



# FIB nanotomography examination of superconducting wires

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# Motivation

- The presence of porosity in superconductors produced by powder-in-tube (PIT) technology is unavoidable
- Porosity generally reduces the useful superconductor volume in the composite
- If porosity is distributed inside the superconducting phase, like in Bi-2223, Bi-2212 and  $\text{MgB}_2$ , it can block the supercurrent.

A better understanding of the porosity formation and redistribution can help to optimise the processing procedures in order to increase the critical current density.

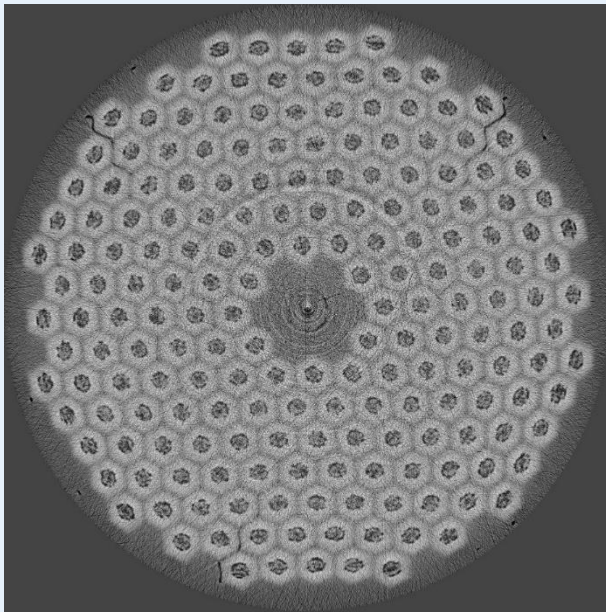


## X-ray tomography

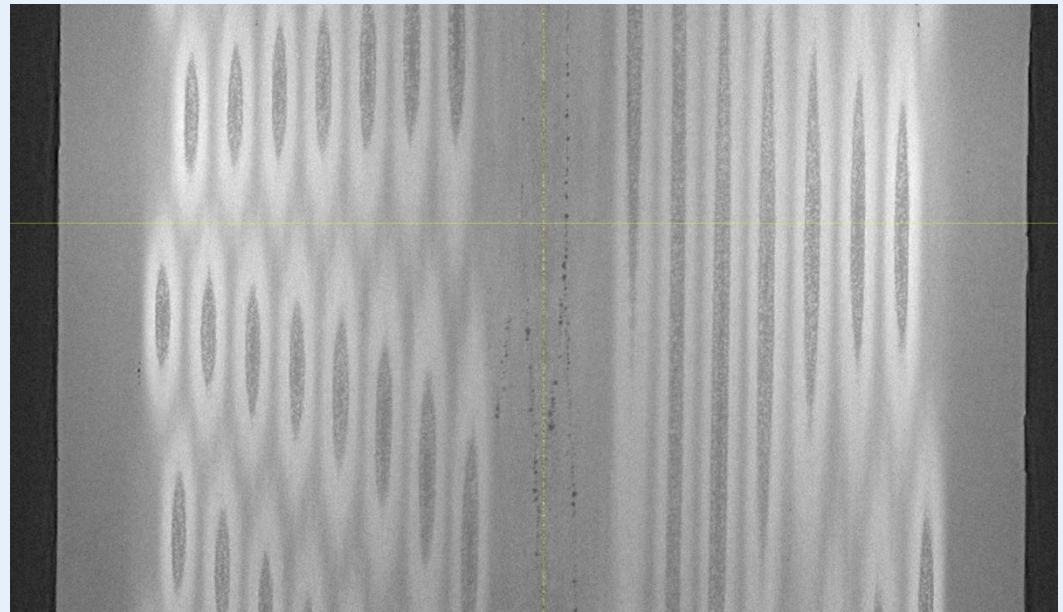
# $\mu$ -CT of a PIT-type $\text{Nb}_3\text{Sn}$ wire

The spatial resolution of  $\mu$ -CT is not sufficient to resolve the porosity in a PIT-type  $\text{Nb}_3\text{Sn}$  wire

(a)



