EFFECT OF CURE ON MECHANICAL PROPERTIES OF A COMPOSITE

Part 1 of 2

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Composites Knowledge Network

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Zwick Roell

zwickroell.com

SUPPORTED BY:

Technical University of Munich

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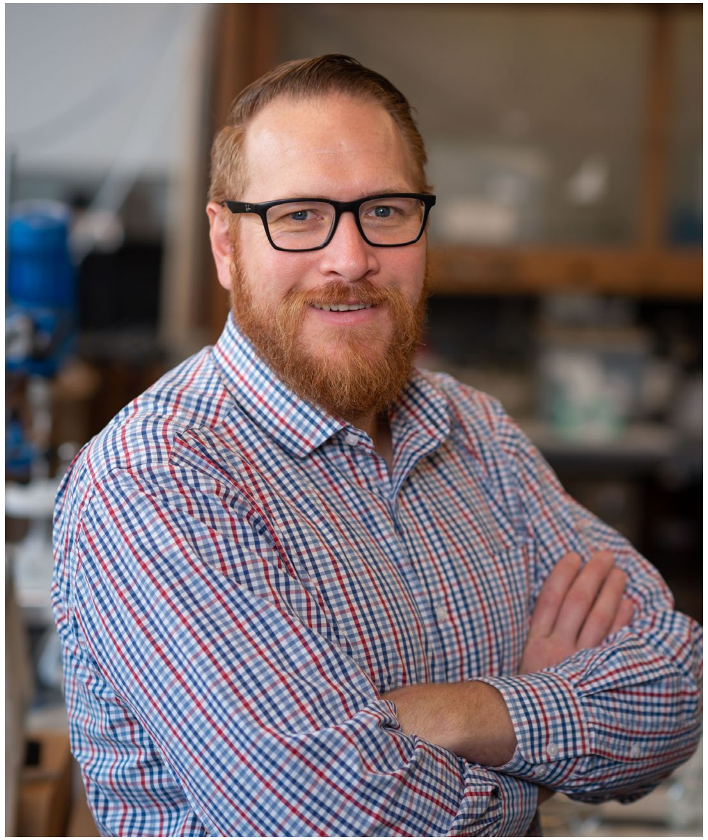
YOUR HOSTS



Casey Keulen, Ph.D, P.Eng.
Assistant Professor of Teaching, University of British Columbia
Co-Director of Advanced Materials Manufacturing MEL Program, UBC
Director of Knowledge in Practice Centre, CKN

- Ph.D. and M.A.Sc. in Composite Materials Engineering
- Over 15 years experience in industry and academia working on polymer matrix composites in aerospace, automotive, marine, energy, recreation and others
- Experience working with over 150 companies from SME to major international corporations
- Expertise in liquid composite moulding and thermal management

YOUR HOSTS

Scott Nesbitt, MASc., P.Eng.
Research Engineer, (CRN/CKN) University of British Columbia

- BSc. in Mechanical Engineering
- MASc. in Materials Engineering (Composites)
- 5 years industrial experience with carbon fibre overwrapped pressure vessels (COPVs)
- Expertise in wet layup and filament winding
- Experience with composite monocoque structures & composite tooling manufacture

YOUR HOSTS

Zwick Roell

Hannes Koerber, PhD, Dipl.-Ing.
Global Industry Manager Composites, Zwick Roell

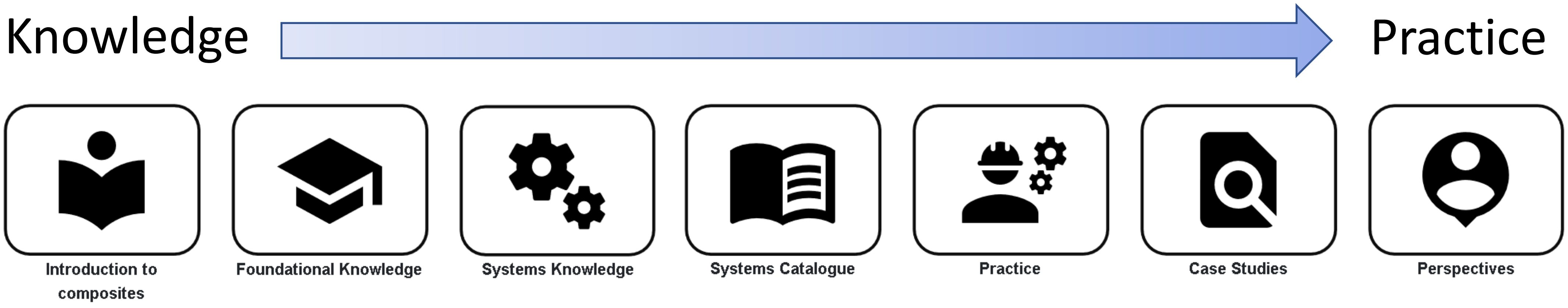
- Aerospace engineering degree from University of Stuttgart, Germany
- PhD on “Mechanical Response of Advanced Composites under High Strain Rates” from University of Porto, Portugal
- Longstanding experience from industry and academia on characterization of composites
- Member of DIN and ISO committees for standardization of composites testing

KNOWLEDGE IN PRACTICE CENTRE (KPC)

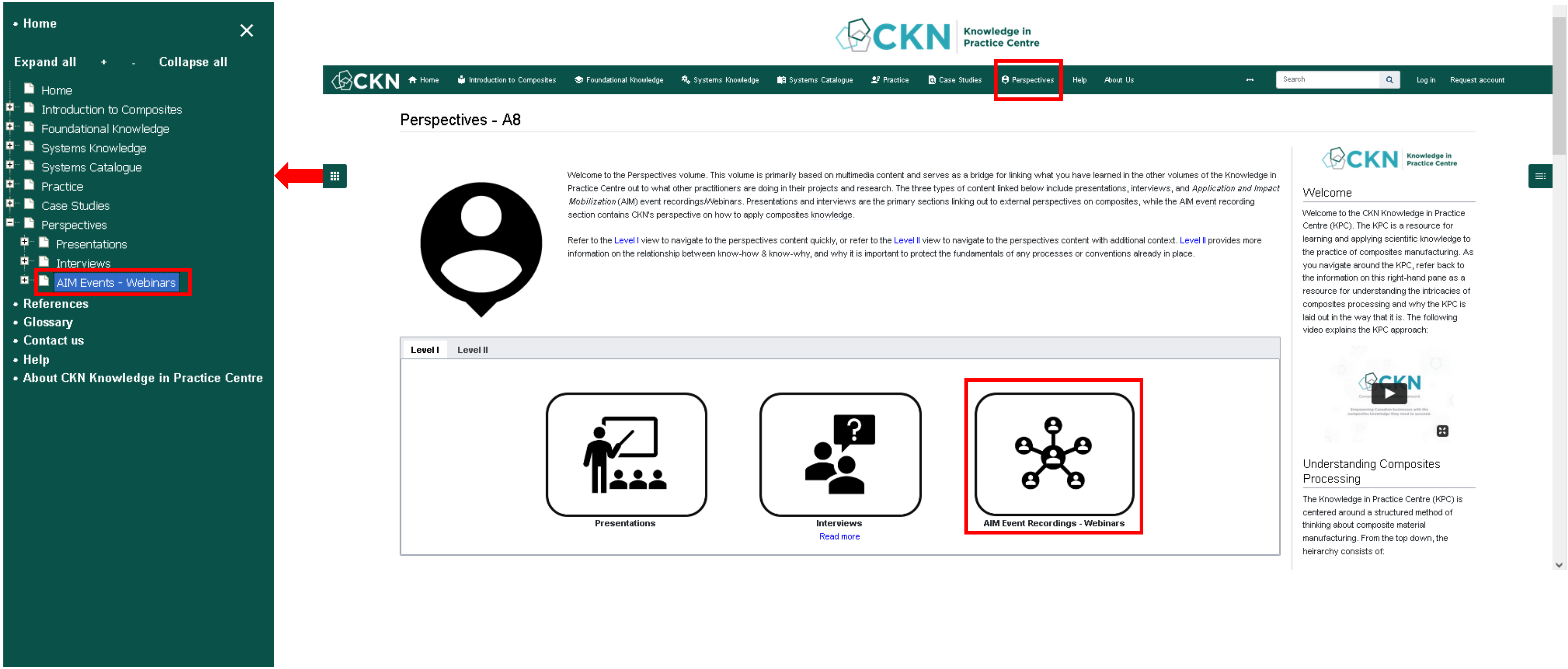
- A freely available online resource for composite materials engineering:

compositeskn.org/KPC

- Focus on practice, guided by foundational knowledge and a systems-based approach to thinking about composites manufacturing



PAST WEBINAR RECORDINGS AVAILABLE



TODAY'S TOPIC:

Effect of cure on mechanical properties of a composite: Part 1 of 2

Part 2 will be presented on February 23, 2022

OUTLINE

- Introduction, learning objectives
- Thermal management of a thermoset curing process
  - 1D thermal system
- How curing issues can arise in practice
- Experimental investigation of under cure/MRCC/thermally degraded material
  - Tensile
  - Interlaminar shear
  - Compression
  - In-plane shear

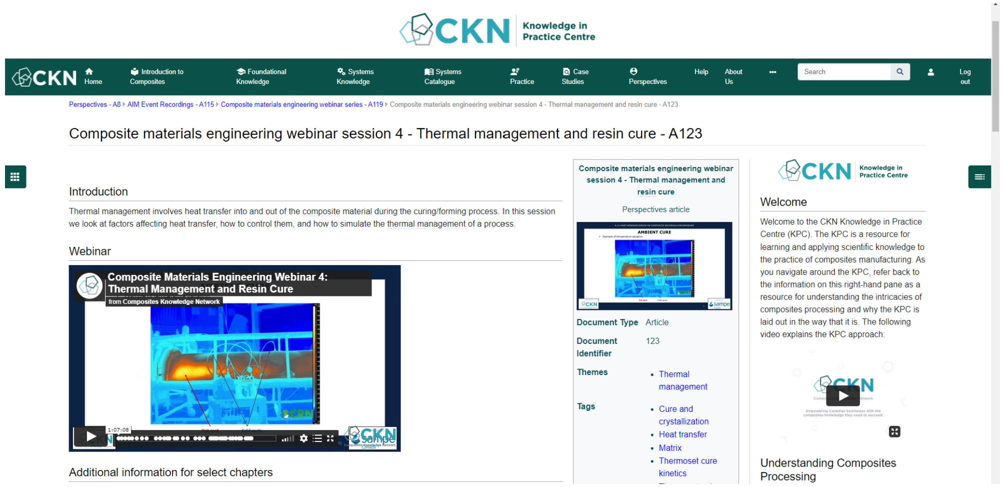
INTRODUCTION

- Today we'll be talking about the effect of thermal management on a composite's properties
  - *How does the cure affect the performance of a composite?*

- Learning objectives:
  - Understand degree of cure
  - Understand the basics of heat transfer during cure of a thermoset part
  - Understand the effect of thermal management on mechanical properties and which properties are more/less affected
  - Understand how this shows up in a real situation

THERMAL MANAGEMENT

- We've discussed thermal management in the past
- We know why it's important in terms of measurable thermo-chemical properties, but what about mechanical properties?



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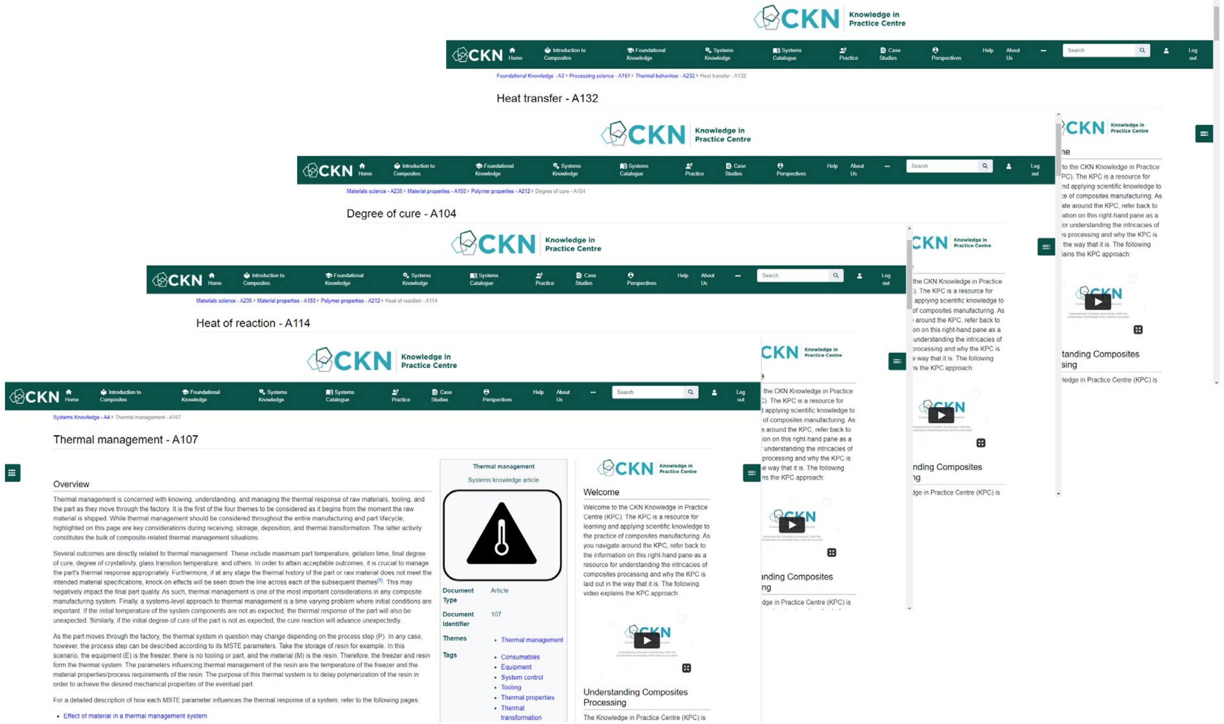

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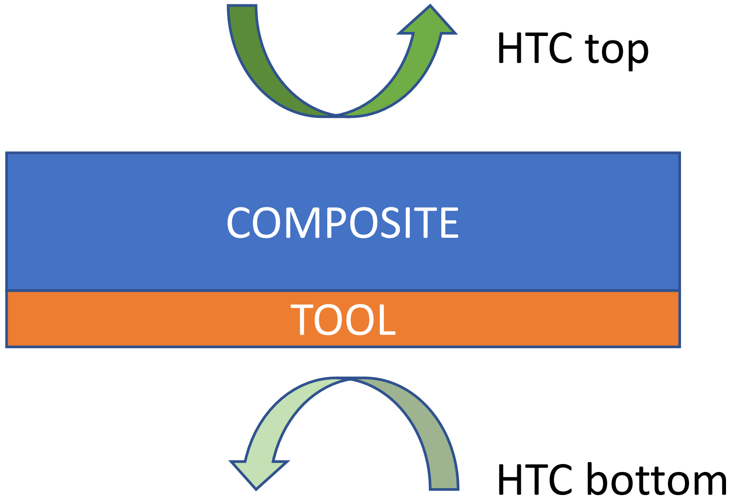
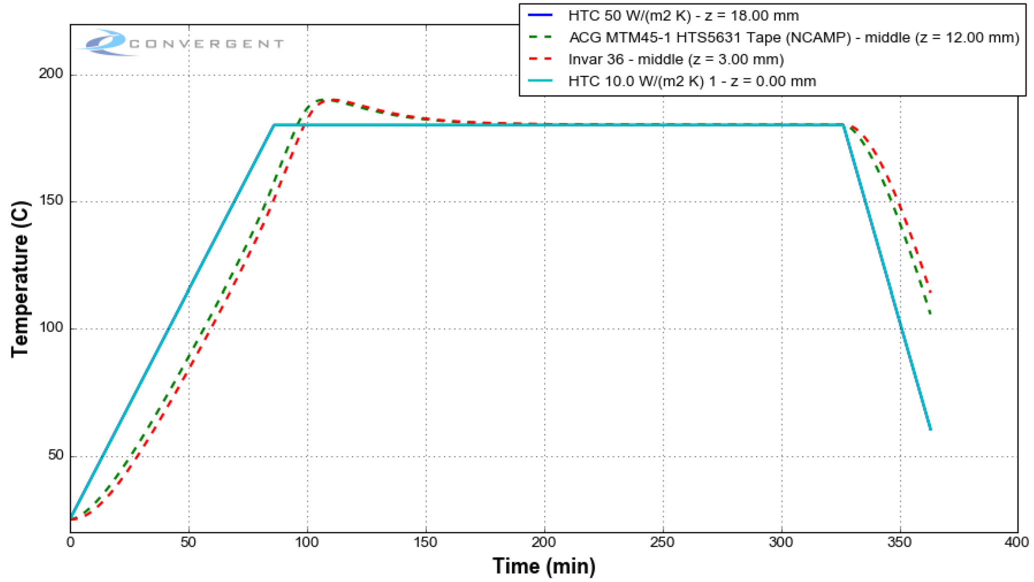
THERMAL MANAGEMENT

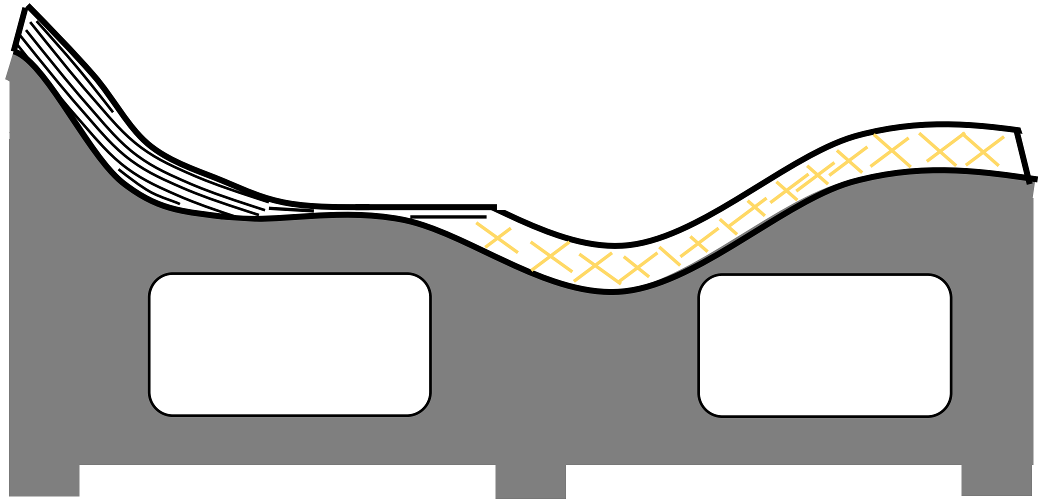
- 1D heat transfer model
  - Composite generates heat (H_R)
  - Tool has geometric and thermal properties
  - Both are in a curing environment (HTC, could be conduction)
  - Modelling and commercial simulation tools can be used to model this





WHERE CAN WE RUN INTO PROBLEMS?

