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# Thermal conductivity measurements of impregnated Nb<sub>3</sub>Sn coil samples in the temperature range of 3.5 K to 100 K

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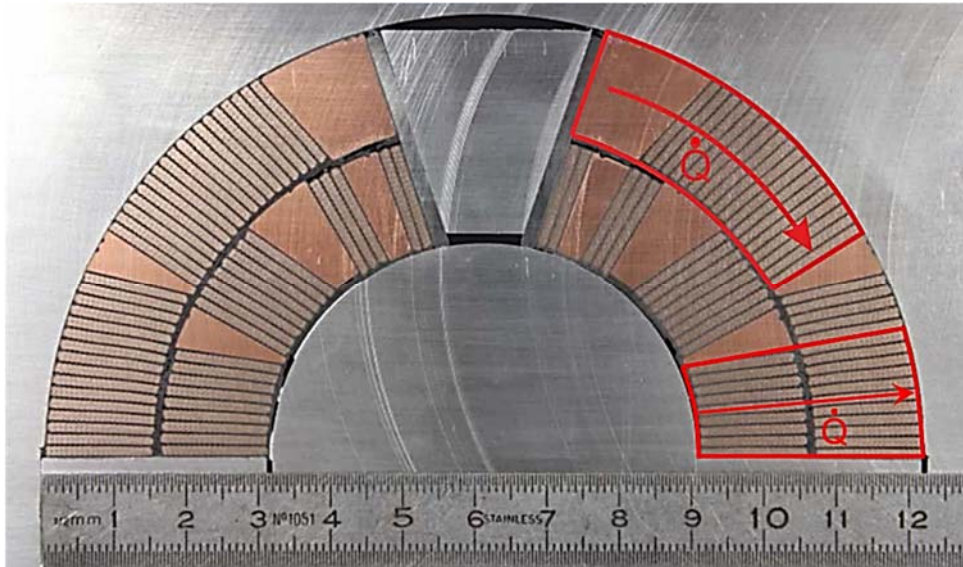
**Abstract.** In the framework of the luminosity upgrade of the LHC, high-field magnets are under development. Magnetic flux densities of up to 13 T require the use of Nb<sub>3</sub>Sn superconducting coils. Quench protection becomes challenging due to the high stored energy density and the low stabilizer fraction. The thermal conductivity and diffusivity of the combination of insulating layers and Nb<sub>3</sub>Sn based cables are an important thermodynamic input parameter for quench protection systems and superfluid helium cooling studies. A two-stage cryocooler based test stand is used to measure the thermal conductance of the coil sample in two different heat flow directions with respect to the coil package geometry. Variable base temperatures of the experimental platform at the cryocooler allow for a steady-state heat flux method up to 100 K. The heat is applied at wedges style copper interfaces of the Rutherford cables. The respective temperature difference represents the absolute value of thermal conductance of the sample arrangement. We report about the measurement methodology applied to this kind of non-uniform sample composition and the evaluation of the used resin composite materials.

## 1. Tested sample geometries

Samples for thermal conductivity are cut from one of the first 11T dipole model superconducting coils. The typical two layer coil structure (one half shell) is shown in figure 1. The coil is encapsulated in a metal structure to enable machining of slices of the coil. A coil sample with an overall length of 40 mm was provided to carry out the thermal conductivity tests [1]. Further machining of the coil has been done in the indicated way to create two representative samples. The samples are probing the thermal conduction along the azimuthal direction along the Rutherford cable stack with the impregnation layers in between or across the two layers of the coil in radial direction, figure 1. The Rutherford cable is composed of Nb<sub>3</sub>Sn filaments embedded in a copper matrix of 0.7 mm strand diameter. The Rutherford cable consists of 40 strands. The cable insulation is composed of a Mica tape of 80 µm thickness shaped in the form of a “C” around the cable, and surrounded by a 70 µm thick braiding of S2-glass fibre [2]. The whole coil undergoes a thermal cycle at 650°C under Argon atmosphere and after it is impregnated with epoxy resin CTD-101K<sup>®</sup> from Composite Technology Development. During the winding step, a binder is brushed onto the model coils to allow their manipulation, CTD-1202 [System CTD-1202, from Composite Technology Development]. The intercoil layer, present between the two coil layers in case (B) shown in figure 2, consists of a S-2 Glass [933 S-2 Glass 66TEX, from AGY] weave and binder CTD-1202. The binder undergoes a pyrolysis with the thermal cycle and the byproducts will also be incorporated in the epoxy resin. The nominal thickness of the insulation under 35 MPa of pressure is 100 µm. The thickness of the insulation was measured for this specific sample with an optical



