



Liquid Metal Embrittlement – LME Gleeble-Based Testing Procedure

Developed in collaboration with S/AP

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January 13, 2019



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1. Test Description and Objective

A procedure was developed to identify at what temperature (400 – 800 °C, 25 °C increments) and stroke rate (5, 50, 500 mm/s) various materials experience LME. Testing compared the galvanized material coating to its uncoated counterpart. Tests performed are similar to a hot tensile pull, materials were heated to a set temperature and then a predetermined stroke rate was applied until fracture. The Gleeble 3800 allowed for collection of force (kN) and stroke position (mm) data which was used to quantitatively determine material susceptibility to LME. The subheadings below will discuss in detail sample design and preparation prior to testing, the Gleeble testing procedure, qualitative and quantitative data collection, and data post-processing.

2. Sample Design

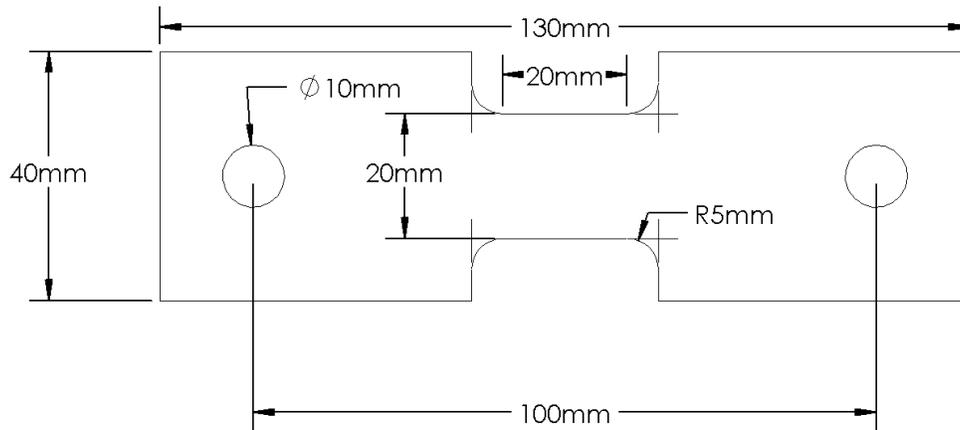


Figure 1: Gleeble tensile testing dimensions

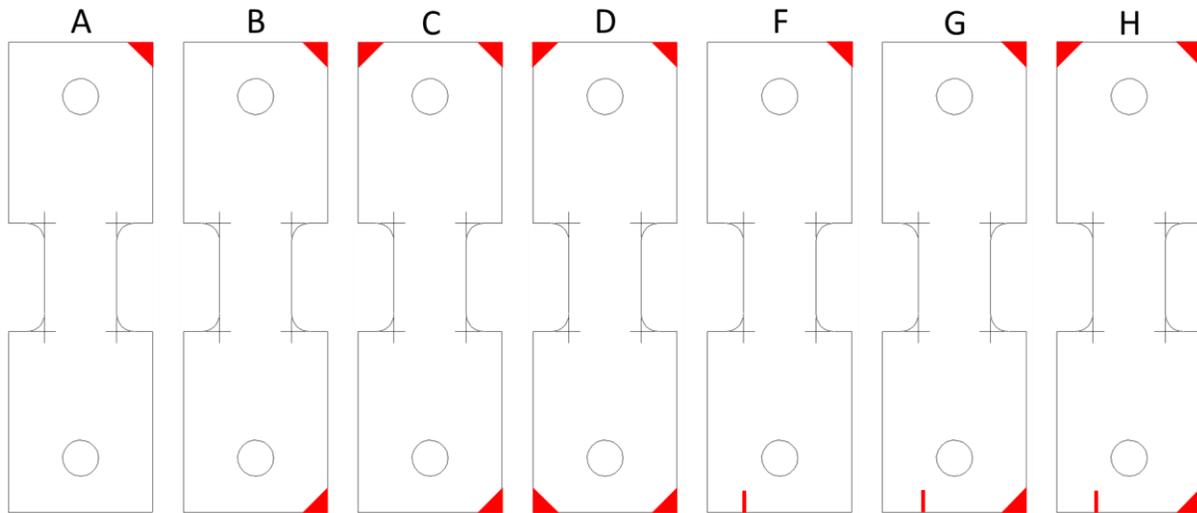


Figure 2: Gleeble tensile coupon identification

3. Tests Stages

- 1) Prepare sample
 - a. Scribe lines
 - b. Etch and strip based on test
- 2) Attach thermocouples
- 3) Run test and verify cracking
- 4) Graph results in Origin
- 5) Analyze data



4. Preparing Samples: Scribe Lines

Each sample requires a transverse line scribed across the length of its reduced section. This scribe line acts as a reference when etching the samples for the test. To scribe line each sample:

- 1) Make two small scribe points down the middle of two samples shown in Figure 3.

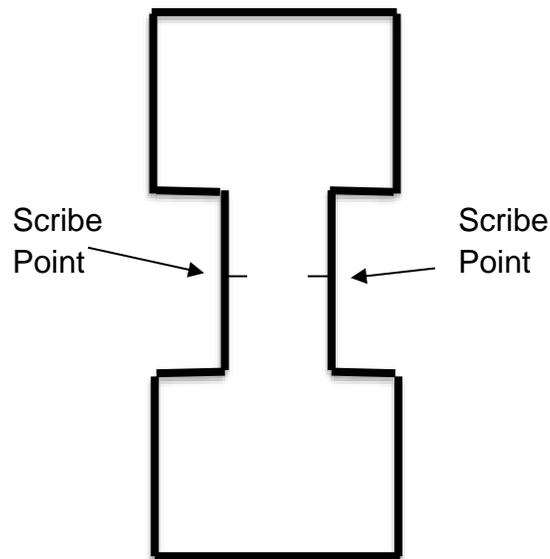


Figure 3: Scribe points example

- 2) Lay several samples in-line with each other, placing the two samples with scribe points at the ends of the row. This arrangement is illustrated in Figure 4.

NOTE: Verify all samples are aligned with a straight edge/ruler to ensure each scribe line is made through the center of every sample.

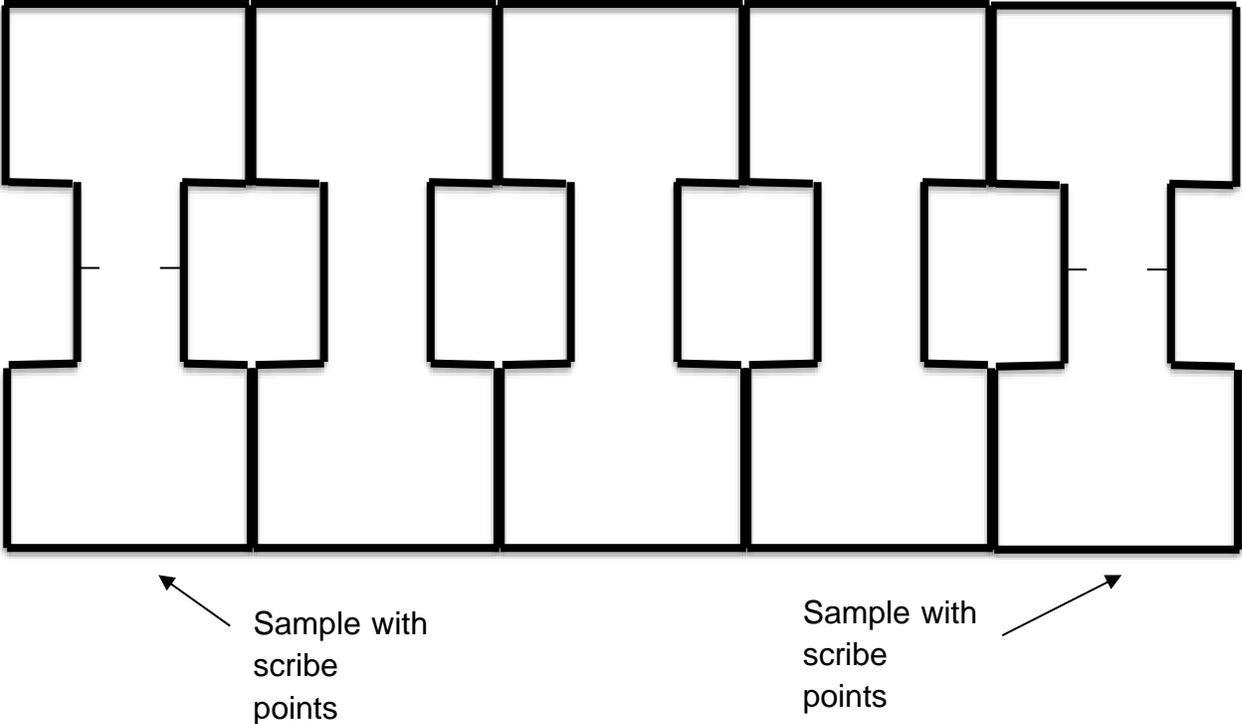


Figure 4: Alignment of pre-scribed samples

- 3) Place a straight edge, such as a ruler, across the four scribe points and scribe a line down the middle of all samples using a hard, sharp object, such as a hobby knife. A depiction of the final scribed samples can be seen in Figure 5.

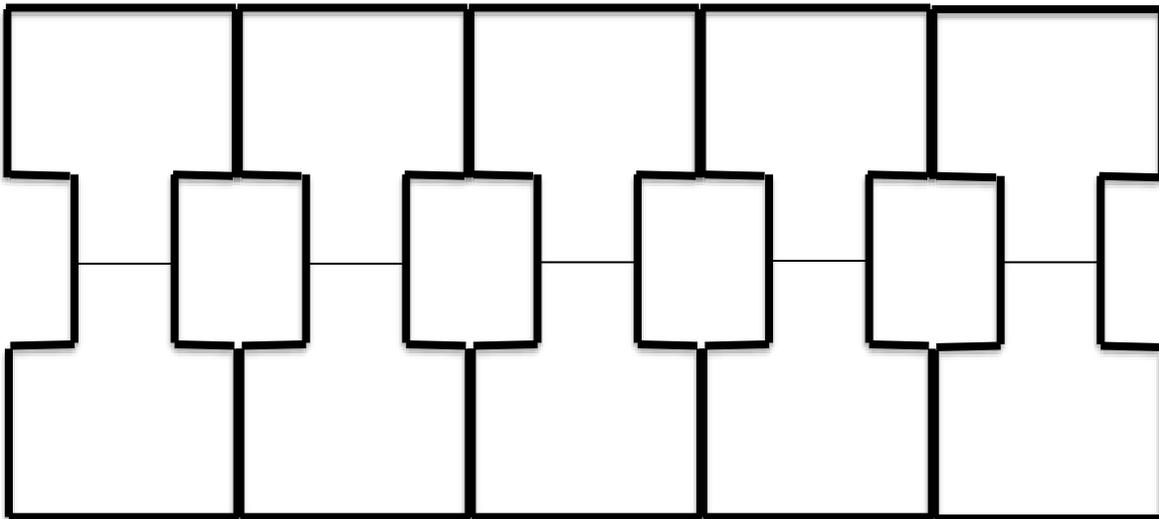


Figure 5: Row of completed scribed samples



- 4) Completed reference lines should lie across the middle of every sample. Dimensional errors less than 0.5 mm are acceptable.

5. Preparing “Coated” Samples: Etching for Thermocouples

The scribe lines are then used as a guide for proper placement of the welded thermocouples during Gleeble testing. In order to create sufficient welds between the thermocouples and the samples, the galvanized zinc layer needs to be etched off. This process is described in the following steps:

- 1) Bring prepared samples to the fume hood and locate the following materials:
 - a. 2 glass petri dish
 - b. 1 cotton swab with a wooden shaft
 - c. Concentrated (12M) Hydrochloric Acid (HCl)
 - d. Ethanol spray bottle
 - e. 2 Plastic trays
 - f. Paper towels
 - g. 1000mL Beaker
 - h. Hot blower
- 2) Break or cut the cotton swab in the middle of its wooden shaft to create a sharp taper point as seen in Figure 6. The sharper point will provide more precision when etching the samples with HCl.



Figure 6: Broken shaft of cotton swab

- 3) Working under the fume hood, place a small amount (~30 mL) of concentrated HCl in a petri dish. **WARNING:** Concentrated HCl is very corrosive and will damage clothing and skin if contact is made.
- 4) Place the concentrated HCl back into its storage unit.
- 5) Place a plastic tray outside of the fume hood and cover with several paper towels to help samples dry in future steps.
- 6) Place another plastic tray and ethanol spray bottle underneath the fume hood.
- 7) Place the samples to be etched onto the plastic tray under the fume hood in one single file, unstacked line with the scribe lines facing upwards to be visible
 - a. To expedite the procedure, heat the samples with the blow dryer before etching. The heat will act as a catalyst and reduce the iterations etchant must be applied to the thermocouple weld locations.
- 8) Dip the tapered wooden tip of the cotton swab into the concentrated HCl. Be sure to wipe off any excess HCl using the rim of the petri dish.



- 9) Using the tapered cotton swab, carefully apply HCl onto two small spots along the ends of a sample's scribe line as depicted in Figure 7. Each spot should be ~0.2mm in diameter.

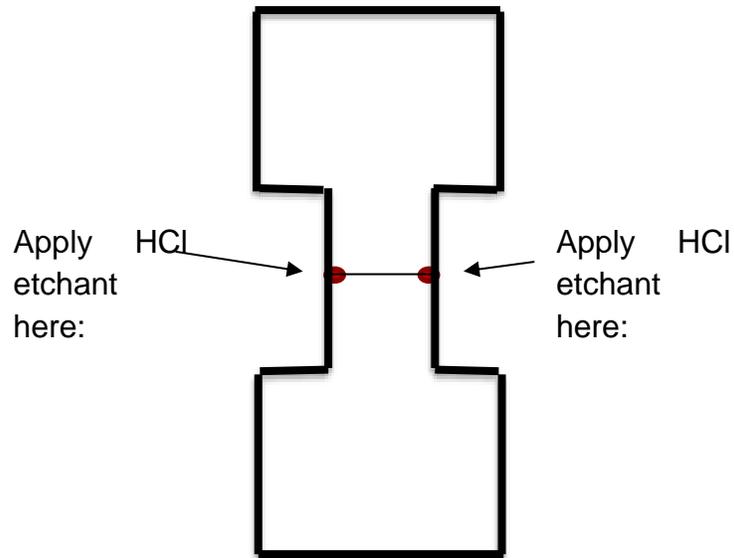


Figure 7: Etchant Spots

- a. **NOTE:** If an etched spot appears to be too large, the sample may need to be discarded. Do not panic; it is preferable to discard a sample rather than performing a Gleeble test that yields skewed or unusable data.
- 10) Repeat step 8 for all samples on the plastic tray under the fume hood.
- 11) Rinse the etchant off the samples with tap water in the sink next to the fume hood and immediately blow dry the samples with the hot blower over the sink. **WARNING:** Drying samples with paper towels causes sample oxidation. Be patient and use the hot blower until the sample is free of liquid.
- 12) Repeat steps 10-12 until thermocouple weld spots are etched to bare metal.
- 13) Under the fume hood, rinse all completed samples with ethanol over a designated petri dish catch then immediately dry the samples over the sink with the hot blower.
- 14) If more samples need to be etched, repeat steps 6-13 until all samples are finished.
- 15) Rinse the wooden end of the broken cotton swab in the sink to wash away any residual HCl. Once completed, discard in the appropriate trash bin.
- 16) Carefully pour any residual HCl on the plastic tray into the petri dish of concentrated HCl. Be sure not to splash or spill this solution while pouring.
- 17) On the wall to the right of the fume hood, complete the chemical log sheet for the chemical waste container. Fill out the chemical waste type (ethanol and HCl), approximate total volume, and date onto the log sheet.
- 18) Carefully pour the contents of both the ethanol and HCl petri dishes into the chemical waste container.
- 19) Remove all items from the hood and wash the plastic trays and petri dishes.



