Recreating Ductility-Dip Cracking via Gleeble®-Based Welding Simulation

Simulated strain ratcheting was used to recreate welding thermomechanical histories leading to ductility-dip cracking

BY S. LUTHER AND B. ALEXANDROV

ABSTRACT

Face-centered cubic alloys, such as nickel-based alloys and austenitic stainless steels, are important to many industries, notably nuclear power generation and petrochemical. These alloys are prone to ductility-dip cracking (DDC), an intermediate-temperature, solid-state cracking phenomenon. They experience an abnormal elevated-temperature ductility loss, which leads to cracking upon applying sufficient restraint. A unified mechanism for DDC has been elusive. To learn more about DDC, an experimental procedure has been designed and evaluated for use in future studies. It is a thermomechanical test that replicates welding conditions via simulated strain ratcheting (SSR) using the Gleeble thermomechanical simulator. This study evaluates SSR and aims to establish the procedure is reproducible and adequately optimized for producing DDC. A design of experiments was created with four alloys tested at varying preloads, elevated temperature strains, and a number of thermomechanical cycles. Mechanical energy imposed within the DDC temperature range was used for quantification of the effect of thermomechanical cycling on the DDC response. The materials tested were 310 stainless steel and Nickel 201 base metals as well as nickel-based filler metals 52M and 52MSS. The SSR successfully recreated DDC while maintaining higher fidelity to actual production conditions than past laboratory tests and offered a more controlled environment than large-scale weld tests. Therefore, the SSR will provide a viable experimental procedure for learning more about the DDC mechanism.

KEYWORDS

- Ductility-Dip Cracking (DDC) • Nickel-Based Alloys
- Stainless Steels • Thermomechanical Testing
- Weldability

is being developed. The industry-driven motivation is the mitigation of ductility-dip cracking (DDC) in nickel-based groove welds and structural weld overlays (SWOLs) on nuclear pressure vessel nozzles. This cracking phenomenon has been known to exist for almost 100 years but has not been a major problem in this industry until the relatively recent development of high-chromium filler metals, such as Alloys 52 and 82. Alloy 52M, an Alloy 52 variant, was later selected as an overlay material to protect underlying metals undergoing primary water stress corrosion cracking, and DDC has been observed in all three alloys (Refs. 1, 2). DDC is not fully understood; therefore, fundamental research is needed to elucidate a micro-mechanistic explanation. In general, DDC involves anomalous intermediate-temperature ductility loss. High restraint during welding leads to cracking, especially in multipass welding. The current explanation is stress combined with elevated temperatures encourages grain boundary (GB) sliding. This sliding enables void formation and these voids eventually grow to form cracks, which are free to propagate (Refs. 3–7). Many researchers also contend sulfur plays a key role in initiating cracking (Refs. 8–10). However, clarification is needed on both fronts because the finer details of the mechanisms on the microstructural level are unclear. The utility of clarification is more effectively avoiding DDC in real-world welding. DDC can lead to expensive rework due to the low flaw tolerance in nuclear power.

Design of experiments (DOE) is used in this study. DOE is a statistical analysis technique utilized to minimize the number of samples needed to evaluate the effect of factors on a response. Each factor is tested at two levels, low and high, and at least one replicate is needed, thus leading to a number of experimental runs equal to two times two to the power of the number of factors. For example, this study involves several DOEs with two factors, so each DOE has eight runs. Similar to multiple linear regression, DOE analysis results in an equation with each factor multiplied by a coefficient, and each coefficient's significance is represented by a P-value.

Introduction

Background

A thermomechanical simulation designed to imitate the multipass welding conditions experienced by austenitic alloys

Literature Review

Numerous researchers have developed ways to artificially

Fig. 1 — Geometry used for tensile samples. Dimensions are in inches.

produce DDC in metals of interest. One specifically designed to study DDC is the strain-to-fracture (STF) test (Ref. 11). Others that have been used to study DDC include the programmable-deformation-crack (PVR), hot ductility, hot tensile, and fissure bend tests, though these were not originally designed for DDC (Refs. 12–15). Through this body of research, it is possible to rank DDC-susceptible alloys relative to one another, and each has contributed insight into the mechanism. However, information has also been gained by welding large imitation mockups to create DDC as it would form in the field. Researchers who use these methods often argue artificial laboratory tests are lacking because they do not mimic actual welding conditions (Refs. 15, 16). Even the prolific susceptibility ranking completed using STF only provides this ranking on the basis of relative comparison of crack counting rather than a holistic theory capturing the unexplained micromechanistic details of DDC. Both the externally restrained laboratory tests and the self-restrained field methods have made significant contributions to the understanding of DDC. However, further research will attempt to mimic production conditions, thereby addressing the fidelity issues of past laboratory tests. It will also utilize a highly controlled and reproducible test procedure and a sample design, which is easily characterized, to overcome drawbacks of available field tests. Thus, a new laboratory test method has been developed with inspiration from McCracken and Tatman's narrow groove weld test (Ref. 16). They demonstrated the ability to correlate DDC crack locations in a narrow groove weld with areas of high strain accumulation, or strain ratcheting, as predicted by a finite element analysis model using Sysweld™. The newly developed experimental procedure, described in the experimental methodology section, generally applies this strain ratcheting approach to a weld-metal-containing sample, which is heated using a welding temperature profile experienced in multipass welding. This procedure is termed simulated strain ratcheting (SSR).

