DTU

INFLUENCE OF FIBRE-BRAGG-GRATING STIFFNESS ON IN-SITU MEASUREMENTS OF CURE-INDUCED SHRINKAGE IN THERMOSETS

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Overall motivation – Fundamentals of the Study

- Cure-induced shrinkage leads to residual stresses in composite manufacturing.
- This may lead to manufacturing defects such as shorter fatigue life with more aggressive cure profiles [1].
- At DTU Wind, this approach is applied to determine the characteristics while curing with Fibre-Bragg-Gratings.

[1] - https://orbit.dtu.dk/en/publications/influence-of-curing-cycle-on-the-build-up-of-residual-stresses-an

In this Study:

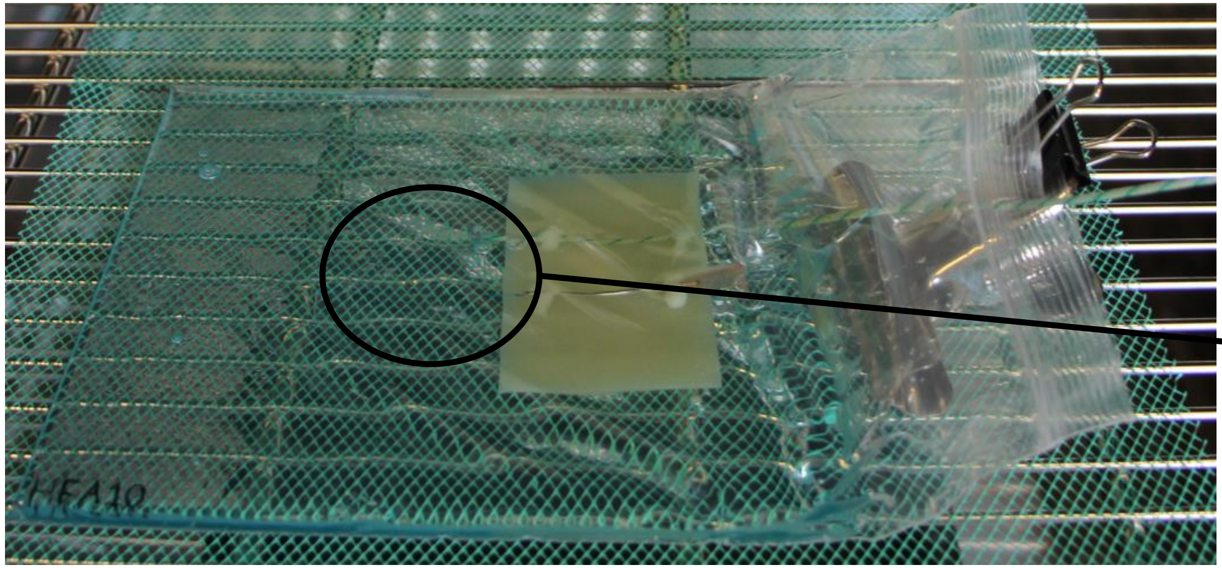
Measuring cure shrinkage through Fibre-Bragg-Gratings in an **assumed stress-free state**

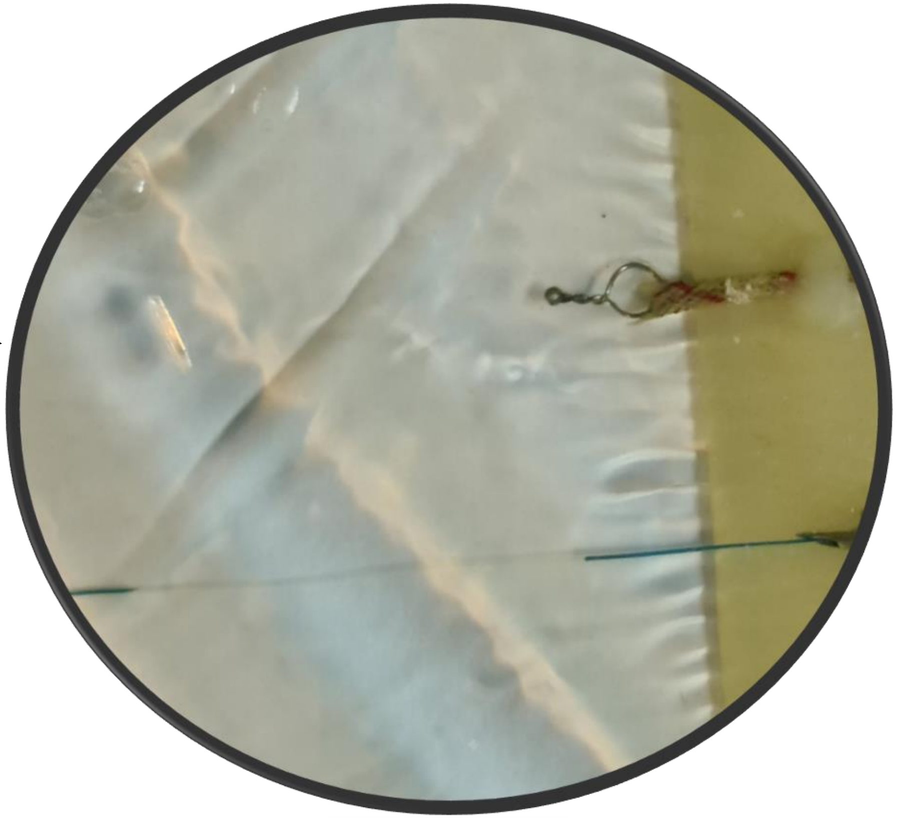
1. Glass-based FBG (G-FBG) – High stiffness: $E_{\text{G-FBG}} = 70\,\mathrm{GPa}$

2. Polymer-based FBG (P-FBG) – Low stiffness: $E_{\text{P-FBG}} = 2.5\,\mathrm{GPa}$

We will come back to **why** it is relevant to investigate a P-FBG

Experimental concept for measuring cure-induced strain



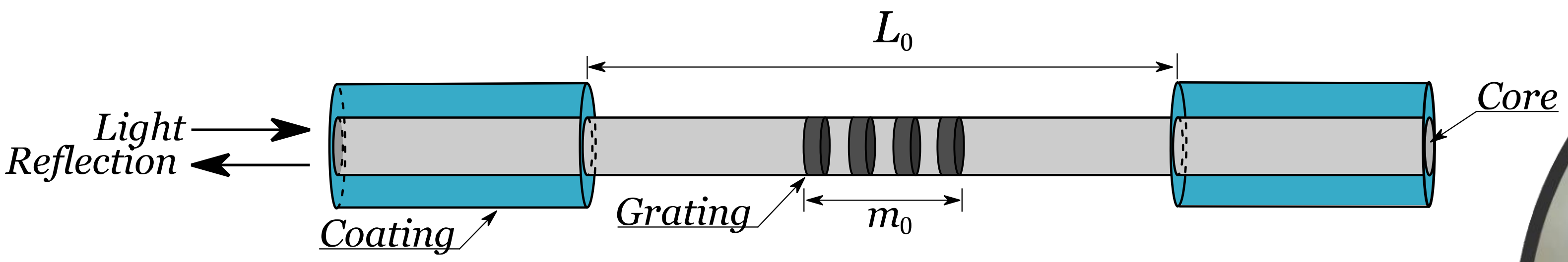


Liquid thermoset in a bag = Unconstrained thermoset
= Free movement while curing.

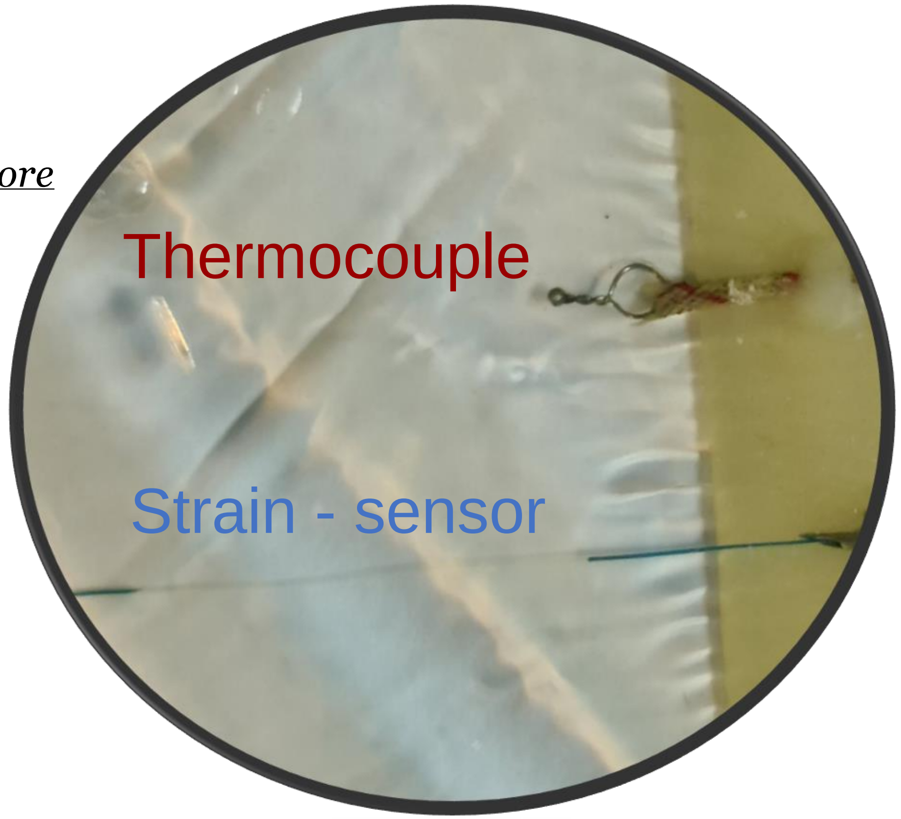
The thermoset <u>SHOULD</u> be in a stress-free state!

[2] - Mikkelsen et al. (2023) http://doi.org/10.5281/zenodo.7868314 [Pre-print]

Experimental concept for measuring cure-induced strain



Silica glass Fibre-Bragg-Grating Sensor (G-FBG)

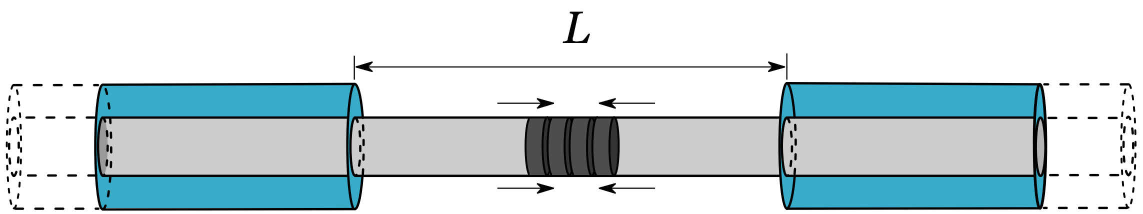


Comment

The temperature behaviour of the resin is given by a thermocouple monitoring the behaviour inside the resin.

A strain sensor in the form of an optical silica glass FBG measures the load transferred strain during curing.

