Synchrotron radiation techniques for the characterization of Nb_3Sn superconductors

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Outline

1) Why using high energy synchrotron radiation for characterizing Nb_3Sn superconductors?
2) Case studies:
   I. Synchrotron x-ray diffraction for phase analysis during *in-situ* reaction heat treatment of Nb_3Sn strands
   II. Fast synchrotron micro-tomography for monitoring void formation during *in-situ* reaction heat treatment of Nb_3Sn strands
   III. High resolution diffraction for measuring the strain state in Nb_3Sn composite strands during *in-situ* tensile loading
3) Conclusion and outlook

Why using high energy synchrotron radiation for characterizing Nb_3Sn strands? (I)

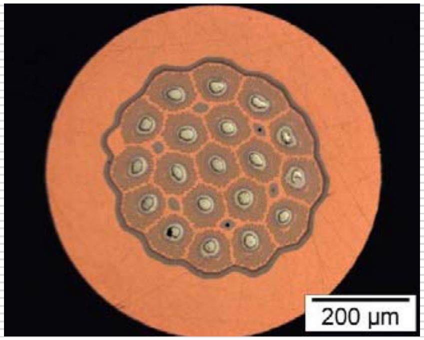
- Electron microscopy is the most important tool for the materials characterization of Nb_3Sn superconductors. Non-destructive studies can be a useful complement.
- In the past non-destructive materials characterization of Nb_3Sn composite strands has been done by neutrons. Neutron high resolution diffraction measurements have provided for instance important results about the strain state within Nb_3Sn composite conductors.
- The performance of synchrotron sources and insertion devices is continuously improving. The flux of monochromatic x-rays that can be provided by state-of-the-art high energy scattering beam lines exceeds the neutron flux of the most powerful neutron sources by many orders of magnitude.
- Due to the very high x-ray flux, synchrotron experiments can be very fast and only relatively small sample volumes are needed.

Why using high energy synchrotron radiation for characterizing Nb_3Sn strands? (II)

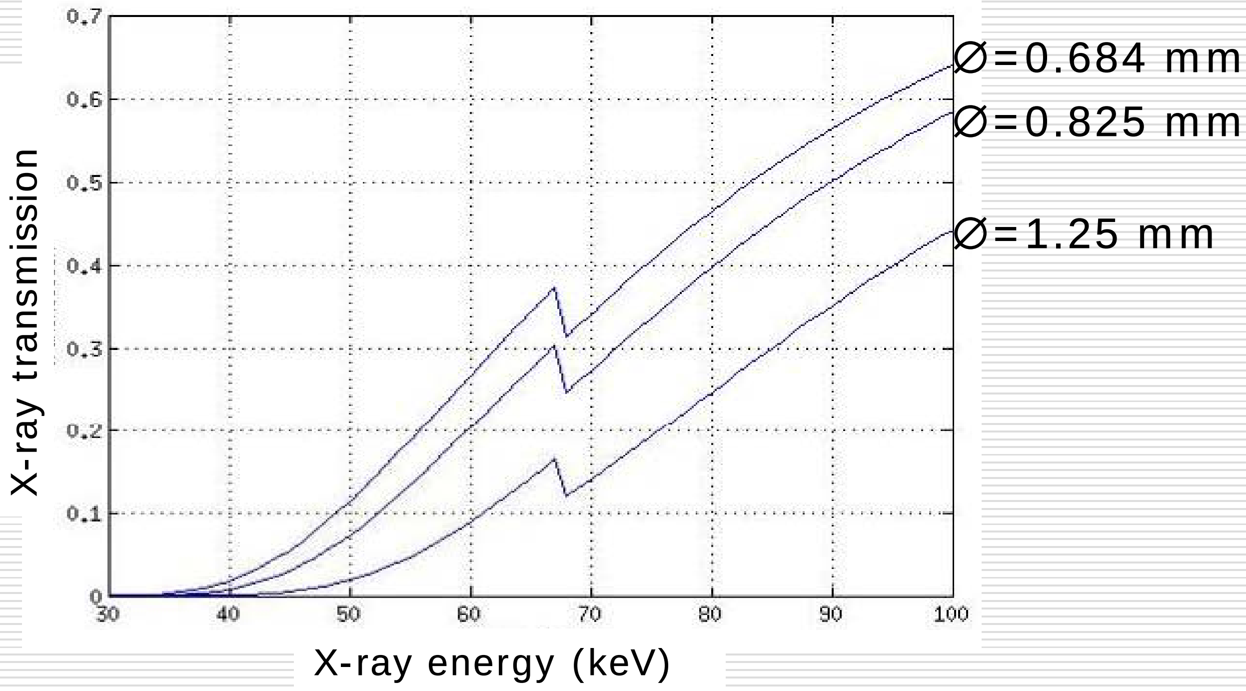
- The comparatively small scattering angles of high energy x-rays make it easier to add auxiliary equipment (furnace, cryostat, tensile rig, etc.) to the experiment and measurements can be performed *in-situ.*
- Different techniques can be combined in one experiment (e.g. diffraction and micro-tomography during an *in-situ* reaction heat treatment).
- High energy x-rays allow to perform non-destructive measurements and sample preparation artefacts can be avoided.

X-ray transmission through a Nb_3Sn strand as a function of strand diameter and x-ray energy

- For fast, *in-situ* studies of the highly absorbing Nb_3Sn strands a very high flux of high energy x-rays is required.
- The x-ray transmission for obtaining optimum signal-to-noise ratio in tomography is about 20 %.

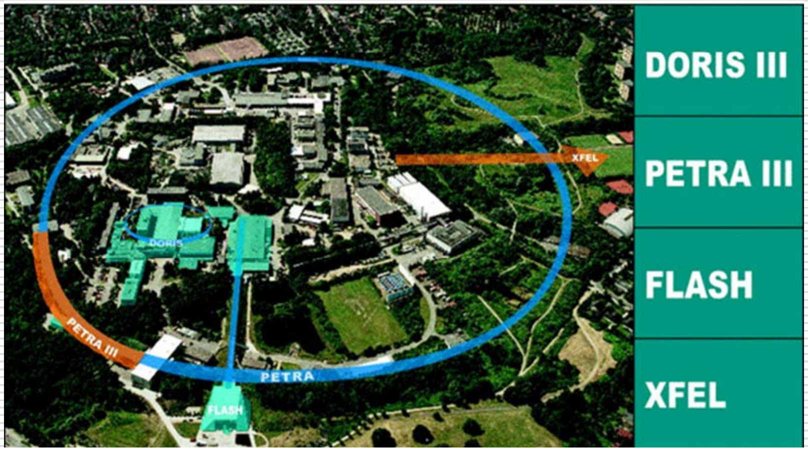


X-ray transmission as a function of x-ray energy through an IT Nb_3Sn strand with different diameters.



