Auto/Steel Partnership

Laboratory Procedure

ASP LP#3.1 (06-2023)

Rapid Liquid Metal Embrittlement Test Procedure for Coated Sheet Steel

Version: 1.0

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Previous Editions:

- None

Changes:

- 1st Version

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Disclaimer

This document is a suggested procedure for anyone to use at their own risk. Anyone using this procedure must following all prevailing safety procedures. A/SP and parties involved in creating the procedure, do not any responsibility or liability for its use. A/SP requests anyone encountering an issue with this procedure to contact A/SP immediately so that the issue can be corrected.

1.0 Introduction

1.1 Purpose

The purpose of this procedure is to provide a quick qualitative assessment of the susceptibility of coated sheet steel to liquid metal embrittlement during joining. The procedure is specifically designed for the following purposes:

- To induce LME crack formation in coated steels
- To predict which of several types of LME cracks a given coated steel might exhibit when welded in a manufacturing environment
- To allow comparison of LME susceptibility of various grades and/or lots of steel
- To protect automakers against LME cracking for worst-case manufacturing conditions
- To produce cracked spot welds for mechanical testing

<u>This test is not intended nor suitable to develop production welding parameters.</u> Its sole purpose is for suppliers, users and researchers to evaluate the relative LME susceptibility of various steel grades and coatings.

1.2 Scope

When certain metallic coatings (such as zinc) come in contact with a steel substrate held under sufficient tension at elevated temperature, the molten metal coating may penetrate into the solid steel substrate and embrittle the steel grain boundaries. The embrittlement can lead to cracking, known as Liquid Metal Embrittlement (LME).

A/SP has applied high temperature (Gleeble) tensile testing to characterize the LME behavior in coated steels using a procedure co-developed with the Ohio State University (*ASP LP#2.1 (06-2023): Liquid Metal Embrittlement - Gleeble-Based Testing Procedure*). Gleeble testing is preferred for a quantitative assessment of LME susceptibility but tends to be expensive and time consuming. As an alternative, A/SP has developed the Rapid LME test procedure using resistance spot welding, which can provide a qualitative means of assessing LME susceptibility at lower cost and reduced time.

In this test, a series of welds are made at progressively greater power input to push a material to its characteristic LME activation point. Below the expulsion current, the location of an LME crack indicates the relative cracking temperature that Gleeble testing would have quantified. Unfortunately, above the expulsion point the mode of LME cracking becomes less predictable, due to inherent instability in momentary weld force and input power associated with RSW expulsion. Consequently, the crack repeatability is better for welds made below the expulsion point.

This test was configured to be used on 1.1 and 1.5 ± 0.1 mm steel sheet thickness. The parameters for 1.5 +/- 0.1mm material were developed using batches of various automotive grades of galvanized steels for which Gleeble LME data were available. The weld parameters were adjusted until the Rapid LME Test results correlated well with Gleeble results.

1.3 Referenced Documents

- ASP LP#2.1: LP#2.1 (06-2023): Liquid Metal Embrittlement - Gleeble-Based Testing Procedure.

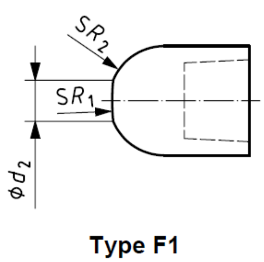
2.0 Test Plan

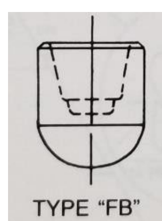
2.1 Testing Equipment

The following equipment is required to conduct the test:

1. Mid frequency direct current (MFDC) Resistance Spot Welding machine capable of producing the required weld force and the special pulsed weld current profiles (pulsed upslope). The WTC brand controller was used to develop and calibrate this procedure.
2. Electrode caps (tips) with a 6R40 dressed face, (face may be dressed *in-situ* or purchased with this common global industry weld face from suppliers).

   Either ISO Type F1 or RWMA Ball nose may be used:





ISO 5821 Standard RWMA Standard

Either of these standard electrode caps is acceptable, provided they are:

- 16mm O.D.,
- radius 8 dome (SR2 = 8mm), and
- 6R40 dressed weld face (d2 = 6mm, SR1 = 40mm)
- Material may be C15000 or C18150 copper
- Copper hardness shall be Rb 72 minimum

3. Weld current analyzer with millisecond graphing capability.

   Simultaneous plots of Current, Voltage, and Power are required for each weld.

4. Chemicals and laboratory equipment for safely stripping zinc coating from welded coupons.

   Note: The stripping chemicals are typically concentrated acids and shall only be handled carefully by qualified persons under a chemical fume hood, with emergency shower and eyewash stations standing by. Users shall consult the Material Safety Data Sheets (MSDS) for all chemicals prior to handling.

5. Wet saw for cutting weld samples. The purpose of cutting is to produce metallographic samples for evaluation.

6. Typical metallographic laboratory equipment for sample preparation and evaluation of weld cross sections for microstructural examination. This includes, but not limited to grinding and polishing equipment.

7. Etchants (including Picral, Super Picral, Nital-Picral or any suitable etchant that can reveal the underlying microstructural details in welded steel samples).

   Note: Etchants are chemicals that shall be handled carefully. Etching shall be done under a fume hood that can safely vent acid fumes. Users shall consult the Material Safety Data Sheets (MSDS) for all chemicals prior to handling.

8. An optical macroscope capable of zoom magnification in the range 3x-25x.

   An optical metallograph microscope with a capability of 25X and higher. Metallograph shall be equipped with provisions to measure crack depths. Photographic capability to record both macro and micro images.

2.2 Material Requirements

Approximately three-square meters of flat production-intent steel sheet is required for this test. Coupons for weld sample preparation shall be sheared to the approximate dimensions 37 mm x 125 mm, (1½ inch x 5 inch).

3.0 Cracking Test Welding Parameters (Schedule) and Set-up

3.1 Electrode Alignment

The upper and lower electrodes shall be aligned. Prior to start of testing, the alignment of the electrode tips (caps) must be verified by making carbon imprints, or by other quantitative means. Electrode alignment is considered acceptable when the carbon imprint from both the upper and lower tips are circular in shape, uniform in color and the diameter meets the specified pre-dressed value (the default face diameter is 6 mm).

3.2 Water Flow Rate

A water flow rate of 3.8 liters per minute (1 gallon per minute) per electrode tip is required for this procedure.

3.3 Welding Test Parameters

3.3.1 For sheet thickness 1.5mm +/- 0.1

Squeeze time:	**1000 milliseconds (ms)**
Impulse timing:	130 ms Heat, plus 40 ms Cool
Slope Profile:	8 ***Progressive Sloping Impulses*** (such that the first impulse begins at 5ka and the last impulse ends at a designated final amperage).
Hold time:	250 ms
Electrode Force:	4.0 kN

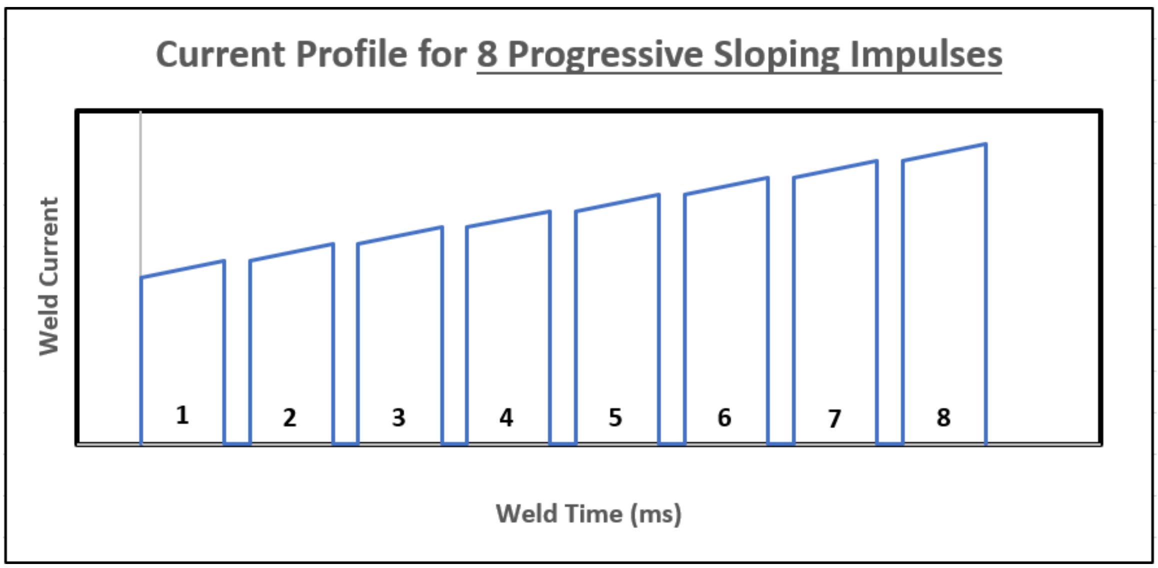


Figure 1. The required profile of weld current for this procedure.

