Optimization and Application of the Strain-To-Fracture Test for Studying Ductility-Dip Cracking in Ni-base Alloys

THESIS

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Abstract

The use of non-ferrous alloys in the nuclear power industry has increased over the past several decades due to industry demands for improved materials properties. Specifically, Inconel Alloy-690 and matching filler metal, FM-52, have been developed with an increased Cr-content (28-30 wt. %), which has demonstrated improved properties in corrosive environments. The development of high-Cr, Ni-base alloys has however, led to weldability concerns, such as ductility-dip cracking, that have challenged manufacturers and users of these consumables.

Concerns with ductility-dip cracking in FCC materials has led to the development of the Strain-To-Fracture test at Ohio State, which is a weldability test designed to quantify DDC susceptibility in austenitic materials. A Gleeble® 3800 is used to generate a thermo-mechanical response cycle in which a material is isolated within the ductility dip temperature range, and subsequently strained to a desired level. Due to variability in the testing environment and the desire to employ Ni-alloy and FM’s in the STF test, a “re-optimization” study was conducted to ensure parameters were in a robust region that would produce repeatable results. Insights into DDC mechanisms were also made by studying commonly used Ni-alloys with compositional/microstructural variations as well as specimens that were generated with various fabrication methods. It was determined that composition has a large influence on DDC performance, and “pure” materials tend to be most susceptible. Also, fabrication method does appear to have some influence over
DDC formation in heats of FM-52M. A digital image correlation study was also conducted to confirm strain levels in sample gauge sections.
Dedication

To my loving parents, for their support and guidance has shown me what is possible through patience, passion, and perseverance, and that doing what you love is a privilege that should not be taken for granted. For that and many other things, I am extremely grateful.
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Table of Contents

Abstract................................................................................................................................. ii
Dedication ............................................................................................................................... iv
Acknowledgments ................................................................................................................ v
Vita ........................................................................................................................................ vi
Publications .......................................................................................................................... vi
Fields of Study ..................................................................................................................... vi

Table of Contents ................................................................................................................. vii

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

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

Chapter 1: Introduction ...................................................................................................... 1

Chapter 2: Background .......................................................................................................... 6

2.1 Ductility Dip Cracking Definition ................................................................................ 6

2.2 Ductility Dip Cracking in Ni-base Alloys .................................................................... 7

2.3 Ductility Dip Cracking: Microstructural Characteristics ........................................... 11

2.4 Ductility Dip Cracking Formation in Various Ni-base Alloys and Filler Metals ... 18

2.5 DDC Mechanisms .......................................................................................................... 19

2.6 STF Testing Method ....................................................................................................... 21
Chapter 3: Objectives.............................................................................................................. 28

Chapter 4: Experimental Approach ....................................................................................... 30

Chapter 5: Experimental Procedure ....................................................................................... 33

5.1 Materials.......................................................................................................................... 33

5.1.1 Base Metal Compositions......................................................................................... 33

5.1.2 Ni-base Filler Metals .............................................................................................. 35

5.2 STF Re-Optimization ...................................................................................................... 36

5.2.1 GTA Spot Weld Parameter Optimization................................................................. 37

5.2.2 STF Optimization .................................................................................................... 39

5.3 Sample Preparation .......................................................................................................... 42

5.3.1 Heat Treatments ....................................................................................................... 43

5.3.2 Oxygen Additions .................................................................................................... 44

5.3.3 Limited Material Sample Preparation ................................................................... 45

5.3.4 STF Digital Image Correlation ............................................................................... 46

5.4 Round Robin Sample Fabrication and Preparation .......................................................... 47

5.4.1 WSI/AZZ Sample Fabrication .............................................................................. 48

5.4.2 Special Metals Sample Fabrication ....................................................................... 51

5.4.3 EPRI Sample Fabrication ...................................................................................... 53

5.5 Sample Characterization ................................................................................................ 54
Chapter 6: Results and Discussion ................................................................. 57

6.1 Phase I: STF Parameter “Re-Optimization” ........................................ 58
  6.1.1 GTA Spot Weld Optimization ...................................................... 59
  6.1.2 Gleeble Parameter Optimization .................................................. 65

6.2 Phase II: Insight to DDC Mechanism .................................................. 73
  6.2.1 STF Testing of Various Ni-base Alloys ........................................ 74
  6.2.2 Alloy 200 .................................................................................. 75
  6.2.3 Inconel Alloy-600..................................................................... 79
  6.2.4 Inconel Alloy-625 ..................................................................... 81
  6.2.5 The Effect of Oxygen on DDC susceptibility ................................ 84
  6.2.6 STF Sample Heat Treatments .................................................... 85

Phase III: 6.3 STF Round Robin Study ....................................................... 88
  6.3.1 NX4720TK ............................................................................ 89
  6.3.2 NX5213TK ............................................................................ 91
  6.3.3 EXOA51P ............................................................................. 93
  6.3.4 Compiled Round Robin Results ................................................ 95
  6.3.5 Effect of Oxygen Concentration on STF Round Robin Study ......... 101
Phase IV: 6.4 “New” STF Testing Procedure ......................................................... 103

Phase V: STF Digital Image Correlation ............................................................... 105

Chapter 7: Conclusions ......................................................................................... 108

Chapter 8: Recommended Future Work .............................................................. 112

References ............................................................................................................. 115

Appendix A ........................................................................................................... 118

Gleeble QuickSim Program for STF Test .............................................................. 118
List of Tables

Table 1: Historically Significant DDC Studies in Ni-base Alloys and FM’s. ............... 19
Table 2: Theorized DDC Mechanisms Summary ................................................................. 21
Table 3: GTA Spot Weld parameter schedule for STF testing method ....................... 24
Table 4: Compositional data (wt. %) for Ni-base materials used in the presented STF study .............................................................................................................................................. 35
Table 5: Ni-base FM, FM-52M, compositional data (wt. %) for the 3 heats used in the round robin study (NX4720TK, EXOA51P, NX5213TK) ................................................................. 36
Table 6: STF optimization parameter schedules for GTA spot weld. ....................... 38
Table 7: Gleeble parameters used during the spot weld optimization study. ........... 39
Table 8: Gleeble-based parameters for the STF testing procedure, developed by Nissley [15]. ................................................................................................................................................................. 40
Table 9: Gleeble parameter optimization schedules for INCONEL Alloy-600. .......... 42
Table 10: Gleeble parameter optimization schedules for INCONEL Alloy-690. ....... 42
Table 11: Heat treatment schedules used on STF samples in Phase II .................. 44
Table 12: Weld procedure specification for the fabrication of STF samples in the round robin study. ................................................................................................................................. 48
Table 13: Parameters associated with the fabrication of WSI’s STF samples. ........... 50
Table 14: GTAW spot welding schedule. Highlighted values indicate variables that were examined in the optimization study. .................................................................................. 61
Table 15: Spot welding DOE used in optimization study for Inconel Alloy-690 specimens .......................................................... 64

Table 16: Gleeble DOE during the optimization study for Inconel Alloy-600. ............... 70

Table 17: Gleeble optimization test runs for Inconel Alloy-690. ................................. 72

Table 18: Sample heat treatment schedules employed in Phase II STF testing, including grain size measurements in the spot weld microstructure after GTA spot welds were generated in sample gauge sections. .......................................................... 87

Table 19: Round robin STF data for FM-52M heat NX4720TK .................................. 91

Table 20: Round robin STF data for FM-52M heat NX5213TK .................................. 93

Table 21: Round robin STF results for FM-52M, heat EXOA51P ............................... 95

Table 22: Complied STF results for round robin testing ........................................... 96

Table 23: Round robin STF data comparing a 98Ar/2O2 shielding gas mixture and a 100Ar shielding gas during the GTA spot weld stage of the STF test. ..................... 102

Table 24: STF data for the “new” STF testing procedure, allowing testing of material in limited concentrations .......................................................... 105
List of Figures

Figure 1: Ductility-temperature profile of an alloy that undergoes ductility-dip phenomena [8]. .......................................................... 8

Figure 2: Ductility-temperature curve representing the relationship between restraint and crack susceptibility [8].......................................................... 10

Figure 3: Representation of solidification microstructure present in single phase austenitic weld metals, exhibiting grain boundary migration [36].................................. 12

Figure 4: Photomicrograph of a ductility-dip crack along a migrated grain boundary in a thick section weldment of filler metal 52 (FM52). Arrows represent the onset of recrystallization due to high local strain concentrations at these boundaries [41]. ........... 14

Figure 5: Strain distribution map of FM 52 heated to 986ºC and strained at 2.6%. Thin lines represent high angle boundaries. Black is open cracks present in the WM. Color is strain distribution with blue being lowest and red being highest [34]............................. 15

Figure 6: SEM photomicrographs of WM MGBs in a) FM82 showing pinning via MC carbides (arrows) and b) FM52 [9, 23]................................................... 17

Figure 7: STF sample geometry.......................................................... 23

Figure 8: Copper block used for the autogenous GTAW spot weld input in the gauge section of all STF samples. .......................................................... 23

Figure 9: Autogenous GTAW spot welding microstructure. Current downslope schedule induces columnar growth in a 360º radial direction causing multiple grain boundary

xiii
orientation relative to the applied loading direction. DDC may typically be found 45°
with respect to the loading direction [18]. ............................................................ 24

Figure 10: Gleeble 3800® at The Ohio State University, utilized for STF testing.......... 27

Figure 11: Thermo-mechanical cycle generated by the Gleeble to conduct the STF testing
procedure. The blue line represented the temperature history, while the orange line
represents the mechanical history. ............................................................................. 27

Figure 12: Historical output for each spot weld test run, S1-S4.................................. 39

Figure 13: STF sequence for “newly” developed STF testing procedure. ..................... 46

Figure 14: DIC painted speckle pattern on STF sample gauge section. ....................... 47

Figure 15: Schematic of WSI/AZZ’s STF sample fabrication. ..................................... 50

Figure 16: Special Metals Co. STF sample fabrication for the round robin STF study. .. 52

Figure 17: EPRI sample fabrication for round robin STF study................................. 54

Figure 18: STF output data for the STF spot weld optimization study. ....................... 65

Figure 19: Cracking vs. Strain data for STF testing of Inconel Alloy-600 re-optimization
study ......................................................................................................................... 71

Figure 20: Stroke-Strain relationship used to predict strain values in Inconel Alloy-600
STF specimens............................................................................................................ 71

Figure 21: Stroke rate vs. Strain threshold data showing an increasing trend between
stroke rate and minimum strain required to induce DDC........................................... 72

Figure 22: Stroke rate vs. Strain threshold data for Alloy-690 STF specimens showing
similar trend to Alloy-600 specimens................................................................. 73
Figure 23: STF cracking data at 950°C for Phase II testing, comparing Alloy 600, 625, and 200 DDC susceptibilities. ................................................................. 75

Figure 24: Ni-200 photomicrographs exhibiting DDC formation along WM MGBs. A.) 100X magnification B.) 200X magnification. ................................................................. 77

Figure 25: SEM Images of a Ni-200 STF sample with increasing magnification to observe fracture morphology features. A flat intergranular morphology is present with no evidence of micro-ductility. .................................................................................. 78

Figure 26: Alloy-600 photomicrographs depicting DDC formation along WM MGBs. A.) 500X magnification B.) 1000X magnification. ................................................................. 80

Figure 27: SEM images of Ni-600 STF specimen showing a smooth intergranular fracture morphology indicative of DDC. ................................................................. 81

Figure 28: Alloy-625 photomicrograph showing Interdendritic phase formation (NbC and Laves Phase) within the austenite matrix at 500X magnification........................................ 83

Figure 29: STF strain vs cracking envelope comparing 100% Argon (blue) shielding gas vs a shielding gas mixture containing 2% oxygen (red). ................................................................. 85

Figure 30: Figure depicting the observed effect of grain size on sample DDC susceptibility. ................................................................................................. 88

Figure 31: STF round robin results by company ....................................................................... 99

Figure 32: Round Robin STF data as compared by FM-52M heat.............................................. 101

Figure 33: STF Round Robin study comparing oxygen presence in shielding gas mixture to a 100% Ar shielding gas. .................................................................................. 103

Figure 34: Graphical depiction of the data provided in Table 24.............................................. 105
Figure 36: STF DIC strain map before STF testing................................. 107

Figure 37: STF DIC strain map after STF testing. Strain distribution may be viewed via the legend on the right as percent strain......................................................... 107

Figure 38: QuickSim program used to generate STF testing procedure..................... 120
Chapter 1: Introduction

The use of non-ferrous alloys in the nuclear power industry has increased over the past several decades due to industry demand for improved material properties, including; elevated temperature strength, corrosion resistance, and increased strength to weight ratio [1]. More specifically, the use of Inconel alloy 600 and matching filler metal, FM82 (AWS A5.14 ERNiCr-3), have been employed in steam generator tubes and hardware in nuclear reactors for the electric power generation industry since the 1950’s [2], initially exhibiting good resistance to generalized corrosive attack and localized corrosion. However, long exposure to primary water environment has led to concerns with stress corrosion cracking (SCC) of moderate Cr-alloys (14-22 wt. %), such as alloy 600, and matching filler metal FM82, leading to the development of higher Cr alloys such as Inconel alloy 690 (28-30 wt. % Cr) [3-5]. Inconel alloy 690, and its series of matching filler metals (FM52 family: AWS A5.14 ERNiCrFe-7(A)) have shown considerable resistance to corrosion in most water-reactor environments [6] leading to the replacement of alloy 600 in nuclear steam generators [7]. However, the use of higher-Cr alloys in the nuclear power industry has led to significant weldability issues, such as ductility dip cracking (DDC), which has evolved into a weldability problem that has challenged manufacturers and users of these consumables [8, 9]. Considerable research has been conducted to avoid all types of cracking in these environments in order to increase the degree of safety in nuclear components carrying high pressure radioactive steam.
Due to problems with ductility dip crack (DDC) formation in the first generation of FM-52 (AWS A5.14 ERNiCrFe-7), a second generation FM (Inconel FM-52M, AWS A5.14 ERNiCrFe-7A) was developed with additions of B, Nb, and Zr to improve the grain boundary character and creep properties associated with DDC formation. Nb additions result in low melting eutectic formations at the end of solidification, which act to pin migrated grain boundaries, resulting in a tortuous grain boundary character that is resistant to DDC formation. This however has resulted in problems with solidification cracking due to the expansion of the solidification temperature range (STR), which is known to be the driving force behind this cracking mechanism [9]. Continuous efforts are being made to improve the cracking resistance of all high-Cr, Ni-base FM’s employed in safe end nozzles of nuclear steam generators [1] and further derivatives of the FM-52 family are being engineered to be resistant to DDC as well as other forms of cracking that may be problematic in high temperature service environments.

DDC is a solid-state, intergranular, cracking mechanism associated with the weld metal and heat affected zone (HAZ) of austenitic (FCC) materials. Several engineering materials including nickel-based alloys, austenitic stainless steels, titanium alloys, and copper alloys [8-10] have experienced this form of cracking, leading to the development of DDC resistant materials. A material susceptible to this form of solid-state cracking generally experiences a sudden, precipitous, drop in ductility at temperatures ranging from the alloys effective solidus temperature (Ts) to half this temperature (0.5Ts) [9], and unlike other weld cracking mechanisms, DDC generally occurs in “clean” materials which have significant grain boundary mobility [1, 11, 12]. The absence of secondary
phases allows for significant grain boundary migration at elevated temperatures, leading to large grains and relatively straight migrated grain boundaries (MGBs), increasing a materials susceptibility to DDC. Under high restraint conditions, often exhibited in thick sectioned, multi-pass welding procedures, strain localization at these grain boundaries and grain boundary triple points leads to a “creep-like phenomena” [13], which eventually leads to DDC propagation. While DDC cracks may be rather minute in size, it can be a relatively costly failure and preventative measures must be taken to forego this type of cracking.

The Strain-To-Fracture (STF) testing method, developed at The Ohio State University (OSU) by Nathan Nissley and John Lippold [1, 14-16] is a Gleeble®-based testing procedure designed to rank a materials susceptibility to DDC formation. The STF testing method allows the user to test a variety of alloys and microstructures at various temperatures and strains [15], to produce temperature-strain envelopes designed to represent a materials susceptibility to this form of cracking. The STF testing procedure is unique from all other DDC testing methods in that it isolates the sample within the materials ductility-dip temperature range (DTR) and allows for precise control over the testing conditions [1]. It is, however, unclear at what level of strain an actual weldment must undergo to produce such forms of cracking. Due to this factor, further investigation must to be conducted in order to relate DDC formation in the STF testing procedure to DDC formation in an actual weld mockup.

There is a lot of speculation surrounding the external factors that influence DDC formation in Ni-base alloys and filler metals, and many mechanisms relating to the causes
of DDC have been proposed. The presence of DDC in an austenitic (FCC) alloy was first reported as early as 1912 by Bengough [17], and since that time many researchers have conducted studies focusing on DDC formation in multiple alloy systems, giving insight into the mechanism governing this cracking phenomenon.