- 20) Sort the etched samples and store them in a dry environment (i.e. desiccator).
- 21) The samples are ready to be tested in the Gleeble as “coated” samples.

6. Preparing “Bare” Samples: Acid Immersion Process

- 1) Under a fume hood, mix a solution of 1:1 hydrochloric acid and deionized (DI) water.
- 2) Fill the 1000mL beaker with solution to fully cover the sample.
- 3) Place spacers in the beaker to keep the samples off the bottom of the glass.
- 4) Under a fume hood, immerse three to four samples in the beaker of solution until bubbling stops.
- 5) After bubbling stops, remove the sample from the beaker using tongs and rinse in the sink with tap water thoroughly.
- 6) Dry the specimen using the hot blower over the sink.
- 7) Under a fume hood, rinse the sample with ethanol over a petri dish catch and blow dry again.
- 8) As the total number of samples that have been immersed increases, the bubbling action will be reduced. This indicates the solution needs to be replaced for further stripping. Follow steps 9 and 10 before replacing the solution. Begin with step 1.
- 9) On the wall to the right of the fume hood, complete the chemical log sheet for the chemical waste container. Fill out the chemical waste type (ethanol and HCl), approximate total volume, and date onto the log sheet.
- 10) Carefully pour the beaker of solution and ethanol petri catch dish into the chemical waste container.
- 11) Remove all items from the hood and wash the two plastic trays, 1000 mL beaker, and petri dish.
- 12) Sort the etched samples and store them in a dry environment (i.e. desiccator).
- 13) The samples are ready to be tested in the Gleeble as “bare” samples.

7. Turning on the Gleeble Machine

- 1) Check the clear tube on the back of the high bay chiller to ensure that it has a sufficient water level.
- 2) If the chiller does not have enough water, open the valve (~2 seconds) until the water level rises to an acceptable level in the tube on the back of the chiller (indicated by green lines on the tube).

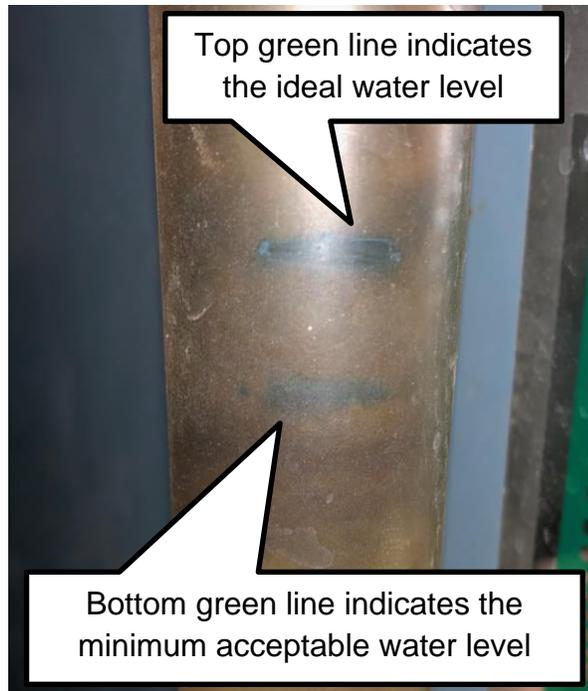


Figure 8. High bay chiller water level

- 3) Place the “GLEEBLE IN USE” sign on the chiller.



Figure 9. “GLEEBLE IN USE” sign

- 4) Turn the high bay chiller to “ON.”

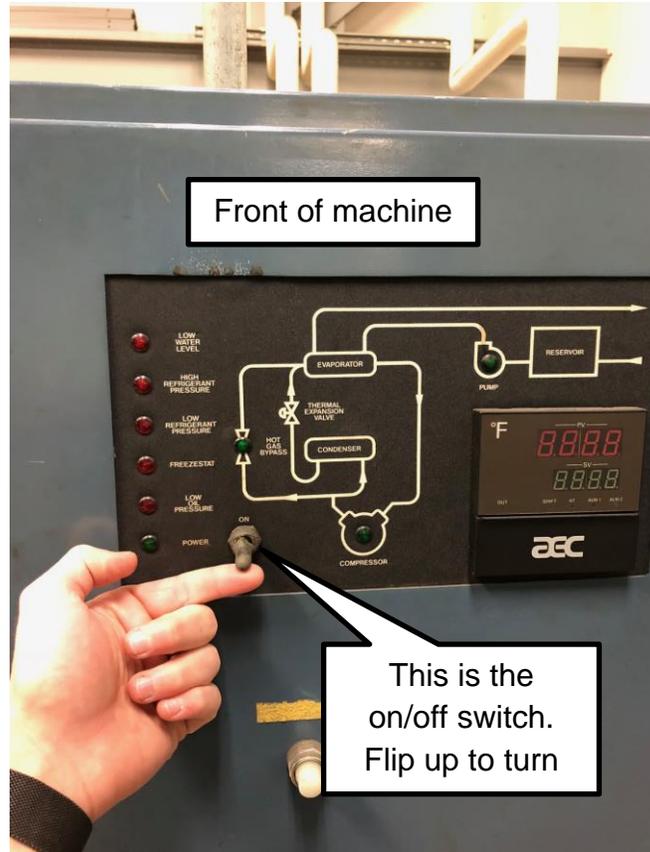


Figure 10. High bay chiller “ON” switch

5) Turn the chiller pump next to the Gleeble machine to “LOCAL.”

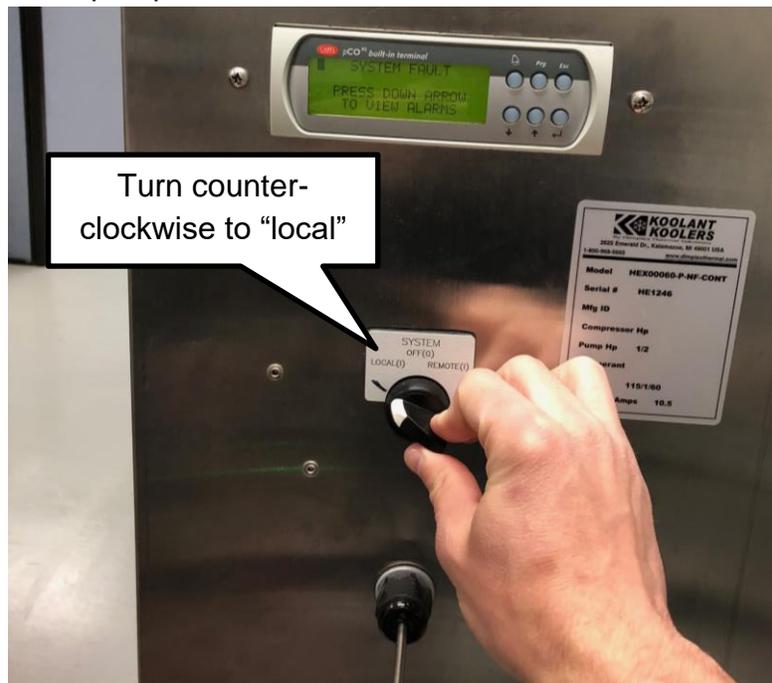


Figure 11. Chiller pump “LOCAL” switch



- 6) Open the argon tank valve to allow gas to flow to the Gleeble. The right gauge on the tank should be above 150 psi (gas level in the tank). If the right gauge is below 150 psi (the last red tick on the dial), replace the argon tank appropriately. See Figure 12 for reference.
- 7) The left gauge on the argon tank should read 40 psi (gas pressure running to the Gleeble machine). If the left gauge is below 40 psi, replace the argon tank appropriately.

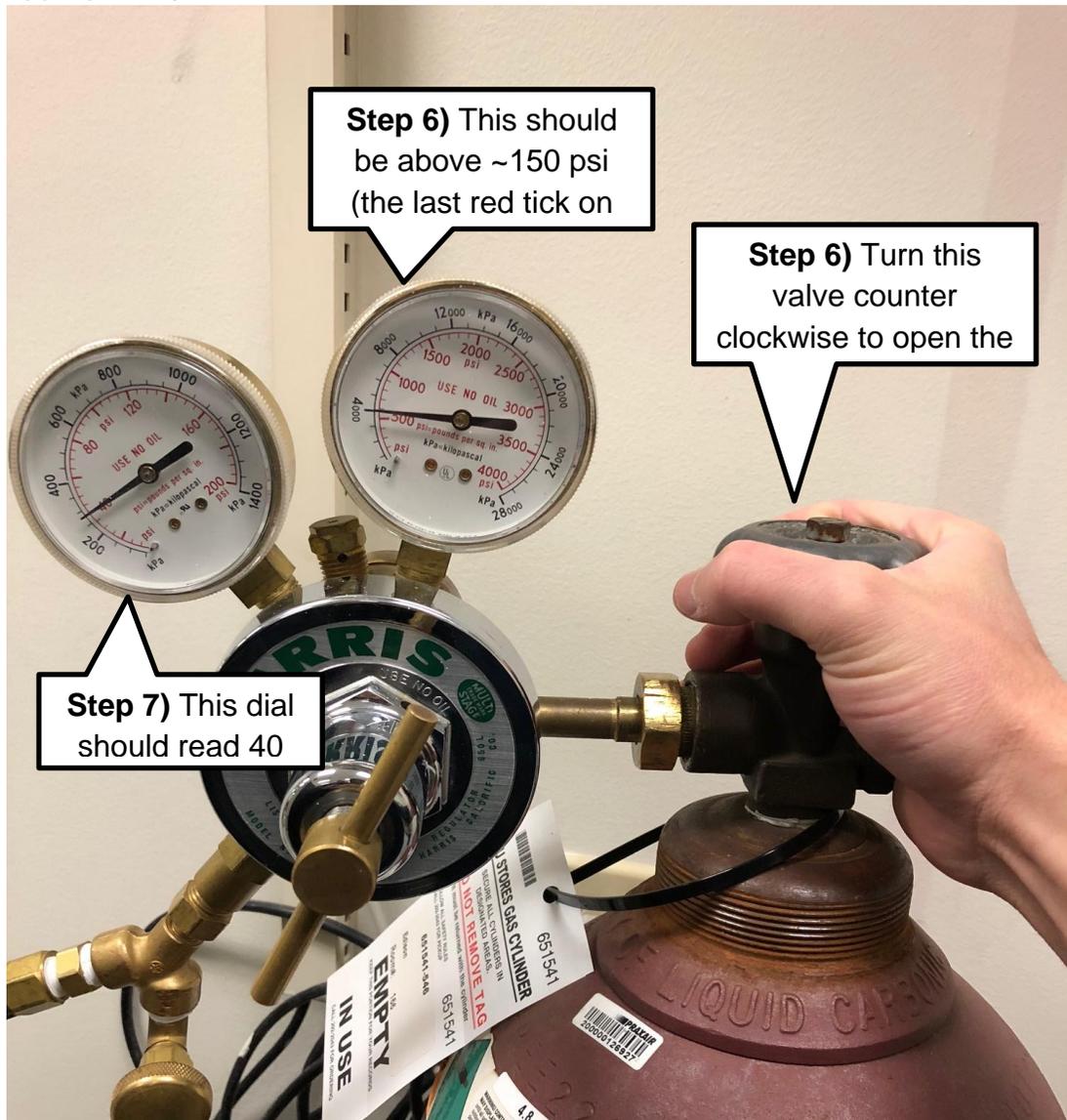


Figure 12. Gleeble argon gas tank gauges



8) Turn the Gleeble machine to “ON.”



Figure 13. Gleeble “ON” button

8. Welding the Thermocouples to the Part

- 1) Put on clean, latex gloves.
- 2) Turn the thermocouple welder to “ON.”