3.3.2 For sheet thickness 1.1mm +/- 0.1

Squeeze time:	**1000 milliseconds (ms)**
Impulse timing:	67 ms Heat, plus 10 ms Cool
Slope Profile:	11 ***Progressive Sloping Impulses*** (such that the first impulse begins at 4ka and the last impulse ends at a designated final amperage).
Hold time:	250 ms
Electrode Force:	2.6 kN

4.0 Test Procedure

4.1 Methodology Overview

Spot welds are made at progressively higher weld currents under specified conditions to determine the current level at which various types of LME cracks are generated. The order of welding is specified. Test welds are made and evaluated for location and types of cracks that may have occurred.

Once equipment has been properly set up and verified, welding may begin. An oscilloscope trace of each weld is recorded by photography or other digital means. Recordings shall include at a minimum, dynamic traces of secondary weld current and voltage. Dynamic resistance and power traces are also helpful but not required.

After welding is complete, both visual and metallographic evaluations are performed to determine if cracking has taken place:

1. The weld surface is macrophotographed in the as-welded condition. Any visible surface cracks must be captured on photographs. Both top and bottom surfaces of all welds must be examined for possible cracks.
2. Zinc coating is then removed from the surface of the weld by pickling with muriatic acid. The weld surfaces are again macrophotographed as in step 1 for a side by side comparison. Often, LME cracks are easier to notice after the zinc coating is removed (post-weld).
3. Next, the welds are marked for metallographic sectioning to cross-section the plane of the greatest amount of visible LME cracking. The chosen cross-section plane should include the axial center of the spot weld whenever possible.
4. After sectioning the samples in the marked orientation, typical metallographic preparation is performed. The default etchant used is Picral. Other suitable etchants, such as nital or nital- picral mixture may be used with certain steel alloy grades to better reveal microstructure and LME cracks. Samples are etched sufficiently to reveal the underlying microstructure of weld fusion zone (FZ) and heat affected zone (HAZ).
5. Each cross-section is photographed at roughly 25X magnification. The location and relative depth of all cracks is marked and measured. Record crack penetration depth normal to the surface, as a percentage of the sheet thickness (or percentage of the weld thickness, depending on the location of the crack). Use higher magnification if needed, but always capture a standard low magnification photo for the final report.

Report write-up:

1. In the test report, one page per weld is used to document the following photographs:
    a. Oscilloscope trace
    b. Macro view of the most cracked side of the weld – As-welded
    c. Macro view of the most cracked side of the weld – After pickling away the coating.
    d. 25x micrographic view of cross-section, with cracks marked and measured
2. For the final report, document the progression of welds by arranging the photos onto one (or two) page per weld in a standard format. Then arrange the pages according to the order of welding, beginning with the lowest amperage.
3. Use a standard report format to facilitate review and comparison of overall results.
4. When possible, draw conclusions relative to the LME susceptibility of the subject material.

Guidelines for test interpretation are included at the end of this procedure.

4.2 Welding Sequence on Each Coupon:

The welds are made in a predetermined order on the coupons (so that each coupon yields two test welds):

1. A Shunt Weld is made at the center of the weld coupon stackup.
2. The First Test Weld (spaced 30 mm away from the shunt weld).
3. The Second Test Weld (spaced 30 mm away from the shunt weld in the opposite direction of the first test weld). A test variant for dissimilar stackups: the second test weld may be of opposite polarity if specified.

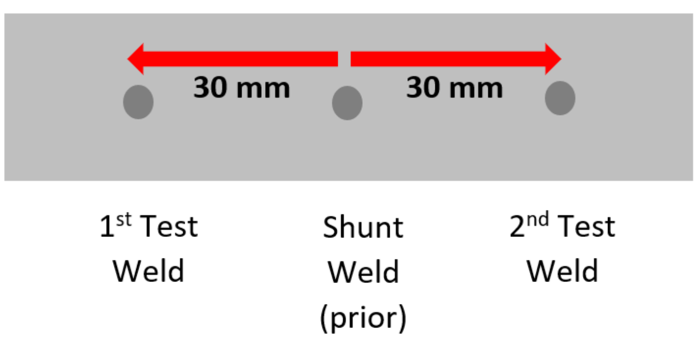


Figure 2. Location of shunt and test welds on test coupons

4.3 Programming the Weld Controller:

The weld schedule is programmed using the Progressive Sloping Impulse method as follows.

For 1.5mm material, the WTC weld schedule sequence for a Finishing Current of 10.5 ka:

SQUEEZE 1000	Ms	
IMPULSE	130 heat ms, 40 cool ms	(function 60)
SLOPE	8 IMP 5000 A TO 10500 A	(function 45)
HOLD	250 ms	

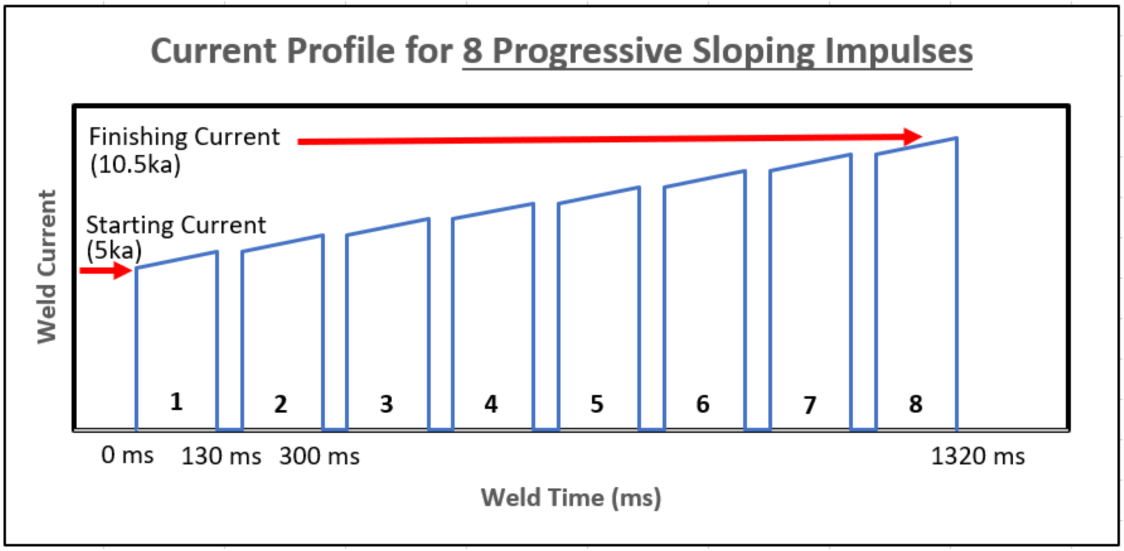


Figure 3. Expected current profile resulting from the above P.S.I. program

As written, the above program begins the first pulse at 5ka and ends the last pulse at 10.5ka.

For the purposes of this procedure the **test current refers to the finishing current**, (therefore in this example the **test current** is recorded as 10.5ka).

- For 1.5mm material the starting current is never changed from 5.0 ka.
- For 1.1mm material the starting current is never changed from 4.0 ka.

4.4 Conditioning Welds:

After the electrode tips are pre-dressed and aligned, make 8-10 conditioning spot welds, one each on different weld coupon sets using the weld schedule described in Section 4.3 with a Test Current of 7 ka. These conditioning welds help to reduce the scatter of test data and to condition the surface of the electrode tips for the high currents to be used. Test welds may be placed on different coupons and used as shunt welds as defined above.

Note: To make tip conditioning welds use a test current of 7.0 kAmp. Peel at least one shunt weld to verify at least a 3 mm weld is formed. (Adjust test current if needed).

