TECHNISCHE UNIVERSITÄT WIEN
Vienna University of Technology

USTEM

Microstructural characterization of advanced superconducting materials for different components of the CERN Future Circular Collider (FCC-hh)

Alice Moros, PhD student at TU Wien – USTEM

EASITrain

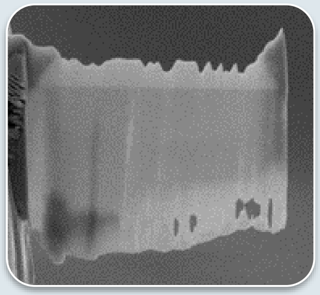
EASISchool 3 – Student Workshop
CNR-SPIN Genoa, 8th - 9th October 2020

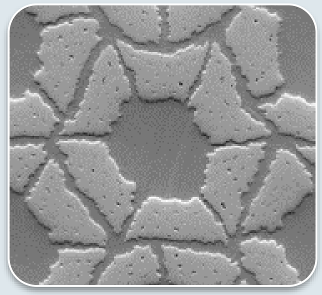
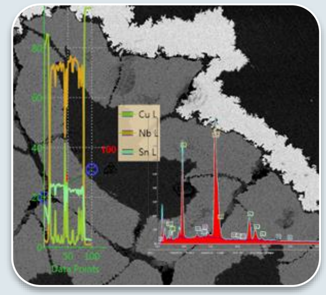
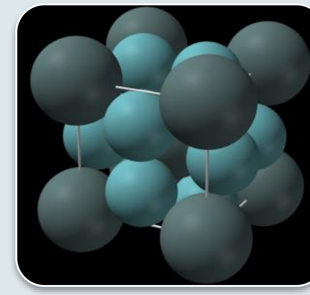
ESAS
The European Society for Applied Superconductivity

The Microstructure Analysis

“Seeing is believing”

Manfred Von Heimendahl *in his* ***introduction to Electron Microscopy of Materials****,1980*



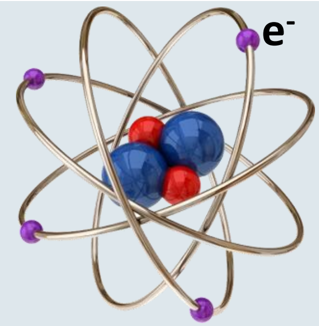




Sample preparation	SEM & TEM	Imaging	Composition analysis	Structure analysis

Mikros (small)   Skopeo (look at)

Greek Origin

The Microstructure Analysis



	LIGHT MICROSCOPE	ELECTRON MICROSCOPE
Source of illumination	The ambient light source is light for the microscope	Electrons are used to “see” – light is replaced by an electron gun built into the column
Lens type	Glass lenses	Electromagnetic lenses
Magnification method	Magnification is changed by moving the lens	Focal length is charged by changing the current through the lens coil
Viewing the sample	Ocular	Fluorescent screen or digital camera
Use of vacuum	No vacuum	Entire electron path from gun to camera must be under vacuum

The Microstructure Analysis

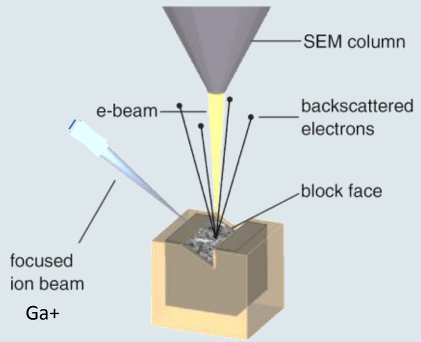
"We see past time in a telescope and present time in a microscope. Hence the apparent enormities of the present"

Victor Hugo, Victor Hugo's Intellectual Autobiography: (Postscriptum de ma vie) (1907)

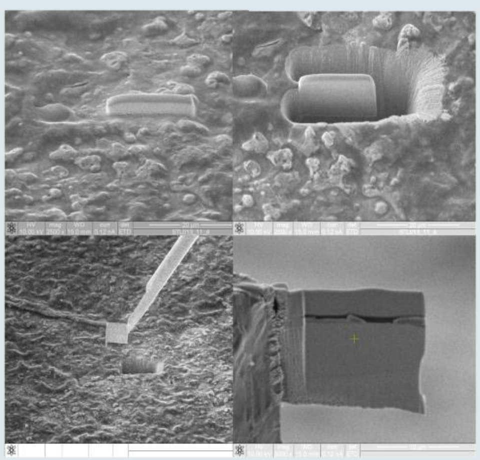
	TEM	SEM
Electron Beam	Broad, static beams	Beam focused to fine point; sample is scanned line by line
Voltages Needed	TEM voltage ranges from 60-300,000 volts	Accelerating voltage much lower; not necessary to penetrate the specimen
Interaction of the beam electrons	Specimen must be very thin	Wide range of specimens allowed
Imaging	Electrons must pass through and be transmitted by the specimen	Information needed is collected near the surface of the specimen
Image Rendering	Transmitted electrons are collectively focused by the objective lens and magnified to create a real image	Beam is scanned along the surface of the sample to build up the image

