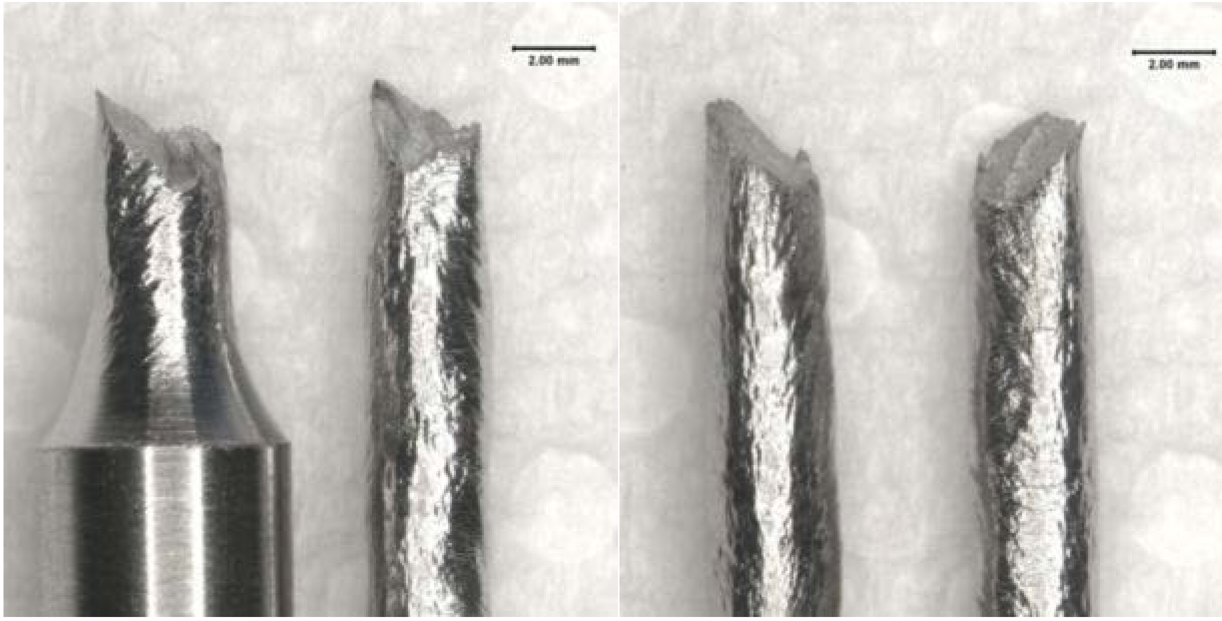
(a) SSR-8 (b) SSR-10 (c) SSR-11

**Figure 27: Macrograph of tensile specimens tested at room temperature in sour-gas environment**



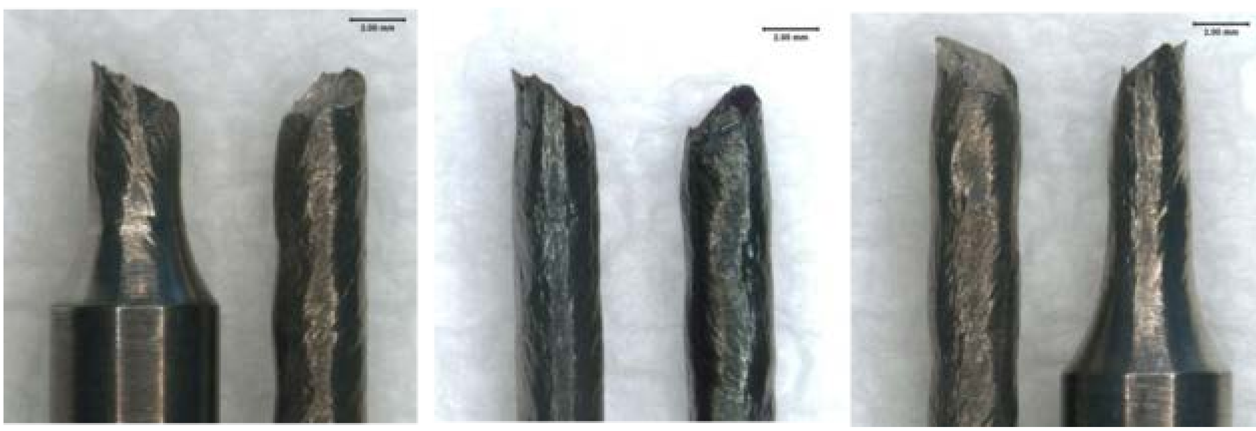

(a) SSR-1 (b) SSR-2

**Figure 28: Macrograph of tensile specimens tested at room temperature in air environment**



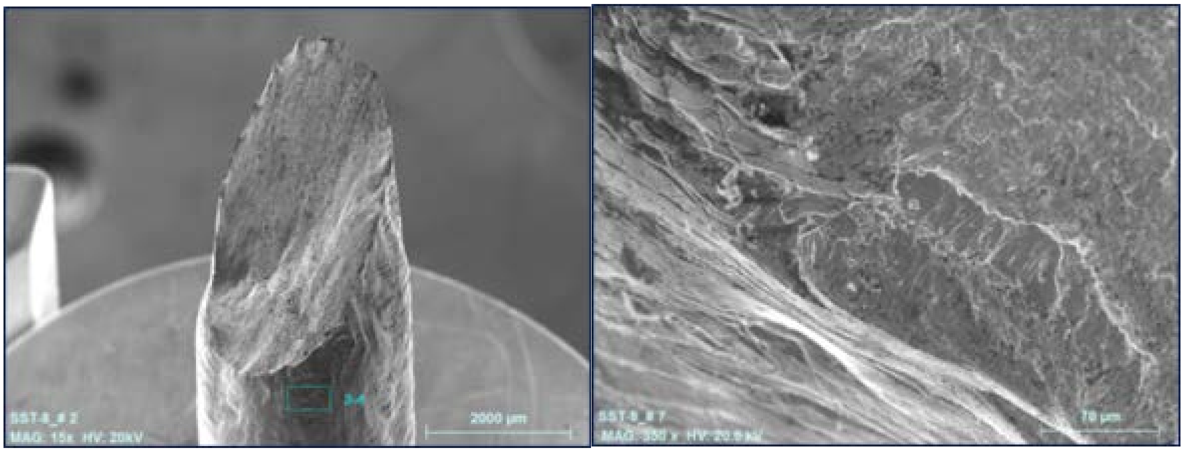

(a) SSR-3 (b) SSR-4

**Figure 29: Macrograph of tensile specimens tested at 350 °F in air environment**



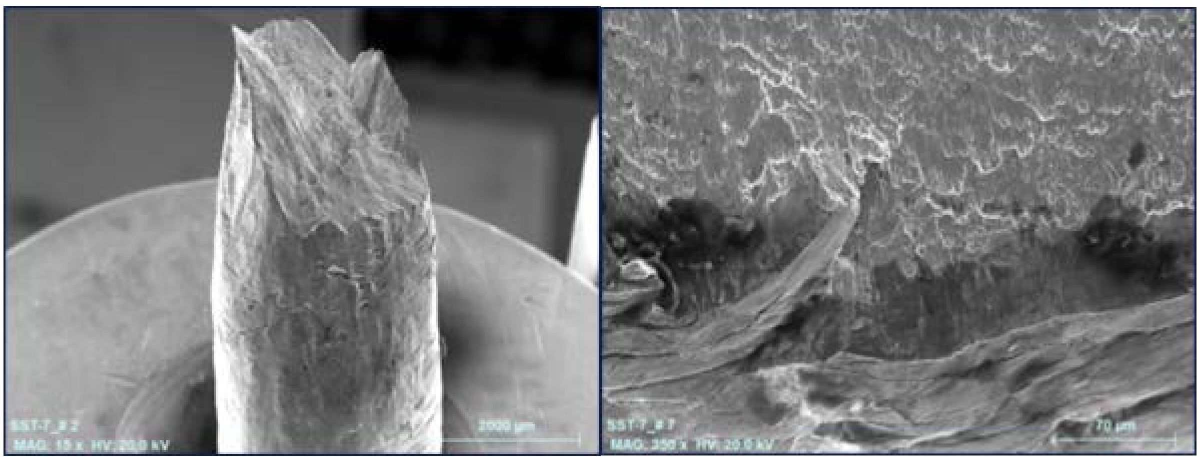
(a) SSR-6 (b) SSR-7 (c) SSR-9

**Figure 30: Macrograph of tensile specimens tested at 350 °F in sour-gas environment**




(a) Low-magnification image (b) High-magnification image

**Figure 31: Typical room-temperature microstructure of fracture surface in sour-gas environment**




(a) Low-magnification image (b) High-magnification image

**Figure 32: Typical 350 °F microstructure of fracture surface in sour-gas environment**

## 8.3 Bent-Beam Stress Corrosion Cracking (SCC) Testng

The specimen loading condition was first determined by plotting the load versus displacement curve for a typical specimen. From this curve, which is analogous to a stress-strain curve, one can determine the displacement to put on the test specimens to achieve the desired stress condition. As seen in Figure 33, the specimen was in elastic stress until 1040 pounds was exerted by the test frame as determined by an offset from the initially parallel line representing elastic behavior. The offset line is parallel to the elastic portion of the curve and intersects the displacement axis at the displacement where the load-displacement curve deviates from linear elastic behavior. From this yield load, the displacements at 95%, 110% and 120% of apparent yield load (AYL) were determined. Three replicates were tested under each of these conditions; specimens were held for 30 days in the HPHT environment. None of the specimens showed signs of cracking or pitting, indicating minimal attack by the HPHT sour-gas environment. Figure 34 shows macrographs of specimens from each of the stress levels evaluated.

