Thermomechanical behaviour of Nb$_3$Sn magnet constituent materials

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Outline

• Ingredients for the thermomechanical modelling of linear elastic materials behaviour
  • Linear elastic and isotropic materials behaviour
  • Thermal expansion coefficients
  • Friction coefficients

• Additional challenges in the modelling of the HL-LHC Nb₃Sn magnet thermomechanical behaviour
  • Elastic anisotropy
  • Cu yielding and flowing
  • Nb₃Sn brittleness and low temperature lattice softening
  • Nb₃Sn conductor non linear and irreversible thermal expansion

• Nb₃Sn conductor block mechanical behaviour
  • Young’s moduli in axial, transverse and radial direction
  • Nb₃Sn and Cu loading strain and stress

• Measuring strain and stress distribution in 11 T dipole coils
  • Residual strain measurement
  • Post mortem metallographic characterisation

• Conclusion
Elastic modulus definitions

“Specifying how stress and strain are to be measured, including directions, allows for many types of elastic moduli to be defined. The three primary ones are:”

• “Young's modulus (E), is a mechanical property that measures the stiffness of a solid material .... in the linear elasticity regime of a uniaxial deformation.

• The shear modulus (G) describes an object's tendency to shear (the deformation of shape at constant volume).

• The bulk modulus (K) describes the material's response to (uniform) hydrostatic pressure.“

From: https://en.wikipedia.org/wiki/Elastic_modulus
Young’s modulus vs tangent and chord moduli

• Young’s modulus is a materials property (which ideally is independent on strain and stress).

• For materials that exhibit nonlinear stress-strain behaviour (for instance stainless steel), the value of tangent or chord modulus is sometimes used to describe the magnitude of strain at a certain applied stress.

• Tangent modulus is stress dependent and chord modulus is stress range dependent.

https://cds.cern.ch/record/1463298/files/BO2kndPxouGDY.pdf?
Samples for Young’s modulus measurements

- Dynamic tests with long thin beams with rectangular cross section
  (3 mm × 9 mm × 100 mm)
- Uniaxial static tensile tests with flat samples DIN 50125-E3 × 8 × 30
- Uniaxial static compression tests with cylinder samples Ø 10 mm, height 15 mm

Standardized test samples for dynamic and static tensile and compression tests. All samples have been extracted from a Ti-6Al-4V pole wedge by electrical discharge machining. Courtesy CERN central workshop team.
Dynamic E-modulus measurements

- Highly accurate Young’s moduli can be derived from dynamic tests.
- Measurement uncertainty is mainly defined by the mass and the dimensions of the specimen [1].
- A measurement uncertainty < 1 % is realistic [2,3].
- Typical differences between dynamic and static Young’s moduli (adiabatic vs isothermal modulus) are <1 % [2].
- Temperature dependent dynamic Young’s modulus measurements are possible when the thermal expansion coefficients are precisely known.

Static Young’s modulus measurement in tension and compression

• For the materials discussed here, stress strain curves in tension and compression are nearly identical.

• Direct strain measurement using an extensometer is crucial.

• Static Young’s modulus measurement by tensile tests uncertainty is typically 3%.

• For materials that do not exhibit pronounced linear stress strain behaviour (e.g. stainless steel), the uncertainty of the E-modulus derived from static tests can be >10% [5].

• Static Young’s modulus measurement by compression tests uncertainty is typically 5%.

• Additional constraints for compression tests:
  • Sample height to diameter ratio should be small in order to avoid buckling.
  • Friction between contact surfaces needs to be minimised.

Comparison of tensile and compressive stress-strain curves of Ti-6Al-4V and DISCUP up to 0.5 % strain [iv].

Relation of elastic constants in homogeneous isotropic materials

- The Young’s modulus ($E$), shear modulus ($G$), bulk modulus ($K$) and Poisson’s ratio ($\nu$) of homogeneous isotropic materials are related (Equation 1):

$$E = 2G(1+\nu) = 3K(1-2\nu)$$  

Equation 1

Comparison of static and dynamic Young’s moduli ($E$), dynamic shear modulus ($G$), Poisson’s ratio ($\nu$) measured by extensometry, and $\nu$ calculated from $E$ and $G$ ($\nu=E/2G-1$).

<table>
<thead>
<tr>
<th>Sample</th>
<th>E-static tension (GPa)</th>
<th>E-static compr. (GPa)</th>
<th>E-dynamic (GPa)</th>
<th>G-dynamic (GPa)</th>
<th>$\nu=(E/2G-1)$</th>
<th>$\nu$ measured</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti-6Al-4V</td>
<td>115±1 (L)</td>
<td>120±2 (L)</td>
<td>115 (L)</td>
<td>43.6</td>
<td>0.32</td>
<td>0.32±0.03</td>
</tr>
<tr>
<td>Al 7175</td>
<td>68.9</td>
<td>not measured</td>
<td>68.5</td>
<td>25.4</td>
<td>0.35</td>
<td>0.33 [7]</td>
</tr>
<tr>
<td>316 LN</td>
<td>183±7 (L)</td>
<td>not measured</td>
<td>191 (L)</td>
<td>74.5</td>
<td>0.28</td>
<td>0.25 [8]</td>
</tr>
</tbody>
</table>

[6] $E=114$ GPa, $G=42.1$ GPa, and $\nu=0.34$ reported in: [http://www.matweb.com/search/DataSheet.aspx?MatGUID=10d463eb3d3d4ff48fc57e0ad1037434](http://www.matweb.com/search/DataSheet.aspx?MatGUID=10d463eb3d3d4ff48fc57e0ad1037434)
[7] $E=72$ GPa, $G=27$ GPa, and $\nu=0.33$ reported in: [http://www.matweb.com/search/datasheet_print.aspx?matguid=13ca3d21315b4632b4330965f0e01c31](http://www.matweb.com/search/datasheet_print.aspx?matguid=13ca3d21315b4632b4330965f0e01c31)
Young’s modulus temperature dependence-example Ti-6Al-4V

• Young’s modulus is determined by the binding forces between atoms.
• Young’s modulus is not strongly influenced by alloying elements and by the degree of strain work.
• Young’s modulus is temperature dependent (for many metals it is at 4.2 K roughly 10% higher than at RT.

