

Simultaneous measurement of critical current, stress, strain and lattice distortions in high temperature superconductors

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Outline

- High energy synchrotron X-ray diffraction (XRD)
- Mechanical behaviour of Nb₃Sn composites in 4.2 K
- HTS samples
- Universal Test Machine (UTM)
- Electromechanical properties of different HTS
 - uniaxial tensile loading
 - transverse compressive loading
- Integration of the UTM in a synchrotron beamline
- Simultaneous XRD, I_c , stress and strain measurements
- Conclusion

Synchrotron Radiation Techniques for the Characterization of Nb_3Sn Superconductors

Christian Scheuerlein, Marco Di Michiel, and Florin Buta

Abstract—The high flux of high energy X-rays that can be provided through state-of-the-art high energy synchrotron beam lines has enabled a variety of new experiments with the highly absorbing Nb_3Sn superconductors. We report different experiments with Nb_3Sn strands that have been conducted at the ID15 High Energy Scattering beam line of the European Synchrotron Radiation Facility (ESRF). Synchrotron X-ray diffraction has been used in order to monitor phase transformations during *in-situ* reaction heat treatments prior to Nb_3Sn formation, and to monitor Nb_3Sn growth. Fast synchrotron micro-tomography was applied to study void growth during the reaction heat treatment of Internal Tin strands. The elastic strain in the different phases of fully reacted Nb_3Sn composite conductors has been measured by high resolution X-ray diffraction during *in-situ* tensile tests.

Index Terms—Diffraction, superconducting wires and filaments, tomography.

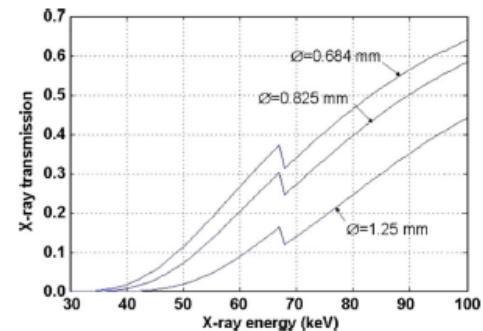
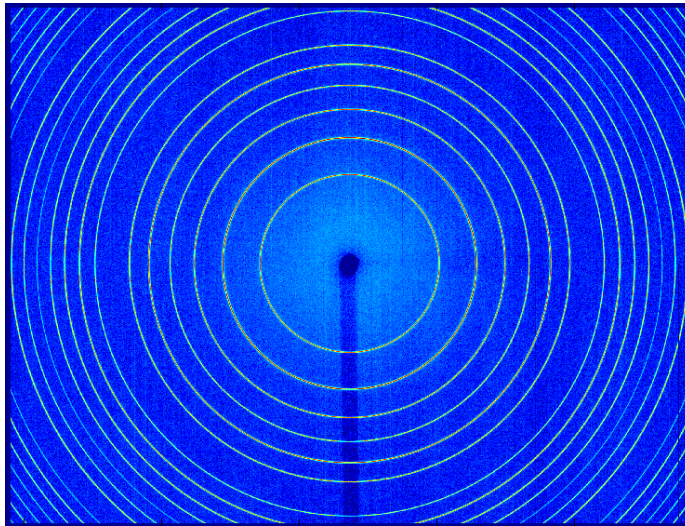


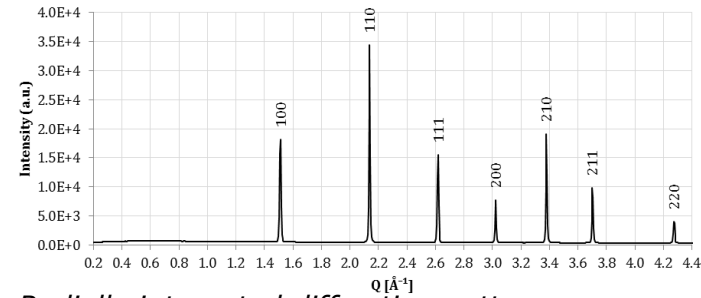
Fig. 1. X-ray transmission as a function of X-ray energy for an Internal Tin Nb_3Sn strand with different diameter. The X-ray transmission for obtaining optimum signal-to-noise ratio in tomography is about 20%.

- High X-ray energy \rightarrow enables non-destructive XRD in transmission geometry
- High X-ray flux \rightarrow enables fast acquisition of diffractograms and time resolved studies

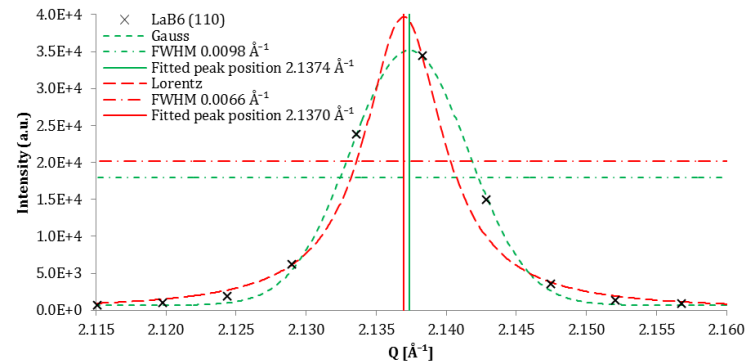
Angular dispersive high energy synchrotron XRD



Diffraction pattern of LaB₆ reference powder.



Radially integrated diffraction pattern.

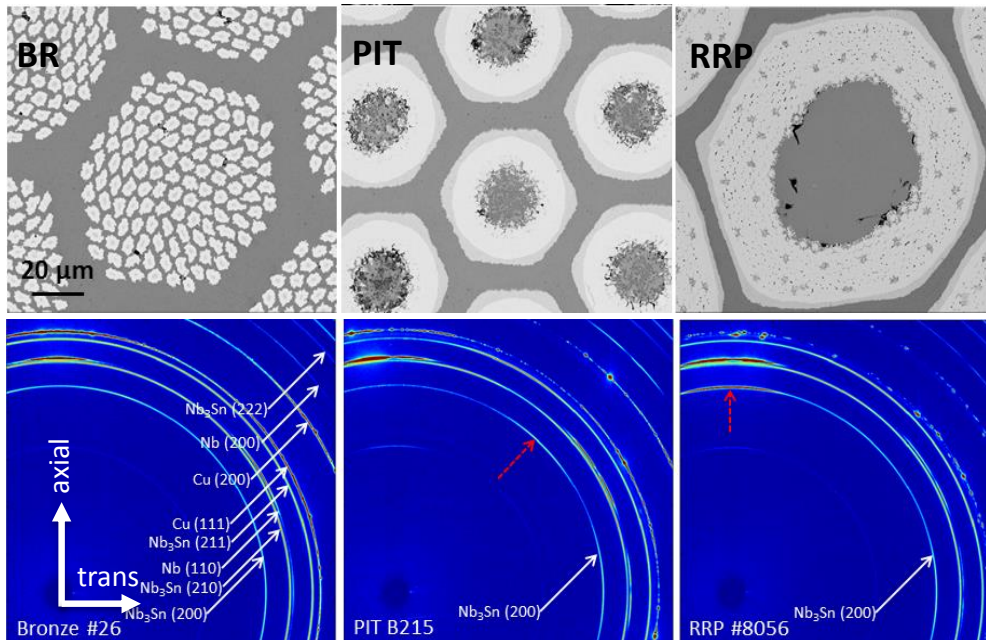


*Fit of the LaB₆ (110) diffraction peak.
Courtesy J. Kadar.*

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Nb₃Sn texture and elastic anisotropy

- Intensity fluctuations in the diffraction rings are a sign for texture [1,2].
- Nb precursor filaments exhibit a strong <110> fiber texture in axial wire direction.
- Different preferential Nb₃Sn grain orientation in the three Nb₃Sn wire types:
 - No Nb₃Sn texture in the Bronze Route (BR) wire.
 - Nb₃Sn <110> texture in PIT type superconductors.
 - Nb₃Sn <100> texture in RRP type superconductors.
- Texture causes an elastic anisotropy [3].



