TECHNISCHE UNIVERSITÄT WIEN
Vienna University of Technology

USTEM

Microstructural characterization of superconducting materials

Alice Moros, ESR12, WP2 - Materials
TU Wien - USTEM

EASITrain

Sample preparation & electron microscopy techniques

Grinding/polishing machines

Vibromet: fine polishing

Precision Ion Polishing System

SEM

SE, BSE imaging, EDX, EBSD (normal & transmission mode)

TEM

Imaging and STEM, EDX EELS, electron diffraction, HRTEM

FIB

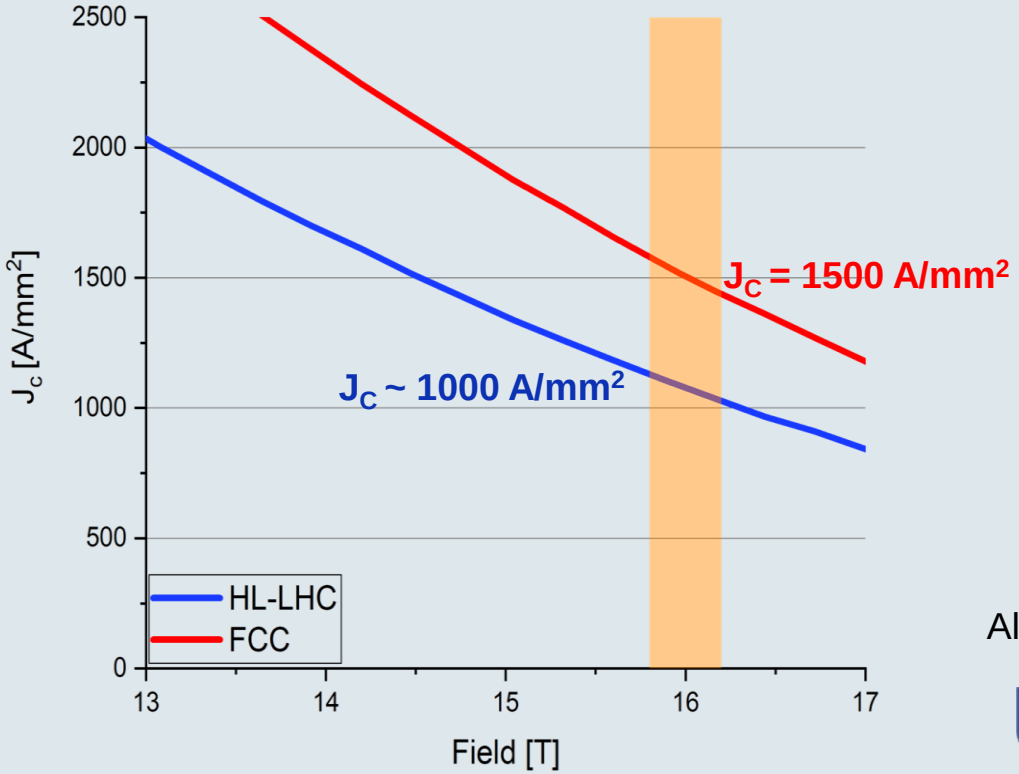
Lamellas for TEM analysis

A route towards the Future Circular Collider

Magnets	Nb_3Sn	JSC VNIINM	MgB_2 ASG SUPERCONDUCTORS	YBCO BRUKER
Beam Screen	Tl-1223	SPIN		
RF Cavities	NbN	UNIVERSITÄT SIEGEN		

Nb_3Sn (A15)

FCC requirements: J_C (non Cu) = 1.5 kA/mm² at 16 T & 4.2 K



J_c ↑

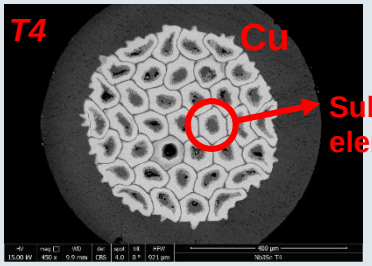
- **Inhomogeneities** ↓
- Grain size ↓
- Artificial defects

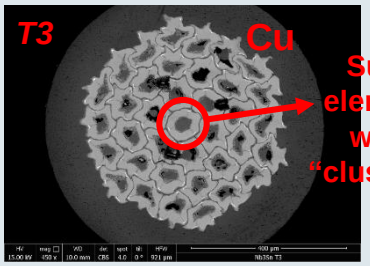
Alice (ESR12) ⟲ Mattia O. (ESR13)