Transmission electron microscopy

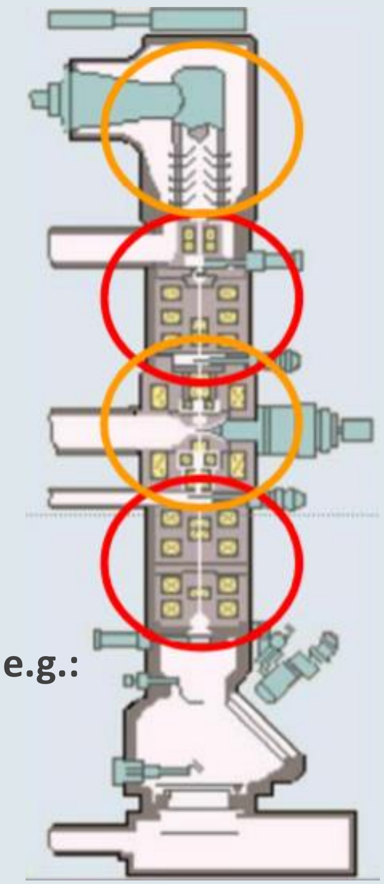
Sample preparation



Focused Ion Beam - FIB



TEM - scheme



Electron source

Condensor

Objective lens, sample

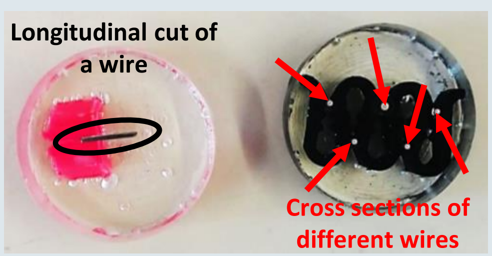
Magnification system

Detectors, e.g.:
STEM
EDX
EELS
GIF

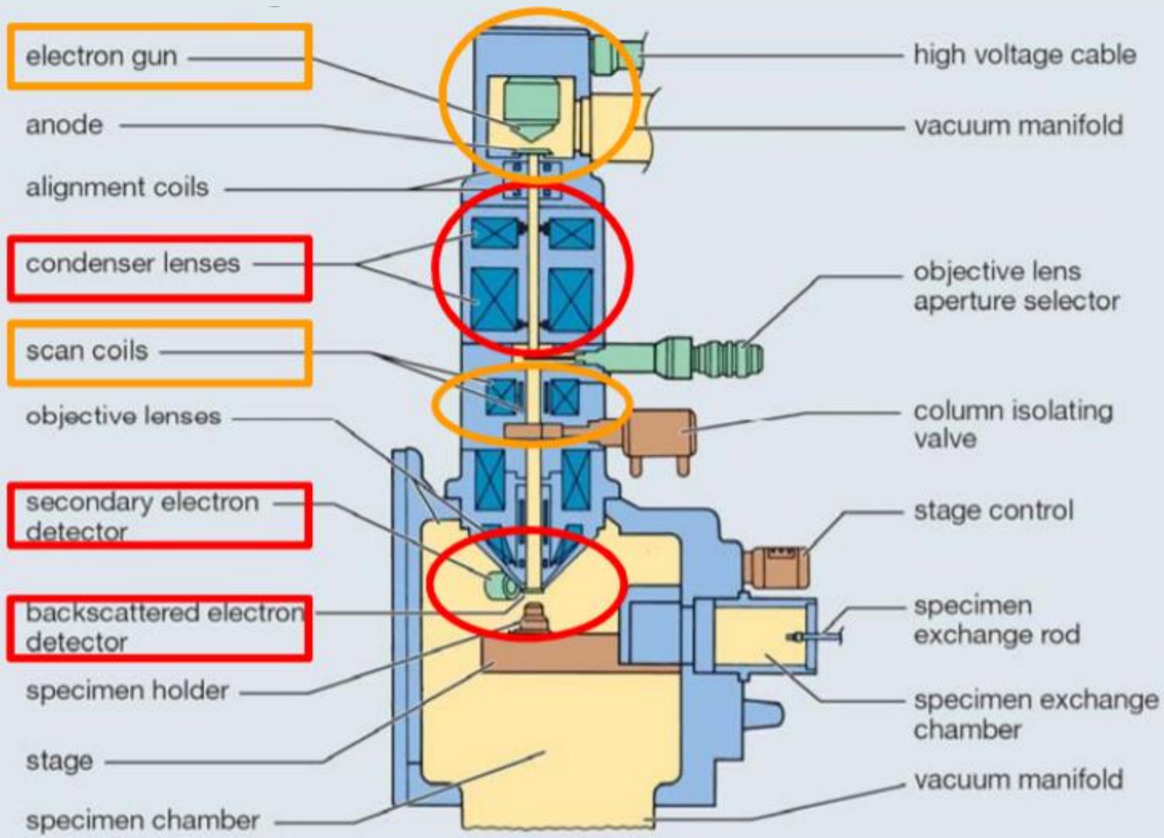
Camera / CCD

Scanning electron microscopy

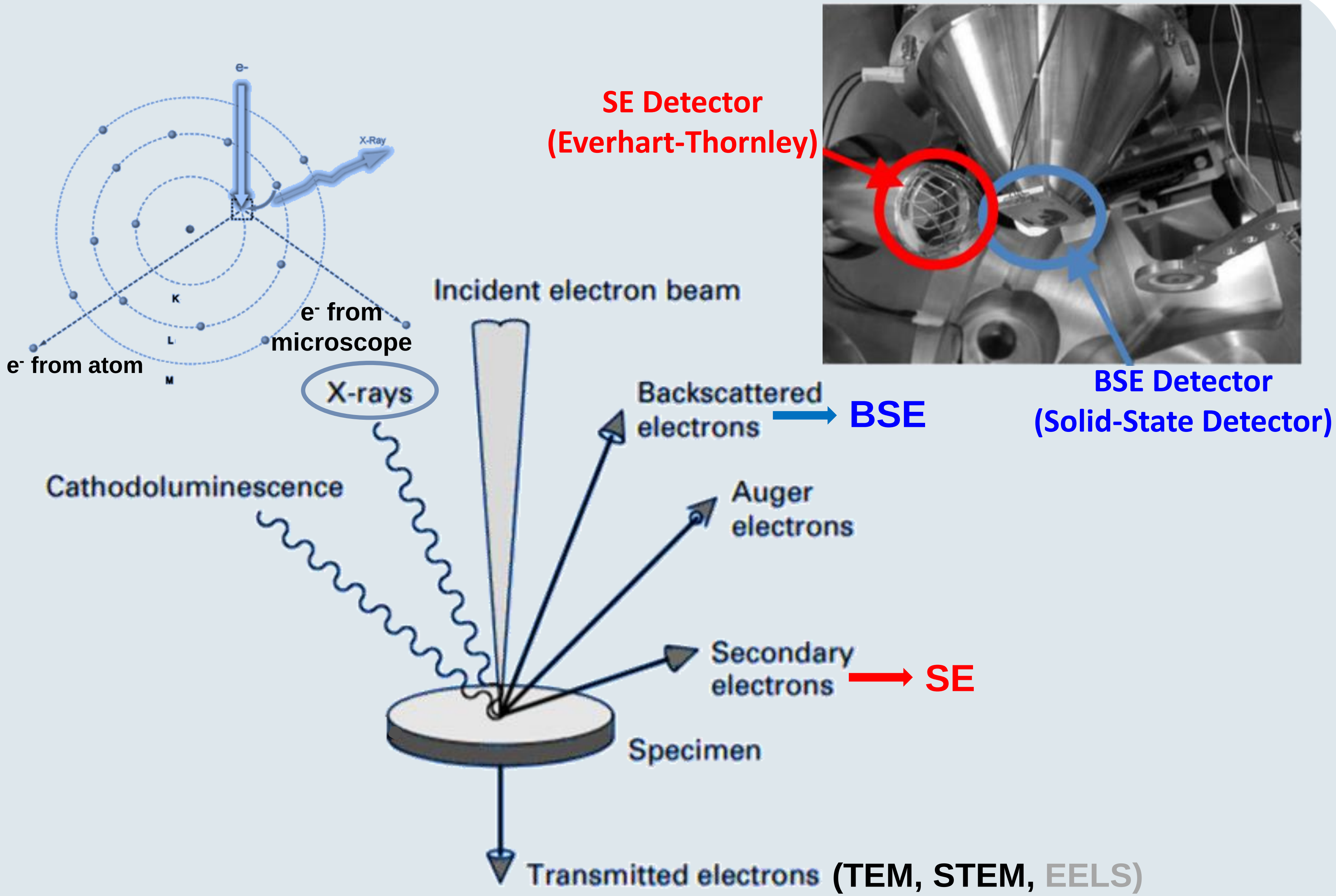
Sample preparation



SEM - scheme

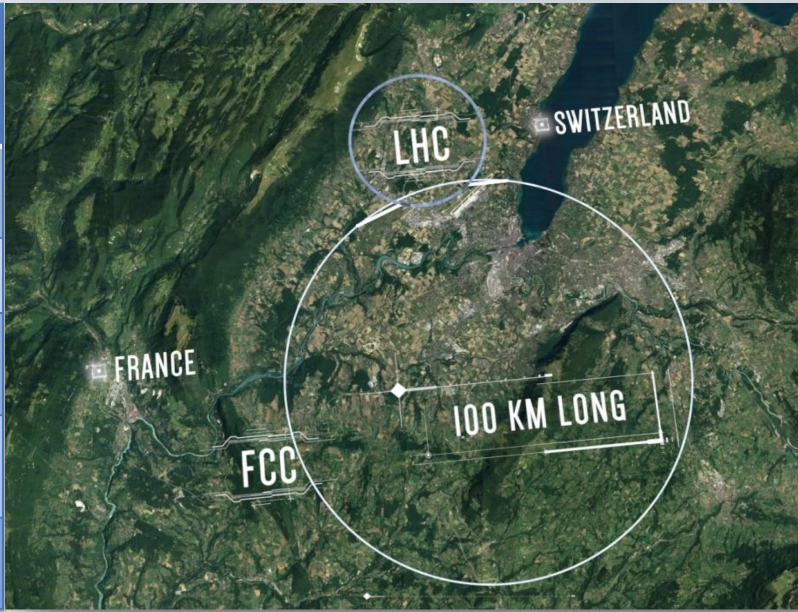


Electron – Matter Interaction



Towards the CERN Future Circular Collider

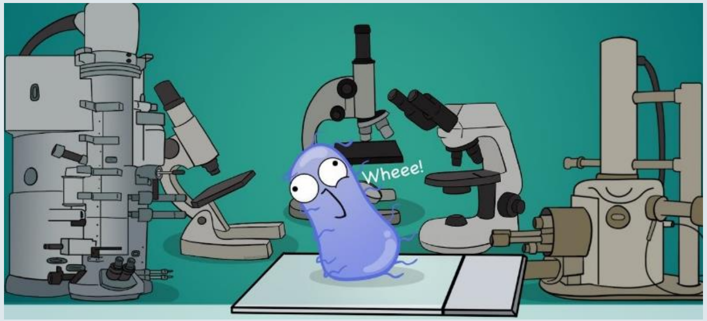
Parameter	LHC	FCC-hh
Collision energy [TeV]	14	**100**
Dipole field [T]	8.33	**16**
Circumference [km]	26.7	**97.75**
Beam current [A]	0.58	**0.5**
Synchr. rad. power [kW]	3.6	**2400**



- **High-field superconducting bending magnets:** **Nb_3Sn**
  Critical current density J_c = 1.5 kA/mm^2 at 16 T & 4.2 K

- **Low surface resistance beam screen:** **Tl1223**
  Operation T = 50 K

- **Superconducting links:** **MgB_2**



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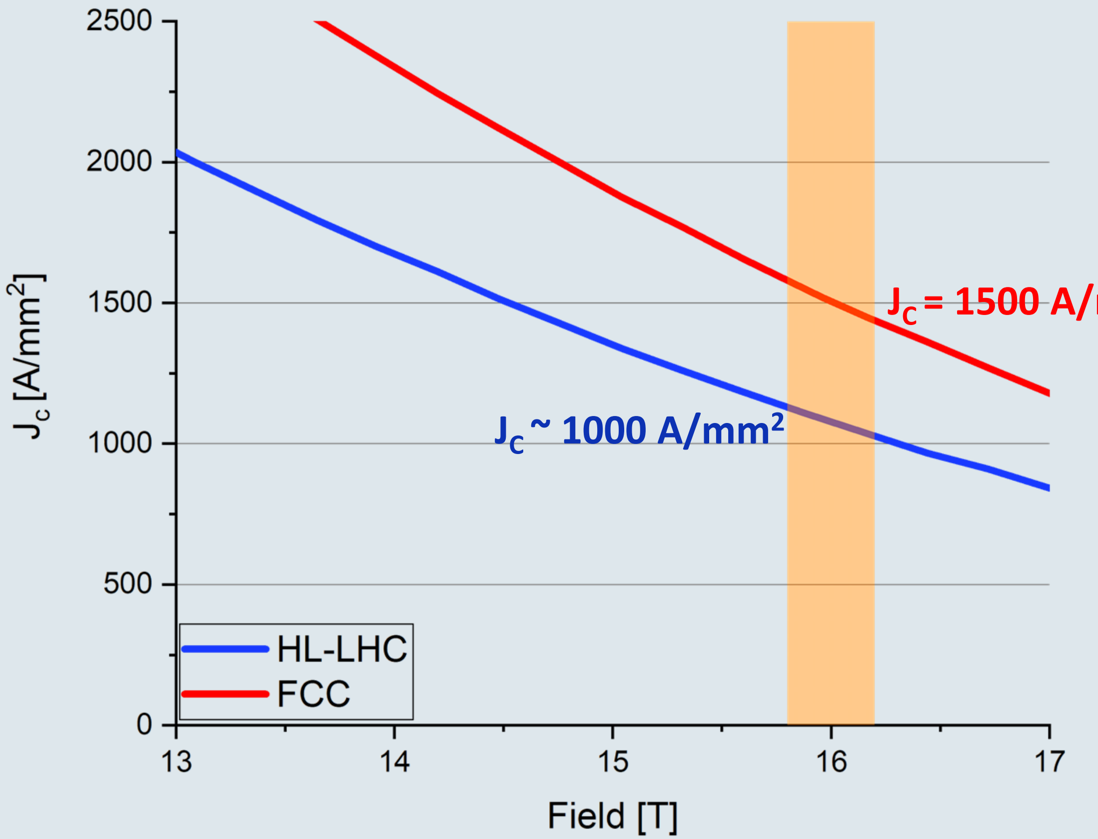
The critical current density J_c

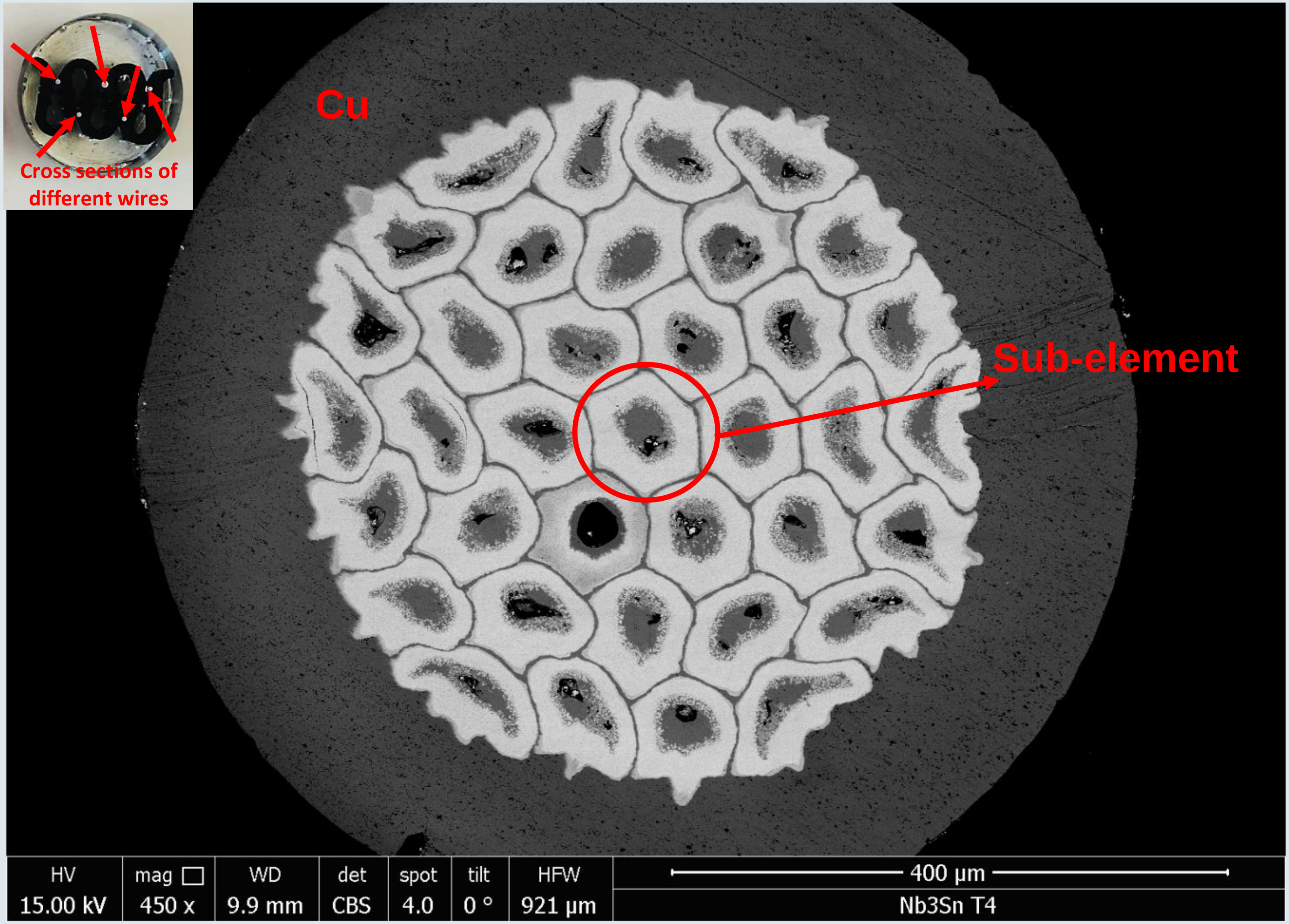
Critical current density J_c = 1.5 kA/mm² at 16 T & 4.2 K → Nb_3Sn



