Experimental assessment of Nb_3Sn impregnated coil samples in He II:

temperature margin evaluation and investigation of aging mechanisms in the impregnation

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special thanks to Kirtana Puthran (TE/MSC), Enes Ilbuga (TE/CRG), EN/MME team

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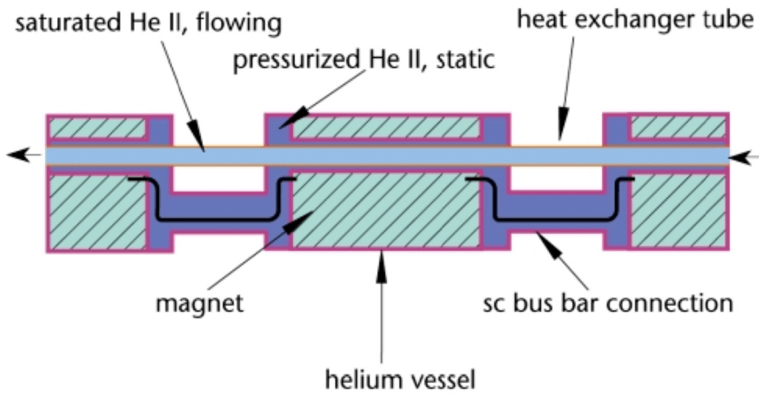
Introduction

- **He II Heat Transfer experimental set-up** has been established at the Central Cryogenic Laboratory at CERN to qualify the thermal performance of samples of superconducting magnet coils
- Adapted to test **Nb_3Sn impregnated coil samples** in an **ongoing test program**
- Program evolved into experimental campaign + numerical simulations to answer **open questions on thermal behaviour of Nb_3Sn impregnated coils**, esp. quench limits as function of locally deposited power
- Set-up has been recently used for studies on **aging effects on the coil samples due to thermal cycling**

Introduction

- The **main outputs** of the experimental campaign are:
  - Thermal interface resistance between the coil surface and the He II bath;
  - Temperature gradients generated in cable layers as a function of heat flux;
  - Heat deposition regime above 25 mW/cm^3 remained unexplored and required assessment

- Measurements carried out in a set-up where the **coil sample is immersed in a pressurized He II stagnant bath** which is in turn in contact with a saturated He II bath for heat extraction.
  - Method is complex and time-consuming.
  - A **complete measurement run in saturated He II conditions** was carried out to validate whether results are qualitatively and quantitatively comparable to the standard method.



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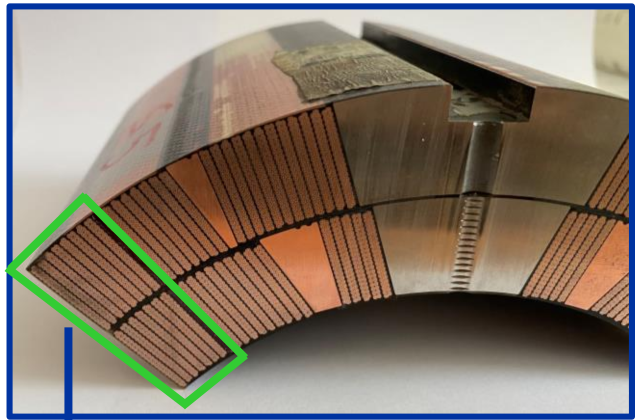
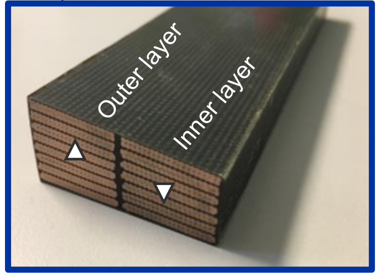
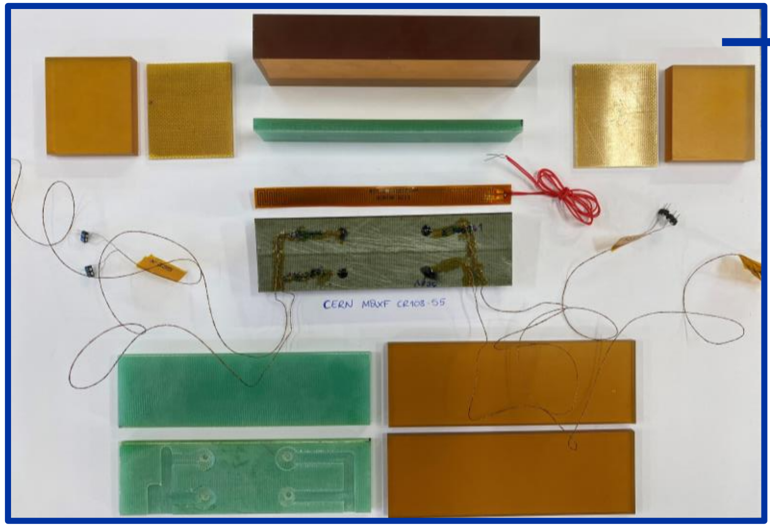
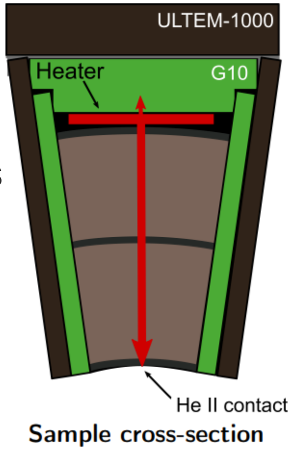
Sample preparation



Sample cut from larger MQXF/D11T section (8 cables in outer layer, 7 cables in inner layer)

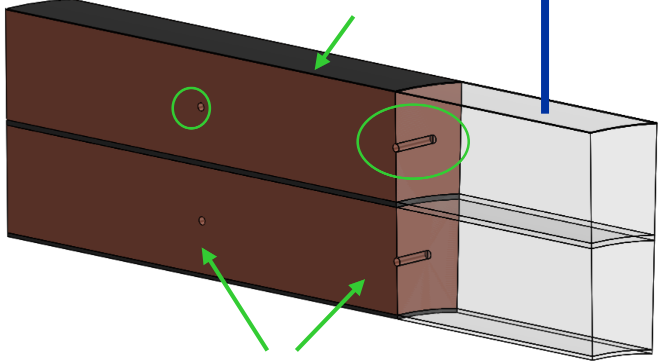






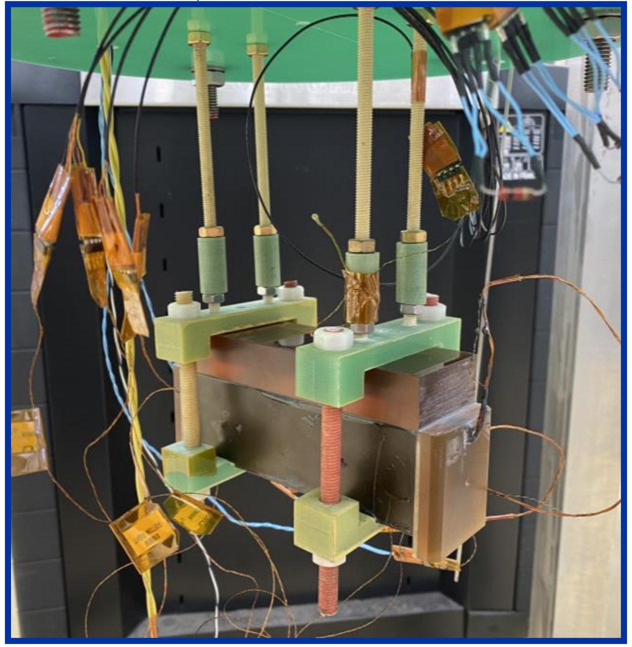
instrumented sample is encased in 2 layers of **insulation** on all sides (**except for inner surface**, He contact)

Polyimide **electric heater** bonded to the outer insulation



2 temperature sensors per layer (45 mm apart), each **probing centre** of the cut sample cross-section

Sample is mounted on test set-up and **ready for measurements in saturated He II**



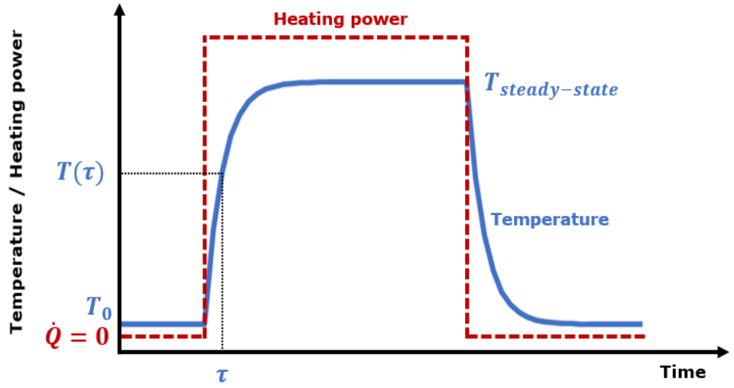
* Maximum length allowed by setup ~140 mm, longer than twist pitch

Experimental set-up & measurement procedure

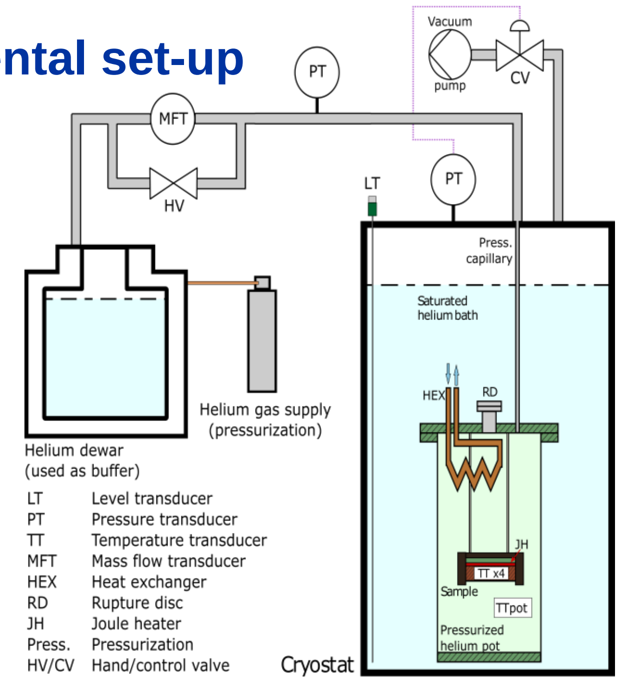
Typical measurement run:

- Heat load sweep from 0 to ~ 1000 W/m^2
- Sweeps done for bath temperatures of 1.8 K, 1.9 K, 2.0 K and 2.1 K
- From each measurement point, a **steady-state temperature** and **time constant** (with associated temperature) **are extracted for each sensor**

Typical measurement for each heat input



Experimental set-up



Experiment allows for extraction of:

- **Steady-state temperature rise** as function of applied heat load
- Temperature **response as function of time**
- Global and interlayer **heat exchange coefficients***
- Solid material properties, **Kapitza resistance**
- Qualitative and quantitative* **information about He presence in the sample** as well as its **location** within the coil

