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Swiss Accelerator Research and Technology

UNIVERSITY OF TWENTE.

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Progress in materials and processes at PSI

This work was performed under the auspices of and with support from the Swiss Accelerator Research and Technology (CHART) program (www.chart.ch)

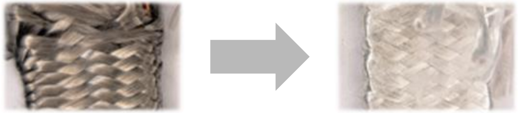
Acknowledgment

Many thanks to the US Magnet Development Program and the Lawrence Berkeley National Laboratory for providing the Nb_3Sn cable.

Overview

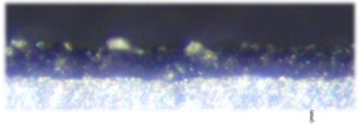
Burning stuff

a route to «cleaner» magnets



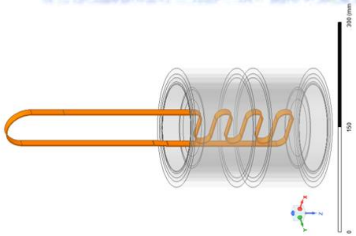
A glass ceramic coating for stainless steel

improved insulation



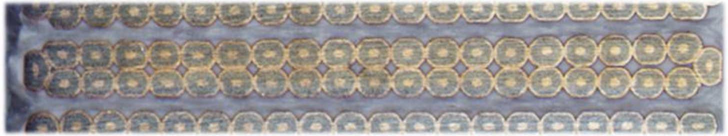
The Box programm

fast turnaround testing of impregnation systems

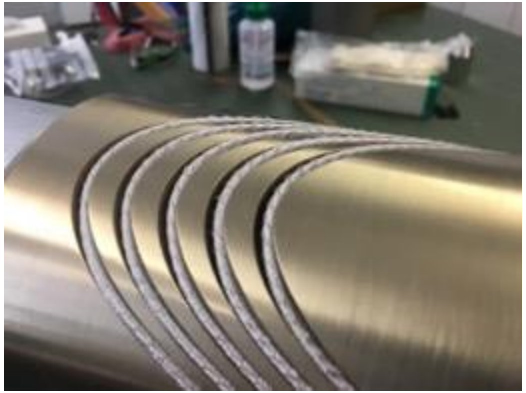


Filled impregnation systems

lower CTE and increased modulus

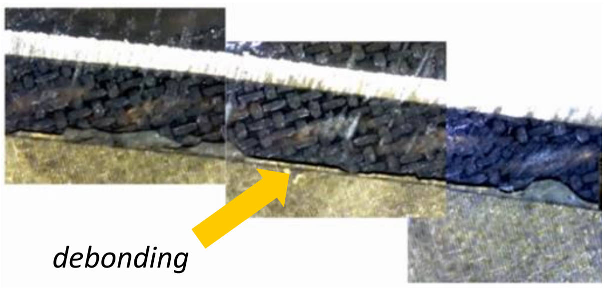


Burning stuff - motivation



before heat treatment

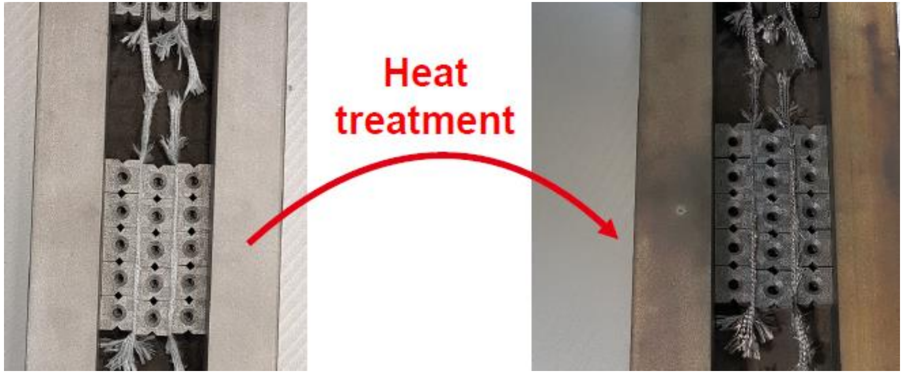
after heat treatment

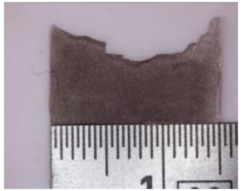


Courtesy D. Arbelaez, LBNL.

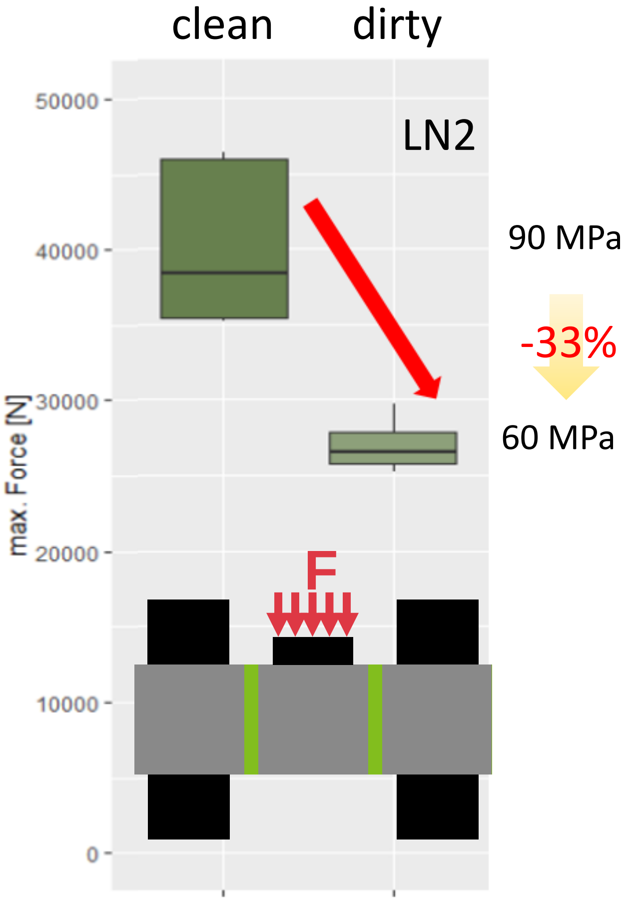
after impregnation and cool-down

Adhesion– polluted surfaces





Polluted surface on a fractured epoxy fragment

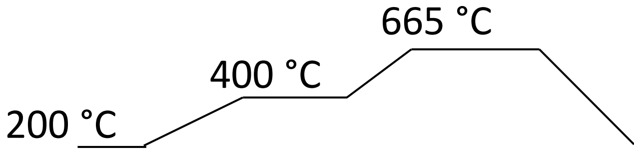


significant drop in adhesion

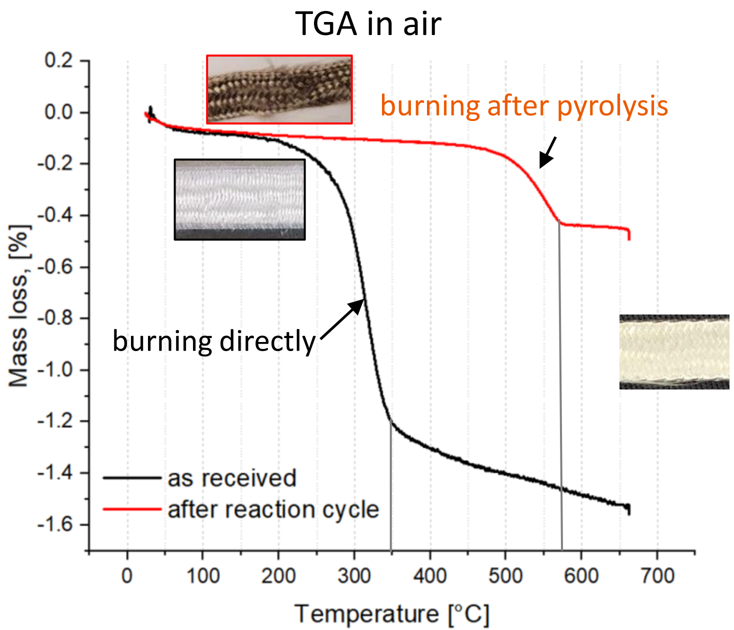
prior cleaning is not sufficient

- *burn organic residues during heat treatment*

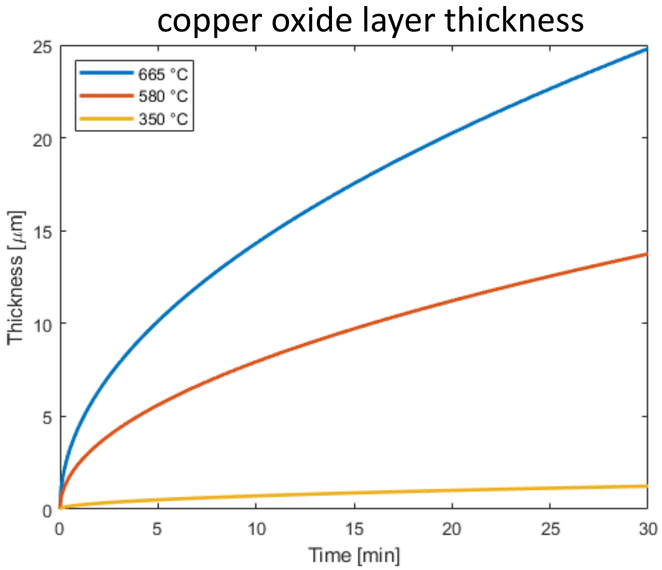
When to burn



combustion vs. oxidation of copper



TGA measurements of glass fiber braids in atmosphere at 1 °C/min.
As received: braid as received on the cable – no cleaning
After reaction cycle: braid as received after a complete reaction cycle



Build up of CuO layer at various temperatures in atmosphere, calculated with a parabolic rate law, Ea = 92.5 kJ/mol.

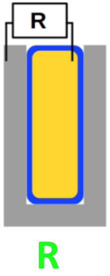
Equipment - oven and sensors

Inlet:

- Ar
- O2

Chamber:

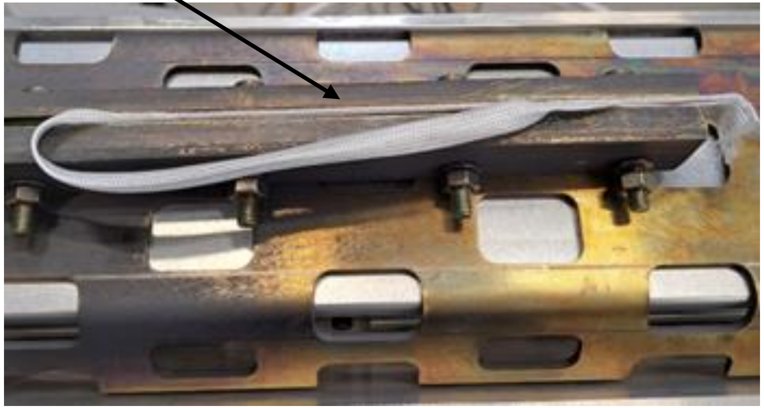
- single cable in a channel
  - ➢ measure resistivity between cable and channel



Exhaust:

- O2 probe, Zirox **XS22**
- CO2 sensor, **MH-Z19C**
- gas flow meter
- vacuum line

quartz tube furnace – reactive gases and vacuum



single cable in a channel

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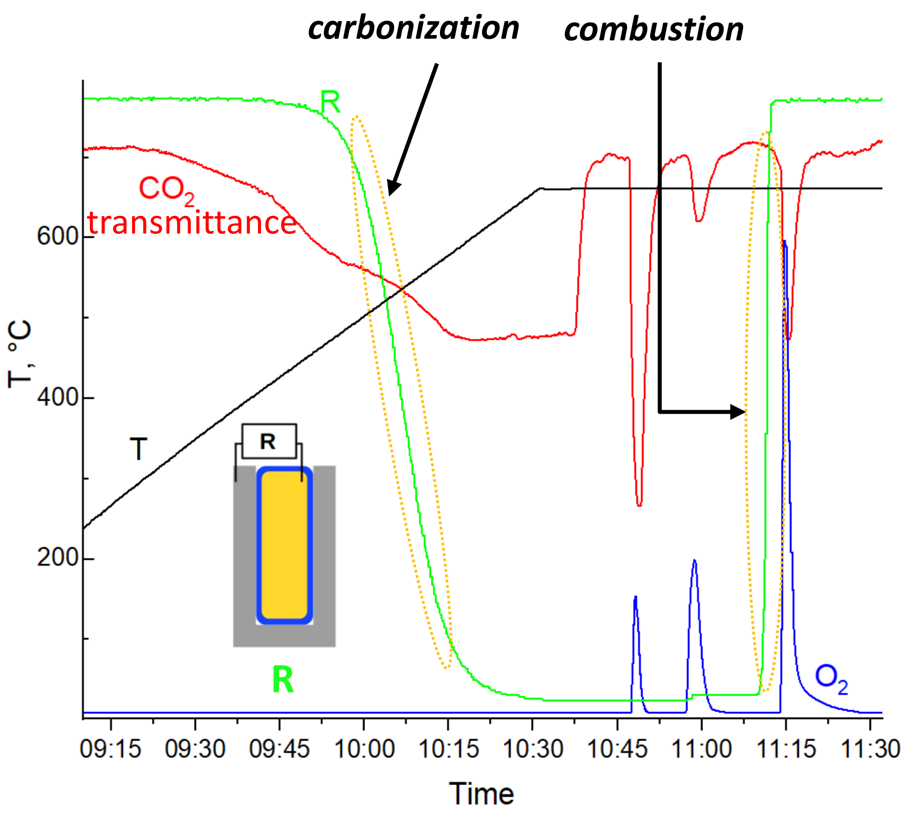
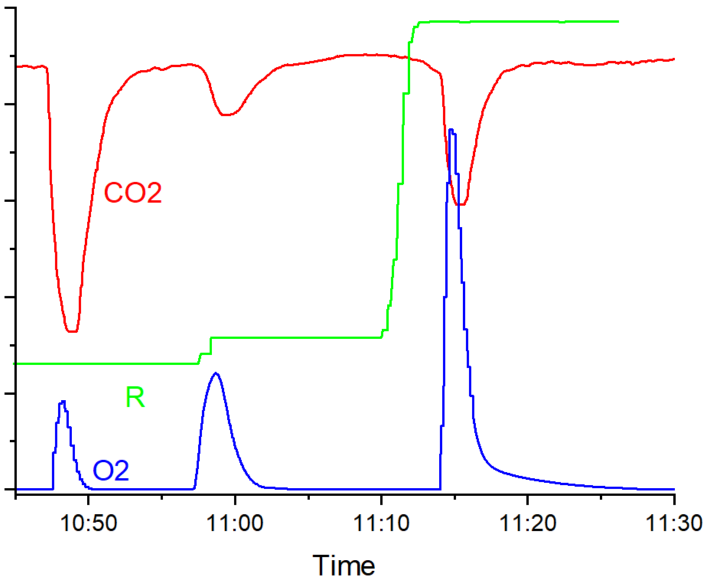
Realization – live monitoring



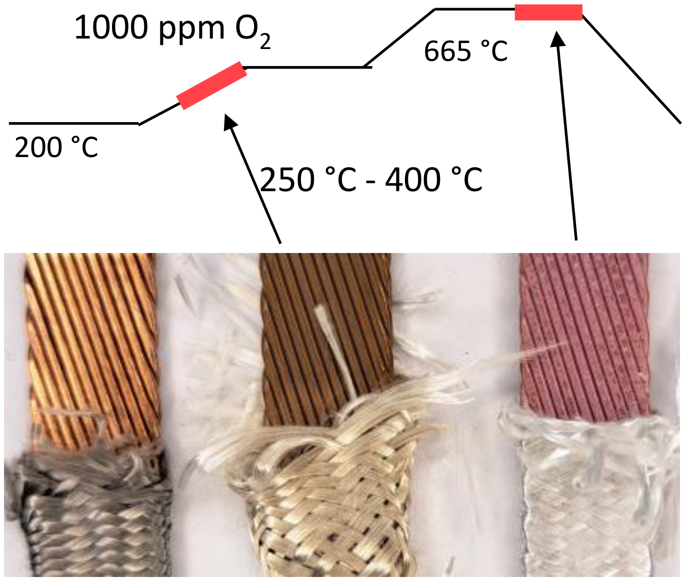


stop when R rises

and/or

when no CO_2 peak occurs after a O_2 pulse

Validation



reference in argon | 250-400 °C | 665°C in vacuum

	$RRR_{292/18}$	RRR reduction
Reference	532	-
250-400 °C	521	2%
665°C **in vacuum**	443	16%*

** in line with the reduction in cross-section due to the oxide layer*

Many more process parameters

- with or without vacuum
- dedicated isotherm for cleaning
- cleaning at multiple temperatures
- post reduction with H_2
- ...

Conclusion and Outlook

- Carbon residues can be removed with limited oxidation of the copper

- Optimization potential
  - improve CO_2 and R detection
  - explore other temperatures and conditions
  - reduction of copper oxide with H_2

- Conduct further analysis
  - XPS measurements of braid surface
  - SEM/EDX to check the oxide layer thickness
  - measure contact resistance between wires
  - check if adhesion and mechanical performance is better – composite

Glass ceramic coating

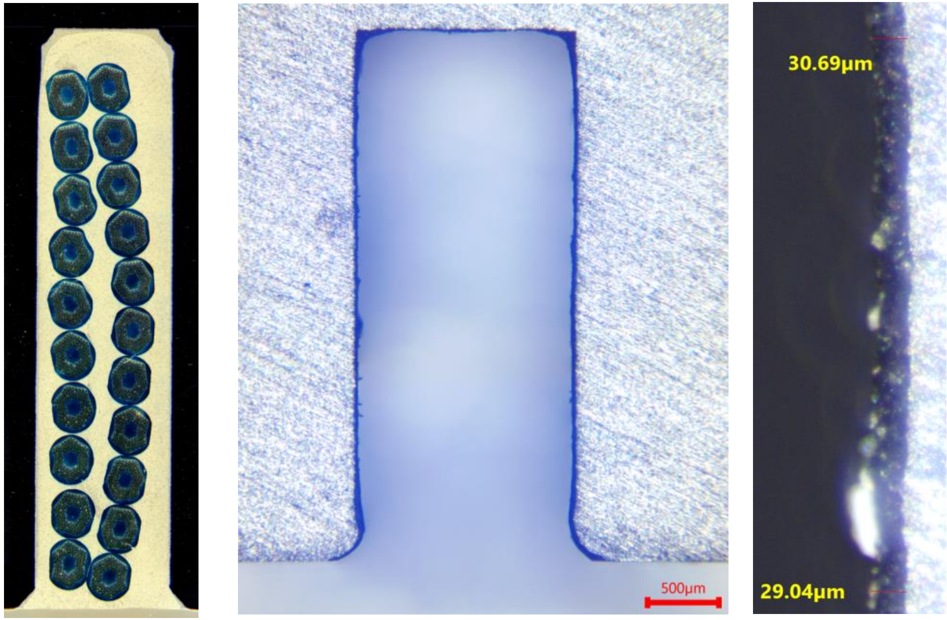

glass ceramic coating on box channel

Various commercial ceramic coatings were tested as an alternative to line of sight coating methods

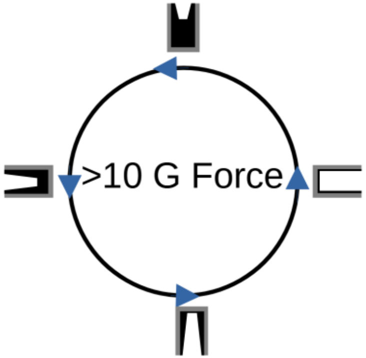
Aremco SGC4000-HT was chosen as a suitable coating

- temperature resistance to 760 °C, requires glazing at 720 °C
- 40 V/µm dielectric breakdown strength
- simple dilution with solvents (adjustable viscosity)
- suitable for spraying (fine particles), dip-coating, spin-coating etc.
- good adhesion to stainless steel
- survives the reaction cycle and cool-down

Spin coating channels



coating of high aspect ratio channels is possible



DBS measurements **40 V/µm**

layer thickness (µm)	breakdown voltage (V)	expected DBS value (V)
11	490	444
21	750	822
32	1160	1244

tested in a BOX

and BigBOX

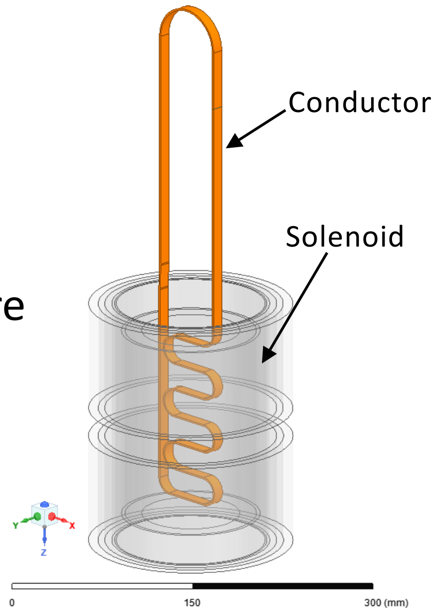
M. Daly et al., 2021
M. Daly et al., 2022
S. Otten et al., 2023

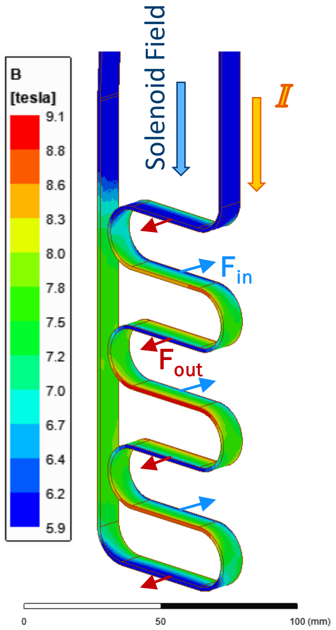
D. M. Araujo et al. 2023

Box program - concept

BOnding e**X**periment

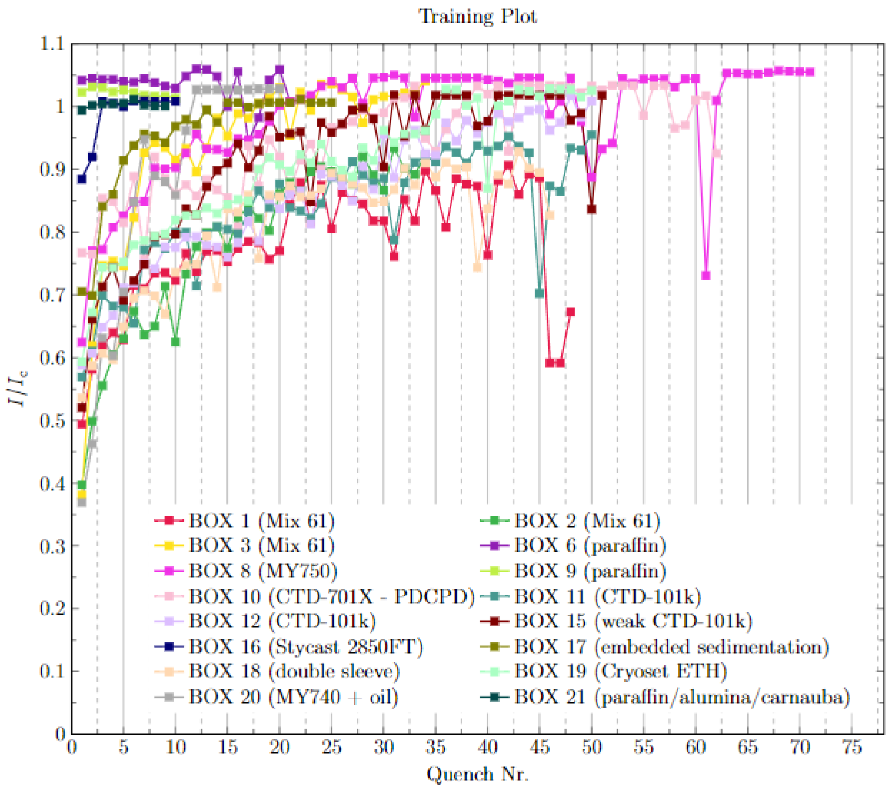
- cable in a channel
- bonding of the composite to structure
- assess conductor limits
- 9T background field
- training behavior can be adressed