- Uneven cure can arise in a variety of scenarios
  - Poor/uneven HTC in oven/autoclave
  - Part with varying thickness
  - Sandwich part
  - Improper oven/autoclave control



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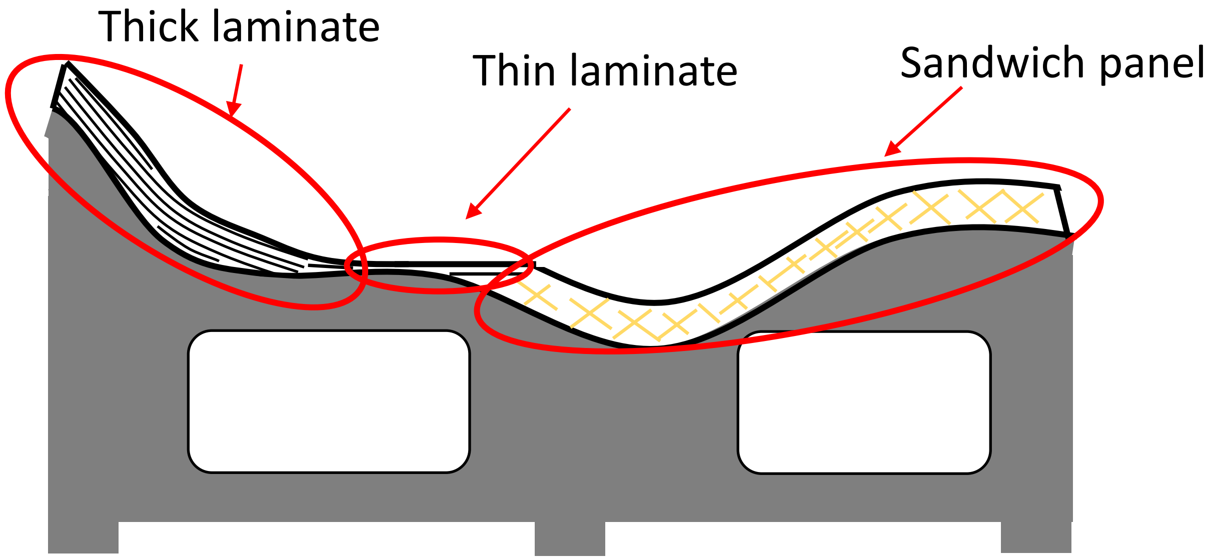

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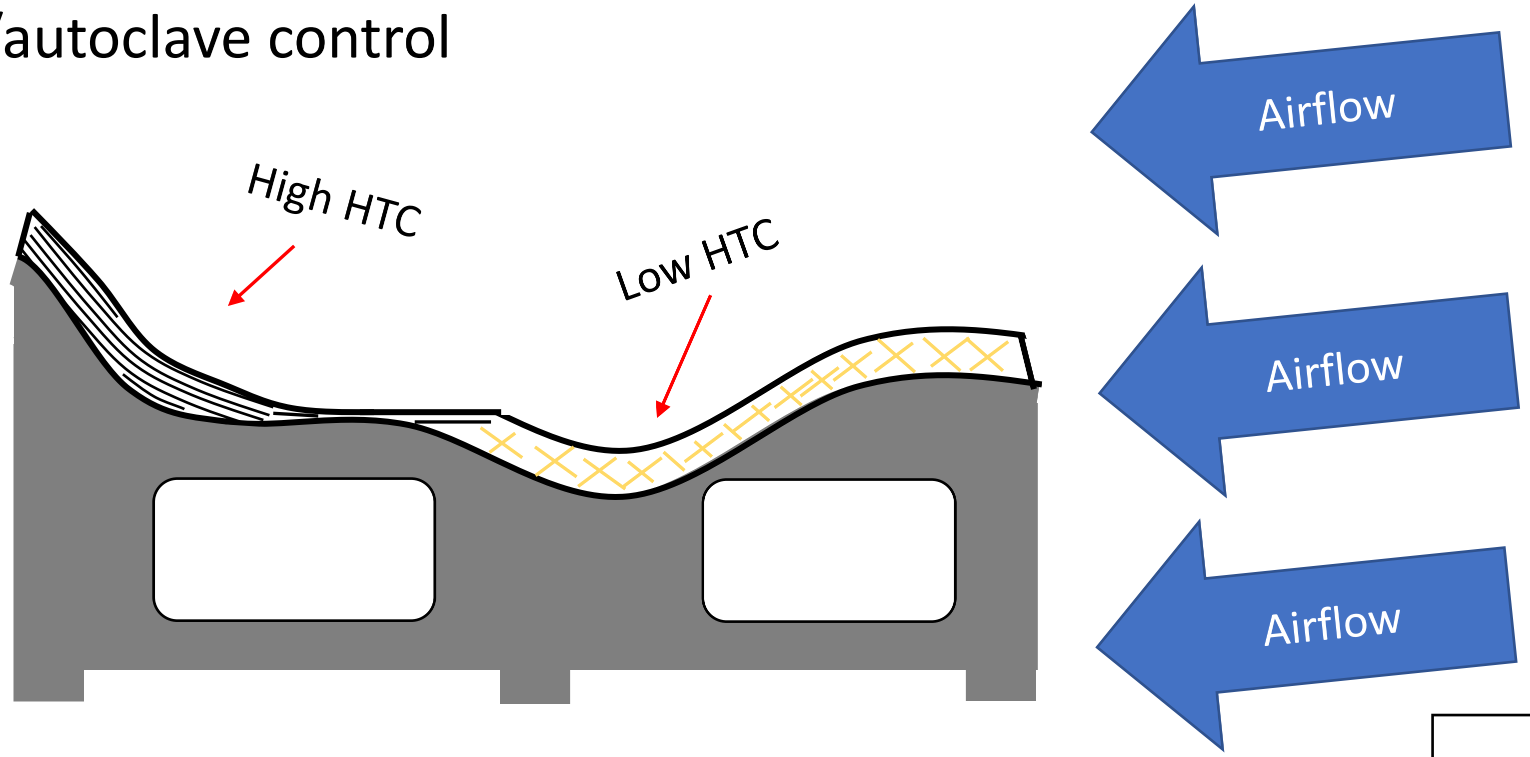

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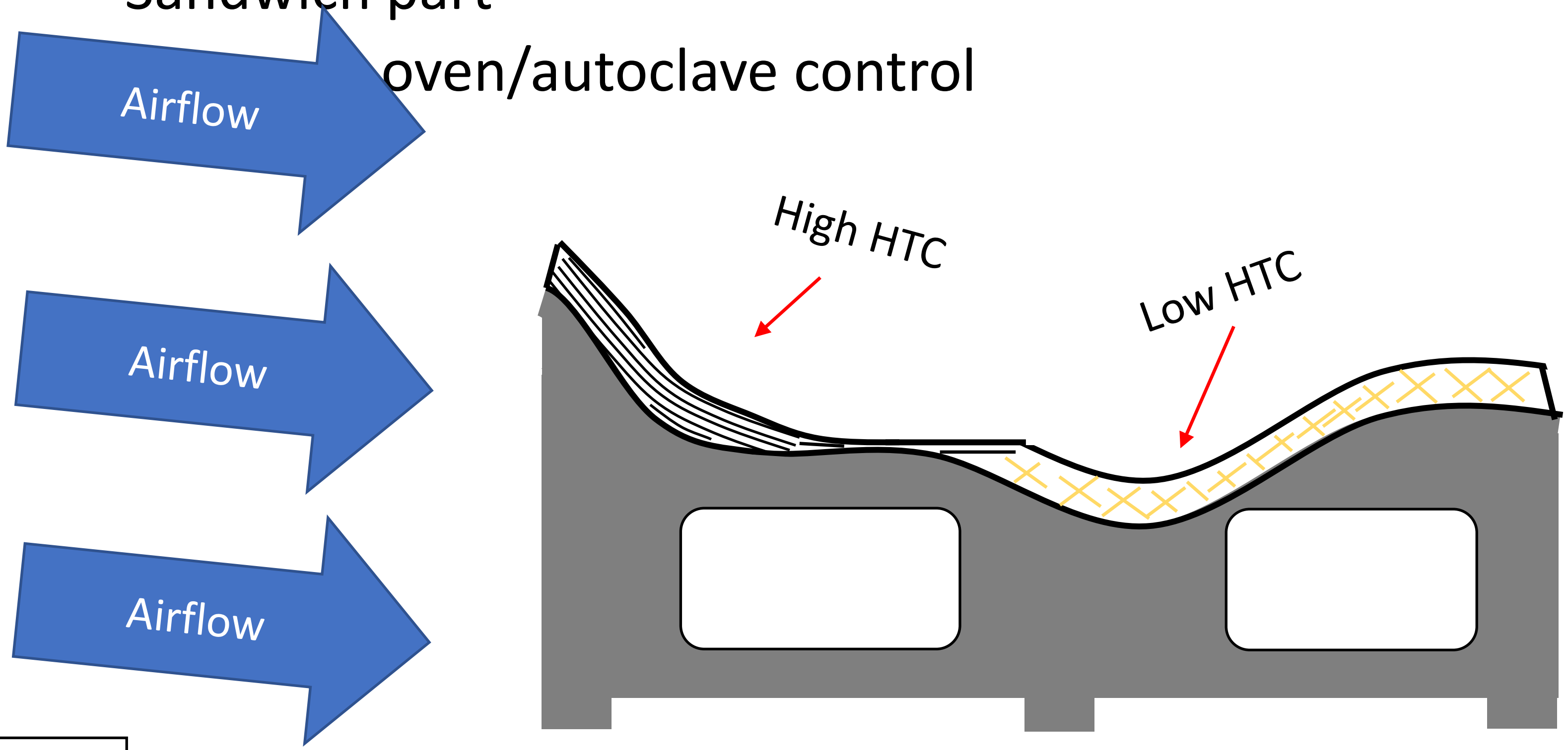

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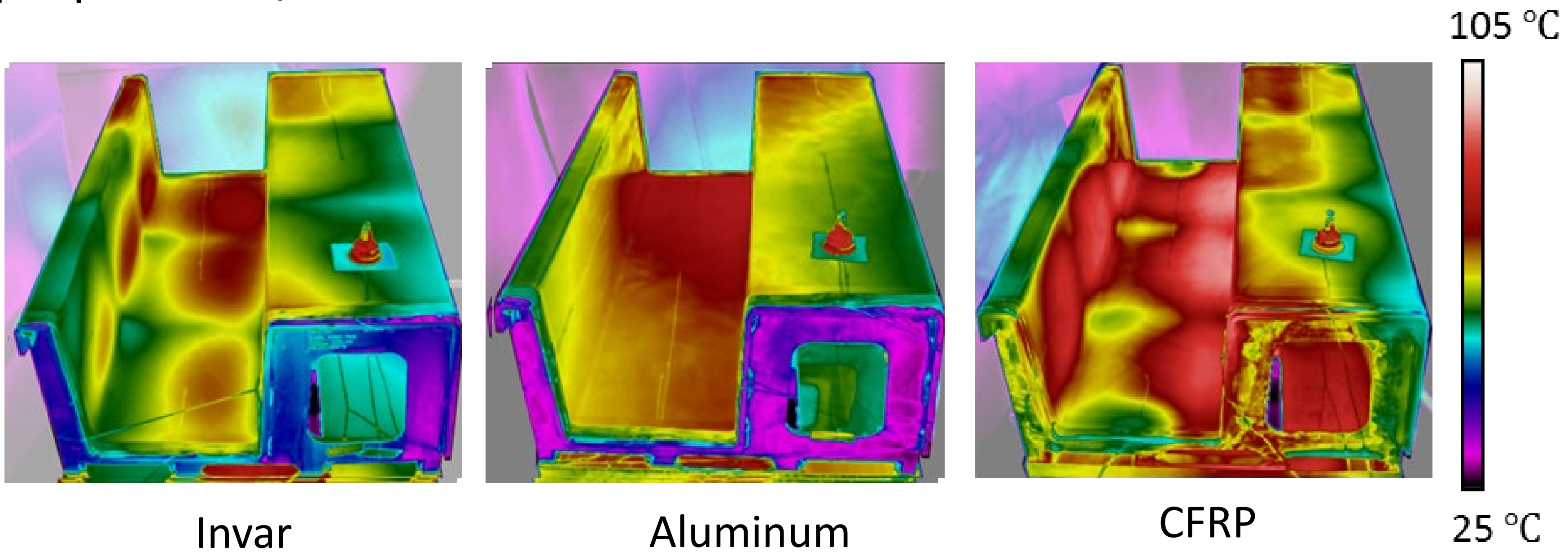


IR images of three tools immediately after a heating cycle of 5 °C/min to 100 °C

LET'S LOOK AT THE EFFECTS THROUGH A CASE STUDY

- The stage is set:
  - We know what's happening during cure
  - We know how these issues can arise during processing
  - What are the outcomes?
    - Let's look at the mechanical properties of parts with different thermal/cure histories
  - 'Goldilocks and the Four Bears' case study

Extreme under-cure
Designated "1A"

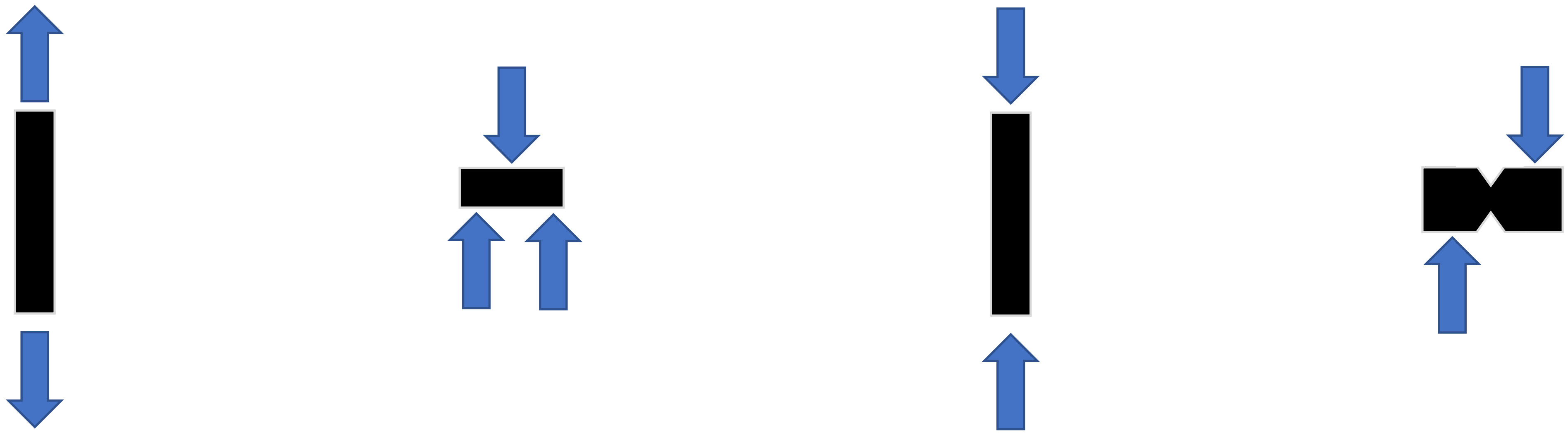
Under-cure
Designated "2A"

MRCC cured
(NCAMP Baseline 'MH Cure')
Designated "3A"

Thermally degraded
Designated "4A"

'GOLDILOCKS AND THE FOUR BEARS' CASE STUDY

- Performed the following mechanical tests:



Tensile
ASTM D3039

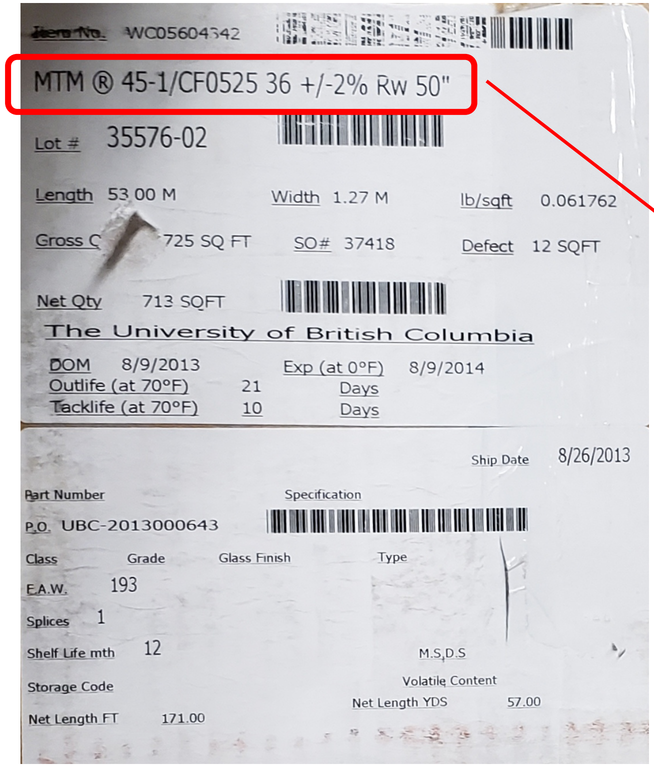
Short Beam Shear
ASTM D2344

Compression
Combined Loading
ASTM D6641

In-plane Shear
Iosipescu
ASTM D5379

'GOLDILOCKS AND THE FOUR BEARS' CASE STUDY

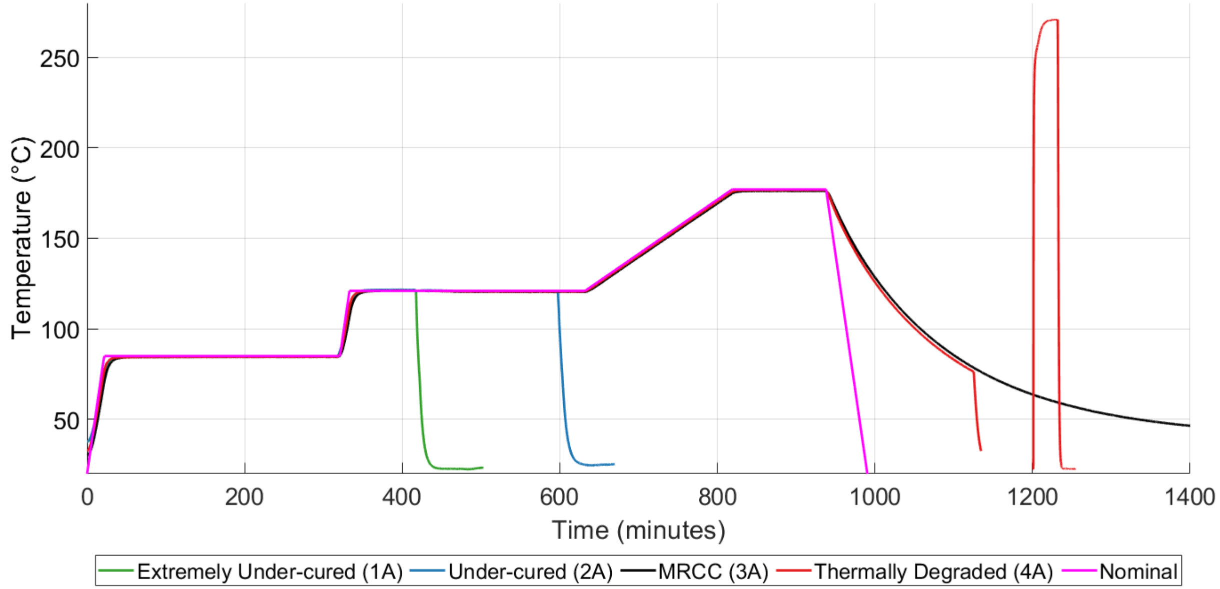
- Material:
  - MTM 45-1, woven AS4-3K, 193 gsm FAW, 36%RW
  - 14 layers, 0/90 plain weave; 2.81 mm nominal thickness
- NCAMP has results of MRCC published for reference