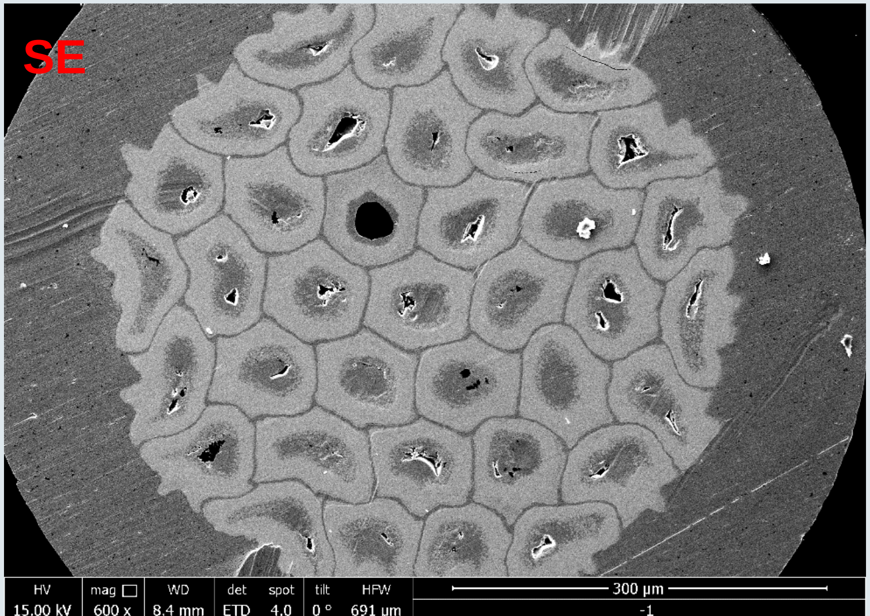
J_c ↑

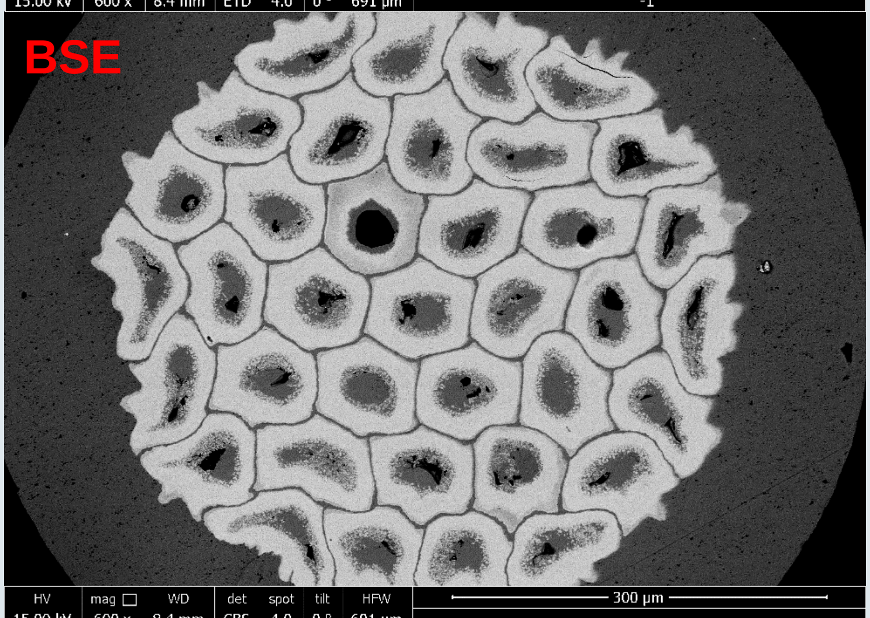
- **Inhomogeneities ↓**
- Grain size ↓
- Artificial defects

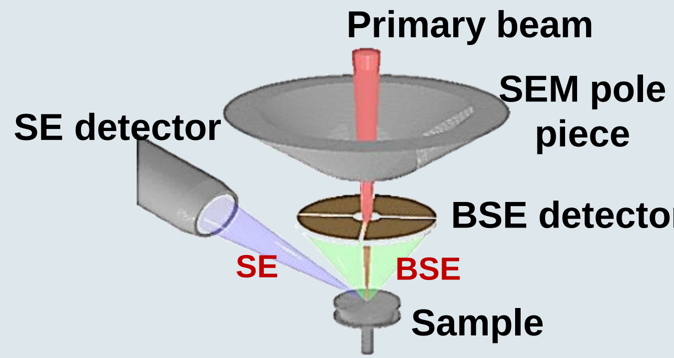
Nb_3Sn wires

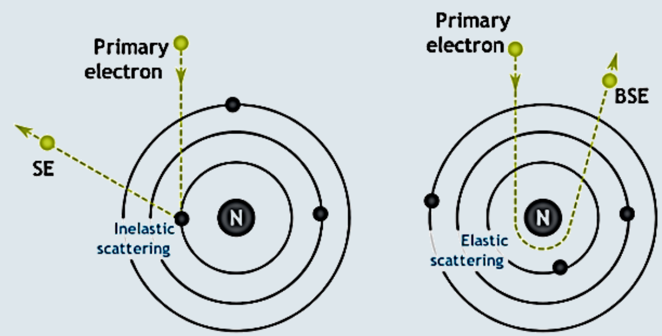

Cross sections of different wires
Cu
Sub-element
HV
15.00 kV
mag
450 x
WD
9.9 mm
det
CBS
spot
4.0
tilt
0 °
HFW
921 µm
400 µm
Nb3Sn T4