Fig. 14: Tensile stress–strain curve of annealed Ti-6Al-4V at different temperatures (from Ref. [9])

Resonance test for measuring the temperature dependence of $E$, $G$, and $\nu$

- During $\text{Nb}_3\text{Sn}$ reaction heat treatment the coil materials and the reaction mold are heated to typically 650 °C.
- The temperature dependence of the elastic properties of different coil and reaction mold materials has been derived from the resonance frequencies measured \textit{in situ} during heat cycles in a vacuum furnace.

Stainless steel $\text{316LN}$, Ti-6Al-4V and DISCUP C30 temperature dependence of (a) Young’s modulus ($E$) and (b) shear modulus ($G$). (c) Poisson’s ratio ($\nu$) temperature dependence of $\text{316LN}$ and Ti6Al4V [4].
Linear thermal expansion coefficients

Coefficient of thermal expansion of different polymers and metallic magnet constituents (reference temperature $T_0=20\, ^\circ C$).

Relative length changes of Cu, DISCUP C3/30, Ti-6Al-4V, stainless steel 316LN and Nb (literature). Reference temperature $T_0=20\, ^\circ C$ [4].
Friction coefficients

- Example: Ti-6Al-4V against 316LN under conditions close to those between the pole wedge and the stainless-steel shims of the 11 T Nb$_3$Sn magnets.
- At RT in air stable sliding with a friction coefficient of about 0.4.
- At 4.2 K strong stick-slip effect.
- Application of the solid lubricant MoS$_2$ (spray) lowers the friction coefficient to about 0.08 [10].
- Friction can be further reduced by an optimized coating method, for instance with sputtered MoS$_2$ coatings [11].


Additional challenges in the modelling of the HL-LHC Nb$_3$Sn magnet thermomechanical behaviour

- Elastic anisotropy
- Yielding and flowing
- Nb$_3$Sn brittleness and low temperature lattice softening
- Non linear and irreversible Nb$_3$Sn conductor thermal expansion
Calculation of the angular dependence of the Young’s moduli: The models of Reuss, Voigt and Hill

• In order to determine the angular dependence of Young’s moduli, neutron texture measurements have been performed and elastic anisotropy was calculated from the diffraction data and single crystal elastic constants (courtesy W. Gan, STRESS-SPEC materials science beamline at MLZ).

• Young’s moduli of polycrystalline materials can be calculated with the assumptions that either the strain (Voigt) [12] or the stress (Reuss) [13] are constant in the entire material.

• In most cases, the experimentally determined Young’s moduli of polycrystalline metals are between the upper (Voigt) and lower (Reuss) bounds predicted by these models.

• For many materials, the experimentally determined Young’s moduli are close to those calculated according to the Hill model (arithmetic mean of the Reuss and Voigt values) [14].

Magnetil yoke and YUS-130 collar elastic anisotropy

- The yoke of the LHC dipole and the 11 T dipole is made of hot rolled sheets of an ultralow carbon steel (trademark “Magnetil”). The texturing produced by the rolling process is optimised for the magnetic properties of the yoke.
- The 3 mm-thick collars of the 11 T dipole magnets are made by fine-blanking of austenitic steel X8CrMnNiN19-11-6 sheet (tradename YUS-130)
- Young’s moduli measured by the resonance method are within the upper and lower bounds calculated according to Reuss and Voigt, and are close to the calculated Hill values \(E_{\text{magnetil-L}}=196\ \text{GPa}, \ E_{\text{magnetil-T}}=219\ \text{GPa}, \ \text{and} \ E_{\text{YUS-130L}}=196\ \text{GPa},\ E_{\text{YUS-130T}}=192\ \text{GPa}\).

\[\text{Fig. 3. Magnetil Young's modulus as a function of the angle with respect to the rolling direction L (at 0°), whereby T is the transverse direction. The circular symbols indicate the experimentally determined Young's moduli in L and T directions [4].}\]

\[\text{Fig. 5. YUS-130 Young's modulus as a function of the angle with respect to the rolling direction L (at 0°), whereby T is the transverse direction. The circular symbols indicate the experimentally determined Young's moduli in L and T directions [4].}\]

DISCUP coil wedges elastic anisotropy

- The strong texture causes an elastic anisotropy of about 40%.
- The simple relation between $E$, $G$ and $\nu$ does not apply in the case of anisotropic materials (note the Poisson’s ratio calculated with $\nu=E/2G-1$ is negative).
- The DISCUP Poisson’s ratio determined directly by extensometry is $\nu=0.43$.

<table>
<thead>
<tr>
<th></th>
<th>$E$-tension (GPa)</th>
<th>$E$-compr. (GPa)</th>
<th>$E$-dyn. (GPa)</th>
<th>$G$-dyn. (GPa)</th>
<th>$\nu=E/2G-1$</th>
<th>$\nu$ meas.</th>
</tr>
</thead>
<tbody>
<tr>
<td>DISCUP C30/3</td>
<td>96.5 (L)</td>
<td>91±1</td>
<td>87±2</td>
<td>54</td>
<td>-0.2</td>
<td>0.43±0.02</td>
</tr>
</tbody>
</table>

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. Young’s moduli derived from stress-strain compression tests are shown for comparison [15].
Additional challenges in the modelling of the HL-LHC Nb$_3$Sn magnet thermomechanical behaviour

• Elastic anisotropy
• Yielding and flowing
• Nb$_3$Sn brittleness and low temperature lattice softening
• Non linear and irreversible Nb$_3$Sn conductor thermal expansion
Yielding magnet materials

- Most magnet materials do not exhibit linear elastic behaviour, even at low loads.
- In particular the yielding of annealed Cu causes a major challenge in the modelling of Nb$_3$Sn accelerator magnets thermomechanical behaviour.