Experimental concept for measuring cure-induced strain



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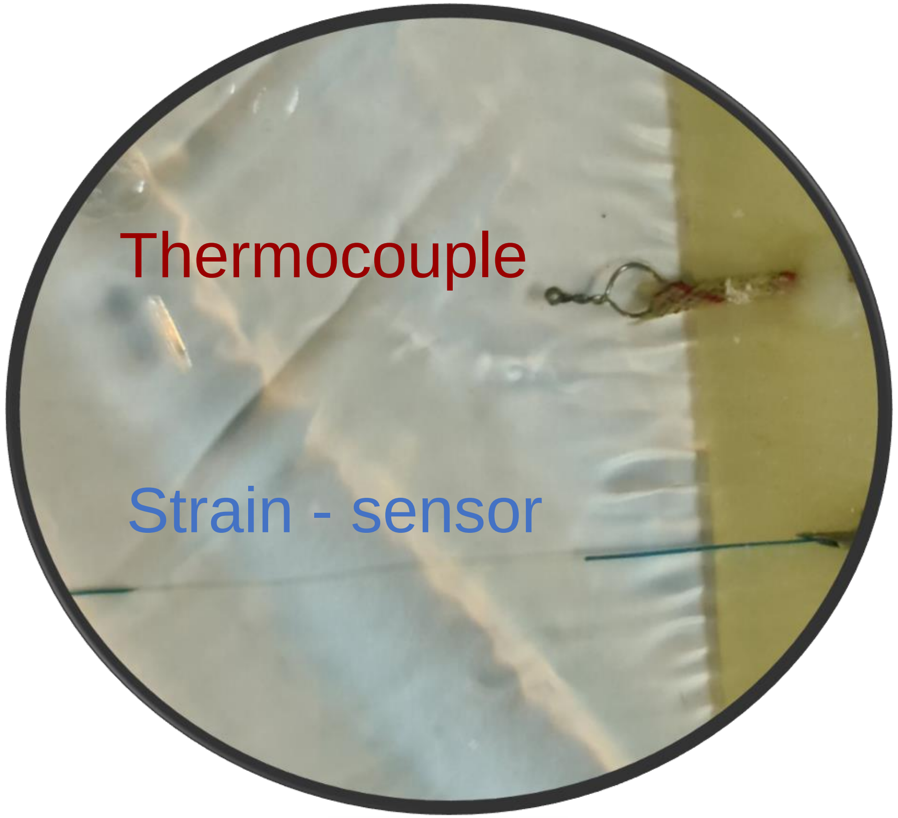
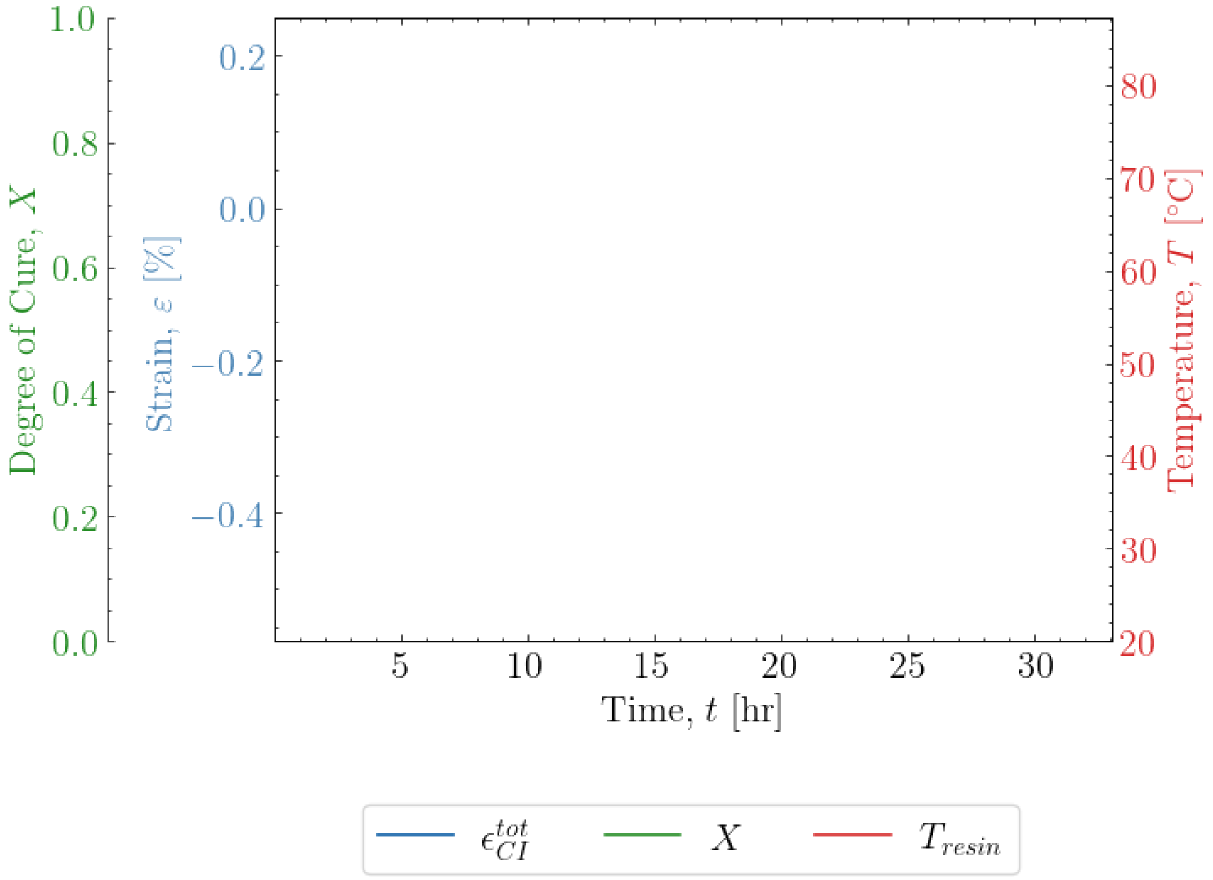


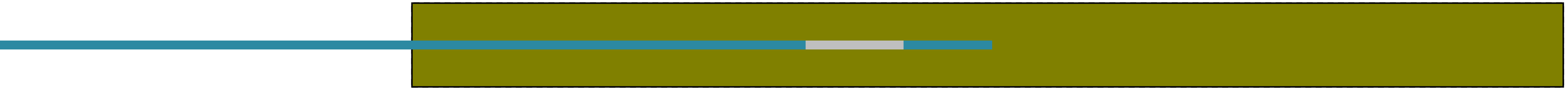
The Experimental Concept – Starting Point

Comment

The experiment can be illustrated as a graph plotting the developing cure, temperature and strain as a function of time.

Below is a principle sketch of the experiment.

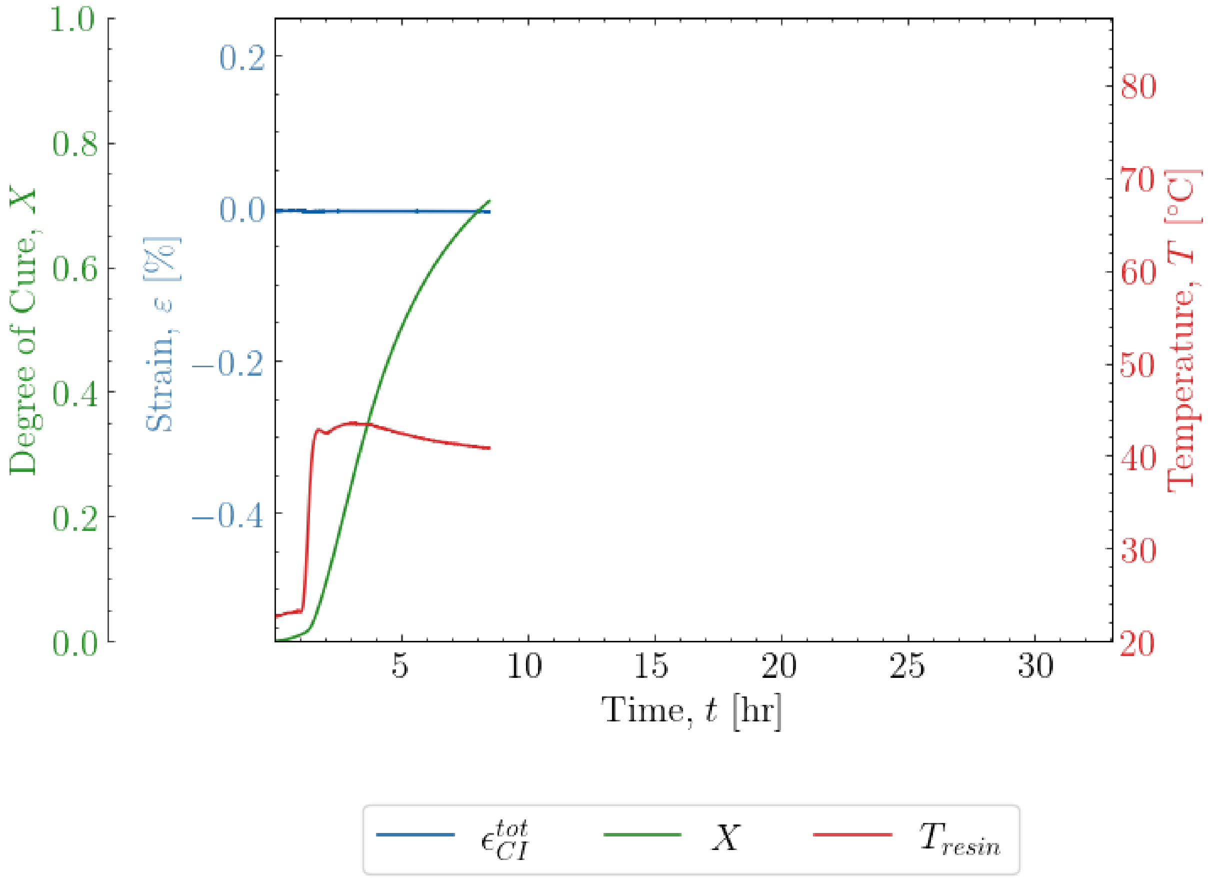


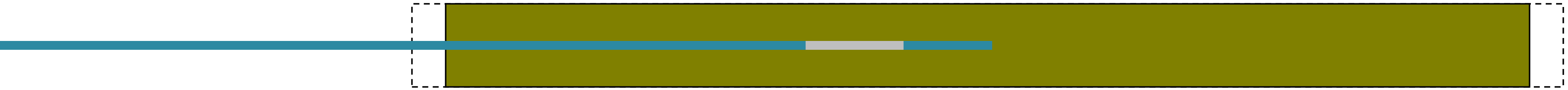


The Experimental Concept – Liquid Shrinkage

Comment

As the temperature rises in a pre-cure state, the degree of cure rises and the thermoset shrinks in the liquid phase. Hence, no strains are developing at this point as the material cannot transfer load.



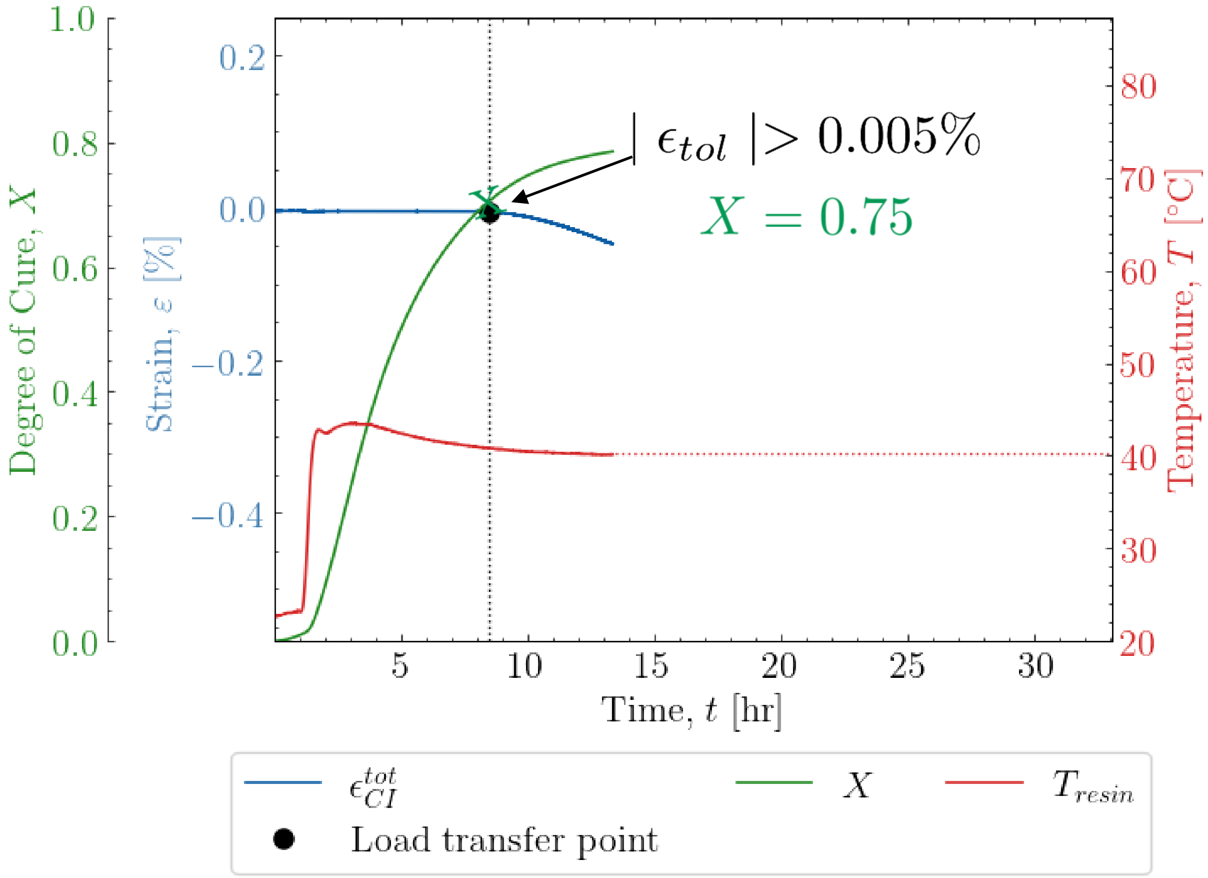


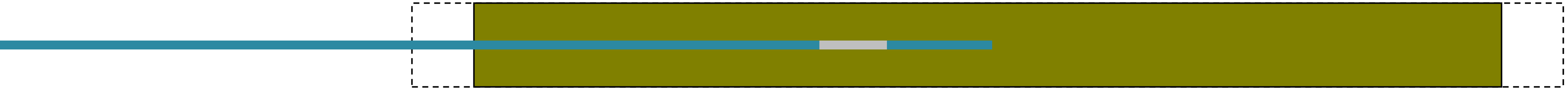
The Experimental Concept – Shrinkage Solid Phase

Comment

At a point of time, the thermoset is able to transfer load to the FBG seen as the strains become different from 0. This point is defined as the load-transfer point.