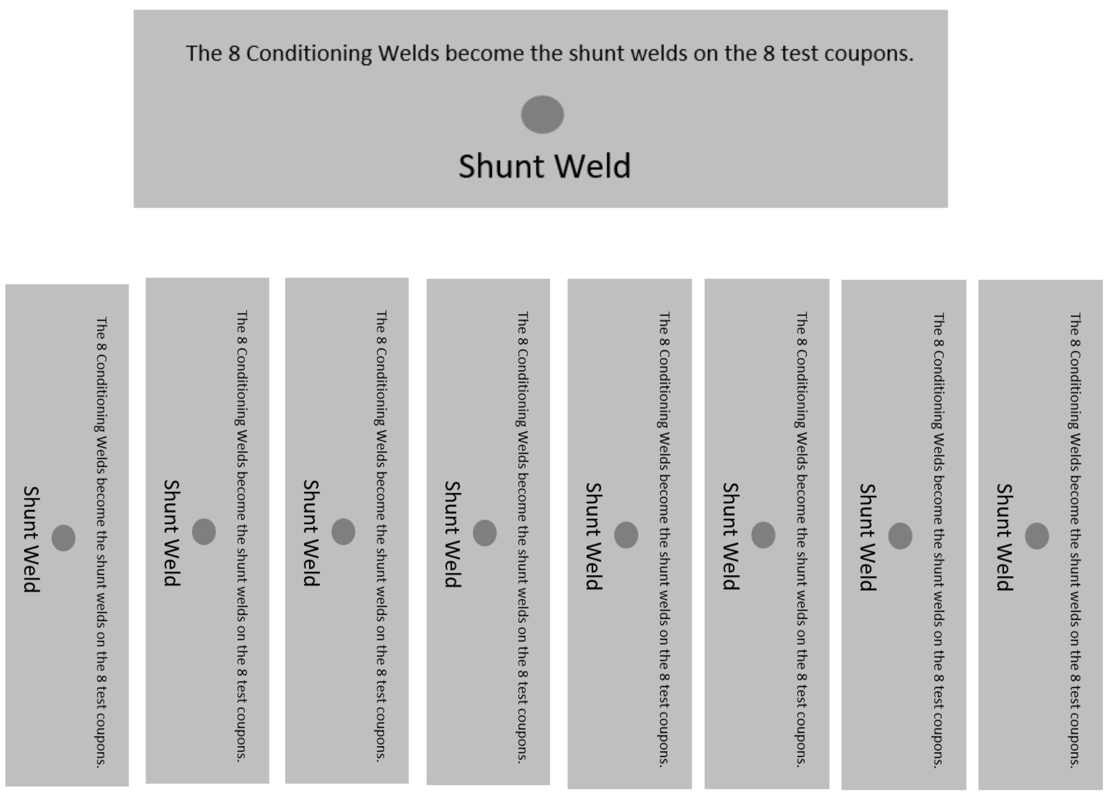


Figure 4. The conditioning welds later serve as shunt welds on each test coupon.

4.5 The Progression of Test Welds:

Coupon Set	Weld Number	Test Current,
1	1	7.
	2	7.
2	3	8.
	4	8.
3	5	9.
	6	9.
4	7	10.
	8	10.
5	9	11.
	1	11.
6	1	12.
	1	12.
7	1	13.
	1	13.
8	1	14.
	1	**14.**

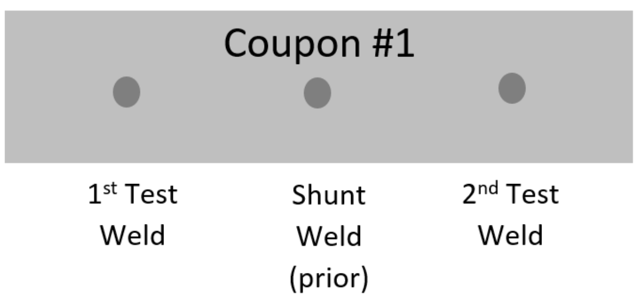


Figure 5. Repeat the pattern of Coupon #1 through all 8 Coupons.

Carefully label the welding current (in kAmps) of all test weld samples. Mark with an "X" or "EXP" any test weld that exhibits expulsion. For each weld, expulsion is to be monitored by two methods. Either event confirms expulsion:

- Visual observation of flash during welding
- Electrical observation of an abrupt drop in power signal on the oscilloscope

Carefully and descriptively label each coupon and weld in a sequence that denotes the progression of welding (such as welding current at which each weld was made, and whether expulsion occurred). Make all 16 test welds regardless of how hot the last welds may seem. This is for full LME characterization below and above the expulsion point.

5.0 Post Weld Evaluation

5.1 Surface Macrophotos

After welding has been completed, photograph both sides (top and bottom) of each weld at approximately 3X to 5X magnification to document any visible cracks and post-weld surface conditions of the zinc coating and the area surrounding the weld.

5.2 Coating Removal

Next, strip the zinc coating by pickling samples with muriatic acid or inhibited hydrochloric acid. Rinse and clean samples. After the coating is stripped from each sample, re-photograph all welds and record any additional cracks revealed after zinc removal.

5.3 Cutting and Mounting of Samples

The next evaluation process is a cross section of the welds focusing on capturing crack depth and location. Under the stereoscope identify any cracks and draw an intersecting perpendicular line to the crack so when followed with a cut the crack depth will be revealed. Make sure all samples are deburred and that the side of interest showing the weld is flat and burr-free and doesn't have any rounded edges before mounting into the press. Carefully cut all samples down to size so they will fit into the mounting press but still show the best representation of any cracks that have occurred. Mount the samples into metallography pucks and prepare for polishing.

5.4 Polishing, Etching and Metallographic Review

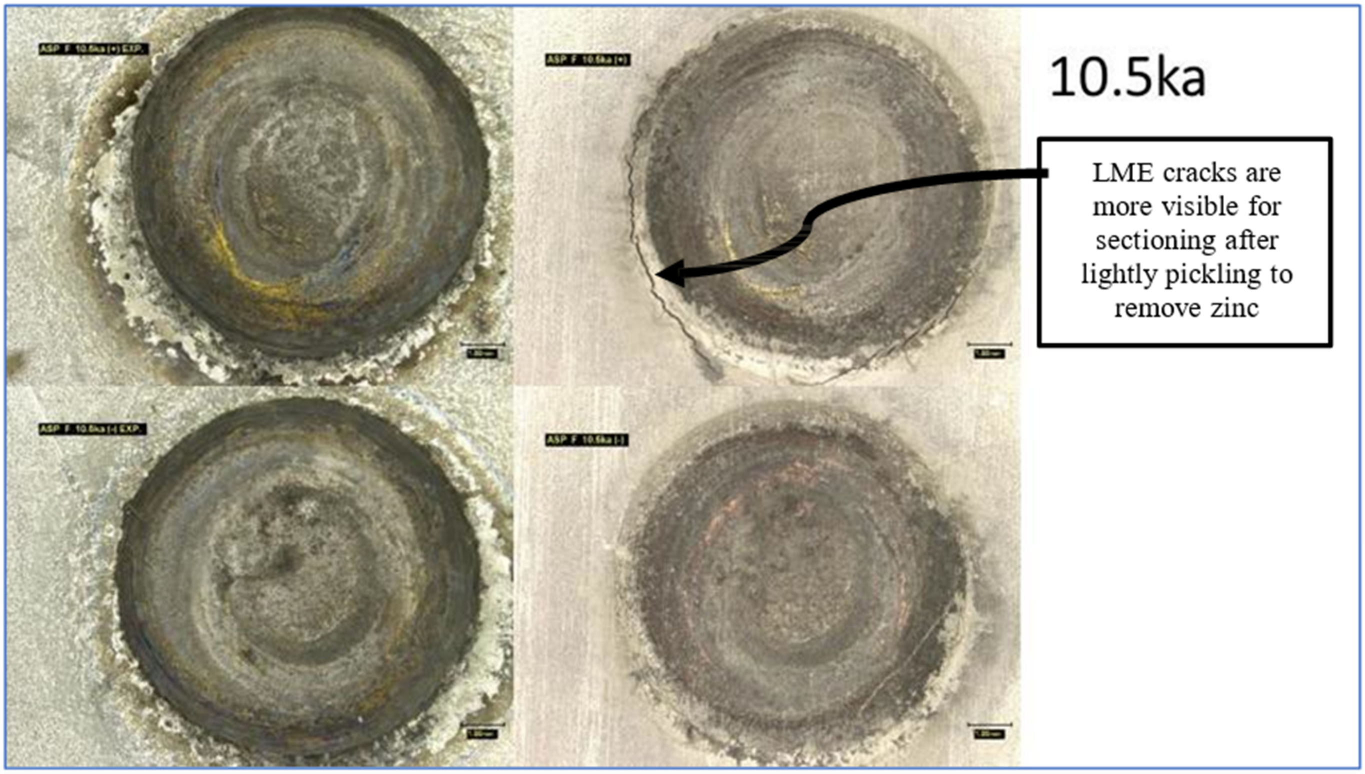
Polish each sample so that microstructures will be identifiable after chemical etching. Etch the metallographic sample to expose the nugget formation, general microstructure and heat affected zone. Evaluate the samples under a microscope and take standard photo views for documentation. Measure the crack depth into the substrate and circle the location of each crack on the photo for ease of identification during subsequent review.

