# Uncertainties in the measurement of the mechanical properties of Nb<sub>3</sub>Sn coil constituents and cable stacks

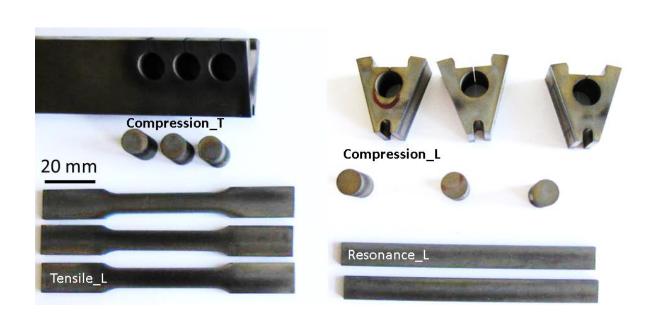
C. Scheuerlein, F. Lackner

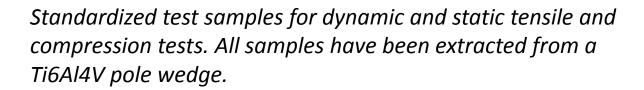
Nb<sub>3</sub>Sn Rutherford cable characterization for accelerator magnets workshop, 17<sup>th</sup> November 2017, CIEMAT, Madrid, Spain

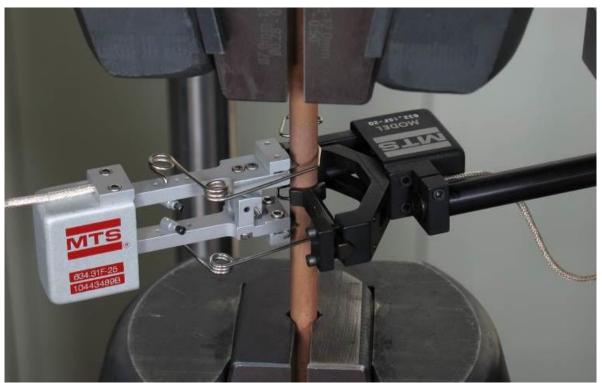
#### Outline

- Test sample geometries
- Uncertainties in dynamic Young's modulus measurements
- Uncertainties in stress-strain measurements using homogeneous samples with ideal testing geometry
  - Stress strain behaviour dependence on the load direction
  - Non linear stress-strain behaviour
  - Elastic anisotropy of the coil constituent materials
- Uncertainties in Nb<sub>3</sub>Sn composite wire stress-strain measurements
- Uncertainties in the determination of the Nb<sub>3</sub>Sn mechanical properties
- Uncertainties in stress-strain measurements of reacted and impregnated Nb<sub>3</sub>Sn Rutherford cable stacks
- Conclusion

#### Sample geometries for standardised mechanical testing



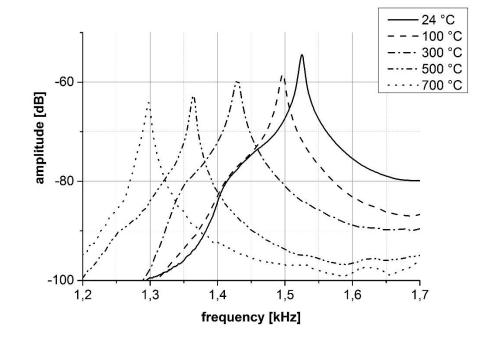




Sample for Poisson's ratio measurement and extensometers for axial and transverse strain measurements. The sample has been extracted from a DISCUP coil wedge.

### Dynamic Young's modulus measurements

- For dynamic test methods (resonance and impulse excitation) the key elements defining the measurement uncertainty are the mass and physical size of the specimen [i].
- For both test methods a measurement uncertainty < 1 % is realistic [ii,iii].
- Typical differences between dynamic and static Young's moduli (adiabatic vs isothermal modulus) are <1 % [ii].
- Since dynamic Young's modulus measurements are non destructive temperature dependent measurements are possible [iv].



Resonance peak of the first bending oscillation of rectangular 316LN specimen at different temperatures [iv].

[i] J.D. Lord, R. Morrell, NPL Good Practice Guide No. 98 "Elastic Modulus Measurement", NPL London 2006

[ii] F.H. Newmann, V.H.L. Searle, The general properties of matter. Issue 5, Edward Arnold, 1962

[iii] ASTM E 1875 "Standard Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio by Sonic Resonance". 2013, ASTM International.