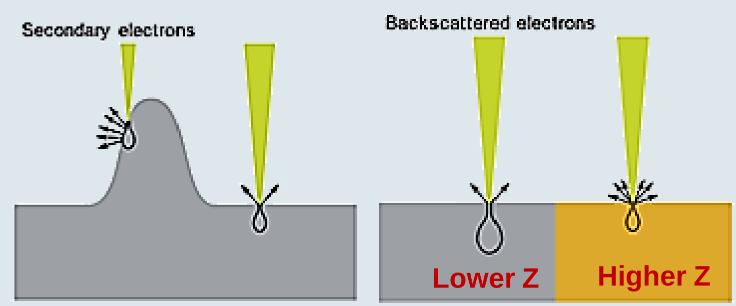
SEM imaging: SE vs BSE



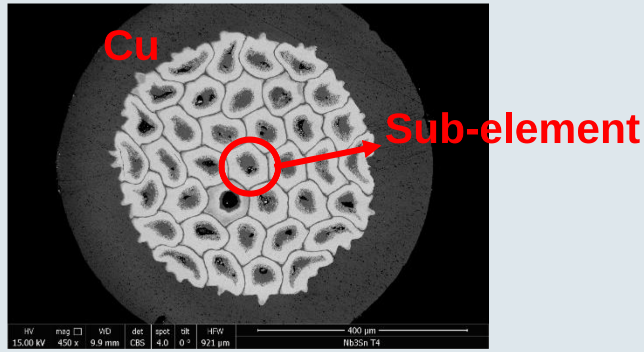




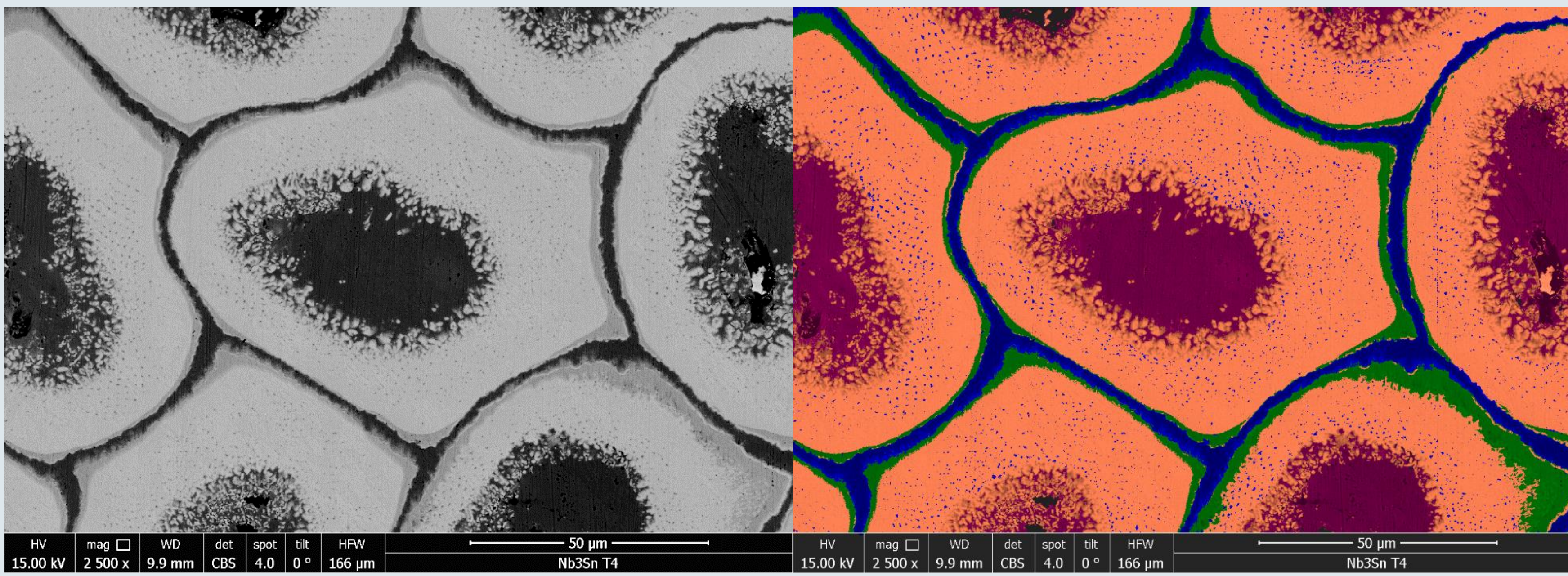




Distributed Nb



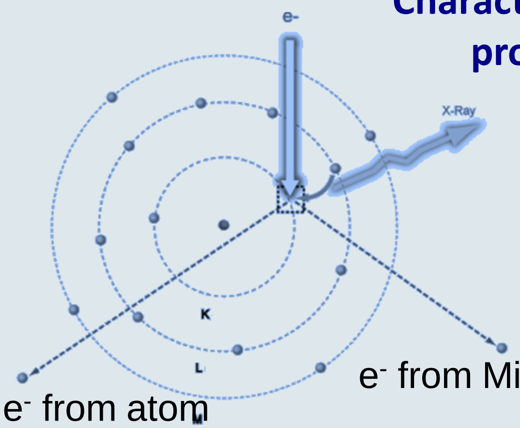
- Nb_3Sn
- Nb
- Cu
- Cu-Sn



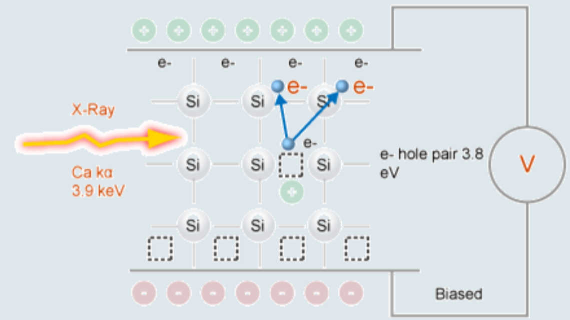
SEM EDX - Homogeneity investigation

➢ **SEM-EDX line scans over different sub-elements along the radial direction → Sn gradient evaluation**

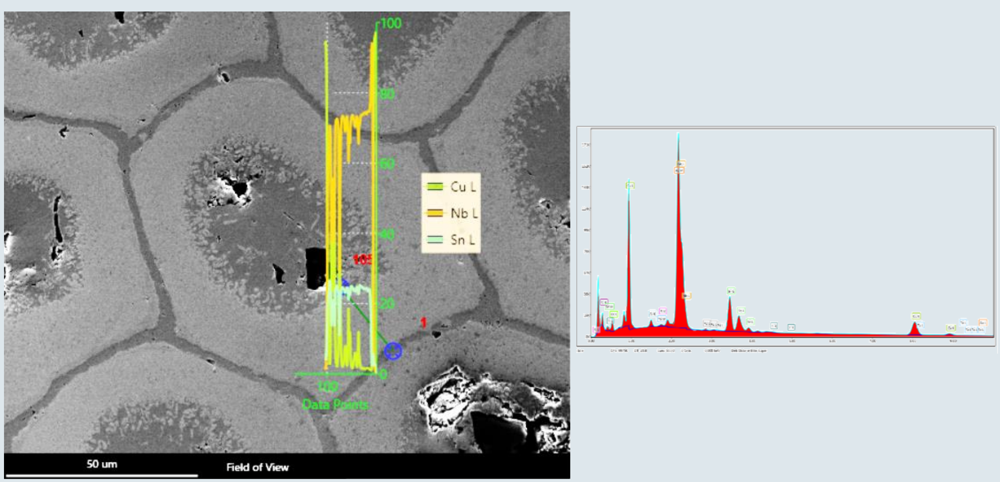
Characteristic X-Ray production



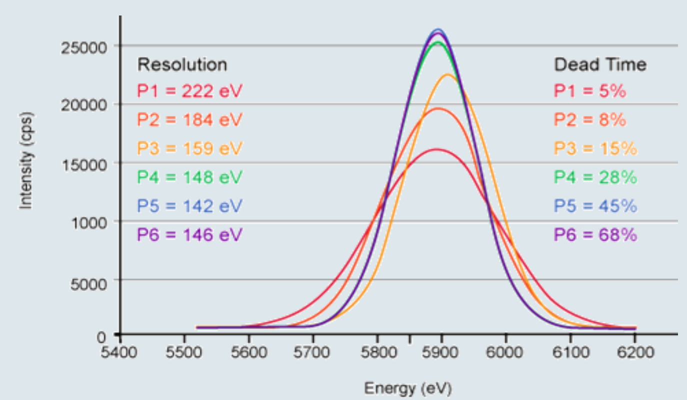
Silicon Drift Detector - SDD



Spectrum: I (counts) vs E (eV)

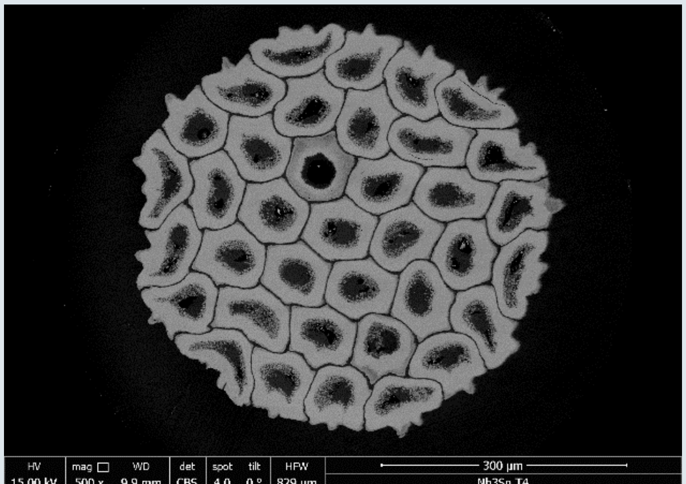


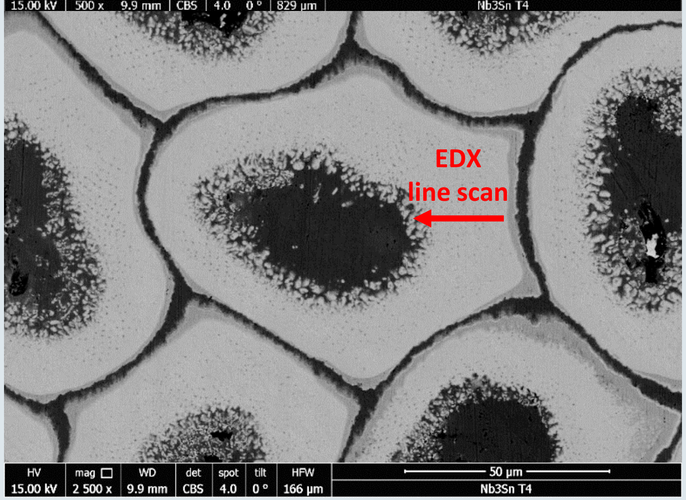
Process time, Resolution and Dead time

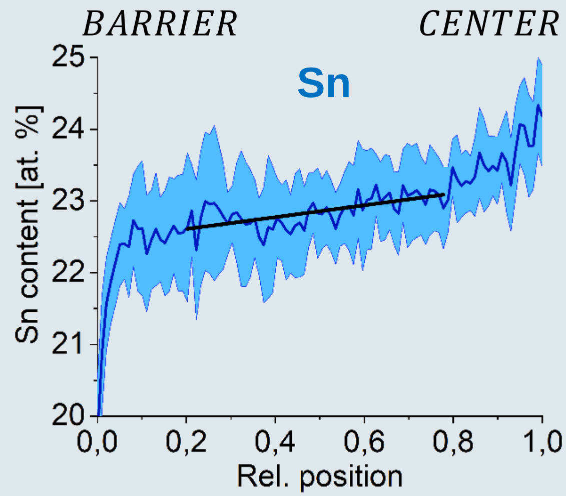


SEM EDX - Homogeneity investigation

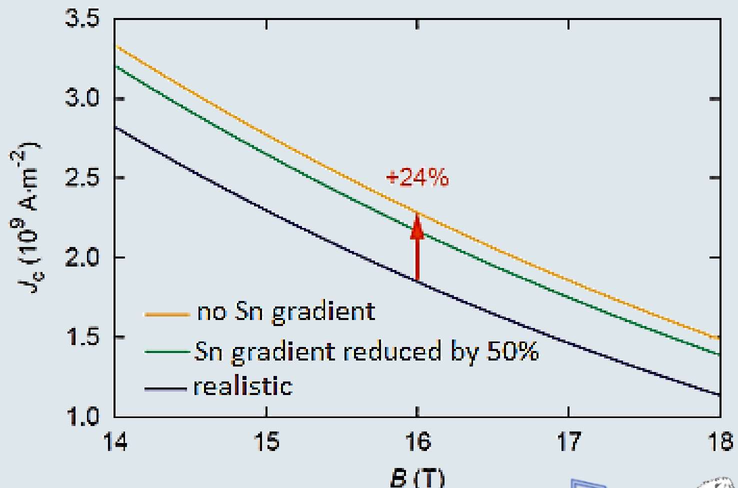
➢ **SEM-EDX line scans over different sub-elements along the radial direction → Sn gradient statistical analysis**