5.5 Report/Final Determination of Material

Photos and documentation may be compiled into a report devoting two pages per weld, showing:

- Oscilloscope waveform
- As-welded condition (worst side only),
- Stripped condition (worst side only),
- Cross-section

Interpretation of results must be done by persons qualified to do so.



AS WELDED (both sides) AFTER PICKLING (both sides)

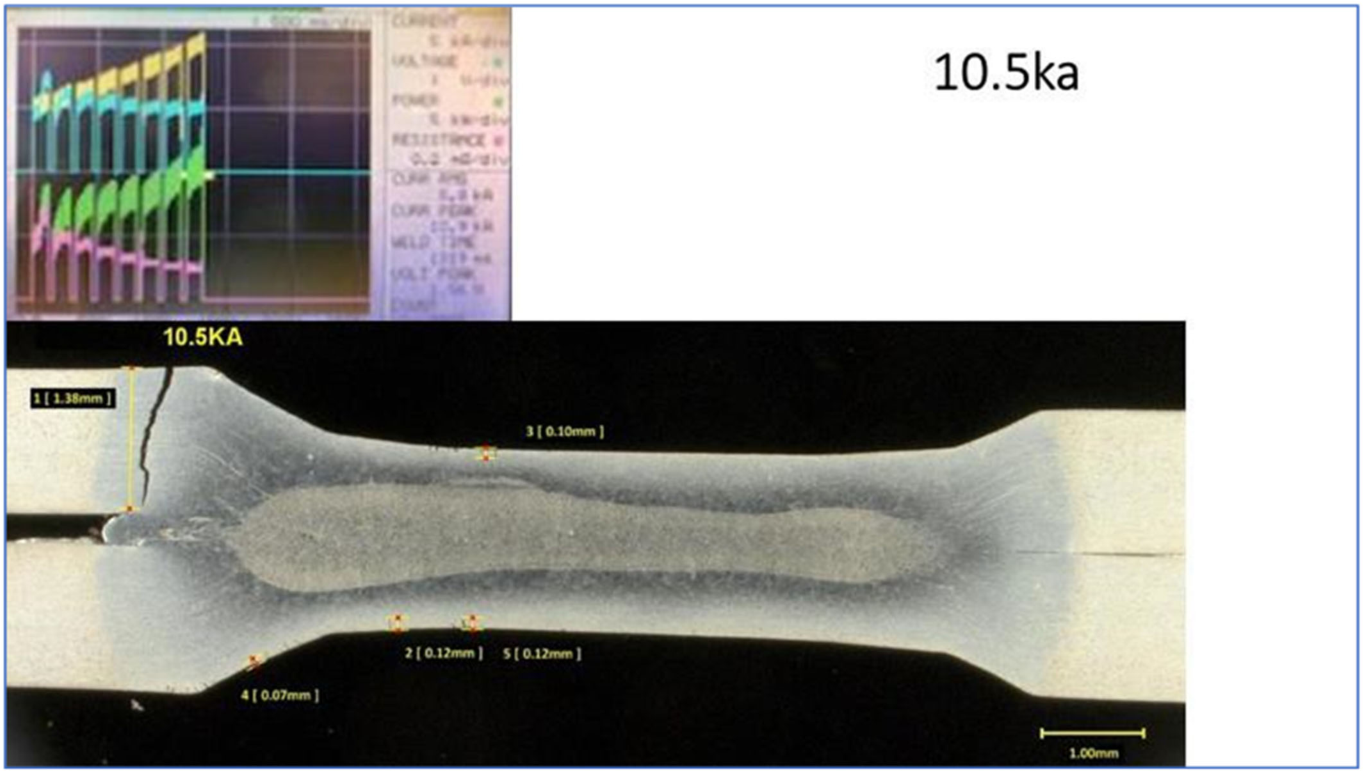


Figure 6. Sample two-page format showing typical data resulting from one test weld.

6.0 Interpretation and acceptability of LME cracks:

6.1 TYPE A - High Temperature LME (LME above 650C)

LME cracks directly under the electrode footprint are understood to represent LME in the temperature range >650° C. It occurs with most galvanized steel grades when welding significantly above the expulsion point. High Temperature LME an acceptable result during this test if it only occurs during severe over-welding. Below expulsion these cracks are only acceptable if crack depth is less than 0.10 mm (100 micrometers).

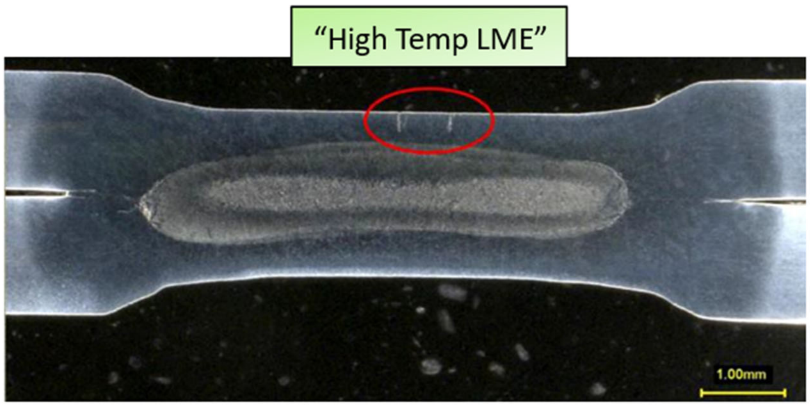


Figure 7: A weld cross-section etched to reveal examples LME cracks, judged to be High Temperature LME due to their location, touching the electrode face.

6.2 TYPE B - Low Temperature LME (LME below 650C)

LME cracks outside the electrode indentation are believed to represent LME in the temperature range <650° C. This type of LME is not considered an acceptable result for this test.

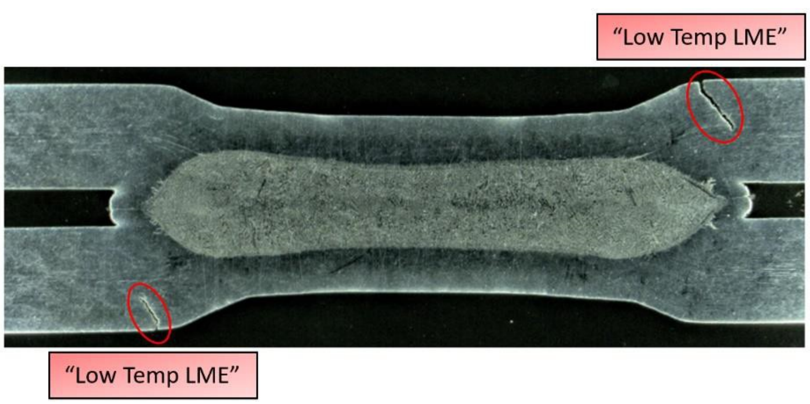


Figure 8. Weld cross-section showing examples of LME cracks, judged to be Low Temperature type due to their location just outside of the electrode imprint area.

6.3 TYPE D - Threshold Temperature LME (LME near 650C)

LME cracks in the outer transition of the electrode imprint are believed to represent LME at the threshold temperature (~650C) between the two LME modes.