The strain tolerance gives the time and the degree of cure at this time.

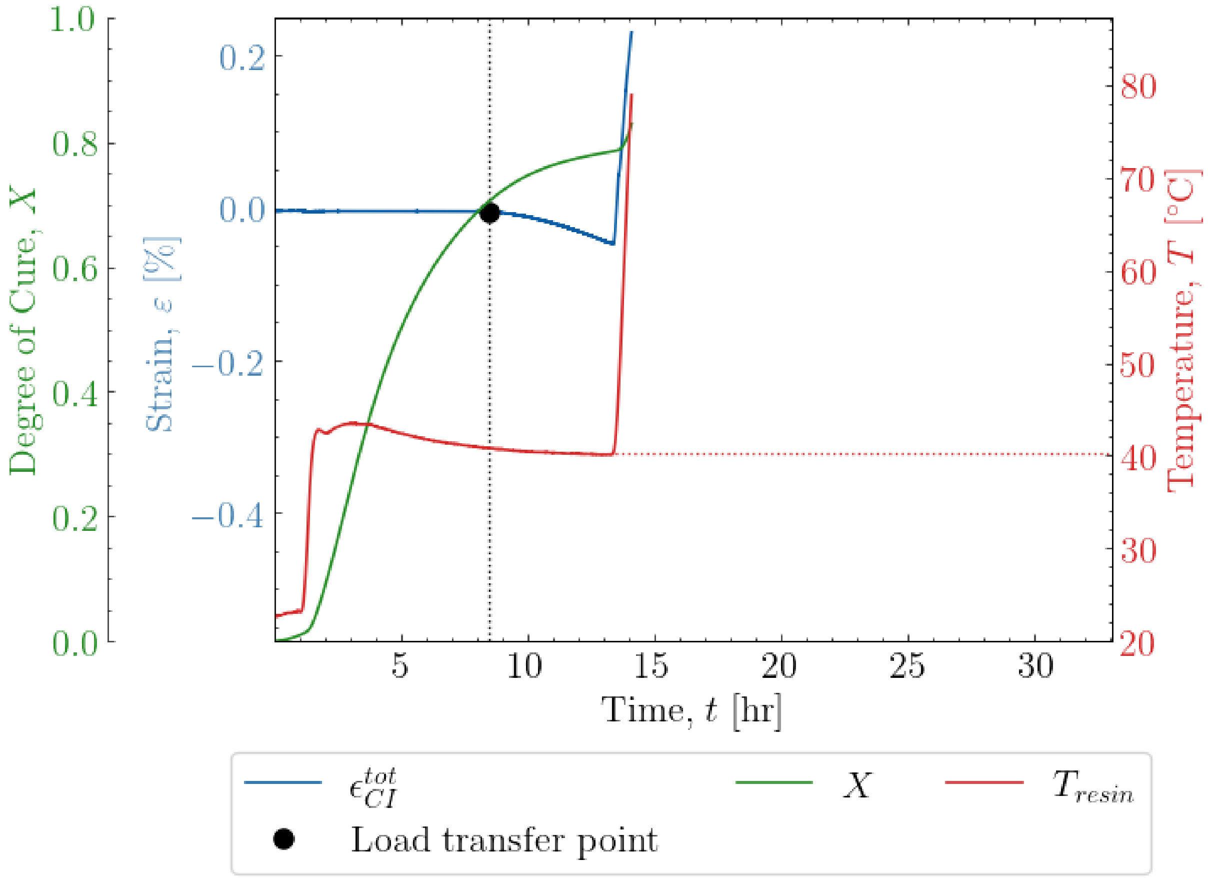


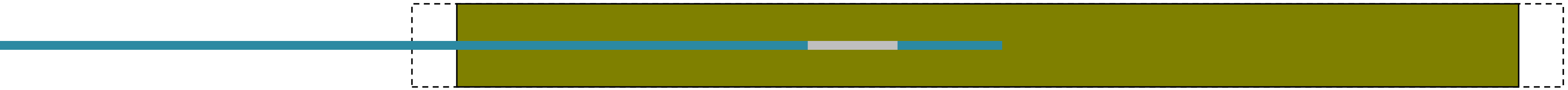


The Experimental Concept – Shrinkage Solid Phase

Comment

As the temperature rises towards the post-cure the FBG captures the expansion too.

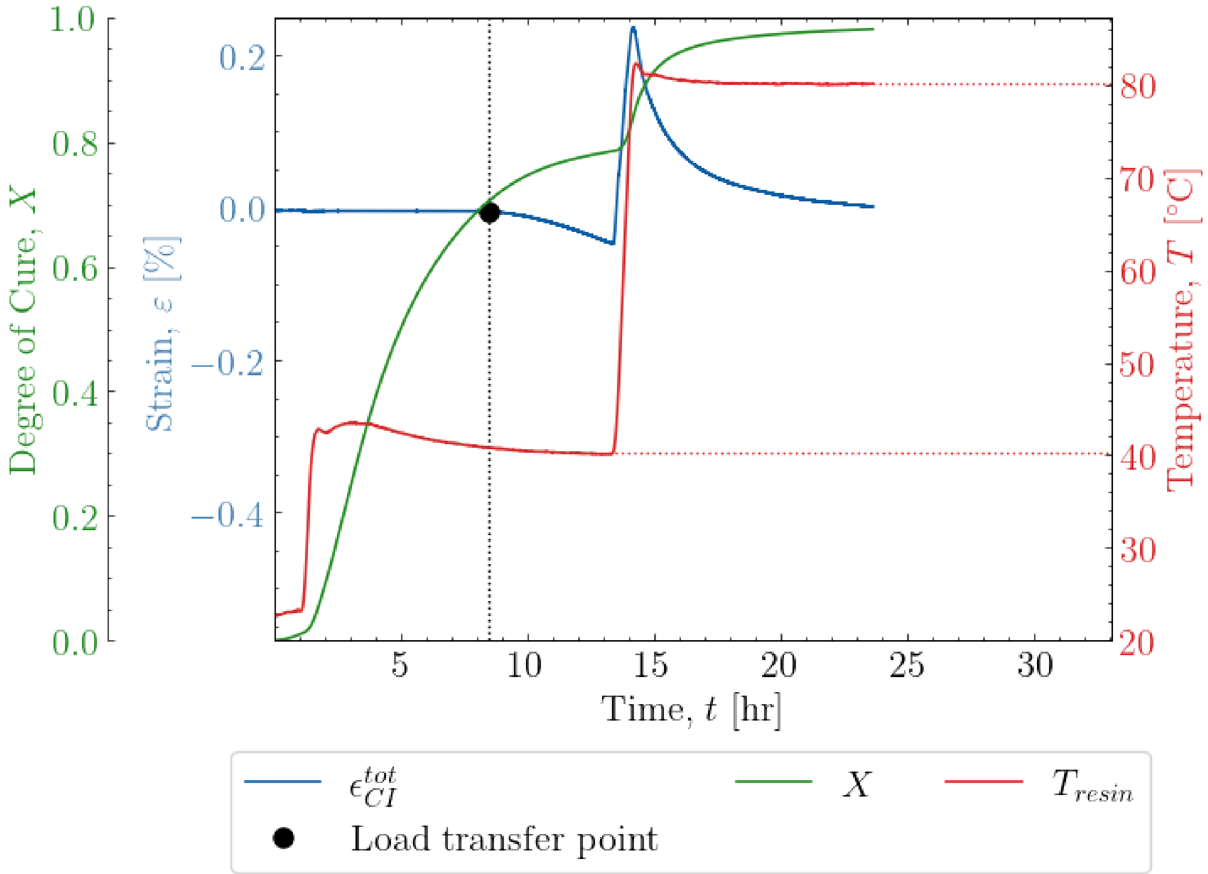


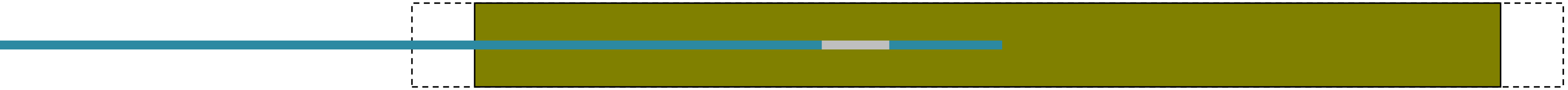


The Experimental Concept – Shrinkage Solid Phase

Comment

For the post-cure temperature, the strain is observed as a drop in the strains due to the contraction as a result of the cross-linking.





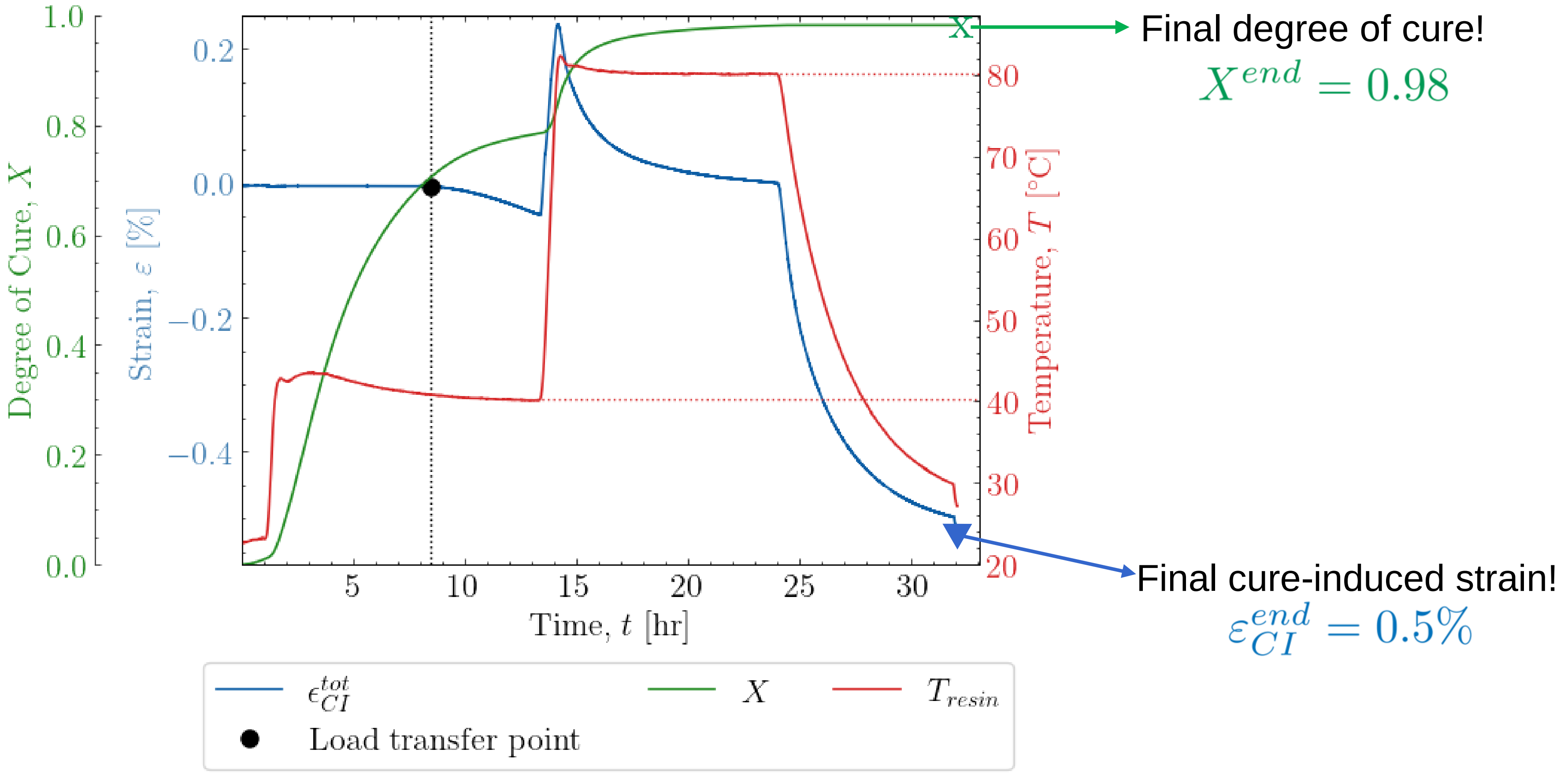
The Experimental Concept – Shrinkage Solid Phase

