Status of the analysis of the epoxy resin impregnation process

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Content

1. Introduction

Motivation and objectives

The observed variation of glass transition temperature (T_g) of the resin along and between different coils, and its potential impact on their performance have led to the following objectives:

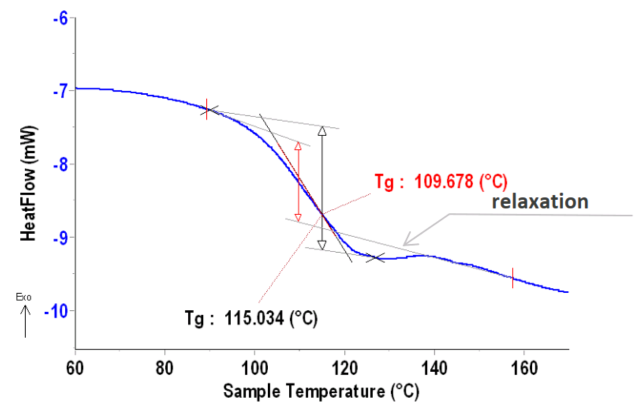
1. Investigate and identify the source of the variation of T_g:
   - Curing and post-curing temperatures.
   - Pressure, and soaking time vs. pot life.
   - Presence of contaminant/residues (e.g. mica degradation by-products).
2. Investigate the impact of a variation of T_g in the mechanical properties of the epoxy resin:
   - Tension/Compression properties.
   - Fracture behaviour.
3. Investigate the quality of impregnation in terms of cracks and void content.
4. Establish an acceptable range of curing and post-curing temperatures, and resulting T_g, for the acceptance of the coils.
5. Define a reliable quality control set of parameters and methods for the resin impregnation of the coils.

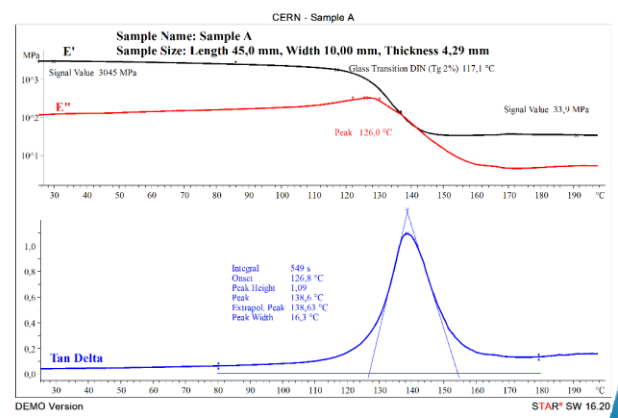
1. Introduction

Thermal analysis – *Some concepts*

1. The state of cure, C, is related to the extent of conversion, and can be estimated a posteriori by measuring the glass transition temperature, T_g, of the epoxy resin.
2. The T_g can be defined as the temperature at which a transition between a glassy state and a rubbery state occurs, as a result of the activation of long-range translational motion of the polymer chains.
3. The T_g can be measured by different techniques, including DSC and DMA:
    1. The DSC method relies on an observable step change in the specific heat capacity, during the phase transition.
    2. The DMA method relies on the relation between the measured storage and loss modulus of the material under cyclic loading.

$$C = \frac{T_g - T_{g0}}{T_{g\infty} - T_{g0}}$$





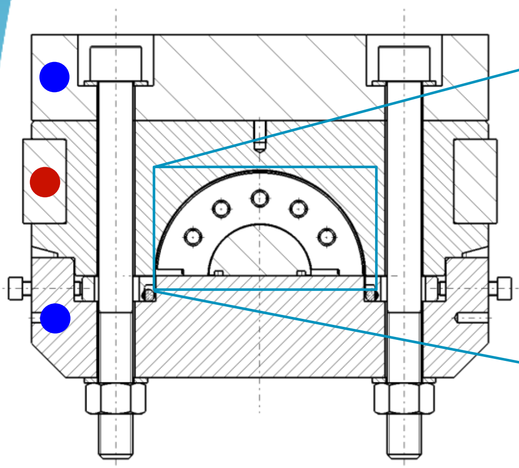
2. Experimental

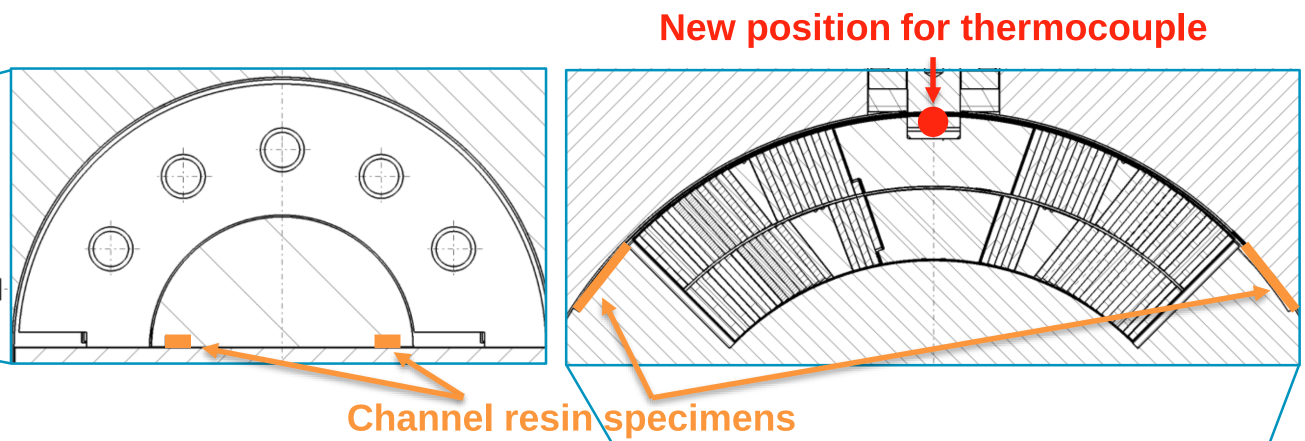
Plan – Summary

1. Investigation of the temperature during impregnation and extraction of resin samples from the impregnation mould and coil.
2. Production of reference samples in a dedicated mould, with the variation of the following parameters:
    1. Curing and post-curing temperatures.
    2. Impact of contaminants.
3. Measurement of T_g.
4. Measurement of the mechanical properties of produced and extracted samples.
5. Dye penetration tests.