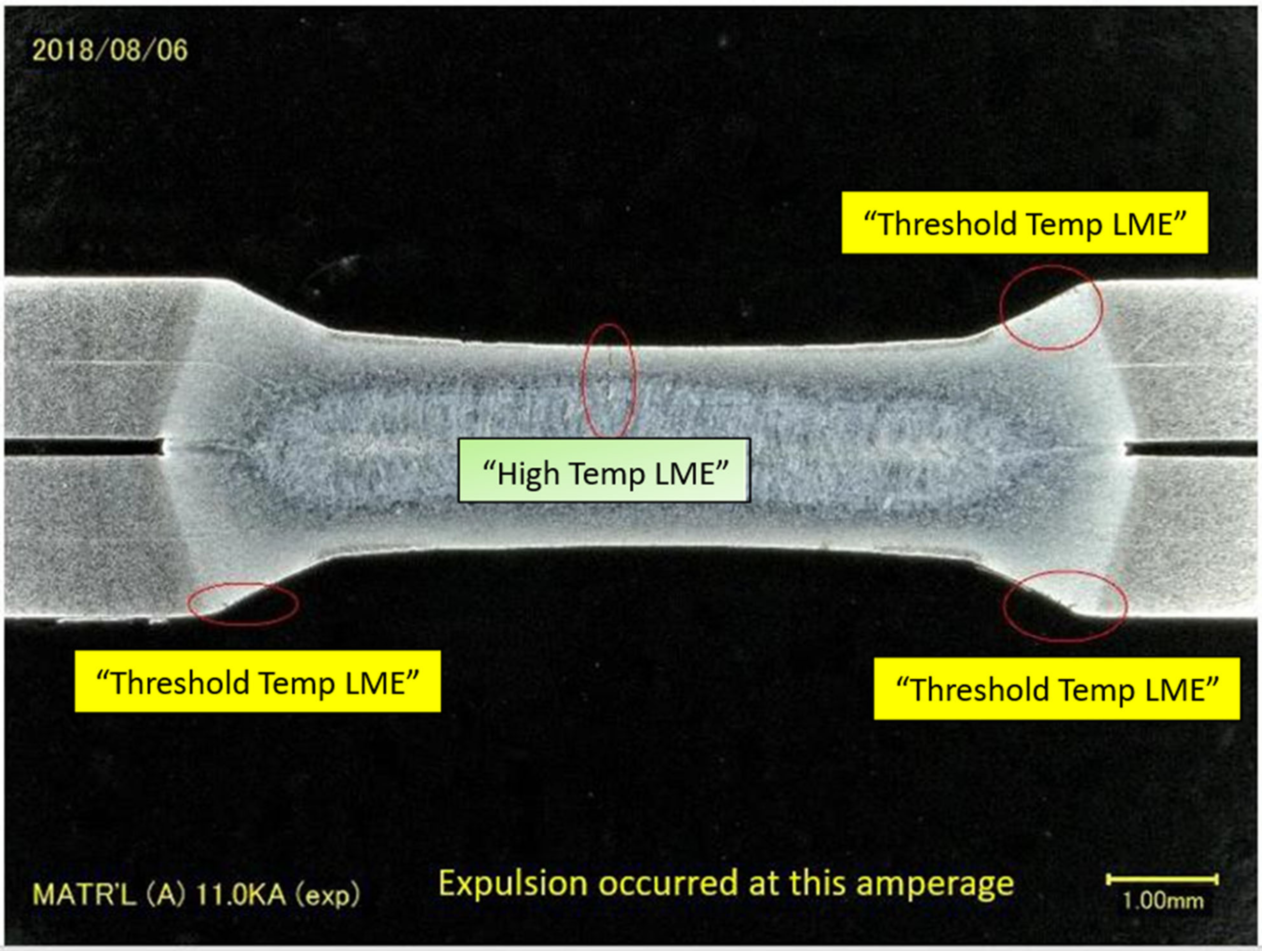


Table 1: General Acceptability of LME Cracks (By Location and Depth)

(THESE ACCEPTANCE CRITERIA APPLY TO THIS TEST ONLY, AND DO NOT IMPLY A PRODUCT QUALITY STANDARD)

	4 Crack Types by Relative Temperature:			
	Type A	Type B	Type C	Type D
Relative Test Current	High Temp LME (>650C)	Low Temp LME (<650C)	Interfacial LME (? rare)	Threshold Temp LME (~650C)
Below Expulsion	> 10% NOK	> 5% NOK	> 5% NOK	> 5% NOK
At (or above) Expulsion	OK	> 5% NOK	> 5% NOK	> 10% NOK

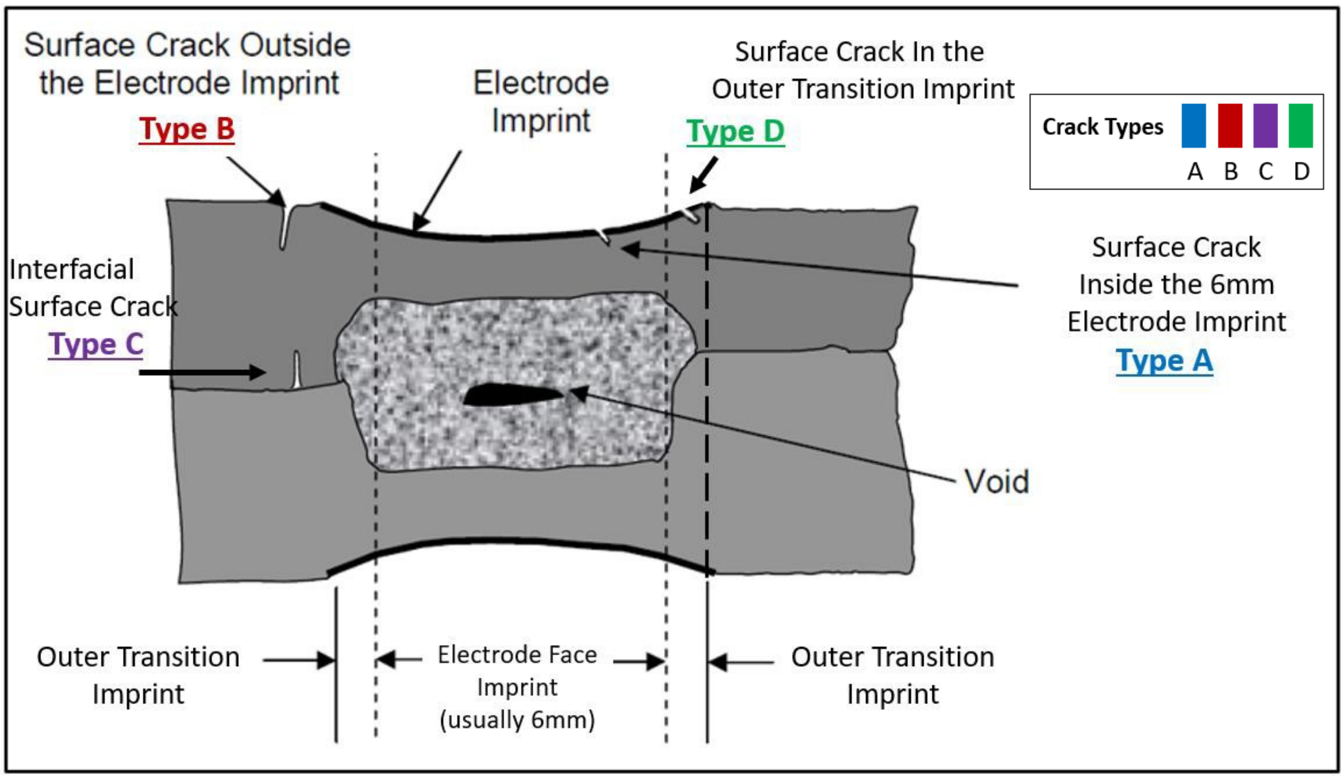


Figure 10: Crack location map, to be used in conjunction with Table 1 to interpret results.