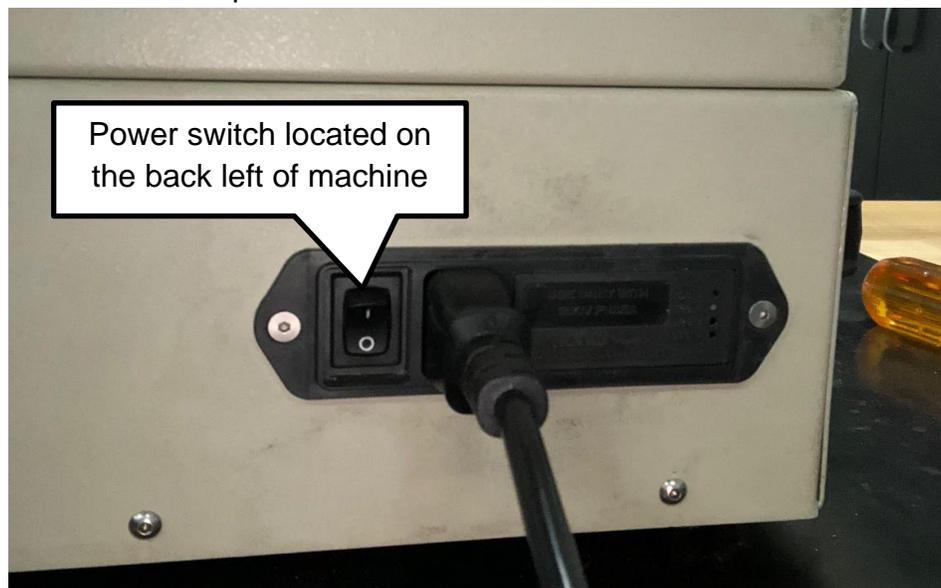


Figure 14a. Thermocouple welder “ON” switch



- 3) Set the thermocouple welder voltage to 35V

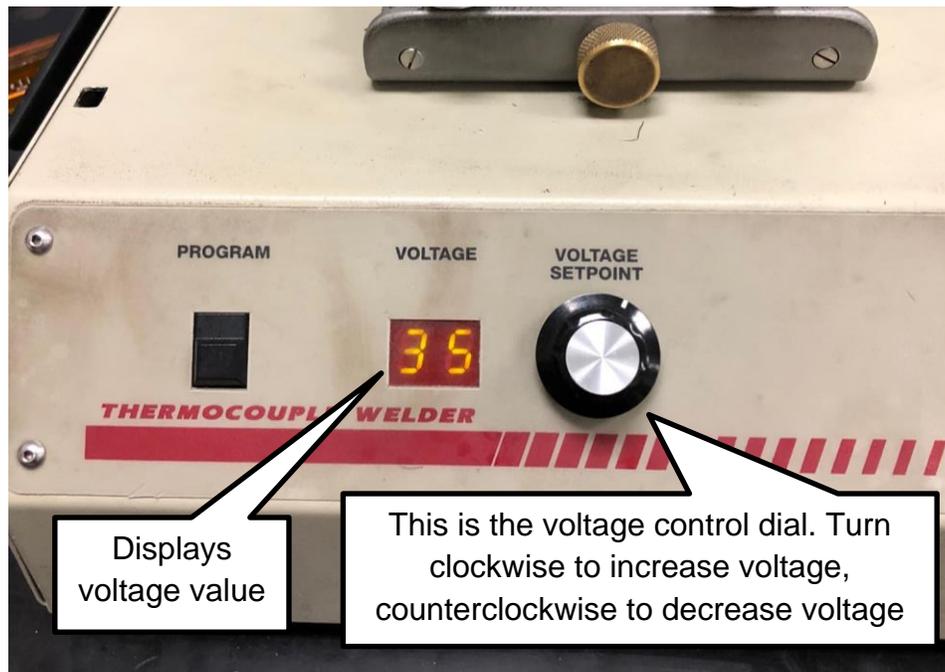


Figure 14b. Thermocouple welder: voltage set point and display

- 4) Find two thermocouple wires; one wire should be magnetic for the black (negative) terminal on the Gleeble, and the other wire should not be magnetic for the red (positive) terminal on the Gleeble. **NOTE:** Test to see if the wire is magnetic by using the magnet next to the Gleeble machine.
- 5) Ensure that the thermocouple wires are long enough to extend from the sample to the terminal in the Gleeble without experiencing tension.
- 6) Feed the wires through their respective insulation material to prevent the wires from shorting during testing. Ensure there is means to indicate both the positive and negative wires
- 7) Select an end of the thermocouple wire to be welded by locating a straight (not bent) portion of the wire close to that end.
- 8) Use wire cutters to cut the wire at the face of the straight portion. This cut is made at a 45° angle to create a “tip” at the end of the wire.
- 9) Wash the sample with ethanol again and dry with a “Kimwipe” prior to welding to remove any contaminants and reduce chances of oxidation during testing.
- 10) Place the sample to be tested in the thermocouple welding machine
- 11) Use the screw and lock mechanism to place the weld trigger so that it holds the thermocouple ~3 mm above the area on the sample to be welded. Turn clockwise to raise the trigger, counterclockwise to lower the trigger.

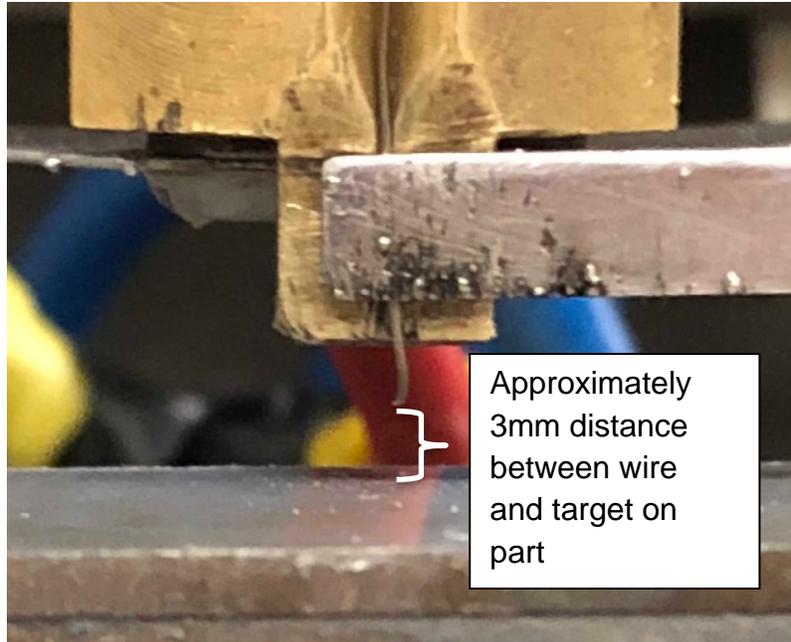


Figure 15. Distance from thermocouple wire and sample

12) Secure a thermocouple wire into the wire guide and clip.

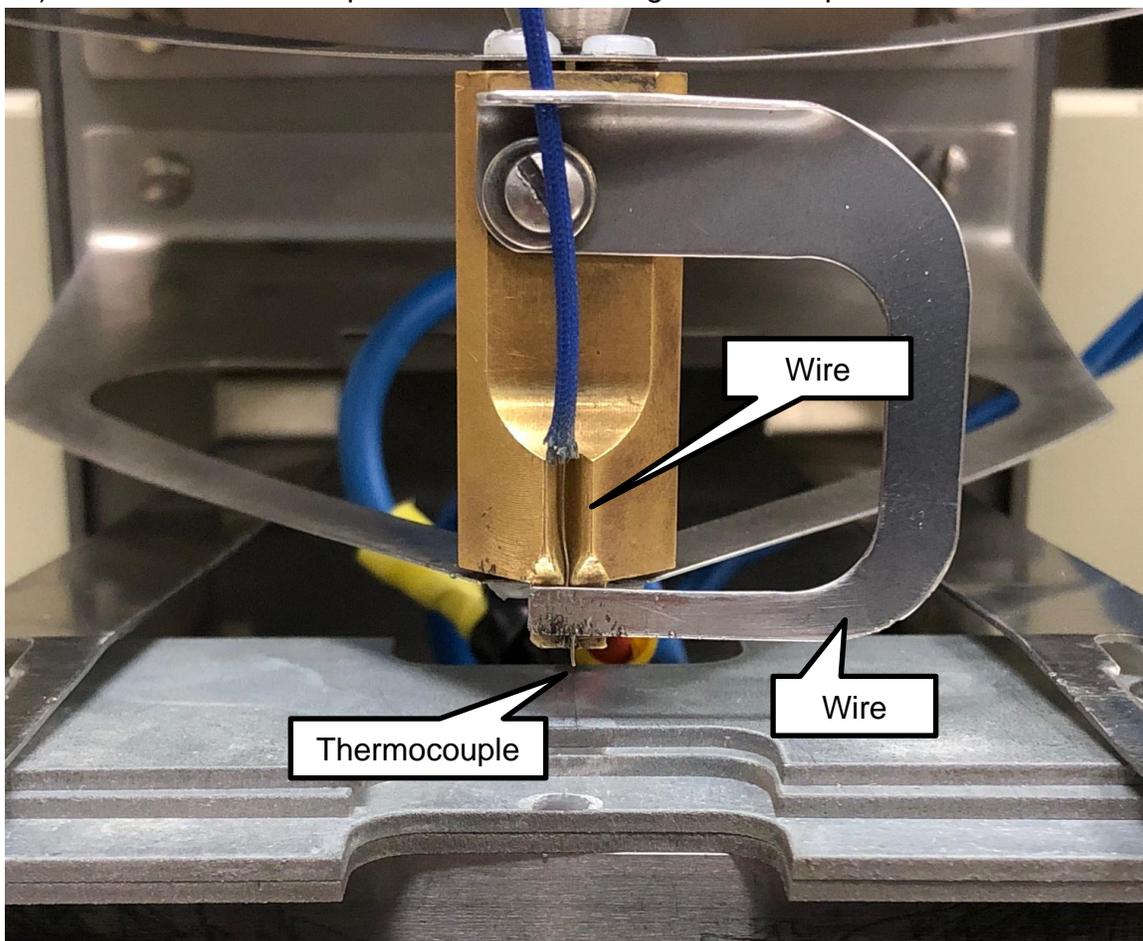


Figure 16. Thermocouple wire, wire guide, and wire clip



- 13) Align the wire with the location of the intended weld along the scribe line edge.
- 14) Set the wire length so that it is just touching the sample at weld completion.
- 15) Raise the thermocouple and its guide so that it rests on the weld trigger.
- 16) While looking away or using the weld shield, press the “WELD” button.
- 17) Repeat steps 5-12 for the other wire. Be sure to give the other wire slack and not break the weld.

9. Conducting the Gleeble Test

- 1) Open the valve to the Gleeble chamber to release any potential vacuum.

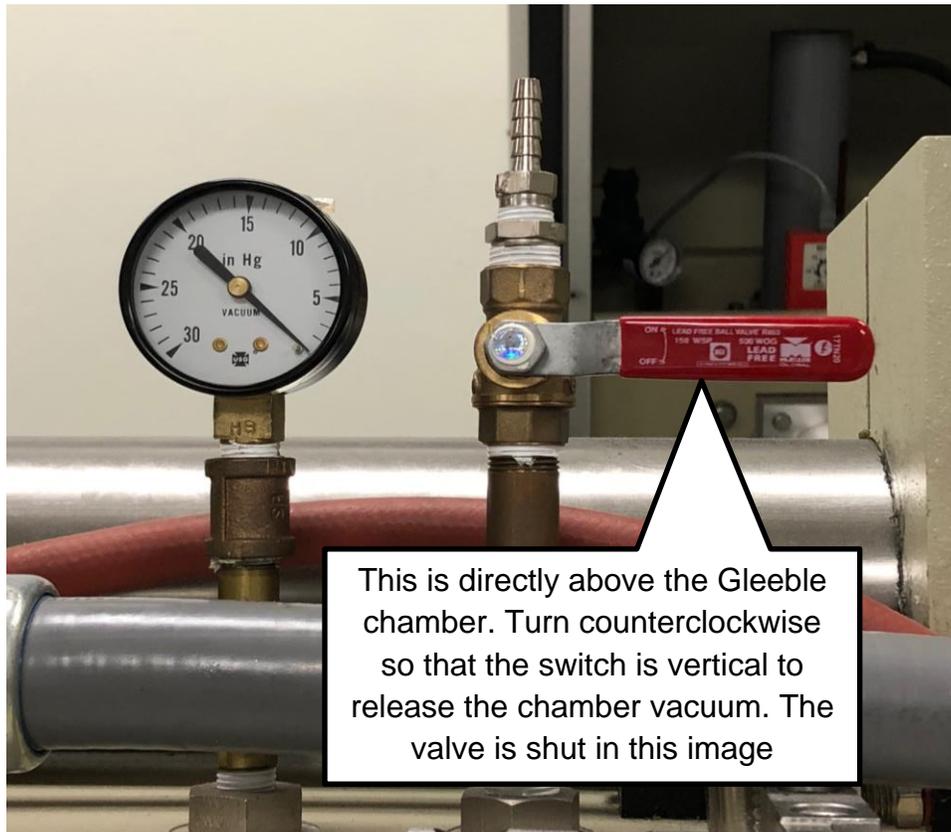


Figure 17. Gleeble vacuum release valve and gauge



- 2) Press the “DOOR RELEASE” button on the Gleeble and open the door.

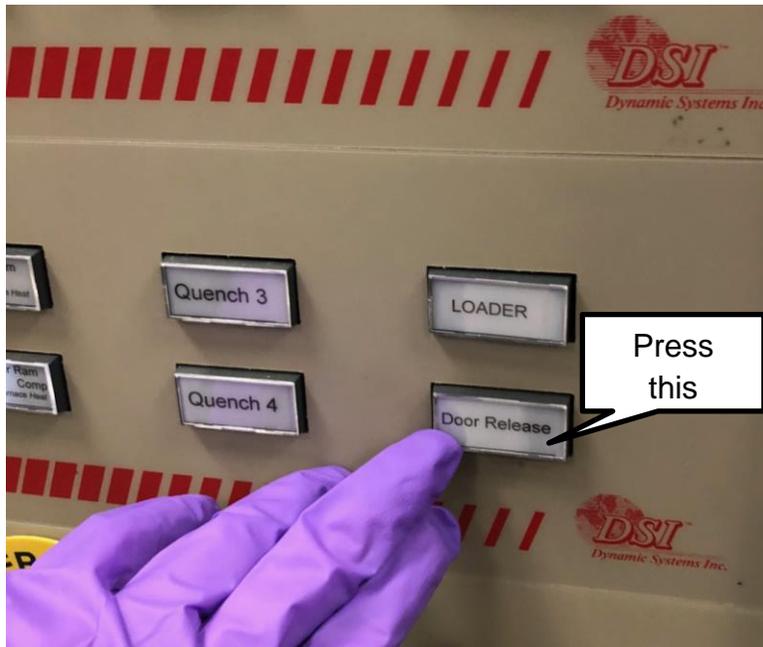


Figure 18. Gleeble “DOOR RELEASE” button

- 3) Set the copper sample mounts into the chamber.
- 4) Transfer the sample with welded thermocouples to the Gleeble, being careful not to separate the thermocouples.
- 5) Set the sample into the copper sample mount as seen in Figure 19.