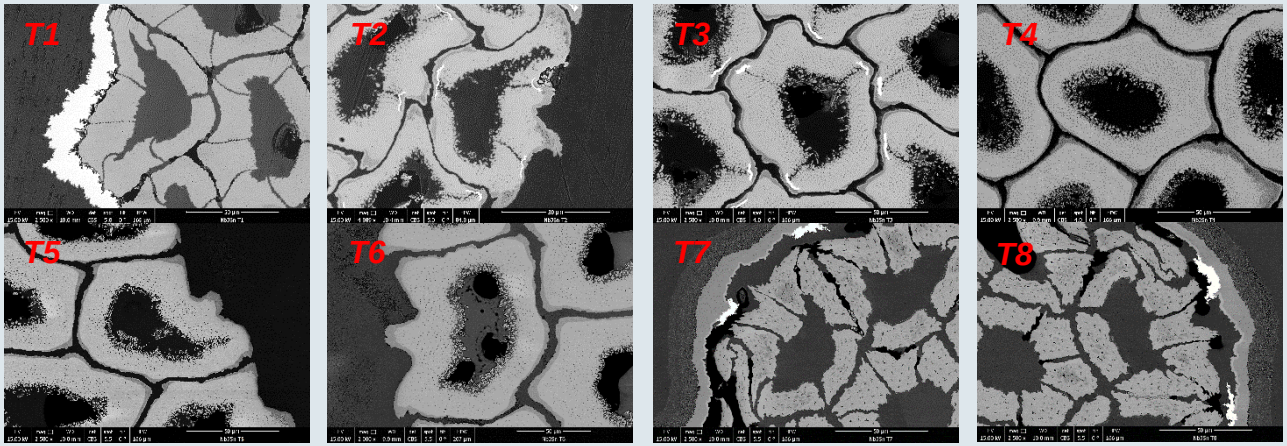
Magnet applications

Internal Tin Nb_3Sn wires

Wire identification	T1	T7; T8	T4; T5	T6	T2; T3
Wire dia, mm	0.7	0.7	**0.7**	1	T2: 0.36; T3: **0.7**
Barrier	**Common Ta**	**Common Nb + Ta**	**Distributed Nb**	**Distributed Nb**	**Distributed Nb + Ta**
Heat treatment	370 °C, 100 h + 665 °C, 100 h	T7: 200 °C, 24 h +380 °C, 50 h + 570 °C, 112 h + 660 °C, 100 h; T8: 380 °C, 48 h + 580 °C, 48 h + 700 °C, 92 h	**T4: 210 °C, 50 h + 400 °C, 50 h + 665 °C, 50 h**; T5: 370 °C, 100 h + 665 °C, 50 h	370 °C, 100 h + 665 °C, 50 h	**210 °C, 50 h+400 °C, 50 h+665 °C, 50 h**