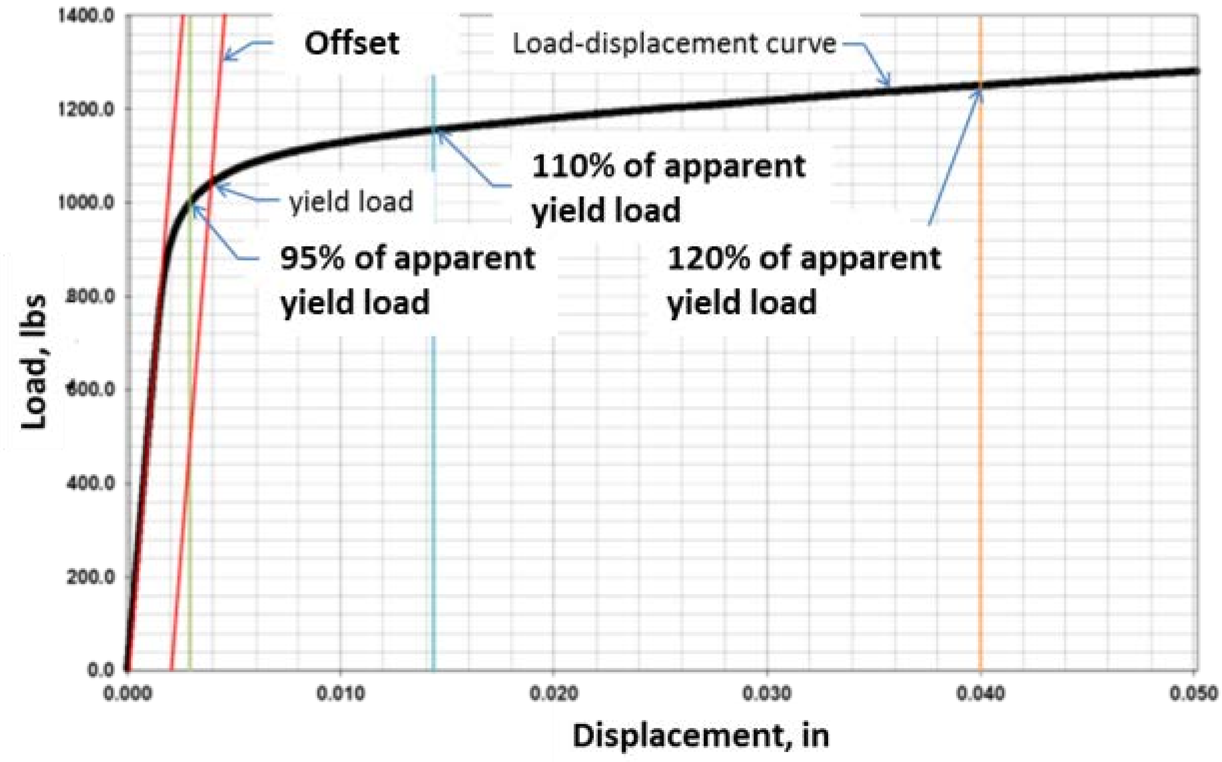



**Figure 33: Determination of displacements imparted to three sets of SCC specimens**


(a) Specimen 2622-3 tested at 95% of AYL

(b) Specimen 2622-4 tested at 110% of AYL

(c) Specimen 2622-7 tested at 120% of AYL

**Figure 34: Macrographs of specimens from four-point SCC bend tests for Inconel 625 cladding subjected to HPHT sour-gas conditions for 30 days**

### 8.4 Fracture Toughness Testing

As mentioned earlier, to determine the crack length during testing under sour-gas conditions, an electrical current was passed through the specimen and the potential drop used to determine the crack length. This requires that a calibration run be completed, especially for the mixed metals of the specimens tested in this project. The calibration routine was completed in air at 350 °F, with the results shown in Figures 35 and 36 for the upper and lower clad layers, respectively.

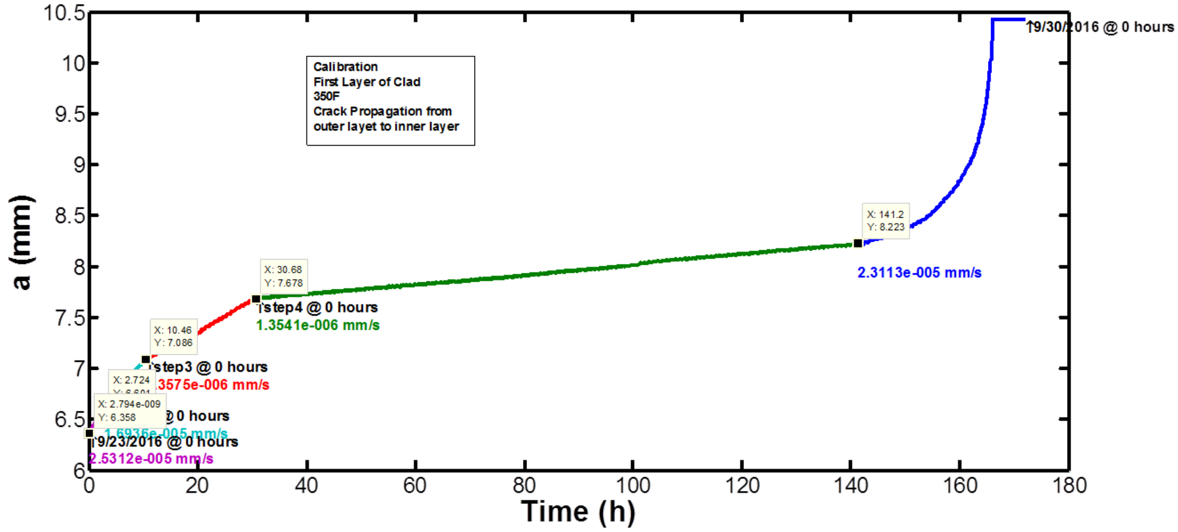



**Figure 35: Fracture toughness calibration curve (crack extension versus time) for the upper Inconel 625 clad layer**

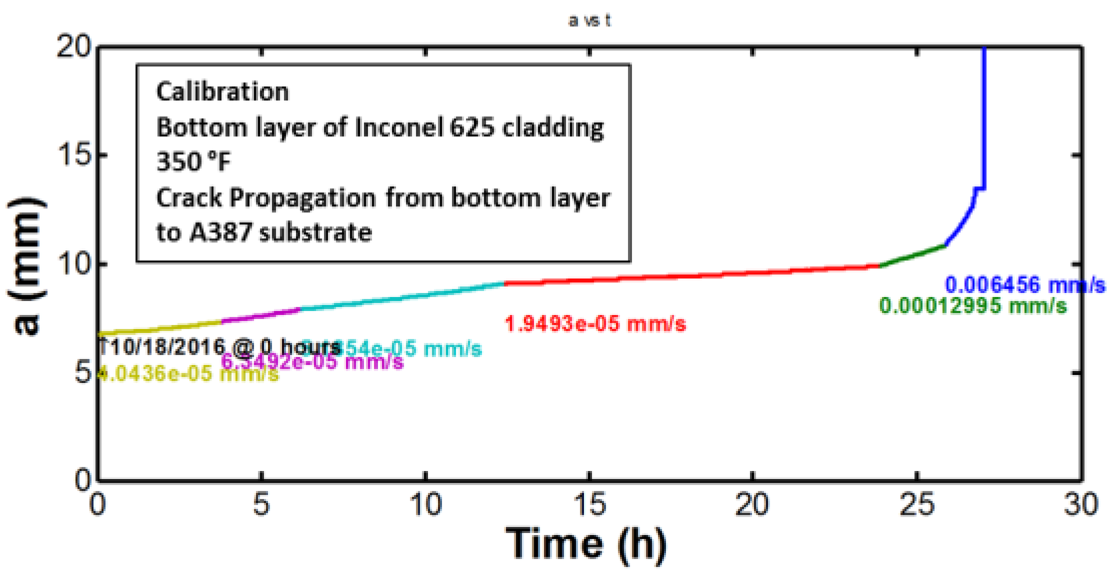


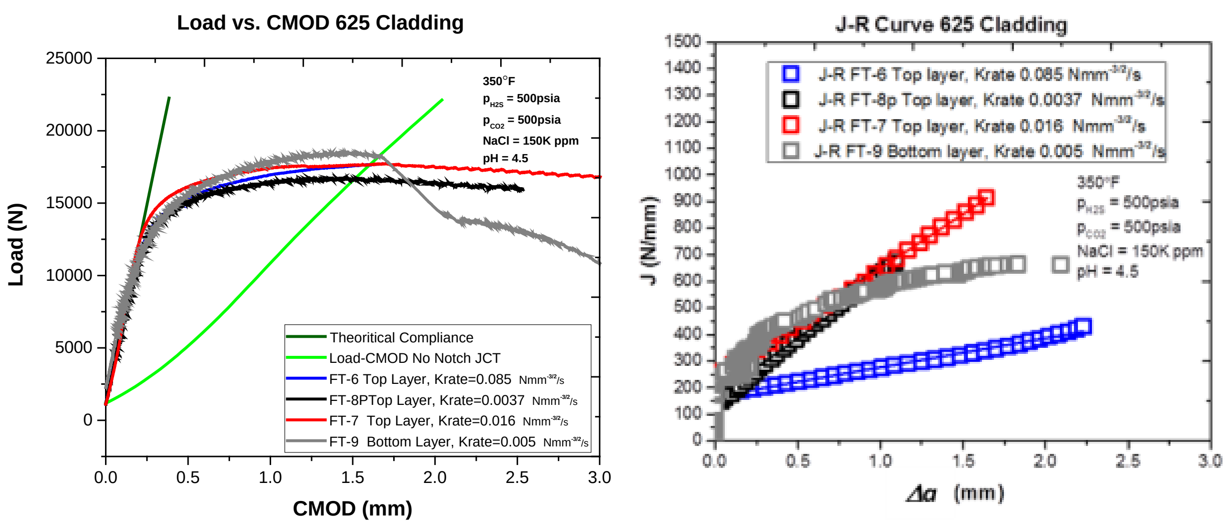

**Figure 36: Fracture toughness calibration curve (crack extension versus time) for the lower Inconel 625 clad layer**

Using the above calibration curves, fracture toughness specimens were tested under the HPHT sour-gas conditions defined in Table 5. Three fracture toughness specimens were tested from the upper layer while only one specimen was tested from the lower clad layer. The three specimens from the upper clad layer were tested at varying *K* (stress intensity factor) rates to establish a *K*-rate value for subsequent fracture toughness testing. The resulting *J* values are listed in Table 11. Several values are shown, corresponding to various positions on the *J* versus $\Delta a$ (change in crack length) curves shown in Figure 37. There did not appear to be a significant sensitivity to *K*-rate. The initiation toughness of the lower clad layer is slightly higher with a $J_{th}$ value of 247

N/mm. The *R*-curve of the lower layer exhibits a much shallower slope compared to the upper layer, suggesting slightly higher susceptibility to crack propagation. One fracture toughness specimen failed to provide meaningful data, while the final two fracture toughness specimens were used to generate the calibration curves for subsequent tests.

**Table 11: Fracture Toughness Results in HPHT Sour-Gas Environment**

| Specimen ID | Notch Location | *K*-rate (N/mm$^{-3/2}$•s) | $J_{th}$ (N/mm) | $J_{0.2\ mm}$ (N/mm) | $J_{1.0\ mm}$ (N/mm) | $J_{MaxLoad}$ (N/mm) |
|---|---|---|---|---|---|---|
| Fracture Toughness Measure | | | Threshold | Value at 0.2 mm CMOD | Value at 1.0 mm CMOD | Value at Maximum Load |
| FT-6 | Upper | 0.085 | 168.8 | 192.8 | 275.8 | -- |
| FT-7 | Upper | 0.016 | 234.9 | 325.4 | 642.0 | 474.3 |
| FT-8P | Upper | 0.0037 | 160.7 | 253.8 | 604.7 | 300.7 |
| FT-9 | Lower | 0.005 | 247 | 344 | 576 | 380 |




(a) Crack-mouth opening displacement (CMOD) (b) *J-R* curve

**Figure 37: Fracture toughness results**

With no evidence of load drops in the upper layer, ductile tearing in this clad layer is likely occurring. Figure 38 shows an SEM image of the fracture surface of Specimen FT-6, a specimen tested in the upper clad layer. Figure 39 shows similar information for Specimen FT-9, which was tested in the lower clad layer. No sign of intergranular or transgranular cracking was observed; however, ductile voids can be seen as the crack front advances from the fatigue pre-crack. This suggests that the clad layers are not susceptible to environmentally assisted cracking under the tested conditions. The above results compliment the results from the SSR and 4-point bend SCC testing, which indicated that there was no measureable susceptibility to environmentally assisted cracking under the tested conditions. Testing at high levels of plastic deformation both in the unnotched (SSR and 4-bend SCC at applied stress greater than yield strength) as well as notched condition (fracture toughness) suggests that under the test conditions, clad Inconel 625 appears to be very resistant to SCC. Higher threshold values on the lower layer could be a result of a slightly higher YS closer to the fusion line. The lower layer *J*-

*R* curve (see Figure 37(b)) is significantly "flat" as the crack tip advanced towards the fusion line.