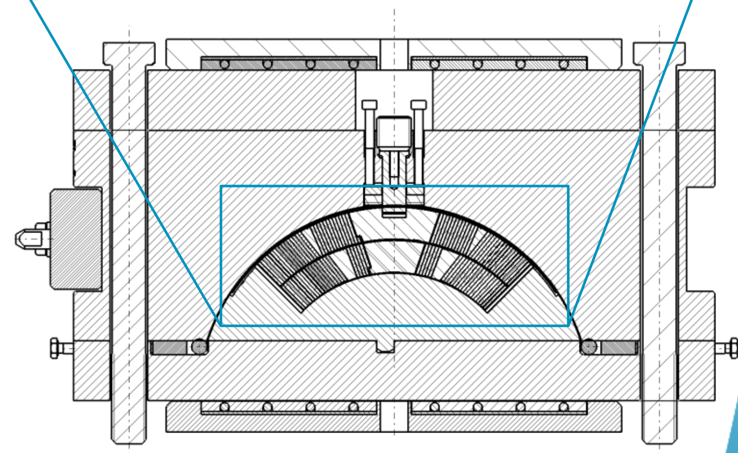
2. Experimental

1. Investigation of the temperature during impregnation and extraction of resin samples from the impregnation mould and coil

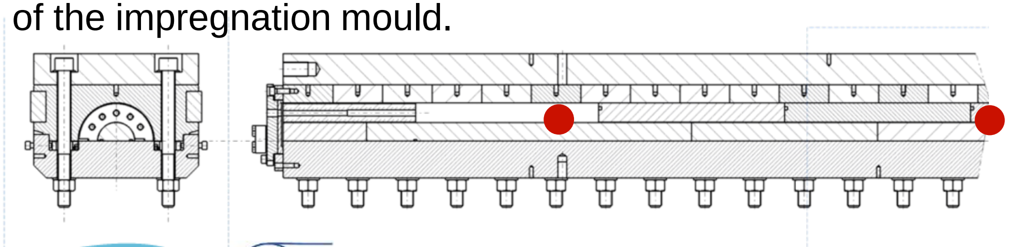




- 🔴 Position of the thermocouples for temperature analysis.
- 🔵 Position of the thermocouples for the heating system feedback control.



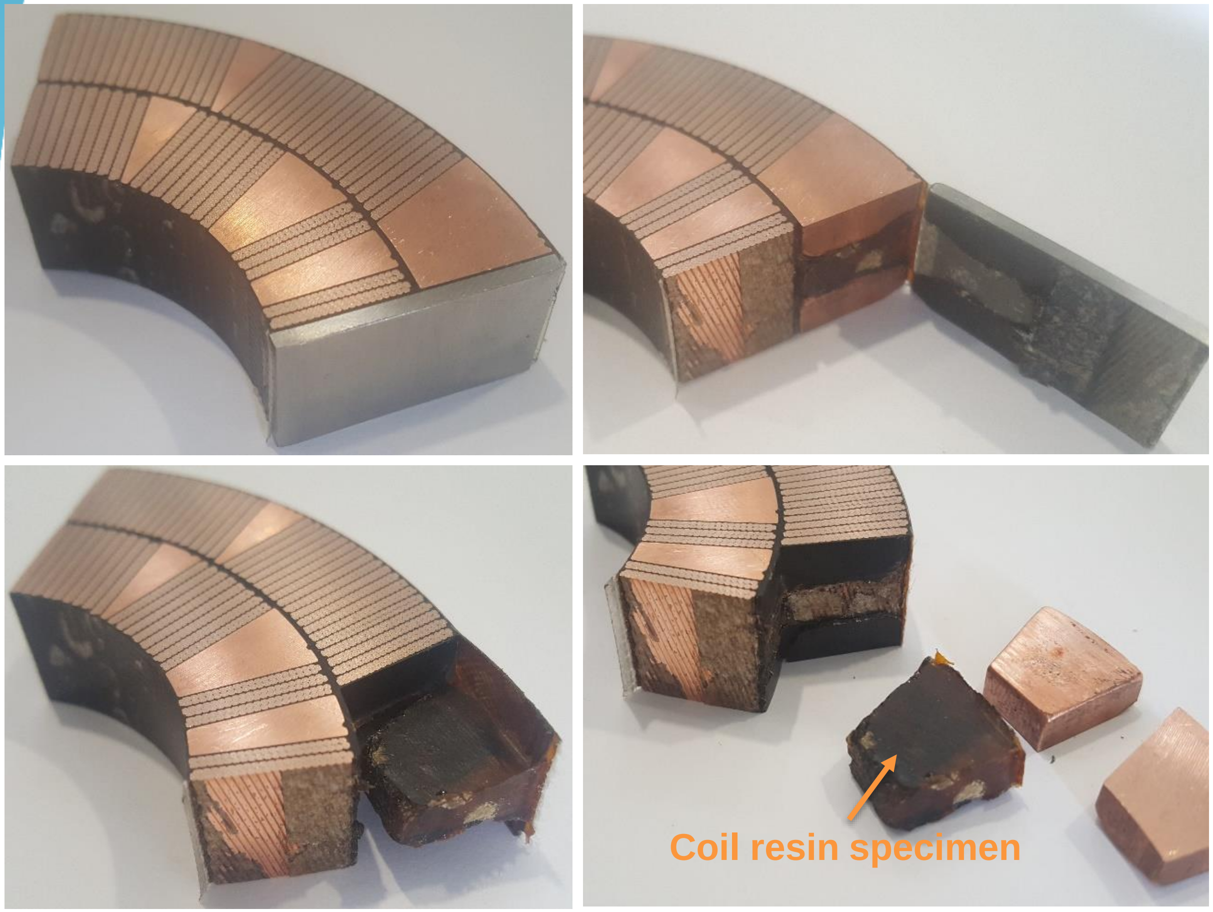
Sensors are positioned in different blocks along the length of the impregnation mould.



- Resin specimens can be extracted for each impregnation from the channels within MQXF and 11T impregnation moulds.

2. Experimental

1. Investigation of the temperature during impregnation and extraction of resin samples from the impregnation mould and coil

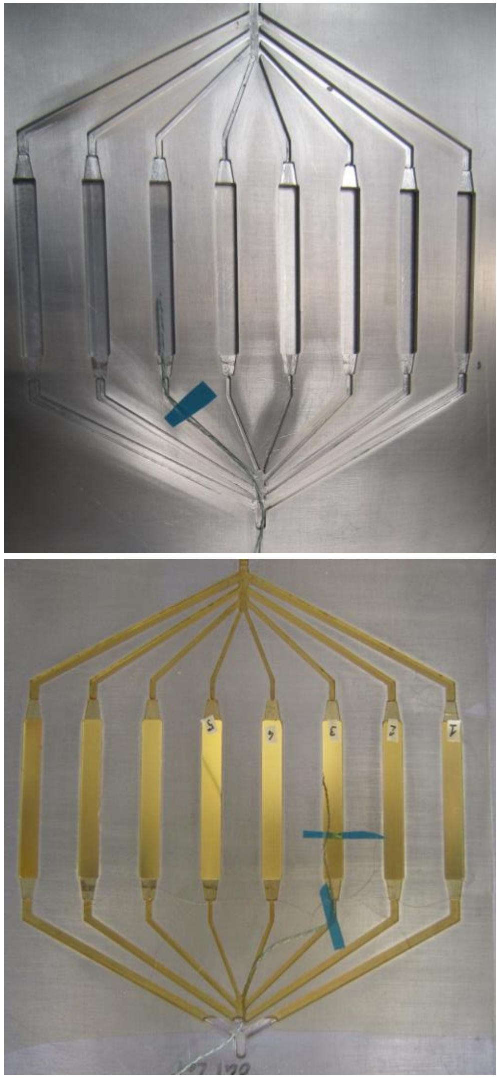


- A specimen within 11T coil GE02 has been extracted from the space between two copper wedges.

2. Experimental

2. Production of reference samples

Resin samples are impregnated in an aluminium mould with different parameters. The temperature is monitored in the furnace, mould and within the resin during the curing and post-curing processes. For each impregnation eight specimens of 10 x 4 x 80 mm are produced.



Sets to be produced:

- Analysis of curing and post-curing temperatures:
  - Nominal cycle (used for long coils).
  - Nominal cycle + 10 °C (curing and post-curing).
  - Nominal cycle – 10 °C (curing and post-curing).
  - MDT cycle: Pre-cycle at 80 °C for ~ 12 hours + Nominal cycle (used for short coils).
- Analysis of the impact of contaminants (**Work in progress**):
  - Reacted mica.
  - Reacted glass fibre insulation system + binder.
  - Reacted mica + glass fibre insulation system + binder.