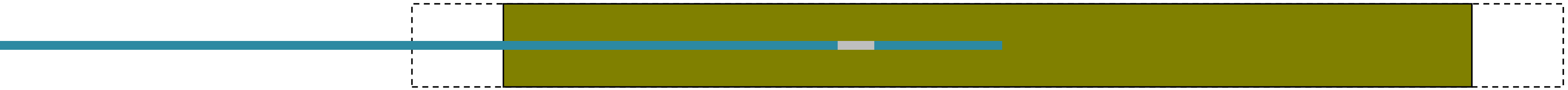
Comment

After the post-cure, the material is cooled down to room temperature and the panel contracts a final time.

The final degree of cure is found as 0.98 and the material is considered fully cured.

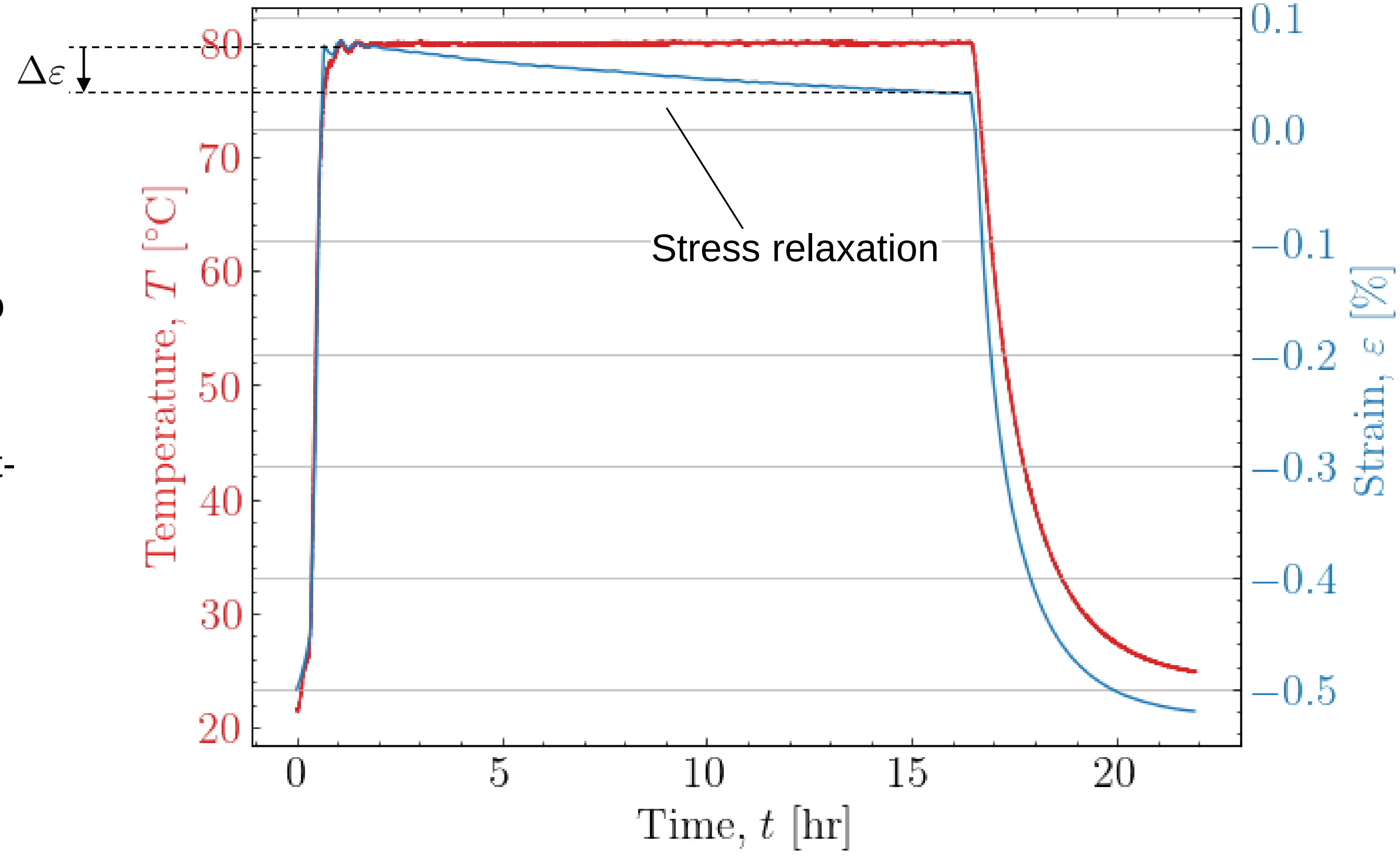
The final cure-induced strain is 0.5% which is the linear shrinkage and not the volumetric shrinkage.





Relaxation Measured Through G-FBG

- Reheat of samples
- Samples assumed to be fully cured
- Held at a typical post-cure temperature for a typical cure time.



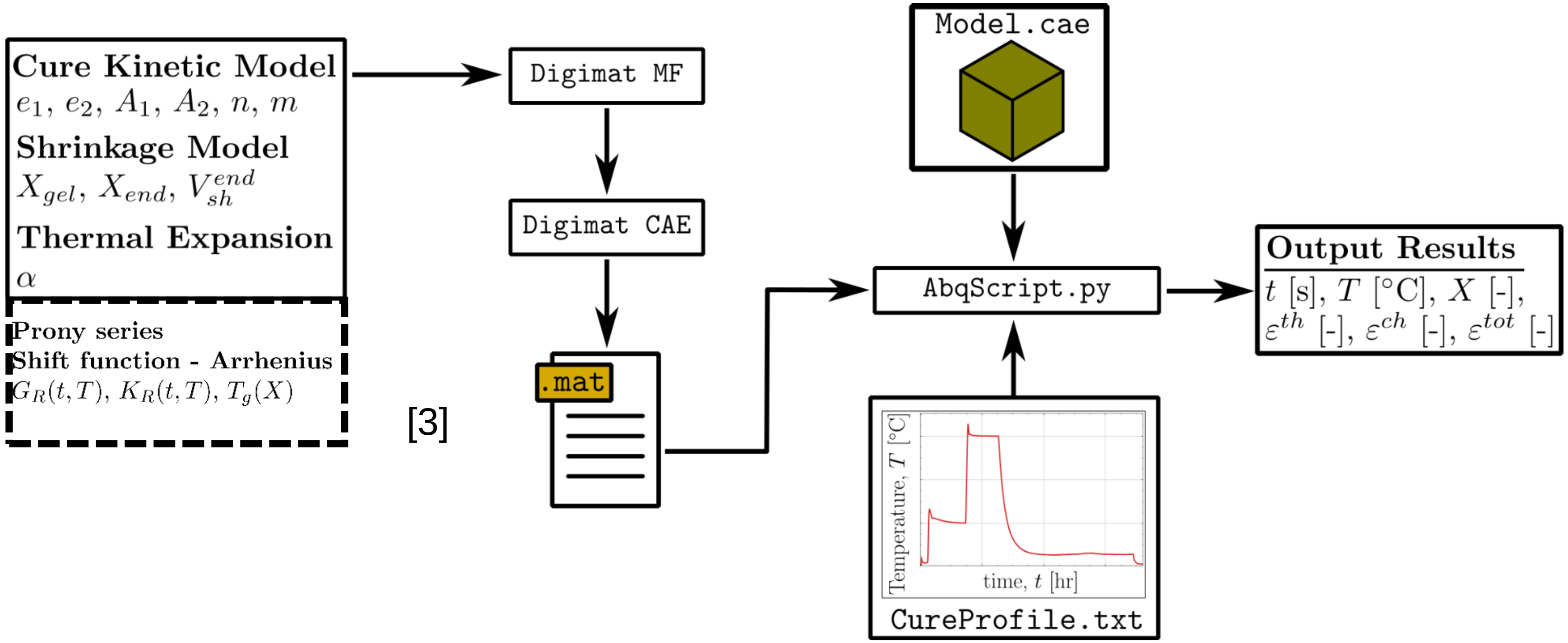
Modelling with Digimat and ABAQUS – G-FBG

Thermo linear elastic (**T-LE**)

$E_{\text{resin}} = \text{constant}$

or

Thermo-viscoelastic (**T-VE**)



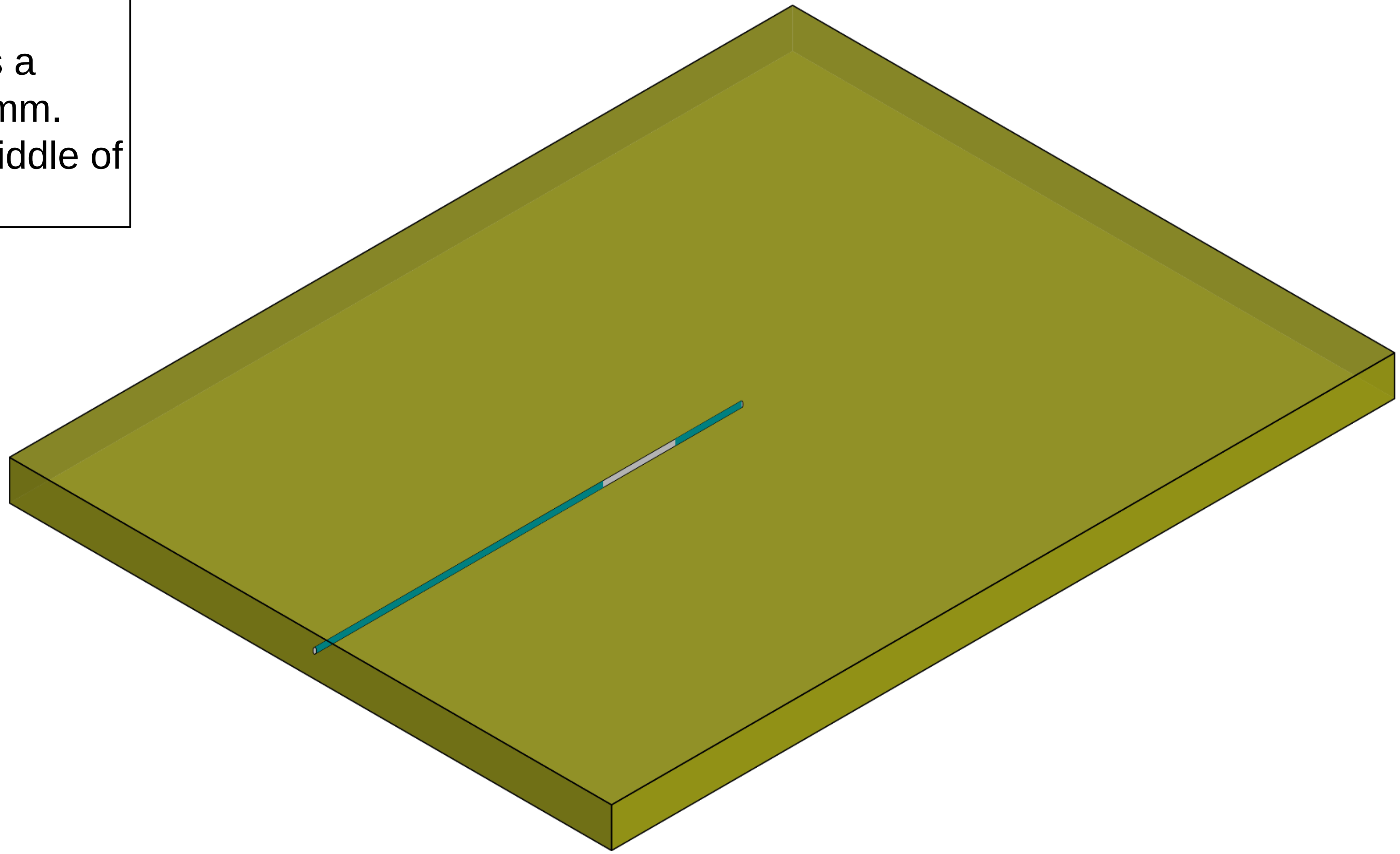
[3]