Material Specification – ACGM1001	Nomenclature	Fibre/Style	Fibre areal weight [FAW] (g/m^2)	Resin weight [RW] (%)	Fibre volume fraction [Vf] (%)	Cured ply thickness [CPT] (mm)
1001-03	MTM45-1/4581-35%RW	AQIII/ 8HS fabric	288	35	50.01	0.262
1001-04	MTM45-1/GF0103-35%RW	E-glass/ 7781 8HS fabric	300	35	46.42	0.254
1001-06	MTM45-1/IM7-145g/m^2-32%RW	IM7/ Unidirectional	145	32	58.72	0.140
1001-07	MTM45-1/CF0525-36%RW	AS4/ 3K PW fabric	193	36	54.34	0.201
1001-10	MTM45-1/CF2425-36%RW	AS4/ 6k 5HS fabric	375	36	54.34	0.389
1001-11	MTM45-1/AS4-145g/m^2-32%RW	AS4/ Unidirectional	145	32	58.72	0.140
1001-12	MTM45-1/6781-35%RW	S2-glass/ 8HS fabric	300	35	47.02	0.257
1001-13	MTM45-1/CF0526-36%RW	G30-500/ 3K PW fabric	193	36	54.48	0.201
1001-14	MTM45-1/HTS5631-145g/m^2-32%RW	HTS5631/ Unidirectional	145	32	58.86	0.140

'GOLDILOCKS AND THE FOUR BEARS' CASE STUDY

- Panels were cured under four conditions:
  - Extremely under-cured – "1A"
  - Under-cured – "2A"
  - Manufacturer's recommended cure cycle (MRCC) - "3A"
    - NCAMP Baseline "MH Cure"
  - Thermally degraded – "4A"



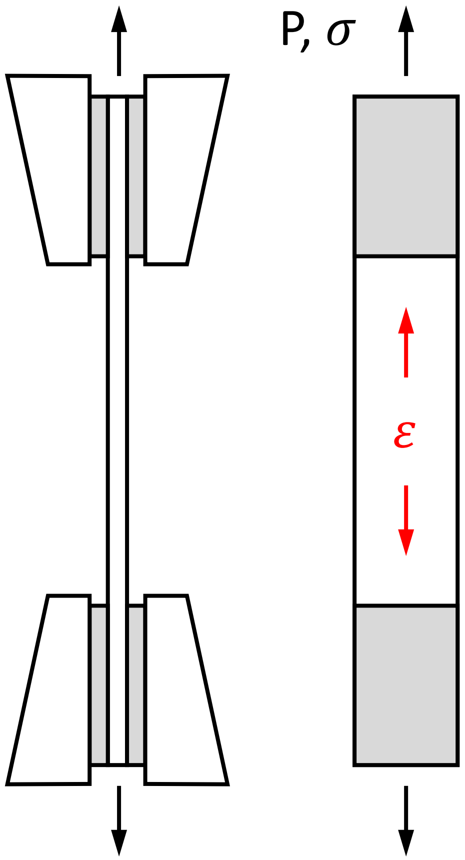
Oven Load

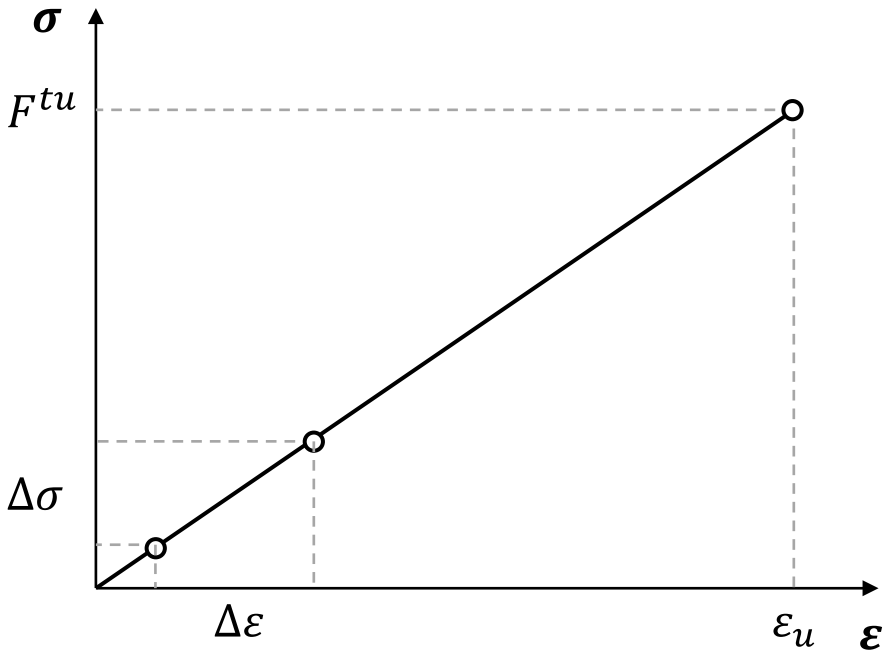
Thermal "Quench"

EXPECTATIONS AND CONVENTIONAL WISDOM

- What results do we expect to see?
- Which properties are fibre dominated, which are resin dominated?
- How much effect, if any will under-cure/thermally degraded have?

TENSILE TESTING – ASTM D3039





$$E_t^{chord} = \frac{\Delta\sigma}{\Delta\varepsilon}$$

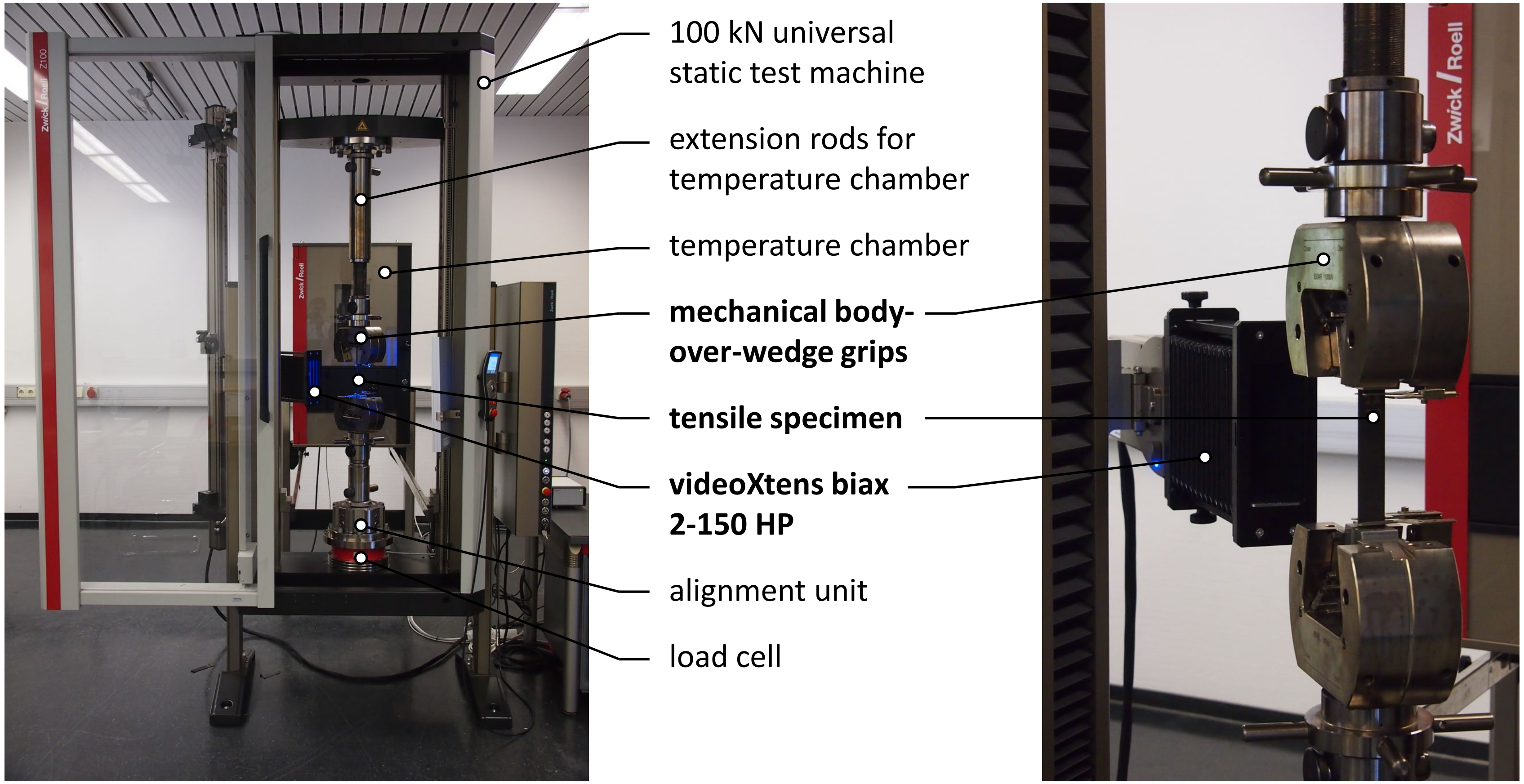
$$F^{tu} = \frac{P^{max}}{A}$$

where:

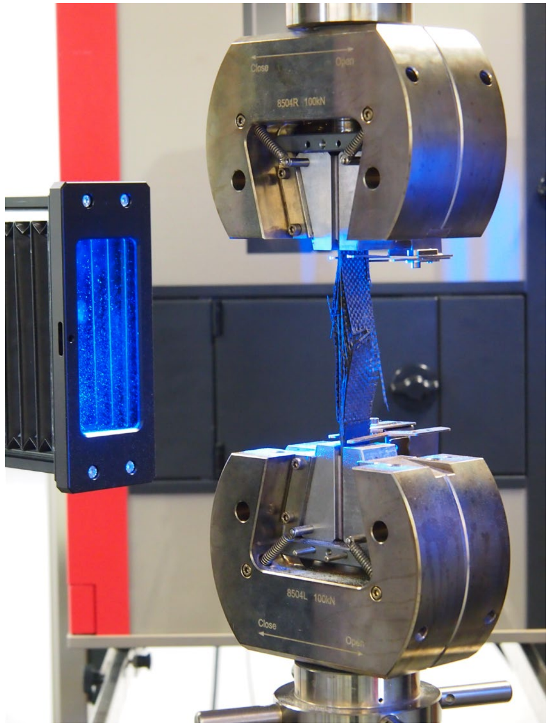
E_t^{chord}	tensile chord modulus of elasticity
F^{tu}	ultimate tensile strength
P^{max}	maximum measured force
A	cross-sectional area of specimen
$\Delta\varepsilon$	difference between strain points (1000 to 3000 $\mu\varepsilon$)
$\Delta\sigma$	difference between applied tensile stress between defined strain points
ε_u	strain at failure

TENSILE TESTING – ASTM D3039

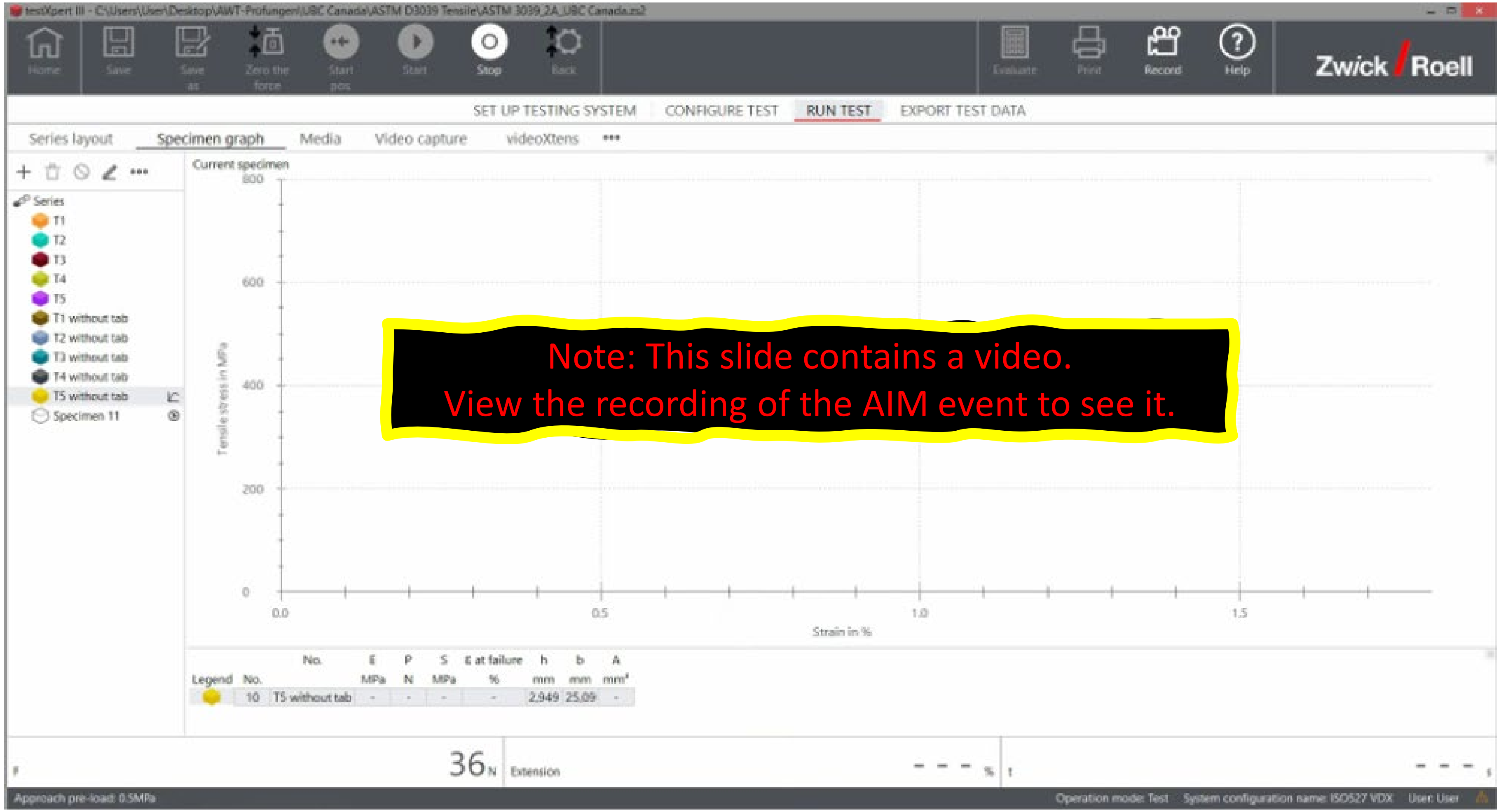
Test setup



Tested specimen of test series 1A



TENSILE TESTING – ASTM D3039



Note: This slide contains a video.
View the recording of the AIM event to see it.

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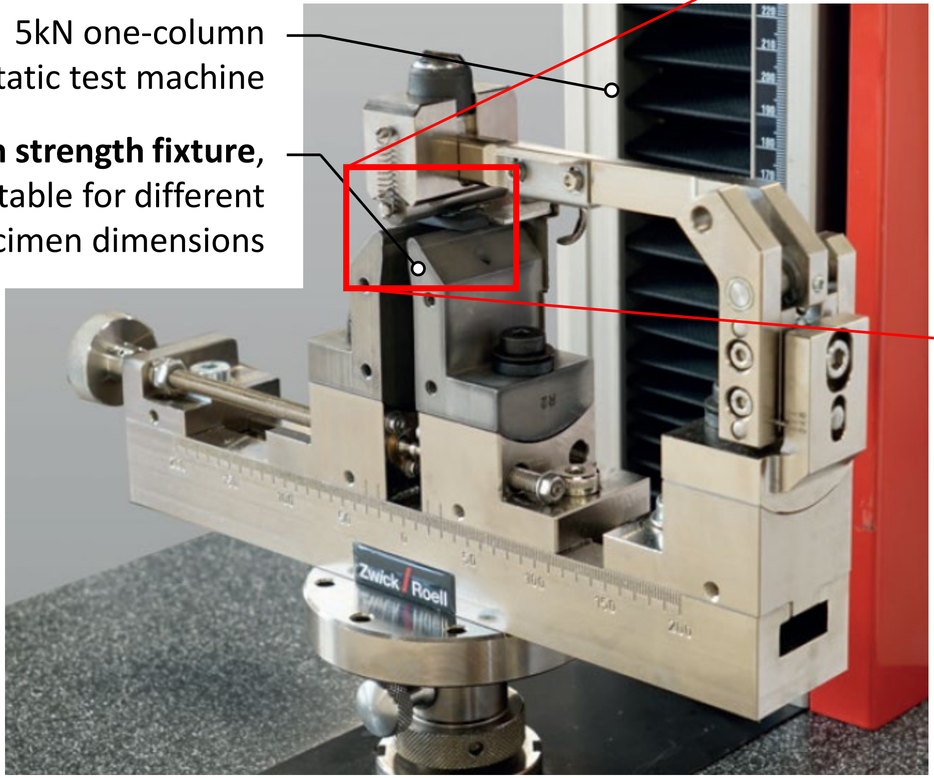
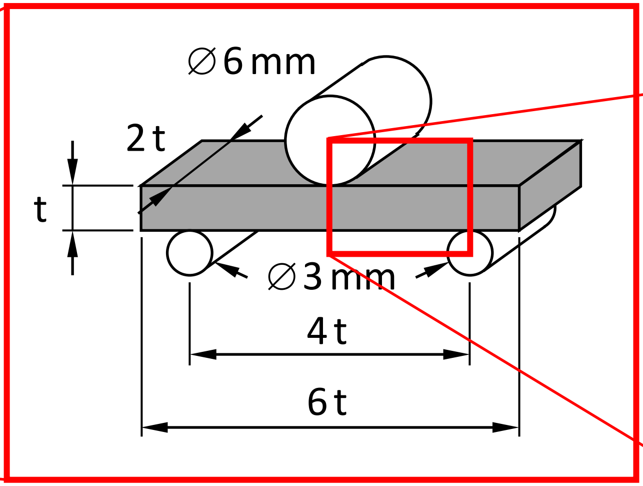
SHORT BEAM SHEAR TESTING (ASTM D2344)

Test setup

5kN one-column universal static test machine

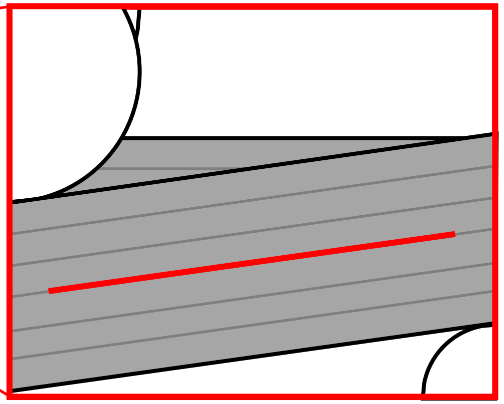
short-beam strength fixture, fully adjustable for different specimen dimensions