[iv] C. Scheuerlein, F. Lackner, F. Savary, B. Rehmer, M. Finn, C. Meyer, "Thermomechanical behavior of the HL-LHC 11 Tesla Nb<sub>3</sub>Sn magnet coil constituents during reaction heat treatment", IEEE Trans. Appl. Supercond., submitted

# Stress-strain behaviour dependence on the load direction at the example of stainless steel

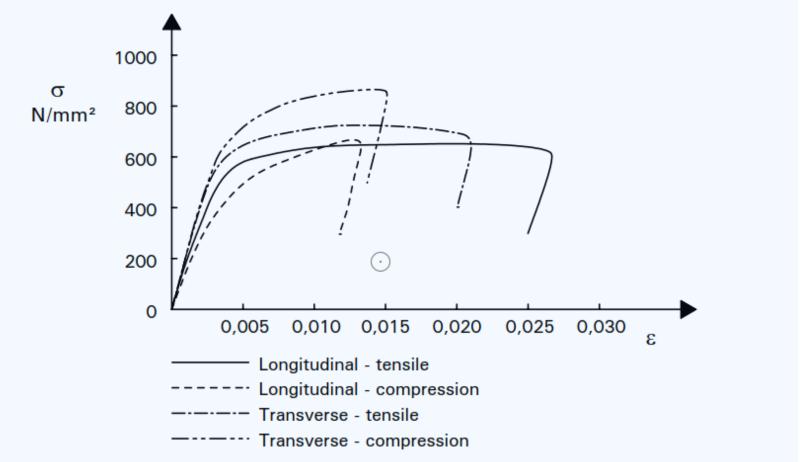
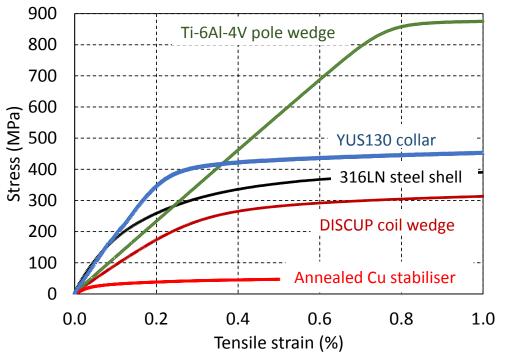


Figure 3.2 Typical stress-strain curves for grade 1.4318 cold worked to strength level C850

From: The European Stainless Steel Development Association Euro Inox and The Steel Construction Institute, Design manual for structural stainless steel-Commentary (2<sup>nd</sup> Edition), 2003

#### Uncertainties in static Young's modulus measurements

- Precise Young's moduli can be derived from stress-strain measurements of materials that exhibit pronounced linear elastic behaviour (e.g. Ti6Al4V).
- Comparatively large uncertainty in the Young's modulus results of materials that do not exhibit linear behaviour, like stainless steels.



1000 -E = 224 and 189 GPa E = 115 and 114 GPa σ [MPa] 500 316LN 316LN regression line --- Ti-6AI-4V - - Ti-6Al-4V regression line 0.0 0.2 0.4 0.6 0.8 1.0 ε [%]

Comparison of the RT engineering stress-strain curves of different coil and magnet materials [iv].

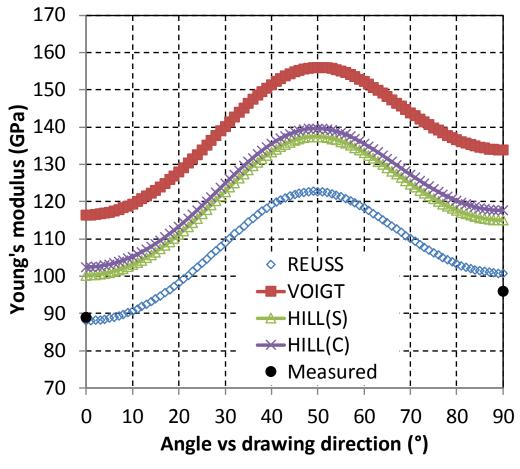
Comparison of 316LN and Ti6Al4V stress-strain curves and linear fits for the determination of Young's modulus [v].