Synchrotron sources with high energy scattering beam lines

ESRF, Grenoble



DORIS III, Hasylab, Hamburg (from 2009 PETRA III)

APS, Chicago

SPring-8, *RIKEN, Japan*

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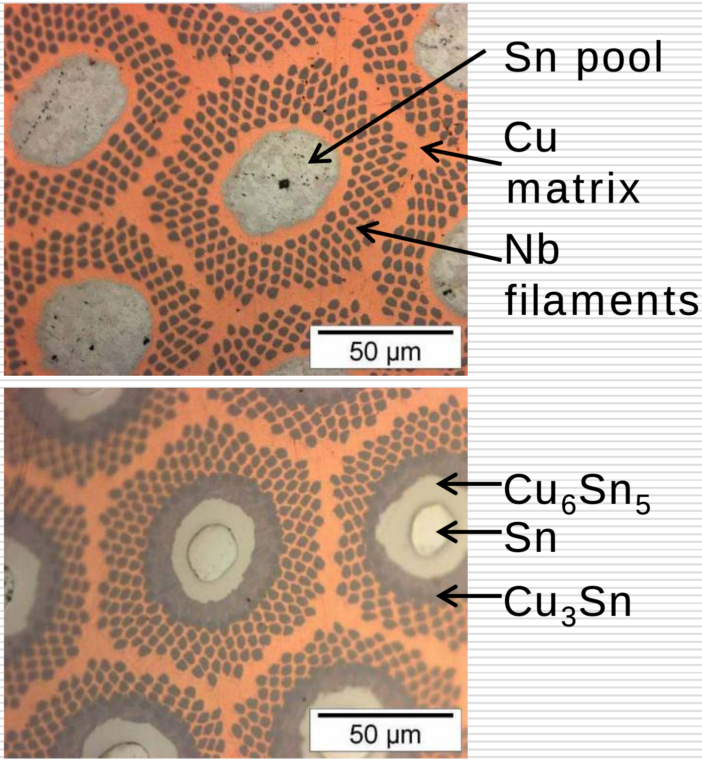
Phase transformations during the superconductor reaction heat treatment (HT)

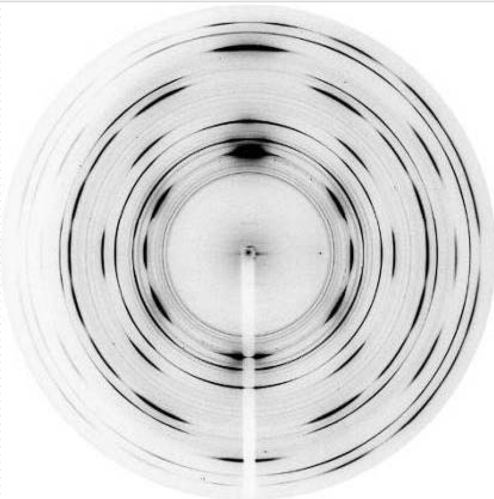
- In Nb_3Sn superconductors the brittle A15 phase is produced from the pre-cursor phases by solid state diffusion during a reaction HT at roughly 700 °C.
- The phase transformations prior to Nb_3Sn nucleation and growth can influence the microstructure and microchemistry of the fully reacted strand.
- The phase transformations can be studied by energy dispersive x-ray spectroscopy (EDS), using metallographic strand cross sections, prepared after *ex-situ* HT.
- X-ray diffraction is another technique that can be used for phase analysis.



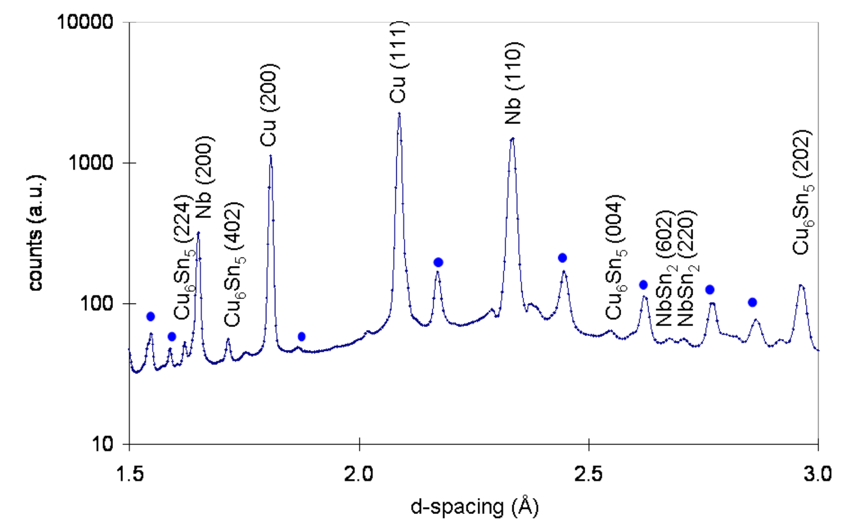
Metallographic cross sections of an IT Nb_3Sn strand as received and after 9 days at 220 °C.

Monitoring phase transformations by synchrotron diffraction during *in-situ* HT

Using high energy x-rays, diffraction measurements of Nb_3Sn strands can be performed in transmission geometry. In combination with a x-ray transparent furnace this allows to do diffraction measurements *in-situ* during strand heating cycles.

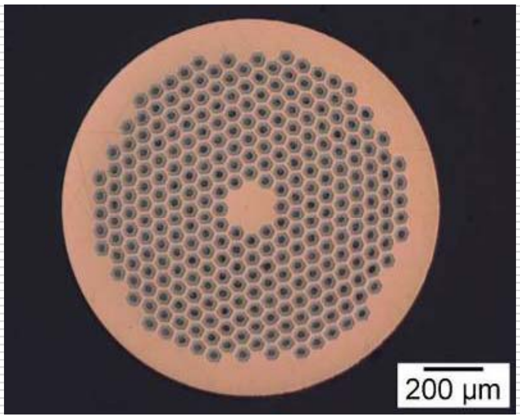


Diffraction pattern acquired with a MAR 345 image plate detector.