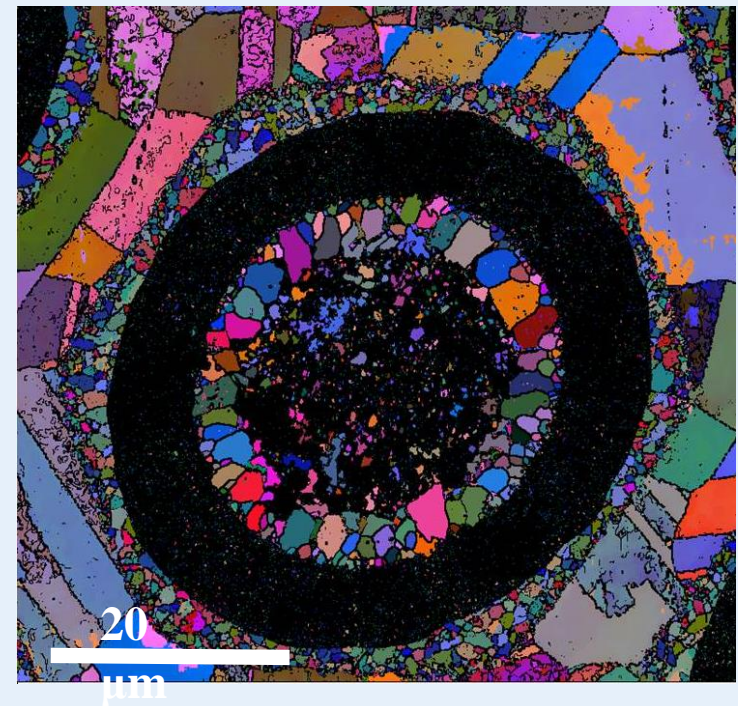
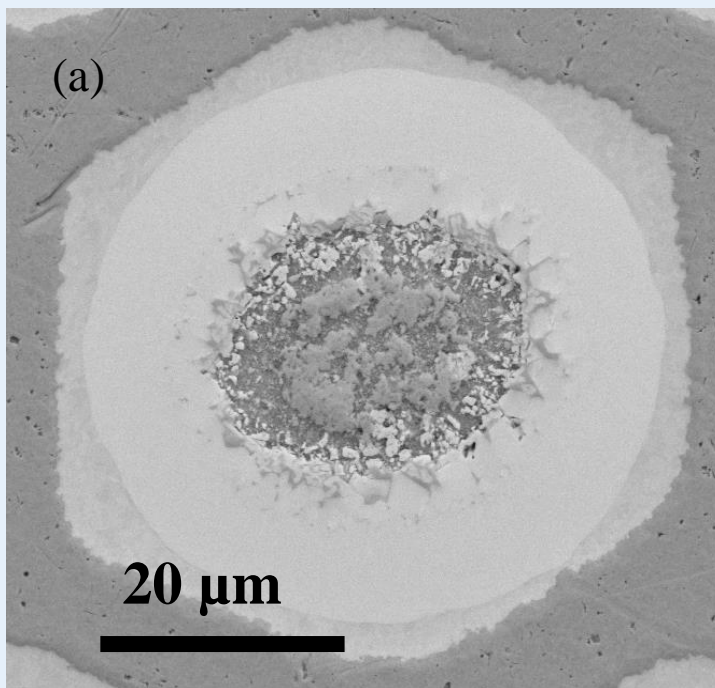
(b)



(a) Transverse and (b) longitudinal  $\mu$ -CT cross section of an entire PIT  $\text{Nb}_3\text{Sn}$  wire ( $\text{Ø}=1.25$  mm). Courtesy A. Rack, ESRF ID19.

## Mechanical Sectioning

# Conventional metallographic cross sections

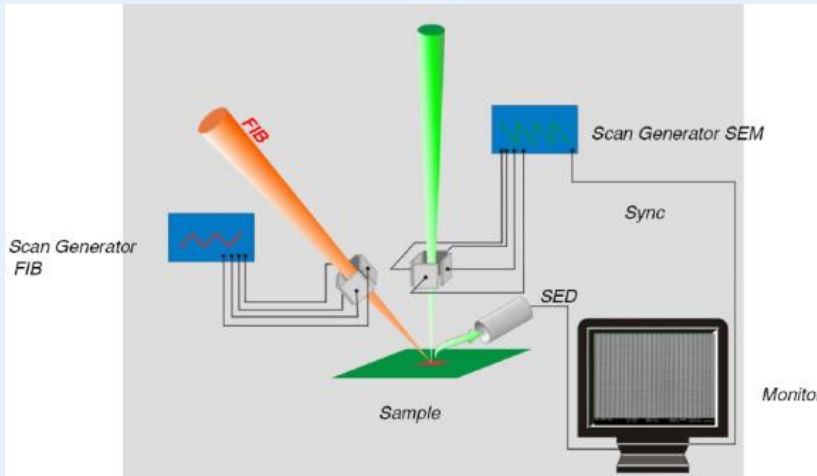


*Metallographic cross sections of a  $\text{Nb}_3\text{Sn}$  filament prepared by mechanical grinding and polishing. (a) Backscatter electron (BE) and (b) Electron Backscatter Diffraction (EBSD) contrast. Courtesy P. Alknes, CERN.*



# SEM/FIB Systems

## Focused Ion Beam



Zeiss Company

Incorporates:

- SEM for non destructive imaging
- FIB for sight specific material removal

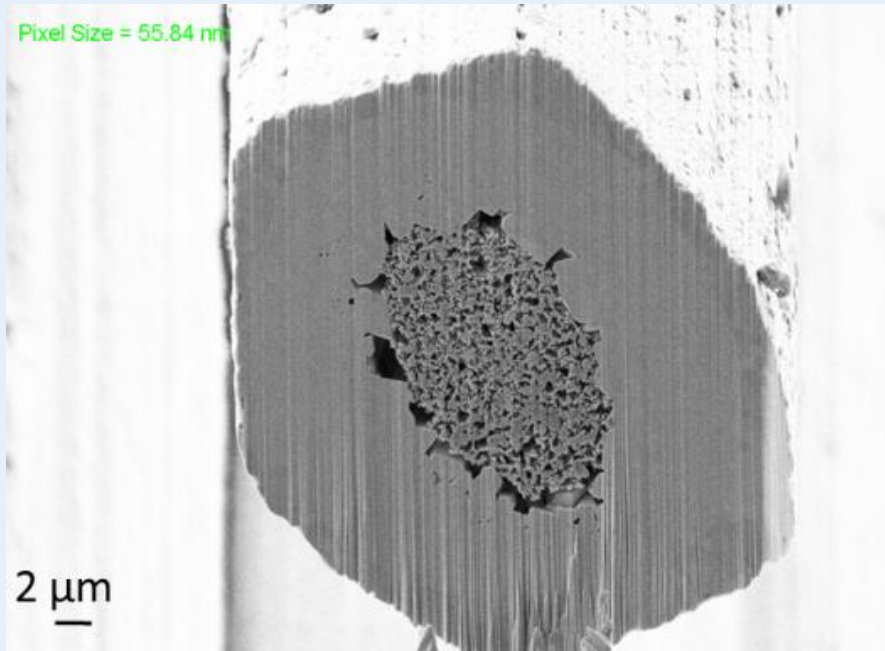


Zeiss Neon EsB

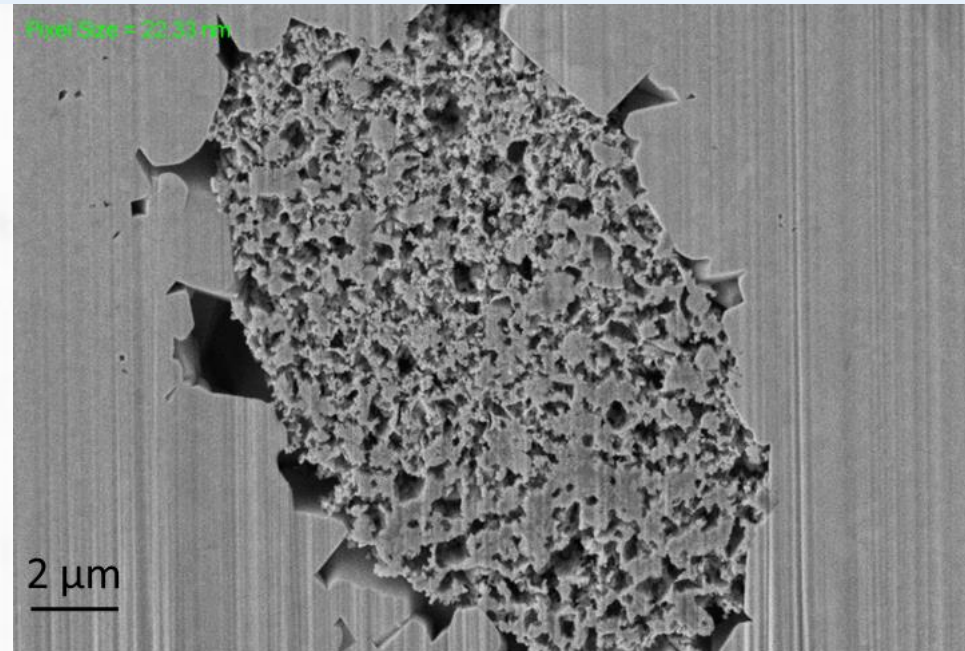


# FIB Cross-Section

(a)



(b)