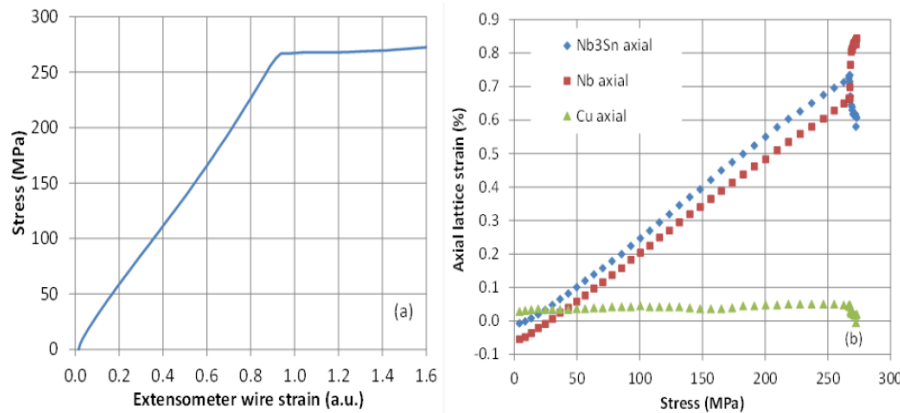
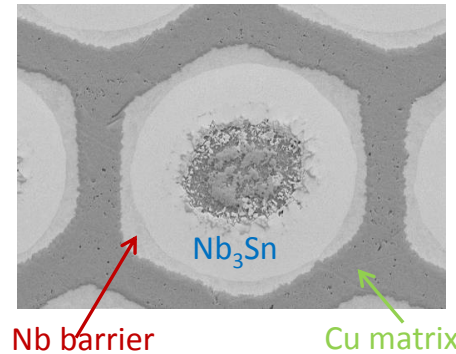
[1] SuST. 27, (2014), 044021
[2] SuST. 27, (2014), 025013
[3] IEEE TAS, 25(3), (2015),
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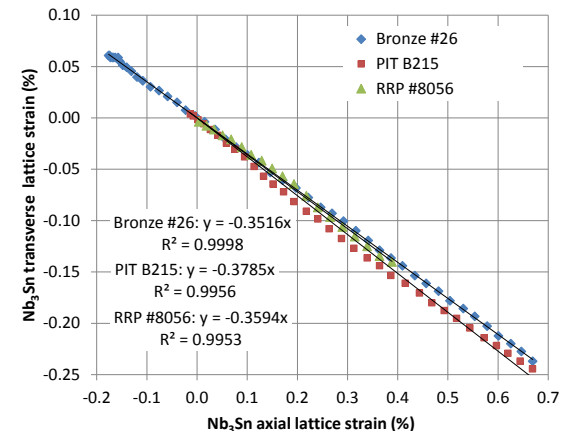
Mechanical behaviour of Nb₃Sn composite wires studied by simultaneous stress-strain-XRD measurements at 4.2 K

Experiment could be performed for the first time in a **University of Geneva-ESRF-CERN** collaboration.

- XRD-stress-strain results show
 - Nb and Nb₃Sn axial pre-compression
 - Linear elastic behaviour of Nb and Nb₃Sn
 - Load transfer from Nb₃Sn to Nb
 - Almost no load is carried by the annealed Cu matrix



(a) 4.2 K stress-strain curve and (b) axial lattice strain vs uniaxial tensile stress.



Comparison of the 4.2 K Nb₃Sn Poisson ratio in the BR, PIT and RRP wire.

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Nb₃Sn lattice distortions and superconducting properties

- The maximum critical current (I_c) in Nb₃Sn composite wires is obtained when applying a certain tensile strain ϵ_{max} at which the Nb₃Sn lattice parameters in axial and in transverse direction are approximately the same.
- Reasonable agreement between XRD results of axial pre-compression and ϵ_{max} results is found in several cases, but not always. As an example, ϵ_{max} of the Nb₃Sn PIT B215 wire measured with a Walters spring is 0.18%, while lattice parameter measurements show an axial pre-compression of 0.02% only.

Nb₃Sn nearly stress free lattice parameters and approximate Nb₃Sn axial pre-strain at 4.2 K estimated from the crossing of the axial and transverse lattice parameters [SuST. 27, (2014), 044021].

Sample	Nb ₃ Sn axial=Nb ₃ Sn trans	Nb ₃ Sn axial pre-compression
RRP #8056	5.276 Å	0.03 %
BR #26	5.275 Å	0.2 %
PIT B215	5.277 Å	0.02 %

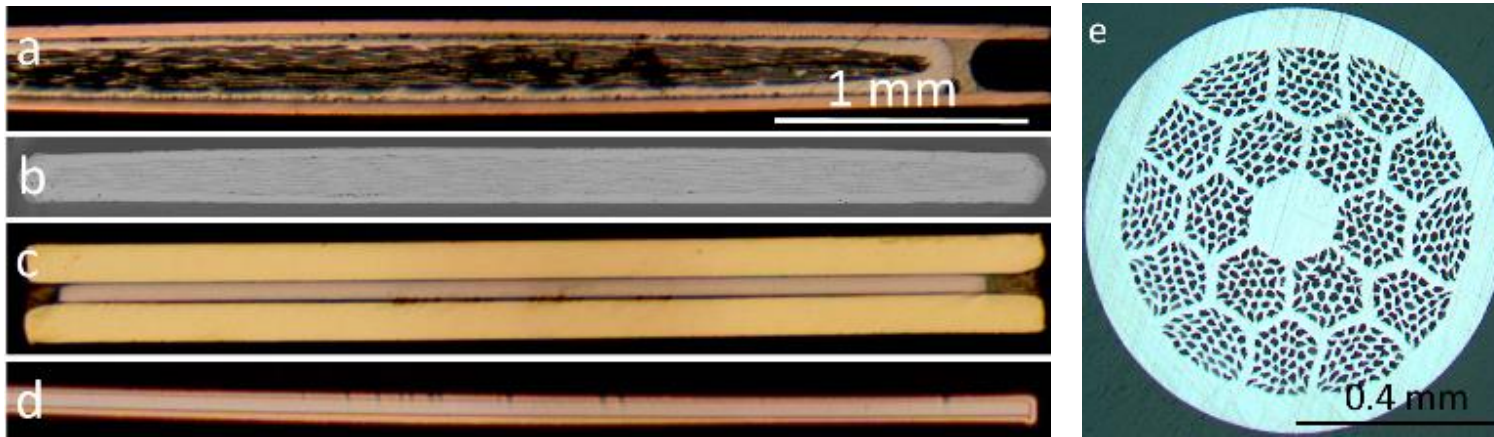
I_c and lattice parameter comparison uncertainties

- A comparison of lattice distortions with superconducting properties measured with different experiments is not always straight forward:
 - Influence of sample holder material and solder on the stress state in the superconductor
 - Sample geometry
 - Sample inhomogeneity
 - Previous sample manipulation
 - Thermal history of the sample
 - Strain measurement uncertainties
- These uncertainties can be eliminated or reduced when measuring superconducting properties and lattice distortions at the same time.
- Such measurements can most conveniently be performed with high temperature superconductors (HTS) in an open liquid nitrogen cryostat, and without applied field.