M. Daly et al., 2021
M. Daly et al., 2022
S. Otten et al., 2023

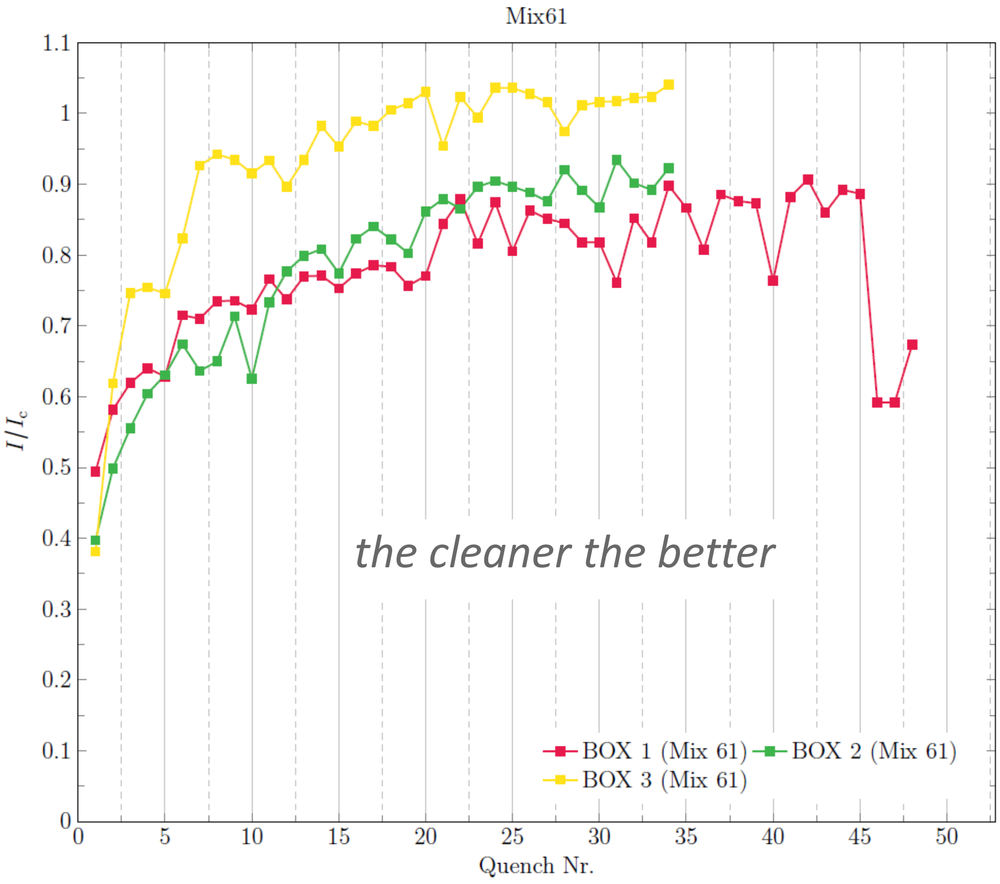
BOX program – fast turnaround



M. Daly et al., 2021
M. Daly et al., 2022
S. Otten et al., 2023

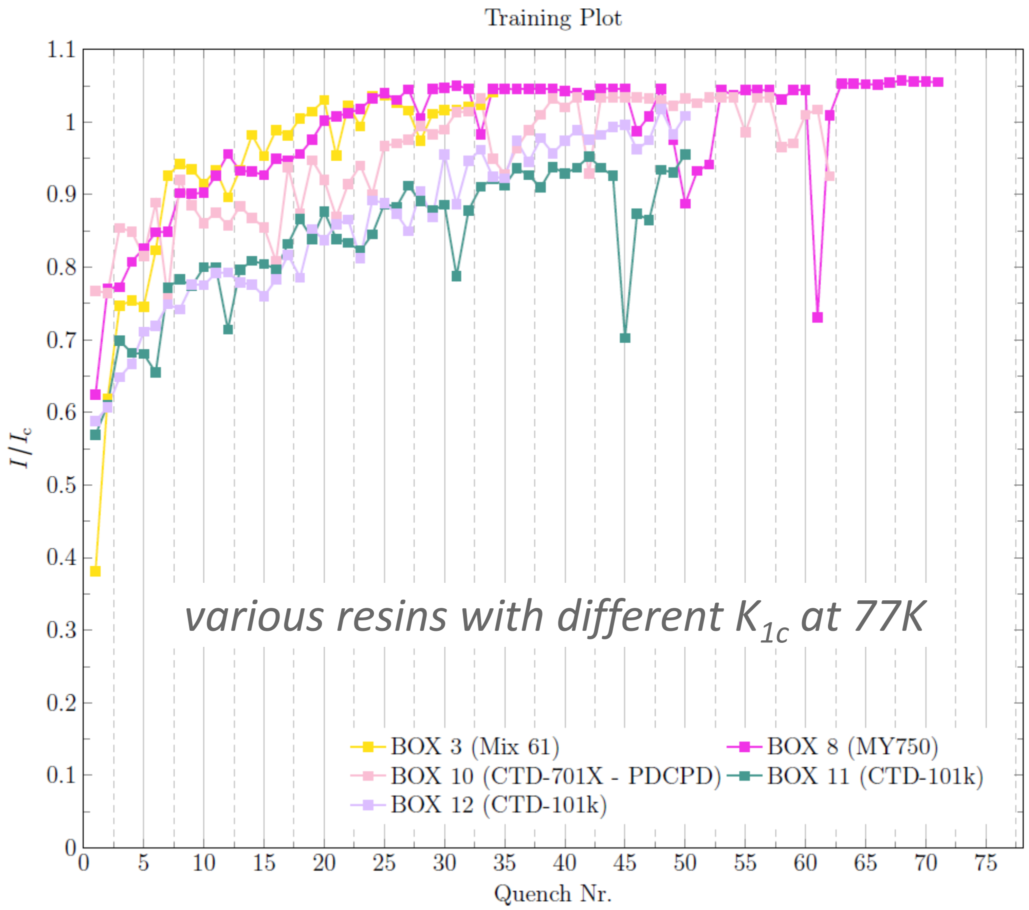
the chance to explore and compare a vast amount of impregnation systems

BOX program – procedure improvement



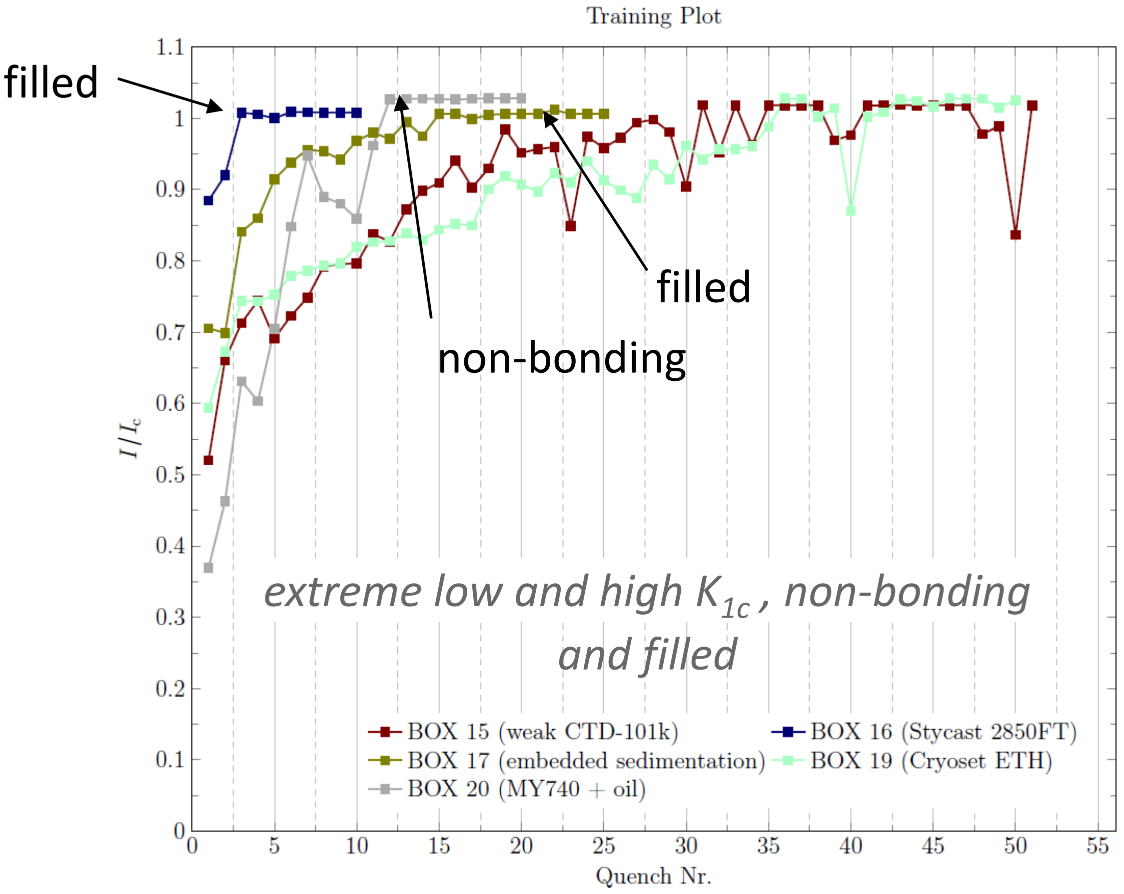
M. Daly et al., 2021
M. Daly et al., 2022
S. Otten et al., 2023

BOX program – pure resins



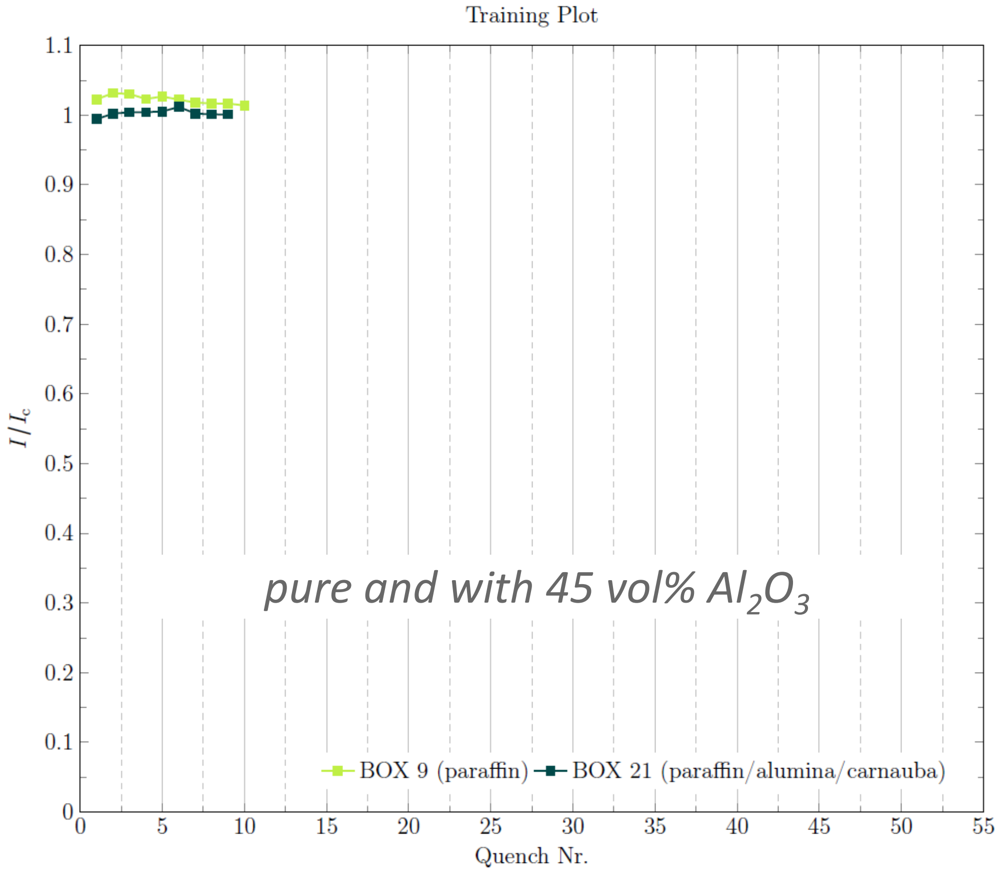
M. Daly et al., 2021
M. Daly et al., 2022
S. Otten et al., 2023

