Updates on Impregnation and Epoxy Studies at FNAL

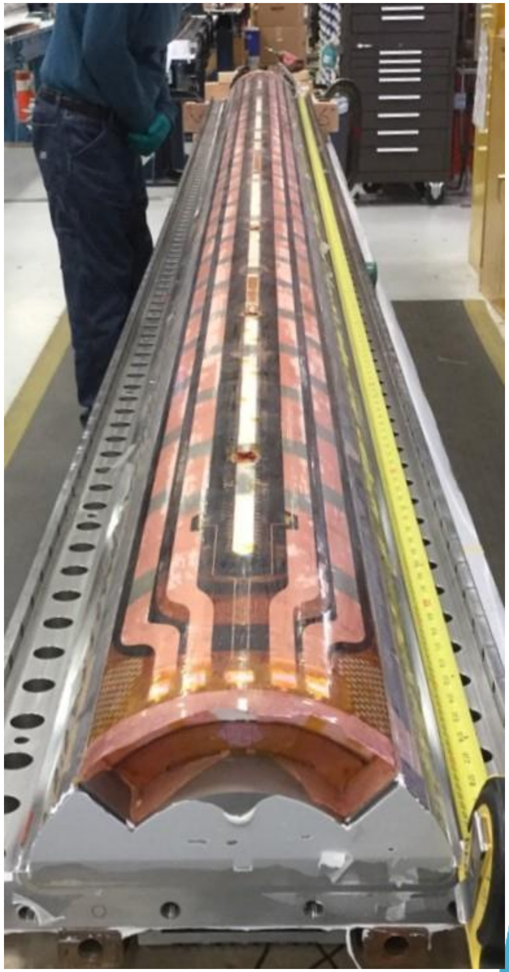
Steve Krave
Fred Nobrega

9th HL-LHC CM Meeting — 16 October 2019

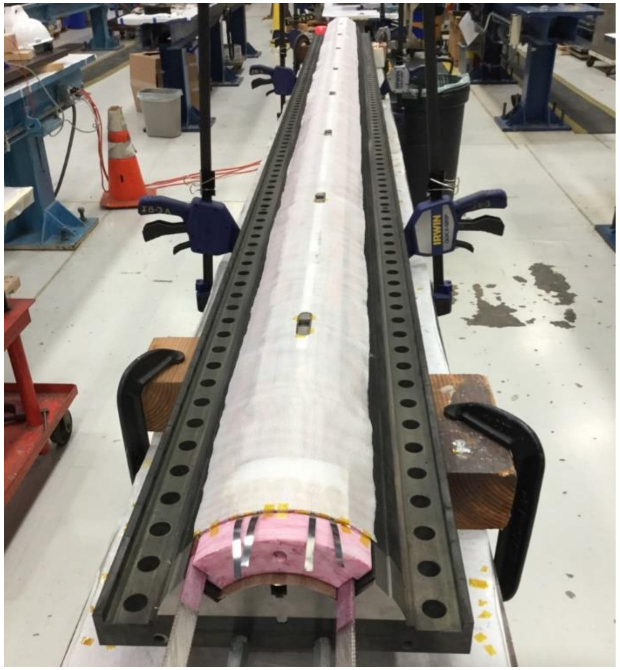
- Impregnation Procedure Review
- Investigation into Tg
- Process evolution
- Look at Shear Strength of Nb_3Sn Composite
- Mold Release Peel Testing

Coil Impregnation Process

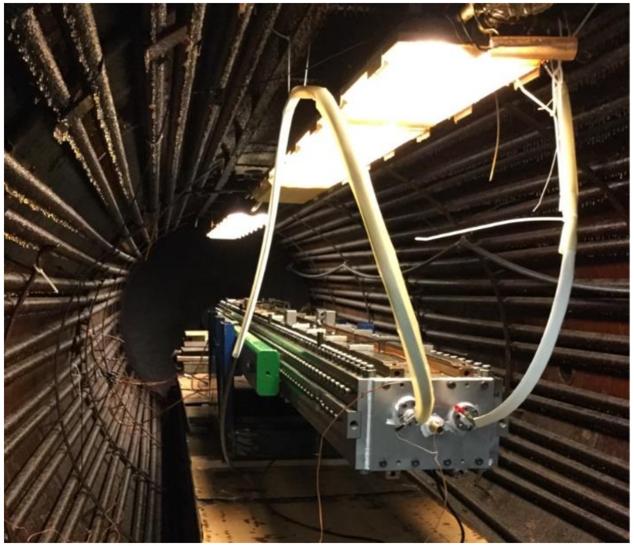
- Goals of impregnation
  - Provide electrical insulation
    - Epoxy fills the voids in the glass insulation and allows for reasonably high insulation dielectric strength
  - Provide mechanical support
    - Epoxy fixes turns in place such that pre-load may be applied in a safe fashion and the magnet will maintain a magnetic cross section
    - Prevent conductor movement during powering to reduce magnet quenching
  - Allow for efficient handling of coils
    - Without impregnation Nb_3Sn coils are extremely delicate

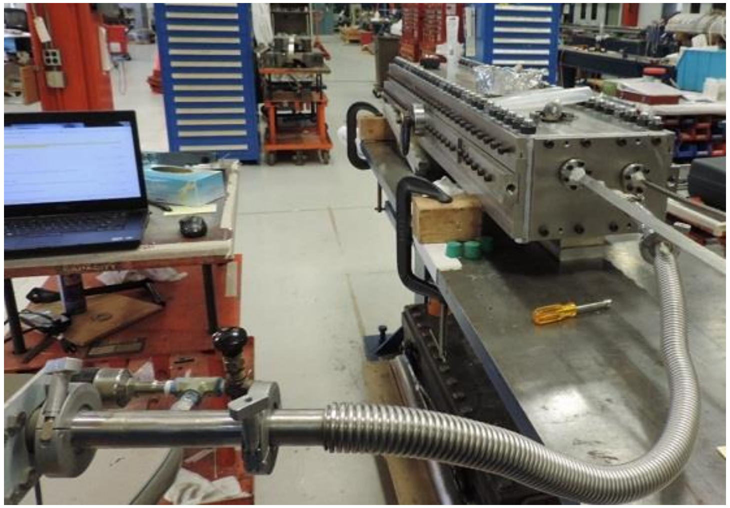


Impregnation Outline



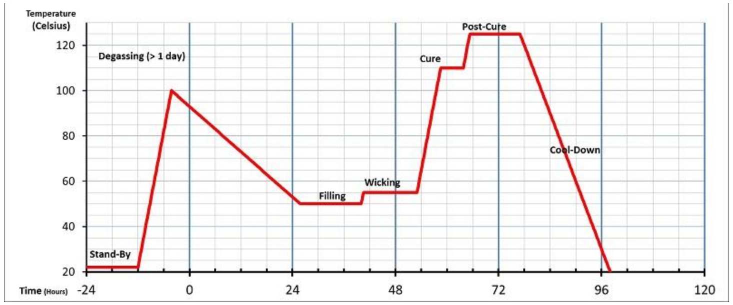
- Coil Packaging
  - Mold release tooling
  - Install Quench Heaters
  - Install fiberglass
- Coil Leak Test