microscope, finding  $140 \mu\text{m} \pm 10 \mu\text{m}$  as the actual insulation thickness, providing a total insulation between coil turns of  $280 \mu\text{m} \pm 20 \mu\text{m}$ . In the radial direction, the nominal insulation thickness is  $700 \mu\text{m}$  ( $500 \mu\text{m}$  of inter layer insulation +  $2 \times 100 \mu\text{m}$  of conductor insulation). The measured thickness of insulation was  $1240 \mu\text{m} \pm 90 \mu\text{m}$ . Bubbles were visible on the surface of the epoxy between the two coil layers, covering roughly one third of the surface area. The depth of the cavities was difficult to determine in an optical measurement, but they extended to a depth of at least  $130 \mu\text{m}$ .



**Figure 1.** Picture of the 11T dipole impregnated coil sample. Cross sectional view of the  $\text{Nb}_3\text{Sn}$  superconducting Rutherford cable in machining support structure. The two tested sample geometries are indicated, the upper one in azimuthal direction of the cable stack with indication of later on applied heat flow (original copper wedge kept). The second sample was cut from the right bottom part across the coil layers with indication of later on applied heat flow direction. The scale shows the diameter of the coil of 122 mm.

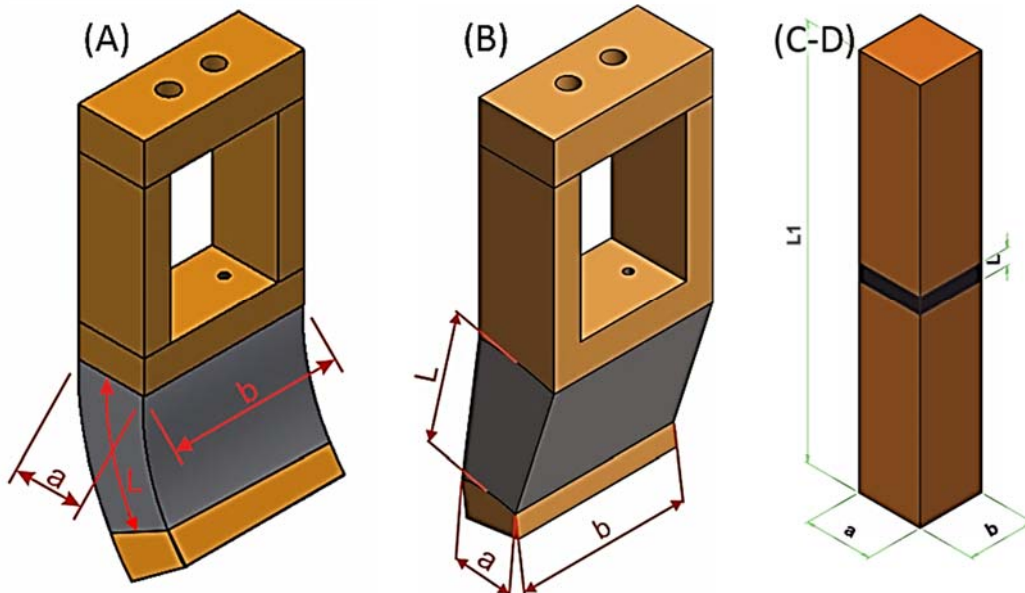
## 2. Thermal conductivity measurement set-up

The steady-state measurement method is used to determine the thermal conductivity in the temperature range between 3.3 K and 30 K (up to 100 K by an additional interface) and a quasi steady-state method during warm up in the range from 100 K – 270 K. The thermal conductivity  $\lambda$  and the thermal conductance  $\Lambda_{tot}$  were determined from the proportionality between applied heat flux  $\dot{Q}$  and recorded temperature difference  $\Delta T$ :