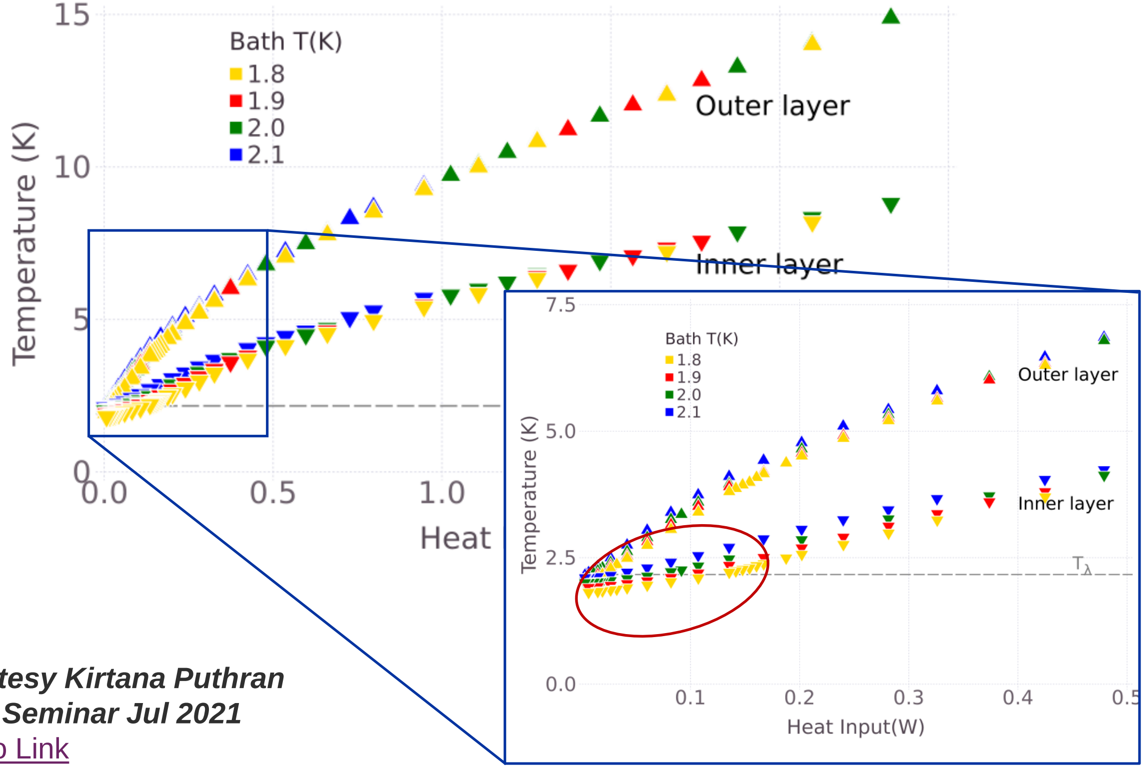
Contents

Interpretation of steady-state results

Example: D11T GE-02

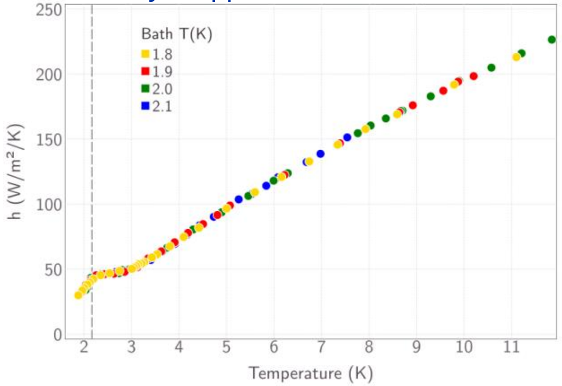
- Steady-state results are plotted as **steady-state temperature** as a function of **heat input** from the resistive heater

Steady-state temperature rise

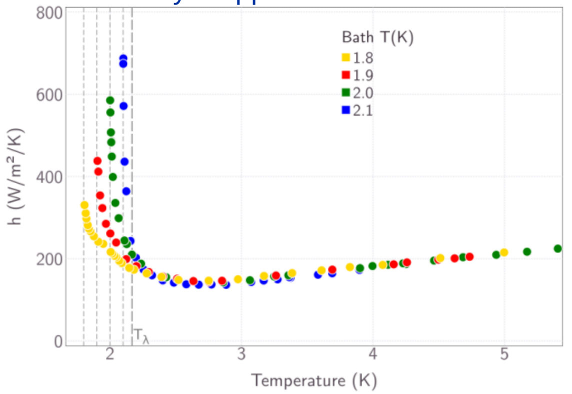


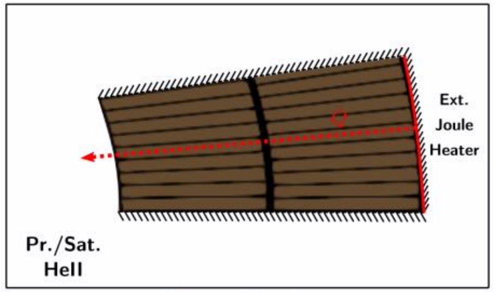
Courtesy Kirtana Puthran
MSC Seminar Jul 2021
Indico Link

Interlayer approx. heat transfer coeffs.



Inner layer approx. heat transfer coeffs.





External heating

- Gives indication of **temperature rise** in cable layers
- Allows for extraction of **maximum inner surface** as well as **interlayer heat transfer coefficients***
- Allows for extraction of **Kapitza resistance** between coil sample and He II bath
- Overall shape of curve below T_λ gives **indication of He presence** in the sample**

- Resistive heater on outer surface → constant heat input $\dot{Q}$ [W].
- Despite best efforts in insulating the sample, significant amount of heat lost through surfaces not directly in contact with He II.
- **Efforts ongoing to calibrate the heat load effectively crossing the sample.**

Interpretation of transient results
Example: D11T GE-02

Time constant of sample defined as:

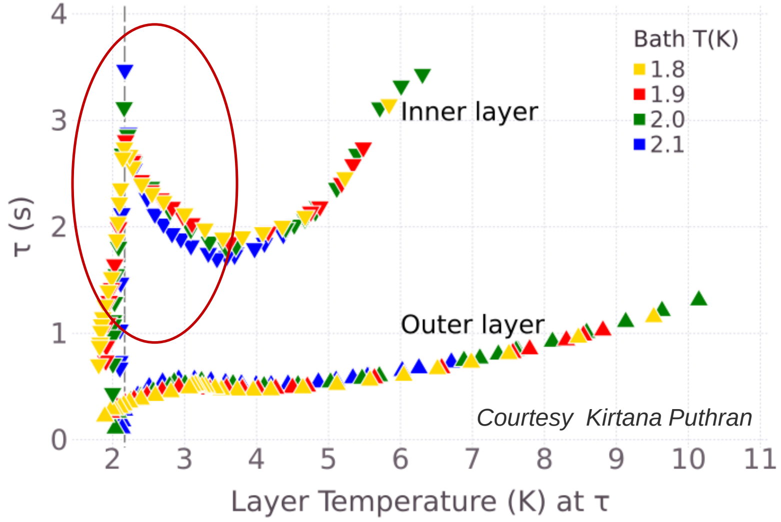
$$\tau = \sum_{i=0}^{n} \frac{\rho_i {c_p}_i V_i}{h A_s}$$

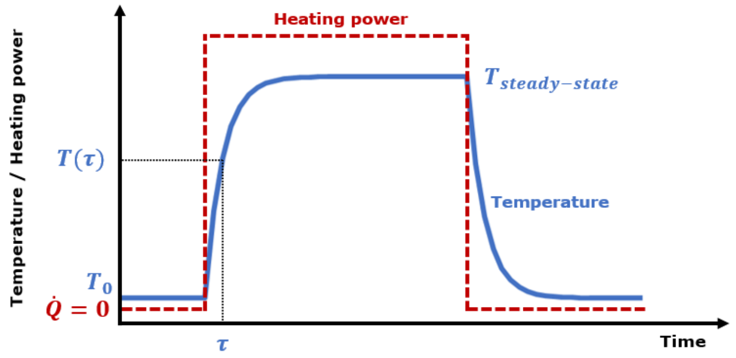
i refers to different contributions to the time constant:

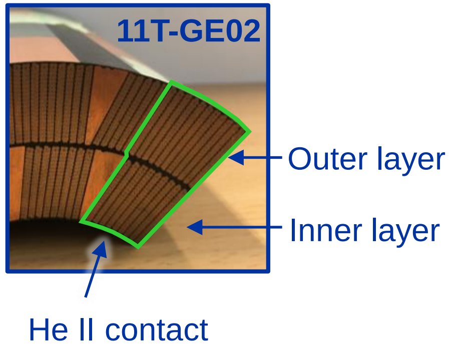
- Materials that make up the coil sample (Cu, Nb_3Sn,epoxy, glass fibre…)

D11T GE-02 inner layer shows unusual behaviour

Time constants as function of layer T







- A_S, ρ_i, V_i do not change (appreciably) with temperature
- h does not suffer abrupt changes
- c_p is vanishingly small at $T \approx T_\lambda$ for most solid materials.
- **What can produce a peak in the sample's heat capacity** (seen in its measured time constant) **at or around T_λ**?

Interpretation of transient results

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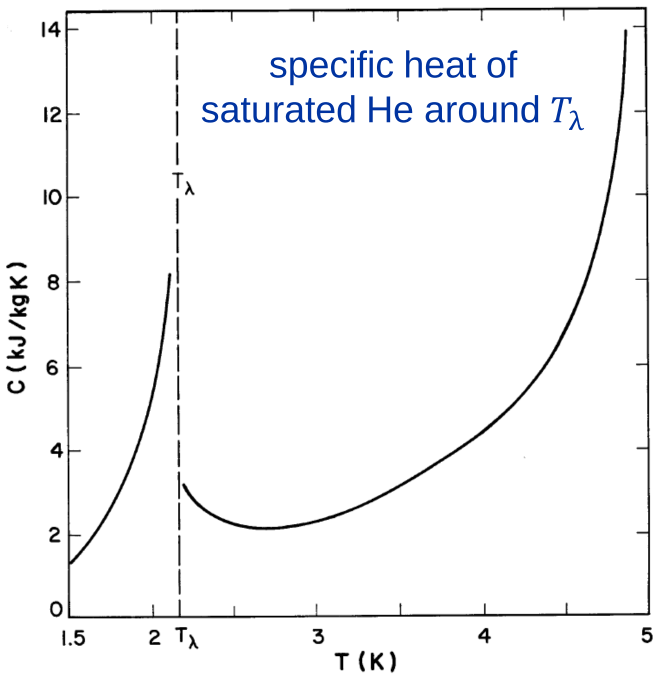
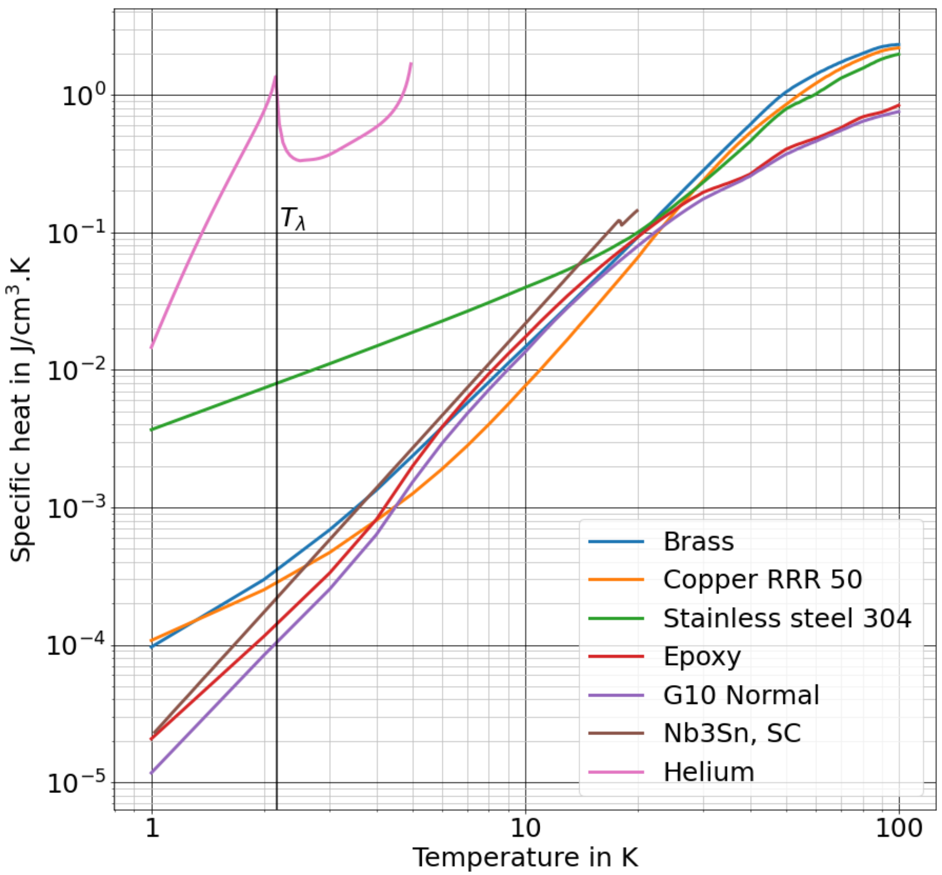
- Materials that make up the coil sample (Cu, Nb_3Sn,epoxy, glass fibre…)
- Additional insulation, glue, He that has penetrated/permeated the sample…

- c_p is vanishingly small at $T \approx T_\lambda$ for most solid materials, **but He II has an unmistakable peak at its transition from He I to He II**