One area where DDC knowledge is lacking is in explaining certain features that can be found on DDC fracture surfaces called thermal faceting. Achieving this end is not the purpose of this article; however, a brief introduction to the phenomenon is given here to provide background on some

Fig. 2 — Final Nickel 201 samples used in SSR testing with the welded gauge section.

of the results presented herein. Thermal faceting is known to occur predominantly in austenitic alloys, and it simply refers to a change in equilibrium crystal shape, which happens at elevated temperatures to minimize surface or interfacial energy (Ref. 17). Thermal faceting manifests as very small terraces or steps on the order of 50–200 nm in size, which can be present along GBs or on fracture surfaces. Incidentally, this change occurs in the DDC temperature range. Very little research has been done to see if this phenomenon has any significance to DDC or if it is just coincidence. Future work in this project will further investigate the topic.

Objective

The overarching goal of this research is to advance the fundamental understanding of DDC toward a mechanistic explanation for cracking through exploring the potential involvement of localized GB phenomena, such as sulfur-induced amorphization and GB faceting. To reach this end, material properties will be identified and quantified that directly relate to DDC cracking susceptibility and GB decohesion or sliding. This will be made possible by simulating thermomechanical welding conditions in austenitic alloys to study DDC and strain ratcheting, or by using SSR. This new procedure has been designed to mimic production conditions to reduce the artificiality of past testing and ultimately clarify the micromechanistic details of DDC. In this study, the authors describe the process of creating and optimizing the SSR procedure, which will be used in future research on this subject.

Experimental Methodology

Materials and Test Sample Preparation

The chemical composition of each material used in this study is shown in Table 1. These alloys were selected because they represent a range of DDC susceptibilities according to past research. The order from least to most susceptible is Alloy 52MSS, 310 stainless steel (SS), Alloy 52M, and Nickel 201 (Refs. 11, 18, 19). The proposed SSR procedure

Fig. 3 — Image of final 310 SS sample with the welded gauge section and spot weld.

was expected to produce more severe cracking in the more susceptible alloys to demonstrate its sensitivity to DDC susceptibility, and it was expected to do so consistently to prove its reproducibility. 310 SS and commercially pure Nickel 201 base metals were obtained in the form of 0.25-in. rolled plate. Bead-on-plate (BOP) welds were completed autogenously using gas tungsten arc welding (GTAW) according to the parameters shown in Table 2. Welds were spaced such that dog-bone tensile samples could be extracted with weld metal centered in the gauge section.

Figure 1 shows the geometry used for these tensile samples, and Fig. 2 is an image of the final Nickel 201 samples. After preliminary testing with Nickel 201 showed an absence of DDC, spot welds were added to the front and back of the center of the gauge section of the 310 SS samples us-

Fig. 4 — Computer-aided design drawing of the machining plan for the Alloy 52M overlay.

ing autogenous GTAW with the parameters in Table 2, and samples were extrinsically strained during heating. Figure 3 shows an image of the final 310 SS samples. Nickel-based filler metal 52M was obtained by machining samples from a mock-up SWOL used for successful welding qualification. The samples were made entirely of weld metal. Figures 4 and 5 show the process of extraction of these samples. Nickel-based filler metal 52MSS samples were created by

Table 1 — Material Compositions (wt-%) of 310 SS, Ni 201, Alloy 52M, and Alloy 52MSS

Element	310 SS	Ni 201	Ni 52M	Ni 52MSS
Al	—	—	0.059	0.29
B	—	—	< 0.0001	—
C	0.045	0.018	0.034	0.03
Co	0.270	—	0.018	0.001
Cr	25.41	—	30.1	28.7
Cu	0.20	0.01	0.22	0.04
Fe	53.147	0.004	9.47	5.42
Mn	0.86	0.25	0.76	0.44
Mo	0.28	—	0.15	3.49
N	0.038	0.28	—	—
Nb	0.006	—	0.83	1.31
Ni	19.15	99.47	57.9	58.1
P	0.023	0.001	0.007	0.014
S	0.001	0.0002	0.015	0.001
Si	0.57	0.08	0.09	0.15
Ta	—	—	—	1.66
Ti	—	—	0.22	0.23
W	—	—	0.071	—
Zr	—	—	0.002	—
Others	—	—	0.056	0.124

Table 2 — GTAW Parameters Used for BOP and Spot Welding

Weld Type	Arc Voltage	Travel Speed	Downslope	Current	End Current	Shielding Gas	Gas Flow Rate
BOP	12 V	3 in./min	N/A	200 A	N/A	99.995% Ar	40 ft ³ /min
Spot	12.5 V	N/A	12.7 s	120 A	20 A	99.995% Ar	40 ft ³ /min

Fig. 5 — Final machined 52M samples.

groove welding 0.25-in. Alloy 690 plates in a butt joint configuration, then extracting samples in a manner similar to the 310 SS and Nickel 201. A cold wire pulsed GTAW process was used on two Alloy 690 base plates in the butt joint configuration, each with a 45-deg bevel and a $\frac{1}{4}$ -in. flat at the root. Welding parameters for the Alloy 52MSS plates are presented in Table 3. Circular notches of 0.25 in. in diameter were added to the gauge sections of the Alloy 52MSS samples in the weld metal after failure was observed in the base metal during preliminary testing. Figure 6 shows the final Alloy 52MSS sample geometry used in testing.