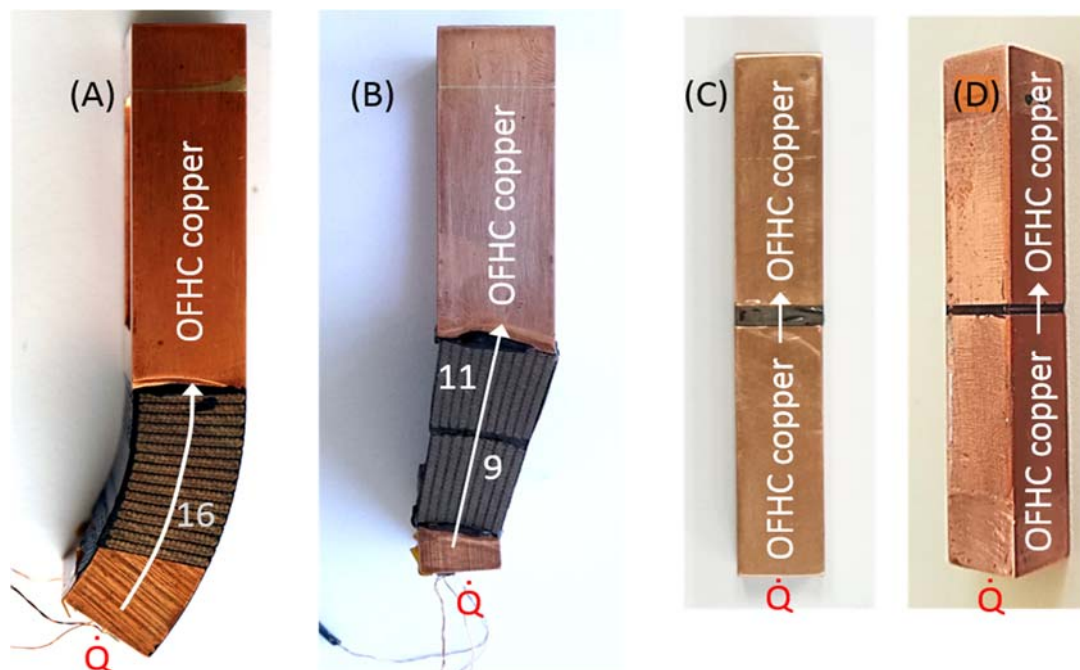
$$\lambda = \dot{Q}L/A\Delta T \quad \text{and} \quad \Lambda_{tot} = \dot{Q}/\Delta T, \quad (1)$$

where  $\lambda$  contains the characteristic dimensions of length  $l$  and cross section  $A$  of the sample, see figure 2. The specimens are cut from the magnet coil and have variable cross section in the heat flux direction, thus  $\Lambda_{tot}$  describes the thermal conductance of the defined coil specimen. Thermal conductivity values are calculated by using average data for cross section (smallest value is used in case of coil (B)) and the length of the impregnated coil. The experimental platform is thermally weakly attached via a copper rod to the second stage of the pulse tube cryocooler (PTC), which reaches 2.9 K no load temperature and can provide up to 1 W cooling power at 4.2 K, see figure 4. The insulation vacuum is pumped to the order of  $10^{-5}$  mbar before cooldown. Higher platform temperatures are achieved by heating the platform or the second stage of the PTC including the thermal shield around the sample. The 1<sup>st</sup> stage of the PTC minimizes the heat load of all incoming instrumentation wires to the 2<sup>nd</sup> stage

and is actively cooling the outer thermal shield at around 35 K. Both thermal shields have a copper 'can' construction, which is covered with one blanket of 10 layer superinsulation that surrounds all respective components.



**Figure 2.** CAD design of the test samples, demonstrating the dimensions used to calculate thermal conductivity values. Left - tangential coil sample (A), middle - radial coil sample (B) and right - resin sample (C and D). The upper part is mounted to the experimental platform, see figure 3 and figure 4.

