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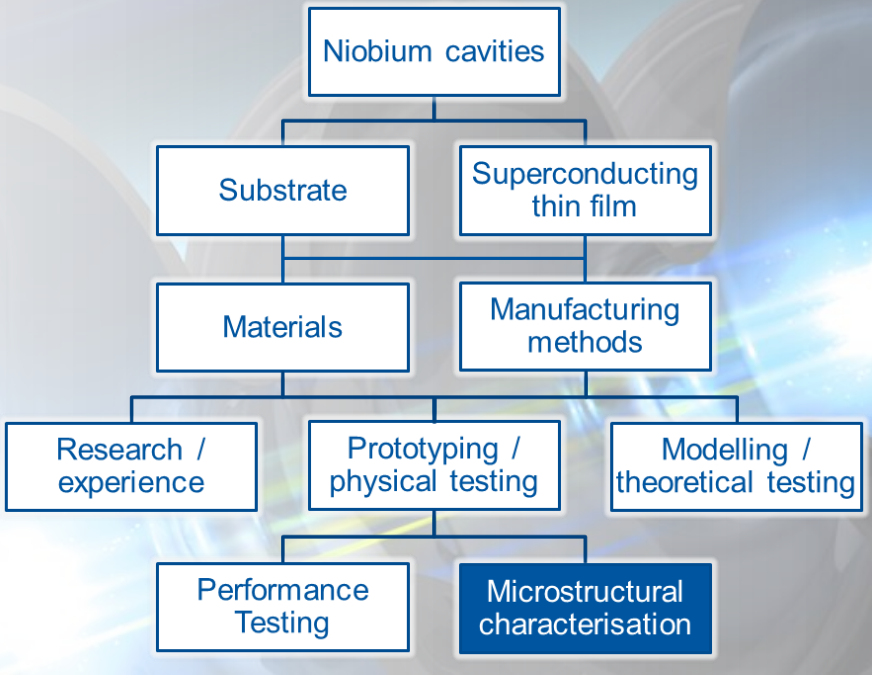
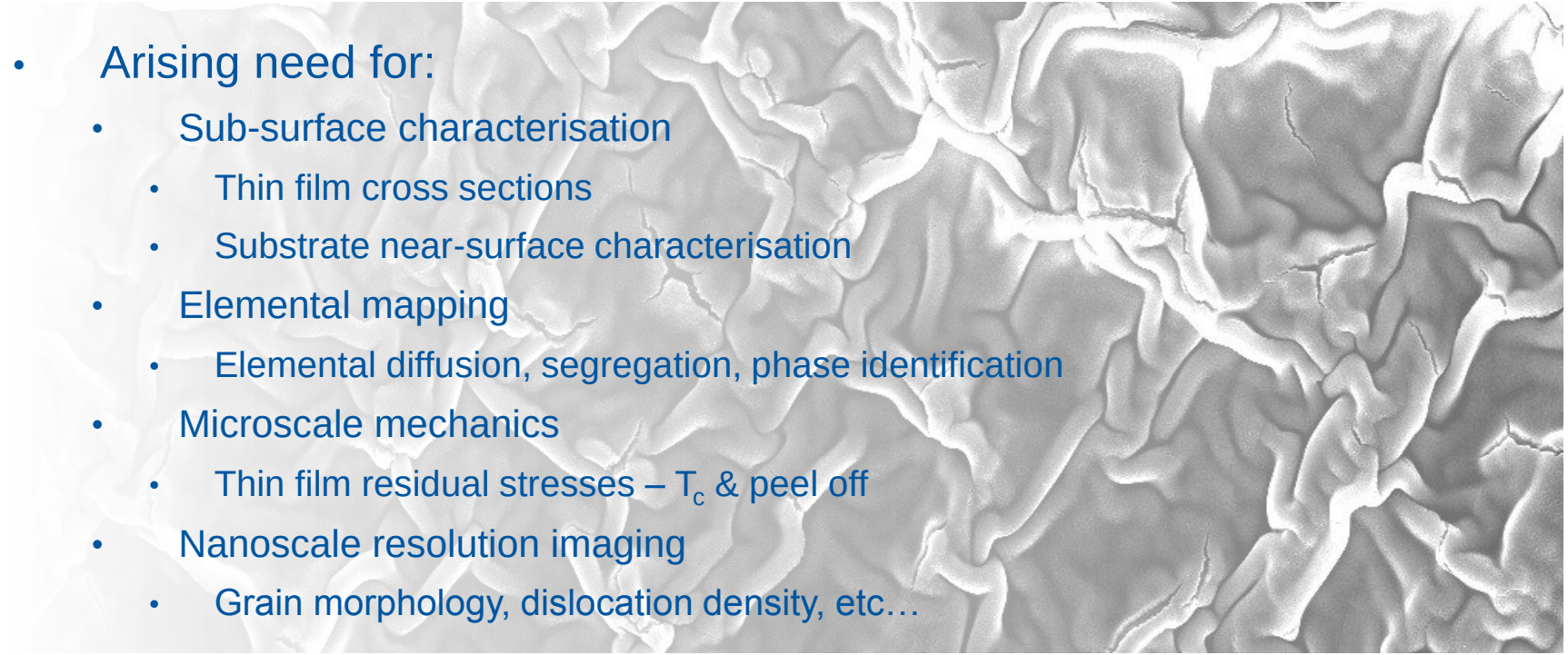
Micro-to-nanoscale characterisation of SRF cavity coatings for the FCC using advanced FIB microscopy