Testing Procedures

Samples were loaded into the Gleeble thermomechanical simulator after a single Type-K thermocouple was attached to the center of the gauge section. Stainless steel blocks with carbide grips were used to secure the sample in the Gleeble jaws. The U-jacks in the chamber were first handtightened to settle the blocks against the U-jacks and the grips onto the sample. Then, an approximately 70% room temperature yield strength tensile load was applied to the sample until the force remained steady to ensure the blocks were fully mating with the jaw surface. U-jacks were tightened to their final position while the hydraulic system was still engaged. A dilatometer was then attached to the center of the gauge section such that the arms of the dilatometer were in-line with the thermocouple and the linear variable displacement transformer position was at its center point. The dilatometer measured changes in the transverse cross section. This reading was converted to an axial measurement in-line with the loading direction during data

Fig. 6 — Final 52MSS sample with circular notches in the weld metal.

Fig. 7 — Image of SSR setup in the Gleeble chamber.

processing using the materials' Poisson's ratio.

Figure 7 is an image of the experimental setup in the Gleeble chamber. The Gleeble chamber was sealed and the roughing vacuum was used until the chamber pressure readout was less than 0.05 torr. In addition, 99.998% argon was pumped into the chamber at 40 ft³/min until a partial vacuum of 400 torr was reached. The chamber was once more pumped down and refilled with argon at the same parameters to ensure a more complete oxygen purge (Ref. 18). Finally, the SSR user-defined Gleeble script was executed and testing commenced.

The basic SSR procedure involves simulation of multiple welding thermal cycles combined with fixed displacement plus tensile stress preload and/or on-heating extrinsic straining within the DDC temperature range. The aim is to replicate the thermomechanical history in highly restrained multipass welds. A welding thermal cycle, which was obtained from the

Table 3 — Multipass Welding Parameters Used for 52MSS Groove Welds

Layer	Number of Beads	Layer Depth	Current	Voltage	Travel Speed	Wire Feed Speed
Butter	0	0.27 in.	—	—	—	—
1	3	0.209 in.	165/145 A	9.2 V	3.2 in./min	30/30 in./min
2	3	0.136 in.	165/145 A	9.2 V	3.2 in./min	30/30 in./min
3	3	0.075 in.	165/145 A	9.2 V	3.2 in./min	30/30 in./min
4	3	0.028 in. (above flush)	85/165 A	9.9 V	3.0 in./min	41/41 in./min

Fig. 8 — Schematic of a single preloaded SSR cycle.

thermocouple data recorded during multipass welding, was applied to the test sample. A peak temperature was selected, which allows the material to pass through the DDC temperature range without reaching the solidus temperature. This temperature corresponds to a point about 50°–100°C below the 99% fraction solid temperature predicted by a Scheil solidification simulation in Thermo-Calc using the material composition of the sample. The peak temperature for Nickel 201, 310 stainless steel, and Alloy 52M was 1200°C, and the peak temperature for Alloy 52 MSS was 1100°C. Heating occurred at about 140°C/s and air cooling was about 3°–6°C/s depending on the material. After reaching peak temperature, the sample was free cooled to 100°C before the next cycle began.

After the desired number of thermomechanical cycles was reached, the hydraulic system was turned off, the chamber was vented and opened, and the sample was retrieved from the Gleeble. Data processing of SSR outputs was completed using MATLAB® R2019a. These outputs included stroke, time, temperature, force, power angle, and dilatometer reading at 10 Hz during testing.

Design of Experiments

The basic SSR procedure was applied in three separate

Fig. 9 — Schematic of a single thermal cycle from an SSR test.

Fig. 10 — Example of the integrated stress/strain curve to calculate the imposed mechanical energy. Taken from 310 SS sample 1, 0 DDC strain, and 1 cycle. Result is shown in mega-joules (MJ).

two-factor DOE studies to evaluate the effect of number of applied thermomechanical cycles, room temperature preload, and strain ratcheting on the DDC response in welds of the four tested alloys.

Table 4 — DOE Setup

Sample Number	Number of Thermomechanical Cycles	DOE 1: Nickel 201		DOE 2: Alloy 52M		DOE 3: 310 SS, Alloy 52MSS		
		DDC Strain, %	Preload, %	DDC Strain, %	Preload, %	310SS DDC Strain, %	52MSS DDC Strain, %	Preload, %
1	1	0	0	1.35	0	0	0	0
2	1	0	90	1.35	90	2.7	0.9	0
3	10	0	0	1.35	0	0	0	0
4	10	0	90	1.35	90	2.7	0.9	0
5	1	0	0	1.35	0	0	0	0
6	1	0	90	1.35	90	2.7	0.9	0
7	10	0	0	1.35	0	0	0	0
8	10	0	90	1.35	90	2.7	0.9	0

Note: Nickel 201 did not have any DDC strain applied and the preload for DOE 3 was 0% for all tests.

Fig. 11 — Graphed SSR data from 310 SS sample 4: A — Stress and temperature vs. time; B — strain and temperature vs. time; C — stress and strain vs. time; D — stress and strain vs. temperature; E — stress vs. strain; F — stroke vs. time.

The DOE test matrices are shown in Table 4. The number of thermomechanical cycles was a factor in all three DOEs and varied between 1 and 10. DOE 1 and DOE 2 utilized room temperature preload applied immediately prior to starting to simulate artificial restraint. The low/high preload values were 0 and 90% of the tested material room temperature yield strength. Figure 8 shows a schematic of this preload procedure.