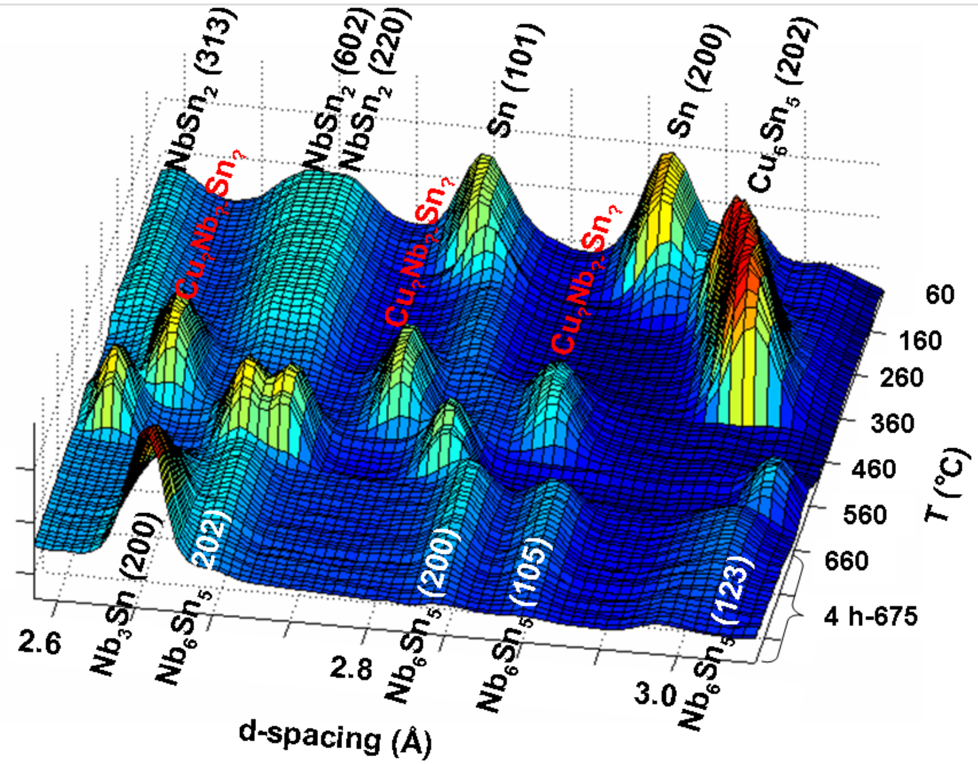


Radially integrated diffraction pattern of a PIT strand acquired at RT subsequent to 490 °C HT. The diffraction peaks of a Cu-Nb-Sn phase are indicated by full dots.

Phase transformations during the reaction HT of a state-of-the-art Nb_3Sn PIT strand



PIT strand B215 for NED, ∅=1.25 mm



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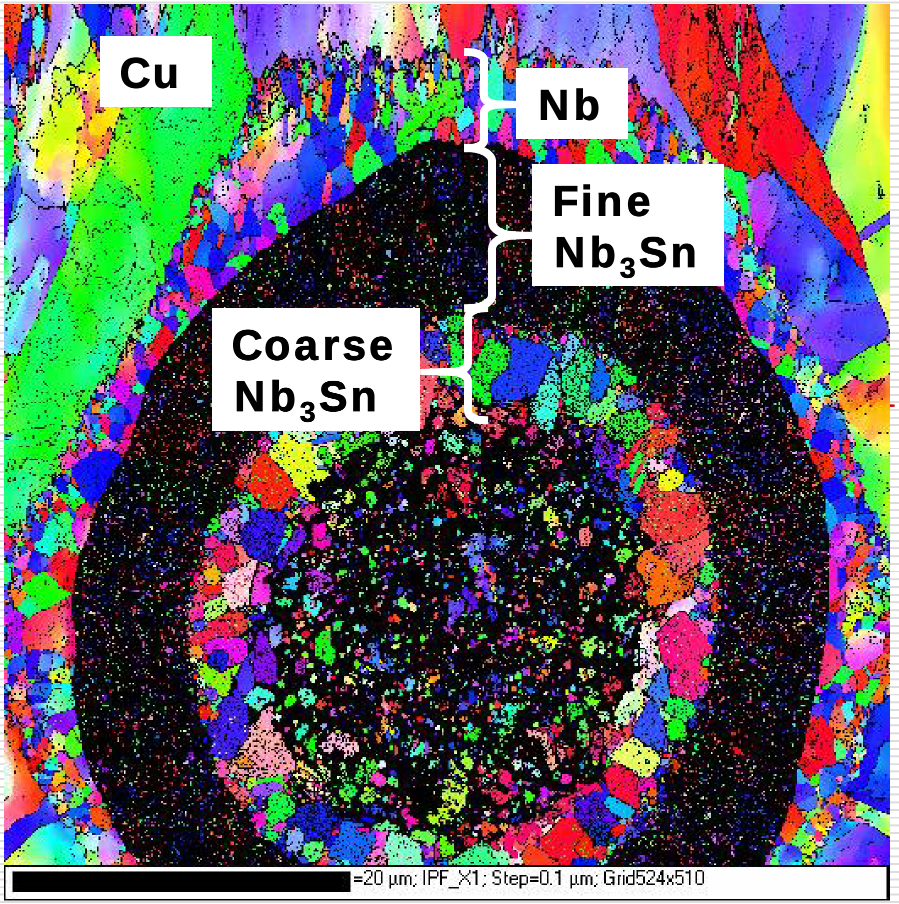
Sequence of radially integrated diffraction pattern acquired during in-situ reaction HT of PIT B215 strand.

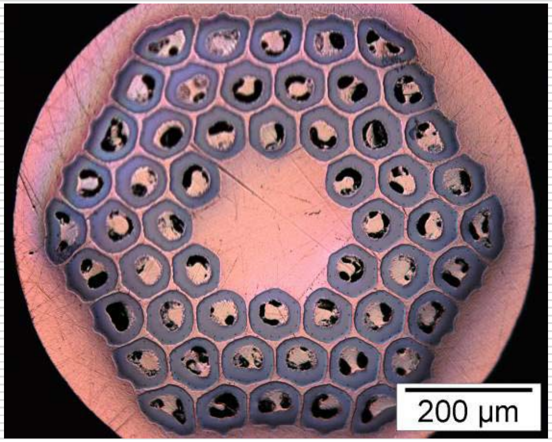
Growth of coarse Nb_3Sn grains in PIT strands subsequent to the formation of Nb_6Sn_5