Collins et al. determined that impurity elements, S and P, have a negative influence on DDC formation, resulting in sufficiently lower strains required to produce cracking when these elements are present in increased concentration [18-22]. While “clean” materials tend to be most susceptible to this cracking phenomenon, impurity element concentration may have some influence over grain boundary cohesive strength, which is likely lowered in the presence of S, P, H, or O [18, 23].

Certain elemental additions, such as Nb and Ti, have proven to increase a materials resistance to DDC by influencing the nature of the secondary phase precipitates that form within these austenitic alloys [8, 9, 11]. The formation of Nb-rich, M(C,N) precipitates, at the end of solidification has the most profound effect on DDC, since these precipitates are most effective at pinning MGBs [11]. Generally speaking, materials with refined grain size and more tortuous grain boundary character tend to be more resistant to DDC formation, making “clean” materials most susceptible to DDC formation. As an example, “Commercially-Pure,” Ni-alloy 200 has increased DDC response due to the absence of secondary phases that might pin MGBs, refining grain size, and producing tortuous grain boundary character [12].

While the exact mechanism for DDC formation may be under relative speculation, it is generally accepted that the underlying cause of the ductility-dip is a
grain boundary sliding phenomenon (similar to creep-rupture failure), where void coalescence at temperatures below the recrystallization temperature contribute to DDC formation and eventual failure [8, 9, 13, 24, 25]. In fact, a recent study by Chen [26] measured the effect of grain boundary sliding and MGB misorientation angle on DDC formation, concluding that grain boundary sliding contributes to crack formation and propagation, while misorientation angle is associated with the DDC initiation site.

This work aims to study the DDC response in several Ni-base alloys while utilizing the Strain-to-Fracture testing method which was developed at Ohio State. Severe compositional variations in the alloys studied will give some insight into specific factors that influence DDC susceptibility, and STF parameter development will also be under examination to determine an optimized range for testing. A “round robin” study was also conducted which aims to understand the influence of sample fabrication methods as it relates to DDC response in Ni-base FM 52M. Results in this document provide an understanding to the specific mechanism(s) which govern DDC susceptibility in Ni-base alloys and FM’s and may be used as a guide to reduce this weldability issue in future industrial applications.
Chapter 2: Background

2.1 Ductility Dip Cracking Definition

Until recently, ductility dip cracking (DDC) in welded structures was considered more of a curiosity than a serious weldability issue [9]. This is due to the inconsistent and confusing language and literature concerning DDC formation in multiple alloy systems. DDC has been referred to as hot cracking, hot tearing, hot fissuring, micro-fissuring, reheat cracking, and polygonisation in welding literature [1, 13, 27, 28], causing confusion amongst academics in the metallurgical profession. Haddrill and Baker [29] clearly define DDC as the loss in ductility, over a temperature range below the solidus, sufficient to produce cracking under the influence of thermal strain caused by welding. Essentially referring to DDC as a solid-state cracking phenomenon that is induced based on a certain level of restraint imposed to a specified area in a multi-pass weldment. Hemsworth [27] confirms this definition of DDC formation, referring to DDC as occurring above 0.5Tm (Tm = melting temperature in K), but occurring at boundaries that are free from liquid films. In fact, it is Hemsworth and Boniszewski [27] who categorizes various high temperature cracking mechanisms as either Type 1.) Segregation cracking or Type 2.) DDC, separating liquation cracking mechanisms from solid-state cracking mechanisms. DDC may be further divided into occurring in the heat affected zone (HAZ) of a material, the weld metal (WM), or the reheated weld metal based on the conditions exhibited during welding and the amount of restraint imposed on a specified
area. This separation criterion proposed by Hemsworth [27] does not, however, provide the specifics to define a single cracking mechanism [9], requiring further evaluation in the efforts to define DDC formation.

2.2 Ductility Dip Cracking in Ni-base Alloys

Figure 1 provides a useful representation of the ductility-dip curve associated with DDC susceptible, Ni-base alloys and filler metals [8]. A precipitous drop in ductility over a narrow temperature range below the effective solidus temperature (Ts) (represented by the shaded area in Figure 1), may lead to a solid-state, intergranular, form of cracking, known as DDC. Specifically, Ni-base alloys and filler metals commonly exhibit this “signature” ductility-dip between 750-1150°C, with a minimum ductility typically occurring around 950°C [15, 18, 30]. In the presence of sufficient strain levels (via extrinsic loading or intrinsic weld restraint experienced in thick sectioned weldments), a materials available ductility may be “exhausted,” leading to void formation and eventual DDC via a grain boundary sliding phenomenon [8, 9] in the reheated WM or HAZ of a weldment.

It should be noted, that each individual alloy susceptible to this form of cracking has its own “signature” ductility-dip curve and threshold strain value required to initiate cracking, making some alloys more susceptible to DDC than others. It is generally known that a wide DTR and low $\varepsilon_{\text{min}}$ to initiate fracture represent the most DDC susceptible materials. In fact, Zhang et al. [31] determined that for a material to be considered resistant to DDC formation, the DTR should be less than 100°C wide and/or the threshold strain should be greater than 15%. Compositional and microstructural influences on a
materials elevated temperature ductility will be discussed in further detail in section 2.5, Proposed DDC Mechanisms.

Figure 1: Ductility-temperature profile of an alloy that undergoes ductility-dip phenomena [8].

Figure 2 [8], represents the relationship between residual strain concentration due to a materials intrinsic restraint level or contraction stresses induced on cooling and DDC formation. As depicted by the figure, if the strain level (restraint) exceeds a certain threshold level, \( \varepsilon_{\text{min}} \), within the DTR crack formation is likely to occur. This condition is represented by the “high restraint” line in Figure 2 and would ultimately crack due to a grain boundary sliding phenomenon along the materials MGBs. However, if the restraint level could be kept under control and held below the \( \varepsilon_{\text{min}} \), cracking would be arrested and the material may continue to be employed as usual. DDC may therefore be characterized by a strain threshold to induce crack formation, \( \varepsilon_{\text{min}} \), and a ductility dip temperature range.
(DTR), in which crack formation is possible [1, 8-10, 15, 30, 32]. It should be noted that the DTR exists over a temperature range where no liquid film formation is likely to occur, making DDC a solid-state cracking mechanism. There have however, been few reports linking the presence of DDC as propagating from a liquation crack and in some cases, the brittle temperature range (BTR) and the DTR may overlap [33].

The onset of the ductility-dip in DDC susceptible alloys contrasts several engineering alloys that are generally resistant to DDC formation. Elevated temperature ductility of these alloys is generally quite high at increased temperatures, close to the effective solidus (Ts), followed by a steady decline in ductility as the temperature is reduced to room temperature [8]. However, alloys that are susceptible to DDC formation, generally experience a ductility-dip phenomenon at temperatures just below the recrystallization temperature [34]. Within this temperature range, voids have time to form and coalesce via grain boundary shearing and DDC formation occurs [9].
Exposure to a temperature within the recrystallization temperature range leads to the formation of new grains and therefore grain boundaries along parent MGBs, making void formation and coalescence very difficult [8, 9, 30]. At these temperatures the ductility is recovered and DDC formation is not likely to occur. The upper bound of the DTR curve is therefore represented by temperatures at or around the recrystallization temperature (1050-1200°C), where ductility recovery is influenced by the onset of new grain formation [18, 23, 34].

The lower bound of the DTR curve may be linked to the creep properties of the subjected material due to the “creep-like” nature of DDC formation [13]. It is generally known, that creep rupture is limited at lower temperatures and the dislocation motion associated with grain boundary sliding may not occur below certain temperatures. Due to
limited creep properties at lower temperatures, ductility recovery occurs close to 0.5Ts, representing the lower temperature limit in which DDC is likely to occur. Increased creep strength at low temperatures is thought to make DDC formation impossible below 0.5Ts.

2.3 Ductility Dip Cracking: Microstructural Characteristics

As previously stated, DDC is a solid-state, intergranular, form of cracking that is most often observed in single phase, austenitic (FCC) materials exhibiting long and non-tortuous MGBs throughout the γ matrix [28, 35]. The formation of MGBs within a single phase, FCC material, may be represented by the schematic shown in Figure 3 [36]. This figure shows the solidification microstructure characteristics associated with single phase, Ni-base, WM’s upon cooling from the solidus temperature (Ts). As represented by the figure, MGB formation originates from the parent solidification grain boundary (SGB). As cooling persists, the crystallographic portion of the SGB acts to straighten itself out, migrating away from the compositional portion of the SGB. If no secondary phase formation is present to “pin” the migration of the MGB, long and non-tortuous, high angle grain boundaries will be present in the weld microstructure. Migration of MGBs is driven by the high energy associated with the increased misorientation angle of the crystallographic portion in SGB’s [37]. MGBs are generally thought to be very susceptible to DDC formation, especially if the nature of the boundary is in a non-tortuous condition [8, 9, 30, 38]. High purity materials tend to form with non-tortuous MGBs due to the absence of secondary phases and solute elements that may act to “pin” or “drag” this boundary via a grain boundary locking mechanism or solute drag effect [8,
“Free” migration of MGBs tends to result in weld metal microstructures that contain relatively large grains and very straight boundaries, making high purity materials most susceptible to DDC formation [9, 12, 35].

The DDC susceptibility of large grained, single phase, structures tends to be increased due to the reduction in grain boundary area per unit volume exhibited by a particular large grained materials [19, 33, 34, 39, 40]. Limiting the GB area per unit volume decreases the paths for deformation mechanisms that may be necessary in the presence of loading conditions, often exhibited when welding highly restrained materials. This leads to localized strain concentrations at MGBs and grain boundary triple points, where DDC formation commonly initiates [9, 18]. If sufficient strain is concentrated
along a particular MGB, the available ductility may be “exhausted” [8, 9], leading to grain boundary shearing mechanisms associated with void formation and DDC propagation. Strain localization along MGBs can in fact be observed in Figure 4 [41] and 5 [34], proving that DDC formation may only be induced when sufficient strain levels exceed the ductility minimum governed by the ductility trough in DTR.

Figure 4 is a photomicrograph of a thick sectioned weldment of high-Cr, Ni-base, filler metal 52 (FM52), exhibiting severe DDC formation and recrystallization along MGBs [9]. Arrows indicate areas along MGBs that are exhibiting new grain formation via a recrystallization mechanism, resulting from high strain concentrations along these boundaries at elevated temperatures [9]. Dynamic recrystallization often occurs in the presence of localized strain concentrations at elevated temperatures, proving that intrinsic strain, resulting from restraint conditions, tends to be localized along MGBs. As previously stated, if sufficient strain levels are present along MGBs, ductility exhaustion may occur leading to de-cohesion of these boundaries (DDC formation).
Figure 4: Photomicrograph of a ductility-dip crack along a migrated grain boundary in a thick section weldment of filler metal 52 (FM52). Arrows represent the onset of recrystallization due to high local strain concentrations at these boundaries [41].

The effect of strain localization along MGBs was also investigated by Collins, et al [23, 34] using Electron Back Scatter Diffraction (EBSD) to produce a strain distribution map along a material subjected to the Strain-To-Fracture (STF) testing method. Figure 5 depicts the use of the strain distribution map on a STF sample of FM52 subjected to 2.6% strain at approximately 950°C. It is clear from this figure, that the highest levels of strain concentration are observed along high angle, MGBs, and grain boundary triple points. DDC is therefore only likely to occur along MGBs due to the presence of localized strain concentrations which may exceed the available ductility present at the specified boundary, leading to crack formation and propagation.
Figure 5: Strain distribution map of FM 52 heated to 986°C and strained at 2.6%. Thin lines represent high angle boundaries. Black is open cracks present in the WM. Color is strain distribution with blue being lowest and red being highest [34].

“Free” grain boundary mobility, often associated with high purity, Ni-base weld metals, may be reduced by influencing the secondary phase formations via compositional alterations to DDC susceptible, Ni-base filler metals. The presence of secondary carbides limits grain boundary mobility and grain growth leading to a structure with increased grain boundary area per unit volume. Ramirez, et al [9, 24, 34] reported that formation of second phase precipitates are effective at pinning MGBs, preventing grain growth and resulting in tortuous MGB character. It is in fact proven that additions of Nb to DDC susceptible weld metals result in the formation of Nb-rich carbides (NbC) at the end of solidification. These precipitates are effective at pinning MGBs, via GB locking mechanisms, which acts to produce a more tortuous GB character within the solidification microstructure [9]. Secondary phase formation also limits grain growth, contributing to an overall increase in GB area per unit volume.
The nature of secondary carbide phase formation on DDC susceptibility in Ni-base FM’s and WM’s has been the focus of many studies \([3, 18, 24, 42]\) in an effort to determine the underlying mechanism associate with DDC formation. It was determined that alloys with increased concentrations of Ti and Nb, such as in FM82, tend to have increased resistance to DDC formation \([18]\) in comparison with FM’s absent of these elemental additions. The formation of MC carbides act to mechanically lock MGBs leading to boundaries which resist grain boundary sliding and have a more tortuous GB character \([18]\). The influence of carbide formation on MGB character, in various Ni-base FM’s can be observed in Figure 6, a and b \([9, 23]\).

Figure 6, a, is a SEM photomicrograph of Ni-base, FM82 exhibiting GB pinning effects from the MC type carbides indicated by the arrows. It is apparent from this figure that MGBs become pinned in place at the location of Nb-rich MC carbides, leading to a more tortuous structure that may be more resistant to DDC formation \([23]\). Figure 6, b, is a similar SEM photomicrograph of Ni-base, FM 52, exhibiting long, non-tortuous MGBs \([9]\). The difference in MGB character may be attributed to Nb additions. While FM82 contains approximately 2.5 wt. % Nb, FM 52 only contains approximately 1 wt. % Nb \([9]\). The reduced concentration of Nb in FM52 results in limited formation of NbC at the end of solidification, leading to insufficient GB pinning effects.

It should be noted that not all secondary phase precipitates influence DDC formation as significantly as MC carbides. Some carbides, such as \(M_{23}C_6\), form outside the DTR and have shown little influence on GB tortuosity, and therefore DDC \([9, 24]\). Studies conducted by Noecker and Dupont \([25, 43]\) correlate increased DDC resistance
with M$_{23}$C$_6$ carbide formation, however, it is unclear if they are of sufficient size to promote GB pinning. The influence of other elemental additions on DDC susceptibility will be discussed further in section 2.5, Proposed DDC Mechanisms.

Figure 6: SEM photomicrographs of WM MGBs in a) FM82 showing pinning via MC carbides (arrows) and b) FM52 [9, 23].
2.4 Ductility Dip Cracking Formation in Various Ni-base Alloys and Filler Metals

Although DDC is generally not encountered in most weldments, it has been a problem in structural weld overlays (SWOL’s) of power plant piping and safe ends in nuclear steam generators [39] and metal thermo-mechanical processing at elevated temperatures [16]. With the introduction of high-Cr, Ni-base alloys and filler metals in the nuclear power industry, the prevalence of DDC has increased, leading to many studies which focus on the prevention and causes of DDC formation [11, 12, 15, 18, 26, 42, 44-46]. This form of cracking can be a serious weldability issue, especially in thick sectioned weldments, exhibiting high levels of restraint [9], and must be combatted via various testing methods. Table 1 provides a summary of some of the most recent and relevant studies relating to DDC formation in Ni-base alloys and filler metals. A variety of testing techniques have been employed in an effort to provide some insight into the factors which influence elevated temperature ductility and therefore DDC formation. Studies such as these also help set up a ranking criterion of susceptible materials, which may help influence industrial demands for material selection. While these studies focus predominantly on DDC formation in Ni-base alloys and filler metals, it is important to note that DDC formation may occur in many engineering alloys including stainless steels, copper alloys, and titanium alloys [1].
Table 1: Historically Significant DDC Studies in Ni-base Alloys and FM’s.

<table>
<thead>
<tr>
<th>Material</th>
<th>Testing Method</th>
<th>Reference</th>
<th>Year</th>
</tr>
</thead>
<tbody>
<tr>
<td>Commercially pure Ni-alloy 200, INCONEL 600, and INCONEL 625</td>
<td>STF Test</td>
<td>Kreuter [12]</td>
<td>2014</td>
</tr>
<tr>
<td>FM52 and FM52M</td>
<td>STF Test</td>
<td>Nissley, Lippold [1, 11, 45, 46]</td>
<td>2005</td>
</tr>
<tr>
<td>INCONEL alloy 690</td>
<td>Hot Ductility Test</td>
<td>Dave and Cola [47]</td>
<td>2004</td>
</tr>
<tr>
<td>FM52 and FM82</td>
<td>STF Test</td>
<td>Ramirez, Sowards, Lippold [24, 42, 44]</td>
<td>2006</td>
</tr>
</tbody>
</table>

2.5 DDC Mechanisms

Until recently, the mechanisms which govern ductility-dip cracking in Ni-base alloys and FM’s were rather unclear. The introduction of the STF testing method and other weldability testing methods, which focus on the formation of DDC, have allowed significant advancement in the understanding of this specific cracking phenomenon. Throughout the years of research focusing on DDC, many factors have been proposed which aim to understand the exact mechanism governing the formation of the ductility-dip and therefore crack formation in many alloy systems. Table 2 summarizes proposed mechanisms that have been theorized over the past 50 years.

