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 Nb₃Sn 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$_3$Sn 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
  - ~0.75 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 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
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).

* Dip from identified inconsistency in thermocouple mounting
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 stress.
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.

* 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 ratio of ~4.
    - This is do-able

<table>
<thead>
<tr>
<th>Average</th>
<th>σ</th>
<th>CV</th>
</tr>
</thead>
<tbody>
<tr>
<td>1202</td>
<td>9.68 MPa</td>
<td>0.811 MPa</td>
</tr>
<tr>
<td>1290</td>
<td>8.91 MPa</td>
<td>0.502 MPa</td>
</tr>
</tbody>
</table>
Observations from SBS Testing

Shear Testing and Energy Release

Assuming that $K = \text{constant and some basic spring stuff and conserving energy}$:

$$F = kx$$

$$x = F/k$$

$$E = \frac{1}{2}kx^2$$

$$E = \frac{1}{2}kF^2/k^2$$

$$= \frac{1}{2}kF^2$$

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

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 reevaluating 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