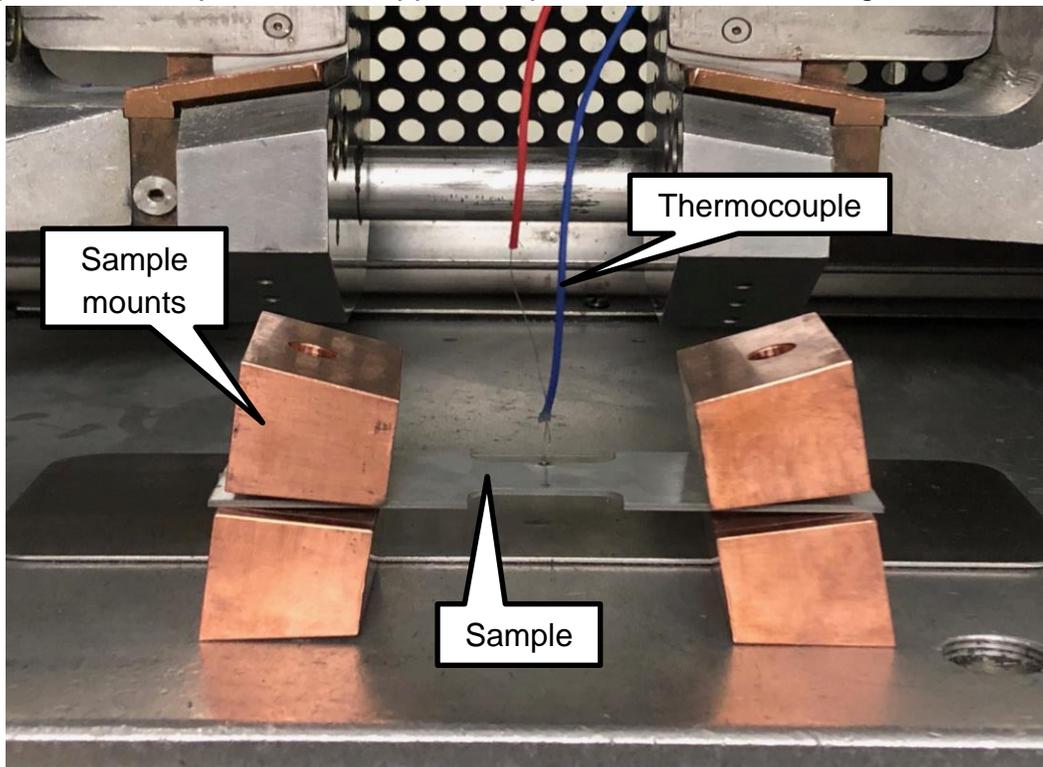


Figure 19. Copper sample mounts and mounted sample



- 6) Place the sample and sample mounts into their respective Gleeble jaws.
- 7) On the Gleeble machine press “RUN” followed by “MECHANICAL.” Both buttons should be illuminated



Figure 20. Gleeble “RUN” and “MECHANICAL” buttons

- 8) Use the “STROKE” dial to tighten the jaws on the Gleeble so that the Gleeble force gauge reads 2.5 kN. Turning the dial counter-clockwise loosens the jaws (reduces the force), and turning the dial clockwise tightens the jaws (increases the force).

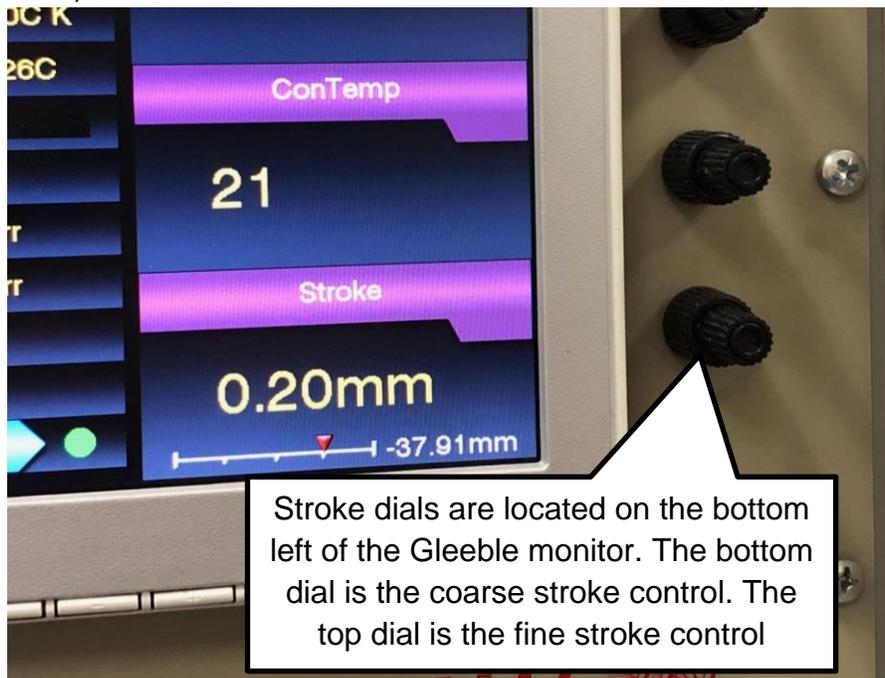


Figure 21. Gleeble “STROKE” dial



- 9) Place the U-spacers between their respective sample mounts and Gleeble jaws.

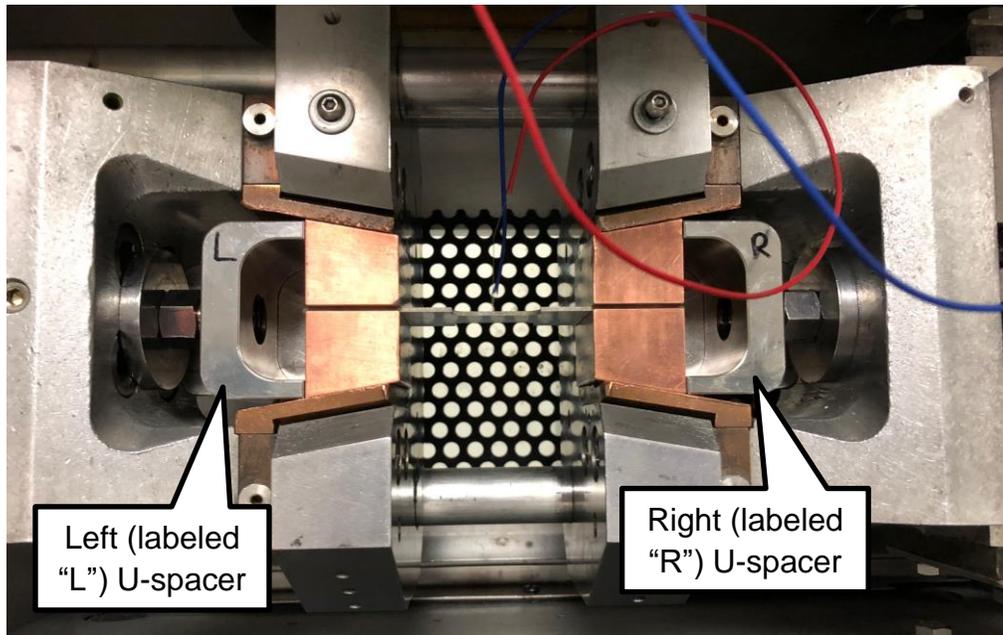


Figure 22. U-spacers between sample mounts and Gleeble jaws

- 10) Tighten the U-spacers to snug fit. Turn the left spacer nut “up” to tighten and the right spacer nut “down” to tighten.
- 11) Connect the thermocouples to the T/C2 terminals in the Gleeble machine. The magnetic thermocouple goes to the negative (black) port, and the non-magnetic thermocouple goes to the positive (red) port.
- Be sure that no bare thermocouple wires are touching
 - If two pairs of thermocouple wires are to be used, connect the “reference” wires to the T/C2 port and the other pair to the T/C3 port

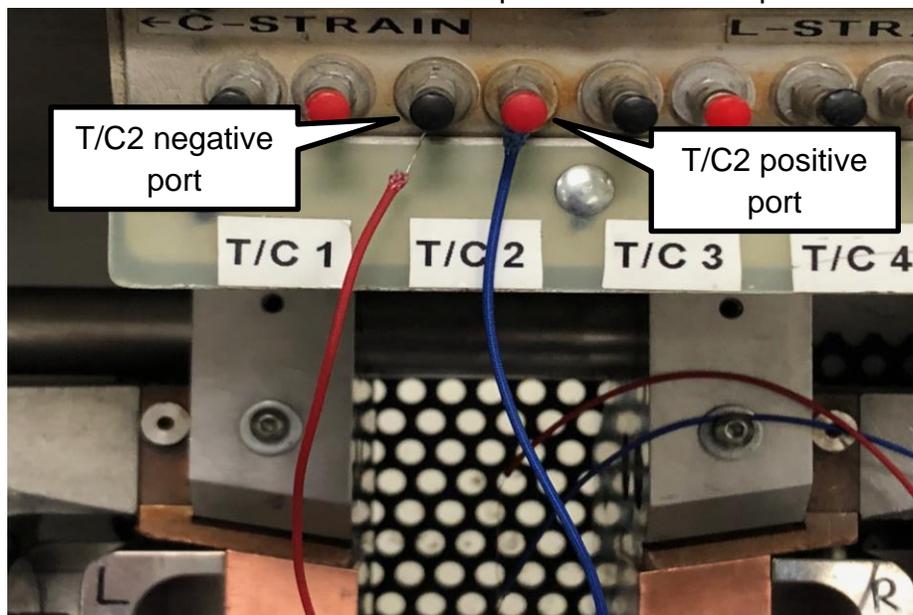


Figure 23. Thermocouple terminals in the Gleeble



- 12) Close the Gleeble vacuum chamber valve.
- 13) Set the Gleeble vacuum to “ROUGHING” then press “VACUUM RUN”

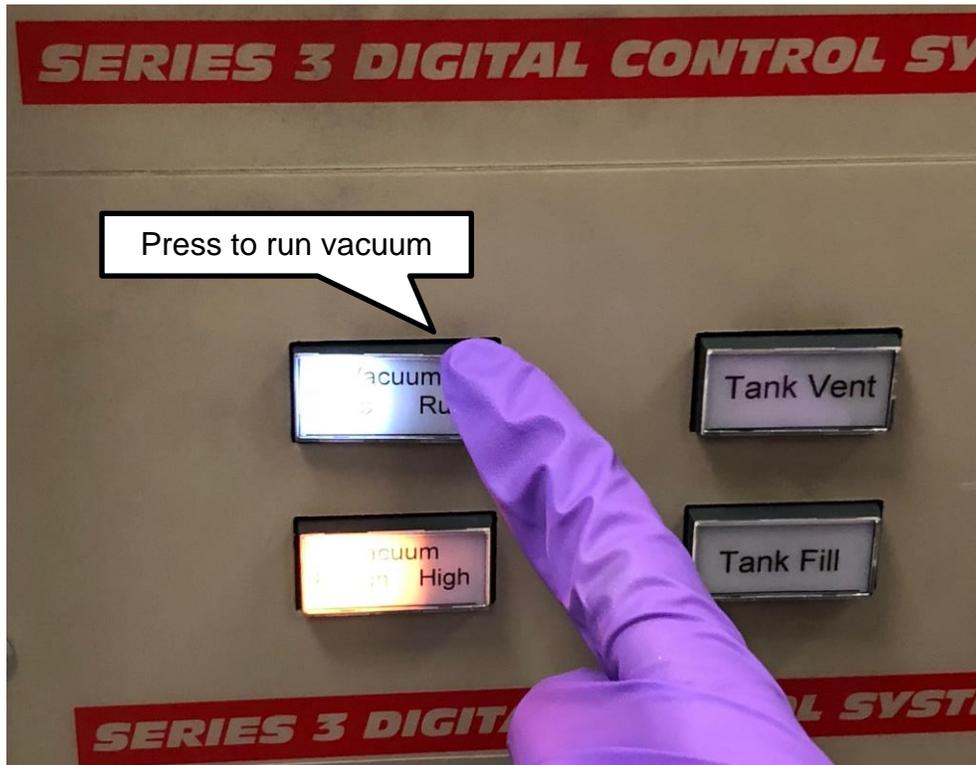


Figure 24. Gleeble “VACUUM RUN” and “VACUUM ROUGHING” buttons

- 14) Monitor the vacuum level until it reaches 3.2×10^{-1} torr.

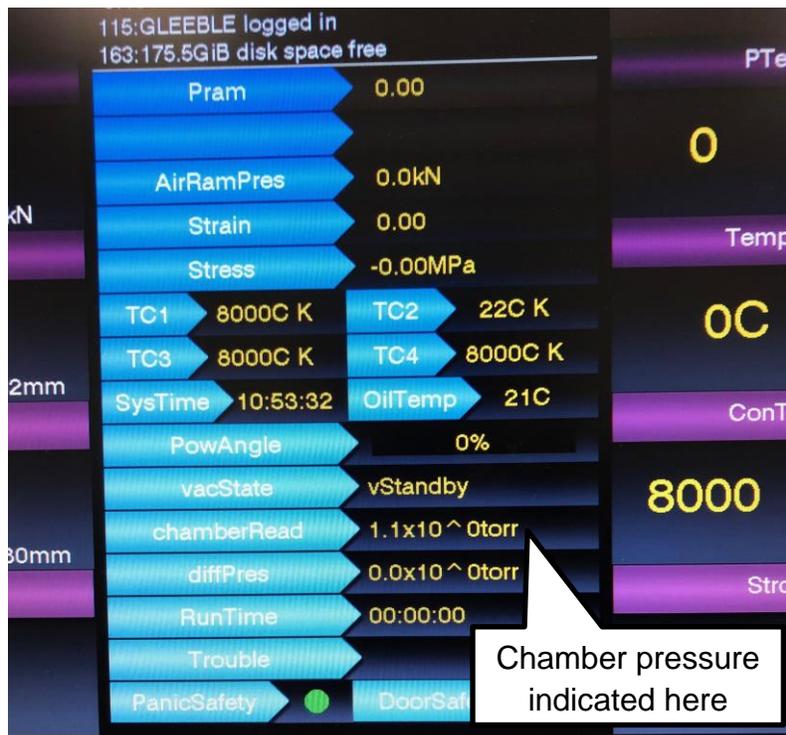


Figure 25. Gleeble monitor screen



- 15) Once the Gleeble control panel reads the chamber pressure as 3.2×10^{-1} torr, press “VACCUUM STOP” then “TANK FILL” to fill the chamber with argon gas.



Figure 26. Gleeble “TANK FILL” button

- 16) When the dial above the Gleeble chamber that measures the pressure in the chamber reaches 15 mmHg, press “TANK FILL” again to stop the argon gas.
- 17) Press the Gleeble “VACUUM RUN” button again.
- 18) Once the Gleeble control panel reads the chamber pressure as 3.2×10^{-1} torr, press “VACCUUM STOP” then “TANK FILL” to fill the chamber with argon gas.
- 19) When the dial above the Gleeble chamber that measures the pressure in the chamber reaches 15 mmHg, press “TANK FILL” again to stop the argon gas.
- 20) Use the stroke dial on the Gleeble control to zero the force reading (with a tolerance of ± 0.3 kN).
- 21) On the computer, double click “GLEEBLE-SHORTCUT”
- 22) Double click “Henry”
- 23) Double click the sample identification letter (ex: “F”)
- If the sample identification letter is not there, create a new folder with the sample’s identification letter
- 24) Double click your sample shape (i.e.: “Notch” or “Sub-Size”)
- If the sample shape is not there, create a new folder for the sample’s shape
- 25) Open any .qst file in this folder and edit your file accordingly as seen in Figure 27.