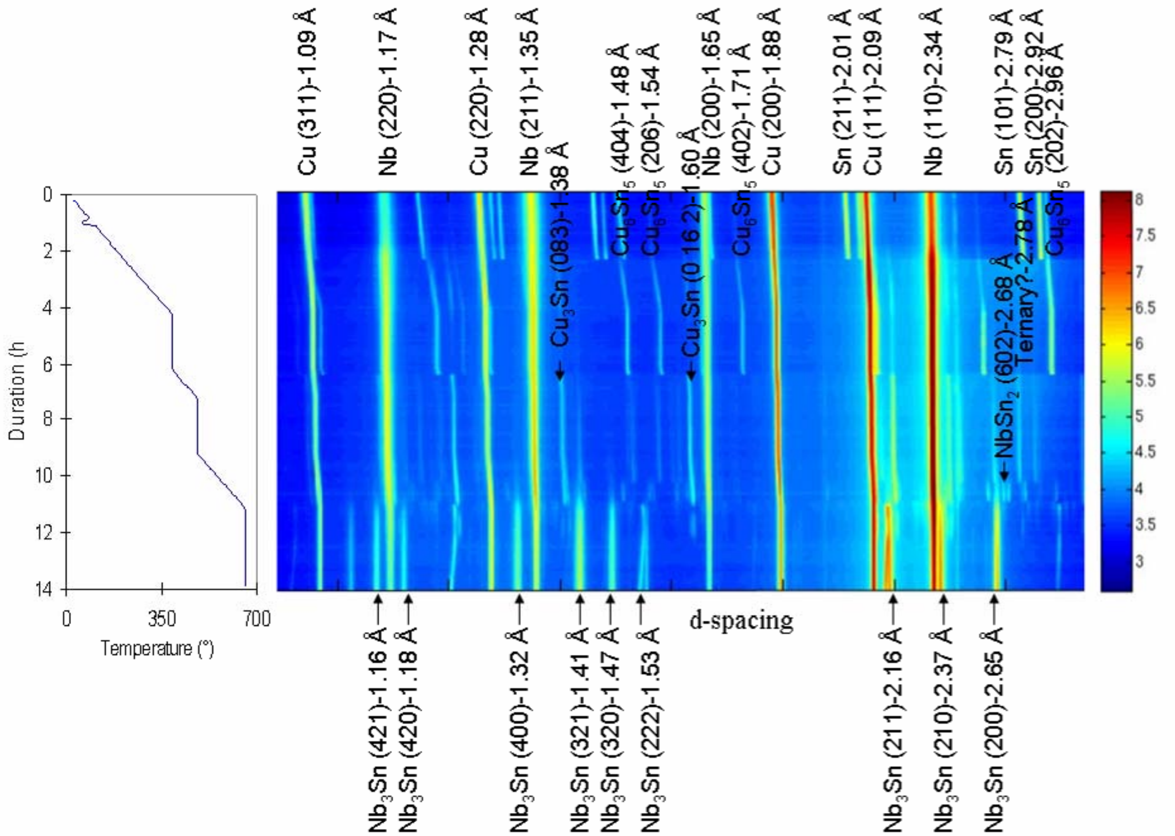
Nb_6Sn_5 formation inside the Nb precursor tubes of PIT strands prior to Nb_3Sn formation causes the subsequent growth of coarse Nb_3Sn grains.

Grain size and grain orientation distribution in a fully reacted PIT B215 cross section as determined by Electron Backscatter Diffraction (step size 100 nm). Courtesy G. Nolze, BAM, Berlin.

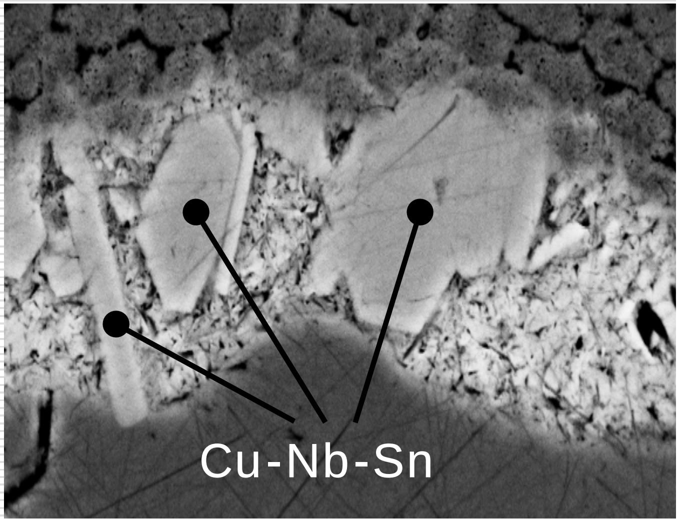
Phase transformations during the reaction HT of a state-of-the-art high J_c RRP® strand