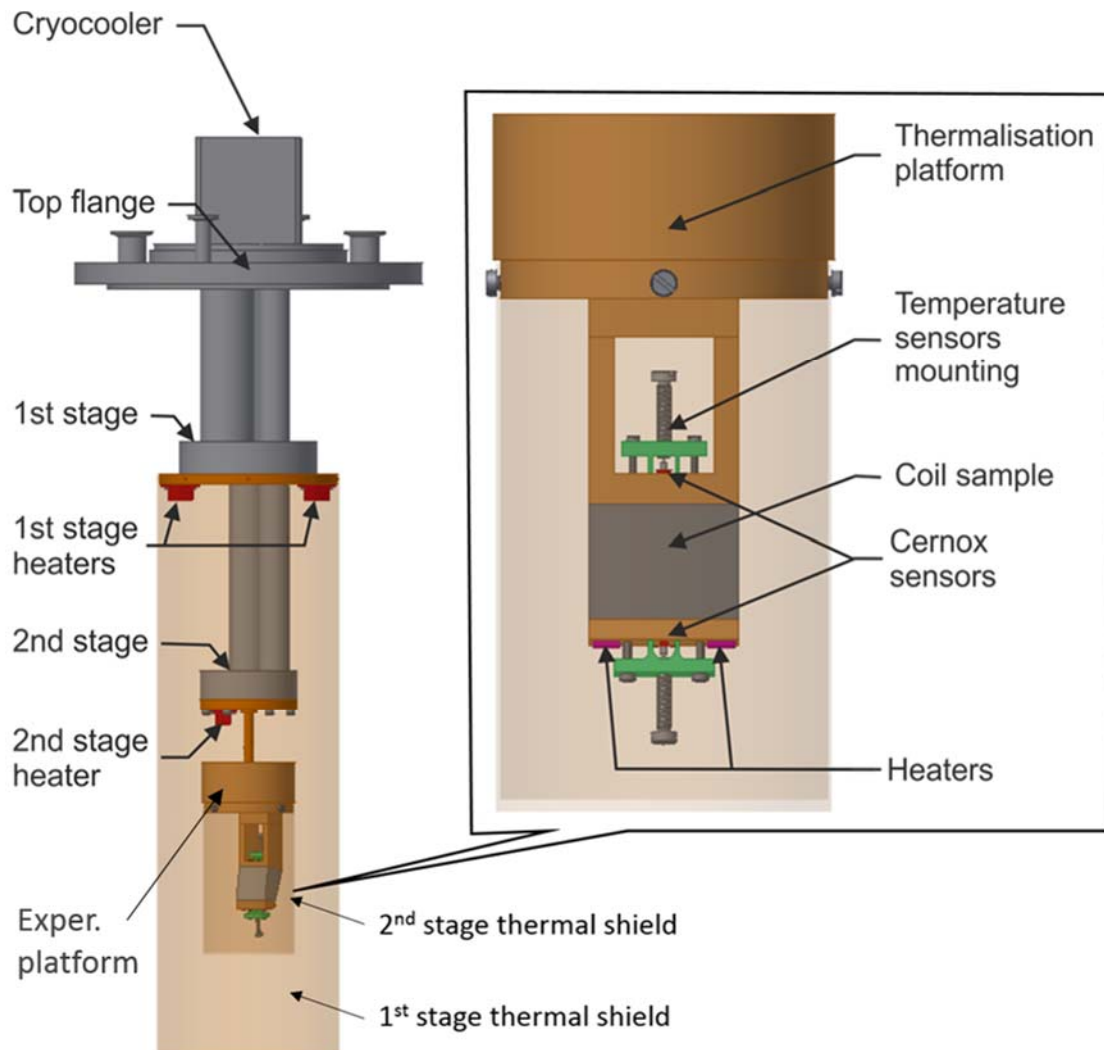


**Figure 3.** Picture of the samples bonded to the oxygen free high conductivity (OFHC) copper supports. The sample geometry for Stycast - sample (C) and CTD-101K – sample (D) is equivalent, see table 1. The stated numbers for samples (A) and (B) are the number of Rutherford cable layers in the respective stack.