#	L	Time	Axis 1	Axis 2	Axis 3	Comment
1		System	Setup	Limits: Compression=-150mm, Force=50000kaf, Heat=100%		
2		Stress/Strain	Axial strain using Stroke, l = 12.00mm, d = 10.00mm			
3		Acquire	Force	Force.line	PowAngle	PTemp Stroke TC2 TC3
4		*				
5		*				
6		*				
7		Start	<input checked="" type="checkbox"/> Mechanical	<input checked="" type="checkbox"/> High	<input checked="" type="checkbox"/> Thermal	
8		Mode	Force(kN)	Wedge(cm)	TC2(C)	
9		Sample	10.0Hz			
10		00:05.0000	1.0			25 Lig
11		00:05.0000	0.0	0		25 Return to zero stress
12		00:00.5000		0		25 Hold to stabilize force
13		Sample	1000.0Hz			
14		00:01.4500	0.0	0		750 HR 500C/s
15		00:00.2000	0.0	0		800 Prevent overshoot, reduce HR to 250C/s
16		00:00.2000	0.0	0		800 Hold to stabilize temperature
17		Sample	5000.0Hz			
18		GSL>	set thermal to off			
19		GSL>	delay 5 msec			
20		Zero	Stroke			
21		Mode	Stroke(mm)	Wedge(cm)	TC2(C)	
22		GSL>	delay 5 msec			
23		GSL>	50 msec	if Force > 12 kN		
24		GSL>	go stop			
25		GSL>	end			
26		00:00.0600	30.00			25
27		Sample	10.0Hz			
28		00:05	35.00			25
29		End	<input checked="" type="checkbox"/> Mechanical	<input type="checkbox"/> High	<input type="checkbox"/> Ther	

When changing the program temperature, remember to edit the heating time to maintain constant heating rates. To do this, change the Time value in row 14 using the equation $Time = \frac{(Row\ 14, Axis\ 3) - (Row\ 12, Axis\ 3)}{500}$

In this example, $Time = \frac{750 - 25}{500} =$

To change your program temperature, edit the Axis 3 column of rows 14, 15, and 16 where rows 15 and 16 is your program temperature and row 14 is your program temperature minus 50

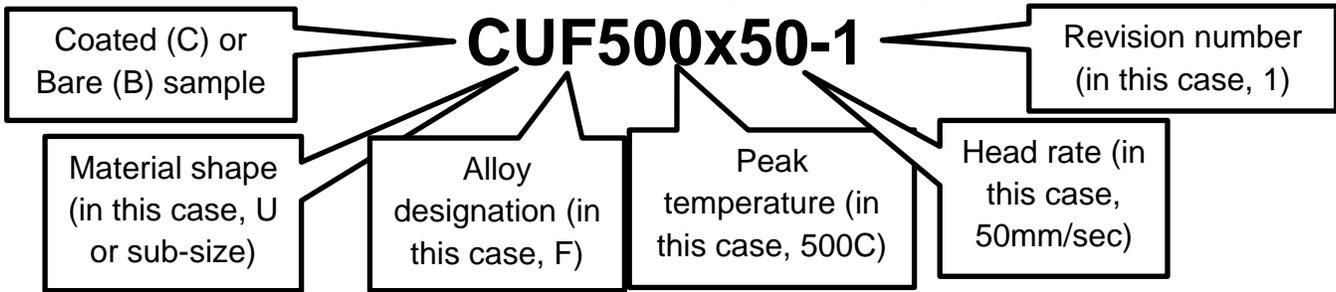
To change your head rate, edit the time (units in seconds) value in row 26 using the equation $Head\ Rate = \frac{Distance}{Time}$ where the value 30.00 mm in row 26

Be sure to check that all .qst files have this change prior to running. Otherwise when testing at strain rates of 500 and 50 mm/s the force will not zero prior to stroking.

Figure 27. Gleeble .qst file set-up



26) Hit "SAVE AS" and rename the file using the following naming convention:



27) Hit "RUN" on the computer.

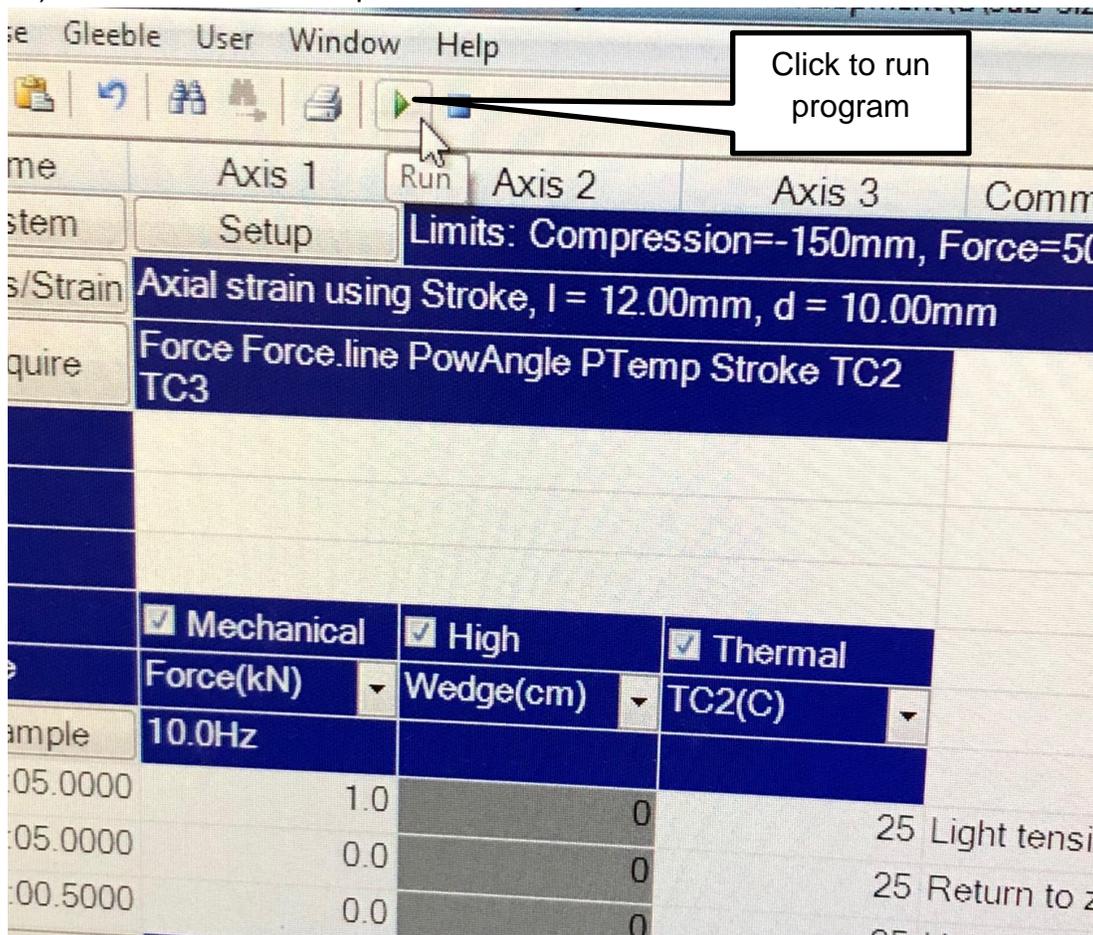


Figure 28. Gleeble .qst "RUN" button on the computer

28) After the Gleeble test is complete and the sample has fractured, let it cool in the chamber for two minutes to prevent oxidation.

- 29) Save the Gleeble data onto a flash drive or immediately upload to Buckeye Box.
- If you save the data to a flash drive, upload it to Buckeye Box immediately.

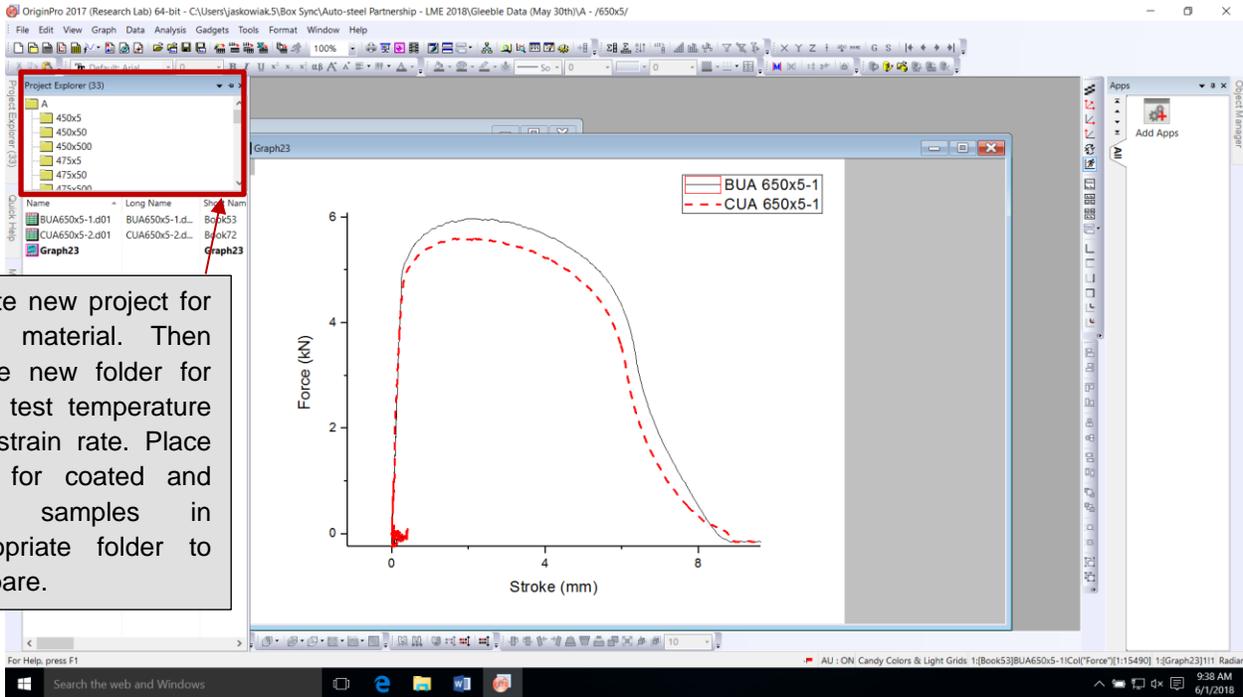


Figure 29. Origin folder creation

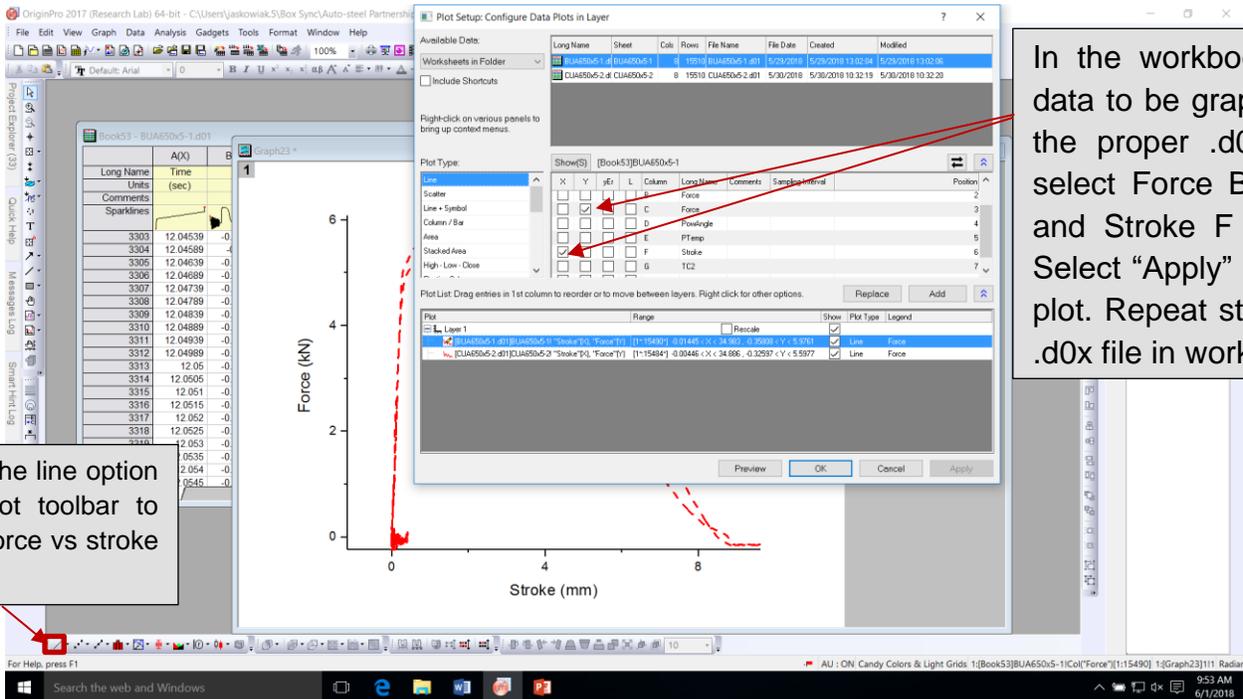


Figure 30. Origin force v. stroke plot creation

- 30) Open the .d0x file for the test to analyze the data in Origin. Ensure that the test ran and recorded the data to completion and verify that the TC2 temperature and program temperature at the maximum programmed temperature are within an acceptable value.



- a. Create a new project folder in origin each time a new material is tested.
 - b. Plot FORCE vs. STROKE in Origin.
 - c. Compare plots to check for liquid metal embrittlement and validity of data.
- 31) Vent the chamber by opening the chamber release valve on the machine.
 - 32) Press “DOOR RELEASE” and open the door to the Gleeble chamber.
 - 33) Loosen and remove the U-spacers between the sample mount and the Gleeble jaws.
 - 34) Place the aluminum tool between the two sample mounts as seen in Figure 31. Adjust the stroke dial as necessary to get the aluminum tool in place.