DOE 2 and DOE 3 utilized tensile straining at a constant rate within the DDC temperature range of 800° and 1050°C to reflect the strain ratcheting effect in highly restrained multipass welds found by McCracken and Tatman (Ref. 20). The amount of DDC temperature range strain applied per

thermal cycle is approximately 9% of the threshold strain to complete separation so ten thermal cycles would total to 90% of strain to complete separation. Strain was applied based on jaw displacement as a fraction of the gauge section in the free span between the jaws. For example, a jaw displacement of 2.5 mm with a free span of 25 mm results in 10% strain across the gauge section. The DDC strain was constant in DOE 2 and varied between 0 and 9% in DOE 3. Figure 9 provides a visual representation of one SSR cycle with applied DDC strain. To reiterate, all extrinsic straining occurs on heating and the remainder of testing is conducted under fixed displacement, where intrinsic strain accumulates due to thermal expansion or contraction.

Table 5 — SEM Parameters Used for Fractography

Accelerating Voltage	Beam Current	Working Distance	Stage Bias	Detector
9 kV	50 pA	5–15 mm	–4 kV	T2

Table 6 — Complete DOE Results for Each Material

Material	Sample	Energy, MJ	Cracking	P-Values	Coefficients, MJ	R-Squared, %
Nickel 201	1	2.1012	None	Cycles: < 0.001 Preload: 0.953 P*C: 0.039	Cycles: 1.660 Preload: –0.0009 P*C: –0.00695	98.13
	2	0.9093	None			
	3	18.4291	None			
	4	8.3648	None			
	5	2.1540	None			
	6	0.9918	None			
	7	14.6862	None			
	8	12.1653	None			
Alloy 52M	1	1.3890	None	Cycles: < 0.001 Preload: 0.107 P*C: 0.038	Cycles: 2.136 Preload: 0.0285 P*C: –0.00679	99.15
	2	3.9873	None			
	3	23.2733	Profuse			
	4	19.5568	Profuse			
	5	1.6822	None			
	6	4.1930	None			
	7	19.5719	Profuse			
	8	16.0772	Profuse			
310 SS	1	2.1554	None	Cycles: 0.012 Strain: 0.691 S*C: 0.038	Cycles: 1.542 Strain: –0.69 S*C: 0.772	94.62
	2	1.7501	None			
	3	15.9546	None			
	4	25.6883	Some			
	5	1.8297	None			
	6	1.7634	None			
	7	14.7967	None			
	8	43.7896	Some			
Alloy 52MSS	1	1.7940	None	Cycles: 0.003 Strain: 0.568 S*C: 0.053	Cycles: 1.757 Strain: –2.55 S*C: 1.748	95.76
	2	0.9609	None			
	3	18.0273	None			
	4	38.5063	Failure			
	5	1.8575	None			
	6	1.1144	None			
	7	17.075	None			
	8	23.5176	Failure			

Prior to running the DOEs, the threshold strain to complete separation was determined to ensure samples were not overloaded and fractured during the high-cycle tests. This procedure consisted of running a SSR test with 5% DDC temperature range strain added per cycle until the sample completely separated. For 310 SS, this complete separation strain was 30%, for Alloy 52M it was 15%, and for Alloy 52MSS it was 10%.

The response variable was imposed mechanical energy within the DDC temperature range, which was calculated by integrating the stress-strain curve generated from testing in the temperature range of 800°–1050°C. The heating and cooling portions were calculated separately and added together. Since GB sliding can occur during stress relaxation or stress accumulation, the absolute value of imposed mechanical energy was used for DOE calculations (Refs. 2, 18). Figure 10 shows an example of the integrated

stress/strain curve with the imposed mechanical energy result across the entirety of the curve. This was achieved using MATLAB's built-in trapezoidal area function. Imposed mechanical energy was selected to characterize the effect of strain ratcheting on GB movement/strain because it captures the stress/strain behavior throughout SSR's low-cycle, fatigue-type procedure. Furthermore, the absolute value was used because both stress/strain accumulation and relaxation could potentially cause GB sliding, but in opposite directions. Thus, the response variable needed to account for both directions of sliding.

With two factors and one replicate, the total number of runs was eight per DOE. Four center points were initially planned; however, statistical analysis showed the assumption of linearity remained valid for the first eight runs. The Nickel 201 tests followed the preload/number of thermo-mechanical cycles in DOE 1, the Alloy 52M tests followed

Fig. 12 — 310 SS DDC fracture surface showing thermal faceting.

Fig. 13 — Alloy 52M DDC fracture surface showing thermal faceting.

Fig. 14 — Alloy 52MSS DDC fracture surface showing thermal faceting.

Fig. 15 — 310 SS metallography cross section showing large overloading cracks alongside smaller DDC cracking. The dark spots at interdendritic regions are etching artifacts. The base metal is shown at the bottom of the image.

DOE 2 with preload/number of thermomechanical cycles and a constant DDC strain applied each cycle, and the 310 SS and Alloy 52MSS tests followed DOE 3 with DDC strain/number of thermomechanical cycles and no preload. DOE calculations were performed using Minitab® 18.