Nominal cycle for CTD-101K impregnated Nb_3Sn coils:
- Curing: 5 hours at 110 °C
- Post-curing: 16 hours at 125 °C.

Pictures courtesy of R. Gauthier, CERN

2. Experimental

3. Measurement of T_g

Analysis parameters:

DSC measurements using a SETARAM 131:

- ASTM E1356 standard
- Crucible: aluminium 30 µL without lid
- Gas: nitrogen
- Gas flow rate: 50 mL/min
- Form of test specimen: single piece of cubic shape
- Temperature range: 30 °C – 200 °C
- Temperature ramp rate (heating): 10 °C/min
- Temperature ramp rate (cooling): 15 °C/min

DMA measurements by Mettler Toledo:

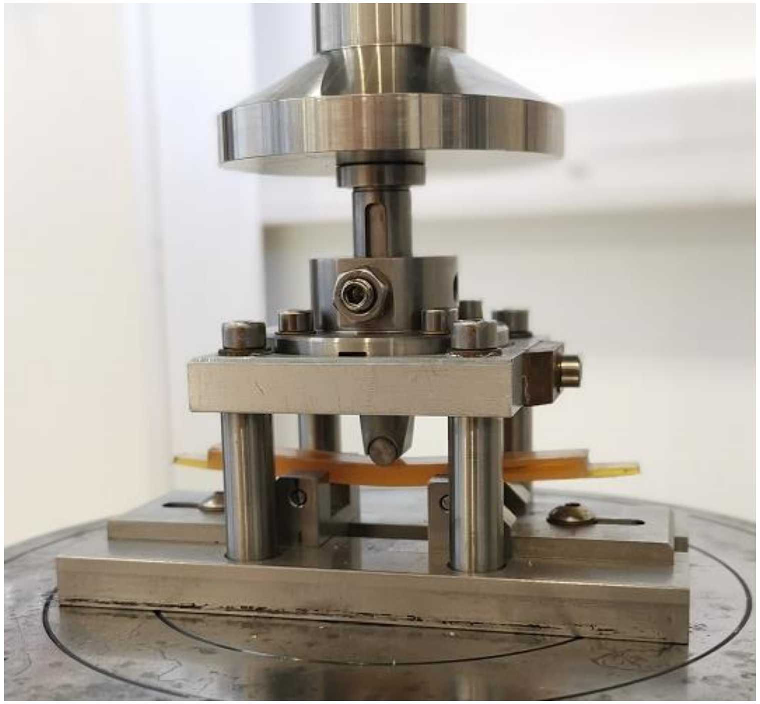
- Method: Three point bending
- Temperature range: 25 °C – 200 °C
- Temperature ramp rate (heating): 3 °C/min
- Displacement: 40 µm.
- Frequency: 1 Hz

Resin specimens that contain glass fibre, and that are intended for T_g measurements, will be further analysed by thermogravimetric techniques, in order to determine the mass fraction of glass fibre and its potential influence on the results.

2. Experimental

4. Measurement of the mechanical properties of produced and extracted samples.

The mechanical test used for the characterisation of specimens is the three point loading based on ASTM D 790, in which the bending strength and modulus is measured.



Analysis parameters:

- Crosshead speed: 0.3 mm/min.
- Span: 30.5 mm.
- Support dimeter: 3 mm.
- Nose diameter: 6 mm.
- Load cell capacity: 1kN.

$$E_b = \frac{L^3 m}{4bd^3} \quad \sigma_f = \frac{3PL}{2bd^2}$$

Measurement conditions:

- Room temperature.
- 77 K (**Work in progress**).

Pictures courtesy of M. Crouvizier, CERN

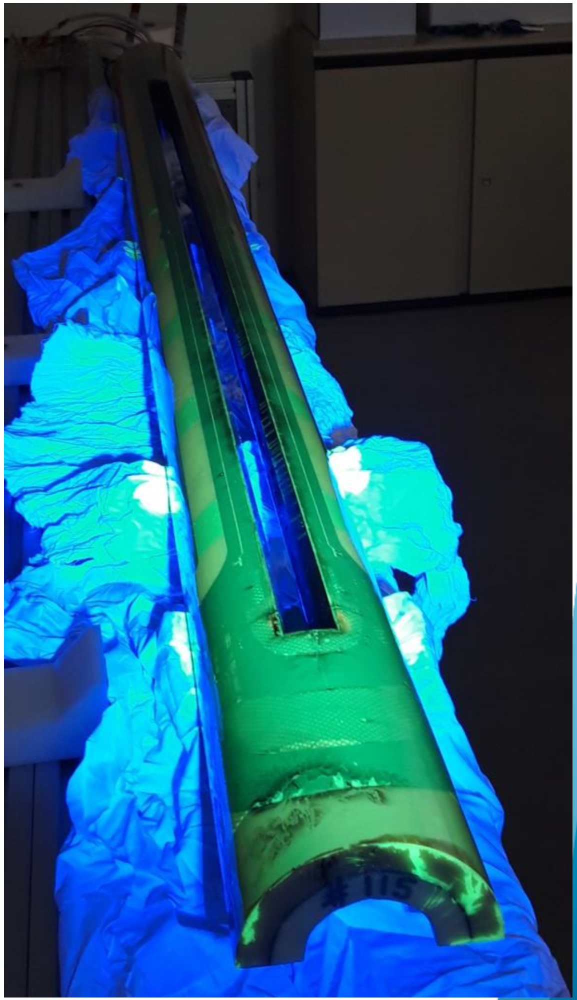
2. Experimental

5. Dye penetrant test

The dye penetrant test is used to investigate the quality of impregnation, including porosity and existence of possible cracks in the epoxy resin.

Experimental examination of 11T short coil 115:

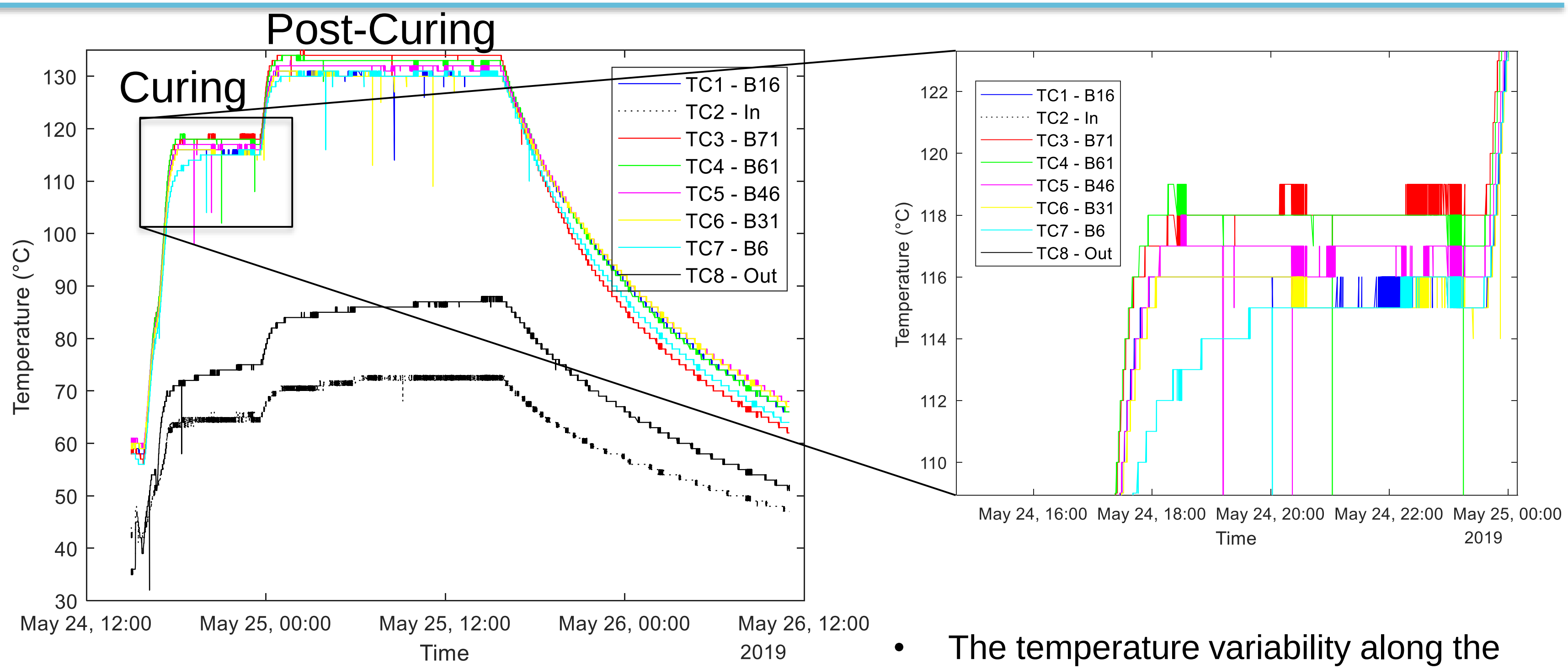
1. Application of dye penetrant to the outer radius of the coil.
2. Removing/cleaning the dye from the outer surface.
3. Application of the developer.
4. Inspection of the outer and inner surfaces of the coil.

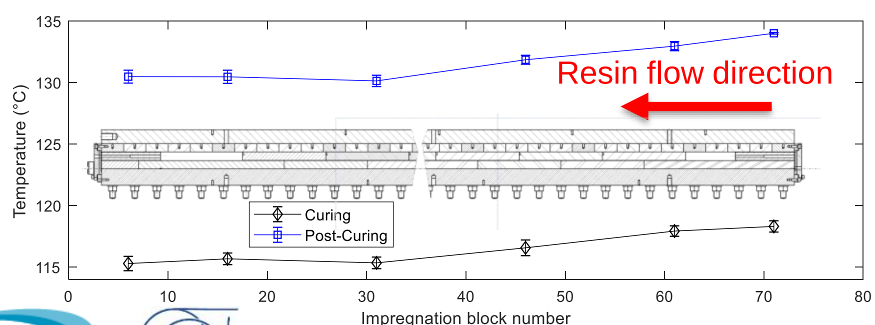


Pictures courtesy of A. Porret, CERN

3. Initial results

Temperature during impregnation – Analysis of 11T coil GE-12

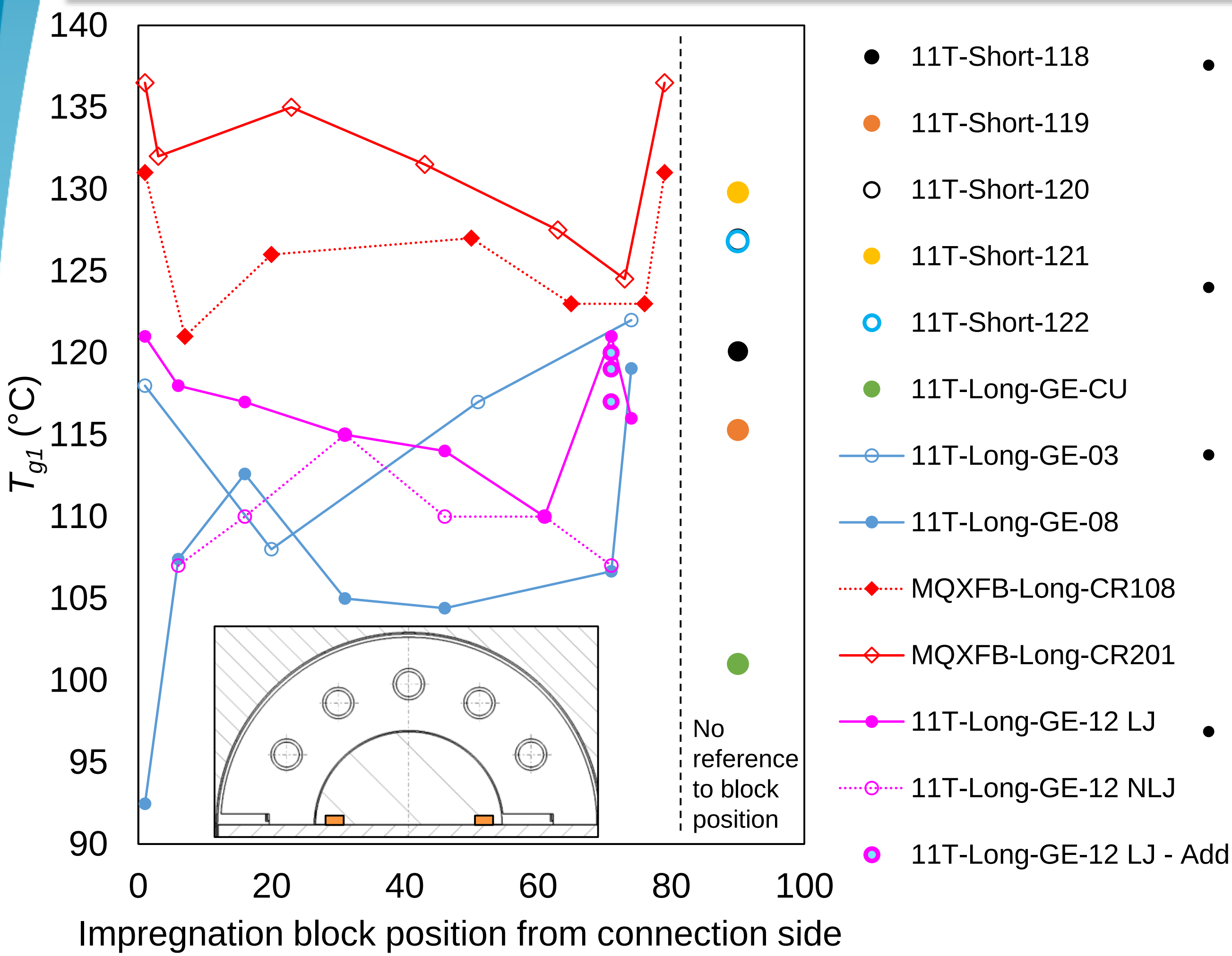




- The temperature variability along the length of the impregnation mould during curing and post-curing is relatively low (i.e. < 5 °C).
- There is an apparent trend in which the temperature decreases from the mould's inlet to outlet.

