Gas Tungsten Arc Welding of 304L and 21-6-9 Austenitic Stainless Steel with Penetration Enhancing Compounds

THESIS

Presented in Partial Fulfillment of the Requirements for the Degree Master of Science in the Graduate School of The Ohio State University

By

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The Ohio State University
2014

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Abstract

The penetration capability of gas tungsten arc (GTA) welding of stainless steels is influenced greatly by small amounts of trace elements, such as sulfur and oxygen. Due to the small amounts of sulfur in modern austenitic stainless steels, a shallow weld penetration occurs through a surface tension related phenomena, known as the Marangoni effect. Increasing the penetration efficiency and reducing variations of GTA welds has been investigated since the 1960’s using a number of different oxide combinations including the three used in this study; TiO₂, SiO₂, and Cr₂O₃. ASM has identified these oxides as penetration enhancing compounds (PEC) and the process has been given the name A-TIG or activated TIG. The use of PEC’s has been shown to increase penetration in GTA welding through arc constriction and surface tension driven fluid flow resulting in a favorable Marangoni effect for penetration. The process of adding PEC’s to the GTA welding process has relied upon a paint based application method requiring additional processing steps and adequate use of quality control methods to maintain paint deposition consistency. Precise control is necessary to reduce the amount of weld penetration variability. The development of a 308L composition PEC cored wire eliminates the additional steps required during setup of the paint on process, and allows for consistent weld pool application.
A comprehensive analysis of the A-TIG process and microstructure of welds with PEC additions were evaluated using 304L and 21-6-9 base materials. PEC flux cored wire of 0.045” diameter was used along with 308L solid wire for comparison of weld microstructures. A small loss in penetration capability with the use of cored wire was surpassed through ease of application. A penetration enhancement of 293% was measured in welds of low sulfur 304L. A single pass, full penetration tube weld on 304L of 3/8” wall thickness was successfully developed. Internal pressurization of the backing gas was required and achieved using a multi-stage pressure regulator on the supply of argon, with a back pressure regulator and Magnehelic® gauge on the exit side of the weld purge system. A back pressure of 3 to 5 inches of water column (InWC) was required to support the 3/8” full penetration weld puddle in the weld joint.

Microstructural analysis of the resultant welds was completed using standard metallographic preparation techniques various etching techniques. Optical microscopy, macro and micro hardness indentations, as well as ferrite content analyses were used to examine the welds. Advanced microscopy and chemical analysis techniques were also used, including scanning electron microscopy, electron dispersive spectroscopy and laser ablation mass spectroscopy. Increased alloying of PEC constituents in full penetration welds resulted in a max of 0.78 wt% titanium measured at the root of the weld. Partial penetration welds of similar heat input have reduced alloying of PEC constituents resulting in a max of 0.03 wt% titanium. The entrapment of slag elements or formations of intermetallic compounds were not seen in welds with PEC addition.
Dedication

To my lovely wife Katie, for her patience and limitless support.
Acknowledgments

Thanks to my advisor John Lippold for being the person I didn’t want to let down, and whom I looked up to for advice and stewardship. In addition, thanks to Dave Farson for supporting myself and the project from its early conceptual days through completion. A special thanks to Adam Hope, David Tung, Ryan Smith, and Daniel Tung for the advice, friendship, help, and support they provided throughout my pursuit of this degree.

I would like to thank Honeywell FM&T for the opportunity to chase down a personal goal and complete it while maintaining employment. The technical fellowship program and plant directed research and development programs were instrumental in making this possible. Without the support of my manager, Mark Mehl, and 2nd level manager, Brian Heineman this would not have been possible.

Additional thanks are needed for support from David Weiliczka, and Tammy Pond at Honeywell FM&T’s Analytical Laboratory for helping with Energy Dispersion and Laser Ablation Spectroscopy.
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Fields of Study

Major Field: Welding Engineering
Table of Contents

Abstract.......................................................................................................................................... ii

Dedication ...................................................................................................................................... iv

Acknowledgments......................................................................................................................... v

Vita.............................................................................................................................................. vi

Fields of Study ............................................................................................................................... vii

Table of Contents ........................................................................................................................... viii

List of Tables ................................................................................................................................. xi

List of Figures ............................................................................................................................... xiii

Chapter 1 : Introduction / Objective ............................................................................................ 1

1.1 : Process Characterization ........................................................................................................ 4

1.2 : Material Characterization ...................................................................................................... 4

1.3 : Full Penetration Capability .................................................................................................. 5

1.4 : Arc Constriction Verification .............................................................................................. 6

Chapter 2 : Background ................................................................................................................ 7

2.1 : Penetration Enhancement ..................................................................................................... 7

2.2 : Ferrite Content ..................................................................................................................... 8
List of Tables

Table 1: PEC Oxides with Reported Penetration Enhancement [3, 4] .........................8
Table 2: Tensile Tests of 304 Austenitic Stainless Steel Welds [8] ........................ 12
Table 3: EDS Results from SEM image of 304L Weld Center Region [12] ...............13
Table 4: BCC and FCC Interstice Spacing's [32] ......................................................33
Table 5: Atomic size of Interstitial Elements in Iron [32] ........................................33
Table 6: Solubility of Carbon and Nitrogen in Austenite and Ferrite [32] ...............33
Table 7: 304L 1/2" Thick Plate Material Specs .......................................................50
Table 8: Material Composition .............................................................................58
Table 9: Material Chemistry ................................................................................67
Table 10: Material to Material Weld Penetration Variability .................................78
Table 11: Tube Material Chemical Composition ....................................................84
Table 12: Full Penetration Study (Magnehelic® gauge measures: inches of water
        column) .........................................................................................................88
Table 13: Properties of Oxides[41] .......................................................................101
Table 14: Material Chromium and Nickel Equivalency Values .............................102
Table 15: 304L, Solidification Mode vs. % Nitrogen Addition, Balance: Argon ......138
Table 16: 21-6-9 Solidification Mode with Nitrogen Additions to Shielding Gas,
        Balance: Argon ..........................................................................................147

xi
Table 17: Initial Weld Trial Data .................................................................185
Table 18: Experimental Design with Data .....................................................186
Table 19: MC308L, PEC Flux Cored Wire Composition ................................187
Table 20: Wire Feed Speed Tests - 304L Plate ..............................................188
Table 21: Material Variability Data ...............................................................190
Table 22: Full Penetration 3/8" Tube Weld Data .........................................191
Table 23: Material Compositions .................................................................192
Table 24: SEM, EDS Summary of 300 Amp, Autogenous Partial Penetration
    Weld ........................................................................................................193
Table 25: SEM, EDS Summary of 300 Amps, 20 ipm PEC Cored Wire Addition,
    Partial Penetration Weld .........................................................................193
Table 26: SEM, EDS Summary, 200 Amp Autogenous Partial Penetration Weld ....193
List of Figures

Figure 1: A) Typical U-Groove type joint (1/4” Wall Thickness), B) 3/8” wall thickness completed weld using narrow U-Groove joint design ....................3

Figure 2: Ferrite Number Reported with use of PEC [3] .................................................................................10

Figure 3: Mechanical and Corrosion Properties using SS-7 PEC. Hardness Plot is of 316L welds. [7, 10] ...................................................................................11

Figure 4: SEM Image of 304 Weld Center Region [12] .........................................................................................13

Figure 5: Surface Tension Gradient A) Negative, B) Positive Marangoni Effect [13] ..........................................................16

Figure 6: Penetration vs. Oxygen Content –volume% Shielding Gas [15], and ppm content [18] .................................................................17

Figure 7: Effect of Oxygen Concentration on Surface Tension [17] .................................................................19

Figure 8: Fe-O Phase Diagram of Equilibrium O-contents due to the dissociation of different oxides [17] .................................................................................20

Figure 9: Penetration efficiency of c) Chromium, d) Silica, and e) Titanium oxides [23] .........................................................................................21

Figure 10: Size of Oxide particles effects contribution to Oxygen content. A) 0.8µm, B) 4µm, C) 25µm [22] .................................................................................22

Figure 11: Arc Constriction model by Howse and Lucas [24] .................................................................................24
Figure 12: Top Row: Without PEC, Bottom Row With PEC addition:  A) GTAW, B) PAW, C) Laser, D) Electron Beam [24] ..........................................................25

Figure 13: Recorded Images of Arc to quantify arc constriction [3] ........................................26

Figure 14: Schematic of Keyhole mode A-TIG weld [25] ................................................27

Figure 15: Weld Pool Depression due to Lorentz Forces without PEC [28] ..................28

Figure 16: a) BCC b) FCC atom positions [13] ................................................................31

Figure 17: Tetrahedral and Octahedral Sites in a) BCC and b) FCC [13] ..........................32

Figure 18: Iron - Carbon Phase Diagram [14] ................................................................35

Figure 19: M23C6 Carbide Precipitation Curves [34] ................................................36

Figure 20: Iron - Chromium Phase Diagram [32] ............................................................37

Figure 21: Strengthening effects through alloying additions[33] ....................................41

Figure 22: Relative Strength of Alloying Elements a) Ferrite Formers b) Austenite

Formers [38] ........................................................................................................42

Figure 23: Fe-Cr-Ni Psuedo-Binary Phase Diagrams A) Solidification modes

(70% Fe) B) Composition and Temperature [9, 29] .................................................43

Figure 24: Cracking Susceptibility via Solidification Mode [29] ....................................44

Figure 25: Jetline 9500 Gantry System with Thermal Arc 400GTSW Power

Supply ..............................................................................................................48

Figure 26: 3/32” Thoriated Electrode ........................................................................50

Figure 27: Experimental Design Penetration Response Plot ........................................51

Figure 28: Pareto Chart and Main Effects Plot – Penetration ....................................53

Figure 29: Main Effects Plot - Penetration ................................................................54
Figure 30: Interaction Plot of Penetration .................................................................55
Figure 31: Histogram of Weld Penetration (Autogenous vs. PEC Deposition) ............56
Figure 32: Histogram of Weld Width (Autogenous vs. PEC Cored Wire) ...................56
Figure 33: Metallographic Sections from DOE .........................................................57
Figure 34: 308L Penetration Box Plot ......................................................................59
Figure 35: 308L Experimental Design Results, A) Pareto Analysis, Width B) Pareto Analysis, Penetration C) Interaction Plot, Penetration D) Main Effects Plot, Penetration .................................................................60
Figure 36: Metallographic Sections from DOE ..........................................................61
Figure 37: Application Steps for Paint on PEC [4] .....................................................62
Figure 38: Paint on PEC ...........................................................................................63
Figure 39: Paint on PEC - After Scraping to 0.3 mm ...............................................64
Figure 40: Paint vs. Cored Wire Deposition, Penetration and Weld Width ...............65
Figure 41: No PEC, 300 Amps, 11 Volts, 2.5 ipm Travel Speed (1/2" plate thickness) .........................................................................................................................66
Figure 42: 300 Amps, 11 Volts, 2.5 ipm Travel Speed (1/2” plate thickness) A) Paint on PEC ~0.033 g/inch, B) Cored Wire PEC, 20 IPM WFS ...........66
Figure 43: Wire Feed Speed Test: 275 Amps, 13.5 Volts, 4 ipm Travel Speed ............69
Figure 44: Penetration vs. Wire Feed Speed, 200 Amps ............................................71
Figure 45: Penetration vs. Wire Feed Speed, 300 Amps ............................................71
Figure 46: Penetration Contour Plot - Max 200 Amps – 4 IPM Travel Speed .............73
Figure 47: Penetration Contour Plot – Max 300 Amps - 4 IPM Travel Speed ..........73
Figure 48: Weld Surface Photographs of Nitronic 40 and 304L Welds.........................77

Figure 49: Penetration vs. Heat Number with and without additions of PEC flux cored wire..........................................................................................................................79

Figure 50: Representative Cross Sections of Material to Material Variability.............81

Figure 51: Effect of Sulfur Content on Penetration (commercial 304L plate)............82

Figure 52: Current vs. Weld Joint under Evaluation ....................................................83

Figure 53: Machined Tube Samples: Wall Thicknesses, ¼” (Left) and 3/8” (Right) .................................................................................................................................84

Figure 54: Weld Tube Setup with Compression Purge Gas Setup (A), and Silicon Baffle System (B). ........................................................................................................85

Figure 55: Catastrophic Weld Drop-Thru Failure .......................................................86

Figure 56: 3/8” Full Penetration Scatterplot – Amps vs. Travel Speed (ipm)..............89

Figure 57: Metallographic Cross Sections of Full Penetration Weld Trials. A) 200 Amps, 11.5 Volts, B) 200 Amps, 11 Volts, C) 200 Amps, 10.5 Volts, D) 250 Amps, 10.5 Volts, E) 275 Amps, 10.5 Volts. Wire Feed Speed was varied for all welds from 20 IPM to 5 IPM. ........................................90

Figure 58: 3/8” Wall Thickness - 250 Amps, 11 Volts, 62E) 4 InWC, 62D) 5 InWC ..............................................................................................................................91

Figure 59: Full Penetration Welds with Varying Internal Pressure (275 Amps, 11 Volts)..................................................................................................................92

Figure 60: Weld Reinforcement with Slag Layer .........................................................93

Figure 61: Sample 64C root of weld.............................................................................93
Figure 62: Internal Weld Surface (Clean in foreground, oxidized in background) ..........94

Figure 63: Full Penetration Weld, Sample 48E, 9.5 mm wall thickness, 275 amps, 10.5 volts, 3ipm linear travel speed, 5ipm wire feed speed, 4.5 InWC ..........97

Figure 64: Solidification Mode, Pseudobinary Phase Diagram Relationship [29] ........100

Figure 65: DeLong Diagram, 304L only, [43] ........................................................................................................105

Figure 66: WRC-1992 Diagram, 304L only [29] ........................................................................................................106

Figure 67: Hull Diagram [44] .........................................................................................................................107

Figure 68: Espy Diagram with 21-6-9 heat locations [45] ..................................................................................108

Figure 69: Ferrite Number vs. Consumed grams of PEC per linear inch of weld. Paint and Wire addition (300 Amps, 11 Volts, 2.5 IPM Travel Speed) .........................................................................................................................111

Figure 70: Ferrite Number vs. Wire Feed Speed (200 Amps, 300 Amps) ......................................................112

Figure 71: 304L Base Material and Filler Material Ferrite Number Comparison (55 = autogenous, 55-308L = 308L solid wire addition, 55F = PEC cored wire addition). .........................................................................................................................113

Figure 72: Ferrite Numbers for 304L Heats .................................................................................................114

Figure 73: Ferrite Numbers for 21-6-9 Heats .................................................................................................116

Figure 74: Nitronic 40 Ferrite Number Comparison .........................................................................................117

Figure 75: Commercially available ½” thick plate welded autogenously at, 275 Amps, 13.5 Volts, 4 IPM Travel Speed, Originally 200x magnification .........................................................................................................................119
Figure 76: Commercially available ½” thick plate welded with 5ipm PEC cored wire addition at, 200 Amps, 11 Volts, 2.5 ipm Travel Speed. SEM image (QBSD, 20kV, 9.5mmWD) (7.7 FN measured on top surface of weld) ...............................................................119

Figure 77: 304L Fusion Boundary ...................................................................................121

Figure 78: 304L Transition Region .............................................................................121

Figure 79: Weld Morphology at Center of Weld .........................................................121

Figure 80: Sample 55, 304L, A: Autogenous B: Cored Wire Added (200x) .............123

Figure 81: 304L with PEC flux cored wire addition ..................................................123

Figure 82: Autogenous: 200 Amps, 0.050" Arc Gap, 4ipm Travel Speed (1000x Optical Magnification, Chromic Acid Etchant) ........................................124

Figure 83: PEC Flux Cored Wire Addition (1000x Optical Magnification, Chromic Acid Etchant) .................................................................124

Figure 84: Solidification Mode - A) Hull, B) DeLong [36] ........................................126

Figure 85: Hull Diagram with predicted ferrite content[44] .......................................126

Figure 86: Weld Morphology at Fusion Boundary of Nitronic 40 ............................128

Figure 87: Weld Morphology at Transition Region of Nitronic 40 ...........................128

Figure 88: Weld Morphology at Weld Center of Nitronic 40 ....................................128

Figure 89: Autogenous Weld, Nitronic 40 - Center of Fusion Zone, 400x Original Magnification .................................................................................129

Figure 90: Cored Wire Added to Nitronic 40 - Center of Fusion Zone, 400x Original Magnification .................................................................................129
Figure 91: Sample 56C, root of weld at 100x original optical magnification

Figure 92: Sample 56C, Transition in solidification direction, Skeletal Ferrite

Figure 93: Rockwell B Hardness Indents: 1/16" ball, 100kgs weight – 304L

Figure 94: Rockwell B Hardness Indents: 1/16" ball, 100kgs weight - Nitronic 40

Figure 95: Micro hardness Linear Segment

Figure 96: Nitronic 40, 6 Pass GTA weld

Figure 97: 304L Single Pass Full Penetration, Single Pass GTA weld

Figure 98: 304L Autogenous Welds with Nitrogen Shielding Gas Additions

Figure 99: 304L Welds with PEC Flux Cored Wire Additions and Nitrogen

Figure 100: 304L 308L Filler Wire and Nitrogen Shielding Gas Additions

Figure 101: 304L Unmixed Zone with Cored Wire Addition, Ar +10N₂ DIC prism

and polarizer used, ferritic phase is white

Figure 102: 304L with Cored Wire Added, A) Ar +5%N₂ B) Ar +10%N₂

Figure 103: Micro Hardness Values Across Unmixed Zone of 304L

Figure 104: 21-6-9 Autogenous Welds with Nitrogen Shielding Gas Additions

Figure 105: 21-6-9 Welds with PEC Flux Cored Wire and Nitrogen Shielding Gas Additions

Figure 106: 21-6-9 with 308L Filler Metal and Nitrogen Shielding Gas additions to Argon Shielding Gas

Figure 107: Micro Hardness Traverse across Unmixed Zone of Nitronic 40 Alloy

Figure 108: Alloy 21-6-9 with 5% Nitrogen added to Shielding Gas
Figure 109: Solidification Mode near Center of Welds - Optical Micrograph -

1000x Original Magnification.................................................................................152

Figure 110: Analysis of PEC ...........................................................................................154

Figure 111: Avg. Compositional Analysis Values (wt%) through EDS at

Analytical Laboratory, Kansas City.................................................................156

Figure 112: Example EDS Trace .....................................................................................157

Figure 113: EDS trace of slag adhered to surface of weld. (Rectangular scan area)......158

Figure 114: Titanium slag elements found in mount removed from surface of weld

(Rectangular scan area)............................................................................................158

Figure 115: FEI Quanta 200 at Center for Electron Microscopy and Analysis

(CEMAS)[48].............................................................................................................159

Figure 116: EDS Analyses of Slag Remnants, spot and full frame.........................160

Figure 117: Full Penetration EDS Analysis.................................................................162

Figure 118: Element Trace Information of Full Penetration Welds .......................163

Figure 119: Solidification Growth Directions of Partial and Full Penetration

Welds  (A: Sample 5A Transverse, B: Sample 48E Transverse, C:

Sample 64C Longitudinal Section)..............................................................................165

Figure 120: Comparison of Welding Arcs, A) Autogenous, B) with Paint on oxide

layer, C) with flux cored wire addition.........................................................................167

Figure 121: Image Intensity Evaluation...........................................................................168

Figure 122: Representative Sample of Captured Images by WeldQC Camera

System, Autogenous Weld.......................................................................................169
Figure 123: Surface Active Oxides Present in Circulation of Pool (Clockwise) ..........170
Figure 124: Thermo Scientific X Series 2 ICP-MS with New Wave Laser [50] ..........172
Figure 125: Sample 36 after analysis by Laser Ablation Mass Spectroscopy ..........173
Figure 126: Residual Titanium Content Using ICP-MS with Laser Ablation, 200 Amps ..........174
Figure 127: Residual Titanium Content using ICP-MS with LASER Ablation, 300 Amps ..........174
Figure 128: Residual Silicon Content in Partial Penetration Welds, 200 Amps ..........175
Figure 129: Residual Silicon Content in Partial Penetration Welds, 300 Amps ..........175
Figure 130: Sample 34, 300 Amps, Autogenous .........................................................194
Figure 131: Sample 36, 300 Amps, 10ipm PEC Cored Wire Addition .........................194
Figure 132: Sample 34, 300 Amp Autogenous, Weld Center ........................................195
Figure 133: Sample 36, 300 Amps, 10ipm PEC Cored Wire Addition, Weld Center .........................195
Figure 134: 308L Solid Wire Composition Ranges ..........................................................196
The use of gas tungsten arc (GTA) welding in the joining of materials is essential in many industries to maintain weld cleanliness and quality requirements of thick joint sections. Many of these applications are on tubular components with circular cross sections. This choice of processes is due to the cleanliness of the root pass and subsequent fill passes. GTA welding is preferred in full penetration welds over processes such as plasma arc, electron beam and laser in which issues of spatter and keyhole mode can affect internal components. These can be overcome through use of backing features and step features; however these cannot always be used and can have negative impacts on mechanic stress concentrations, crevice corrosion resistance and machining costs. Issues that limit the use of gas tungsten arc welding are the shallow depth of penetration, high heat input, and variations in penetration. This is especially true in the use of austenitic stainless steels in which variations in penetration are commonly caused by small variations in base metal constituents such as sulfur and oxygen. This phenomenon is known as the Marangoni effect and is discussed further in Chapter 3. Steps have been taken in many industries using austenitic stainless steels to control the sulfur content of the base material at additional cost. The variation in penetration is hard to avoid in the production process when refurbishment of older components using less restrictive heats of material end up in the standard process.
The typical full penetration gas tungsten arc weld joint without a backing feature is machined to include a U-groove as shown in Figure 1A. This typical design is also used in thicker sections, requiring the same small land thickness for root penetration. A completed weld of 3/8” joint thickness is shown in Figure 1B. The typical number of welds passes to fill a u-groove joint varies depending upon machining tolerances and weld parameters used however five to six passes are typically specified for ¼” joint thicknesses and seven to eight for 3/8” wall thickness joints. The number of passes has a direct effect on process cycle time and critical applications typically involve small root welds to control joint distortion and excessive penetration. The root weld pass is also typically run at low arc currents to allow for control of the welding process. With improvements in penetration and reduced variability between welds of different material heats, a more efficient process could provide reduced cycle time through welding. Increasing the efficiency of the GTA process has included the use of modified equipment to focus current towards a direct contact anode grounding probe as well as with additions of oxide compounds on the top surface of the weld. Modifications to the weld process cannot always be used a final closure weld with limited access or small inner tube diameters are used. The use of oxides known as penetration enhancing compounds (PEC) allow for the increase in penetration efficiency of gas tungsten arc welding. The addition of oxide compounds to a welding process using tightly controlled base material compositions requires additional scrutiny of PEC material alloying contents and slag evolution through the welding process. The use of a flux cored wire consisting of PEC
and 308L nominal composition will be used in the weld analysis of partial and full penetration welds.