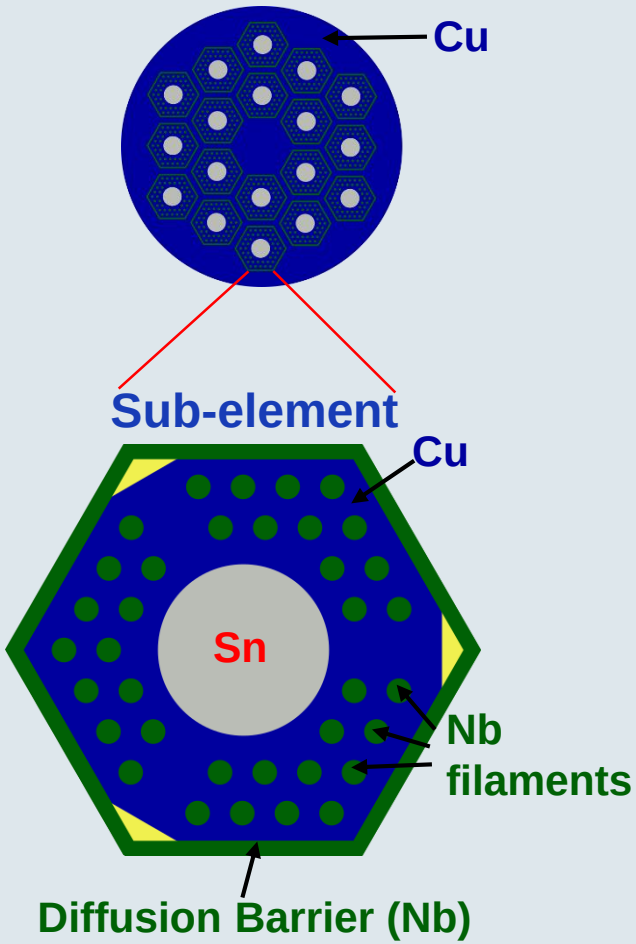




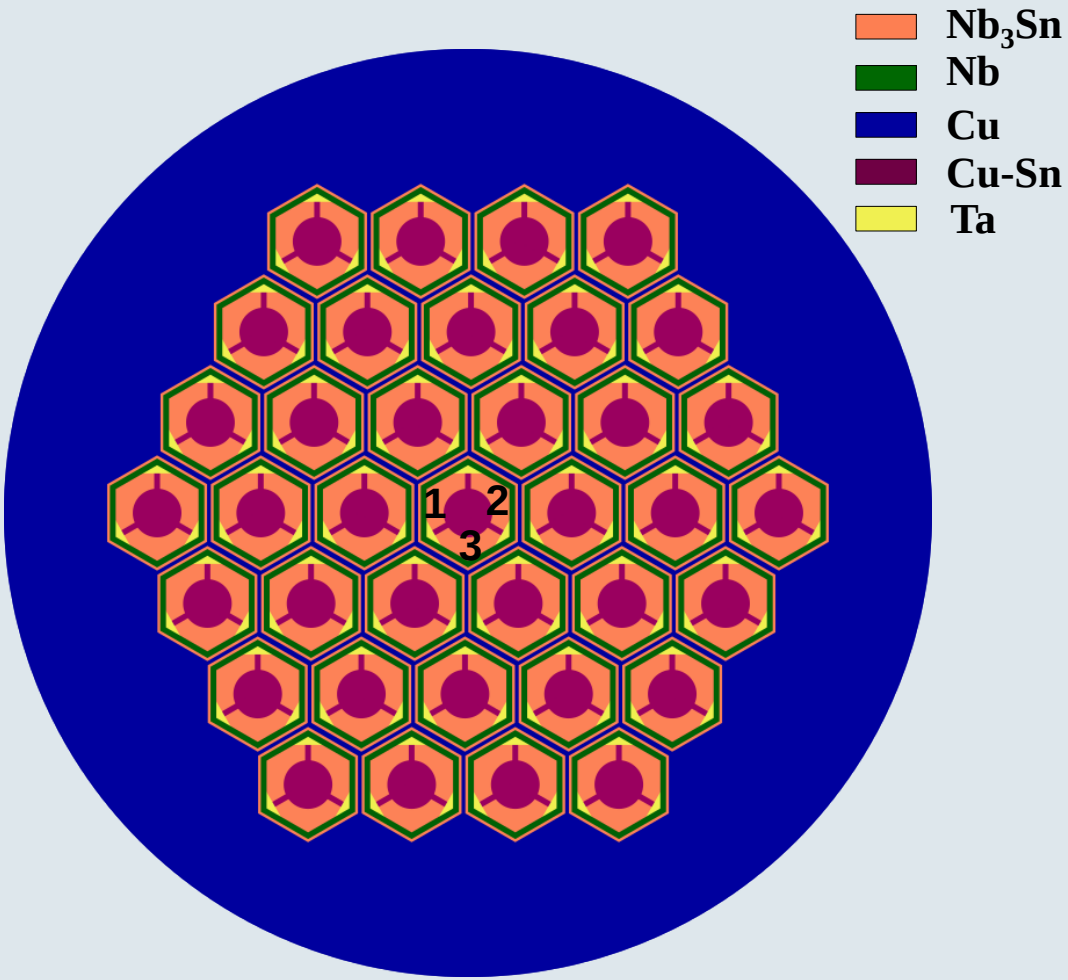


Magnet applications

Internal Tin Nb_3Sn wires

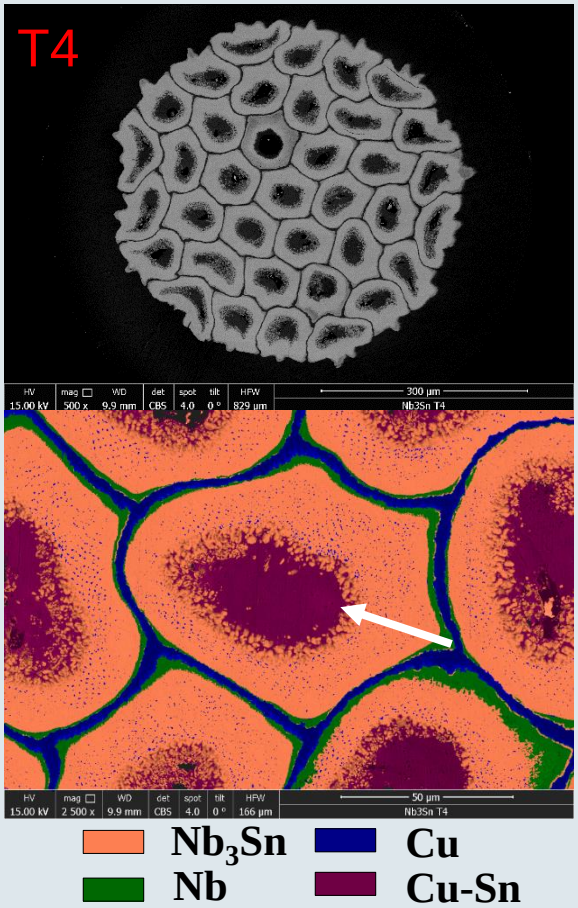


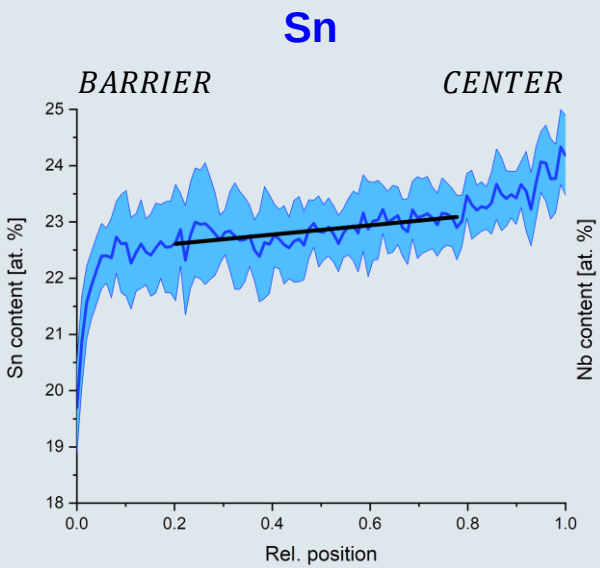
After heat treatment



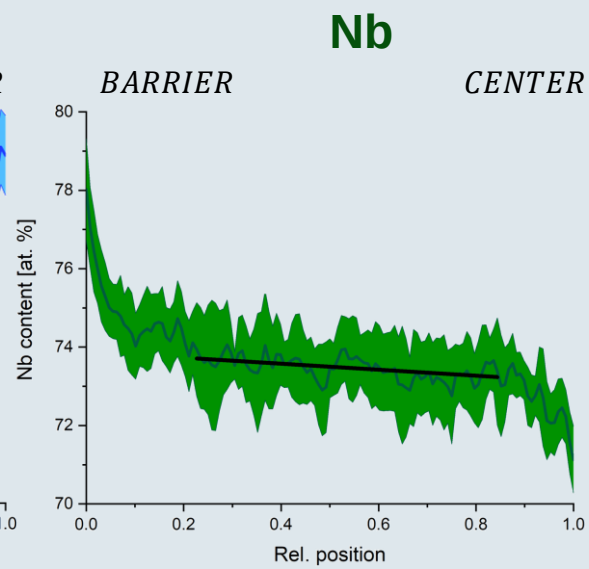
Homogeneity analysis: composition gradients