3. Initial results

Measurement of T_g – Summary of channel specimens

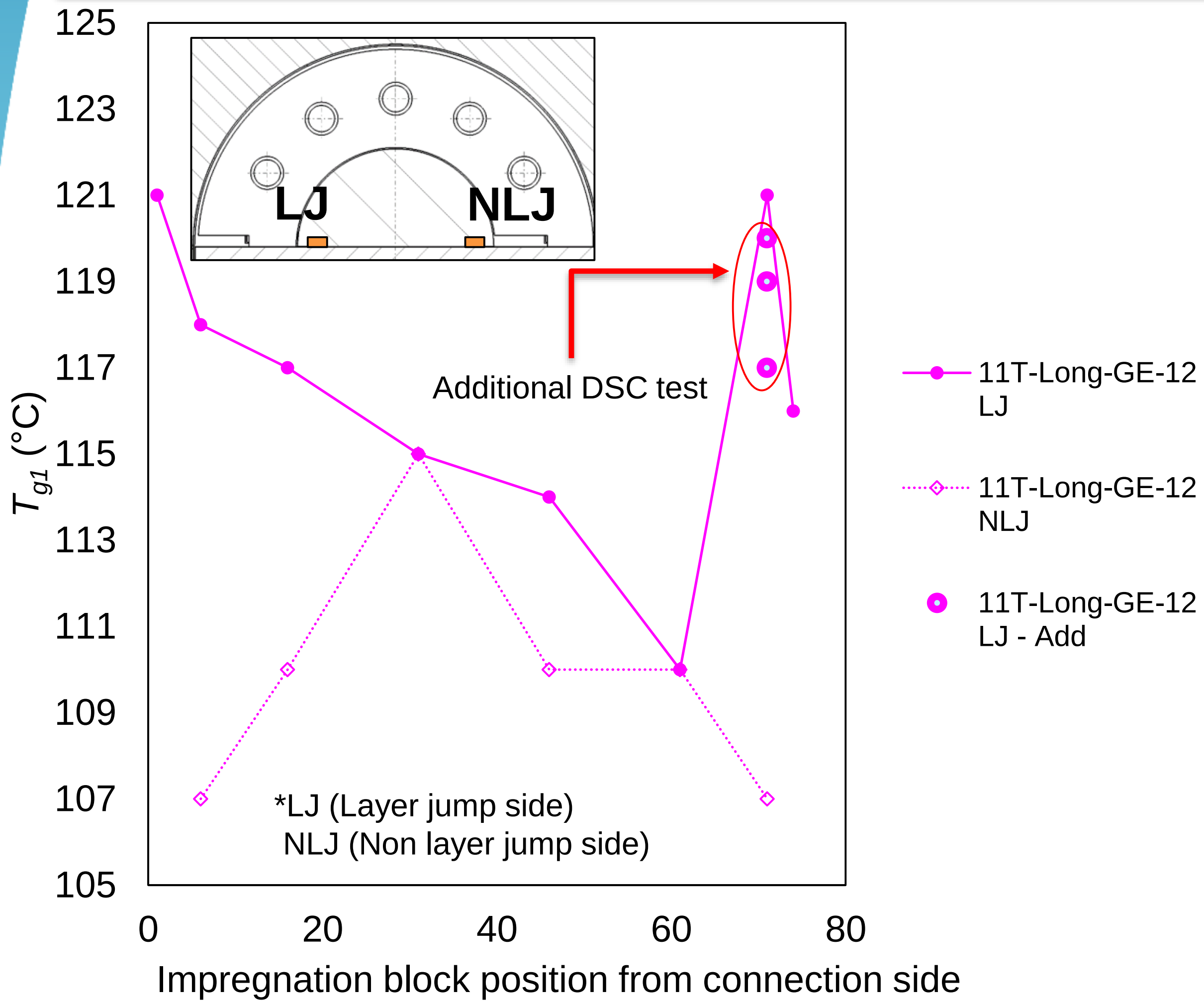


- The overall variability of T_g for the CTD-101K impregnated coils is ~40 °C.
- The maximum variability of T_g along the length of a single coil is ~25 °C.
- MQXF channel specimens has consistently higher T_g in relation to 11T specimens.
- The relatively high variation of T_g in 11T is observed for short and long coils.

DSC measurements carried out by D. Ternova and B. Teissandier following ASTM E1356

3. Initial results

Measurement of T_g – Summary of channel specimens

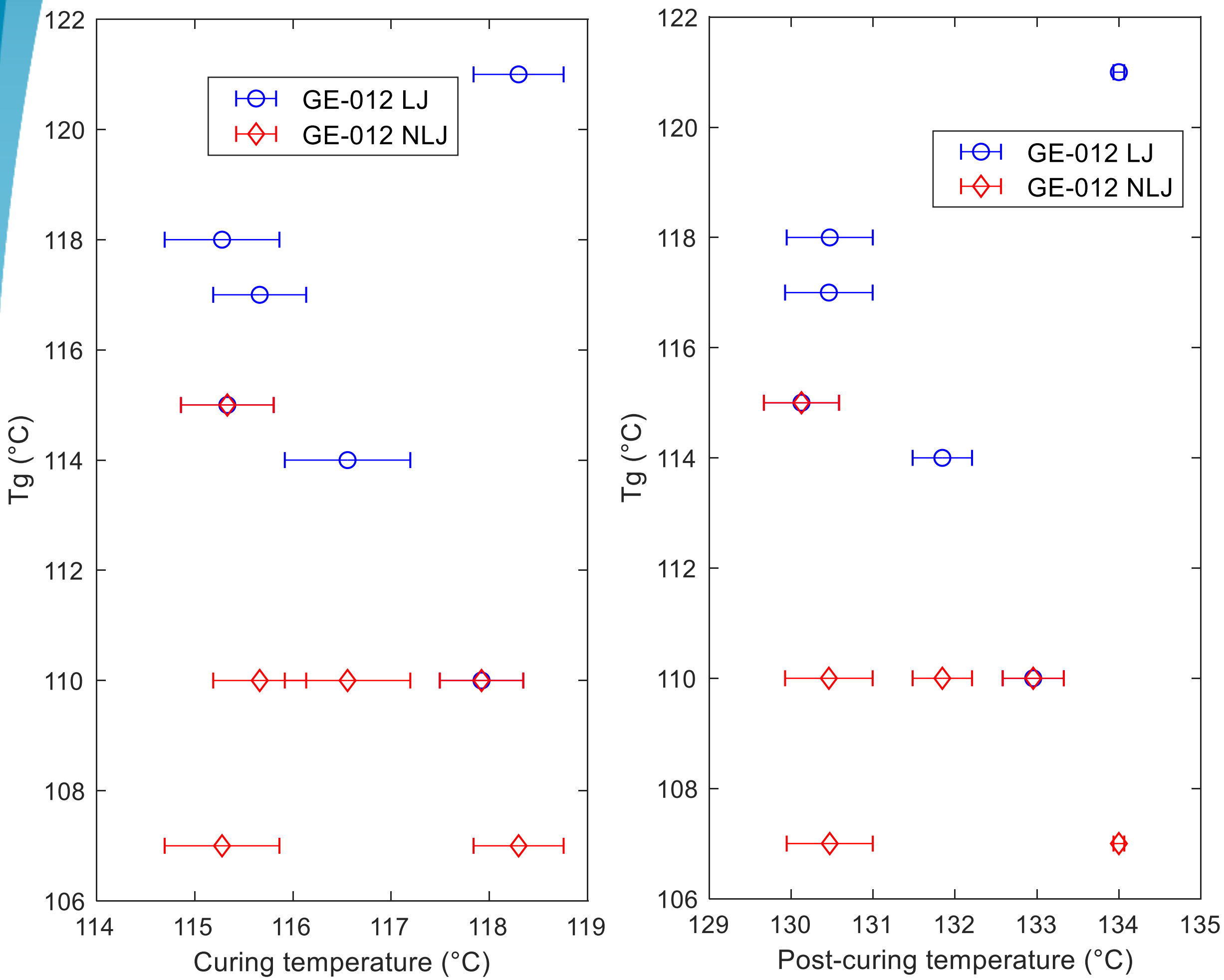


- The difference in T_g between two samples taken from the same longitudinal position, but different channels, is up to ~15 °C.
- The additional DSC tests carried out on material from block 71 and LJ side revealed that the test repeatability (i.e. < 5 °C) can't explain the difference between LJ and NLJ channels.
- Assuming that the temperature in both channels, for the same cross-section, should be similar, differences in T_g should be dependent on other parameters (e.g. resin mix and/or contaminants uniformity).