EXAMPLE A

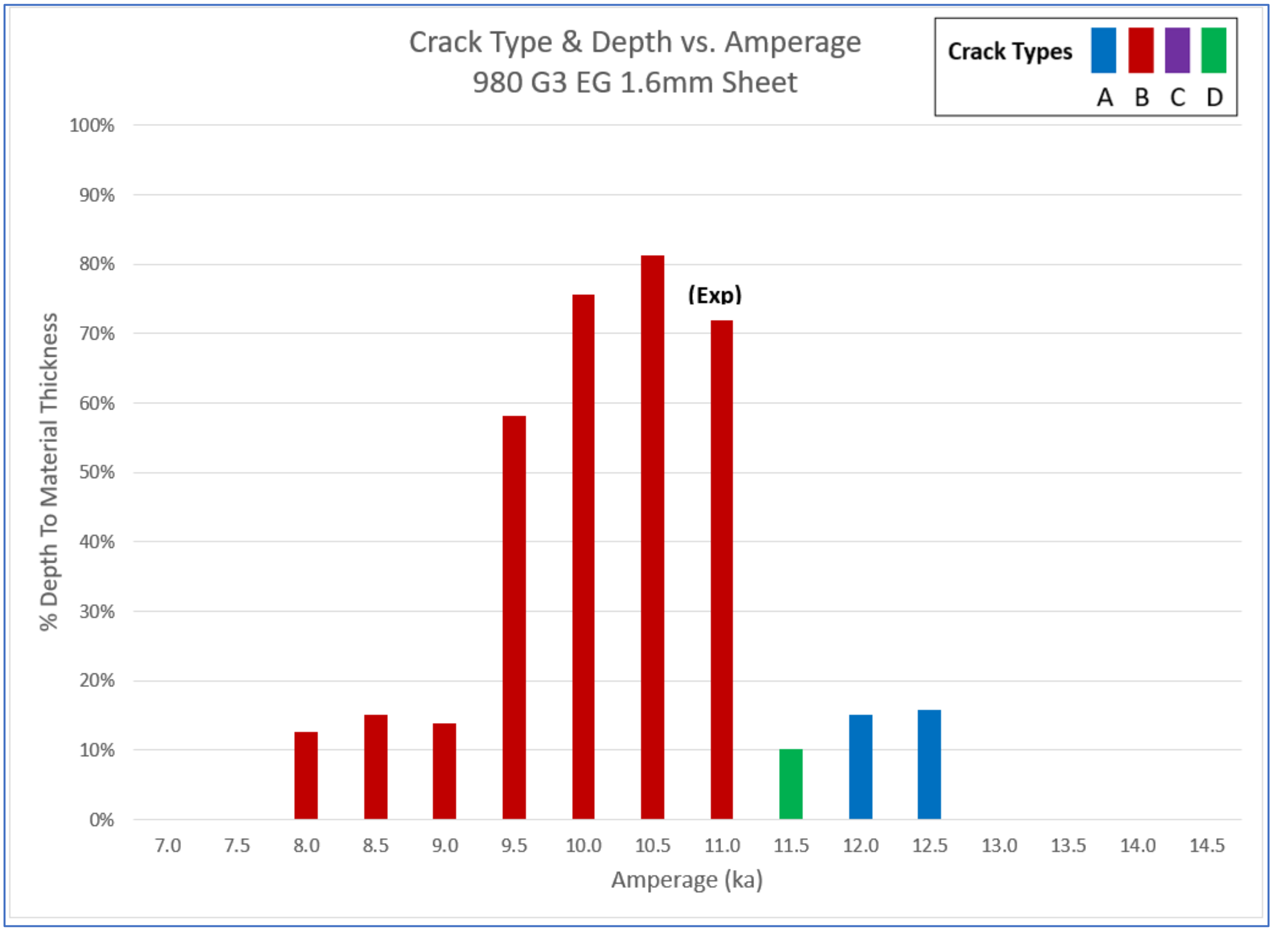
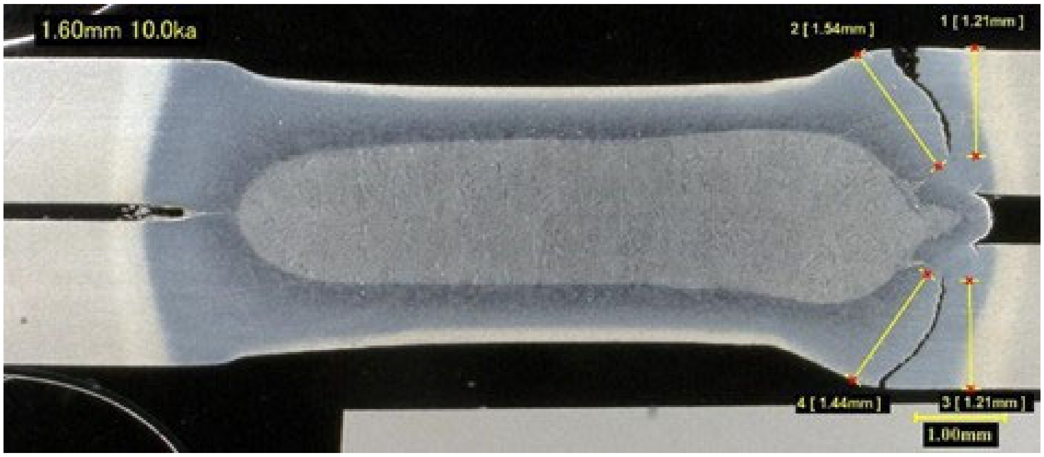


Figure 10. LME Susceptibility Graph - Sample result of one complete test.

Interpretation:

- 15% Hi Temp LME (Type A) above Expulsion – ACCEPT
- 80% Low Temp LME (Type B) below Expulsion -REJECT
- 10% Threshold LME (Type D) above Expulsion – REJECT

Conclusion: <u>Highly susceptible material – REJECT BATCH</u>



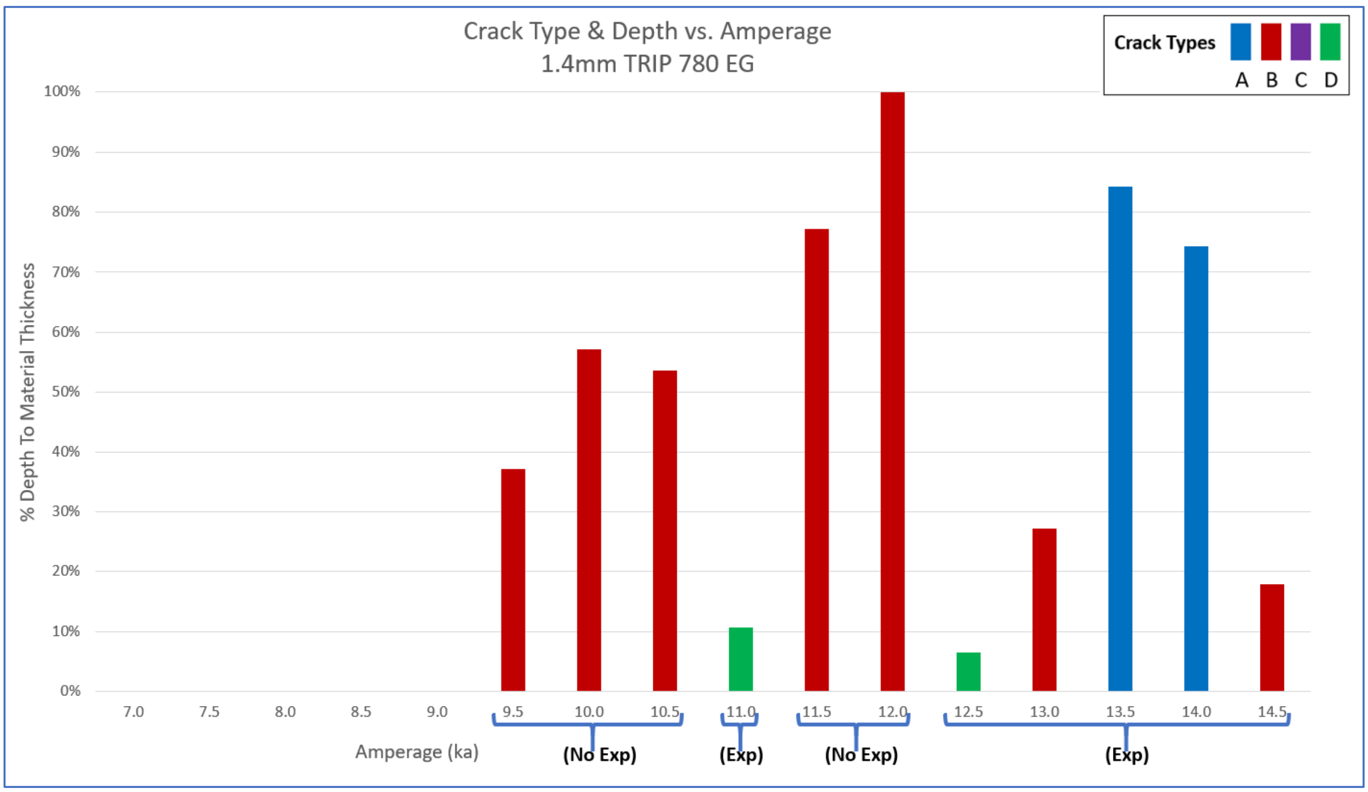


Figure 11. LME Susceptibility Graph - Sample result of one complete test.