- **SEM-EDX line scans over 10 sub-elements along the radial direction**





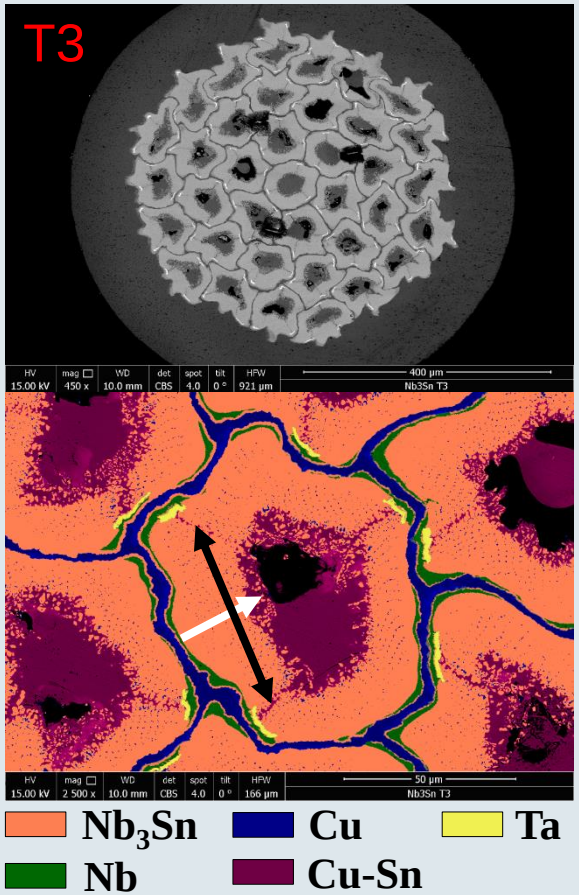
$$Slope: \ 0.83 \pm 0.12$$

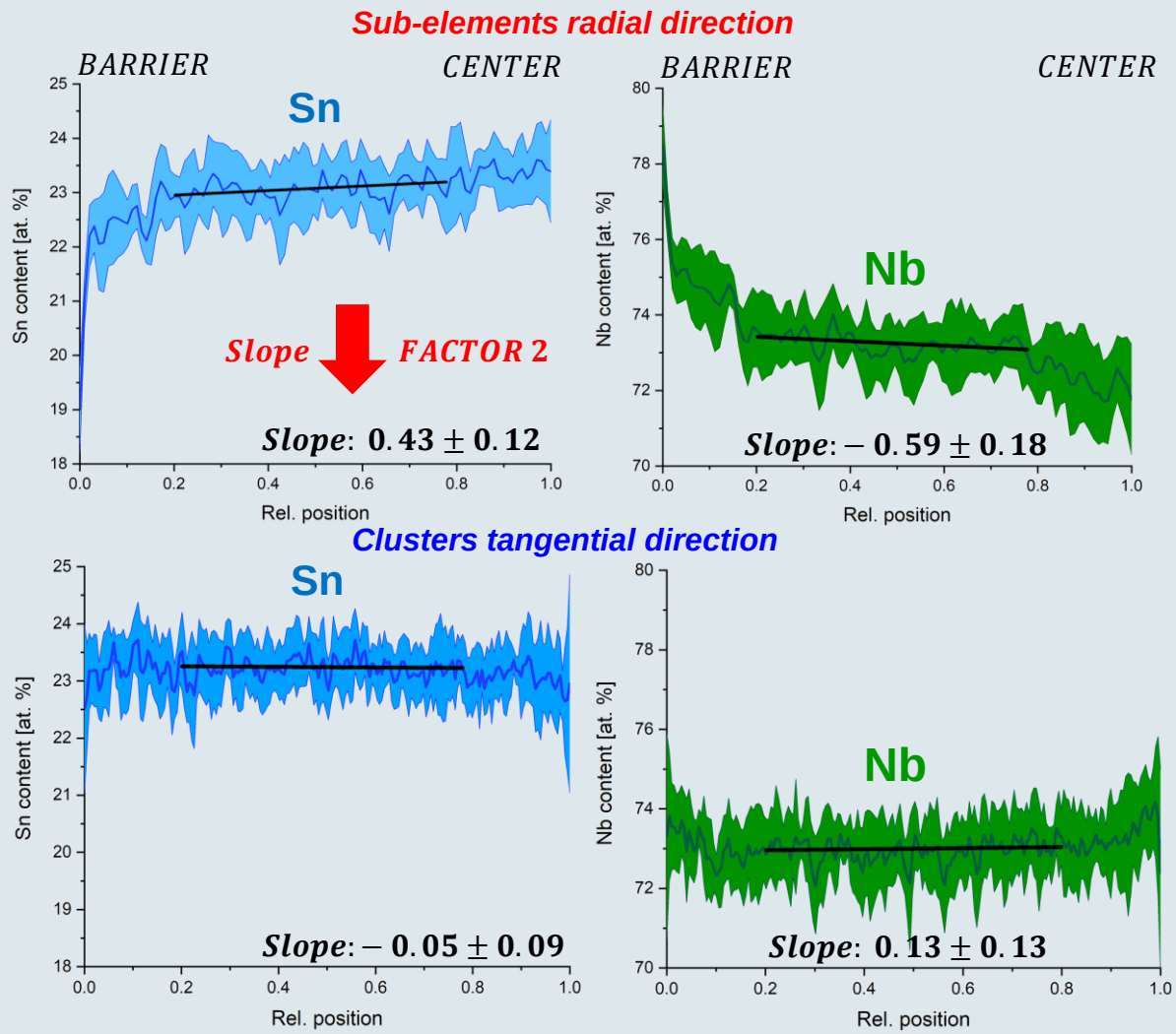


$$Slope: -0.76 \pm 0.14$$

Results presented at ASEM Workshop, Graz (25th-26th April 2019)

Homogeneity analysis: composition gradients

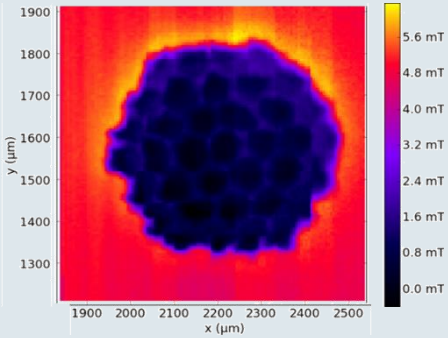




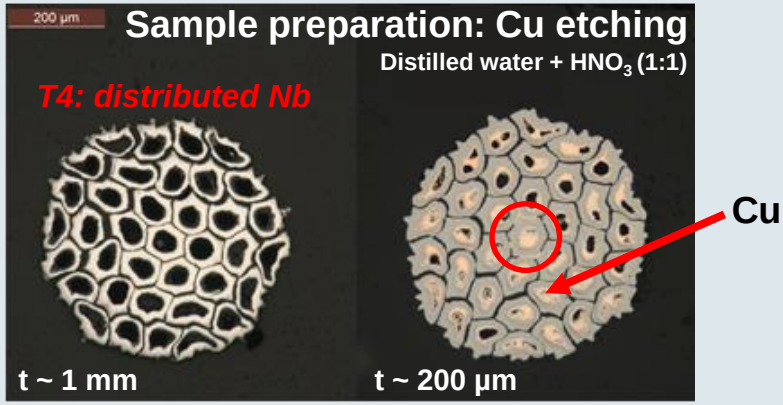
Results presented at ASEM Workshop, Graz (25th-26th April 2019)