[iv] C. Scheuerlein, F. Lackner, F. Savary, B. Rehmer, M. Finn, C. Meyer, "Thermomechanical behavior of the HL-LHC 11 Tesla Nb<sub>3</sub>Sn magnet coil constituents during reaction heat treatment", IEEE Trans. Appl. Supercond., submitted

[v] C. Scheuerlein, F. Lackner, F. Savary, B. Rehmer, M. Finn, P. Uhlemann, "Mechanical properties of the HL-LHC 11 Tesla Nb<sub>3</sub>Sn magnet constituent materials", IEEE Trans. Appl. Supercond., 27(4), (2017), 4003007

#### Elastic anisotropy in the 11 T dipole DISCUP coil wedges

- The DISCUP Young's moduli derived from stress-strain compression tests are 89 GPa in the wedge extrusion direction and 96 GPa perpendicular to the extrusion direction [v].
- These values are substantially lower than the values between 115 to 130 GPa found for ODS Copper in literature.
- The angular dependence of Young's modulus has been calculated from texture data obtained by neutron diffraction and from Cu single crystal elastic constants.
- The DISCUP wedges are strongly textured (multiples of random orientation MRD=16), which causes a strong elastic anisotropy of about 30%.



Angular DISCUP Young's modulus dependence with respect to the wedge extrusion direction. Calculated assuming equal strains (Voigt) and equal stresses (Reuss) in all grains, respectively. Measurement results from [v] are shown for comparison. Courtesy of W. Gan, Helmholtz-Zentrum Geesthacht.

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### Summary: Uncertainties in Young's modulus measurement with ideal test samples, according to well established standards

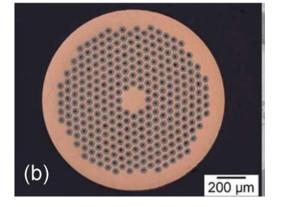
- Dynamic Young's modulus measurement uncertainty <1 %.</li>
- Static Young's modulus measurement by tensile tests uncertainty is typically 3% [ii], provided the material exhibits pronounced linear elastic behaviour.
- Static Young's modulus measurement by compression tests uncertainty is typically 5% [iii].
- For materials that do not exhibit pronounced linear stress strain behaviour (e.g. stainless steel and annealed copper), the uncertainty of the E-modulus derived from static tests can be >10% [v,vi].
- Important elastic anisotropy of textured coil materials (as an example DISCUP elastic anisotropy is>30%).

[vi] Ch. Weißmüller, H. Frenz, "Messunsicherheit bei der Ermittlung des E-Moduls im Zugversuch an Stahl", Materials Testing, vol. 55, no. 9, 2013, pp. 643-647

Uncertainties in stress-strain measurements of

Nb<sub>3</sub>Sn/Cu composite wires

- The most easy to study wire load case is uniaxial tensile loading in the wire drawing direction.
- Nb<sub>3</sub>Sn/Cu composite wires are not suitable tensile test samples.
  - the wire constituents are not stress free
  - the filaments contain porosity
  - at zero axial load the wires are always bended
- No linear part in the loading stress-strain curve.
  Therefore, the wire stiffness is estimated from the linear slope of unloading curves.
- The Nb<sub>3</sub>Sn/Cu wire stiffness is stress and strain dependent, even in the wire drawing direction.



Strain (%)	Stiffness (GPa)
0.02	139
0.04	120
0.06	117
0.08	118
0.10	118
0.12	116
0.14	117
0.16	117

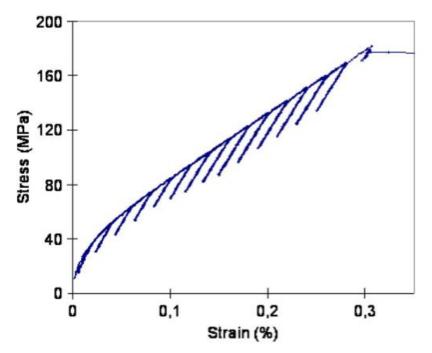
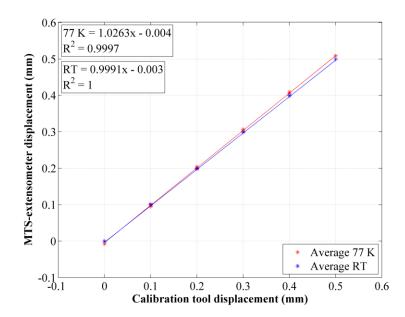


Fig. 2. Engineering stress-strain curve of the reacted PIT B215 composite strand measured at RT. The stress has been partly released every 0.02% in order to measure the apparent composite E-modulus as a function of axial composite strain. Courtesy B. Rehmer and M. Finn, Federal Laboratory for Materials Research, Berlin.