BOX program – experimental systems



M. Daly et al., 2021
M. Daly et al., 2022
S. Otten et al., 2023

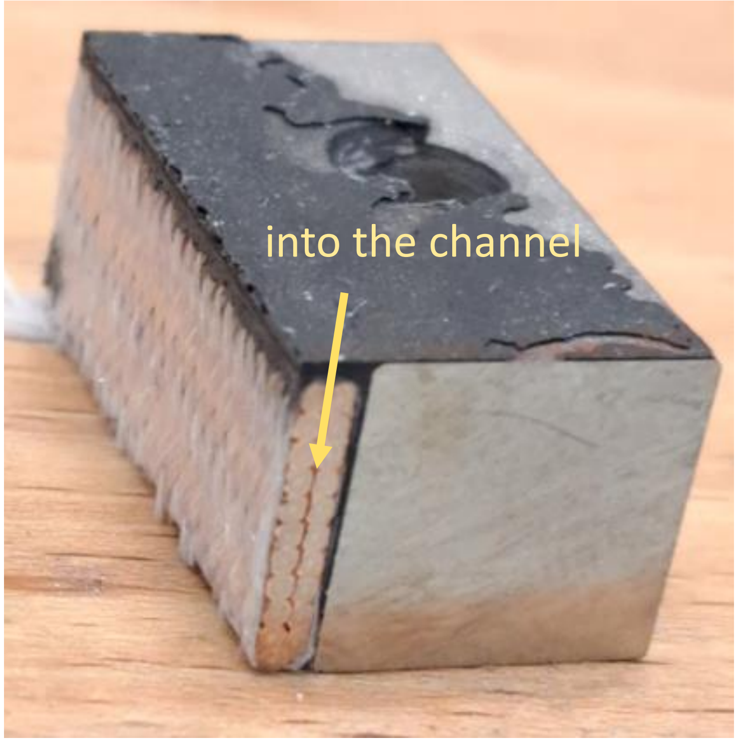
BOX program – wax and filled wax



M. Daly et al., 2021
M. Daly et al., 2022
S. Otten et al., 2023

Filled systems

flow direction



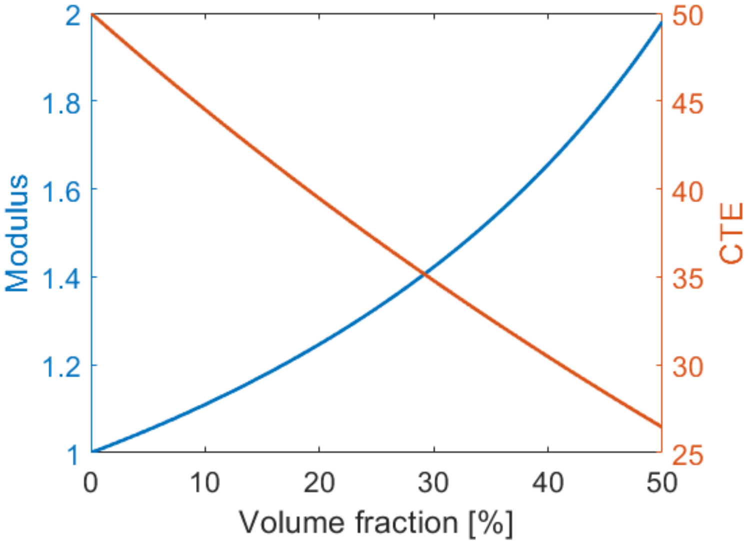
along the channel

Stycast 2850 FT + Cat 23 LV

Advantages and obstacles of filled systems

$E_m = 1$
$E_f = 100$

$CTE_m = 50$
$CTE_f = 5$

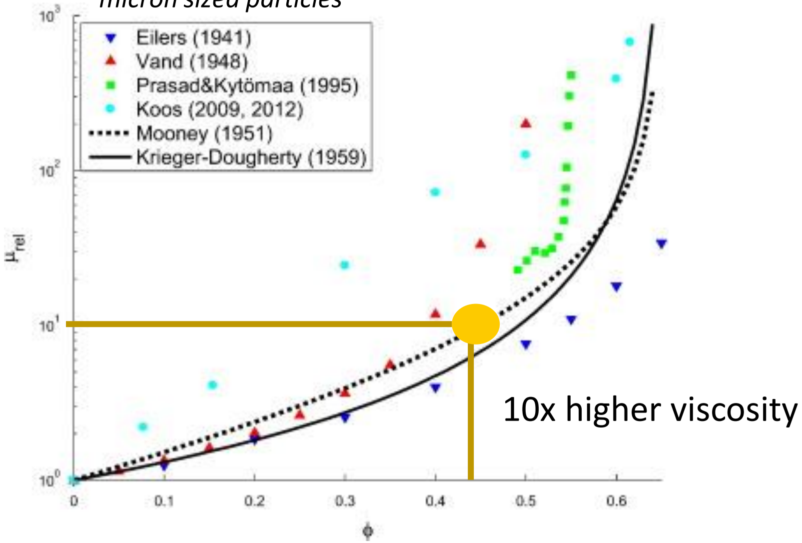


$$E_c = \left(\frac{f}{E_f} + \frac{1-f}{E_m} \right)^{-1}$$

particle filled system series model

lower boundaries

micron sized particles



B.J.Konijn et al. 2014

$e.g.\ if\ \eta_s = 80\ Pas$
$with\ 45\ vol\%\ \ \eta = 800\ Pas$

$$\eta_{rel} = \frac{\eta}{\eta_s}$$

challenging for epoxies

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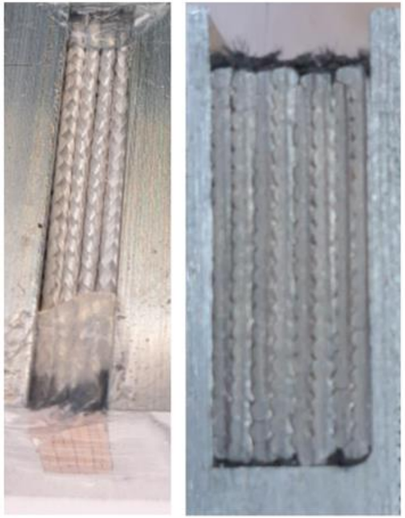
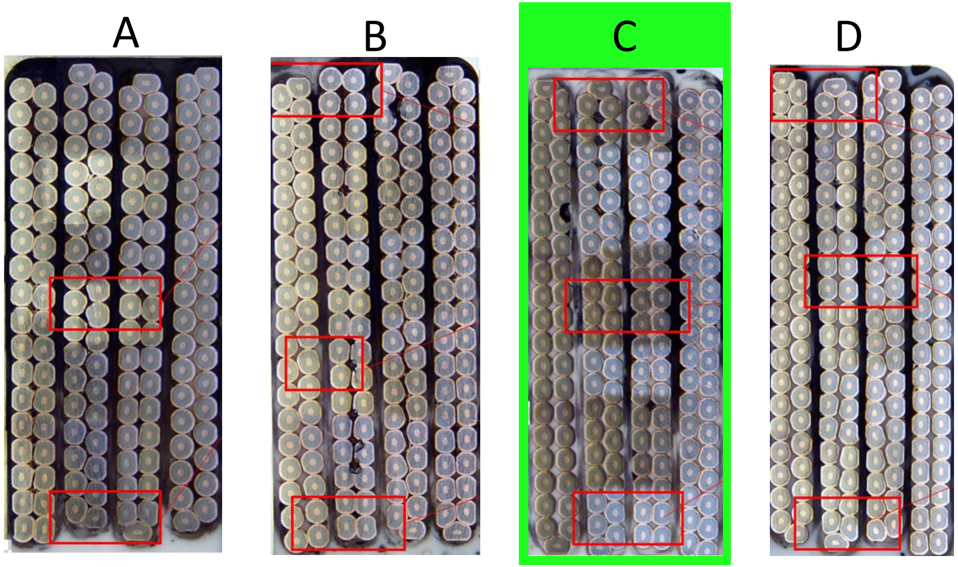
Pre trials - filled systems – Epoxy (MY750)

Label	Shape	Size, D50 µm	Specific surface m^2/g
A-2µm	Spheres	2.18	1.27
B-1µm	Spheres	1.08	1.69
C-1µm	Shards	1.05	2.4
D-0.1µm	Spheres	0.1	7.5

Submicron particles can pass through the glass fiber insulation but **viscosity was too high**



BOX cable without mica



40 vol% filler content

Filled wax – up to 45 vol% of particles

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filled wax:

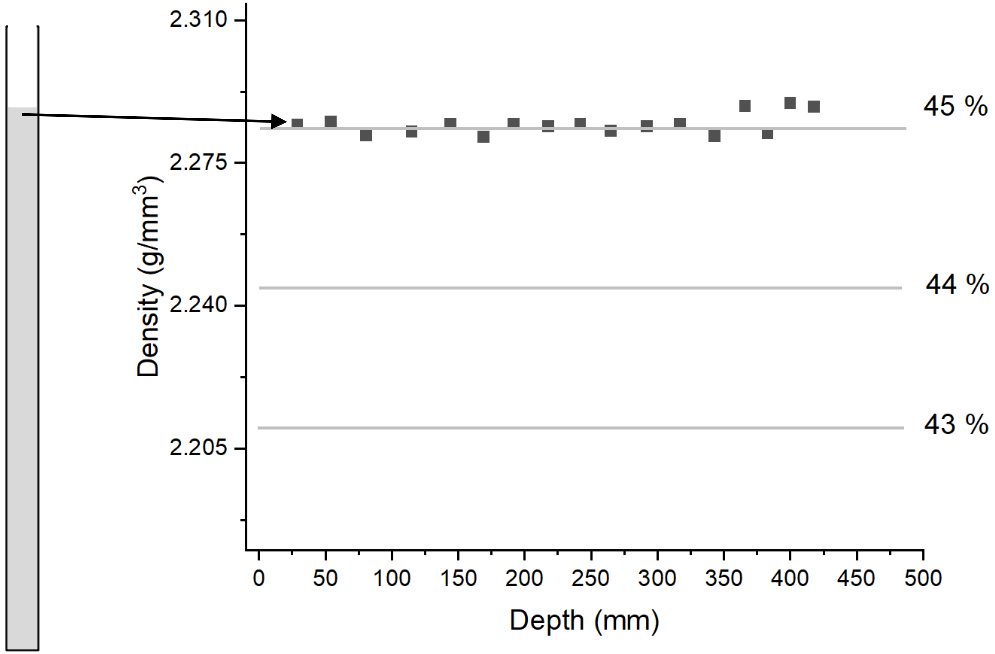
wax with well dispersed submicron alumina particles which go through the glass fiber insulation.

Preliminary results - processing

Sedimentation

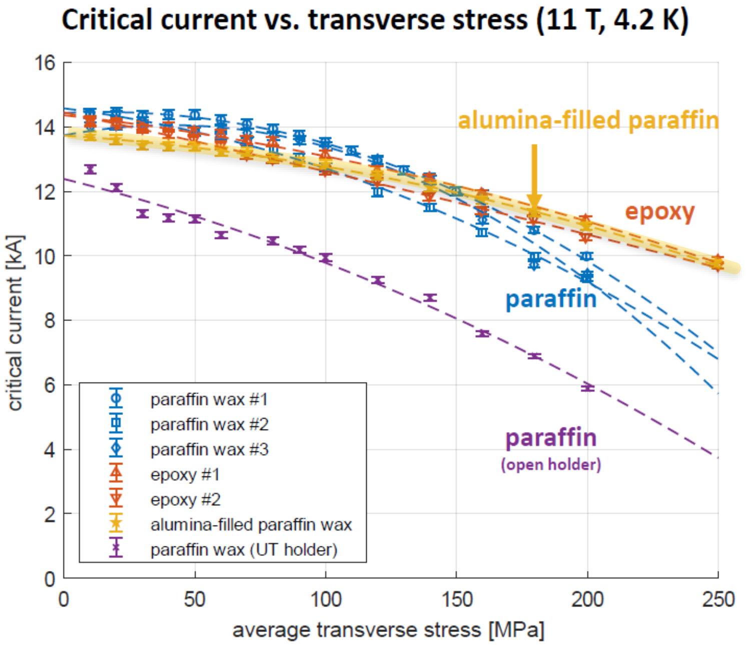
500 mm high column
filled with wax at
90 °C for 3 hours

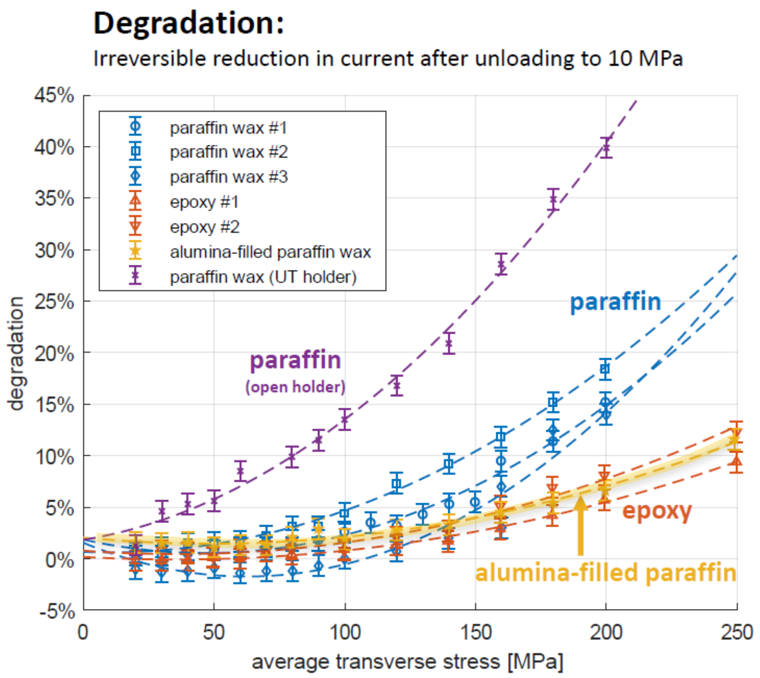
cool down and cut sections
for density measurements



no visible sedimentation over 3 hours, longer times are being tested

Critical current vs. transverse stress





Michael Daly, Bernard Auchmann, André Brem, Christoph Hug, Serguei Sidorov, Simon Otten, Marc Dhallé, Zichuan Guo, Anna Kario and Herman ten Kate