Figure 1: A) Typical U-Groove type joint (1/4” Wall Thickness), B) 3/8” wall thickness completed weld using narrow U-Groove joint design

Previous articles investigating the use of penetration enhancing oxides during GTA welding have conclusively shown improvement to weld penetration. The goal of this project is to investigate the process capability, mechanical properties, and evaluate the effects of PEC use on the weld microstructure of austenitic stainless steels. Since the weld metal composition is not expected to be modified through use of PEC additions, the mechanical properties are not expected to be altered to a significant extent. To test the material weldability, cleanliness of weld deposits, and process efficiency two austenitic material types were selected. 304L and 21-6-9 were selected due to their widespread use in industry.
1.1: Process Characterization

Commercially procured heats of hot rolled 304L with low and high sulfur content were used in ½” and 1” thick, 4” x 8” plates. Bead on plate welds with the aid of experimental designs were used to determine process variable influence and penetration capability of GTA welding with PEC flux cored wire additions. Amps, Volts, Travel Speed, and PEC additions using flux cored wire and paint on applications were varied to measure penetration responses. Comparisons were then made to similar welds completed autogenously and with 308L solid wire addition. Minitab and JMP were used to analyze the data recorded from metallographic cross sectioning and SEM analysis of weld metal deposits. As found in this study the penetration enhancement measured was up to 293%, very near the published maximums reported in literature up to 300%.

1.2: Material Characterization

Six heats of 304L and four heats of 21-6-9 forging material were used in addition to the hot rolled 304L plate material to determine penetration variability as influenced by small variations in material chemistry. Process parameters were fixed to allow comparison between heats of material and welds made autogenously, with 308L solid wire addition, and with PEC flux cored wire addition. Fifty welds were made between 304L and 21-6-9 to evaluate the material variability and resulting weld microstructures. Welds were evaluated for ferrite content, hardness, and solidification microstructure. Ferrite content was measure by magnetic permeability using a Fischer Feritscope FMP30. Hardness values were measured through selection of representative samples for macro Rockwell B
hardness indentation and micro Vickers hardness indentations. Linear traverse and planar hardness maps were produced to evaluate hardness of the weld region. Solidification microstructure analysis consisted on optical and electrical microscopy using oxalic, chromic, and nitric acid etching techniques along with energy dispersion spectroscopy in the SEM for chemical analysis of welds with and without PEC alloying additions. Laser ablation mass spectroscopy was also used on samples with and without PEC addition at low and high heat inputs to determine alloying content of PEC constituents. The addition of nitrogen shielding gas was included to determine the feasibility of using small additions of nitrogen to the coaxial shielding gas for reduction of ferrite content in welds with PEC addition. Welds were made of matching weld parameters with additions of 2, 5, and 10 volume % nitrogen and argon on 304L and 21-6-9 with and without PEC’s. The addition of PEC’s to the arc welding process allowed for additional retention of nitrogen in the weld metal vs. welds with 308L solid wire and autogenous welds. This was seen through the formation of porosity near the unmixed zones in 304L welds only when using PEC’s, and the reduction in retained weld metal ferrite content. This reduction in ferrite content occurred in both 304L and 21-6-9, resulting in measurements below 3FN, a lower limit usually used to avoid crack susceptibility.

1.3: Full Penetration Capability

Full penetration welds were developed using 1/4” and 3/8” wall thickness 304L tubing. Square butt joints were machined onto 5” outside diameter, 2” long tube sections for testing of full penetration capability. The use of backing gas pressurization was required
to maintain the weld pool in the weld joint of 3/8” wall thickness joints. Single pass full penetration welds were accomplished with root and surface reinforcement using 4 to 5 inches of water column (InWC) or 0.11 to 0.18 psi backing pressure. Weld chemical analysis revealed additional alloying content in full penetration welds of 0.78 wt% titanium vs. 0.03 wt% in partial penetration welds of similar heat input. The entrapment of slag elements or intermetallic formations such as silicon or titanium carbides or nitrides was not seen in any weld cross sections. Weld solidification structure and ferrite content of the full penetration welds were type III FA mode with a ferrite contents averaging 6.5 FN.

1.4: Arc Constriction Verification

Arc constriction has been said to occur for welds using PEC’s. Techniques involving light spectroscopy to measure the amount of arc constriction have not been well published due to the many different wave lengths of light emitted and asymmetry of the recorded data. During testing using a weld vision camera system a small cylindrical arc column could be seen between the electrode tip and work piece. Through image analysis of captured images of autogenous welds and welds with paint on and wire fed PEC addition a constriction of the arc plasma column was recorded. A 16% reduction in the diameter of the arc column was measured in welds with PEC addition at a value just above FWHM of the image intensity. This is thought to be more accurate than measurements using saturated images of the arc; however this method does exclude much of the arc equaling lesser intensity.
Chapter 2: Background

The use of surface active elements to enhance penetration in GTA welds initiated from the Paton Welding Institute in the former Soviet Union during the 1960’s [1]. The primary materials that have seen benefits through use of this technology are: titanium, nickel alloys, low carbon / low alloy steels, and austenitic stainless steels. Select terminology has been developed for these compounds and the process using them. Many terms have been used in the past referring to the compounds as a flux, or mixture of oxides, but are now widely known as penetration enhancing compounds (PEC). Use of PEC in gas tungsten arc welding has been identified as a process called activated TIG or A-TIG [2]. The constituents making up the PEC has been closely guarded by manufacturers of these compounds and is protected as proprietary information with patent control. PEC compositions are primarily based on oxides, halides, and metal powder compounds and vary by the material application [3].

2.1: Penetration Enhancement

For stainless steels a mixture of oxides are primarily used. EWI and Liburdi use titanium based oxides, with additions of SiO₂ and Cr₂O₃ in the EWI manufactured PEC[2]. A review of current published literature has revealed a variety of different oxides evaluated for penetration efficiency, summarized in Table 1. Of these, TiO₂ is the most commonly
referenced oxide internationally, and forms the basis of many commercially available compositions. Results of tests conducted using the various flux compounds generally produce similar results. A maximum penetration enhancement of near 300% has been reported by different investigators using different combinations of oxides [3-6].

Table 1: PEC Oxides with Reported Penetration Enhancement [3, 4]

<table>
<thead>
<tr>
<th>PEC</th>
<th>Penetration Enhancement</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>295%</td>
</tr>
<tr>
<td>TiO₂</td>
<td>214%</td>
</tr>
<tr>
<td>MoO₃</td>
<td>273%</td>
</tr>
<tr>
<td>MnO₂</td>
<td>204%</td>
</tr>
<tr>
<td>ZnO</td>
<td>280%</td>
</tr>
<tr>
<td>Cr₂O₃</td>
<td>Not Available</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>Not Available</td>
</tr>
</tbody>
</table>

The application of PEC to increase penetration efficiency has been evaluated for many years with limited application. A resurging interest in the application of PEC for GTA welding has occurred as more companies are trying to increase production efficiency.

2.2: Ferrite Content

The effect on ferrite content is important for a number of reasons including crack susceptibility and corrosion resistance. For 304L welds with gas tungsten arc welding a ferrite number (FN) between 5 and 10 is expected. The addition of oxides, namely
Chromium, titanium, and silicon will impart some added alloying content to the weld metal. Because of the ferrite stabilizing tendency of these alloy additions an increase in ferrite content is expected. Ferrite content on welds of 300 series welds were reported to increase by as much as 20%, 25%, and 54% compared to autogenous welds of the same arc parameters [3-5]. Micrographs of various oxide weld deposited welds can be seen in Figure 2. An increase in ferrite content was reported in all papers reviewed with additions of TiO$_2$, and SiO$_2$ oxides. Measurements were taken through magnetic permeability using a Feritscope on the top surface of the completed welds and averaged.

Analyses completed on ferrite content of welds focus on the heat input of welds with PEC increasing due to increased arc voltage. The greater heat input is deemed responsible for the increased ferrite content [3-5].

Reports through EWI, the manufacturer of the PEC used in testing reported insignificant influence on ferrite content in austenitic stainless steels [6, 7]. Development of welds of full thickness penetration reported a decrease in weld joint ferrite content when comparing to a 7 pass weld completed with the addition of 308L solid filler metal [8]. This is due to the composition difference between 304L and 308L.

Ferrite content of austenitic stainless steel welds is important for corrosion properties and mechanical properties. A ferrite number between 3 and 10 is desired for resistance to
solidification cracking as well as prevent mechanical property degradation at high temperature that can occur above 10 FN [8, 9].

![Figure 2: Ferrite Number Reported with use of PEC][3]

2.3: Mechanical Properties

Welding of Austenitic Stainless Steels under normal processing conditions with arc welding results in the formation of retained ferrite through weld solidification. This was captured in data shown above through measurements of ferrite content. The amount of
ferrite present has an effect on the mechanical properties of the resultant weld joint. Tests conducted to evaluate the hardness and tensile properties of welds using SS-7 PEC are shown below. Type 316L and 304 austenitic stainless steels were tested. Improvements were seen in all welds when comparing to autogenous welds of the same base metal, as shown in Figure 3. Type 316L samples welded autogenously resulted in a hardness value of 168 HVN. The same arc parameters with oxide additions resulted in a slightly higher hardness of 183 HVN, appreciably lower than the base metal hardness of 254 HVN. Critical pitting resistance was also found to increase slightly with PEC additions [7].

![Figure 3: Mechanical and Corrosion Properties using SS-7 PEC. Hardness Plot is of 316L welds. [7, 10]](image)

Tensile testing of 316L and 304L austenitic stainless steels resulted in minor changes in tensile strength in welds with PEC addition compared to conventional welds with 308 filler metal additions, as shown in Figure 3 and Table 2. All samples failed in the fusion zone with little change in ultimate tensile strength. Values of 81.5 and 82.6 ksi UTS were obtained. Bend tests were also completed successfully.[8]

<table>
<thead>
<tr>
<th>Material</th>
<th>Condition</th>
<th>Tensile Strength (ksi)</th>
<th>Hardness (HVN)</th>
<th>Critical Pitting Resistance (CPT, °C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2507</td>
<td>No Flux w/ N₂ Purge</td>
<td>126</td>
<td>294</td>
<td>47.7</td>
</tr>
<tr>
<td>2507</td>
<td>No Flux w/ Ar Purge</td>
<td>125</td>
<td>300</td>
<td>39.0</td>
</tr>
<tr>
<td>2507</td>
<td>Fluoride + Oxide</td>
<td>130</td>
<td>290</td>
<td>48.7</td>
</tr>
<tr>
<td>2507</td>
<td>Oxide</td>
<td>141</td>
<td>315</td>
<td>55.0</td>
</tr>
<tr>
<td>316L</td>
<td>Base Metal</td>
<td>164</td>
<td>355</td>
<td>86.3</td>
</tr>
<tr>
<td>316L</td>
<td>No Flux</td>
<td>89</td>
<td>168</td>
<td>6.9</td>
</tr>
<tr>
<td>316L</td>
<td>Fluoride + Oxide</td>
<td>79</td>
<td>240</td>
<td>0.7</td>
</tr>
<tr>
<td>316L</td>
<td>Oxide</td>
<td>92</td>
<td>183</td>
<td>1.8</td>
</tr>
<tr>
<td>316L</td>
<td>Base Metal</td>
<td>106</td>
<td>254</td>
<td>13.7</td>
</tr>
<tr>
<td>254 SMO</td>
<td>No Flux</td>
<td>92</td>
<td>211</td>
<td>0.5</td>
</tr>
<tr>
<td>254 SMO</td>
<td>Fluoride + Oxide</td>
<td>111</td>
<td>231</td>
<td>49.9</td>
</tr>
<tr>
<td>254 SMO</td>
<td>Oxide</td>
<td>114</td>
<td>221</td>
<td>45.0</td>
</tr>
<tr>
<td>254 SMO</td>
<td>Base Metal</td>
<td>157</td>
<td>376</td>
<td>60.9</td>
</tr>
</tbody>
</table>
Table 2: Tensile Tests of 304 Austenitic Stainless Steel Welds [8]

<table>
<thead>
<tr>
<th>Weld Type</th>
<th>Ultimate Strength (ksi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conventional (NNS-SS1 and NNS-SS1)</td>
<td>85, 87.3, 87.6</td>
</tr>
<tr>
<td>Flux Root w/Cap Pass (SS7T-15 and SS7T-13)</td>
<td>83.4, 83.4, 83.0</td>
</tr>
<tr>
<td>Flux Root (SS7T-14 and SS7T-21)</td>
<td>81.5, 81.6, 81.8</td>
</tr>
<tr>
<td>Nonflux (NNS7T-2)</td>
<td>82.2, 82.6, 82.6</td>
</tr>
<tr>
<td>Base Metal (035-005NN)</td>
<td>85.2, 85.9, 85.9</td>
</tr>
<tr>
<td>ASTM A312 Minimum</td>
<td>75</td>
</tr>
</tbody>
</table>

2.4: PEC Entrapment

Very little information is available on optical microscopy or spectroscopy techniques being utilized to evaluate PEC additions to welds. No external publications could be found using advanced microscopy techniques on evaluation of the weld metal. X-ray diffraction patterns and energy dispersive spectroscopy have been used to analyze oxide layers and ferrite content was found in literature provided by Loureiro[5], and Huang[11]. The only information containing weld metal data was provided in a report through EWI internal research and development. EWI reported low levels of titanium in the body and root of welds tested and were reported to be within the levels present in the base material, 0.03 to 0.04 wt% [12]. Additional evaluation of weld metal oxide entrapment is necessary. See Table 3 and Figure 4 for SEM and EDS results from the weld center.
Table 3: EDS Results from SEM image of 304L Weld Center Region [12]

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight%</th>
<th>Atomic%</th>
</tr>
</thead>
<tbody>
<tr>
<td>C K</td>
<td>1.69</td>
<td>7.28</td>
</tr>
<tr>
<td>Si K</td>
<td>0.56</td>
<td>1.04</td>
</tr>
<tr>
<td>Ti K</td>
<td>0.03</td>
<td>0.04</td>
</tr>
<tr>
<td>Cr K</td>
<td>18.70</td>
<td>18.65</td>
</tr>
<tr>
<td>Mn K</td>
<td>1.82</td>
<td>1.71</td>
</tr>
<tr>
<td>Fe K</td>
<td>69.09</td>
<td>64.13</td>
</tr>
<tr>
<td>Ni K</td>
<td>8.11</td>
<td>7.16</td>
</tr>
<tr>
<td><strong>Totals</strong></td>
<td><strong>100.00</strong></td>
<td><strong>100.00</strong></td>
</tr>
</tbody>
</table>

Figure 4: SEM Image of 304 Weld Center Region [12]
There are several mechanisms that have been investigated for penetration enhancement using penetration enhancing compounds. Many investigations into determining the primary mechanism have explored two main driving forces: Marangoni flow, and arc constriction. There are multiple theories involved with arc constriction including, quasi-keyhole and cathode focused GTA welding. The primary focus of research has been on the fluid flow and arc constriction theories. Most reports investigating mechanisms of enhanced penetration have focused on proving a single mechanism while disqualifying the other. The continued publication of reports identifying alternate primary mechanisms demonstrates the need for better understanding of each with evaluation of synergetic effects. A review of penetration enhancing mechanisms is provided below.

3.1: Marangoni Effect

The Marangoni effect describes the change in surface tension of a liquid with temperature. A fluid either increases in surface tension with temperature or decreases, directly influencing the motion of the fluid when in non-equilibrium conditions. The affected fluid will want to move to areas of greater surface tension, therefore when the surface tension gradient is positive with increased temperature the flow of material will be to the center of the weld pool, towards the heat source. A negative surface tension
gradient will have the opposite effect, in which the low temperature fluid at the edges of the weld pool will be of higher surface tension, causing the outward flow of material from the center. This is shown schematically in Figure 5. Marangoni fluid flow is a mechanism initially discussed by Heiple and Roper for variations in penetration of 21-6-9 by minor element effects in GTA welds [13]. Elements such as sulfur, selenium, aluminum, and oxygen were found to influence the surface tension of the weld pool. To no surprise the oxygen content in welds with oxide additions increases. This has been recorded by many investigators using chemical analysis on welds with and without PEC additions in stainless steels [7, 8, 10]. Oxygen is also known to effect the Marangoni flow of welds [14], therefore is the primary suspect in the mechanistic evaluation of fluid flow when adding PEC to the weld. The addition of oxygen and the resulting Marangoni effect have been recorded in additions through oxide dissolution and shielding gas additions [15]. The penetration enhancing effect of oxygen can be seen in Figure 6. In all cases the effect of oxygen occurs through arrange of about 70-300 ppm weld metal content [16].

The addition of Oxygen through decomposition of oxides is more complex, invoking not only oxygen content, but surface active elements through unconsumed oxide particles, and slag components. A model has been developed called the surface phase transition (SPT) model by Sandor, Mekler, Doranszky, and Kaptay [17]. Calculations of oxide particle dissolution to absorbed oxygen content was calculated and experimentally measured to be 33% and 34% respectively with a linear relationship to oxide density [17].

[17]
Figure 5: Surface Tension Gradient A) Negative, B) Positive Marangoni Effect [13]
The SPT model consists of a surface wetting phenomenon discussed originally by Cahn, to explain the wetting characteristics of multiple solutions [19]. The SPT model discusses the role of a Fe-O thin film fully wetting the surface of the molten pool. When this occurs, the surface tension of the weld pool is affected by the liquid / gas interface. As shown in Figure 7, the sign of the surface tension gradient is altered by the oxygen content corresponding to the wetting of the surface by a film of Iron oxide. It has been found that penetration is more affected by the sign of the surface tension gradient than the numeric calculated value [17, 20]. An interfacial force is applied to the surface of the weld pool according to Equation 1.