**Table 1.** Dimensions of the tested samples for impregnated coils and resin/epoxy adhesive.

Sample	L in mm	Dimensions a x b in mm	Cross section in mm <sup>2</sup>	Remarks
(A) Coil sample	25.5	15.7x39.75	624	A centre length of 25.5 mm is chosen
(B) Coil sample	31.5	13.8x39.75	548	Smallest cross section is used 9 layers of Rutherford cable (12 upper part)
(C) Stycast FT2850	2.1	8.0x8.0	64	Catalyst: Loctite CAT 9
(D) CTD-101K	1.0	8.0x7.5	60	Shrinkage during curing $\Delta b = -0.5$ mm

Given the geometry of the coil samples, the epoxy encapsulant Stycast 2850FT [LOCTITE STYCAST 2850FT, catalyst LOCTITE CAT 9, Henkel], was used as an adhesive to bond some sample surfaces to copper blocks. It consists of an epoxy resin with high content of aluminum oxide in fibrous form.



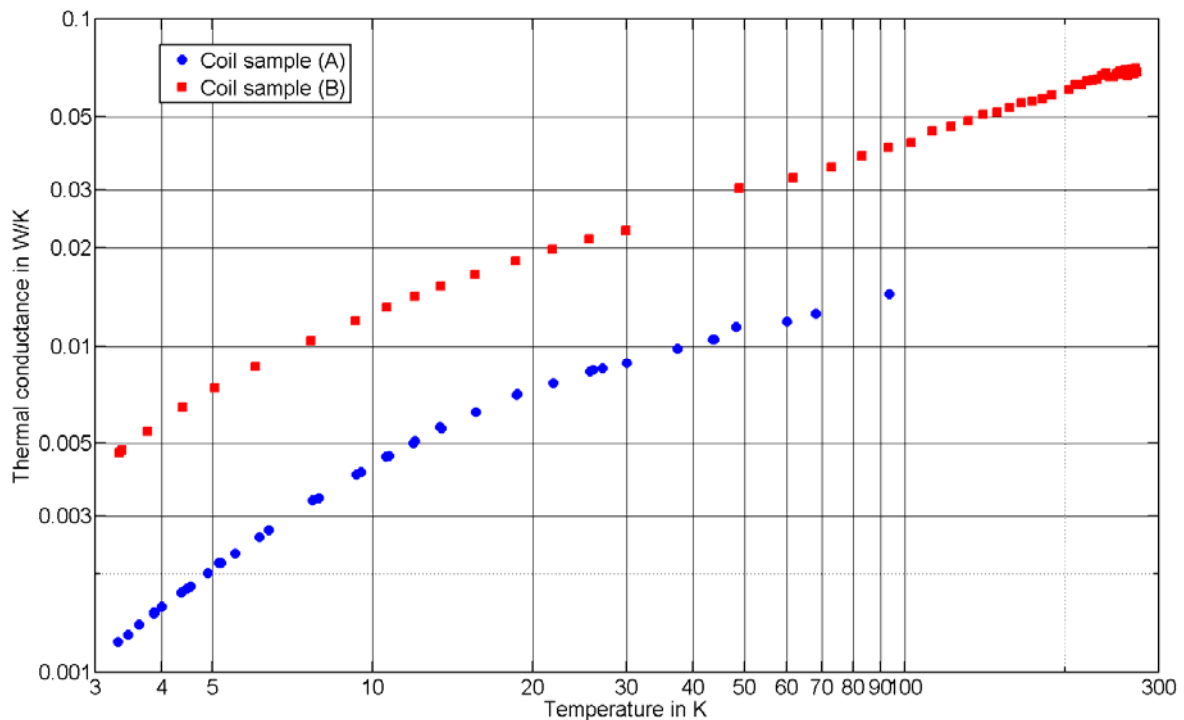
**Figure 4.** Picture of the overall test stand demonstrating the integration of the samples at the thermal conductivity tests stand. The experimental platform is attached to a two-stage pulse tube refrigerator providing 1 W cooling power at 4.2 K.