Conclusion

- submicron particles work with the Box cable insulation
  - size distribution is important
  - shape should be spherical/round

Standard PB products (Typical values)		PB4MAR
Crystalline phase (%)		
Specific Surface Area (m^2/g) *BET*		4
PSD (µm) *Laser diffraction* *Horiba LA950*	d_{10}	0.64
	d_{50}	0.77
	d_{90}	0.91

Baikowski

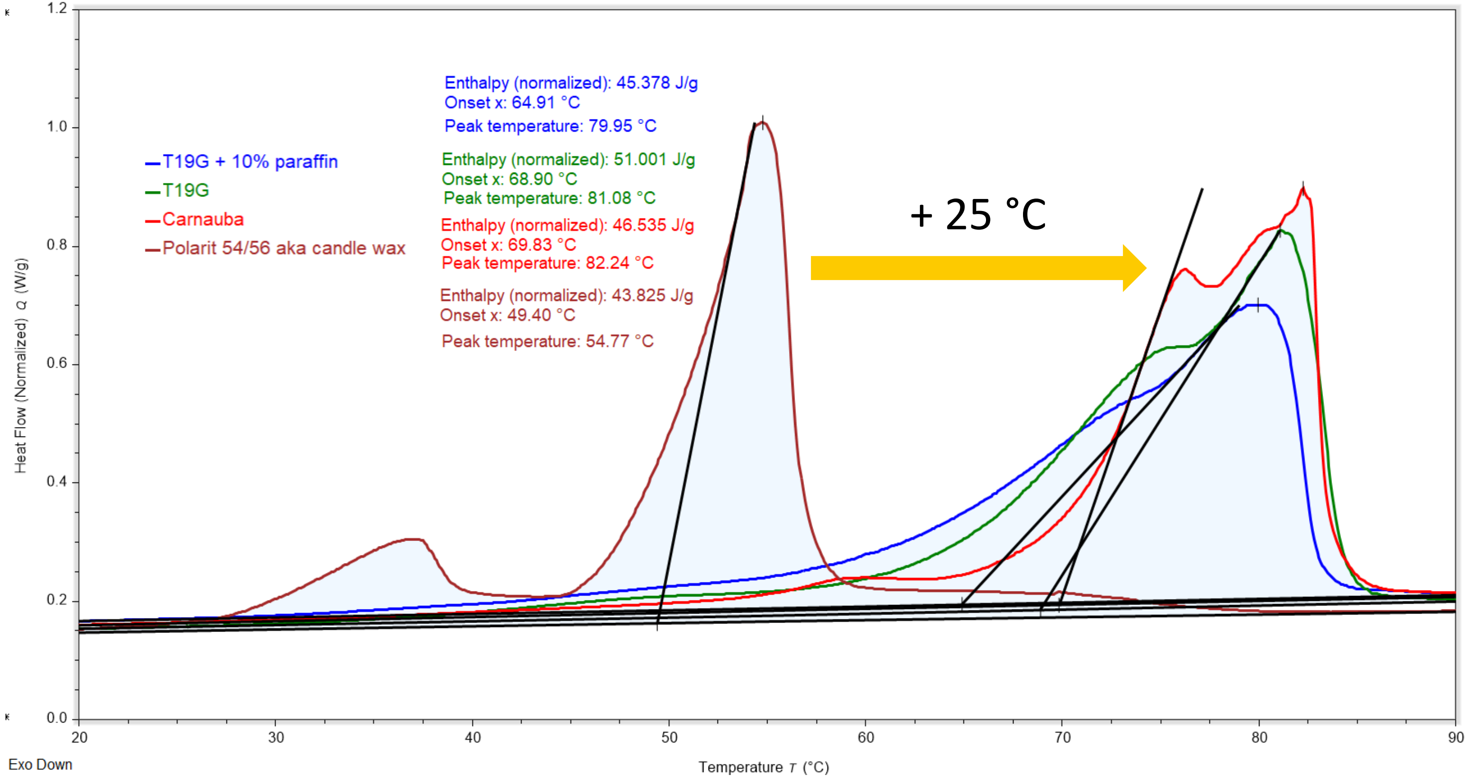
- specific surface area has to be kept low (< $4m^2/g$)
  - keeps the relative viscosity reasonable

- for each particle and matrix system the dispersion process may change
  - mixing techniques
  - mixing sequence
  - dispersing aids
  - η_s has to be below 30 mPas

TABLE II
FILLED WAX - 100 ML AT RT

FRP wax	45.5 g
Carnauba	5.0 g
alumina	180 g

Outlook - higher melting point wax



filled with 45 vol% alumina particles

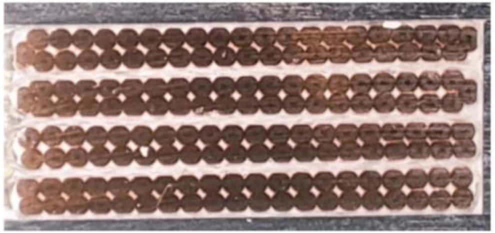
Summary

burning organic material during RHT followed by RRR measurements

- clean magnets are feasible

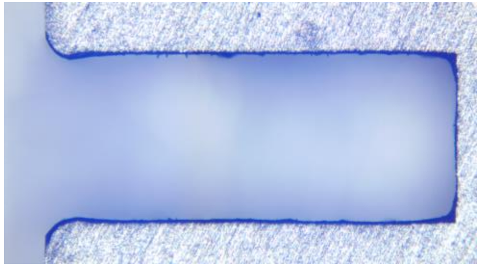
a glass ceramic coating, which survives a dT of 900 °C, as an insulation for steel parts.

- primary or secondary insulation



the box program

- an excellent tool to investigate impregnation systems

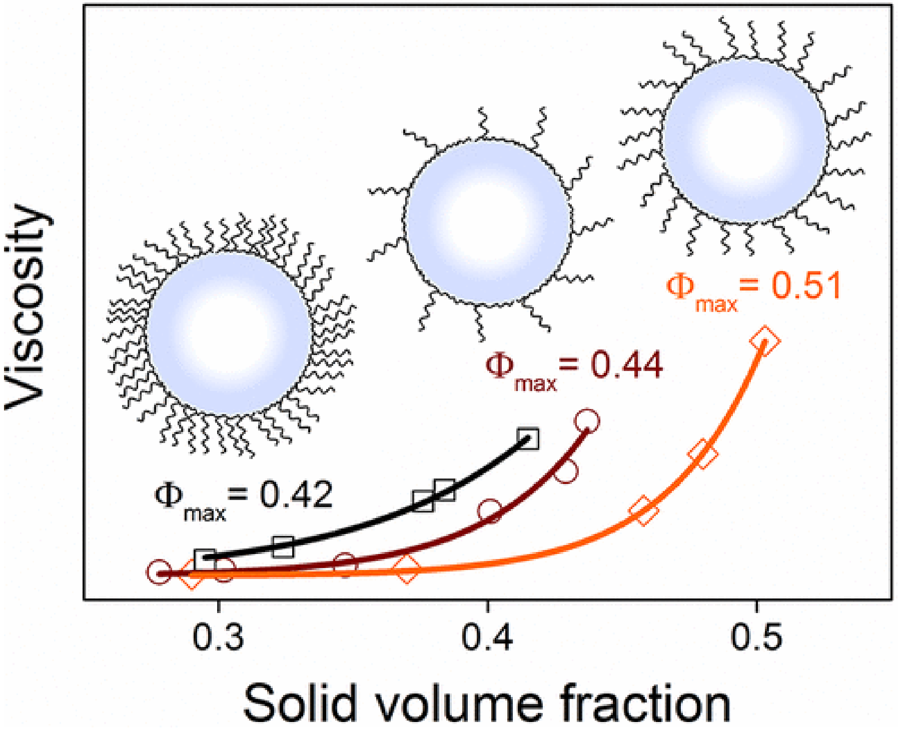


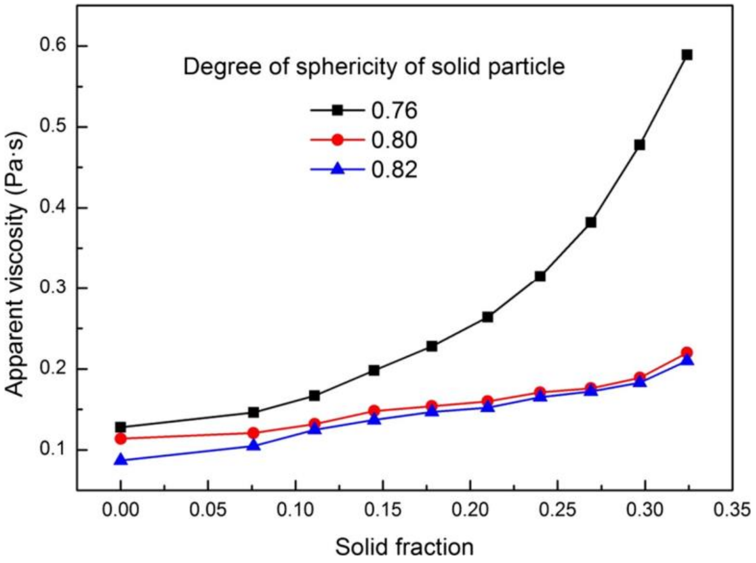
impregnation of filled hot wax of a 4-stack shows a promising route to better properties at cold

- increased modulus and lower CTE

Questions

Particle shape and dispersant concentration





https://doi.org/10.1016/j.powtec.2017.12.019

Viscosity and particle size

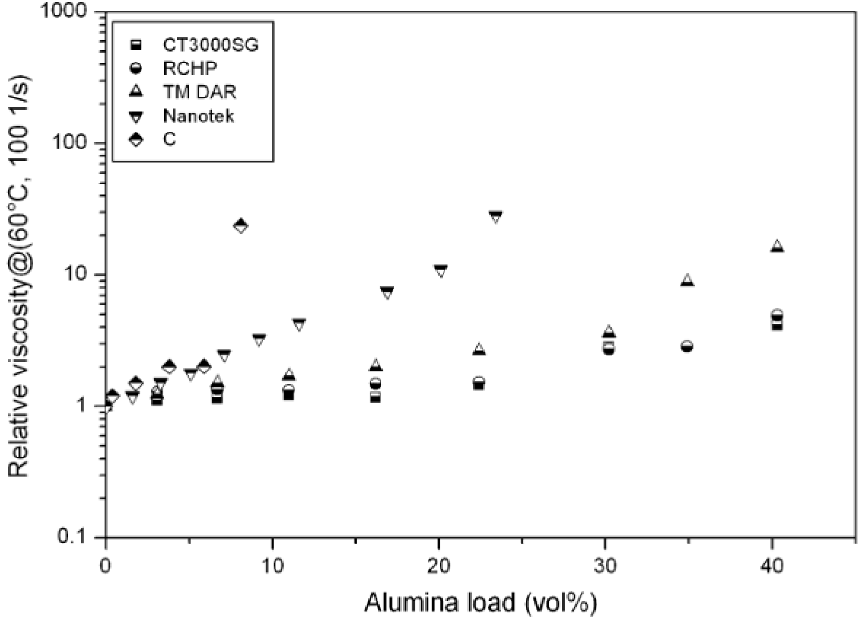


Fig. 5. Relative viscosity change with alumina load at 60 °C and a shear rate of 100 s^{-1}.