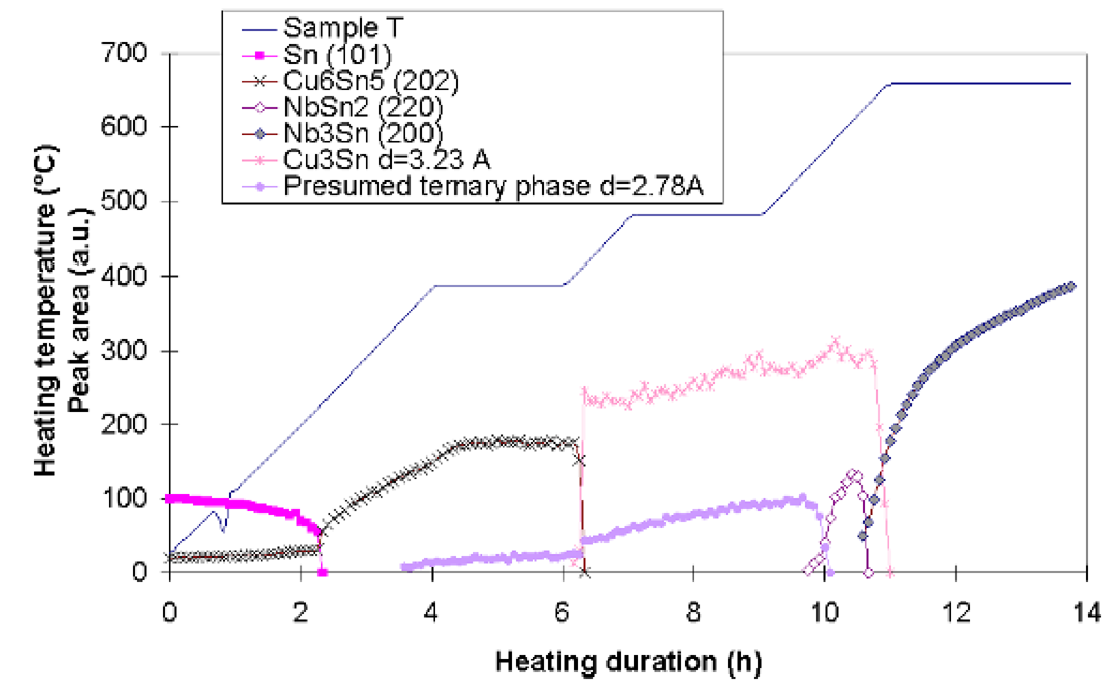
OI-ST RRP® strand, ∅=0.80 mm



Nb dissolution in a Cu-Nb-Sn ternary phase in IT strands with high Sn content

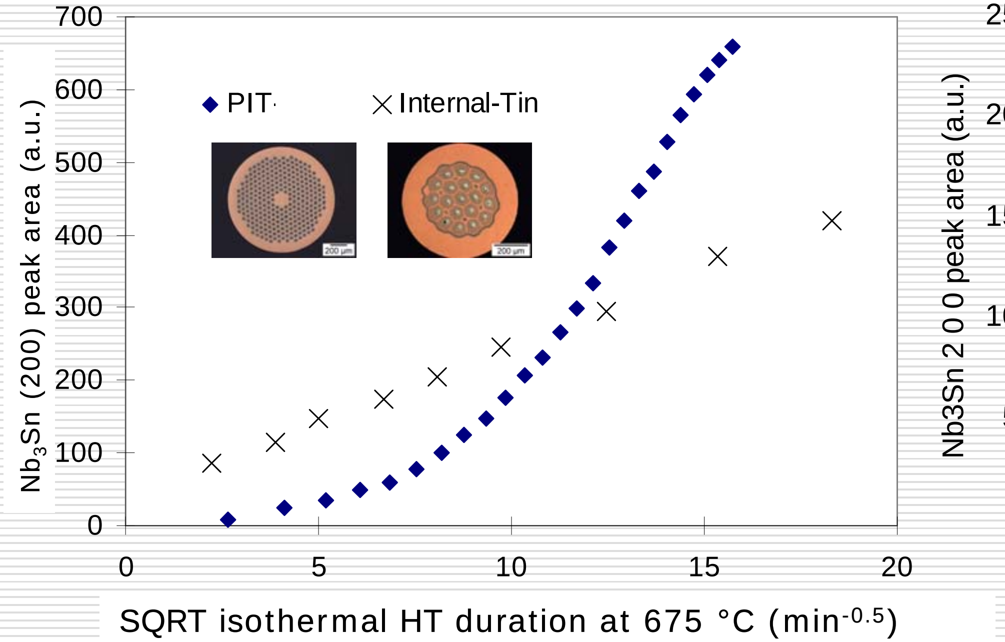


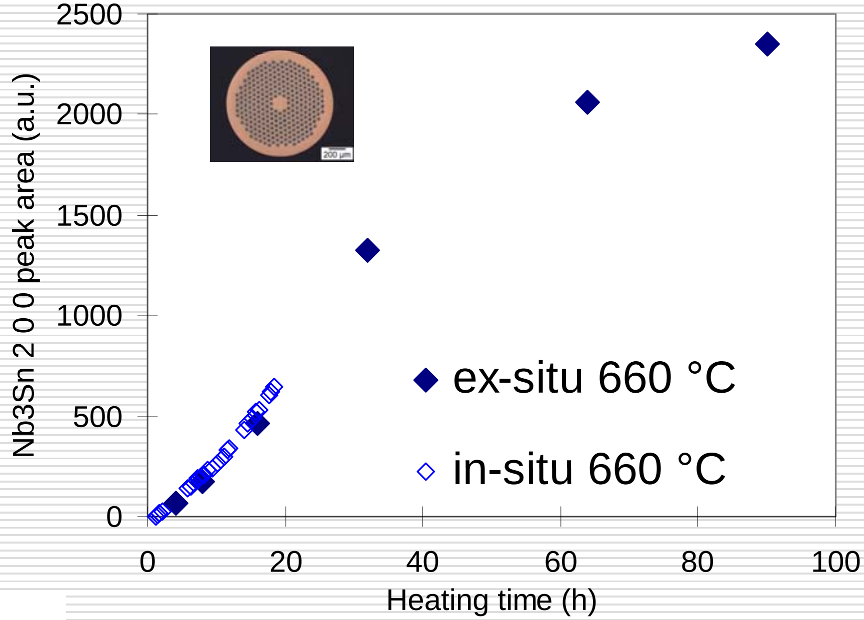
Backscatter electron image of the Cu-Nb-Sn ternary phase in the RRP® strand. SEM/EDS analysis courtesy of G. Arnau, CERN.



Summary of the phase transformations during reaction HT of the OI-ST RRP® strand. An important advantage of the RRP® design over the PIT design is that there is only little or no Nb_6Sn_5 formed in the RRP® strand (i.e. the amount of Nb_6Sn_5 remains below the detection limit of the experiment).

Nb_3Sn growth monitored by synchrotron x-ray diffraction





A15 phase growth at 675 °C in an IT strand with low Sn content and in a PIT strand. In the IT strand the Nb_3Sn growth during the first hours 675 °C HT follows a parabolic law, as expected for a fully diffusion controlled process.

A15 phase growth in the PIT B215 strand during isothermal 660 °C HT.

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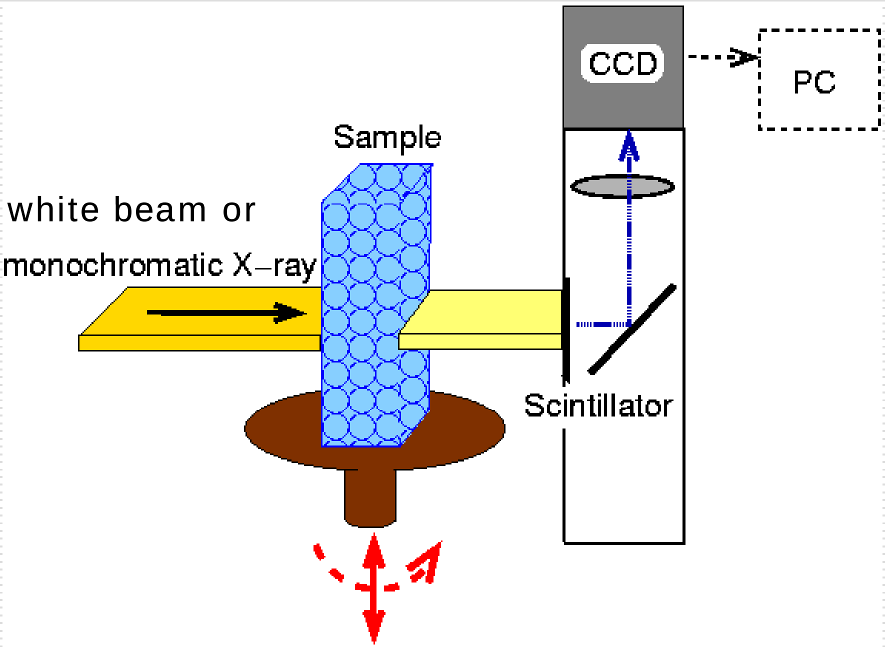
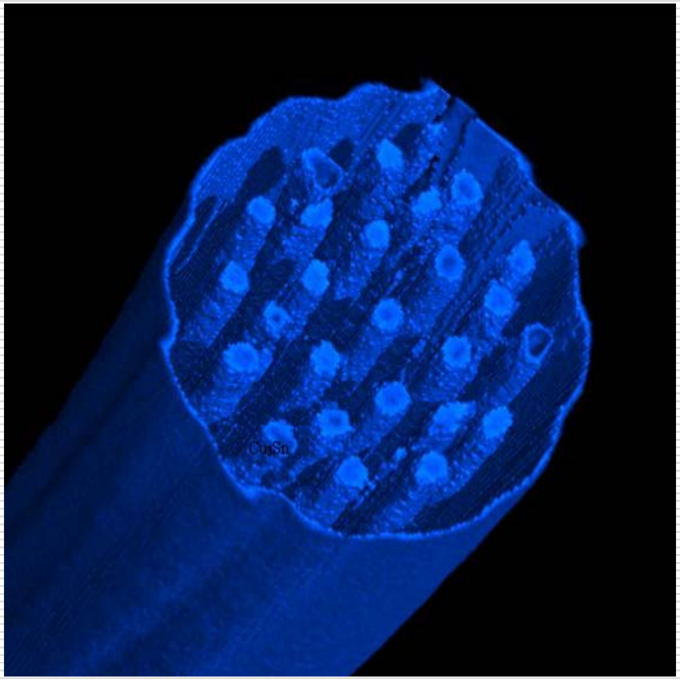
Synchrotron micro-tomography





Sketch of a tomographic setup.