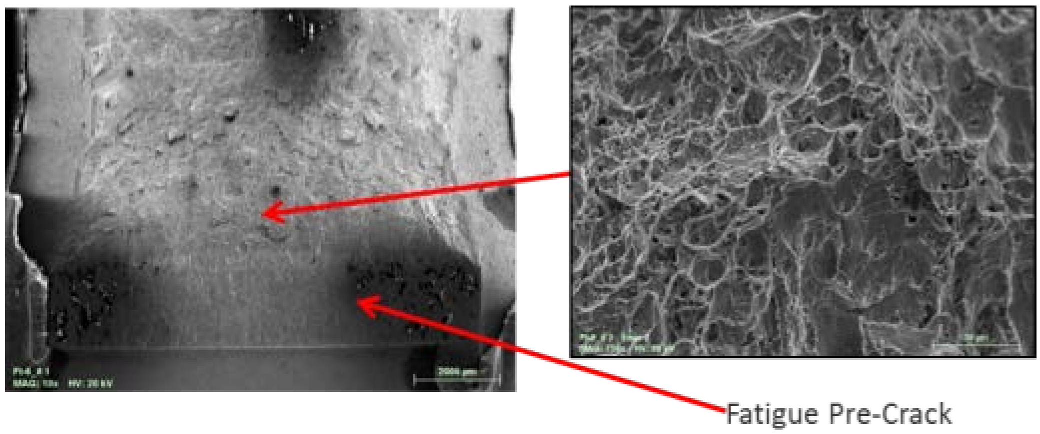



**Figure 38: SEM image of fracture surface of upper layer fracture surface**

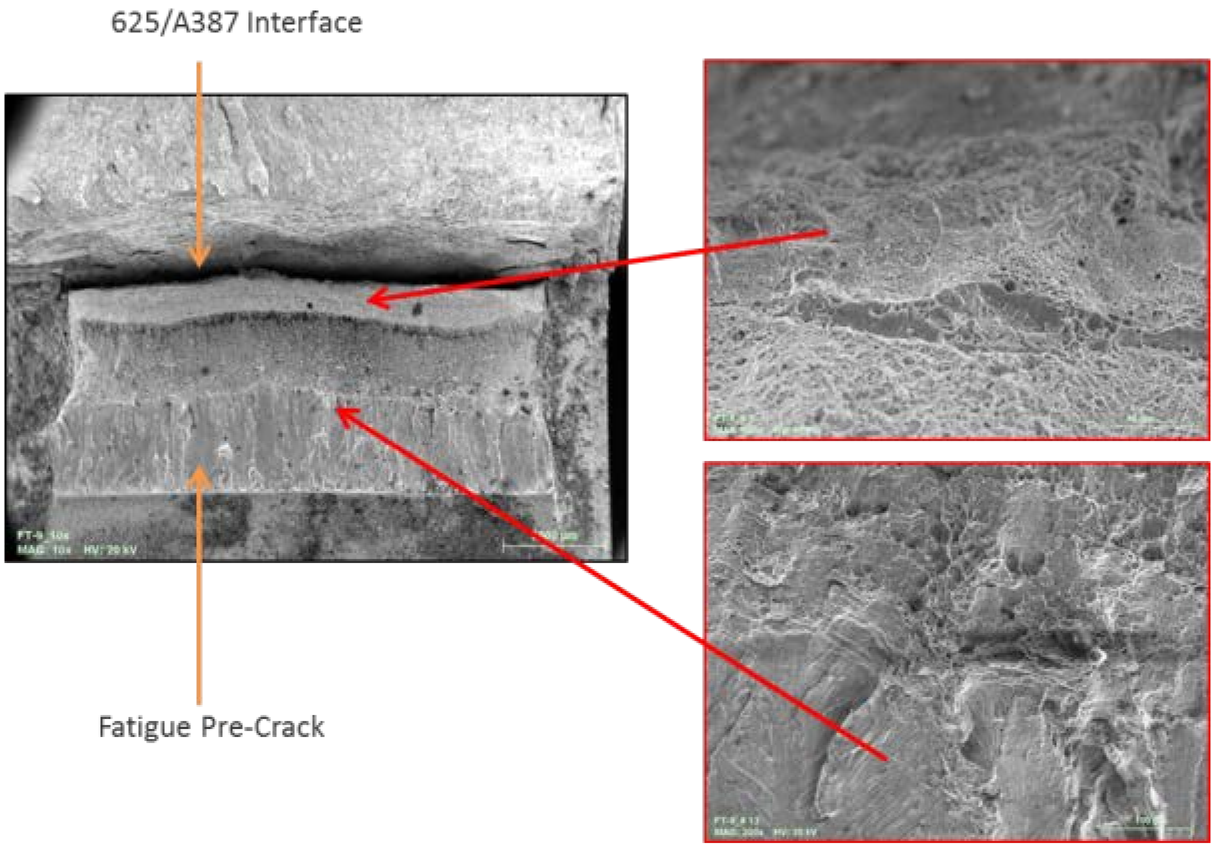



**Figure 39: SEM image of fracture surface of lower layer fracture surface**

## 8.5 Fatigue Testing

Table 12 summarizes the results of fatigue testing. Early in the execution of the fatigue tests smooth-walled fatigue test specimens (FAT-9 and FAT-32) were evaluated in air. Neither of these specimens failed within the desired number of load cycles (several hundreds to several thousands). Specimen FAT-32 along with two other specimens (FAT-30 and FAT-35) were then modified by machining a notch at the mid-length of the test region (as noted in Figure 17) to a notch sensitivity of 3.0 by machining a notch having a root radius of 0.005 inch. As with the earlier tests, none of these fatigue specimens failed within the desired number of cycles. Other specimens were then machined with a notch having a 0.0034-inch root radius and tested. (CTC considered machining a notch root radius less than 0.0034 inch too risky as minor machining errors consistent with machining variability would lead to undesirable variations in the test results.) In addition, the peak cyclic stress was also increased in an attempt to achieve the desired number of cycles to failure. While none of the specimens tested in air met the desired number of cycles to failure, as noted in Table 12, the test team agreed to use the findings of the in-air tests and apply them to testing in HPHT sour-gas conditions. The early testing was completed on specimens with the peak cyclic stress below the yield strength of the Inconel 625 cladding. Failure within the desired number of cycles was not achieved. Subsequent tests were completed at increasingly higher values of peak cyclic stress. Eventually, the peak cyclic stress during testing exceeded the yield strength of the Inconel 625 cladding. As the peak cyclic stress was increased to 88 ksi (the maximum peak cyclic stress tested, which was 134% of yield strength), the desired number of cycles to failure ($N_f$) was achieved, as noted in the results of Specimen FAT-34 in Table 12. When pristine data[9] are plotted on a log-log scale, the trend looks well behaved – see Figure 40. Three curves are shown here: 1) the best-fit linear relationship (labeled $N_f$), 2) the best-fit, lower-bound linear relationship using one-sided statistics with 97.5% confidence (labeled $N_{f,97.5\%}$) and 3) the best-fit, lower-bound linear relationship using one-sided statistics with 99% confidence (labeled $N_{f,99\%}$). Using a method defined in Reference 26, the linear relationships using the best fit and the one-sided statistics were defined.

[9] Pristine data are those from HPHT sour-gas test environment that 1) progressed to failure using only one peak cyclic stress value and one *R* value, 2) experienced no anomalies during testing and 3) were tested at a frequency of 0.3 Hz.

**Table 12: Fatigue Test Results**

| Environment | Temperature (˚F) | Specimen ID | Notched | *R* | $\sigma_{max}$ (ksi) | *f* (Hz) | $N_f$ (Actual) | Failure |
|---|---|---|---|---|---|---|---|---|
| Air | 350 | FAT-30 | Y | 0.13 | 60 | 2 | 580,129 | Y |
| | | FAT-9 | N | 0.13 | 60 | | 25,206 | N |
| | | FAT-32 | N/Y | 0.13 | 60 | | 488,715 | Y |
| | | FAT-35 | Y | 0.13 | 60 | | 2,163,834 | N |
| | | FAT-13 | Y | 0.13 | 52 | | 145,526 | Y |
| | | | | 0.13 | 52 | | | |
| | | | | 0.13 | 52 | | | |
| | | FAT-16 | Y | 0.13 | 63.7 | | 249,025 | Y |
| | | | | 0.13 | 63.7 | | | |
| | | | | 0.13 | 63.7 | | | |
| | | FAT-3 | Y | 0.30 | 60 | | 2,334,719 | N |
| | | FAT-33 | Y | 0.30 | 60 | | 80,267 | Y |
| | | FAT-31 | Y | 0.75 | 60 | | 10,460,808 | N |
| HPHT Sour-Gas 500-psia $H_2S$ 500-psia $CO_2$ | 350 | FAT-7 | Y | 0.13 | 52 | 0.3 | 1,629,040 | N |
| | | FAT-7 | Y | 0.13 | 72 | 0.3 | 25,523 | Y |
| | | FAT-11 | Y | 0.13 | 60 | 0.3 | 101,060 | N |
| | | FAT-8 | Y | 0.13 | 60 | 0.1 | 162,138 | Y |
| | | FAT-10 | Y | 0.13 | 60 | 0.1 | 32,461 | Y |
| | | FAT-2 | Y | 0.13 | 63.7 | 0.3 | 50,836 | Y |
| | | FAT-15 | Y | 0.13 | 63.7 | 0.3 | 1,425,360 | N |
| | | FAT-15 | Y | 0.13 | 68 | 0.3 | 27,312 | Y |
| | | FAT-14 | Y | 0.13 | 70 | 0.3 | 18,600 | Y |
| | | FAT-36 | Y | 0.13 | 75 | 0.3 | 132,927 | N |
| | | FAT-17 | Y | 0.13 | 85 | 0.3 | 16,332 | Y |
| | | FAT-4 | Y | 0.13 | 85 | 0.3 | 11,445 | Y |
| | | FAT-34 | Y | 0.13 | 88 | 0.3 | 4,852 | Y |

Specimens FAT-7 and FAT-15 were initially tested at the lower of the two stress conditions without failure after a large number of cycles. The stress level was then increased and fatigue testing was restarted.
*Only the number of cycles to failure after notching is shown for Specimen FAT-32.