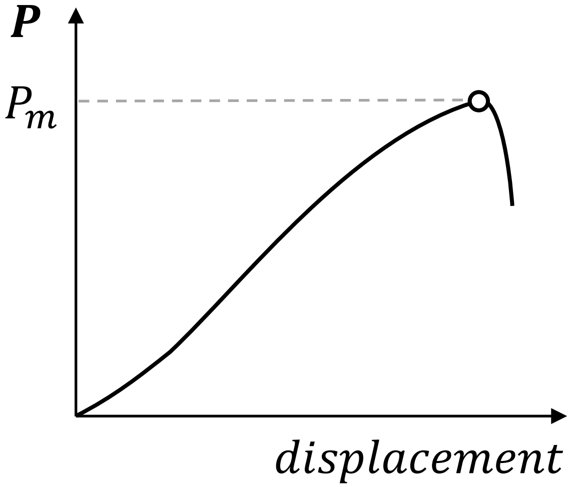




deformed with interlaminar shear failure

initial state



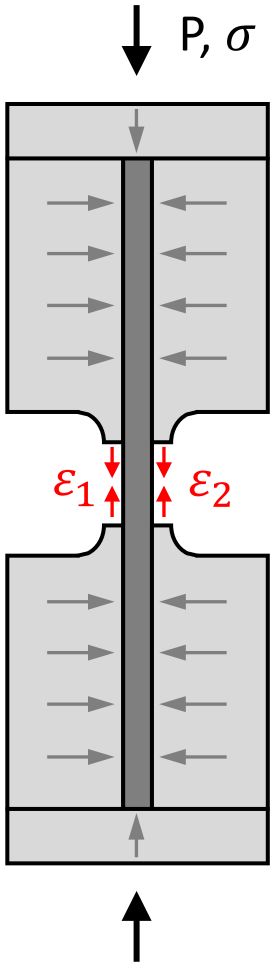


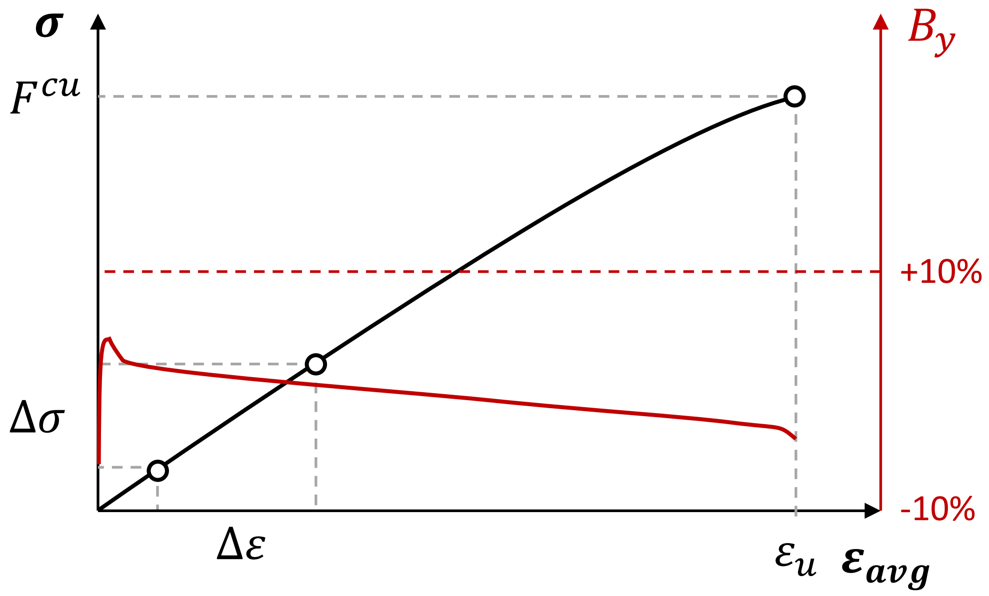
$$F^{sbs} = 0.75 \times \frac{P_m}{b\,h}$$

where:

F^{sbs}	short-beam strength
P_m	maximum applied load
b	specimen width
h	specimen thickness

COMPRESSION TESTING – ASTM D6641





$$E_c^{chord} = \frac{\Delta\sigma}{\Delta\varepsilon}$$

$$F^{cu} = \frac{P^{max}}{A}$$

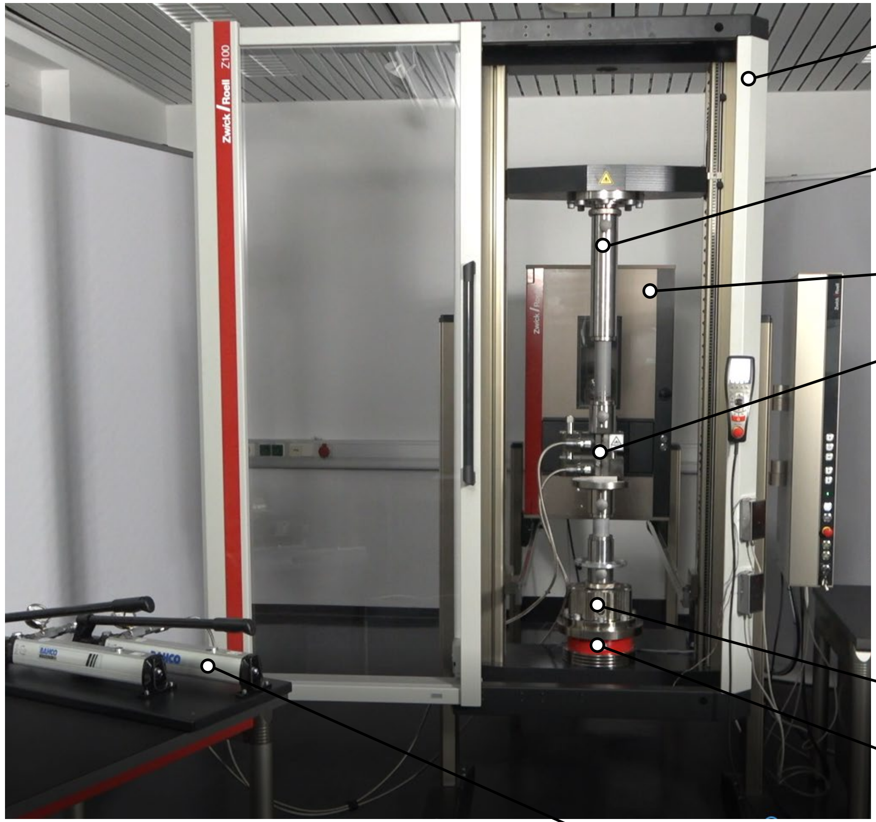
$$B_y = \frac{\varepsilon_1 - \varepsilon_2}{\varepsilon_1 + \varepsilon_2} \times 100$$

where:

E_c^{chord}	compression chord modulus of elasticity
F^{cu}	ultimate compression strength
P^{max}	maximum measured force
A	cross-sectional area of specimen
$\Delta\varepsilon$	difference between strain points (1000 to 3000 $\mu\varepsilon$) of averaged axial strain measurement
$\Delta\sigma$	difference between applied tensile stress between defined strain points
ε_u	strain at failure
ε_1	axial compression strain at strain measuring position 1
ε_2	axial compression strain at strain measuring position 2
B_y	percent bending criteria

COMPRESSION TESTING – ASTM D6641

Test setup



100 kN universal static test machine

extension rods for temperature chamber

temperature chamber

Hydraulic Composites Compression Fixture (HCCF)

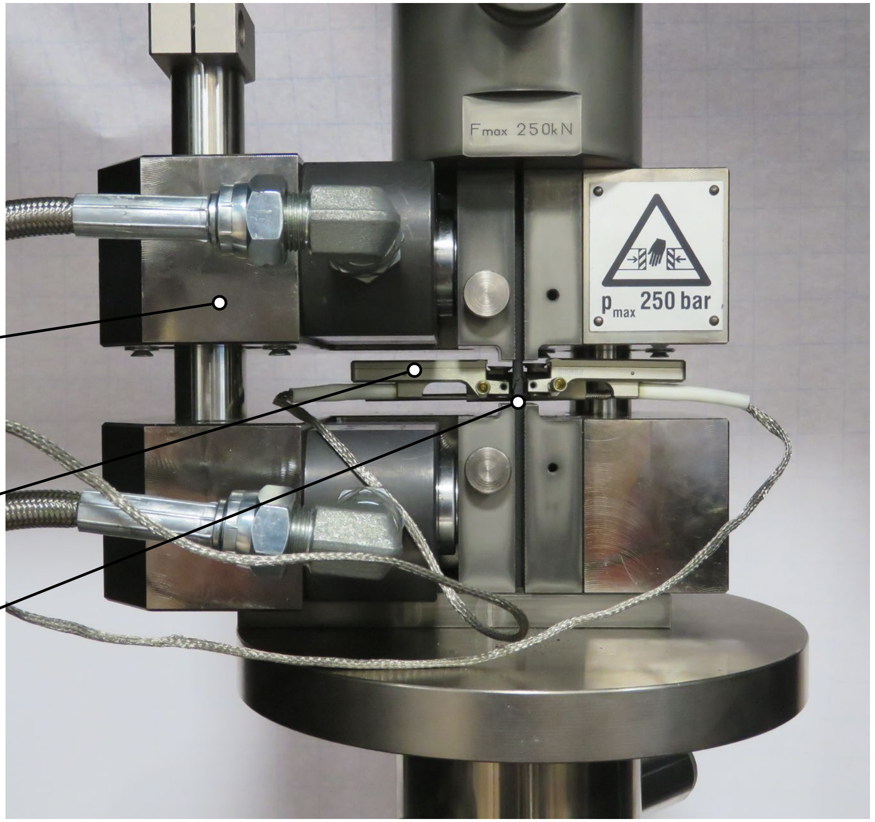
double-sided axial clip-on extensometer

compression sample

alignment unit

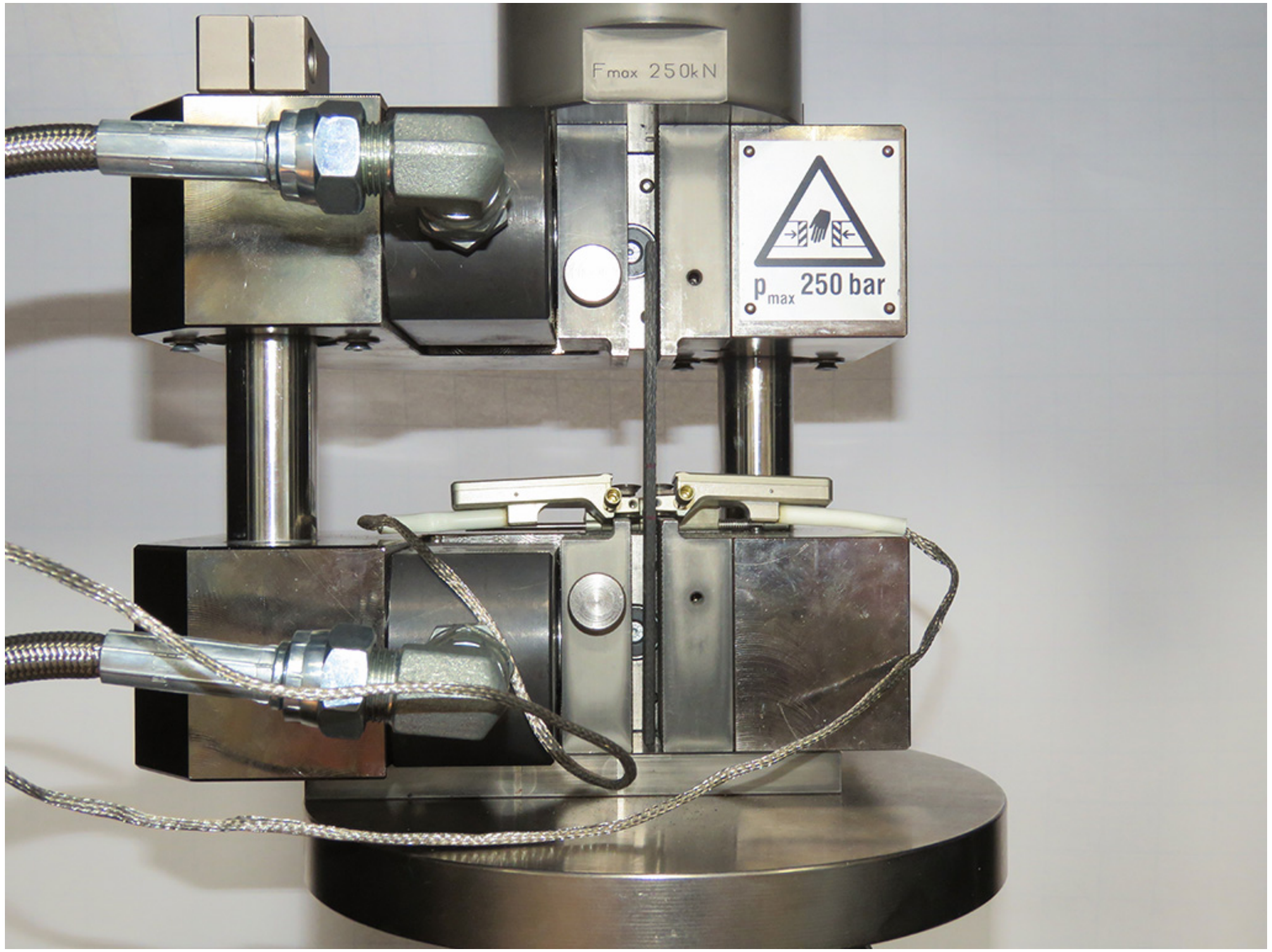
load cell

hydraulic hand pumps



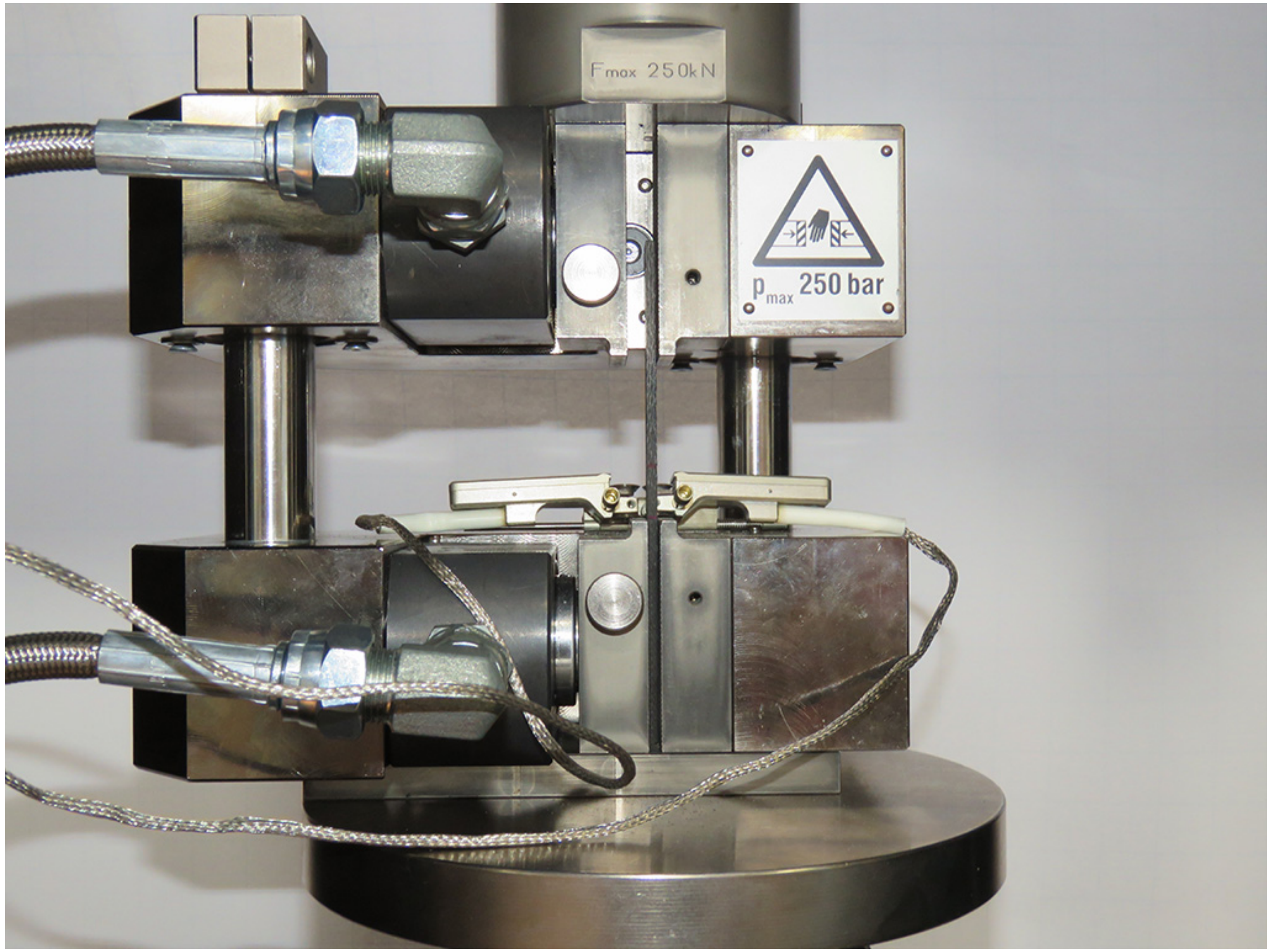
COMPRESSION TESTING – ASTM D6641

Test sequence: Inserting the specimen into the test fixture



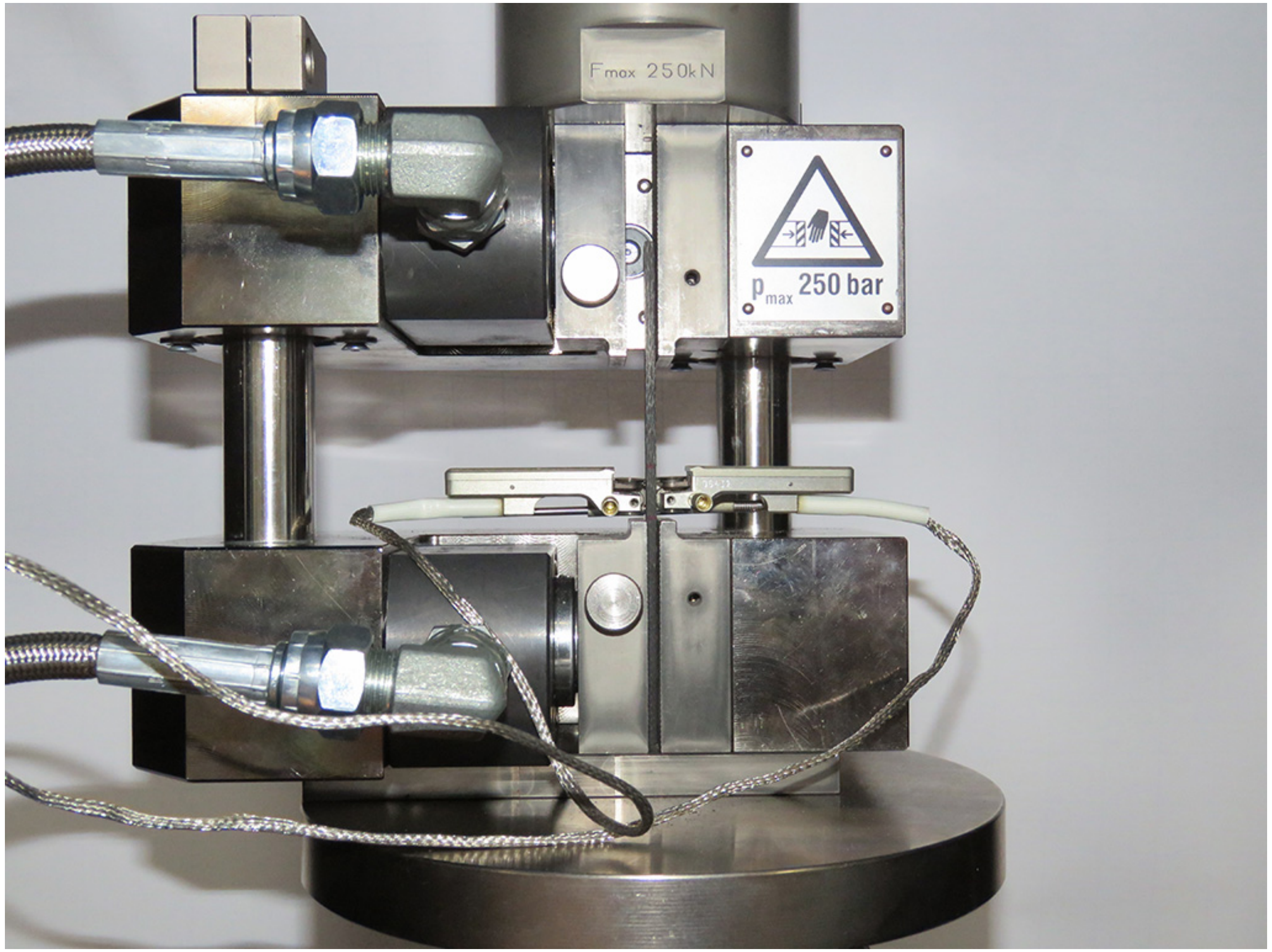
COMPRESSION TESTING – ASTM D6641

Test sequence: Closing the lower grip (holding the specimen in place)