Alexander Lunt

SRF Development



Microstructural characterisation



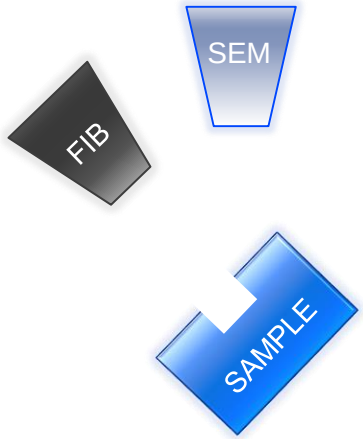
- Arising need for:
  - Sub-surface characterisation
    - Thin film cross sections
    - Substrate near-surface characterisation
  - Elemental mapping
    - Elemental diffusion, segregation, phase identification
  - Microscale mechanics
    - Thin film residual stresses – T_c & peel off
  - Nanoscale resolution imaging
    - Grain morphology, dislocation density, etc…

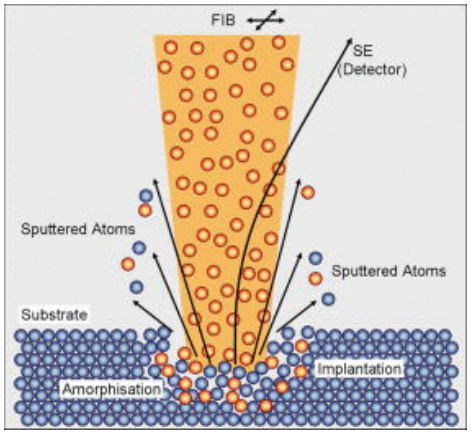
Focused ion beam (FIB) analysis

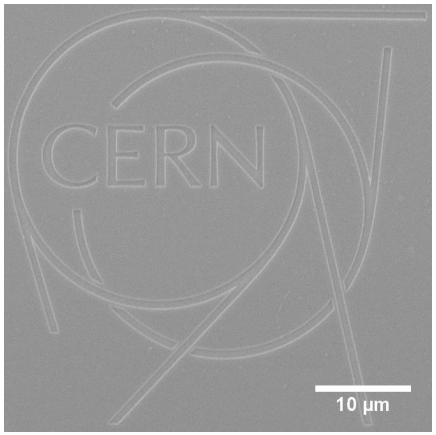
- Anticipated arising need
- 2nd May 2016
- Zeiss XB540 FIB-SEM
  - Sub nanometre resolution
  - Ga^+ with Pt and C deposition
  - Energy Dispersive Spectroscopy (EDS)
  - Atlas 5
  - Scanning Transmission Electron Microscopy (STEM) detector

What is FIB analysis?