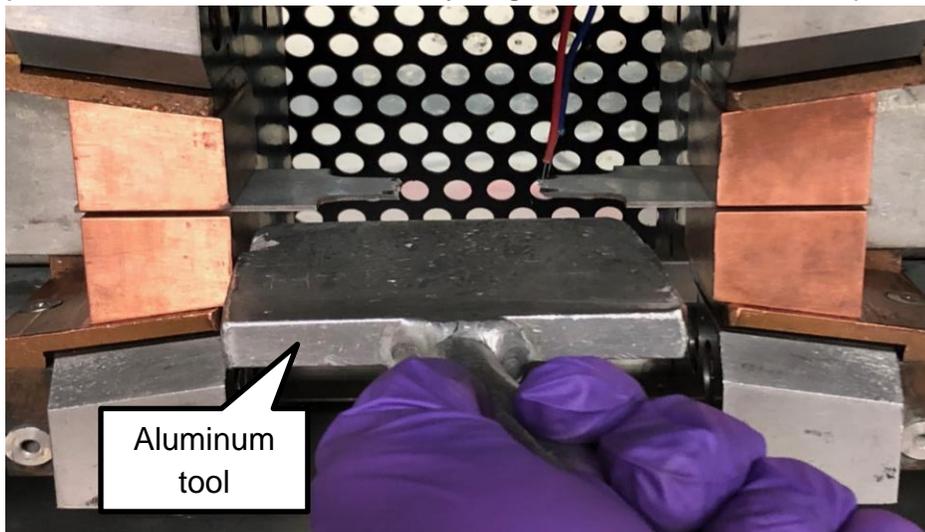


Figure 31. Aluminum tool for sample removal from Gleeble jaws

- 35) Press “RUN” and “MECHANICAL” on the Gleeble controls then slowly turn the stroke dial until one of the sample mounts loosens from the Gleeble jaws. Remove the sample half from the sample mount.
- 36) Place the aluminum tool against the remaining sample mount as seen in Figure 32. Adjust the stroke dial as necessary to get the aluminum tool in place.

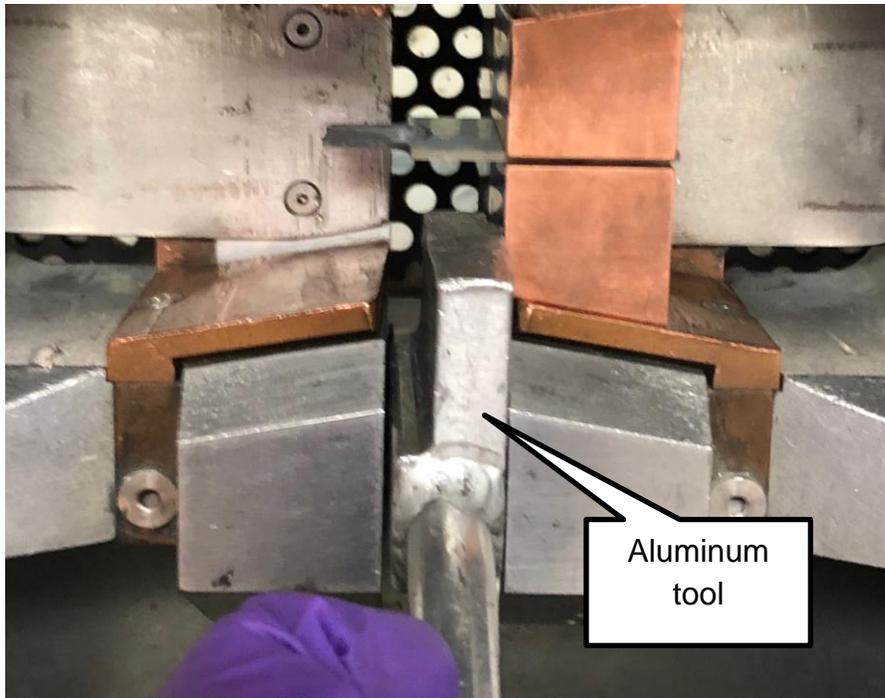


Figure 32. Aluminum tool to remove remaining sample mount from Gleeble jaw

- 37) Turn the stroke dial until the sample mount loosens from the Gleeble jaw. Remove the sample half from the sample mount.
- 38) Label one of the sample halves with a Sharpie, tape the parts together, and place in a small plastic bag. **NOTE:** Be careful not to scrape the crack interface against the other half of the sample.
- 39) Using the stroke dial on the Gleeble controls, return the Gleeble jaws to their original starting position.

10. Turning Off the Gleeble Machine

- 1) Press “MECHANICAL” and “RUN” until unilluminated (off) on the Gleeble controls.
- 2) Wipe down the Gleeble chamber with a “Kimwipe” and ethanol.
- 3) Close the Gleeble chamber door, shut the chamber valve, and press “VACUUM RUN.”
- 4) When the vacuum gauge above the Gleeble chamber reads 15 mmHg, press “VACUUM STOP” to stop the vacuum.
- 5) Press the “OFF” button on the Gleeble.
- 6) Tighten shut the argon tank behind the Gleeble.
- 7) Turn off the thermocouple welder.
- 8) Turn off the cooling unit next to the Gleeble.
- 9) Turn off the high bay chiller and remove the “GLEEBLE IN USE” sign.



11. Troubleshooting

1. The Gleeble is aborting my program mid-test (“46 – aborted” is likely displayed on the Gleeble console).

While there may be other problems causing this program to abort, it is likely that the force applied to your sample is exceeding a certain threshold. In the program on line 24, there is a command stating “event safeload if Force > X kN” which can be seen in Figure 33.

#	L	Time	Axis 1	Axis 2	Axis 3	Comment
1		System	Setup	Limits: Compression=-150mm, Force=50000kgf, Heat=100%		
2		Stress/Strain	Axial strain using Stroke, l = 12.00mm, d = 10.00mm			
3		Acquire	Force Force.line PowAngle PTemp Stroke TC2 TC3			
4		*				
5		*				
6		*				
7		Start	<input checked="" type="checkbox"/> Mechanical	<input checked="" type="checkbox"/> High	<input checked="" type="checkbox"/> Thermal	
8		Mode	Force(kN)	Wedge(cm)	TC2(C)	
9		Sample	20.0Hz			
10		00:05.0000	1.0	0		25 Light tension to seat jaws/grips
11		00:05.0000	0.0	0		25 Return to zero stress
12		00:00.5000	0.0	0		25 Hold to stabilize force
13		Sample	2000.0Hz			
14		00:01.4500	0.0	0		750 *HR 500C/s (test temp -50C)
15		00:00.2000	0.0	0		800 *Prevent overshoot, reduce HR to 250C/s (test t
16		00:00.2000	0.0	0		800 *Hold to stabilize temperature (test temp)
17		Sample	10000.0Hz			
18		GSL>	set thermal to off			
19		00:00.0050	0.0	0		25 Extra safeguard for: "set thermal to off"
20		GSL>	delay 5 msec			
21		Zero	Stroke			
22		Mode	Stroke(mm)	Wedge(cm)	TC2(C)	
23		GSL>	delay 5 msec			
24		GSL>	event safeload if Force > 8 kN			
25		GSL>	go stop			
26		GSL>	end			
27		00:06.0000	30.00	0		25
28		Sample	10.0Hz			
29		00:12.0000	35.00	0		25 Buffer to prevent rapid stopping of jaws
30		End	<input type="checkbox"/> Mechanical	<input type="checkbox"/> High	<input type="checkbox"/> Thermal	

Line 24 in the program, and the focus of this solution

Figure 33. Gleeble test code error “safeload”

This means that if the force exceeds “X”, the program will abort. The current safeload, not shown in Figure 33, is set at 35 kN, but previous programs may have a safeload set at a lower level (8 kN in Figure 33). If your event safeload is less



than 35, increase it to 35 and try again. If it is set at 35 kN and you are still encountering this problem, open your program and follow these steps:

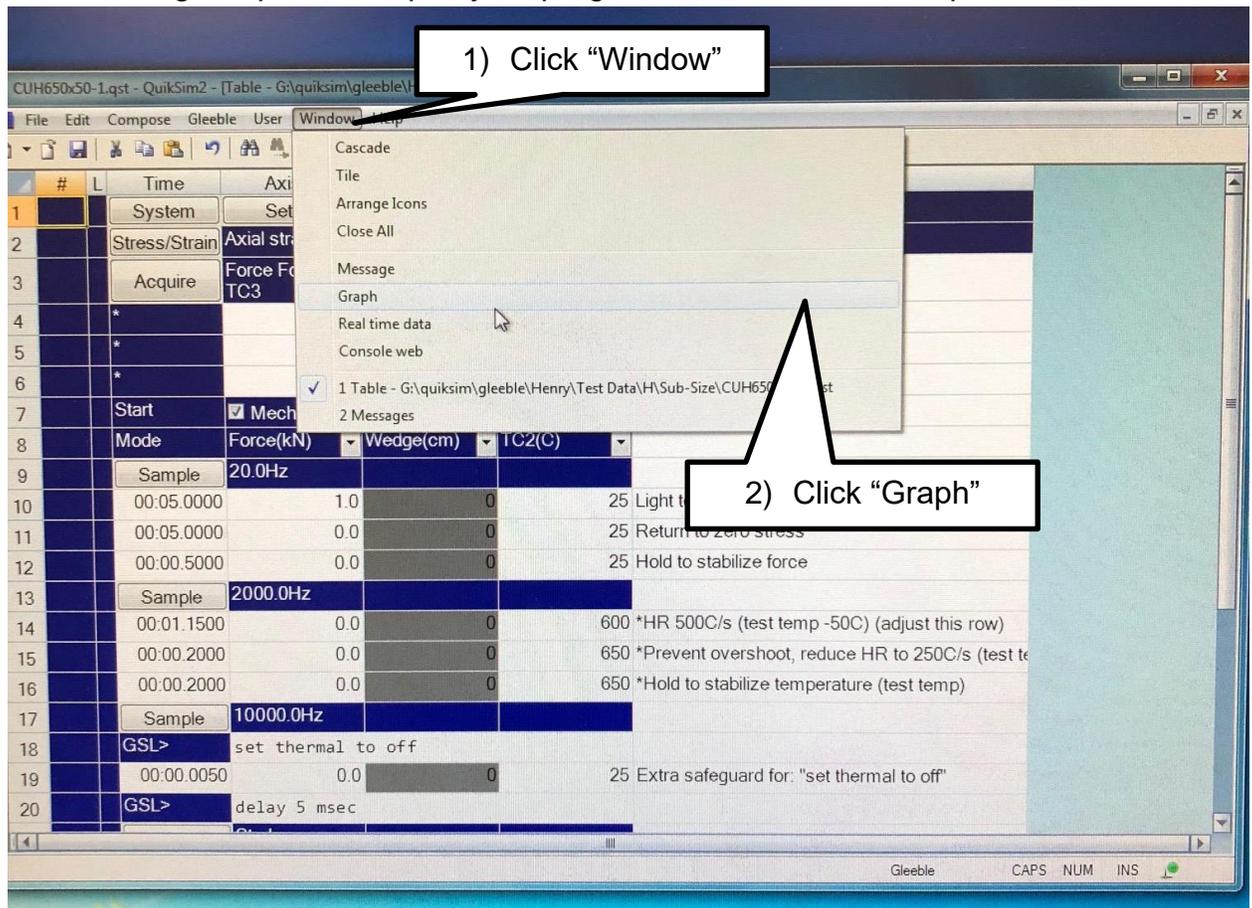


Figure 34. Gleeble troubleshooting manual step one and two

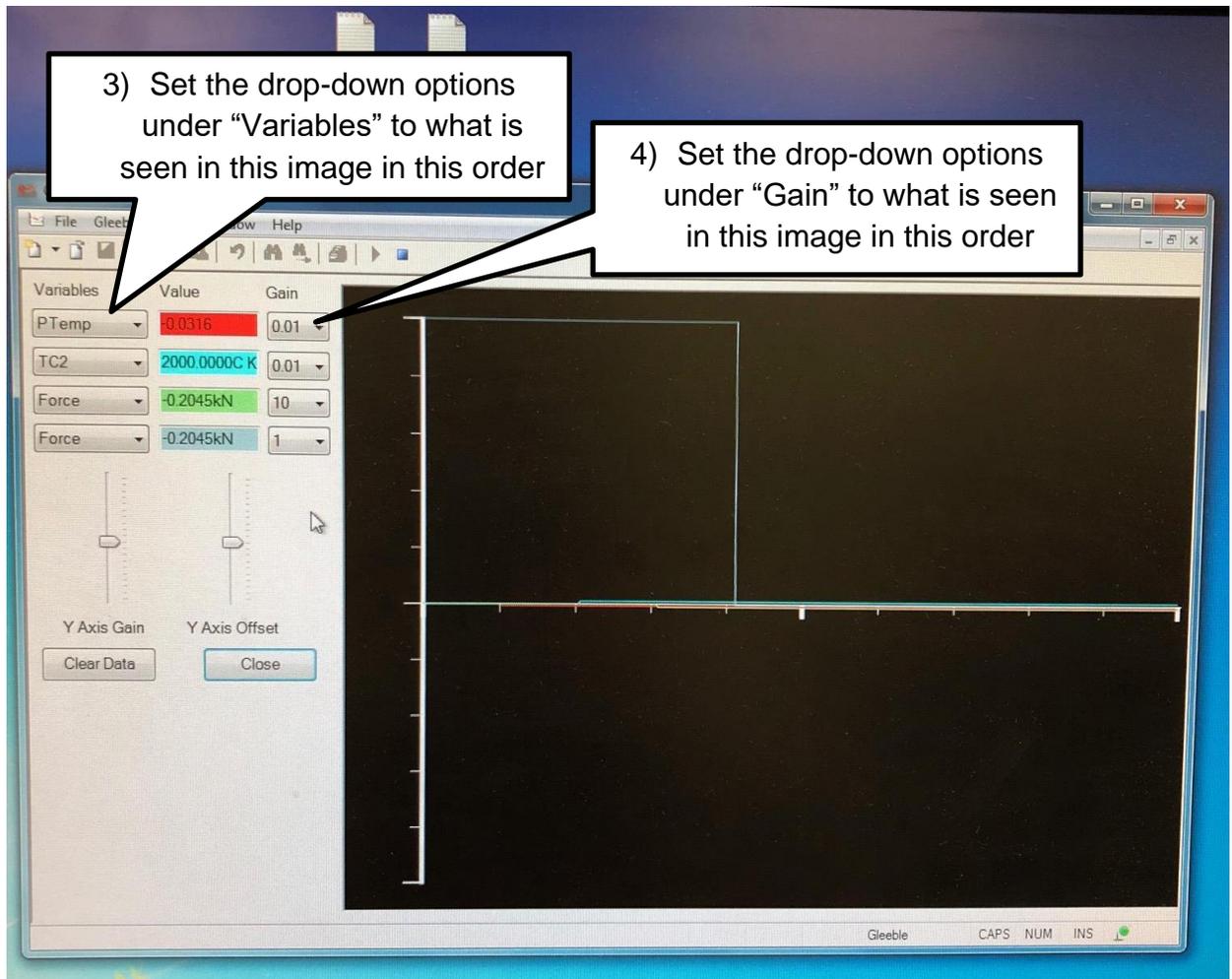


Figure 35. Gleeble troubleshooting manual step three and four

Following these steps will bring up a live stream of data acquired from the Gleeble. This is important because if the program is aborted, the Gleeble deletes the last 5 seconds of data in the event of an abort, meaning you would not be able to review your useful data after the test is ran. Run the test and monitor the force value on the live graph. Take note of the highest force reached in the test on the monitor. Using this force, go back to line 24 in your program and edit it so that your force is above the force exerted on the graph. A good number to enter is 5 kN above the maximum force that you monitored.