Source: Sciver, S.W. (2012), Helium cryogenics: Second edition

Specific heat of (some) constituent materials of coil sample



Material data from: HePak, Cryocomp, MATPRO

Interpretation of transient results

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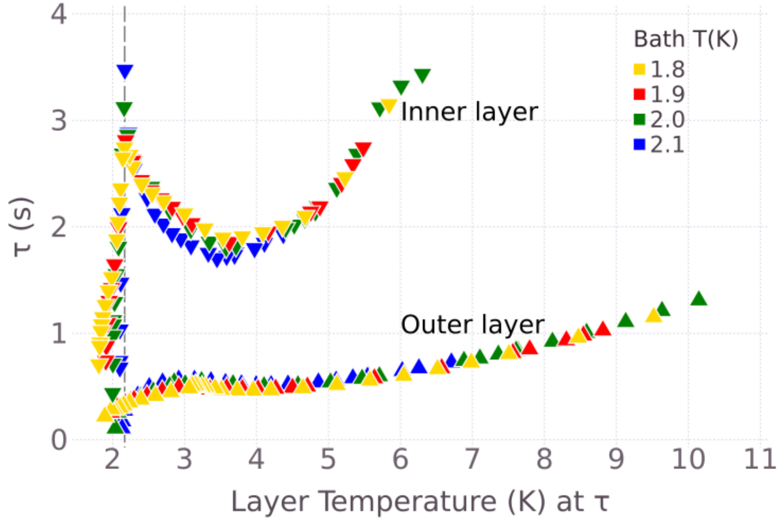
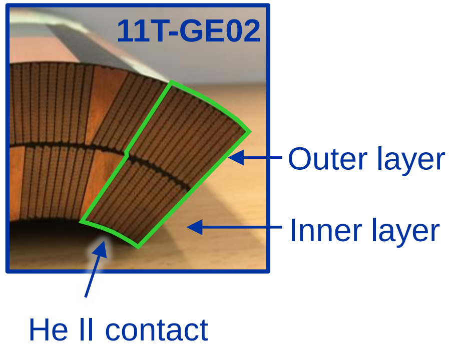
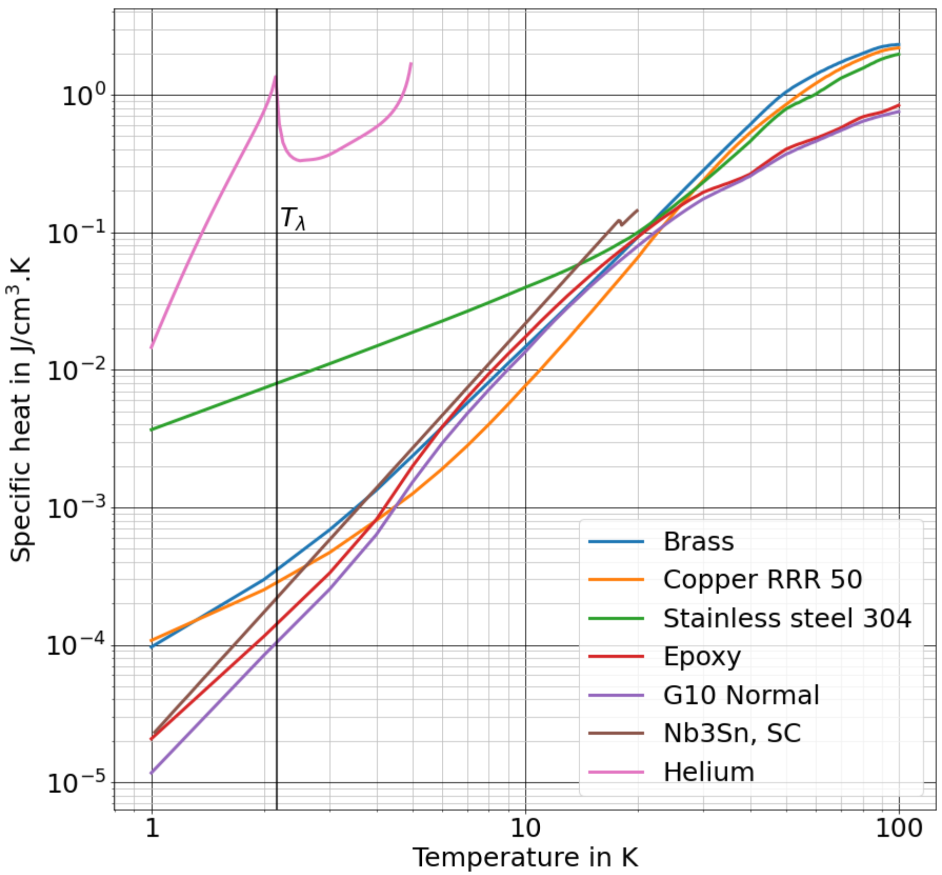




D11T GE-02 inner layer shows He signature (0.32% by volume fraction estimated)

Specific heat of (some) constituent materials of coil sample



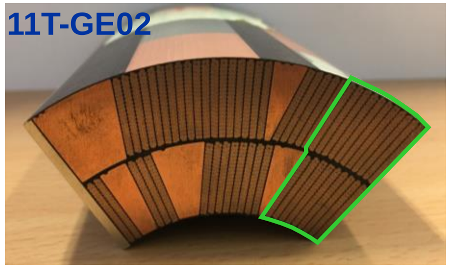
Material data from: HePak, Cryocomp, MATPRO

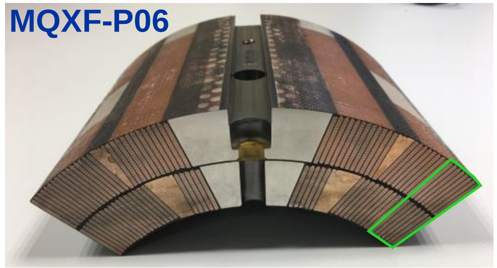
History of measured samples @ the Cryolab

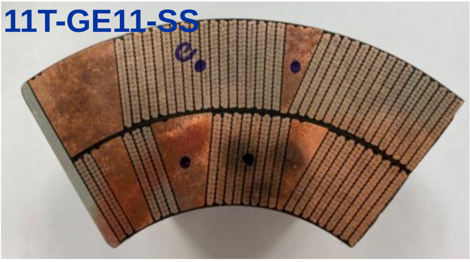
See link for Kirtana's presentation

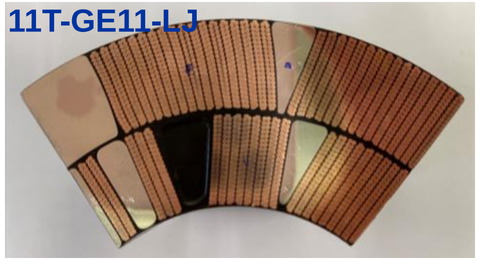
Cryolab Name	MQXF / 11 T	Production / Prototype	Manufacturer	Type of test	Period of testing	# Meas. Runs	He content?	Comments	Coil trace
				Transfer	2017	1		Induction heating	In-house
MQXF LARP07	MQXF	Prototype	USA	He II Heat Transfer	2018/19/20	3		Induction heating	LARP (tested in MQXFS3)
D11T GE02	11 T	Production	CERN	He II Heat Transfer	2020	1		Induction and Joule Heating	Coil GE02 (HCMBH_C005-01000002)
MQXF P06	MQXF	Production	USA	He II Heat Transfer	Feb-21	2		Joule Heating , Press + Sat conditions	Coil P06, MQXFAP1b
MQXF CR108	MQXF	Production	CERN	He II Heat Transfer	Oct-21	2		Joule Heating, saturated cond.	Coil CR108 S5 cut, CR108-S5-840-975
D11T GE11-LJ	11T	Production	CERN	He II content / cycling	Summer 2021	1		Virgin coil, layer jump	Coil C11, GE11, connection side
		Production	CERN	He II content / cycling	Summer 2021	1		Virgin coil, straight section	Coil C11, GE11, connection side

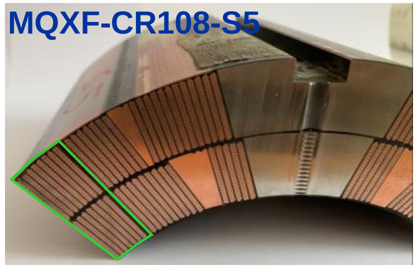
This presentation











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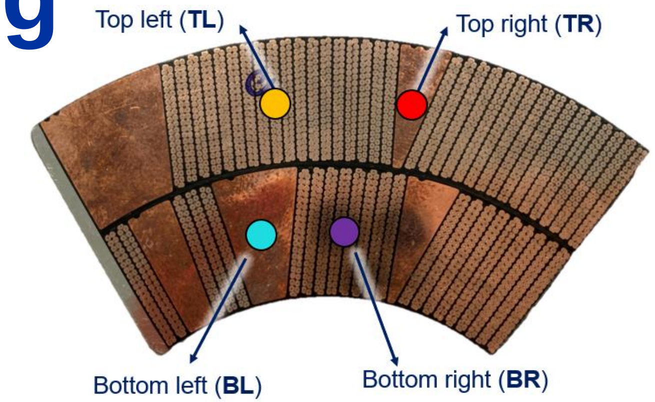
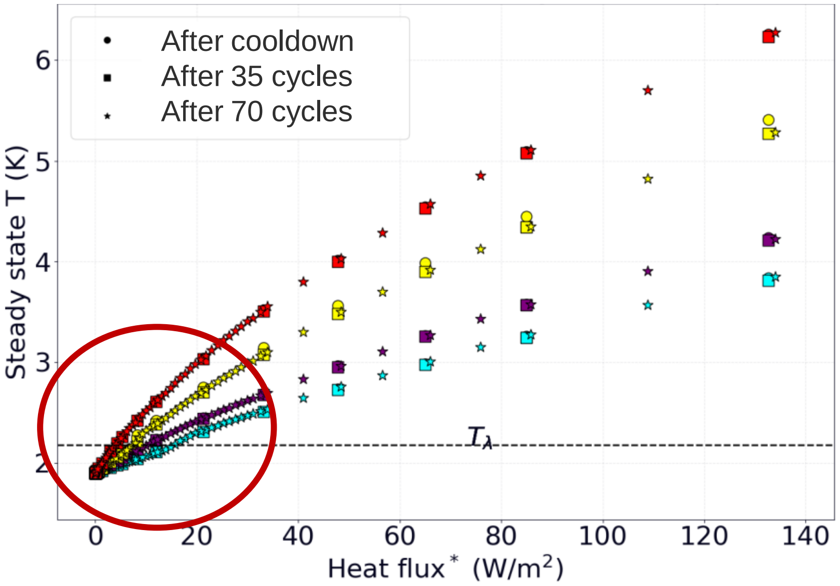
11T virgin coil GE11 – Thermal cycling

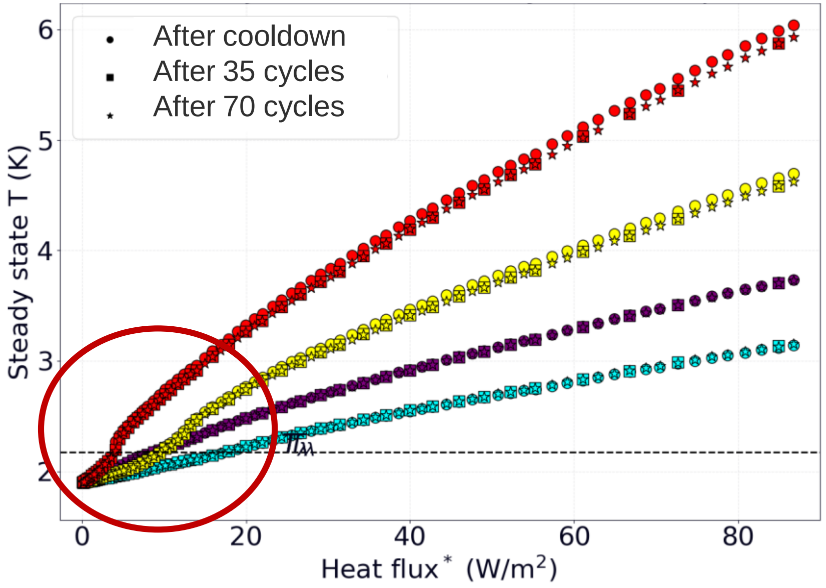


- Triggered by the 11T Task Force, report in EDMS no. 2649442 (link)
- Both samples from connection side of 11T coil **GE11 (virgin coil),** one from the **straight section** and one from the **layer jump** zone
- **Thermal cycling** (from 1.9 K to 25 K): scans pre-cycle, after 35, after 70 cycles.