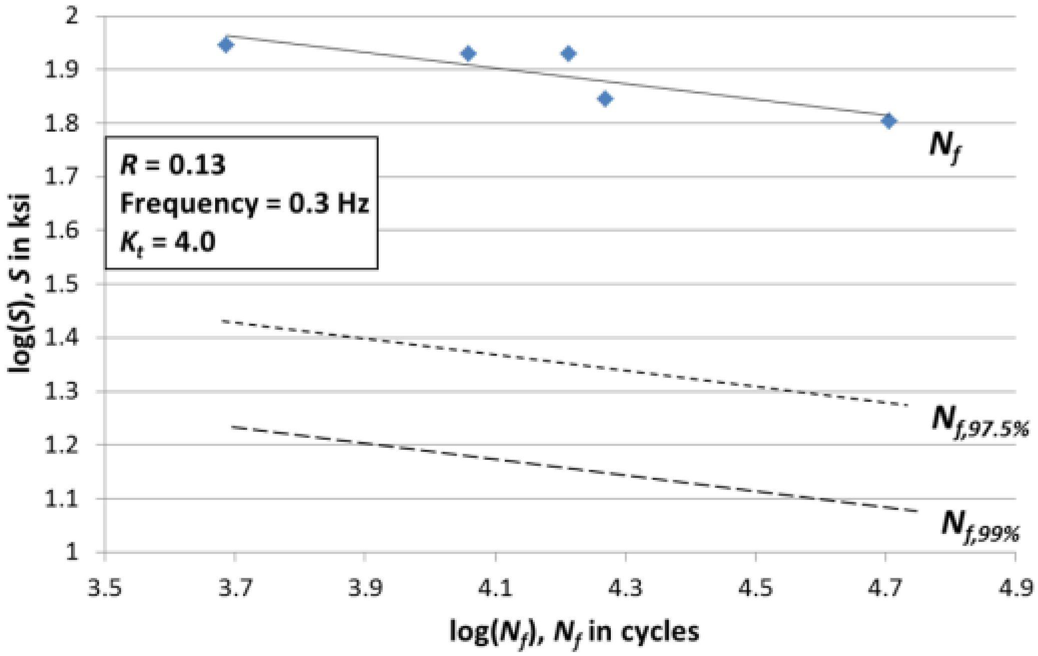



**Figure 40: Curve fit to fatigue test results using pristine data for Inconel 625 cladding under HPHT sour-gas conditions**

As other HPHT sour-gas fatigue data are plotted, they align very well with the trend of the pristine data. These added data points can be seen in Figure 41, where the red dots represent two specimens (FAT-7 and FAT-15) that were "uploaded" and the green stars represent fatigue tests completed at a frequency of 0.1 Hz. Uploading is when a specimen tested at a certain peak cyclic stress level that has not yet failed is restarted at a higher peak cyclic stress level. This was done to Specimens FAT-7 and FAT-15 as a result of observing no signs of imminent fatigue failure at the initially applied stress values after more than 1,000,000 cycles. Rather than continuing to test under conditions that far exceeded the desired number of cycles to failure, completing the testing at a higher peak cyclic stress provided some insight on the fatigue results from increased peak cyclic stress. Only the number of cycles at the higher stress level is shown for these specimens in Figure 41. The linear relationships shown in Figure 41 are identical to those in Figure 40; in other words, the statistical curves were not altered, but are included in Figure 41 for reference.

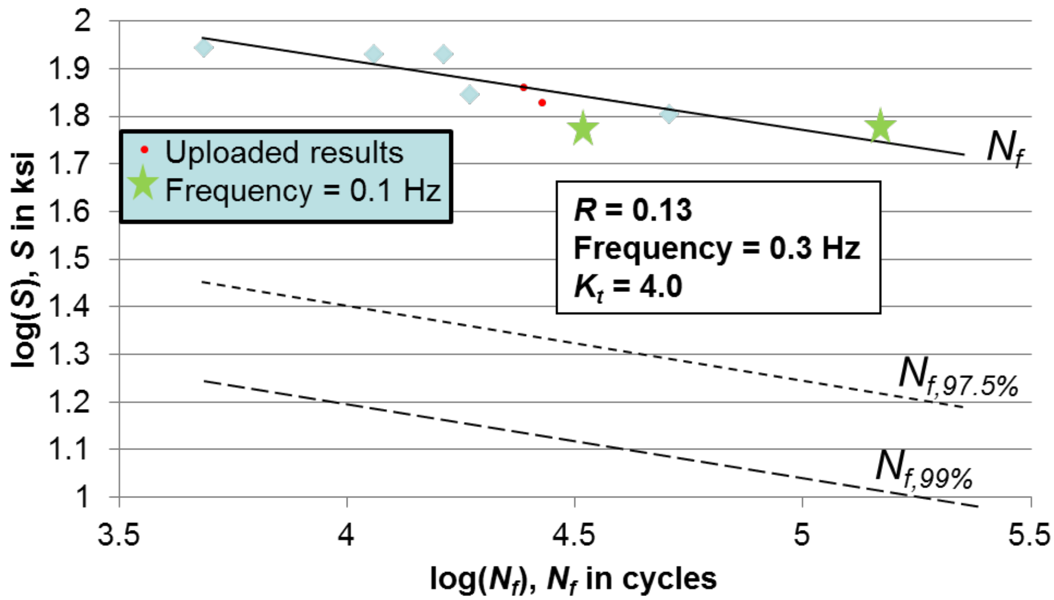



**Figure 41: Curve fit to fatigue test results using all data for Inconel 625 cladding under HPHT sour-gas conditions**

### 8.6 Fatigue Crack Growth Rate (FCGR)

After completing the calibration testing, frequency scans were taken for specimens in both the upper and lower clad layers. Fatigue crack growth rate frequency scan tests were performed at a constant $\Delta K$ of 25 ksi•in$^{1/2}$ and $K_{max}$ of 42.5 ksi•in$^{1/2}$ on both the upper and lower layers of the Inconel 625 cladding. Results of these frequency scans are summarized in Figure 42, which shows an increase in FCGR as the test frequency decreases. This may be due to selective attack on the cladding by the HPHT sour-gas environment. However, at the very lowest scan frequencies (i.e., those approaching 0.0001 Hz), static crack growth and/or increased HPHT exposure may be contributing significantly to the crack growth rate. Note also that the crack growth appears to be higher (by an order of magnitude) on the lower clad layer, i.e., the more diluted, layer. This suggests that crack growth may significantly accelerate in fielded equipment once an exterior crack has penetrated through the outer clad layer into the lower, more highly diluted, clad layer. While testing at low frequencies is desirable to more closely mimic the conditions experienced during oil and gas exploration and extraction, testing at frequencies approaching 0.0001 Hz is not practical as this represents one cycle for every 10,000 seconds or one cycle every 2.8 hours. Therefore, the majority of FCGR tests were completed at a test frequency of 0.001 Hz.

DNV attempted to determine a set of Paris law curves under increasing $\Delta K$ conditions, starting at an initial $\Delta K$ of 12–15 ksi•in$^{1/2}$ and $R$-ratio of 0.4. Based on the results above, DNV attempted to run the test at 1 mHz. However, in performing these tests on both the upper layer and lower layers no crack growth was observed. Subsequently a test was performed at a constant $R$-ratio of 0.4 at a high $\Delta K$ of 25 ksi•in$^{1/2}$. The FCGR appeared to decrease sharply as $\Delta K$ was reduced. The test frequency was then increased to 0.1 Hz and $\Delta K$ increased to determine if it was possible to sustain crack growth; however, it appeared that the crack was not able to reinitiate the growth.

Another test was performed starting at a high $\Delta K$ at a test frequency of 1 mHz. This test exhibited sustained fatigue crack growth rate until 800 N•mm$^{-3/2}$ (23 ksi•in$^{1/2}$).

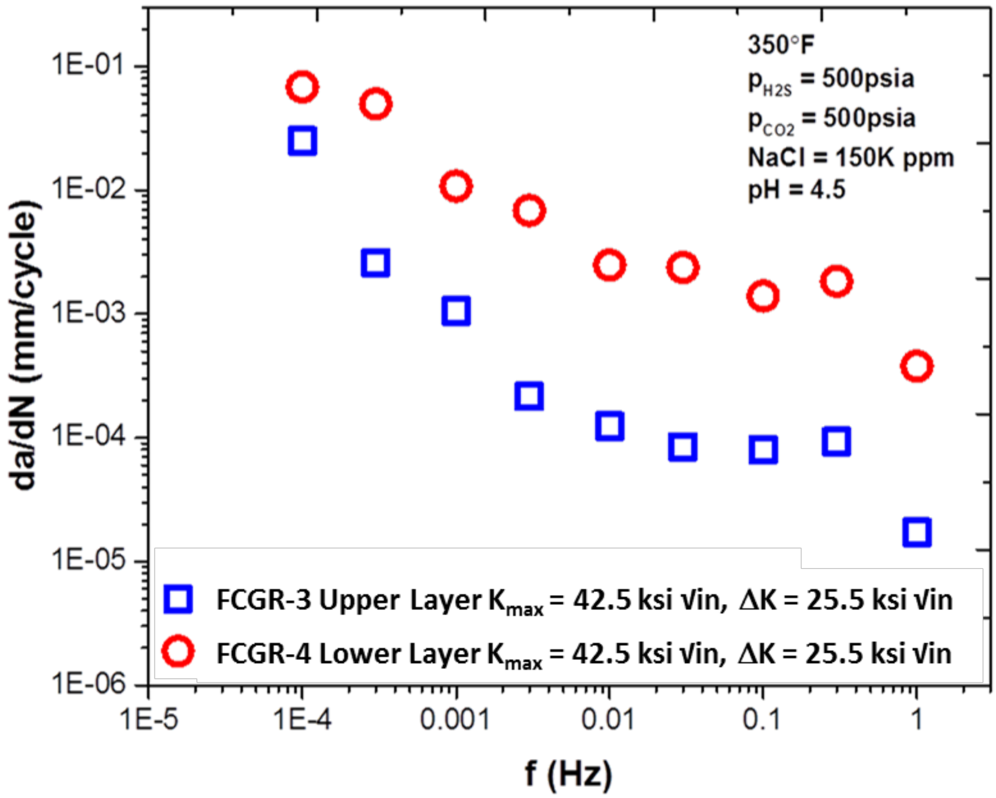



**Figure 42: FCGR frequency scans of Inconel 625 cladding under HPHT sour-gas conditions**

Of the FCGR tests completed, only one in the lower layer and two in the upper layer provided valid results, which are shown in Tables 13 and 14, respectively. Values in Table 13 were developed with a constant $K_{max}$ and variable $\Delta K$ (change in stress intensity factor). For these data $\Delta K$ and $R$ are not independent. Values in Table 14, the upper clad layer results, had two useful FCGR data sets. The first, from Specimen FCGR-8P, used a constant $R$ value of 0.4 and the maximum and minimum stress intensity factors, $K_{max}$ and $K_{min}$, respectively, were varied. The second group, from Specimen FCGR-11 was developed with a constant $K_{max}$ and variable $\Delta K$. Values of $\Delta K$ and $R$ from FCGR-11 are not independent. Data in both Tables 13 and 14 can be used in lookup table methods to predict FCGR behavior under similar fatigue behavior in Inconel 625 clad onto ASTM A387 Grade 22, Class 2 steel substrate.