*RT engineering stress-strain curves of different HL-LHC magnet materials*
Mechanical properties of pure Cu

• Mechanical properties of strain hardened Cu are drastically changed during annealing heat treatment.
• Fully annealed high purity Cu starts to yield at very low stress in the order of 10 MPa.
• Annealed Cu is a strain hardening material, i.e. its yield stress increases with increasing plastic deformation.
• Cu flows (creeps) when the flow stress (the applied stress needed to maintain plastic flow of a material) is exceeded.

Stress-strain curves of Cu wire cold-drawn and after subsequent 695 °C HT [16].

<table>
<thead>
<tr>
<th></th>
<th>E_a (GPa)</th>
<th>R_p0.2 (MPa)</th>
<th>R_m (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cold-worked Cu wire</td>
<td>127 ± 0.8</td>
<td>397 ± 15</td>
<td>427 ± 5.9</td>
</tr>
<tr>
<td>Annealed Cu wire</td>
<td>108 ± 2.1</td>
<td>46.2 ± 2.6</td>
<td>204 ± 1.6</td>
</tr>
</tbody>
</table>

Strain hardening of Cu

Effect of Cyclic loading of copper in tension and compression:
• (A) Strain worked copper.
• (B) Annealed copper: the initial yield surface expands (significant isotropic hardening).

Hysteresis loops measured during cyclic reversed strain controlled (±0.55% strain) loading of (A) strain hardened and (B) annealed copper [17].

Monotonic stress strain curve and unloading hysteresis loop of annealed Cu wire [16].

Measuring Young’s modulus from linear part of the hysteresis loop

• Young’s modulus of materials that do not exhibit pronounced linear elastic behaviour is often determined from the slope of unloading stress-strain curves.

• When stress is released the elastic strain is recovered, but also reverse plastic straining may occur upon unloading (the Bauschinger strain).

• In order to measure Young’s modulus the slope of the initial straight part of the unloading curve is measured.

• Reverse plastic straining during unloading is also observed in composites.

https://lib.dr.iastate.edu/cgi/viewcontent.cgi?referer=https://www.google.de/&httpredir=1&article=4151&context=rtd
Additional challenges in the modelling of the HL-LHC Nb$_3$Sn magnet thermomechanical behaviour

- Elastic anisotropy
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Mechanical behaviour of Nb$_3$Sn wire constituents under tensile axial loading

Combined X-ray diffraction and uniaxial tensile stress-strain measurements at 4.2 K [19]:

- Almost no load is carried by the annealed Cu matrix
- Linear elastic behaviour of Nb and Nb$_3$Sn
- Brittle Nb$_3$Sn fracture at a wire stress of about 270 MPa and a strain of about 0.7% (load transfer from Nb$_3$Sn to the ductile Nb)

Nb$_3$Sn Young’s modulus

- Preferential Nb$_3$Sn grain orientation in RRP and PIT wires causes elastic anisotropy.
- Nb$_3$Sn elastic anisotropy was calculated from:
  - Nb$_3$Sn grain orientation distribution determined by neutron diffraction [14] and by Electron Backscatter Diffraction (EBSD) [20],
  - Nb$_3$Sn single crystal elastic constants (SEC) determined from ultrasonic measurements [21].
- Nb$_3$Sn Young’s moduli calculations rely on the availability of accurate SEC.
- Young’s moduli in axial direction of the RRP and PIT wires are 130 GPa and 140 GPa, respectively with an estimated uncertainty $<\pm 10\%$ [20].

The unusual temperature dependence of the Nb$_3$Sn Young’s modulus

• During cool down Nb$_3$Sn undergoes a phase transformation at roughly 40 K.
• This causes a Nb$_3$Sn lattice softening [21].
• The reduction of Nb$_3$Sn Young’s modulus in recent Nb$_3$Sn RRP and PIT type wires is confirmed by combined XRD-stress-strain measurements at RT and at 4.2 K.
• At 4.2 K the axial PIT Nb$_3$Sn subelement Young’s modulus is 27% smaller than the corresponding RT value.

Comparison of RT and 4.2 K Nb$_3$Sn filament stress strain curves (stress is normalised to non-Cu cross section of the PIT wire) [19].

Nb$_3$Sn Poisson’s ratio at 4.2 K

- Derived from Nb$_3$Sn lattice parameter measurements by high energy synchrotron X-ray diffraction during uniaxial tests in liquid He.

- It is assumed that the Nb$_3$Sn lattice changes are not strongly effected by the annealed Cu matrix.

- Poisson’s ratio can be calculated as the quotient of the transverse and axial lattice parameter change.

$$\nu_{Nb_3Sn-4.2K} = 0.36$$

$Nb_3Sn$ transverse lattice strain as a function the axial lattice strain measured during uniaxial tensile loading of BR, PIT and RRP wires in liquid He [19].
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Thermal expansion of unconfined Nb$_3$Sn conductor during RHT

Nb$_3$Sn RRP type wire axial and radial expansion during RHT. The thermal expansion of Cu is shown for comparison [20].

11 T dipole Nb3Sn Rutherford cable axial length change during RHT [23].


Thermal expansion of the Nb$_3$Sn conductor block

- Expansion of initially unreacted 11 T dipole cable ten stack samples during heat treatment up to 650 °C measured with high temperature extensometer at the StressSpec materials science neutron beamline of MLZ.
- In axial direction at minimal applied stress the unconfined cable length change behaviour is reproduced.
- Volume changes are strongly dependent on the stress applied during heat treatment. Data analysis is ongoing.

Set-up for simultaneous diffraction and stress strain measurements during Nb$_3$Sn Rutherford cable ten stack RHT up to 650 °C. 

*Courtesy M. Hofmann and MLZ StressSpec team.*
Nb$_3$Sn conductor block mechanical behaviour

- Young’s moduli in axial, transverse and radial direction
- Nb$_3$Sn and Cu loading strain and stress
Samples for uniaxial compressive stress-strain measurements

- Samples made of Nb$_3$Sn 11 T dipole Rutherford cables with mica and S2 glass insulation.
- (a) Ten-stack samples impregnated with epoxy resin CTD-101K with different epoxy volume fractions.
- (b) Cubes machined out of 11 T dipole conductor block, containing 8 Rutherford cables and part of the adjacent coil wedges to compensate for the cable keystone angles.
- (c) Non impregnated ten-stack samples.