Characterization

As-welded sections from each material were examined using destructive metallography to ensure no cracks were present prior to testing. 310 SS was etched using the electrolytic technique in 10% oxalic acid with 6 V and 1 A for 3 s. The nickel-based filler metals were etched using electrolytic technique in 10% chromic trioxide solution with 6 V and 1 A for 3 s. The Nickel 201 base metal was unetched. Little or no etching was intentionally used to avoid confusion between etching artifacts and cracking. An Olympus GX51 optical microscope was used for this task. Also, samples were imaged using differential interference contrast to further enhance surface topography

and hence improve crack identification. Tested samples were prepared and examined following the same metallography procedure. Fractured samples were imaged using a Thermo Fisher Apreo low-vacuum field emission scanning electron microscope (SEM) operated in optiplan mode with the parameters shown in Table 5. Sample preparation consisted of ultrasonic cleaning, ethanol rinse, and bake-out in a light furnace for 20 min.

Results

SSR Test Outputs

There were 32 samples tested across the three DOEs and four materials presented here, and six graphs were generated for each sample using the acquired thermomechanical data. One representative set of graphs is shown in Fig. 11 from 310 SS sample 4, which was subject to high strain and a high number of thermomechanical cycles. In order, the

Fig. 16 — Alloy 52M metallography showing DDC.

Fig. 17 — Alloy 52MSS metallography cross section showing DDC and primary fracture.

graphs show stress and temperature vs. time, strain and temperature vs. time, stress and strain vs. time, stress and strain vs. temperature, stress vs. strain, and stroke vs. time.

DOE Results

The results for each DOE are compiled and summarized in Table 6. Recall the factors for 310 SS and Alloy 52MSS are DDC strain and number of thermomechanical cycles, and the factors for Nickel 201 and Alloy 52M are preload and number of thermomechanical cycles. The critical P-value in this design is 0.05. The P*C or S*C values show the interaction between preload and cycles or DDC strain and cycles. The coefficients shown in Table 6 can be combined to form predictive equations for the imposed mechanical energy (E) in each material's DOE. Equation 1 is for Nickel 201, Equation 2 is for Alloy 52M, Equation 3 is for 310 SS, and Equation 4 is for Alloy 52MSS, where E is the imposed mechanical energy, C is the number of thermomechanical cycles, P is preload, and S is the DDC strain.

- (1) $E = 1.660C - 0.0009P - 0.00695P*C$
- (2) $E = 2.136C + 0.0285P - 0.00679P*C$
- (3) $E = 1.542C - 0.69S + 0.772S*C$
- (4) $E = 1.757C - 2.55S + 1.748S*C$

Optical and Electron Microscopy Observations

Samples with full separation failures from 310 SS, Alloy 52M, and Alloy 52MSS were examined with SEM fractography. These full separation failures came from the stress tests conducted prior to the DOEs, which were used to determine the complete separation strain. All showed the presence of thermal faceting. SEM results for 310 SS, Alloy 52M, and Alloy 52MSS are shown in Figs. 12–14, correspondingly. Sample 4 from each material DOE was selected for destructive metallography since these samples typically showed signs of cracking when viewed macroscopically. Light optical micrographs of 310 SS, Alloy 52M, and Alloy 52MSS are shown in Figs. 15–17, correspondingly. All cracks were intergranular and often oc-

curred along straight, migrated grain boundaries with one or more crack tips located at triple points. Some cracks were found in the process of formation and gave the grain boundary a perforated appearance as though many voids formed along the boundary and would eventually connect to form DDC. An example of this is shown in Fig. 18.

Discussion

Main Points

In general, Table 6 and Equations 1–4 show the number of thermomechanical cycles significantly impacts the imposed mechanical energy and preloading/straining does not. Throughout testing, several important aspects of the procedure were deliberately altered to improve the effectiveness of SSR, such as elevated temperature straining, spot welding where needed, elimination of preloading, and reduction of the gauge cross section. Here, “effectiveness” is the ability for SSR to recreate severe welding conditions to the extent of causing DDC in these materials. It would be of little value if the procedure were unable to do so. Despite the changes during testing and minor differences in material properties, the number of thermomechanical cycles has a similar trendline coefficient, about 1.5–2.1 MJ per cycle, across the four materials in Table 6 and Equations 1–4. The R^2 values in excess of 94%, the imposed energy equations (Equations 1, 2, 3, and 4), and the consistency of number of thermomechanical cycles coefficients indicate strong reproducibility. However, larger variations were found between Samples 3 and 7 as well as Samples 4 and 8 of each material, which were tested at equal conditions as shown in Table 6. This is likely because longer tests (i.e., tests with ten cycles instead of one) have greater opportunity for deformation and hence changes in stress-strain behavior over the course of the test. Figure 11C is an example exhibiting this change over time.

Assessment of SSR Testing

Referring to the SSR output graphs shown in Fig. 11

Fig. 18 — Alloy 52M DDC along the “perforated” grain boundary. Taken from a complete separation failure sample.

(310 SS Sample 4 with ten cycles and 2.7% strain per cycle), these results can be informative about sample behavior during testing in many ways. First, on the stress and temperature vs. time graph (A), initial hardening was evident after the first few cycles, then the sample began to weaken toward the end as peak stress dropped. Due to the fixed displacement, as the temperature rose, stress tended to drop or even become compressive as the sample thermally expanded and was subsequently resisted by the jaws. The opposite occurred on cooling. The test sample was subjected to fatigue loading with dominating tensile cycles since the sample spent a relatively longer time cooling and contracting than heating and expanding. On the strain and temperature vs. time graph (B), strain accumulated each cycle up to a maximum of about 40%. It must be noted this was a measurement of total strain on the sample, not just the strain added during heating, and hence there was an initial drop each cycle due to thermal expansion.