#### Extensometry for wire uniaxial stress-strain measurements

- The precision of stress-strain measurements relies mainly on careful extensometer strain measurements.
- Extensometers for strain measurements in wire drawing direction are commercially available.
- Extensometer calibration at RT can be performed by the supplier.
- Calibration at 77K and 4.2 K is not easily available and requires the setting up of dedicated calibration tools [vii].





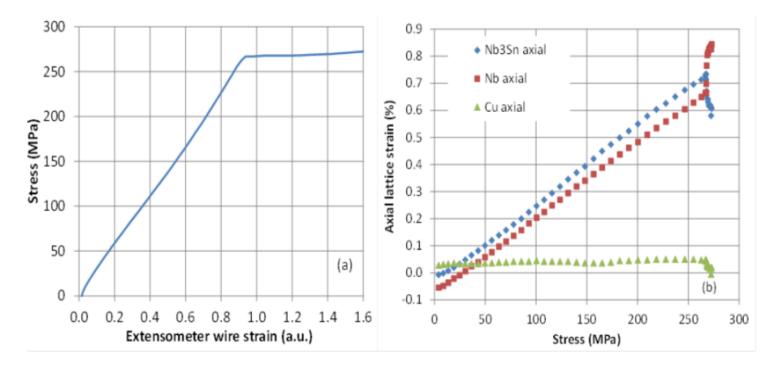


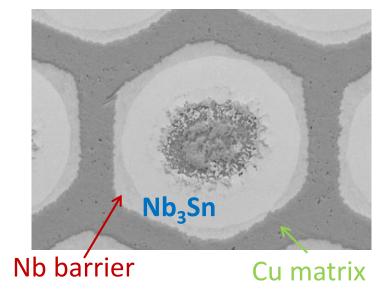
(a) MTS-clip on extensometer type 632.27F-21 with gauge length of 25 mm and (b) calibration tool.

### Mechanical behaviour of Nb<sub>3</sub>Sn/Cu PIT wire constituents studied by simultaneous stress-strain-XRD measurements at 4.2 K

The effect of different wire constituents on the wire mechanical behaviour can be understood from combined X-ray diffraction-stress-strain results:

- Almost no load is carried by the annealed Cu matrix
- Linear elastic behaviour of Nb and Nb<sub>3</sub>Sn
- Nb and Nb<sub>3</sub>Sn axial pre-compression in the unloaded wire
- Load transfer from Nb<sub>3</sub>Sn to Nb at a wire stress of about 270 MPa and a strain of about 0.7%





[viii] C. Scheuerlein, M. Di Michiel, F. Buta, B. Seeber, C. Senatore, R. Flükiger, T. Siegrist, T. Besara, J. Kadar, B. Bordini, A. Ballarino, L. Bottura, "Stress distribution and lattice distortions in Nb<sub>3</sub>Sn/Cu multifilament wires under uniaxial tensile loading at 4.2 K", Supercond. Sci. Technol. 27, (2014), Q44021.

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

### Summary: Sources of uncertainty in uniaxial tensile stressstrain measurements of Nb<sub>3</sub>Sn/Cu composite wires

- Nb<sub>3</sub>Sn/Cu composite wires are not suitable for precise tensile tests. Their constituents are not stress free, the filaments contain porosity and at zero axial load the wires are always bended.
- There is no linear part in the wire loading stress-strain curves.
- The wire stiffness is commonly estimated from the linear slope of unloading stress-strain curves. The slope of these curves is stress and strain dependent.
- Wire stiffness results depend strongly on test and data analysis procedures.
- Wire stress-strain results cannot be easily compared with results obtained with alternative test methods.
- Only few laboratories perform the challenging stress-strain measurements at 4.2 K.