Table 1
Specifications of the used micro- and nanosized alumina

Type	Vendor	Specific surface area (m^2/g)	Measured particle sizes (μm)		
			d_{10}	d_{50}	d_{90}
CT3000SG	Almatis	6–8[a]	0.071	0.266	1.002
RCHP	Baikowski	7–9[a]	0.242 (d_{20})	0.335	0.575
TM DAR	Taimei	12.8	0.104	0.165	0.297
Nanotek	Nanophase	34	0.125	0.155	0.192
C	Degussa	107	0.125	0.155	0.205

[a] Vendor's data.

https://doi.org/10.1016/j.ceramint.2007.08.007

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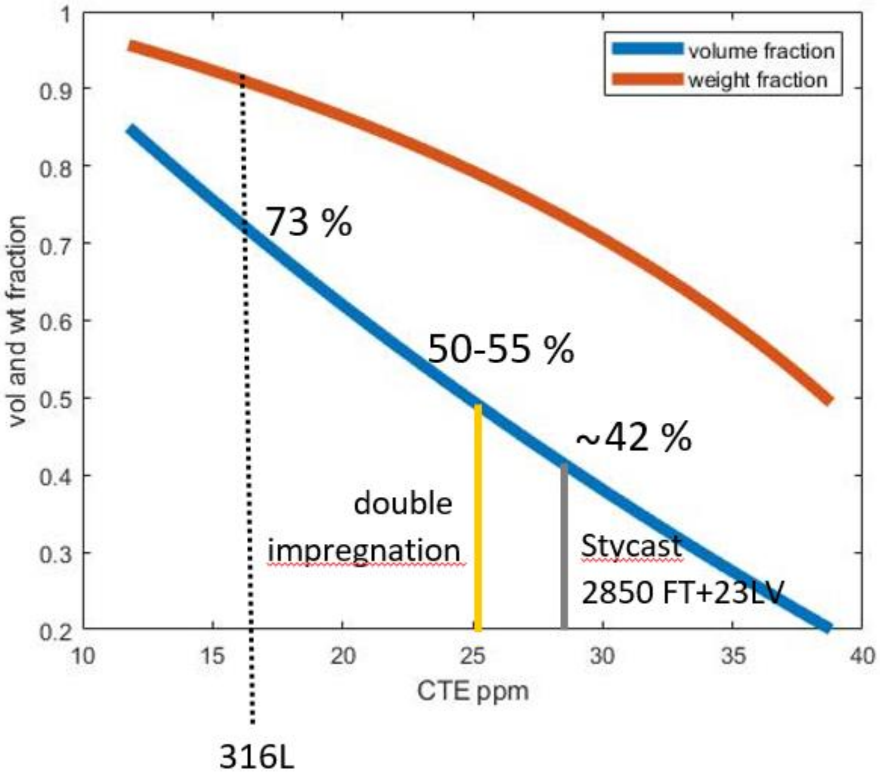
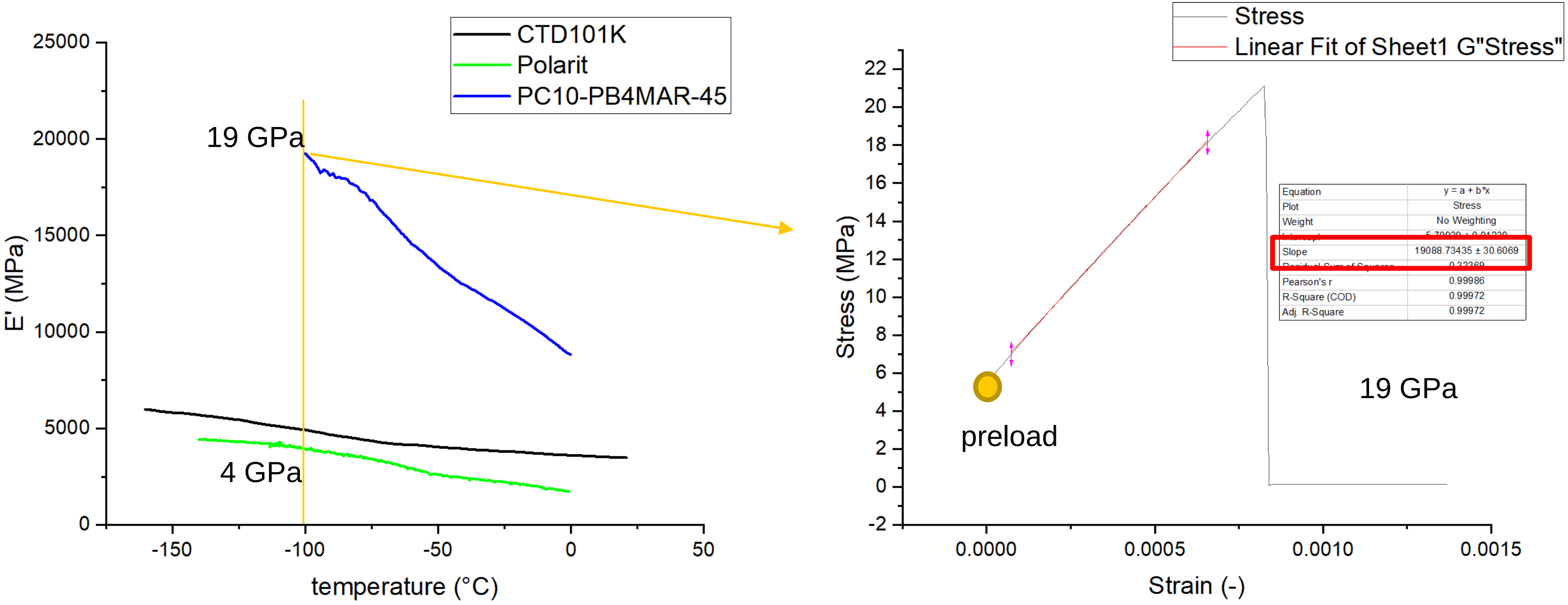
double impregnation with ceramic slurry



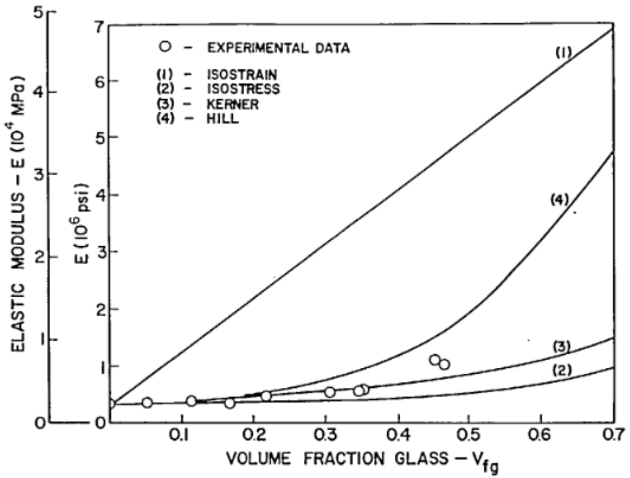
Preliminary results - DMA and 3 PB at -100 °C



at -100 °C, 4 GPa for pure Wax, 19 GPa for the filled wax – 4.75 times higher

Literature

- https://doi.org/10.1016/j.compositesb.2008.01.002
- https://doi.org/10.1177/0021998375009002
- https://doi.org/10.1016/S0263-8223(98)00033-6
- https://doi.org/10.1023/A:1008975323486



Kerner model more apropriate

Figure 2. Experimental and predicted elastic modulus variation with glass content.

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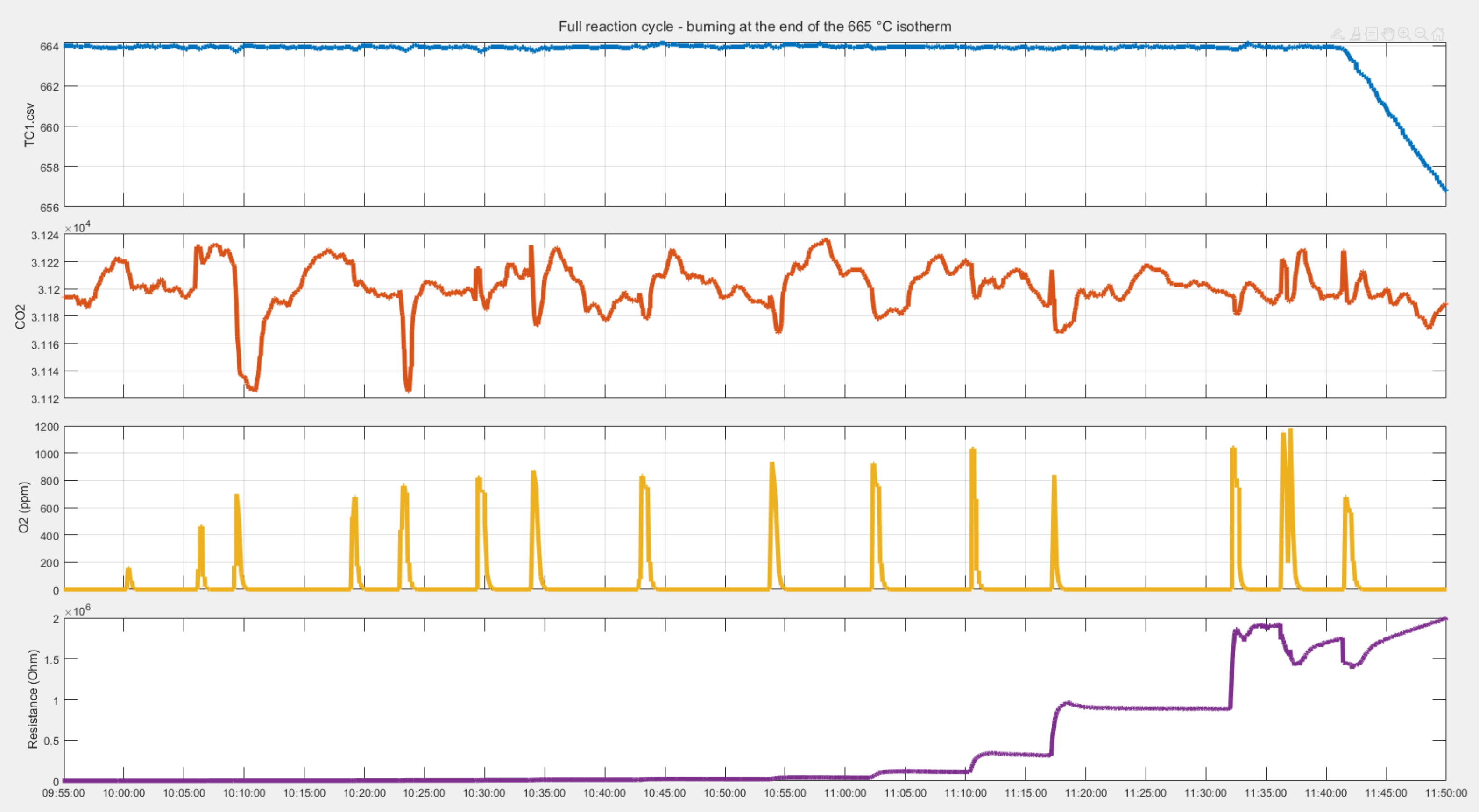

XPS Sizing

3.3 Carbon 1s

Regarding carbon, fitting for three peaks also gives the most sensible results (see figure 9)). When looking at the reference peak 2 is most likely an overlap of C-C and C-O bonds [10]. Peak 3 seems to be the C-Si bonds from the silane [10]. Peak 1 looks to be the carboxyl groups from fatty acid residues in lubricants that are used when braiding the GFs.

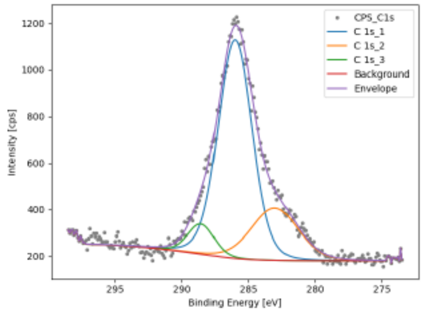

Figure 9: Reference Carbon 1s peak in the spectrum. Averaged over 5 cycles.

For sample 1, the carbon peak has the most significant reduction in intensity. First of all, peak 1 stemming from the carboxyl groups of fatty acids seems to have completely disappeared. Furthermore, peak 2 representing C-C and C-O bonds is significantly smaller. Only peak 3, the silicon-bound carbon, is still close to the same, if not more intense (see figure 10).

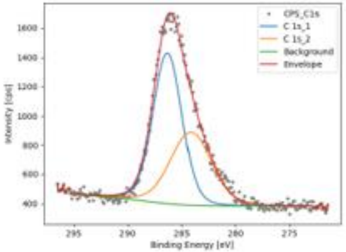

Figure 10: Sample 1 Carbon 1s peak in the spectrum. Averaged over 5 cycles.

The same can be said for sample 2. However there is a strange 3rd peak that only appeared here and we could not assign with sufficient certainty. It is most likely some form of contamination.

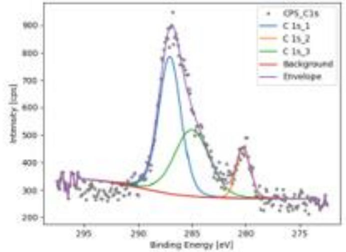

Figure 11: Sample 2 Carbon 1s peak in the spectrum. Averaged over 5 cycles.

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cable

- Rutherford cable made of 21 strands of Bruker OST RRP 108/127 Nb_3Sn 0.85 mm in diameter following the LARP QXF specification 150505 and insulated with fibreglass braid. The 21-strand CCT cable was manufactured by the Lawrence Berkeley National Laboratory (LBNL) and delivered to the Paul Scherrer Institute (PSI) as part of a collaboration between PSI and LBNL.