Based on these proposed factors influencing DDC formation, it is clear that there are four main theories which have been used to describe DDC formation in FCC materials. Precipitate morphology, concentration of impurity elements (S, P, O, and N), grain
boundary strain concentrations, and grain boundary sliding have all been described as a factor which influence DDC formation. Young et al. [3] studied the effect of precipitate morphology on DDC formation and theorized that strain concentrations exist adjacent to partially coherent precipitates which act as a crack initiation point. This however, contradicts work conducted by Ramirez et al. [12, 42] which states that precipitates may be added to reduce DDC susceptibility via GB “locking” mechanisms, and that “clean” materials are most susceptible to DDC formation.

The introduction of impurity elements, such as S or P, have also been considered a factor which promotes DDC response in FCC alloys. While research by Collins [18] shows an increased susceptibility to DDC in the presence of these elements, it does not explain the increased DDC susceptibility observed in commercially pure materials. Impurity elements definitely have some influence over DDC formation, however their existence does not describe the underlying mechanisms associated with DDC. The extent to which these elements influence DDC response is still under current investigation.

It is generally accepted that grain boundary sliding is the underlying mechanism governing DDC formation. GB sliding is a “creep-like” phenomenon which occurs along high angle grain boundaries due to ductility “exhaustion” in the presence of increased strain levels [9, 30, 35]. This phenomenon has been observed by Ramirez [44] via an “in-situ” STF testing method, and recently quantified by Chen [26]. This mechanisms is still under current investigation although it is clear that GB sliding is the mechanism which governs DDC formation in FCC materials.
### 2.6 STF Testing Method

The STF testing method was designed by Nathan Nissley in 2002 [15] as a method to quantify DDC susceptibility in multiple alloy systems. The test has been employed by multiple researchers to compare FM’s susceptibility to DDC under high restraint and to improve understanding of the mechanisms associated with this form of deterioration.
cracking. The following will discuss the process procedure of the STF testing method as it was developed by Nissley.

Samples of the desired tested material are machined by water jet cutting ¼” thick plate to the sample geometry shown in Figure 7. Sample gauge sections are machined with a smaller area, as compared to the rest of the bar, in order to concentrate strain and heating in this area. Once machined, sample gauge sections are polished to remove surface contaminants and then given an autogenous GTA spot weld with the parameters listed in Table 3. A specialized copper molding system has been constructed specifically for this stage of testing and may be seen in Figure 8. Weld parameters are specifically designed to control the solidification rate and grain growth in the spot weld of each sample. The current downslope induces columnar growth in the radial direction as seen in Figure 9, giving a 360° radial array of MGBs within the spot weld microstructure. Grain boundary orientation relative to the applied strain has proven to be an important variable for repeatability in accordance with this experiment, and cracking tends to occur in the spot weld +/- 45° relative to the normal direction of the applied load.
Figure 7: STF sample geometry.

Figure 8: Copper block used for the autogenous GTAW spot weld input in the gauge section of all STF samples.
Figure 9: Autogenous GTAW spot welding microstructure. Current downslope schedule induces columnar growth in a 360° radial direction causing multiple grain boundary orientation relative to the applied loading direction. DDC may typically be found 45° with respect to the loading direction [18].

Table 3: GTA Spot Weld parameter schedule for STF testing method

<table>
<thead>
<tr>
<th></th>
<th>Time (S)</th>
<th>Value</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shielding Gas Pre-flow</td>
<td>10</td>
<td>20</td>
<td>Ft^3/h</td>
</tr>
<tr>
<td>Initial Current</td>
<td>0.1</td>
<td>20</td>
<td>Amps</td>
</tr>
<tr>
<td>Upslope</td>
<td>5</td>
<td>24</td>
<td>Amps/Second</td>
</tr>
<tr>
<td>Weld Current</td>
<td>10</td>
<td>140</td>
<td>Amps</td>
</tr>
<tr>
<td>Downslope</td>
<td>12.7</td>
<td>9.5</td>
<td>Amps/Second</td>
</tr>
<tr>
<td>Final Current</td>
<td>0.1</td>
<td>20</td>
<td>Amps</td>
</tr>
<tr>
<td>Shielding Gas Post-flow</td>
<td>20</td>
<td>20</td>
<td>Ft^3/h</td>
</tr>
<tr>
<td>Voltage</td>
<td></td>
<td>11-12.5</td>
<td>Voltage</td>
</tr>
</tbody>
</table>

Shielding Gas Argon (99.98%)
Following the completion of the spot weld on each sample, gauge sections were polished through 800 grit silicon carbide paper to help make crack detection more observable. Indentation marks ~8mm apart were then placed in the gage section in order to measure the induced strain in each sample. The distance was measured before and after the STF test with the use of an optical microscope and strain was calculated by dividing length change over original length of the indentation marks. This technique allows for localized strain measurements in the sample gauge section and avoids the complicated and time consuming setup of a strain gauge. It should be noted that Digital Image Correlation (DIC) technology was used in a later stage of this testing, and strain measurements via the indentation measurement method have been confirmed.

The strain-to-fracture test is conducted using a Gleeble® 3800 [48] (shown in Figure 10), where thermal cycles as well as mechanical loading conditions may be applied to simulate overlay welding procedures. Samples are fit with a type K thermocouple which connects to the Gleeble in order to verify accurate temperature readings throughout the sample gauge section. Prepared samples are then mounted into the Gleeble’s stainless steel high force jaw system which contains knurled tool steel grips in order to prevent slippage. A jaw spacing of 30 mm was used in these tests to allow for accurate positioning of each sample. Once samples are mounted and positioned appropriately, a rough vacuum system depressurizes the chamber (10⁻² torr) followed with a back purge of 99.998% argon. This process is repeated twice to prevent sample contamination during testing.
Programming of the Gleeble cycle is generated with the use of QuickSim (snapshot of the QuickSim interface macro is located in Appendix A), which allows for input of multiple variables. Programming for this test was generated to heat samples to a peak temperature of 950°C at a heating rate of 100°C/s. 950°C was chosen as the test temperature based on previous data which suggests that this temperature is the low ductility point in Ni-base alloys ductility dip temperature range (DTR). Samples were held at the peak temperature for 10 seconds before the stroke was applied to allow for the temperature to stabilize across the gauge section. After the hold time, the sample was strained by moving the jaws at a constant rate of 0.6 mm/s. Maximum stroke inputs were variable in order to allow for different strains in each sample. Upon completion of the stroke cycle, heating was turned off and samples were free cooled down to room temperature. A zero load condition was applied to the sample on cooling to account for thermal contraction stresses which suppressed cracks that may have formed on cooling. Figure 11 represents the thermo-mechanical cycle that is currently generated for the STF testing procedure in the Gleeble simulator. It should be noted that three mobile conversion units (MCU) are available for the Gleeble at Ohio State for various forms of testing; pocket jaws (high and low force) and a torsion unit may be changed out depending on the testing requirements.
Figure 10: Gleeble 3800® at The Ohio State University, utilized for STF testing.

Figure 11: Thermo-mechanical cycle generated by the Gleeble to conduct the STF testing procedure. The blue line represented the temperature history, while the orange line represents the mechanical history.
Chapter 3: Objectives

The Strain-To-Fracture (STF) test has been used for years to evaluate the Ductility-Dip Cracking (DDC) susceptibility in several alloy systems. Many researchers have employed this testing method to determine specific factors that contribute to a materials DDC response in an effort to avoid weldability challenges in industrial applications. However, application of the STF testing method to actual practice has proved to be a challenge and the validity of specific quantification methods have been in question. The goal of this study is to optimize the testing methods used at The Ohio State University to evaluate DDC response, as well as determine the influence of weld procedure and STF sample fabrication methods on DDC susceptibility. More specifically, the objectives of this study are as follows:

1. Optimize the STF testing procedure to ensure sample repeatability when utilizing Ni-base alloys and FM’s.

2. Evaluate the cracking response of several Ni-base alloys and filler metals to observe how compositional influence and solidification microstructure variations influence DDC formation, including Nickel 200, Inconel 600, Inconel 625, Inconel 690, and FM52M.

3. Using the optimized STF procedure, determine the influence of weld procedure and STF sample geometry on DDC susceptibility in a round robin study using FM52M.
4. Provide insight into the mechanism(s) that govern DDC formation in Ni-base weld metals and determine how composition and solidification microstructure influences DDC formation in these materials.

5. Determine the effect of a 98Ar/2O₂ mixed shielding gas, employed during the GTA spot weld, on the formation of DDC in Inconel 600, 625, and FM52M.
Chapter 4: Experimental Approach

The Gleeble®-based, Strain-To-Fracture (STF) testing procedure was developed at The Ohio State University in 2002 by Nissley and Lippold [14-16] and has become one of the most repeatable, cost effective, and reliable testing methods for quantifying DDC susceptibility in a variety of alloy systems. This test is unique from all other hot cracking tests in that samples are isolated within the ductility dip temperature range (DTR), meaning that crack formation is limited solely to DDC formation. The isolated temperature field within the DTR limits the confusion of other DDC testing methods, such as the Varestraint Test [49] or PVR Test [50], which may be used to produce various forms of hot cracking phenomenon, such as solidification cracking, liquation cracking, and ductility dip cracking. The formation of other types of high temperature cracking while employing these various testing methods may result in stress relief in the testing area, which will add to the complexity of the testing format, especially since these tests require strain measurements as the quantification methodology.

The Gleeble-based STF testing method has been determined to be the best approach for distinguishing slight variations in DDC cracking resistance between a variety of Ni-base weld metals and filler metals [18]. The flexibility of the thermal control system allows for a variety of heating and cooling rates, which may aid in determining temperature dependent mechanisms that govern DDC formation in tested alloys. A variety of isothermal heat treatments may be employed to isolate the sample
within the DTR, which limits crack formation to DDC. Solutionization heat treatments may also be devised to produce a variety of precipitation reactions which may help produce a more tortuous grain boundary structure that is more resistant to DDC formation. These heat treatments are often used in conjunction to provide a variety of microstructural characteristics in the specific alloys tested resulting in a variety of DDC formation as well as strain quantification figures. The mechanical loading system in the Gleeble is also very flexible and may be used to control the amount of strain localization that is present within the gauge section of the samples tested. Since crack formation is directly related to the amount of strain localization a sample experiences at a MGB, various strain amounts may be used to provide a range of cracking susceptibilities in materials tested. Strain rate is also a flexible variable controlled by the Gleeble thermo-mechanical system. This variable, however, appears to have a very significant effect on the resistance to DDC formation in all alloys tested. Increasing strain rate has shown to reduce a samples susceptibility to DDC formation, which is likely due to reduced microstructural creep properties at increased strain rates [51].

This study focuses of the improvement of current testing techniques associated with the Strain-To-Fracture test, in order to provide a robust parameter set that may be used to provide a repeatable testing method for the formation of DDC in multiple alloy systems. This was initiated with the re-optimization of the STF testing parameters when employing Ni-base alloys and filler metals, which was necessary as the test was originally developed with the use of 310 stainless steel. Once the testing parameters were optimized, a variety of tests may be run to give insight into the mechanisms which govern
DDC susceptibility in multiple alloy systems. The repeatability of the STF test allows for a quick and cost effective way to examine DDC response. A variety of Ni-base material systems were examined in this study to give some insight into compositional and microstructural variation on DDC formation. Process parameters used to develop STF samples were also examined in order to determine the effect of weld process parameters as they relate to DDC formation in Ni-base systems. Work in this study aims to relate weld procedure specification (WPS’s) used in the nuclear power industry to DDC performance.
Chapter 5: Experimental Procedure

5.1 Materials

The following section includes all of the materials that were employed in the STF study including tables with compositional information. Ni-base metals will be discussed first followed by Ni-base FM’s.

5.1.1 Base Metal Compositions

Four commonly used Ni-base alloys were examined in this study for DDC propensity via the STF testing procedure. Inconel alloys 600, 625, and 690 as well as commercially pure Ni-200 were all used to study the respective susceptibility to DDC formation based on compositional variations. Compositions (weight %) for these alloys may be seen in Table 4. It should be noted that Inconel alloys 600 and 625 as well as commercially pure Ni-200 compositions are presented as nominal compositions, while Inconel alloy-690 is presented as the exact composition for the specific heat used. This is a result of unidentified heat numbers used for a portion of testing.

Due to Alloy-690’s increased susceptibility to DDC formation, it was used as a baseline material for the “re-optimization” of the STF testing parameters (Phase I). It was determined that this alloy would be useful in producing repeatable results at relatively lower strains, which would be quicker and more cost effective to run. Nb, Ti, and Al additions are kept at a minimum in this alloy which prevents precipitation of carbides that
would otherwise act to pin MGBs. This results in solidification microstructure that has relatively straight MGBs at room temperature which have limited resistance to DDC formation. Reduced secondary phase formation also results in increased grain sizes as the alloy cools to room temperature due to the fact that grain boundary mobility is not restricted.

Inconel alloys 600, 625, and commercially pure Ni-200 were used as baseline materials for Phase II of testing due to the dramatic compositional variations between the three materials. This study was designed to focus on the effect of alloying additions on DDC susceptibility in various materials. Phase II gives some insight into factors that may influence DDC formation in Ni-base materials.
Table 4: Compositional data (wt. %) for Ni-base materials used in the presented STF study.

<table>
<thead>
<tr>
<th>Element</th>
<th>Inconel Alloy-600</th>
<th>Inconel Alloy-625</th>
<th>Inconel Alloy-690 (NX9620H)</th>
<th>Nickel 200</th>
</tr>
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<tbody>
<tr>
<td>Ni</td>
<td>72</td>
<td>58</td>
<td>60.5</td>
<td>99</td>
</tr>
<tr>
<td>Fe</td>
<td>10</td>
<td>5</td>
<td>9.13</td>
<td>0.4</td>
</tr>
<tr>
<td>Cr</td>
<td>17</td>
<td>23</td>
<td>29.5</td>
<td>-</td>
</tr>
<tr>
<td>C</td>
<td>0.02</td>
<td>0.1</td>
<td>0.03</td>
<td>0.15</td>
</tr>
<tr>
<td>Mn</td>
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<td>0.5</td>
<td>0.15</td>
<td>0.35</td>
</tr>
<tr>
<td>Si</td>
<td>0.5</td>
<td>0.5</td>
<td>0.05</td>
<td>0.35</td>
</tr>
<tr>
<td>Cu</td>
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<td>-</td>
<td>0.01</td>
<td>0.25</td>
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<tr>
<td>S</td>
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<td>0.002</td>
<td>0.01</td>
</tr>
<tr>
<td>P</td>
<td>-</td>
<td>0.015</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Nb</td>
<td>0.15</td>
<td>4.15</td>
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<td>Mo</td>
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<tr>
<td>Co</td>
<td>-</td>
<td>1</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

5.1.2 Ni-base Filler Metals

Three heats of FM-52M were examined and tested in this study via the STF test and their nominal compositional properties (wt. %) may be seen in Table 5. All heats of FM-52M were used in a “round robin” study to examine the effect of weld procedure development on DDC formation as well as to examine the significance of heat-to-heat variation as it relates to DDC development. The results of the “round robin” study will be discussed in chapter 6 of this text.
Table 5: Ni-base FM, FM-52M, compositional data (wt. %) for the 3 heats used in the round robin study (NX4720TK, EXOA51P, NX5213TK).

<table>
<thead>
<tr>
<th>Element</th>
<th>FM 52M (NX4720TK)</th>
<th>FM52M (EXOA51P)</th>
<th>FM52M (NX5213TK)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni</td>
<td>60.23</td>
<td>60.37</td>
<td>60.05</td>
</tr>
<tr>
<td>Cr</td>
<td>29.56</td>
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<td>29.99</td>
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<td>Fe</td>
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</tr>
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<td>C</td>
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<td>Nb + Ta</td>
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<tr>
<td>Ti</td>
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<td>Al</td>
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<tr>
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<td>0.02</td>
<td>0.01</td>
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<td>0.004</td>
<td>0.003</td>
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<tr>
<td>Zr</td>
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<td>0.015</td>
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</tr>
<tr>
<td>B</td>
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<td>0.004</td>
<td>0.001</td>
</tr>
<tr>
<td>Other</td>
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<td>&lt;0.5</td>
<td>&lt;0.5</td>
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5.2 STF Re-Optimization

The interest of high-Cr, Ni-base alloys and filler metals in the nuclear power industry fueled the “re-optimization” of STF testing procedure while employing these alloys. STF parameter schedules (in both the spot weld stage and the Gleeble simulation stage) were created with Type 310 stainless steel during the developments of the STF test in 2002 [15]. For this reason, it was necessary to conduct a study focusing on parameter development as it relates to DDC susceptibility for Ni-base alloys and filler metals. Spot
weld parameter optimization was looked at initially followed by parameter optimization of the Gleeble-based simulation. For testing repeatability reasons, it was necessary to find a range of parameters that have little or no influence of the quantification method of the STF test. Important variables examined in each stage of testing will be discussed in the following sections. INCONEL alloy 690 was used as the baseline material for all DOE testing.