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The HTS samples



- (a) Bi-2223 tape type HT-CA with Cu reinforcement, from Sumitomo Electric Industries (SEI)
- (b) Bi-2223 tape type G, from SEI (without reinforcement)
- (c) ReBCO tape, type 8700 with brass reinforcement from American Superconductor (AMSC)
- (d) ReBCO tape, SCS-4050 from SuperPower
- (e) Bi-2212 wire, from Oxford Superconducting Technology (OST), overpressure processing by NHMFL-ASC

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Universal Test Machine (UTM) for electromechanical characterisation of superconductors at 77 K

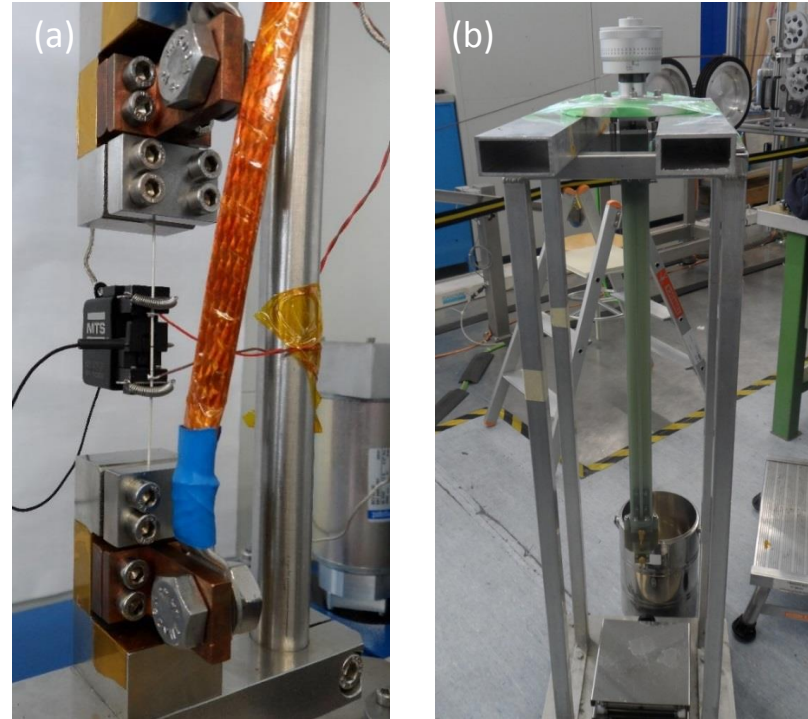
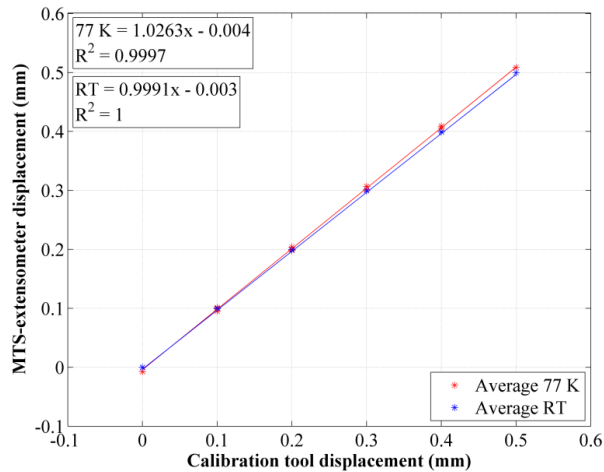
- Commercial UTM from Hegewald & Peschke MPT GmbH.
- Load capacity of 5 kN
- UTM has been equipped with a reverse load frame, such that grips and samples instrumented with extensometer and current leads can be immersed in liquid nitrogen.
- Measurements can be performed in force or in strain control.



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Extensometer

- MTS-clip on extensometer (type 632.27F-21)
- Gauge length of 25 mm
- Calibrated at RT by the supplier
- Calibrated at CERN at RT and at 77K



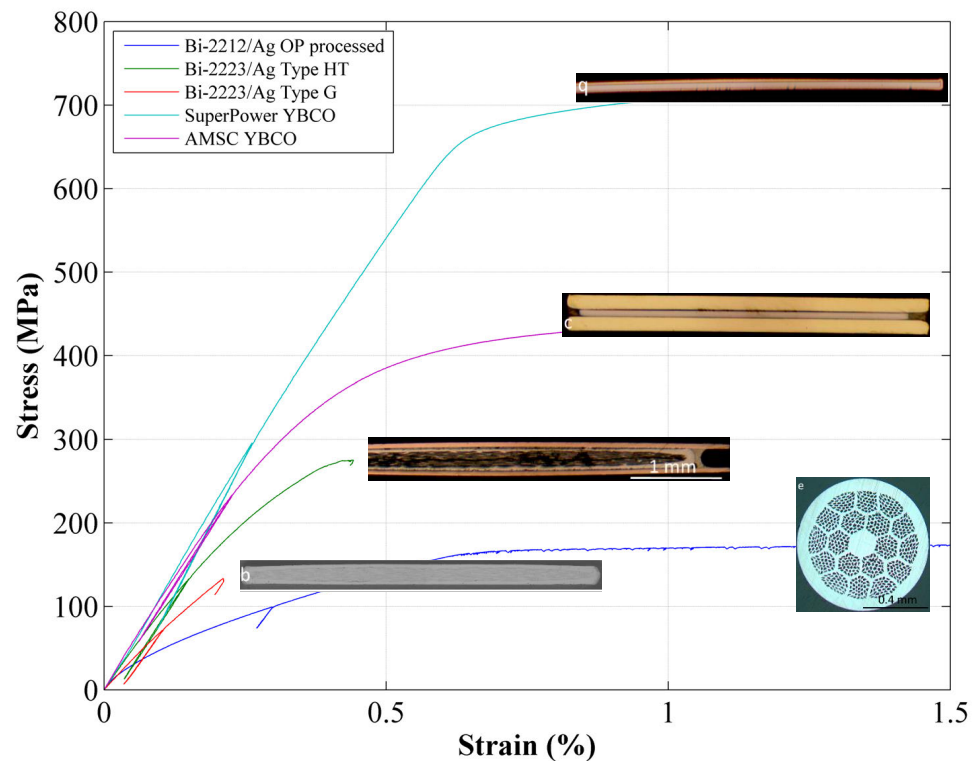
(a) Clip on extensometer and (b) calibration tool.

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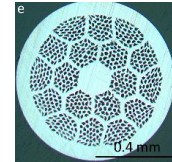
Comparison of the 77 K stress-strain curves

- YBCO tapes have highest strength because of the strong substrate materials used (Hastelloy or Ni-W alloy).
- The OP processed Bi-2212 wire has a relatively low ultimate strength, but comparable strain at rupture as the YBCO tapes.
- The Bi-2223 tapes rupture at relatively low strain.