Looking at stress and strain vs. time graph (C) now, it becomes apparent strain begins to accumulate more slowly as the test progresses and peak stress begins to drop. The stress and strain vs. temperature graph (D) is more difficult to interpret. The test cycle begins at the bottom left of the graph where stress was near zero. On the first heating cycle, the stress grew to about 60 MPa in compression, increased to 70 MPa in tension while the extrinsic strain was applied in the DDC temperature range, and then decreased to about 25 MPa during the subsequent heating to 1200°C. On cooling to room temperature, the stress gradually increased to about 150 MPa. In the subsequent SSR cycles, the stress applied to the test sample remained tensile, varying between about 20 and 220 MPa, and followed similar trends of decreasing on heating, except during extrinsic straining, and increasing on cooling. The strain growth accumulated steadily, with local drops due to thermal expansion within the heating cycles, then gradually increased to about 40% at the final cycle.

The stress vs. strain graph (E) shows what would eventually be used to calculate imposed mechanical energy. The graph begins at zero strain and progresses briefly to the left as compressive strain was experienced during the very first cycle, then to the right as the sample was strained in a combination

Fig. 19 — Nickel 201 Sample 4 stress and temperature vs. time plot showing effect of preload on stress over many cycles.

of on-heating extrinsically applied strain and on-cooling intrinsic strain ratcheting.

Finally, the stroke vs. time graph (F) shows the displacement of the Gleeble jaws as the test is underway. The sample was held fixed during most of the heating and all of cooling, and the steps corresponded to the extrinsic strain applied on heating. The information shown on these graphs for each material is helpful in monitoring the sample's behavior during testing and will aid in future work aimed at determining how these behaviors are related to DDC. In addition, the graphs help to visualize how the weld metal service behavior is artificially recreated within the SSR test.

It was initially difficult to produce DDC in samples that were primarily composed of base metal with welds partially penetrating the gauge section. Weld metal is more susceptible to DDC due to the presence of migrated grain boundaries, so if strain does not accumulate in it, there will be no DDC (Ref. 2). After the removal of preload and replacement with straining, DDC formed more readily and consistently, so future work will continue to include SSR to artificially produce DDC where needed.

DOE

The consistent high significance of number of thermomechanical cycles shown in Table 6 and Equations 1–4 across all materials indicates it is the factor that most strongly affects imposed mechanical energy. This is indicated by the coefficients for number of cycles having the largest numerical magnitude in the equations and the P-values, which are all less than 0.05 in Table 6. This is an unsurprising result, though it is interesting how consistent the numerical effect of cycling is across multiple materials and loading conditions. Perhaps this is due to similar thermomechanical properties among the materials involved.

Preload was initially added to act as an artificial restraint, but graphs of the data show the preload usually only affects the first one or two cycles and each subsequent cycle is more like nonpreloaded behavior. Figure 19 is an example graph showing this behavior taken from a high-cycle, high-preload Nickel 201 sample. Preload then turned out to be statistical-

ly insignificant after running the DOE calculations, so it was eliminated. This was determined by examining the coefficients for preload in Equations 1–4, which are small compared to number of cycles, and by examining the P-values for preload in Table 6, which are all in excess of 0.05.

With the P-value larger than 0.05, the extrinsically applied strain within the DDC temperature range does not show up as a statistically significant factor. However, its combined effect with number of thermomechanical cycles is significant and its coefficients in the imposed energy equations are comparative with those of the number of thermomechanical cycles. The DOE results in Table 6 show that DDC strain, used to simulate strain ratcheting in highly restrained multipass welds, greatly increases the likelihood of DDC forming during testing. The mechanical energy imposed within the DDC temperature range accounts for both the applied extrinsic strain on heating and the intrinsic strain accumulated as a result of fixed displacement on cooling. Samples that contained more DDC did experience higher imposed mechanical energy, thus there is a correlation between cracking probability and imposed mechanical energy. As a result, the imposed mechanical energy can be used to quantify the thermomechanical effect of the SSR procedure on the DDC response in tested materials.

The DOE results in Table 6 could not be used to rank DDC susceptibility due to variation in procedure and sample preparation. However, some of the qualitative results combined with the DOE do reflect trends observed in past research. The established ranking of susceptibility from lowest to highest is Alloy 52MSS < 310 SS < Alloy 52M < Nickel 201 (Refs. 11, 21). 310 SS, despite experiencing greater total strain in Samples 4 and 8, showed less cracking and more signs of ductility, such as necking and surface deformation, than similar samples of Alloy 52M. Nickel 201 and Alloy 52MSS had somewhat anomalous behavior here because no DDC was observed in Nickel 201, and Alloy 52MSS experienced complete separation failures on the final cycle of Samples 4 and 8. However, the Nickel 201 DOE was conducted without any DDC strain, and Alloy 52MSS had the greatest difference in sample geometry compared to the others, so making a comparison with the rest of the materials may be inappropriate.

Procedure Refinement

During the Nickel 201 DOE, it became clear changes would need to be made to the procedure/sample preparation to produce DDC. No cracking was observed in Nickel 201 even though past research suggests it was the most susceptible material tested in the current study (Ref. 21). This is likely due to the gauge section containing base metal, which was able to bear more of the plastic deformation where the weld metal was unable to do so. McCracken and Tatman's computational model was closely examined to find other factors that may impact DDC susceptibility (Ref. 16). It predicted elevated-temperature strain accumulation, so this was added to the SSR procedure. Furthermore, past research on STF involved preparing samples with gauge sections composed entirely of groove welds with surface spot welds on both sides of the sample to produce favorable grain orientations for cracking (Ref. 11). Likewise, spot welds were

added to both sides of all 310 SS samples to melt through any unmelted base metal, since they were not entirely composed of weld metal prior to this change. Figure 15 shows mainly cracking from overloading in 310 SS after testing with some small DDC cracks. The absence of abundant secondary DDC cracking besides the final failure indicates a low susceptibility to DDC. The increase in gauge section length, as the sample is strained, is accommodated by plastic deformation without significant grain boundary sliding, leading to DDC.