5.2.1 GTA Spot Weld Parameter Optimization

The importance of the spot weld on STF results was discussed in the background section, 2.6. A designed current downslope is used to induce columnar growth in the radial direction, creating a 360 degree radial array of migrated grain boundaries (MGBs) that may be susceptible to DDC formation (shown in Figure 9). It has been proven that grain boundary orientation relative to the applied load has some influence on DDC formation, with 45° relative to the applied loading direction being the most susceptible orientation for the formation of DDC. Grain size and grain boundary tortuosity also have proven to influence DDC formation. For this reason, it was necessary to examine the spot welding parameters which have the ability to effect solidification microstructure and therefore DDC susceptibility. Variables that were examined include, peak current, current downslope time, and weld time (time at peak current). It was necessary to determine if slight variations in spot welding parameters significantly affected microstructural characteristics and therefore STF strain thresholds to induce crack formation. If this was the case, special attention must be given to the spot weld parameters so to ensure (i.e. arc
length, cooling rate, heat input) all are exactly the same from one sample to the next. It may also be necessary to find a more robust parameter set if slight variations in spot weld parameters produce inconsistent STF results.

A simple design of experimentation (DOE) for spot welding variables influence on DDC formation was conducted focusing on three main variables (peak current, time at peak current, and current downslope time). Table 6 includes a list of the various parameter sets (S1-S4) used to generate the spot weld in each sample, as well as the resultant STF strain threshold data and spot weld diameters (mm). Spot weld parameters were made in accordance with Table 6 parameter sets (S1-S4) and the remainder of the Gleeble-based testing was conducted with the parameters shown in Table 7 (developed by Nissley). A graphical depiction of all of the spot welding current-time outputs were generated and are shown in Figure 12. It should be noted that testing schedule S1, depicted by the blue line in Figure 12, is the original spot weld parameter schedule developed by Nissley [14-16] when the STF test was first developed. Results of the GTA spot weld parameter optimization will be discussed in the results section of this thesis.

<table>
<thead>
<tr>
<th>Test Schedule</th>
<th>Current (Amps)</th>
<th>Time @ Peak Current (seconds)</th>
<th>Current Downslope Time (Second)</th>
<th>Voltage (Volts)</th>
<th>Spot weld diameter (mm)</th>
<th>Sample Threshold (E(W))</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>140</td>
<td>10</td>
<td>12.7</td>
<td>10.63</td>
<td>10.8</td>
<td>8.9</td>
</tr>
<tr>
<td>S2</td>
<td>140</td>
<td>20</td>
<td>12.7</td>
<td>10.63</td>
<td>10.8</td>
<td>10.9</td>
</tr>
<tr>
<td>S3</td>
<td>140</td>
<td>10</td>
<td>25</td>
<td>5.40</td>
<td>11</td>
<td>9.4</td>
</tr>
<tr>
<td>S4</td>
<td>200</td>
<td>10</td>
<td>12.7</td>
<td>15.35</td>
<td>13.5</td>
<td>12.5</td>
</tr>
</tbody>
</table>
Table 7: Gleeble parameters used during the spot weld optimization study.

<table>
<thead>
<tr>
<th>STF Gleeble Parameters</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Stroke Rate (mm/s)</td>
<td>0.6</td>
</tr>
<tr>
<td>Peak Temp (°C)</td>
<td>950</td>
</tr>
<tr>
<td>Time at peak temp (s)</td>
<td>10</td>
</tr>
</tbody>
</table>

Figure 12: Historical output for each spot weld test run, S1-S4.

5.2.2 STF Optimization

The next stage of testing consisted of parameter verification and optimization for the Gleeble parameters used throughout the STF testing procedure. This stage of testing is required to induce strain localization and temperature homogenization within the sample’s gauge sections. A set of standard conditions for the STF test was developed by Nissley in 2002 [14-16], and may be seen in the following table (Table 8). Preliminary
testing during the development stages of the STF test employed Type 310 stainless steel as the baseline alloy. For this reason, it was necessary to optimize the chosen parameters for Ni-base alloys, which are commonly employed in the STF test. Like the spot weld optimization, it was necessary to find a robust parameter set for the Gleeble-based portion of testing to ensure that slight variability in parameters will produce repeatable results.

Table 8: Gleeble-based parameters for the STF testing procedure, developed by Nissley [15].

<table>
<thead>
<tr>
<th>Step</th>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Heating Rate (°C/Sec)</td>
<td>100</td>
</tr>
<tr>
<td>2</td>
<td>Peak Temperature (°C)</td>
<td>950</td>
</tr>
<tr>
<td>3</td>
<td>Time at Peak Temperature (s)</td>
<td>10</td>
</tr>
<tr>
<td>3</td>
<td>Stroke (mm)</td>
<td>Controls strain - Any Value</td>
</tr>
<tr>
<td>4</td>
<td>Stroke Rate (cm/s)</td>
<td>0.06</td>
</tr>
<tr>
<td>5</td>
<td>Cooling Rate (°C/s)</td>
<td>~1 (Free Cool)</td>
</tr>
</tbody>
</table>

Variables that were considered for further testing include: heating rate, peak temperature, time at peak temperature, the effect of multiple heating cycles, and stroke rate. It is known already that peak temperature may influence the position within the DTR that the sample is tested. This means that as long as the test temperature is kept consistent, results may be considered repeatable. Heating rate and the use of multiple heating cycles were considered non-significant variables, and were not examined in this optimization process. In fact, when Nissley developed the STF testing procedure, only
stroke rate and time at peak temperature seemed to have influence on the quantification method. For this reason, only these two variables were examined further in the optimization process. INCONEL alloy 600 and 690 were used as baseline alloys for Gleeble DOE testing. These two alloys were chosen due to their historical significance in the nuclear power industry as well as to ensure repeatability when employing parameter sets.

Parameter sets for this experimentation may be viewed in Table 9 (INCONEL alloy 600) and Table 10 (INCONEL alloy 690). Tests A-E were designed to examine the effect of stroke rate and time at peak temperature on STF strain threshold. Values highlighted in red indicate the changed variable for the particular parameter set. It should be noted that only 1 variation to “time at peak temperature” was conducted to confirm that the sample gauge section was of uniform heat as the stroke was imposed on the sample. If this is not the case, a longer time must be used to confirm temperature homogenization along the sample gauge section. It should also be noted that test run C (for both alloys 600 and 690) is the original parameter set that was developed by Nissley during the creation of the STF testing procedure. Results to the Gleeble optimization testing and the significance of tested variables on strain threshold results will be discussed further in the results section of this thesis.
Table 9: Gleeble parameter optimization schedules for INCONEL Alloy-600.

<table>
<thead>
<tr>
<th>Run</th>
<th>Stroke Rate (cm/s)</th>
<th>Time at Peak Temperature (s)</th>
<th>Peak Temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>G-1</td>
<td>0.6</td>
<td>10</td>
<td>950</td>
</tr>
<tr>
<td>G-2</td>
<td>0.2</td>
<td>10</td>
<td>950</td>
</tr>
<tr>
<td>G-3</td>
<td>0.06</td>
<td>10</td>
<td>950</td>
</tr>
<tr>
<td>G-4</td>
<td>0.06</td>
<td>1</td>
<td>950</td>
</tr>
<tr>
<td>G-5</td>
<td>0.1</td>
<td>10</td>
<td>950</td>
</tr>
</tbody>
</table>

Table 10: Gleeble parameter optimization schedules for INCONEL Alloy-690.

<table>
<thead>
<tr>
<th>Run</th>
<th>Stroke Rate (cm/s)</th>
<th>Time at Peak temperature (s)</th>
<th>Peak Temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GS-1</td>
<td>0.6</td>
<td>10</td>
<td>950</td>
</tr>
<tr>
<td>GS-2</td>
<td>0.2</td>
<td>10</td>
<td>950</td>
</tr>
<tr>
<td>GS-3</td>
<td>0.06</td>
<td>10</td>
<td>950</td>
</tr>
<tr>
<td>GS-4</td>
<td>0.06</td>
<td>1</td>
<td>950</td>
</tr>
<tr>
<td>GS-5</td>
<td>0.1</td>
<td>10</td>
<td>950</td>
</tr>
</tbody>
</table>

5.3 Sample Preparation

A variety of techniques were used in this study to focus on specific aspects that relate to DDC susceptibility in Ni-base alloys and FM’s. The following material describes specific factors that were influenced in order to alter the STF response. This section also covers the sample preparation for the round robin STF study.
5.3.1 Heat Treatments

In order to give further insights into some of the mechanisms which govern DDC formation in Ni-base alloys, heat treatments were employed on various STF samples. The Lindberg Horizontal Furnace at Ohio State, was used as a means to conduct heat treatments on STF samples in order to examine the effect of increased grain size and varying precipitate morphology on ductility-dip crack formation. Two heat treatments were employed on 4 STF samples constructed from ¼” thick plate of Inconel Alloy-600 and compared to the as welded Inconel Alloy-600 samples testing in the initial stages of phase II. Heat treatment schedules for phase II of testing may be viewed in Table 11, along with average grain size measurements. Grain size was measured by averaging the width per grain of consecutive grains in a radial pattern. Heat treated samples were then run through the STF testing procedure to examine the effect of grain size on the STF quantification thresholds ($\varepsilon_{\text{min}}$).

It should be noted that increasing a materials grain size will result in increased strain concentration per grain boundary, (due to a decrease in grain boundary area per unit volume), and therefore a reduced amount of grain boundaries to distribute localized strain induced in each sample. This may result in STF threshold values that are much lower than originally seen in the as welded samples. Special attention was given to the degree of tortuosity of MGBs in these heat treated samples. The results section of this thesis will make an effort to relate grain boundary tortuosity as well as grain size measurements to DDC susceptibility.
Table 11: Heat treatment schedules used on STF samples in Phase II.

<table>
<thead>
<tr>
<th>Heat Treatment</th>
<th>Grain Size (Microns/grain)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As Welded</td>
<td>176</td>
</tr>
<tr>
<td>1200°C for 10 hrs.</td>
<td>225</td>
</tr>
<tr>
<td>1200°C for 20 hrs.</td>
<td>247</td>
</tr>
</tbody>
</table>

5.3.2 Oxygen Additions

The effect of impurity elements on crack formation has been studied in many alloy systems. Specific to this study, Collins sought out the effect of hydrogen and sulfur on DDC formation in two nickel-based filler metals [18]. It was found that both impurities, H and S, increased the susceptibility of DDC and lowered the effective $\varepsilon_{\text{min}}$. Hydrogen, which was added in through the shielding gas, seemed to be most significant in lowering the strain threshold.

Continued efforts to study the significance of impurity element concentration on DDC formation was examined in this study with the use of a mixed Ar-2%O₂ shielding gas. Autogenous GTAW spot welds were made on nickel 600 and 625, utilizing this shielding gas mixture to determine the effect oxygen has on the STF strain threshold. Once spot welds were completed on specified STF samples, the samples were run through the STF testing procedure as usual and results were compared to samples prepared with 100% Ar shielding gas. The effects of oxygen concentration on DDC
formation will be discussed in depth in the results section of this thesis. Previous studies have confirmed that increased oxygen concentrations result in grain boundary embrittlement and therefor reduced increased temperature ductility [52].

5.3.3 Limited Material Sample Preparation

A new STF testing procedure was developed for testing materials in limited supply. This was initially an attempt to conduct STF testing on wire for the “development of a high-Cr, Ni-base alloy for nuclear power applications” project. Conical holes were drilled into the gauge section of existing STF samples in order to make a space for new material to be welded in. Small weight buttons were then made via an autogenous GTAW torch in order to fit into the conical hole within the STF sample. A Jetline GTAW side beam was then used to melt the button into the conical hole of the STF sample for further testing. Excess material was then ground flat before continuing with the spot weld and Gleeble testing. A step by step figure of the “new” STF procedure developed at OSU is shown in Figure 13: STF sequence for “newly” developed STF testing procedure. This test proved useful for testing materials that were in limited supply and STF tests were run for Hf-bearing alloys that were in the process of being developed for a FM development project currently being conducted at OSU. Further testing may be required to confirm the repeatability of this method. It is thought that there may be some lack of fusion effect influencing DDC formation, but this has yet to be concluded.
5.3.4 STF Digital Image Correlation

The final stage of this work was to conduct a digital image correlation (DIC) of STF specimen gauge sections via a spray paint pixel methodology. DIC software, NCorr, converts a painted speckle pattern (as shown in Figure 14), on STF sample gauge sections to a strain gradient based on pixel displacement, allowing for accurate and localized strain measurements on samples tested. This was conducted in an effort to confirm strain calculations that had been previously measured via an indentation spacing on the side of each sample. Indentation marks were measured before and after testing and were used to determine the percent strain for the particular sample. DIC analysis was also used as a means to identify strain concentrations which may be associated with crack initiation sites. The use of DIC to observe grain boundary strain concentrations was the original intention of this work, however problems with high resolution imaging made this
difficult. Future work related to DIC analysis on STF specimens will attempt to complete this work.

Figure 14: DIC painted speckle pattern on STF sample gauge section.

Equation 1: Strain equation

\[ \varepsilon = \frac{\Delta l}{l} \times 100 \]

5.4 Round Robin Sample Fabrication and Preparation

Phase III of testing in this experimentation was a STF “round robin” study comprising samples manufactured from three participating companies (WSI, Special Metals, and EPRI). Each respective company fabricates STF samples in a way that represents their weld procedures specification in industrial applications. The design of this round robin study was to determine how weld procedure influences DDC formation in three heats of Ni-base FM-52M (EXOA51P, NX4720TK, and NX5213TK). This phase
of testing also helps gives some further insight into the formation of DDC in Ni-base FM’s and may help influence future weld procedure developments in nuclear power applications. Filler metal compositions for each heat of FM-52M used in this study may be observed in section 5.1 in Table 5, followed by weld procedure specifications that were used by each respective company to fabricate their STF samples in Table 12. Further details on STF sample fabrication will be discussed in the following sections.

Table 12: Weld procedure specification for the fabrication of STF samples in the round robin study.

<table>
<thead>
<tr>
<th>Company</th>
<th>Samples</th>
<th>Heat</th>
<th>BM</th>
<th>FM</th>
<th>Weld Process</th>
<th>Wire diam.</th>
<th>I (amps)</th>
<th>V (volts)</th>
<th>Polarity</th>
<th>Shielding Gas</th>
<th>Interpass Temp</th>
<th>TS (in/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>WSI/AZZ</td>
<td>10</td>
<td>NX5213TK</td>
<td>-</td>
<td>52M</td>
<td>GTAW</td>
<td>0.035</td>
<td>200</td>
<td>9.8</td>
<td>DCEN</td>
<td>100% Ar</td>
<td>350</td>
<td>3.3</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>EX0A51P</td>
<td></td>
<td></td>
<td>GMAW</td>
<td>0.023</td>
<td>200</td>
<td>9.8</td>
<td>DCEP</td>
<td></td>
<td>350</td>
<td>3.3</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>NX4720TK</td>
<td></td>
<td></td>
<td></td>
<td>0.035</td>
<td>200</td>
<td>9.8</td>
<td>DCEN</td>
<td>100% Ar</td>
<td>350</td>
<td>3.3</td>
</tr>
<tr>
<td>Special Metals</td>
<td>11</td>
<td>NX5213TK</td>
<td>690</td>
<td>52M</td>
<td>GTAW</td>
<td>0.045</td>
<td>220</td>
<td>30</td>
<td>DCEP</td>
<td>100% Ar</td>
<td>350</td>
<td>7</td>
</tr>
<tr>
<td></td>
<td>11</td>
<td>EX0A51P</td>
<td></td>
<td></td>
<td>GMAW</td>
<td>0.045</td>
<td>200-240</td>
<td>29-31</td>
<td>DCEP</td>
<td></td>
<td>350</td>
<td>7</td>
</tr>
<tr>
<td></td>
<td>11</td>
<td>NX4720TK</td>
<td></td>
<td></td>
<td></td>
<td>0.045</td>
<td>200-240</td>
<td>29-31</td>
<td>DCEN</td>
<td>100% Ar</td>
<td>350</td>
<td>7</td>
</tr>
<tr>
<td>EPRI</td>
<td>11</td>
<td>NX5213TK</td>
<td>600</td>
<td></td>
<td>GTAW</td>
<td>0.035</td>
<td>155</td>
<td>10</td>
<td>DCEN</td>
<td></td>
<td>N/A</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>11</td>
<td>EX0A51P</td>
<td></td>
<td></td>
<td></td>
<td>0.045</td>
<td>180/150</td>
<td>9.8</td>
<td>DCEN</td>
<td></td>
<td>N/A</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>11</td>
<td>NX4720TK</td>
<td></td>
<td></td>
<td></td>
<td>0.035</td>
<td>160/150</td>
<td>10.2</td>
<td>DCEN</td>
<td></td>
<td>N/A</td>
<td>4</td>
</tr>
</tbody>
</table>

5.4.1 WSI/AZZ Sample Fabrication

As represented by Table 12, 10 samples of each respective filler metal of FM-52M were supplied by AZZ/WSI LLC for round robin STF testing. The point of this testing was to measure the propensity for DDC in each of these heats of FM-52M and to relate that to
sample fabrication mockups generated by AZZ/WSI. The results of the round robin study will give further insight into fabrication techniques that may be used to avoid micro-fissuring in Structural Weld Overlays (SWOL’s) that are generated by this company [53].