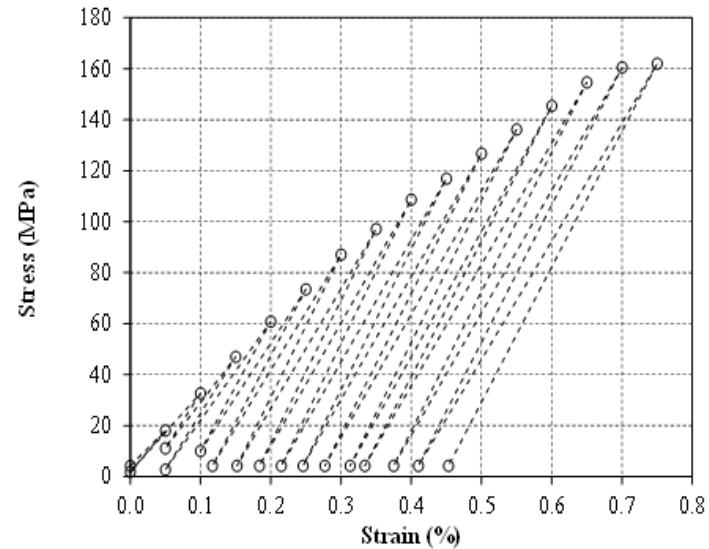
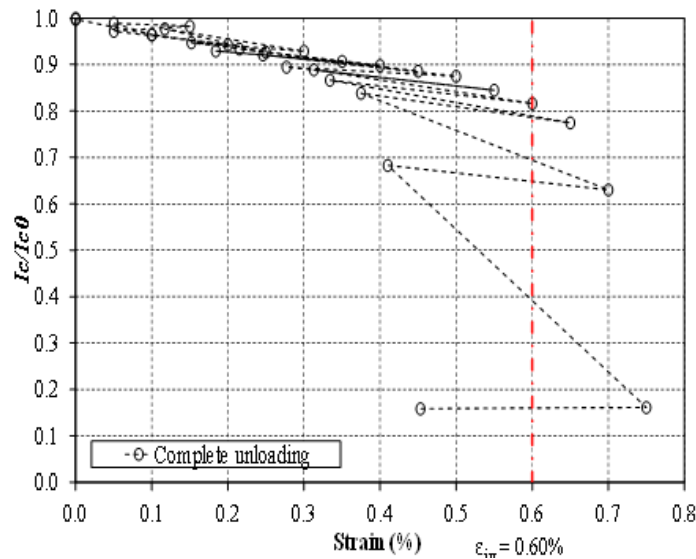


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Simultaneous I_c vs strain and stress measurement of highly dense Bi-2212 wire



- Irreversible I_c degradation of 5% $\epsilon_{\text{irr-5\%}}=0.60\%$ (at a stress $\sigma_{\text{irr-5\%}} \approx 150$ MPa)

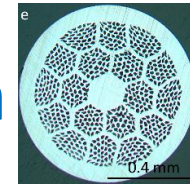


Permanent wire elongation after stress relaxation.

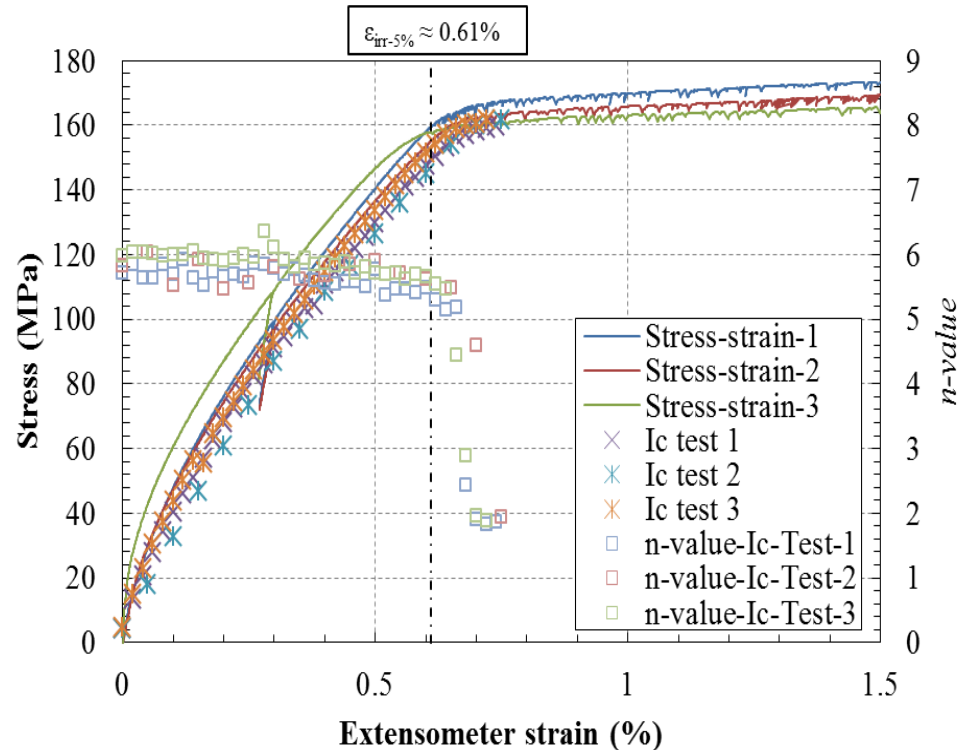
OP processed Bi-2212 wire provided by NHMFL-ASC
Supercond. Sci. Technol. 28, (2015), 062002

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Comparison of stress and n -value vs strain



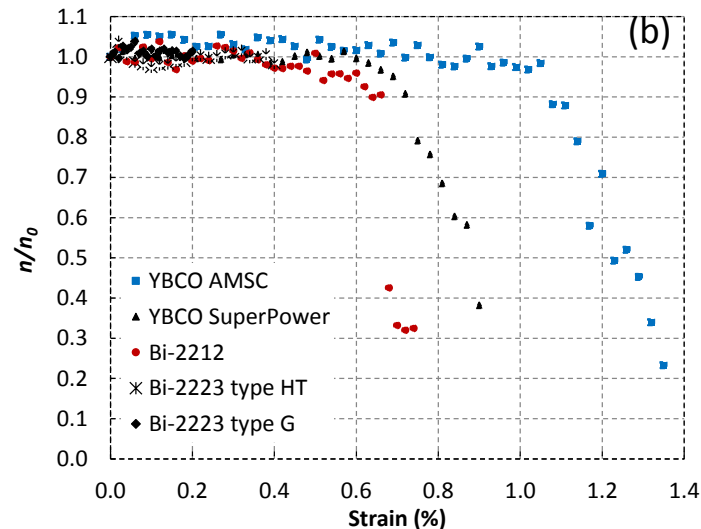
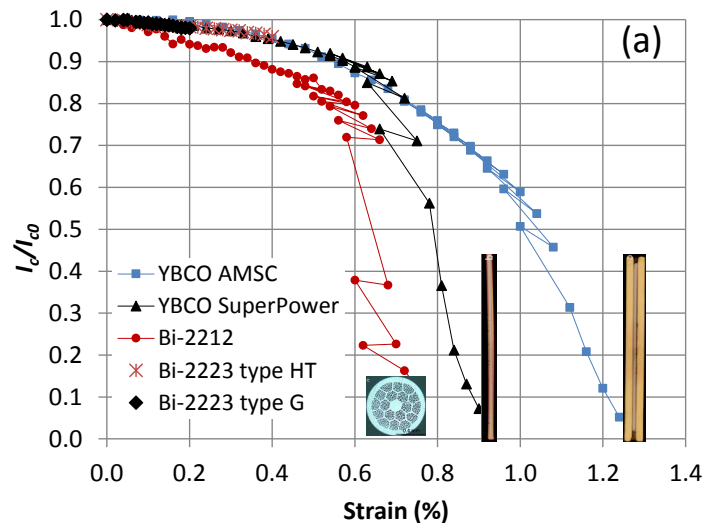
- When the wire strain exceeds about 0.65%, the stress reaches a plateau and the n -value decreases drastically, indicating strong filament damage.