Coil Outgassing

- Coil is outgassed
  - QXF Coils are run through a pre-impregnation outgassing cycle: Fast ramp to 100C and long cool down (~30 Hours)
  - After outgassing cycle coil volume at inlet side is roughly 10X Chamber pressure
  - Tooling in ~30-90 mTorr (0.04-.12 mBar)
    - Ultimate chamber pressure depends on quality of door seal and behavior of other components external to coil circuit

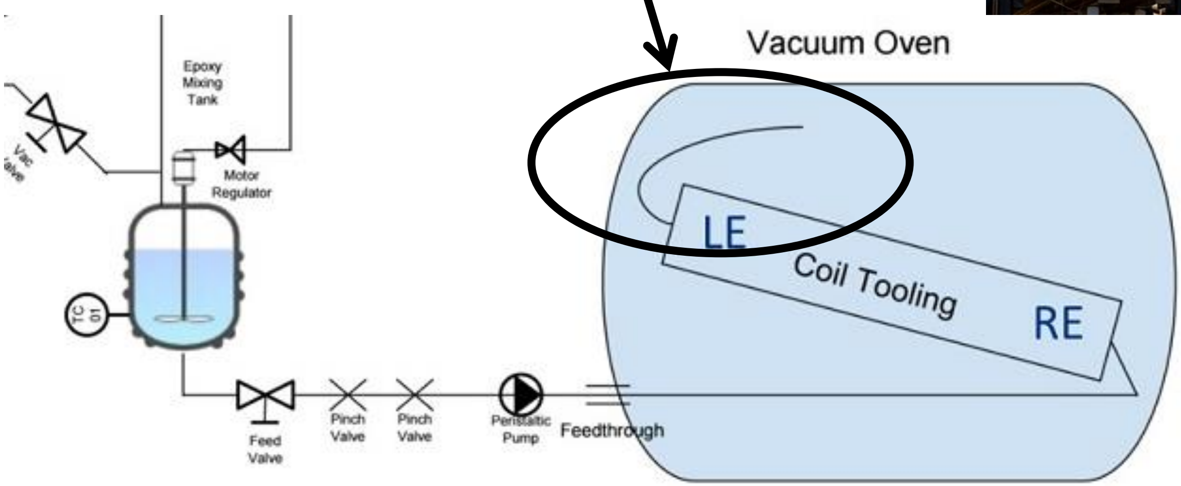


Filling

- Epoxy mixed and degassed for 1 hour at 55°C
- Injection into coil by Peristaltic pump at 10 ml/min effective flow rate
  - ~.75 liters of epoxy per meter of coil ~3.5 Liters of epoxy in the coil. ~5 hours of fill time
- After fill is complete, Chamber is let to atmosphere and any resin level change is recorded.

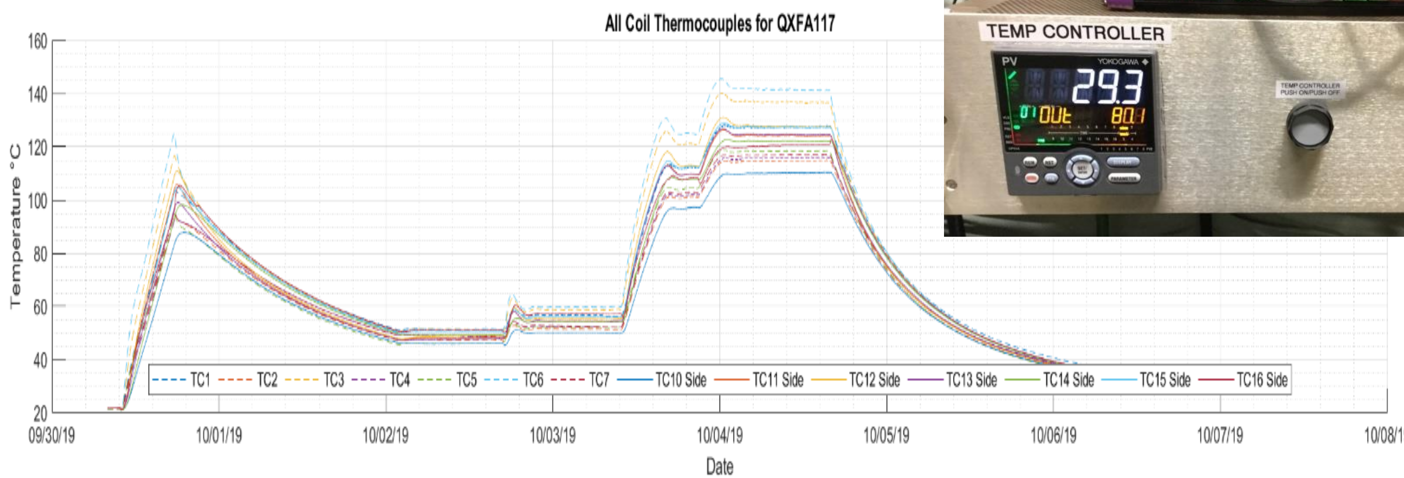
Coil in Vacuum Oven

300 ml Epoxy Reservoir
(Overflow tube)



Coil Curing

- Coil Curing follows CTD recipe
  - 110 °C for 5 hours
  - 125 °C for 16 hours
- Temperature control uses locally cooler area to drive tooling

