Material studies for the ATLAS Phase-II Upgrade for the High Luminosity LHC

Carbon fibre laminae measurements and investigation of moisture expansion of an adhesive used in support structures

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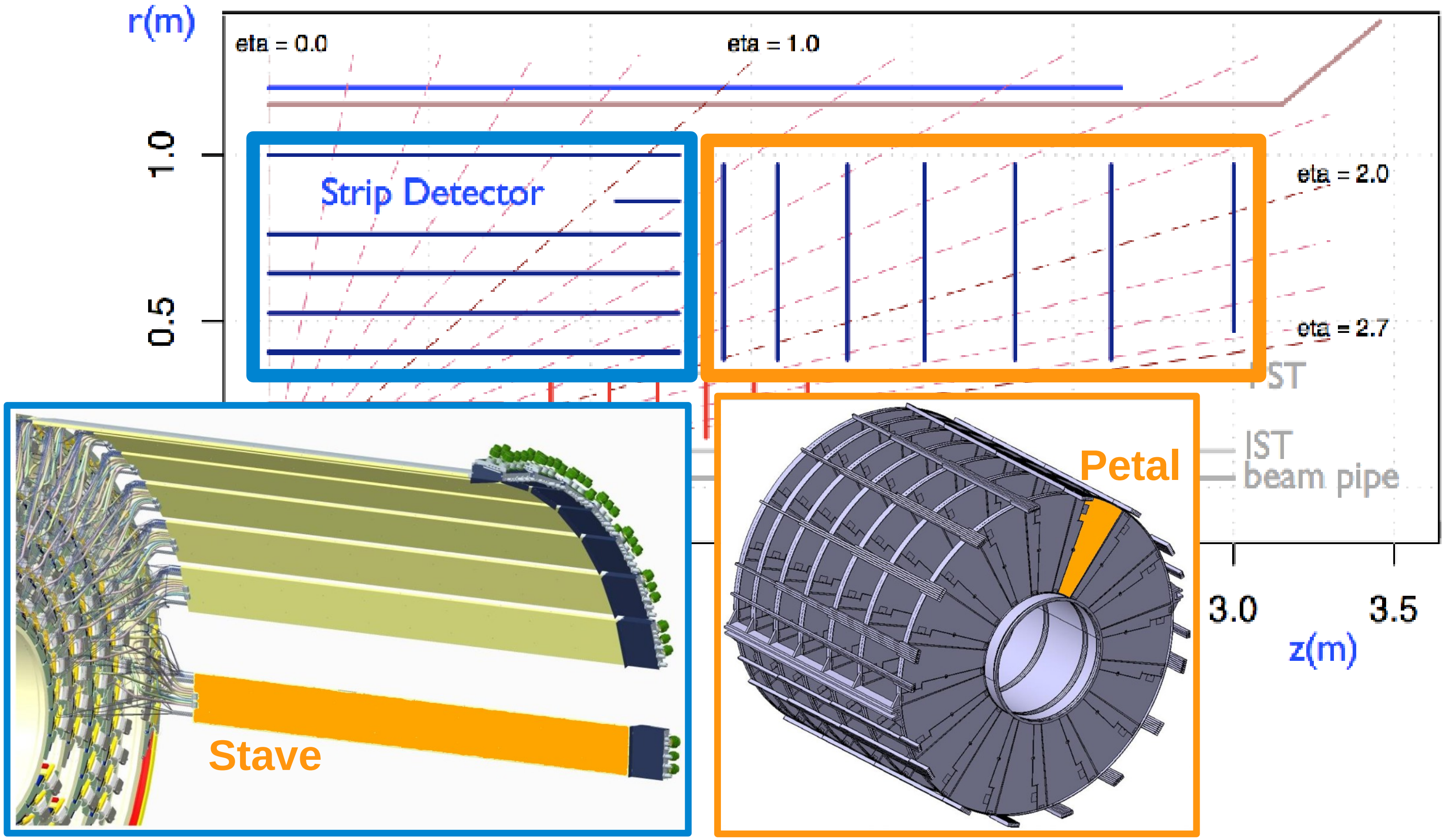
The future ATLAS strip tracker

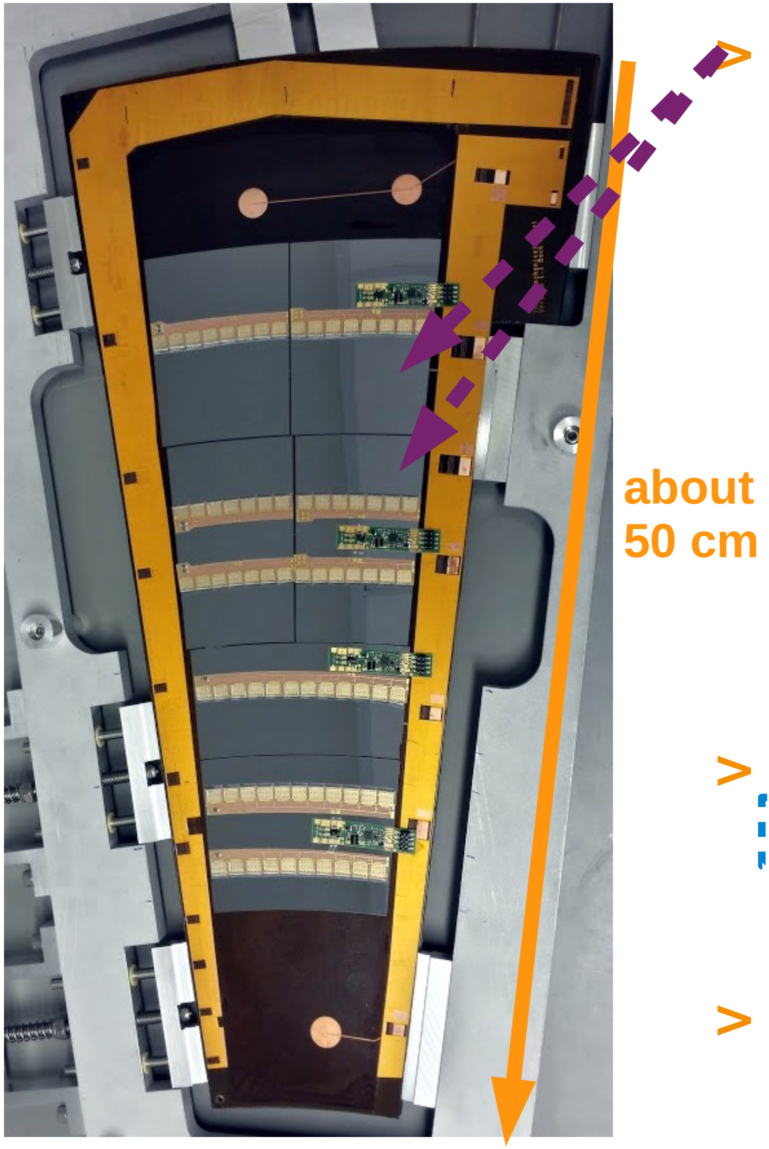


Barrel, sensors parallel to beam, consisting of staves

Endcap, sensors perpendicular to beam, consisting of petals

Staves and petals have the same structure, vary mostly in geometry

Petals for the new end-cap



- Modules are glued on to support structure consisting of
  - Carbon fibre face sheets
  - Carbon fibre honeycomb structure
  - Closeouts
  - Carbon foam
  - Titanium pipes (for CO_2 cooling)
- Everything held together by Hysol 9396 glue
- Face sheets and glue studies presented today

Carbon fibre face sheets

Carbon fibre: K13C2U, impregnated with 45 % (mass) resin (EX1515)

→ by volume: 61 % resin, 39 % carbon fibre

→ density 1.58 g/cm^3

61 % of the carbon fibre volume consists of resin we know nothing about but its density

- Modulus: 900 GPa (fibre), 4 GPa (resin)

  → modulus along fibres (0°): 353 Gpa (higher for higher fibre content)

  → modulus across fibres (90°): 6 Gpa (higher for higher resin content)

- Modulus for intermediate angles can be calculated

- Theoretical properties of an ideal cured carbon fibre lamina

  → this is what would be used in a simulation

  → measurements conducted for laminae produced under realistic circumstances

Making carbon fibre face sheets

- Flat, even metal plate (larger than face sheets to be built) is covered with release film

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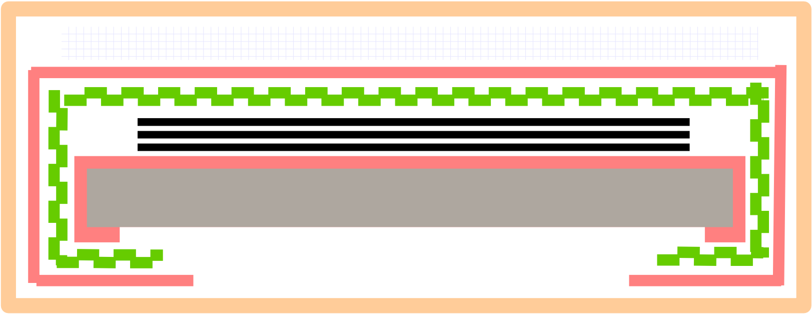
Making carbon fibre face sheets



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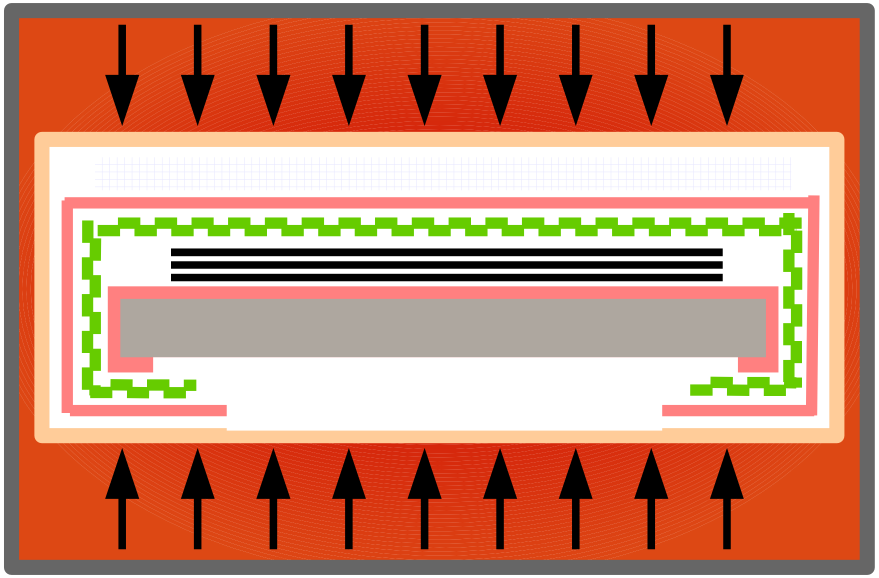