Heat intercepts are attached to the 1<sup>st</sup> and 2<sup>nd</sup> stage of the PTC for thermalizing instrumentation wires made out of manganin, which are installed in twisted pair scheme. The wires of the electrical heater, which itself is connected to the bottom of the sample are optimized for minimum heat inleak by adapting the cross section of the copper or bronze wires respecting the expected thermal conductivity behavior of the sample (necessary heating power to achieve 0.2 K to 0.3 K temperature gradient across the probed sample distance). The goal is to balance the effect of self-heating and thermal conductance of the current wires, making sure that the temperature difference between the sample heater itself and the experimental platform is smaller than 0.5 K.

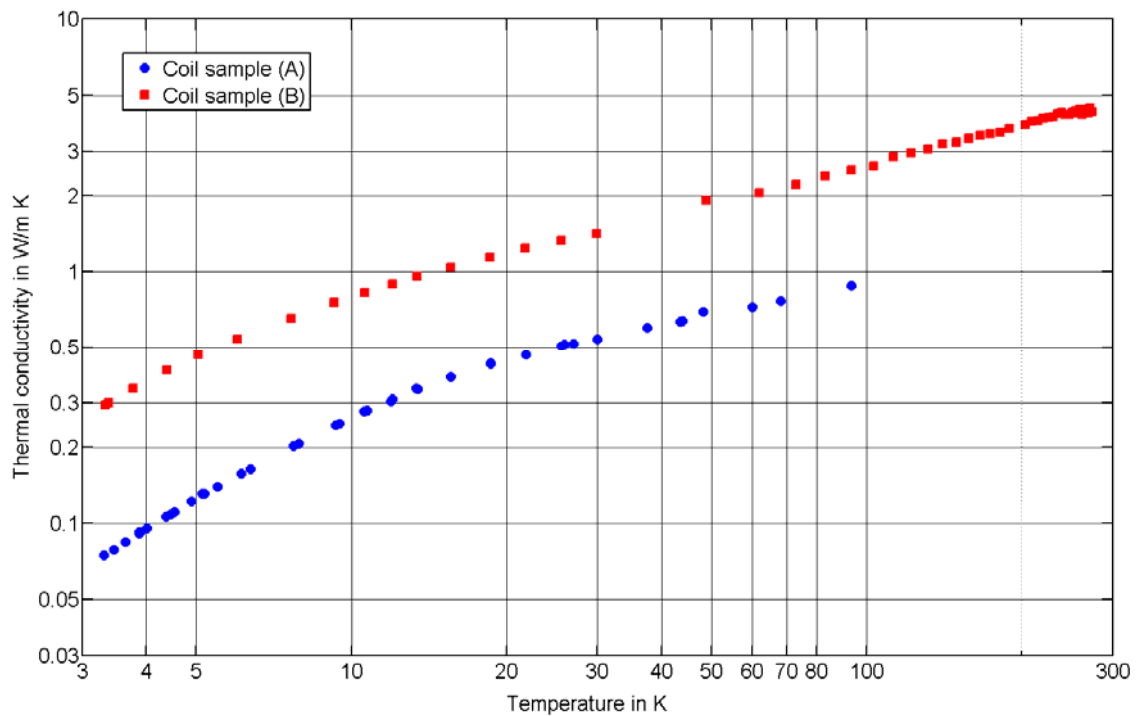
The estimation of the measurement accuracy includes all data acquisition by precision current sources, the nano-voltmeter including a switching card and the heater current source. Sample dimensions are respected with different uncertainties for the coil samples because of the use of averaged length and cross section data compared to the resin tests (C) and (D). The relative error for the thermal conductivity below 10 K is  $\Delta\lambda = \pm 3\%$  and above  $\Delta\lambda = \pm 8.5\%$ . The absolute error for the temperature values is estimated to be  $\Delta T = \pm 8$  mK below 30 K rising to  $\Delta T = \pm 120$  mK at room temperature caused by the reduced sensitivity of the installed Cernox<sup>®</sup> 1050 SD temperature sensors.

### 3. Measurement results

The measured thermal conductance of the coil samples (A) and (B) is shown in figure 5. The difference in sample conductance is a factor 3 to 4 with higher values for sample (B) due to the very different inner geometry. The 17 layers of resin in between the Rutherford cables (16 layers CTD-101K and one layer Stycast FT2850 of 0.2 mm) are dominating the thermal performance of coil sample (A). Coil sample (B) has only 1 layer of CTD-101K in between the cables and 2 layers of 0.2 mm Stycast FT2850 for bonding it to the copper interfaces.