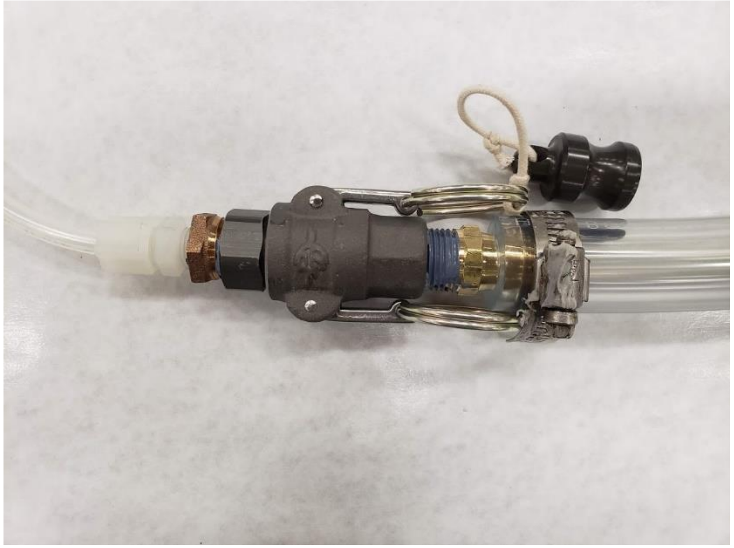


Process Evolution

- Throughout LARP and AUP, we have been seeking to reduce variability and risk from coil to coil by
  - Reducing un-necessary steps
  - Integrating fixed processes
  - Reducing technician fabricated or configured items
  - Reducing sensitivity to process variables

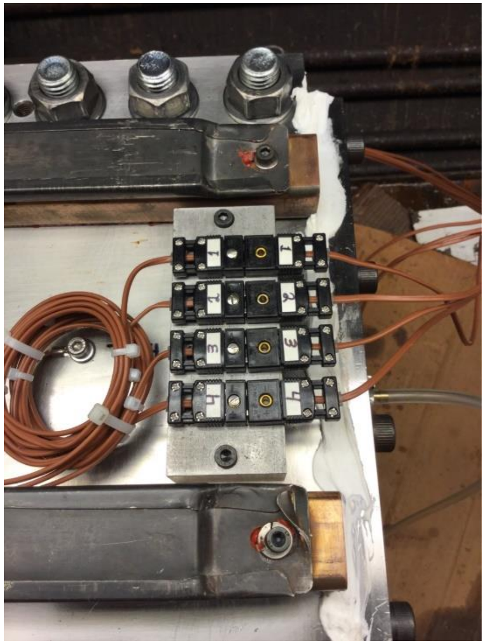
Process Evolution: Feedthroughs and Plumbing

- All “valves” between epoxy tank and coil are of the pinch type.
- No breaks in line with semi-disposable feedthrough design
- Herbie Clips are handy and disposable. They avoid some of the troubles of hose clamps

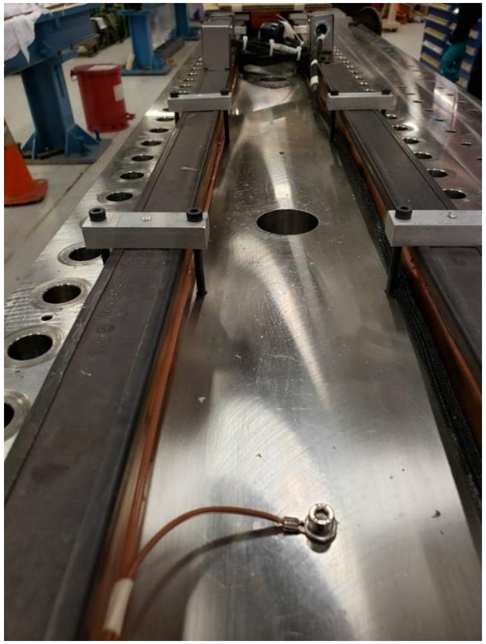


Process Evolution: Instrumentation Thermocouples and wiring

Instrumentation Thermocouples and wiring



Robust, Permanently mounted thermocouples



Well-Dressed cabling and wire routing

Improved granularity on measurement.

- Soon to come: Permanent, robust mounting

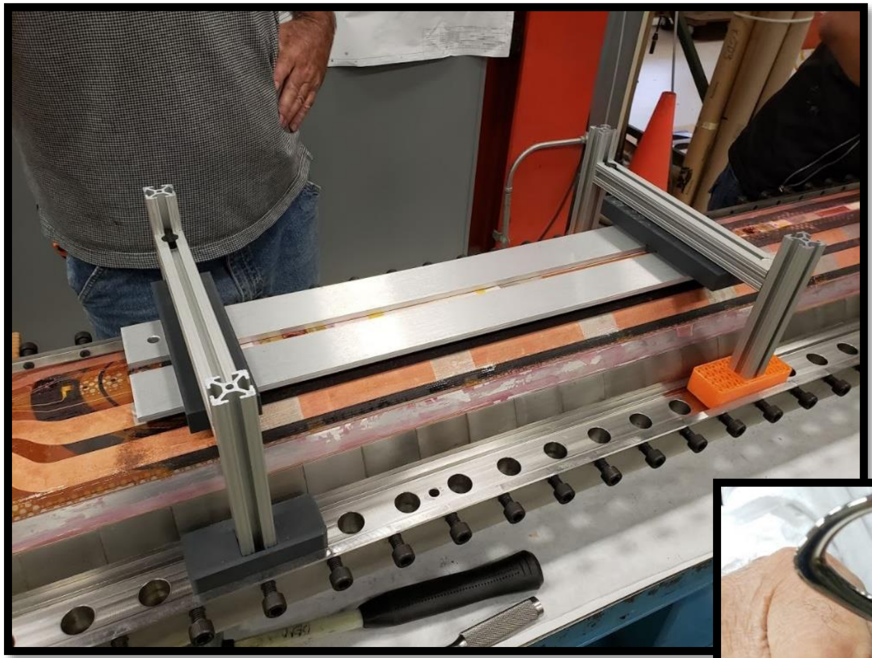
Coming Next Coil: automatically logged vacuum gages