- Combined with Scanning Electron Microscope
- Accelerate and focus a beam of ions

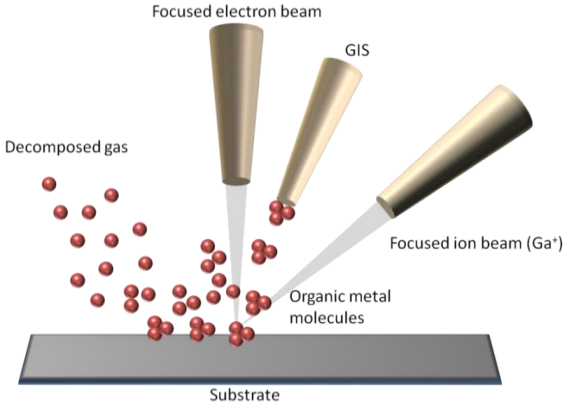


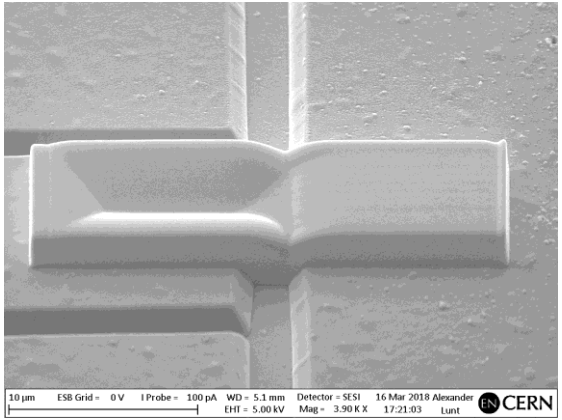




FIB deposition

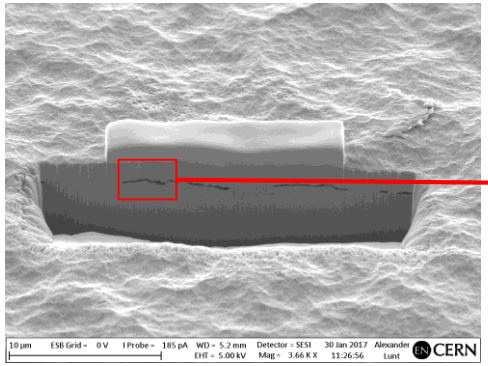
- Interaction between precursor and ion beam
- Atomic species of interest deposited

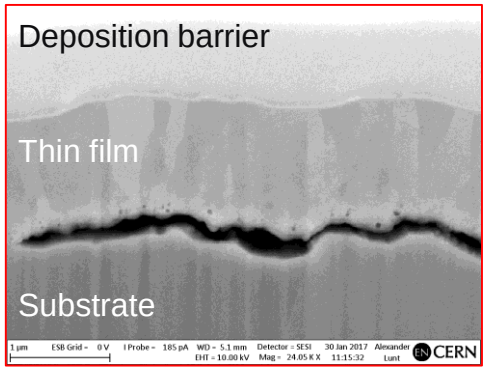


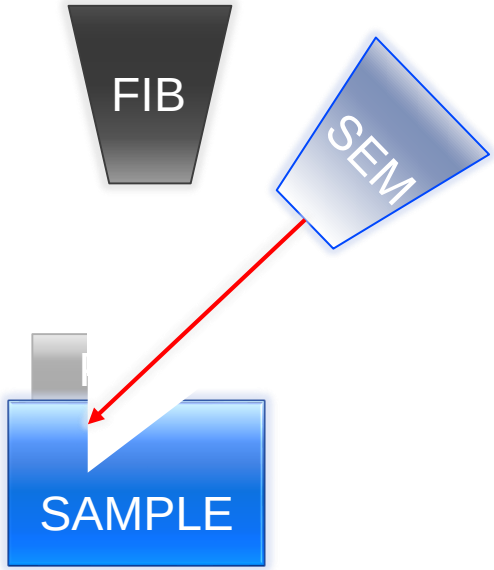


FIB cross sectional milling

- Pt deposition barrier – protect surface
- FIB milling to produce cross section
- Tilt corrected SEM imaging