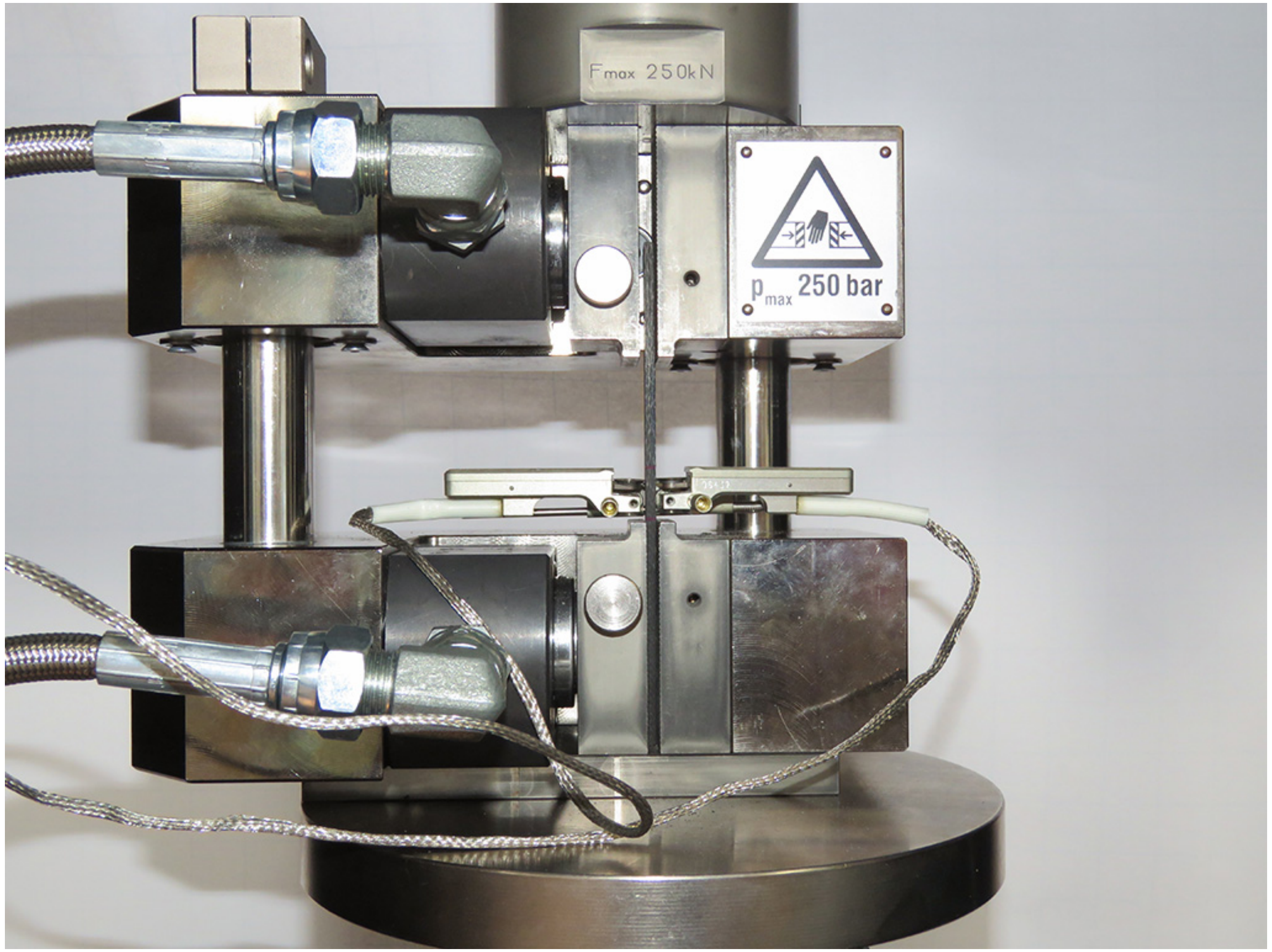
COMPRESSION TESTING – ASTM D6641

Test sequence: Attaching the double-sided clip-on extensometer



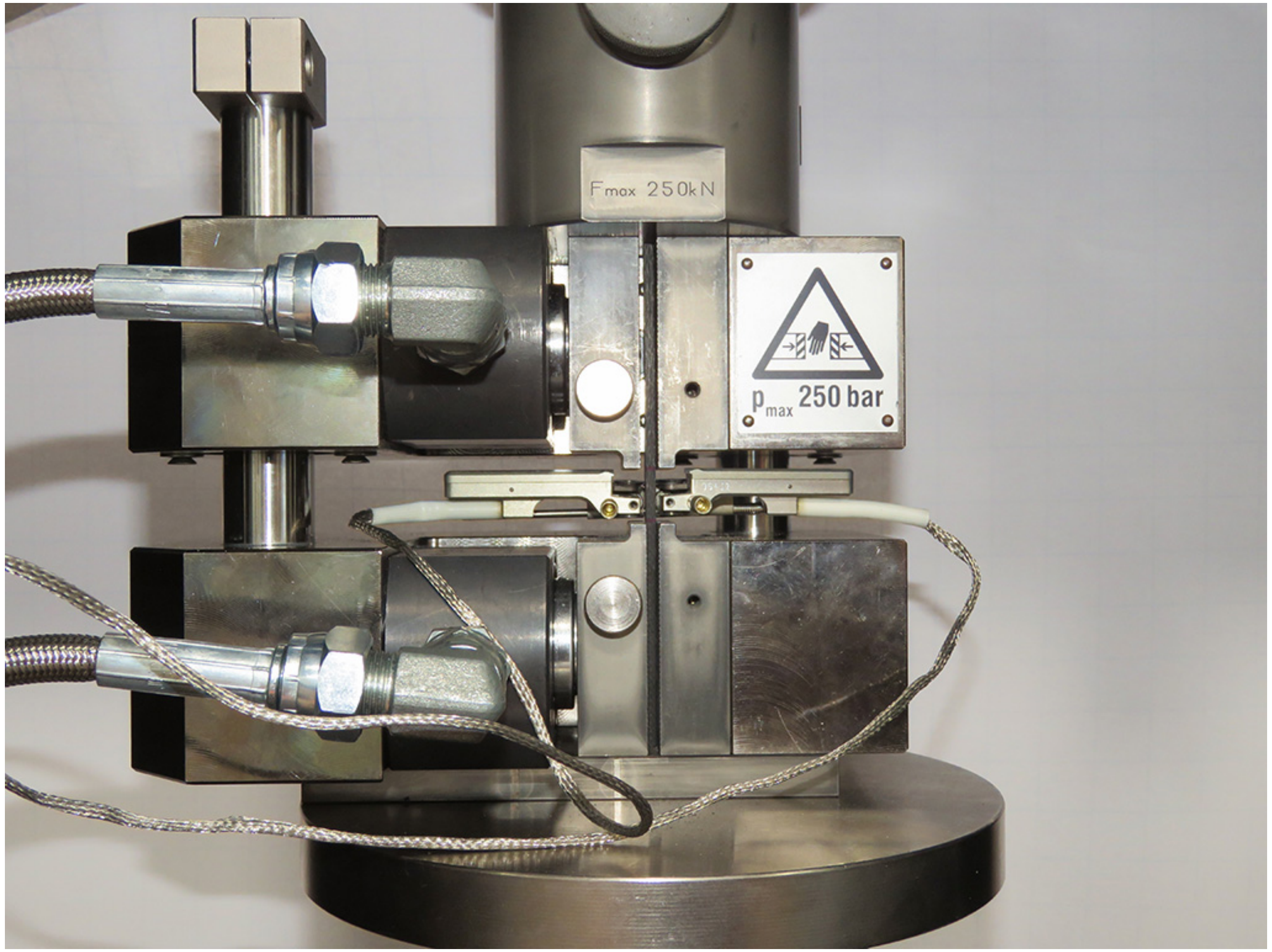
COMPRESSION TESTING – ASTM D6641

Test sequence: Closing the upper grip (a small gap remains)