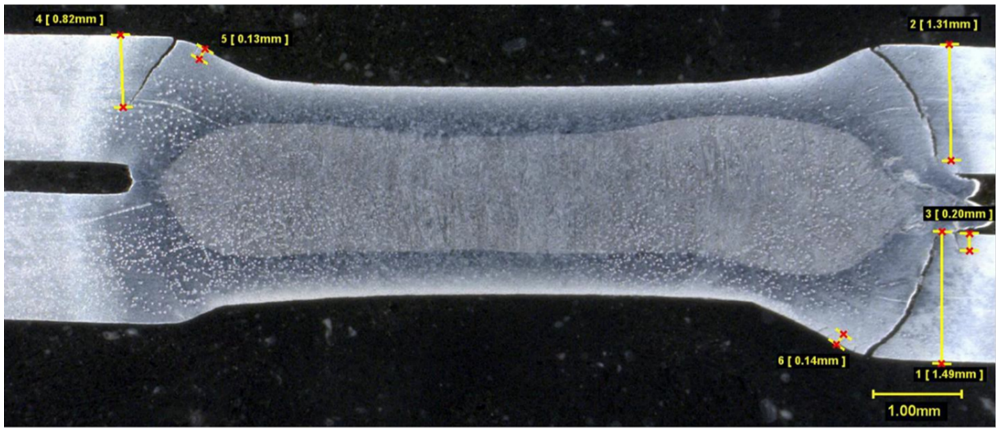
EXAMPLE B

Interpretation:

- 85% Hi Temp LME (Type A) above Expulsion – ACCEPT
- 100% Low Temp LME (Type B) above Expulsion -REJECT

- 10% Threshold LME (Type D) above Expulsion – ACCEPT

Conclusion: <u>Highly susceptible material – REJECT BATCH</u>



EXAMPLE C

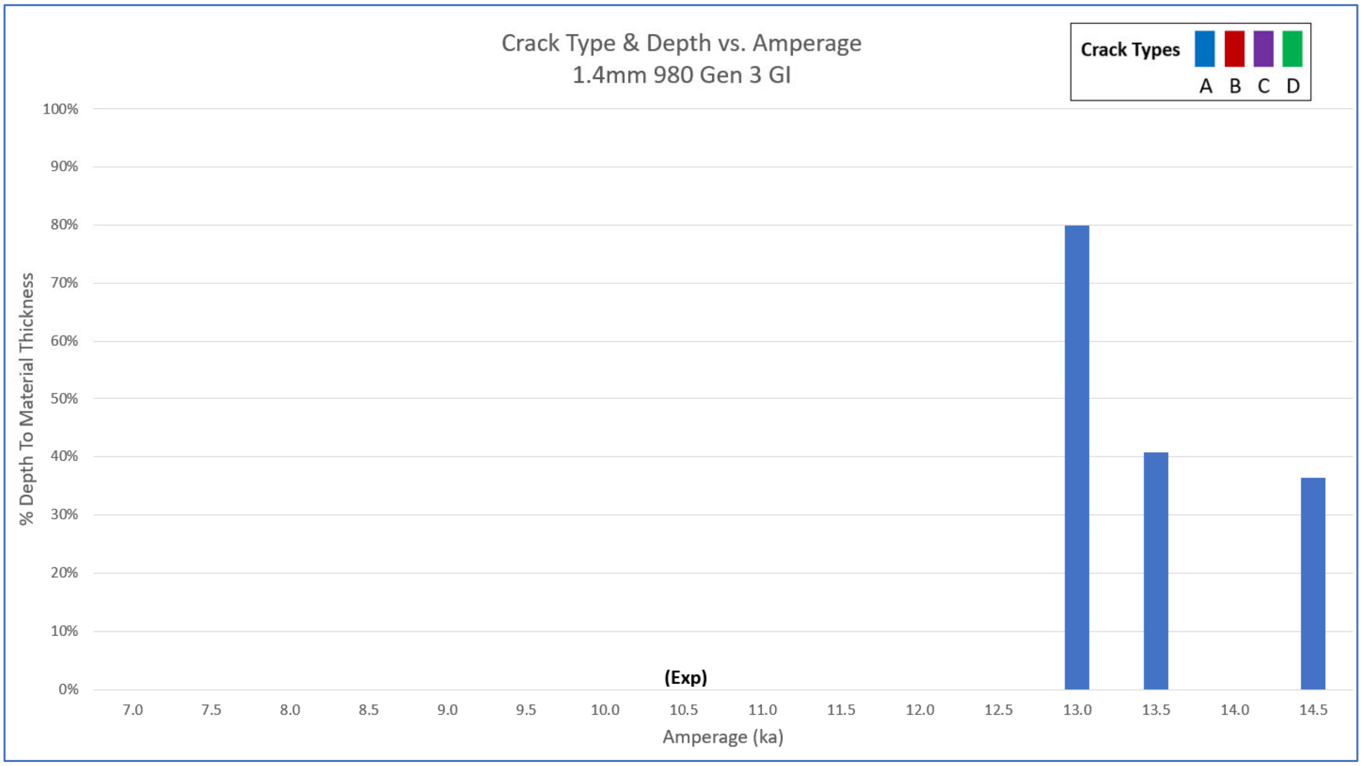
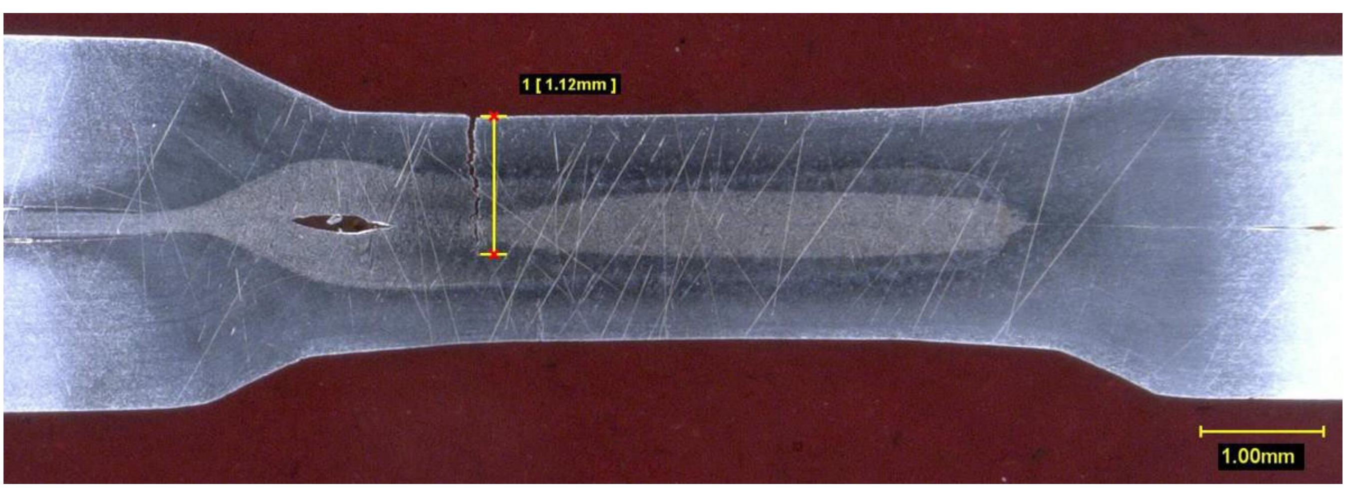


Figure 12. LME Susceptibility Graph - Sample result of one complete test.