D11T-GE11-SS (straight section)



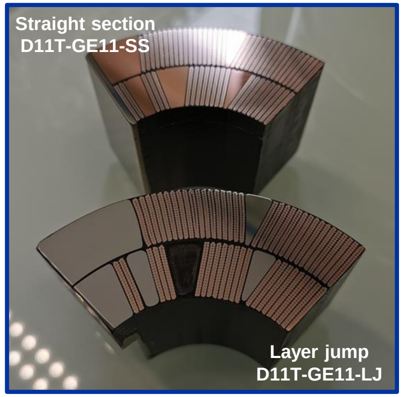
D11T-GE11-LJ (layer jump)



- **Steady state temperature plots hint at He presence** (shown by shape of curve close to T_λ)
- **Effect more pronounced in the layer jump** sample, especially in the outer layer (red+yellow sensors)

* Applied heat flux

11T virgin coil GE11 – Thermal cycling



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- **Peaks at or near T_λ indicate presence of He in the coil**

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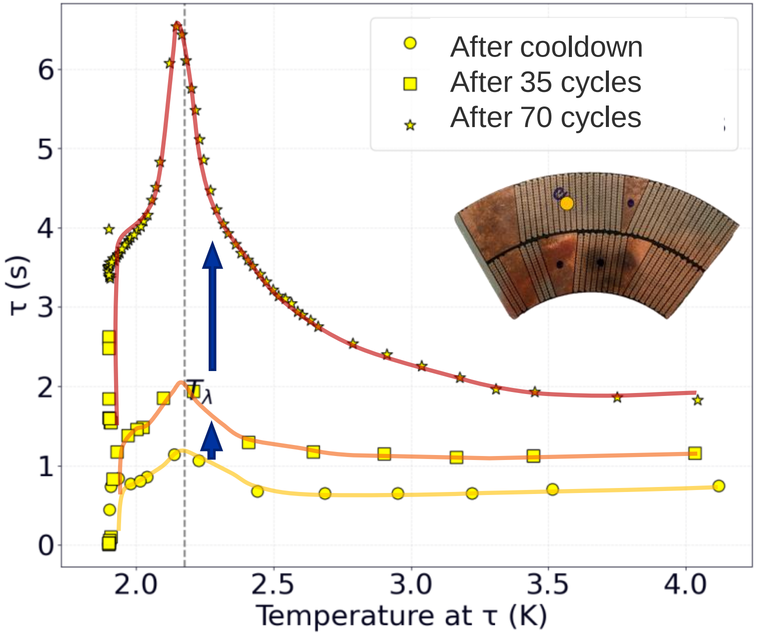
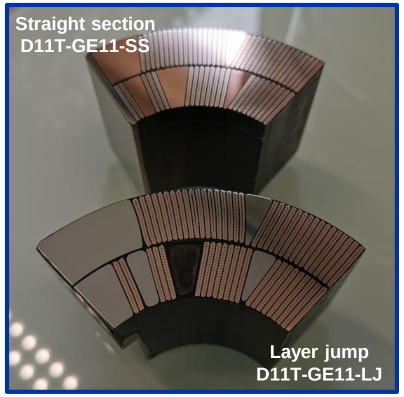


11T virgin coil GE11 – Thermal cycling



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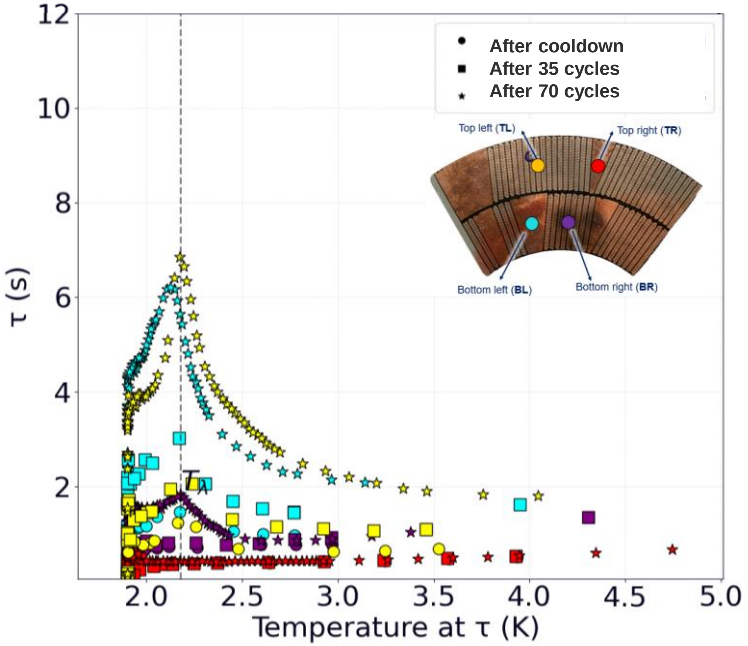
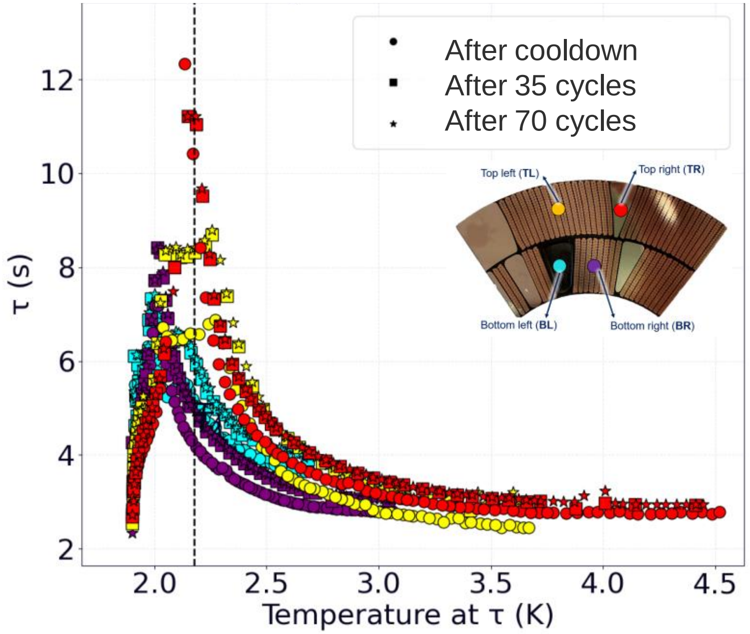


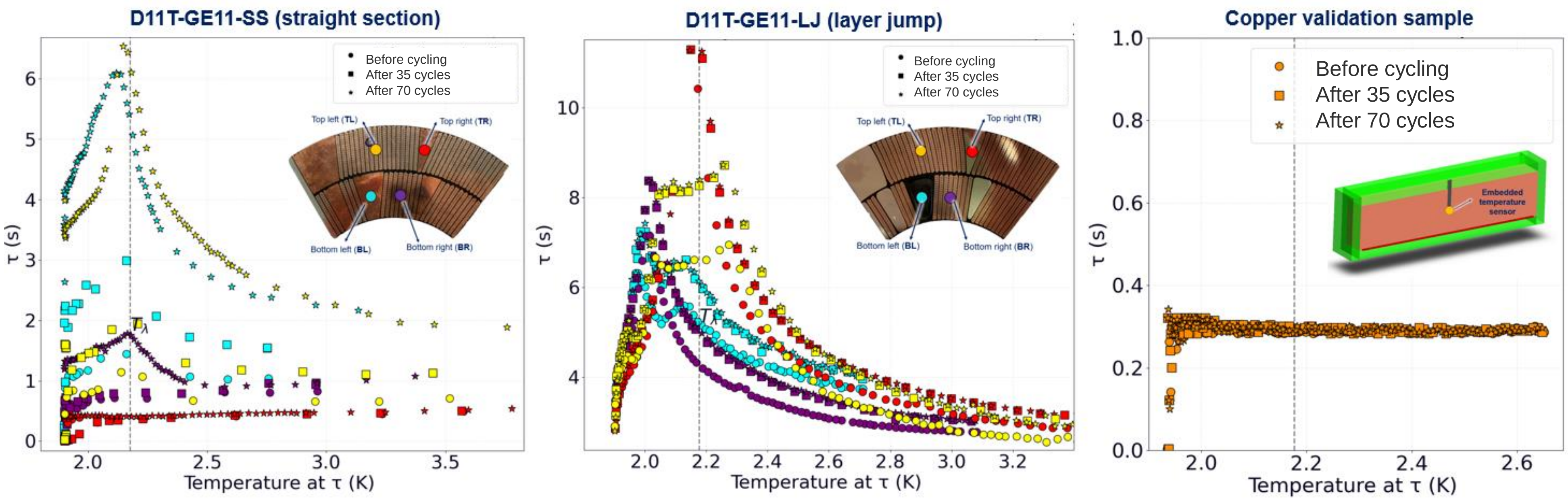
D11T-GE11-LJ (layer jump)



- Results **show a clear He signature that is present even before cycling and that evolves dramatically with increased number of (modest) thermal cycles**.
- In more inhomogeneous parts of the coil, **He presence is pronounced from the start** but does not noticeably increase further.

11T virgin coil GE11 – Thermal cycling

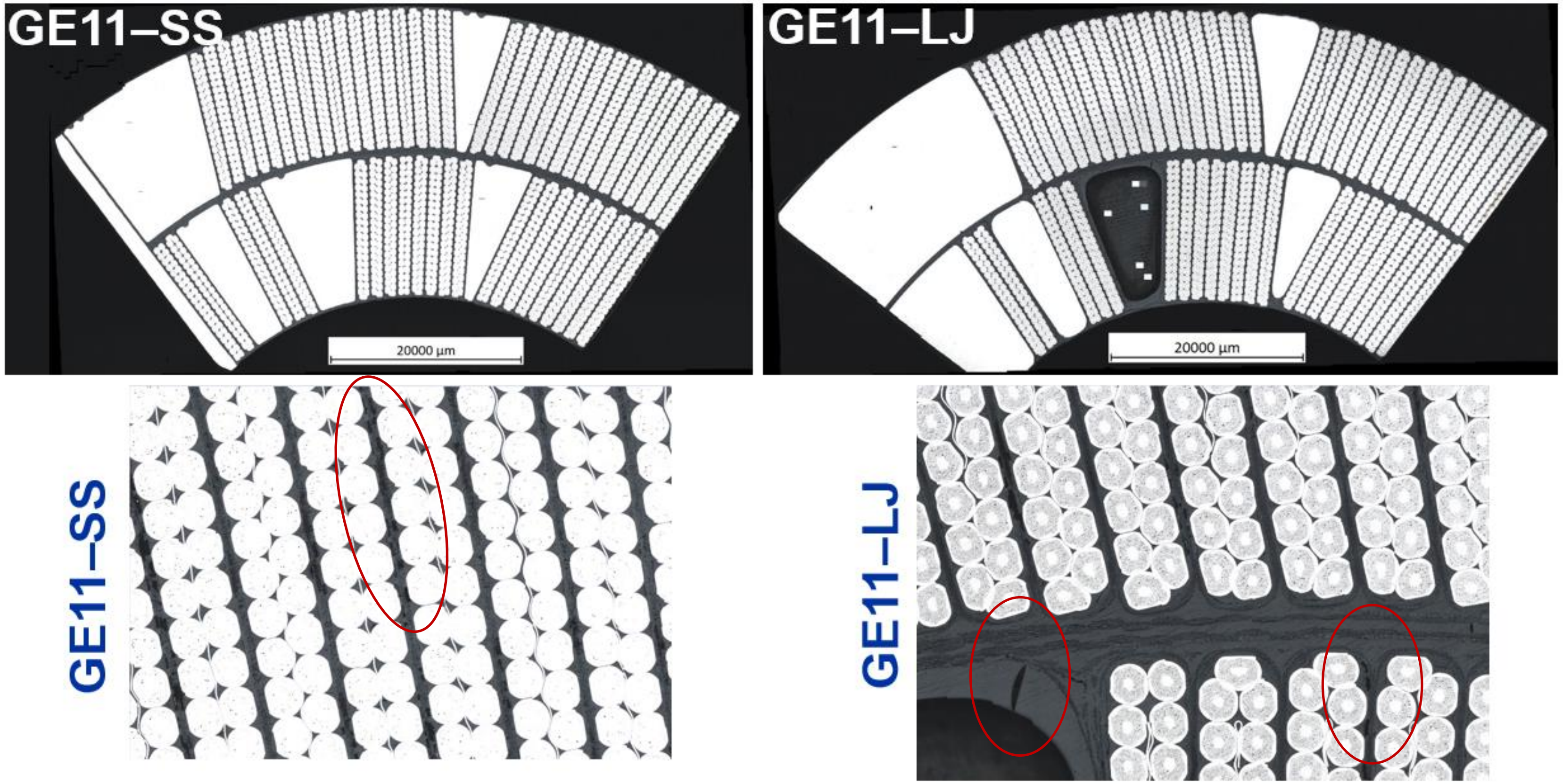
- The question was raised about the **effect that the sensor holes and surrounding epoxy could have on the measurement results,** namely that the He presence detected could come as a result of sample preparation
- A **copper validation sample** was prepared to test this hypothesis, prepared in the same way as the coil samples (epoxy-filled hole for sensor + insulation)
- Time constants obtained are low, as excepted for a bulk sample, ≈ 0.3 s. Data set confirms measurement principle and that **for temperatures around T_λ there is no observable He presence in the sample**.