DSC measurements carried out by D. Ternova and B. Teissandier following ASTM E1356

3. Initial results

Measurement of T_g – Analysis of channel specimens



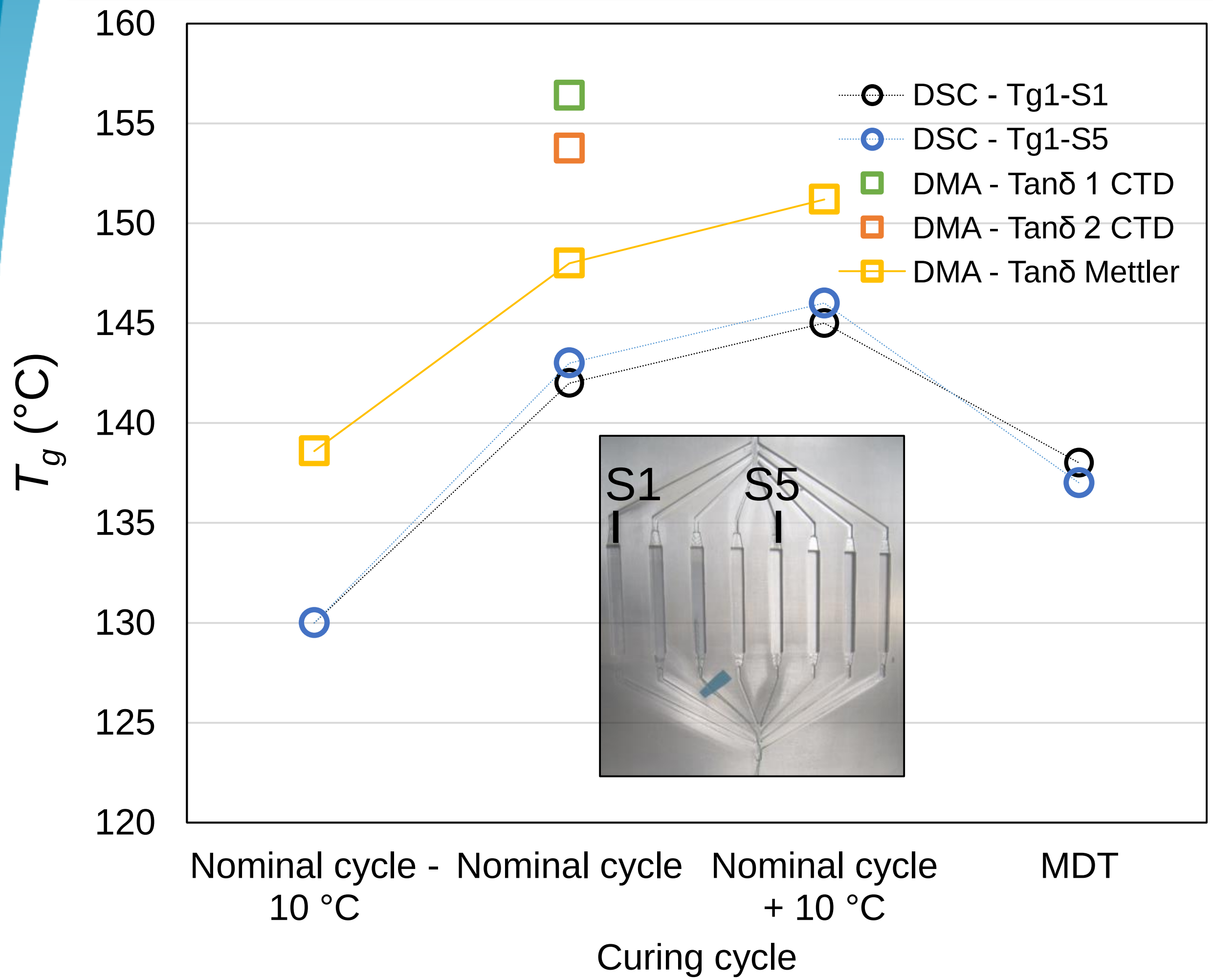
- The analysis of each particular position along the length of the coil, for LJ and NLJ sides, revealed no strong correlation between measured T_g and block average temperatures during curing and post-curing cycles.
- In line with the previously discussed point concerning differences in T_g for LJ and NLJ sides, the lack of clear correlation between mould temperatures and T_g might indicate a higher sensitivity of T_g to other parameters.

*LJ (Layer jump side)
NLJ (Non layer jump side)

DSC measurements carried out by D. Ternova and B. Teissandier following ASTM E1356