[3] - Jørgensen et al. (2023) [Pre-print]

Modelling the Experiment – G-FBG

Comment

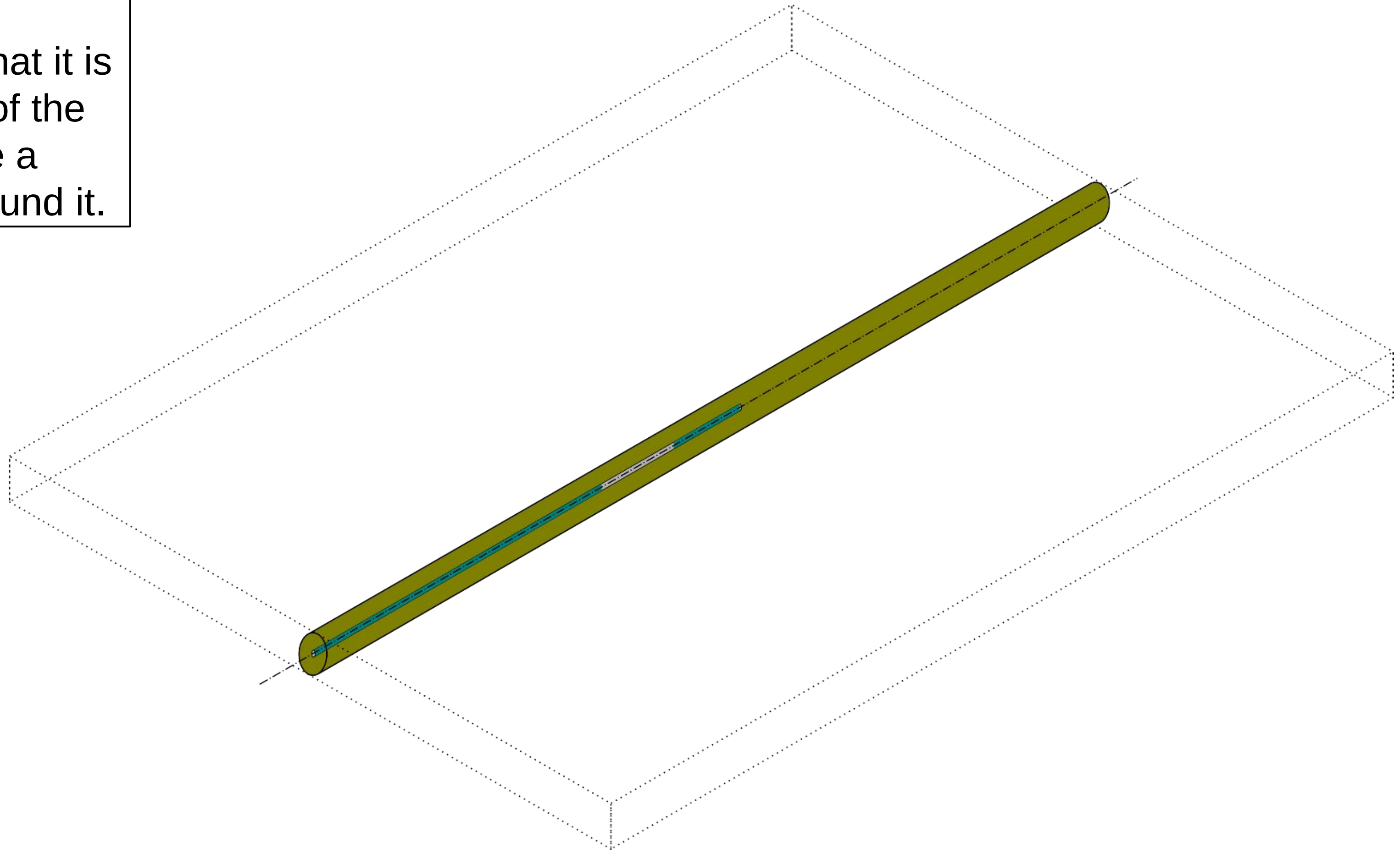
The panel in the experiment is a panel of around 80 x 100 x 4 mm. The G-FBG is placed in the middle of the thickness.



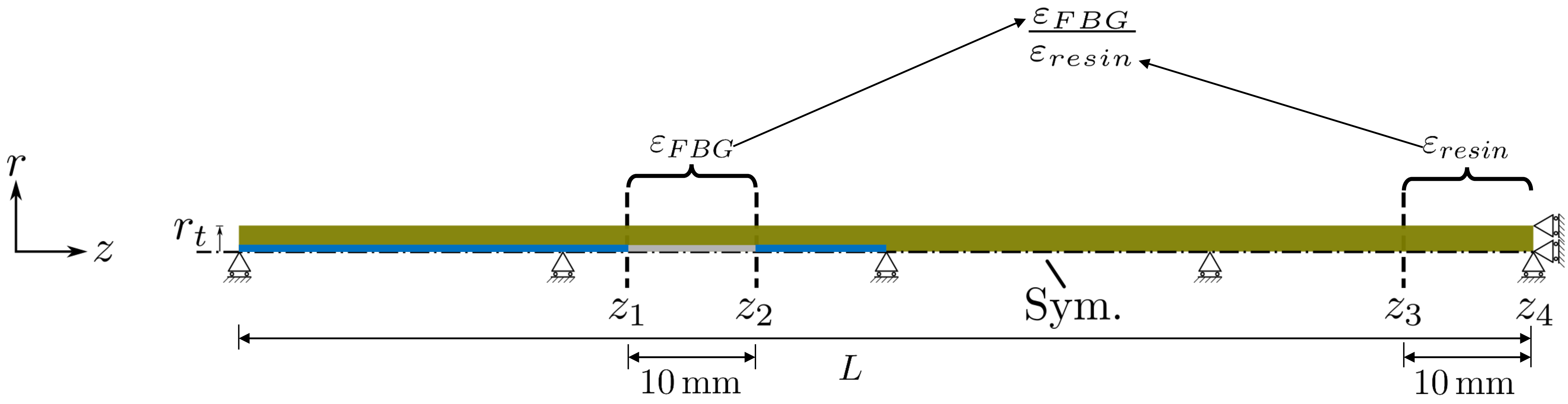
Modelling the Experiment – G-FBG

Comment

For modelling, it is assumed that it is sufficient to model only a rod of the panel as the FBG will only see a certain amount of material around it.



Modelling the Experiment – G-FBG



$r_{FBG} = 0.0625\,\mathrm{mm}$ $\quad E_{\text{G-FBG}} = 70\,\mathrm{GPa}$

$r_t = 2\,\mathrm{mm}$

$L = 100\,\mathrm{mm}$

Comment

As the model is a cylindrical rod it is enough to consider it as a 2D axisymmetrical model.

The strain is computed in the FBG and the resin. The values are found far from each other to make sure they do not influence each other. The ratio between the two is evaluated.

Model Results – G-FBG

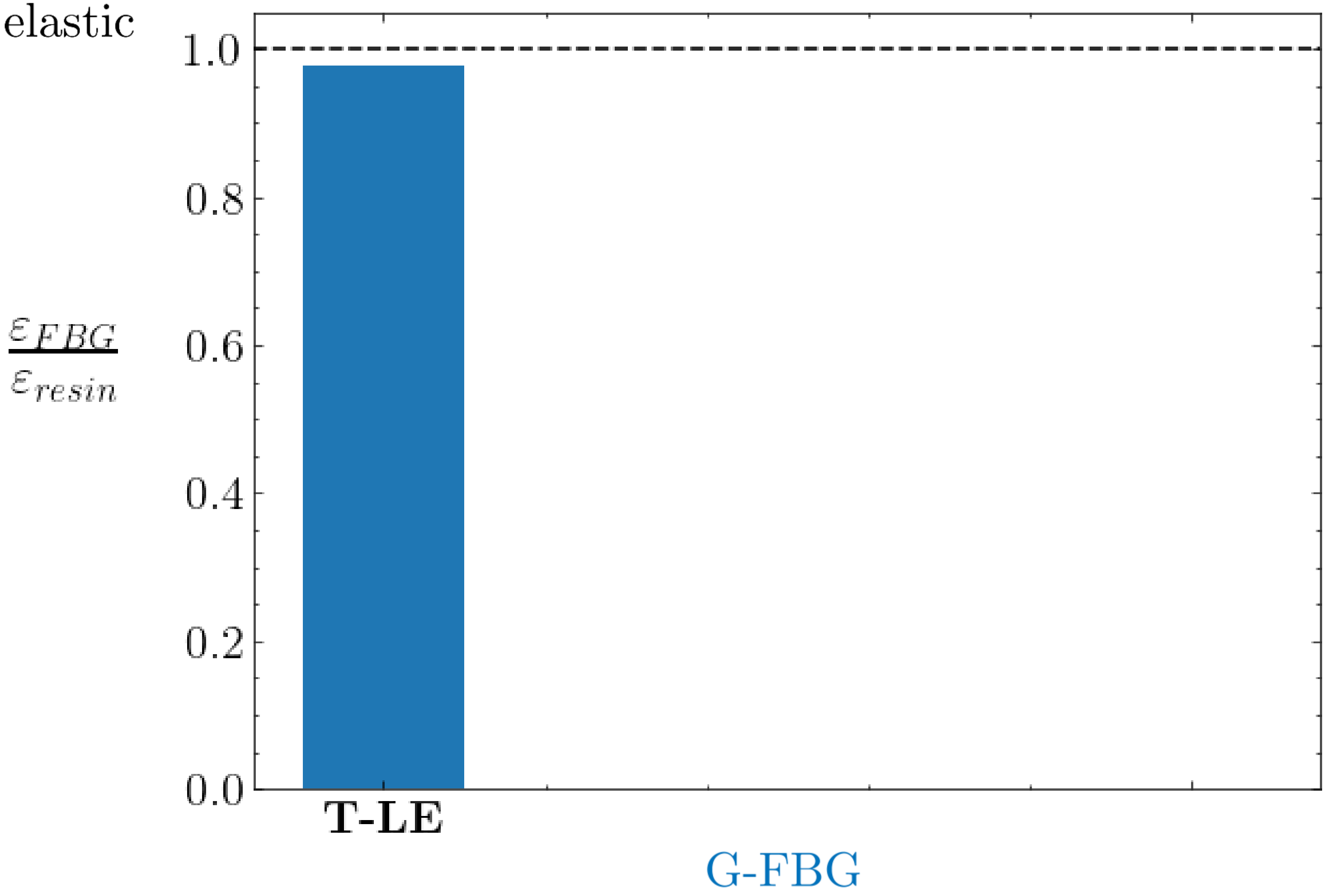
$\mathbf{T\text{-}LE} = \text{Thermo - linear elastic}$

$E_{\text{G-FBG}} = 70\,\text{GPa}$

$E_{\text{resin}} = \text{constant}$

Comment

The model with the Thermo – linear elastic properties shows very little deviation between the two measured strains.