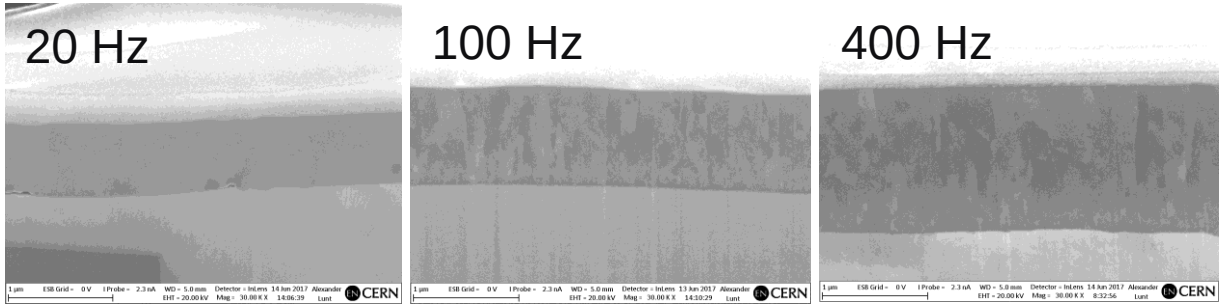


Example Studies

Katsiaryna Ilyina-Brunner 10:48 Berlage zaal (1.9)
Guillaume Rosaz 11:06 Berlage zaal (1.9)

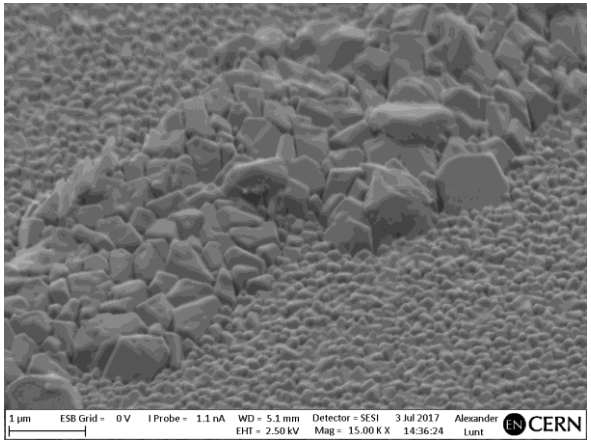
- High-Power Impulse Magnetron Sputtering
  - Pressure, frequency, voltage bias, coating position
- 18 samples studied

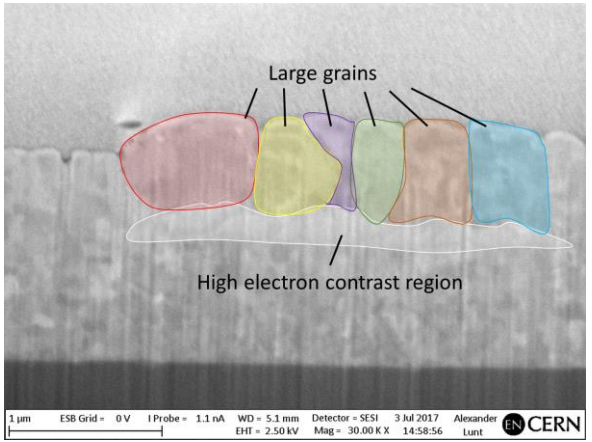
Increased frequency = higher coating rate



Surface contamination

- Cu rich deposits observed
- What is the influence on the microstructure?





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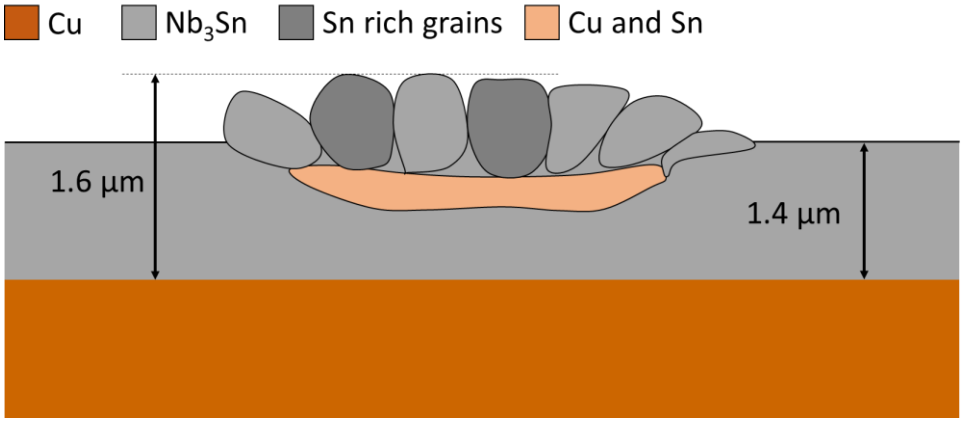
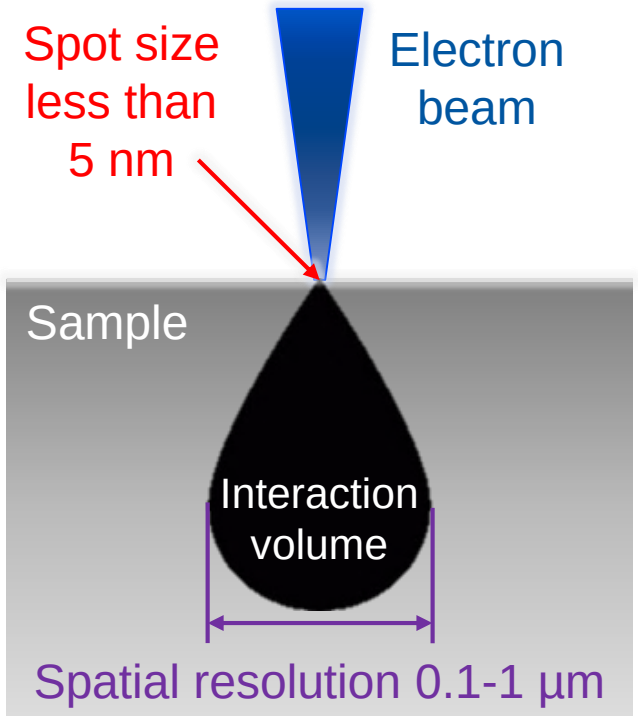