11T virgin coil GE11 – Results EN/MME

- **Work carried out by EN/MME**, their findings reported in EDMS 2646199 (link)
- Macro- and micro-optical imaging of samples before the thermal cycling tests
- **Cavities** can be observed **in the resin/glass fibre system located in the interlayer and between cables**.

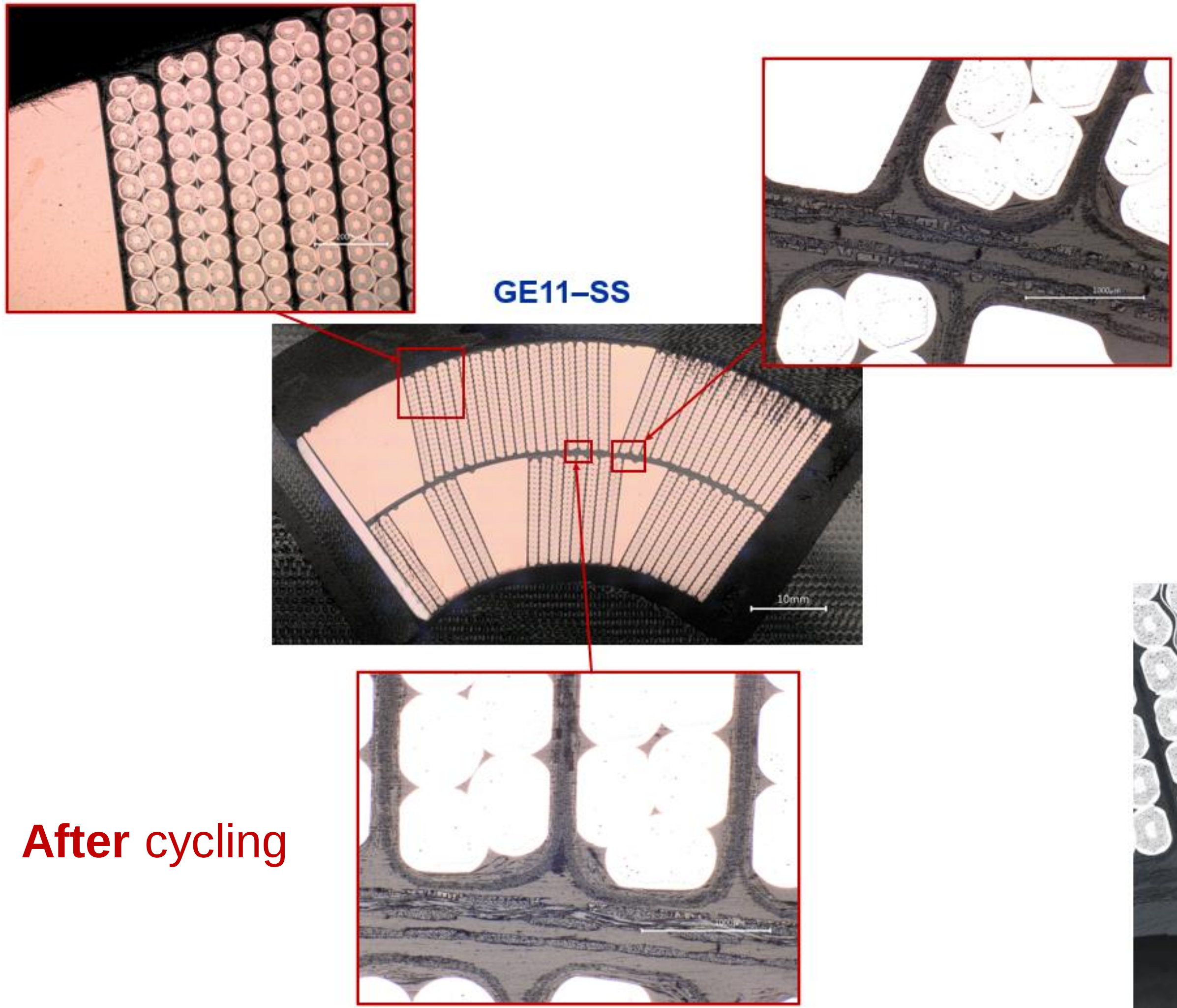
Before sample prep + cycling



Courtesy M. Crouvizier, S. Sgobba

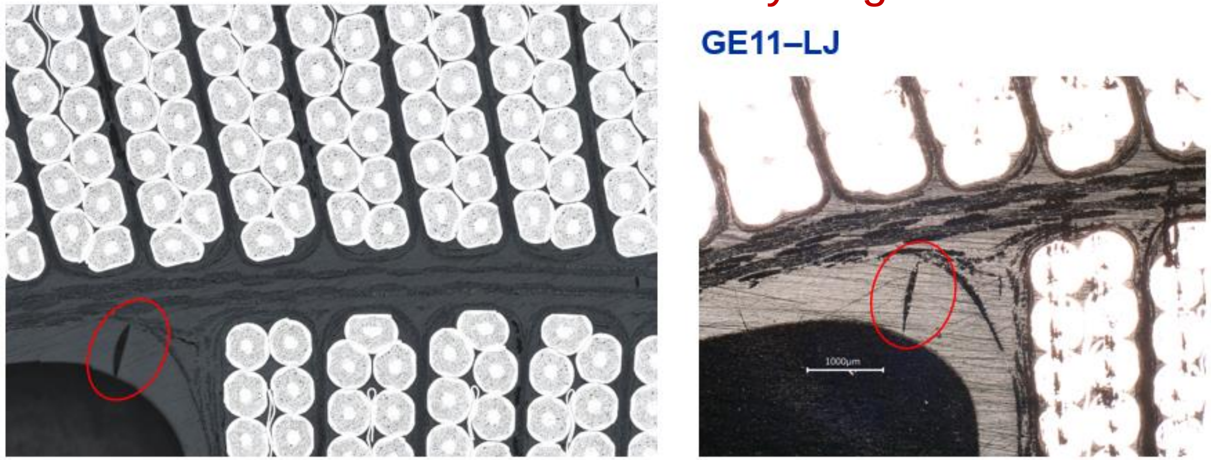
11T virgin coil GE11 – Results EN/MME

- Extra machining/preparation required, making **one-to-one comparison difficult**
- **GE11-SS:** new population of imperfections observed, however the same kind and number of cracks and cavities were observable in the insulation after the test.
- **GE11-LJ:** a deep cavity could still be observed after removal of approx. 150 µm.
- To the extent of present examination, **thermal cycling in He II seems not to lead to a visible further development of micro-cracks and cavities**.



After cycling

Before → **after** cycling



Before helium thermal cycling test

After helium thermal cycling test

Courtesy M. Crouvizier, S. Sgobba

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- Experimental campaign
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  - Set-up description and measurement procedure
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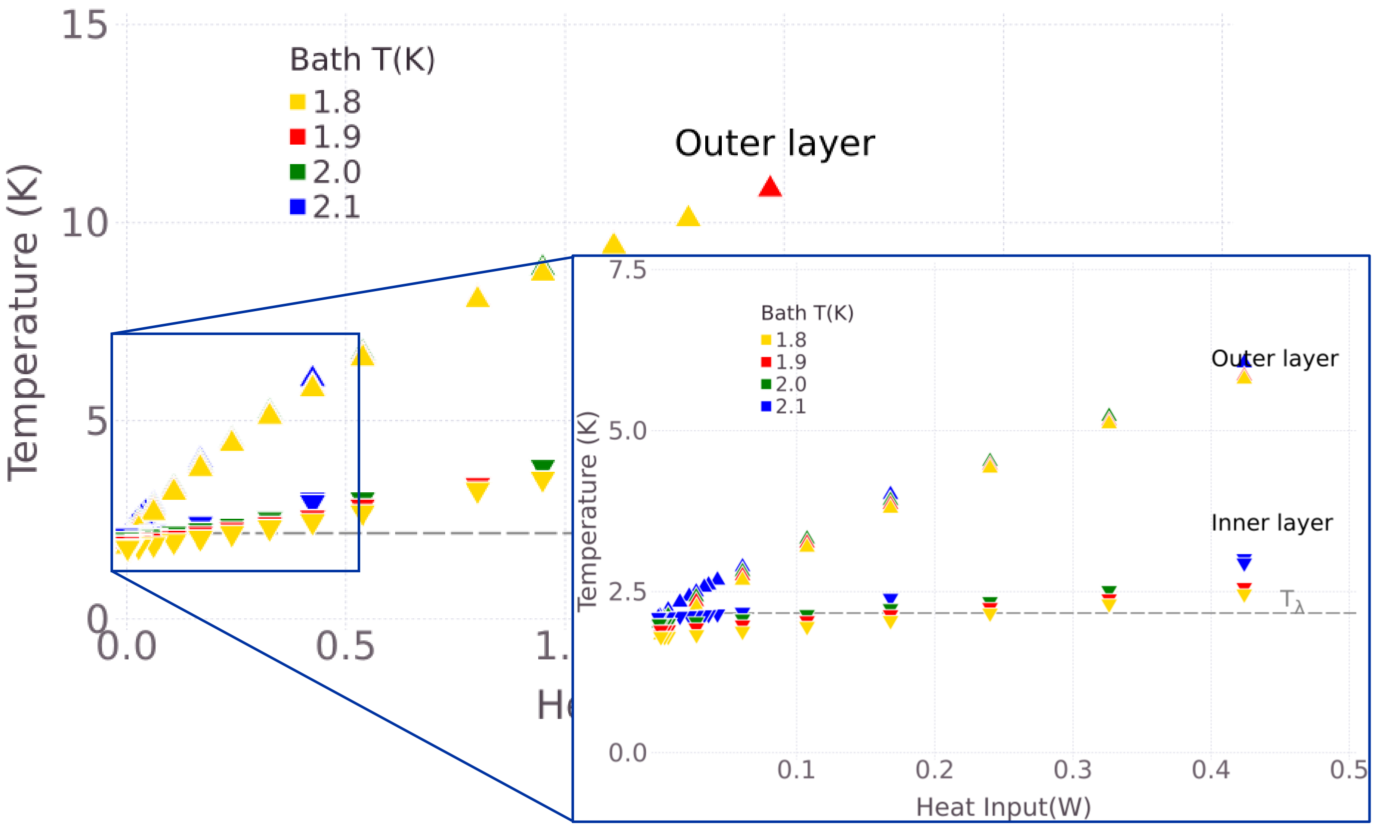
  - Example of 11T coil GE-02
- 11T virgin coil campaign: thermal cycling / He content
- MQXF coil (U.S. and CERN production) campaign: heat transfer
- Summary
- Outlook

MQXF coil P06 (U.S. production) – Results

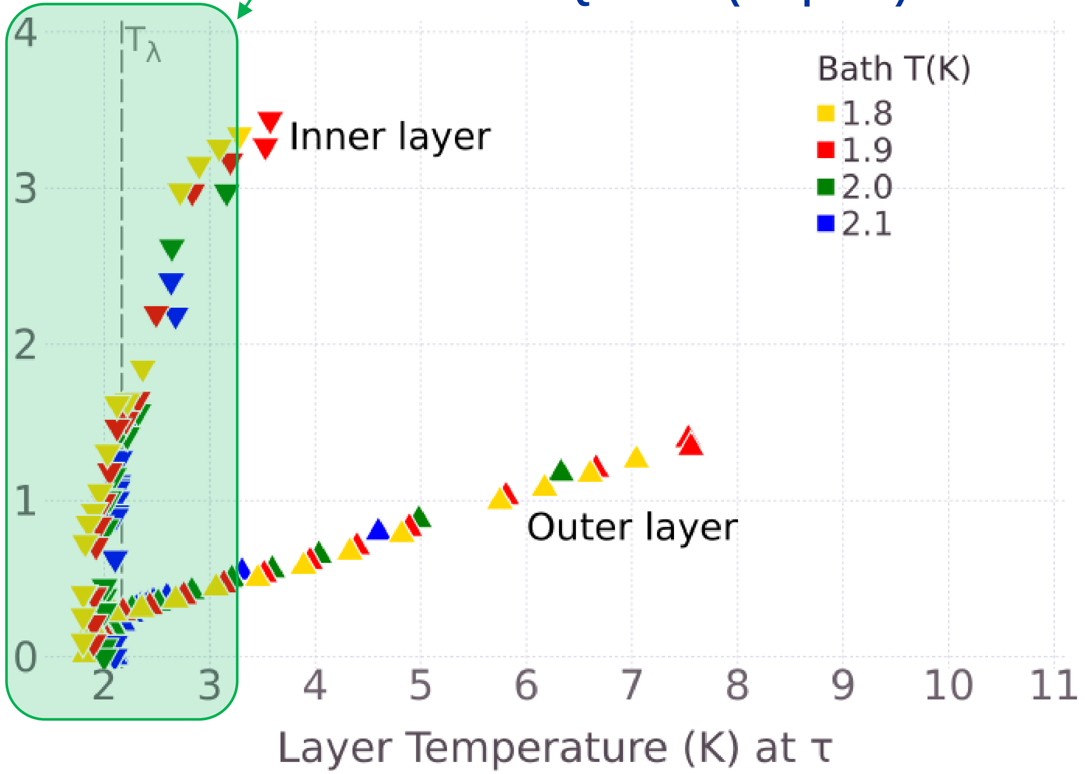
- MQXF specimen cut from coil P06, which was tested in MQXFAP1b
- It is made of the final conductor (RRP108/127)

No signs of He presence

Steady-state behaviour – MQXF P06 (US prod.)