The temperature gradient from the center of the weld pool to the outside edges is negative, \( \frac{dT}{dr} \), therefore a negative value for \( \frac{d\sigma}{dT} \) results in a positive force, \( F_\sigma \) away from...
the center of the weld pool. If the oxygen content reaches a critical value the sign of the surface tension gradient changes and the force reverses, causing an inward flow. The application of oxygen to the weld pool then becomes the next concern.

\[ F_\sigma = A \frac{d\sigma}{dT} \frac{dT}{dr} \]

Equation 1: Force applied onto surface through Marangoni Effect [21]

Many oxide species have been tested for effectiveness in penetration enhancement, see Figure 2. The efficiency of these oxide powders varies and can be related to the oxygen dissolution temperatures. Figure 8, plots dissociation amounts of various oxides with the SPT line for iron. According to this plot the dissociation of anatase (TiO\(_2\)) provides just enough oxygen along the entire range of temperatures to alter the surface tension of the weld pool. Silica, SiO\(_2\), is another experimentally efficient oxide composition that lies to the right of the SPT line providing sufficient oxygen at all temperatures. Excess oxygen is provided in the case of silica, reducing the magnitude of the surface tension gradient. A lesser Marangoni force is expected through calculations.[17]
Figure 7: Effect of Oxygen Concentration on Surface Tension [17]
Many tests have been done to determine the optimum oxide density needed to apply to the weld joint for penetration efficiency. Tests conducted by S. Lu using TiO₂, Cr₂O₃, and SiO₂, revealed optimum levels of penetration at limited oxide additions. TiO₂ provided consistent penetration with increased oxide content, see Figure 9. The use of SiO₂ and Cr₂O₃ resulted in penetration maximums oxygen contents of 500 to 600 ppm in the weld metal. The process of dissolution and alloying into the weld pool is a complicated process that relies upon not only the amount of oxide, but also the size of the oxides. The effect of particle size on penetration efficiency is shown in Figure 10 for SiO₂ of 0.8µm, 4µm, and 25µm. Maximum penetration efficiency was seen at the two small oxide particle sizes with varying decays in penetration behavior. The large 25µm particle size resulted in a
negligible increase in penetration at all oxide densities tested. The smaller oxide particle sizes allow for more efficient transfer of Oxygen into the weld metal. [22]

Figure 9: Penetration efficiency of c) Chromium, d) Silica, and e) Titanium oxides [23].

The process of using PEC’s has been rumored to be troublesome due to variations during welding. The proper transition from the research and development labs to the production environment is essential for the successful application of the paint on process. Due to variations with oxide particle size and deposition density, weld penetration variations are likely without constant oversight and strong quality control measures in place.
The mechanism of Marangoni flow has demonstrated its involvement in the penetration mechanism activated by using penetration enhancing compounds. Oxygen plays a large role in the influence of weld pool convection.

Figure 10: Size of Oxide particles effects contribution to Oxygen content. A) 0.8µm, B) 4µm, C) 25µm [22]
3.2: Arc Constriction

There are three main theories of arc constriction that are commonly presented in literature. The theories consist of formation of a plasma column, keyhole mode, and electrically isolating surface oxide. The main arguments of each theory will be discussed. Arc constriction as studied by Howse and Lucas [24], involve arc ionization effects amplified through the application of PEC’s to form a plasma column. A modified theory of Simonik’s electron absorption theory was used to evaluate the increased penetration capability. This theory relies upon the concept of four regions of the GTA arc.

1. Plasma Column: Current carried by the electrons produced through ionization of the shielding gas.
2. Anode/Cathode: High potential drop between the anode and cathode to maintain the flow of current.
3. Cathode: Thermionic emission is provided through bombardment by positive ions.
4. Anode: Electrons transfer kinetic energy to the work piece.
Figure 11: Arc Constriction model by Howse and Lucas [24]

Howse and Lucas concluded their results after experimentation was completed using oxide and halogen compounds with GTA, plasma arc, laser and electron beam welds. Arc constriction was deemed the dominant mechanism through comparison of the GTA welds to those utilizing higher arc densities. Little to no enhancement in penetration was seen in the PAW, Laser, and Electron Beam welds. The use of PAW, Laser, and EB change the partial pressures involved at the liquid gas interface of the weld pool. This change also would affect the transfer efficiency of the oxygen to the molten pool. Although the tests conceptually appear to be worthwhile, the comparisons appear to be biased by years of electron beam experience.
A simpler approach to defining arc constriction has been taken by other investigators. Figure 13, shows images captured through video recording of welds using PEC compounds. The conical shape present in welds can be measured and compared to determine if the arc width changed during welding with PEC’s. Measurement techniques published are crude but show a narrower arc profile is present when using PEC’s. These results are typical of published material.

The keyhole mode theory is a subset of the plasma column theory that has reemerged with publications in the last decade as a mechanism of penetration enhancement. Nearly all papers reporting keyhole mode or quasi-keyhole have come from the Paton Welding Institute. Claims of power densities reaching $10^4$ W/cm$^2$ have been made, with reference to weld pool depressions shown schematically in Figure 14.[25, 26]
Another approach to the arc constriction mechanism focuses, like Marangoni flow, on the surface of the weld pool. The formation of an oxide rich layer on the surface of the weld pool creates an electrically isolating film. No experimental work defines this theory; however the net effect is arc constriction. The electrically isolating flux on the surface of the weld restricts the cathode to a small surface area in the center of the weld pool. This

Figure 13: Recorded Images of Arc to quantify arc constriction [3]
is similar to the method of cathode focused GTA welding in which a second electrode is
placed on the root side of the weld focusing the current from the arc source through the
secondary electrode. This method has proven to be valid [27].

![Figure 14: Schematic of Keyhole mode A-TIG weld [25]](image)

The arc constriction theories have one mechanistic feature in common. Lorentz forces are
generated in GTA welding without the use of PEC’s and cause depression of the weld
pool, increasing penetration. An extreme form of utilizing this process is called buried arc
GTA welding. An article by Rokhlin and Guu investigated arc parameters and a term
called arc force, relating Lorentz forces to weld pool depression. A schematic of arc
depression measured by Rokhlin is shown in Figure 15. Weld pool depressions of up to 4
mm were recorded at 350 amps. The use of the term, keyhole, is normally restricted to
processes such as PAW, laser, and electron beam welding. During the mechanistic
evaluation of A-TIG welding, there are a few researchers who mention a transition
between dominant penetration mechanisms dependent upon arc parameters. Just as Rokhlin and Guu reported, a transition occurs in which penetration increases more rapidly due to the depression of the weld pool [28]. This transition was at 200 amps, for the combination of electrode size, and material in tests conducted by Rokhlin. The interactions of the plasma column and weld pool are affected by the current density, surface tension, and density of the weld pool [28].

![Figure 15: Weld Pool Depression due to Lorentz Forces without PEC [28]](image)

There is an arc column interaction occurring during the process of A-TIG welding. The increase in arc voltage of approximately 0.5 to 0.8 Volts has been reported by many authors [3, 7, 8, 12]. More investigations need to be completed to determine the exact reason for arc constriction.
Stainless steels are an expensive subset of steels with intentional alloying additions to balance properties with increased corrosion resistance. The corrosion resistance is improved through the addition of Chromium which forms an oxide layer on the surface of the alloy, shielding the steel below from exposure to corrosion environments. A value of 12% Cr or more is defined as the start of the stainless steels. However this does not imply immunity. In the welding of stainless steels, the main forms of corrosion that occur are through mechanisms termed intergranular corrosion (IGC) and stress corrosion cracking (SCC) [29]. To understand the metallurgical principles involved, an understanding of steel and stainless steel alloying effects is needed. This document will cover the basics of stainless steel metallurgy with a focus on alloying additions and resulting effects of PEC compound constituents.

4.1: Basic Structure of Steels

Every metal consists of atoms that are orientated in specific ways. The positions of atoms and the way they are stacked in relation to one another can vary between and within the same metal groups. Atomic arrangements that make up a solid can be categorized into one of 14 crystallographic orientations. These crystal structures are based on the unit cell, which is the smallest group of atoms that if repeated would form the overall structure of
the crystal lattice [30]. Steels consist of two primary crystal structures, body centered cubic (BCC) and face centered cubic (FCC). Atom positions for these two crystal structures are depicted in Figure 16. These crystal structures are often identified by their Greek symbols in steel alloys as, \( \alpha \), and \( \gamma \) for BCC and FCC respectively. Both structures consist of eight atoms at the corners of the cubic structure. In a body centered cubic atomic structure an atom is located centrally to the cubic unit cell. The face centered cubic structure contains the same 8 corner atoms with additional atoms at the center of each face of the cubic cell. These atom positions primarily consist of the parent material atom, in this case iron. Substitution of these atom locations occurs through alloying additions in which for example, a chromium or nickel atom may take the place of an iron atom in the crystal structure forming a substitutional solid solution.

Not all alloying additions replace solvent atoms at lattice points depicted by the bcc and fcc structure. You can imagine the structure is similar to a ball pit in which smaller atoms may intertwine with the bcc or FCC structure and position themselves between the atoms making up the unit cell. In the bcc and FCC crystal structure there are two common sites termed, interstitial sites in which an atom may occupy. These are shown pictorially in Figure 17 for clarity.
The strength of a material can be influenced greatly by occupation of these interstitial sites. This strengthening mechanism is significant in the bcc atomic structure where shear stresses imparted by the interstitial element react strongly with dislocation movement [31]. The FCC atomic structure has a weak strengthening effect due to shear. In the iron based unit cells, α-iron is more loosely packed than γ-iron, with larger atomic spacing’s at tetrahedral sites between the edge and central atoms of joining unit cells. As it turns out, the FCC structure is more tightly packed than the BCC structure; however maintains a larger interstitial spacing located at the octahedral sites, shown in Figure 17(b) and numerically in Table 4.
The distances between atoms in the unit cell can vary depending upon the size of the atoms in each atomic position, however are dominated by the principle element. In iron systems the interstitial spacing’s for BCC and FCC are shown in Table 4. Due to the size limitations of the interstitial atomic positions, only very small elements may reside at these locations. The most common of these elements are Carbon and Nitrogen. Potential interstitial elements can be seen in Table 5.
The addition of carbon to iron forms the basis of steel metallurgy. Through additions of carbon, the strength of iron increases dramatically. A phase diagram of Fe-C can be seen in Figure 18. Chemical solubility of carbon and nitrogen varies between the two phases, $\alpha$ and $\gamma$. Solubility of carbon and nitrogen is greater in austenite as expected from the size of potential interstitial sites. These solubility limits, listed in Table 6, are exploited to produce carbides through heat treatment, adding a secondary strengthening mechanism to the iron alloy. As shown in Figure 18 the formation of Fe$_3$C occurs at concentrations
greater than 0.02 wt% in ferrite at 723°C. Carbon will remain in solution in the austenitic phase field up to 2 wt% at 1147°C. Through non equilibrium cooling, such as during welding, super saturation of \( \alpha \)-iron (ferrite) can occur in which the formation of carbides will result. This occurs due to the ease of diffusion of carbon and nitrogen at room temperature in BCC ferrite. The lower solubility of carbon in ferrite and greater rate of diffusion due to the less dense atomic packing factor of BCC crystal structures allows for the formation of iron carbides preferentially at \( \alpha-\gamma \) grain boundary interfaces [32]. Limiting the amount of carbon in the base material has a direct effect on carbide precipitation as seen in Figure 19. A decarburizing process is now used to obtain very small amounts of carbon less than 0.03 wt%. A low carbon designation, L, is added to steels that utilize this process, such as 304L. [32, 33]

There is much more that could be discussed in terms of carbon content and phase morphologies in steels. Stainless steels, particularly 304L and 21-6-9 investigated in this study purposely limit carbon content to avoid carbide precipitation and sensitization. The book Steels, by Bhadeshia and Honeycombe is recommended for additional information about steel metallurgy.
Figure 18: Iron - Carbon Phase Diagram [14]
4.2: Stainless Steels

Stainless steels are a subset of steels that take advantage of chromium additions for corrosion resistance. Above 12 wt% Cr, a stable oxide layer will develop on the surface of the alloy resulting in a protective coating. At 13 wt% a fully ferritic microstructure can be maintained from solidification to room temperature, however is subject to a ductile to brittle transformation [32]. An Iron-Chromium phase diagram can be seen in Figure 20.

With additions of $\gamma$ stabilizers, the gamma loop can be expanded to obtain a fully austenitic microstructure at room temperature. Additions of Nickel and Manganese are commonly added to expand the gamma loop for austenitic alloys. The most common of these is the 18Cr-8Ni, 300 series of alloys.
Sigma phase (σ) seen in Figure 20, can significantly reduce mechanical properties of the material. The formation of σ phase occurs over long periods of time in chromium rich areas usually near austenite grain boundaries. Elements such as titanium and molybdenum achieve a further acceleration of sigma formation. [29, 32]

There are many alloying elements added to stainless steels to affect phase balance, affecting mechanical and corrosive properties. Each alloying element typically can be divided between two categories of α or γ stabilizers. As seen above the addition of these elements can be balanced to either expand the gamma loop of the Fe-Cr diagram or
decrease it. With increased alloying additions, the alloy system can become complicated quickly. The use of pseudo-binary phase diagrams becomes important in determining phase stability and weld microstructure evolution.

4.3: Ferrite Stabilizers (α)

Chromium is added primarily for corrosion resistance through the formation of an oxide layer on the surface of the steel protecting the material from attack. The formation of chromium rich carbides such as M23C6, depletes the surrounding area of chromium leading to corrosion sensitive areas. This process is called sensitization. Because the chromium is no longer in solution and is tied up in large concentrations in the carbide, the protective film breaks down leading to intergranular corrosion.[29]

Silicon is also a ferrite stabilizing element when added at concentrations greater than 1 wt%. Concentrations of silicon are usually kept below 1 wt% due to its known segregation behavior and formation of low melting eutectics and iron silicides. It is commonly an intentional addition in filler metals to improve fluidity of the weld puddle. [29]

Titanium is a potent ferrite stabilizing element due to its strong affinity for carbon and nitrogen, neutralizing these strong austenite stabilizers. Alloys that utilize titanium additions are called stabilized grades due to the high temperature formation of Ti2C and Ti2N which are very stable. The formation of these carbides is often used for precipitation
hardening due to the fine dispersion of carbides that remains at high temperatures, resulting in good creep resistance. The formation of carbides at high temperatures also acts as a grain refiner in weld deposits pinning grain boundaries. [29, 32, 33]

Molybdenum is also a strong ferrite stabilizer with benefits to corrosion resistance. The addition of Molybdenum suppresses active corrosion sites by forming a oxy-hydroxide or molybdate [35]. Additions of up to 6 wt% are used to improve pitting and crevice corrosion resistance with improved high temperature strength. This increase in strength can make the alloy more difficult to hot work. A carbide former itself, it also has an effect on the kinetics of nucleation and growth, slowing carbide formation in steels. [29, 32]

4.4: Austenite Stabilizers (γ)
Nickel is the primary austenite stabilizer used in stainless steels. The combination of Cr and Ni promotes the retention of austenite to room temperature.

Manganese is an austenite stabilizing element that is potent at low concentrations, but diminishes at concentrations above 4-6% [36]. Manganese improves resistance to solidification cracking through removal of low melting point elements such as sulfur [36]. Manganese is also added to increase the Nitrogen solubility which can reach 0.4 wt%. [29]
Carbon and Nitrogen are potent austenite stabilizers and solid solution strengtheners. The potent solid solution strengthening of common alloying additions can be seen in Figure 21. Increasing amounts of nitrogen improve the resistance to localized corrosion and intergranular corrosion due to precipitation of the Cr$_2$N nitride causing less chromium depletion in the vicinity of the grain boundaries. Nitrogen additions are found in some base metals of austenitic grades, but are primarily found in duplex stainless steels. Welding of nitrogen bearing steels such as 21-6-9 (Nitronic 40) causes desorption of the nitrogen if insufficient austenite is formed at high temperature. The solubility of nitrogen is lower in ferrite causing the loss of nitrogen during welding. This is often overcome by the addition of nitrogen in the shielding gas of duplex stainless steel welds. Nitrogen has also been known to inhibit the precipitation of intermetallic compounds such as sigma phase. Increased corrosion resistance was found in nitrogen bearing steels through increased dissolution of iron freeing chromium near the surface which blocks active corrosion. [35-37]

The relative strength of stabilization through alloying elements is shown graphically in Figure 22. The strong austenite stabilizing elements, carbon and nitrogen are shown greatly surpassing the effects of other austenite stabilizers. As for ferrite stabilizers it is not surprising that some of the strong ferrite stabilizers are also strong carbide and nitride formers essentially negating the effects of carbon and nitrogen.
Nitrogen has been used in austenitic stainless steels containing manganese additions to increase solubility in austenite. 21-6-9 (Cr-Ni-Mn) austenitic stainless steel takes advantage of this increased solubility by reducing the amount of Ni needed to maintain austenite at room temperature. The trade name Nitronic is given to a series of alloys that utilize nitrogen alloying additions. The 21-6-9 material used in this study is a Nitronic 40 alloy. The strength of steels with nitrogen additions are greatly improved as seen by the strong effect on yield stress in Figure 21.
4.5: Welding of 304L

304L has been a very commonly used austenitic stainless steel due to its very tolerant welding characteristics, strength, toughness, and corrosion resistance. The material is based on the 18Cr-8Ni alloy series with less than 0.02 wt% Carbon content. Few metallurgical precautions must be taken with 304L. Through use of predictive diagrams, such as the WRC-1992 and Delong diagrams, weld metal ferrite content can readily be calculated by alloy content. The materials included in this scope of work were plotted on the WRC-1992 diagram shown in Figure 66, resulting in a residual ferrite content of 6 to 10 wt% or Ferrite Number (FN). Solidification mode is predicted by the ratio of chromium stabilizing elements to austenite stabilizing elements known as Chromium and Nickel equivalents. Through use of pseudo-binary phase diagrams based on the Fe-Cr-Ni
ternary system the solidification mode for arc welding can be predicted. As shown in Figure 23b, the solidification mode of a 20Cr-10Ni alloy is predicted to initiate as primary ferritic solidification, known as delta ferrite (δ). This initial solidification of delta ferrite is rich in chromium, approximately 26 wt% upon initial solidification and decreases to nominal chromium content as the temperature approaches the solidus temperature. The last ferrite formed through solidification did not undergo the drastic segregation and enrichment in chromium as the initial delta ferrite. A solid state transformation of delta ferrite to austenite will occur as the material cools within the two phase field of α and γ. The later ferrite to solidify having near nominal Cr composition will transform to austenite leaving only the initial delta ferrite enriched in chromium to remain. A skeletal ferrite and austenite weld microstructure is common in 304L arc welds.

Figure 23: Fe-Cr-Ni Pseudo-Binary Phase Diagrams A) Solidification modes (70% Fe) B) Composition and Temperature [9, 29]
The primary ferrite and secondary austenite solidification mode, with nomenclature FA is desirable in austenitic stainless steel welding due to its low cracking susceptibility. Cracking susceptibility is qualitatively expressed in Figure 24, in which cracking susceptibility of austenitic stainless steels in drastically reduced through type FA solidification mode. Because solidification mode can be altered by cooling rate, not all welding processes will exhibit the same solidification mode in the same steels. Laser and Electron beam welding applications typically require higher chromium and nickel equivalency to avoid solidification cracking. Cracking susceptibility cannot be correlated to retained ferrite content [39].