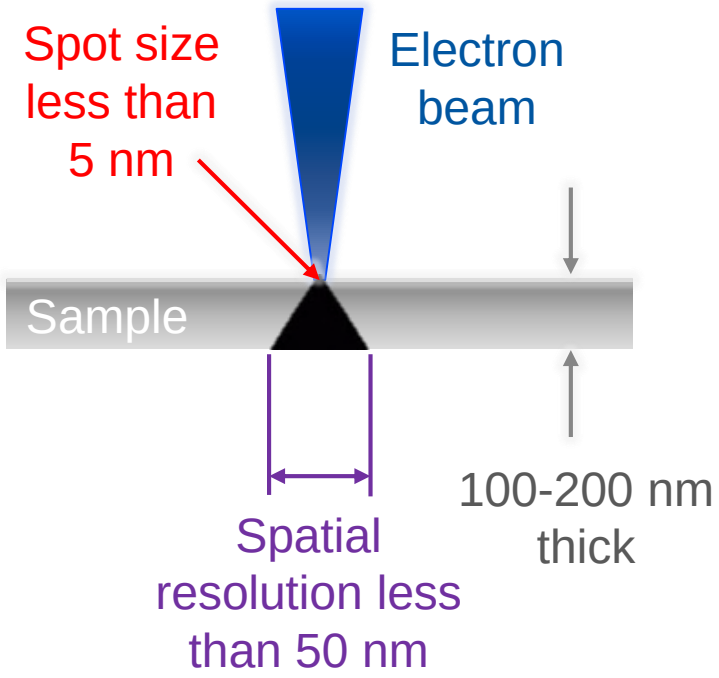
High resolution elemental mapping

- Need to examine elemental diffusion
- Limiting factor on EDS resolution is interaction volume not beam spot size
- Resolution insufficient for thin film samples



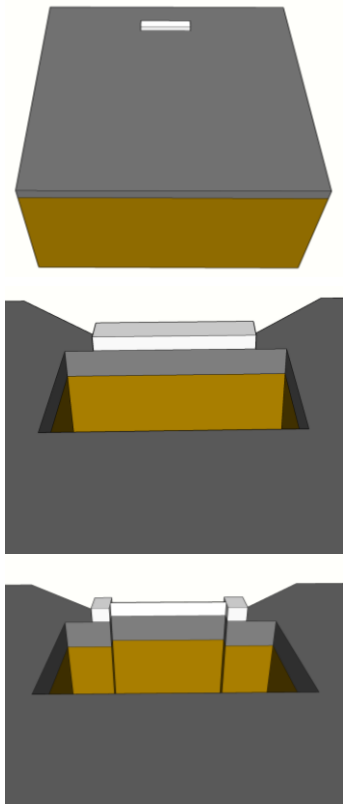
Transmission EDS

- Improve resolution – reduce interaction volume
- Lamella 100-200 nm thick
- Nanoscale mapping possible
- Problem:
  - 10-20 hours milling time
  - Complex experimental process – prone to failure
  - Many samples need investigating