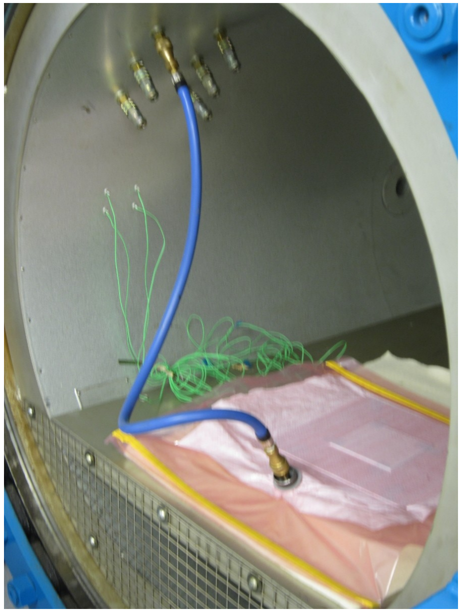

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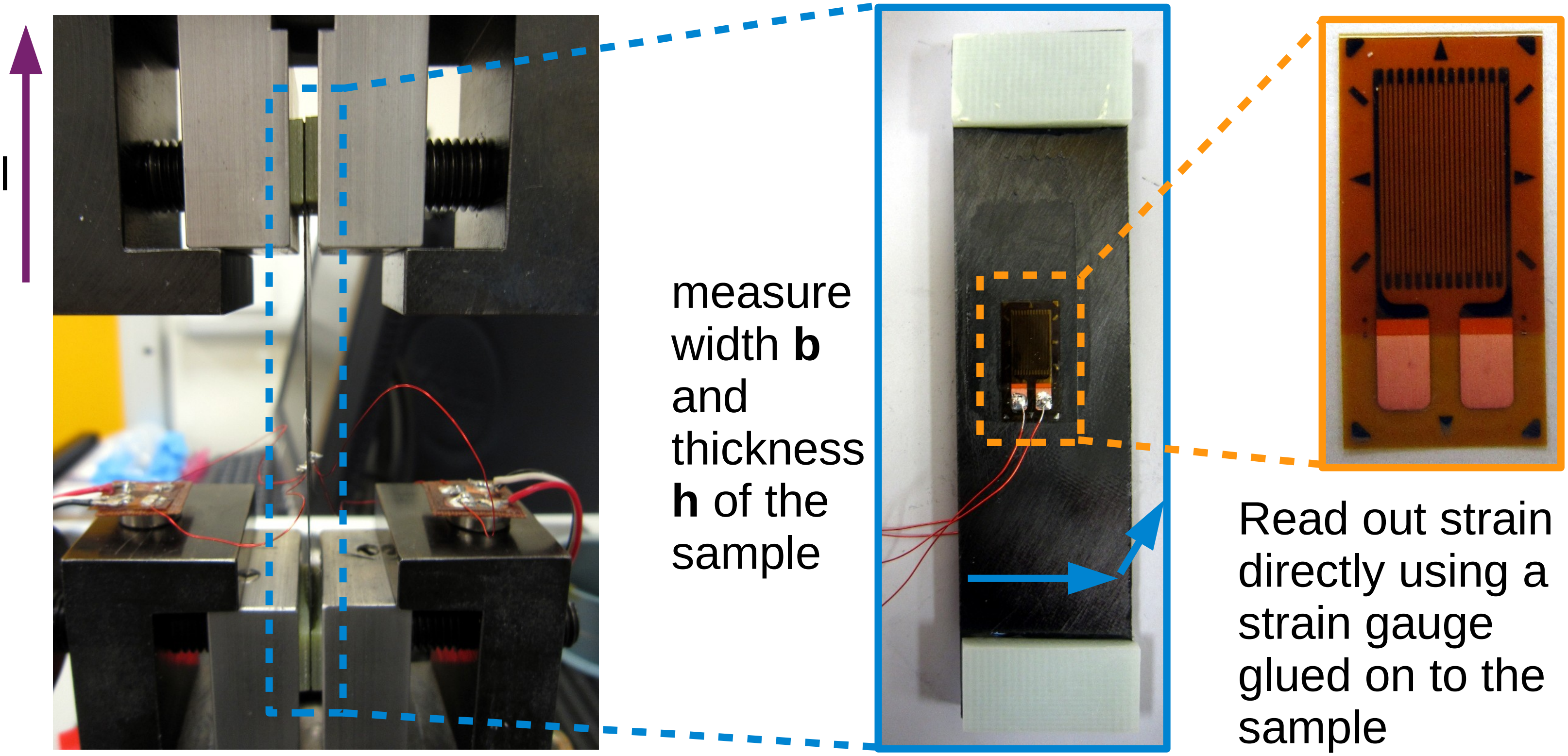
- The whole package is put in a vacuum back with a breather layer to distribute vacuum better, then sealed and evacuated
- Whole package is then placed in an autoclave and cured at high pressure and high temperature

Modulus of elasticity measurements

- Unidirectional laminae produced (fibres in all layers aligned in parallel)
- Strips cut at angles between 0° and 90° from different laminae
- Modulus measured in material tester: $E = \frac{\text{stress}}{\text{strain}} = \frac{F/(b \cdot h)}{\varepsilon}$



pull force **F** applied in material tester

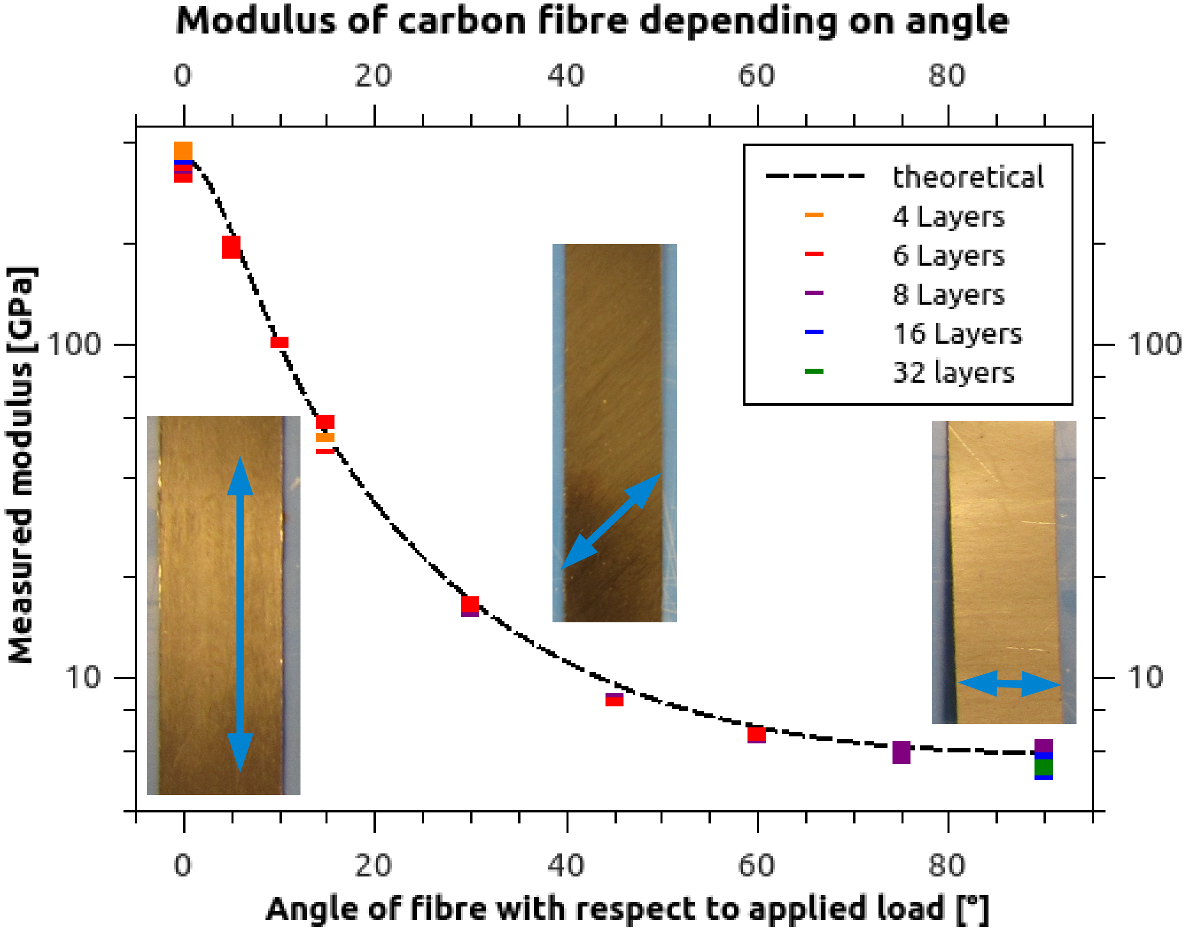
measure width **b** and thickness **h** of the sample

Read out strain **ε** directly using a strain gauge glued on to the sample

Modulus measurements

$$\frac{1}{E_x} = \frac{1}{E_1}\cos^4\alpha + \left(\frac{1}{G_{12}} - \frac{2\nu_{12}}{E_1}\right)\sin^2\alpha\cos^2\alpha + \frac{1}{E_2}\sin^4\alpha,$$

- Samples were found to be (qualitatively) close to the expected modulus calculated from
- Modulus at 0° (E_1)
- Modulus at 90° (E_2)
- Shear modulus (G_{12})
- Major Poisson's ratio (ν_{12})



- Modulus of steel: 210 GPa

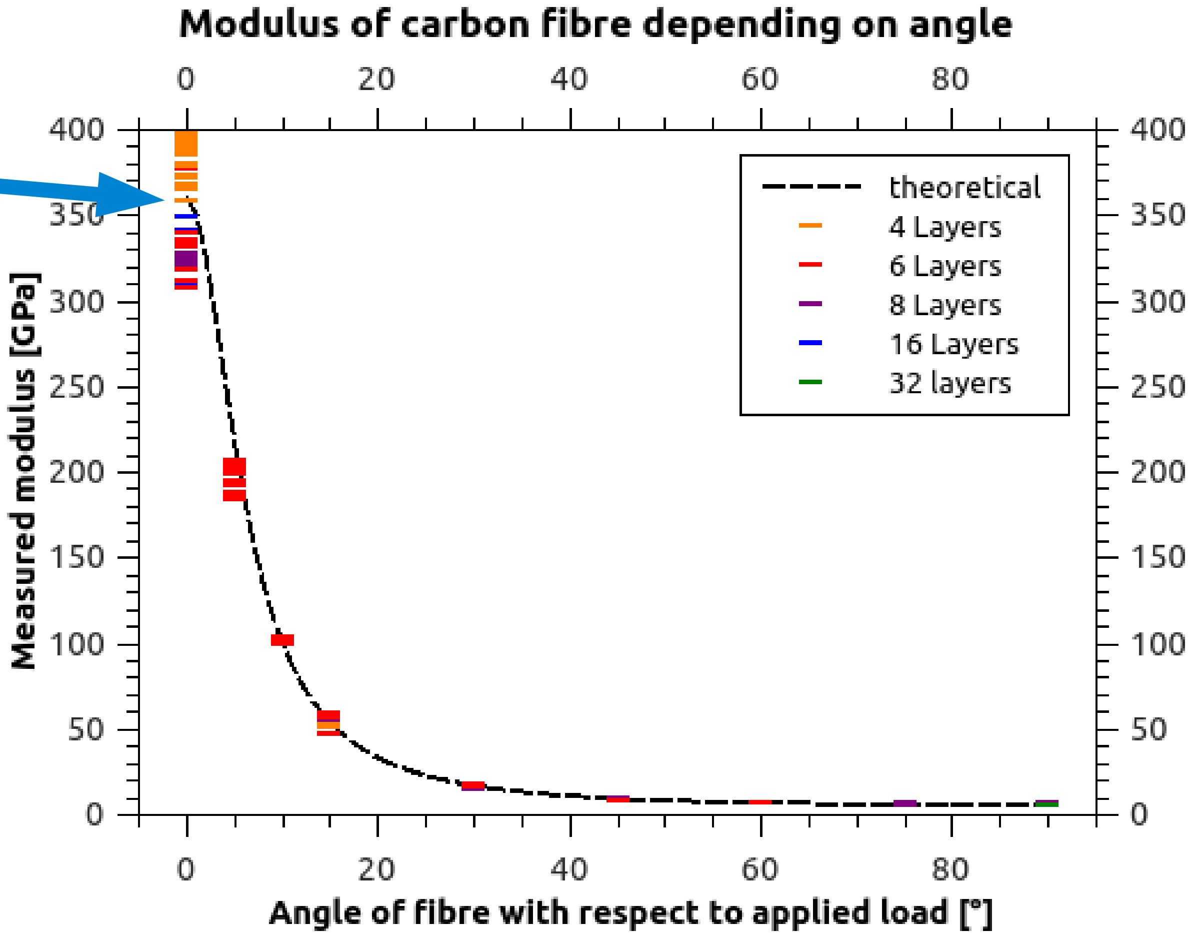
Modulus measurements

But on a linear scale …

- discrepancy of ± 50 GPa for 0° angle
- ±10 % for higher angles

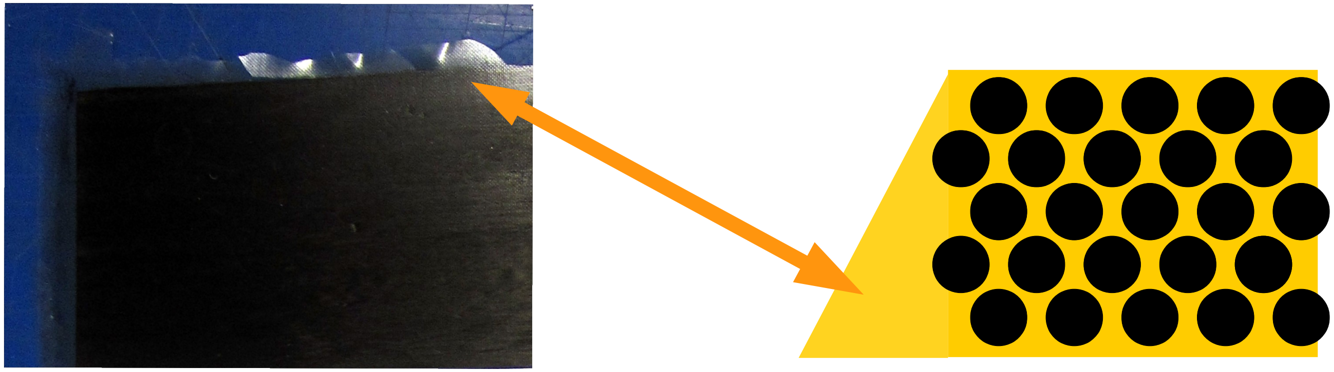
→ look for the source of these variations