Sample fabrication is conducted by generating a GTAW overlay buildup of the desired filler metals to be tested, in this case FM-52M, on an ER308L stainless steel buffer layer, as shown in Figure 15. Weld procedure specification for each pass of the SWOL mockup may be seen in Table 13, indicating that 5 passes are made to complete the mockup. STF samples are then plasma cut from the overlay to the desired geometry, yielding an STF specimen that is composed entirely of deposited weld metal [53]. This is in contrary to the typical STF specimen where samples are fabricated either entirely from the desired base material via a ¼” thick plate or a via a groove joint configuration with a dissimilar metal weld. 10 samples for each heat of FM-52M were fabricated this way and then STF tested with the parameters generated during the optimization phase of this research.
Figure 15: Schematic of WSI/AZZ’s STF sample fabrication.

Table 13: Parameters associated with the fabrication of WSI’s STF samples.

<table>
<thead>
<tr>
<th>Layer</th>
<th>Amps</th>
<th>Volts</th>
<th>Travel (in/min)</th>
<th>Wire</th>
<th>Inter-pass Temp.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stainless Buffer</td>
<td>180</td>
<td>9.8</td>
<td>3.3</td>
<td>90</td>
<td>212°F</td>
</tr>
<tr>
<td>#1</td>
<td>186</td>
<td>9.7</td>
<td>3.6</td>
<td>67</td>
<td>350°F</td>
</tr>
<tr>
<td>#2</td>
<td>215</td>
<td>9.9</td>
<td>3.1</td>
<td>70</td>
<td>350°F</td>
</tr>
<tr>
<td>#3</td>
<td>200</td>
<td>9.8</td>
<td>3.3</td>
<td>90</td>
<td>350°F</td>
</tr>
<tr>
<td>#4</td>
<td>200</td>
<td>9.8</td>
<td>3.3</td>
<td>90</td>
<td>350°F</td>
</tr>
<tr>
<td>#5*</td>
<td>200</td>
<td>9.8</td>
<td>3.3</td>
<td>90</td>
<td>350°F</td>
</tr>
</tbody>
</table>
5.4.2 Special Metals Sample Fabrication

As indicated by Table 12, 10 samples of each heat of FM-52M were prepared by Special Metals Corporation and sent to OSU for round robin STF testing. Special Metals Co. fabricates their STF samples via the traditional groove joint configuration, shown in Figure 16, with a dissimilar metal welding operation. A 75 degree (included angle) groove is machined into a ¾” thick plate of INCONEL Alloy-690 base material. A GMAW process utilizing 0.045” diameter filler wire of INCONEL FM-52M is used to fill the groove, and represents the gauge section of each STF sample. Upon completion of welding, samples are water-jet cut to a thickness of just over ¼” and then ground to the final thickness of 0.25”. It should be noted that samples from Special Metals Co. were received without the sample gauge section, requiring additional manufacturing before testing could be completed. Weld procedures specifications for the fabrication of these samples may be viewed in Table 12. Samples provided by Special Metals are the only samples out of the three participating companies that utilized a GMAW process as a fabrication method. It should be noted that historically, samples provided by Special Metals have performed worse than those provided by competing companies. This may be due to the increased heat input required to deposit weld metal via a GMAW process or the increased travel speed utilized in the weld fabrication technique. Insights into why this might be the case will be discussed in the results section of this thesis as well as results of the round robin STF testing.
Sample Preparation

Figure 16: Special Metals Co. STF sample fabrication for the round robin STF study.
5.4.3 EPRI Sample Fabrication

The Electric Power Research Institute (EPRI) provided 11 samples for each heat of FM-52M to complete STF round robin testing at OSU. Fabrication was completed using a ½” U-groove with a 2” A-36 steel backing plate to prevent distortion during welding. INCONEL Alloy-600 was used as the base material and 3 U-grooves were machined into the 18” long plate, as can be observed in Figure 17. U-grooves are filled with a GTAW process with the desired filler metal for testing (in this case 3 heats of FM-52M) in accordance with the weld procedure specification listed in Table 12. After welding, the A-36 steel backing plate is removed and then flattened in a press in order to reduce any distortion that may have occurred during welding. The top of the plate is then machined flat and then flipped over to machine to the final ¼” thickness required for the STF samples. STF samples are then water-jet cut from the remaining ¼” thick plate to the desired geometry, resulting in specimens that are produced from the top 2/3rds of the joint configuration. A special current pulsing mechanism is used to produce the welds with a 180 peak current and 150 background current. STF results for these specific specimens are recorded and compared in the results section of this thesis.
5.5 Sample Characterization

5.5.1 Optical Microscopy

The use of optical microscopy is necessary to examine tested sample surfaces to for crack formation as well as to observe solidification microstructure that may have led to or helped resist DDC formation. Crack detection is conducted with a Phillips XL-30-FEG as well as a Nikon SMZ1000 from 30-70X magnification. Due to the depth of cracks on the sample surface, cracks may easily be detected by intergranular “dark areas,” where light does not return through the optical lens. Cracks within the spot welds are recorded on each side of the sample gauge sections and then averaged for each particular sample. It should be noted that cracks that are observed outside of the spot
welded area should not be counted as a ductility-dip crack unless otherwise specified by the user.

Photomicrographs were taken of selected sample that have gone through the STF testing procedure. Selected samples were prepared using standard polishing and etching techniques. The use of an electrolytic, 10% chromic acid etch at 5 volts and 1 amp for approximately 5-10 seconds was used to reveal sample microstructures. Special attention was paid to grain size, grain boundary tortuosity, precipitate morphology, and crack formation/location. Various alloy samples were compared to one another to give some insight to the mechanisms governing DDC formation in Ni-base alloys. Photomicrographs of selected samples may be viewed in the results section of this thesis.

5.5.2 Scanning Electron Microscope

A Scanning Electron Microscope (SEM), located at the Center for Electron Microscopy and Analysis (CEMAS) [54], at The Ohio State University in Columbus, Ohio was used for all electron microscopy in this study. Selected samples were prepared and brought to CEMAS for imaging and Energy Dispersive Spectroscopy (EDS) chemical analysis. The Phillips XL-30 ESEM was used for general imaging as well as EDS chemical analysis. Fracture surface morphological features were given special attention with ESEM imagining to ensure crack morphology was that of DDC. A smooth intergranular fracture surface was to be expected for DDC formation and images as well as morphological fracture features may be further examined in the results section of this thesis. Chemical analysis also helped discover compositional gradients adjacent to
fracture features and at precipitates to examine their effect on crack formation. The Quanta SEM, which uses a tungsten electrode source, was generally used for lower resolution optical microscopy due to the limited resolution of this SEM. It should be noted that all SEM analysis was performed in a high vacuum (HV) at 15-20 KeV with a spot size of 3-5.
Chapter 6: Results and Discussion

The following section includes STF results for Phase’s I-V in this study, which are as follows:

2. Phase II: STF testing of various Ni-base alloys to give insight into factors promoting DDC formation in these alloys.
3. Phase III: Round robin STF study determining the influence of fabrication method and heat to heat variation on DDC response.
4. Phase IV: Develop a new STF testing procedure for materials that are in limited quantities.
5. Phase V: Conduct a digital image correlation (DIC) of an STF specimen to produce a macroscopic strain map on sample gauge sections.

A discussion will be presented in conjunction with the results to give insights into why specific materials responded certain ways and potential mechanisms that resulted in the formation of DDC. Photomicrographs are presented as well as fracture morphology studies in order to characterize DDC in selected materials. Further metallographic studies
may be required to make more insights into the mechanisms governing DDC formation in Ni-base alloys and FM’s, and will be presented in the future work section of this thesis.

6.1 Phase I: STF Parameter “Re-Optimization”

In the past decade or so, problems with ductility dip cracking has been reported in a number of Ni-base alloys used in the nuclear power industry [2, 5]. The push for DDC resistant alloys in these industries has led to the “re-optimization” of the Strain-To-Fracture testing procedure that was originally developed at Ohio State with the use of Austenitic Stainless Steels (304 and 310) [14-16]. It was imperative to ensure that the STF testing procedure was optimized in accordance with Ni-base alloys to ensure the accuracy of the results that were being produced. For this reason, Phase I of testing in this research focuses initially on, the optimization of the GTA spot welding procedure associated with the STF test, followed by the optimization of some important variables used in the Gleeble-based portion of testing. It was necessary to find a range of parameters that would be robust to the STF test and insensitive to slight variable changes. The initial GTA spot weld is conducted on the gauge section of every STF specimen before testing continues, making the parameters used to generate this autogenous weld very important. It was imperative to see if slight variable changes in the spot welding parameters would result in microstructural changes that would yield different strain thresholds to produce fracture. If this is the case, it would be necessary to ensure that the same exact variables were used for every GTA spot weld produced (i.e. voltage, current, arc length, etc.). The same applies to the Gleeble-based portion of testing. Variables must
be consistent in producing results even if there are slight changes in stroke, stroke rate, peak temperature, etc. A design of experimentation (DOE) was conducted on both portions of the STF test in order to ensure a robust parameter set was being used in both phases of testing for Ni-base alloys.

The following sections discuss the results of the GTA spot weld optimization as well as the Gleeble-based portion of testing, including variables that were evaluated as well as how individual variables influenced STF outputs. It should be noted that Inconel Alloy-690 was used as the baseline alloy for the spot weld optimization testing and Inconel Alloy-600 as well as Inconel Alloy-690 were used for the Gleeble-based portion of testing. Alloy compositions for both of these Ni-base alloys were provided in section 5.1 in Table 4. These alloys were used as the baseline alloys due to their predominant use in the nuclear power industry as well as their inherent problems with DDC in highly restrained weldments at intermediate temperatures.

6.1.1 GTA Spot Weld Optimization

The importance of the autogenous GTA spot weld on the gauge section of each individual STF specimen cannot be understated. Spot welding parameters were designed to induce columnar growth in a 360 degree radial pattern in order to produce a multitude of migrated grain boundaries (MGBs) with various grain boundary orientations relative to the applied loading direction, which would later be experienced in the Gleeble. Many researchers have studied the effect of grain boundary orientation relative to the applied loading direction [13, 51] and found that creep rupture (DDC is a creep-like phenomenon [13]) only occurs in a transverse (+/− 45°) orientation relative to the applied loading
direction. This means that DDC is never found in a parallel orientation to the applied loading direction in the autogenous spot weld on STF specimens, due to the mechanism of creep rupture in austenitic (FCC) alloys [51].

DDC formation in Ni-base alloys has also proven to be highly dependent on grain size and MGB tortuosity [8, 9, 18, 35, 42]. The tortuosity of a MGB is a measure of its relative “curviness” with more tortuous grain boundaries being less susceptible to crack propagation. Specimens with increased grain size experience a reduced grain boundary area per unit volume, which would act to increase the amount of strain localized on a specific grain boundary under loading or restraint conditions. Due to the fact that DDC occurs due to ductility “exhaustion” at a grain boundary [9], specimens with increased grain size would experience an increased susceptibility to DDC formation. A reduced grain boundary area per unit volume results in a limited distribution of localized strain concentration along these boundaries making grain size a critical feature in predicting DDC susceptibility of a particular alloy.

The fact that DDC susceptibility is highly dependent on microstructural features of a particular STF specimen means that the autogenous GTA spot welding procedure, that precedes STF testing in the Gleeble, must produce repeatable microstructural features from specimen to specimen. Current autogenous GTA spot welding parameters that are used to generate sample spot welds may be found in Table 14. Highlighted features in this table indicate variables that were examined further in the optimization process. The time at peak current, the peak current, and the current downslope time have the most significant impact of the weld metal microstructure size and distribution of
MGBs due to their influence on the relative heat input from sample to sample. For this reason, a DOE was generated which would examine the effect of these three parameters on weld metal microstructure and their influence on the strain threshold required to induce intergranular fracture within a sample spot weld. It should be noted that all other variables were kept consistent during the optimization process in order to examine the individual effects of each variable tested. The interaction of variables was ruled out in this DOE due to results that were found by Nissley in his developmental work of the spot welding parameters. It should also be noted that a copper heat sink was used to hold STF specimens during the autogenous GTA spot weld, seen in Figure 8, which was kept constant from testing conducted by Nissley in 2002.

Table 14: GTAW spot welding schedule. Highlighted values indicate variables that were examined in the optimization study.

<table>
<thead>
<tr>
<th>GTAW Spot Weld Variable</th>
<th>Time</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shielding Gas Pre-flow</td>
<td>10 Seconds</td>
<td>Argon</td>
</tr>
<tr>
<td>Initial Current</td>
<td>0.1 Seconds</td>
<td>20 Amps</td>
</tr>
<tr>
<td>Current Upslope</td>
<td>5 Seconds</td>
<td>27 Amps/Second</td>
</tr>
<tr>
<td>Peak Welding Current</td>
<td>10 Seconds</td>
<td>140 Amps</td>
</tr>
<tr>
<td>Current Downslope</td>
<td>12.7 Seconds</td>
<td>~9.5 Amps/Second</td>
</tr>
<tr>
<td>Final Current</td>
<td>0.1 Seconds</td>
<td>5 Amps</td>
</tr>
<tr>
<td>Shielding Gas Post Flow</td>
<td>30 Seconds</td>
<td>Argon</td>
</tr>
<tr>
<td>Arc Length</td>
<td>-</td>
<td>2 mm</td>
</tr>
<tr>
<td>Shielding Gas Flow Rate</td>
<td>-</td>
<td>20 CFM</td>
</tr>
</tbody>
</table>
Test runs (S1-S4) for the spot weld optimization study may be seen in Table 14. Test run S-1 was the original GTA spot weld testing schedule which was generated during the development stages of the STF test in 2002 [15]. These parameters may be observed in a time/value domain in section 5.2.1 in Figure 12, where all values are represented from arc on to the shielding gas post flow rate that is implemented after welding is finished. It should be noted that test schedule S-1 was included in the DOE as a control schedule in order to examine the individual effects of the changed parameters in test schedules S2-S4.

The highlighted value in Table 15 represents the changed variable for the particular test schedule. The column second from the right in Table 15 represents how shifting the highlighted variable influences the size of the spot weld on the gauge section in a STF specimen comprised of Inconel Alloy-690. From these values, it appears that peak current has the most significant effect on spot weld size with a spot weld diameter of 12.5 mm’s when the peak current is increased to a value of 200 amps. Increasing the heat input via peak current or time at peak current results in more heat conduction into the STF specimen which is the result of the increased spot welding diameter. This may also influence the cooling rate of the specimen which could potentially give more time for grain boundary migration or grain growth in the weld cooling period. The rate of columnar growth is controlled by the current downslope time which was examined in spot weld test schedule S3.

The STF output (strain threshold, $\varepsilon_{\text{min}}$) was compared for all spot welding test schedules and may be seen in the most right column in Table 15. Based on empirical
results of the spot welding diameter changes from test runs S1-S4, it could be predicted that shifting spot welding parameters (especially the ones that result in increased heat input into the STF specimen) would result in variable STF strain thresholds to induce DDC formation. This however, was not the case as the strain threshold values for testing schedules S1-S3 were all around 8% strain. This means that it took about 8% localized strain to cause intergranular crack formation in the spot weld of each sample tested. The strain threshold for test run S4 was at about 10% which is actually the opposite of what was predicted. It was initially thought that increasing the peak current, resulting in increased heat input and therefore slower cooling rate, would result in a sample with larger grains that is known to be more susceptible to DDC formation. This testing schedule may require further analysis as a limited amount of samples were used for this optimization study. It is still not clear how many samples must be tested via the STF testing procedure to produce statistically validated results. From this testing however, it can be concluded that slight variations in spot welding parameters have no significant effect on the STF output, meaning that the parameter set originally chosen by Nissley is in a robust range that ensures sample repeatability. Cracking vs. strain charts for each spot weld test run may be observed graphically in Figure 18 to get a better sense of the STF performance for each trial run. All test runs (S1-S4) followed the same general pattern in the crack vs strain chart confirming the robustness and repeatability of the parameter sets chosen. This might signify that microstructural changes were not significant enough from one test run to another to produce variation in strain threshold to
cause cracking. Microstructural variations may need to be much more significant in order to influence the STF output.

Table 15: Spot welding DOE used in optimization study for Inconel Alloy-690 specimens.