Transient behaviour – MQXF P06 (US prod.)

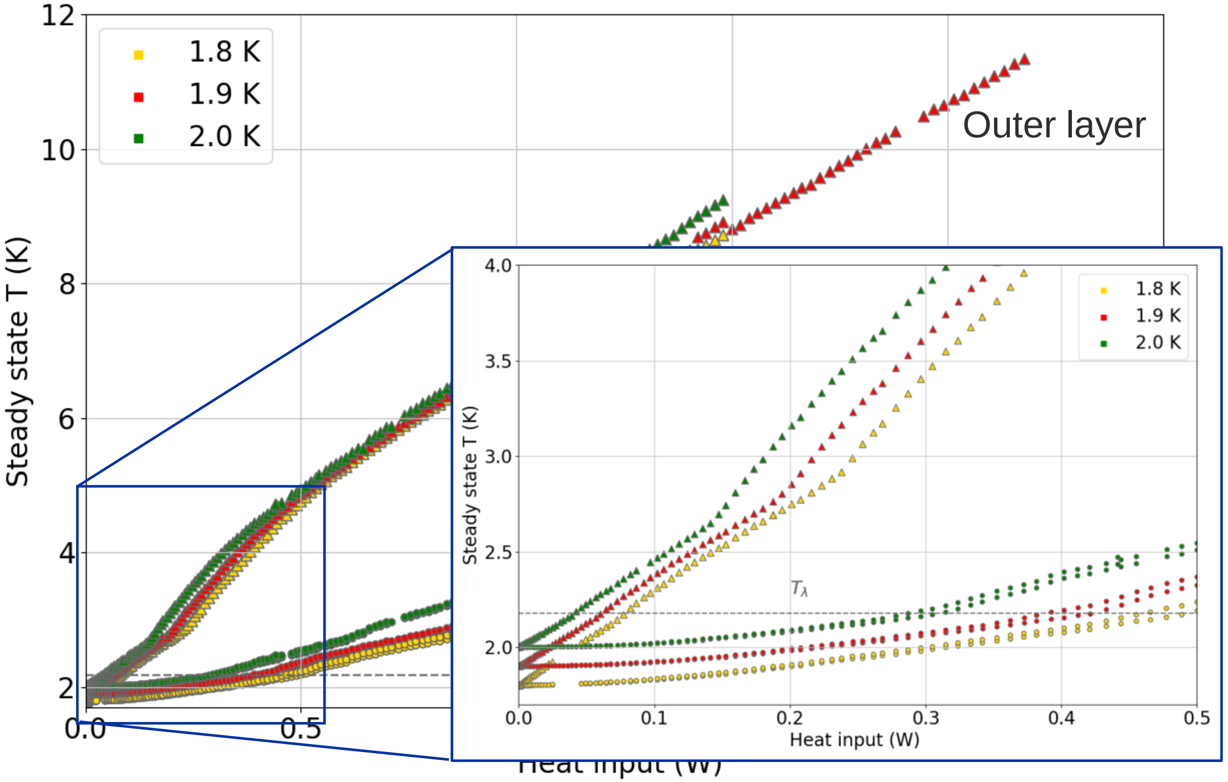


MQXF coil CR108 (CERN production) – Results

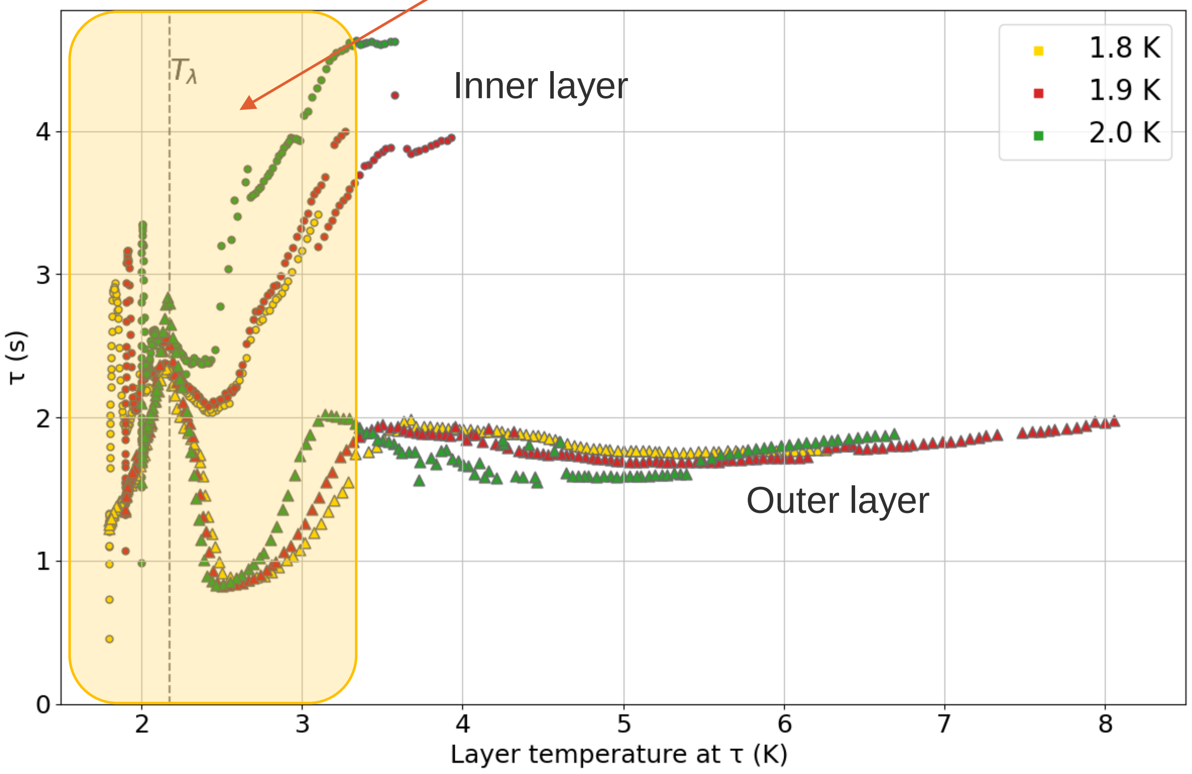
- MQXF specimen cut from coil CR108, section S5, which was tested in MQXFBP1
- Source: https://indico.cern.ch/event/1034788/

He presence signature, *i.e.* λ-peak shape due to c_p of helium

Steady-state behaviour – MQXF CR108-S5 (CERN prod.)



Transient behaviour – MQXF CR108-S5 (CERN prod.)



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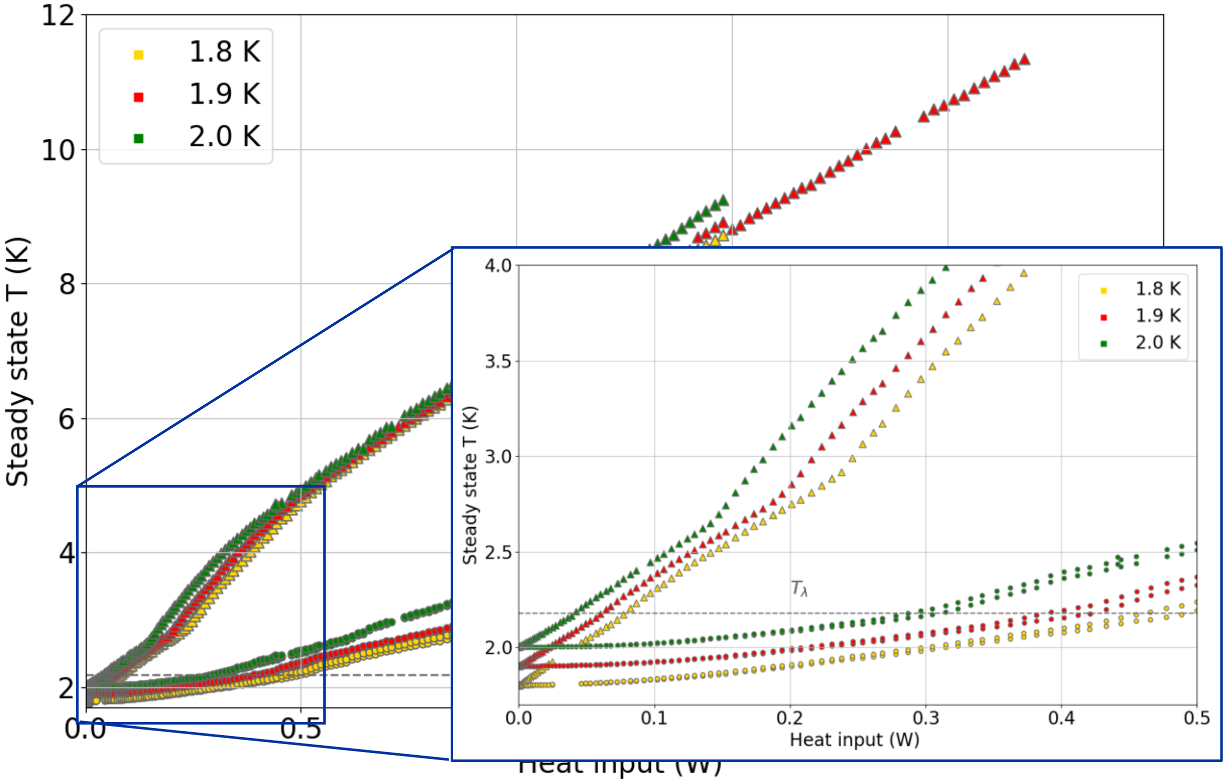