(strain gauge misalignment, influence of strain gauges on mechanics etc. taken into account)

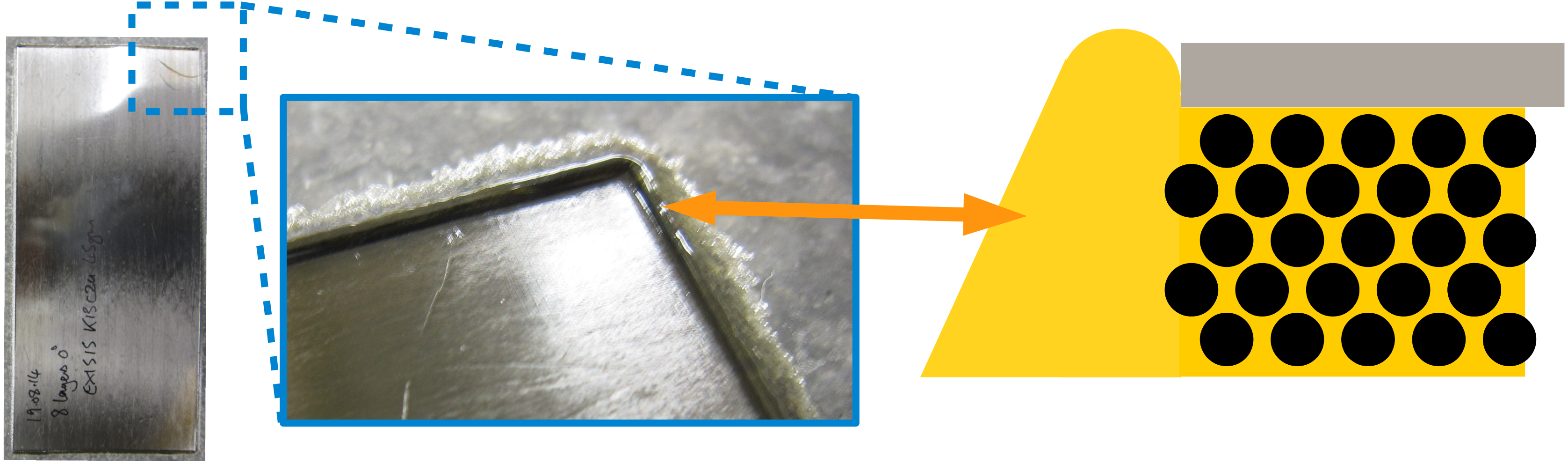


Modulus measurements

- Observed during face sheet construction: resin oozing out at edges

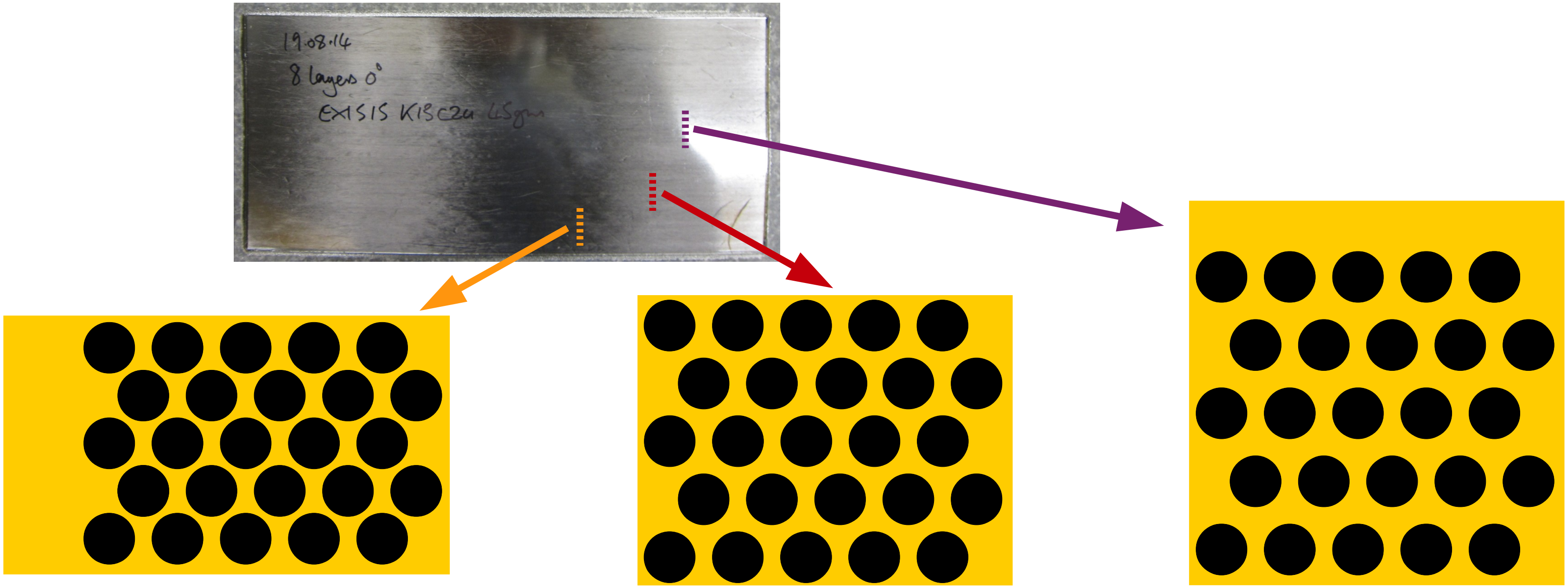


- Tried in a different setup with metal plate on top



- In these areas, the resin content would be lower than nominal value

Resin content impact on modulus



Edge region:
resin oozed out
same fibre volume

resin content < 45 %
fibre content > 55 %

Longitudinal
modulus > 360 GPa
Transverse
modulus < 6 GPa

Normal region:

45 % resin content
55 % fibre content

Longitudinal
modulus 360 GPa
Transverse
modulus 6 GPa

Center region
(depending on curing setup)
resin accumulated
same fibre volume

resin content > 45 %
fibre content < 55 %

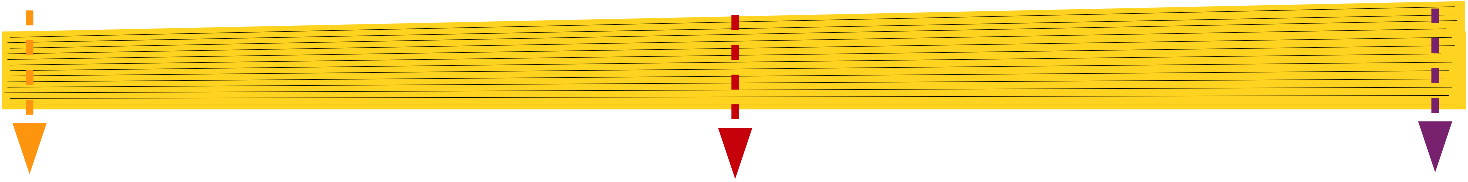
Longitudinal modulus < 360 GPa
Transverse modulus > 6 GPa

Simple estimate of actual modulus

- Depending on the curing setup of a lamina and the region on the lamina, thickness and modulus can differ for the same type of prepreg

- absolute fibre content per area (45 g/cm^2 per layer) does not change

  (thicknesses actually measured on different samples)



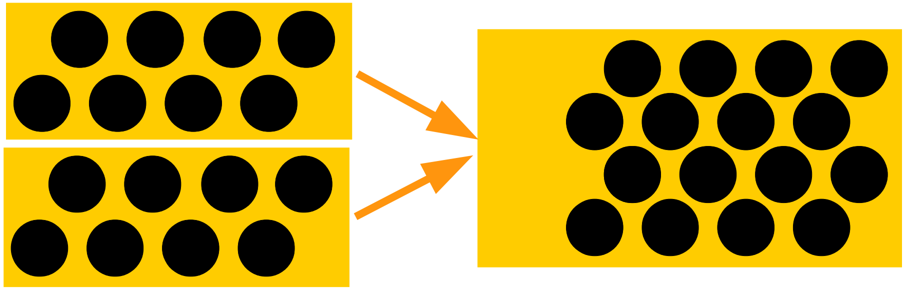
45 µm per layer (edge region)	52 µm per layer (nominal thickness)	55 µm per layer (center region)
20.5 µm (46 %) fibre 24.5 µm (54 %) resin → 416 GPa	20.5 µm (39 %) fibre 31.5 µm (61 %) resin → 353 GPa	20.5 µm (37 %) fibre 34.5 µm (63 %) resin → 336 GPa

Estimate actual modulus of a carbon fibre lamina

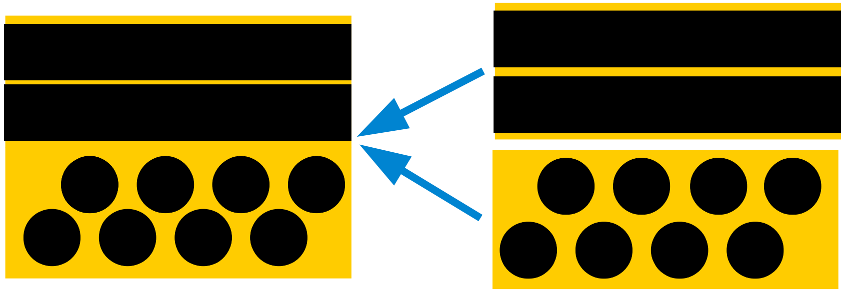
- Normally known for commercially available prepreg:
  - Modulus of fibre and resin (E_{fibre}, E_{resin})
  - Nominal contents of resin (by mass) and fibre (g/cm^2)
  - Densities of fibre and resin
- Step 1: calculation of
  - Nominal volume fractions of fibre and resin ($V_{nominal,\ fibre}$, $V_{nominal,\ resin}$)
  - Calculate nominal layer thickness $d_{nominal}$
- Step 2: measurement of the actual lamina thickness
  - Calculate actual layer thickness d_{actual} = lamina thickness / number of layers
- Step 3: calculation of
  - The actual fibre volume content $V_{actual,\ fibre} = V_{nominal,\ fibre} \cdot d_{nominal} / d_{actual}$
  - The actual resin volume content $V_{actual,\ resin} = 1 - V_{actual,\ fibre}$
- Step 4: Calculation of actual modulus $E = V_{actual,\ fibre} \cdot E_{fibre} + V_{actual,\ resin} \cdot E_{resin}$

Estimate carbon fibre modulus for angle laminae

- Face sheets for support structures: usually not unidirectional (fibres in all layers are parallel), but angle laminae (high longitudinal modulus in one direction compensates low modulus in another layer)
- Rough estimate $E_{lamina} \approx (\sum E_{layer})$ / number of layers
- Compression effect occurs mostly between parallel layers



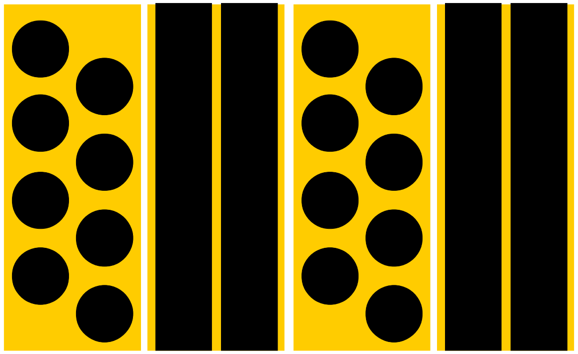
Unidirectional lamina: 0-0



Angle lamina: 90-0

- Compressive effects (+ resin oozing out) within a layer can occur for each layer
- Compressive effects between layers can only occur for adjacent layers with parallel fibres

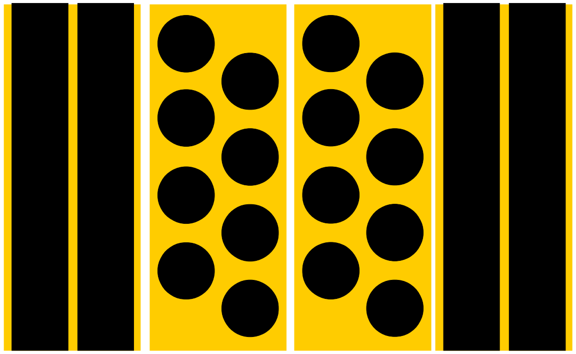
Accounting for layer compression in simuation



- Compression/lower resin content will occur similarly within each layer

  → all layers can be treated equally

  → four layers of similar thickness and modulus



- Compression/lower resin content will occur to a higher extent between the two middle layers