**Table 13: FCGR Results for the Lower Clad Layer**

| ***ΔK*** **(N•mm$^{-3/2}$)** | **d*a*/d*N*** **(mm/cycle)** | ***Δa*** **(mm)** | ***R*** |
|---|---|---|---|
| | Crack growth rate | Change in crack length | Ratio of $K_{min}$ to $K_{max}$ |
| **Specimen FCGR-9** – tested at constant $K_{max}$ of 1470 N•mm$^{-3/2}$ | | | |
| 1279.3 | 0.00101 | 0.5591 | 0.130 |
| 1267.5 | 0.00226 | 0.1328 | 0.138 |
| 1250.5 | 0.00233 | 0.06833 | 0.150 |
| 1234.3 | 0.00241 | 0.04191 | 0.160 |
| 1219.4* | 0.00829 | 0.1930 | 0.171 |
| 1219.4* | 0.01371 | 0.02870 | 0.171 |
| 1194.0 | 0.00140 | 0.03175 | 0.188 |
| 1176.9 | 0.00100 | 0.03302 | 0.200 |
| 1158.8 | 8.00E-04 | 0.05080 | 0.212 |
| 1139.5 | 7.50E-04 | 0.06274 | 0.225 |
| 1119.0 | 7.00E-04 | 0.03556 | 0.239 |
| 1097.3 | 6.00E-04 | 0.03353 | 0.254 |
| 1074.2 | 5.84E-04 | 0.03378 | 0.269 |
| 1049.7 | 5.20E-04 | 0.03429 | 0.286 |
| 1023.7 | 4.30E-04 | 0.03454 | 0.304 |
| 996.1 | 4.00E-04 | 0.03478 | 0.323 |
| 966.7 | 3.00E-04 | 0.03505 | 0.343 |
| 935.6 | 2.70E-04 | 0.03353 | 0.364 |
| 709.3 | 2.00E-05 | 0.03404 | 0.518 |
| 605.5 | 3.00E-05 | 0.03327 | 0.588 |

*These values far exceed the trend of the other data.

**Table 14: FCGR Results for the Upper Clad Layer**

| **$\Delta K$ (N•mm$^{-3/2}$)** | **d*a*/d*N* (mm/cycle)** | **$\Delta a$ (mm)** | ***R*** |
|---|---|---|---|
| | Crack growth rate | Change in crack length | Ratio of $K_{min}$ to $K_{max}$ |
| **Specimen FCGR-8P** – tested at a constant R-ratio of 0.4 | | | |
| 897.5 | 3.87E-04 | 0.03556 | 0.4 |
| 832.3 | 2.07E-04 | 0.04572 | 0.4 |
| 817.4 | 1.84E-04 | 0.03962 | 0.4 |
| 802.2 | 2.61E-04 | 0.08661 | 0.4 |
| 786.2 | 1.82E-04 | 0.07010 | 0.4 |
| 744.8 | 2.00E-04 | 0.07137 | 0.4 |
| 611.4 | 2.18E-04 | 0.14376 | 0.4 |
| 600.5 | 1.81E-04 | 0.07087 | 0.4 |
| 589.7 | 3.46E-05 | 0.07493 | 0.4 |
| 583.1 | 3.01E-04 | 0.07315 | 0.4 |
| 549.3 | 1.55E-04 | 0.02296 | 0.4 |
| 517.2 | 3.87E-04 | 0.07087 | 0.4 |
| **Specimen FCGR-11** – tested at constant Kmax of 1470 N•mm−3/2 | | | |
| 1279.3 | 0.01842 | 0.363982 | 0.13 |
| 1279.3 | 0.02442 | 0.109982 | 0.13 |
| 1258.4 | 0.0109 | 0.049022 | 0.14423 |
| 1237.0 | 0.01677 | 0.073914 | 0.15878 |
| 1215.3 | 0.00688 | 0.030988 | 0.17355 |
| 1195.6 | 0.02045 | 0.089916 | 0.18693 |
| 1174.5 | 0.01413 | 0.0635 | 0.20126 |
| 1154.9 | 0.01135 | 0.051054 | 0.2146 |
| 1132.5 | 0.01553 | 0.068326 | 0.22985 |
| 1109.1 | 0.01871 | 0.084074 | 0.24577 |
| 1084.9 | 0.02019 | 0.0889 | 0.26224 |
| 1059.7 | 0.01501 | 0.067564 | 0.27937 |
| 1032.3 | 0.02301 | 0.103632 | 0.29797 |
| 1002.7 | 0.01959 | 0.086106 | 0.31812 |
| 971.3 | 0.01304 | 0.058674 | 0.33946 |
| 936.2 | 0.00491 | 0.021599 | 0.36338 |
| 898.3 | 0.01326 | 0.05969 | 0.38911 |
| 858.4 | 0.0034 | 0.014977 | 0.41626 |
| 817.6 | 0.00103 | 0.004653 | 0.444 |
| 774.3 | 4.34E-04 | 0.001908 | 0.47341 |
| 730.7 | 0.00377 | 0.016954 | 0.50307 |

FCGR results are presented in Figures 43 and 44 in log-log format, for the lower and upper clad layers, respectively. The trend shown in the lower clad layer is strikingly well behaved. The individual values are well represented by a straight line fit in log-log format. However, the results from the upper clad layer show some scatter, as expected for welded metal, around the best-fit straight line.

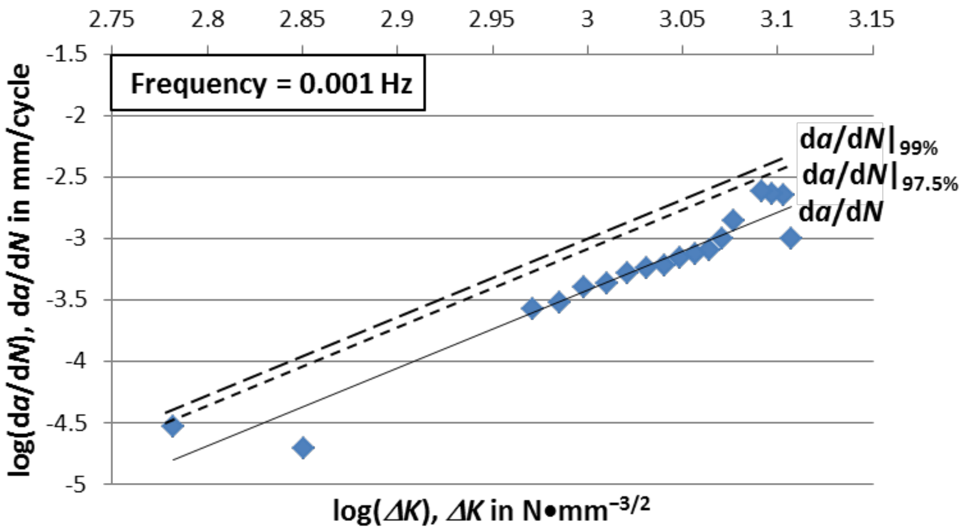



**Figure 43: Summary of FCGR for the lower clad layer under HPHT sour-gas conditions**

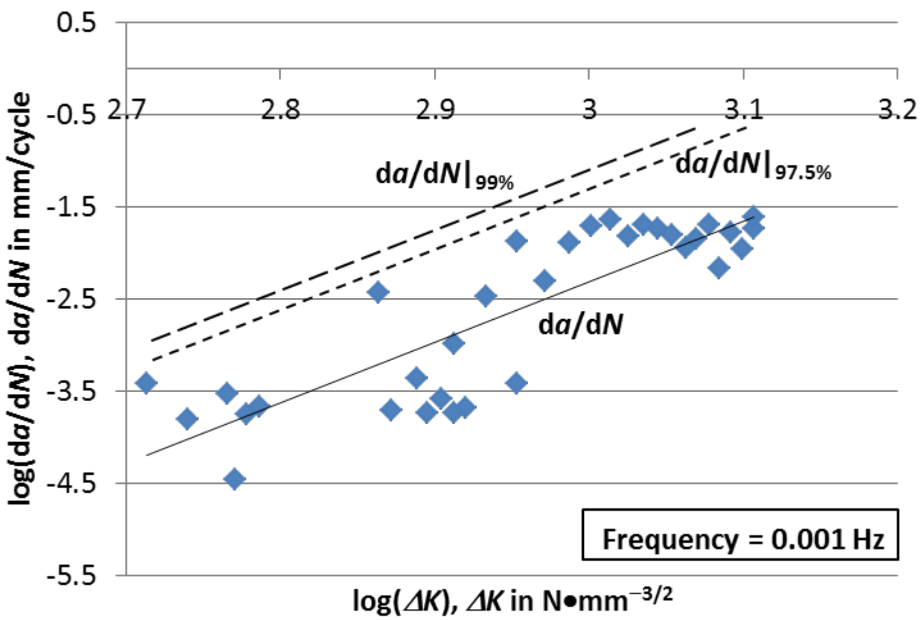



**Figure 44: Summary of FCGR for the upper clad layer under HPHT sour-gas conditions**

## 9.0 CONCLUSIONS

Based upon the findings, the following conclusions are offered.

1. The data provided in this report are a good start towards having a broad collection of publically available fatigue and fracture data for use by designers, failure analysts and regulatory bodies within the oil and gas exploration and extraction industry for clad components subjected to HPHT sour-gas conditions.