Process Evolution: Mixing and Degassing

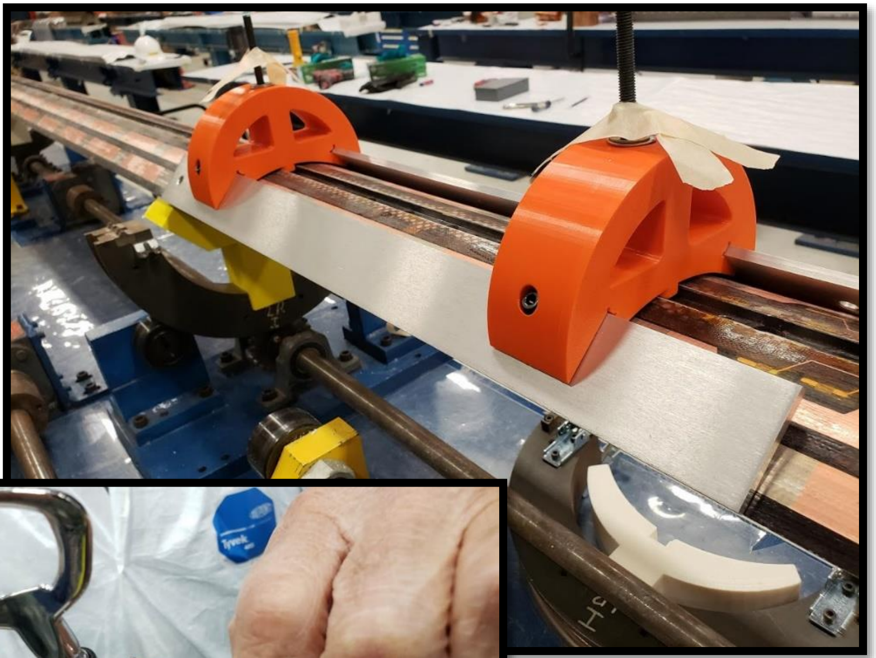
- A Bigger Step
  - A purpose built mixing-degassing vessel is due next week.
  - Integrated system
  - Less user configuration required

Process Evolution: Coil Clean Up

Pole Trimming Fixture



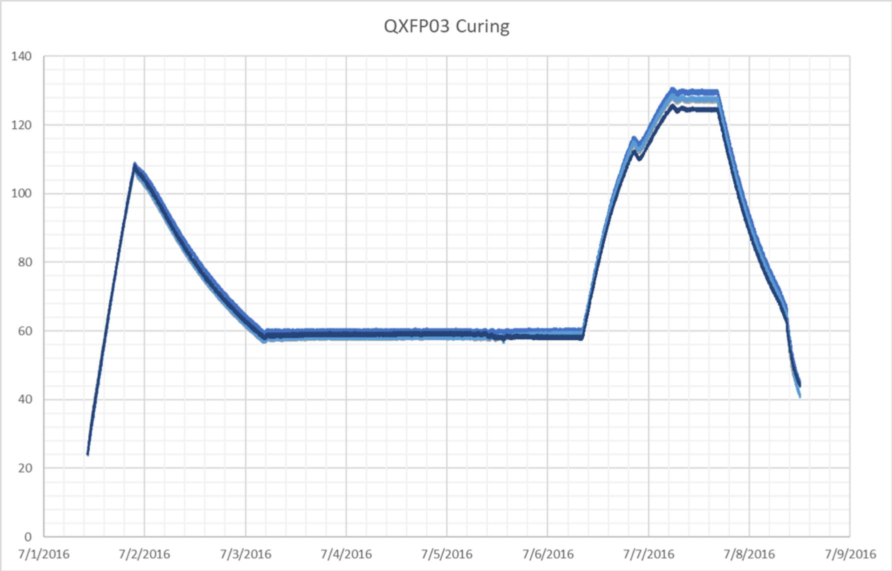
Midplane Trimming Fixture



Multipurpose Plug Puller

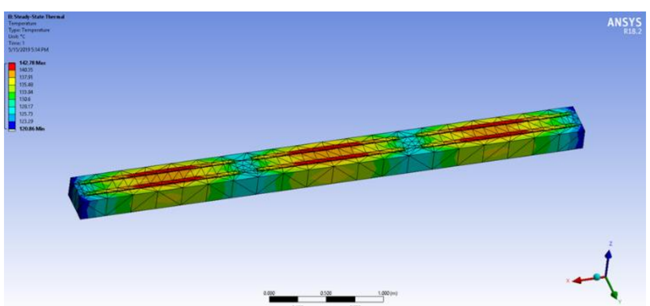
Coil Temperature Distribution

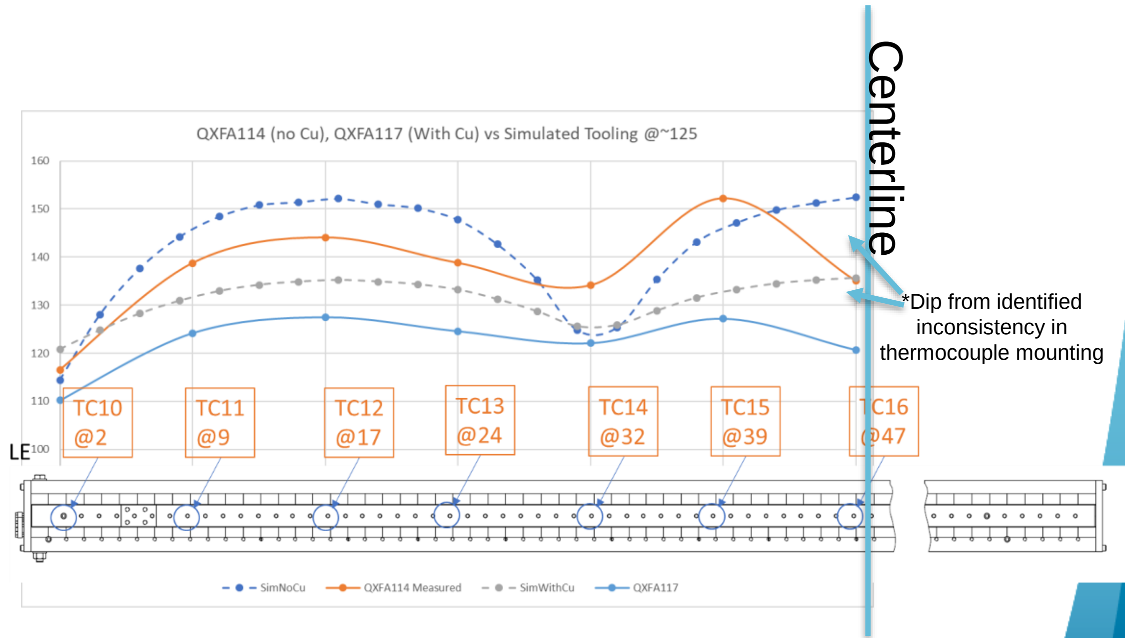
- Tooling is long and mostly has essentially Fixed power to mass and cross section
  - Except the ends
  - So we added a little insulation to compensate for added heat loss
  - Things generally look good



But conditions were different than assumed

- Periodic heaters and laminated blocks make for a worse than originally anticipated temperature distribution
  - Important to sample at >Nyquist rate
- Added copper bars (old magnet conductor) to double longitudinal thermal conductivity (and roughly halve gradients).