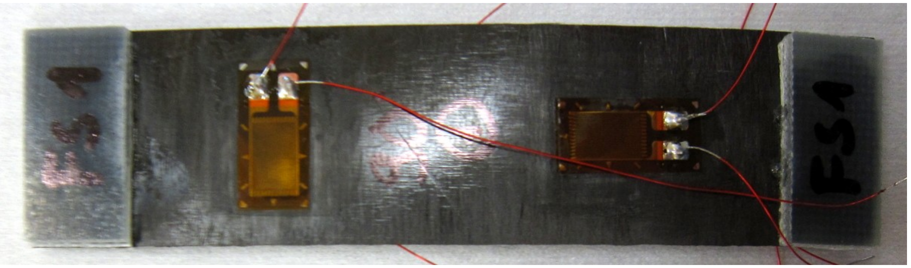
  → middle layers will probably have a lower resin content and higher modulus

  → account for effect in simulation (e.g. thinner layers in centre with higher modulus)

→ Simulated properties of angle laminae (calculated using results from unidirectional laminae) were found to agree well with measurements

(Major) Poisson's ratio

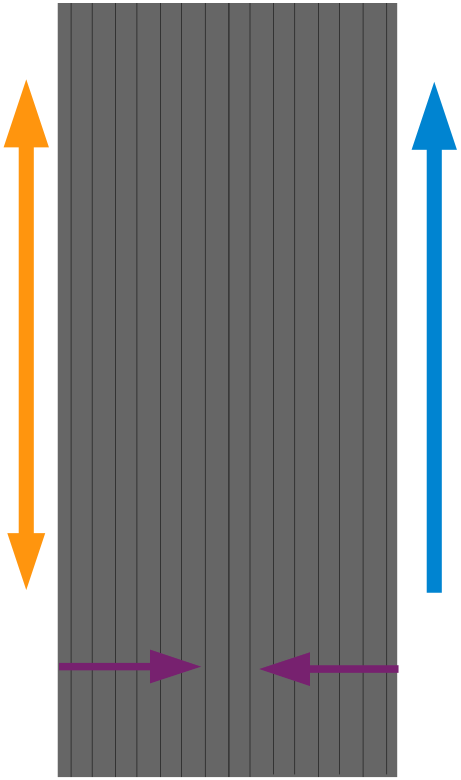
- Strain occuring perpendicular to fibres if stress is applied parallel to fibres
- Measured in material tester for 0° samples



- Measure strain occurring both parallel and perpendicular to fibre direction simultaneously using strain gauges
- Poisson's ratio

$$\nu_{12} = -\frac{\text{strain (perpendicular)}}{\text{strain (parallel)}} \approx 0.3$$

(fibres don't contract, only (unidirectional) resin between fibres)



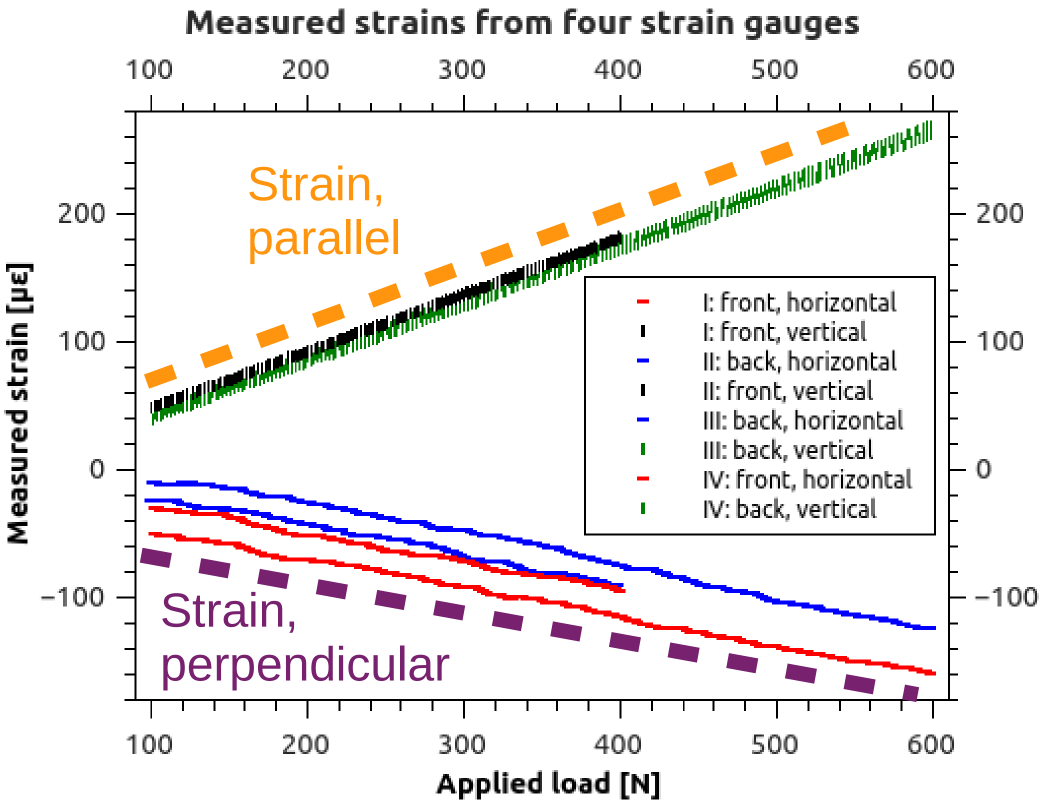
fibre direction

applied stress

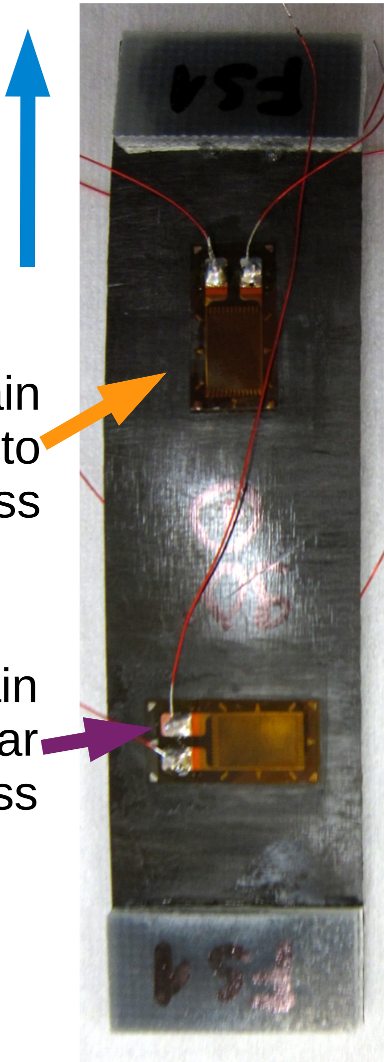
contraction occurring perpendicular to applied stress

Major Poisson's ratio measurement

- Strains measured both on front and back side of sample with one horizontal and one vertical strain gauge on each side



Measured strains from four strain gauges



applied stress

measure strain parallel to stress

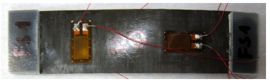
Measure strain perpendicular to stress

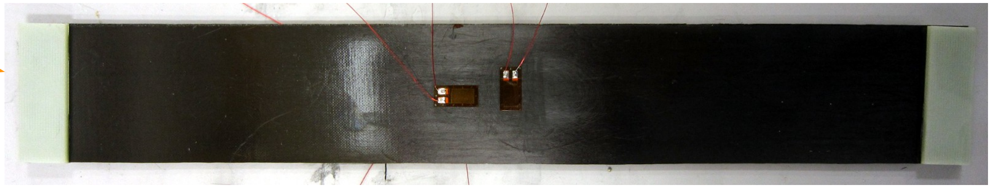
- Poisson's ratio: 0.53±0.03 (expected: 0.3)

Major Poisson's ratio measurements

> Investigate possible impact by sample geometry

- Single-sided measurement: 0.50 ± 0.02
- Double-sided measurement: 0.53 ± 0.03
- Different prepreg (100 g/cm^2): 0.47 ± 0.02
- Thick sample (16 layers): 0.58 ± 0.09
- Large strip: 0.45 ± 0.14





→ Combined: 0.49 ± 0.04 → much higher than expected

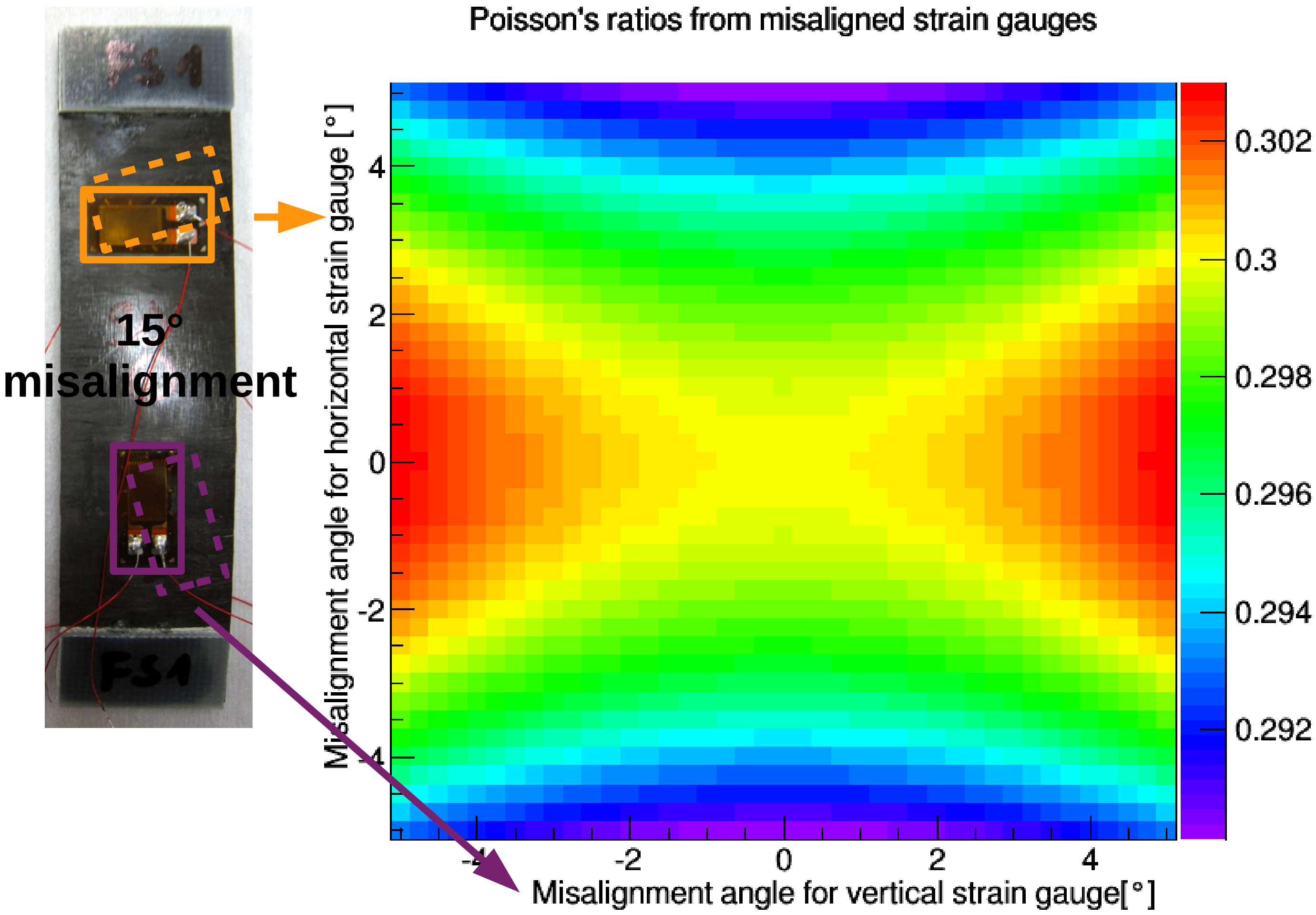
> Check if method is working using an aluminium strip

→ expected: 0.34, measured: 0.33 ± 0.01

→ Poisson's ratio for K13C2U really higher than expected?