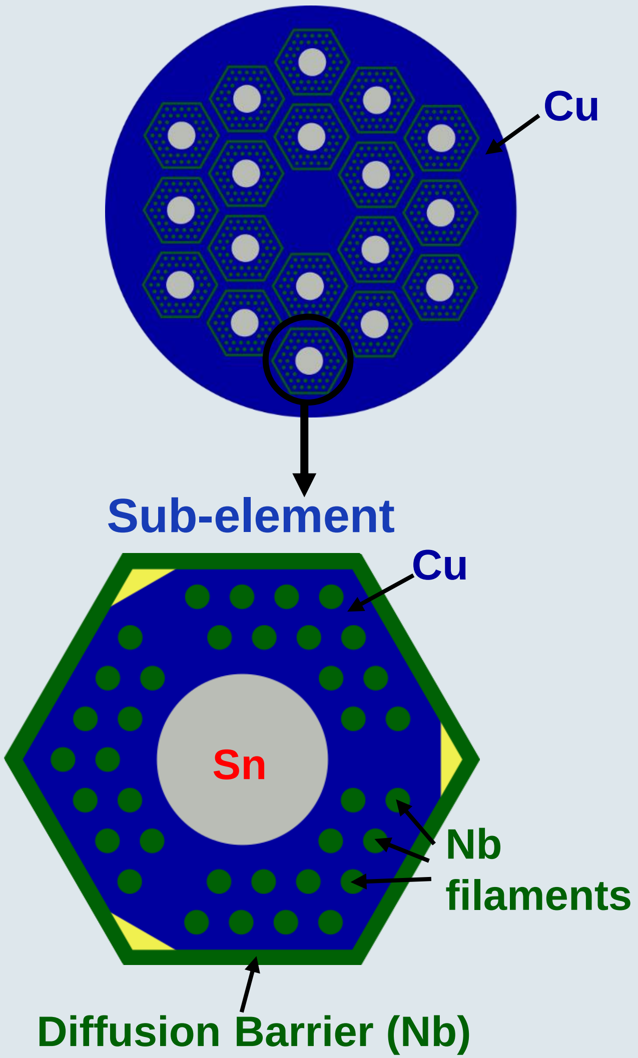


Gradient:
(0.025 ± 0.004) at. %/µm

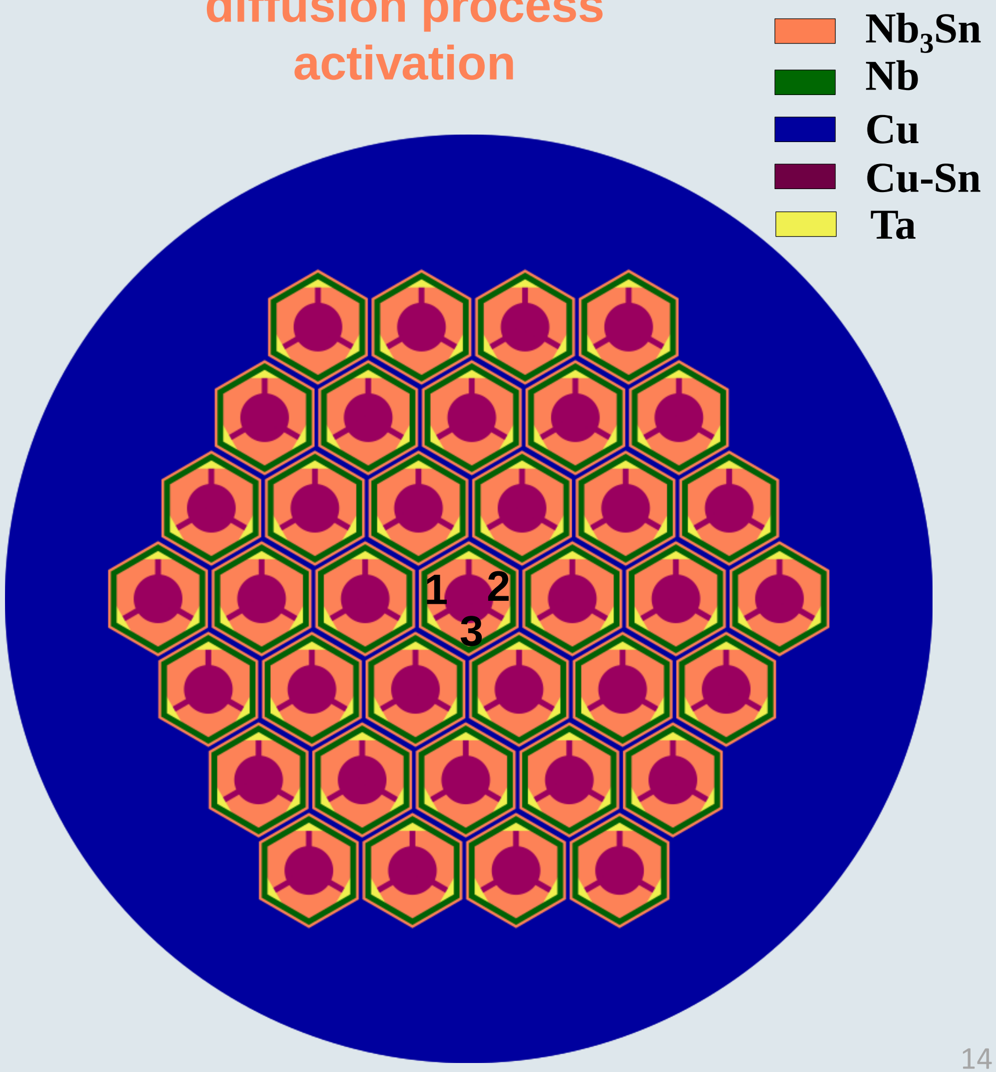


Thomas Baumgartner, ATOMINSTITUT, TU Wien

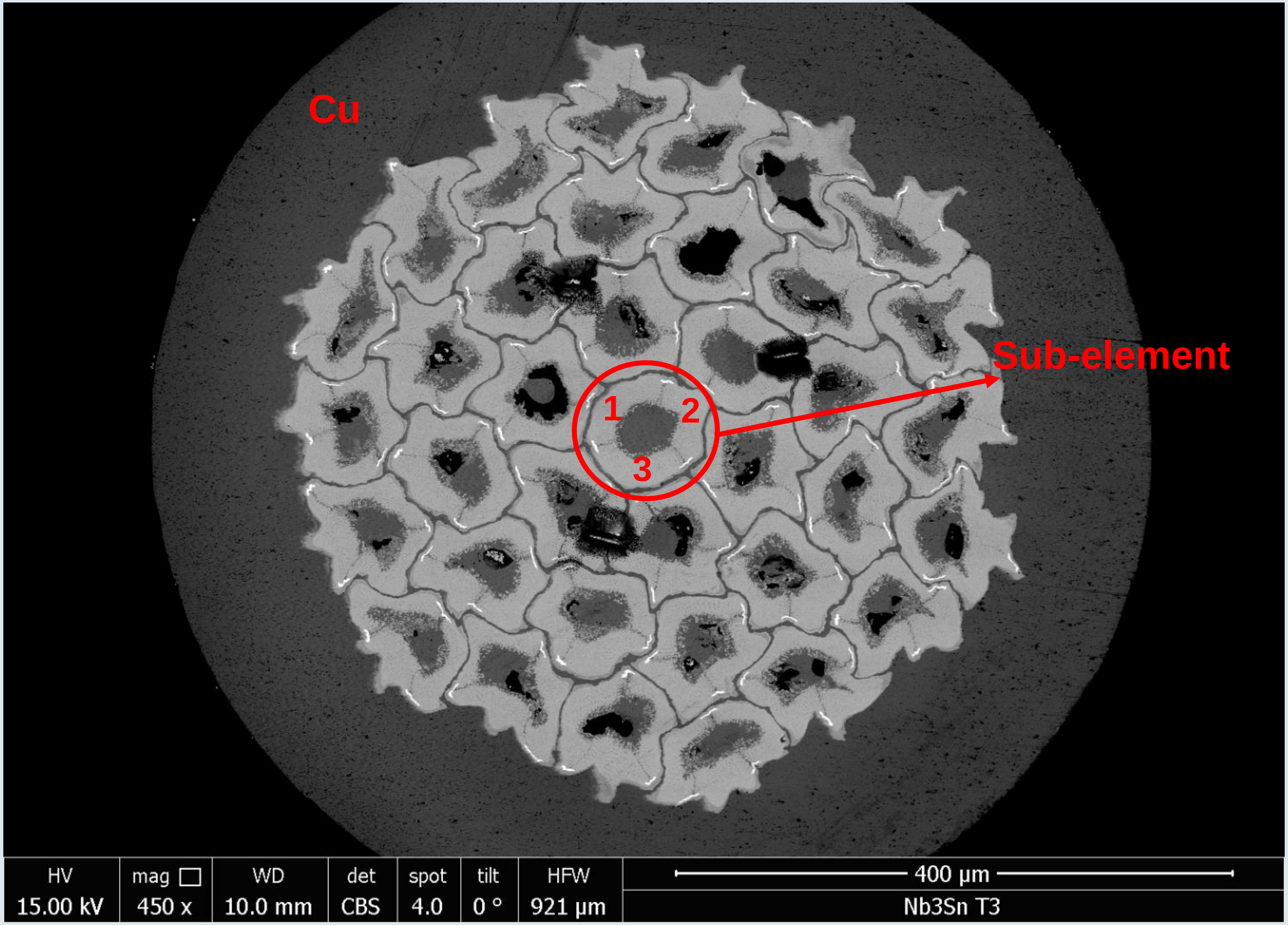
Internal Tin (IT) wire overview



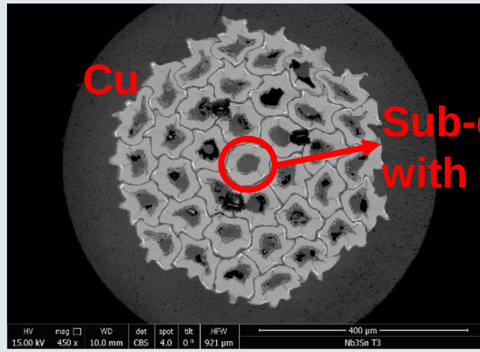
After heat treatment and diffusion process activation