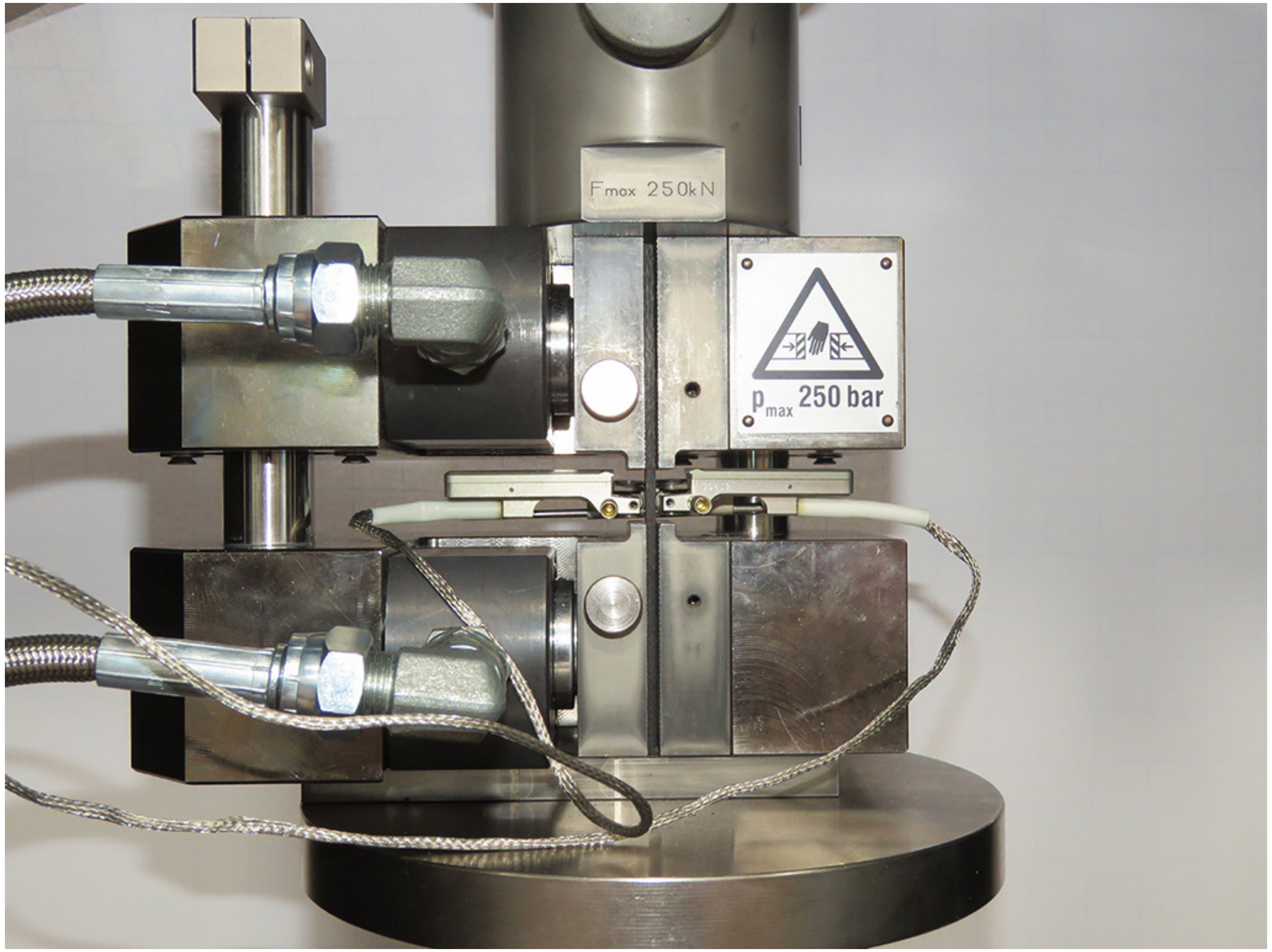
COMPRESSION TESTING – ASTM D6641

Test sequence: Lowering the upper grip



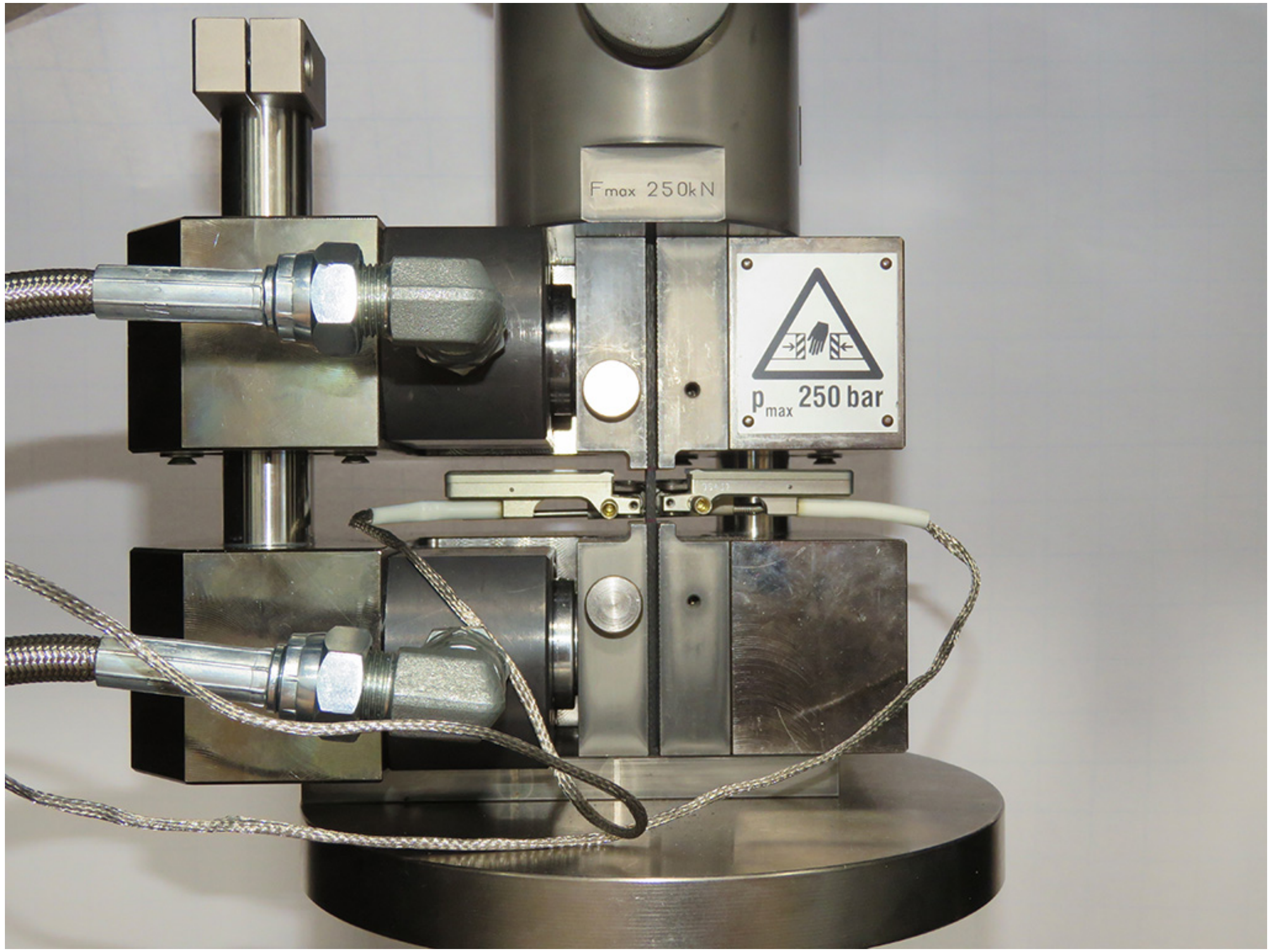
COMPRESSION TESTING – ASTM D6641

Test sequence: Closing the upper grip



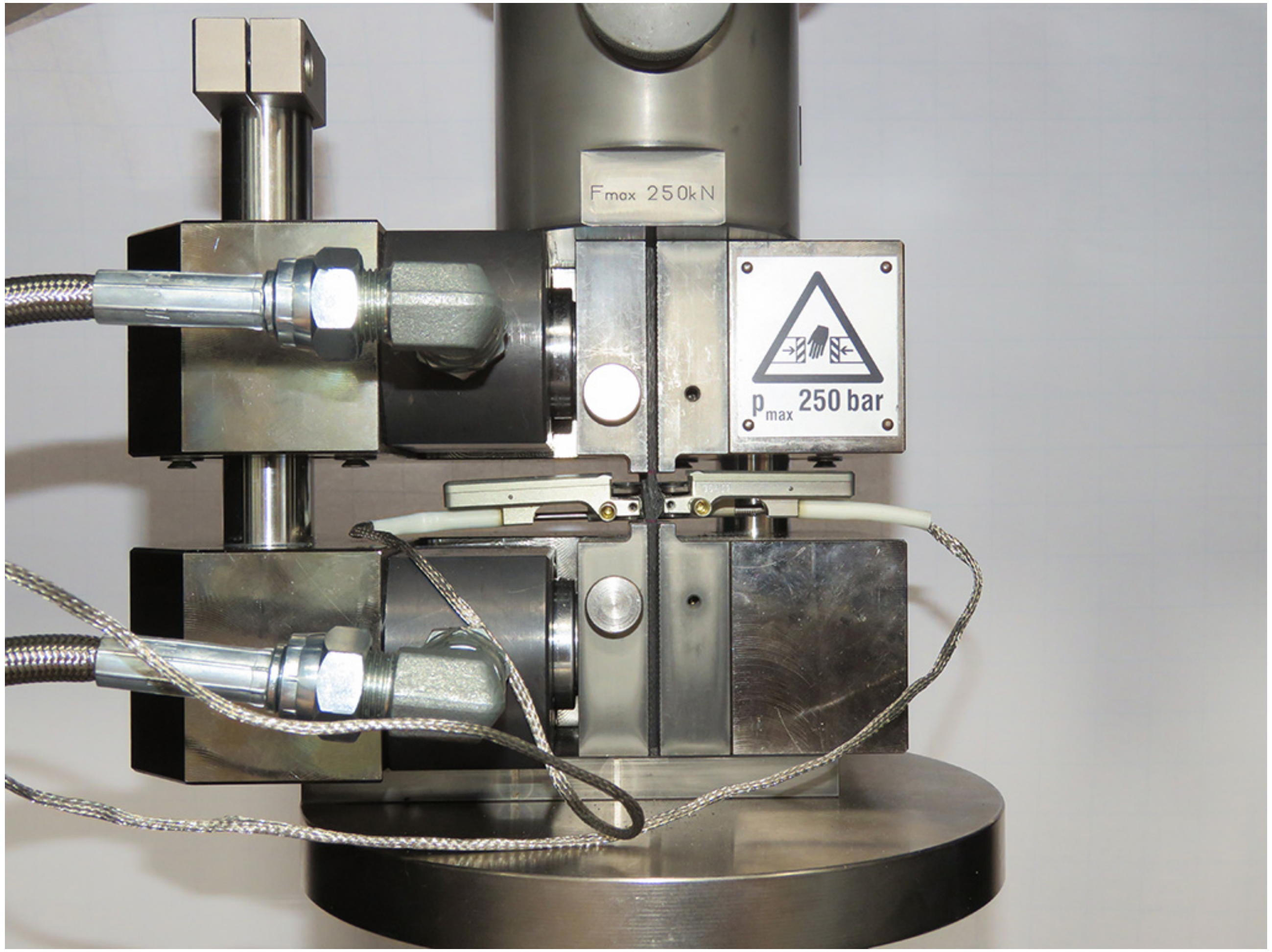
COMPRESSION TESTING – ASTM D6641

Test sequence: Apply full closing pressure to lower and upper grip

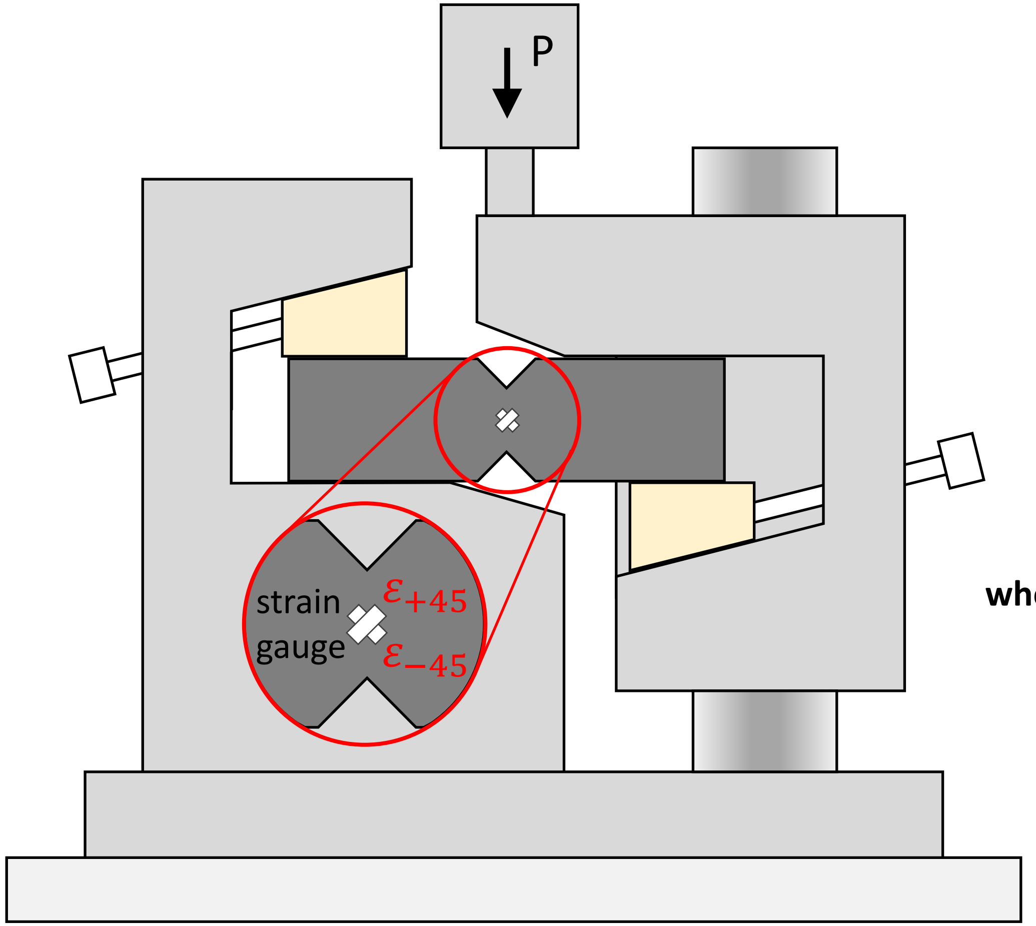


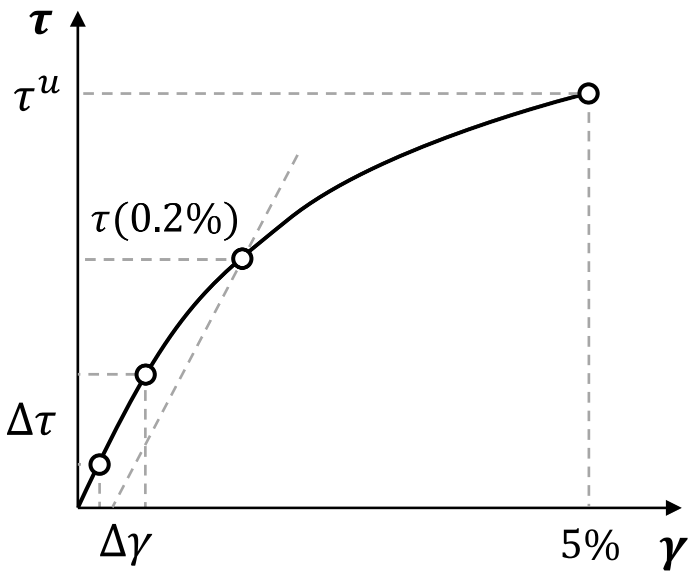
COMPRESSION TESTING – ASTM D6641

Test sequence: End of test with specimen broken in gauge section



IN-PLANE SHEAR TESTING – ASTM D5379





$$\gamma = |\varepsilon_{+45}| + |\varepsilon_{-45}|$$

$$\tau = \frac{P}{A}$$

$$G^{chord} = \frac{\Delta\tau}{\Delta\gamma}$$

$$\tau^u = \frac{P^u}{A}$$

where:

γ	shear strain
τ	shear stress
P	applied load
A	cross-sectional area at v-notch
ε_{+45}	strain of +45° strain gauge
ε_{-45}	strain of -45° strain gauge
G^{chord}	shear chord modulus of elasticity
$\Delta\gamma$	difference between shear strain points (2000 to 6000 $\mu\varepsilon$)
$\Delta\tau$	difference of applied shear stress between defined strain points
τ^u	shear strength (at 5% shear strain)
P^u	applied load at 5% shear strain

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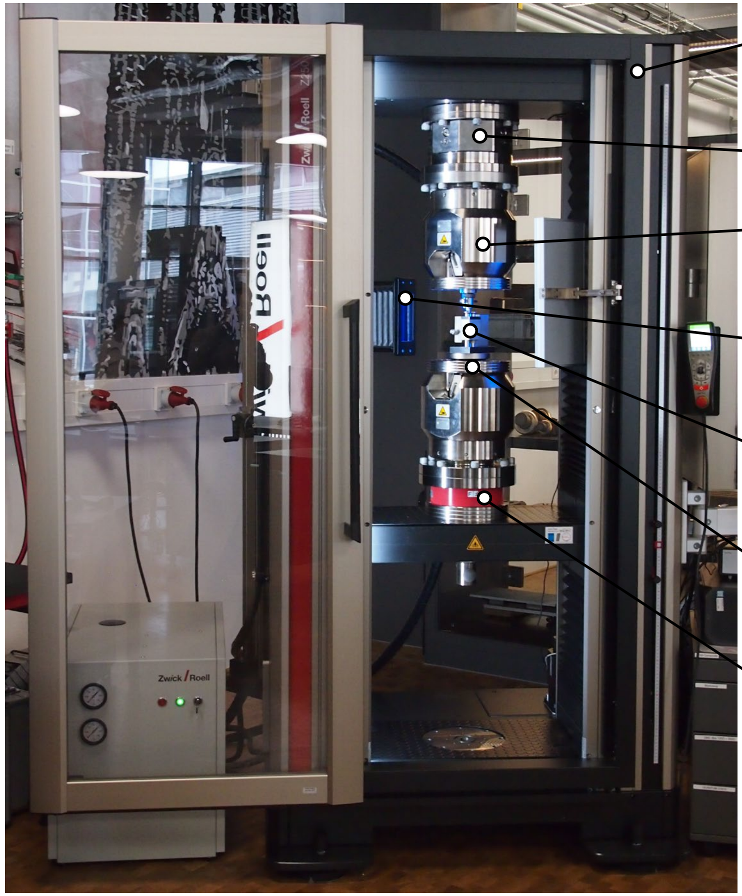
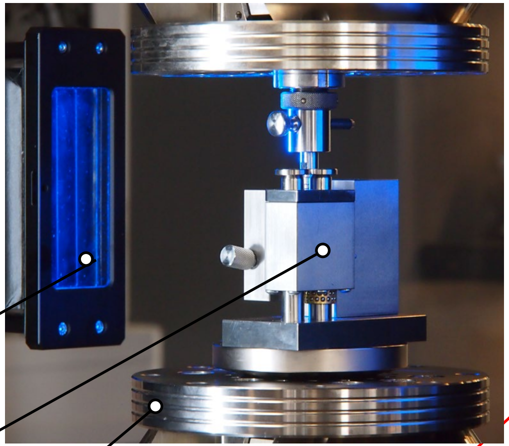
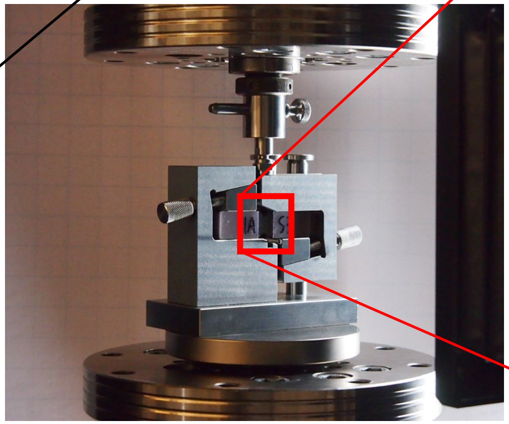
IN-PLANE SHEAR TESTING – ASTM D5379

Test setup

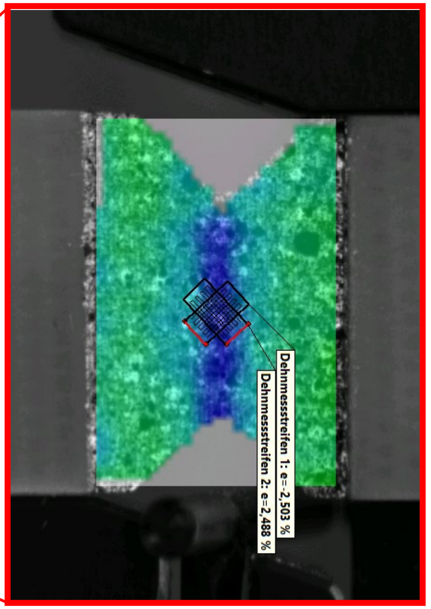


- 250 kN universal static test machine
- alignment unit
- hydraulic body-over-wedge grips
- **videoXtens biax 2-150 HP**
- **Iosipescu shear fixture with sample**
- **modular adapter system for grips**
- load cell



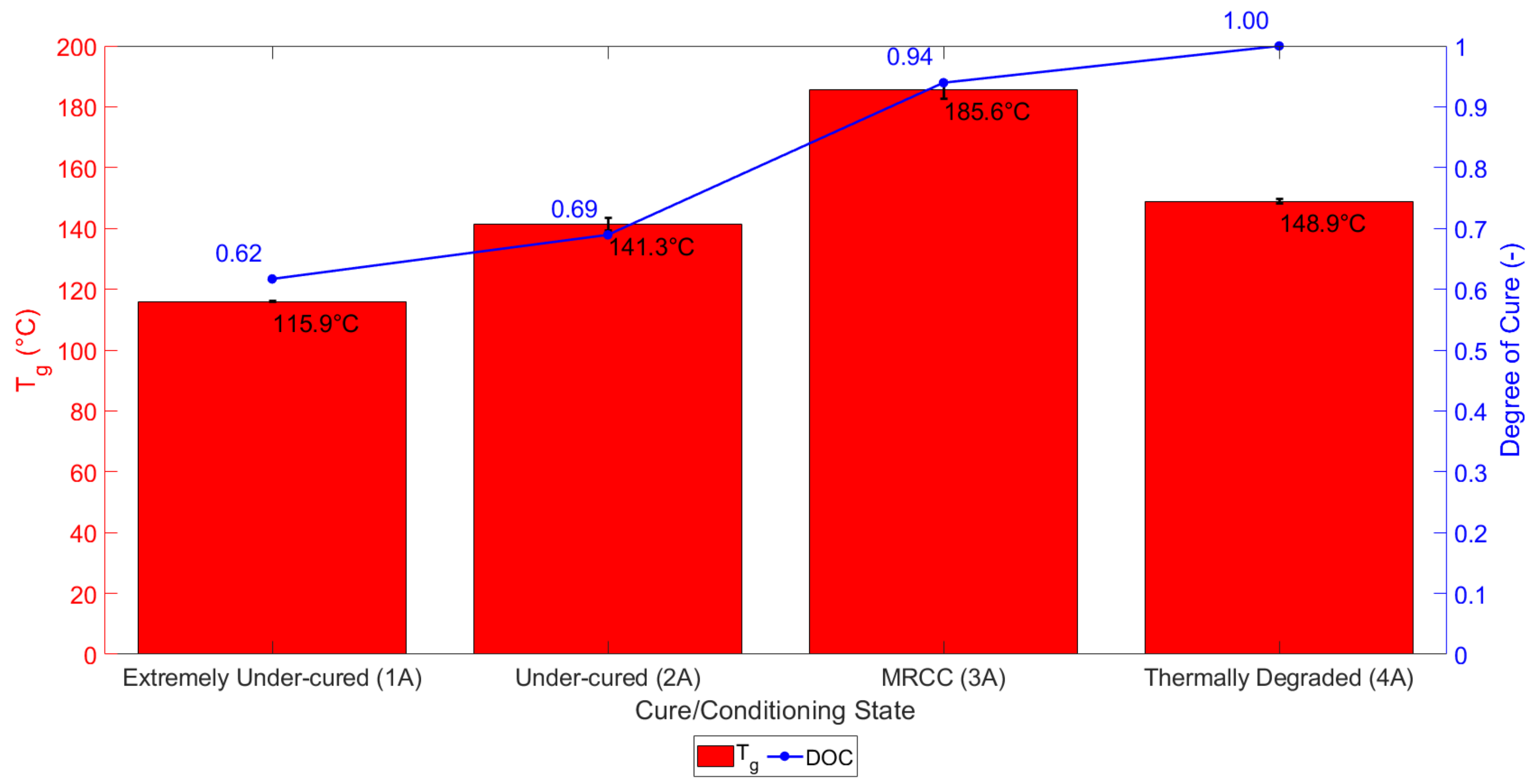


- Shear strain field obtained with DIC software module of the videoXtens
- ±45° virtual strain gauge for strain measurement



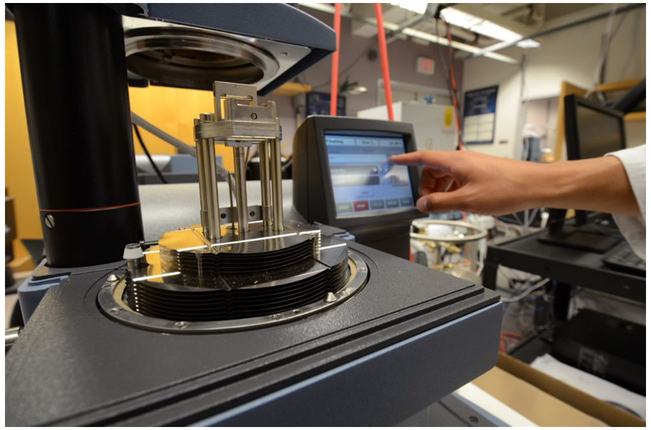
Analytical Characterization (T_g & DOC)

- Degree of Cure (DOC) from DSC
- T_g from DMA



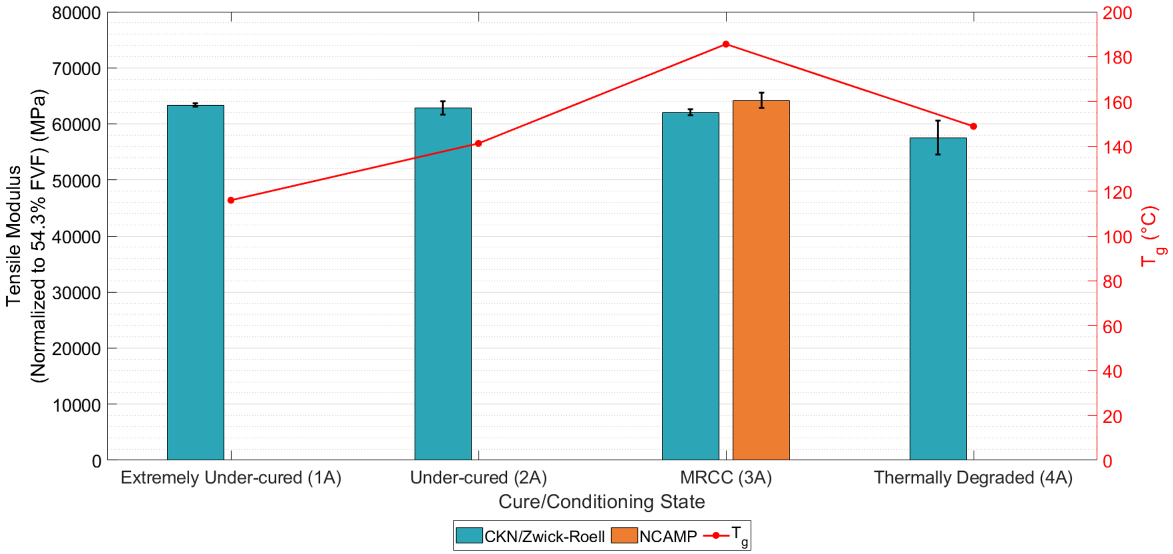
Differential Scanning Calorimetry (DSC)