Figure 24: Cracking Susceptibility via Solidification Mode [29]
4.6: Welding of 21-6-9

21-6-9 has a few significant differences when comparing to 304L. The most important of these differences is the addition of nitrogen in the base metal. As shown earlier this has a drastic effect on the strength of the material. A difficulty in the welding of these alloys has been the retention of nitrogen in weld metal deposits. Due to the preferential weld solidification through primary ferrite, nitrogen evolution from the weld metal occurs due to the lower solubility of nitrogen in ferrite and low partial pressure above the weld pool [35]. Retention of weld metal nitrogen under normal gas tungsten arc welding with 100% argon shielding gas requires fast cooling rates to allow formation of a sufficient quantity of austenite to maintain nitrogen content. This can be achieved in processes such as laser and electron beam. Low heat input GTA welds may be used; however, production rates would be impacted severely. A careful balance must therefore be taken to control nitrogen desorption from the weld pool, requiring consideration for heat inputs and cooling rates. To maintain the nitrogen contents after welding, 2 to 5 % nitrogen by volume is sometimes added to the shielding gas. This is primarily done in duplex welding where a 50/50 phase balance is sought. The addition of excess nitrogen can have a detrimental effect on formation of porosity in welds and chromium nitrides, depleting the chromium content [35].
4.7: Corrosion of Austenitic Stainless Steels

Austenitic stainless steels are often chosen due to their corrosion resistance, however can become sensitive to two main types of corrosion: intergranular corrosion (IGC), and stress corrosion cracking (SCC) [29]. Intergranular corrosion typically occurs in austenitic stainless steels when the alloy has been said to be “sensitized”. In effect the material after welding has imparted a thermal gradient in the heat affected zone (HAZ) that precipitates chromium rich carbides, \( \text{Cr}_2\text{C}_6 \), at grain boundaries. The depletion of chromium from the surrounding matrix in the HAZ causes the material to be sensitive to intergranular corrosion. Stress corrosion cracking can also occur in austenitic grades of stainless steel. Specifically alloys with a nickel content between 5 and 20 wt% are susceptible [29]. With SCC you must satisfy three criterions for cracking to occur; stress, corrosive environment, and susceptible material. The materials, 304L and 21-6-9, both are susceptible, and with welding can have sufficient restraint. Minimization of weld stress through post weld heat treatments or joint designs can be used to avoid SCC. Proper material selection for chlorine environments is pivotal to avoid SCC. [29]
Weld testing was completed at the Welding Engineering Laboratory in the department of Materials Science and Engineering at The Ohio State University. A Jetline 9500 system controller with mechanized gantry and rotary axis were used. Welding was completed in the 1G welding position for bead on plate and rotary tube welds. Photographs of the equipment can be seen in below in Figure 25. 2% Thoriated electrodes were used in all tests of either 3/32”, or 1/8” diameter. 100% Argon was also used for all tests unless otherwise mentioned.

The PEC tested in this analysis is produced by EWI as DeepTIG®, in their SS-7 formulae and provided in metallic powder and cored wire forms. The exact composition is proprietary, but consists of: TiO$_2$ (20 to 60%), Cr$_2$O$_3$ (0-50%), and SiO$_2$ (0-20%). The cored wire consumable consists of 3% PEC, 7% Fill ratio of components by weight in 0.045” wire. The wire was manufactured to be nominally 308L composition by Electric Welding Limited (ESAB) in Hanover, PA.

Weld parameter development relies upon an understanding of the process and each variable that influences the end product. This is well understood when using the conventional gas tungsten arc welding process. No publications could be found to
describe the parameter interactions when using a PEC cored penetration enhancing wire. The addition of wire to the molten weld pool causes a cooling effect which may affect the maximum penetration of the weld. Traditionally, the root pass, is completed autogenously to ensure full penetration, followed by additional fill weld passes using a solid consumable weld wire. The use of the DeepTIG wire introduces filler wire to the root pass, without joint preparation. The target material for this study was 304L, a common commercially available stainless steel. A design of experiments was then conducted to determine the process variability and influence on weld penetration, width, and reinforcement.

Figure 25: Jetline 9500 Gantry System with Thermal Arc 400GTSW Power Supply
Initial tests with the wire were based on previous experience with the gas tungsten arc welding process. Voltage values were tested from 9 to 17 volts to determine a suitable arc height for wire addition. Eleven volts was determined to be the minimum value used for the design of experiments. Lower values of voltage resulted in very small arc heights that were below the surface of the original plate material and caused wire feeding and arc stability issues. Values for voltage, during fill welding can typically be greater than 17 volts. This allows for a greater arc height allowing the addition of wire to be readily accomplished at high deposition rates. High deposition rates were not sought, and after a few initial welds, a value of 13 volts was settled upon for the design of experiments. The arc voltage controller was used in this experiment to give practical weld development responses.

5.1: Experimental Design

An understanding of the variables and their interaction efficiencies was sought through the use of a standard 3 variable, 2 factor experimental designs. Variables used were; Amps, Volts, and Wire Feed Speed. The weld matrix can be seen in Table 18. Arc parameters were varied between 150 to 200 Amps, 11 to 13 Volts, and 0 to 20 ipm of wire feed speed. All tests were conducted using 4 ipm travel speed, 100% Argon shielding gas, and a 3/32” electrode with 5° electrode tip angle, as shown in Figure 26. To baseline the weld penetration response, 308L solid wire was used in addition to autogenous welding. These two methods will allow for comparison to a baseline standard for penetration and weld metallurgy.
Initial welds were completed on 4” x 8” x ½” 304L plate material. The base material specifications can be seen below in Table 7. Bead on plate welds were utilized to eliminate influence of the parameter response due to small joint variances. Two quasi-independent experimental designs were completed combining the data extracted from the autogenous welds with the PEC cored and solid 308L welds independently. Welds were first made between welds with PEC cored wire addition and autogenous welds.

<table>
<thead>
<tr>
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<th>P</th>
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<th>Co</th>
<th>Mo</th>
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</table>

Table 7: 304L 1/2” Thick Plate Material Specs

Penetration was maximized through increased amperage with wire feed speed. Although no determination could be made on the efficiency of the PEC due to the near constant...
penetration response at all wire feed speeds, the effect of PEC addition was dramatic. A box plot of penetration values can be seen in Figure 27. Penetration of approximately 3.76 mm was achieved throughout the range of currents and voltages, with the addition of PEC, resulting in a large process window.

Several techniques were used to determine the process variable influence and interdependency relationships between input variables. When the experimental design was analyzed through a Pareto analysis to determine significant factors, amperage, wire...
feed speed or the combinations of the two were not statistically significant factors influencing penetration. This could not be resolved through the scope of this design of experiment. In terms of the practical response during welding, it is evident that a significant increase in penetration was found when using higher amperage and wire feed speed. Penetration increases of 220% at 150 amps, 11 volts, and 232% at 200 Amps, 13 volts were measured. Of importance to note is that although not statistically significant, wire feed speed, therefore PEC concentration had a larger effect on weld penetration than amperage, voltage, or any combination of weld parameters. This holds true for welds up to 200 amps, in which arc force is not a significant penetration mechanism. Above 200 amps the influence of arc force on weld pool depression increases penetration without the aid of PEC’s. This increase in the Lorentz forces above 200 amps reducing the efficiency of the PEC’s. Further analysis of these parameters is needed to delineate the penetration response between the variables of this experimental design. Additional analysis tools, such as a main effects plot and interaction plot can reveal tendencies for parameter influence on a desired output. These tools were used to calculate the parameter influence on penetration discussed below.

When welding with PEC, voltage did not play a significant role in penetration depth when varied between 11 and 13 volts. This was not the case when welding without PEC. A decrease of 19% penetration was seen without PEC at 200 Amps. A main effects plot, Figure 29, shows the individual parameter effectiveness whereas the interaction plot, in Figure 30, allows for additional analysis of net effects at the extreme values of the
experimental design. The data was analyzed primarily for penetration response. Other responses such as weld width, reinforcement, and aspect ratio were recorded and analyzed. Please see the appendix for this information.

![Figure 28: Pareto Chart and Main Effects Plot – Penetration](image)

It was found that wire feed speed has the most significant influence on penetration in this experiment. Not surprisingly, current was close behind. The ability of the PEC to provide efficient penetration at increasing voltage is significant for gas tungsten arc welding. There are several potential advantages that can be achieved through exploitation of this capability. This is important when considering the application of the PEC through wire addition. The additional arc height needed for weld wire addition does not inhibit or decrease the penetration capability of the wire. Although a slight negative effect can be
seen in Figure 30, the effect was not noticeable in the welds with PEC addition. This negative effect can be attributed to the welds without PEC. Weld width changed drastically when adding PEC to the weld pool. A maximum weld width of 10.4 mm was measured at 13 volts and 200 Amps. For comparison the width was reduced to 8.8 mm when PEC was added at 20 ipm wire feed speed. This resulted in a range of weld widths from 7.1 mm to 10.4 mm for welds without PEC addition. When PEC was added to the weld, the weld width was reduced to an average 6.5 mm. A narrower weld width and higher weld aspect ratio was recorded. Histograms of the penetrations and weld widths are shown in Figure 31, and Figure 32.

Figure 29: Main Effects Plot - Penetration

![Main Effects Plot for Penetration (mm)](image)

Figure 29: Main Effects Plot - Penetration
Although a limited number of samples exist in this DOE, a distinct population difference can be seen for welds with PEC vs. autogenous welds. This effect was drastic and very noticeable as welded as shown in Figure 33. The mechanisms that increase penetration using PEC’s are discussed in Chapter 3.

Figure 30: Interaction Plot of Penetration
Figure 31: Histogram of Weld Penetration (Autogenous vs. PEC Deposition)

Figure 32: Histogram of Weld Width (Autogenous vs. PEC Cored Wire)
<table>
<thead>
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<td>Sample 1C</td>
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Figure 33: Metallographic Sections from DOE
5.2: 308L Solid Wire Addition

A 25 pound spool of wire was purchased from Harris Products of 0.045” diameter, solid 308L wire. The current process that is being evaluated for use of penetration enhancing compounds through cored wire addition uses solid 308L fill passes to complete the weld joint. Evaluation of the PEC cored wire vs. Autogenous welds and 308L solid wire is needed to fully characterize and evaluate the process. The PEC cored wire supplied by ESAB is nominally 308L in composition. Penetration was increased with addition of 308L solid wire with no PEC. Weld widths also were slightly reduced. The increase in penetration when using 308L filler metal may have been due to an increase in Sulfur content or through the disturbance of the weld pool due to the addition of the wire.

<table>
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Table 8: Material Composition

The penetration response on the 308L solid wire is shown in Figure 34. Penetration was greatest at maximum current, wire feed speed and lowest voltage. The increase in penetration with filler metal addition is not typical however are not significant. The addition of sulfur in the welding wire is added to minimize variations in penetration that have caused significant issues when welding of stainless steels.
A summary of the experimental design responses can be seen in Figure 35. Review of A and B reveal results typical of gas tungsten arc welding. Current had the highest influence on penetration and weld width. Although weld width is normally correlated to arc voltage, it is dependent upon electrode size, conical angle, tip diameter, and travel speed. Travel speed for these tests was relatively fast at 4 ipm, (1.69 mm/sec) therefore current had a larger impact on weld width. For stainless steels with low sulfur content a shallow and wide weld penetration profile is to be expected [13]. In Figure 35 part C and D, current had a large impact on penetration at 11 volts. Penetration dropped dramatically at 13 volts resulting in a loss of 19% and 30% at 200 amps for autogenous and welds with solid 308L filler metal, respectively.

Figure 34: 308L Penetration Box Plot
Figure 35: 308L Experimental Design Results, A) Pareto Analysis, Width B) Pareto Analysis, Penetration C) Interaction Plot, Penetration D) Main Effects Plot, Penetration
5.3: Cored Wire vs. Paint on PEC Additions

Penetration enhancing compounds have been in use for around 50 years. The deposition of the PEC on the weld joint has depended upon use of solvent that is then painted on to the surface of the weld joint. The solvent then evaporates leaving a painted layer of PEC.
on the surface of the weld joint. The process adds additional steps to the welding process including grinding, mixing, and painting, as shown in Figure 37. The solvents recommended for use with EWI’s SS-7 powder are acetone or isopropyl alcohol. EWI provides the SS-7 PEC pre-mixed and ground.

To compare the properties of the paint on PEC to the PEC cored wire, a small section of wire was cut and measured for weight. Calculations for the amount of PEC added during cored wire addition can be calculated by incorporating the travel speed, wire feed speed as well as the PEC wt% composition of the wire itself. An amount of PEC equal to 3 wt% total was used in the manufacture of the PEC cored wire at ESAB. Other alloying elements were added for a total fill of 10wt %.

Multiple methods of painting the PEC on top of the weld surface were tested. Scraping blades with steps of 0.1, 0.2, and 0.3 mm were used to obtain desired paint thickness levels. This was not successful, as the oxides would form clumps, dragging additional particles through the deposited layer, leaving a bare swath in the weld area. Without a
suitable way to obtain a uniform paint thickness, attempts to deposit the PEC on the surface in a uniform way were conducted. The only moderately acceptable method was using a paintbrush as directed and painting the PEC on the surface with one stroke. Any attempt to repair an area through brushing was unsuccessful.

Figure 38: Paint on PEC
Approximate weights of deposition were calculated by careful measurements of the PEC, paint brush, mixing dish, and dry PEC weights individually. Before and after measurements were taken of each item resulting in a net loss of weight. This loss in weight was therefore the deposited weight of PEC on the plate. Careful measurements and markings on the plate surface were used to draw the target box area for PEC. An area of 88.9 mm (3.5 inches) x 25.4 mm (1 inch) was used. The total PEC density was then calculated for the deposited layer on the 304L plate material. A weld schedule of 300 amps, 11 volts, and 2.5 ipm was used to test the penetration efficiency of the PEC, see Figure 38. Test results of the wire feed tests and paint on PEC can be seen in Figure 40. A PEC consumption of approximately 50% was used to calculate how much PEC was consumed by the weld. This value was calculated through area measurements of the painted on surface and weld. PEC content is reported in grams/inch of weld for both cases; paint on and cored wire deposition.
Several issues were found after the completion of this experiment, namely the depth of penetration exceeded expectations, reaching a depth of penetration equal to 10.39 mm (82%) of the plate thickness of 12.7 mm (1/2 inch). Results of the penetration tests found that penetration was not significantly different than the cored wire process, see Figure 40. The added steps required for paint on deposition and difficulty in preparing a sample of specified paint thickness or PEC density only limits the desirability further. No benefits were found to support using the paint on process in lieu of cored wire deposition.

Metallographic cross sections of an autogenous, paint on PEC, and Cored Wire PEC weld are shown in Figure 41, and Figure 42.
Figure 41: No PEC, 300 Amps, 11 Volts, 2.5 ipm Travel Speed (1/2” plate thickness)

Figure 42: 300 Amps, 11 Volts, 2.5 ipm Travel Speed (1/2” plate thickness)  A) Paint on PEC ~0.033 g/inch,  B) Cored Wire PEC, 20 IPM WFS
5.4: Wire Feed Speed Tests

Penetration efficiency of the PEC cored wire was sought through additional tests of varying wire feed speed with all other variables fixed. Due to the deep penetration obtained in the previous tests, weld at 300 Amps were completed on 1” 304L plate material. Welds at 200 amps were completed on the ½” material previously used. Chemical compositions can be seen in Table 9. The same 3/32”, 2% Thoriated electrode used in the experimental design was used for this test. See Figure 26. Tests using current values above 200 amps utilized a 1/8”, 2% Thoriated electrode purchased to the following specifications:

- 7” Length
- 1/8” Diameter
- 30° Taper / Grind Angle
- 0.045” Tip Flat Diameter
- Standard Surface Finish

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<th>Mn</th>
<th>P</th>
<th>Si</th>
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</table>

Table 9: Material Chemistry

The effect of wire feed speed, hence PEC density, on the weld penetration achieved in bead on plate trials was completed initially at 275 amps, 13.5 Volts, 4 ipm travel speed, and up to 120 ipm on the ½” 304L plate material. Penetration increased for the initial test
at 30 ipm of wire feed speed, however penetration decreased at greater wire feed speeds. This can be seen in Figure 43. Although the increased wire feed speeds allowed for greater ease of application, the penetration did drop off dramatically past 30 inches per minute. Parameters were chosen to provide a stable and consistent arc transfer mode at the high wire feed speeds. At wire feed speeds above 30 ipm, a significant amount of arc energy was required to melt the consumable wire reducing the energy input to the work piece. As more energy was consumed by the wire addition less and less base metal dilution occurred resulting in the cladding of the work piece by the filler wire. Reverting back to the results of the design of experiments, and recognizing the dramatic downward trend in weld penetration, refined parameters were used in the next tests. Most notably, reduced arc height / voltage, and lower wire feed rates.

Reduction of wire feed speeds below 30 ipm along with reduced arc height, increased penetration efficiency. The reduced wire feed speed did require careful placement of the wire in the weld pool. Wire feed speeds were reduced to 5, 10, 15, and 20 ipm. See Figure 44, and Figure 45. At less than 20 ipm the wire has to be fed directly into the front of the weld pool to prevent burn back on the wire or an inconsistent globular transfer across the arc. Maximum penetration was attained at 5 ipm; the lowest wire feed speed used. At 5 ipm of wire feed speed, the wire was directly fed into the weld puddle at the leading edge. This prevented undesired burn back of the wire during welding and provided consistent addition of filler and PEC material to the weld pool. At 200 amps, 2.5 ipm travel speed, and 5 ipm wire feed speed, a maximum increase in penetration of 240%
was measured. The penetration enhancement was not as significant at 300 amps where a maximum increase of 125% was measured. These tests were conducted at 2.5ipm (1.06 mm/sec) and 4 ipm (1.69 mm/sec) of travel speed.

The greater arc forces involved at higher amps reduced the effectiveness of added PEC as compared to the welds completed at less than 200 amps with PEC addition. At lower arc energies the penetration is dominated by hydrodynamic forces based upon the fluid flow and surface tension of the material. PEC additions at currents below 200 amps have a significant effect on penetration due to the change in surface tension gradient in stainless steels. At increased energy densities provided by arc currents above 200 amps, the electromagnetic contributions from the arc cause a depression of the molten weld pool.

Figure 43: Wire Feed Speed Test: 275 Amps, 13.5 Volts, 4 ipm Travel Speed
through a force termed, arc force. This force on the weld pool is significant in that the depression of the weld pool focuses the energy of the arc into the weld pool. A nonlinear relationship ensues in that penetration is rapidly increased through the contributory effects of the hydrodynamic and electromagnetic forces [28]. The penetration mechanisms involved in the use of PEC’s include these arc and surface tension effects seen in high amperage gas tungsten arc welding. Measurements of arc force have been taken by Rokhlin and Guu [28]. Results from their welds on steel plates revealed that weld pool depression did not occur at welds below 200 amps. Above 200 amps, Lorentz forces and hydrodynamic forces were attributed to the weld pool depression and increase in penetration depth [28]. These values are affected by the material, electrode geometry, shielding gas, and arc length.
Figure 44: Penetration vs. Wire Feed Speed, 200 Amps

Figure 45: Penetration vs. Wire Feed Speed, 300 Amps
5.5: Summary of Experimental Design

Several observations could be made after completion and analysis of the DOE and wire feed speed tests. First and foremost, a minimal amount of PEC is needed from the wire to promote maximum penetration efficiency. Second, penetration does not drop off quickly allowing for greater deposition rates, if needed. A study of oxide PEC quantity on penetration depth found that titanium oxide has been shown to maintain deep penetration in welds at higher PEC contents [16]. This was not the case in their study including, Cu₂O, NiO, and SiO₂ oxides[16]. Titanium, Silicon, and Chromium oxides make up the constituents of EWI’s SS-7 PEC. The ability of Titanium oxides to maintain penetration enhancement at higher PEC concentrations may be why it is used in EWI’s SS-7 formulation. Wire placement has also been used in manufacturing as a method of using minimal amounts of filler wire in a weld joint for weld repairs and high energy density processing such as laser and electron beam welding. It is possible that a pre placed wire on top of the weld joint would give increased penetration, however this was not tested. The additional steps required diminish the cost benefit of using the PEC cored wire.

Penetration contour plots are useful in initial process development and equipment characterization. Plots were created for the GTA process with PEC cored wire through a consolidated data set of welds made using PEC. Figure 46 shows contours of all data up to 200 amps at 4 ipm (1.69 mm/sec) travel speed. Figure 47 includes the data shown in Figure 46, with added data for welds at 300 amps, also at 4 ipm (1.69 mm/sec).
Figure 46: Penetration Contour Plot - Max 200 Amps – 4 IPM Travel Speed

Figure 47: Penetration Contour Plot – Max 300 Amps - 4 IPM Travel Speed
Chapter 6 : Heat to Heat Variability

Weld variability of stainless steels has been well documented and theorized for effects of sulfur content. Welds at Honeywell FM&T have been influenced detrimentally by the effects of low sulfur content minimizing the penetration of gas tungsten arc welds. One of the benefits claimed by EWI for the SS-7 PEC is reduced variability due to small differences in chemical composition [8]. Low sulfur contents below 100 ppm (0.01 wt%) have shown rapidly decreasing penetration. Much of the work focused on using the PEC material has been reported comparing partial penetration gas tungsten arc welds without PEC to those of full penetration welds with PEC. A direct comparison between partial penetration welds was sought in this study.