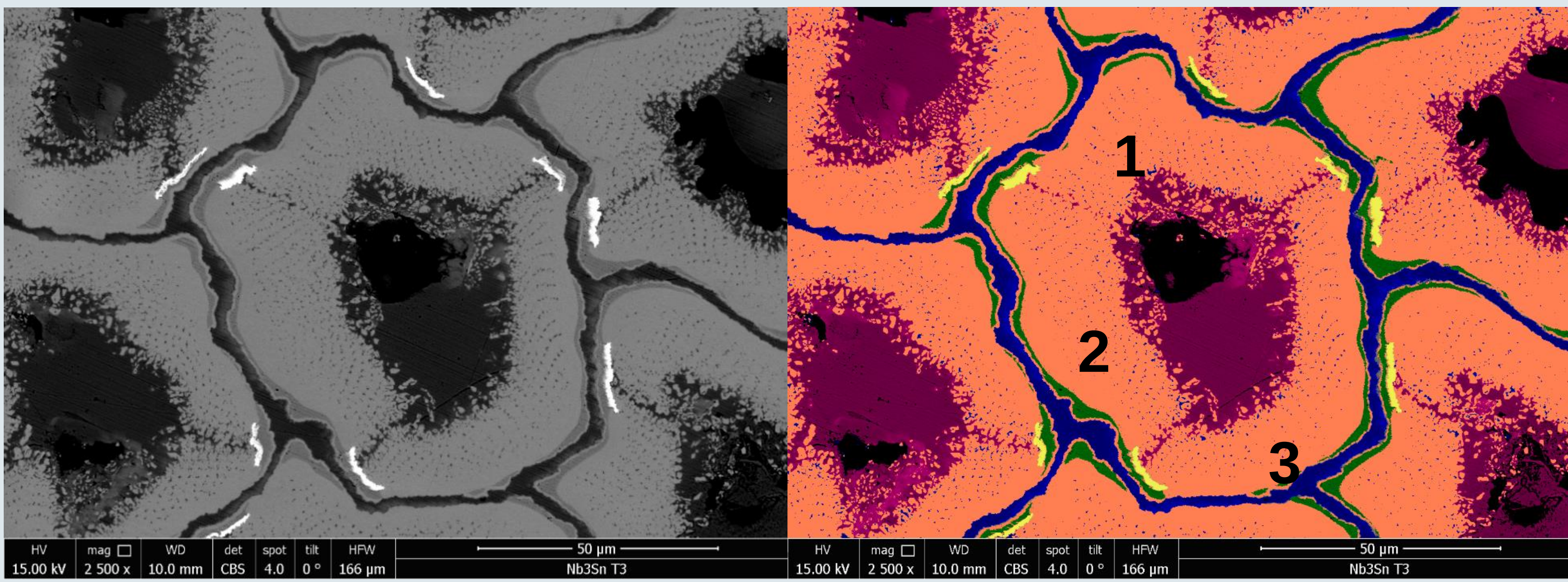
“Clusters” in Nb_3Sn wires



Distributed Nb + Ta

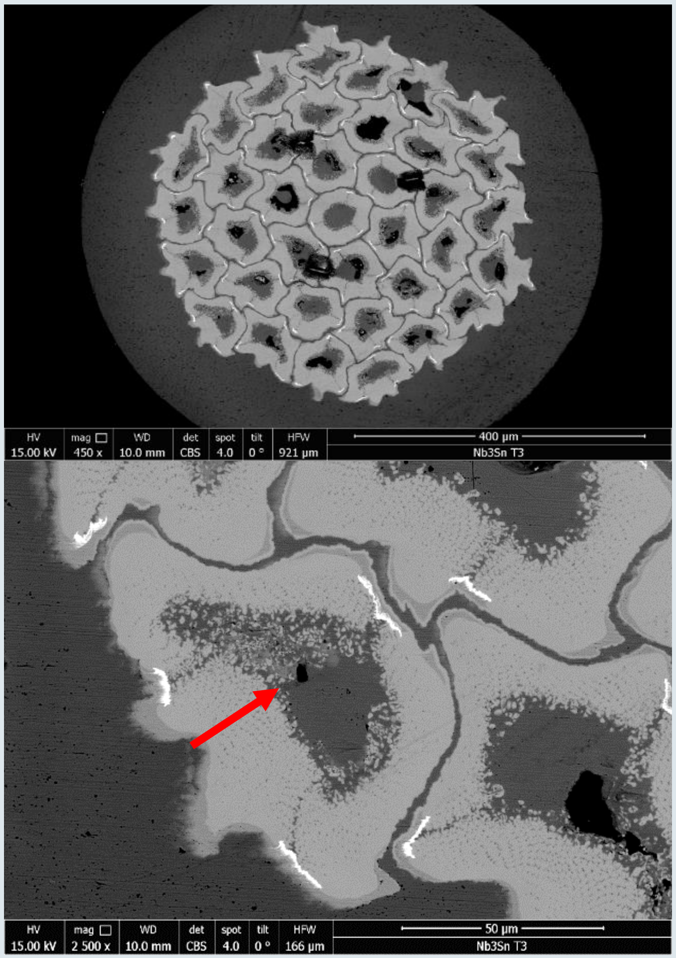


- Nb_3Sn
- Nb
- Cu
- Cu-Sn
- Ta

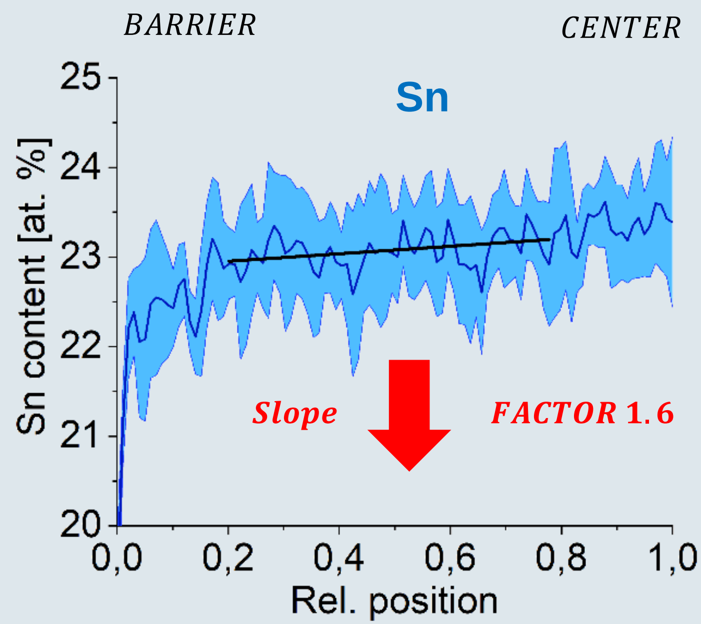


SEM EDX - Homogeneity investigation

- SEM-EDX line scans over different sub-elements along the radial direction → Sn gradient statistical analysis

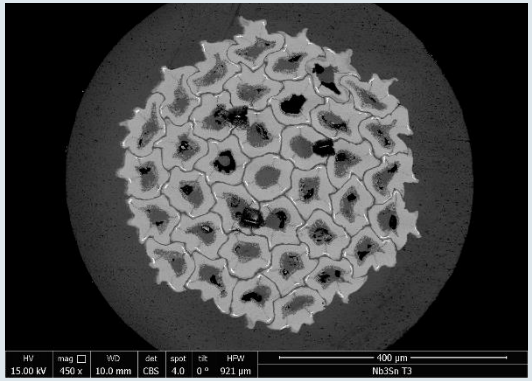


Sub-elements radial direction



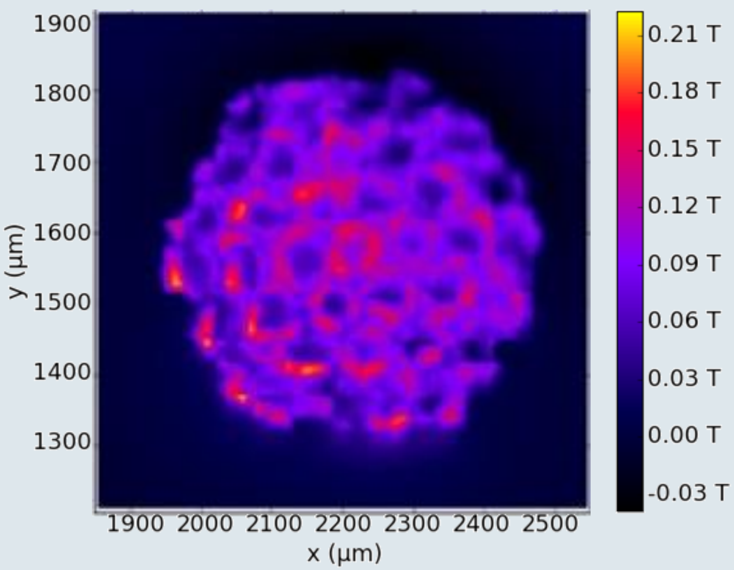
Gradient:
(0.015 ± 0.004) at. %/µm

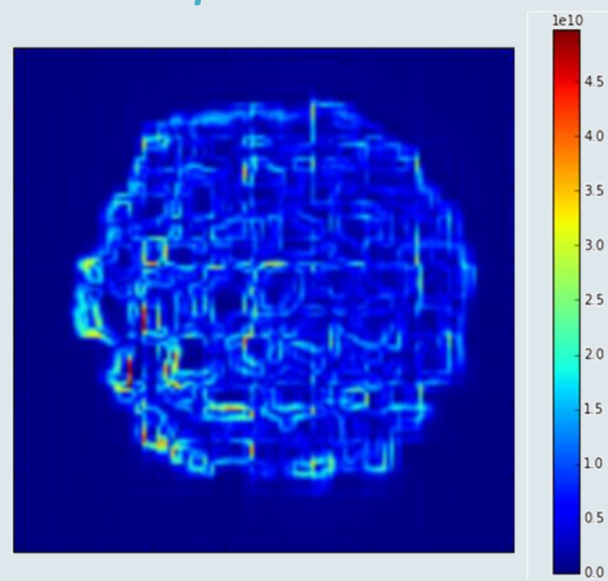
Preliminary J_C Measurements



Cluster layout

Scanning Hall Probe Microscopy (SHPM) – local transport measurements



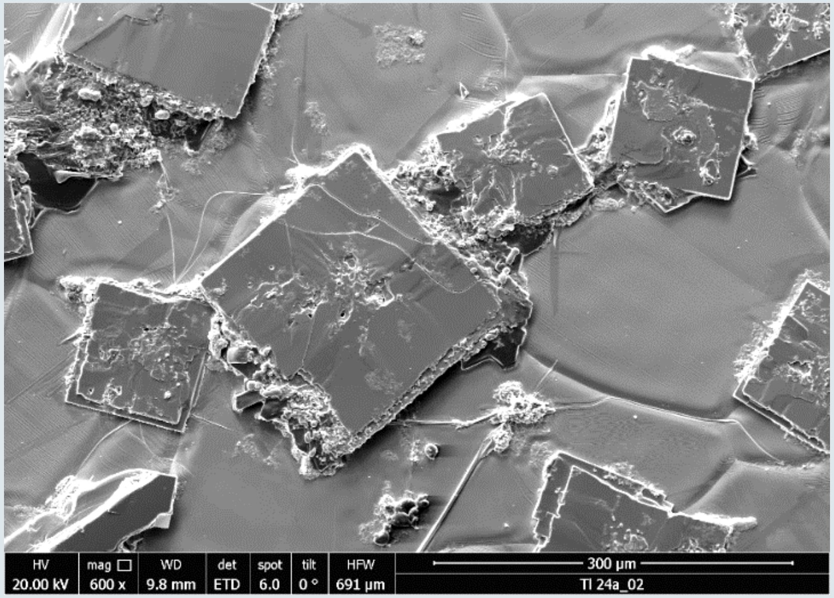


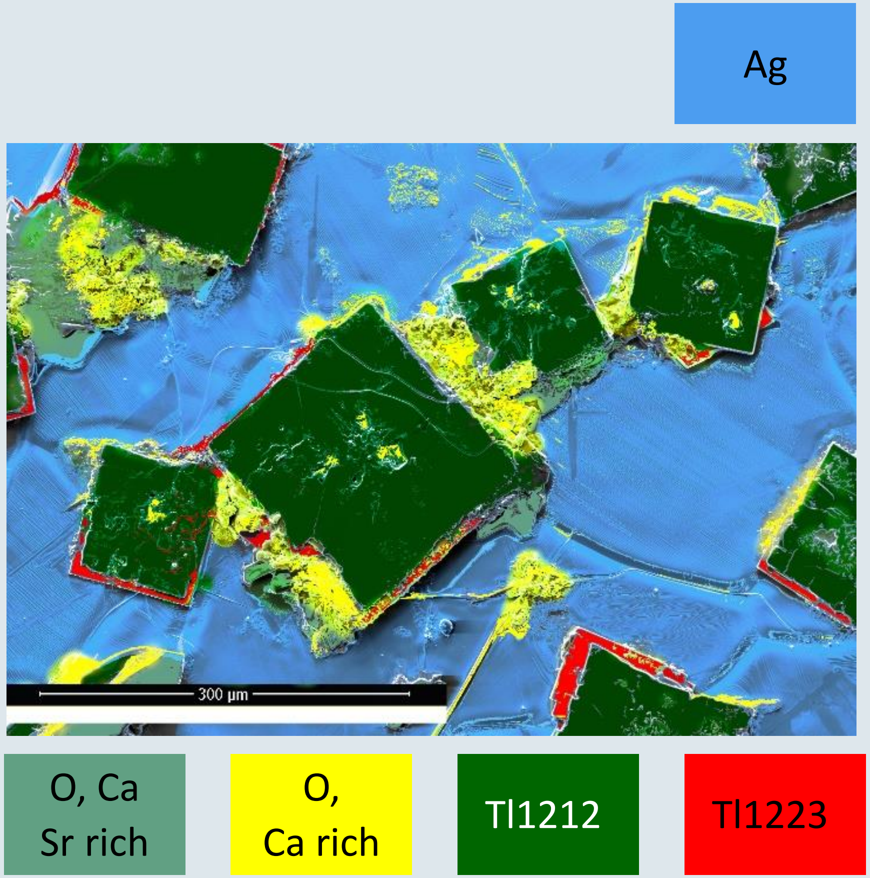
Remanent-field scans used for local current evaluation