2. No observable cracking or pitting was observed in any of the SCC specimens (three replicates each were tested at 95%, 110% or 120% of apparent yield load), which were subjected to the HPHT sour-gas environment for 30 days.
3. Fatigue and fracture differences were noted between the inner and outer layers of the two-layer weld cladding evaluated in the present project. The differences can be attributed to the iron (Fe) dilution that primarily occurred in the inner layer. Treating each clad layer as a "separate" material in fatigue and fracture assessments is justified.
4. Slow strain rate tensile tests performed in the HPHT sour-gas environment did not show any evidence of environmentally assisted cracking. The fracture surface exhibited a ductile failure mode with no measureable evidence of attack by the Inconel 625 cladding from the HPHT sour-gas environment.
5. Fracture toughness tests performed in air and sour-gas environments in both the upper (low Fe dilution) and lower (high Fe dilution) Inconel 625 clad layers indicated the fracture toughness of both clad layers is high (threshold value of $J > 240$ N/mm in the lower clad layer and J ~ 190 N/mm in the upper clad layer, where $J$ is fracture toughness). Plane-strain plastic-elastic fracture toughness ($J_{Ic}$, defined as the $J$ value at a crack mouth opening displacement of 0.2 mm) averaged 257 N/mm for the upper clad layer; the singular $J_{Ic}$ value for the lower clad layer was 344 N/mm. The fracture surfaces exhibited ductile features, suggesting that neither clad layers were susceptible to environmentally assisted fracture.
6. FCGR frequency scan tests on both the upper (low Fe dilution) and lower (high Fe dilution) Inconel 625 clad layers did not exhibit a strong frequency dependence between 1 Hz and 3 mHz. However, between 1 mHz and 0.1 mHz, FCGR increased by about 100×. Although this suggests that chemical attack occurs at the crack tip, thereby making the material more susceptible to crack <u>growth</u> over time, effects of static growth rate, especially at the lowest test frequencies, may also have played a role in the increased FCGR at low test frequencies. During frequency scans, the lower layer (i.e., the one more highly diluted with substrate material) was found to have a higher FCGR by about an order of magnitude (i.e., 10×) over the upper layer. When the material was tested in the Paris law region, the FCGR of the lower layer was about twice that of the upper layer. These results suggest any crack that starts from the exterior of a cladded component may accelerate its growth rate once the outer clad layer has been completely penetrated and the crack grows into the lower clad layer.
7. To achieve failure within a few hundred to a few thousand cycles, cyclic fatigue specimens must be notched with a stress concentration factor of about 4.0 and subjected to nominal stresses that exceed yield. (For the Inconel 625 cladding evaluated here, the measured 0.2% offset yield strength at 350 °F was 65.9 ksi.) Preliminary fatigue tests on smooth bar tests resulted in runouts, after which additional fatigue tests were performed on notched specimens. Fatigue failures occurred between 4000 to 10,000 cycles in the peak cyclic stress range of 60 to 88 ksi. The log-log relationship between the number of cycles to failure and peak cyclic stress followed a linear relationship with minimal scatter around the best-fit curve, which included peak cyclic stresses both below and above the Inconel 625 cladding yield strength.
8. While the HPHT sour-gas environment may lead to greater scatter (~5%) in tensile elongation, reduction of area and time to failure during slow-strain-rate testing, the <u>mean</u>

values of these tensile properties were not significantly altered (~1%) from values measured in air at 350 °F.

9. Additive manufacturing methods were useful for providing material to the top of cladding without impairing its original microstructure/mechanical properties and enable physical completion of fracture and FCGR specimens.

## 10.0 RECOMMENDATIONS

1. Additional testing of material made by other clad vendors (and from multiple iterations of cladded materials from any given vendor) would provide information on the expected variability in fatigue and fracture behavior of cladding during oil and gas exploration and extraction. Future efforts should consider a test summary as defined in Appendix A.
2. Similarly, while cyclic fatigue testing was completed at a single stress ratio ($R$ = 0.13), completing additional cyclic fatigue tests at other stress ratios (and possibly with other than sinusoidal stress versus time cycles) would provide the industry with additional valuable data.
3. Since other cladding alloys are either being used or are being considered for use by the oil and gas exploration and extraction industry, complementing the present work by assessing the fatigue and fracture behavior of these other materials would also benefit the industry.
4. To determine low-cycle stress-based fatigue curves for common cladding materials, the test should start with nominal peak cyclic stresses just above and just below the yield strength of the cladding.

## 11.0 REFERENCES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# APPENDIX A: CERTIFICATION FOR SUBSTRATE PLATE

02/16/2016 WINGATE ALLOYS, INC.

PL#: F15283

Item: 1 (1 PC) 1-1/4" X 22" X 48"

ARCELORMITTAL PLATE LLC

TEST CERTIFICATE

PAGE NO: 01 OF 02
FILE NO: 0284-01-05
MILL ORDER NO: 26331-001
MELT NO: U0099
SLAB NO: 9A
DATE: 01/31/14

01-C

**STEEL PLATE DIMENSIONS / DESCRIPTION**

| TOTAL QTY | GAUGE | WIDTH | LENGTH | DESCRIPTION | PIECE WEIGHT |
|---|---|---|---|---|---|
| 1 | 1.25" | 120" | 480" | RECTANGLE | 20419# |

**CUSTOMER INFORMATION**

CUSTOMER PO: 97797-NY

PART NO. 3

**SPECIFICATION(S)**

THIS MATERIAL HAS BEEN MANUFACTURED AND TESTED IN ACCORDANCE WITH PURCHASE ORDER REQUIREMENTS AND SPECIFICATION(S).

ASTM A387 YR 10 GR 22 CL 2
ASME SA387 EDITION 13 GRADE 22 CLASS 2
THE MANAGEMENT SYSTEMS FOR MANUFACTURE OF THIS PRODUCT ARE CERTIFIED TO ISO 9001:2008 (CERTIFICATE NO. 30130) AND ISO 14001 (CERTIFICATE NO. 009496).

**CHEMICAL COMPOSITION**

| | C | MN | P | S | CU | SI | NI | CR | MO |
|---|---|---|---|---|---|---|---|---|---|
| MELT:U0099 | .15 | .52 | .009 | .005 | .21 | .20 | .16 | 2.26 | .94 |

| | V | TI | B | AL | CB | SB | AS | SN |
|---|---|---|---|---|---|---|---|---|
| MELT:U0099 | .004 | .001 | .0001 | .025 | .001 | .0020 | .0030 | .008 |

**MANUFACTURE**

ELECTRIC FURNACE QUALITY - FINE GRAIN PRACTICE

**HEAT TREAT CONDITION.**

| MATL OR TEST | HEAT TREAT DESCRIPTION | NOM TEMP | HOLD MINS | COOL MTHD |
|---|---|---|---|---|
| PL/TEST | NORMALIZE | 1675F | 34 | AIR COOL |
| PL/TEST | TEMPER | 1280F | 85 | AIR COOL |

Certified a true copy of the [illegible]

DO 3/16/14

WE HEREBY CERTIFY THE ABOVE INFORMATION IS CORRECT:

ARCELORMITTAL PLATE LLC
QUALITY ASSURANCE LABORATORY
139 MODENA ROAD
COATESVILLE, PA 19320

Elinore Zaplitny
SUPERVISOR – TEST REPORTING
ELINORE ZAPLITNY

02/16/2016 WINGATE ALLOYS, INC.

Item: 1 (1 PC) 1-1/4" X 22" X 48" PL#: F15283

TEST CERTIFICATE

PAGE NO: 02 OF 02
FILE NO: 0284-01-05
MILL ORDER NO: 26331-001
MELT NO: U0099
SLAB NO: 9A
DATE: 01/31/14

TENSILE PROPERTIES

| SLAB NO. | LOC | DIR | YIELD STRENGTH PSI X 100 | TENSILE STRENGTH PSI X 100 | ELONGATION GAGE LGTH | % | %R.A. |
|---|---|---|---|---|---|---|---|
| 9A | BOT. | TRANS. | 820 | 1000 | 2.00" | 22.0 | 72.0 |

GENERAL INFORMATION

ALL STEEL HAS BEEN MELTED AND MANUFACTURED IN THE U.S.A.
TEST CERTS. ARE PREPARED IN ACCORD. WITH PROCEDURES
OUTLINED IN EN 10204:2004 TYPE 3.1.
MERCURY OR MERCURY COMPOUNDS ARE NOT USED IN THE
MANUFACTURE OF ARCELORMITTAL PLATE LLC PRODUCTS.
ACID SOLUBLE ALUMINUM
FOR MORE INFORMATION AND PROCESSING GUIDELINES, REFER TO
WWW.ARCELORMITTAL.COM/PLATEINFORMATION

B/L #41843 TTPX 805483

WE HEREBY CERTIFY THE ABOVE
INFORMATION IS CORRECT:

ARCELORMITTAL PLATE LLC
QUALITY ASSURANCE LABORATORY
139 MODENA ROAD
COATESVILLE, PA 19320

Elinore Zaplitny
SUPERVISOR – TEST REPORTING
ELINORE ZAPLITNY

# APPENDIX B: CLADDING STATEMENT OF WORK

Concurrent Technologies Corporation (CTC) will supply a steel plate (ASTM A387 Grade 22) with approximate dimensions of 48 in x 22 in x 1.25 in. The vendor shall clad one face of the steel plate with Inconel 625 alloy using the weld overlay process. The following scope of work shall be performed to produce the weld overlay.

1. Fabrication welding and welders/welding operations should be qualified in accordance with applicable internationally recognized standards such as ASME Section IX, ANSI/NACE MR0175/ISO 15156, or equivalents.
2. Prepare and clean one (1) face of the steel plate for weld overlay deposition.
3. The overlay weld shall be made using Alloy 625 weld wire (ERNiCrMo-3).
4. Apply weld overlay over an area of 48 in x 20 in on the face of the steel plate.
5. The weld overlay must run in the longest direction of the steel plate.
6. The final weld overlay shall be near flat and must have a minimum thickness of 0.25 inches. The cladded plate shall not be machined.
7. Surface inspection of the weld overlay shall be conducted to detect surface imperfections via penetrant inspection in accordance with applicable ASME or ASTM standards for non-destructive inspection (NDI).
8. The steel plate with the un-machined weld overlay surface shall be sent to Juan J. Valencia, CTC, 128 Industrial Park Road, Johnstown, PA 15904-1942. CTC will complete final stress relief.
9. The process parameters used to create the weld overlay shall be provided with the cladded plate.

## SCHEDULE AND DELIVERABLES FOR CLAD PLATE

The manufacturing facility shall provide a sound un-machined weld overlay steel plate. Also, both an electronic and hardcopy of the welding procedures, wire weld material certificates and NDI results in accordance with the reporting requirements of the applicable specifications shall be provided to CTC.

# APPENDIX C:  WELD OVERLAY CERTIFICATION

Hi-Tech Weld Overlay Group, LLC
1695 SE Decker St.
Lee's Summit, MO 64081

**Welding Procedure Specification (WPS)**

WPS No.: **CRO F43-P5A H**   Date: **10/4/2010**   Rev. No.: **0**   Page 1 of 2
Supporting PQR(s): **625-P5A 2G**
Welding Process(es) / Type(s): **(1) GMAW / Semiautomatic and Machine**

**Joint Design (QW-402)**
Weld Type: Corrosion resistant overlays
WELD JOINT DESCRIPTIONS SHOWN ARE NOT INCLUSIVE OF ALL THOSE FOUND ON A JOB. WELD JOINT DESIGN REFERENCE IN AN ENGINEERING SPECIFICATION OR A DESIGN DRAWING SHALL TAKE PRECEDENCE OVER WELD JOINTS SHOWN IN THIS WPS.