[Image of samples and measurements]

Courtesy Felix Wolf

11 T conductor block sample was prepared by CERN central workshop team

Two set-ups used for compressive stress-strain measurements

Set-up at CERN inserted in a hydraulic press

Set-up at MLZ StressSpec materials science neutron beam line

Ten-stack sample with clip-on extensometer Epsilon 3442-006M-010-LT Class B-1 with 6 mm gauge length.

Sample with clip-on extensometer Instron 2620-602 with 12 mm gauge length. 50 kN load frame with load cells (HBM Typ 03, 50 kN)

Courtesy Felix Wolf

Courtesy M. Hofmann and MLZ StressSpec team
Axial Young’s modulus: Experiment vs rule of mixture (ROM) estimate

- Stress strain measurements in axial compression with ten-stack samples with different epoxy volume fraction.
- Assuming $E_{\text{wire-axial}}=126$ GPa and $E_{\text{epoxy}}=4$ GPa.
- Ten-stack stiffness can be predicted assuming iso-strain conditions (according to Voigt). The load is essentially carried by the wire, and the stress in the epoxy remains small.

**Comparison of stress-strain curves of Nb$_3$Sn wire (axial tension) and ten-stack samples wire different volume fraction ($V_{\text{wire}}$) (axial compression) [24].**

**ROM estimation (upper bound) of the axial compression stiffness dependence on $V_{\text{wire}}$ and comparison with experiment.**
Transverse Young’s modulus: Effect of previous load cycles and unloading stress:

- Cyclic loading hysteresis is strongly reduced in second load cycle.
- A strong creep behaviour is observed when the transversal load exceeds about 125 Mpa.
- Small effect of previous load cycle on ten-stack Young’s modulus.

Courtesy Felix Wolf
Transverse Young’s modulus of the 11 T dipole conductor block and effect of epoxy volume fraction

• Young’s modulus decreases with increasing epoxy volume fraction in the ten-stack samples.
• 11 T dipole conductor block Young’s modulus corresponds with that of ten-stack with about 25% epoxy.
• Identical Young’s modulus vs unloading stress behaviour measured with CERN and MLZ set-ups, with extensometer gauge lengths of 6 mm and 12 mm, respectively.
• $40 \text{ GPa} < E_{\text{transverse}} < 55 \text{ GPa}$ in the unloading stress range from 50 to 125 MPa

**Courtesy Felix Wolf**
Set-up for combined stress-strain and diffraction measurements

- Lattice strain (elastic strain) in Nb$_3$Sn and Cu is measured by neutron diffraction in axial, radial and transverse directions.
- Gauge volume $5 \times 5 \times 5$ mm$^3$ in the centre of the samples.
- Macroscopic sample strain is measured simultaneously with an extensometer.

(a) Load frame mounted in Eulerian cradle in the Stress-Spec beamline. (b) Ten-stack sample and extensometer mounted for combined stress-strain and diffraction measurements.

Calculation of loading strain and stress

- The \( \text{Nb}_3\text{Sn} \) and Cu loading strains caused by the externally applied stress are determined from the \( \text{Nb}_3\text{Sn} \) (321) and Cu (220) scattering angles.

- Loading strains in transverse, axial, and radial directions are calculated with respect to the assumed stress free scattering angle.

\[
\varepsilon_{hkl} = \frac{d_{hkl} - d_{0,hkl}}{d_{0,hkl}} = \frac{\sin(\theta_{0,hkl})}{\sin(\theta_{hkl})} - 1
\]

- Loading stresses are calculated assuming that the transverse, axial and radial directions are the principal stress directions in the sample, and that there are no shear stresses in the sample.

- Elastic constants and Poisson’s ratios have been calculated from single crystal elastic constants (\( E_{\text{Nb}_3\text{Sn}(321)} = 131 \text{ GPa}, \nu_{\text{Nb}_3\text{Sn}(321)} = 0.363, E_{\text{Cu}(220)} = 138.9 \text{ GPa}, \nu_{\text{Cu}(220)} = 0.333 \)) [21,26,27].

\[
\sigma_{ii} = \frac{E_{hkl}}{1 + \nu_{hkl}} \left( \varepsilon_{ii} + \frac{\nu_{hkl}}{1 - 2\nu_{hkl}} (\varepsilon_{11} + \varepsilon_{22} + \varepsilon_{33}) \right)
\]


Transverse compression: Nb₃Sn and Cu loading stresses

- Transverse Nb₃Sn and Cu loading stresses increase linearly with the externally applied transverse stress (iso-stress conditions in the composite in transverse load direction).
- In axial and radial directions, the isotropic Cu pressure induces an important axial and radial tensile stress in Nb₃Sn upon uniaxial transverse compression.

Comparison of Nb₃Sn (321) and Cu (220) loading stress evolution in (a) transverse, (b) axial and (c) radial direction [25].
Axial compression: Nb$_3$Sn and Cu loading stresses

- When the macroscopic axial pressure on the cable stack exceeds about 100 MPa, the Cu elastic strain remains constant and the load is mainly carried by Nb$_3$Sn (and Nb) filaments.
- At 150 MPa externally applied pressure the axial Nb$_3$Sn (321) and Cu (220) loading stresses are about 300 MPa and 100 MPa, respectively.

(a) Nb$_3$Sn and (b) Cu loading stresses in impregnated cable stack as a function of axial compressive stress. Encircled data points show the residual strain when the external stress is released.
Axial compression: Nb₃Sn loading strain vs macroscopic sample strain

• When the macroscopic strain exceeds 0.1%, the elastic Nb₃Sn strain increases with a slope close to unity, confirming that the strain in all conductor constituents is equal.
• Transverse and radial Nb₃Sn lattice spacing changes are mainly caused by the Poisson effect (assuming a Poisson’s ratio of about 0.36).