2. The Gleeble runs my test, but when I go back to analyze my data, it appears as if the testing data was cut short and not reading the entire test.

This issue is similar to the problem encountered with the program aborting. When a certain force is exceeded, the Gleeble attempts to abort the program. However, if you are conducting the test at a higher stroke rate (500 mm/sec or sometimes 50 mm/sec), the test will "finish" before the Gleeble can abort the program. When



the Gleeble tries to abort a program, it will delete the last 5 seconds of recorded data from your test even if the test still finished, resulting in the “cut-off data” problem. To solve this problem, follow the same solution as described in [Problem 1](#) with the Gleeble aborting the program.

3. The vacuum on the Gleeble is pumping down slow or is not achieving the level of vacuum that I need for my test.

This indicates that there is a leak in your vacuum system. There are four likely reasons why this is occurring:

First, open the front door and check both the O-ring and the back of the front door where the O-ring would sit for debris. Debris can cause leaks and prevent a vacuum. To remove debris from the door, take a paper towel with ethanol and wipe down the area where the O-ring would sit on the door to remove all of the debris. If debris is on the O-ring itself, run your finger across the entire length of the O-ring. The debris will stick to your finger and allow you to achieve a clean seal. After you wipe down the O-ring, be sure to reapply vacuum grease to the O-ring.

Second, check the O-ring to see if there is sufficient vacuum grease on it. There should be a visible coating across the O-ring’s entire length. If there is not, apply a new coat of vacuum grease to the O-ring.

Third, check the clear panels on the front, back of the Gleeble, and ensure there is enough vacuum grease across the O-rings behind these panels. There should be a healthy amount of vacuum grease to prevent outside air from leaking into the Gleeble chamber. If there is not, seek Ed, Antonio Ramirez, or someone else in removing the panel to apply a new layer of grease to these panel O-rings.

Finally, check the inside of the Gleeble chamber for moisture, coolant, or any other liquid. Liquid can keep a vacuum from being formed and must be removed. To do this, take a paper towel soaked in ethanol and wipe down wherever liquid is present in the chamber. You may need to remove/open parts in the machine to fully clean these areas. Seek Ed or Antonio Ramirez for help if you believe you need more access in cleaning the chamber of liquid.

If none of these solutions work, talk with Ed or Antonio and see what they believe your problem could be. You may have to contact DSI for help on their machine.

4. I am getting oxidation on my parts after running a test. What can I do to prevent this?

Oxidation can potentially skew our testing results. If you are getting oxidation on parts there are four likely causes.

First, there may be a small leak in the air hose connecting the Gleeble chamber to the argon tank. This can allow oxygen into the chamber causing oxidation. As of



May 10th, 2018, this is the most likely cause of oxidation issues if you are using the “Tank Fill” control on the Gleeble console. Finding a way to properly rig the argon tank directly to the Gleeble chamber safely and without leaks may vary from one situation or another, so talk to Antonio Ramirez about how to safely do this. If you are already using a method that connects the argon tank to the Gleeble chamber (such as running a hose from the argon tank to the vent valve on the Gleeble), your oxidation issues may be coming from another source.

Second, the samples may have picked up grease on the surface between cleaning and testing, even if the grease may not be immediately visible. To remove this grease, simply run down the sample with ethanol before welding thermocouples on, and then wipe them down with lint-free wipes. The sample should be dry before welding the thermocouples on.

Third, the samples may not be properly cleaned after being spot etched or stripped bare. If you believe this is the cause, rinse the samples with tap water, then spray with deionized water, then spray with ethanol, and then use a blow-dryer to dry the samples and prevent rusting. In the future, make sure you follow proper cleaning procedure following spot etching or stripping.

Finally, there may be a leak in Gleeble chamber itself. To test this, vacuum down the Gleeble to around 5×10^{-1} torr. Let the Gleeble sit at this vacuum level for a while. The pressure in the Gleeble will likely slowly creep up above this 5×10^{-1} torr level, which is acceptable. However, if the pressure in the chamber rises at a rapid rate, you likely have a substantial leak in the Gleeble chamber. Seek someone like Antonio Ramirez or Ed for advice on what to do. You may have to contact DSI for assistance.

- 5. When I plot the Force vs Stroke graphs, I get a weird jump in the data as seen in Figure 36:**

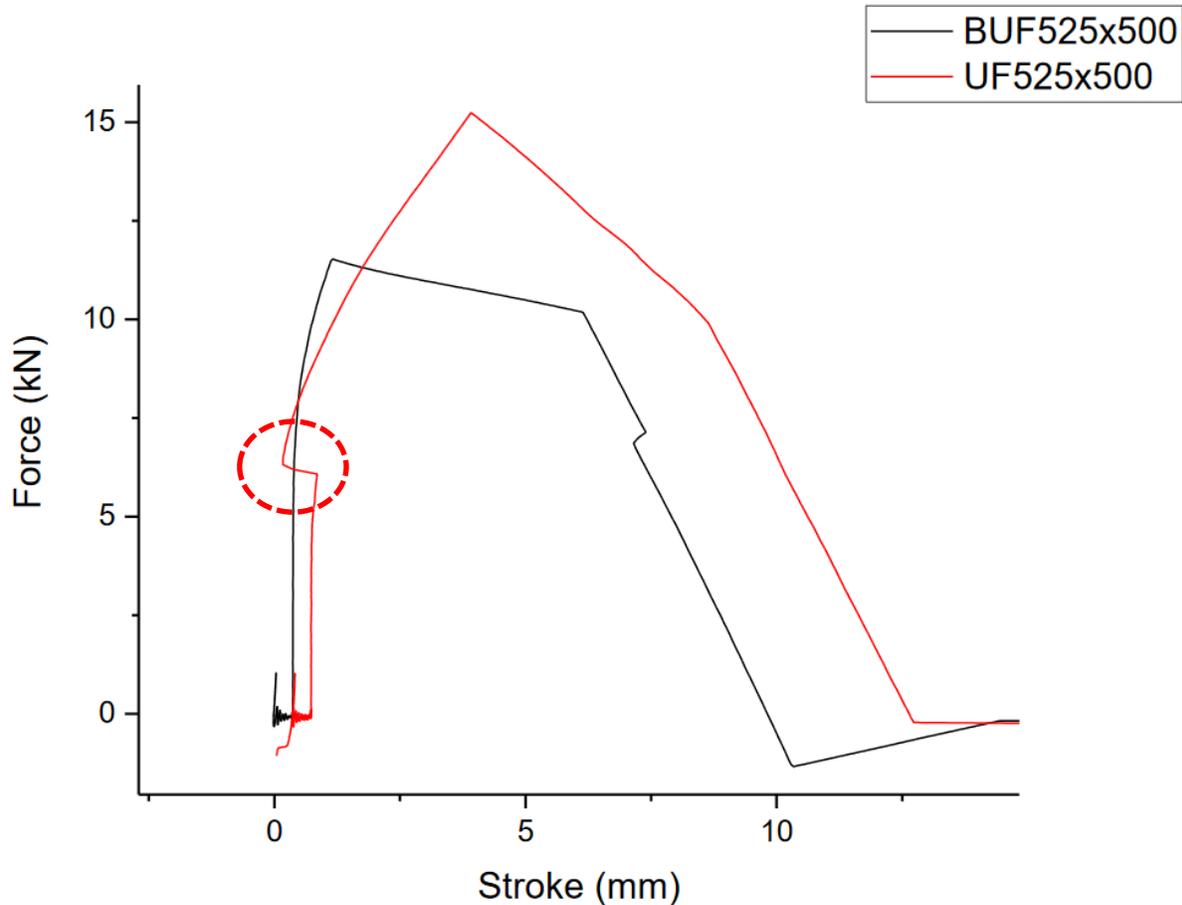


Figure 36. Force v. Stroke curve example

This is caused by the Gleeble initiating stroke movement before it has the time to zero the stroke position. This typically happens at a stroke rate of 500 mm/sec due to how fast the samples are pulled at this rate. To fix this problem, go to line 23 (see Figure 37) in the Gleeble program. It should read "delay X msec". This delays the pulling of the sample by X seconds, giving the Gleeble time to zero in the stroke. As of May 10th, 2018, this delay is set at 50 msec, however, this may need to be incrementally adjusted if issues are still being encountered. Older programs may have a different delay time (Figure 37 has a delay of only 5 msec)



#	L	Time	Axis 1	Axis 2	Axis 3	Comment
1		System	Setup	Limits: Compression=-150mm, Force=50000kgf, Heat=100%		
2		Stress/Strain	Axial strain using Stroke, l = 12.00mm, d = 10.00mm			
3		Acquire	Force Force line PowAngle PTemp Stroke TC2 TC3			
4		*				
5		*				
6		*				
7		Start	<input checked="" type="checkbox"/> Mechanical	<input checked="" type="checkbox"/> High	<input checked="" type="checkbox"/> Thermal	
8		Mode	Force(kN)	Wedge(cm)	TC2(C)	
9		Sample	20.0Hz			
10		00:05.0000	1.0	0	25	Light tension to seat jaws/grips
11		00:05.0000	0.0	0	25	Return to zero stress
12		00:00.5000	0.0	0	25	Hold to stabilize force
13		Sample	2000.0Hz			
14		00:01.4500	0.0	0	750	*HR 500C/s (test temp -50C)
15		00:00.2000	0.0	0	800	*Prevent overshoot, reduce HR to 250C/s (test t
16		00:00.2000	0.0	0	800	*Hold to stabilize temperature (test temp)
17		Sample	10000.0Hz			
18		GSL>	set thermal to off			
19		00:00.0050	0.0	0	25	Extra safeguard for: "set thermal to off"
20		GSL>	delay 5 msec			
21		Zero	Stroke			
22		Mode	Stroke(mm)	Wedge(cm)	TC2(C)	
23		GSL>	delay 5 msec			
24		GSL>	event safeload if Force > 8 kN			
25		GSL>	go stop			
26		GSL>	end			
27		00:06.0000	30.00	0	25	
28		Sample	10.0Hz			
29		00:12.0000	35.00	0	25	Buffer to prevent rapid stopping of jaws
30		End	<input type="checkbox"/> Mechanical	<input type="checkbox"/> High	<input type="checkbox"/> Thermal	

Adjusts delay time to allow Gleeble to zero the stroke position

Figure 37. Gleeble stroke delay screenshot

6. My program will not start even though I followed the procedure correctly.

This is likely caused by the machine not reading the chamber door as shut (even if it appears to be shut). If this is the case, there will be a yellow circle next to "DoorSafety" on the Gleeble monitor. To fix this, vent the chamber, open the door, and then shut it again, this time making sure that the circle next to DoorSafety is green. This indicates that the Gleeble reads the door as shut and should allow you to run the test. You will need to pump down again before you run your test to prevent oxidation.

If this does not fix your problem, contact someone such as Ed or Antonio Ramirez for help as there may be something else wrong with the machine or program.



12. Analysis

1. During Testing

- a. During testing the priority analysis to be conducted is to visually determine how much LME is present. This is done by visually inspecting the sample and determining if deep, shallow, threshold, or no LME is present.
 - i. LME is usually found in higher temperatures and strain rates, then gradually falls off as temperature and strain rate decrease.
 - ii. LME should not be present in the bare samples. If LME is found in a bare sample it likely means that the Zinc layer was not properly removed prior to testing and will need to be rerun.

		Material B: CR340Y590T-DP-HDG																
Head Rate mm/s		450	475	500	525	550	575	600	625	650	675	700	725	750	775	800		Color Legend:
500					453	451	449	447	445	442	440	438						Deep LME
50																		Shallow LME
5																		Threshold
																		No LME
																		Bad Data
		Material B: (Coating Removed)																
Head Rate mm/s		450	475	500	525	550	575	600	625	650	675	700	725	750	775	800		
500					452	450	448	446	444	443	441	439						
50																		
5																		

Figure 38. LME Green Line Data: Material B

- iii. Figure 38 shows visual analysis for Material B. It is color coated to show deep, shallow, threshold, and no LME.
- iv. Examples of LME:

1. Deep

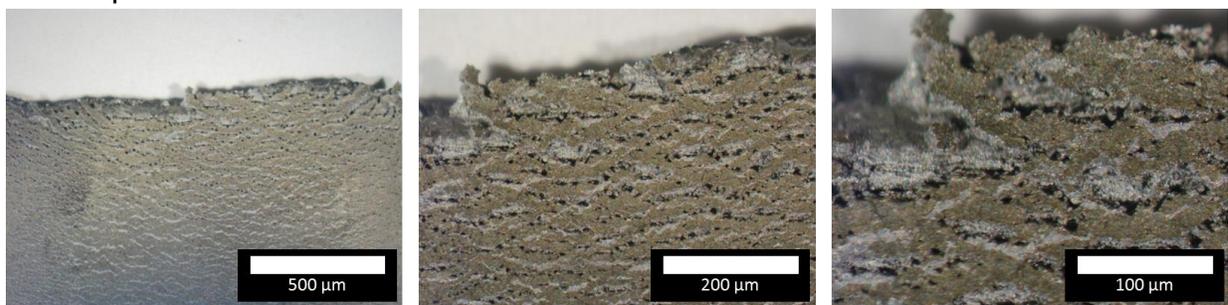


Figure 39. Deep LME

- Deep cracks clearly visible to the naked eye
- Brittle surface fracture
- Significant cracking throughout center of the sampe

2. Shallow

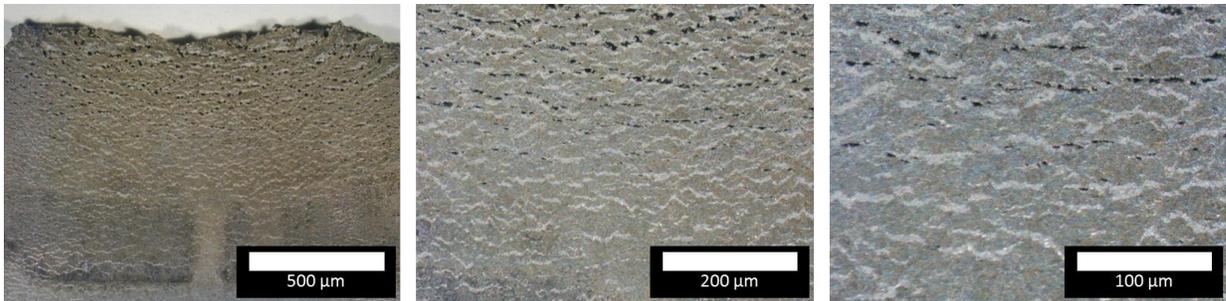


Figure 40. Shallow LME

- Deep cracks visible the naked eye, less when compared to deep LME
- Illuminated magnifier confirms significant cracks present
- Crack penetration fails to penetrate through entire sample thickness