Next steps

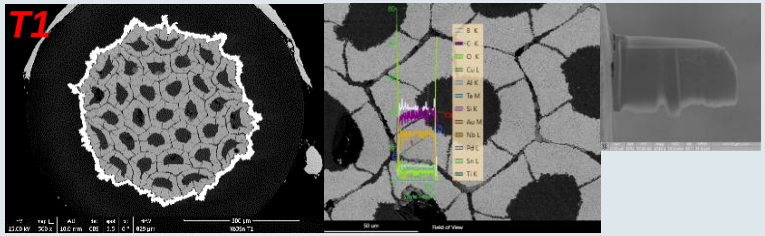
Scanning Hall Probe Microscopy: Meissner-state measurements (T range: 5 – 19 K)

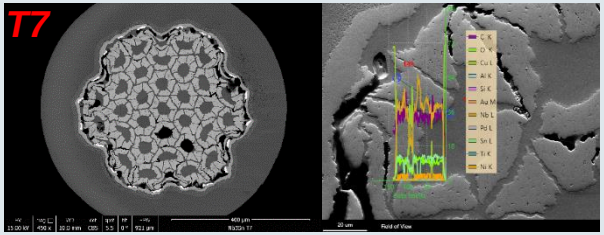


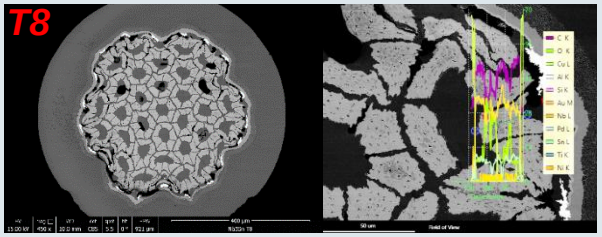
Mattia O., ESR13



Other samples SEM homogeneity analyses & TEM investigations







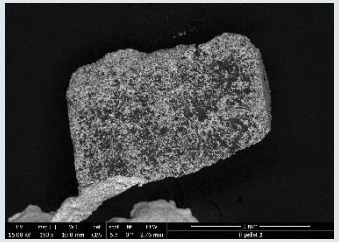
Sample preparation → Microstructure ⟲ Magnetic & supeconducting characterization

$(Tl,Bi)(Pb,Ba,Sr)_2Ca_2Cu_3O_x$ → Tl-1223

Alice (ESR12) USTEM Aisha (ESR6) SPIN Sigrid (PhD student) ATOMINSTITUT

Beam screen → **Operation T: *50 K*** → ***HTS*: *lower surface resistance* than Cu at 50 K**