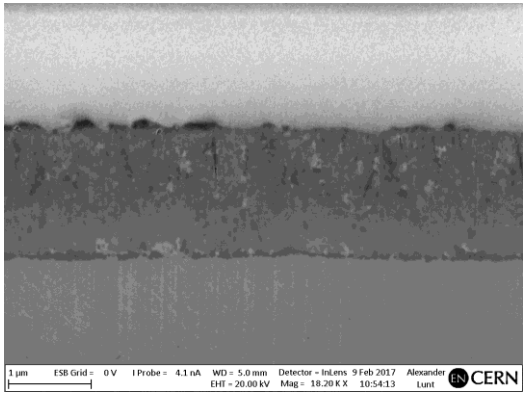
Solution: ‘Rapid’ TEDS

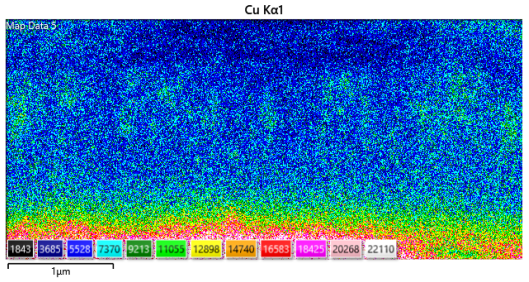
- New approach based on ‘in-situ’ lamella
- No need for risky removal of lamella
- Significant reduction in milling time (4 hours)
- Similar nanoscale resolution EDS mapping possible.



Contamination assessment

- Electron contrast in cross sectional analysis
- The origins of this:
  - Phase segregation
  - Contamination
  - Electron channelling from grain orientation
- TEDS can provide answers

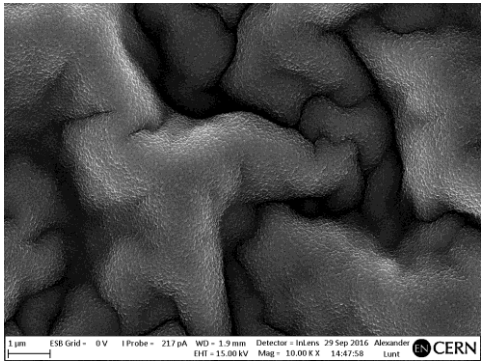




Microscale residual stress analysis

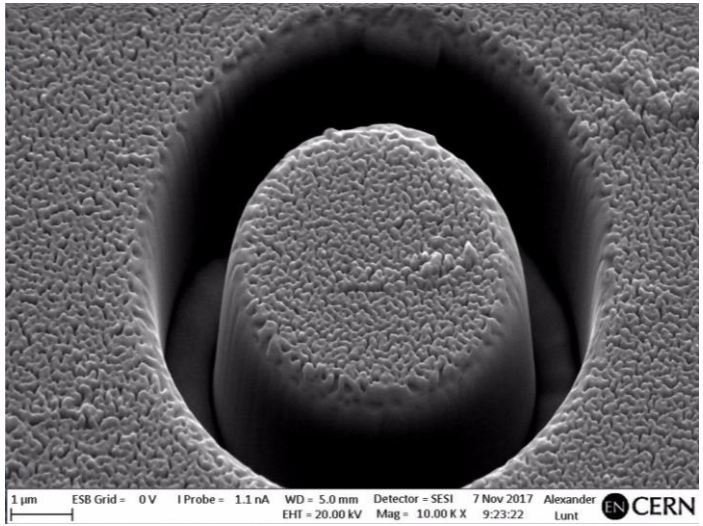
- Residual stress in thin films
  - Peel off & T_c
- X-ray diffraction conventional approach
  - Average over large area
- Need to quantify at specific locations

  – origins of failure



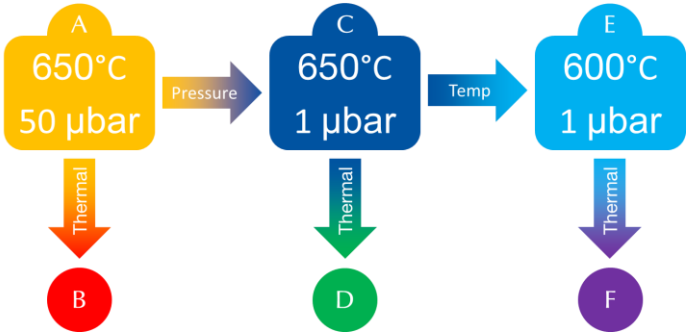
Ring-core residual stress analysis

- FIB based technique
- Incremental annular milling
- Relaxation of core
- Record SEM images
- Quantification using digital image correlation
- Compare to finite element simulations
- Quantify residual stress originally present within core