3. Initial results

Measurement of T_g – Summary of reference samples

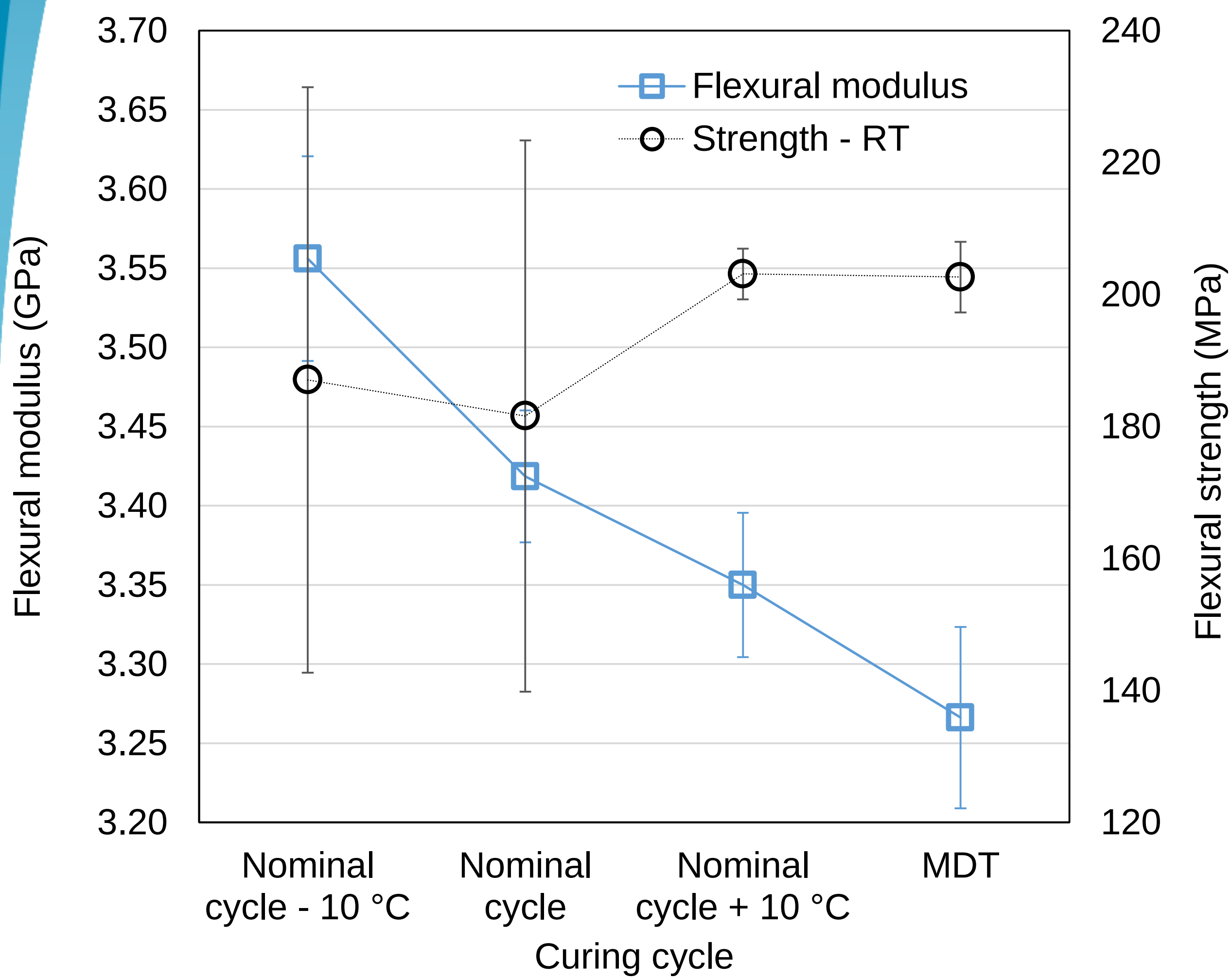


- The DSC measured T_g is consistent between different specimens within the mould.
- The observed trend with regards to the curing cycle is equivalent for measurements carried out by DSC and DMA techniques.
- The observed offset between DMA and DSC measurements is due to the different technique used, and is in line with previously reported trends in literature.
- The DMA measured T_g values reported by CTD for this resin batch (i.e. □ and □) are slightly higher than the DMA value for the nominal cycle reference sample, which might indicate high sensitivity of the resin to curing and post-curing conditions.

DSC measurements carried out by D. Ternova and B. Teissandier following ASTM E1356

3. Initial results

Measurement of mechanical properties – Summary of reference samples

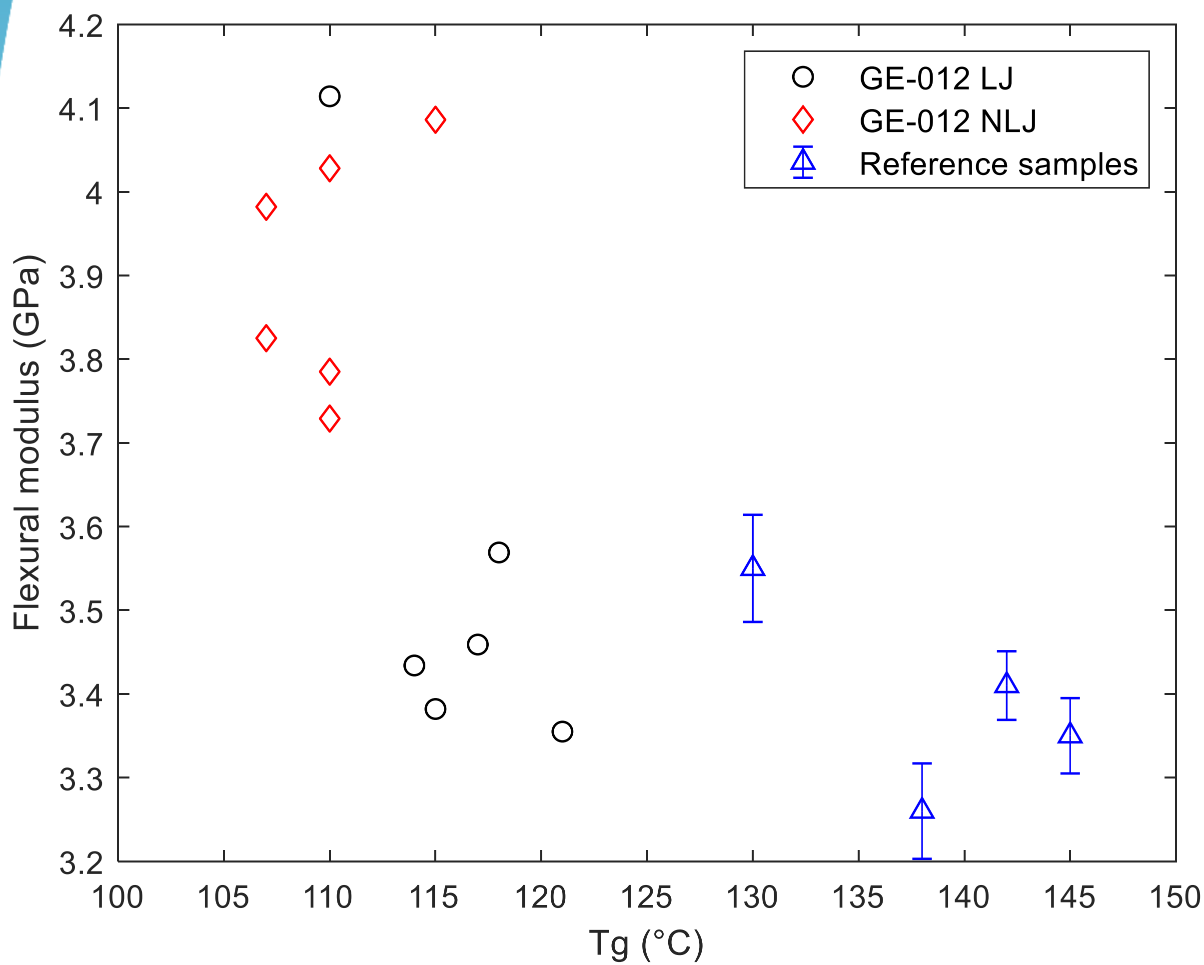


- The measured values of flexural modulus exhibit an inverse relation with regards to the temperature during curing and post-curing cycles. Further, the additional pre-cycle seems to lower the flexural modulus as well (i.e. MDT cycle).
- The values of flexural strength don't show statistically significant differences as the standard deviation is too high, due to the high dependency of strength on the random distribution of micro-cracks and the relatively low number of tested specimens (i.e. three).