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I_c and n -value degradation

- Relatively strong reversible I_c degradation under uniaxial tensile loading at 77 K.
- ReBCO AMSC $\epsilon_{\text{irr-5\%}}=1.0\%$; ReBCO SuperPower $\epsilon_{\text{irr-5\%}}=0.7\%$; Bi-2212 $\epsilon_{\text{irr-5\%}}=0.6\%$.
- n -value changes are an indication for irreversible damage.

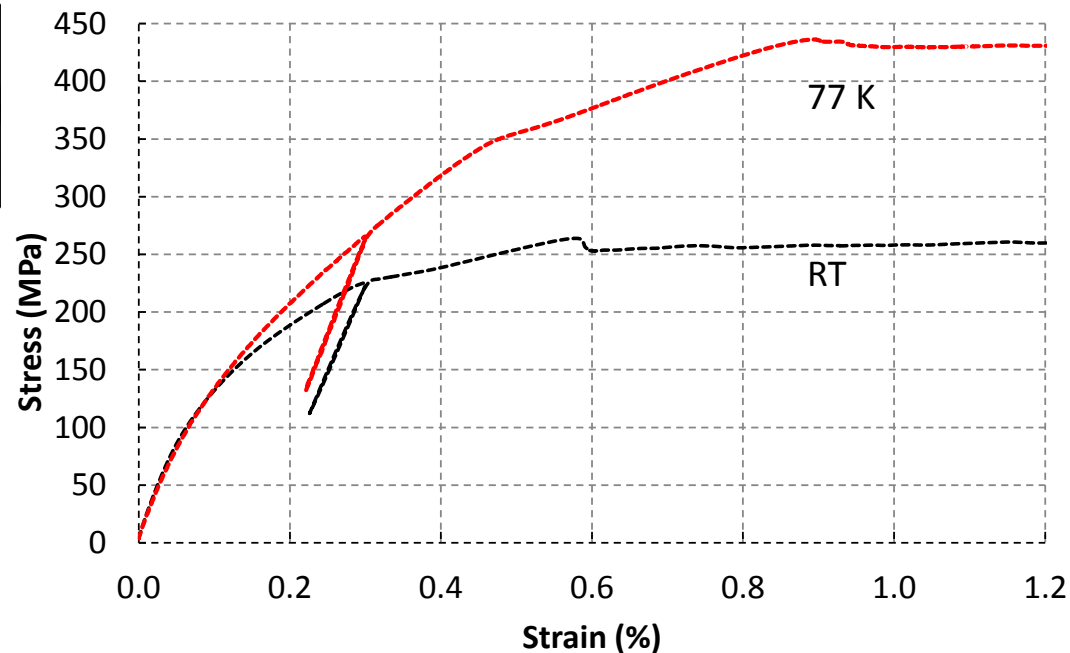
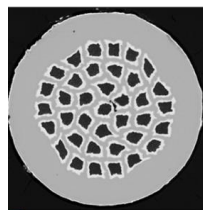


Comparison of (a) the I_c and (b) n -value variation of the different HTS as a function of uniaxial tensile strain.

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MgB₂ wire electromechanical properties

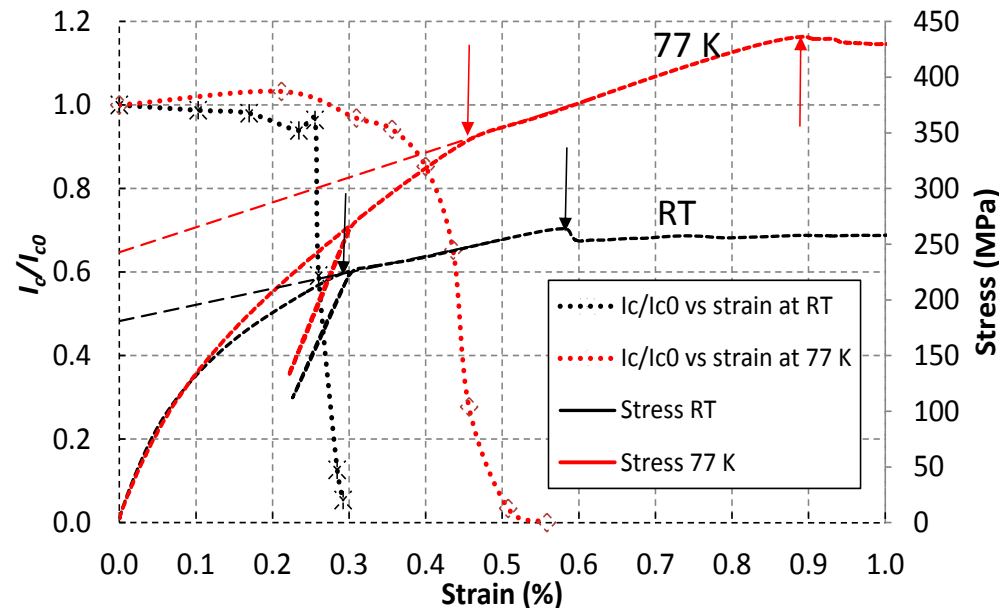
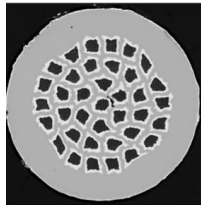
- Stress acting on a MgB₂ wire, e.g. during cabling, must not damage the filaments.
- The irreversible strain and stress limits of MgB₂ wires can be studied by applying a well defined strain with the UTM, and by subsequent I_c measurement in liquid He.



Stress-strain curves of an ex situ MgB₂ wire acquired at RT and at 77 K.

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MgB₂ irreversible I_c degradation as a function of uniaxial tensile strain applied at RT and at 77 K



Comparison of the stress-strain curves of an ex situ MgB₂ wire with the relative I_c variation measured at 4.2 K at 3 Tesla as function of the uniaxial tensile strain to which the wire was submitted at RT or at 77 K before the I_c measurement.