**Base Metals (QW-403)**
P-No. 5A   Thickness Range: 0.25 in. minimum

**Preheat (QW-406)**
Minimum Preheat Temperature: 400 °F
Maximum Interpass Temperature: 550 °F
Preheat Maintenance: Temperature sensing crayons
Heat pads or rosebud torch

**Postweld Heat Treatment (QW-407)**
PWHT Type: PWHT below lower transformation temperature
PWHT Temperature: 1250 °F
PWHT Holding Time: 1.0 hr./in., 0.25 hr. minimum

Initial and Interpass Cleaning: With stainless steel brush clean area to be overlayed.
Method of Back Gouging: Remove overlay defects by grinding only.

We certify that the statements in this record are correct and that the test welds were prepared, welded, and tested in accordance with the requirements of Section IX of the ASME Code.

Accepted By: Jeff Burkin   10/4/2010 (Date)   QC Manager

Hi-Tech Weld Overlay Group, LLC
**Welding Procedure Specification (WPS)**

WPS No.: **CRO F43-P5A H**   Rev. No.: **0**   Page 2 of 2

**First Process:** GMAW   **Type:** Semiautomatic and Machine

**Filler Metal (QW-404)**
Weld Deposit Limits: 0.1875 in. minimum
AWS Classification: ERNiCrMo-3   SFA Specification: 5.14   F-No.: 43
A-No. or Chemical Composition: N/A
Filler Metal Product Form: Bare (Solid)
Supplemental Filler Metal: None

**Position (QW-405)**
Position of Joint: Flat & Horizontal
Weld Progression: N/A

**Gas (QW-408)**
Shielding: 100% Argon / 41-54 CFH
Trailing: None / - CFH

**Electrical Characteristics (QW-409)**
Current Type and Polarity: DCEP (reverse)
Transfer Mode: Pulsating arc
Max. Heat Input, 1st Layer (J/in): 11315
Energy/Power: Thermal Arc Power Master

**Technique (QW-410)**
Stringer or Weave Bead: Stringer bead
Nozzle / Gas Cup Size: 3/8" to 5/8"
Oscillation: None
Peening: None
Contact Tube to Work Distance: 3/4" - 1"
Number of Electrodes: 1
Multiple or Single Layer(s): Multiple layer

**First Process Welding Parameters**

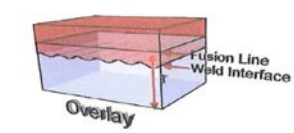
| Layer(s) and/or Pass(es) | Filler Metal | | Current | | Wire Feed Speed (in/min) | Voltage Range | Travel Speed Range (in/min) |
|---|---|---|---|---|---|---|---|
| | AWS Classification | Size (in.) | Type and Polarity | Amperage Range | | | |
| 1-n | ERNiCrMo-3 | 0.045 | DCEP (reverse) | 190-210 | 350-450 | 22-25 | 28 |
| 2-n | ERNiCrMo-3 | .045 | DCEP (reverse) | 190-210 | 350-450 | 22-25 | 28 |

Hi-Tech Industrial Services, Inc.
1695 SE Decker St.
Lee's Summit, Mo. 64081

**Procedure Qualification Record (PQR)**

PQR No.: **625-P5A 2G**   Date: **10/1/2010**   WPS No.: **CRO F43-P5A H**   Page 1 of 2
Welding Process(es) / Type(s): **(1) GMAW / Machine**

**Joint Design (QW-402)**
Weld Type: Corrosion resistant overlay




**Base Metals (QW-403)**
Specification Type and Grade: SA-387, Grade 21, Cl. 2
P-No. 5A   Group No. 1
Thickness (in.): 0.25

**Preheat (QW-406)**
Minimum Preheat Temperature: 400 °F
Preheat Maintenance: Temperature crayons
Maximum Interpass Temperature: 600 °F
Maintained preheat using heating pads.

**Postweld Heat Treatment (QW-407)**
Type: PWHT performed below lower transformation temp.
PWHT Temperature: 1250 -1350 °F
PWHT Holding Time: 0.25 hr.
Ambient to 200°F at a moderate rate, 200°F to 1250°F - 1350°F at a rate not to exceed 133°F/hr. Hold 1250°F - 1350°F for .25 hours minimum 1250°F - 1350°F to 400°F at a rate not to exceed 133°F/hr 400°F to ambient cool in still air.

**First Process:** GMAW   **Type:** Machine

**Filler Metals (QW-404)**
AWS Classification: ERNiCrMo-3
SFA Specification: 5.14   F-No.: 43
A-No. or Chemical Composition: N/A
Filler Metal Trade Name: Inconel 625
Filler Metal Product Form: Bare (Solid)
Supplemental Filler Metal: None
Min Qualified 't' (in.): 0.1875

**Positions (QW-405)**
Position of Joint: 2G - Horizontal
Weld Progression: N/A

**Gas (QW-408)**
Shielding: 100% Argon / 45 CFH
Trailing: None / - CFH

**Electrical Characteristics (QW-409)**
Current Type and Polarity: DCEP (reverse)
Transfer Mode: Pulsating arc

**Welding Details**
Filler Metal Size (in.): 0.045 | 0.045 | -
Amperage Used: 200 | 205 | -
Wire Feed Speed (in/min): 400 | 400 | -
Voltage Used: 24 | 25 | -
Travel Speed (in/min): 28 | 28 | -
Max. Heat Input, 1st Layer (J/in): 10286

**Technique (QW-410)**
Thermal Processes: No
Stringer or Weave Bead: Stringer bead
Nozzle / Gas Cup Size: 5/8"
Contact Tube to Work Distance: 3/4" - 1"
Oscillation: None
Number of Electrodes: 1
Multiple or Single Layer(s): Multiple layer

**Hi-Tech Industrial Services, Inc.**

**Procedure Qualification Record (PQR)**

PQR No.: 625-P5A 2G    Page 2 of 2

**Guided Bend Tests (QW-160)**

| Type and Figure No. | Result | Type and Figure No. | Result |
|---|---|---|---|
| Perpend. per QW-453 | Acceptable | Perpend. per QW-453 | Acceptable |
| Perpend. per QW-453 | Acceptable | Perpend. per QW-453 | Acceptable |

Visual Examination: No Indications

Liquid Penetrant Test: Satisfactory per QW 195.2

Macro-Examination Test: None

Chemical Analysis: C=0.03%, Cr=21.20%, Mo=8.71%, Ni=61.64%, Mn=0.05%, Si=0.04%, P<0.008%, S<0.005%, Fe=3.57%, AL=0.32%, Tl=0.31%, Nb=3.53%, Cu<0.01%, Co<0.01%

Welder's Name: Hubbard, Jason    I.D.: Q7    Stamp No.: Q7

PQR was done and welding of coupon was witnessed by: Hi-Tech Industrial Services, Inc.

Test conducted by: Sherry labs    Lab Test No.: 10100059-002-v1

We certify that the statements in this record are correct and that the test welds were prepared, welded, and tested in accordance with the requirements of Section IX of the ASME Code.

Accepted By: ____ Jeff Burkitt    10/1/2010 (Date)    QC Manager

**N.D.E. V.T. REPORT**

H-Tech Industrial Services Inc.
1695 SE Decker St.
Lee's Summit, Mo. 64081

| | |
|---|---|
| Customer Name: Concurrent Technologies Corporation 128 Industrial Park Road Johnstown PA, 15904 | Job Number: 4018 |
| | Unit Number: |
| Unit ID: Test Coupon | |
| Examination Technique: | Light Intensity Measurement: (1000 lux min.) |
| Procedure Number: VT-1, Rev 1 | 500 Watt Quartz Light |
| | > 2500 lux at 24" |

Identification of area examined:

**48"x 20" overlayed plate after 1st pass of Inconel 625**

Examination results ( location of rejected indications or area free from indications)

**2 rejectable indications found and repaired after which no indications found**

Operator Signature    Date: 3/10/2016    Cert. #: 0409-016

**N.D.E. D.P.T. REPORT**

Hi-Tech Industrial Services, Inc.
1695 Decker Street
Lees Summit, MO 64081

| | |
|---|---|
| Customer Name: Concurrent Technologies Corp | Job Number: 4018 |
| | Unit Number: |
| Penetrate Type: Color Contrast | Procedure Number: P.T.- **DPT-1 Rev. 2** |
| Brand Name: Spot Check | Examination Technique: Direct |
| Manufacturer : Magnaflux | |
| Batch No.: | Light Intensity Measurement: (100 fc min.) >100 fc |

**Identification of area examined:**

48"x20" plate with a double pass of 625 Inconel overlay

**Examination results (location of rejected indications or area free from indications):**

No rejectable indications noted at time of examination.

**Comments:**

| Operator Signature | Level | Date | CERT. # |
|---|---|---|---|
| | | 3/14/2016 | 0409-016 |

**HI-TECH** WELD OVERLAY GROUP

# POSITIVE MATERIAL IDENTIFICATION REPORT

| | |
|---|---|
| Report Number: | 1 |
| Job Number: | 4018 |
| Calibration Date: | 03/23/15 |

| | | | | | |
|---|---|---|---|---|---|
| Client: | CTC | Procedure Number: | Hi-Tech PMI | Revision: | 0 |
| Project: | Double Pass Inco 625 on P5A | Equipment Type: | Niton XL3t 980 | Serial Number: | 83196 |

| Test | Description | Material | Heat | Cr % | Ni % | Mo % | Mn % | Cu % | Fe % | Time/Sec | Accept | Reject |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Pass #1 Inconel 625 on P5A | 625 Inconel | QQ756 | 21.10 | 54.76 | 7.80 | | | 12.27 | | | |
| 2 | Pass #2 Inconel 625 on P5A | 625 Inconel | QQ756 | 21.10 | 61.72 | 8.61 | | | 1.8 | | | |
| 3 | | | | | | | | | | | | |
| 4 | | | | | | | | | | | | |
| 5 | | | | | | | | | | | | |
| 6 | | | | | | | | | | | | |
| 7 | | | | | | | | | | | | |
| 8 | | | | | | | | | | | | |
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| 10 | | | | | | | | | | | | |
| 11 | | | | | | | | | | | | |
| 12 | | | | | | | | | | | | |
| 13 | | | | | | | | | | | | |
| 14 | | | | | | | | | | | | |
| 15 | | | | | | | | | | | | |
| 16 | | | | | | | | | | | | |
| 17 | | | | | | | | | | | | |
| 18 | | | | | | | | | | | | |

Comments: (Operator shall check Analyzer for Calibration prior to each use with "Certified Reference Material" provided with the Analyzer.)

| | | | |
|---|---|---|---|
| Operator Name: | Greg Richards | Client Representative: | |
| Date: | 3/11/2016 | Date: | |

Note: (Next Analyzer Calibration Due on: 3/23/18)

| | | | |
|---|---|---|---|
| Quality Control: | Don Jones | Hi-Tech Representative: | |
| Date: | 3/11/2016 | Date: | |