Investigation of high Poisson's ratio

- While investigating possible sources of errors, we considered strain gauge misalignment

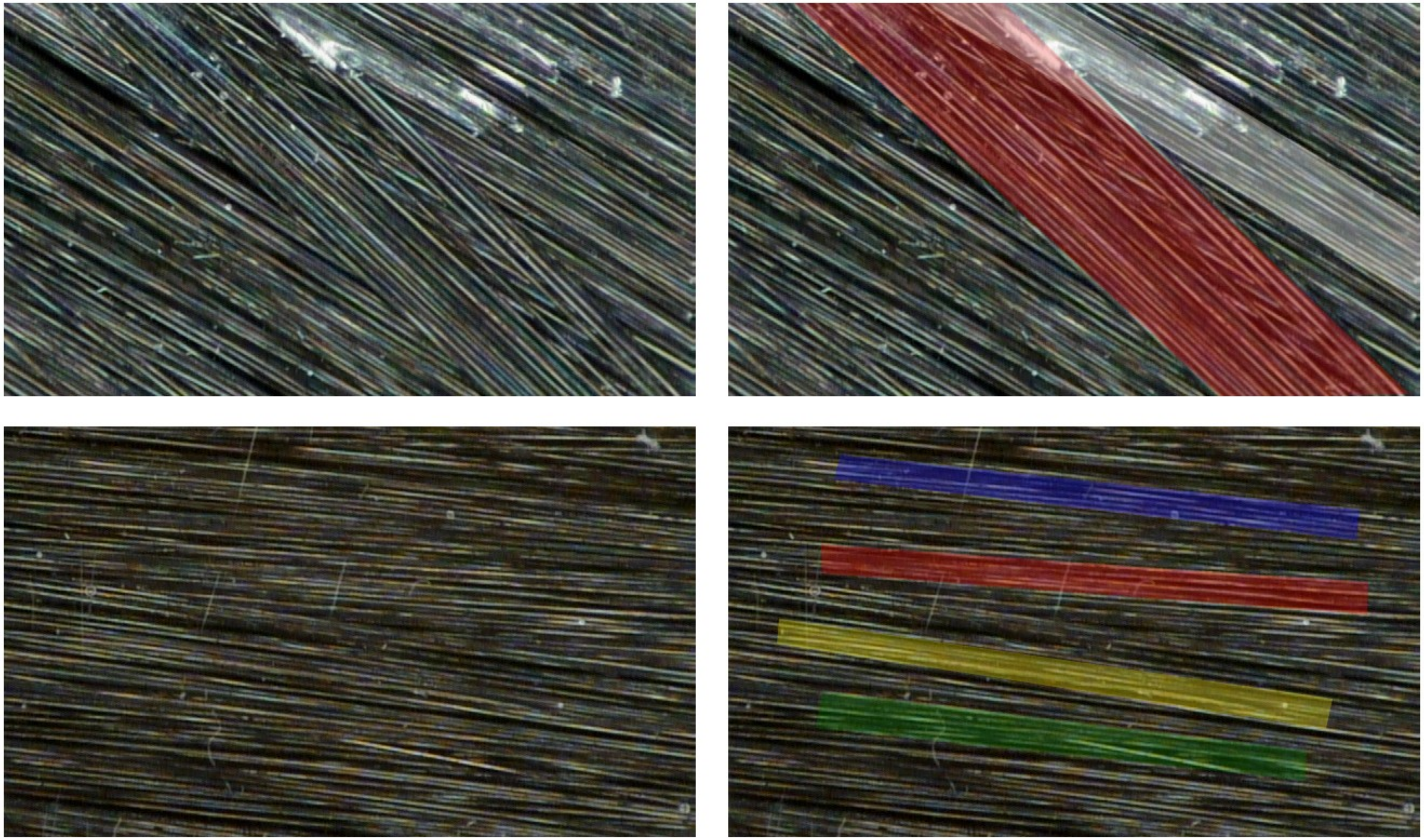


→ Poisson's ratio of 0.5 requires vertical strain gauge misalignment >> 5° and perfectly aligned horizontal strain gauges

→ does not explain our results, but gave us an idea what to look for

Investigation of high Poisson's ratio

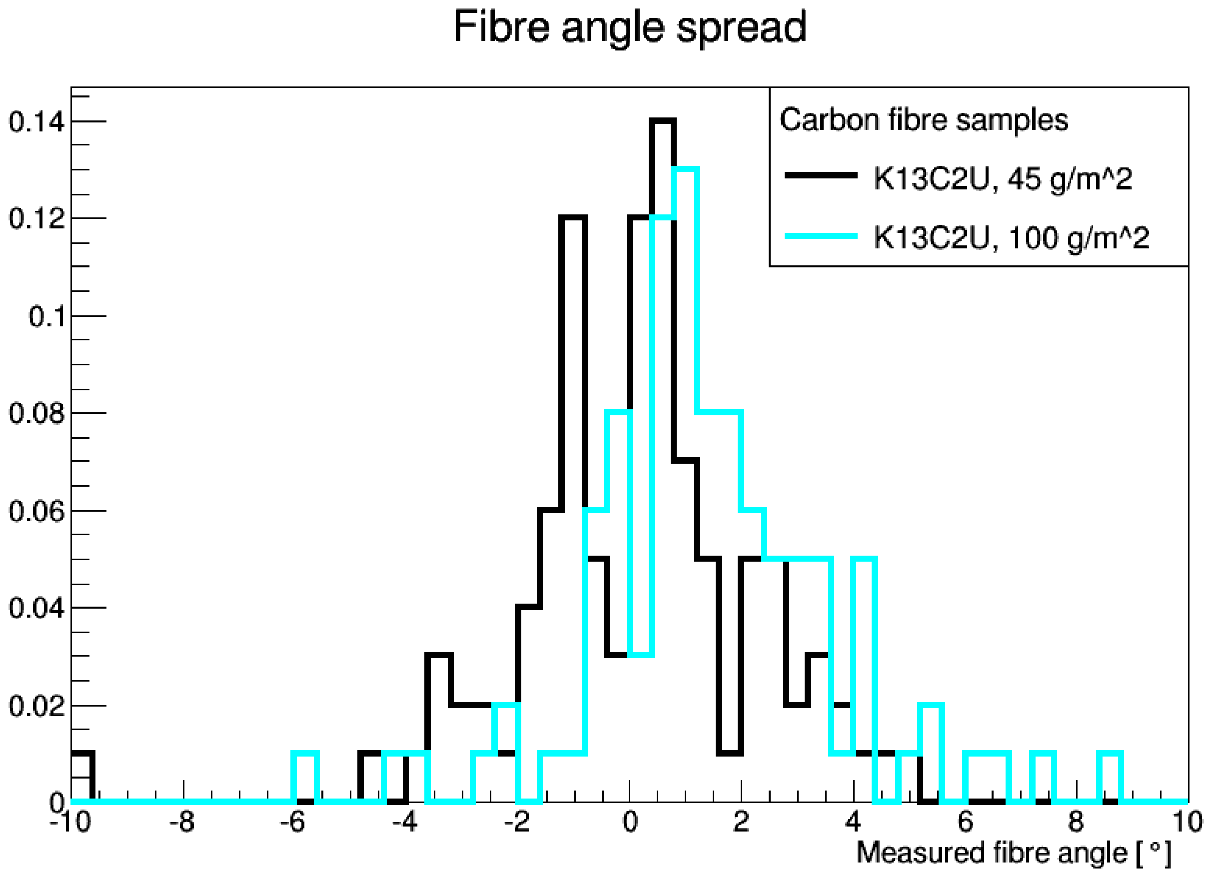
- Taking a closer look at our samples



- Parallel fibres in a prepreg / unidirectonal lamina are not really parallel

Quantifying fibre angle spread

- Poisson's ratio measurement samples placed under Smartscope, measured angles of individual, random fibres



- Gaussian fit of 100 measured angles showed a spread of fibre angles of σ = ± 2.6° and σ = ± 2.8° around expected angle

Investigating high Poisson's ratio measurements

> Large fibre angle spread could explain increased Poisson's ratios if

  - Strain parallel to fibre is dominated by small angles (stabilising)
  - Strain perpendicular to fibre is dominated by large angles (more inward movement)

> Estimated impact on overall Poisson's ratio with tool developed in parallel to mechanical measurements

→ for each layer in a lamina, stiffness matrix is calculated (how much strain occurs in which direction depending on direction of applied stress)

→ stresses and strains in each layer are calculated

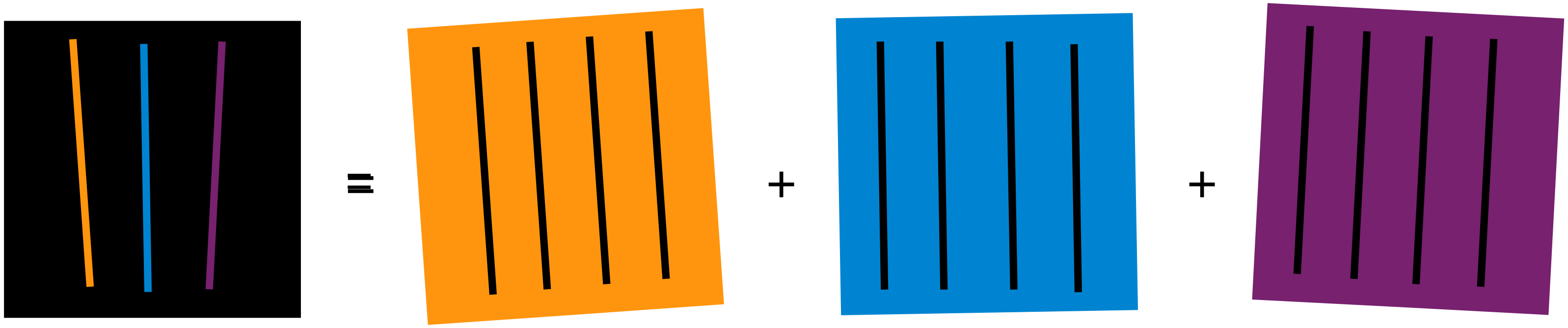
→ stresses and strains in the full lamina are calculated

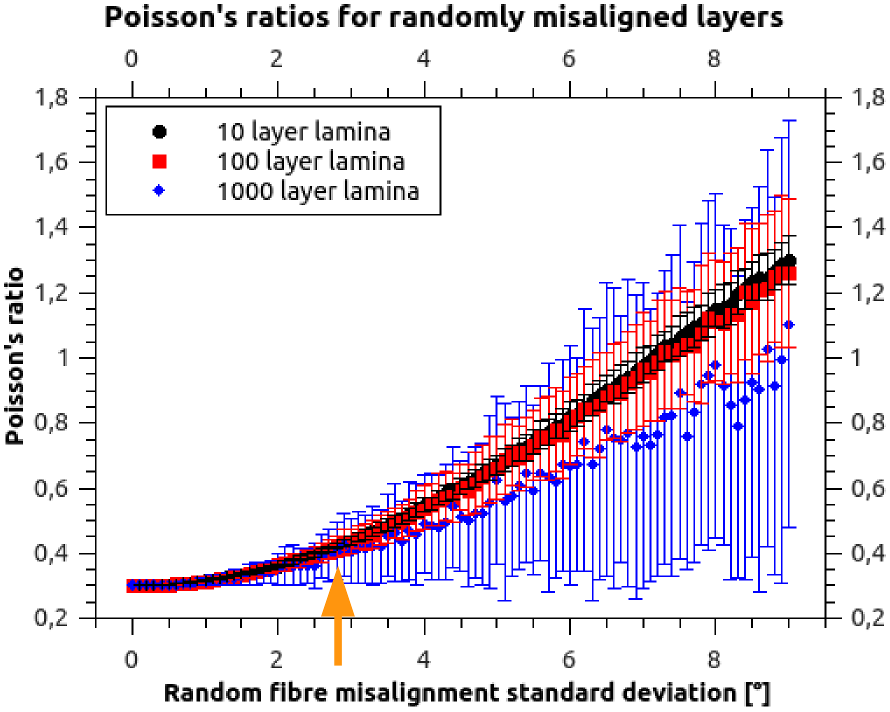
→ overall characteristics (modulus, Poisson's ratio) of the full lamina are calculated

→ different lamina setups can be studied theoretically

Investigating high Poisson's ratio measurements

- No possibility to simulate fibres with different angles in one layer
  - → approximate with thinner sub-layer with random angles