In contrast, the Alloy 52M showed extensive secondary cracking after a high number of thermomechanical cycles, as shown in Figs. 16 and 18. This is because the samples were composed entirely of weld metal. There was no irregularity in microstructure in the gauge section (i.e., no transition from base metal to weld metal), and it is suspected this factor was the most impactful in causing a distinct absence of DDC in Nickel 201 in addition to the lack of elevated-temperature straining. This base/weld metal transition can be problematic in mechanical testing because the base metal and weld metal have different strength and ductility, which means differing behavior during testing (Ref. 3).

After making these significant changes to the procedure and observing the DDC generated in this testing, it is clear this will be a viable method for studying the DDC cracking mechanism in future work. The DDC observed in Alloy 52M along with the fracture surfaces from Alloy 52M, Alloy 52MSS, and 310 SS all match the expected morphology and size scale of cracking, which has been observed in past research from both the authors and other researchers (Refs. 1, 18, 22). Also, the recorded thermomechanical behavior from testing closely corresponds to a past study that modeled thermomechanical behavior in a multipass weld (Ref. 20). These past studies represent both laboratory testing, such as STF and large-scale multipass welds. The strengths of performing a mechanistic investigation with this method are that it closely mimics actual production conditions while providing a carefully controlled environment for cracking to nucleate and propagate without the influence of confounding variables, which may be encountered in a large-scale testing, such as oxygen/hydrogen contamination and fusion-related defects.

Cracking Morphology and Fractography

The secondary cracking observed in the 310 SS (Fig. 15), Alloy 52M (Figs. 16 and 18), and Alloy 52MSS (Fig. 17) matches the expected DDC size, shape, and location as documented in past research (Refs. 3, 6, 18, 22). The thermal faceting observed on the DDC fracture surface of 310 SS (Fig. 12), Alloy 52M (Fig. 13), and Alloy 52MSS (Fig. 14) has been a consistent finding in these alloys in past research from the authors and others (Refs. 1, 22). Thus, SSR recreates DDC in terms of morphology and location in the microstructure. Also, this is not the first time thermal faceting has been observed on DDC fracture surfaces, though it is perhaps the first time it has been mentioned by name alongside DDC in decades of published literature (Refs. 1, 22). Collins et al. observed thermal faceting on artificially generated DDC fracture surfaces, and Hemsworth et al. observed it on a fracture surface extracted from a multipass weld (Refs. 1, 22). Very few researchers ob-

serve thermal faceting to begin with, likely because it is difficult to image due to its small size and is likewise easily obscured by thin oxide layers. Furthermore, sometimes it is found and referred to as slip lines or plastic deformation artifacts in error (Refs. 11, 22). At present, it is not yet clear how thermal faceting influences DDC, if at all, but future work will consider and investigate the potential implications it may have.

Conclusion

Optimized Procedure

The SSR procedure diagram shown in Fig. 9 remains accurate with the following stipulations:

- 1) The gauge section contained in the free span of the SSR samples should be made entirely of weld metal for microstructural consistency;
- 2) In the event it is somehow impossible to meet the previous requirement, spot welds should be added to the front and back of the sample;
- 3) No preloading is necessary; and
- 4) Gauge section should be of uniform geometry (i.e., no notches, reduced thickness, etc.) for even stress and strain distribution.

The number of cycles used in testing can be varied at will based on the desired level of welding restraint and thermal cycles. Elevated-temperature strain ratcheting should be added in the DDC temperature range to encourage crack formation. Based on results shown here, cracking appears near 90% of the complete separation strain. The outputs of the test are quantitative in the form of data acquired during the test, such as dilatometer, jaw displacement, stress, and temperature. These outputs can be processed and analyzed to calculate the imposed mechanical energy on the sample, which has been correlated with DDC occurrence here. The qualitative outputs are metallographic imaging and extent of cracking.

Summary

The SSR procedure has been adequately optimized to reliably produce DDC in a variety of materials and sample geometries with several different levels of susceptibility. Multiple thermomechanical cycles have a greater effect on imposed mechanical energy on samples than preloading. Overall strain accumulation is required for DDC to form. A significant interaction was found between the strain ratcheting, simulated through straining in the DDC temperature range, and the number of thermomechanical cycles. The mechanical energy imposed within the DDC temperature range can be used for quantification of the effect of thermomechanical cycling on the DDC response in tested materials. SSR will be used in future research to study the effects of impurities and grain boundary evolution during simulated welding thermal cycles.

The cracking generated by the SSR procedure has both similar fracture surface topography and cross-sectional cracking morphology as previous documented cases of DDC, both from laboratory tests and service failures. SSR can be used to simulate welding conditions, in particular multipass welding conditions, which often lead to DDC in high-chromium nickel-based

alloys. This represents an improvement over past laboratory tests because the thermomechanical conditions are more similar to production conditions without sacrificing the control over variables introduced by multipass weld tests, such as contamination and weld defects.