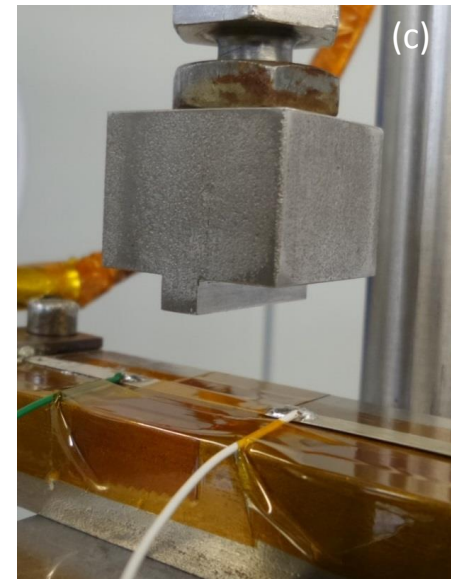
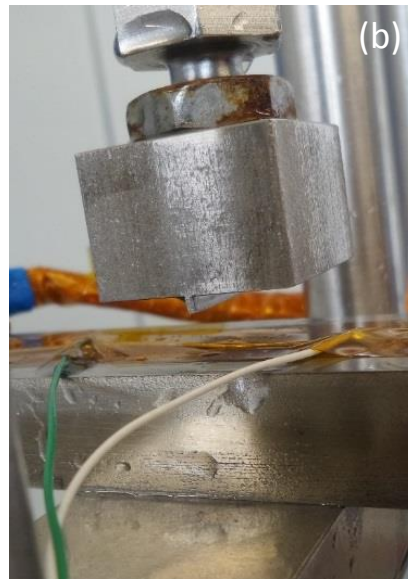
Courtesy P. Alknes, poster 3A-WT-P-05

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I_c vs transverse compression experiments

- Samples are mounted on a flat stainless steel plate that is connected to the bottom of the UTM reverse load frame.
- The maximum nominal stress that can be applied on a 4 mm-wide tape with a 1 mm-wide pressing tool and a 1 kN load cell is 250 MPa.

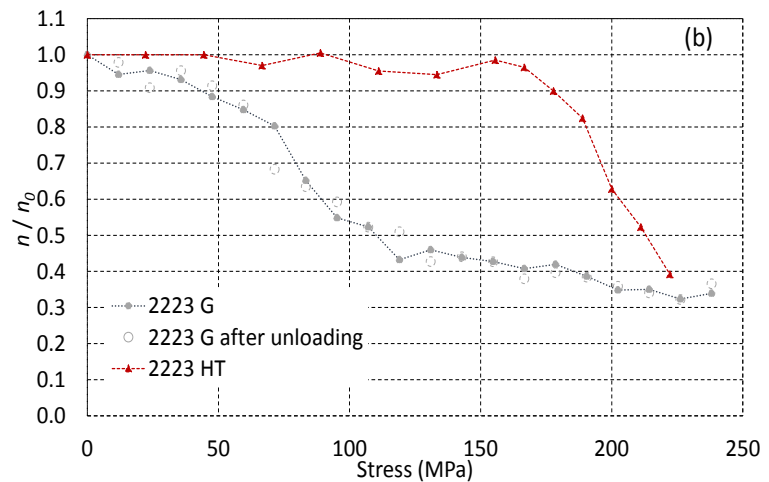
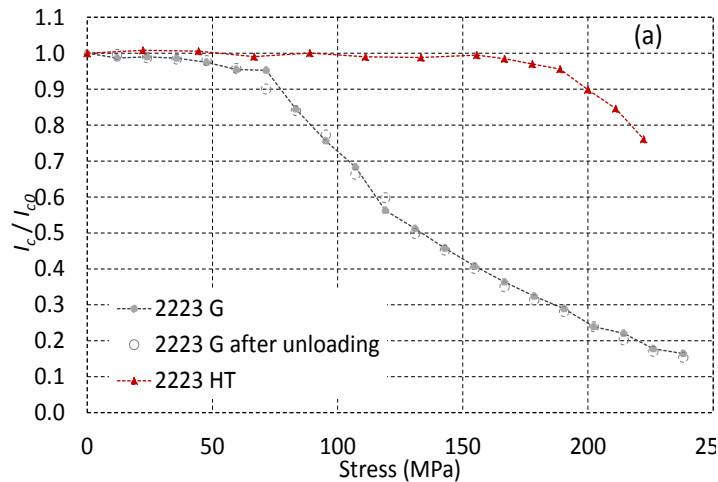
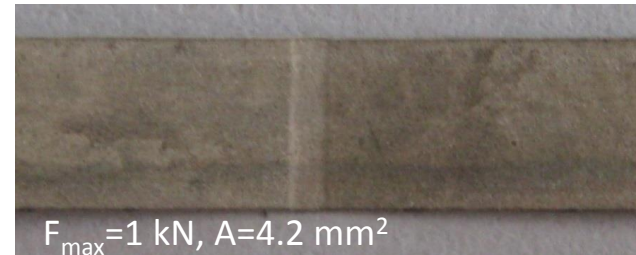


(a) Sample holder for I_c vs transverse stress measurements with (b) 1 mm-wide and (c) 5 mm-wide pressing tool.

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I_c vs transverse compressive stress of Bi-2223

- I_c degradation under transverse compressive stress is entirely irreversible.
- The reinforcement of the Bi-2223 tape is effective to increase its critical transverse stress to above 150 MPa.



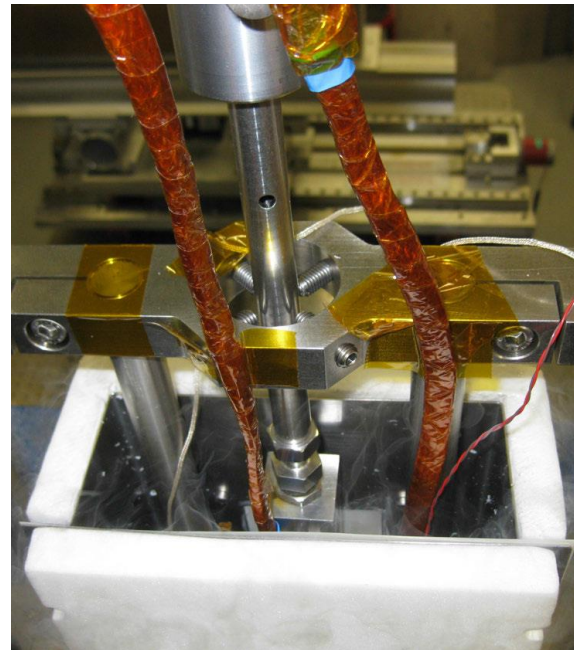
(a) I_c / I_{c0} and (b) n / n_0 vs transverse compressive stress applied using 1 mm-wide pressing tool onto the non reinforced (type G) and reinforced (type HT) Bi-2223 tapes.