Mechanical measurements carried out by M. Crouvizier, CERN

3. Initial results

Measurement of mechanical properties – Overall analysis



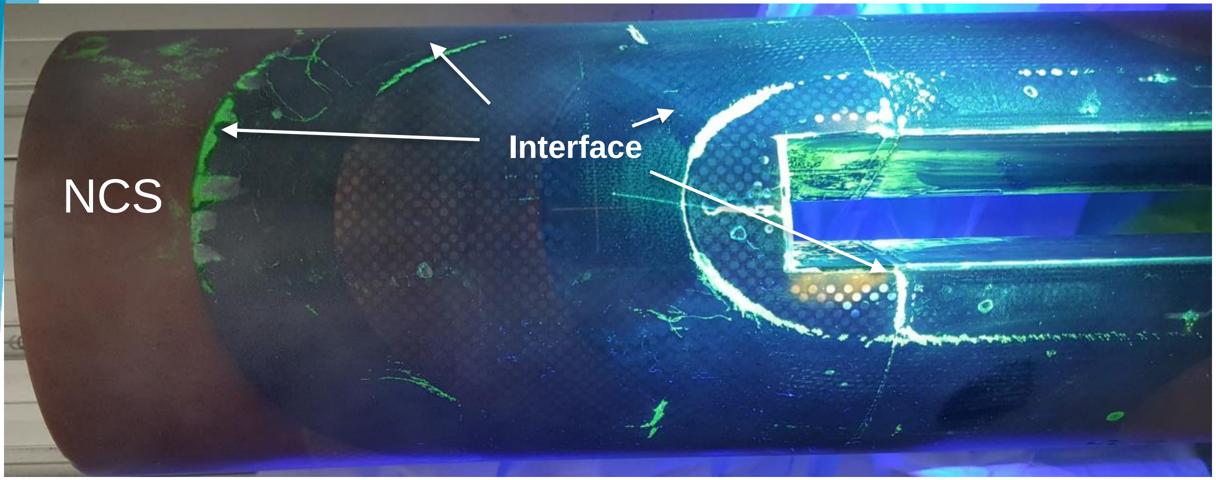
- The measured flexural modulus of reference and channel specimens show an apparent inverse relation with respect to T_g.
- Similar inverse dependencies can also be found in literature*. This relation is normally linked to the molecular packing of the resin.

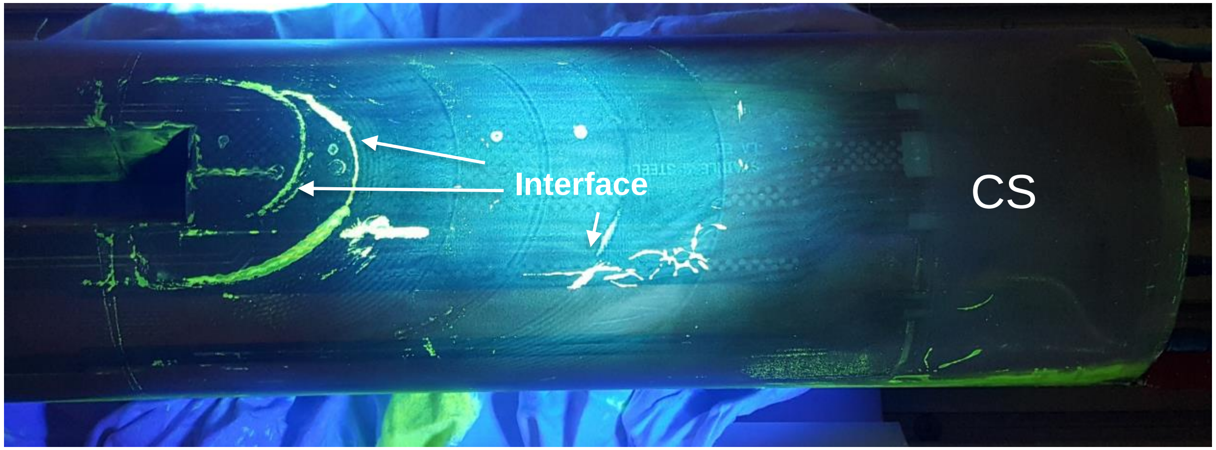
* Detwiler, Andrew T., and Alan J. Lesser. "Aspects of network formation in glassy thermosets." *Journal of Applied Polymer Science* 117, no. 2 (2010): 1021-1034.

Mechanical measurements carried out by M. Crouvizier, CERN

3. Initial results

Dye penetrant test after cold magnet testing – Outer radius surface C115

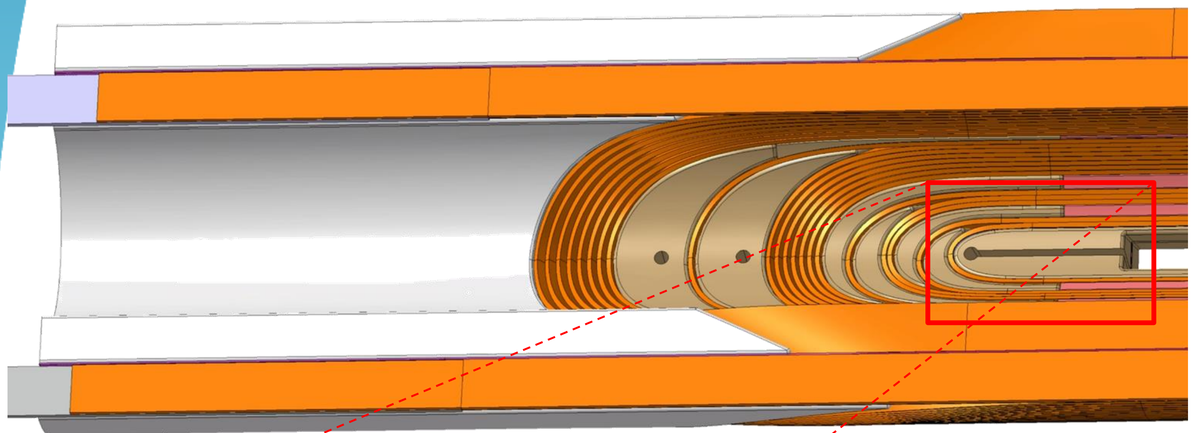




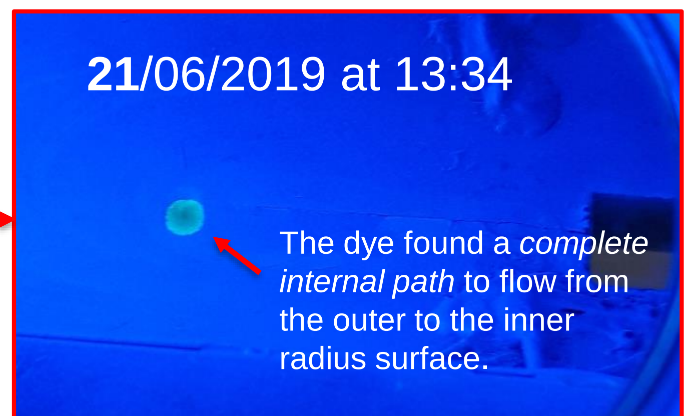
- The inspection of the outer radius exposed dye marks, mainly in the interfacial regions between different components, indicating partial debonding in these regions.

3. Initial results

Dye penetrant test after cold magnet testing – Inner radius surface C115



- The examination of the inner radius revealed a small dye mark. It seems that there is an internal *complete paths* for the dye to flow from the outer to the inner radius surface.



4. Next steps

1. Investigation of the temperature during impregnation:
   a) Measurement of the temperature in a position close to the MQXF coil's pole of C204.
2. Production of additional reference samples:
   a) *Additional specimens for curing and post-curing temperatures analysis, in order to provide enough mechanical results so the variation with respect to parameters is statistically significant.*
   b) *Samples with the presence of contaminants*.
3. Measurement of T_g by DSC method:
   a) *Coil extracted resin block.*
   b) *Samples with the presence of contaminants*.
   c) *Additional channel extracted resin samples.*
4. Measurement of the mechanical properties:
   a) Additional reference samples at RT.
   b) Reference samples at 77K.
5. Chemical analysis:
   a) Energy-dispersive X-ray spectroscopy (EDS) to investigate the presence of contaminants in resin samples.
6. Dye penetration tests:
   a) Investigation of sections from coil 11T coil GE-02 and 10 stack samples.

Thanks!