Model Results – G-FBG

T-LE = Thermo - linear elastic

$E_{\text{G-FBG}} = 70\,\text{GPa}$

$E_{\text{resin}} = \text{constant}$

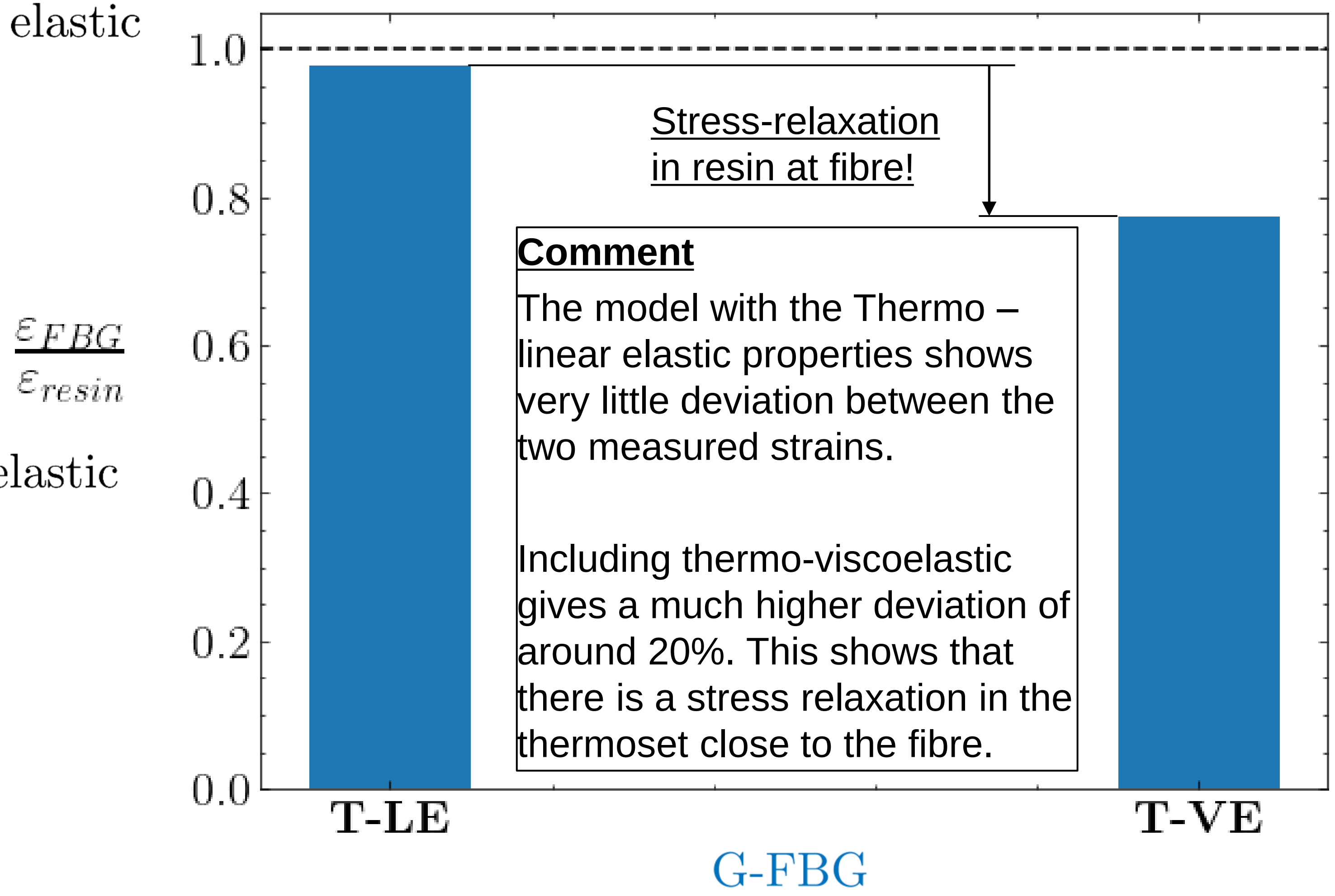
T-VE = Thermo - viscoelastic

$E_{\text{G-FBG}} = 70\,\text{GPa}$

Prony series

Shift function - Arrhenius

$G_R(t,T),\ K_R(t,T),\ T_g(X)$



Comment

The model with the Thermo – linear elastic properties shows very little deviation between the two measured strains.

Including thermo-viscoelastic gives a much higher deviation of around 20%. This shows that there is a stress relaxation in the thermoset close to the fibre.

Introduction to P-FBG

Polymer-based FBG (P-FBG)

- Material is a Cyclo-Olefin-Polymer – Zeonex® [4]
  - Excellent optical properties
  - Moduli of $E_{\text{P-FBG}} = 2.5\,\mathrm{GPa}$

Comment

Therefore, it makes sense to look at a low stiffness FBG like the P-FBG.

[2] - Woyessa et al. (2017) http://doi.org/10.1364/ome.7.000286

Model Results – G-FBG and P-FBG

T-LE = Thermo - linear elastic

$E_{\text{G-FBG}} = 70\,\text{GPa}$

$E_{\text{resin}} = \text{constant}$

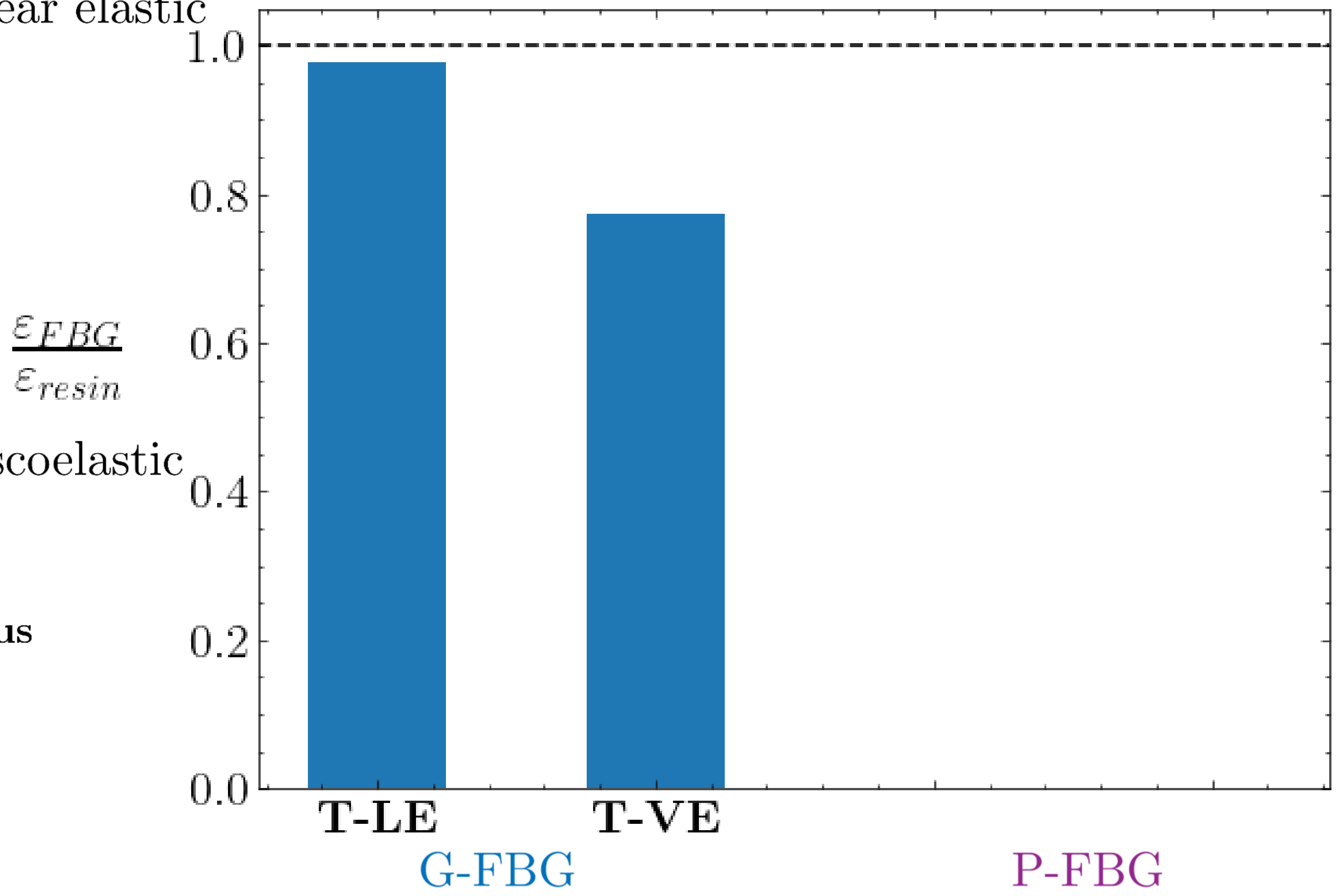
T-VE = Thermo - viscoelastic

$E_{\text{G-FBG}} = 70\,\text{GPa}$

Prony series

Shift function - Arrhenius

$G_R(t,T),\ K_R(t,T),\ T_g(X)$



Comment

Recalling the results of the G-FBG we can compare with the results from the P-FBG.