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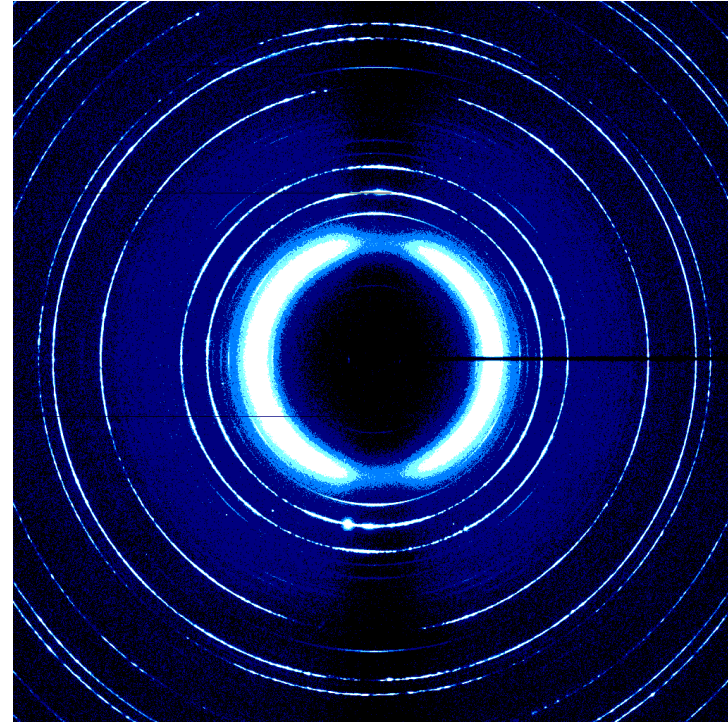
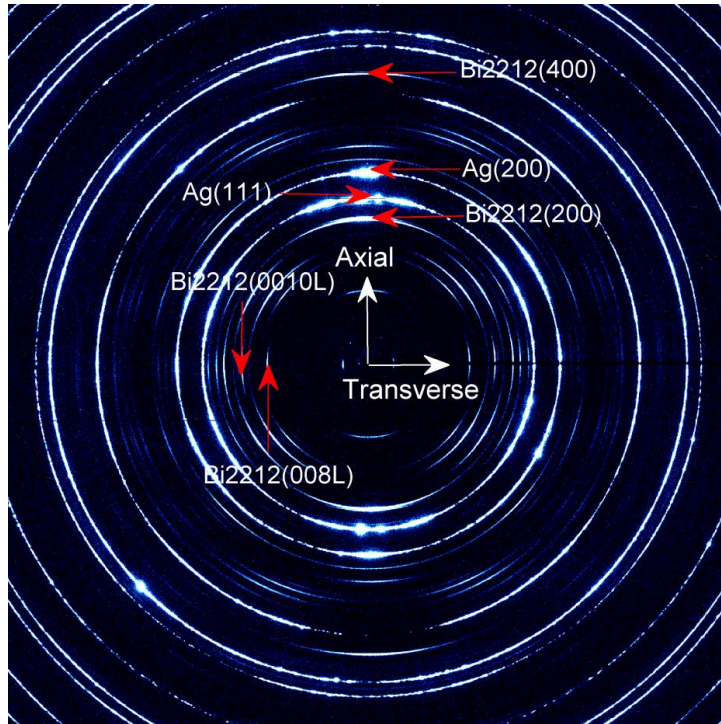
The X-ray transparent cryostat

- Dismountable cryostat made of stainless steel, encapsulated by Styrofoam. Windows are made of sheets of PET, in which X-ray absorption is negligible.
- Absorption of 90 keV X-rays in 7 cm of liquid nitrogen is about 50%.
- Refill of the cryostat is required after about 20 min.



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Diffraction pattern in air and in liquid nitrogen

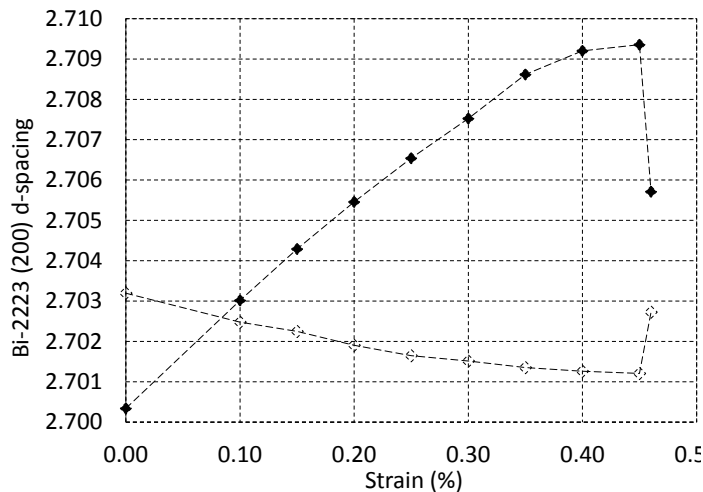


Diffraction pattern of the Bi-2212 wire in air.

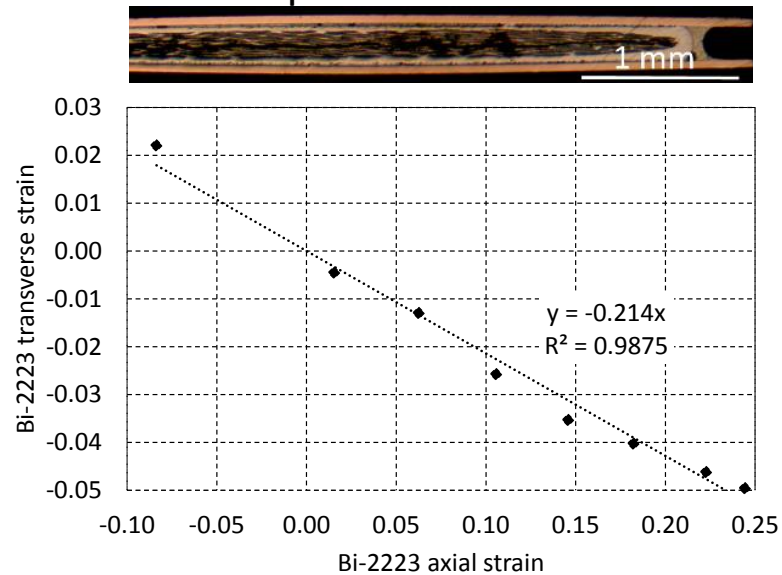
Diffraction pattern of the Bi-2212 wire in liquid nitrogen.

Elastic strain of the Bi-2223 filaments in the HT-CA tape at 77 K

- Before application of an external load the Bi-2223 filaments are in axial pre-compression of 0.09%.
- Linear elastic behaviour up to about 0.4% strain.
- Filament relaxation at 0.45% due to filament rupture.
- Bi-2223 Poisson ratio $\nu = 0.21$.