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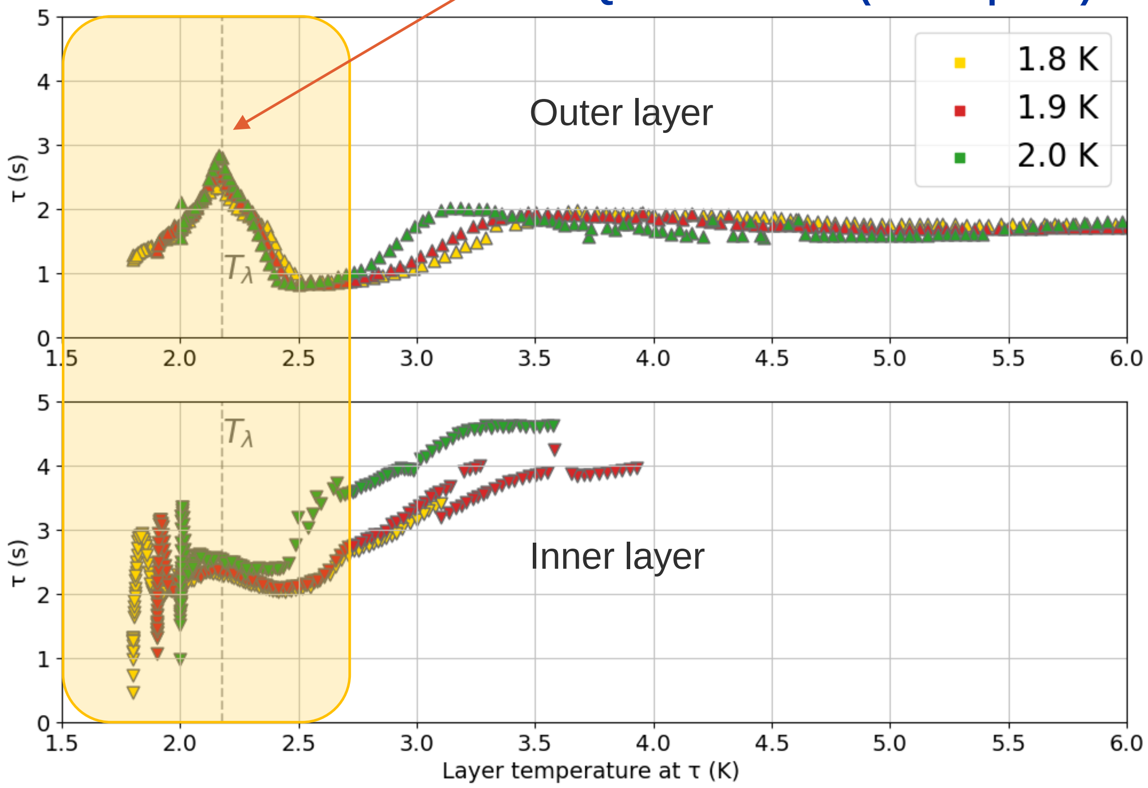
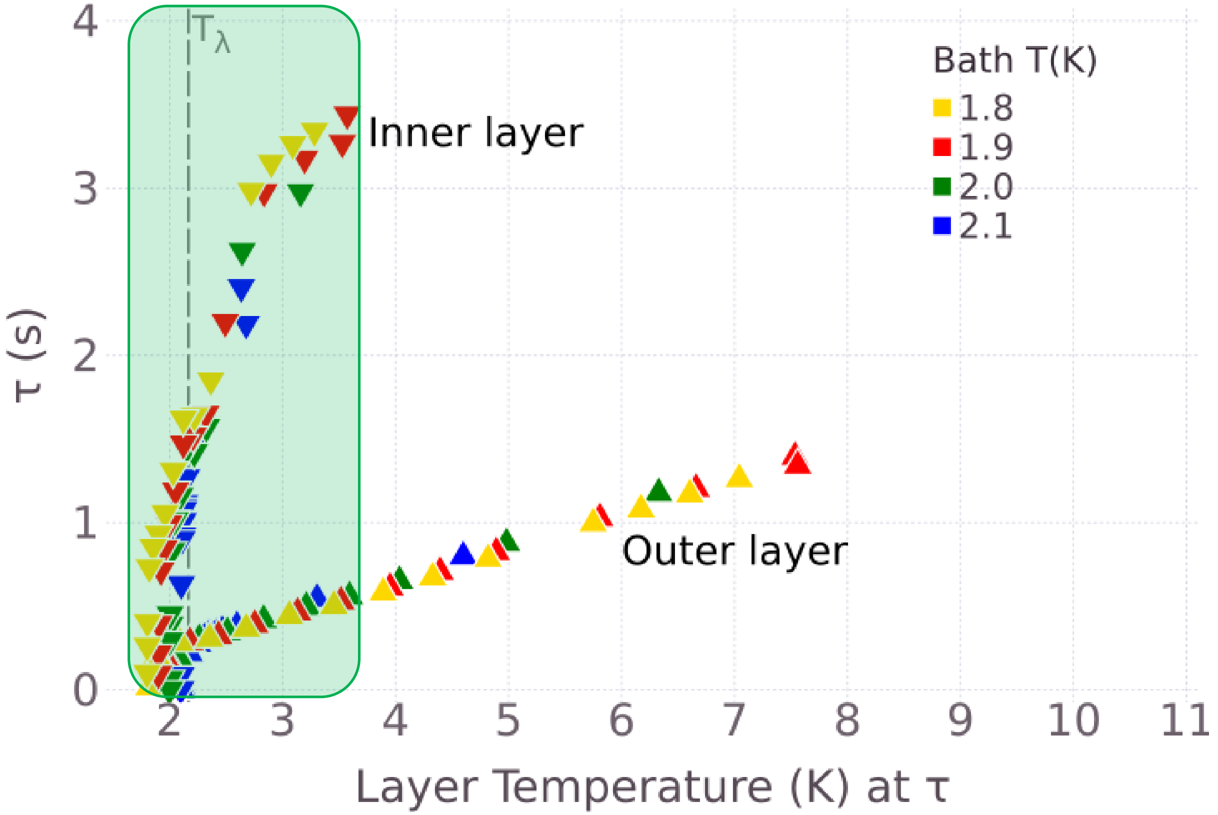


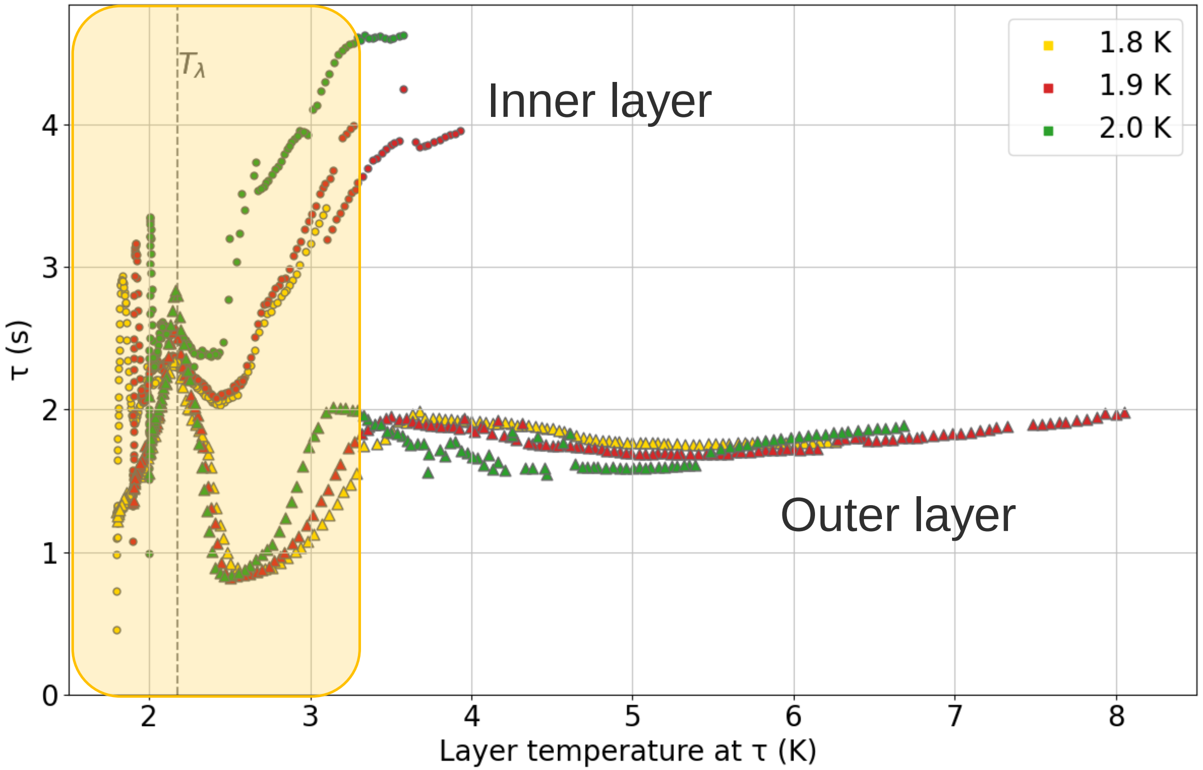
Side-by-side comparison: MQXF P06 *vs.* CR108

Transient behaviour – MQXF P06 (US prod.)



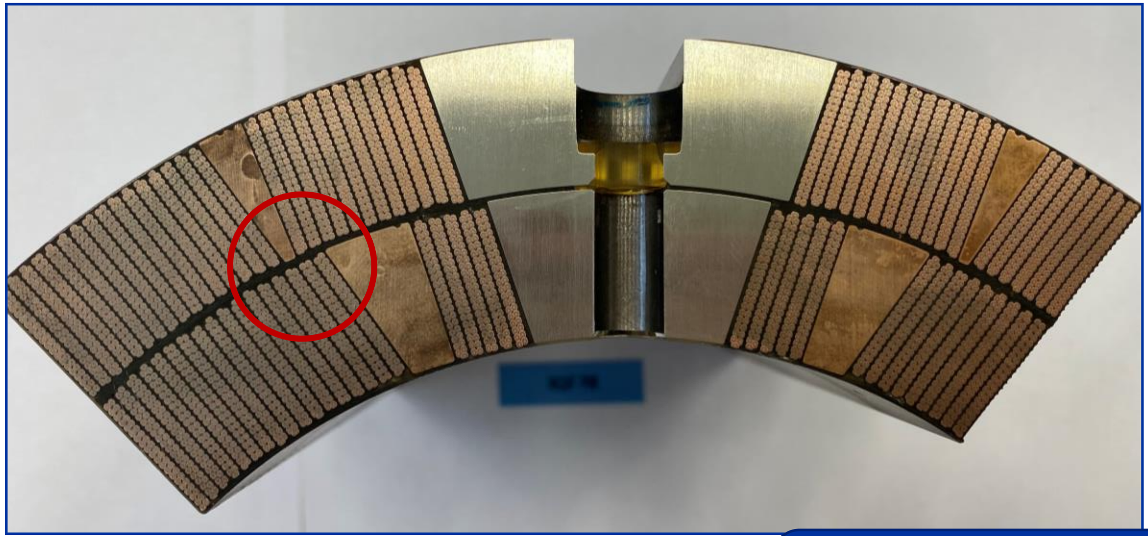
P06: No signs of He presence

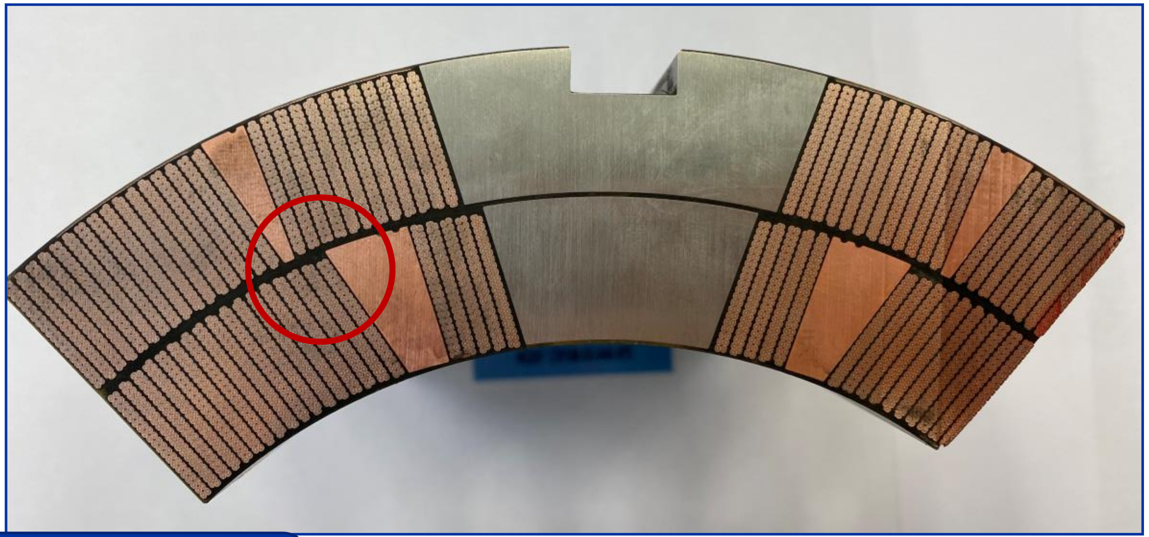
Transient behaviour – MQXF CR108-S5 (CERN prod.)