A 3-dimensional heat flow scenario using partial penetration welds on 1” thick sections cut from 6 heats of 304L and 4 heats of Nitronic 40 were used. Chromium equivalents of available material may be seen in Table 14, followed by chemical composition of each alloy in Table 23. To predict the solidification mode and ferrite content of welded austenitic stainless steels, chromium and nickel equivalency formulas have been developed. Delong and WRC-1992 diagrams were used for prediction of weld microstructure in the 304L alloys, Figure 65 and Figure 66. Nitrogen bearing alloys cannot be accurately predicted through the WRC and DeLong diagrams, leading to the
development of specialized diagrams for nitrogen alloys. Diagrams developed by Hull and Espy were used for prediction of the 21-6-9 Nitronic alloy solidification mode and ferrite content. A detailed analysis of the solidification mode and ferrite content will be completed in Chapter 8. Penetration variability within the same family of material can be influenced by small variations in composition. Sulfur and oxygen have been shown to significantly affect the molten puddle fluid flow in gas tungsten arc welding [13]. The commercially available 304L contains the highest amount of sulfur at 0.028 wt%. This value is near the max allowable content level of 0.030 wt%. Linear welds were made on each heat of material autogenously, with 308L solid wire and with PEC flux cored wire. 308L solid wire filler metal welds were completed for later evaluation as part of the metallurgical characterization to be discussed in Chapter 8. Welding parameters were held constant including arc height. For this study the arc voltage controller was disabled and a fixed electrode standoff of 0.050” was used. Parameters for this study are:

- 3/32” Diameter Electrode (see Figure 26)
- 200 Amps
- 0.050” Electrode Standoff – Arc Height
- 0.125” Electrode Extension beyond Gas Cup
- 30 cfh Argon Shielding Gas
- 4 ipm Travel Speed
- 10 ipm Wire Feed Speed
Weld penetration efficiency was more pronounced in this study than the previous parameter study. A difference in arc voltage, approximately 1 volt, was witnessed for autogenous welds, and welds with PEC. Welds with 308L exhibited a decreased voltage due to the addition of material without the formation of a weld pool depression, thereby lessening the arc gap. This increase in arc voltage for welds with PEC has been reported in previous literature, and is the subject of some debate for influence on microstructural evolution [3, 4, 7]. Some authors argue the increased voltage increases heat input, therefore affecting the cooling rate of the weld pool. Photos of representative samples can be seen below in Figure 48.

At an arc height of 0.050 inches, the voltage feedback registered at 12 Volts for welds without PEC. When welding with PEC, the value would toggle between 12 and 13 volts on the welding power supply. This does coincide with previous reports of increased arc voltage when using PEC. Penetration did increase more significantly in this test, compared to the experimental design completed earlier using an arc height controller. This is likely due to the shallower penetrating autogenous weld due to higher arc height and resultant voltage. Previous welds with the arc height controller would follow the puddle downward with the depression created through arc force. The increase in penetration seen in these test, an average of 293% for heats of Honeywell 304L, validates claims of up to 300% by EWI. Further claims by EWI to eliminate heat to heat variation were also validated through testing [6].
Figure 48: Weld Surface Photographs of Nitronic 40 and 304L Welds
Numerically the variation between heats was similar for welds without PEC and with PEC. Values of approximately 0.45 and 0.101 were measured for the standard deviation between 304L welds and Nitronic 40 welds respectively. Penetration variability is shown in Table 10. The improvement is relative to the depth of penetration, where, as a percentage of penetration, the variability was reduced by 14% in 304L and 2% for Nitronic 40. The true numerical variability between heats of material was not improved with the addition of PEC flux cored wire. Standard deviations of approximately 0.45mm were calculated for welds made autogenously and with PEC flux cored wire on 304L base materials. A scatterplot of penetration vs. material heat can be seen in Figure 49.

<table>
<thead>
<tr>
<th>Material</th>
<th>Average Penetration (mm)</th>
<th>Autogenous</th>
<th>Std. Dev.</th>
<th>304L</th>
<th>Std. Dev.</th>
<th>MC304L PEC</th>
<th>Std. Dev.</th>
</tr>
</thead>
<tbody>
<tr>
<td>304L</td>
<td>1.845</td>
<td>0.440</td>
<td>2.213</td>
<td>0.068</td>
<td>4.731</td>
<td>0.456</td>
<td></td>
</tr>
<tr>
<td>304L*</td>
<td>1.667</td>
<td>0.050</td>
<td>2.213</td>
<td>0.068</td>
<td>4.893</td>
<td>0.407</td>
<td></td>
</tr>
<tr>
<td>Nitronic 40</td>
<td>1.984</td>
<td>0.091</td>
<td>1.986</td>
<td>0.059</td>
<td>4.477</td>
<td>0.114</td>
<td></td>
</tr>
</tbody>
</table>

* Honeywell Material Only

Table 10: Material to Material Weld Penetration Variability

Sulfur content can negatively impact a manufacturer when reprocessing of components causes variations in heats to be intermixed into components to be welded. The intermixing of different heats of low and high sulfur material can cause a qualified weld schedule to no longer provide the desired weld profile. This can cause issues when qualifications are tightly controlled and costly. Welds were made between the commercially available low sulfur material and high sulfur material with PEC and without. A significant increase in penetration was seen for the 304L material with 0.028 wt% Sulfur without PEC. As reported by Heiple and Roper, Sulfur content influenced the
weld penetration of our welds through Marangoni fluid flow increasing penetration by 150% in the autogenous weld [13]. This is one of the mechanisms proposed for penetration enhancement of the PEC. Weld Penetration was stabilized with the addition of PEC as can be seen in the Micrographs in Figure 51.

Figure 49: Penetration vs. Heat Number with and without additions of PEC flux cored wire.

The addition of PEC flux cored wire induced increased penetration variability in welds on low sulfur heats of 304L forgings. This material is tightly controlled for sulfur content, reducing the variability through shallow and consistent penetration. The addition of 0.028wt% sulfur drastically increases the penetration (150%) and drastically increases the variability of the weld penetration. Avoidance of this scenario is not always possible

79
when refurbishment of old components may provide varying sulfur contents in the welded base materials. The numerical variability of autogenous 304L welds with low sulfur content was recorded to be a low 0.05mm of penetration, or 3%. This value increased to 8.3% with the addition of PEC flux cored wire, although at significantly greater penetration depth. When adding the high sulfur heat of material to the analysis, the standard deviation increased to 23.8%. A slight increase to 9.6% was measured in the same high and low sulfur heats of 304L with PEC flux cored wire addition. A decreased penetration variability was found with the use of PEC flux cored wire when welding low and high sulfur 304L base materials. Penetration variability in the heats of 21-6-9 was reduced from 4.5% in autogenous welds to 2.5% with the addition of PEC flux cored wire.
<table>
<thead>
<tr>
<th></th>
<th>304L (201155)</th>
<th>Nitronic 40 (201094)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Autogenous</td>
<td><img src="image1.png" alt="Image" /></td>
<td><img src="image2.png" alt="Image" /></td>
</tr>
<tr>
<td>308L</td>
<td><img src="image3.png" alt="Image" /></td>
<td><img src="image4.png" alt="Image" /></td>
</tr>
<tr>
<td>PEC</td>
<td><img src="image5.png" alt="Image" /></td>
<td><img src="image6.png" alt="Image" /></td>
</tr>
</tbody>
</table>

Figure 50: Representative Cross Sections of Material to Material Variability
<table>
<thead>
<tr>
<th>Autogenous</th>
<th>With PEC Addition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low Sulfur – Autogenous (1.813 mm)</td>
<td>Low Sulfur – With PEC (4.350 mm)</td>
</tr>
<tr>
<td>High Sulfur – Autogenous (3.082 mm)</td>
<td>High Sulfur – With PEC (4.288 mm)</td>
</tr>
<tr>
<td>Low to High Sulfur – Autogenous (3.661 mm)</td>
<td>Low to High Sulfur – With PEC (4.785 mm)</td>
</tr>
</tbody>
</table>

Figure 51: Effect of Sulfur Content on Penetration (commercial 304L plate)
A principal goal of this project is to test full penetration tube welds of 304L material. The current process being evaluated utilizes 8 to 9 weld passes to fully fill the weld joint. A U-groove joint design is currently used requiring a small root pass without backing, followed by additional fill passes with 308L solid wire addition, see Figure 52. A full penetration weld removing the joint preparation would simplify the machining and processing of the component leading to a significant cost savings. Weld samples for this study included two wall thicknesses, ¼” and 3/8”. Weld samples were machined on all surfaces to provide the specified wall thickness and approximate 5” outside diameter. Procured tube samples were ordered of 304L material, see Table 23, and cut to 2” tube lengths, with machined weld surfaces to leave sharp corners. Completed samples are shown in Figure 53.
Weld samples were manually tack welded together using a v-block and up to a max of 125 amps using a Miller Syncrowave 350LX TIG weld power supply. Four weld samples were tack welded together and positioned on the Jetline rotary axis for welding in the 1G weld position, Figure 54. There were three methods used for purge gas containment and pressurization, internal disk and O-ring seals, disk and rubber compression seal (shown in Figure 54), and silicon baffles. The multiple systems were tried to improve the quality of the purge gas and containment of the backing pressure. The 1/8” diameter welding electrode was used for all tests of welding the 3/8” tube wall thicknesses. Wire was fed from the leading edge of the weld pool at an angle of approximately 30°.

Figure 53: Machined Tube Samples: Wall Thicknesses, ¼” (Left) and 3/8” (Right)
Early test welds on $\frac{1}{4}''$ and $\frac{3}{8}''$ wall thickness tubing revealed easy penetration of the $\frac{1}{4}''$ tube without backing pressure, $\frac{3}{8}''$ wall thickness samples were then tested for parameter optimization and root cleanliness. Of considerable concern was the validation of a clean root surface. Weld penetration trials were originally attempted at travel speeds of 3 to 4 ipm, and varying wire feed speeds from 5 to 20 ipm. Upon onset of full penetration, the dynamics of the weld pool would cause immediate drop-thru of the weld puddle into the tube.

Pressurization of the internal tube volume was required after attempts to minimize the amount of penetration failed to prevent this catastrophic failure. Internal pressurization was required due to the absence of a backing feature on weld samples and in the target application of this technology. Backing pressure was obtained through use of an additional Argon gas cylinder with a multi-stage pressure regulator allowing for accurate and consistent control of supplied pressure. A 0 to 30 psi back pressure regulator was

Figure 54: Weld Tube Setup with Compression Purge Gas Setup (A), and Silicon Baffle System (B).
attached to the output side of the purge gas system. To monitor internal backing pressure a Magnehelic® gauge measuring inches of water displacement (inches WC) was attached to a T-joint on the exit side of the pressure system. Adjustment of the Argon source pressure regulator was required to maintain internal pressure during welding, due to the loss of leak path occurring during welding.

Figure 55: Catastrophic Weld Drop-Thru Failure
7.1: 3/8” Full Penetration Welds

Initial weld tests were conducted with backing gas of varying back pressure and plotted for analysis to determine a potential operating window. Wire feed rates for these tests was varied during welding from 20 ipm wire feed speed downward. With the available electrode sizes of 3/32” and 1/8”, amperage was kept below 350 amps. For this reason travel speed was reduced to decrease required amperage and prevent excessive electrode breakdown during welding. This suitable range of welding current for the 1/8” electrode is less than 350 amps. Review of Current vs. Travel Speed and Arc Voltage scatterplots of initial tests revealed a target operating range of 3ipm travel speed, 10.5 to 11 volts, and 250 to 300 Amps. These scatter plots can be seen in Figure 56. Table 12 shows specific welding data for test welds leading to full penetration of the 3/8” tube wall.

A single pass, full penetration weld was achieved through use of PEC cored wire addition at 250 amps. In order to minimize root penetration, variables were slowly modified to obtain full penetration. As a result of testing a full penetration weld with a conical shape with a wide reinforcement area and small root width was not achievable. Once full penetration was achieved the fluidity of the weld pool allowed for a wide penetration profile. The use of internal pressure also had an effect on achieving full penetration in that too much pressure could suppress full penetration. A suitable range of internal pressure was found to be of 3 to 5 inches of water column (InWC), or 0.11 to 0.18 psi on the Magnehelic® gauge. Utilizing this pressure range resulted in welds with a root appearance showing appreciable convexity to a slight concavity.
<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Amps</th>
<th>Volts</th>
<th>Travel Speed (ipm)</th>
<th>Wire Feed Speed (ipm)</th>
<th>Internal Pressure (InWC)</th>
<th>Full Penetration</th>
<th>Penetration (mm)</th>
<th>Root Width (mm)</th>
<th>Root Reinforcement (mm)</th>
<th>Weld Width (mm)</th>
<th>Cap Reinforcement (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>48A</td>
<td>200</td>
<td>11.5</td>
<td>3</td>
<td>20</td>
<td>no</td>
<td>7.67</td>
<td></td>
<td></td>
<td></td>
<td>8.87</td>
<td>1.17</td>
</tr>
<tr>
<td>48B</td>
<td>200</td>
<td>11</td>
<td>3</td>
<td>20</td>
<td>no</td>
<td>6.76</td>
<td></td>
<td></td>
<td></td>
<td>7.97</td>
<td>1.40</td>
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<td>275</td>
<td>10.5</td>
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<td>5</td>
<td>4.5</td>
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<td>4.92</td>
<td>0.16</td>
<td>7.32</td>
<td>1.62</td>
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<tr>
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<td>275</td>
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<td>3</td>
<td>10</td>
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<td>6.59</td>
<td>3.11</td>
<td>7.01</td>
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<tr>
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<td>10</td>
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<td>8.23</td>
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<td>7.63</td>
<td>0.16</td>
<td>8.69</td>
<td>1.85</td>
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<tr>
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<td>10</td>
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<td>-0.78</td>
<td>9.09</td>
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<td>10</td>
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<tr>
<td>62F</td>
<td>250</td>
<td>11</td>
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<td>10</td>
<td>5</td>
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<td>9.53</td>
<td>4.86</td>
<td>-0.51</td>
<td>7.88</td>
<td>1.74</td>
</tr>
</tbody>
</table>

Table 12: Full Penetration Study (Magnehelic® gauge measures: inches of water column)
Micrographs of weld penetrations leading up to full penetration can be seen in Figure 57. Control of the weld root convexity was achieved through careful adjustment of the supplied Argon pressure regulator to maintain internal weld tube pressure. The use of a multistage pressure regulator allowed for steady adjustments to be made. Although maximum penetration efficiency was found to be with 5 ipm of wire feed speed, the little amount of additional filler metal reduced the amount of material available for the root and cap reinforcement. Welds with 10 ipm of wire feed speed were chosen to be a good compromise between amount of added PEC, penetration efficiency, and amount of filler material added for root and cap reinforcement. Welds were made with varying arc
parameters and varying internal pressures from 3 to 5 InWC to determine the optimum weld schedule.

Figure 57: Metallographic Cross Sections of Full Penetration Weld Trials. A) 200 Amps, 11.5 Volts, B) 200 Amps, 11 Volts, C) 200 Amps, 10.5 Volts, D) 250 Amps, 10.5 Volts, E) 275 Amps, 10.5 Volts. Wire Feed Speed was varied for all welds from 20 IPM to 5 IPM.

Welds completed at 250 amps were found to have a small processing window. Two successful welds completed at 250 amps are shown in Figure 58. The penetration profile varied from the weld surface to the root of the weld with the change in pressure. An increase in wire feed speed to 15 ipm resulted in the loss of full penetration. A larger process window was found at 275 amps. Due to the larger volume of material at the root of the weld, the puddle was more resistant to the changes in pressure. This resulted in a subtle transition from root convexity to concavity. A value of 4 to 4.5 InWC was found to provide a slightly convex to flat root surface at 275 amps. The addition of 15 inches per
minute of wire feed speed resulted in similar weld profiles, shown in Figure 59. Sample photographs of weld cap and root appearance are shown in Figure 60, Figure 61, and Figure 62.

![Sample photographs of weld cap and root appearance](image)

62E (10WFS, 4 InWC) 62D (10WFS, 5 InWC)

Figure 58: 3/8" Wall Thickness - 250 Amps, 11 Volts, 62E) 4 InWC, 62D) 5 InWC

Root convexity is desired to minimize weld stresses on the interior surface of the weld. A low internal pressure of approximately 4 InWC, and 10 to 15 ipm of wire feed addition was found to provide adequate root and cap reinforcement. Due to the formation of a adhered slag layer on the top surface of the weld, mechanical removal through abrasive grinding or machining is required.
Figure 59: Full Penetration Welds with Varying Internal Pressure (275 Amps, 11 Volts)
Figure 60: Weld Reinforcement with Slag Layer

Figure 61: Sample 64C root of weld
Figure 62: Internal Weld Surface (Clean in foreground, oxidized in background)
7.2: 1/4” Full Penetration Welds

Full Penetration was easily achieved in ¼” wall thickness tubing. Previous papers have demonstrated ability to weld ¼” tubing without the need for backing pressure using the paint on PEC [6]. Test welds were made using the PEC cored wire with parameters derived from previous literature using paint on PEC [3, 40]. Values of 200 to 250 Amps were used in published reports for full penetration of ¼” stainless steel plates. Full penetration was achieved at 175Amps, 11Volts, 3IPM Travel Speed, and 10 IPM of Wire Feed Speed without backing pressure. A suitable weld root penetration and reinforcement were obtained. Because of the limited cost savings provided at ¼” wall thickness, the focus of the experimentation remained on 3/8” wall thickness welds.

7.3: Summary

The use of internal pressure to sustain a weld puddle within the weld joint in 9.5mm wall thickness 304L tubing was achieved through use of a multi-stage pressure regulator and manual control of the input pressure. A back pressure regulator was also used on the exit side of the system. The penetration of the tube wall thickness was easy to obtain using the PEC flux cored wire. When optimized, a weld bead with both a root and surface convexity could be maintained. A weld with optimum weld joint geometry is shown in Figure 63. The use of 5ipm of PEC flux cored wire results in a minimal amount of filler metal, reducing the tolerance for pressure variations during welding. This would be a difficult weld to obtain day to day in a production environment. The pressure regulators used could supply a constant pressure to the system, however were not capable of
controlling the pressure to the extent needed without manual adjustments. With the sealing of the tube, the leak path inherent in the weld joint closed as welding continued around the circumference of the pipe. This resulted in a buildup of pressure during welding. As a result the pressure regulator usually required a correction to lower the input pressure of the system. Pressure is reduced by limiting the flow of Argon from the cylinder to the system. This also has an effect on the back purging and shielding of the root of the weld. Manipulating the system input pressures and the air flow restriction at the output to maintain a steady flow of purge gas and maintain the correct pressure was difficult. Oxidized welds did occur, with increased frequency in full circumferential welds. The addition of PEC cored wire likely introduces oxygen to the interior of the pipe. Without a suitable flow of argon, a buildup of oxygen emanating from the oxygen rich weld will lead to oxidation of the root surface.

A production ready system will require pressure and flow regulators with additional resolution at 0.1 to 0.2 psi with the capability to adjust both accordingly. This system may require a programmable logic controller.
Figure 63: Full Penetration Weld, Sample 48E, 9.5 mm wall thickness, 275 amps, 10.5 volts, 3ipm linear travel speed, 5ipm wire feed speed, 4.5 InWC
Chapter 8 : Metallurgical Characterization

The introduction of PEC cored wire to the weld adds a few constituents not traditionally used in the gas tungsten arc welding of austenitic stainless steels. The composition of the PEC cored wire is nominally 308L with some of the constituents consisting of oxides. Additions of oxygen, titanium, chromium, and silicon are alloyed into the weld metal affecting the dynamics of the weld pool and resultant weld morphology. Of these constituents titanium would be of the greatest concern due to its high reactivity.

Questions still remain as to the mechanistic impact the PEC’s have on the weld pool as well as the evolution of the oxides in the resultant weld metal. A major focus of this project was to seek information on the microstructural evolution of the oxide constituents. Unpublished literature from EWI, the manufacturer of the PEC, claims that most is removed in the slag, with a few SEM pictures to justify this response. A more detailed and independent analysis was desired for this project. Also of interest is the resultant metallurgy and comparison of solidification modes with and without PEC.