Dynamic Mechanical Analysis (DMA)

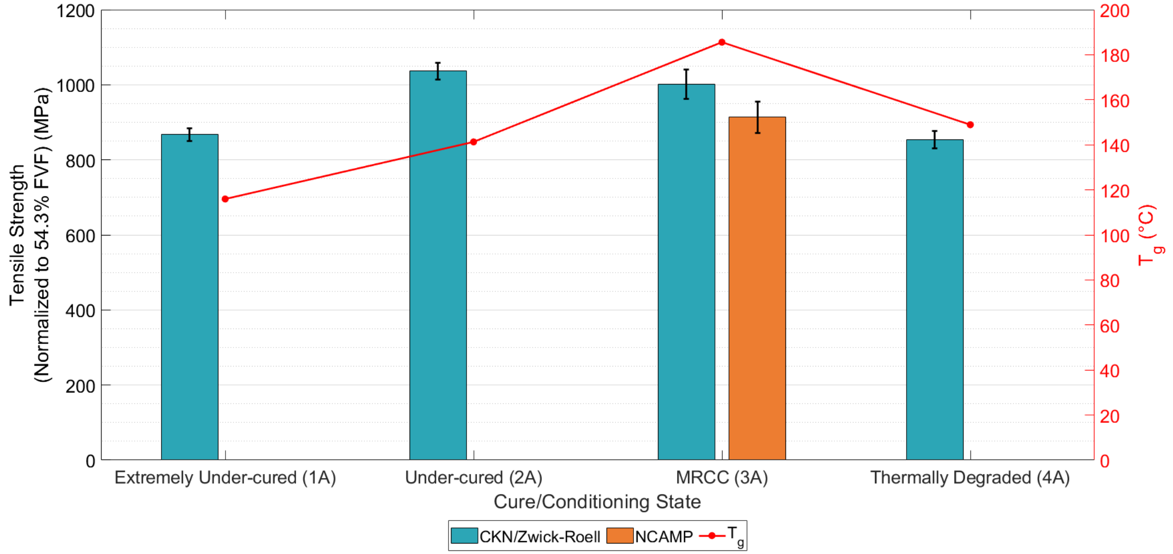


TENSILE TESTING – ASTM D3039

Tensile Modulus



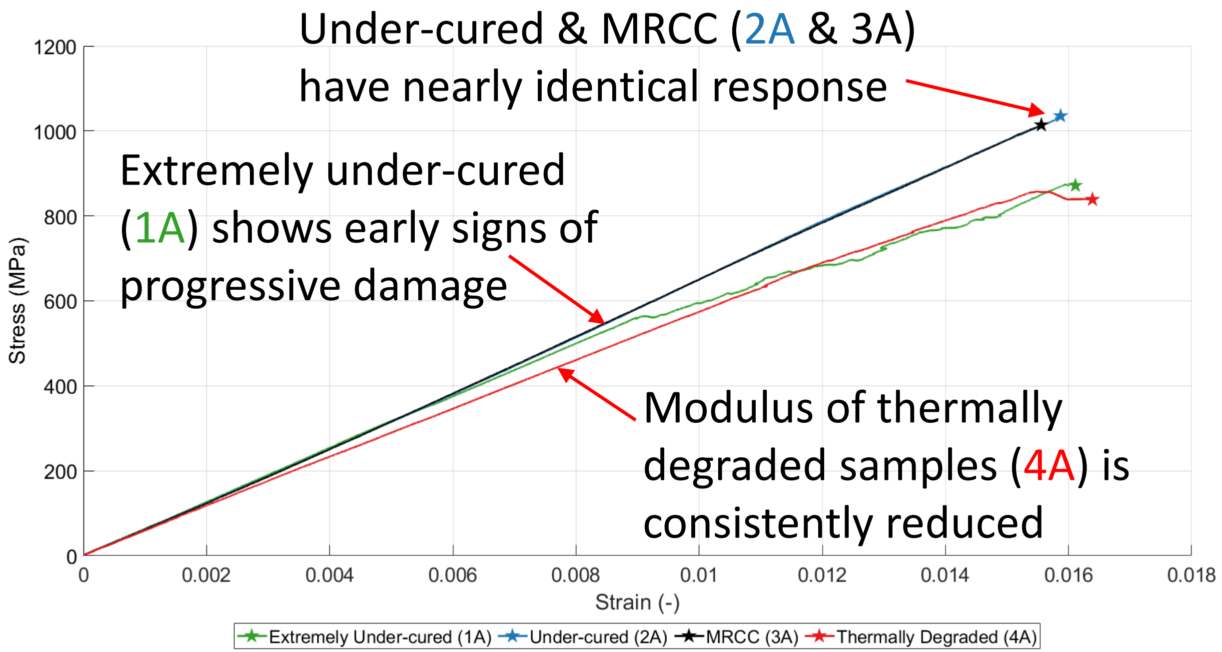
Tensile Strength



- Modulus is generally unaffected by curing conditions as it is dominated by the stiffness of the fibres
  - Some average reduction in modulus when thermally degraded, but scatter is also high
- Strength is substantially affected by extreme under-cure (1A) and thermal degradation of matrix (4A)
  - Tensile strength is not strictly a fibre dominated property; load sharing and ability to redirect load to adjacent fibres as filaments begin to break is an important role of the matrix
  - These results show that at room temperature dry conditions, we are seeing higher tensile strength with a moderately under-cured (2A) sample; but reduced T_g indicates the potential for an issue for use in hot and hot/wet conditions

TENSILE TESTING – ASTM D3039

REPRESENTATIVE CURVES



- Under-cured (2A) and MRCC (3A) show substantially similar response, with the under-cured strength and failure strain slightly higher
- Thermally degraded (4A) modulus is consistently less due to some effect of matrix degradation
- Extremely under-cured (1A) stiffness begins nominally, but low matrix strength results in early onset of progressive damage

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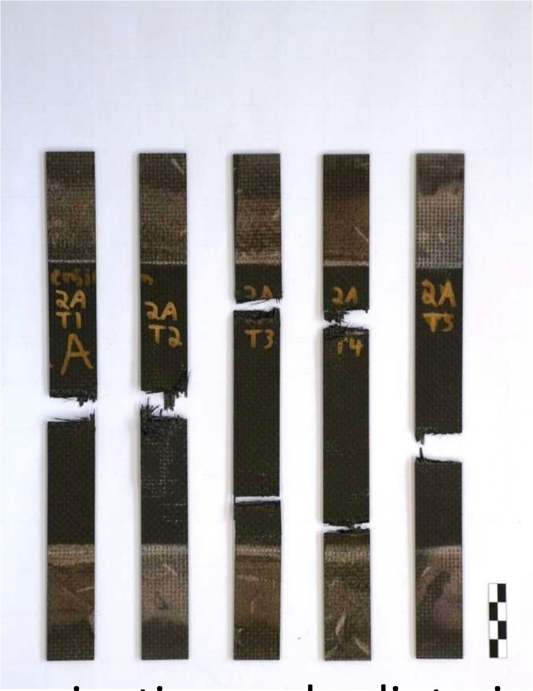
TENSILE TESTING – ASTM D3039

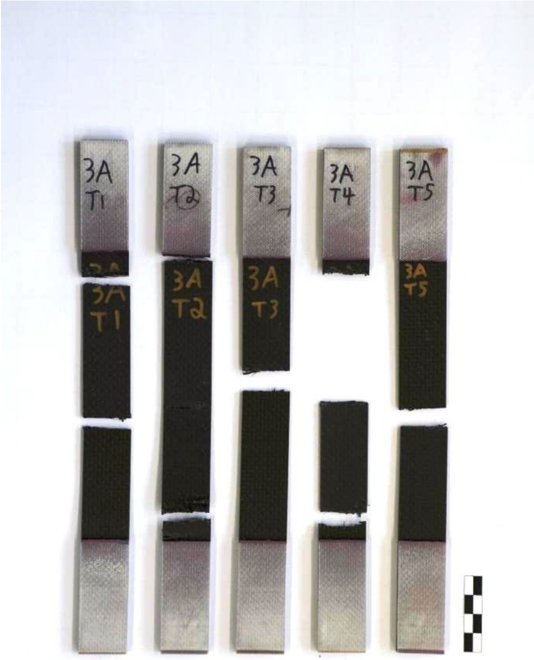
Failure modes

Extremely Under-Cured (1A)

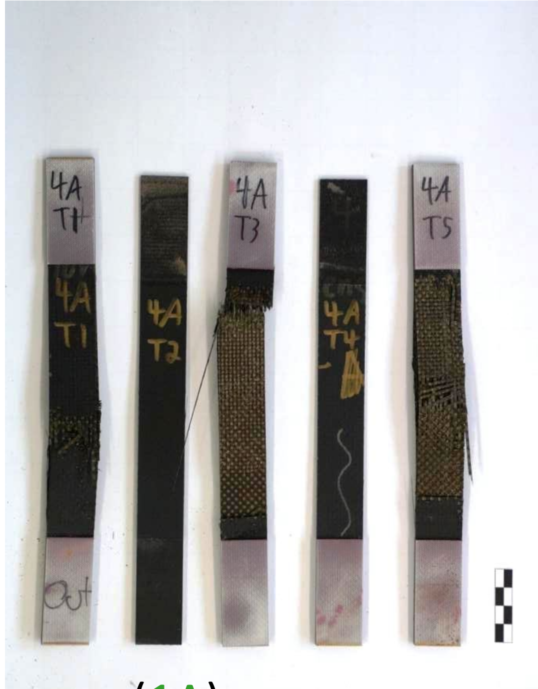
Under-cured (2A)



MRCC (3A)

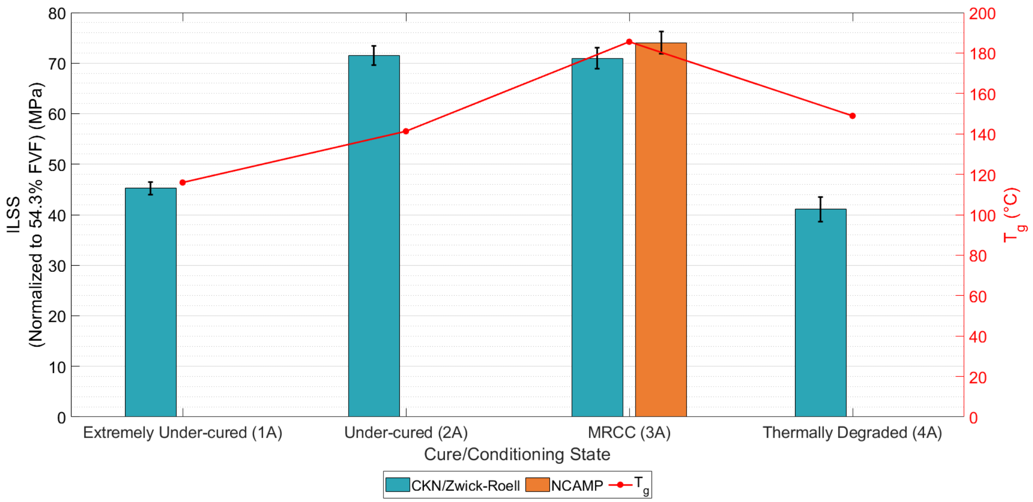


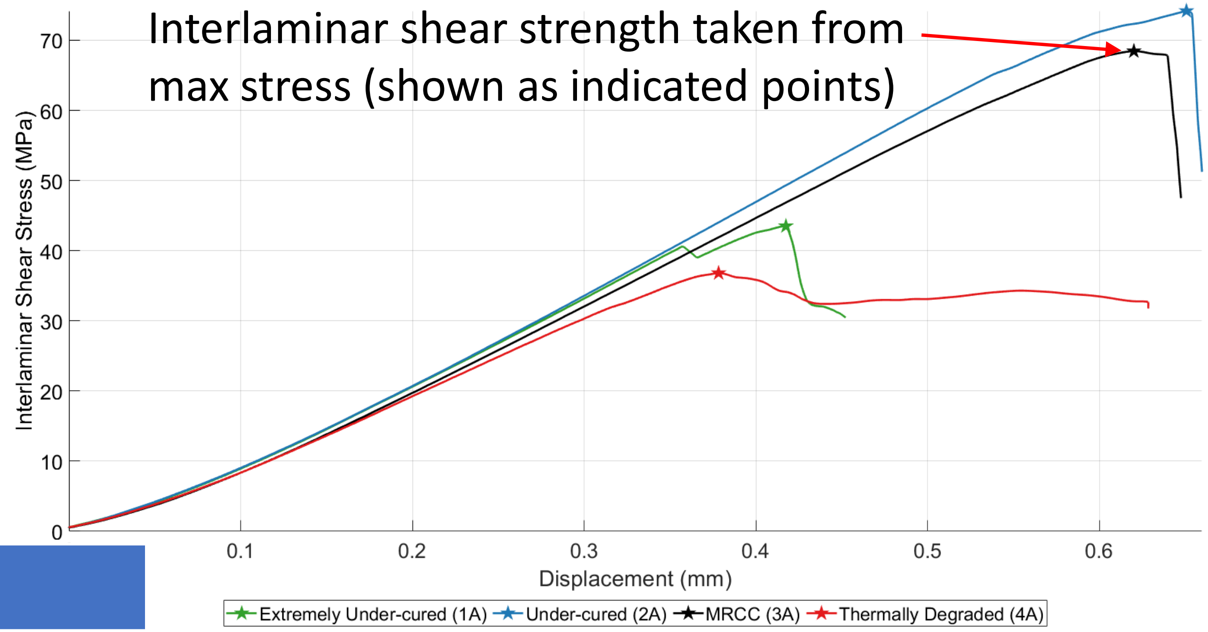
Thermally degraded (4A)

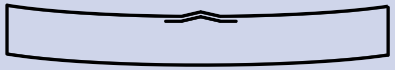
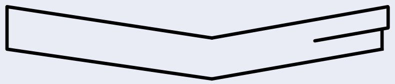


- Extensive delamination and splintering is seen in the extreme under-cure (1A) case as a result of low matrix strength
- Reduced matrix integrity in the thermally degraded (4A) case results in similar delamination and splintering
- Under-cured and MRCC (2A & 3A) samples show a more typical sudden lateral failure

SHORT BEAM SHEAR TESTING (ASTM D2344)





Cure Condition	Failure Mode	
1A & 4A	Flexure Compression	
2A & 3A	Interlaminar Shear	

- Result is substantially affected by extreme under-cure (1A) and thermal degradation (4A)
  - Matrix dominated property
  - This will have important consequences for compressive strength
  - Sensitivity of this test and its simple coupons and procedure make it a good test for quality assurance
- Difference in failure modes clearly seen in mechanical response

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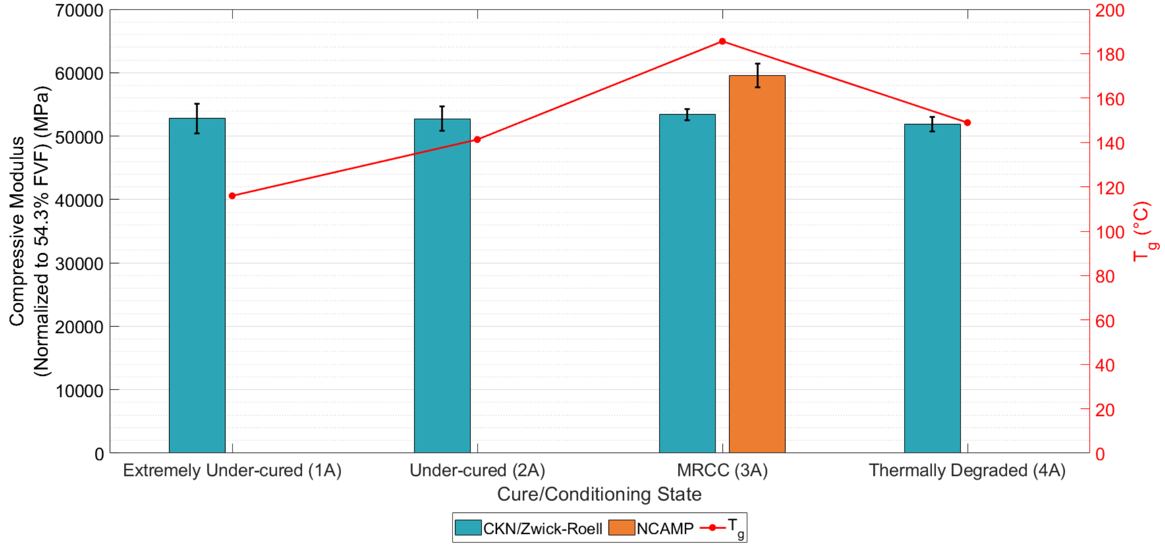
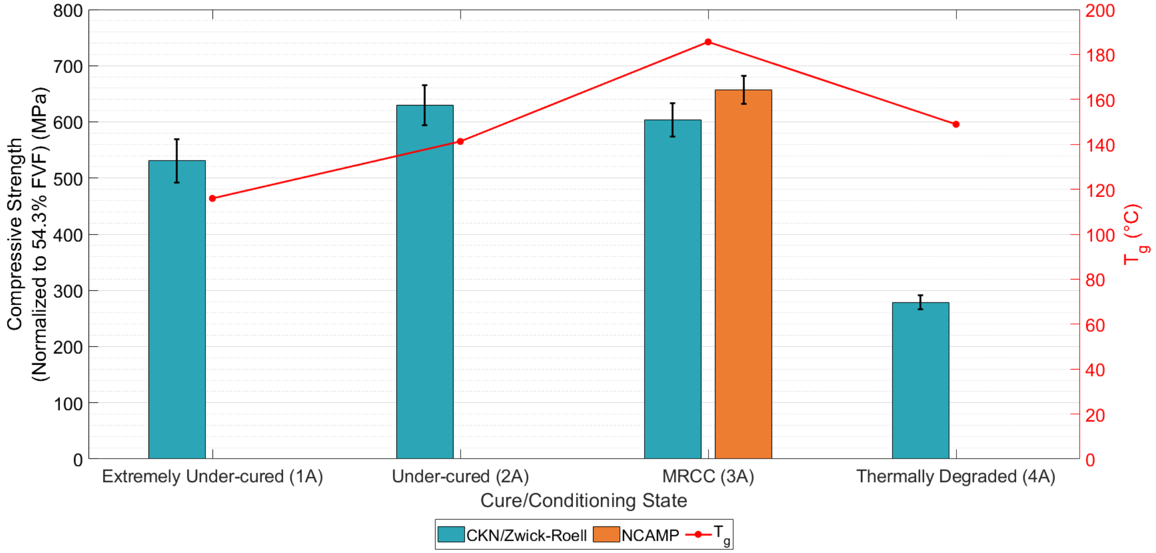
COMPRESSION TESTING – ASTM D6641