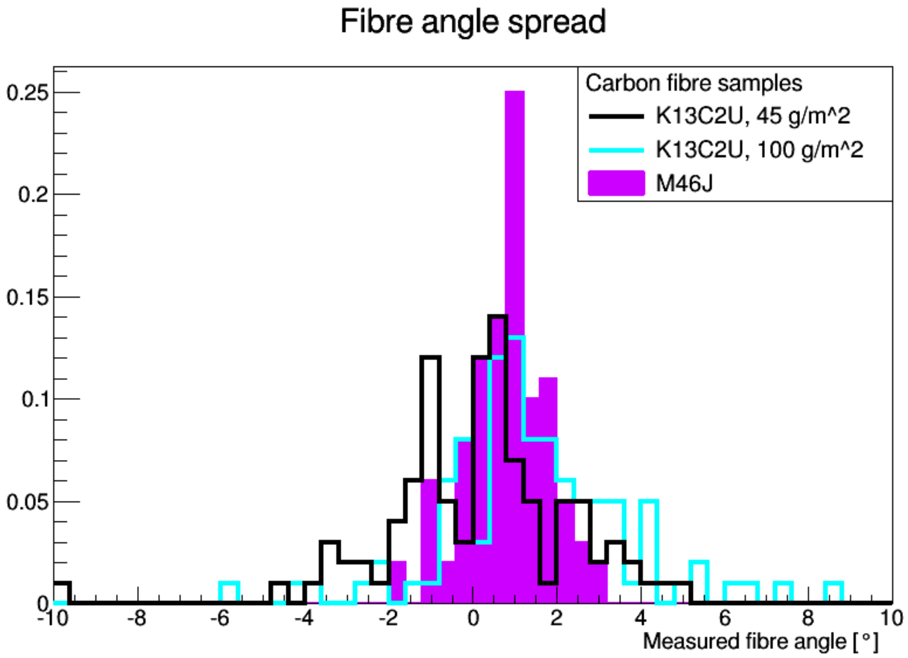


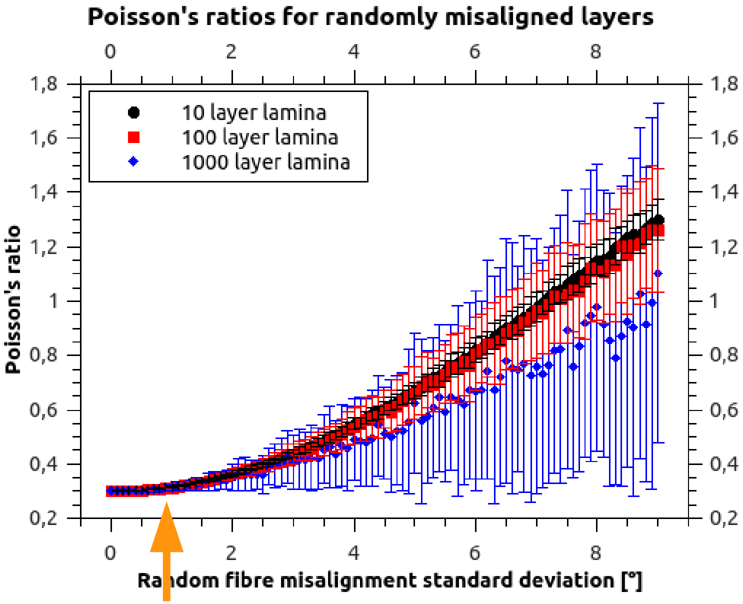


- For Gaussian distributed, random angles of individual layers, the Poisson's ratio increases with sigma of the Gaussian distribution
  - → for angle distributions measured on fibre (2.6° and 2.8°), Poisson's ratio already well above 0.4
- This could be the explanation

Investigating high Poisson's ratio measurements

- Theory checked with samples M46J fibre (stiffer, lower modulus)
  - → smaller angular spread measured in Smartscope





- angular spread: ± 0.9°, measured Poisson's ratio: 0.29 ± 0.02
  - → expected Poisson's ratios measured for aluminium and parallel fibre
- Poisson's ratio for K13C2U probably really is 0.49 ± 0.04 due to large angle variations of fibres

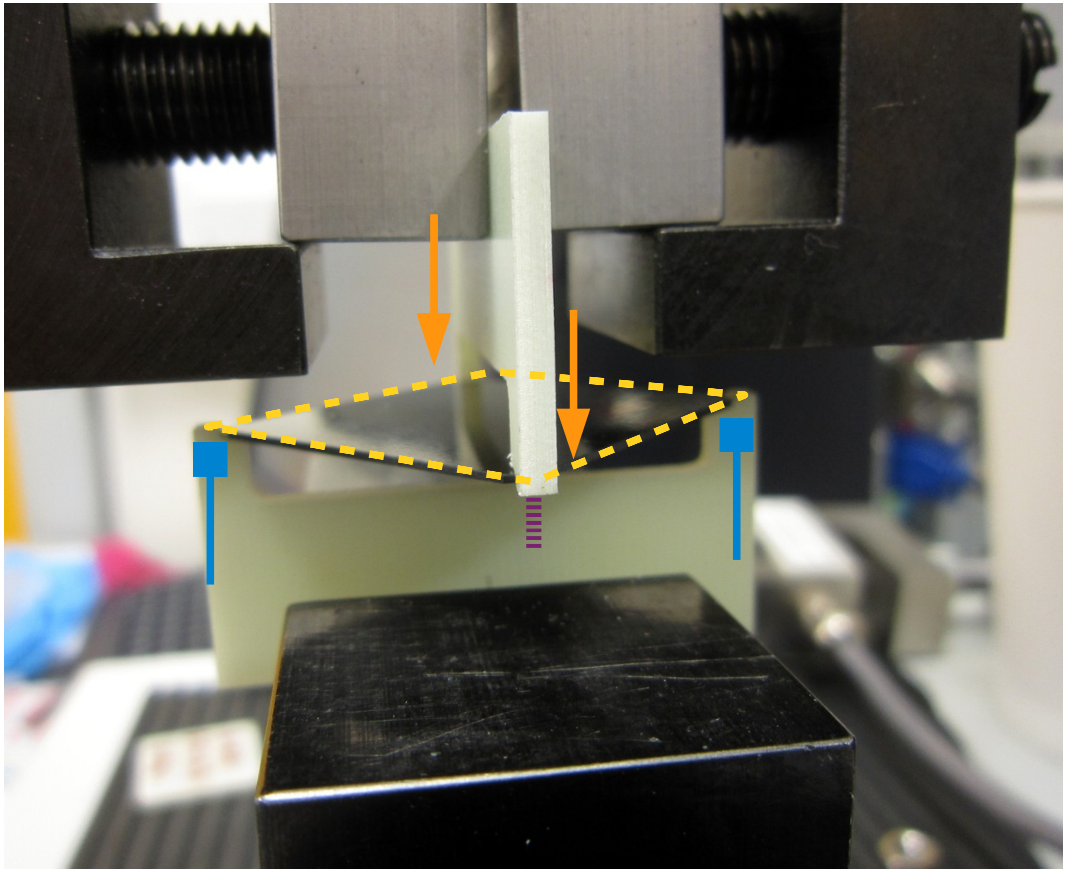
Shear modulus measurements

Measured in bending setup

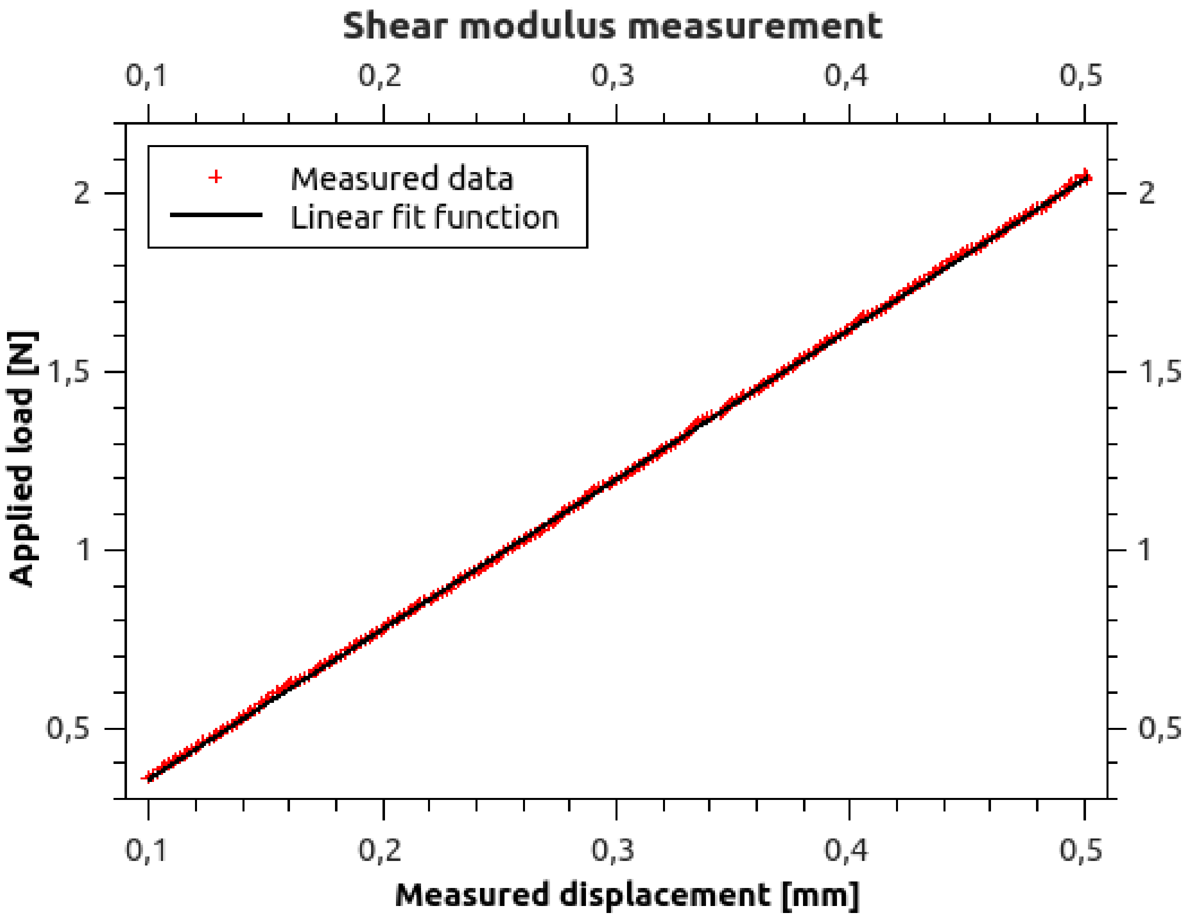
- Square plate of unidirectional lamina
- Load applied on two corners from above
- Two corners supported from below

→ measure corner displacement as a function of applied load

→ calculate shear modulus



Shear modulus measurements



Load vs displacement in good agreement with linear function

→ calculate shear modulus from slope (and geometrical properties of sample)

> Shear modulus measured to be 3.8 ± 0.1 GPa for different samples

→ consistent with data sheet expectations

Carbon fibre measurement conclusion

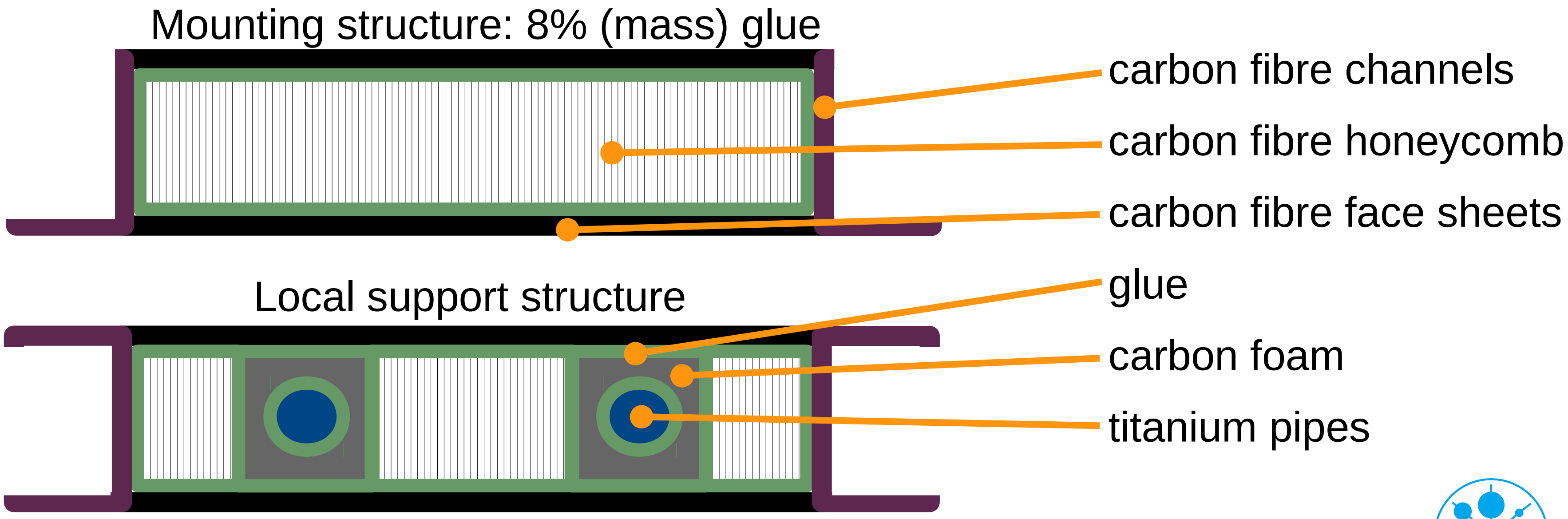
- Lots of samples measured
- Full mechanical characterisation of fibre as used in future local support structures:
  - Modulus depending on alignment angle
  - Shear modulus
  - Poisson's ratio
- Input for realistic simulations of face sheets

Coefficient of moisture expansion of glue

- Concern: support structures are glued in clean rooms (about 40% humidity)
- In ATLAS, the detector will be flushed with dry gas
  - → Moisture will slowly be reduced to 0%
  - → concern: contraction of materials while drying
- Measure coefficient of moisture expansion of glue