Optical microscopy, electron microscopy including electron diffraction spectroscopy, laser ablation mass spectroscopy, and magnetic ferrite measurements were used in the analysis of weld metallurgy.
Welds were first cut into manageable sections for mounting through use of a band saw and precise abrasive cut off saws. Standard metallographic sample techniques were used to mount polish each sample. These steps included the mounting of the cut sample, usually a transverse weld cross section in Bakelite using a mounting press of 1.25” or 1.5” diameter. Most samples were polished down to 1µm diamond paste, with additional polishing of 0.05µm colloidal silica being used on some samples intended for SEM analysis. This provided a polished sample surface free of visible scratches and ready for electrolytic etching. Three etchants were used in the analysis of the weld microstructures; 10% oxalic acid, 60% Nitric Acid, and 10% Chromic Acid. A potentiostat was used to provide a set voltage across the cathode and anode in solution to preferentially etch the sample surface revealing the different phases that comprise the weld microstructure. Ferrite is preferentially etched for all three etchants used.

8.1: Solidification Structure
Weld metallographic analyses of the welds with PEC and without PEC were analyzed through optical microscopy and electron microscopy. Diagrams describing solidification mode were discussed in Chapter 4, and duplicated in Figure 64. As shown in Chapter 4, 304L consists of approximately 20 wt% Chromium, and 10 wt% Nickel. This composition lies just to the right of the triangular eutectic region in Figure 64, resulting in the initial solidification of delta ferrite, δ. First to solidify, the ferrite will be highly enriched with Chromium, a max of about 26 wt%. This composition of delta ferrite will be stable through cooling to room temperature as shown with the dotted line in Figure 99.
64b. The remainder of the weld pool will solidify as a mixture of austenite and ferrite, however this secondary ferrite will undergo a diffusional transformation to austenite upon further cooling [9].

![Solidification Mode, Pseudobinary Phase Diagram Relationship](image)

Figure 64: Solidification Mode, Pseudobinary Phase Diagram Relationship [29]

Through process characterization, the maximum enhancement in penetration occurred at minimum operating ranges of alloying content (5ipm WFS). This reduces the amount of extra alloying elements inserted into the weld metal that are not inherent of the base metal composition. This minimal amount of added PEC constituents were not expected to change the solidification behavior of the stainless steel, however the incorporation of titanium in the weld metal is not well published.
Welds made to characterize the weld penetration variability using PEC flux cored wire were subsequently used for metallographic analysis. This consisted of Nitronic 40, also known as 21-6-9 (Cr-Ni-Mn), and 304L austenitic stainless steel in the commercially available rolled plate, and forgings. Detailed chemical compositions can be seen in Table 23.

<table>
<thead>
<tr>
<th>Oxide</th>
<th>Melting Point</th>
<th>Crystal Structure</th>
</tr>
</thead>
<tbody>
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<td>TiO₂</td>
<td>1840°C</td>
<td>Tetragonal</td>
</tr>
<tr>
<td>Cr₂O₃</td>
<td>2435°C</td>
<td>Hexagonal</td>
</tr>
<tr>
<td>SiO₂</td>
<td>1600°C</td>
<td>Tetrahedral</td>
</tr>
</tbody>
</table>

Table 13: Properties of Oxides[41]

The use of very stable oxides in welding causes concerns of PEC entrapment in the weld microstructure. Titanium Dioxide (TiO₂), Chromium Oxide (Cr₂O₃), and Silicon Oxide (SiO₂) have melting temperatures that are stable above the liquidus temperature for stainless steels. The melting temperatures of these oxides are shown in Table 13. Because of this high temperature stability, the potential for unconsumed PEC to reside in the fusion zone is high. The use of a cold wire feed technique increases the potential further for entrapment of unconsumed oxides. Entrapment of oxides may be tolerated in fine dispersions, as they are insoluble in metals and provide high temperature strength [31].

Predictive diagrams are extensively used for stainless steels. Chromium and Nickel equivalents can be calculated from predictive equations and plotted to predict
solidification mode and retained ferrite content. A number of different predictive diagrams were used in the analysis of the 304L and 21-6-9 alloys. The DeLong and WRC-1992 were used for 304L and can be seen in Figure 65 and Figure 66, respectively. Due to the nitrogen content in 21-6-9, diagrams such as the Espy diagram, and the Hull diagram can be used to predict ferrite content. The Hull and Espy diagrams are included in Figure 67, and Figure 68. The chromium and Nickel equivalents calculated can be seen in Table 14.

<table>
<thead>
<tr>
<th>Material</th>
<th>Sample</th>
<th>WRC 1992</th>
<th>DeLong</th>
<th>Hull</th>
<th>Espy</th>
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<tr>
<td></td>
<td></td>
<td>CrEq</td>
<td>NiEq</td>
<td>CrEq</td>
<td>NiEq</td>
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<tr>
<td>21-6-9</td>
<td>201094</td>
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<td>12.28</td>
<td>19.75</td>
<td>12.39</td>
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<td></td>
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<tr>
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<td>12.44</td>
<td>20.60</td>
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<td>11.55</td>
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<td>18.76</td>
<td>10.43</td>
<td>19.17</td>
<td>11.87</td>
</tr>
</tbody>
</table>

Table 14: Material Chromium and Nickel Equivalency Values

The available heats of 304L as predicted by the DeLong and WRC-1992 diagrams result in a ferrite content between 6 and 9 FN for the forging material and 9-10 for the commercial rolled plate. The solidification mode is predicted by the WRC-1992 diagram.
to be Type FA for the forging material. The commercial 304L plate is predicted by the
diagram to solidify as Type F. With the addition of chromium, silicon, and titanium, an
increase in ferrite content is expected in both alloys with increasing additions of PEC.

Welding of stainless steels containing nitrogen can detrimentally deplete the weld metal
of nitrogen content. The desorption of nitrogen in stainless steels is driven by the strong
affinity of nitrogen to form N₂ gas, solubility of nitrogen in the weld puddle, and the
difference in partial pressures present in the arc. A loss of nitrogen commonly occurs. To
counter this nitrogen is sometimes added to the shielding gas to maintain nitrogen
content. Solidification mode also plays a role in nitrogen desorption in which solubility in
ferrite is lower than austenite, causing the segregation and desorption of nitrogen if
insufficient austenite is not formed [42].

Several methods are to be used to analyze the metallurgical aspects of welds with PEC
additions. Retained ferrite content and solidification mode will be primarily addressed in
this chapter. Ferrite content can easily be measured through magnetic permeability,
magnetic detachment force, and through point counting methods after metallography. A
Fisher feritscope fmp30 was used to measure the ferrite content of welds through
placement on the undisturbed weld surface, unless otherwise mentioned. Analysis of the
solidification mode primarily relied upon proper etching of the polished weld cross-
section and optical microscopy and/or scanning electron microscopy. Ferrite content of
gas tungsten arc welds are often taken to verify the weld deposit is within limits set
during weld procedure qualification. This verifies that the weld process was completed as qualified and indicates the level of crack susceptibility. As such, it will be discussed first.
Figure 65: DeLong Diagram, 304L only, [43]
Figure 66: WRC-1992 Diagram, 304L only [29]
Figure 67: Hull Diagram [44]
Figure 68: Espy Diagram with 21-6-9 heat locations [45]
8.2: Ferrite Measurements

Ferrite content of each weld was recorded by use of a Fisher Feritscope FMP-30. This allowed for the measurement of retained ferrite content in the weld metal. Conflicting reports have been found for oxide effects on ferrite content of austenitic stainless steels. Reports of increased ferrite content have been reported when analyzing welds using a single oxide constituent. Research papers authored by Tseng and Loureiro report an increase in ferrite content when using TiO₂, SiO₂, MnO₂, and Al₂O₃ oxides [3, 5]. The SS-7 cored wire composition consists of both Titanium and Silicon oxides; therefore an increase in ferrite number may be expected. Reports using the EWI composition of SS-7 have reported a reduced ferrite content [10].

Ferrite content was measured through magnetic permeability using a Fisher feritscope fmp30 and calibrated according to AWS A4.2. Ferrite measurements were taken on the undisturbed surface of the weld. Removal of the oxide layer through use of a powered rotary wire brush revealed an increase of up to 0.5FN in both 304L and 21-6-9 when compared to the measurements through the oxide layer. Measurements taken on transverse cross sections after final polishing contained a ferrite content increase of 1.5FN and 1FN for 304L and 21-6-9 respectively compared to measurements through the oxide layer on the surface of the weld.
8.3: 304L Ferrite Measurements – Partial Penetration Welds

Ferrite content is a good indicator of material composition and cooling rate of weld metal. The ferrite content was measured on samples welded autogenously, with paint on PEC, with 308L solid filler metal, and with PEC cored wire addition for comparison. Samples prepared during the paint vs. wire testing reduce variation by reducing the influence of cooling rate. Due to the similar depth of penetration and weld schedule of the welds with and without PEC addition, the cooling rate would also be similar. Samples prepared with the additions of paint on and wire fed PEC delivery were measured for ferrite content on mounted and polished metallographic transverse cross sections. Ferrite content of welds using paint and wire delivery of PEC increased the ferrite content in the resultant weld metal. This is shown in Figure 69. Ferrite number increased from approximately 7.7FN for autogenous welds to 9.5FN with PEC addition.

\[
\text{Heat Input} = \frac{\text{Amps} \times \text{Volts}}{\text{Travel Speed}} \times 60 \ (\text{Joules per Inch})
\]

Equation 2: Heat Input Calculation

Additional tests including the variation seen with cooling rate were completed through use of varying amperage, travel speed, and PEC flux cored wire speeds. The ferrite content of the wire feed speed analysis were measured to determine the effect of increasing PEC additions and heat input on ferrite content. A slight increase in ferrite content was measured as wire feed speeds increased. Welds of different heat inputs were used in this analysis from 33k Joules/Inch to 79.2k Joules/Inch, calculated using Equation 110.
2. With the addition of ferrite stabilizing elements the ferrite content does increase with the addition of PEC, however ferrite content was influenced more directly by travel speed than by alloying content. The average ferrite content measured was between 7.5 and 8.5FN. This is shown in Figure 70. These measurements were taken through the oxide layer. An increase of 0.5FN was measured in samples with the oxide layer removed.

Figure 69: Ferrite Number vs. Consumed grams of PEC per linear inch of weld. Paint and Wire addition (300 Amps, 11 Volts, 2.5 IPM Travel Speed)
A value of 10 ipm of wire feed speed was determined through process characterization to be a compromise between properties of penetration efficiency and filler metal dilution. A dilution amount of approximately 87% was calculated in the material variability study when adding PEC flux cored wire at 10ipm. Because the PEC flux cored wire was manufactured to meet the chemical composition of 308L filler metal a small increase in ferrite content is expected due to the change in chemical composition. Because of the small dilution amounts, a minimal effect on ferrite number is expected in welds with PEC addition. Use of the WRC-1992 diagram, Figure 66, predicts a ferrite content of 7 to 9
FN in the weld metal if no filler metal is used. A comparison of ferrite content was made in autogenous welds to welds with 308L solid wire and PEC cored wire addition. Ferrite content was measured on the top surface of the weld after allowing the weld to cool to room temperature. As shown in Figure 71, the use of 308L filler metal increased the ferrite content from an average 7.19FN in autogenous welds to 7.94FN with 308L filler metal. The value recorded in welds with PEC cored wire averaged 7.04FN, prior to a correction of approximately 0.5FN to account for the oxide layer.

Figure 71: 304L Base Material and Filler Material Ferrite Number Comparison (55 = autogenous, 55-308L = 308L solid wire addition, 55F = PEC cored wire addition).
The average ferrite content of all welds made on 304L was 7.13 FN and 7.02 FN for autogenous and PEC cored wire assisted welds, respectively. A minimal change in ferrite number was recorded in welds with PEC cored wire addition, as shown in Figure 72. Measurements were taken on the top surface of each weld after cooling to room temperature, without removal of the oxide layer.

![Figure 72: Ferrite Numbers for 304L Heats](image)

8.4: 304L Ferrite Measurements – Full Penetration Pipe Welds

The chemical composition of the pipe welds are shown in Table 23. Ferrite measurements of full penetration tube welds were recorded from the weld reinforcement, as well as the
root side of the weld. Average values of 5.67 FN were measured on the weld reinforcement, and values of 6.61FN were measured from the root surface. Measurements were taken on the reinforcement without removal of the oxide layer, as well as on the clean root surface. A convex weld surface existed in all samples tested using the ferrite probe on the weld reinforcement. A flat to slightly convex weld surface at the root of the full penetration welds were analyzed.

8.5: Nitronic 40 Ferrite Measurements
As mentioned earlier the surface appearance of the welds on Nitronic 40 was significantly altered by the addition of PEC. A rough surface appearance was found on these samples with sharper solidification boundaries. An average ferrite number of 9.69 were measured on samples welded autogenously. Ferrite content dropped significantly with the addition of the PEC flux cored wire, corresponding to an average ferrite number of 4.35. Ames, Johnson and Lippold have reported retained Nitrogen content in welds with SS-7PEC on SAF 2507 super duplex steel [7]. The same concept of retained Nitrogen content is expected to have occurred for the Nitronic 40 alloy. This concept infers the ability of the slag layer formed on the surface of the weld to resist evolution of nitrogen from the weld metal. This would account for the reduction in ferrite content in the welds. Recordings were taken on the undisturbed weld surface using the Feritescope FMP-30 on each sample of the 4 heats of material used in this study. Ten measurements were taken on each sample, resulting in a total of 40 measurements for each condition. This data is shown in Figure 73. Measurements for welds with additions of solid 308L
wire were also measured for comparison to the autogenous and cored wire additions for a specific alloy heat of material, 2011098. Ferrite content for welds with 308L wire were slightly less than those welded autogenously, see Figure 74.

Figure 73: Ferrite Numbers for 21-6-9 Heats
Figure 74: Nitronic 40 Ferrite Number Comparison
8.6: 304L Base Metal Weld Solidification Analysis

Weld solidification with PEC and without PEC exhibited primarily FA type solidification as predicted by the pseudobinary phase diagram and WRC-1992 diagram. In a few cases type F solidification, which is a little unusual for austenitic stainless steels, were seen in welds on the commercially procured 304L plate material. The chromium and nickel equivalents of this material are calculated in Table 14. Plotting these values on the WRC-1992 diagram resulted in a predicted ferrite content of 9 FN. This positions the alloy composition just below the predicted Type F solidification mode line. Type F solidification mode occurred in these welds with and without any addition of filler metal. Ferrite content of these welds was calculated to be approximately 7.5 in both welds as measured from the top surface of the weld. Micrographs are shown in Figure 75, and Figure 76 of the Type F solidification mode seen.

For type F solidification, the first solidification product is ferrite. In the zone of type F solidification, Austenite is not formed until further cooling in which transformation to austenite occurs through a solid state transformation. Austenite begins to nucleate at the prior delta ferrite grain boundaries and forms a continuous band of austenite encompassing all prior ferrite grains. Acicular austenite can be seen emanating from the grain boundaries into the interior of the prior delta ferrite grain. This morphology was seen in welds with and without PEC addition in welds of similar heat input, and in the commercially available ½” plate material. The focus of metallurgical analysis was completed on forgings of 304L that did not exhibit this solidification mode.
Figure 75: Commercially available ½” thick plate welded autogenously at, 275 Amps, 13.5 Volts, 4 IPM Travel Speed, Originally 200x magnification

Figure 76: Commercially available ½” thick plate welded with 5ipm PEC cored wire addition at, 200 Amps, 11 Volts, 2.5 ipm Travel Speed. SEM image (QBSD, 20kV, 9.5mmWD) (7.7 FN measured on top surface of weld)
This same plate material was used in subsequent testing of heat to heat variability; however FA mode dominated at the lower heat input welds (<35k joules per inch).

Welds completed for material weld penetration variability study were used for weld microstructure analysis. The common weld schedule of 200 amps, 4 ipm travel speed, and 0.050” electrode standoff allows for the comparison of weld microstructure between autogenous welds and welds with 308L and PEC flux cored wire filler metal. As seen previously the ferrite content of welds with 308L and PEC flux cored wire were increased. Microstructural analysis of transverse cross sections prepared through chromic acid etching revealed nearly identical weld microstructures near the center of the welds. Type FA solidification mode was seen in all welds. The fusion boundary and transition regions vary in dendrite spacing due to differences in cooling rate. Micrographs taken at the fusion boundary, transition region, and weld centerline can be seen in Figure 77, Figure 78, and Figure 79. Skeletal ferrite dominates the solidification mode of all three evaluated weld types. An increase in lathy ferrite can be seen near the weld centerline. This transition between skeletal and lathy ferrite is driven by changes in composition and cooling rate and was seen sporadically in all welds. This can be explained through thermodynamic driving forces prefer the formation of small laths of ferrite intermixed with austenite due to limitations in diffusion at high rates of undercooling and chemical solubility [31].
Figure 77: 304L Fusion Boundary

<table>
<thead>
<tr>
<th>304L Fusion Boundary, 200 Amps, 0.050” Arc Height, 4 ipm Travel Speed</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
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</tbody>
</table>

Figure 78: 304L Transition Region

<table>
<thead>
<tr>
<th>304L Transition Region, 200 Amps, 0.050” Arc Height, 4 ipm Travel Speed</th>
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<tbody>
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<td>A</td>
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</tbody>
</table>

Figure 79: Weld Morphology at Center of Weld

<table>
<thead>
<tr>
<th>304L Weld Center, 200 Amps, 0.050” Arc Height, 4 ipm Travel Speed</th>
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<tbody>
<tr>
<td>A</td>
</tr>
</tbody>
</table>
Micrographs taken at 200x optical magnification of the fusion zone of welds with and without cored wire addition are shown in Figure 80, and Figure 81. No significant differences were seen in the central region of welds with and without PEC flux cored wire. Figure 82 and Figure 83 are photographs taken of the microstructure evolution at the center of the weld. A slightly darker region can be seen between the prior delta ferrite regions of the welds made autogenously. This may be caused by changes in composition causing a difference in etching response, or the result of an etching.
Figure 80: Sample 55, 304L, A: Autogenous B: Cored Wire Added (200x)

Figure 81: 304L with PEC flux cored wire addition
Figure 82: Autogenous: 200 Amps, 0.050" Arc Gap, 4ipm Travel Speed (1000x Optical Magnification, Chromic Acid Etchant)

Figure 83: PEC Flux Cored Wire Addition (1000x Optical Magnification, Chromic Acid Etchant)
8.7: Nitronic 40 Base Metal Weld Solidification Analysis

The characterization of the solidification mode was more difficult to recognize in the low ferrite bearing weld material. As shown by Suutala [36], the solidification mode of austenitic stainless steels with Manganese contents greater than 5wt% cannot be predicted accurately by the DeLong diagram. A drastic underestimation of the ferrite content and solidification mode occurs. Ferrite Number and Solidification mode are more accurately predicted by Hull’s equations. This is due to the transition of the manganese from a strong austenite promoting element to a weak stabilizer at higher concentrations (5-8 wt %). [36] Chemical compositions of the four Nitronic 40 alloys used in this study can be seen in Table 23. Nitrogen contents in these alloys are 0.24, and 0.25 wt% nitrogen.

Diagrams presented by Suutala using DeLong and Hull to predict solidification mode are shown in Figure 84[36]. In these diagrams the solidification mode predicted by DeLong was Type A and a prediction of Type FA in the Hull diagram. Weld metal ferrite content is known to be under predicted by the DeLong diagram; therefore the Espy diagram was developed for high nitrogen austenitic stainless steels and is shown along with the Hull diagram in Figure 85. Ferrite content predicted through use of the Hull diagram using chill castings is 5%. Espy predicts a similar ferrite content of 4-5 FN. The Espy diagram can be seen in Figure 68.
Figure 84: Solidification Mode - A) Hull, B) DeLong [36]

Figure 85: Hull Diagram with predicted ferrite content[44]
Solidification occurred in the Type FA solidification mode. As revealed by ferrite content measurements the amount of retained delta ferrite was significantly less than the autogenous welds and welds with 308L filler metal additions. The reduced ferrite content was evident throughout the weld from the fusion boundary to the center of the weld. This can be easily seen in the Figure 86C through Figure 88C. Although more susceptible to solidification cracking, the low ferrite content interspersed within the austenite is beneficial for a number of applications involving temperature and corrosion resistance [29]. The amount of ferrite retained in the weld metal is enough to provide a crack resistant weld solidification mode.