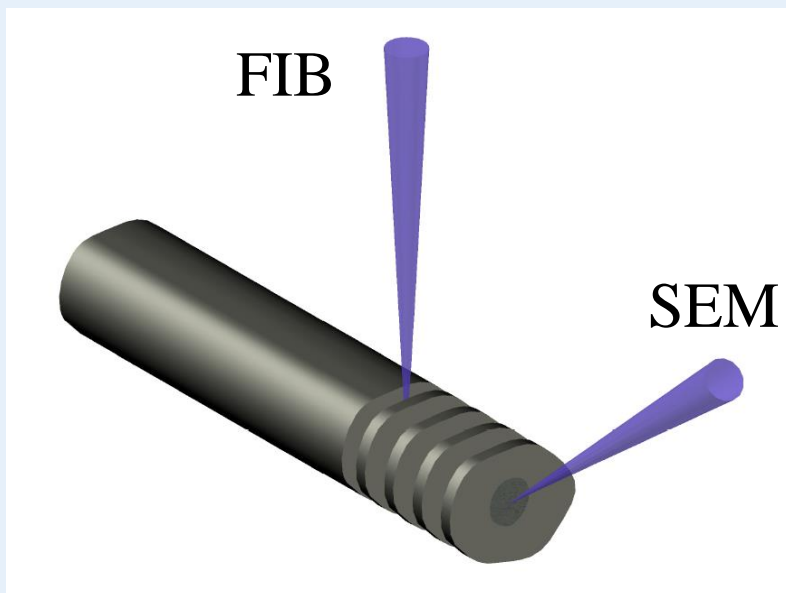


*Secondary electron image of the cross section of an extracted  $Nb_3Sn$  filament prepared by Focused-Ion-Beam milling. The porosity between the coarse grains and inside the filament core is clearly visualised.*



# What is FIB Nanotomography?

## FIB nanotomography



### 3D structural analysis

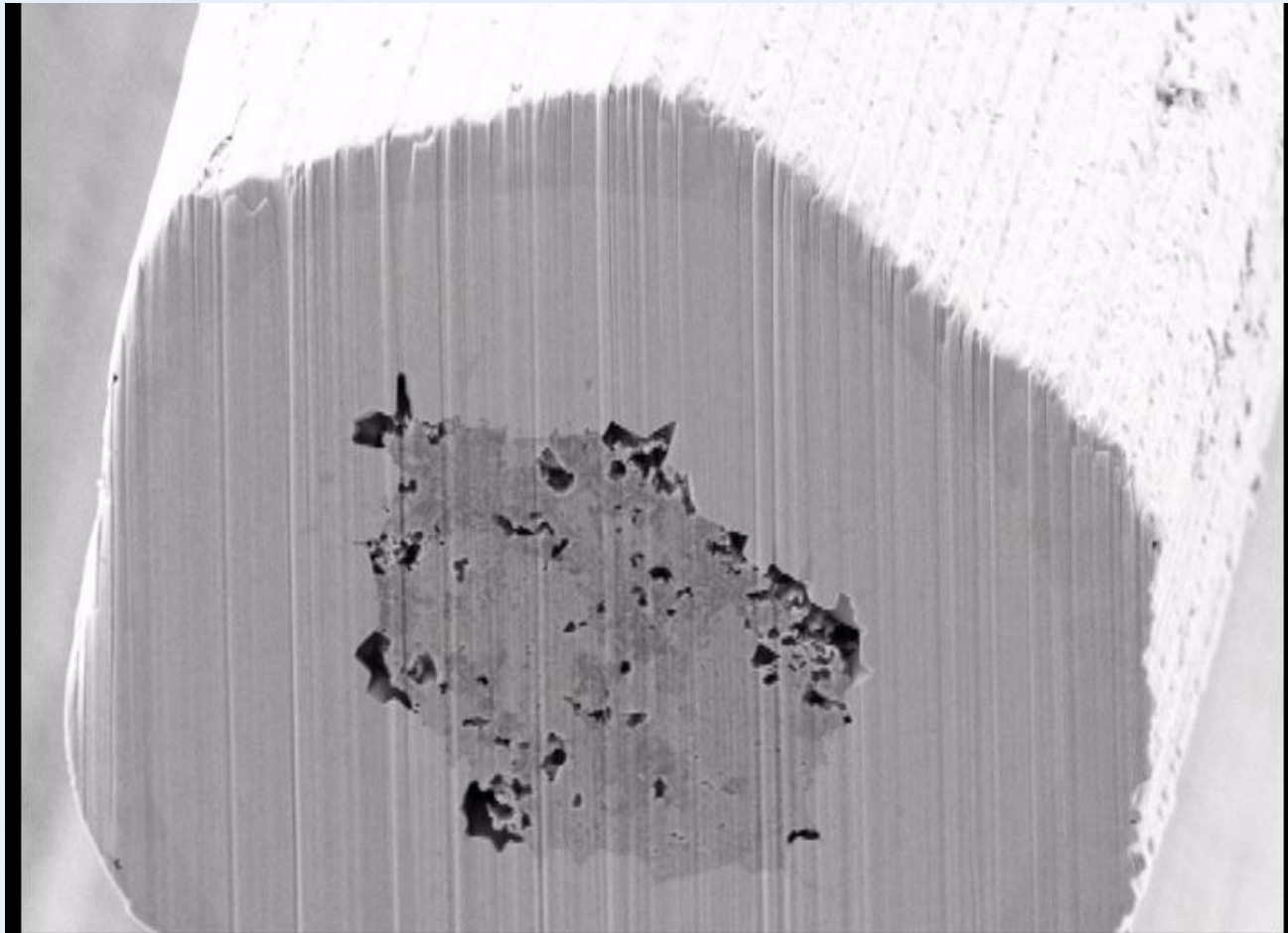
- voxel size down to 10 nm
- higher resolution than X-ray
- larger volume than TEM

FIB tomography fills the gap between classical tomography methods (e.g. X-ray) and high resolution TEM tomography