<table>
<thead>
<tr>
<th>Run</th>
<th>Peak Current (I)</th>
<th>Time at Peak Current (s)</th>
<th>Current Downslope Time (s)</th>
<th>Avg Spot Diameter (mm)</th>
<th>Strain Threshold ($\epsilon_{min}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S-1</td>
<td>140</td>
<td>10</td>
<td>12.7</td>
<td>8.9</td>
<td>8</td>
</tr>
<tr>
<td>S-2</td>
<td>140</td>
<td>20</td>
<td>12.7</td>
<td>10.9</td>
<td>7.9</td>
</tr>
<tr>
<td>S-3</td>
<td>140</td>
<td>10</td>
<td>25</td>
<td>9.4</td>
<td>8.3</td>
</tr>
<tr>
<td>S-4</td>
<td>200</td>
<td>10</td>
<td>12.7</td>
<td>12.5</td>
<td>10</td>
</tr>
</tbody>
</table>
6.1.2 Gleeble Parameter Optimization

The Gleeble-based portion of the STF testing procedure has the most direct effect on the STF testing results. This stage of testing is required for temperature homogenization of the sample gauge section to a temperature within the ductility dip temperature range (DTR) as well as strain localization to exceed the strain threshold of each particular sample in order to induce DDC formation. A preliminary DOE was conducted during the developmental stages of the STF testing procedure with type 310 SS in order to develop a set of initial standard Gleeble-parameters, which may be viewed in section 2.6 of this thesis. Due to industrial demands for the use of Ni-base alloys in the nuclear power generation industry, the Gleeble portion of the STF testing procedure must be “re-optimized” to ensure that slight variations in Gleeble parameters will yield the
same result when utilizing Ni-base alloys and filler metals. It is necessary to create a robust parameter set that will ensure sample repeatability and accuracy when dealing with specialty alloys like those used in the nuclear power industry. Like the spot welding optimization, a new DOE must also be conducted for the Gleeble thermo-mechanical parameters used to run the STF testing procedure. Testing was conducted with two Ni-base alloy systems in this case to ensure sample repeatability. INCONEL Alloy-600 as well as INCONEL Alloy-690 were run through a series of testing procedures to check the influence on individual parameters generated by the Gleeble.

Initially, multiple variables were considered for the optimization of the Gleeble-based portion of the STF testing procedure. Testing temperature, stroke rate, heating rate to test temperature, hold time at the testing temperature, and the use of multiple heating cycles were considered important variables in this stage of testing. It was later determined that a few of these variables were non-essential variables in the optimization of the STF test due to their insensitivity to the testing output.

It is already determined that the peak testing temperature (°C) controls the point in the ductility-dip temperature range that the specific sample is in. As long as this variable is kept constant from one sample to the next, without much fluctuation (+/- 5°C), the position in the DTR would remain the same and therefore the strain threshold would as well. Due to this knowledge, the testing temperature was kept at a constant 950°C for the remainder of the testing procedure. Previous research indicated that 950°C is a typical low point within the DTR envelope of Ni-base alloys and FM’s which would eliminate variation in output data given some temperature fluctuation.
Heating rate to the testing temperature was initially considered a significant variable due to the variations in testing microstructure that may occur with different heating rates. This was however thrown out, due to the fact that diffusion rates in FCC materials are generally slower [30] resulting in similar sample microstructures regardless of heating rate. It was also determined by Nissley that heating rates of 10°C/sec and 100°C/sec yielded no difference in producing threshold values. Accordingly, a value of 100°C/sec was selected for the remainder of the tests, due to the fact that it closely matches what occurs in a GTA weld as well as to reduce the time required to get to the testing temperature.

The use of multiple heating cycles was considered an important and practical factor for the STF testing environment. Multiple heating cycles may resemble multi-pass welding conditions and allow for “subsequent” passes to be made on the same specimen. This variable was later thrown out however, due to the complexity it would add to the testing environment. While this may be an important variable to test in future studies, it was not selected for the “re-optimization” phase of the STF testing procedure. As long as only one heating cycle was generated for each respective sample that was tested, results would yield a repeatable value. The presumption here is that DDC occurs on-heating during the first reheat cycle into the DTR.

According to the literature [31, 55, 56], the value of stroke rate, or the rate at which the jaws induce the strain within the sample, is considered an important variable in influencing grain boundary sliding characteristics. Due to the fact that DDC is a grain boundary sliding phenomenon [9], the stroke rate parameter must be further evaluated in
the optimization study. Four separate stroke rate values (0.06 cm/s, 0.1 cm/s, 0.2 cm/s, and 0.6 cm/s) were evaluated in this re-optimization study to examine the general trend stroke rate has on DDC formation in Ni-base alloys. It was determined that increasing the stroke rate reduced the susceptibility an alloy has to DDC formation. Increased stroke rates, for both Inconel Alloy-600 and Inconel Alloy-690, were found to yield increased strain threshold for DDC formation, which can be seen in Figure 21 and Figure 22, respectively. This result is in agreement with studies conducted by Nissley, where increasing the stroke rate resulted in a decreased severity to DDC formation in 310 SS. A value of 0.6mm/s was eventually settled on based on estimates of actual strain rates imposed via welding as proposed by the literature.

Time at peak temperature was also considered an important variable due to the sole consideration of temperature homogenization within the samples gauge section. It was thought that at a low time at peak temperature, the sample gauge section might not be completely homogenized, resulting in a non-uniform strain distribution or variations in the peak testing temperature. A low value of 1 second was compared to the original value of 10 second to observe this effect. It was determined that this variable has no significant influence on STF results and repeatable tests may be conducted at either of these values.

Table 16 and Table 17, indicate the testing runs used for the Gleeble parameter optimization study for Inconel Alloy-600 (G1-G4) and Inconel Alloy-690 (GS1-GS-4), respectively. As previously discussed, only stroke rate and time at peak temperature were examined in this optimization study, as they were the only variables considered to have an influential impact on the repeatability of the testing results. Like the spot weld testing
DOE, highlighted variables indicate the changed variable for the particular test run and test run G-3 (Alloy-600) and GS-3 (Alloy-690) indicate the original testing schedule that was developed by Nissley to be used as a control test run.

The results indicate that the stroke rate is the only important variable in altering the repeatability of the test runs, with an increased stroke rate resulting in an increased strain threshold required to induce DDC formation in both Ni-alloys tested. The relationship between the stroke rate and the strain threshold value is represented in Figure 21 for Alloy-600 and Figure 22 for Alloy-690, both of which show a similar trend. It was however, determined that there is a robust parameter value between 0.06cm/s and 0.1cm/s (test runs G3/GS3 and G5/GS5) indicating that slight variations in the stroke rate would yield repeatable results. This means that the testing stroke rate variable that was generated by Nissley during the developmental stages of the STF test in 2002 is also valid for Ni-base alloys.

As previously stated, the DOE found no significance in variations of the time at the testing temperature as both test runs, G3/GS3 and G4/GS4, yielded the same strain threshold. Cracking vs. strain information for Inconel Alloy-600 may be viewed in Figure 19 to get a better understanding of the performance of the samples tested. It should be noted that a stroke-strain relationship was developed for Inconel Alloy-600 in order to be able to predict the strain value that would be outputted from the input stroke value. This relationship may be seen in Figure 20, and can be used to produce desired strain levels for Ni-base alloys while employing the STF testing procedure.
Table 16: Gleeble DOE during the optimization study for Inconel Alloy-600.

<table>
<thead>
<tr>
<th>Run</th>
<th>Stroke Rate (cm/s)</th>
<th>Time at Peak Temp (s)</th>
<th>Peak Temp (°C)</th>
<th>Threshold, $\varepsilon_{\text{min}}$ (percent strain)</th>
<th>Slope</th>
</tr>
</thead>
<tbody>
<tr>
<td>G-1</td>
<td>0.6</td>
<td>10</td>
<td>950</td>
<td>10.5</td>
<td>0.68</td>
</tr>
<tr>
<td>G-2</td>
<td>0.2</td>
<td>10</td>
<td>950</td>
<td>9</td>
<td>0.62</td>
</tr>
<tr>
<td>G-3</td>
<td>0.06</td>
<td>10</td>
<td>950</td>
<td>8</td>
<td>5.7</td>
</tr>
<tr>
<td>G-4</td>
<td>0.06</td>
<td>1</td>
<td>950</td>
<td>8</td>
<td>2.2</td>
</tr>
<tr>
<td>G-5</td>
<td>0.1</td>
<td>10</td>
<td>950</td>
<td>8</td>
<td>1.08</td>
</tr>
</tbody>
</table>

Legend:
- Original Parameter Set
- Changed Variable
Figure 19: Cracking vs. Strain data for STF testing of Inconel Alloy-600 re-optimization study.

Figure 20: Stroke-Strain relationship used to predict strain values in Inconel Alloy-600 STF specimens.
Figure 21: Stroke rate vs. Strain threshold data showing an increasing trend between stroke rate and minimum strain required to induce DDC.

Table 17: Gleeble optimization test runs for Inconel Alloy-690.

<table>
<thead>
<tr>
<th>Run</th>
<th>Stroke Rate (mm/s)</th>
<th>Time at Peak Temp (s)</th>
<th>Peak Temp (°C)</th>
<th>Threshold (percent strain)</th>
<th>Slope</th>
</tr>
</thead>
<tbody>
<tr>
<td>GS-1</td>
<td>0.6</td>
<td>10</td>
<td>950</td>
<td>11</td>
<td>7.1</td>
</tr>
<tr>
<td>GS-2</td>
<td>0.2</td>
<td>10</td>
<td>950</td>
<td>10</td>
<td>1</td>
</tr>
<tr>
<td>GS-3</td>
<td>0.06</td>
<td>10</td>
<td>950</td>
<td>8.5</td>
<td>1.3</td>
</tr>
<tr>
<td>GS-4</td>
<td>0.06</td>
<td>1</td>
<td>950</td>
<td>8.7</td>
<td>0.9</td>
</tr>
<tr>
<td>GS-5</td>
<td>0.1</td>
<td>10</td>
<td>950</td>
<td>8.5</td>
<td>1.08</td>
</tr>
</tbody>
</table>
Figure 22: Stroke rate vs. Strain threshold data for Alloy-690 STF specimens showing similar trend to Alloy-600 specimens.

6.2 Phase II: Insight to DDC Mechanism

Phase II of testing was designed to gain some insights into the mechanisms which govern DDC formation in Ni-base alloys. Initially, three commonly used Ni-base alloys, of varying alloying addition and microstructure, were run through the STF testing procedure to examine the effect of compositional fluctuation on DDC formation.
Commercially pure Ni-200, Inconel Alloy-600, and Inconel Alloy-625 were run through the STF testing procedure (in accordance with parameters generated in Phase I) in order to compare the DDC susceptibility of these alloys. Nominal compositions for these alloys (wt. %) were provided in Table 4 in section 5.1 of this thesis. The results of this testing and a subsequent discussion on material performance will follow this introduction.

Metallographic work was also done on each alloy presented to gain an understanding of microstructural features that may be associated with DDC response.

Following the STF work on these Ni-base alloys, a study was conducted in order to understand the significance of oxygen concentration on DDC formation. A 98% Argon, 2% Oxygen, shielding gas mixture was used to generate GTA spot welds on STF samples of Inconel Alloy-600 as well as Inconel Alloy-625. These samples were then run through the remainder of the STF testing procedure and cracking results were compared to similar STF specimens generated with a 100% Argon shielding gas. A SEM metallographic study will be presented and discussed in the following section in order to understand the role oxygen plays in DDC formation.

Final work in Phase II consisted of STF sample heat treatments of Inconel Alloy-600 to study the effect of grain size and precipitate morphology on DDC response. Samples were given various heat treatments at 1200°C for 10 and 20 hours, respectively to study the effect of grain size on DDC formation in these alloys. A full review of the results will be discussed in the following section.

6.2.1 STF Testing of Various Ni-base Alloys
Cracking vs strain data for the STF testing of commercially pure Ni-200, Inconel Alloy-600, and Inconel Alloy-625 are shown in Figure 23. 5-10 STF samples were run at various strains in order to determine a threshold to initiate DDC formation at 950°C in each respective alloy being studied. A ranking criterion of the DDC susceptibility of the alloys testing is as follows (least to most susceptible): Alloy-625, Alloy-600, and Alloy-200. A more in depth review of these results will be presented in the following sections along with metallurgical characterizations of each alloy tested.

![Strain vs. DDC Count](image)

Figure 23: STF cracking data at 950°C for Phase II testing, comparing Alloy 600, 625, and 200 DDC susceptibilities.

6.2.2 Alloy 200

Five Ni-200 STF samples were tested in this investigation and strained at various rates to determine strain threshold values for DDC initiation. This alloy system proved to be the most susceptible to DDC, exhibiting a threshold value of ~1% and a transition to
gross cracking above 2%. Gross cracking can be defined as the strain where cracking becomes so abundant that cracks begin to connect and grow [38]. This is likely due to the significant amount of long and non-tortuous MGBs in the weld microstructure of Ni-200.

Pure materials, such as Ni-200, can be susceptible to weld metal DDC due to their homogenous microstructure that is free of secondary phases. On cooling, the crystallographic portion of the solidification grain boundary will migrate via grain growth and boundary straightening mechanisms, leading to migrated grain boundaries that are long and non-tortuous. The lack of a secondary phase leads to free grain boundary motion within the weld metal; resulting in large grain size and reduced MGB area. Since weld metal DDC forms preferentially along MGBs, Ni-200 has high a susceptibility to DDC. This is in contrast to what was predicted by Young [3], where DDC formation is proposed to be precipitation induced. Due to the fact that Ni-200 has no precipitates within the weld metal microstructure, it can be concluded that Young’s theory of DDC formation may not be applicable to this particular case of DDC formation. That does not mean that strain concentrations do not occur around precipitates leading to void formation and subsequent crack propagation in other alloy systems.

Metallography studies of Ni-200 samples STF tested at ~1% strain are shown in Figure 24 (A and B). A purely austenitic (FCC) microstructure may be seen in both photomicrographs with abundant DDC formation along weld metal MGBs. It is apparent that crack formation initiates at grain boundary triple points in all metallographic sections which were examined, indicating that these areas concentrate the highest amount of
strain. On average, DDC in Ni-200 was around 100 microns in length, and sometimes exceeded 200 microns. This indicated the severity of crack formation in this alloy system, and it may be concluded that highly pure austenitic materials may be very susceptible to DDC formation.

Figure 24: Ni-200 photomicrographs exhibiting DDC formation along WM MGBs. A.) 100X magnification B.) 200X magnification.

The fracture morphology of Ni-200 is shown in Figure 25. Figure 25 A, shows the entire ductility-dip crack with small fractures stemming off of the perpendicular conjoining grain boundaries. From Figure 25 B-D, increasing magnification reveals a smooth intergranular fracture morphology indicative of ductility-dip crack formation. It is evident in this figure that the crack initiated at the grain boundary triple point in the
center of the photo and propagated outward in both directions as strain concentrations increased. This particular crack is rather large and has a crack length of over 600 microns, which reveals the severity of cracking that occurred in this particular specimen.

Figure 25: SEM Images of a Ni-200 STF sample with increasing magnification to observe fracture morphology features. A flat intergranular morphology is present with no evidence of micro-ductility.
6.2.3 Inconel Alloy-600

Seven STF samples of Inconel Alloy-600 were tested in this study to determine the strain threshold required to induce fracture in this system. It was determined that Alloy-600 is resistant to DDC according to DuPont and Lippold [9] by having a strain threshold greater than 6%. Cracking was not observed in this system until the induced strain exceeded 10% and no transition to gross cracking was observed in any of the samples tested. This is likely due to the characterization of the MGBs in this alloy system due to increased alloying additions.

Phase identification software, JMatPro-Ni, suggests the formation of $M_7C_3$ carbides at ~1200°C in Alloy-600. The formation of secondary phases within the austenitic matrix tend to pin grain boundaries via the grain boundary locking mechanism, leading to the formation of tortuous MGBs. Photomicrographs of Ni-600, as seen in Figure 26 A and B, show a relatively fine grained austenitic microstructure with the presence of small carbides along solidification and migrated grain boundaries. Grain boundaries in Alloy 600 tend to be shorter and more tortuous (due to finer grain size), which result in the higher strain threshold required to induce DDC formation exhibited by this material. Cracks also tend to be much shorter with an average crack length of ~40 microns. It is clear from these results that materials with increased alloying additions tend to be less susceptible to DDC formation than commercially pure alloys. The extent of the precipitation in this system is under current review and future work may be conducted on this system to determine the precipitate composition that exists along grain boundaries. It should be noted however, that precipitates in this system form in the solid-state condition.
and not at the end of solidification, which would inhibit any grain boundary locking mechanism that may otherwise occur.

Figure 26: Alloy-600 photomicrographs depicting DDC formation along WM MGBs. A.) 500X magnification B.) 1000X magnification.

Fracture morphology studies were also completed on an STF specimen of Alloy-600 exhibiting ~18% strain. Figure 26 A and B, represent DDC formation along MGBs of a selected specimen of Alloy-600 that has been STF tested. Figure 27 A and B, illustrate the degree of grain boundary cracking at increased strain levels. Smooth intergranular fracture surfaces may be viewed more closely in Figure 27 B, which confirms the presence of DDC in this alloy. Figure 27 A shows how DDC fractures propagate along the grain boundary until the grain boundary changes direction, where the crack propagation tends to
stop. This is due to the tortuosity of the grain at this point, proving that DDC fractures tend to be abundant in materials exhibiting non-tortuous MGBs.

6.2.4 Inconel Alloy-625

Ten samples of Inconel Alloy-625 were STF tested in order to determine a strain threshold required to induce fracture for this system. It was determined that Alloy-625 is very resistant to DDC formation, as no cracking was observed in any of the 10 samples tested (up to 35% strain). This is likely due to the degree of secondary phases that exist in Alloy-625. Crystallographic portions of solidification grain boundaries that would otherwise migrate get “pinned” on secondary phase formations, resulting in a very fine grained structure with tortuous MGBs.