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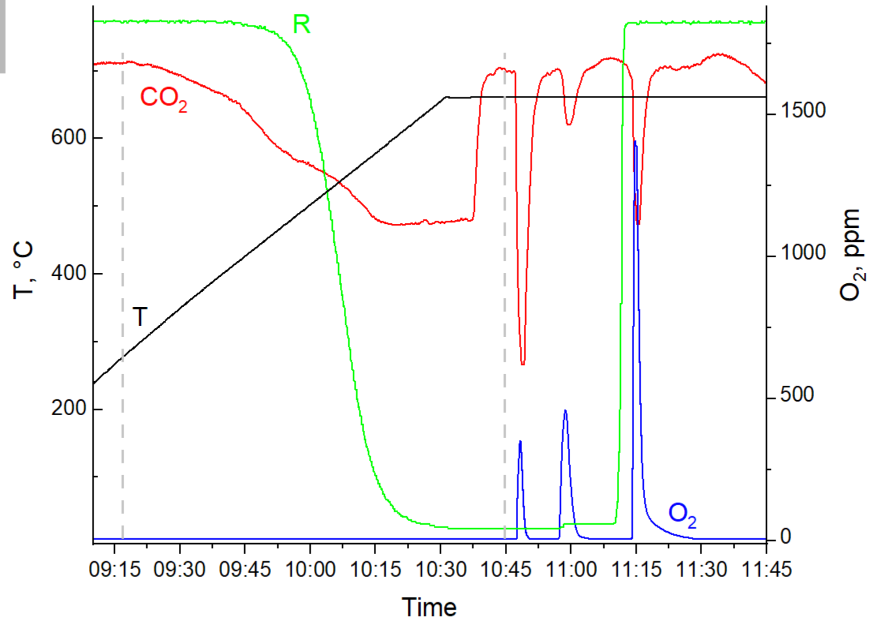
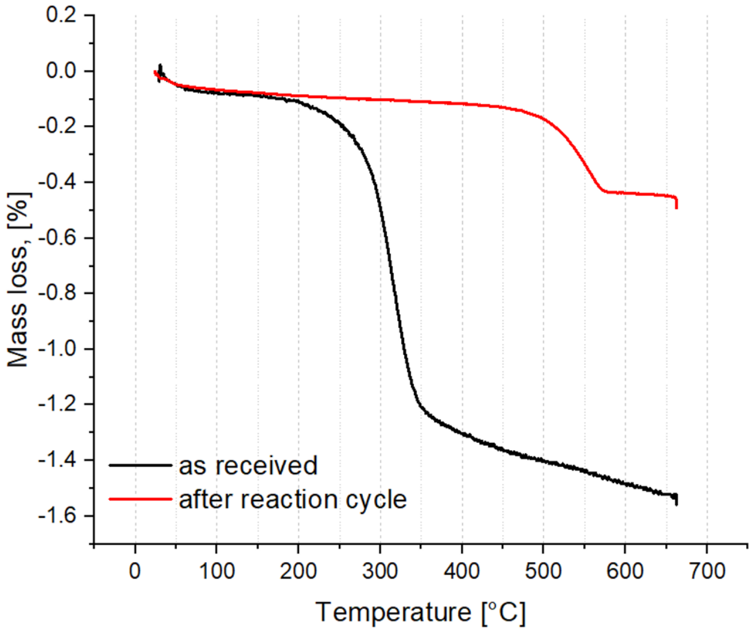
Oven data and TGA





Glass fiber braid Information

is shown in Figure 1.10. The pictured cable is a MQXF cable without mica. The applied yarn is a Type 933 S2 glass by AGY with numerous filaments of 9 μm diameter forming a 145 μm thick glass insulation. The Type 933 S2 glass comes with a thermally stable inorganic sizing that eases the braiding process and remains on the fibreglass after braiding. The electrical magnet insulation is completed with the vacuum pressure

S-2 Glass® Yarns

Filament Diameter US	Filament Diameter Micron	Sliver	Const	Twist	Nominal Yield (yds/lb)	Nominal Yield (TEX)	Denier	Average Tensile Strength (lbs)	Average Tensile Strength (N)	Sizing
D	5	450	1/0	1.0Z	45,000	11	99	2.3	10.2	636

S-2 Glass® Yarns	383	SG150	0.70	Resin		Directly compatible with Resin Systems	Industrial	Epoxy / Polyester / Vinyl Ester
	493	SG150 and SG75	0.45	Resin		Directly compatible with Resin Systems	Industrial	Epoxy / Polyester
	550	SG150 and SG75	0.50	Resin		Directly compatible with Resin Systems	Aerospace Industrial	High Temperature Thermoplastics
	636	SG150 and SG75	1.20	Starch / Lube		General Purpose	Weaving Industrial	General purpose, good processability
	762	SE225	0.42	Resin		Directly compatible with Resin Systems	Industrial	Rubber Compatible
	933	SG75	0.23	Resin		Directly compatible with Resin Systems	Aerospace	"Toughened Epoxy / Thermoplastics Poor Broken Filament Resistance"

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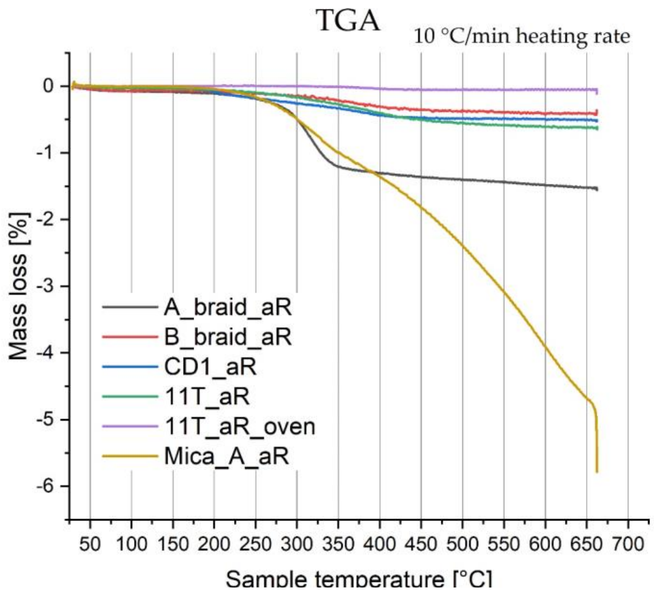

Yarn Type (metric)	Construction	Nominal Twist TPM	Nominal Twist TPI	Sizing	Approximate Yarn Diameter mm	Approximate Yarn Diameter inch	Nominal Bareglass Yield TEX	Nominal Bareglass Yield Yard/Pound	Denier	Nominal Filament Diameter
SCG75 (SC9 66)	1/0	Z40	1.0Z	636	0.192	0.0076	66	7,500	594	"G" or 9 microns
SCG150 (SC9 33)	1/0	Z40	1.0Z	636	0.136	0.0054	33	15,000	297	"G" or 9 microns
SCD450 (SC5 11)	1/0	Z40	1.0Z	636	0.076	0.0030	11	45,000	99	"D" or 5 microns

S-2 Glass® Yarns	383	SG150	0.70	Resin		Directly compatible with Resin Systems	Industrial	Epoxy / Polyester / Vinyl Ester
	493	SG150 and SG75	0.45	Resin		Directly compatible with Resin Systems	Industrial	Epoxy / Polyester
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THERMAL CONTRACTION

Experimental Data and Fits for the Thermal Contraction of Future Magnet Materials at Cryogenic Temperatures

The measurement data provided in this booklet was measured at

CERN

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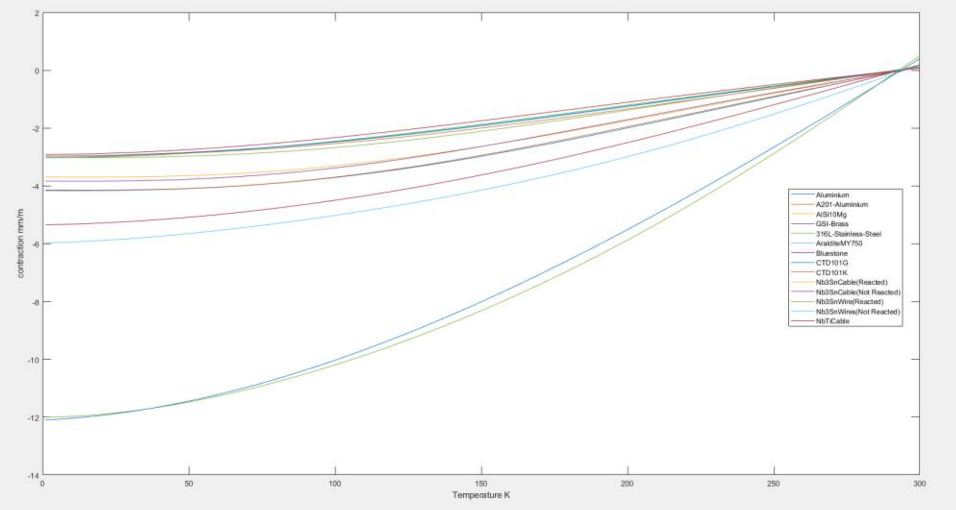
EuCARD²

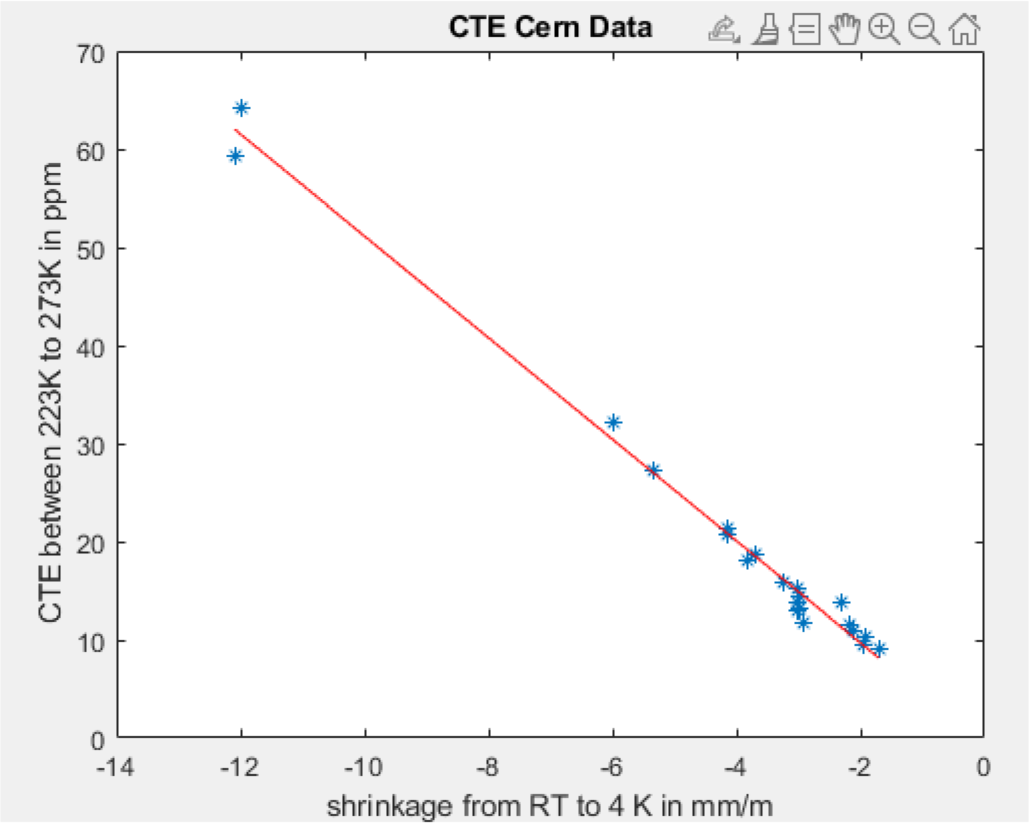
In the making of this booklet the following people were involved