# What is FIB Nanotomography?

## Image acquisition

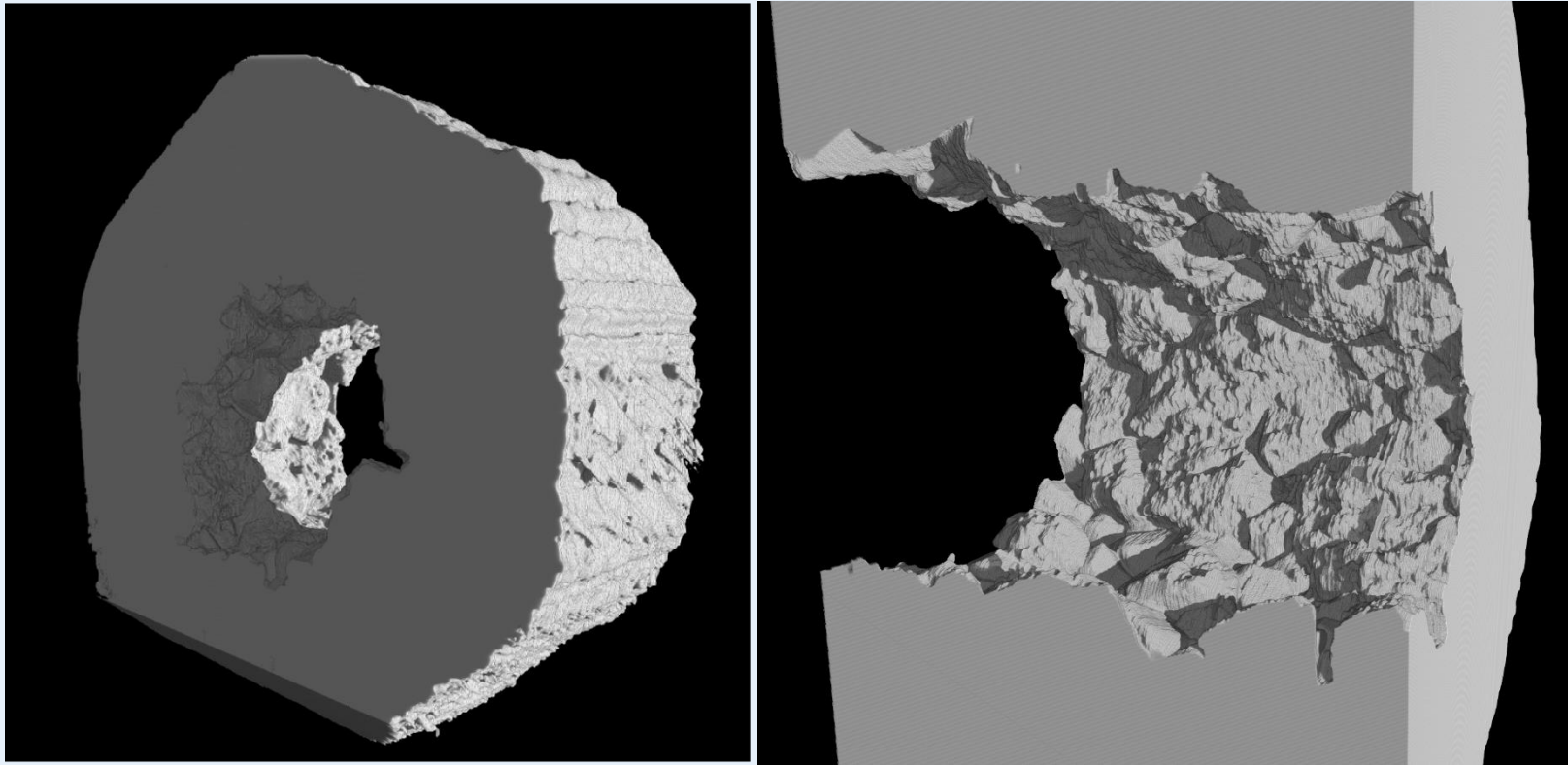






## What is FIB Nanotomography?

### 3D rendering of the coarse $\text{Nb}_3\text{Sn}$ grains

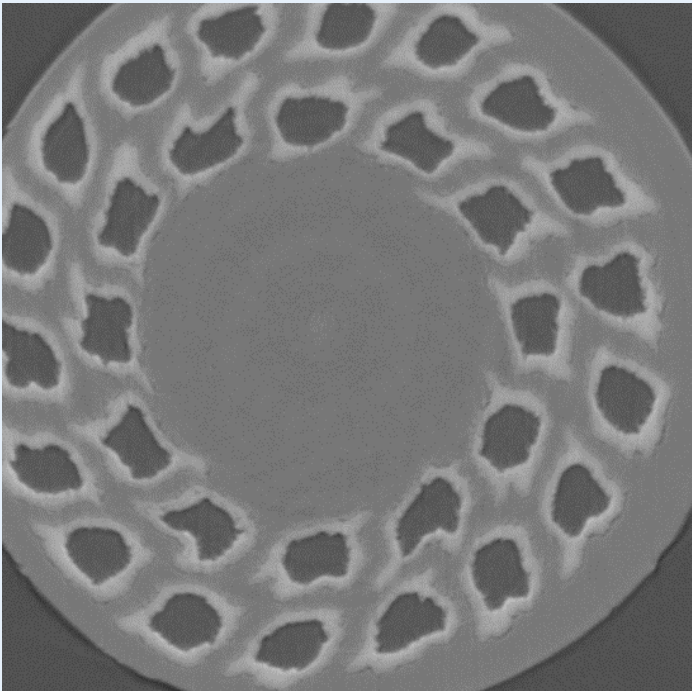


*3D rendering of a  $30\mu\text{m}$  long  $\text{Nb}_3\text{Sn}$  PIT filament. The inner core