Model Results – G-FBG and P-FBG

T-LE = Thermo - linear elastic

$E_{\text{G-FBG}} = 70\,\text{GPa}$

$E_{\text{resin}} = \text{constant}$

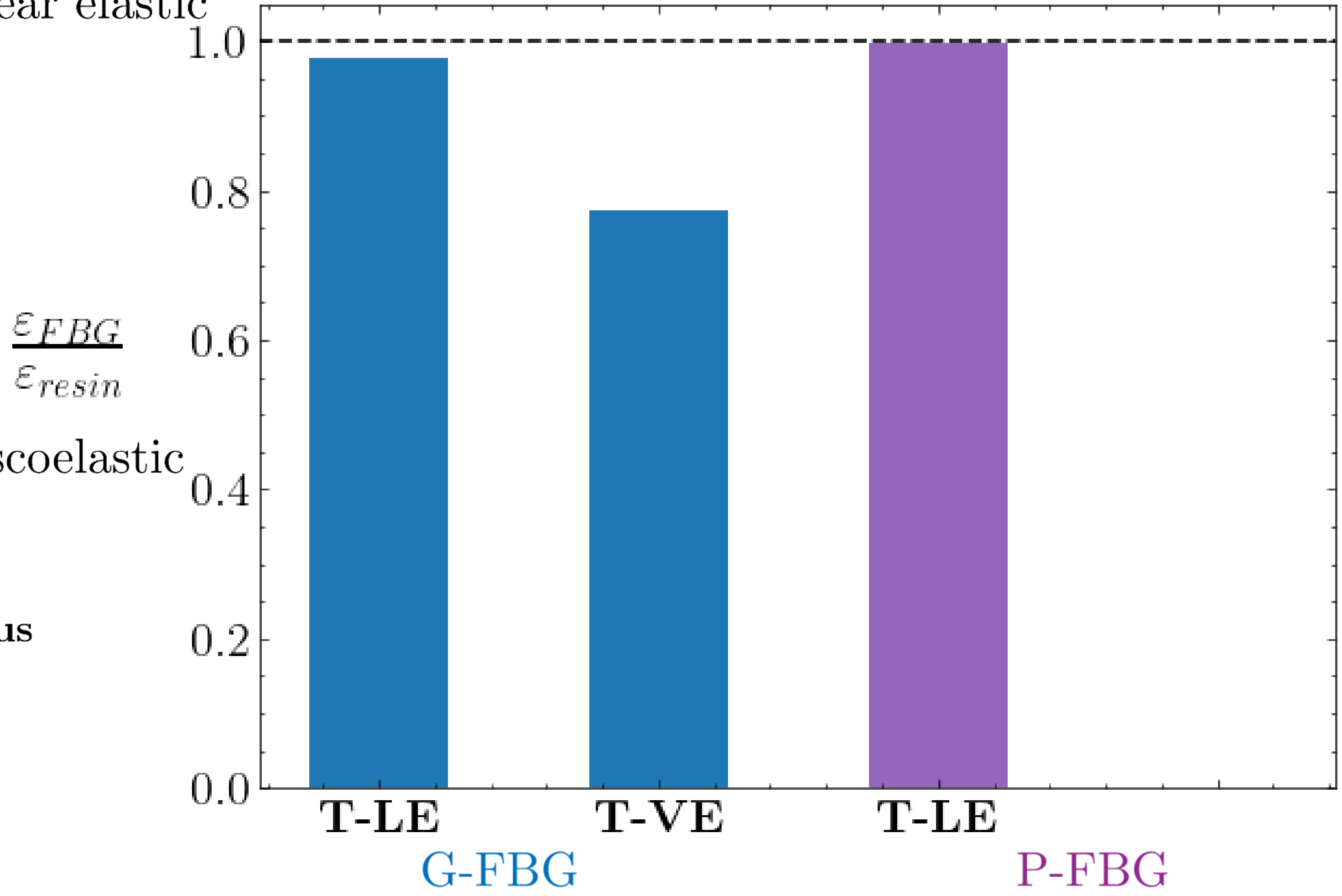
T-VE = Thermo - viscoelastic

$E_{\text{G-FBG}} = 70\,\text{GPa}$

Prony series

Shift function - Arrhenius

$G_R(t,T),\ K_R(t,T),\ T_g(X)$



$E_{\text{P-FBG}} = 2.5\,\text{GPa}$

Comment

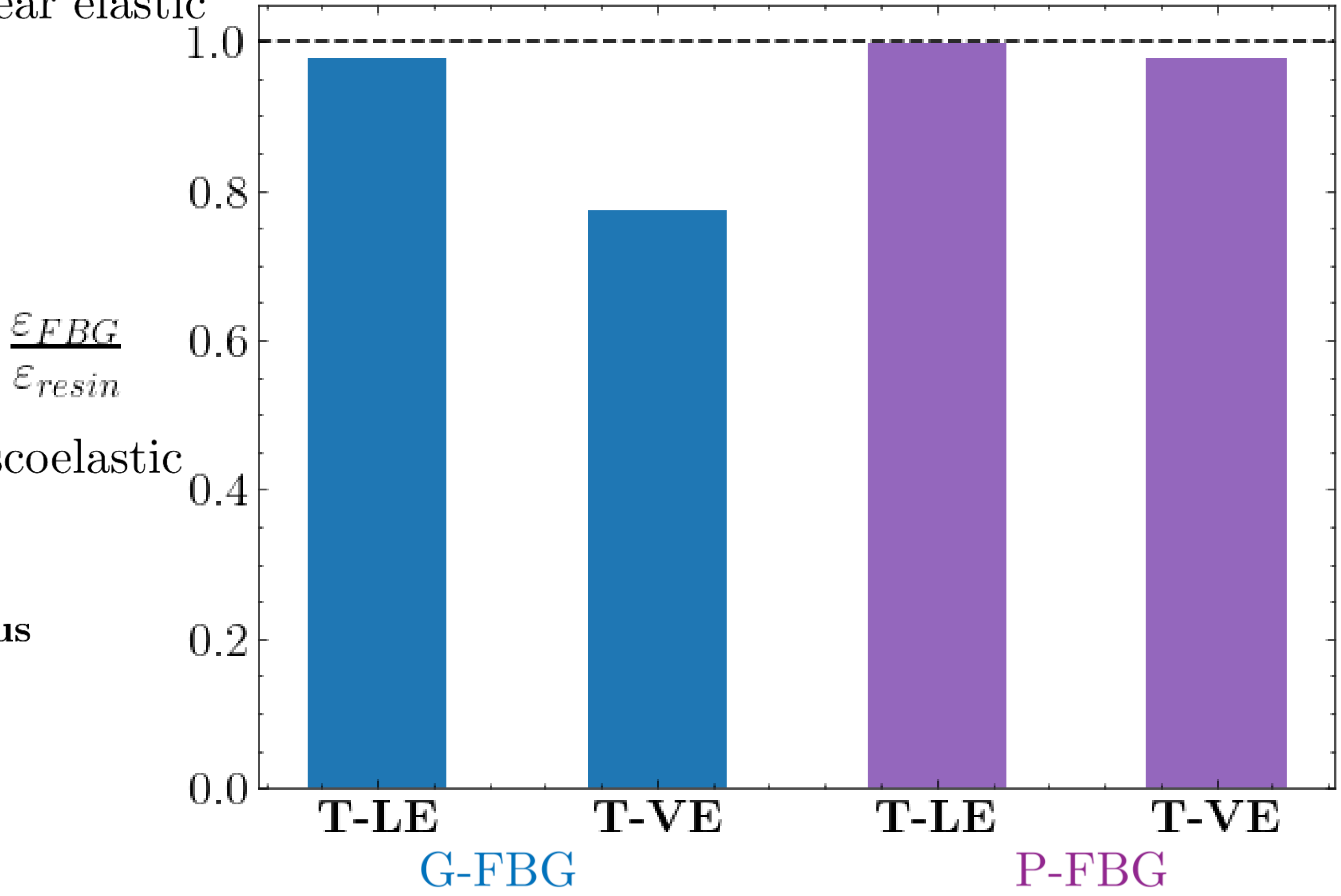
The thermo linear elastic response of the P-FBG model shows close to no deviation.

Model Results – G-FBG and P-FBG

T-LE = Thermo - linear elastic

$E_{\text{G-FBG}} = 70\,\text{GPa}$

$E_{\text{resin}} = \text{constant}$



$E_{\text{P-FBG}} = 2.5\,\text{GPa}$

T-VE = Thermo - viscoelastic

$E_{\text{G-FBG}} = 70\,\text{GPa}$

Prony series

Shift function - Arrhenius

$G_R(t,T),\ K_R(t,T),\ T_g(X)$

Comment

Adding the thermo viscoelastic properties the deviation becomes similar to the G-FBG T-LE solution but still acceptable.

Summary and Ongoing Work

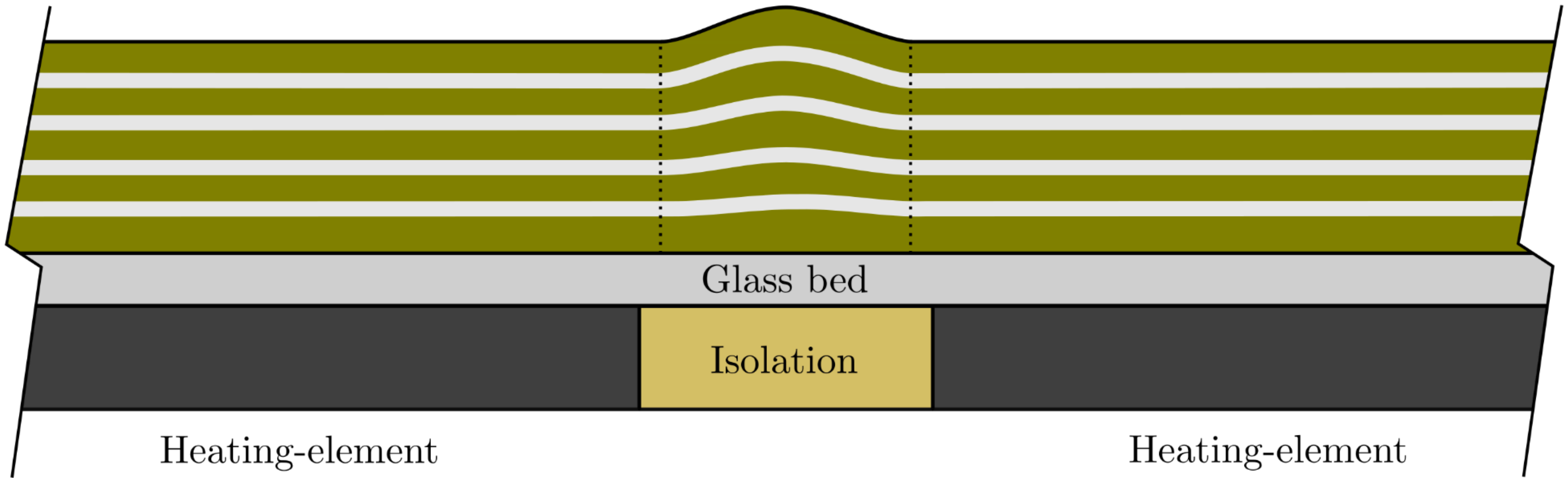
- Currently performing experimental investigations using *both* G-FBG and P-FBG
- Characterising viscoelastic evolution during curing with DMA at DTU and Rheology at National Composites Centre Bristol (NCC).
- Assist the research into cure-induced wrinkles with better knowledge of the thermoset behaviour

Comment

Now that the model has been developed. A validation with experiments is ongoing.

The work with DMA and rheology is ongoing to understand the viscoelasticity better conducted in collaboration between DTU and NCC.

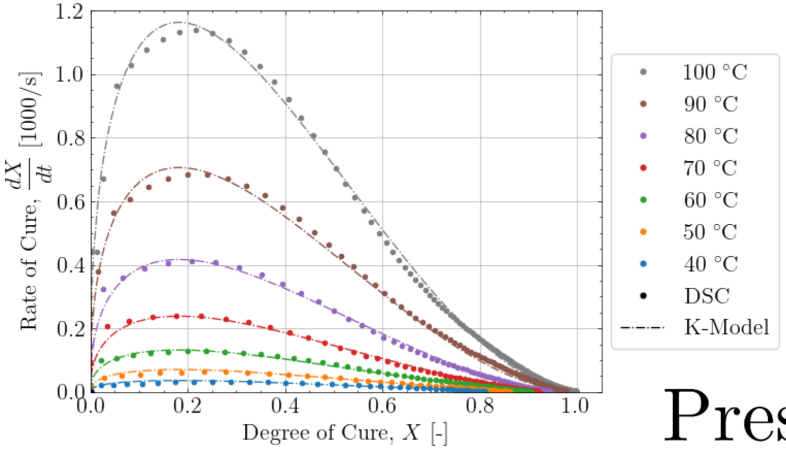
Cure-induced Defects - Wrinkles



Comment

In the end, this research will be used to investigate whether wrinkles can appear due to temperature gradients causing them to happen due to the effects it has on the curing.

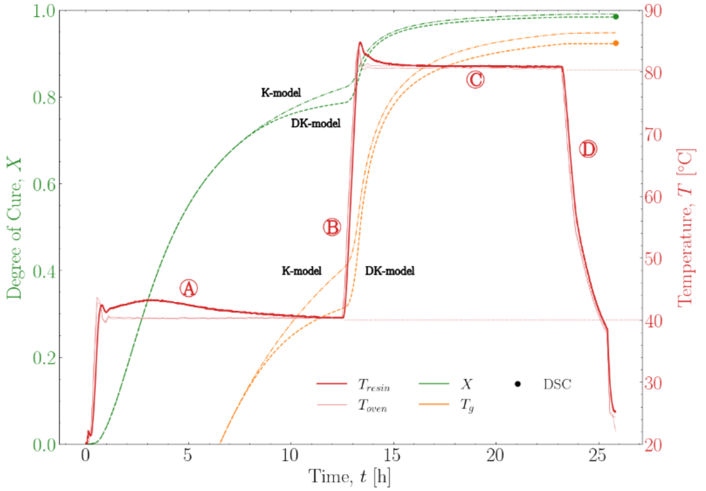
Cure Characterisation to Achieve Model Inputs



Comment

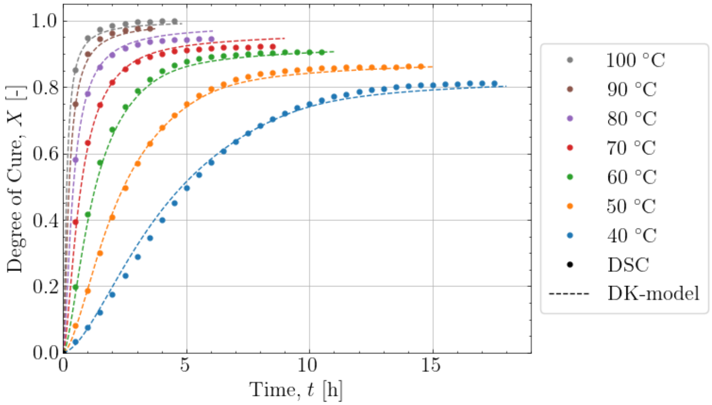
For further interest in how we characterise curing with DSC and make model predictions.

Join the Risø Symposium in september at DTU Wind in Risø, Denmark.



Presented at the Risø Symposium

4th - 7th September!