**Figure 5.** Plot of the thermal conductance values of both impregnated coil samples. The conductance value is given because of the special non-uniform shape/geometry of the cut coil samples, compare figure 2.



**Figure 6.** Plot of the thermal conductivity values using the average geometry data of the cross section and thermal path for both samples of table 1.

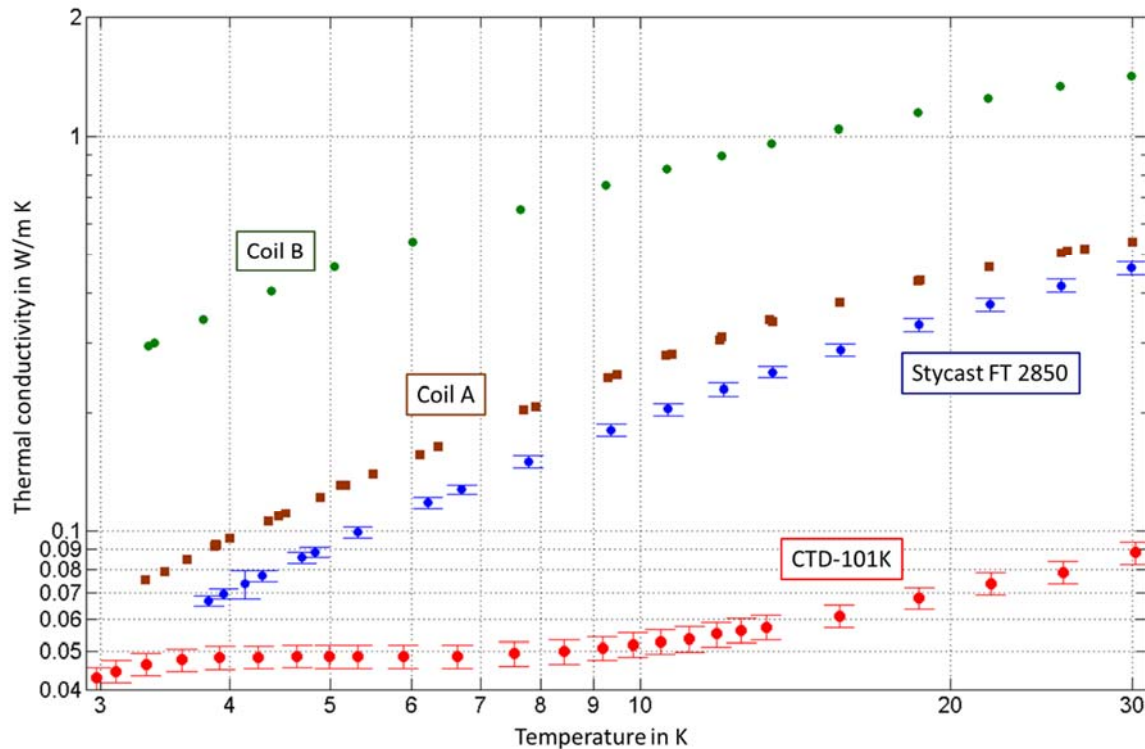
Assuming certain stated geometries for the coil samples (see table 1), it is possible to calculate the thermal conductivity values, shown in figure 6. The overall trend of monotonously rising thermal conductivity values with increasing temperature is maintained.

It is important to check the influence of the used impregnation resin as well as the epoxy adhesive used for bonding the OFHC copper interfaces (bottom copper wedge of sample coil (A) is kept from the original coil structure and has therefore a CTD-101K interface). Both resins have been applied to the same described sample interface used in case (C) and reused for case (D). A precise evaluation of the resin thickness is guaranteed by the measurement of the total length difference at the copper end surfaces before and after preparing sample (C) and (D), compare table 1.