Elastic axial strain derived from neutron diffraction data as a function of the macroscopic sample strain measured with an extensometer.

Average transverse and radial Nb₃Sn (321) strain as a function of axial strain.
Measuring strain and stress distribution in 11 T dipole coils

- Residual strain measurement
- Post mortem metallographic characterisation
Residual strain in unconfined Nb$_3$Sn wires

- The strong mismatch of thermal expansion coefficients of different conductor materials can lead to residual stresses in the composite.
- The Nb$_3$Sn strain state in different straight wires was measured by synchrotron X-ray diffraction in liquid He.
- A significant axial Nb$_3$Sn pre-compression is measured in Bronze Route wires.
- In contrast, in unconfined RRP and PIT type Nb$_3$Sn conductors the residual Nb$_3$Sn strain is small [19].
- Since annealed Cu yields at low stress, it cannot impose substantial stresses on the Nb$_3$Sn filaments in unconfined RRP type and PIT type wires.
- The Cu yielding is a huge advantage in terms of superconductor critical current, in particular in high applied field.
Residual strain in 11 T dipole magnet coils

- Residual Nb\textsubscript{3}Sn (321) strain in the four largest conductor blocks of the 11 T coil segment were measured by neutron diffraction [26].
- In conductor blocks P1, P2 and P3 the residual Nb\textsubscript{3}Sn strain is smaller than the uncertainty of the residual strain measurements (<0.02%).
- A significant Nb\textsubscript{3}Sn residual strain value of -0.056±0.010% is found in P4 conductor block at the coil midplane, which was presumably submitted to the highest stress during prior magnet assembly and cold test.

Residual Nb\textsubscript{3}Sn (321) strain in axial, hoop and radial directions in the center of the four largest conductor blocks of the coil segment extracted from 11 T dipole short model after prior cold test.

• Measuring strain and stress distribution in 11 T dipole coils
  • Residual strain measurement
  • Post mortem metallographic characterisation
Stress distribution during 11 T dipole coil collaring

- The mechanical stresses to which the brittle Nb$_3$Sn filaments are submitted during magnet fabrication and operation must not exceed the Nb$_3$Sn filament fracture stress (which is at RT lower than at 4.2 K).
- Particularly high mechanical stresses can be imposed on the conductor blocks during collaring.
- FE simulations suggest that the stresses in the different coil regions vary over large range.

Critical current limitation of 11 T dipole short model coil #109

- During cold testing of 11 T dipole short model MBHDP102 critical current limitations at the coil mid-plane turns were detected.
- Precise voltage-current measurements revealed a particularly strong critical current and n-value degradation of coil #109, indicating Nb₃Sn filament cracking.
- The degradation is presumably due to excessive mechanical stresses applied during collaring.

V-I curves measured during cold test, indicating a strong critical current degradation in 11 T dipole coil #109. 

Courtesy G. Willering https://indico.cern.ch/event/739669/
Post mortem analysis of coil #109

- Rough cutting of about 30 cm long coil segments
- Precision cutting of 10 mm and 20 mm thick segments with diamond saw.

(a) rough and (b) precision cutting of 11 T dipole short model coil #109. **Courtesy Sylvain Caille**
Visualisation of Nb₃Sn cracks in coil #109 metallographic cross section

Metallographic examination reveals:

• Cracks in some Nb₃Sn filaments
• Deformation contrast in copper
• Cu grains appear smaller in certain regions.

Section 1- Row 1 has the most number of cracks

Section 1->Row1->Strand 1

Metallographic cross section of 11 T dipole coil #109 segment with Nb₃Sn cracks. 
Courtesy S. Balachandran, P.J. Lee, J. Cooper, and NHMFL-ASC team
Nb$_3$Sn crack distribution in coil #109 metallographic cross section

Distribution of cracks in 11 T dipole coil #109
Courtesy S. Balachandran, P.J. Lee, J. Cooper, and NHMFL-ASC team
Absence of cracks in the Nb$_3$Sn filaments in coil GE-CO2 metallographic cross section

- Coil GE-CO2 is a series 11 T dipole coil with optimised insulation scheme.
- It is assumed that the mechanical stress on the conductor during the improved collaring procedure did not exceed the critical stress above which filament breakage occurs.
- V-I measurements during magnet cold test did not reveal any signs of a critical current limitation in the investigated coil part.
- Metallographic cross sections of coil GE-CO2 have been studied in order to confirm the absence of Nb$_3$Sn cracks in series production coils, and as additional confirmation that the cracks visualised in #109 metallographic cross sections are not caused by experimental artefacts.

Metallographic cross sections of Nb3Sn subelements in coil GE-CO2
Courtesy S. Balachandran, P.J. Lee, J. Cooper, and NHMFL-ASC team
Vickers hardness of strain hardened and annealed Cu

- The strain hardening of annealed Cu is an opportunity to determine the maximum stress to which the Cu stabiliser has been exposed when loading the 11 T dipole coils.
- Because of the strong load dependence of HV results at low loads, HV measurements need to be done with a load of at least 100 gf.

(a) Vickers indents in an unreacted Internal Tin Nb$_3$Sn wire. (b) Comparison of conventional HV load dependence of the Cu stabiliser cold-worked and after 24 h-460 °C annealing.
Vickers hardness maps for assessing Cu loading stress

- Preliminary Cu hardness results of ongoing work on 11 T dipole coil segments and cable stacks at NHMFL-ASC
  - #109 HV_{100-max} = 79
  - GE-CO2 (to be done)
  - Ten stack 210 MPa transverse stress HV_{100-max} = 68
  - Ten stack 210 MPa axial stress HV_{100-max} = 54
  - Ten stack unloaded HV_{100-max} = 51

- As expected, the Cu hardness in the axial loaded ten stack is comparatively small (the load is mainly carried by Nb$_3$Sn).

- The maximum loading stress in coil #109 was well above 210 MPa.