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References

1. Hemsworth, B., Boniszewski, T., and Eaton, N. 1969. Classification and definition of high temperature welding cracks in alloys. *Metal Construction & British Welding Journal* 1(25): 5–16.
2. DuPont, J., Lippold, J., and Kiser, S. 2013. *Welding Metallurgy and Weldability of Nickel-Base Alloys*. Hoboken, N.J.: John Wiley and Sons Inc.
3. Lippold, J. 2015. *Welding Metallurgy and Weldability*. Hoboken, N.J.: John Wiley and Sons Inc.
4. Nissley, N., and Lippold, J. 2009. Ductility-dip cracking susceptibility of nickel-based weld metals: Part 2 — Microstructural characterization. *Welding Journal* 88(6): 131-s to 140-s.
5. Noecker, F., and DuPont, J. 2009. Metallurgical investigation into ductility dip cracking in Ni-based alloys: Part II. *Welding Journal* 88(3): 62-s to 77-s.
6. Ramirez, A., and Lippold, J. 2004. High temperature behavior of Ni-base weld metal: Part II — Insight into the mechanism for ductility dip cracking. *Materials Science and Engineering A* 380(1–2): 245. DOI: 10.1016/j.msea.2004.03.075
7. Fink, C. 2016. An investigation on ductility-dip cracking in the base metal heat-affected zone of wrought nickel base alloys — Part I: Metallurgical effects and cracking mechanism. *Welding in the World* 60: 939–950. DOI: 10.1007/s40194-016-0370-4
8. Chen, H., Kalia, R., Kaxiras, E., Lu, G., Nakano, A., and Nomura, K. 2010. Embrittlement of metal by solute segregation-induced amorphization. *Physical Review Letters* 104(15): 1–4. DOI: 10.1103/PhysRevLett.104.155502
9. Nishimoto, K., Saida, K., Okauchi, H., and Ohta, K. 2006. Microcracking in multipass weld metal of Alloy 690 — Part 2: Microcracking mechanism in reheated weld metal. *Science and Technology of Welding and Joining* 11(4): 462–470. DOI: 10.1179/174329306X94309
10. Saida, K. et al. 2012. Influences of phosphorus and sulphur on ductility dip cracking susceptibility in multipass weld metal of Alloy 690. *Science and Technology of Welding and Joining* 17(1): 1–8. DOI: 10.1179/1362171810Y.0000000004
11. Nissley, N., Collins, M., Guaytima, G., and Lippold, J. 2002. Development of the strain-to-fracture test for evaluating ductility-dip cracking in austenitic stainless steels and Ni-base alloys. *Welding in the World* 46(7–8): 32–40. DOI: 10.1007/BF03263388

12. Fink, C., Zinke, M., and Keil, D. 2012. Evaluation of hot cracking susceptibility of nickel-based alloys by the PVR test. *Welding in the World* 56(7–8): 37–43. DOI: 10.1007/BF03321363

13. Noecker, F., and DuPont, J. 2009. Metallurgical investigation into ductility dip cracking in Ni-based alloys: Part I. *Welding Journal* 88(1): 7-s to 20-s.

14. Kadoi, K., Hiraoka, M., Shinozaki, K., and Obana, T. 2019. Ductility-dip cracking susceptibility in dissimilar weld metals of Alloy 690 filler metal and low alloy steel. *Materials Science & Engineering A* 756: 92–97. DOI: 10.1016/j.msea.2019.04.035

15. Lundin, C., DeLong, W., and Spond, D. 1976. The fissure bend test. *Welding Journal* 55(6): 145-s to 151-s.

16. McCracken, S., and Tatman, J. 2016. Prediction of ductility-dip cracking in narrow groove welds using computer simulation of strain accumulation. *Cracking Phenomena in Welds IV*: 119–141.

17. Straumal, B., Kogtenkova, O., Gornakova, A., Sursaeve, V., and Baretzky, B. 2016. Review: Grain boundary faceting-roughening phenomena. *Journal of Materials Science* 51(1): 382–404. DOI: 10.1007/s10853-015-9341-1

18. Nissley, N. 2006. Intermediate temperature grain boundary embrittlement in nickel-base weld metals. PhD diss., The Ohio

State University, Columbus, Ohio.

19. Yushchenko, K., Savchenko, V., Chervyakov, N., Zvyagintseva, A., and Guyot, E. 2011. Comparative hot cracking evaluation of welded joints of Alloy 690 using filler metals Inconel® 52 and 52 MSS. *Welding in the World* 55(9–10): 28–35. DOI: 10.1007/BF03321317

20. McCracken, S., and Tatman, J. 2016. Comparison of ductility-dip cracking to computer modeling with Sysweld™ in a narrow groove multi-pass weld. *Pressure Vessels and Piping Conference*. Vancouver, Canada.

21. Kreuter, V., and Lippold, J. Ductility-dip cracking susceptibility of commercially pure Ni and Ni-base alloys utilizing the strain-to-fracture test. *Cracking Phenomena in Welds IV*: 145–159.

22. Collins, M. 2002. An investigation of ductility dip cracking in nickel-base filler materials. PhD diss., The Ohio State University, Columbus, Ohio.

SAMUEL LUTHER (luther.53@buckeyemail.osu.edu) is a graduate fellow and **BOIAN ALEXANDROV** (alexandrov.l@osu.edu) is a research professor at The Ohio State University, Columbus, Ohio.

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