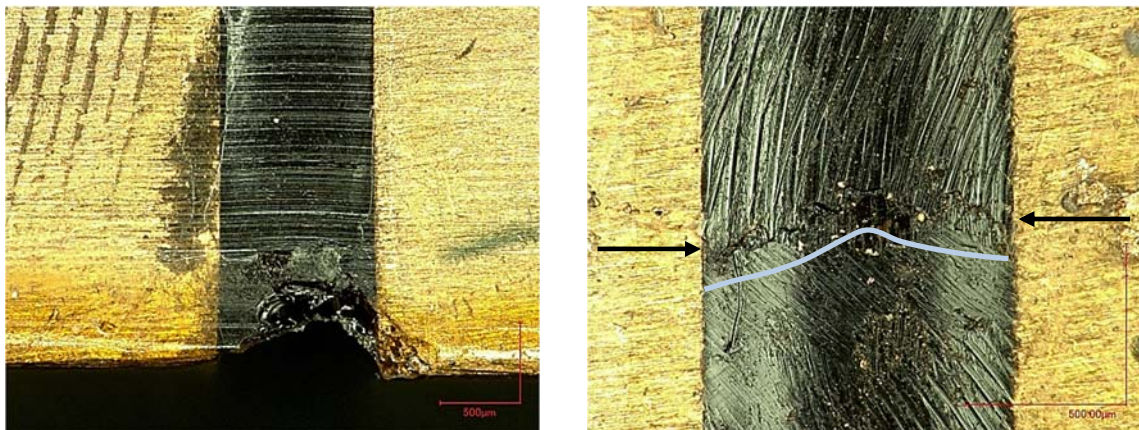
The OFHC copper itself was tested beforehand in the thermal conductivity test stand to evaluate the thermal conductivity data in the required temperature range from 3 K to 290 K. The determined results of e.g.  $\lambda=2250$  W/m K at  $T=25$  K are in good agreement with stated values for RRR=100 data of copper [3]. The typical distance for the temperature sensors to the resin interface layer is 10 mm at each side copper support. The deviation caused by this arrangement is negligible.

Figure 7 compares all measured data including the thermal conductivity values for CTD-101K and Stycast FT2850. CTD-101K, here tested without any filler material, is the matrix material of the insulation composite system and may be present in very small and localized resin rich areas of the coil.

The thermal conductivity data for Stycast are significantly higher than the CTD-101K in the investigated temperature range. Approaching low temperatures  $T < 4$  K the difference in thermal conductivity gets smaller. The measured values of Stycast also support the assumption that the Stycast FT2850 used for bonding the copper support structures to the cut coil packages is not causing a major deviation of the tested coil samples (A) and (B).



**Figure 7.** Plot of the thermal conductivity values of CTD-101K compared to Stycast FT2850 and with CTD-101K impregnated coil samples in the low temperature range. The shown error bars on the CTD-101K data are increased in value to cope with observed micro cracks after warm up and optical inspection. A slight reduction in cross section has been observed at the resin most likely caused during curing of the resin. Exact cross section data is difficult to evaluate, see figure 8.



**Figure 8.** Microscope pictures of the CTD-101K sample in between the two copper interfaces. Left picture shows a reduced cross section for the thermal path. Right picture shows a micro crack in the direction of the thermal path, which is assumed not to have major impact on the measured thermal performance. See the indications by black arrows and the line below the crack.

The two microscope pictures in figure 8 show the CTD-101K sample demonstrating the influence of the geometry to the evaluated thermal conductance and conductivity data. A slight deviation in the copper block alignment is visible and the tendency of micro cracks most likely caused by the thermal cycling during the measurement. These effects are included in our error estimation for CTD-101K plotted in figure 7.

#### 4. Conclusions

A highly versatile measurement set-up to determine thermal conductivity has been built and commissioned in the Central Cryogenic Laboratory of CERN. The set-up is used to determine the thermal conductance and conductivity data of a variety of non-uniform samples. The instrumentation of the set-up is adapted to the expected thermal conductance of the sample minimizing the influence of bypassing heat flows or external heat inleak.

First data of the thermal performance of impregnated Nb<sub>3</sub>Sn coil samples indicate the necessity to further study the influence of the impregnation resin in combination with the filler materials as well as the response of the samples to transient heat loads. That input is essential for numerical simulations of Quench behaviour and time constants for the cooling in a He II bath without direct fluid contact to the Rutherford cable strands.

#### References

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