**Vickers hardness overview in 11 T dipole coil #109, and unloaded and loaded ten-stack samples**

*Courtesy S. Balachandran, P.J. Lee, J. Cooper, and NHMFL-ASC team*

Ten stack samples were provided by F. Wolf.
Cu Vickers hardness distribution in coil #109 metallographic cross section

- Nb$_3$Sn crack density and Cu stabiliser hardness in #109 appear to be correlated.
- Preliminary results suggest that Nb$_3$Sn cracks are formed when the stress exceeds approximately the stress needed for Cu work hardening to HV$_{100}$ = 70.

Vickers hardness distribution in 11 T dipole coil #109 metallographic cross sections

Courtesy S. Balachandran, P.J. Lee, J. Cooper, and NHMFL-ASC team
Correlation between OFE Cu Vickers hardness and yield strength

• Assuming that the maximum stress in the 11 T dipole coils is achieved during collaring at RT, this can be revealed by indentation hardness maps in metallographic coil cross sections.

• For fully strain worked materials yield stress is roughly one third of the Vickers hardness (expressed in identical units).

• Determination of a more precise HV to YS relation for not fully strain worked Cu is ongoing.

\[
y = 2.9152x - 28.673 \\
y = 3.2667x \\
y = 3.4099x
\]

Different models for predicting the relationship between Cu Vickers hardness and yield strength

The residual resistivity ratio (RRR) of pure Cu depends on the degree of strain work.

For Cu Vickers hardness HV<70 the effect of strain work on Cu RRR remains comparatively small.

http://cds.cern.ch/record/1233953/files/Tech%20Note%202010%20005.pdf
Conclusion
Linear elastic and isotropic materials properties

• Thermomechanical properties of the magnet materials exhibiting **linear elastic and isotropic** behaviour are well known.
  • Dynamic and static Young’s moduli are identical within experimental uncertainties.
  • Uniaxial tension and compression results are equivalent within experimental uncertainties.
  • Simple relations between the different elastic moduli and Poisson’s ratio apply.
  • Temperature dependence of $E$, $G$ and $\nu$ can be derived from resonance tests of a single sample.

• **Low temperature friction coefficients**
  • Friction behaviour can strongly change during cooling (for instance stable sliding of stainless steel at RT can change to strong slip stick behaviour at 4.2 K).
  • Low temperature sliding behaviour can be controlled by lubricants and coatings.
  • Better knowing and controlling friction coefficients may be an opportunity for improving magnet performance and the reliability of FE models.
Anisotropic materials and Nb$_3$Sn conductor materials

• For anisotropic materials simple relations between $E$, $G$ and $\nu$ do not apply (e.g. DISCUP C30). All mechanical properties need to be measured.

• Nb$_3$Sn mechanical properties
  • Linear elastic behaviour
  • Brittle fracture
  • Nb$_3$Sn texturing and elastic anisotropy depend on wire fabrication route
  • At RT Young’s moduli in axial direction of the RRP and PIT wires are 130 GPa and 140 GPa, respectively with an estimated uncertainty smaller than ±10%.
  • Unusual Young’s modulus temperature dependence; at 4.2 K the axial Nb$_3$Sn subelement Young’s modulus in PIT wire is about 30% smaller than the corresponding RT value
  • 4.2 K Poisson’s ratio $\nu_{Nb3Sn-4.2K}=0.36$

• Fully annealed Cu mechanical properties
  • Very low yield stress in the order of 10 MPa
  • Flowing when critical stress is exceeded
  • Strain hardening; mechanical properties change with load history

• Nb$_3$Sn conductor thermal expansion during reaction heat treatment
  • Non linear and partly irreversible
  • Different in axial and radial directions
  • Dependent on the mechanical stress applied during reaction heat treatment
Conductor block mechanical behaviour

- The 11 T dipole conductor block (epoxy volume fraction ) Young’s moduli at RT are:
  - \( E_{\text{axial}} = 95 \text{ GPa} \)
  - \( 40 \text{ GPa} < E_{\text{transverse}} < 55 \text{ GPa} \) in the unloading stress range from 50 to 125 MPa
- Assuming that the maximum coil stress is achieved during collaring, for the FE modelling of all subsequent coil load steps the slope of the unloading curves is representative.
- Under cyclic loading (below the maximum stress achieved during collaring) a small hysteresis behaviour typical for composite materials is observed.
- \( \text{Nb}_3\text{Sn} \) coils can be considered as a fibre reinforced composite.
  - Iso-strain conditions in axial load direction
  - Iso-stress conditions in transverse compression
- \( \text{Nb}_3\text{Sn} \) and \( \text{Cu} \) loading stress results are an unique opportunity to validate FE models.
  - Directly measured by neutron diffraction
  - Well defined ten stack samples
  - Well defined mechanical loading conditions
Measurement of strain and stress distribution in coils

- Very small residual Nb$_3$Sn strain (<0.03%) in RRP and PIT conductors and in 11 T dipole coils (unlike in bronze route conductors). The yielding of Cu during cooling from the Nb$_3$Sn formation temperature is highly beneficial since it prevents a strong critical current degradation due to high Nb$_3$Sn residual strain.

- Main results of ongoing metallographic studies at NHMFL-ASC
  - Post mortem metallographic characterisation of 11 T dipole short model coil #109 reveals Nb$_3$Sn filament cracks that can explain the critical current limitation.
  - No Nb$_3$Sn cracks are found in the metallographic cross sections of the optimised 11 T dipole series coil GE-C02.
  - Indentation hardness measurements in the Cu stabiliser in metallographic coil cross sections are an opportunity for post mortem analysis of the stress distribution during collaring.
  - Crack density and hardness of adjacent Cu stabiliser appear to be correlated.
  - The peak stress to which coil #109 has been submitted was well above 210 MPa.
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

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

Calculation of shear modulus from resonance frequencies

For long thin beams with rectangular cross section the shear modulus \( G \) is calculated from the mass \( m \), length \( L \), width \( b \) and thickness \( t \) of the beam, and its fundamental frequency of the beam in torsion \( f_t \).

\[
G = \frac{4Lmf_t^2}{bt} \left( \frac{B}{1 + A} \right) \tag{1}
\]

where

\[
B = \frac{b + t}{t} + \frac{t}{b} - 2.52 \left( \frac{t}{b} \right)^2 + 0.21 \left( \frac{t}{b} \right)^6 \tag{2}
\]

The term \( A \) can be ignored if the ratio \( 1 < b/t < 2 \).