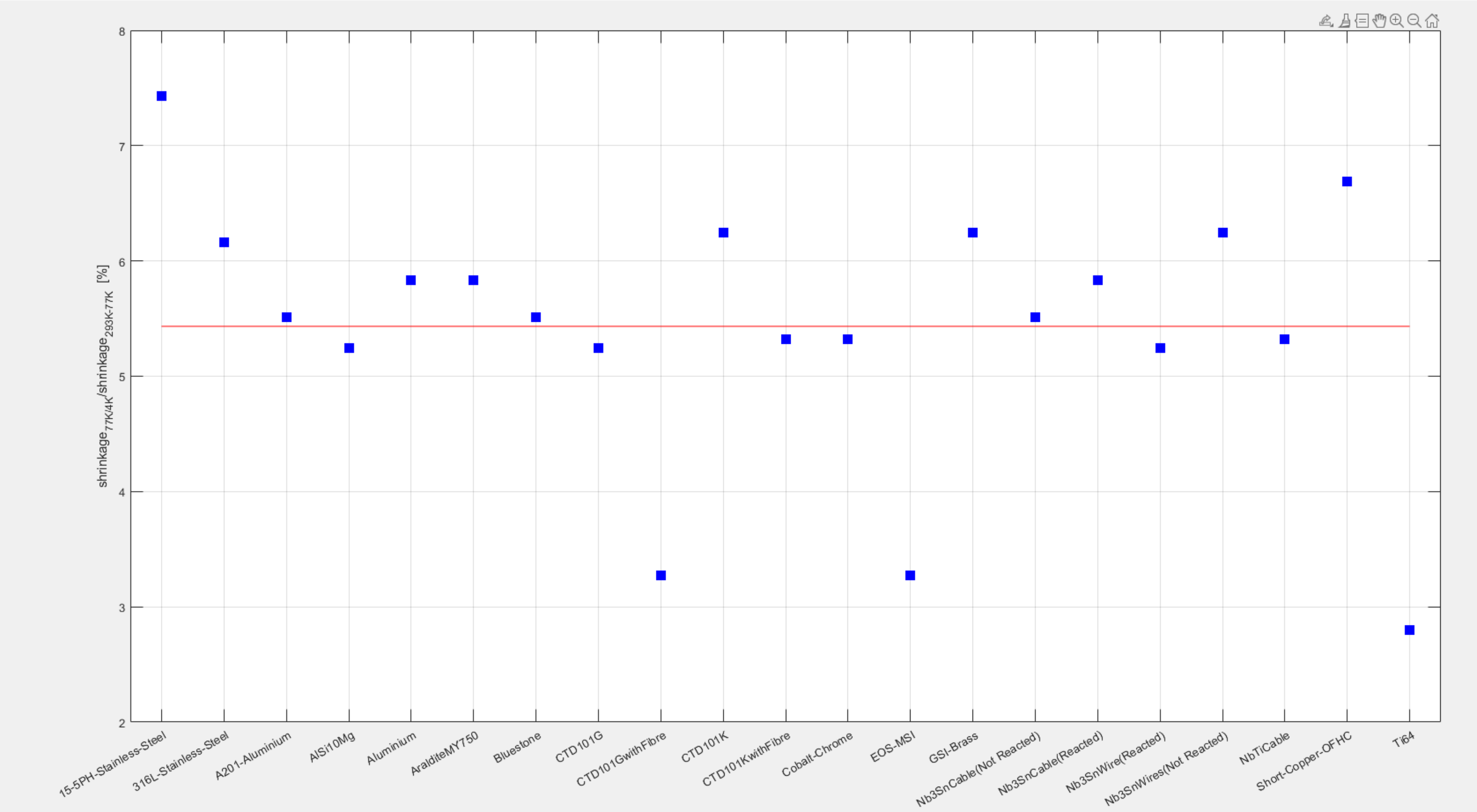
Glyn Kirby, Vladimir Ivanovich Datskov, Sebastien Clement, Antonella Chiuchiolo, Paolo Fessia, Remy Gauthier, Grace Lynch, Jaakko Murtomaki, Jeroen Van Nugteren, Francois-Olivier Pincot, Juan Carlos Perez, Lucio Rossi, Gijs De Rijk, Sandra Tavares, Valentina Venturi

last compilation performed on: October 3, 2016



RT to -50 °C


CTE Cern Data
CTE between 223K to 273K in ppm
shrinkage from RT to 4 K in mm/m
70
60
50
40
30
20
10
0
-14
-12
-10
-8
-6
-4
-2
0



shrinkage$_{77K/4K}$/shrinkage$_{293K-77K}$ [%]
8
7
6
5
4
3
2
15-5PH-Stainless-Steel
316L-Stainless-Steel
A201-Aluminium
AlSi10Mg
Aluminium
AralditeMY750
Bluestone
CTD101G
CTD101GwithFibre
CTD101K
CTD101KwithFibre
Cobalt-Chrome
EOS-MSI
GSI-Brass
Nb3SnCable(Not Reacted)
Nb3SnCable(Reacted)
Nb3SnWire(Reacted)
Nb3SnWires(Not Reacted)
NbTiCable
Short-Copper-OFHC
Ti64

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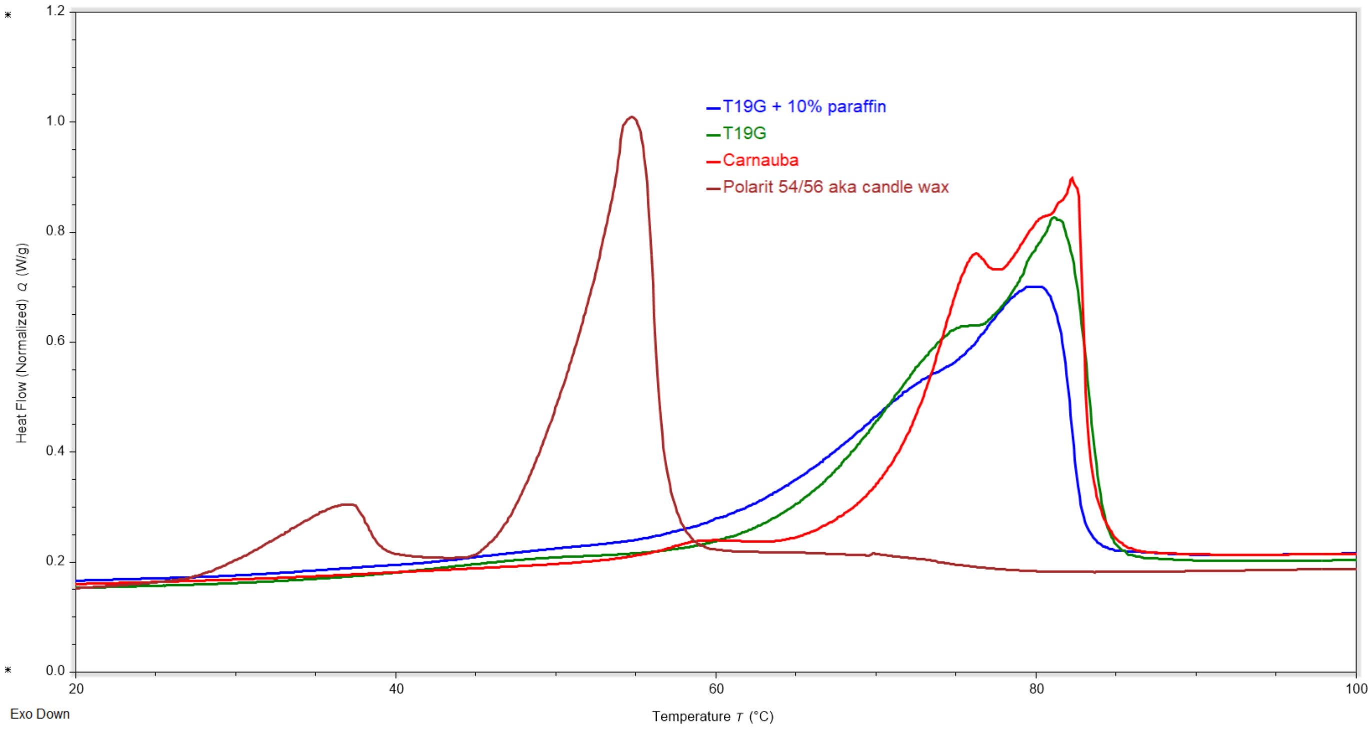

T19G + 10% paraffin
T19G
Carnauba
Polarit 54/56 aka candle wax
Heat Flow (Normalized) Q (W/g)
1.2
1.0
0.8
0.6
0.4
0.2
0.0
20
40
60
80
100
Temperature T (°C)
Exo Down

Filled Wax – lower boundaries

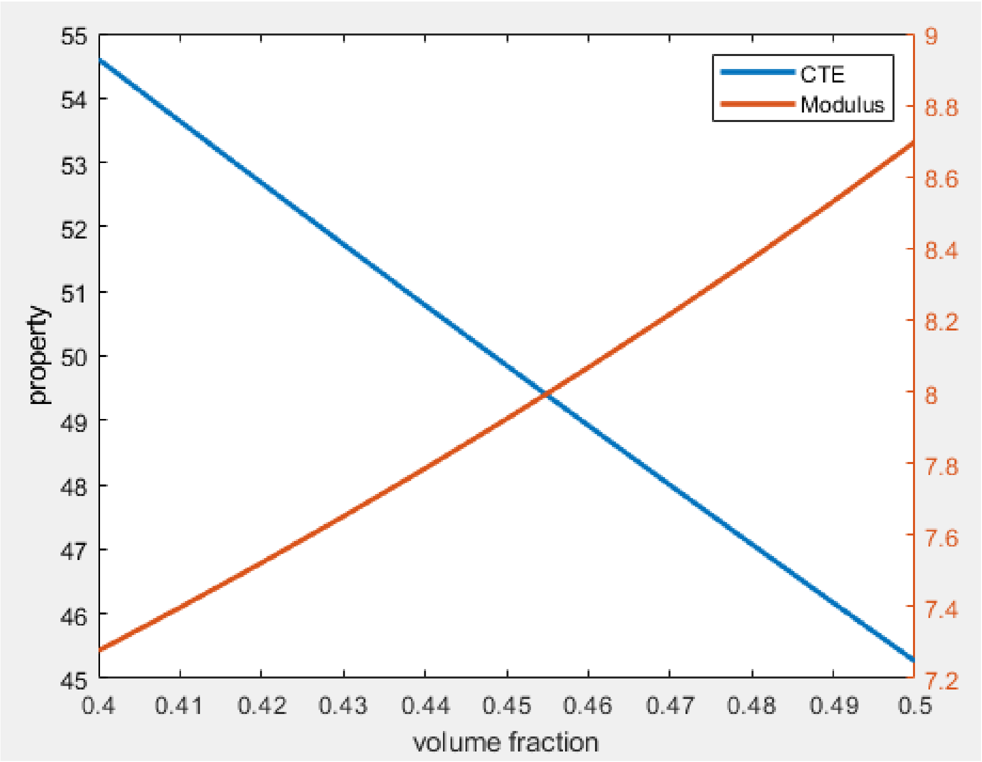
$E_m = 4.4$ $\quad CTE_m = 100$

$E_f = 375$ $\quad CTE_f = 7$

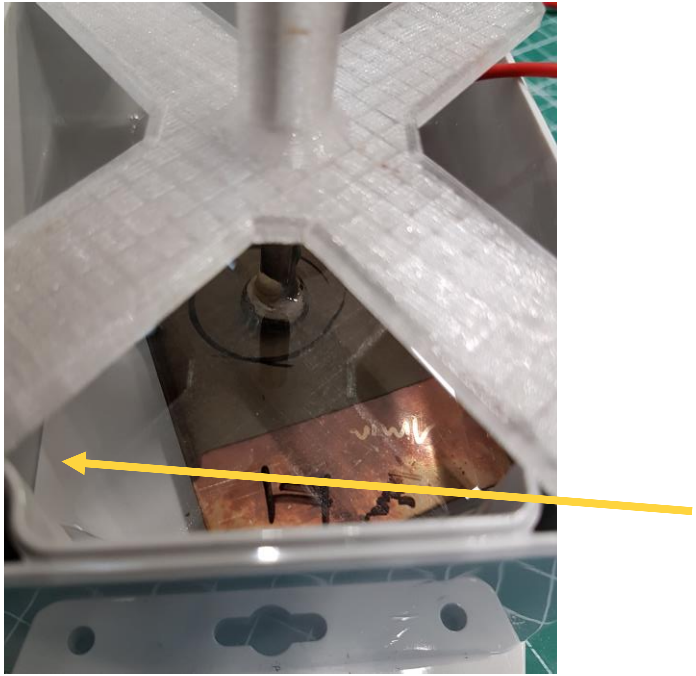
$$E_c = \left(\frac{f}{E_f} + \frac{1-f}{E_m}\right)^{-1}$$

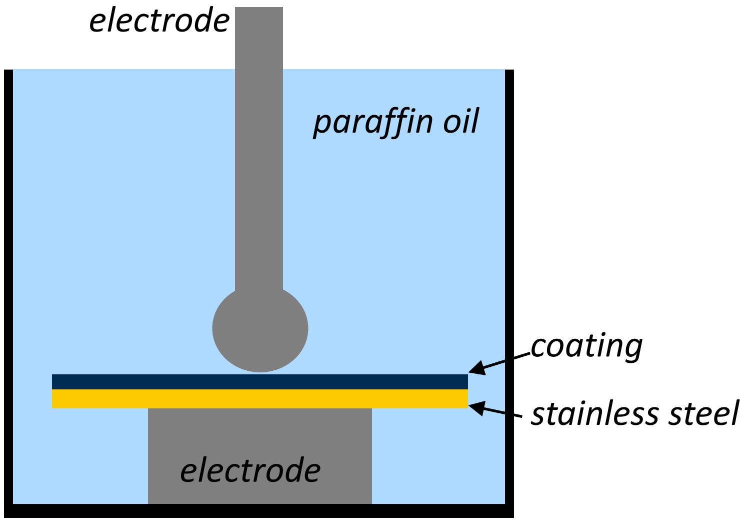
particle filled system

CTE and E-modulus at 77K



Ceramic coating – DBS test setup





Layerthickness measured with a Testboy 70

Voltage applied with a Megger MIT515 in steps