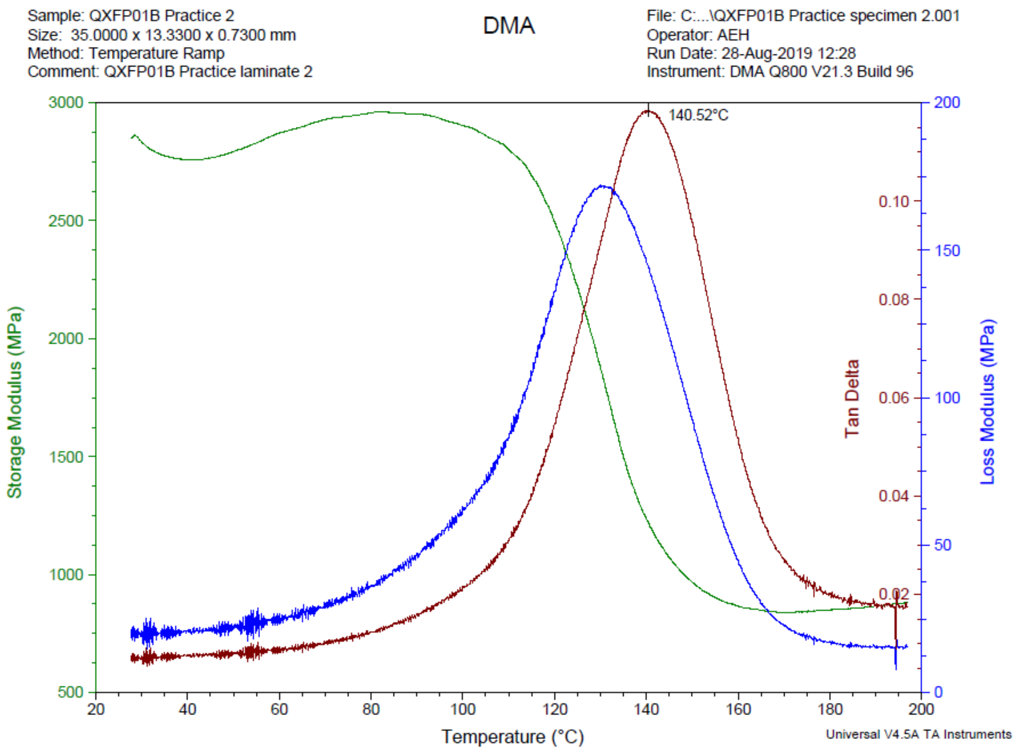


Examining Tg as a result of the above

- Use DSC and DMA tests to determine Tg along the length of coils QXFP03 and QXFP04 from magnet MQXFP1b.
  - Investigate possible impregnation process differences
  - Possibly use DSC for impregnation QA/QC
- DSC and DMA tests have been performed on cable insulation between turns from QXFP1b. DSC results were not usable from this source.
- A DSC test using a sample from the coil OD of QXFA108 along the titanium pole was successful.
- Determine the Tg offset between DMA and DSC.
- Side note: High Tg may not be desired as it results in larger differential thermal stresse.

DMA Test Results

- CTD101K Tg = 140°C ± 5°C based on DMA test.*
- DSC has an offset from DMA and will be established after coil sample testing.