Local J_C (A/m^2) values in line with the state-of-art wires (@ 10K, 0 T)

Tl1223 thin films from CNR SPIN (Genoa-Italy)

- Ag Precursor film + Tl_2O_3 powder in a gold capsule
- Heat treatment: 885 °C/ 10 min



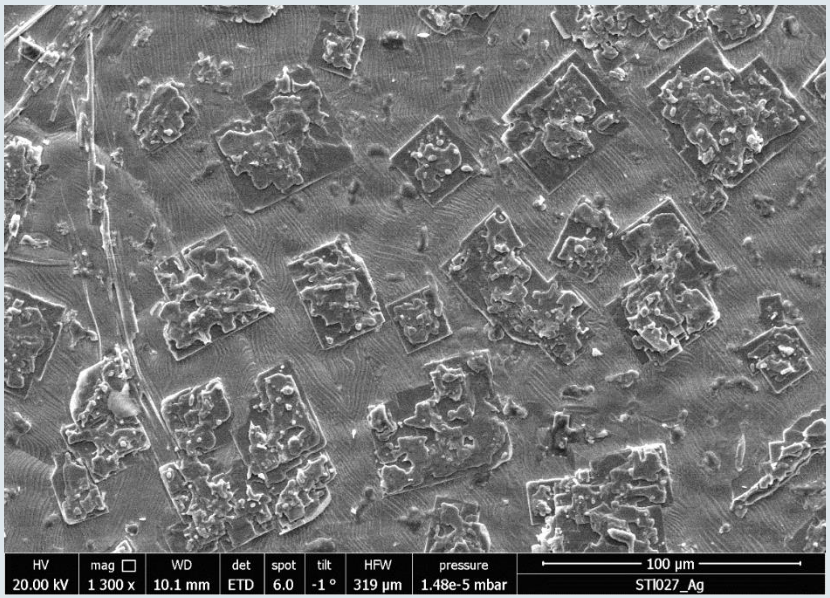


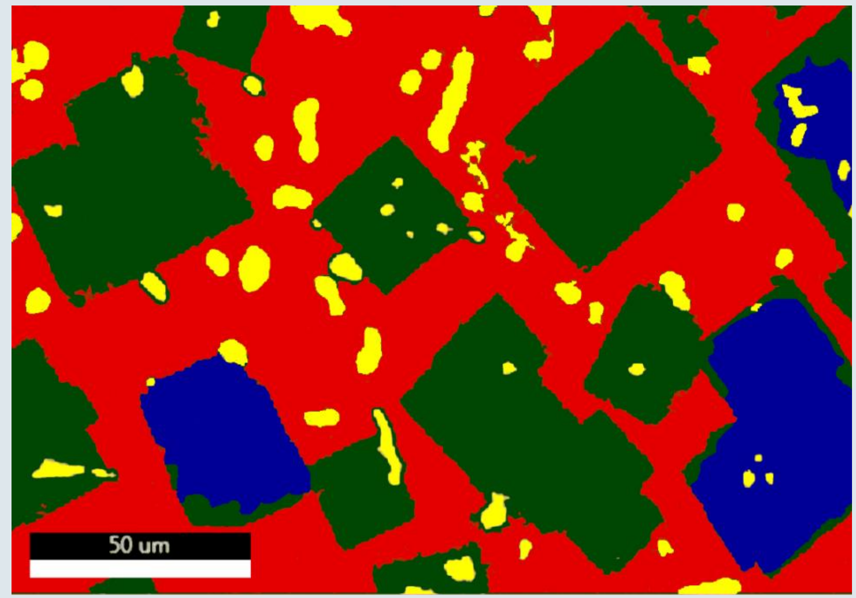
- Plate-like grains
- Large grains → 200µm
- Tl1212 more than Tl1223

Tl1223 thin films from CNR SPIN (Genoa-Italy)

By changing the powder quantity during the thallination process…

Substrate: Ag





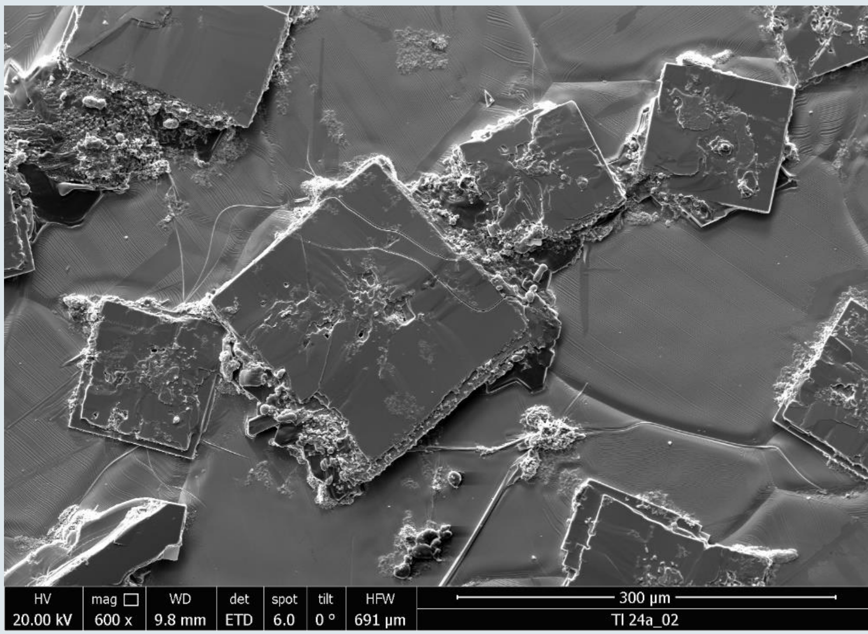
Ag	Tl-1223	O, Ca, Pb rich	Tl-1212

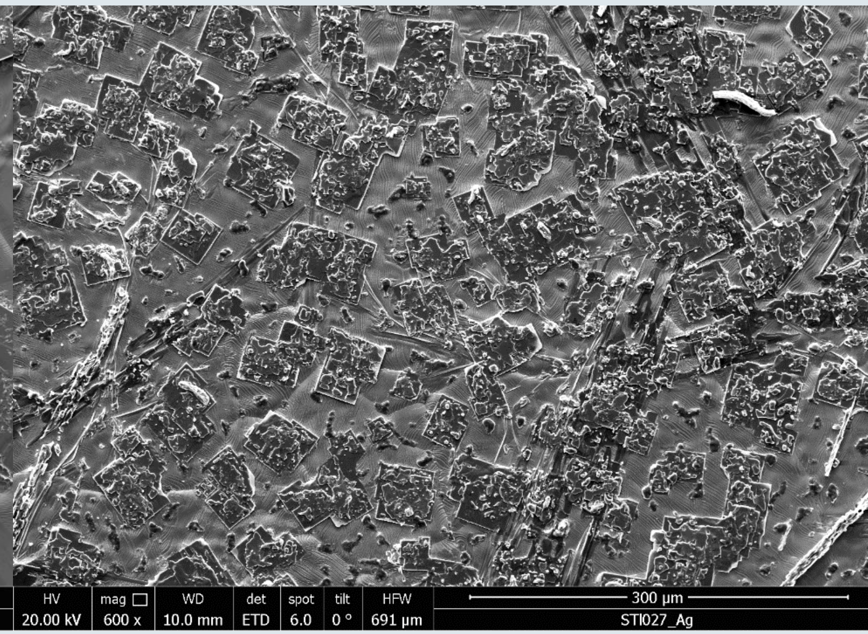
More Tl1223 than Tl1212 ✓

A Leveratto et al 2020 Supercond. Sci. Technol. 33 054004

Tl1223 thin films from CNR SPIN (Genoa-Italy)

Better coverage obtained ✓





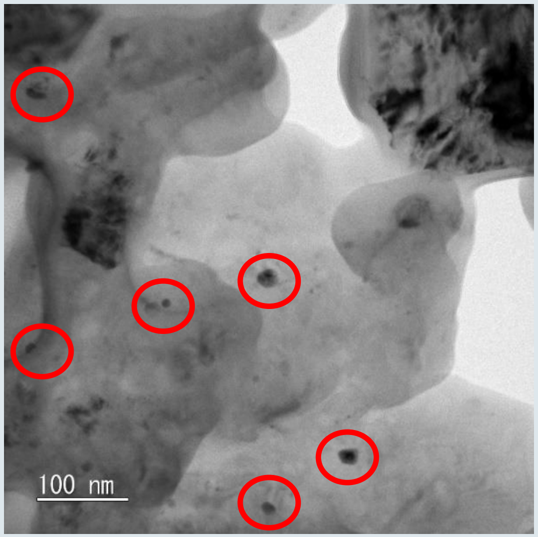
- Sample with big grains
- Ag substrate visible
- Not so good coverage

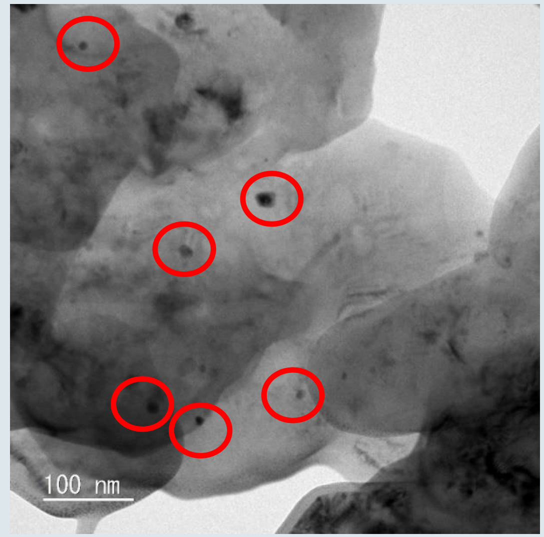
- Large number of plate-like grains
- Better coverage, substrate is less visible
- Better shape of 1223 grains