has been removed in order to expose the coarse  $\text{Nb}_3\text{Sn}$  grain surface.*



# FIB nanotomography $\text{MgB}_2$

## $\mu$ -CT of a $\text{MgB}_2$ wire

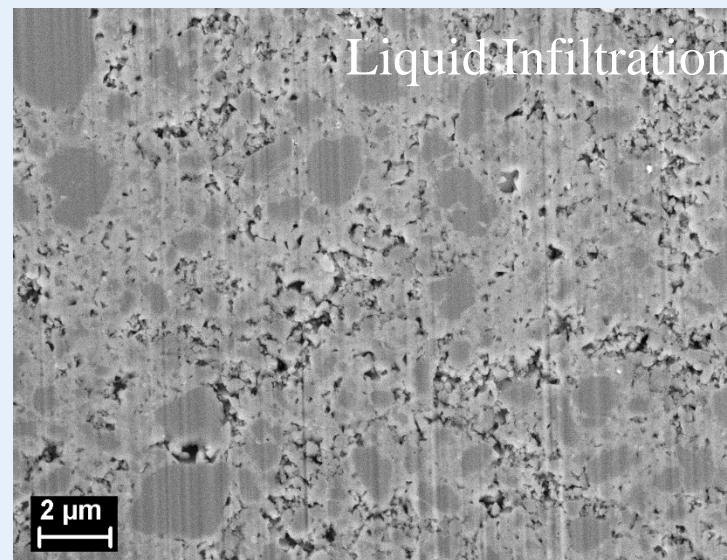
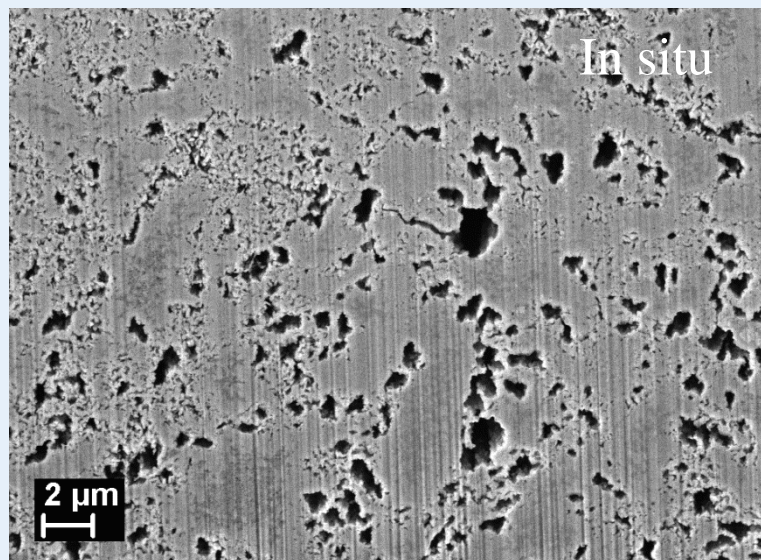
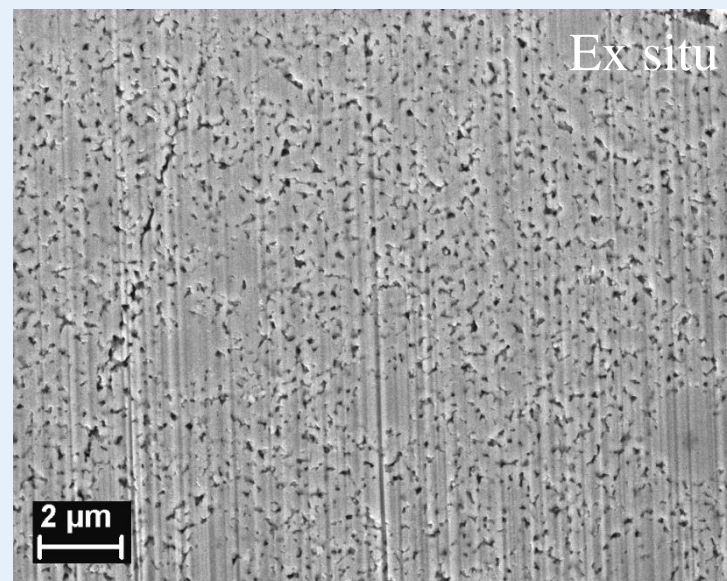
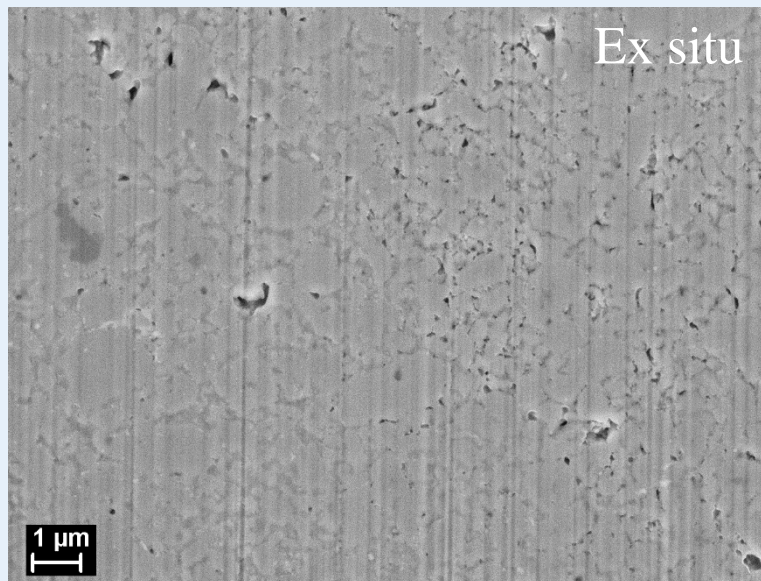