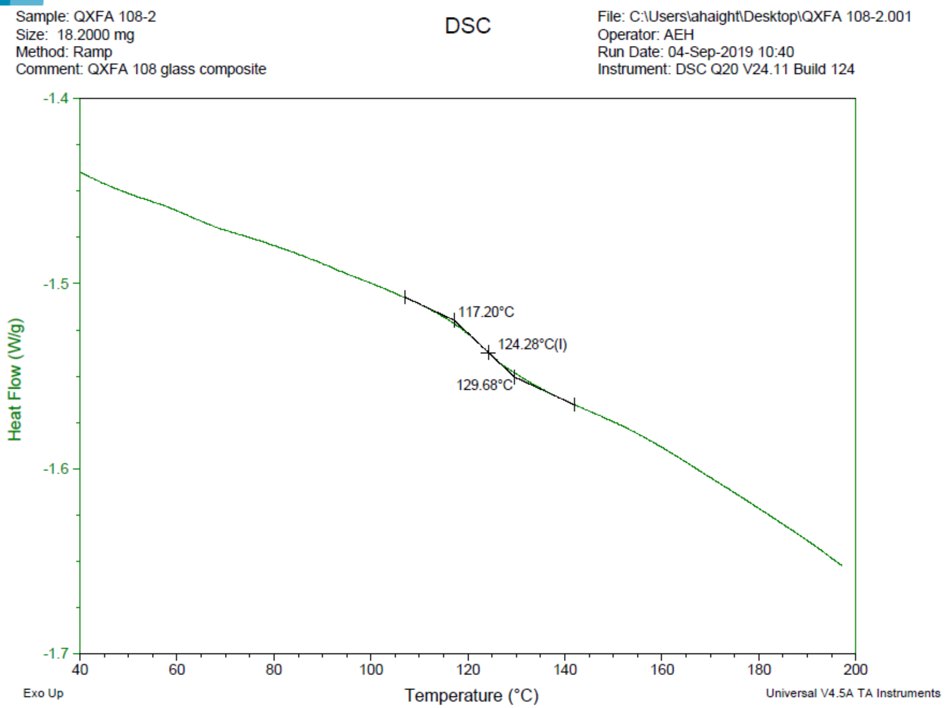


DMA Samples from QXFP1b

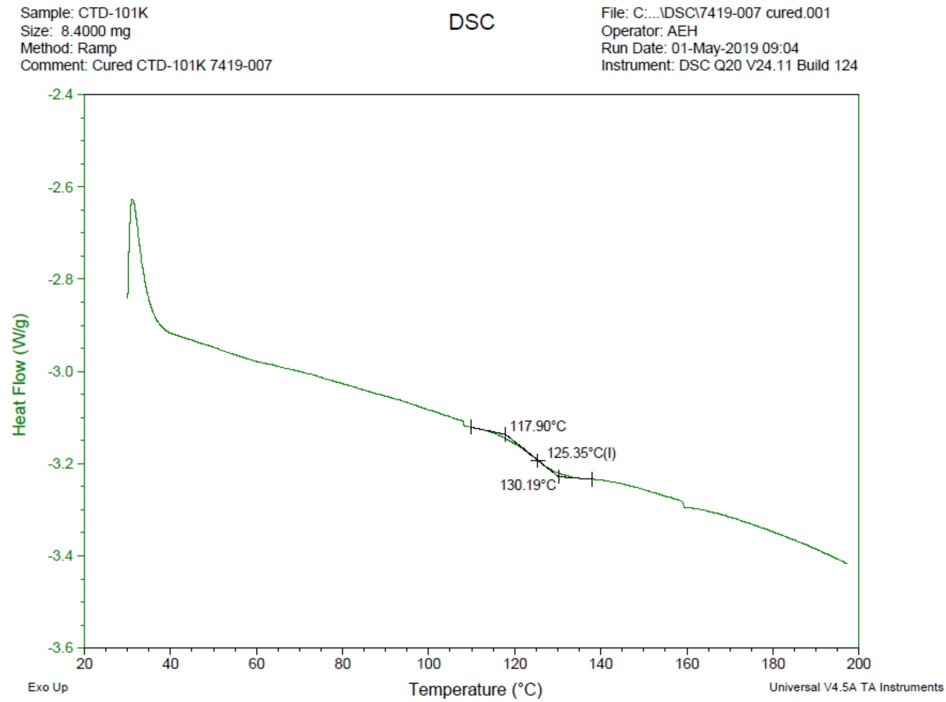
* Paul Fabian, VP of Operations, CTD

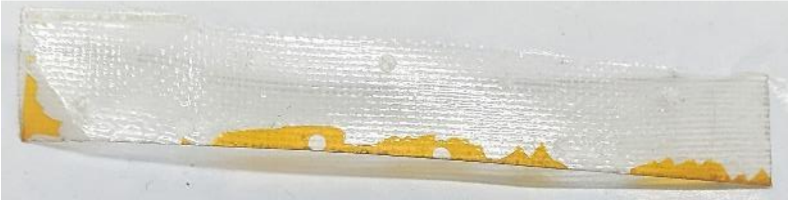
DSC Test Results

QXFA108 sample, Tg=124.3°C



CTD-101K neat resin, Tg= 125.4°C

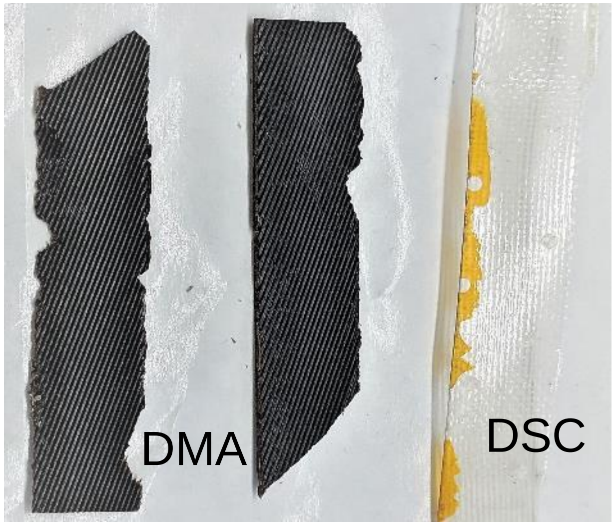


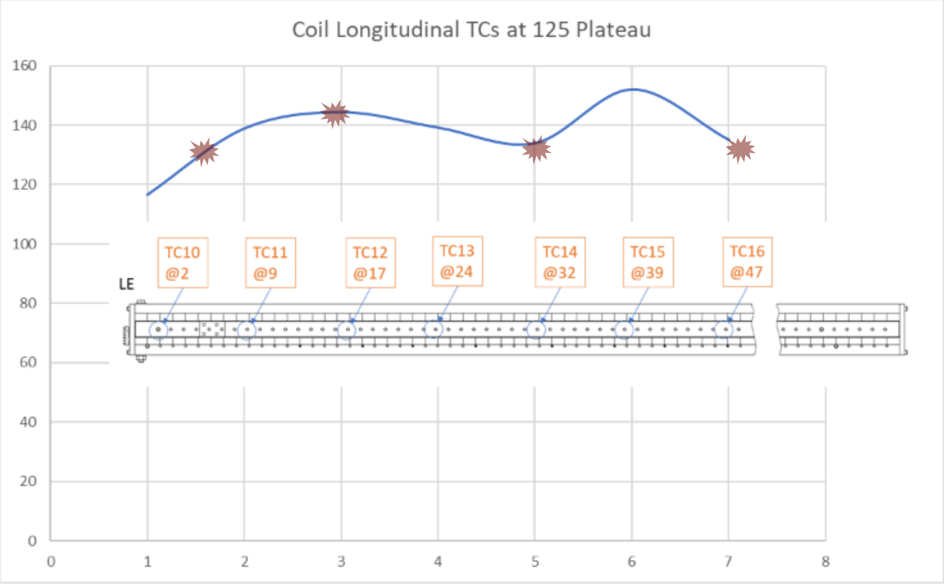


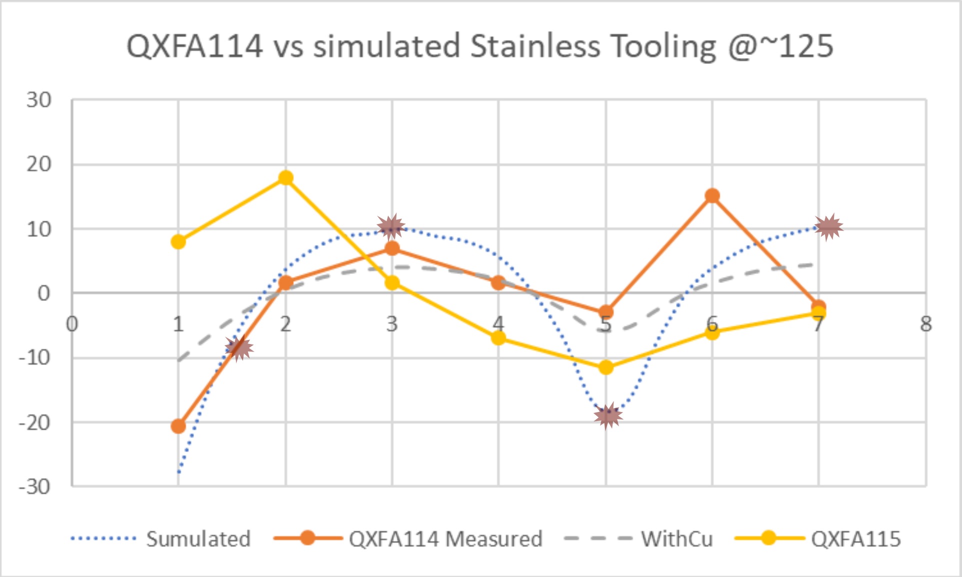
DSC Sample from QXFA108

QXFP Sample Locations

- Obtain 7 samples from high and low temperature locations along the length of the coil
  - DMA – between turns
  - DSC – along pole OD



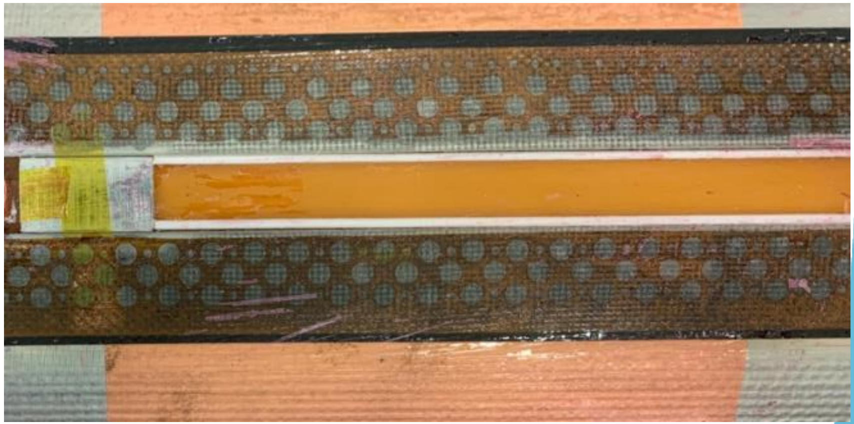


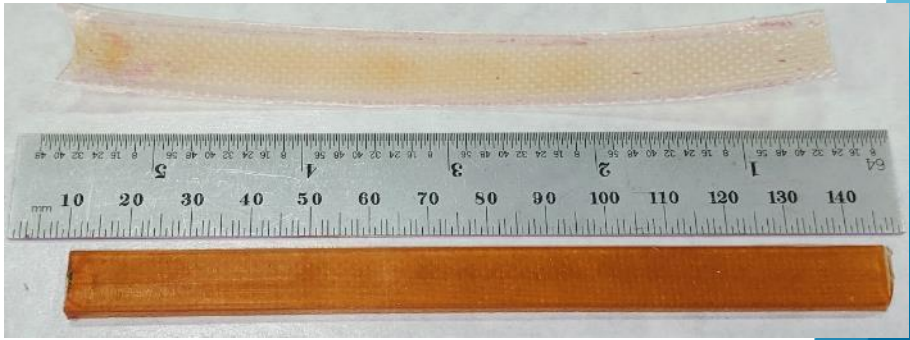


Neat Resin Sample From QXFA116

- Epoxy samples available from coil lead end, center, and return end.
- Neat resin sample cross section:

10.3 mm x 3.4 mm





Tg Summary & Plan

- Preliminary DMA test results indicate coil Tg within tolerance.
- Developed viable samples from existing coils
  - Destructive test
    - Cable insulation between turns
  - Non-destructive tests
    - Neat resin samples from coil OD alignment slot
    - S2 glass from coil OD

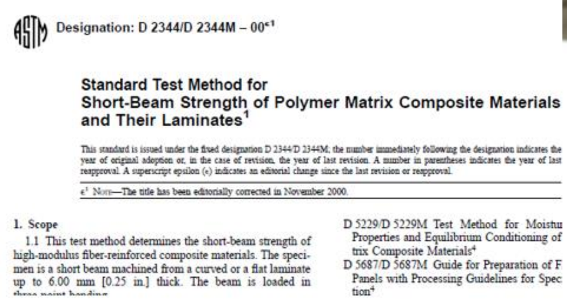
- Plan
  - Obtain 14 samples along the length from coils QXFP03 & QXFP04