Optical images of the fusion zone for welds completed autogenously and with cored wire addition can be seen in Figure 89, and Figure 90. Lathy ferrite is present in both weld microstructures however the vermicular is more prominent.
Figure 86: Weld Morphology at Fusion Boundary of Nitronic 40

Figure 87: Weld Morphology at Transition Region of Nitronic 40

Figure 88: Weld Morphology at Weld Center of Nitronic 40
Figure 89: Autogenous Weld, Nitronic 40 - Center of Fusion Zone, 400x Original Magnification

Figure 90: Cored Wire Added to Nitronic 40 - Center of Fusion Zone, 400x Original Magnification.
8.8: Full Penetration Metallographic Analysis

Full penetration welds made of 3/8” and ¼” joint thickness were welded using 304L tube sections. Chemical compositions can be seen in Table 23, and plotted on the WRC-1992 diagram shown in Figure 66. Welds were completed using Argon coaxial shielding gas and backing gas. Weld solidification mode was not different than the 304L forging material. A higher nickel equivalency predicted a lesser amount of retained ferrite content in the weld metal of approximately 8FN. Small zones of ferritic solidification mode were seen near the root of the welds. This can be seen in Figure 91.

Figure 91: Sample 56C, root of weld at 100x original optical magnification
Skeletal ferritic morphology is dominant from the fusion zone inwards to the center of the weld. The grain growth direction changes from perpendicular to the weld direction near the fusion boundaries to parallel as the weld solidified near the center of the weld. This transition is shown in Figure 92. Weld metal dilution rates varied between 67.8% and 88.7%.

Figure 92: Sample 56C, Transition in solidification direction, Skeletal Ferrite.
Of significant value to the investigation into the use of cored wire, a comparison of hardness was used to baseline the mechanical properties of the resultant microstructure. Because of the small variations in chemical composition resulting in negligible variances in phase fractions, hardness measurements were taken to validate mechanical properties of the resultant weld metal. Hardness was checked using two methods; a micro hardness indenter was used, as well as a macro hardness indenter. The micro hardness tester is able to map out a grid pattern of hardness indents, then automatically measure and record the values.

9.1: Macro Hardness Tests

A Leco hardness tester was used to do macro hardness measurements using a 1/16” ball and 100kg weight. Rockwell B hardness measurements were taken of the base metal and weld metal. Two indentations were performed in the undisturbed base material to each side of the weld metal, and three values within the weld metal. Results of hardness indents for 304L and Nitronic 40 can be seen in Figure 93 and Figure 94 respectively. Welds with cored wire addition retained base metal hardness throughout the weld region for both 304L and Nitronic alloys. The increase in strength of both alloys is likely due to the refinement in grain size seen and retention of Nitrogen in welds with PEC addition.
Figure 93: Rockwell B Hardness Indents: 1/16" ball, 100kgs weight – 304L

Figure 94: Rockwell B Hardness Indents: 1/16" ball, 100kgs weight - Nitronic 40
9.2: Micro Hardness Tests

Micro hardness samples were tested using a 100 gram weight and Vickers indenter. Comparing welds in 304L plate material, the hardness values did not vary significantly as shown in Figure 95. As with the Rockwell hardness data the weld with cored wire addition may have higher weld metal hardness.

![Micro hardness Linear Segment](image)

Figure 95: Micro hardness Linear Segment

A better comparison of hardness values can be extrapolated from the micro hardness maps in Figure 96, and Figure 97. Unfortunately the materials do not match. A weld provided from Honeywell FM&T shown below is of 21-6-9 base material, has a joint thickness of approximately 3/8”, and is welded in 6 weld passes. A tube welds that was welded in 1 pass through use of the PEC flux cored wire is shown for comparison. From results of the Rockwell hardness tests and linear micro-hardness tests, a near uniform hardness distribution can be expected from the single pass weld. A uniform hardness profile across the weld joint in both axial and transverse directions can be seen. The

134
hardness distribution through the 6 pass weld is shown to be highest near the root pass of the joint with lower hardness near the top of the weld. This weld was completed using an autogenous root pass with additions of 308L solid wire for subsequent passes. This change in hardness throughout the joint thickness is a sign of variations in strength throughout the weld thickness. The more uniform behavior of the single pass weld using the cored wire is desirable.

Figure 96: Nitronic 40, 6 Pass GTA weld

Figure 97: 304L Single Pass Full Penetration, Single Pass GTA weld
Chapter 10 : Addition of Nitrogen Shielding Gas

Reports of increased ferrite content were found in previous literature evaluating individual PEC constituents and confirmed through testing of ferrite content in welds made with PEC paint and cored wire [3, 5]. The use of nitrogen shielding gas in duplex stainless steel welding has been used to control the balance of austenite and ferrite in the weld metal to balance properties, such as strength, ductility and corrosion resistance[46]. It was conceived that the use of nitrogen in the shielding gas could benefit the welding of 304L and Nitronic 40 with additions of PEC. A small amount of Nitrogen in the Argon shielding gas could reduce the ferrite number to match the ferrite number of the standard process. For a nitrogen bearing alloy such as Nitronic 40, the additions can be used to maintain weld metal nitrogen content. A careful application of increased nitrogen content can reduce the amount of interconnected ferrite to provide a dispersed ferritic network reducing hydrogen permeability through the weld metal, of concern to some users of austenitic stainless steels. Nitronic 40 contains 0.25 wt% as an intentional alloying element. Once again parameters used previously for the material variation study are used for comparison of nitrogen additions to the shielding gas. The weld schedule is shown below for your reference:
10.1: Additions of Nitrogen to the Shielding Gas during Welding of 304L.

Tests were conducted using a 304L forging composition from the heat to heat variability study, sample 201155. The same welding parameters were used in this study with the addition of small amounts of Nitrogen to the Argon shielding gas. Certified gas cylinders were purchased from Praxair with contents of 2, 5, and 10 volume % Nitrogen. The solidification mode was altered according to Table 15, for welds with the addition of 308L solid wire, PEC cored wire, and welds completed autogenously. Welds with solid 308L wire added maintained ferrite content at all nitrogen addition levels. Solidification modes were primarily delta ferrite at fusion boundaries at the unmixed zone, with austenitic columnar growth extending into the base material. Ferrite remnants were found between dendrite arms. Solidification mode is more definitive in regards to hot cracking susceptibility [39]. Hot cracking sensitivity was increased for all welds, however; according to solidification mode, the welds with 2% Nitrogen addition along with solid
308L and PEC cored wire addition were not altered. The solidification mode of FA, primary delta ferrite and secondary austenite maintains the low hot cracking sensitivity of the weld.

Baseline welds were made autogenously and with 308L solid wire for comparison with PEC cored wire welds. Nitrogen had a great effect on retained ferrite content of the welds tested. Hardness values of the welds are also plotted for comparison. Values for the Autogenous welds with Nitrogen Addition to the shielding gas can be seen in Figure 98. Ferrite content was reduced from a mean of 7.1 FN to 2.5 FN with 2% Nitrogen. Hardness of the weld metal was increased with the addition of 2% Nitrogen; however additions of 5% and 10% Nitrogen did not exhibit the same increase in hardness.

When using the PEC cored wire, the 2% Nitrogen addition resulted in a comparable microstructure and ferrite content of the welds completed autogenously. These are plotted in Figure 99. Additional Nitrogen reduced the ferrite number to a greater extent nearly eliminating the presence of ferrite. At 5 and 10 volume percent nitrogen a unmixed zone was readily apparent. A micrograph was taken using a polarizer and DIC prism to
highlight the formation of ferrite in the unmixed zone at the fusion boundary of the weld in Figure 101. Ferrite in this photo is white and is limited to a small, approximately 50µm wide zone near the fusion boundary. Less ferrite can be seen in the weld metal following the columnar growth direction of grains from this unmixed zone. This increased zone of ferrite content was seen in all welds with Nitrogen addition. In Nitronic alloys the drastic reduction in ferrite content of welds with PEC wire has been theorized to be through retention of base metal nitrogen content. In the case of 304L, the addition of Nitrogen through the shielding gas further reduced the ferrite content. An increase in the nitrogen solubility of the weld metal is theorized through additions of manganese with the PEC cored wire and 308L solid wire alloy additions. Due to the decreased desorption of nitrogen from the weld metal in welds with a surface active oxide layer, the formation of porosity was seen in samples with 5% and 10% Nitrogen. Shown in Figure 102.[29, 32]

Micro hardness traverses were made across the fusion boundary from the base metal to the weld metal of welds with 10% Nitrogen addition. The Vickers hardness values recorded in this region were of near base metal hardness for welds without PEC addition and increased in hardness in the fusion zone. With PEC addition the hardness increased consistently into the fusion zone. See Figure 103.

The effects of nitrogen additions to welds with 308L solid wire addition are shown in Figure 100. An increase of hardness was seen in samples with 308L solid wire additions through increased solubility of nitrogen through alloy additions of manganese. The
increase in hardness was less than PEC cored wire additions, likely due to the increased rate of desorption through absence of a surface active oxide layer. The ferrite contents and hardness values can be seen in Figure 100.
Figure 98: 304L Autogenous Welds with Nitrogen Shielding Gas Additions

Figure 99: 304L Welds with PEC Flux Cored Wire Additions and Nitrogen
Figure 100: 304L 308L Filler Wire and Nitrogen Shielding Gas Additions
The addition of 2% Nitrogen could potentially be used for increased intergranular corrosion resistance due to the reduction of interconnected ferrite. The increase in corrosion resistance also must be balanced by the increased crack sensitivity of the weld during solidification, see Figure 24.

A paper by T.A. Palmer and T. Debroy discuss the role that monatomic nitrogen plays in the absorption and desorption of nitrogen in GTA welds. Through modeling, the transfer and arrangement of nitrogen in welds is directly related to the convection patterns formed in the welds and is influenced by the turbulence of the weld pool [47]. In the case of welding with PEC cored wire, both of these mechanisms are increased. Marangoni flow is directed inward and down, drawing nitrogen into the weld metal, and increased turbulence caused by the PEC additions increase the diffusion coefficient and absorption rate. This may explain why porosity was seen in welds with the addition of nitrogen and PEC but not in autogenous welds. The location of the porosity near the fusion boundary and near the sides of the weld may also be theorized using the same principles. The increase in turbulence and diffusion coefficient in the center of the weld puddle also increases the absorption of nitrogen deep into the weld metal. As turbulence and temperature decrease, the formation of diatomic nitrogen may be preferred due to the decreased diffusion coefficient and decreased solubility of nitrogen in the metal. [47]
Figure 101: 304L Unmixed Zone with Cored Wire Addition, Ar +10N₂ DIC prism and polarizer used, ferritic phase is white.

Figure 102: 304L with Cored Wire Added, A) Ar +5%N₂ B) Ar +10%N₂
10.2: Additions of Nitrogen to the Shielding Gas during Welding of 21-6-9

Results from Nitrogen addition to the shielding gas during welding of Nitronic 40 had a greater effect on weld microstructure than welds on 304L base material. Reduction of ferrite content was more uniform in welds without PEC cored wire with increased Nitrogen content in the shielding gas. Hardness was also more consistent with nitrogen shielding gas added. The formation of porosity was not evident in the Nitronic weld trials; however, no NDE methods were used to characterize porosity formation. The use of nitrogen shielding gas in this study was more focused on the Nitronic alloy due to the intentional alloying additions of nitrogen in the base material. Retention of this nitrogen...
content then becomes a concern to maintain proper phase balance, mechanical properties, and corrosion resistance [37]. With use of the PEC cored wire, ferrite content is drastically reduced with increased additions of nitrogen to the shielding gas. The effects of nitrogen shielding gas additions on ferrite content and hardness can be seen in Figure 104, Figure 105, and Figure 106, for autogenous, PEC cored wire, and 308L solid wire additions respectively.

Solidification mode of the Nitronic alloy was altered by the addition of nitrogen in the shielding gas. With increasing nitrogen content, the amount of retained ferrite decreased correspondingly, while maintaining primary ferrite solidification. The addition of solid wire 308L filler material decreased ferrite content potentially through the increase in weld puddle turbulence caused by wire addition, increasing the absorption rate of Nitrogen. A transition to primary austenitic solidification mode could be seen near the center of the weld when adding 10% Nitrogen to the Argon shielding gas. Addition of PEC cored wire to the Nitronic alloy welds had a drastic effect on ferrite content as mentioned in Chapter 8. The addition of Nitrogen to the shielding gas of welds with PEC cored wire increased the reduction of ferrite content to less than 2%. Solidification mode of these welds was primarily FA in the unmixed region of the weld near the fusion boundary, with a change in solidification mode in the remainder of the weld as primary austenitic. The hot cracking sensitivity of welds with PEC cored wire and Nitrogen shielding gas was increased in the center of the welds, therefore Nitrogen addition to
Nitronic alloys with PEC is not recommended for most applications. The solidification types are shown in Table 16.

Hardness values varied according to ferrite content. As shown in the following figures, the narrowest distribution of hardness was measured in the samples with PEC cored wire addition. These samples also had the least variation in ferrite content amongst the four welding conditions. Autogenous and welds with 308L solid wire addition had larger hardness ranges most likely due to the variations in nitrogen content experienced by the welds. Without a slag formation on the top surface of the weld, the absorption and loss of Nitrogen could not be strictly controlled. Because these are influenced by weld pool turbulence, variability is expected due to the nature of the welds.

<table>
<thead>
<tr>
<th>% Nitrogen</th>
<th>Autogenous</th>
<th>308L Solid Wire</th>
<th>PEC Cored Wire</th>
</tr>
</thead>
<tbody>
<tr>
<td>0%</td>
<td>FA</td>
<td>FA</td>
<td>FA</td>
</tr>
<tr>
<td>2%</td>
<td>FA</td>
<td>FA</td>
<td>FA / AF</td>
</tr>
<tr>
<td>5%</td>
<td>FA</td>
<td>FA</td>
<td>FA / AF</td>
</tr>
<tr>
<td>10%</td>
<td>FA</td>
<td>FA / AF</td>
<td>FA / AF</td>
</tr>
</tbody>
</table>

Table 16: 21-6-9 Solidification Mode with Nitrogen Additions to Shielding Gas, Balance: Argon

Micro hardness traverses were made on welds with 10% Nitrogen addition. Hardness values for welds completed autogenously, with 308L solid wire, and with PEC cored wire exhibited the same trend in hardness across this region. The variations between the three welds were also well within the deviations seen in the hardness maps completed on the
fine scale of micro hardness. A uniform hardness traverse with a moderate hardness increase in the weld metal was recorded for all samples. The 10% Nitrogen addition samples were further evaluated due to the distinct unmixed zone near the fusion boundary.
Figure 104: 21-6-9 Autogenous Welds with Nitrogen Shielding Gas Additions

Figure 105: 21-6-9 Welds with PEC Flux Cored Wire and Nitrogen Shielding Gas Additions
Figure 106: 21-6-9 with 308L Filler Metal and Nitrogen Shielding Gas additions to Argon Shielding Gas
Figure 107: Micro Hardness Traverse across Unmixed Zone of Nitronic 40 Alloy
**Figure 108:** Alloy 21-6-9 with 5% Nitrogen added to Shielding Gas

**Figure 109:** Solidification Mode near Center of Welds - Optical Micrograph - 1000x Original Magnification.
Chapter 11: Electron Microscopy and Analysis

The Center for Electron Microscopy and Analysis (CEMAS) at The Ohio State University and the Analytical Laboratory at the NNSA Kansas City Plant were used to do in depth metallurgical characterization of the resultant weld microstructures. Imaging using Secondary Electron (SE), Back Scattered (BSE) as well as Energy Dispersive X-ray analysis (EDS) were used in this investigation. Samples prepared during the parameter and process characterization studies were re-polished to 0.05µm using colloidal silica to remove etchant when needed.

11.1: PEC Powder Analysis

To better understand the evolution of PEC from the weld material, an analysis was conducted on the stock material provided by EWI. The PEC provided was of the same heat and lot as the material used in the fabrication of the cored wire. As provided, the powder is fine and green in color. Some small clumps or chunks could be seen in the mixture. SEM analysis of the dry powder revealed large chromium oxide particles of approximately 100µm to 200µm in diameter. After mixing with acetone and painting on to the surface of a SEM sample stub, the large particles were still evident on the surface. Large particles of equal size were found after analyzing the painted on PEC. Although evidence of some oxides dissolving through use of the acetone exists, the primary
The purpose of the acetone is to suspend the solution long enough for application onto the surface of the part to be welded. Upon drying, the powder does adhere lightly to the surface, although can be disturbed easily causing it to flake off. Very little change could be seen in the powder after solvent deposition. A more uniform distribution of the fine powder was seen. In Figure 110, the EDS spot analyses of a large oxides present in the powder before and after deposition on the SEM stub can be seen. Large amounts of chromium were measured in all EDS spot and full frame analyses with consistent silicon content. Increased titanium concentrations were found on intermediate sized particles, number 3 in Figure 110, along with chromium and silicon.

![PEC Compound Analysis](image)

<table>
<thead>
<tr>
<th>As Received Powder</th>
<th>After Mixing with Acetone and Painting onto Stub</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image" alt="Image" /></td>
<td><img src="image" alt="Image" /></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>wt%</th>
<th>Cr</th>
<th>Si</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Spot</td>
<td>86.29</td>
<td>9.90</td>
<td>3.81</td>
<td></td>
</tr>
<tr>
<td>2. Spot</td>
<td>87.19</td>
<td>9.09</td>
<td>3.74</td>
<td></td>
</tr>
<tr>
<td>3. Spot</td>
<td>64.66</td>
<td>8.52</td>
<td>26.82</td>
<td></td>
</tr>
</tbody>
</table>

Figure 110: Analysis of PEC
11.2: EDS of Partial Penetration Welds

Initial SEM analyses of welds were completed at the Kansas City Plant, Analytical Laboratory. Partial penetration welds were initially checked for slag inclusions and EDS was used to determine propensity of oxide entrapment. Full frame EDS traces were used to capture as much area of the weld as possible and systematically scanned to include the entire weld profile. Tests were run overnight to capture data at an accelerating voltage of 20kV, spot diameter of 4µm, and working distance of 9.5 mm. Emphasis was placed on the whereabouts of oxide constituents; Chromium, Silicon and Titanium. Titanium is the only alloying element not native to the base metal chemistry and is of the highest concern to Honeywell. Welds were made using the same heat of 304L material with chemical compositions provided by Honeywell for sample analysis. Welds were completed autogenously and with cored wire addition of PEC, and 100% Argon shielding gas.

Comparisons between autogenous and PEC added partial penetration welds revealed little increase in PEC constituents in the weld metal. Traces were completed on welds with cored wire addition at 200, and 300 Amps for comparison to autogenous welds of equal arc parameters. Most traces revealed no discernible amount of titanium in the weld metal. Of the traces captured with titanium peaks, higher levels were found in the 300 Amp welds. Quantification of titanium composition was 0.03 wt% in the weld metal and 0.02 wt% in the weld reinforcement area, see Figure 111. These compositions were limited to a small area of the weld metal and could not be resolved to a specific phase or particle. Other notable differences found in the weld metal of cored wire welds were increased...
Mn, Si, Ti, and Cr, each an alloying constituent of the deposited weld wire. In the manufacture of the PEC cored wire other alloying elements common to 308L were used, including Manganese. This addition can be seen in the EDS traces of welds with cored wire addition, increasing the composition by roughly 0.15 wt% in the weld metal. Analysis of the weld metal only was not definitive in determining PEC evolution through welding.