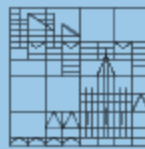
$\mu$ -CT cross sections of an ex situ  $\text{MgB}_2$  wire.

Main problems in  $\mu$ -CT of  $\text{MgB}_2$  wires are the very low x-ray attenuation by the weakly absorbing B and Mg inside a strongly absorbing matrix, and the small size of the porosity inside the  $\text{MgB}_2$  filaments.



# FIB nanotomography $\text{MgB}_2$





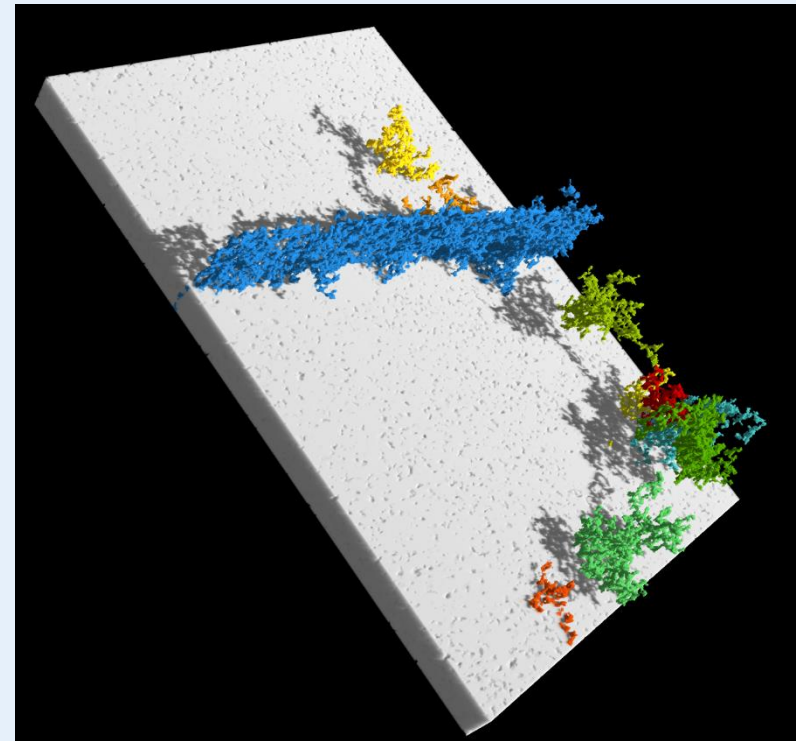
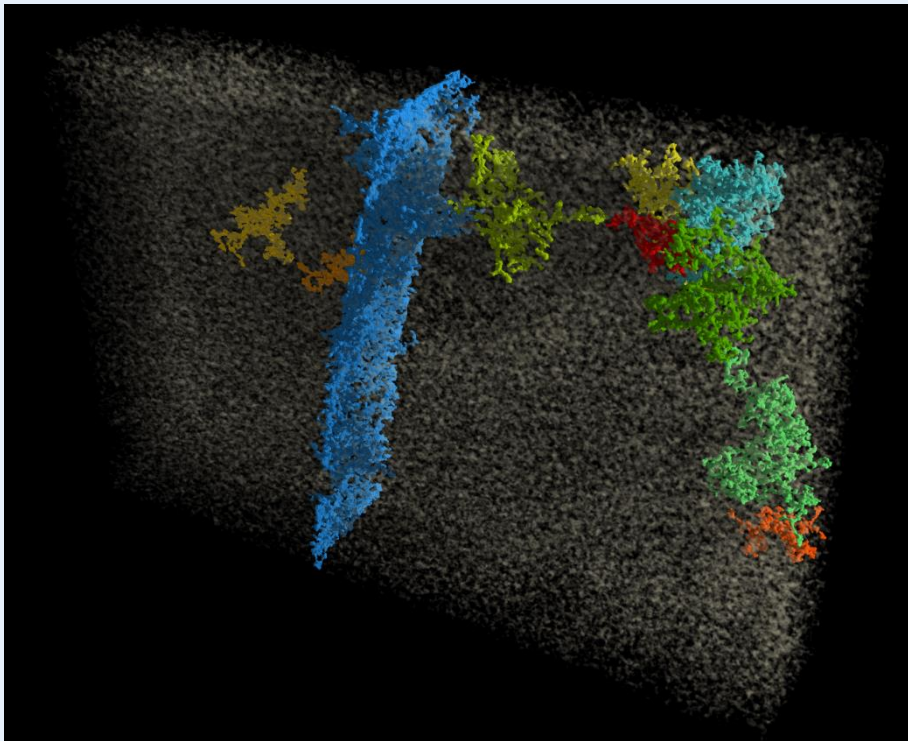
# FIB nanotomography $\text{MgB}_2$