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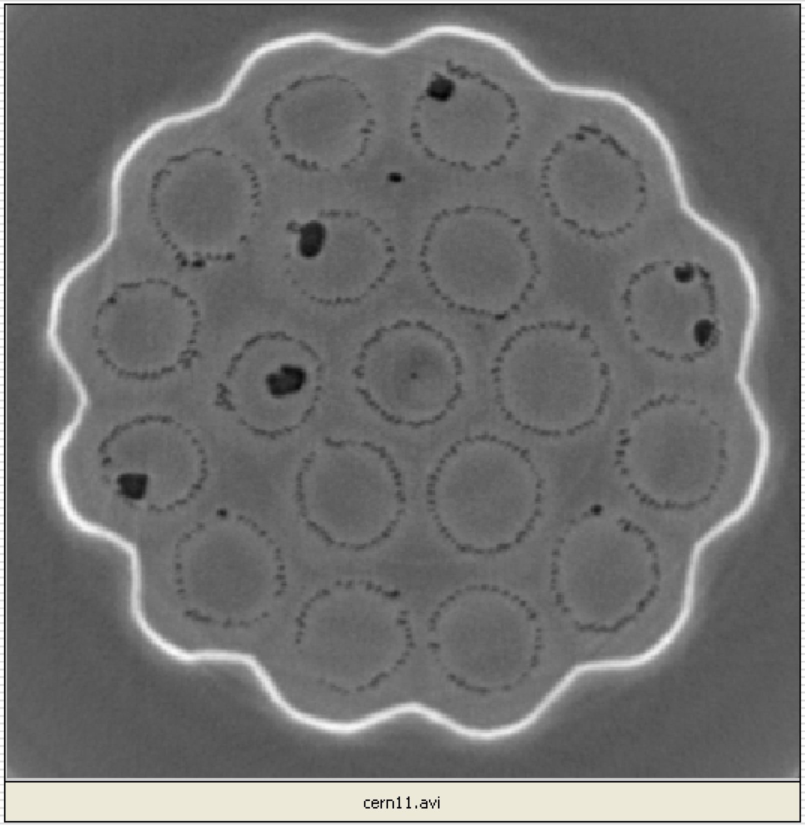
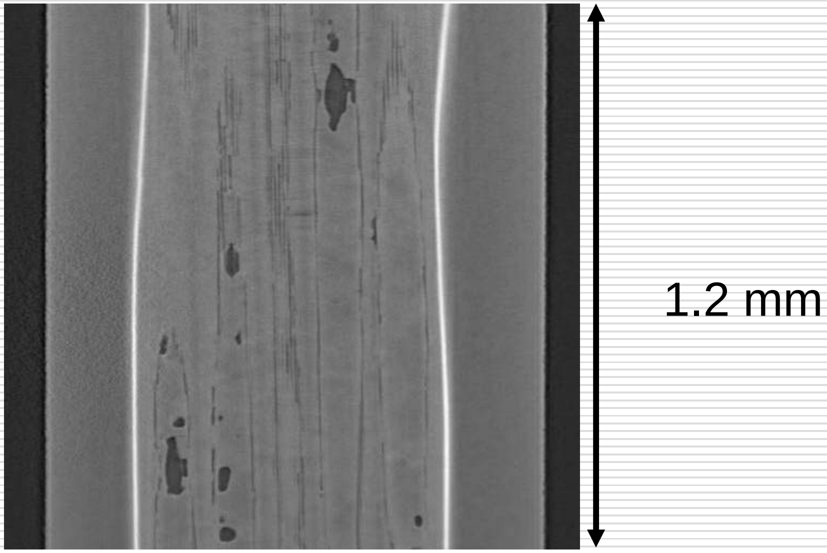
Tomogram of the diffusion barrier and Sn pools of an IT strand after ex-situ 220 °C HT.

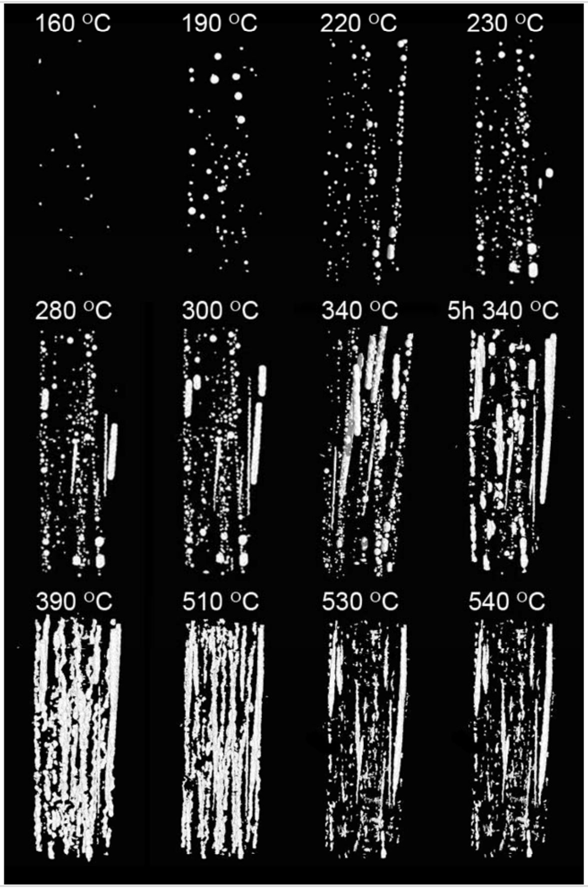
Fast synchrotron micro-tomography at ID15A of ESRF