    - 7x samples for DSC
    - 7x samples for DMA
  - Test neat resin samples from QXFA116
    - 3x samples for DSC
    - 3x samples for DMA
  - Finalize test sample count (34) and submit purchase requisition for testing at CTD.

Other Testing: Short Beam Test

Why Short Beam ~~Shear~~ Testing

An appropriate test to quantify any changes in mechanical performance

- **Binder volume is small so rule of mixtures tells us that modulus is unlikely to be affected.**
- **Compressive behavior is dominated by cable properties**
- **Tensile just wont work**
- **Shear**
  - Binder is likely to affect interface behavior, therefore shear properties
  - Generally shear samples are complicated geometry that can not be realized with cable
  - Short Beam strength uses a symmetric beam with a span to thickness ration of ~4.
    - This is do-able



Designation: D 2344/D 2344M – 00[ε1]

Standard Test Method for
Short-Beam Strength of Polymer Matrix Composite Materials and Their Laminates[1]

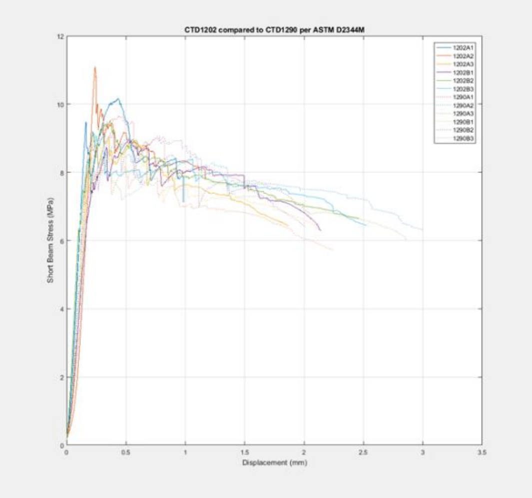
This standard is issued under the fixed designation D 2344/D 2344M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε[1] Note—The title has been editorially corrected in November 2000.