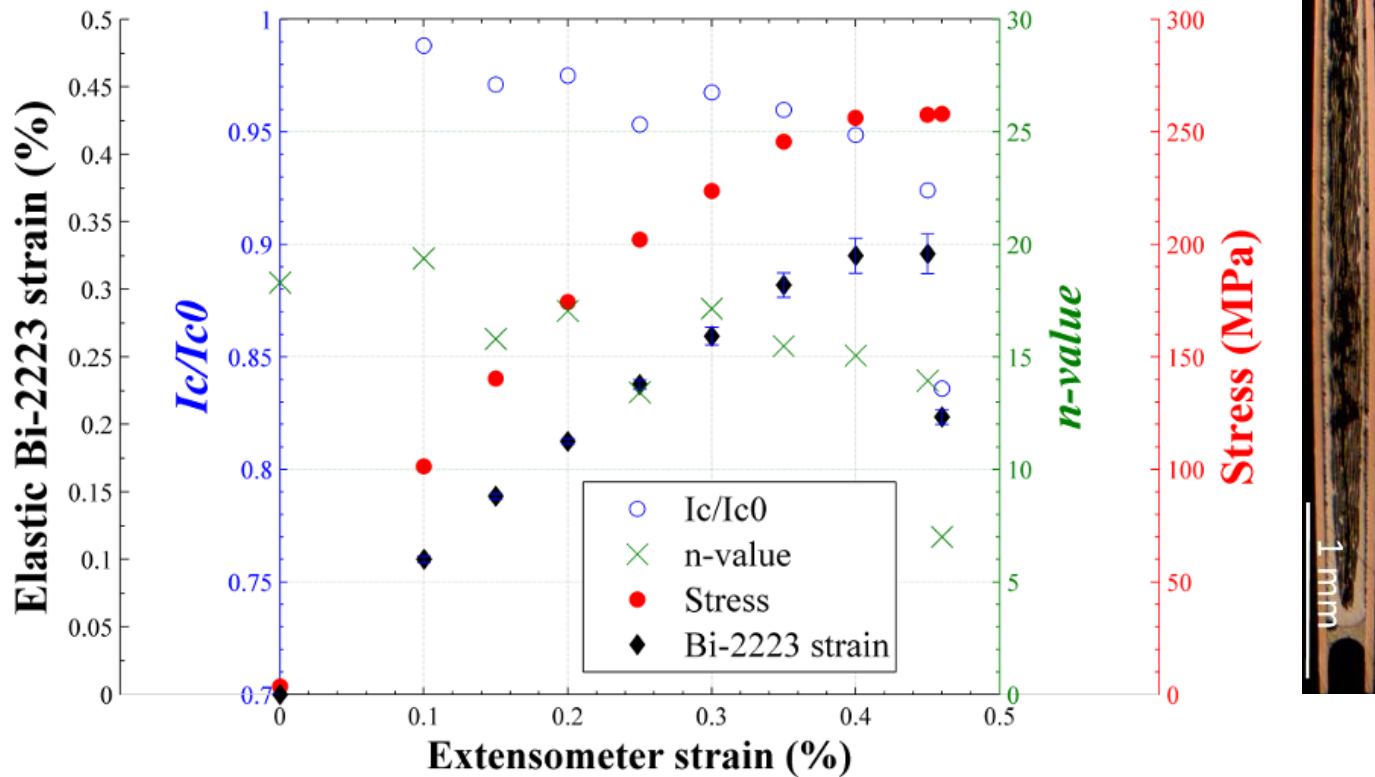
Axial and transverse Bi-2223 (200) d-spacing as a function of the extensometer strain at 77 K.



Transverse vs axial Bi-2223 elastic strain. Strain has been calculated assuming that the stress free Bi-2223(200) d-spacing is 2.7026 Å.

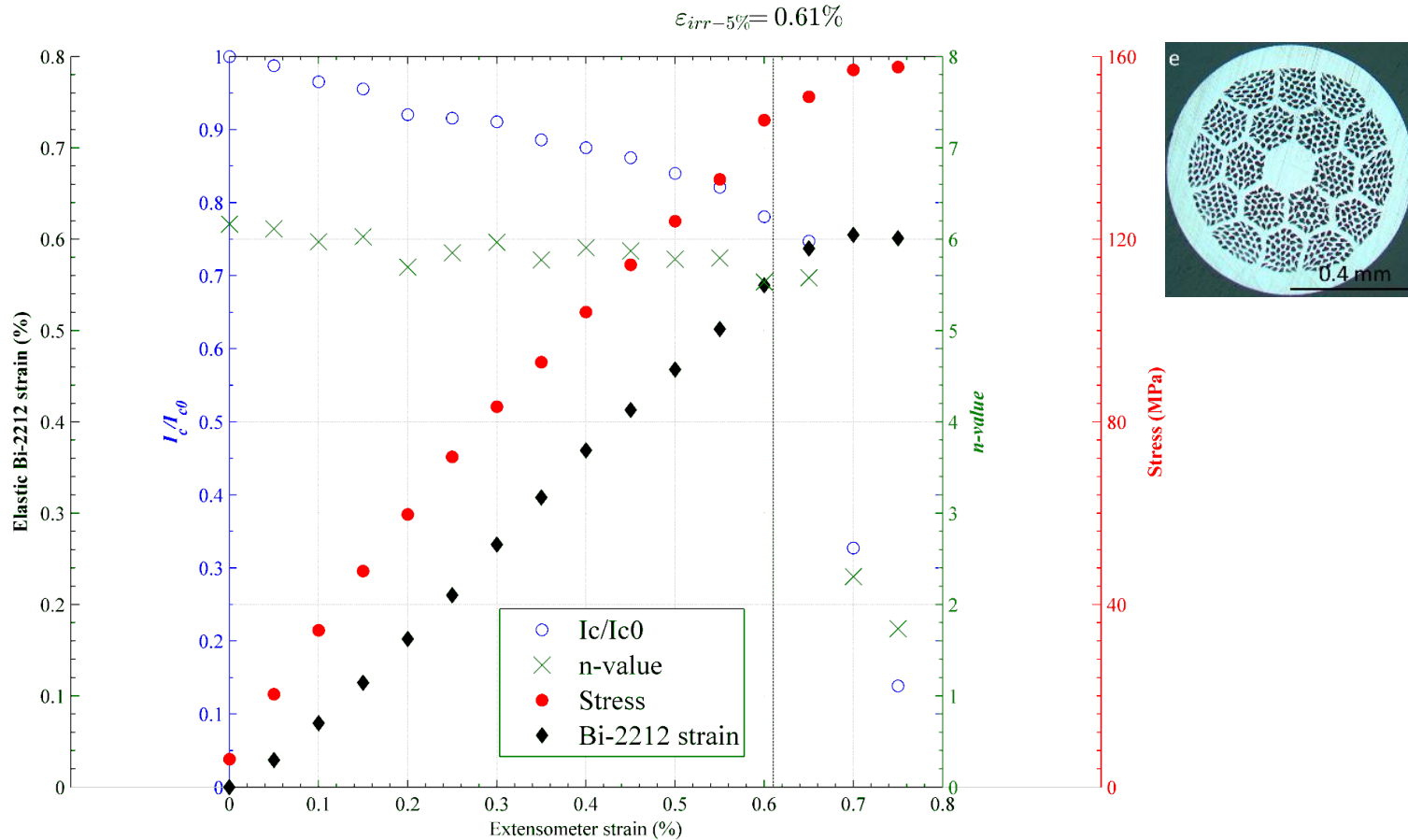
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I_c , n -value, Bi-2223 elastic strain and stress as a function of the macroscopic strain of the Bi-2223 HT-CA tape at 77 K



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I_c , n-value, Bi-2212 elastic strain and stress as a function of the macroscopic wire strain of the OP processed Bi-2212 wire at 77 K



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Conclusion

- An UTM, equipped for I_c measurements as a function of uniaxial tensile strain and transverse compressive stress in liquid nitrogen in self-field, is a convenient tool to study irreversible degradation of HTS.
- Equipping the UTM with a cryocooler would allow to study electromechanical properties of MgB_2 and Nb_3Sn too.
- Advantages of this set-up for studying irreversible I_c degradation of HTS are:
 - Samples do not need to be soldered onto a sample holder
 - The uniaxial tensile stress is measured and controlled during the entire experiment
 - Experiments can be comparatively fast
- Main limitations of the set-up are:
 - Measurements in applied field are not possible
 - Test currents are limited
 - I_c vs axial compression measurements are not possible.
- The UTM can be integrated in a high energy synchrotron beamline for simultaneous XRD, I_c , stress and strain measurements.