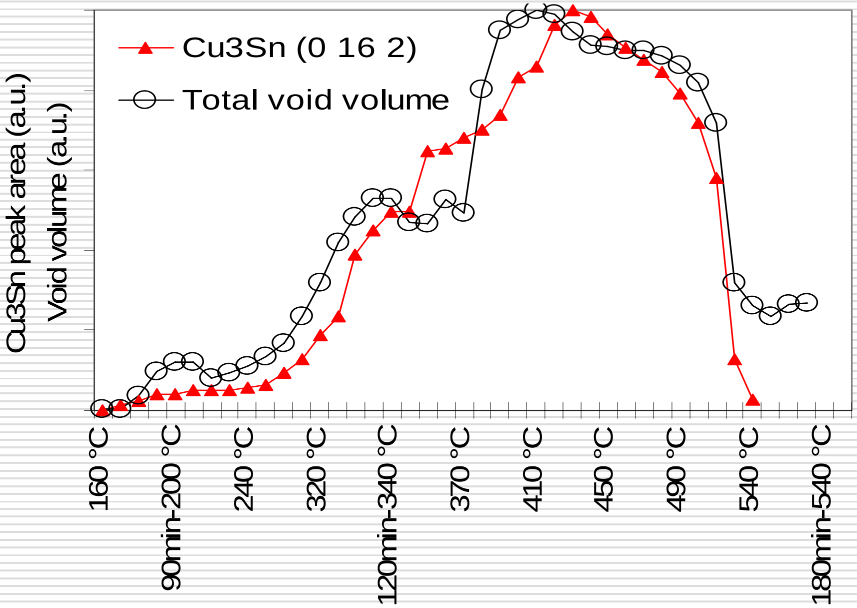


Transverse and longitudinal cross section of an ex-situ processed IT Nb_3Sn strand (∅=0.825 mm), obtained by synchrotron tomography. A tomogram is reconstructed from typically 1000 radiographs, which can be acquired within less than 1 minute. Courtesy M. Di Michiel, ESRF, Grenoble, and H. Reichert, MPI, Stuttgart.

Combined synchrotron micro-tomography and diffraction during *in-situ* reaction HT at ID15A of ESRF



The combination of fast synchrotron micro-tomography and diffraction during one experiment allows to distinguish between different void formation mechanisms in IT strands.



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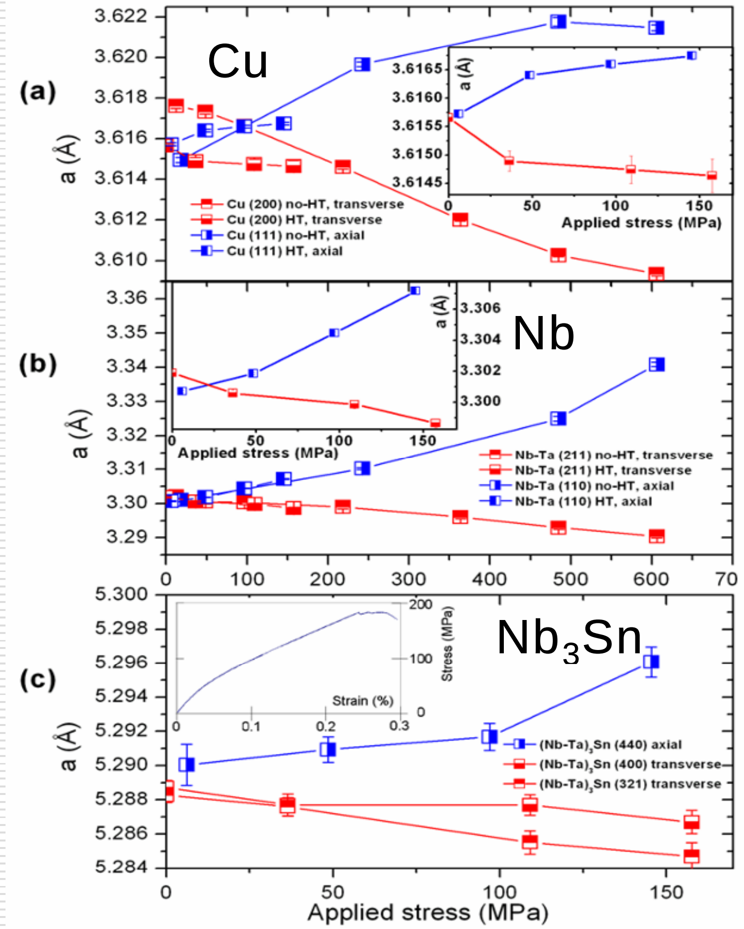
High resolution diffraction during *in-situ* tensile loading of Nb_3Sn strands

- High resolution diffraction measurements can be used as “strain gauges” for measuring the elastic strain in the different strand phases during *in-situ* tensile tests.
- The measurement of the axial and transverse lattice parameter variations allows to monitor the:
  - Internal stress state as a function of the macroscopic composite stress
  - Load transfer between the different strand phases
- High resolution diffraction results should allow to cross-check finite element simulations of the strand deformation.

Internal strain state in PIT strand as a function of uniaxial tensile wire stress measured by neutron diffraction at POLDI of PSI

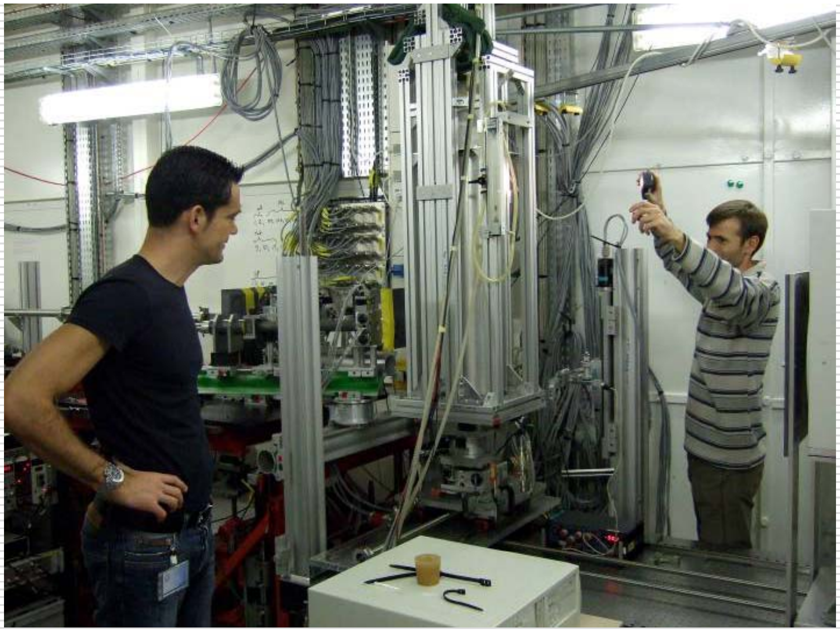
- High resolution neutron diffraction experiments have been performed at the POLDI experiment at the SINQ neutron source of PSI.
- During the diffraction measurements a well defined uniaxial tensile stress could be applied to a single non-reacted and reacted PIT strand.
- The acquisition time for one neutron diffractogram with sufficient signal-to-noise-ratio was several hours.