Inconel Alloy-625 is the most alloyed of all materials tested in this study containing 23 wt. % Cr, 4.15 wt. % Nb, and 10 wt. % Mo. Phase identification software
JMatPro-Ni predicts the formation of niobium carbides (NbC) and Laves Phase at the end of solidification which would act to prevent grain boundary migration and grain growth. Figure 28 shows the solidification microstructure of a STF specimen of Alloy-625. As represented by the photomicrograph, inter-dendritic regions of this material exhibit extensive NbC as well as Laves Phase formations. Although MGB’s are difficult to distinguish in this photomicrograph, they tend to be very tortuous in this alloy, which contributes to the increased resistance to DDC formation.

It may be concluded that significant additions of Nb, Mo, and Cr in Alloy 625 lead to the formation of a complex, multi-phase microstructure, which is resistant to DDC at all intermediate temperatures. This confirms other researchers, such as Ramirez [42], who proposed that DDC may be prevented with the additions of elements that form eutectic constituents, which have a mechanical locking effect on the migration of grain boundaries. This also confirms that DDC susceptible alloys must contain long and non-tortuous MGBs in order for cracking to be prevalent within the weld metal microstructure.
Previous research conducted by Ramirez and Lippold proposed that grain boundary tortuosity has the greatest influence on DDC susceptibility [42]. Studies showed that increasing the level of precipitate-forming elements in nickel-based filler metals, such as niobium and titanium, may lead to grain boundaries which are more tortuous, and therefore less susceptible to DDC. Although tortuous grain boundaries may suppress the formation of DDC, it should be noted that the introduction of precipitate forming elements may widen the solidification temperature range making the alloy more susceptible to solidification cracking. Work conducted in this study supports the grain boundary tortuosity hypothesis and crack formation seems to be suppressed with the addition of precipitation forming
elements in Ni-600 and Ni-625, as compared to commercially pure Ni-200. Continued work is underway to look at the effect of recrystallization behavior on DDC susceptibility.

6.2.5 The Effect of Oxygen on DDC susceptibility

As previously mentioned, the effect of impurity element concentration was examined in this study with the use of a Ar2%O2 shielding gas mixture utilized during the GTA spot welding stage of testing. Three STF samples were prepared utilizing the mixed shielding gas for both Alloys 600 and 625, while keeping all other testing variables constant. Strain vs DDC count charts may be seen in Figure 29 A and B, for Alloy-600 and Alloy-625, respectively. The red line in these charts represents the STF specimens that were tested with the shielding gas mixture while the blue line indicates samples tested in the previous portion of Phase II.

The results showed a significant decrease in strain threshold required for DDC development when oxygen concentrations are increased. The strain threshold for Ni-600 dropped from ~11% to below 7%, whereas the threshold for Ni-625 was established at ~20%. Like hydrogen, oxygen may have some factor in reducing grain boundary cohesion resulting in an increased susceptibility to DDC in materials exhibiting critical concentrations. Surface oxide formations tend to decrease the toughness and ductility of a material due to the brittle nature of the oxide. The extent to which oxygen effects DDC susceptibility and the amount required to shift threshold levels is still under investigation.
Figure 29: STF strain vs cracking envelope comparing 100% Argon (blue) shielding gas vs a shielding gas mixture containing 2% oxygen (red).

6.2.6 STF Sample Heat Treatments

The final stage of Phase II was to determine the effect of grain size on DDC susceptibility in STF samples of Inconel Alloy-600. As previously mentioned, two sample heat treatments were conducted on four STF samples in order to increase the relative grain size in the gauge section of tested samples. Table 18 shows the heat treatments that were used in this study, the average sample grain sizes in the gauge section (microns/grain) for the particular heat treatment employed, and STF strain
thresholds experienced for each respective test. It should be noted that sample grain size was determined based on averaging the microns/grain in a radial direction in the gauge section of each sample. Upon completion of the sample heat treatments, the STF testing procedure was employed as usual. It should be noted that heat treatment schedules were employed before the spot welding stage of testing, which means that there is some degree of epitaxy from the base material into the spot welding microstructure. The significance that this has on spot weld grain size is under current investigation.

As was expected, increasing the sample time in the furnace at 1200°C resulted in increased grain size in the STF sample gauge section. Increased grain boundary mobility and diffusion rates at increased temperatures result in the increase in grain size with high temperature heat treatments. Also, as previously mentioned, increasing sample grain size results in a decrease in grain boundary area per unit volume resulting in increased strain concentration at a particular grain boundary. This is due to the fact that with larger grained samples (reduced grain boundary area), there is less area for strain to be distributed upon external loading or restraint conditions. As a result, each individual grain boundary must accommodate an increased localized strain level, which may result in ductility “exhaustion” and therefore DDC formation at a lower overall load. Figure 30 represents this phenomenon by showing how larger grained samples have a lower strain threshold required to induce DDC formation (which was determined via the STF testing procedure).

In fact, there appears to be a negative linear correlation between larger grain size and strain threshold required to induce DDC in INCONEL Alloy-600 STF samples. For
this reason, it would be recommended to employ materials with finer grain size in the nuclear power industry as to reduce the likelihood that DDC might form within the materials weld metal under increased loading conditions. Further testing is being conducted to confirm this phenomenon with other Ni-base materials and to observe how precipitation behavior affects grain boundary mobility at increased temperatures.

Table 18: Sample heat treatment schedules employed in Phase II STF testing, including grain size measurements in the spot weld microstructure after GTA spot welds were generated in sample gauge sections.

<table>
<thead>
<tr>
<th>Test</th>
<th>Threshold</th>
<th>Grain Size (microns/grain)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As Welded</td>
<td>8</td>
<td>176</td>
</tr>
<tr>
<td>1200°C for 20 hrs</td>
<td>4</td>
<td>247</td>
</tr>
<tr>
<td>1200°C for 10 hrs</td>
<td>4.58</td>
<td>225</td>
</tr>
</tbody>
</table>
Figure 30: Figure depicting the observed effect of grain size on sample DDC susceptibility.

Phase III: 6.3 STF Round Robin Study

Phase III of testing was a “round robin” STF study that was designed to examine the effect of weld process and procedure development on DDC susceptibility in STF samples comprised of Ni-base FM-52M. Three participating companies (WSI, Special Metals, and EPRI) provided STF samples for the study that were fabricated via their respective manufacturing methods commonly employed in the nuclear power industry. As a baseline material, three heats of FM-52M were used to manufacture samples to the desired STF geometry required by the STF testing procedure in section 2.6. Compositional data for the three heats of FM-52M used in this study was provided in Table 5 in section 5.1. Weld procedure specifications for the fabrication of these samples were provided in Table 12 in section 5.4, followed by detailed information on each...
respective company’s method of manufacturing in sections 5.4.1-5.4.3. This information may be insightful in determining mechanisms for the round robin testing results that are compiled in the following sections. Heat to heat variations will be discussed first, followed by a set of compiled data that examines weld procedure developmental aspects that may have influenced STF results. A ranking system will also be provided based on heat to heat variations as well as company fabrication methods. Future work will be conducted in an effort to characterize metallographic characteristics of tested samples, in order to gain some further insight into the performance of selected specimens.

As described in section 5.4, both EPRI and WSI samples were fabricated with the GTAW process, while Special Metals samples were fabricated with the GMAW process. The results clearly indicate that the DDC susceptibility of Special Metals samples was higher, which may be based on heat input, shielding gas coverage, impurity element concentration, or geometrical features during the fabrication of specimens. Travel speed may also be an indication of dissimilar results, as the speed of welding in Special Metals specimens was nearly twice that of both other fabrication methods produced by WSI and EPRI. A more in depth review of weld procedure specifications as they relate to STF results will be discussed in the following sections.

6.3.1 NX4720TK

Compiled data for STF tested samples of FM-52M, heat NX4720TK, may be seen in Table 19. 8-9 STF samples from each participating company were prepared and run through the STF testing procedure in accordance with the parameters specified in section
2.6. Results of individual testing runs may be seen in this table as well as linearly extrapolated strain threshold values for each respective company’s samples. It should be noted that a stroke-strain relationship, developed in Phase I of testing, was used as a means to influence the strain within each tested sample.

Based on the STF results presented in Table 19, for heat NX4720TK, it is clear that the Special Metals samples had the highest DDC susceptibility with a strain threshold of only 3.7%. WSI samples were slightly more resistant to DDC formation with a strain threshold of 6.5%, followed by EPRI samples, which performed the best, having a strain threshold value of 7.2%. As previously mentioned, the strain threshold indicates the required strain to induce DDC formation within a tested sample, therefore all samples tested at a strain over the strain threshold should contain DDC formations along MGBs.

It should be noted that only STF samples produced by Special Metals demonstrated a transition to gross cracking, while all other samples tested showed minimal DDC formations indicating reduced susceptibility. Samples fabricated by EPRI showed a clear favorability in this testing format while employing heat NX4720TK of FM-52M.
Table 19: Round robin STF data for FM-52M heat NX4720TK

<table>
<thead>
<tr>
<th>Company</th>
<th>Heat/Year</th>
<th>Strain (%)</th>
<th># of Cracks</th>
<th>Threshold</th>
</tr>
</thead>
<tbody>
<tr>
<td>WSI</td>
<td>NX4720TK (52M) 2015</td>
<td>3.7</td>
<td>0.0</td>
<td>~6.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>9.7</td>
<td>4.5</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>5.0</td>
<td>0.0</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>7.8</td>
<td>4.0</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>7.2</td>
<td>3.0</td>
<td></td>
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<td></td>
<td></td>
<td>10.7</td>
<td>5.0</td>
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<td></td>
<td></td>
<td>14.1</td>
<td>5.5</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>13.3</td>
<td>7.0</td>
<td></td>
</tr>
<tr>
<td>Special Metals</td>
<td>NX4720TK (52M) 2015</td>
<td>1.7</td>
<td>0.0</td>
<td>~3.7</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2.3</td>
<td>0.0</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
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<td>7.4</td>
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<td>9.5</td>
<td>4.0</td>
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<td></td>
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<td>4.1</td>
<td>0.5</td>
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<td></td>
<td></td>
<td>8.6</td>
<td>7.5</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>7.9</td>
<td>2.5</td>
<td></td>
</tr>
<tr>
<td>EPRI</td>
<td>NX4720TK (52M) 2015</td>
<td>8.6</td>
<td>4.0</td>
<td>~7.2</td>
</tr>
<tr>
<td></td>
<td></td>
<td>7.1</td>
<td>2.5</td>
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<tr>
<td></td>
<td></td>
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<td></td>
<td></td>
<td>3.9</td>
<td>0.0</td>
<td></td>
</tr>
</tbody>
</table>

6.3.2 NX5213TK

Table 20 shows the STF data for round robin specimens produced with heat NX5213TK. Similar to the previous heat tested, 8-9 samples were prepared for each participating company and run through the STF testing procedure with the newly “optimized” parameters. Threshold values, shown in the right column in Table 20, indicate the level of susceptibility that was observed for each companies STF specimens.

STF results, presented in the following table, indicate increased DDC susceptibility in samples fabricated by Special Metals, with a strain threshold of 4%. Samples
fabricated by WSI and EPRI performed very similarly with strain threshold values of 5.8% and 6.1%, respectively. Special Metals samples showed very similar testing results with that of FM-52M heat NX4720TK, while both the WSI and EPRI samples had increased susceptibility during testing of heat NX5213TK. Again, EPRI samples showed the highest resistance to DDC formation, although WSI samples showed similar DDC resistance.

It should be noted that no transition to gross cracking was observed in any of the tested samples (including a Special Metals sample tested at 14.5% strain). While this fact does not speak for the quantification method of the STF test, it does indicate that increased strain levels induced within samples of this heat show increased resistance to DDC formation. Results in samples fabricated with this heat may be due to reduced impurity concentration along weld metal MGBs that can otherwise lead to increased grain boundary decohesion efforts at similar strain values.
Table 20: Round robin STF data for FM-52M heat NX5213TK.

<table>
<thead>
<tr>
<th>Company</th>
<th>Heat/Year</th>
<th>Strain (%)</th>
<th># of Cracks</th>
<th>Threshold</th>
</tr>
</thead>
<tbody>
<tr>
<td>WSI</td>
<td>NX5213TK (52M) 2015</td>
<td>10.4</td>
<td>5.0</td>
<td>~5.8</td>
</tr>
<tr>
<td></td>
<td></td>
<td>9.6</td>
<td>4.0</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>5.7</td>
<td>0.0</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>7.4</td>
<td>1.0</td>
<td></td>
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<td></td>
<td></td>
<td>8.2</td>
<td>3.0</td>
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<td></td>
<td></td>
<td>7.7</td>
<td>2.0</td>
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<tr>
<td></td>
<td></td>
<td>7.7</td>
<td>1.5</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>3.6</td>
<td>0.0</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>6.2</td>
<td>1.0</td>
<td></td>
</tr>
<tr>
<td>Special Metals</td>
<td>NX5213TK (52M) 2015</td>
<td>5.8</td>
<td>0.0</td>
<td>~4.0</td>
</tr>
<tr>
<td></td>
<td></td>
<td>5.4</td>
<td>1.0</td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>4.3</td>
<td>1.0</td>
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<tr>
<td></td>
<td></td>
<td>1.3</td>
<td>0.0</td>
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<tr>
<td></td>
<td></td>
<td>9.3</td>
<td>2.0</td>
<td></td>
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<td>1.3</td>
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<td></td>
<td></td>
<td>14.5</td>
<td>4.0</td>
<td></td>
</tr>
<tr>
<td>EPRI</td>
<td>NX5213TK (52M) 2015</td>
<td>6.1</td>
<td>0.5</td>
<td>~6.1</td>
</tr>
<tr>
<td></td>
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<tr>
<td></td>
<td></td>
<td>11.2</td>
<td>4.0</td>
<td></td>
</tr>
</tbody>
</table>

6.3.3 EXOA51P

Round robin STF results for FM-52M, heat EXOA51P, are shown in Table 21. Results presented in the following table indicate increased DDC resistance for all STF specimens that were fabricated with this particular heat of FM-52M. Special Metals samples demonstrated the highest DDC susceptibility with respect to the other companies’ STF specimens with a resultant strain threshold value of 7.3%. However, this is a considerable increase from the ~4.0% strain threshold that was observed in both other heats of FM-52M. WSI samples showed a strain threshold of 8.6% and EPRI samples showed the highest DDC resistance with a strain threshold of 9.4%. It is clear from
testing that heat EXOA51P produced the best results, showing the lowest DDC susceptibility of all heats tested. No transitions to gross cracking were observed in any of the samples tested and minimal DDC formations were observed in most samples.

Reduced DDC susceptibility, observed with this heat of FM-52M, may be due to the slight increase in Zr and B additions, which are both known to improve creep resistance. Si additions are also reduced as compared to the other heats tested which may act to suppress grain boundary embrittlement phases that may form within the other samples. Nb, Cr, and C concentrations in this heat of FM-52M are also slightly increased which may result in higher volume fraction NbC or M_{23}C_{6} precipitates which may act to pin MGBs on cooling.
6.3.4 Compiled Round Robin Results

The following section will provide insight into the round robin STF testing study that was conducted at Ohio State. Compiled results of the STF round robin test may be seen in Table 22, including strain thresholds for all 9 heats of material that were tested. The following figures (Figure 31 and Figure 32), organize results by company and heat, respectively, to allow comparisons to be made on heat to heat variations as well as weld procedure developmental variations. It should be noted that all STF testing was
conducting with the use of consistent parameters in order to limit possible variations in resultant data.

Table 22: Complied STF results for round robin testing

<table>
<thead>
<tr>
<th>Company</th>
<th>Heat</th>
<th>Strain Threshold, $\varepsilon_{\text{min}}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>WSI/AZZ</td>
<td>NX4720TK</td>
<td>6.5</td>
</tr>
<tr>
<td></td>
<td>NX5213TK</td>
<td>5.8</td>
</tr>
<tr>
<td></td>
<td>EXOA51P</td>
<td>8.6</td>
</tr>
<tr>
<td>Special Metals</td>
<td>NX4720TK</td>
<td>3.7</td>
</tr>
<tr>
<td></td>
<td>NX5213TK</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>EXOA51P</td>
<td>7.3</td>
</tr>
<tr>
<td>EPRI</td>
<td>NX4720TK</td>
<td>7.2</td>
</tr>
<tr>
<td></td>
<td>NX5213TK</td>
<td>6.1</td>
</tr>
<tr>
<td></td>
<td>EXOA51P</td>
<td>9.4</td>
</tr>
</tbody>
</table>

The following figure represents the STF data for the round robin testing conducted at OSU, categorized by the various fabrication methods used to manufacture STF samples. Based on the collected data in Figure 31, it is clear that specimens fabricated by Special Metals showed the highest susceptibility to DDC formation during the STF testing procedure, while EPRI samples generally showed the most resistance to DDC formation. As previously mentioned, EPRI and WSI samples were manufactured with a GTAW process, while Special Metals samples were fabricated via a GMAW process. Weld procedure specifications for the sample mock-ups may be seen in Table 12 in section 5.4 of this thesis.
Samples provided by Special Metals Co. used a GMAW process during STF sample fabrication to fill a 75° included groove in 3/4” 690 plate. Typically, an increased travel speed may be used with GMAW processes due to the increased material deposition rate that is accompanied by the higher heat input of GMAW. This may result in an increased partial pressure of oxygen (less shielding gas coverage) over the welded area, which would result in increased oxygen segregation to weld metal grain boundaries and therefore brittle oxide formations at these boundaries. Increased oxygen content is known to increase DDC susceptibility and may very well be the cause of the increased DDC response observed in Special Metals STF samples.