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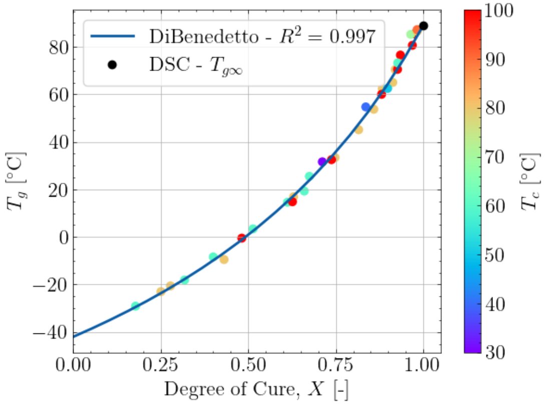


DTU

DTU

43rd Risø International Symposium on Materials Science

Composites for wind energy:
Manufacturing, operation and end-of-life

Composites for wind energy: Manufacturing, operation and end-of-life

4 - 7 September 2023

The focus of the 43rd Risø Symposium is on composites for wind energy. This symposium takes a life cycle perspective and addresses manufacturing, performance during operation and end-of-life of composites for wind energy. Over the last 40 years, wind turbine blades have grown an order of magnitude. Today, the longest blades are exceeding 100 meters and weighing 50 tons. Because of this growth in blade size, the cost of wind energy can now compete with fossil-based energy sources on market terms.

As society is thriving towards a zero-emission future, the wind energy sector is foreseen to further expand. Longer wind turbine blades with improved overall lifetime, reliability, recyclability, sustainability, operability and maintainability are some of the objectives set on this component. To address these upcoming and ambitious requirements, the symposium welcomes contribution dedicated to the manufacturing, the operation and performance, as well as end-of-life strategies for wind turbine blades.

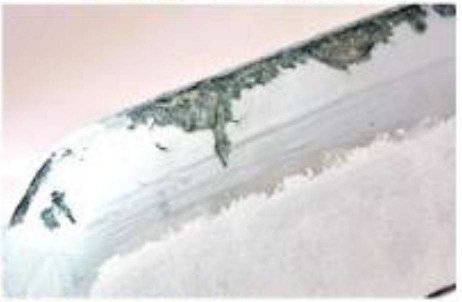
Important Dates

15 March 2023: Abstract submission

01 July 2023: Paper submission

https://www.morressier.com/call-for-papers/63ff34e08d36d800127feb22

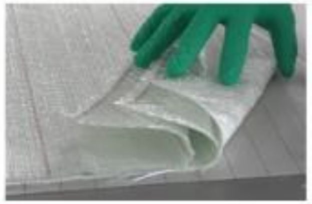
31 August 2023: Registration deadline



Manufacturing

Characterization and development of manufacturing processes for wind turbine blades

Existing and alternative manufacturing technologies, constrains and new opportunities, process characterization, cure kinetics and residual stresses, modelling, manufacturing defects, repair.



Operation

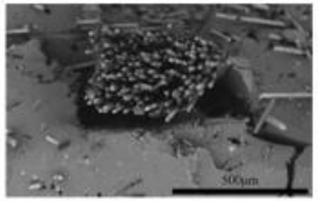
Experimental characterization, mechanical properties and performance of composites for wind turbine blades

Structural design and performance of blade structures; Key composite design properties: stiffness, strength, fracture and fatigue resistance; Materials development: hybrid, bio-based, thermoplastic composites and smart materials; adhesive joints and fibre/matrix interfaces; Leading edge erosion, repair, structural health monitoring. Micro and macro structural characterization using X-ray tomography and ultrasound; Novel test methods for composites under static and cyclic loading; development of test methods for structural elements, e.g. ply-drops and wrinkles, and full-scale testing of blades.

End-of-life

Strategies to address the end-of-life challenges of composites and of wind turbine blades

Reuse and lifetime extension, recyclable composite, composite recycling processes, repurposing, decommissioning, life-cycle analysis (LCA). Recycling of manufacturing waste and end-of-use wind turbines, recycling processes and products incorporating recycled materials, material substitution in wind turbine blades increasing the recycled content.



Registration

The registration fee is DKK 4500 (approx. EUR 600), and covers access to lectures, lunch and refreshments all days, conference dinner, and social arrangements. The registration fee for students is DKK 2000 (approx. EUR 270).

Contact

Lars P. Mikkelsen, Chairman
Justine Beauson, Chairwoman
E-mail: symp43@windenergy.dtu.dk
Website:
https://wind.dtu.dk/about/symposium-on-materials-science

Summary and Ongoing Work

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Questions

Jesper Kjær Jørgensen – PhD Student
Composites Manufacturing and Testing
Technical University of Denmark

References

[1] - M. M. Maduro, “Influence of Curing Cycle on the Build-up of Residual Stresses and the Effect on the Mechanical Performance of Composites”, Risø, Roskilde, Denmark: DTU Wind Energy, 2021.

[2] - Mikkelsen, L.P., Jørgensen, J.K., Mortensen, U.A., Andersen, T.L., “Optical fiber Bragg gratings for in-situ cure-induced strain measurements” [Pre-print]

[3] - Jørgensen, J.K., Mikkelsen, L.P., “Tailored cure profiles for reduction of the cure time and shrinkage of an epoxy thermoset” [Pre-print]

[4] - G. Woyessa, A. Fasano, C. Markos, A. Stefani, H. K. Rasmussen and O. Bang, "Zeonex microstructured polymer optical fiber: fabrication friendly fibers for high temperature and humidity insensitive Bragg grating sensing," Optical Materials Express, vol. 7, no. 1, p. 286, 1 2017.

Extra Slides

Modelling of the Relaxation Behaviour

Cole et al. (1991) - Diffusion-kinetic model

$$\frac{\mathrm{d}X}{\mathrm{d}t} = \frac{k(T)X^m(1-X)^n}{1+\exp\left[C(X-X_c(T))\right]} \qquad (1)$$

$$k(T) = A\exp\left(-\frac{e_a}{RT}\right) \qquad (2)$$

$$X_c(T) = X_{cT}T + X_{c0}$$

A.T. DiBenedetto (1987)

$$T_g(X) = T_{g0} + \frac{\xi X(T_{g\infty} - T_{g0})}{1-(1-\xi)X} \qquad (3)$$

Cole et al. (1991) - https://doi.org/10.1021/ma00011a012 DiBenedetto (1987) - https://doi.org/10.1002/polb.1987.090250914

Modelling of the Relaxation Behaviour

Johnston (1998) shrinkage model

$$V_{sh} = \begin{cases} 0, & X < X_\sigma \\ V_{sh}^{end}\left(\frac{X - X_\sigma}{X_{end} - X_\sigma}\right)^2, & X_\sigma < X < X_{end} \\ V_{sh}^{end}, & X = X_{end} \end{cases} \tag{4}$$

Johnston (1997) - https://www.researchgate.net/publication/253529873_

Modelling of the Relaxation Behaviour

Shear and Bulk Prony series

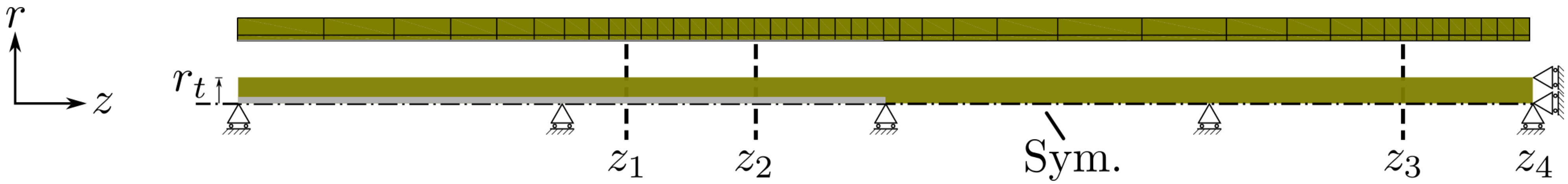
$$G_R(t,T) = G_0(T)\left[1 - \sum_{i=1}^{n} w_i(1 - \exp(-t/\tau_i))\right], \quad G_0(T) = G(t=0,T) \quad (5)$$

$$K_R(t,T) = K_0(T)\left[1 - \sum_{i=1}^{n'} w_i^*(1 - \exp(-t/\tau_i^*))\right], \quad K_0(T) = K(t=0,T) \quad (6)$$

Arrhenius Shift Function

$$\ln A_T(T) = \frac{\Delta U}{R}\left(\left(\frac{1}{T} - \frac{1}{T_0}\right) - \left(\frac{1}{T_g(X)} - \frac{1}{T_g(X_{ref})}\right)\right) \quad (7)$$

Modelling the Experiment – Mesh

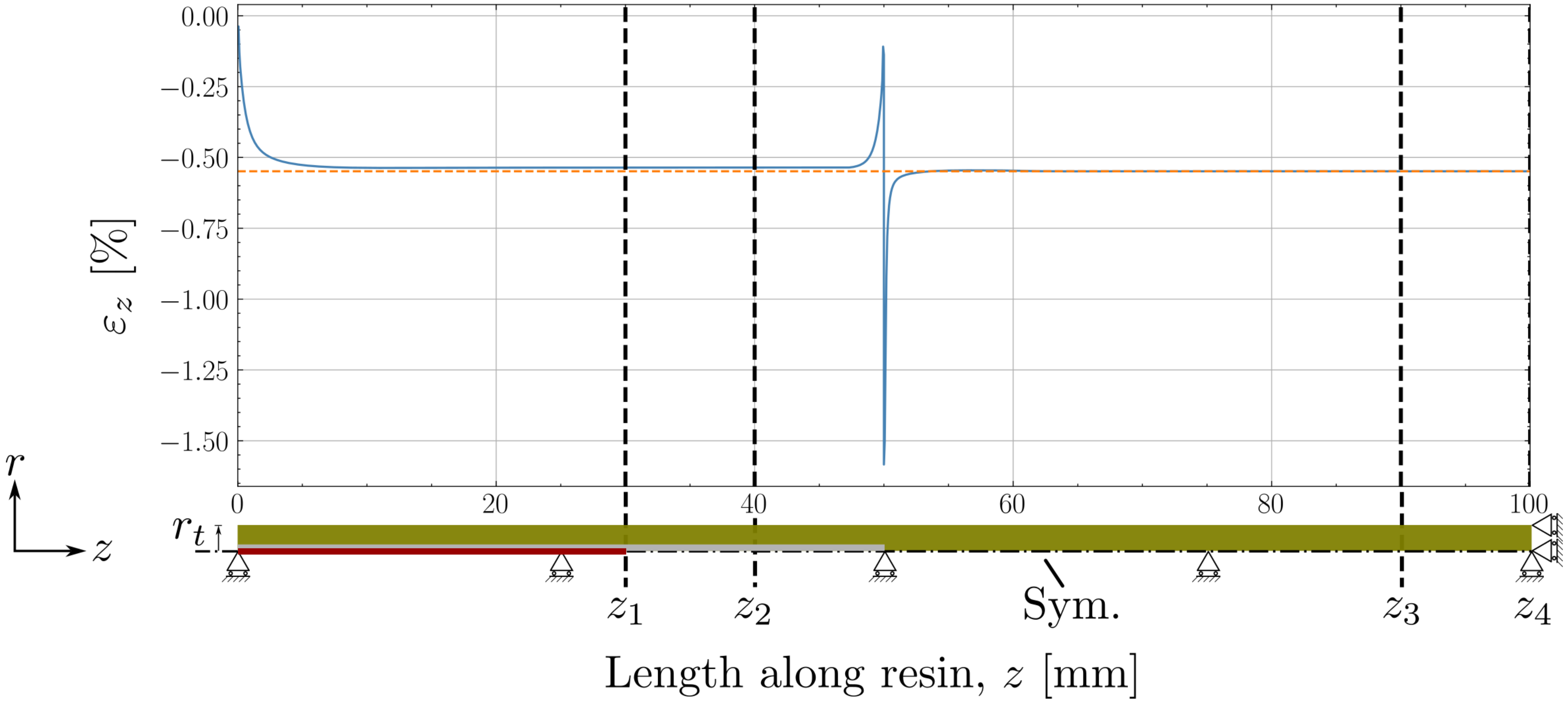


Elements across FBG radius: 4

More elements in region with measurement strain.

Convergence is evaluated by the strain in the resin and the strain change is insiginificant for an element size lower than 0.5 mm.

Modelling the Experiment – Edge Effects



Modelling the Experiment – Radius Effects G-FBG vs P-FBG