Applied Physics Letters, 91(4), 042503, (2007)



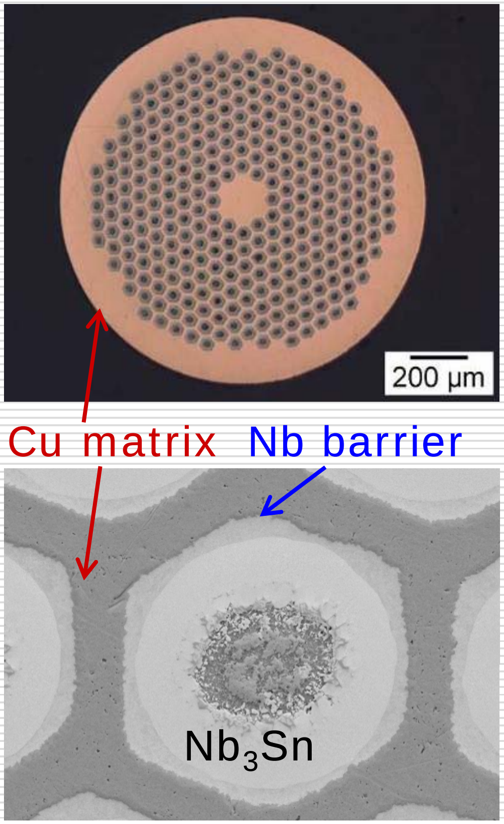
High resolution synchrotron diffraction during *in-situ* tensile tests of Nb_3Sn strands at 4.3 K at ID15B of ESRF

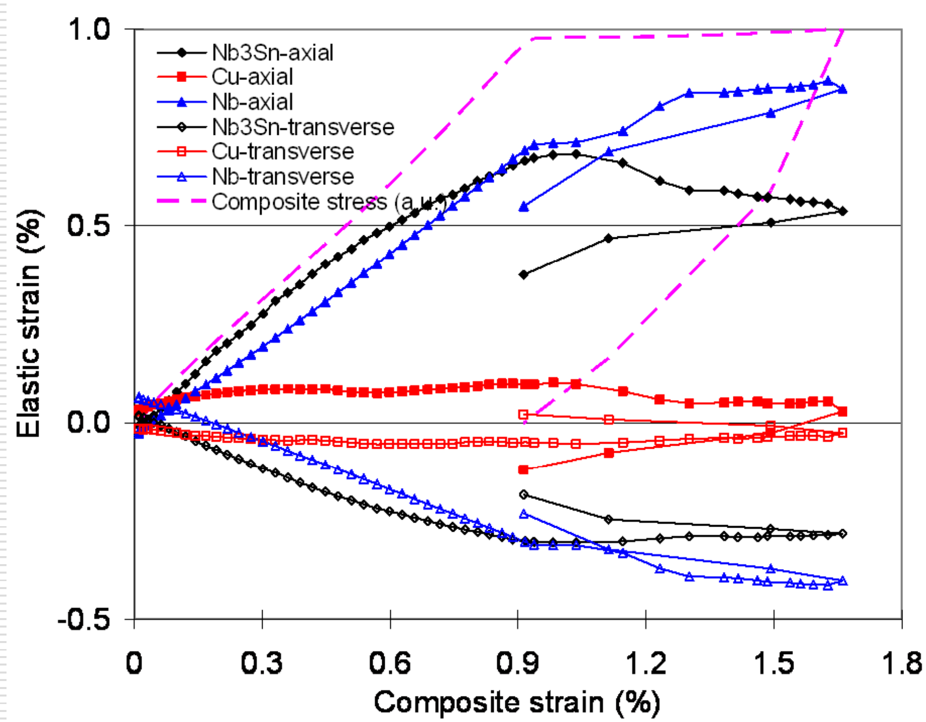
- Collaboration University of Geneva, ESRF and CERN, conducted by F. Buta from Uni-Geneva.
- A dedicated tensile rig from Uni-Geneva within a x-ray transparent LHe glass cryostat has been added to the ID15B beam line.
- The acquisition time for one diffractogram is about 10 seconds.
- The obtainable resolution is better than 10^{-4}.



Uni-Geneva glass cryostat with tensile rig installed at ID15B of ESRF

Elastic strain in the different strand phases as a function of composite strain at 4.3 K (preliminary results)





Elastic strain in Nb_3Sn, Nb and Cu as a function of the PIT B215 composite strain. Synchrotron diffraction and peak fitting courtesy F. Buta and M. Di Michiel.

Internal strain state in a Nb_3Sn strand reacted on a critical current measurement barrel

- Collaboration CERN, University of Poitiers and PSI.
- The strain dependence of the critical properties of Nb_3Sn superconducting strands is a major complication of critical current measurements.
- The lattice parameters of Cu, Nb and Nb_3Sn within a PIT strand reacted on a Ti-6Al-4V barrel have been measured at the POLDI experiment of PSI by high resolution neutron diffraction at RT and at 10 K.

$(Nb\text{-}Ta)_3Sn$ (440) axial	5.2813±0.00081 -0.14 %
$(Nb\text{-}Ta)_3Sn$ (321) transverse	5.2929±0.00081 +0.08 %
Nb-Ta (110) axial	3.2968±0.00050 -0.12 %
Nb-Ta (220) axial	3.2969±0.00047 -0.12 %
Nb-Ta (211) transverse	3.3031±0.00054 +0.07 %
Nb-Ta (110) transverse	3.3040±0.00048 +0.09 %

Lattice parameters (in Å) in PIT B215 strand reacted on a critical current measurement barrel measured at RT, and estimated relative variation with respect to nearly stress free lattice parameters.

Conclusion and outlook

- The very high flux of high energy x-rays that can be provided through state-of-the-art synchrotron beam lines enables a variety of new experiments with Nb_3Sn composite strands.
- Future synchrotron experiments, in combination with electron microscopy and other techniques, may help to answer for instance the following questions:
  - Is the dissolution of part of the Nb filaments in a Cu-Nb-Sn ternary phase in high J_c IT strands inevitable?
  - How can the Nb_6Sn_5 formation in the Nb precursor tubes of PIT strands, prior to Nb_3Sn nucleation and growth, be avoided?
  - What is the influence of ternary and quaterny additions on the phase transformations in Nb_3Sn strands?
  - What determines the Nb_3Sn grain size in fully reacted strands, apart from HT temperature and duration?
  - What is the influence of voids in Nb_3Sn strands on the irreversible J_c degradation?

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- U. Stuhr, Laboratory for Neutron Scattering, Paul Scherrer Institut, Villigen
- A. Haibel, Hahn Meitner Institut, Berlin
- G. Nolze, Federal Laboratory for Materials Research, Berlin