Young’s modulus measurement: uniaxial tensile tests

- Precise Young’s moduli can be derived from stress-strain measurements of materials that exhibit pronounced linear elastic behaviour (e.g. Ti6Al4V).
- 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%.

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.


Extensometry for wire uniaxial stress-strain measurements

- The precision of stress-strain measurements relies mainly on careful extensometer strain measurements.
- 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.

Comparison of calculated Nb$_3$Sn, and measured wire and subelement Young’s moduli

![Image](image.png)

Fig. 2. Euler angle maps of PIT filament (a) after 620 °C-60 h HT and (b) after 650 °C-120 h HT. The scale bar is valid for (a) and (b).

TABLE II

<table>
<thead>
<tr>
<th></th>
<th>PIT B215</th>
<th>RRP #7419</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>RT</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>E$_{\text{axial}}$</td>
<td>130</td>
<td>140</td>
</tr>
<tr>
<td>E$_{\text{trans}}$</td>
<td>135</td>
<td>129</td>
</tr>
<tr>
<td><strong>4.2 K</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>E$_{\text{axial}}$</td>
<td>106</td>
<td>127</td>
</tr>
<tr>
<td>E$_{\text{trans}}$</td>
<td>116</td>
<td>104</td>
</tr>
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</table>

Fig. 3. Stress-strain curves measured at room temperature on a reacted RRP wire and its extracted filaments.

TABLE III

<table>
<thead>
<tr>
<th></th>
<th>E-modulus (GPa)</th>
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<tbody>
<tr>
<td><strong>PIT B215</strong></td>
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</tr>
<tr>
<td>Wire reacted</td>
<td>116±2.3</td>
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<tr>
<td>Filaments reacted</td>
<td>84.8±1.1</td>
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<tr>
<td><strong>RRP #7419</strong></td>
<td></td>
</tr>
<tr>
<td>Wire reacted</td>
<td>124±1.7</td>
</tr>
<tr>
<td>Filaments reacted</td>
<td>114±3.8</td>
</tr>
</tbody>
</table>

Nb₃Sn lattice parameter changes in a PIT wire as a function of externally applied stress

- Measured by high energy synchrotron X-ray diffraction.
- Nearly linear lattice spacing vs stress dependence, both in axial tension and transverse compression.
- Nb₃Sn axial pre-strain in the free standing PIT wire is 0.03% [19].
- Nb₃Sn axial pre-strain of the same PIT wire on a VAMAS barrel is about 0.14% (measured by neutron diffraction) [32].

 Nb₃Sn lattice parameter in axial and transverse direction as a function of axial tensile or transverse compressive stress. The lattice parameters for the same PIT wire on a VAMAS barrel are shown for comparison.

Comparison of the Cu mechanical properties in LHC Nb-Ti and in HL-LHC Nb₃Sn wires

- The Cu in the LHC Nb-Ti wires is partly annealed during a 1 h-190 °C HT at the end of the wire production (Cu Vickers hardness HV0.05 = 79.7±2.0).

<table>
<thead>
<tr>
<th></th>
<th>$R_{p0.2}$ (MPa)</th>
<th>$R_m$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RRP wire #7419</td>
<td>-</td>
<td>185±4.5</td>
</tr>
<tr>
<td>Fully annealed Cu (as in HL-LHC wire)</td>
<td>46.2±2.6</td>
<td>204±1.6</td>
</tr>
<tr>
<td>LHC wire 01K152</td>
<td>282±8</td>
<td>747±8</td>
</tr>
<tr>
<td>Partly annealed Cu in LHC wire</td>
<td>roughly 250</td>
<td>-</td>
</tr>
</tbody>
</table>

RT stress-strain curve of (a) inner LHC Nb-Ti wire 01K152 [33] and (b) reacted RRP type Nb₃Sn wire (OST billet #7419). **Courtesy R. Bjoerstad.**

Monotonic transverse compressive loading of non-impregnated cable stack: **$\text{Nb}_3\text{Sn}$ diffraction angle and peak width changes**

- Average diffraction angles within the $5\times5\times5$ mm$^3$ gauge volume are recorded.
- Diffraction peak broadening indicates that the $\text{Nb}_3\text{Sn}$ loading strain distribution becomes more inhomogeneous upon application of external stress.
- In axial direction the $\text{Nb}_3\text{Sn}$ (321) peak is narrower than it is in radial and transverse directions, and the axial $\text{Nb}_3\text{Sn}$ (321) peak width increase starts at higher stress.

---

**$\text{Nb}_3\text{Sn}$ (321) diffraction angles as a function of externally applied transverse compressive stress.**

**$\text{Nb}_3\text{Sn}$ (321) diffraction peak width evolution as a function of externally applied transverse compressive stress.**
Transverse compression: Nb₃Sn and Cu loading strain and stress

- Loading strains and stresses are calculated from the relative changes of the diffraction angles.
- Cu is under similar compressive stress in all three directions.
- The magnitude of the Cu pressure corresponds approximately with the externally applied transverse compressive stress.
- Uniaxial transverse compression induces an important axial tensile stress in the Nb₃Sn filaments.

(a) Nb₃Sn (321) and (b) Cu (220) lattice strains and (c) Nb₃Sn (321) and (d) Cu (220) loading stresses as a function of externally applied transverse compressive stress.
Strain hardened Cu Vickers hardness load dependence

- At very low loads the HV result depends strongly on experiments and experimental procedures.

*Comparison of the strain worked Cu HV load dependence obtained by instrumented and conventional indentation measurements.*
Is it possible to directly measure stress distributions in collared coils? Experience from neutron diffraction test measurements

- The stresses in the collars and coils are reduced when the hydraulic press is released and the collars are locked by the keys (spring back).
- By neutron diffraction it is possible to measure the lattice spacing in all components in the centre of a collared coil segment (for instance in any collar nose).
- Because of the complex load case, for applied stress measurements neutron diffraction pattern need to be acquired at six different sample orientations.
- Acquisition time can become long, depending on gauge volume (spatial resolution), and on coil segment thickness.
- Distinction between residual and applied stress in the YUS-130 collar can be difficult.