Pellets

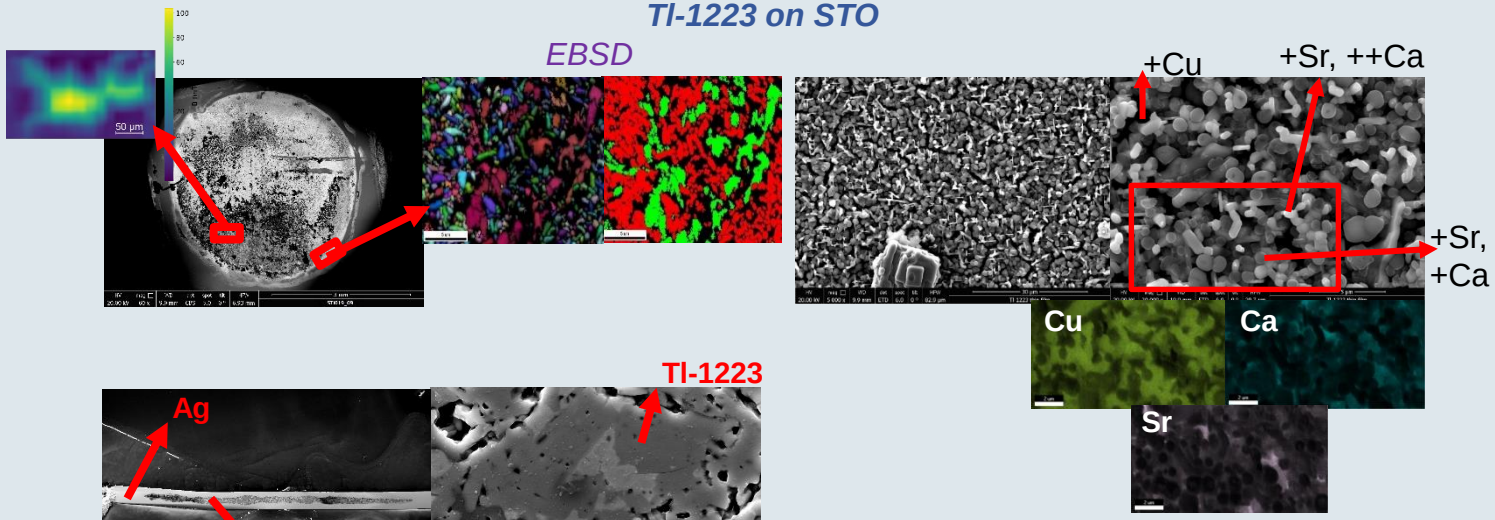


Evaluation of the homogeneity in Tl-1223 phase formation

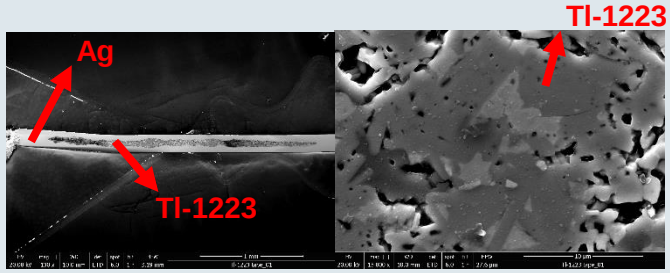
Thin films

Tl-1223 on STO

EBSD



Tapes



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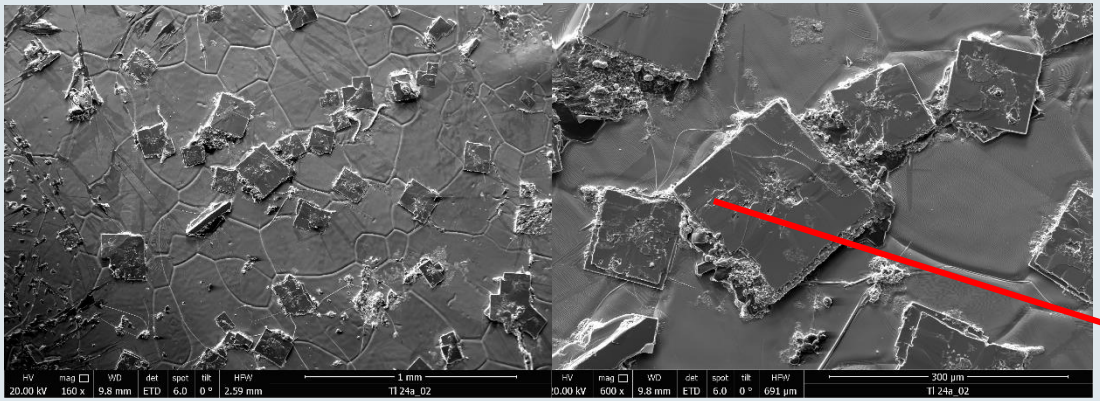
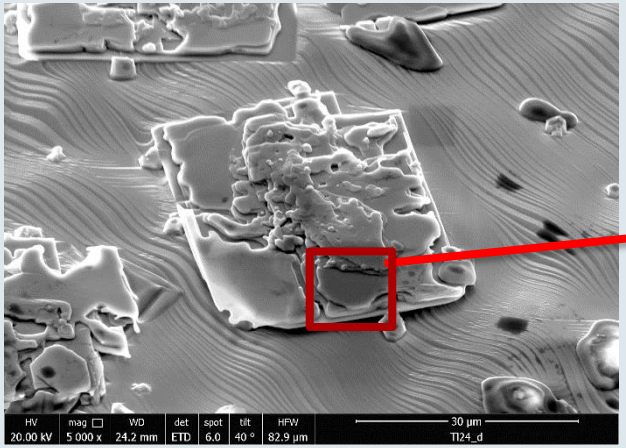
$(Tl,Bi)(Pb,Ba,Sr)_2Ca_2Cu_3O_x$ → Tl-1223

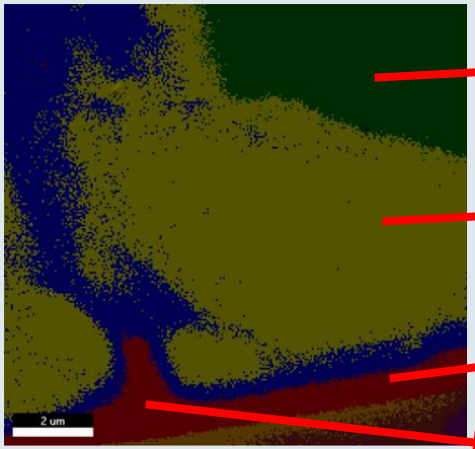
Thin films: ***Tl-1223 on Ag***



Aisha´s secondment at USTEM:
11.02.2019 – 22.02.2019

~ Tl-1212





Sr rich area

~ 1212

~ 1212 (+ Cu)

~ 1234

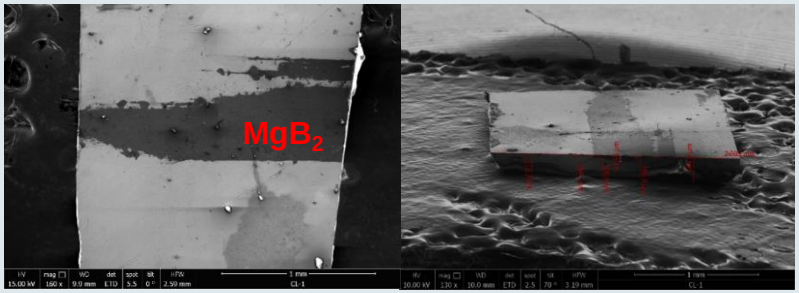
New samples

MgB_2: first steps

Alice (ESR12) USTEM ↔ Mattia D. (ESR7) ASG SUPERCONDUCTORS ↔ Mattia O. (ESR13) ATOMINSTITUT