Compressive Modulus



Compressive Strength



- As with tensile tests, compressive modulus is largely unaffected by curing condition
- Strength is substantially affected
  - Reduced matrix properties (as seen in short beam shear tests) are also seen here in the lower compressive strength of extremely under-cured (1A) and thermally degraded (4A)
- Consequence
  - ➢ Risk of failure in service without a noticeable change in behaviour before failure

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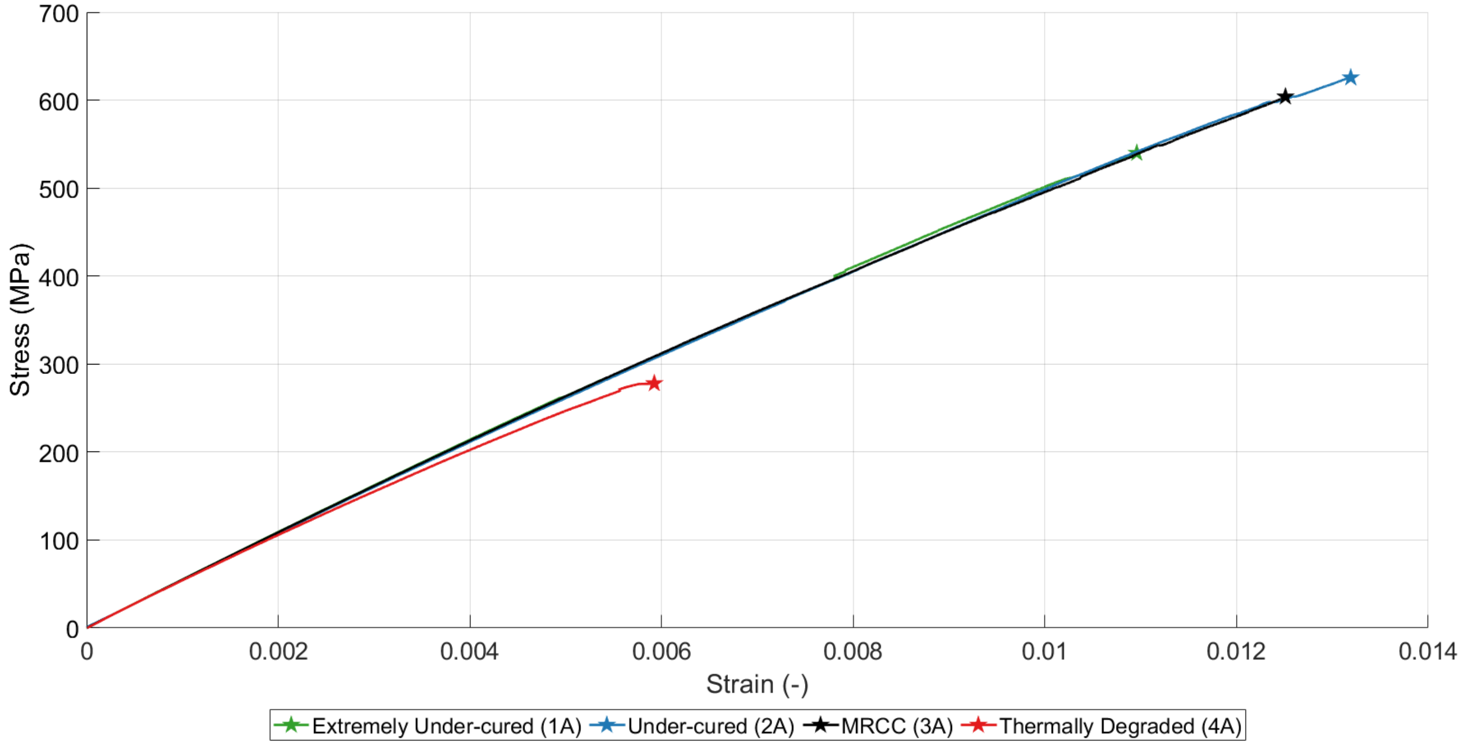
COMPRESSION TESTING – ASTM D6641

REPRESENTATIVE CURVES



- Except for modulus reduction at moderate strains in thermally degraded (4A) conditions, the compressive response is very similar in all coupons with the only difference being a shift in the failure load/strain
  - ➢ Risk of failure in service without a noticeable change in behaviour before failure

COMPRESSION TESTING – ASTM D6641
Failure modes

Extremely Under-Cured (1A)

Under-cured (2A)

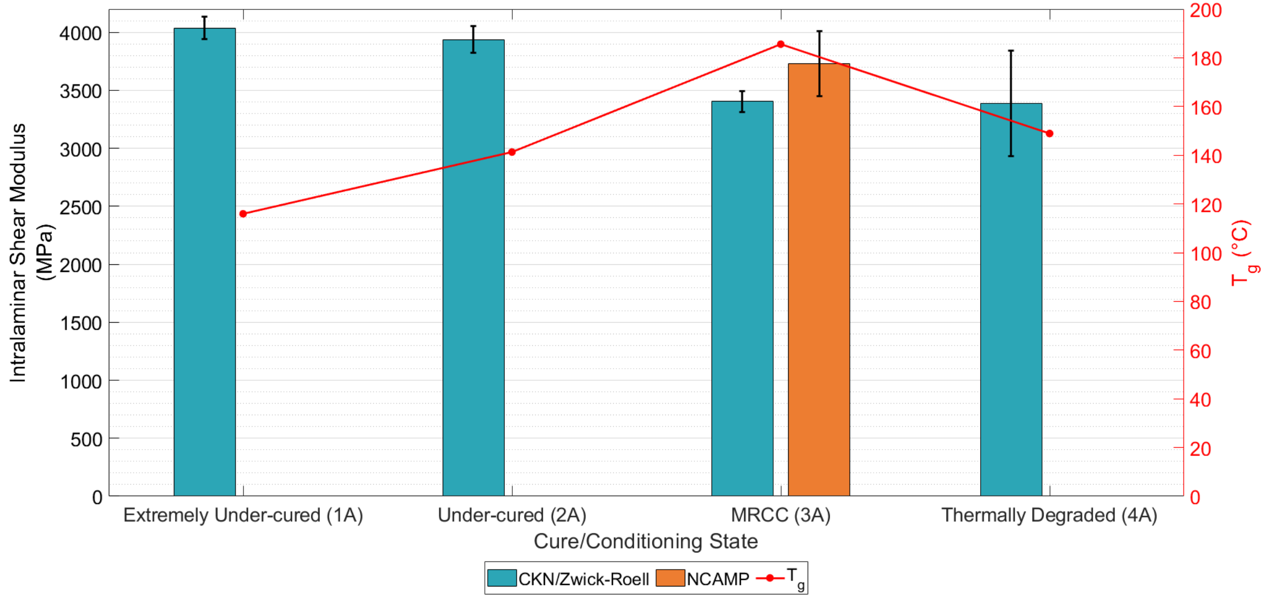
MRCC (3A)

Thermally degraded (4A)

- Substantially more "brooming" and distributed matrix damage in extreme under-cure (1A) case
- Failures in under-cured (2A) and MRCC cure (3A) are localized around kink-band
- Low matrix strength in thermally degraded (4A) coupons results in early failure along a shear plane

IN-PLANE SHEAR TESTING – ASTM D5379

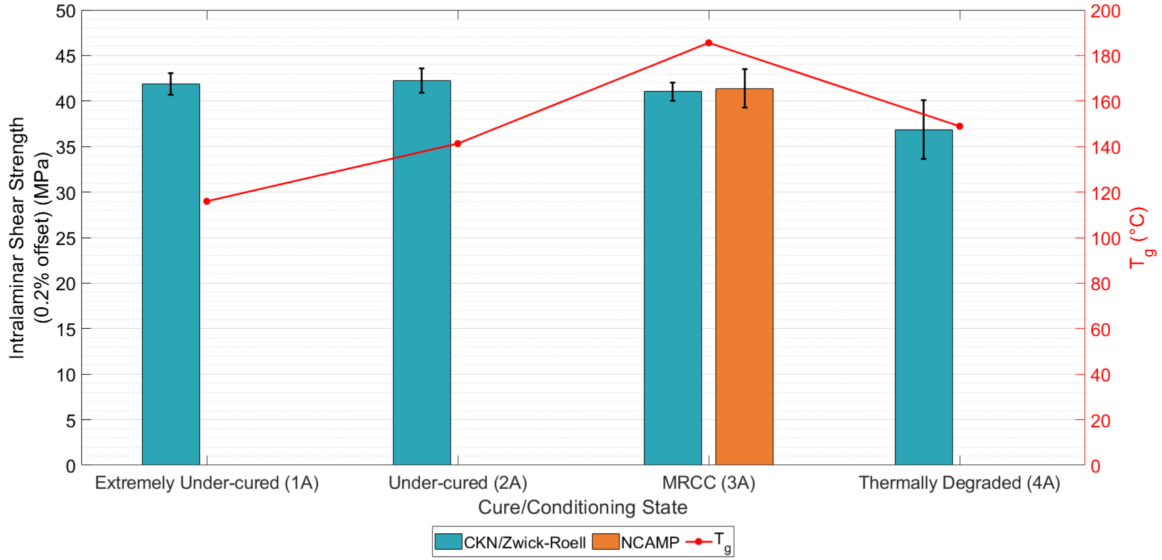
Shear Modulus



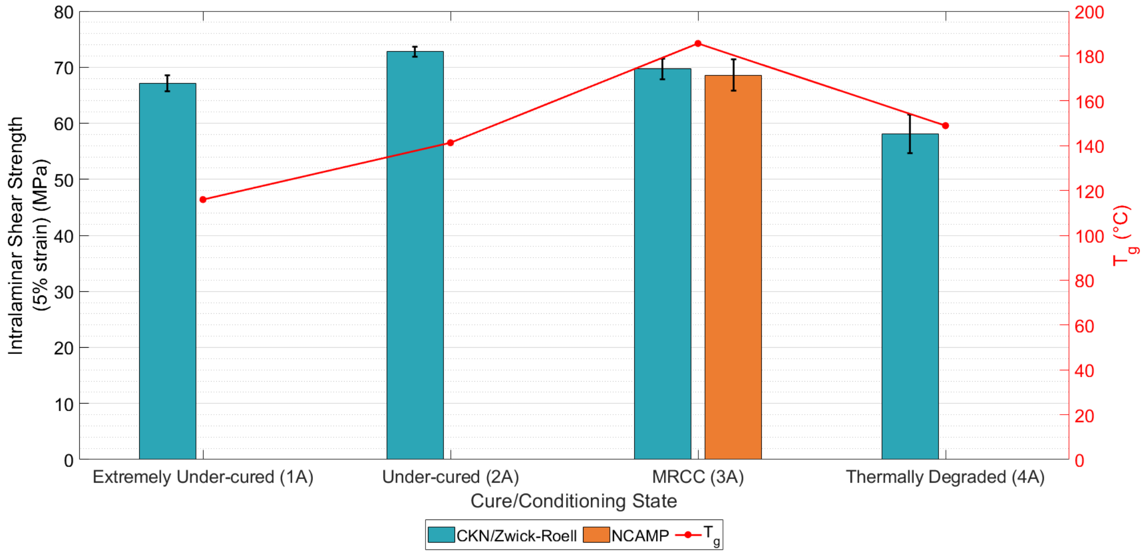
- Variation in shear modulus values is likely due to a tolerance variation of the loading edges
- Accuracy of measurements could be improved with the v-notched rail shear test (ASTM D7078) where specimens are larger and are gripped on their large flat surfaces rather than on the edges as for the Iosipescu test

IN-PLANE SHEAR TESTING – ASTM D5379

0.2% Offset Strength



5% Strain Strength



- Even though extremely under-cured, the gelled & vitrified matrix (T_g ≈ 120°C) in the extremely under-cured condition (1A) has a normal intralaminar shear response at low strain (0.2% offset strength), but a lower strength than under-cured (2A) & MRCC (3A) at 5% shear strain
- Thermally degraded (4A) material has lower strength based on both measurement methods due to pre-existing matrix damage from the thermal degradation

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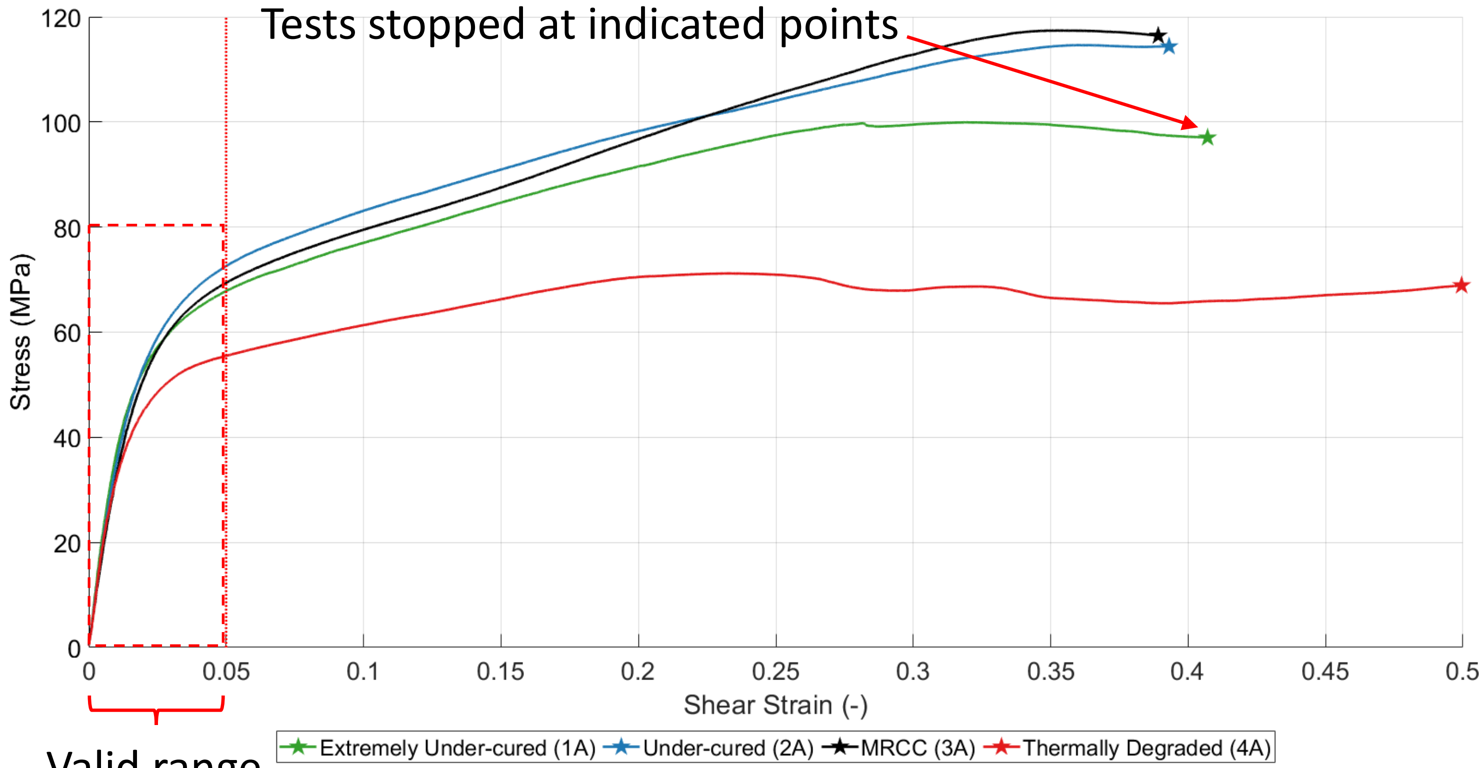
IN-PLANE SHEAR TESTING – ASTM D5379

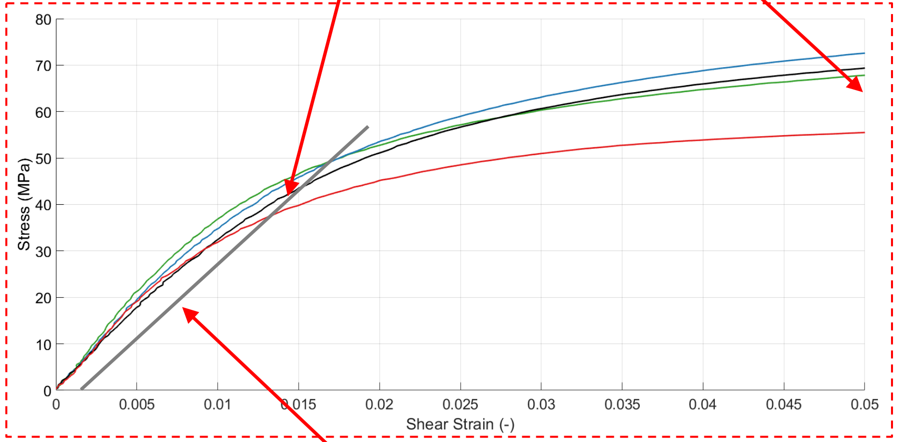
REPRESENTATIVE CURVES



Valid range of strain for test

0.2% Offset Strength (intersection of curves with 0.2% offset line)

5% Strain Strength



0.2% Offset Line

RECAP OF RESULTS

- Under nominal conditions (room temperature, dry), the following was observed:
  - Moduli remain relatively unchanged
  - Strength of extremely under-cured and thermally degraded material was degraded (particularly for ILSS)
  - Strength of under-cured & "fully" cured material are similar
    - We are seeing a trend of slightly higher strength with an under-cured (2A) sample (compared to MRCC/3A); but reduced T_g indicates the potential for an issue for use in hot and hot/wet conditions
  - T_g is lower in both the under-cured and thermally degraded cases
    - Reduces the safe maximum operating temperature of the part

KEY TAKEAWAYS

- Excessive variation in thermal history can result in some locations with an under-cured condition (low DOC) or thermally degraded condition
- Large exotherms can be exceptionally damaging to mechanical properties
- T_g measurement is a useful metric for cure, however it is not possible to tell if the composite is under-cured or thermally degraded with this method
  - Colour of the material can provide an indication
- ILSS measurement is useful for checking if a composite is cured properly and it is more sensitive to thermal degradation
  - Due to its small sample size and ease of test, this method is commonly used for quality assurance

WHAT ABOUT OTHER EFFECTS OF UNDER-CURE/DEGRADATION?

- These specimens were all tested under nominal conditions (room temperature, dry)
  - Is that realistic of composites in-service?
- Key questions that still need to be considered:
  - How does under-cure/thermal degradation affect wet conditions?
  - How does under-cure/thermal degradation affect hot/wet conditions?

We'll discuss this in Part 2 of 2:

EFFECT OF CURE ON MECHANICAL PROPERTIES OF A COMPOSITE

on February 23rd

CKN gratefully acknowledges the support of Zwick Roell and the Technical University of Munich (TUM) for their collaboration with CKN to provide this AIM event

Thank you for joining us!

Keep an eye out for announcements on the next AIM event

Questions?

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