### Nb<sub>3</sub>Sn mechanical properties at RT

- Published RT Nb<sub>3</sub>Sn Young's modulus values vary between 32 GPa to 165 GPa.
- Measuring the mechanical properties of Nb<sub>3</sub>Sn is very tricky because:
  - The intermetallic phase Nb<sub>3</sub>Sn is stable with Sn contents ranging from about 18-25 at.%.
  - Preparation of Nb<sub>3</sub>Sn bulk samples which properties are representative for the Nb<sub>3</sub>Sn filaments in multifilament wires is extremely difficult (e.g. because of different Sn gradients, alloying elements, grain size and orientation distribution, and presence of contaminating phases).
  - Determining Nb<sub>3</sub>Sn/Nb sub-element Young's moduli from tensile tests, for instance using extracted (brittle) Nb<sub>3</sub>Sn filaments, is very delicate.
  - The filaments are a composite, containing Nb<sub>3</sub>Sn, unreacted Nb diffusion barriers, porosity and other material. For such materials rule-of-mixture estimates may not be appropriate.

### The unusual temperature dependence of the Nb<sub>3</sub>Sn mechanical properties and calculation of Nb<sub>3</sub>Sn elastic anisotropy in PIT and RRP type wires

- During cooldown Nb<sub>3</sub>Sn undergoes a phase transformation at roughly 40 K, which causes a lattice softening.
- This is for instance revealed by the slope of the stress-strain curves acquired at RT and at 4.2 K [viii].
- The Nb<sub>3</sub>Sn elastic properties in PIT and RRP wires can be calculated using the Nb<sub>3</sub>Sn grain orientation distribution and published Nb<sub>3</sub>Sn single crystal elastic constants.
- The results illustrate the different elastic anisotropy in RRP and PIT type wires.
- How reliable are the Nb<sub>3</sub>Sn single crystal elastic constants?

TABLE II  $Nb_3Sn$  Elastic Moduli in Axial and Transverse Directions Calculated for the RRP and PIT Wires at RT and at 4.2 K

		PIT B215	RRP #7419
RT	$E_{axial}$	130	140
	$E_{trans}$	135	129
4.2 K	Eaxial	106	127
	$E_{trans}$	116	104

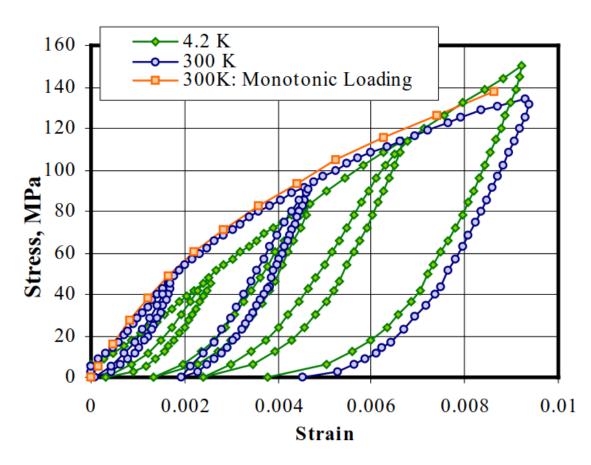
From: "Elastic Anisotropy in Multifilament  $Nb_3Sn$  Superconducting Wires", IEEE Trans Appl. Supercond., 25(3), (2015), 8400605

### Summary: Uncertainties of Nb<sub>3</sub>Sn mechanical properties at RT and 4.2 K

- Preparation of Nb<sub>3</sub>Sn bulk samples which are representative for the Nb<sub>3</sub>Sn in wires for mechanical testing is nearly impossible.
- The mechanical Nb<sub>3</sub>Sn properties in the filaments of IT, PIT and Bronze route wires are different.
- Nb<sub>3</sub>Sn Young's moduli calculations rely on the availability of accurate single crystal elastic constants.
- During cooling to below 77 K a Nb<sub>3</sub>Sn lattice softening occurs. 77 K mechanical test results do not allow to predict 4.2 K Nb<sub>3</sub>Sn mechanical properties.

## Transverse compression test of reacted and impregnated Nb<sub>3</sub>Sn Rutherford cable stacks

- Transverse compressive loading of a composite is a highly complex load case.
- Transverse compression tests of reacted and impregnated Nb<sub>3</sub>Sn Rutherford cable stacks can provide qualitative estimates of the stress-strain behaviour of the Nb<sub>3</sub>Sn conductor in magnet coils under monotonic and cyclic azimuthal loading.
- Reacted and impregnated Nb<sub>3</sub>Sn Rutherford cable stacks are sometimes also used to determine a socalled "Elastic modulus" of the Nb<sub>3</sub>Sn conductor in magnet coils.
- Since Rutherford cable stacks do not exhibit linear elastic behaviour at any stress level, the use of the term "Elastic modulus" in the context of stressstrain measurements of Nb<sub>3</sub>Sn cable stacks is misleading.