MgB_2 *for next generation 10 T magnets*

Tapes

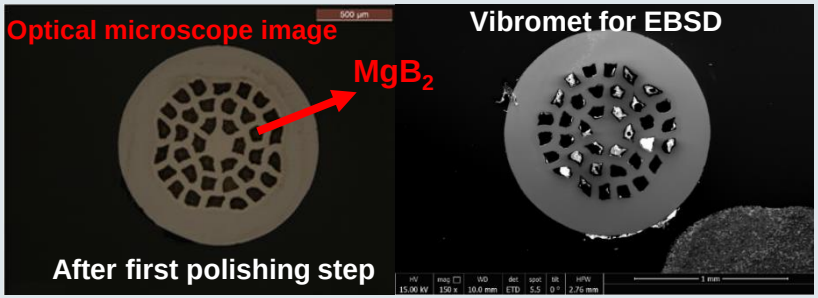


Secondment at

ASG SUPERCONDUCTORS

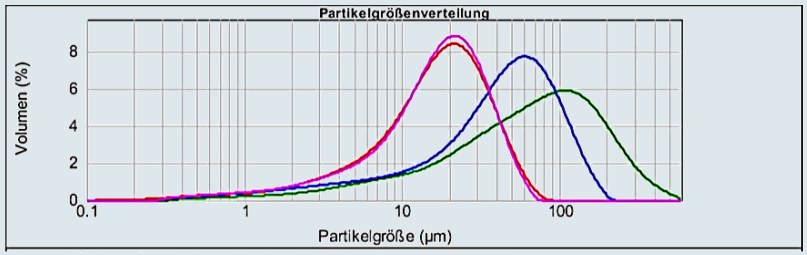
in November 2019

Wires



Grain orientation analysis

Powder granulometry analysis



ICEBE, TU Wien

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$YBa_2Cu_3O_7$: first steps

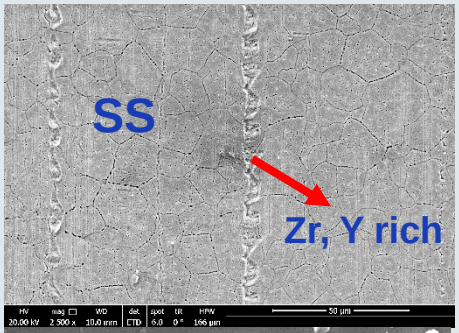
Alice (ESR12) USTEM ⟲ Johannes (ESR2) BRUKER

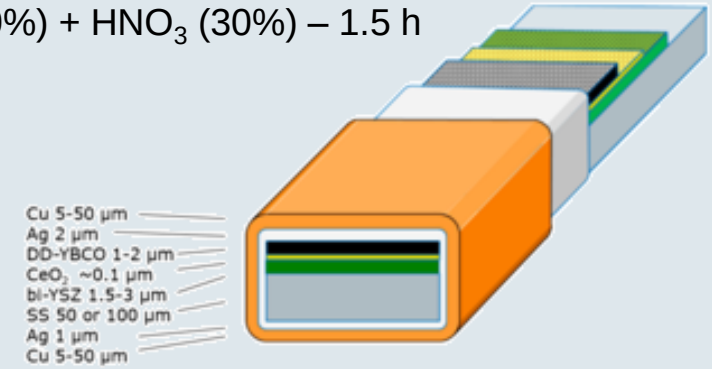
YBCO** coated conductors at **3 T, 77 K** compared to **5 T, 4.2

How the grain orientation/misalignment influences I_c?

EBSD sample prep. → 2 Ag etching methods: **1.** $H_2O_2 + NH_3$ (1:1) – 4 h
2. H_2O (70%) + HNO_3 (30%) – 1.5 h

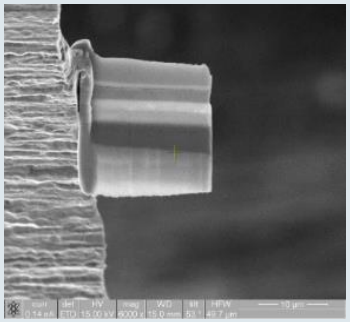
1.





2.

Back scattering imaging for better contrast



Ongoing FIB lamellas preparation for TEM analysis

Secondment at BRUKER → Beginning of 2020

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NbN: first steps

Alice (ESR12) USTEM ⟲ Stewart (ESR14) UNIVERSITÄT SIEGEN

*Deposition process of **LTS thin-film coatings on Cu** substrates* → **Superconducting RF Cavities**

13 samples received: **NbN (B1)** on **OFHC Copper** (~ 2 mm)

Films thickness range: 1-1.4 µm

Nb interlayer onto Cu substrate: ~ 80-90 nm

- Phase identification within the films

↓

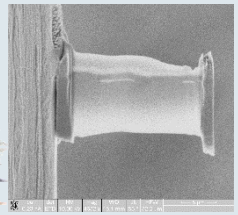
Combined EBSD and EDX

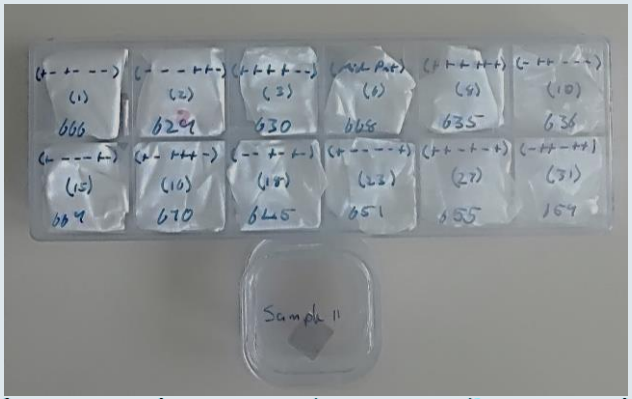
- Cross section evolution from the substrate to the film surface → grain size development, grain orientation/misalignment