C-doped MgB_2 wires

C sources: Hemoglobin, Inulin, C-soot…

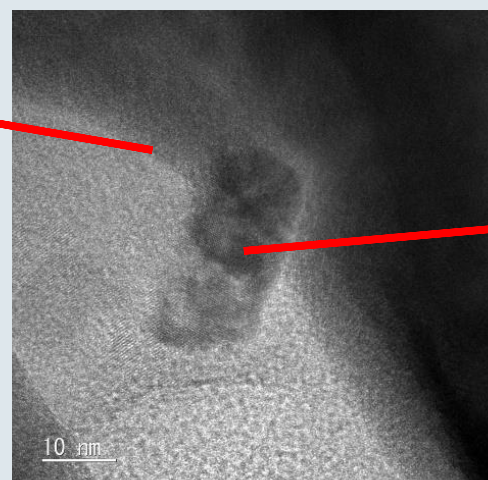
Bright Field TEM images





C clusters → *Pinning centres*

Grain boundary

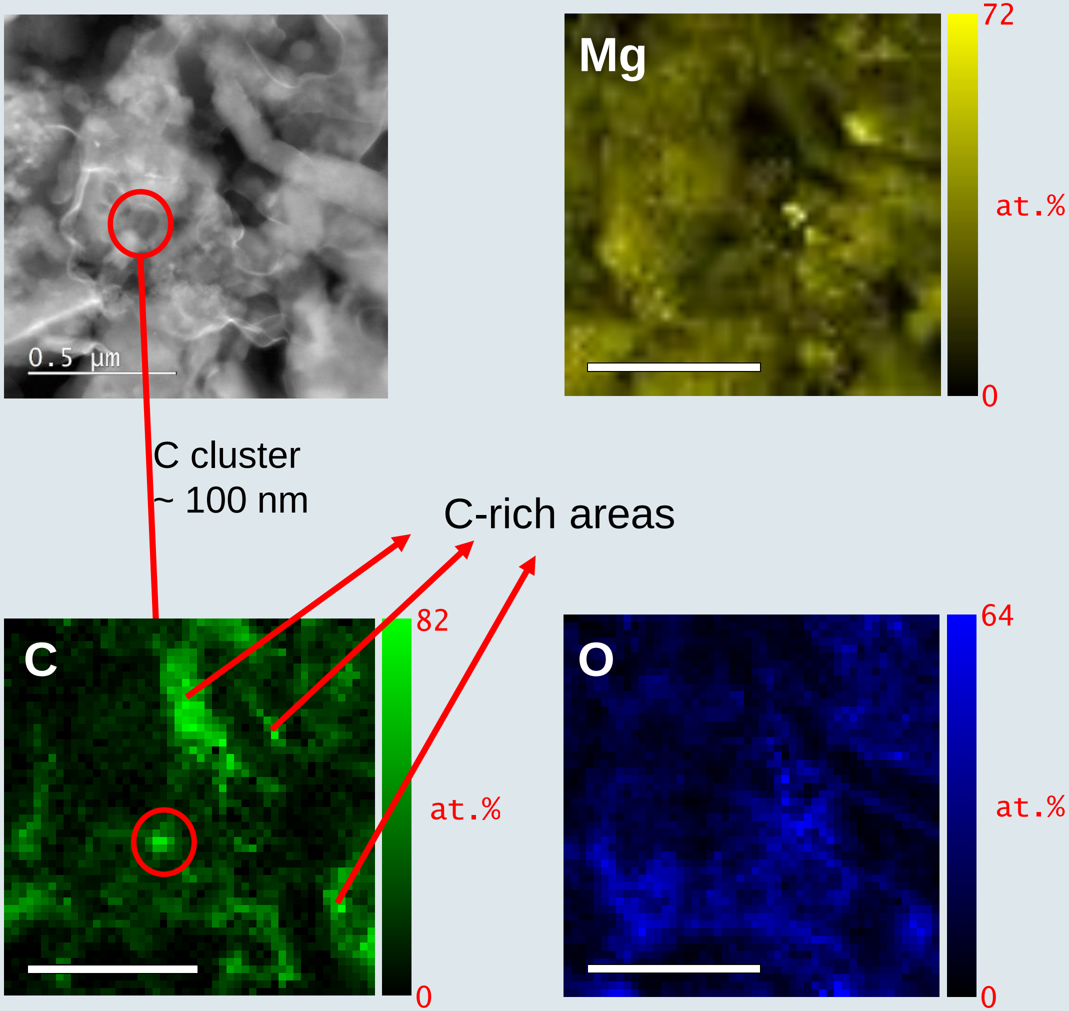


C cluster

HRTEM – High Resolution TEM images

C-doped MgB_2 wires

STEM EDX Map



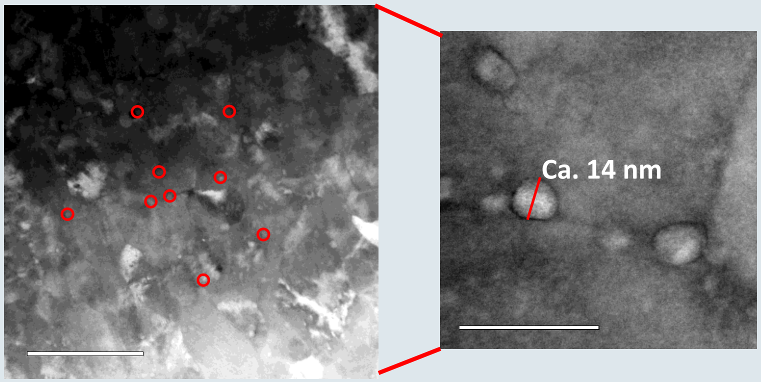
Capra et al "Method for the production of pure and C-doped nanoboron powders tailored for superconductive applications"

Article reference: NANO-126497.R1 – formally accepted by Nanotechnology

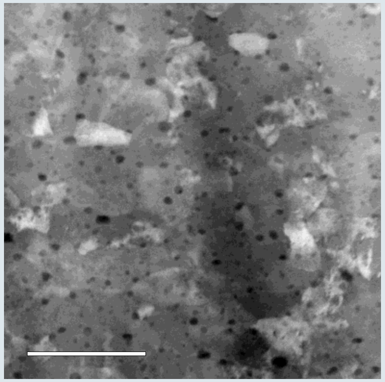
Artificial Pinning Centres Nb_3Sn

STEM images

HfO_2 particles

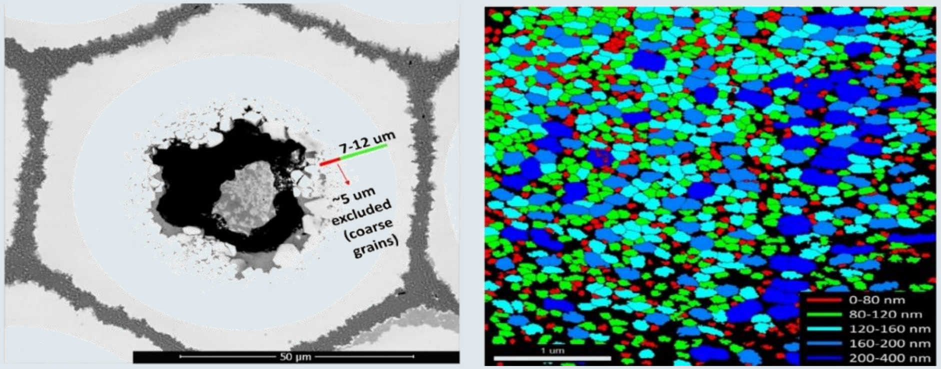


ZrO_2 particles (black spots)

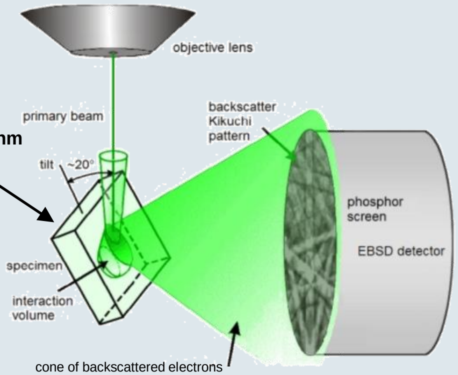


- PP density
- PP size

Grain size evaluation → **E**lectron **B**ack**s**catter **D**iffraction – **EBSD** in transmission mode

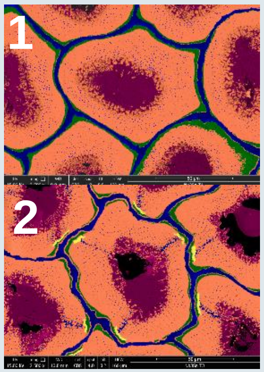


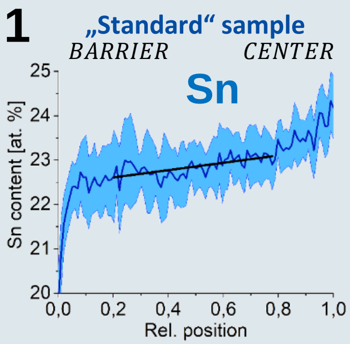
Courtesy of S. Pfeiffer (TU Wien - USTEM)

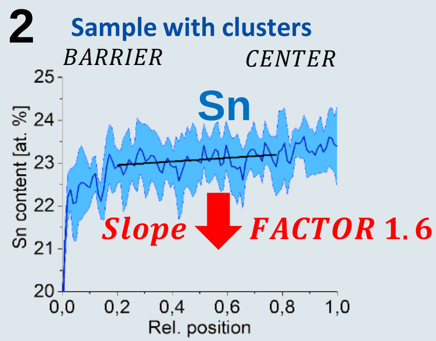


Final remarks

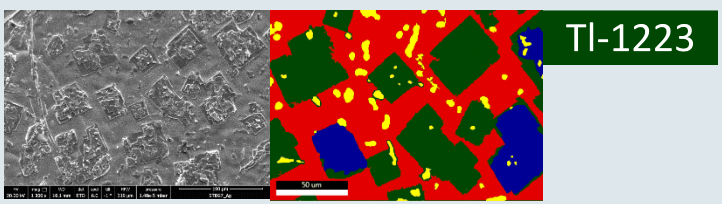
Electron Microscopy  Superconductivity



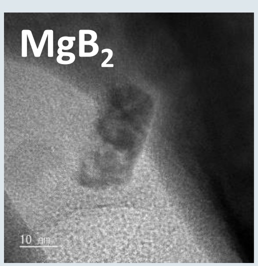


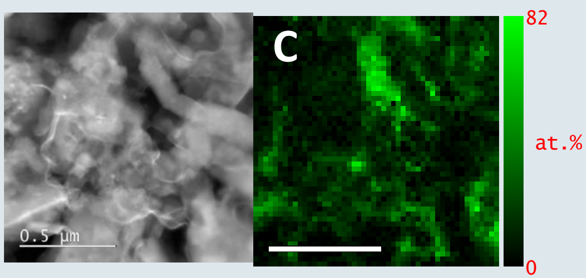


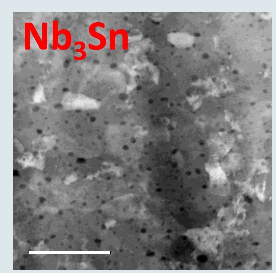
Validation of innovative manufacturing processes



Optimization of the production process parameters







Help analyzing the pinning centres behavior

The End

Thank you for the attention!

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