(a) and (b) Collared 11 T dipole coil segment mounted on the Eulerian cradle on the StressSpec sample stage. (c) Axial, radial and hoop strain in the YUS-130 collar. Lattice strains were calculated assuming that the average axial strain is zero.
Neutron radiography of 11 T dipole collared coil segment

- Neutron radiography was performed at the Antares imaging beam line with cold neutrons ($\lambda=2$ to $5 \, \text{Å}$, field of view $100 \, \text{mm} \times 100 \, \text{mm}$, spatial resolution is about $80 \, \mu\text{m}$).
- Coil alignment imperfections are seen between loading pole, shim, loading plate and at coil midplane.
- In the vicinity of gaps stress peaks should be excluded.

(a) 11 T dipole collared coil segment in Antares beam line. (b) and (c) Neutron radiographs of 4 cm thick collared 11 T dipole coil segment.
### 11 T dipole materials properties

<table>
<thead>
<tr>
<th>Material</th>
<th>$R_{p0.2}$ (MPa)</th>
<th>$R_m$ (MPa)</th>
<th>A (%)</th>
<th>Z (%)</th>
<th>Young’s modulus dynamic (GPa)</th>
<th>Young’s modulus static (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Resonance</td>
<td>Impulse</td>
</tr>
<tr>
<td>316LN_L</td>
<td>351±12</td>
<td>674±2</td>
<td>54±5</td>
<td>63±2</td>
<td>191±0.3</td>
<td>191±0.3</td>
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<tr>
<td>316LN_T</td>
<td>324±4</td>
<td>658±1</td>
<td>53±5</td>
<td>63±1</td>
<td>n.m.</td>
<td>n.m.</td>
</tr>
<tr>
<td>Magnetil_L</td>
<td>117±3</td>
<td>241±1</td>
<td>n.m.</td>
<td>73±3</td>
<td>196±0.8</td>
<td>196±0.3</td>
</tr>
<tr>
<td>Magnetil_T</td>
<td>124±2</td>
<td>267±1</td>
<td>47±2</td>
<td>71±4</td>
<td>219±0.1</td>
<td>218±0.3</td>
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<tr>
<td>YUS-130_L</td>
<td>401</td>
<td>793</td>
<td>71</td>
<td>66</td>
<td>196±0.9</td>
<td>196±0.3</td>
</tr>
<tr>
<td>YUS-130_T</td>
<td>415</td>
<td>749</td>
<td>51</td>
<td>66</td>
<td>193±1.1</td>
<td>192±0.3</td>
</tr>
<tr>
<td>Ti-6Al-4V_L</td>
<td>868±5</td>
<td>930±12</td>
<td>17±1</td>
<td>28±2</td>
<td>115</td>
<td>114</td>
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<tr>
<td>Ti-6Al-4V_T</td>
<td>n.m.</td>
<td>n.m.</td>
<td>n.m.</td>
<td>n.m.</td>
<td>n.m.</td>
<td>n.m.</td>
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<tr>
<td>DISCUP no-HT_L</td>
<td>332±2</td>
<td>387±1</td>
<td>22±2</td>
<td>43±4</td>
<td>92.9±0.2</td>
<td>93.8±0.3</td>
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<tr>
<td>DISCUP no-HT_T</td>
<td>n.m.</td>
<td>n.m.</td>
<td>n.m.</td>
<td>n.m.</td>
<td>n.m.</td>
<td>n.m.</td>
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<tr>
<td>DISCUP HT_L</td>
<td>284±1</td>
<td>376±1</td>
<td>26±1</td>
<td>48±1</td>
<td>96.7</td>
<td>96.3</td>
</tr>
</tbody>
</table>

Elastic properties of some polymers used in superconducting magnets

<table>
<thead>
<tr>
<th>Material</th>
<th>Density (g/cm³)</th>
<th>Poisson’s ratio RT</th>
<th>E-modulus (GPa)</th>
<th>Rₘ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Dynamic</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Compression</td>
<td>Static</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>RT</td>
<td>77 K</td>
</tr>
<tr>
<td>CTD-101K</td>
<td>1.23</td>
<td>n.m.</td>
<td>4.10±0.11</td>
<td>n.m.</td>
</tr>
<tr>
<td>ULTEM 1000</td>
<td>1.28</td>
<td>n.m.</td>
<td>3.80±0.31</td>
<td>n.m.</td>
</tr>
<tr>
<td>ULTEM 9085</td>
<td>n.m.</td>
<td>n.m.</td>
<td>2.20±0.04</td>
<td>n.m.</td>
</tr>
<tr>
<td>Ryton R4-XT</td>
<td>1.68</td>
<td>n.m.</td>
<td>16.5±0.49</td>
<td>n.m.</td>
</tr>
<tr>
<td>EPR S7_axial</td>
<td>1.90</td>
<td>n.m.</td>
<td>19.4</td>
<td>21.0±0.31</td>
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<tr>
<td>EPR S7_trans</td>
<td>n.m.</td>
<td>n.m.</td>
<td>26.4±0.64</td>
<td>n.m.</td>
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<tr>
<td>EPR S1_axial</td>
<td>n.m.</td>
<td>26.6</td>
<td>25.8±0.58</td>
<td>23.5±0.10</td>
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<tr>
<td>EPR S1_trans</td>
<td>n.m.</td>
<td>21.7±1.14</td>
<td>n.m.</td>
<td>n.m.</td>
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<tr>
<td>PMP325</td>
<td>1.76</td>
<td>0.305</td>
<td>13.9</td>
<td>13.9±0.82</td>
</tr>
<tr>
<td>Al 7175_hoop</td>
<td>2.77</td>
<td>0.325</td>
<td>69.2</td>
<td>n.m.</td>
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</table>