721864-1

**HAYNES**
**Wire Company**

159 N. Egerton Rd
P.O. Box 677
Mountain Home, NC 28758
828-692-5791
828-697-9336 FAX

## PRODUCT CERTIFICATION

| WORK ORDER | HEAT NUMBER |
|---|---|
| W02286 | QQ756 |

SALES ORDER / RLS
010563 / 4

CERT ID / REV
00006947 / 01

SOLD TO

Haynes International, Inc.
158 North Egerton Road
Mountain Home, NC 28758
USA

ISO 9001:2008 Certified
AS9100 C Certified

| CUSTOMER P.O. | CUSTOMER PART | QUANTITY | COILS | LADING NO | SHIPMENT DATE |
|---|---|---|---|---|---|
| BF WAREHOUSE | | 6,241 Lbs | 169 | 00011319 | 06/19/2014 |

SPECIFICATION 625-0450-12BP-B00
HAYNES® 625 RTW™ WIRE, 0.0450, 12" DIN 300 Spool, 30.0 lbs, Bright,

CERTIFICATION REQUIREMENTS
UNS N06625
AMS 5837G
AWS A5.14:2009 & 2011 ERNiCrMo-3
ASME SFA-5.14:2004 & 2013 ERNiCrMo-3
BS EN10204:2004 3.1

**Chemical**

| | | | | |
|---|---|---|---|---|
| C | Mn | Fe | P | S |
| 0.01 | 0.03 | 0.22 | <0.003 | <0.001 |
| Si | Cu | Ni | Co | Al |
| 0.05 | <0.01 | 64.5 | 0.02 | 0.11 |
| Ti | Cr | Cb+Ta | Nb/Ta | Mo |
| 0.18 | 22.36 | 3.62 | 3.623 | 8.75 |
| TOE | | | | |
| <0.50 | | | | |

**Mechanical by Lot**

| TEST | UNITS | HIGH | LOW | AVERAGE |
|---|---|---|---|---|
| Diameter | In | 0.0438 | 0.0435 | 0.0436 |

| TEST | RESULT |
|---|---|
| Surface Check | Pass |

HI-TECH INDUSTRIAL SERVICES, INC.
DATE 10/2/14
APPROVED BY QA/QC

This is to certify that all required samplings, inspections and tests have been performed in accordance with the specification requirements. The test report represents the actual attributes of the material furnished and the values shown are correct and true. The material described by this certificate is in full compliance with all order and inspection requirements. We hereby certify that the above data are in accordance with the specification requirements.

Allon Batson
Quality Manager

Page 1

Date Printed 06/30/2014

# APPENDIX D:  HEAT TREATMENT REQUIREMENTS

**Stress Relief of Cladded Inconel 625 on ASTM A387 Grade 22 Steel Plate**
**Statement of Work**
**2016-03-14**

## SCOPE OF WORK

Concurrent Technologies Corporation (CTC) is requesting to conduct stress relief treatment of a steel plate cladded with Inconel 625 alloy.  The steel plate (ASTM A387 Grade 22) has approximate dimensions of 48 in. x 22 in. x 1.25 in. and one face has the cladded a layer.  The Inconel 625 cladded layer has a thickness of approximately 0.3 in and it was produced by the weld overlay process.  The stress relief shall be conducted using the following parameters.

1. The stress relief of the plate shall be conducted in vacuum or in an inert atmosphere.
2. The furnace atmosphere shall be completely free of sulfur, sulfur compounds and other contaminants such as carbon, phosphorous, lead, zinc and carbon containing compounds.
3. Prior to stress relieving the plate shall be free of oil, grease and other contaminants.
4. The plate shall be in a horizontal position with the cladding face up in the furnace.
5. No paint or ink markings shall be made on the cladded plate
6. The cladded plate shall be stress relieved at 1075 °F ± 25 °F and held for 4 (four) hours at temperature.
7. The heating ramp up shall be at 100 °F per hour.
8. The cooling rate shall be equivalent to air cool to below 400 °F.
9. The stress relieved plate shall be properly crated and shipped to CTC at the following address.

    128 Industrial Park Road
    Johnstown, PA  15904-1942
    Attention:  Juan Valencia

## SCHEDULE AND DELIVERABLES

The heat treating facility shall provide the cladded steel plate clean and damage free.  Also, an electronic and/or hardcopy of the thermal history during the stress relief process shall be provided to CTC.

## POINTS OF CONTACT

CTC technical point of contact information is provided below.  The test facility shall identify their technical POC upon award of purchase order.



| | |
|---|---|
| Juan J. Valencia<br>Concurrent Technologies Corp.<br>100 CTC Drive<br>Johnstown, PA  15904-1935<br>Phone/Fax:  814-269-2552<br>valencia@ctc.com | Michael Tims<br>Concurrent Technologies Corp.<br>100 CTC Drive<br>Johnstown, PA  15904-1935<br>Phone/Fax:  814-269-2515<br>tims@ctc.com |

# APPENDIX E: HEAT TREATMENT CERTIFICATION

SOLAR ATMOSPHERES INC.

Solar Atmospheres of Western PA

**Certification**

**Order No.:** 109330
**Date:** 04/04/2016
**Entry Date:** 03/30/2016
**Page:** 1 of 1

**To:**
CONCURRENT TECH. CORP.
100 CTC DRIVE
JOHNSTOWN, PA 15904-1935

**Purchase Order No.:** 160300129
**Packing List No.:**
1 **Material:** INCONEL 625

All work performed subject to Solar Atmospheres Terms Of Sale as presented on form TOS-SAWPI.

| Quantity | Part Number / Part Name / Part Description | Pounds |
|---|---|---|
| 1 | MATERIAL INCONEL 625<br>CLADDED STEEL PLATE<br>REQ: VAL3211403 / 48" X 1.5" X 22' | |

| Insp. Type | Scale | Minimum | Maximum | Number | Other |
|---|---|---|---|---|---|
| **Customer Requirements:** | | | | | |
| Visual | | . | . | . | |

THIS IS TO CERTIFY THAT THE ABOVE LISTED PARTS WERE PROCESSED IN ACCORDANCE WITH YOUR PURCHASE ORDER REQUIREMENTS AND SCOPE OF WORK 1.0 DATED 03/14/2016.

FURNACE RUN#: 70-9098-6004

RAMPED AT 100°F (MAX.) TO 1075°F ±25°F
HELD AT 1075°F ±25°F FOR 4 HOURS +10 / -0 MINUTES
ARGON COOLED

This certification is no guarantee of material performance, properties, or microstructure. Mechanical, physical, and/or metallurgical testing is not performed unless specifically itemized on your purchase order to Solar Atmospheres.

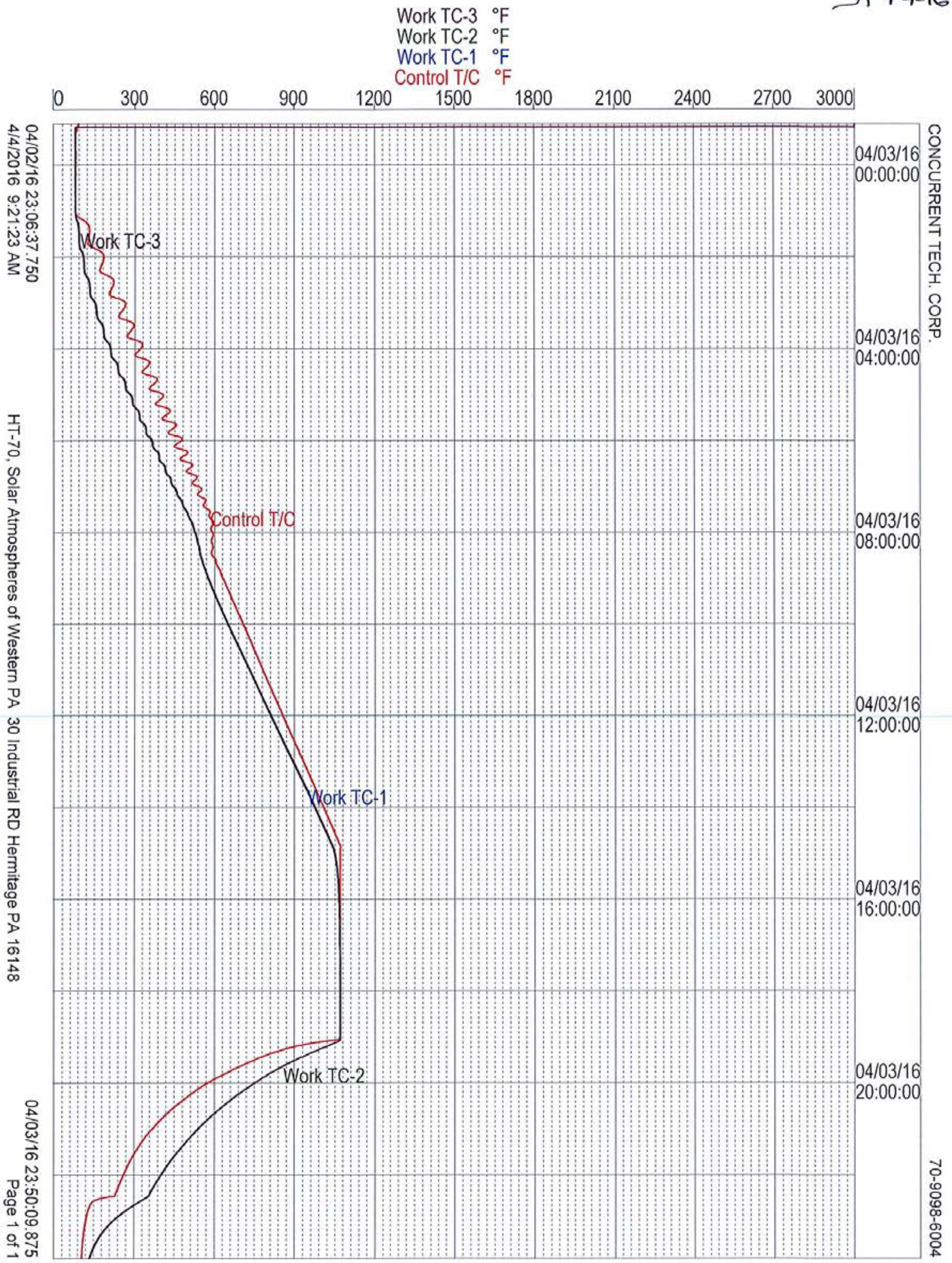
Jack Hardesty

Jack Hardesty
Special Projects Coordinator /
SOLAR ATMOSPHERES INC.

The recording of False, Fictitious, or Fraudulent statements or entries on this document may be punished as a felony under federal statutes including FEDERAL LAW. TITLE 18, CHAPTER 47.

30 Industrial Road Hermitage PA 16148 **Phone:** 724-982-0660 **Fax:** 724-982-0593

H 44-16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Work TC-2 °F
Work TC-1 °F
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Page 1 of 1
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CONCURRENT TECH. CORP.
70-9098-6004
Work TC-3
Control T/C
Work TC-1
Work TC-2