Nb_3Sn thin films

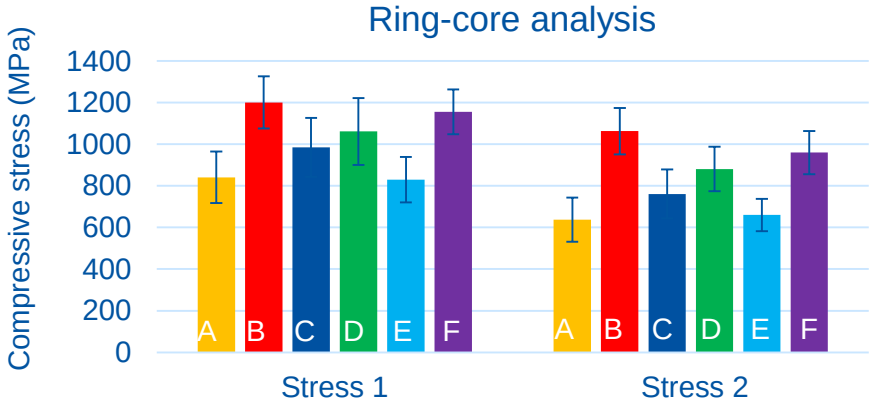
- Influence of coating pressure, temp and thermal processing

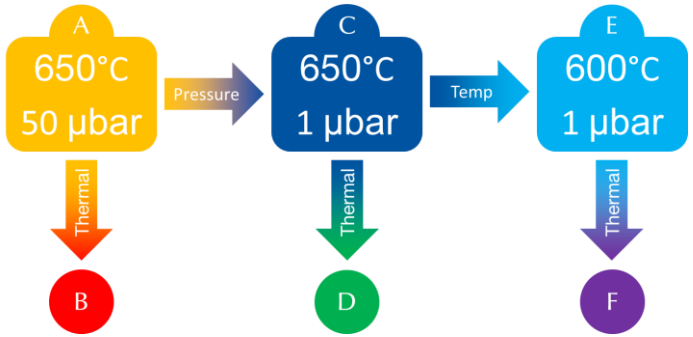


- Thermal treatment = 650°C for 5 hours
- High compressive stress = higher critical temperature

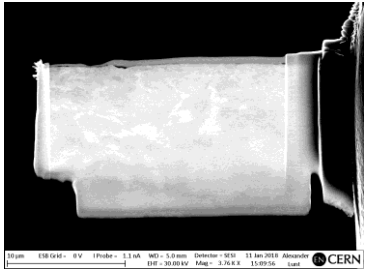
Residual stress results

- Highly compressive stresses*
- Lower temp + higher pressure – reduced stress
- Thermal treatment – increase in stress