1. Scope

1.1 This test method determines the short-beam strength of high-modulus fiber-reinforced composite materials. The specimen is a short beam machined from a curved or a flat laminate up to 6.00 mm [0.25 in.] thick. The beam is loaded in three-point bending.

D 5229/D 5229M Test Method for Moistu[re] Properties and Equilibrium Conditioning of [Ma]trix Composite Materials[4]
D 5687/D 5687M Guide for Preparation of F[lat] Panels with Processing Guidelines for Spec[imen Prepara]tion[4]



Fermilab

	Average	σ	CV
1202	**9.68 MPa**	0.811 MPa	8.37%
1290	**8.91 MPa**	0.502 MPa	5.18%

Observations from SBS Testing

Shear Testing and Energy Release

Assuming that K ≈ constant and some basic spring stuff and conserving energy:

F=kx

x=F/k

E=1/2*k*x^2

E=1/2*k*F^2/k^2

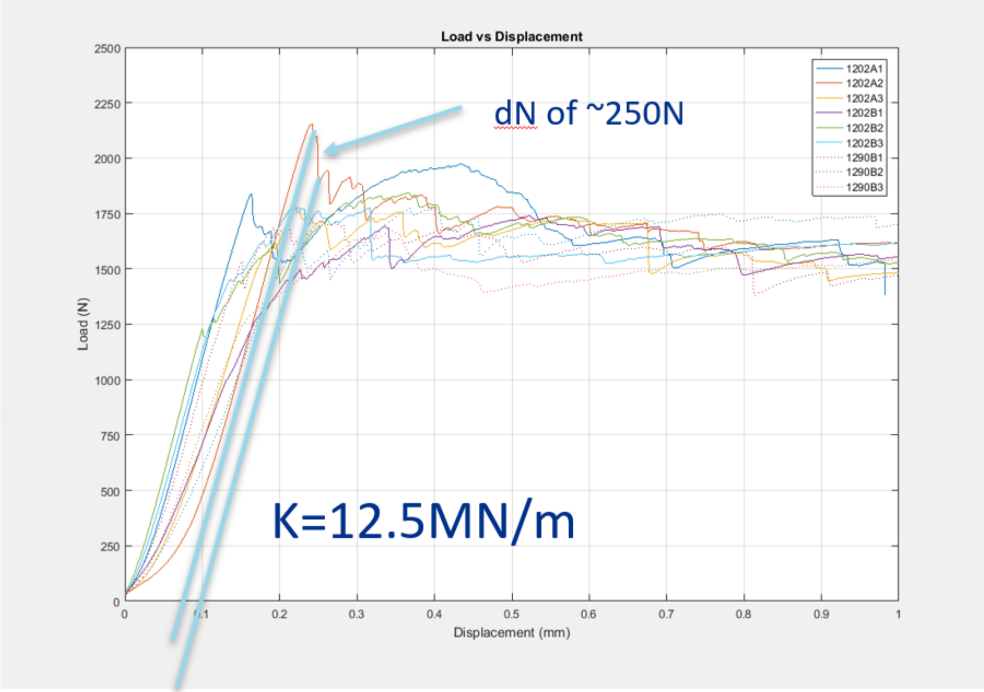
=1/(2k)*F^2

$$\Delta E = \frac{1}{2k}\left(F_1^2 - F_2^2\right)$$

In the highlighted event, the energy release is around 79 mJ

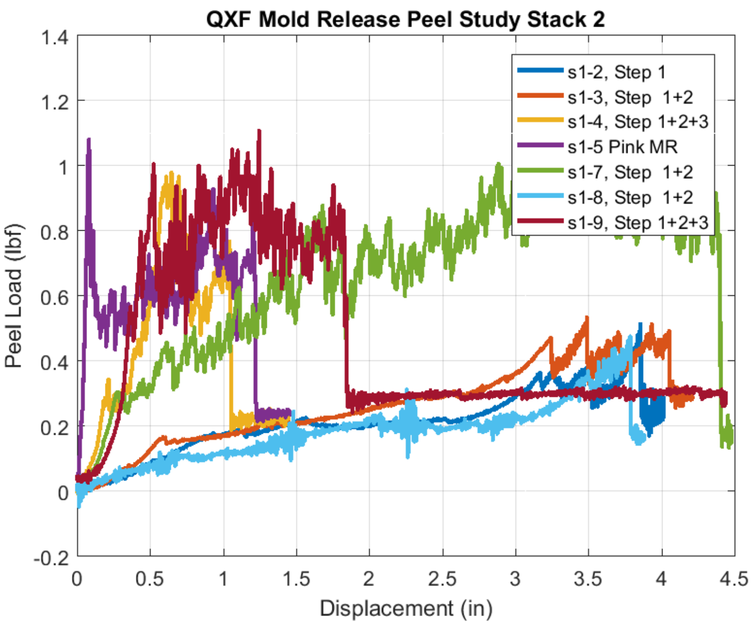
We could assume that the energy is release in the area of the delamination which could be considered at a minimum of~80 uJ/mm^2

Planning on revaluating from a fracture mechanics approach if applicable



Mold Release Peel Testing

- Some recent coils have exhibited adhesion of the coil mid-plane to the impregnation tooling when demolding.
- A Quick comparison of peel strengths of various states of mold release was completed
- Conclusion: Mold release works well when applied in normal circumstances. Teflon works better.



Conclusions

- The FNAL Impregnation process has been evolving to reduce variability and risk
- Process observations have led to additional understanding of system behavior and informed process changes to reduce variability
- We get to learn things by building coils and investigating behavior