Interpretation:

- 80% Hi Temp LME (Type A) above Expulsion – ACCEPT

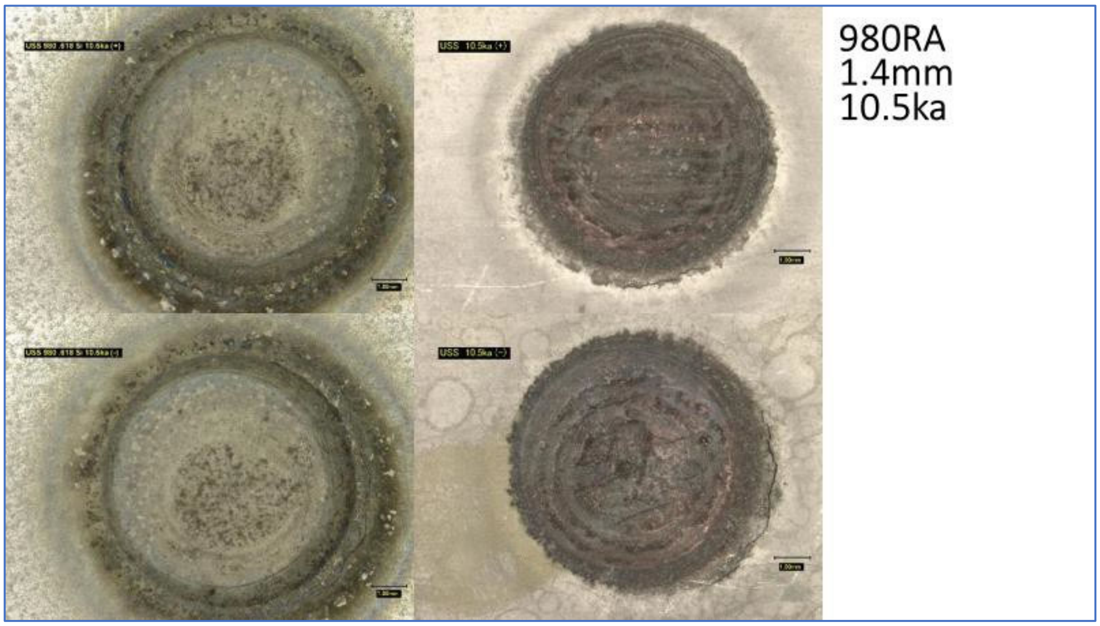
Conclusion: Low LME Susceptibility – ACCEPT BATCH



7.0 Appendix A – Sample report format

Page 1 format (for each of 16 welds)

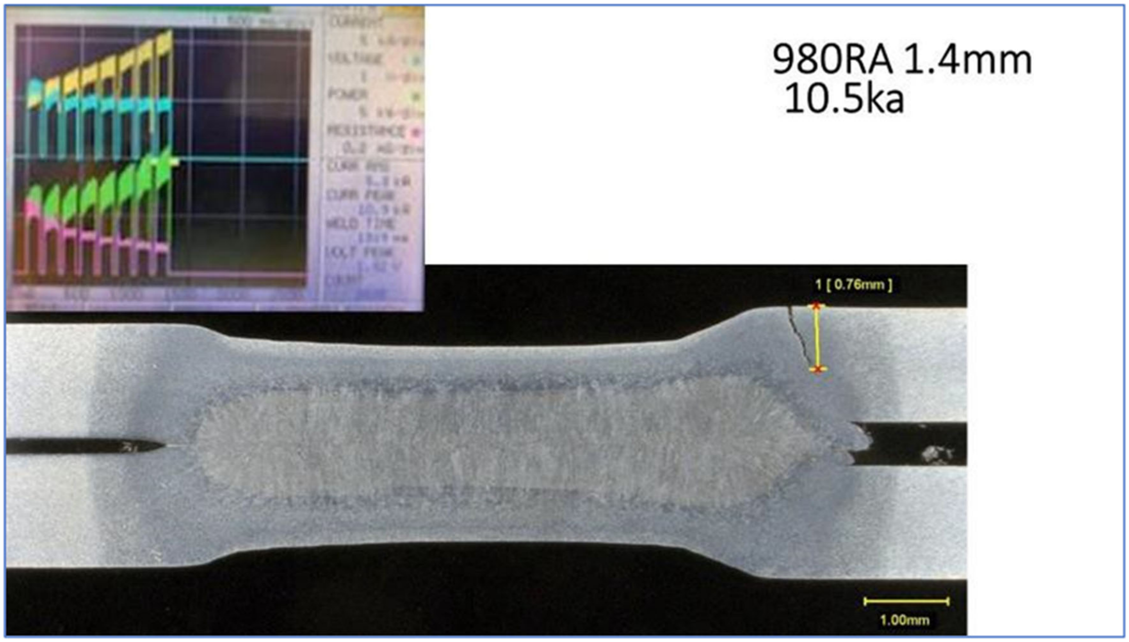
As-welded macro views **Same**
macro views
(each side) **(after pickling off the zinc)**

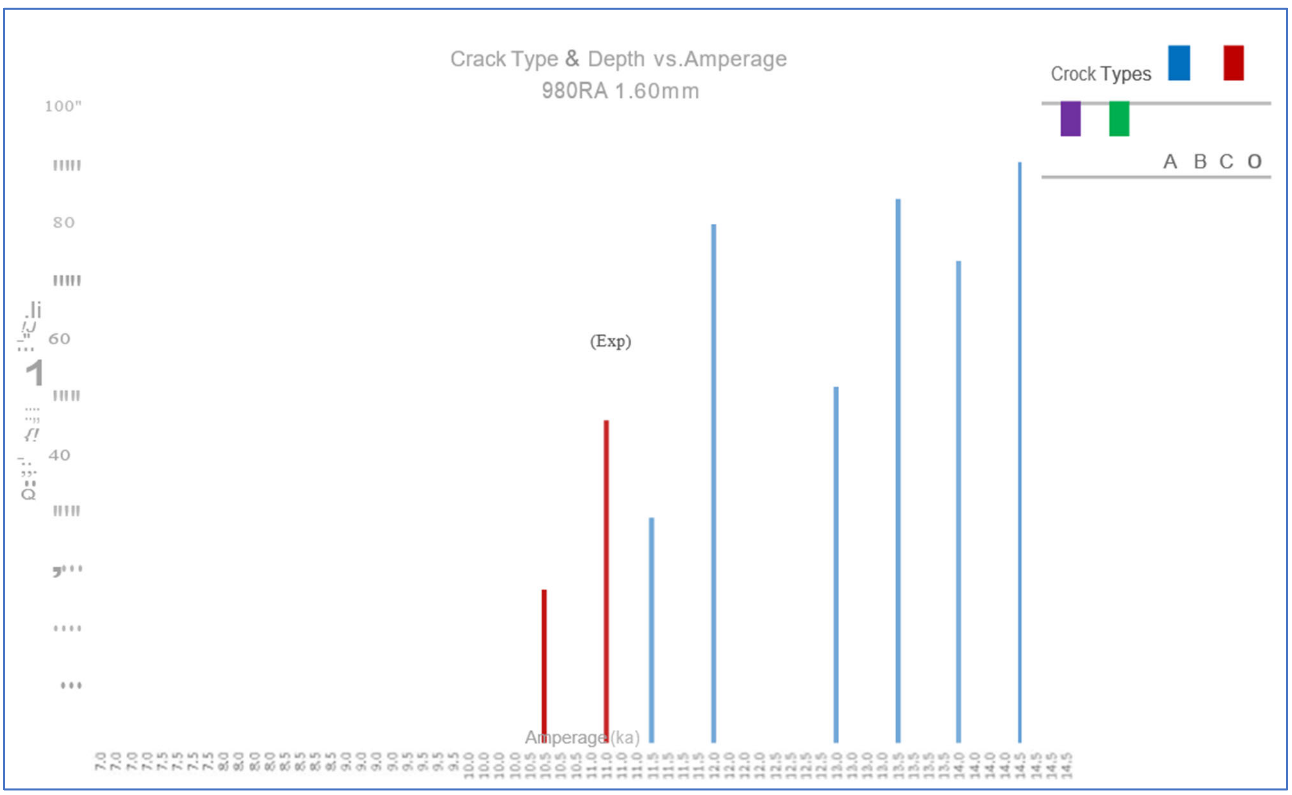


Page 2 format (for each of 16 welds)

Oscilloscope trace of Current, Voltage, Power, Resistance

And sectioned weld at magnification sufficient to find and measure cracks that penetrate deeper than 5% of the base material thickness.





Summary graph of the deepest cracks in a full set of 16 welds for a completed test