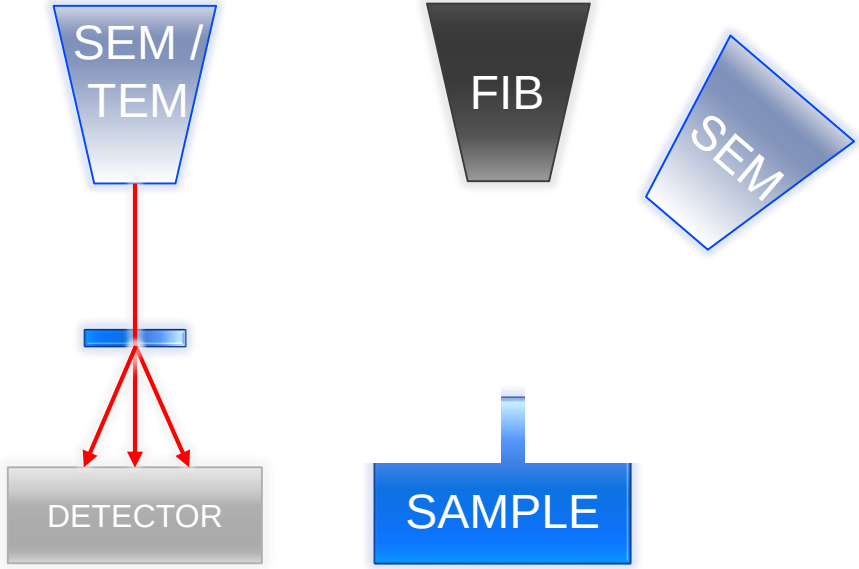




Cavity substrate analysis

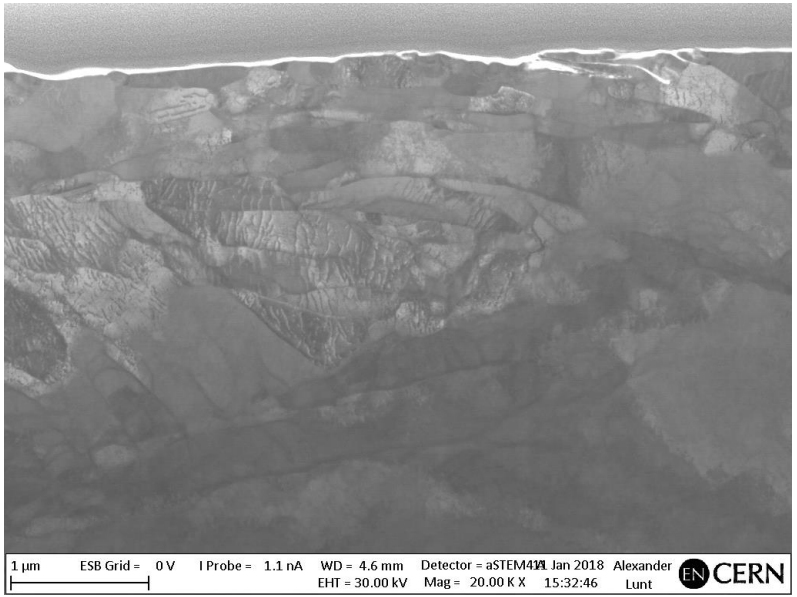


- Investigations into Cu cavity manufacturing techniques ongoing
  - Spinning and electro-hydraulic forming
- Scanning transmission electron microscopy
  - TEM lamella



Potential insights

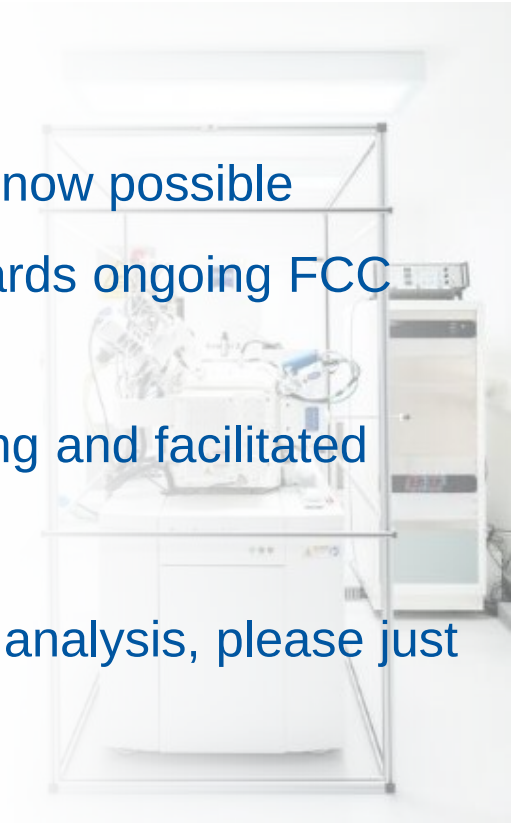
- Nanoscale behaviour
- Dislocation density
- Surface response
- Grain characteristics
- Comparison between inner & outer surfaces



Further discussion – upcoming presentation
Carolina Abajo Clemente 14:30 Berlage zaal (1.9)

Summary

- Powerful tool with broad spectrum of techniques now possible
- Conventional & tailored techniques directed towards ongoing FCC projects
- Useful results which have improved understanding and facilitated design improvements



If you have any studies which may benefit from FIB analysis, please just get in touch!

Any questions?

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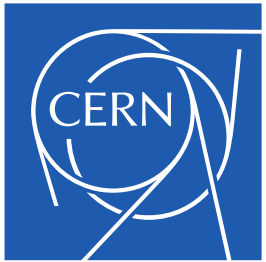
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