**Figure 6**: Load-Unload-Reload Tests.

From: D.R. Chichili, T.T. Arkan, I. Terechkine, J.A. Rice, "Niobium-Tin Magnet Technology Development at Fermilab", Proceedings of the 1999 Particle Accelerator Conference, New York, 1999

### Sources of uncertainties in transverse compression experiments using reacted and impregnated Nb<sub>3</sub>Sn Rutherford cable stacks

- Geometry of Rutherford cable stacks is not suited for precise mechanical testing.
- Stiffness of Rutherford cable stacks is not uniform across the loaded area.
- Geometry of Rutherford cable stacks is only partly representative for the conductor in magnet coils.
- Strong influence of load rate and mechanical cycling on stress-strain behaviour.
- No standard procedures available. Very strong influence of data analysis procedures on stiffness results.
- Stiffness depends on the applied strain and stress.
- Very strong influence of the load direction on stress-strain behaviour.
- Integration of an extensometer in the test set-up is in many cases not possible.
- No easy cross check with other mechanical test methods is possible.

#### Conclusion

- Nb<sub>3</sub>Sn coil and magnet materials properties are anisotropic and load direction dependent.
- Highly accurate Young's moduli can be obtained from dynamic measurements.
- In favourable cases (e.g. Ti6Al4V) Young's moduli can be derived from static stressstrain measurements, using standardised test sample geometries and test procedures.
- Transverse compression tests of reacted and impregnated Nb<sub>3</sub>Sn Rutherford cable stacks can provide qualitative estimates that may be representative for the stressstrain behaviour of the Nb<sub>3</sub>Sn conductor in magnet coils under monotonic and cyclic loading.
- Results of transverse compression tests of reacted and impregnated Nb<sub>3</sub>Sn Rutherford cable stacks cannot be used to precisely predict stress levels in magnets.
- The use of the term Young's modulus or elastic modulus in the context of stress-strain measurements of Rutherford cable stacks is misleading.
- Stress-strain tests at 77 K do not allow to predict Nb<sub>3</sub>Sn behaviour at 4.2 K.

### Back-up slides

### Calculation of Young's modulus from resonance frequencies

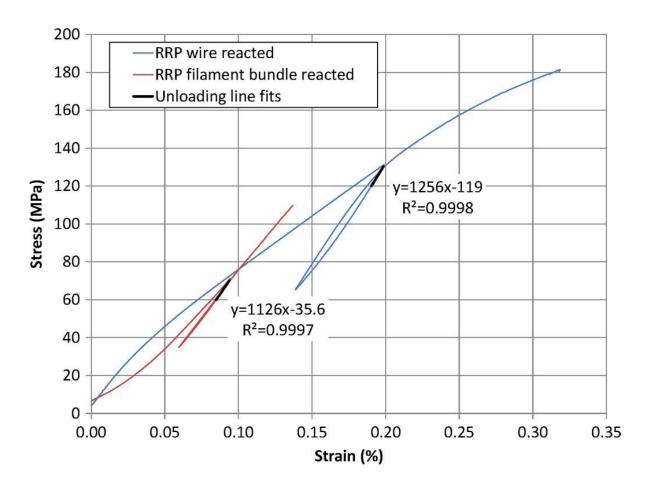
For long thin beams with rectangular cross section the Young's modulus (E) is calculated from the mass (m), length (L), width (b) and thickness (t) of the beam, and its fundamental frequency in bending  $(f_f)$ .