CR108-S5: He presence signature

MQXF: visual inspection of cut samples

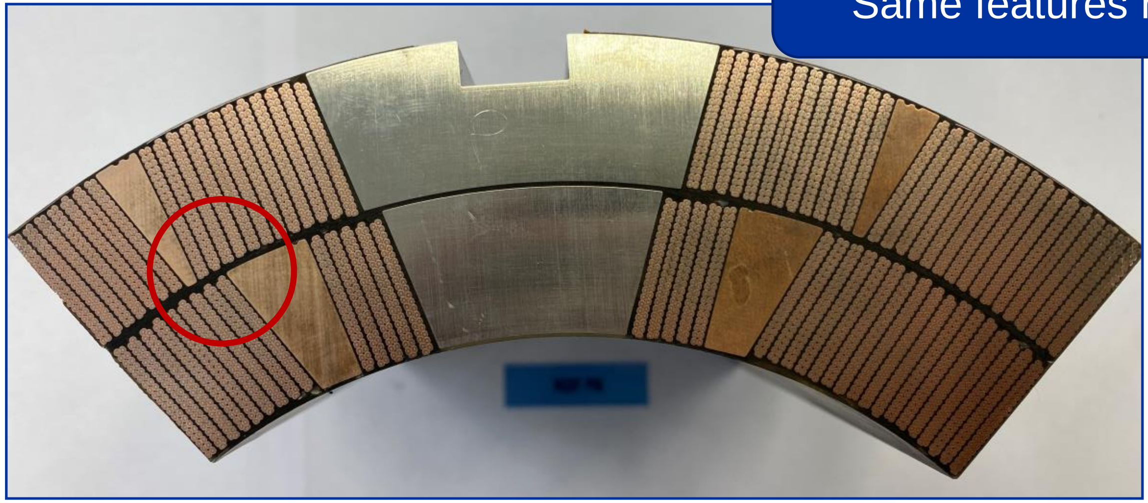


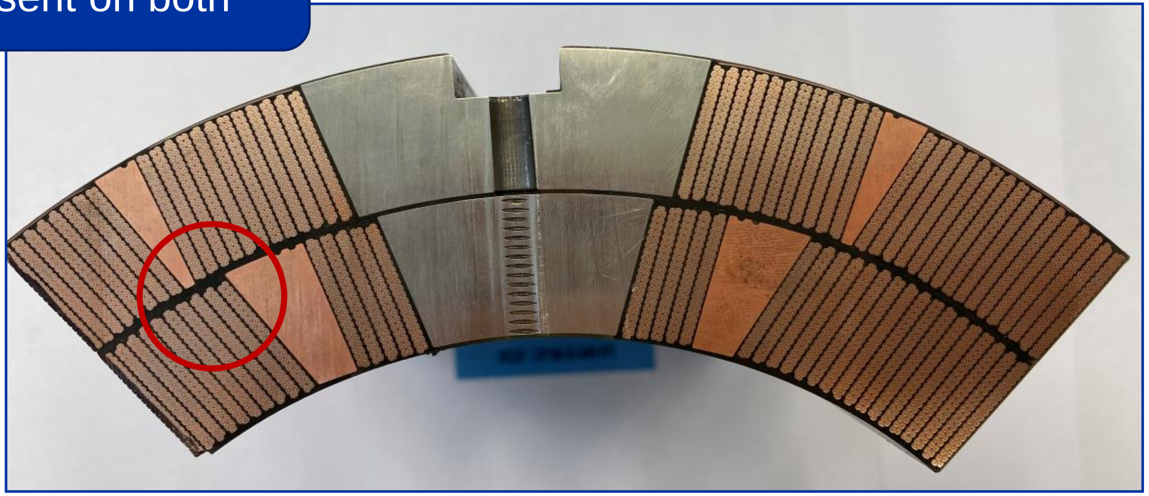


MQXF P06 (U.S. produ

XF CR108 (CERN production)

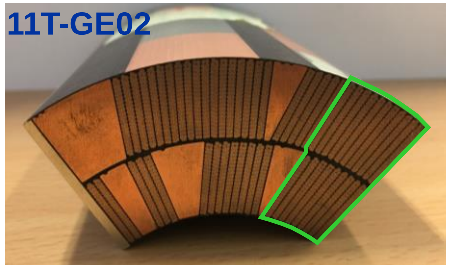
⚠ Only two cross-sections of whole coils,
Same features may be present on both

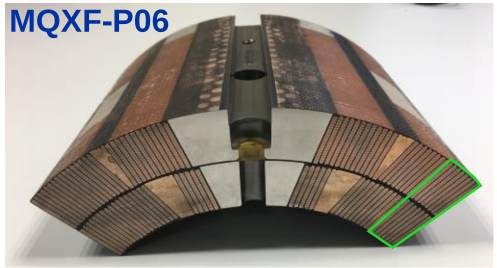


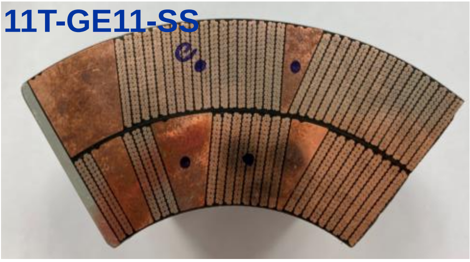


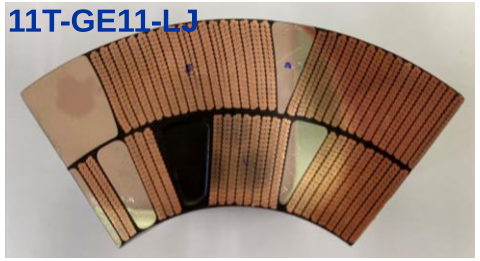
Summary of measured samples @ the Cryolab

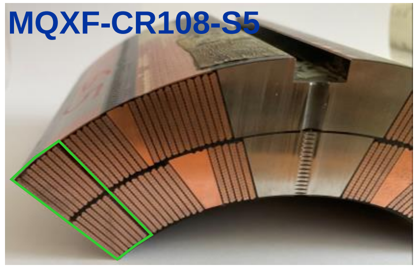
Cryolab Name	MQXF / 11 T	Production / Prototype	Manufacturer	Type of test	Period of testing	# Meas. Runs	He content?	Comments	Coil trace
D11T Proto	11 T	Prototype	CERN	He II Heat Transfer	2017	1	Yes – in both layers	Induction heating	In-house
MQXF LARP07	MQXF	Prototype	USA	He II Heat Transfer	2018/19/20	3	Yes – in both layers	Induction heating	LARP (tested in MQXFS3)
D11T GE02	11 T	Production	CERN	He II Heat Transfer	2020	1	Yes - in inner layer	Induction and Joule Heating	Coil GE02 (HCMBH_C005-01000002)
MQXF P06	MQXF	Production	USA	He II Heat Transfer	Feb-21	2	No indication of He presence	Joule Heating , Press + Sat conditions	Coil P06, MQXFAP1b
MQXF CR108	MQXF	Production	CERN	He II Heat Transfer	Oct-21	2	Yes – in both layers	Joule Heating, saturated cond.	Coil CR108 S5 cut, CR108-S5-840-975
D11T GE11-LJ	11T	Production	CERN	He II content / cycling	Summer 2021	1	Yes - in both layers	Virgin coil, layer jump	Coil C11, GE11, connection side
D11T GE11-SS	11T	Production	CERN	He II content / cycling	Summer 2021	1	Yes - in both layers	Virgin coil, straight section	Coil C11, GE11, connection side











MQXF coil impregnation – CERN process evolution

Tested at Cryolab

108-109 | 201-202-203-204-205-110-111-112-113-114 | 115-116-117-118-119-120 | 121 122-123-124-125 84°C

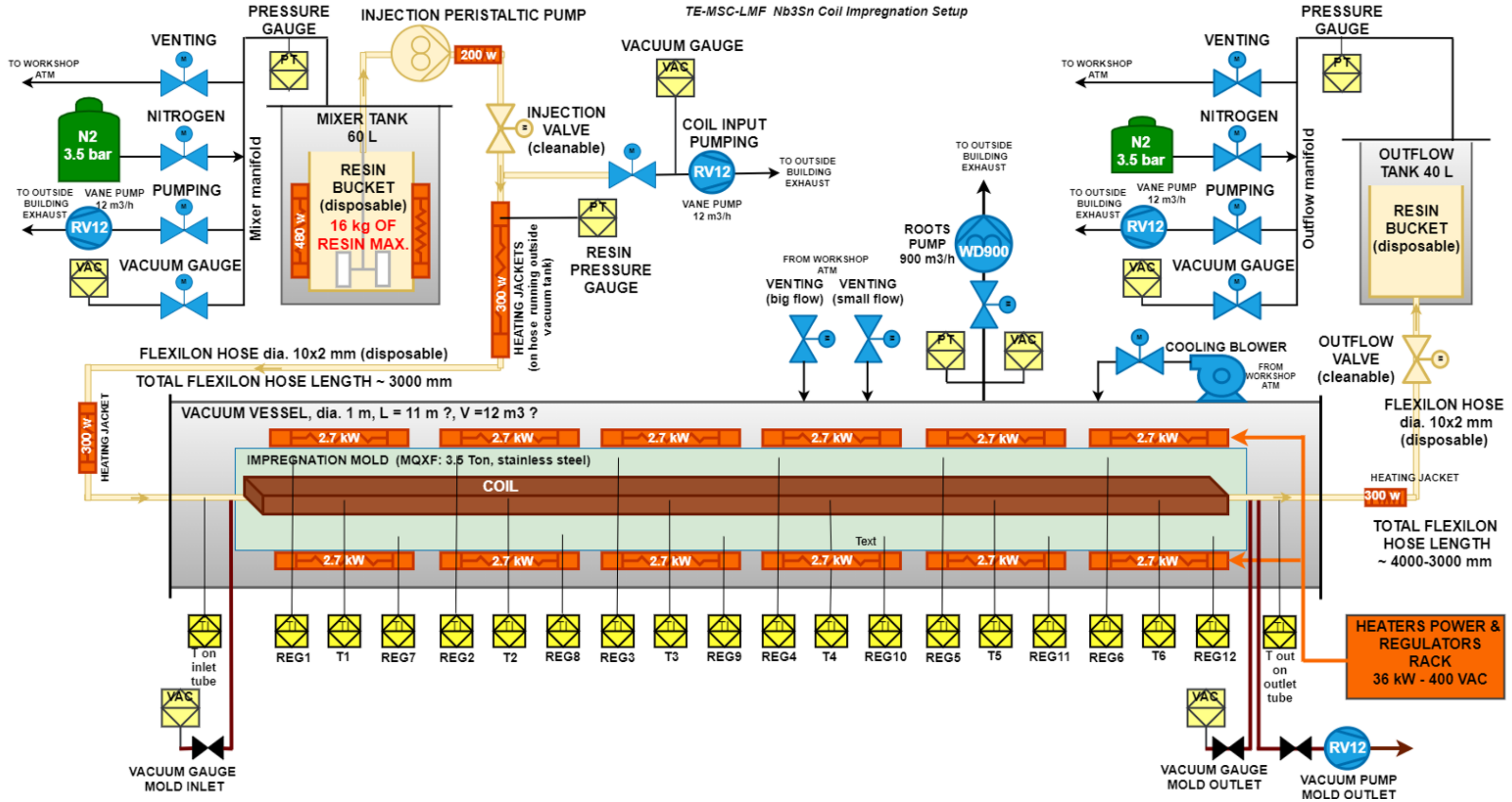
Coils on MQXFBP3

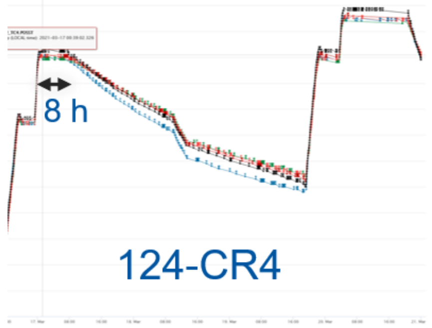
Jan 2019 – Added soaking under vacuum (3 hours)

June 2020 – Added milking process (4 times)

Jan 2021 – Degassing at 110°C, additional pressure gauges

Feb 2021 – New milking criteria, additional vacuum pump





Courtesy: D. Tommasini

Contents

Summary I – General

- Most **CERN-made production samples** of both 11T and MQXF **show a strong signature of He presence**, hinting at differences in the coil impregnation

- Measurements on 11T coil samples show that this **signature is present even for previously virgin** (*i.e.* non-cooled, non-powered) **coils, and that it evolves with thermal cycling**. Visual inspections support this observation. Effects are more pronounced in the more inhomogeneous parts of the coil.

- **US-made production MQXF coil sample (P06) shows no signs of He presence**.

- Temperature at which **resin outgassing** is carried out **differs between US- and CERN-**produced coils (for all measured coil samples): CERN outgassing T = 80 °C (< 3h), for US T = 110 °C (24h)

Summary II – 11T specific

- Thermal cycling tests carried out in **virgin 11T coil GE11 samples show that there are cracks/pores from the start, and that these evolve** with cycling; usually other tested samples have already seen cycling/quenches.

- Metallography analysis carried out by EN/MME **shows cracks in both resin and cables**

- Sample from 11T coil GE02 was sent to TE/VSC for investigation of He diffusion/outgassing. Results show that the **coil sample is porous, and that He is embedded into the bulk**.

- TE/VSC comments that the shape of He outgassing rate is unconventional and **cannot be explained solely by diffusion**. Also observed that there was a significant **amount of water outgassing**, which TE/VSC attributes to the resin and comments is to be expected.

Outlook

- **Ongoing efforts to accurately quantify the heat losses through the insulation**, exploring new sample preparation / measurement solutions
  - Previous and present results provide a **maximum heat transfer coefficient**
  - Further reducing heat losses can provide more accurate values
  - Heat transfer coefficients / Kapitza resistance **can be highly sample-dependent**

- Obtained **material properties are valuable input for simulations** of temperature maps of full coils
  - 11T presented by Kirtana Puthran (link)
  - MQXF ongoing, **results expected this spring**

- Possibility of **testing state-of-the-art coil samples (w.r.t. insulation)** remains open