<table>
<thead>
<tr>
<th>5 ipm Flux Cored Wire</th>
<th>Chemical Certification</th>
<th>SEM + EDS Analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 30</td>
<td>Base Metal</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Base Metal Avg.</td>
<td>18.23</td>
</tr>
<tr>
<td></td>
<td>Weld Metal Avg.</td>
<td>18.64</td>
</tr>
<tr>
<td></td>
<td>Reinforcement Avg.</td>
<td>18.78</td>
</tr>
<tr>
<td></td>
<td>Base Metal Max</td>
<td>18.65</td>
</tr>
<tr>
<td></td>
<td>Weld Metal Max</td>
<td>18.91</td>
</tr>
<tr>
<td></td>
<td>Reinforcement Maximum</td>
<td>18.78</td>
</tr>
<tr>
<td>Cr</td>
<td>18.23</td>
<td>18.64</td>
</tr>
<tr>
<td>Ni</td>
<td>8.07</td>
<td>7.96</td>
</tr>
<tr>
<td>Mn</td>
<td>1.77</td>
<td>2.17</td>
</tr>
<tr>
<td>Si</td>
<td>0.31</td>
<td>0.48</td>
</tr>
<tr>
<td>Ti</td>
<td>0.00</td>
<td>0.00</td>
</tr>
<tr>
<td>Mo</td>
<td>0.32</td>
<td>0.41</td>
</tr>
<tr>
<td>Cu</td>
<td>0.43</td>
<td>0.64</td>
</tr>
<tr>
<td>Fe</td>
<td>balance</td>
<td>69.72</td>
</tr>
</tbody>
</table>

Figure 111: Avg. Compositional Analysis Values (wt%) through EDS at Analytical Laboratory, Kansas City

11.3: EDS of Attached Slag Remnants

As mentioned previously, the slag formation on the top surface of the weld appears to have two different consistencies. A dark, rough, tenacious slag adheres to the surface of the weld in addition to a more smooth, glass type slag that often springs off of the weld surface revealing a clean well shielded weld reinforcement. A characterization of the slag was completed to determine composition.
Spectroscopy of slag elements captured on the surface of the weld in the sample mounts revealed at least two primary slag formations. The first slag elements that were adhered to the surface of the weld are primarily Iron, with increased amounts of Silicon. Some small remnants found on the surface of the weld were determined to be primarily Titanium, Chromium, and Silicon with additions of Iron, Aluminum, and Manganese. As shown in Figure 113, a sample with the top surface of the weld orientated towards the bottom left of the page, reveals increased titanium in this section. Additional titanium slag elements were found in the mounts on slag elements that were removed from the surface during mounting and captured in the Bakelite. Proper orientation was maintained in this sample, Figure 114.

Figure 112: Example EDS Trace
11.4: EDS of Captured Slag Remnants after Welding

The slag found remaining in the mounts from samples with PEC cored wire addition were of limited size and found in small quantities on the samples tested. Additional characterization of slag was performed at the Center for Electron Microscopy and Analysis (CEMAS) at The Ohio State University. This analysis consisted of capturing slag elements after welding. Remnants were allowed to spring off the weld surface during cooling and were captured in a poly bag during flight. Samples were prepared by using mounting tabs with carbon adhesive tape. Random slag elements were placed on the surface to capture representative chemistries and orientations for external and internal slag surfaces. The FEI Quanta 200 at CEMAS was utilized for analysis, Figure 115.
SEM analysis of the slag remnants recovered from the welds consisted mainly of oxide constituents. Chromium, Titanium, and Manganese were found in all locations of the slag. Results of tests can be seen in Figure 116. The morphology of the slag remnants varied greatly from sample to sample with variations in concentration. Dark regions were found to be enriched in titanium, whereas globular areas of light coloration contain significantly more silicon. Through analysis of the slag remnants, the main constituent in slag that is expelled from the surface of the weld is titanium. Due to the difference in solidification temperature and thermal expansion, the expulsion of titanium slag elements is plausible.
Through microstructural analysis of both the weld metal and slag remnants of partial penetration welds, an argument can be made to validate removal of oxide constituents through the formation of the slag. Small fractions of the PEC constituents do remain in the weld metal.

Figure 116: EDS Analyses of Slag Remnants, spot and full frame.
11.5: Full Penetration SEM Analyses

The analysis of partial penetration welds revealed the propensity of PEC removal through the slag. In partial penetration welds the evolution of PEC constituents was directed towards the weld reinforcement, the only exterior surface of the weld. An evaluation of the full penetration welds was sought to determine if the presence of another free surface would alter this process. Samples were prepared through metallographic sectioning and polishing to 0.05µm with colloidal silica. Bulk chemistry values were taken at several locations across the weld through full frame raster scans of the incident electron beam. Analysis was completed at 20kv, 4µm spot size, and 1600x magnification at locations identified in Figure 117. Through analysis of the six EDS traces captured on sample 48E, increased amounts of PEC constituents can be seen in the weld area. Unlike the partial penetration welds, the entrapment of oxide constituents increased to a greater extent. The largest values were found at the root and weld reinforcement area of the weld. Base metal compositions of the tubes can be seen in Table 23. Base material compositions of 18.25 wt % Cr, 0.37wt% Si, and 0 wt% Ti were provided through material certifications from the supplier. The filler metal ranges were provided from ESAB and shown in Table 19. 20-22wt% chromium, <1wt% silicon, 0 wt% titanium were of the filler metal composition. The oxides included as part of the filler wire are additional to those components in compositions of 1-5wt% Cr₂O₃, <1wt% SiO₂, and 1-5wt% TiO₂.

Increases in the compositions of the flux components were seen in the weld metal of the full penetration weld. Electron diffraction spectroscopy was taken at 6 positions on the
weld cross section. Base metal samples were also taken for comparison of local material chemistry that may vary from the certification. Because no titanium was in the base material, all of the titanium measured was found in the weld metal as expected through alloy dilution of the filler metal. An increase up to 0.78 wt% titanium was measured with a maximum value recorded at the root of the weld. A small increase in silicon content was also recorded through the weld zone increasing by 0.38wt%. Entrapment of titanium was increased in the full penetration welds. An average value of 0.58 wt % was measured in the full penetration welds vs. 0.03 wt % in the partial penetration welds. Concentration profiles of the constituents can be seen in Figure 118.

<table>
<thead>
<tr>
<th>Location</th>
<th>Cr</th>
<th>Ni</th>
<th>Si</th>
<th>Ti</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 (Base Metal)</td>
<td>18.71</td>
<td>9.75</td>
<td>0.96</td>
<td>0</td>
<td>70.57</td>
</tr>
<tr>
<td>2 (Reinforcement)</td>
<td>19.39</td>
<td>10.54</td>
<td>1.15</td>
<td>0.62</td>
<td>68.3</td>
</tr>
<tr>
<td>3 (Weld Metal)</td>
<td>19.03</td>
<td>9.64</td>
<td>0.87</td>
<td>0.49</td>
<td>69.96</td>
</tr>
<tr>
<td>4 (Weld Metal)</td>
<td>19.5</td>
<td>9.36</td>
<td>0.35</td>
<td>0.44</td>
<td>70.36</td>
</tr>
<tr>
<td>5 (Weld Root)</td>
<td>19.58</td>
<td>9.57</td>
<td>1.34</td>
<td>0.78</td>
<td>68.73</td>
</tr>
<tr>
<td>6 (Base Metal)</td>
<td>19.99</td>
<td>9.92</td>
<td>0.87</td>
<td>0</td>
<td>69.22</td>
</tr>
</tbody>
</table>

Figure 117: Full Penetration EDS Analysis
The evolution of the oxide constituents is likely driven by not only weld metal fluid flow and density, but also segregation during solidification of the weld pool. In the case of the partial penetration welds the growth directions are normal to the fusion boundary and have a net upward driving force towards the surface of the weld. This is not the case for full penetration welds. As shown in Figure 119, the growth directions of the full penetration welds are largely toward the center of the weld throughout the thickness of the weld joint. Through longitudinal sectioning a slight upward solidification substructure can be seen in the center of the weld that increases in slope as solidification nears the weld reinforcement. Upon solidification in the full penetration weld the PEC compounds would be segregated to the center of the weld region, then would follow the
solidification mode upward to the surface of the weld. The entrapment of PEC constituents in the weld pool can be best explained by the solidification directions and flow of material in the weld puddle. See Figure 119. Based on the solidification structure seen in full penetration welds it is understandable that additional entrapment of oxide constituents would remain in the solidified weld. Solidification directions in partial penetration welds are perpendicular to the fusion boundary. In the case of partial penetration welds the solidification directions are focused to center of the weld reinforcement. This aids in the removal of PEC constituents. Full penetration welds have two walls to remove heat, thereby changing the solidification directions of the weld pool. The cooling direction is therefore perpendicular to the joint design. This implies a more uniform heat distribution across the weld joint with little driving force for removal. In this case not only is the distance for removal of constituents increased, but the assistance due to solidification directions is also reduced. The alloying of the titanium with the weld metal occurs for some of the titanium as measured in the EDS analysis. The remaining portion of the titanium is removed through the oxide layer. Further analysis is needed to explain the evolution of titanium in the weld pool. Through formation of the oxide layer on the surface of the weld, it is likely that titanium, with a high level of reactivity, interacts with oxygen in the arc or on the surface of the weld to form titanium oxides that are then segregated from the weld pool through buoyancy forces.
Figure 119: Solidification Growth Directions of Partial and Full Penetration Welds (A: Sample 5A Transverse, B: Sample 48E Transverse, C: Sample 64C Longitudinal Section)
During initial weld trials an opportunity arose to use equipment at a nearby company named WeldQC. A weld vision camera developed by WeldQC was used to record and analyze welds made with the cored wire addition and paint on PEC. The camera system was developed to allow for greater vision on the electrode and weld puddle through an optical band pass filter that blocks most of the light rays emanating from the arc, but allows for the red wavelength of light emitted by diodes on the opposite side of the camera. Tests were conducted to see if weld pool fluid flow could be seen in the video. The weld pool was readily visible using this camera system however the lack of features in the weld pool made a determination of the molten puddle fluid flow direction inaccurate. It was found that using the camera system an arc column was still visible. One of the methods of increased penetration argues that arc intensity is focused through use of the PEC. Analysis of the images captured from the camera system was completed using Image J to determine the amount of constriction. An intensity profile was used to map the intensity of the arc below the electrode. Each profile was placed the same number of pixels below the tip of the electrode and centered on the weld pool. Reflections from the weld pool were minimized through selective image capture. A full width half maximum measurement was sought however this was not possible due to the weld puddle reflections obscuring the data. The camera iris, focus, and capture rates were not changed.
Throughout the tests to provide the same relative intensities for each weld type. The calculated width in pixels was 38, 37, and 32 for Autogenous, Paint on, and Cored Wire addition respectively.

![Figure 120: Comparison of Welding Arcs, A) Autogenous, B) with Paint on oxide layer, C) with flux cored wire addition](image)

Arc constriction was shown to exist in welds captured with the Weld QC camera. The use of multiple band pass filter images with distance calibration could be used to define the amount of arc constriction. Measurement of arc constriction was completed using a camera system designed to view a narrow band of light for viewing of the weld surface through specific LED’s of this bandwidth. This wavelength was set to be in the dark region of emitted light during gas tungsten arc welding. The filter is not perfect in blocking all light, and is saturated, allowing for the transmission of light from the arc at considerably less intensity than normal viewing. Designed to be a weld vision system, images were captured with paint on and cored wire addition for comparison to autogenous welding. It was this light that was measured for comparison. Because this
filter blocks out a lot of the low level noise that would be recorded through a standard optical fiber and diode, a cleaner signal can be seen of the high intensity arc. Other studies on arc constriction have focused on images without filtering, obtaining saturated signals of limited information [3, 4, 16, 49].

![Comparison of Arc Intensity](image)

**Figure 121: Image Intensity Evaluation**

Through measurements of arc intensity the restriction through paint on and cored wire addition were 3% and 16%, respectively. The main mechanism for penetration seen in this study was Marangoni flow. Arc constriction was seen to influence welds through increased arc force resulting in craters left in welds at arc termination locations. Oxide
constituents were also seen on the surface of the weld pool circulating as described by Sandor [17] and shown in Figure 123.

Figure 122: Representative Sample of Captured Images by WeldQC Camera System, Autogenous Weld
Figure 123: Surface Active Oxides Present in Circulation of Pool (Clockwise)
Additional analysis was completed on partial penetration samples to determine PEC constituent entrapment at the Kansas City Plant Analytical Laboratory using a Laser Ablation Mass Spectrometer. A Thermo Fisher, X series 2 Inductively Coupled Plasma – Mass Spectrometer (ICP-MS) coupled with a New Wave Research/ESI UP-213 laser ablation system was used. The same partial penetration weld samples used in the EDS analysis at KCP were used in this analysis. The ICP-MS was operated in a Time Resolved Analysis (TRA) mode to determine elemental concentration changes from the base metal through the weld metal. A distance of 50µm below the original base material surface was chosen for comparison between shallow non-PEC welds and welds with PEC, Figure 125. Scans were completed using a 10 msec uptake delay for background concentration data and then acquired for each sample with a 10 msec dwell time. The laser was operated under the following parameters:

- 10 Hertz
- 10 µm/sec scan rate
- 100µm spot size
- 100% Power (36.5 J/cm² fluence)
Results of the laser ablation mass spectrometry test were qualitatively good, with uncertain quantitative data. Analysis of the data received, reveals no change in composition from the base metal to weld metal. Due to the process of capturing the fume from laser ablation, the test may not be accurate for discriminating between the scan thru the base metal and the weld metal as performed. Additional scanning time through the base metal may be necessary or separation of tests to ensure clean data. A measured titanium content of 0.003 wt% was measured through laser ablation spectroscopy compared to 0.030 wt% through EDS on partial penetration welds with similar PEC additions and arc parameters. Measurements completed on six samples can be seen for titanium content and silicon content in Figure 127 through Figure 129. In both silicon content and titanium content the lesser wire feed speed retained additional alloying content in the weld metal at 200 amps. This result was not as expected and requires further evaluation. An expected response was seen in the welds at 300 amps, whereas increased PEC addition results in increased weld metal content. The unexpected behavior
at 200 amps may potentially be due to the greater efficiency of penetration at 5 ipm cored wire addition. This increased efficiency may allow for greater entrapment due to weld pool turbulence and/or the formation of slag is diminished through low alloying element availability, leaving increased quantities of the alloying elements in the weld metal. Further investigation is needed to fully understand the variations seen in measurements through the LAMS process.

Figure 125: Sample 36 after analysis by Laser Ablation Mass Spectroscopy
Figure 126: Residual Titanium Content Using ICP-MS with Laser Ablation, 200 Amps

Figure 127: Residual Titanium Content using ICP-MS with LASER Ablation, 300 Amps
Figure 128: Residual Silicon Content in Partial Penetration Welds, 200 Amps.

Figure 129: Residual Silicon Content in Partial Penetration Welds, 300 Amps.
Chapter 14 : Conclusions

The penetration enhancement through use of penetrating enhancing compounds was demonstrated using both paint and flux cored wire addition. The following conclusions are a result of the work presented in this document.

14.1: Process Performance of PEC Flux Cored Wire GTA Welding

1. An increase in penetration of 293% was measured in welds completed on low sulfur heats of 304L. Sulfur contents less than 0.001 wt% were used. Increased sulfur content reduced penetration enhancement however resultant penetration was equal with PEC cored wire addition.

2. The increase in penetration of 21-6-9 with PEC cored wire reached 226%. Although surface texture and arc characteristics of welding 21-6-9 were negatively impacted by use of the PEC cored wire, consistent and clean weld penetration was achieved.

3. 3/8” full penetration, single pass tube welds with root and cap reinforcement was achieved with backing gas pressurization (304L). Full penetration welds were successfully made with the careful adjustment of input pressure and amount of
restriction at the exit port. An internal pressure of 4 to 5 inWC (0.11 to 0.18 psi) was required to maintain the puddle in the weld joint.

4. 14% and 21% reduced penetration variability in 304L and 21-6-9 respectively. Standard deviations of penetration variation were approximately equal in autogenous welds and welds with PEC addition. As a percentage of total penetration the variation was less significant in welds with PEC addition due to greater penetration depth.

14.2: Hardness Tests

1. Rockwell B hardness indents of welded samples maintain hardness through fusion zone of welds in 304L and 21-6-9 with PEC addition. A drop in hardness of approximately 5 to 10 Rockwell B was measured in the fusion zone of 304L welds with 308L filler metal or welded autogenously. A decrease of 10 Rockwell B was measured in the fusion zone of 21-6-9 welds with 308L filler metal or welded autogenously. Base Metal hardness was maintained throughout the fusion zone of welds in 304L and 21-6-9 with PEC additions. The h(BM ~75 Rockwell B, 304L) (BM ~92 Rockwell B, 21-6-9)

2. Additions of nitrogen to the coaxial shielding gas of autogenous welds in 304L revealed an inconsistent hardness response with nitrogen additions. With PEC addition to the welds in 304L, Rockwell B hardness measurements increased with volume % nitrogen surpassing the base metal hardness by up to 10 Rockwell B.
3. Additions of nitrogen to the coaxial shielding gas of autogenous 21-6-9 increased fusion zone hardness similarly to 304L without PEC additions. No correlation was seen with increased additions of nitrogen in the shielding gas. With the addition of PEC flux cored wire to the welds on 21-6-9 base material, the hardness indentations were very consistent across all levels of nitrogen addition. Due to the retention of nitrogen in welds with PEC, retained ferrite content was minimal, having been reduced to 1.12FN with 2% vol. nitrogen. No increase in fusion zone hardness was measured with increased nitrogen shielding gas additions.

14.3: Metallurgical Behavior

1. The retained ferrite content in welds completed using 304L base material and PEC cored wire addition increased 0.5 FN after correction due to measurements through the oxide layer. Ferrite content increases with additions of PEC flux cored wire up to 1 FN at 15 ipm of wire feed speed at 2.5 ipm travel speed. Up to 4 inches of wire feed speed per linear inch of travel resulted in an increase of 0.5 FN. Above 4 inches of wire feed speed per linear inch of travel, the increase in ferrite content was measured to be 1.5 FN after correction.

2. 52% Reduction in ferrite content in 21-6-9 with PEC flux cored wire addition vs. 308L solid wire. A reduction in ferrite content was measured with use of 308L and PEC flux cored wire addition vs. Autogenous welds. (308L: 9.11FN, PEC: 4.35 FN, Autogenous: 9.69FN)
3. No slag inclusions, or formations of intermetallic compounds, such as TiN were found in metallography or SEM analysis of weld joints. The evolution of PEC material was shown to alloy into the weld metal or be removed through formation of a slag layer on the surface of the weld.

4. No change in solidification mode was found in welds of 304L or 21-6-9 using 100% Argon shielding gas. Solidification mode was maintained as Type III FA.

5. An increase in alloying content of PEC elemental constituents was found in full penetration welds when compared to partial penetration welds. A maximum titanium content of 0.78wt% measured in the root of a 3/8” wall thickness tube weld compared to a maximum of 0.03 wt% titanium measured in partial penetration welds.

6. The addition of 2% nitrogen to the coaxial shielding gas reduces ferrite content below the recommended value of 3 for solidification cracking resistance when using PEC during welding of both materials. Due to the increase in nitrogen solubility in 21-6-9, the use of nitrogen shielding gas can be used to further decrease the ferrite content without the formation of porosity in welds. (2.24 FN 304L, 1.12FN 21-6-9)

14.4: Arc Constriction

1. A 16% reduction of the arc plasma column diameter was calculated through image analysis of welds captured through use of a WeldQC ClearVision camera system.
2. A reduction of 3% was measured in welds using paint on flux.
References


183
Appendix A : Process Characterization
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Table 23: Material Compositions
### Table 24: SEM, EDS Summary of 300 Amp, Autogenous Partial Penetration Weld

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<thead>
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<th>Autogenous Chemical Certification</th>
<th>SEM + EDS Analysis</th>
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<td>Sample 34 Base Metal</td>
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<td>Cr</td>
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<td>Mn</td>
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<tr>
<td>Ti</td>
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<td>Mo</td>
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<tr>
<td>Cu</td>
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<tr>
<td>Fe</td>
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### Table 25: SEM, EDS Summary of 300 Amps, 20 ipm PEC Cored Wire Addition, Partial Penetration Weld

<table>
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<th>20 ipm WFS PEC Cored Wire Chemical Certification</th>
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<td>Sample 33 Base Metal</td>
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<tr>
<td>Ni</td>
<td>8.07</td>
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<td>Mn</td>
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<td>Si</td>
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<td>Mo</td>
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<td>Cu</td>
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### Table 26: SEM, EDS Summary, 200 Amp Autogenous Partial Penetration Weld

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<td>Mo</td>
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</tr>
<tr>
<td>Cu</td>
<td>0.43</td>
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<tr>
<td>Fe</td>
<td>balance</td>
</tr>
</tbody>
</table>

Table 24: SEM, EDS Summary of 300 Amp, Autogenous Partial Penetration Weld

Table 25: SEM, EDS Summary of 300 Amps, 20 ipm PEC Cored Wire Addition, Partial Penetration Weld

Table 26: SEM, EDS Summary, 200 Amp Autogenous Partial Penetration Weld
Figure 130: Sample 34, 300 Amps, Autogenous

Figure 131: Sample 36, 300 Amps, 10ipm PEC Cored Wire Addition
Figure 132: Sample 34, 300 Amp Autogenous, Weld Center

Figure 133: Sample 36, 300 Amps, 10ipm PEC Cored Wire Addition, Weld Center
Figure 134: 308L Solid Wire Composition Ranges