$$E = 0.9465 \left(\frac{mf_f^2}{b}\right) \left(\frac{L^3}{t^3}\right) T$$

where

$$T = 1 + 6.858 \left(\frac{t}{L}\right)^2$$

### Uniaxial tensile testing of extracted Nb<sub>3</sub>Sn filaments



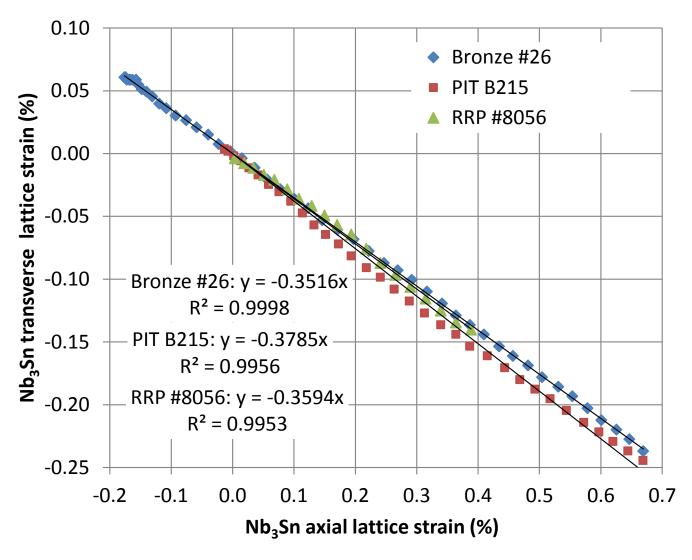
Stress-strain curves of a reacted RRP wire and its extracted filaments measured at room temperature. From: IEEE TAS, 25(3), (2015), 8400605

# Summary of 11 T dipole materials elastic and plastic properties

Material	R <sub>p0.2</sub> (MPa)	R <sub>m</sub> (MPa)	A (%)	Z (%)	Young's modulus dynamic (GPa)		Young's modulus static (GPa)	
					Resonance	Impulse	Tensile	Compr.
316LN_L	351±12	674±2	54±5	63±2	191±0.3	191±0.3	183±7	n.m.
316LN_T	324±4	658±1	53±5	63±1	n.m.	n.m.	203±15	n.m.
Magnetil_L	117±3	241±1	n.m.	73±3	196±0.8	196±0.3	208±19	n.m.
Magnetil_T	124±2	267±1	47±2	71±4	219±0.1	218±0.3	213±3	n.m.
YUS-130_L	401	793	71	66	196±0.9	196±0.3	196	n.m.
YUS-130_T	415	749	51	66	193±1.1	192±0.3	189	n.m.
Ti-6Al-4V_L	868±5	930±12	17±1	28±2	115	114	115±1	120±2
Ti-6Al-4V_T	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	116±2
DISCUP no-HT_L	332±2	387±1	22±2	43±4	92.9±0.2	93.8±0.3	87±1	89±1
DISCUP no-HT_T	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	n.m.	96±2
DISCUP HT_L	284±1	376±1	26±1	48±1	96.7	96.3	91±1	87±2

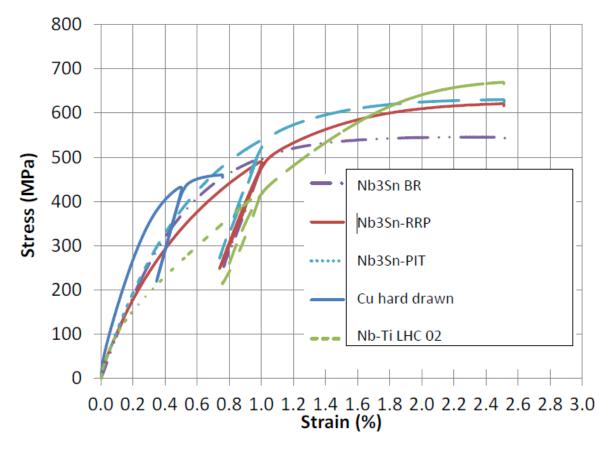
[v] C. Scheuerlein, F. Lackner, F. Savary, B. Rehmer, M. Finn, P. Uhlemann, "Mechanical properties of the HL-LHC 11 Tesla Nb<sub>3</sub>Sn magnet constituent materials", IEEE Trans. Appl. Supercond., 27(4), (2017), 4003007

### Nb<sub>3</sub>Sn Poisson ratio at 4.2 K



Comparison of the 4.2 K  $Nb_3$ Sn Poisson ratio in the BR, PIT and RRP wire. From: Supercond. Sci. Technol. **27** (2014) 044021 (7pp)

### Mechanical properties of unreacted Nb<sub>3</sub>Sn wires



Comparison of the stress-strain curves of unreacted  $Nb_3Sn$  (PIT, RRP and BR), hard drawn Cu and LHC Nb-Ti wire.