3. Threshold

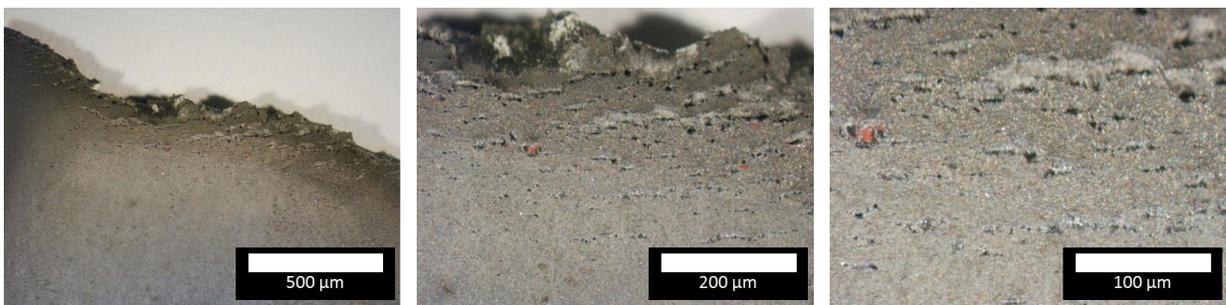


Figure 41. Threshold LME

- Deep cracks visible through the use of illuminated magnifier
- Cracks concentrated near sample fracture surface
- Cracks do not penetrate through sample thickness

4. No LME

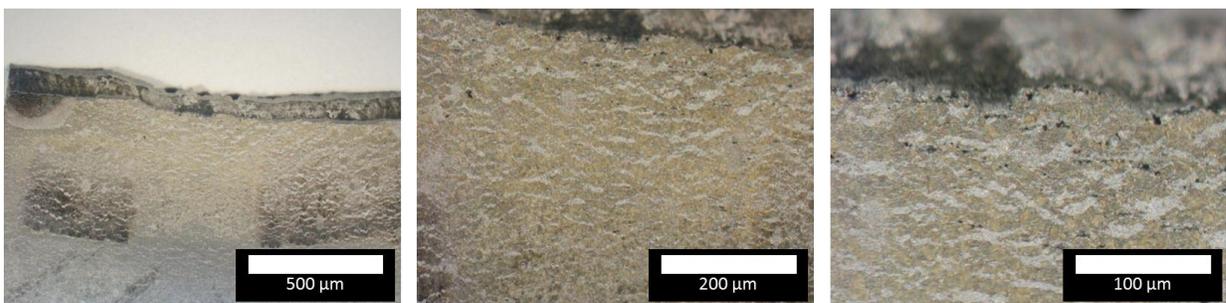


Figure 42. No LME

- No cracks present under visual inspection by illuminated magnifier



- b. Samples must be analyzed to determine if oxidation is present. Oxidation will lead to inaccurate force curves due to the weakening of the sample.
 - i. Example of oxidation:



Figure 43. Oxidation example

- Appears visibly blue in color
- Small amounts of oxidation along fracture surface are acceptable – this indicates oxidation occurred post-testing

2. Post Testing

a. Matlab Processing

- i. After running the sample on the Gleeble and confirming a good test, place the desired .d0# extension file into a folder to run in Matlab.

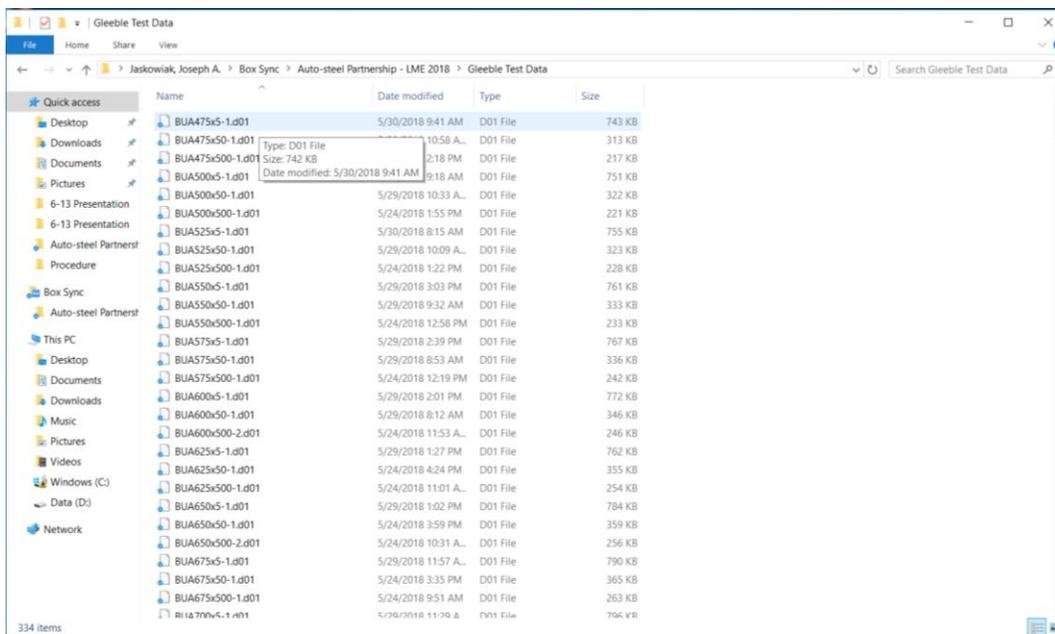


Figure 44. Matlab folder for data processing



- ii. It is recommended to use the “Gleeble Test Data” folder on the box sync account and run Matlab from that directory.
- iii. Once all desired files are placed in the folder to be processed in Matlab, open the Matlab application. Ensure the current version of Matlab has the proper functions in your library. Open and run the functions “ASPMultiData_SmoothAvg” and “SmoothAvg” to organize the data in Matlab. NOTE: These functions are setup for Matlab2016 or newer.

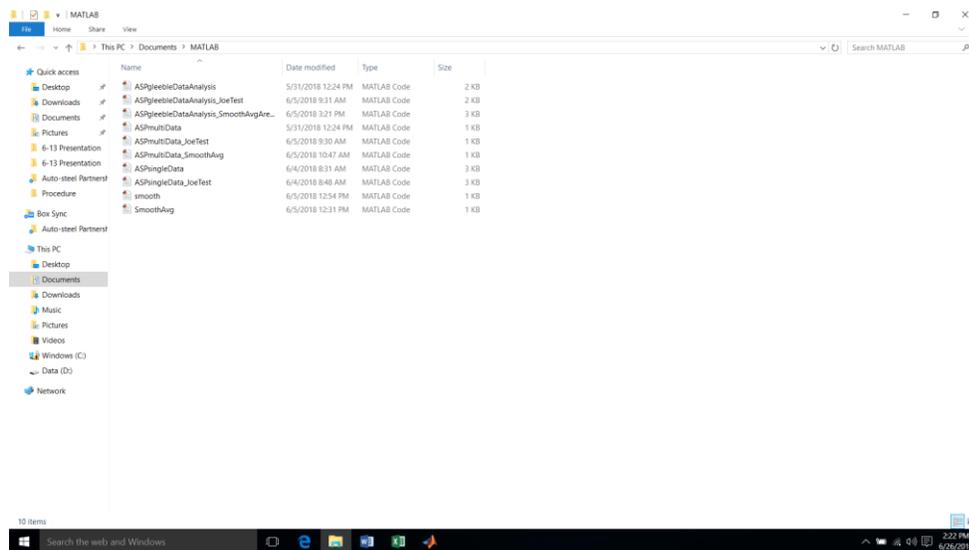


Figure 45. Matlab function folder

- iv. Change the Matlab directory path to the desired folder to execute the Matlab script.

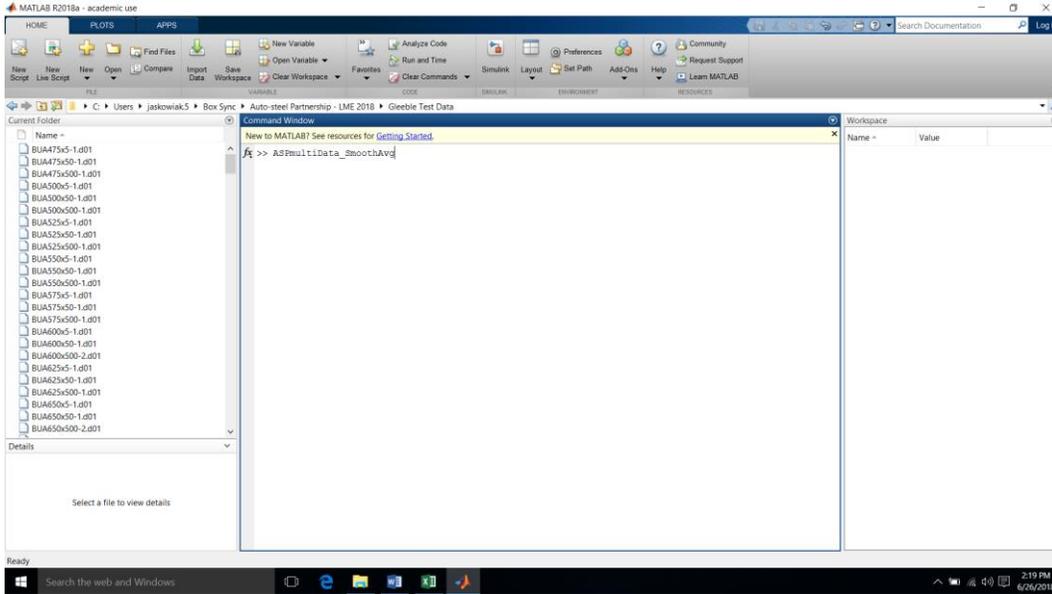


Figure 46. Matlab workbook directory path and function call

v. Copy results of Matlab function to an excel workbook.

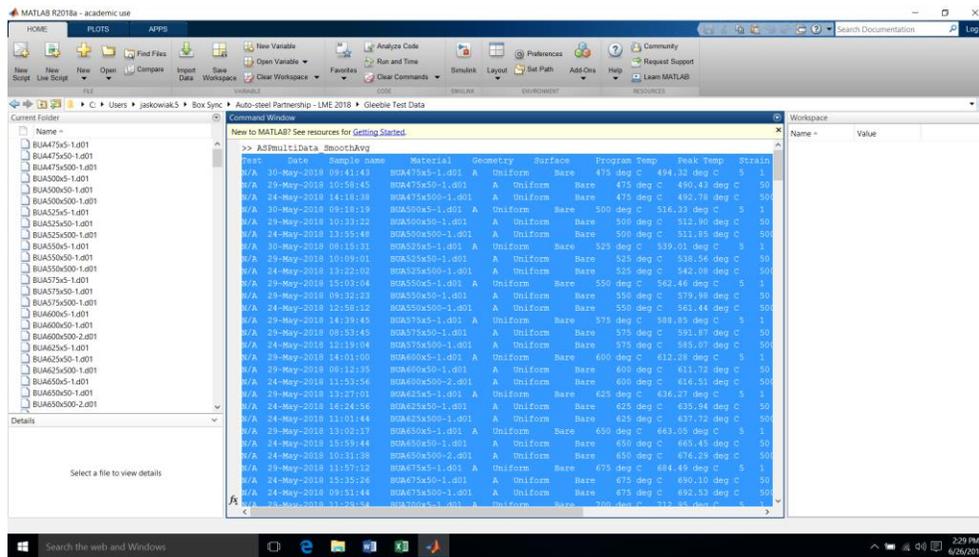


Figure 47. Matlab workbook data results

b. Quantitative Analysis in Excel

i. Open the Excel file “Matlab Test Results ## - ## - #####” with most current date.



ii. Paste Matlab data into the table on sheet titled “Raw Data”.

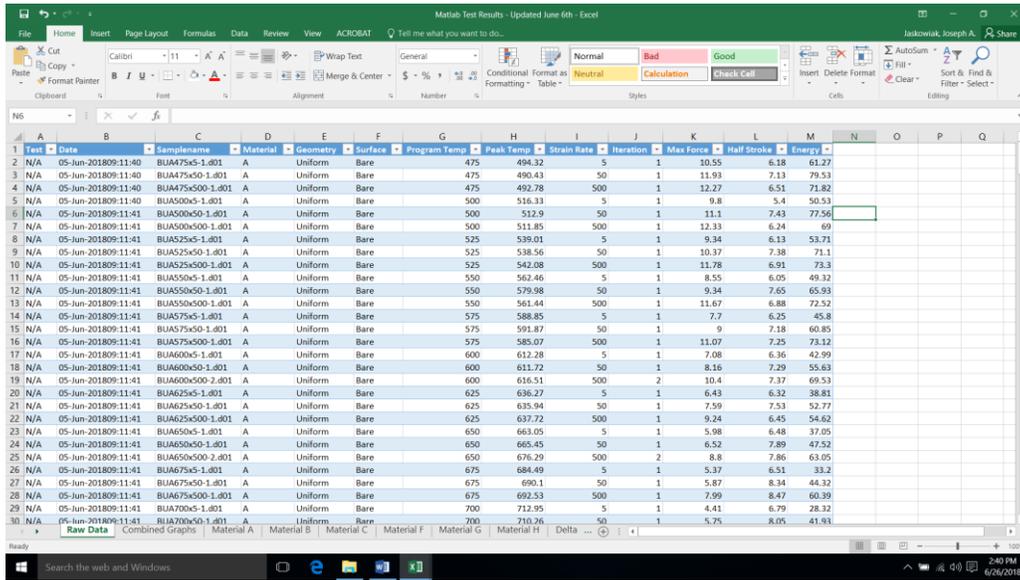


Figure 48. Excel workbook “Raw Data” screenshot

iii. Excel will populate all data for deltas and quantitative LME analysis in respective material sheets.

iv. Each material sheet has LME characterization based off of a comparison between the base sample and coated sample. Parameters to quantitatively determine LME are based off of qualitative analysis.

v. LME Calculation:

$$\frac{(\text{Coated Value} - \text{Bare Value})}{\text{Bare Value}} * 100\% = \text{Percent Change of Delta}$$

vi. LME Characterization:

1. Deep LME: $\Delta \leq -25\%$
2. Shallow LME: $-25\% \leq \Delta \leq -15\%$
3. Threshold LME: $-15\% \leq \Delta \leq -7.5\%$
4. No LME: $-7.5\% \leq \Delta$