# FIB nanotomography $\text{MgB}_2$

## Pore connectivity



The 10 largest connected pore-networks are highlighted in colour



## Conclusion & Outlook

### Conclusion:

- ✓ Porosity is clearly visualised with SEM after FIB preparation
- ✓ Large volumes can be studied with high resolution

### Outlook:

- Quantitative analysis of pore size and distribution
- Comparison with experimental data about superconducting and mechanical properties



# Acknowledgement

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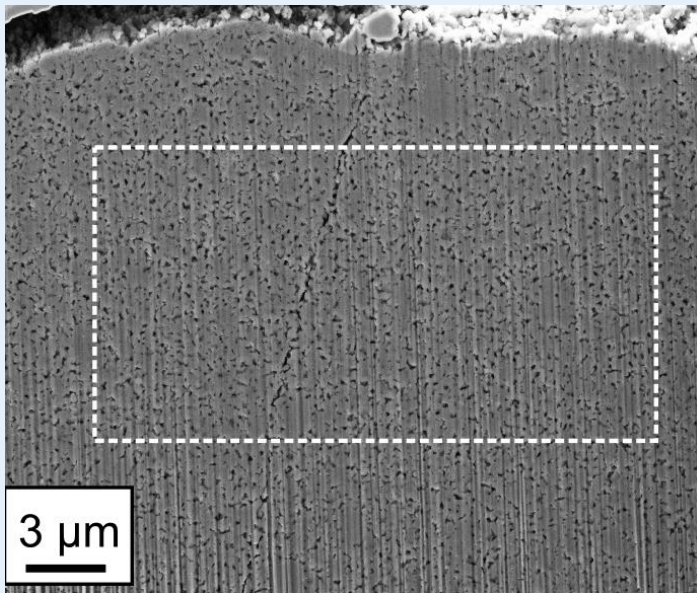
Amalia Ballarino



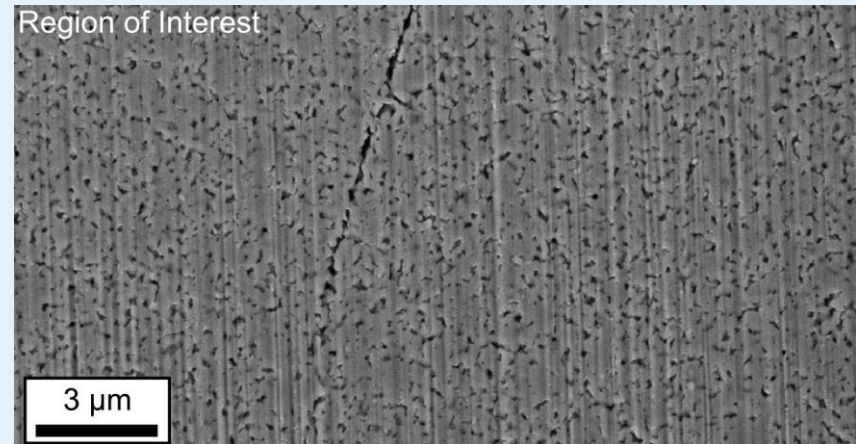
# FIB nanotomography $\text{MgB}_2$

## Workflow of the Reconstruction

1.



2.



3.