CME

$$\frac{\frac{\text{change of length}}{\text{initial length}}}{\frac{\text{mass of absorbed moisture}}{\text{dry mass}}}$$



Samples under investigation

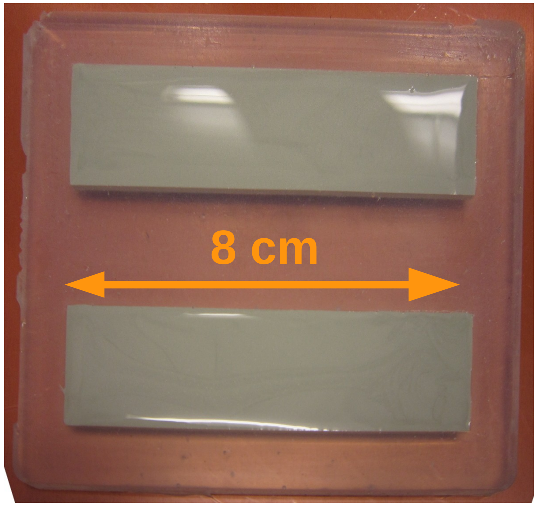
- Glue planned to be used in the detector: Hysol 9396 (two component) with filling for better thermal conduction and higher viscosity

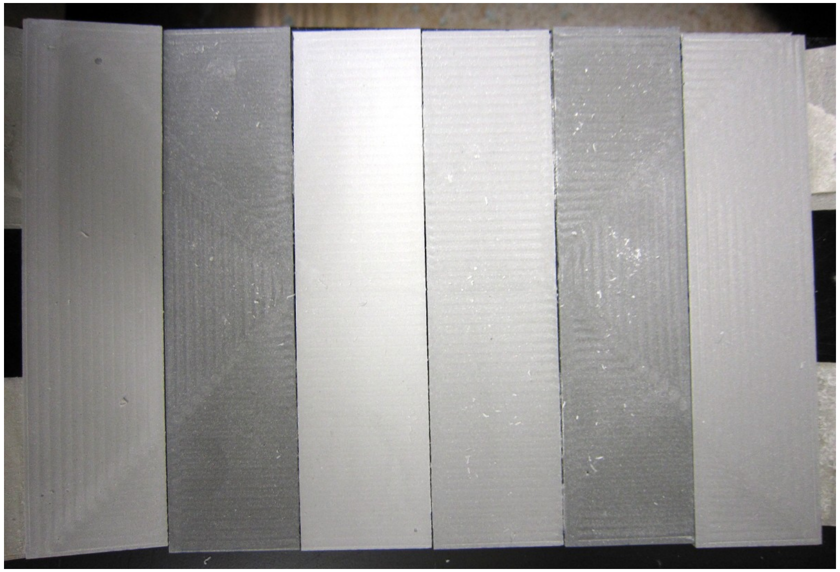
  → mixed samples with 0, 5, 10, 15, 20, 25, 30 % of boron nitride powder to be used in detector

  → two samples per filling percentage (2 cm * 8 cm * 2 mm)

  → half of the samples cured at room temperature, half of them cured at 60°C for one hour

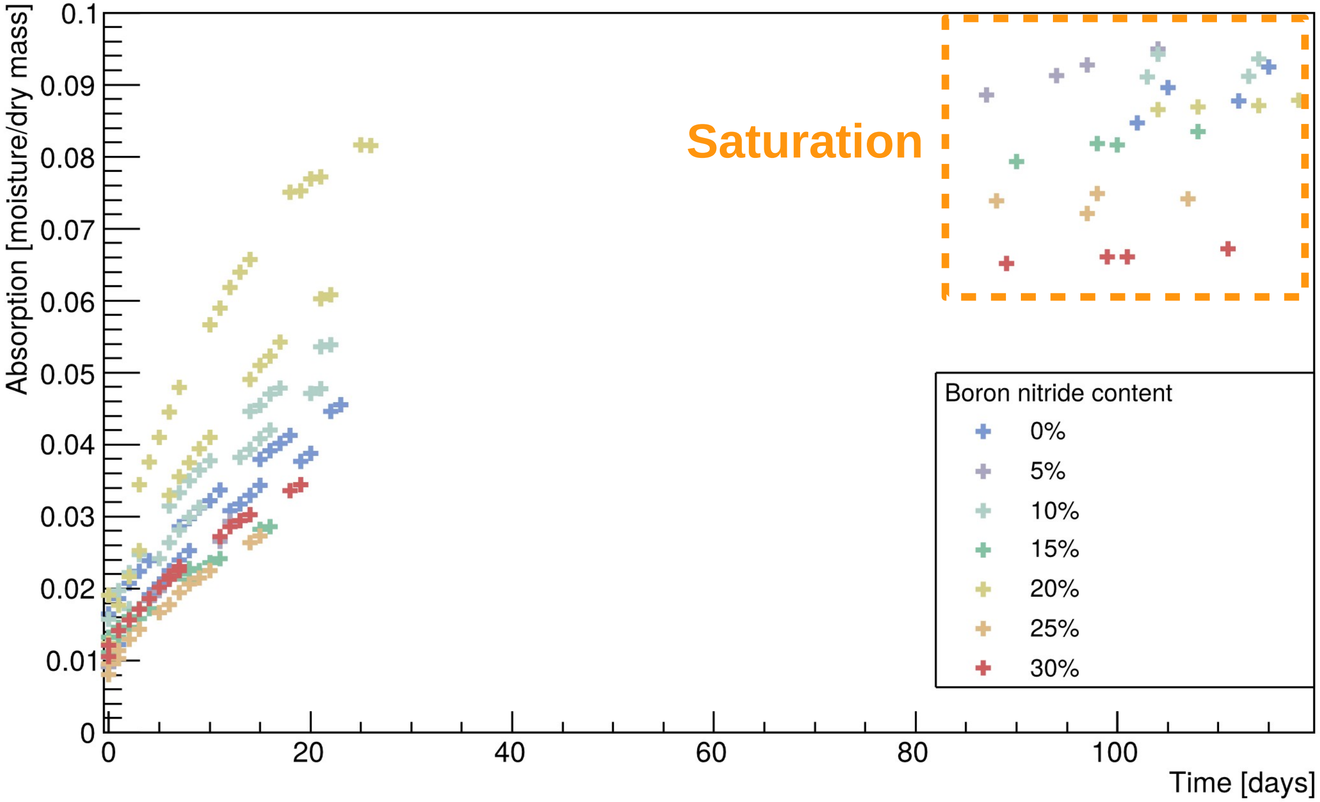
- Surfaces milled flat for easier moisture transfer





Moisture absorption

- Samples dried out completely in a climate chamber
- Then placed in high humidity environment and weighed daily
  - → Estimate maximum absorption



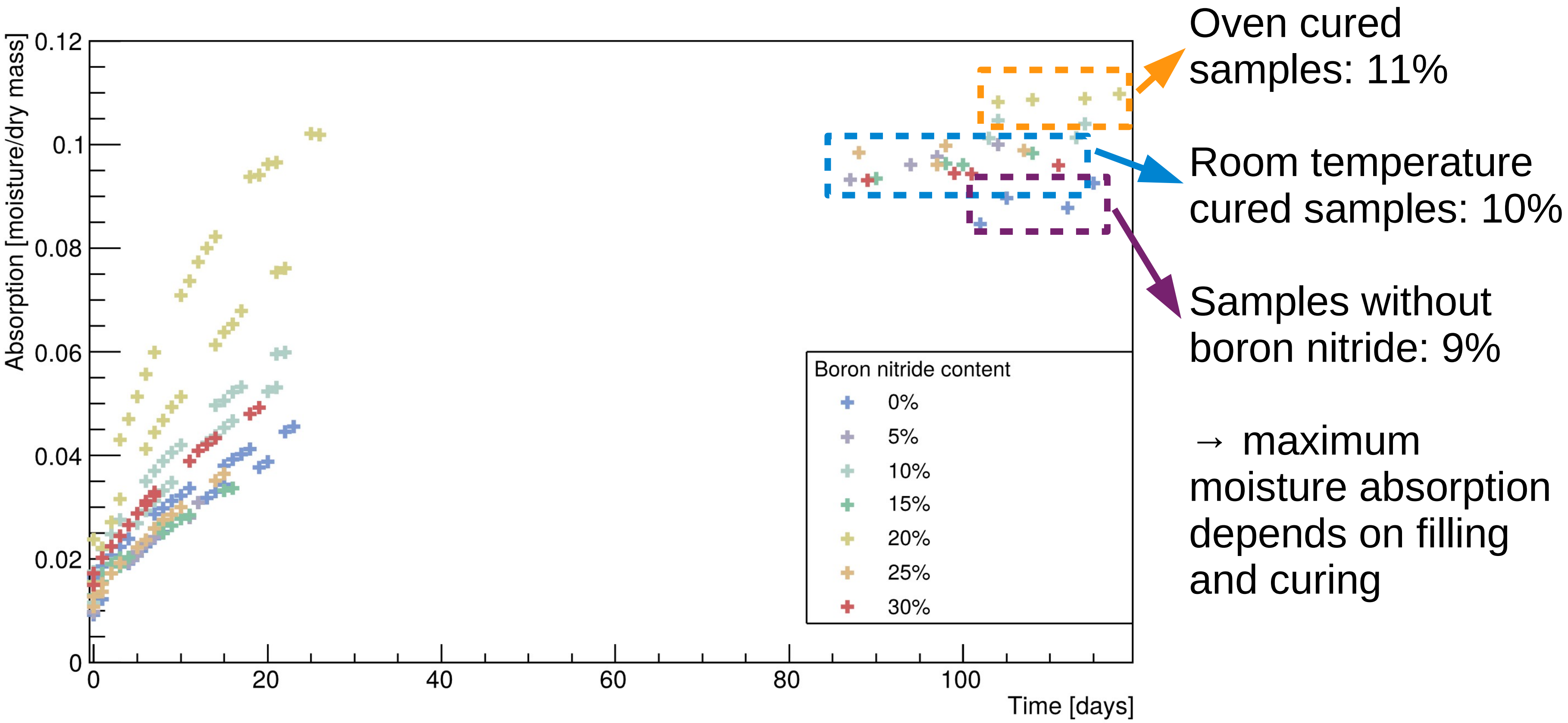
Samples with a higher boron nitride content absorb less moisture

→ expected, as boron nitride is a ceramic (no moisture absorption)

→ account for boron nitride in glue samples

Moisture absorption

- Maximum moisture absorption for glue content only (taking boron nitride content into account): between 9 and 11%



Oven cured samples: 11%

Room temperature cured samples: 10%

Samples without boron nitride: 9%

→ maximum moisture absorption depends on filling and curing

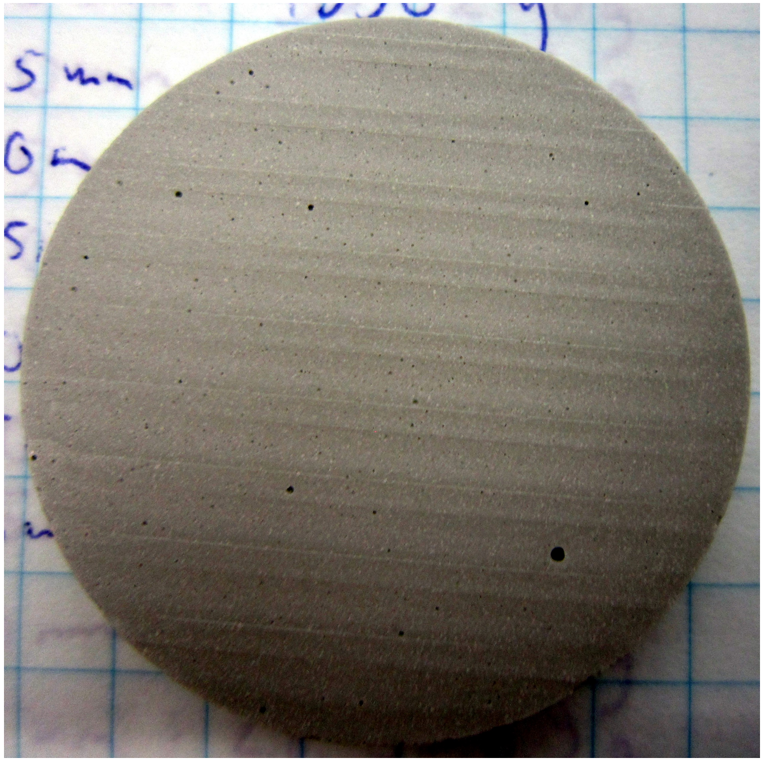
Oven curing vs room temperature curing

- Milled through different glue sample
- Air (from boron nitride powder) trapped in glue
- Effect much stronger for oven curing