It has already been proven, in Phase II of this study, that increasing oxygen concentration in the spot welding stage of STF testing results in an increase in DDC susceptibility. Therefore, reduced shielding gas coverage in samples fabricated by Special Metals (due to increased travel speed, and therefore reduced shielding gas coverage) could very well lead to their hindered performance in the round robin STF study.

Poor performance of Special Metals STF samples in this study may also likely be due to the solidification rate and heat input of the GMAW process employed. With increased heat input, solidification rate as well as cooling rate of the weld pool is reduced, causing more time for migration of grain boundaries or precipitation of secondary phases. Since there are limited alloying elements in the FM-52M heats used to manufacture these specimens, precipitation effects may not be significant enough to pin MGBs, which may otherwise result in a tortuous grain boundary character. This would result in increased time for MGBs to straighten, becoming less tortuous and therefore
more susceptible to DDC formation. GMAW processes also tend to use mixed shielding gasses in order to create arc stabilization in the weld, however this is not the case in this study as 100% Ar shielding gas was employed during the fabrication of samples. This does however create a more unstable arc, which may also be a result of the poor DDC response that was observed in this study.

According to Figure 31, EPRI samples showed the best resistance to DDC in the round robin STF study. This is likely due to the minimal heat input that was used to fabricate these samples, as compared to the other participating companies. A pulsed current schedule was also used to fill U-grooves in Ni-600 plate, which may result in dendrite fragmentation and therefore a refined grain structure that would be more resistant to creep deformation mechanisms, such as DDC.
As previously mentioned, STF heat to heat variations in all three heats of FM-52M showed reduced DDC susceptibility in heat EXOA51P. Figure 32 shows a considerable increase in strain threshold values for each respective company’s tested samples when employing this heat, with a maximum strain threshold value of 9.4% in samples provided by EPRI. This was due to the extent of alloying found in this particular heat of FM-52M as compared to the other two heats tested.

Increased concentrations of Zr and B result in increased creep strength at MGBs. Since DDC is a “creep-like” phenomenon, improving the creep life of a material may have substantial influence on the DDC susceptibility of that particular material. Reduced Si content in this heat, may have also resulted in improved DDC response due to limited
formation of grain boundary embrittlement phases that may otherwise lead to grain boundary decohesion at reduced strain levels. The reduced partition coefficient of Si, results in increased diffusion rates of this element at high temperatures, which may result in increased Si concentrations at SGB’s and sub solidification grain boundaries. The formation of brittle silicide’s at these boundaries (as predicted by JMatPro-Ni) may likely result in decohesive efforts which contribute to DDC formation at reduced strain levels. Reduced Si levels in EXOA51P results in improved DDC response for this reasoning.

Increased Nb, Cr, and C concentrations in heat EXOA51P may also yield an increased resistance to DDC, due to the formation of NbC or M23C6 precipitates at the end of solidification. Secondary phase formation, as shown in Phase II of testing, results in MGB pinning, which results in tortuous grain boundary character that has a difficult time propagating crack formation. While these precipitates may not necessarily stop DDC all together, they do have an effect on the crack propagation rate, which may act to limit the extent of crack formation.

The other two heats of FM-52M tested in this study, NX4720TK and NX5213TK, showed similar STF results due to limited variations in material composition. Slight variation in STF data may be a result of random scatter in the STF data or influence of small compositional variation.
6.3.5 Effect of Oxygen Concentration on STF Round Robin Study

The effect of oxygen on the round robin STF study was examined with the use of an Ar/2%O₂ mixed shielding gas that was employed during the GTA spot welding stage of testing. Like Phase II of this study, the results indicated a significant deleterious effect on DDC response when employing the mixed shielding gas containing oxygen. 2 samples of each heat for each company were tested in this study, one side made with a 100%Ar shielding gas while the other employed the mixed shielding gas. Results are compiled in Table 23 and may be viewed more graphically in Figure 33. It is clear from these results that oxygen has a negative impact on DDC resistance and may likely be due to an embrittlement phase that is present along weld metal MGBs. Again, further work is being conducted to determine the exact phases present along WM MGB’s in the presence of
oxygen as well as quantifying a critical concentration that leads to grain boundary degradation in Ni-base alloys and FM’s.

Table 23: Round robin STF data comparing a 98Ar/2O2 shielding gas mixture and a 100Ar shielding gas during the GTA spot weld stage of the STF test.

<table>
<thead>
<tr>
<th>Company</th>
<th>Heat/Year</th>
<th>Strain (%)</th>
<th># of Cracks (100%Ar)</th>
<th># of Cracks (98%Ar-2%O2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>WSI/AZZ</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>NX4720TK (52M) 2015</td>
<td>7.8</td>
<td>4.0</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td></td>
<td>7.2</td>
<td>3.0</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>NX5213TK (52M) 2015</td>
<td>8.2</td>
<td>3.0</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>7.7</td>
<td>2.0</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>EXOA51P (52M) 2015</td>
<td>7.3</td>
<td>0.0</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>19.7</td>
<td>6.0</td>
<td>17</td>
</tr>
<tr>
<td>EPRI</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>NX4720TK (52M) 2015</td>
<td>11.7</td>
<td>5.0</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td></td>
<td>8.9</td>
<td>3.0</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>NX5213TK (52M) 2015</td>
<td>7.0</td>
<td>1.0</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td></td>
<td>8.0</td>
<td>2.0</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td>EXOA51P (52M) 2015</td>
<td>8.9</td>
<td>0.0</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td></td>
<td>16.4</td>
<td>7.0</td>
<td>10</td>
</tr>
<tr>
<td>Special Metals</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>NX4720TK (52M) 2015</td>
<td>7.4</td>
<td>1.0</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td></td>
<td>9.5</td>
<td>4.0</td>
<td>23</td>
</tr>
<tr>
<td></td>
<td>NX5213TK (52M) 2015</td>
<td>1.3</td>
<td>0.0</td>
<td>0</td>
</tr>
<tr>
<td></td>
<td></td>
<td>9.3</td>
<td>2.0</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>EXOA51P (52M) 2015</td>
<td>18.7</td>
<td>8.0</td>
<td>15</td>
</tr>
<tr>
<td></td>
<td></td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>
Figure 33: STF Round Robin study comparing oxygen presence in shielding gas mixture to a 100% Ar shielding gas.

Phase IV: 6.4 “New” STF Testing Procedure

A new STF testing procedure was designed for testing of material that is in quantities insufficient to produce entire STF samples (typically the case when filler wire is not available or during material development stages). This was in conjunction with a subsequent project being conducted at OSU which has been designed to develop a high-Cr, Ni-base FM for nuclear power applications. The motivation for this work was in result to the limited FM which was produced via compositional optimization studies.
conducted at OSU for this project. Efforts to conduct weldability testing on this new FM were hindered due to limited material availability, which required a new method of testing.

A new STF testing procedure was created by melting small “buttons” of the desired composition into a cavity within the gauge section of an STF sample. An autogenous GTAW process was used to melt the testing material (button) into the specimen cavity, which resulted in samples that contained a sufficient amount of desired testing material within the sample gauge section. Following the melting process, the samples were ground flat and then run through the STF testing procedure as usual. A step by step picture sequence of this process may be seen in section 5.3.3 of this thesis.

In order to gain a better understanding of the testing environment and effectiveness of the new procedure, results for two compositions of a Ni-Cr-Hf FM are included in Table 24 and Error! Reference source not found.. It should be noted that only two samples of each respective material were tested in this phase of testing, but results indicate the effectiveness of the new procedure. The higher Hf material (indicated in Table 24 as Ni-30Cr-2.5Hf), showed improved DDC resistance as compared to the Ni-30Cr-0.25Hf material, which would be expected due to the increased eutectic formation that would subsequently occur during the spot welding stage. As previously mentioned, increased volume fraction eutectic results in highly tortuous MGBs and they are pinned on cooling, which limits their mobility. While initial indications suggest that the new STF testing procedure shows testing accuracy, more samples may need to be run to prove testing repeatability.
Table 24: STF data for the “new” STF testing procedure, allowing testing of material in limited concentrations.

<table>
<thead>
<tr>
<th>Material</th>
<th>Stroke (mm)</th>
<th>Strain (%)</th>
<th>Cracks</th>
<th>Threshold</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni-30Cr-0.25Hf</td>
<td>2</td>
<td>5.23</td>
<td>0</td>
<td>~6.0</td>
</tr>
<tr>
<td>Ni-30Cr-0.25Hf</td>
<td>3</td>
<td>7.65</td>
<td>3</td>
<td>~6.0</td>
</tr>
<tr>
<td>Ni-30Cr-2.5Hf</td>
<td>3</td>
<td>8.31</td>
<td>0</td>
<td>~9.0</td>
</tr>
<tr>
<td>Ni-30Cr-2.5Hf</td>
<td>3.5</td>
<td>9.7</td>
<td>2</td>
<td>~9.0</td>
</tr>
</tbody>
</table>

Figure 34: Graphical depiction of the data provided in Table 24.

Phase V: STF Digital Image Correlation

Phase V of testing was conducted in an effort to observe strain gradients on STF sample gauge sections. DIC analysis was used in order to find regions of high strain.
concentration, where crack initiation might occur. As discussed in section 5.3.4, DIC strain maps are generated via pixel (paint speckle) displacement in highly localized regions and converted to strain measurements via Equation 1 and NCorr software algorithms. Before and after strain maps, generated by the DIC software, are shown in Figure 35 and Figure 36, respectively. Figure 36 shows a concentrated strain measurement of ~20% in the center gauge section after testing, which matches well with the indentation spacing calculation used, which measured ~19.5% strain. This confirms the success of the strain measurement system that is currently in place. DIC maps do however, show that strain in STF samples tends to be concentrated at the center of the sample gauge section, and therefore indentation spacing measurements give a strain value that is averaged over their width rather than a localized measurement that the DIC strain map provides. Future work associated with STF DIC maps will be used to
Figure 35: STF DIC strain map before STF testing.

Figure 36: STF DIC strain map after STF testing. Strain distribution may be viewed via the legend on the right as percent strain.
Chapter 7: Conclusions

1. Phase I: STF Parameter “Re-Optimization” for Ni-base alloys
   
a. The range of GTA spot weld parameters tested showed repeatable STF results, indicating that microstructural variations may need to be quite significant in order to influence DDC susceptibility.
      
i. GTA spot weld parameters: peak current, time at peak current, and current downslope time, showed an insensitivity to STF testing outputs.
   
b. STF optimization of Gleeble parameters showed that stroke rate (mm/sec), or the rate at which the strain is applied to STF sample gauge sections, influences DDC susceptibility quite significantly.
      
i. Increasing the sample stroke rate results in a reduced susceptibility to DDC formation.
   
ii. There exists some range of insensitivity where small variations in stroke rate do not have any effect on the strain threshold required to induce DDC.

iii. All other parameters tested in the STF optimization study showed an insensitivity to STF outputs.
c. STF parameter development in Ni-base alloys shows similar results to those that were developed by Nissley, and STF parameters may be employed as usual.

2. Phase II: Insight Into DDC Mechanisms
   
a. Pure materials, such as Ni-200, appear to have the highest susceptibility to DDC formation with a STF strain threshold of ~1%.
   
i. Extensive cracking was observed in all Ni-200 samples tested.
   
ii. Increased grain boundary mobility, in the absence of secondary phases, results in MGBs that are long and non-tortuous.
   
iii. Fracture surfaces in Ni-200 STF samples showed a smooth intergranular morphology, indicative of DDC.

b. Inconel Alloy-600 and Inconel Alloy-625 were more resistant to DDC formation due to inter-dendritic secondary phase formations within the austenite matrix.
   
i. Inconel Alloy-600 forms intragranular carbides, which have some influence over the DDC susceptibility of this alloy, giving STF specimens comprised of this alloy a strain threshold of ~10%.
   
ii. Inconel Alloy-625 has increased Nb, C, Cr, and Mo contents, which result in NbC precipitates and Laves Phase, making it resistant to DDC at all strain levels tested (up to 35%).
   
iii. Fracture surfaces in Inconel Alloy-600 samples show some evidence of micro-ductility.
3. Phase III: Round Robin STF Study
   a. FM-52M Heat, EXOA51P, was most resistant to DDC formation of all heats tested, which is most likely due to increased additions of Zr, B, Nb, Cr, and C.
   b. Specimens provided by Special Metals Co. showed the highest susceptibility to DDC formation
      i. Increased oxygen levels, most notably associated with the GMAW process, may have contributed to grain boundary decohesive efforts at reduced strain levels.
   c. STF samples provided by EPRI showed the highest resistance to DDC formation
      i. Current pulsing may lead to dendrite fragmentation, and therefore increased nucleation sites for new grain formation, leading to a refined grain size in STF samples that is more resistant to DDC.
   d. While external factors may have influenced STF response in the round robin study, compositional effects are likely the most reasonable cause of susceptibility variations between participating companies.
4. Phase IV: New STF Procedure
   a. The newly developed STF testing procedure for testing materials in limited quantities proved to be an effective testing method.
5. Phase V: STF Digital Image Correlation
a. The DIC analysis verified the current strain measuring system used in the STF testing procedure.

b. Strain in more concentrated to the center of sample gauge sections that originally thought.

6. Oxygen Measurements

a. DDC susceptibility was increased in Alloy-600 and Alloy-625 samples that were tested with the use of an Ar/2%O2 shielding gas during the GTA spot welding stage of testing.

b. The introduction of oxygen in the STF testing procedure likely results in grain boundary decohesion at reduced strain levels.

7. Heat Treatments

a. High temperature heat treatments conducted on Inconel Alloy-600 STF samples resulted in a coarse grain structure that is more susceptible to DDC due to a reduced grain boundary area per unit volume.

   i. Increased sample grain size results in incremented localized strain concentration along sample grain boundaries and grain boundary triple points.

8. STF Testing Procedure

a. The STF testing procedure, as it is employed at OSU, is a verifiable test in determining a materials response to DDC susceptibility.

b. Consistent results in all Ni-base alloys and FM’s tested demonstrate the repeatability of the STF testing procedure.
Chapter 8: Recommended Future Work

Based on the completed work in this thesis, the following is recommended as future work to expand on the comprehensive understanding of DDC formation in Ni-base alloys and FM’s.

1. The effect of oxygen concentration on DDC performance clearly has a negative influence, as determined in the final stages of Phase II and Phase III. Expansion on this knowledge may be critical to understanding the underlying mechanisms which govern DDC formation in these alloys.
   a. EDS testing to observe oxide formation along weld metal MGBs and measurement of oxygen content in already tested samples.
   b. TEM work to identify grain boundary phase formations.
   c. Determining critical oxygen concentrations to hinder DDC performance in Ni-base alloys and FM’s.

2. In depth metallurgical characterizations may be necessary to examine how specific alloying additions influence a materials response in the STF testing procedure. This may also give some heightened insight into the mechanisms which govern DDC susceptibility in FCC materials. Samples used in Phase II may be the best materials to understanding this approach.
3. Nickel-200 was used as a baseline material in order to test a simplified system and gain valuable understanding of DDC formations.
   a. Add alloying elements, such as Nb, Cr, Mo, and C to examine individual effects on DDC response and MGB characterization.
   b. Conduct In-Situ SEM examinations in conjunction with the STF testing procedure to observe GB sliding as it occurs.

4. The effect of precipitate morphology, size, and distribution is known to have some influence on DDC formation in Ni-base alloys and FM's
   a. Conduct a study in which precipitation interactions are closely observed throughout the STF testing procedure.
   b. Vary alloying element concentration (Nb, C, Cr, Si, etc.) in order to vary precipitate formation and observe which is most effective at pinning MGBs.

5. The STF “re-optimization” study neglected to examine the effect of multiple heating cycles on DDC response as well as testing conducted “on-cooling.”
   a. Test a variety of multi-heat cycles in the Gleeble to simulate multi-pass weld procedures used in the nuclear industry. This may result in increased residual stresses which would likely increase the DDC response in a particular alloy.

6. Improve upon the digital image correlation study to examine how strain concentrates along WM MGBs and or precipitate formations.
a. Find a critical localized strain along a WM MGB to lead to void formation and therefore DDC.

b. Observe strain concentrations along crack tips or voids that may lead to DDC propagation.
References


Appendix A

Gleeble QuickSim Program for STF Test

The following figure is the STF-Macro programed via QuickSim2. Controlling variables include temperature (°C) of the sample, stroke or jaw movement (mm), stroke rate (mm/s), time to heat to peak temperature (°C/s), and time at peak temperature before the stroke is applied (s).
Figure 37: QuickSim program used to generate STF testing procedure.