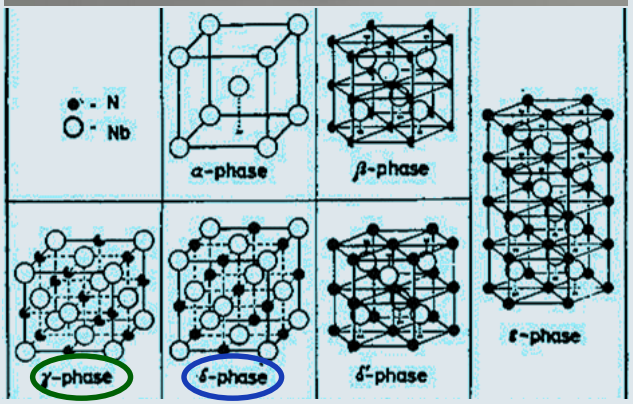
↓

EBSD traditional & transmission mode

- SAMPLE PREPARATION







Nb_4N_3 T_c:12-15 K

NbN_x T_c:15-17.3 K

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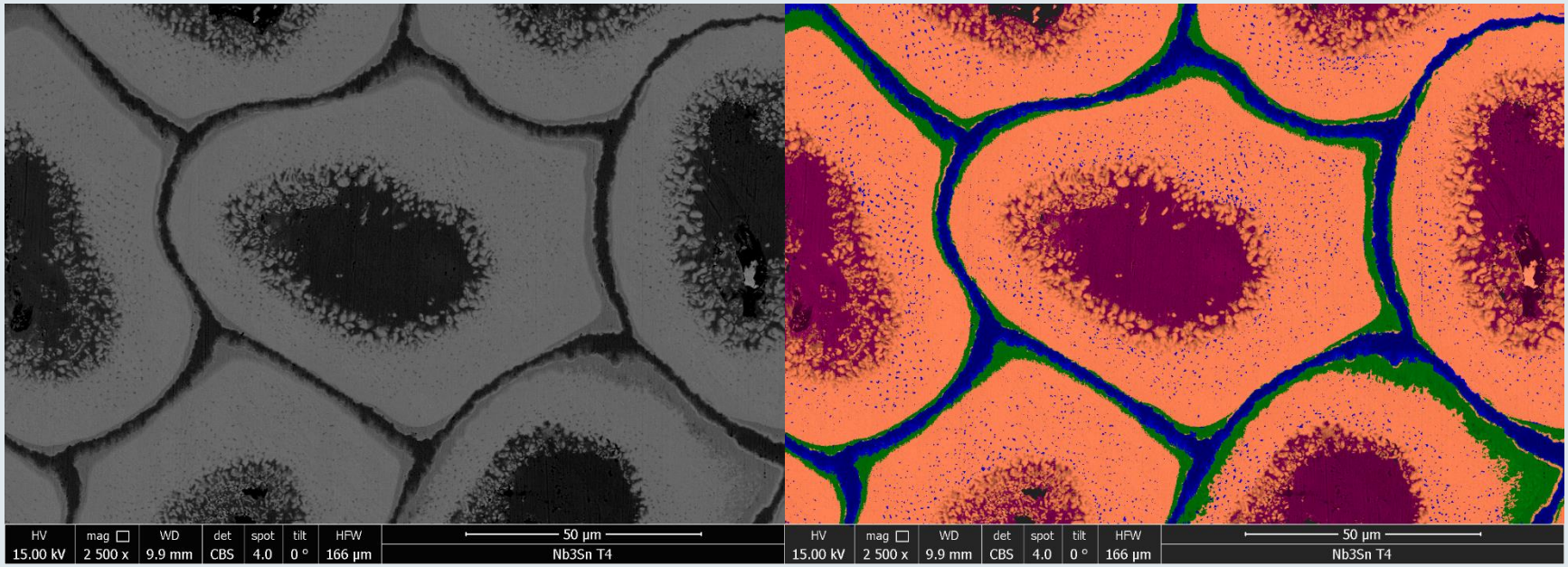
Thank you for the attention!

This work is part of the Marie Skłodowska-Curie Action EASITrain, funded by the European Union's H2020 Framework Programme under grant agreement no. 764879.

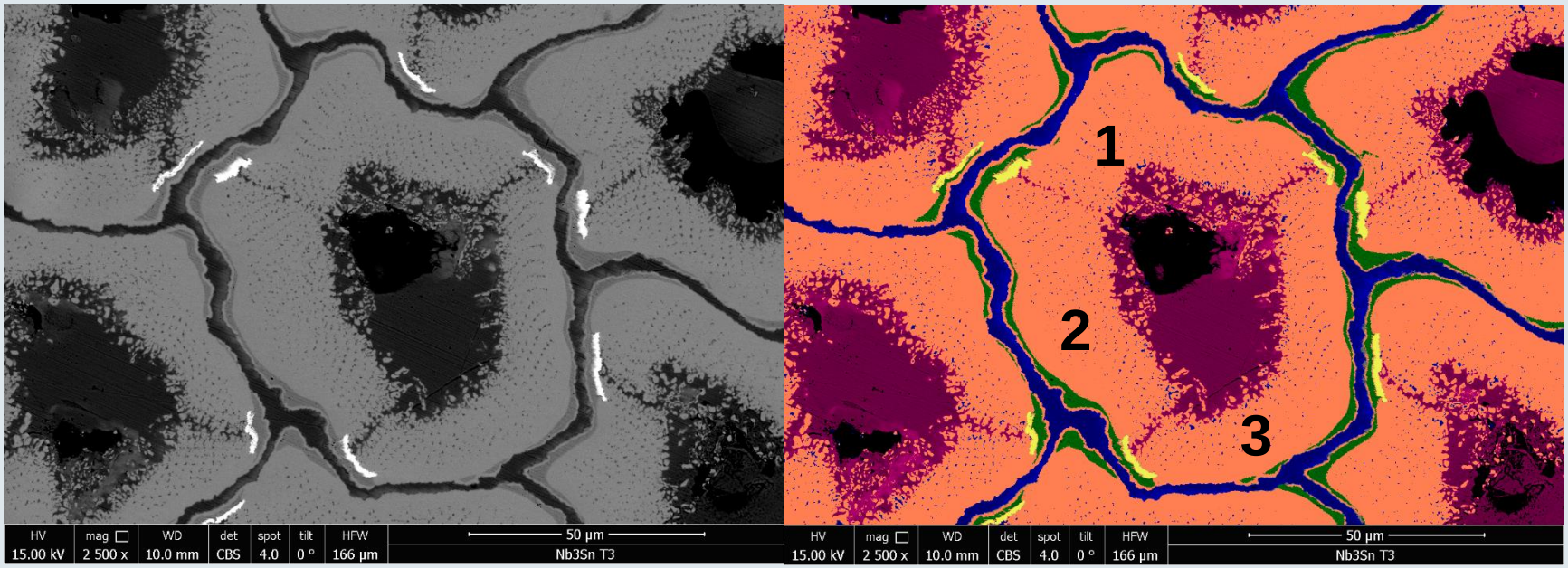
SEM investigation: distributed Nb

T4



- Nb_3Sn
- Nb
- Cu
- Cu-Sn

SEM investigation: distributed Nb + Ta



- Nb_3Sn
- Nb
- Cu
- Cu-Sn
- Ta