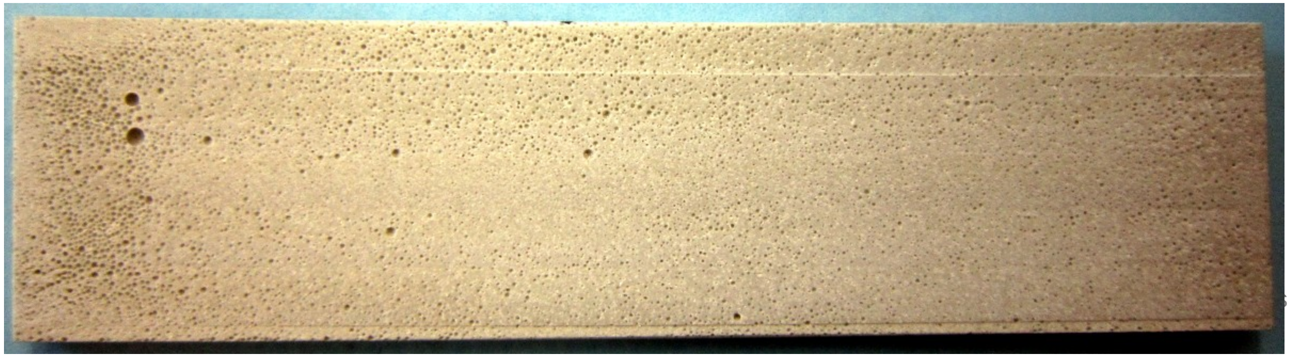
→ glue cures faster at higher temperatures

→ not enough time for air bubbles to escape from glue

→ Moisture absorption can probably be reduced by degassing glue after adding the filling

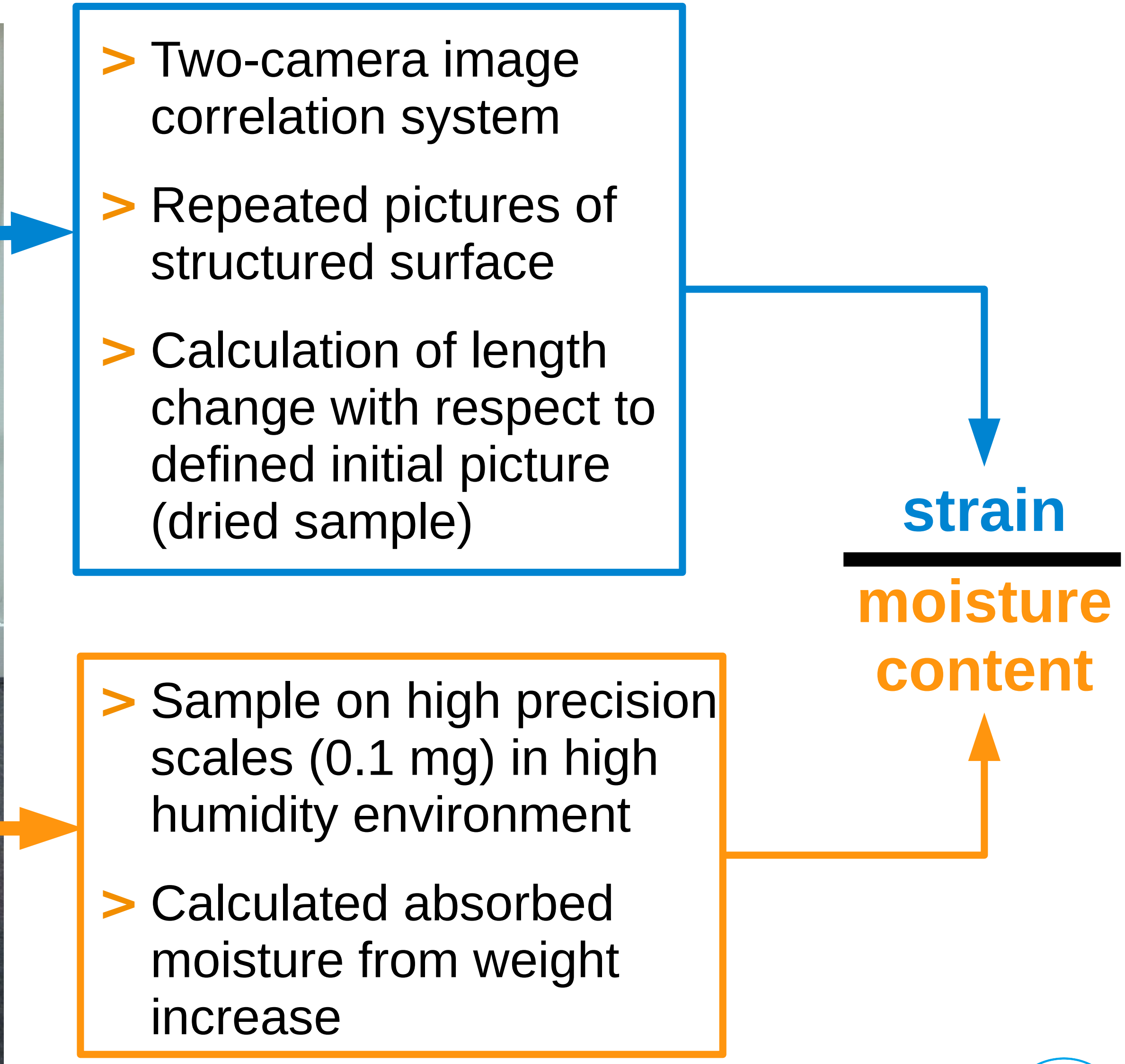


cured at room temperature



cured in an oven

Coefficient of moisture expansion measurement



- Two-camera image correlation system
- Repeated pictures of structured surface
- Calculation of length change with respect to defined initial picture (dried sample)

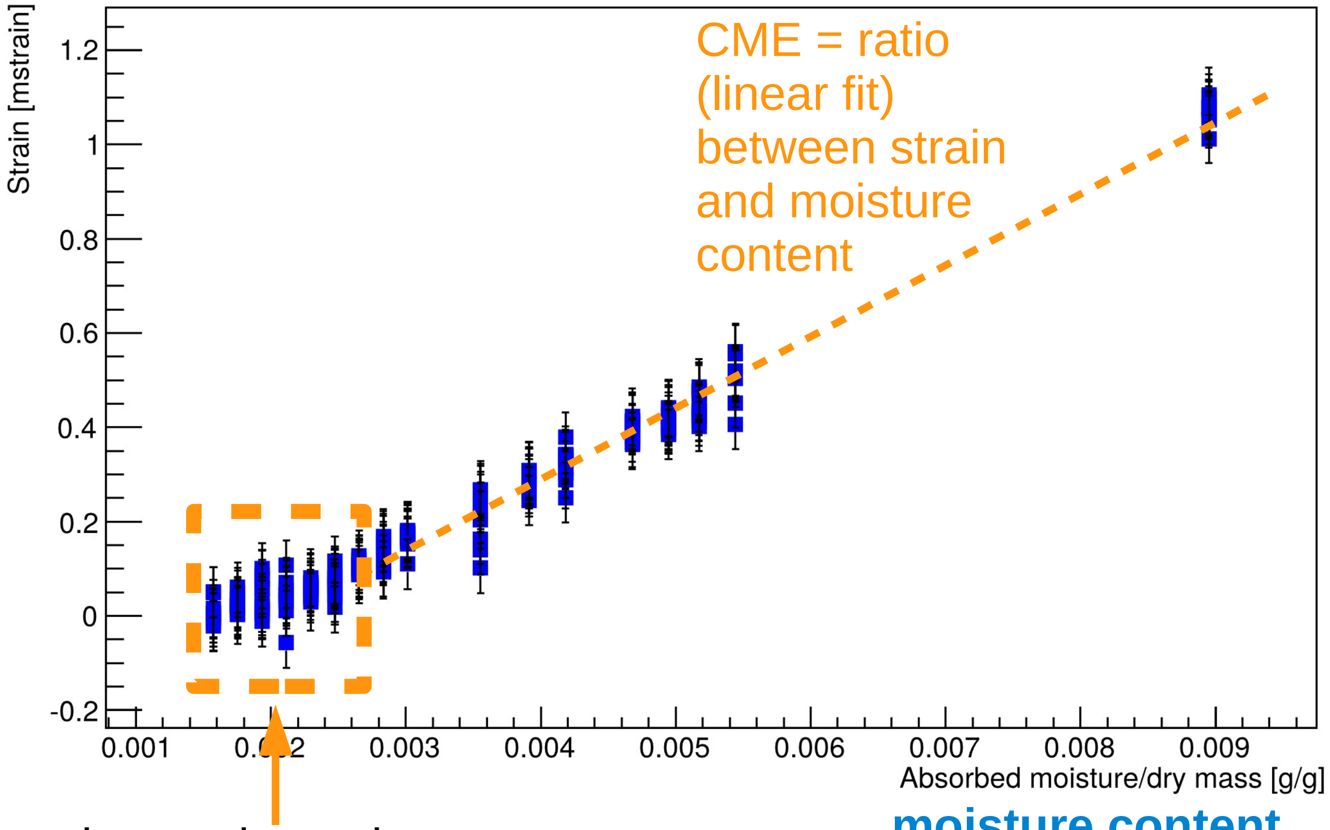
$$\frac{\text{strain}}{\text{moisture content}}$$

- Sample on high precision scales (0.1 mg) in high humidity environment
- Calculated absorbed moisture from weight increase

Coefficients of moisture expansion (example)

strain measured by image correlation system

CME for Hysol with 20% boron nitride



CME = ratio (linear fit) between strain and moisture content

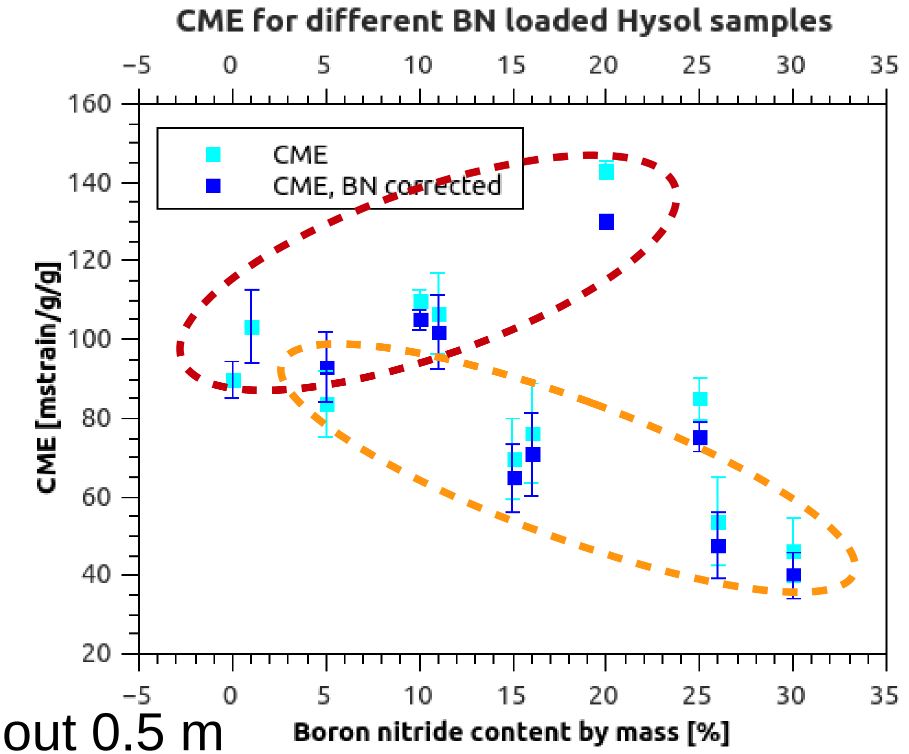
Initial moisture absorption leads to curing of previously uncured glue
→ no expansion

moisture content calculated from

$$\frac{\textbf{weight} - \textbf{dry mass}}{\textbf{dry mass}}$$

Results for different filling percentages

- ≈ 100 mstrain/moisture content
  - → higher for oven cured samples
  - → lower for room temperature cured samples
- Variations can be assumed to be smaller for degassed glue



- Rough estimate of impact:
- Length of a petal support structure: about 0.5 m
  - → assume 40 % humidity during construction
  - → moisture absorption of 0.04 · glue weight (0.1 in 100 % humidity)
  - → contraction during drying: 0.04 · 100 mstrain = 4 mstrain ≈ 2 mm
  - → compared to contraction from temperature change in glue ≈ 2 mm
- This should be taken into account

Still surprising results

Even when we had worked with the glue for some time

- One (slightly warmer) day, the glue was mixed as usual
- On this day, it boiled over

(exothermic glue reaction produces heat, which accelerates curing, which produces more heat

→ air trapped in glue is heated, expands and rises up

→ “boiling over”)

- Never seen before or after