Simplified Methods to Obtain Long-Term Properties of Composites in a wide range of Marine/Offshore Environments

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DNV GL led Joint Industry Project “Affordable Composites

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Project manager Ramin Moslemian

Twelve industrial partners

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Marine Offshore Environment

DNVGL Rules for Composites

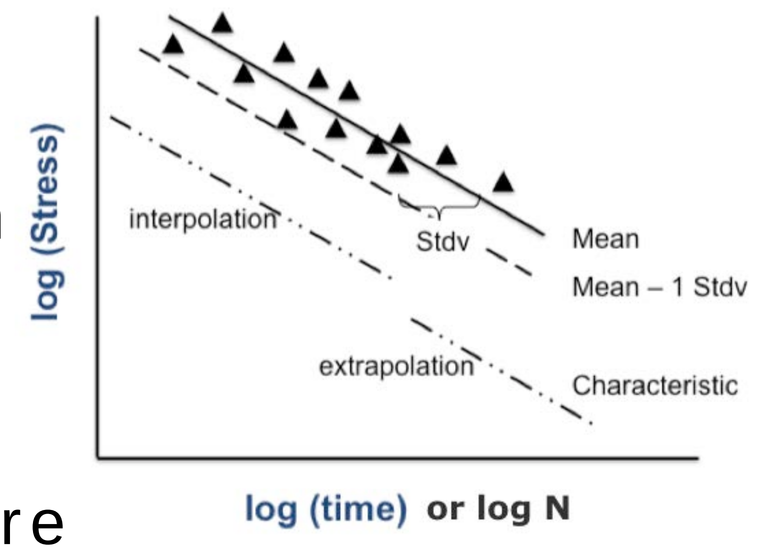
		First issue:
Thermosets:	DNVGL-ST-C501	
	“Composite Components”	(2003)
Thermoplastics:	DNVGL-ST-F119	
	“Thermoplastic Composite Pipes”	(2015)


DNV
Offshore Standard
DNV-OS-C501
COMPOSITE COMPONENTS
2003
DET NORSKE VERITAS


DNV·GL
STANDARD
DNVGL-ST-F119 Edition August 2018
Thermoplastic composite pipes

Short and Long Term Properties in different Environments

- Obtain data for ALL critical failure mechanisms in ALL environments and ALL temperatures
- Usually extreme conditions are sufficient (high and low)
- Suitable engineering approach, but…



Experience

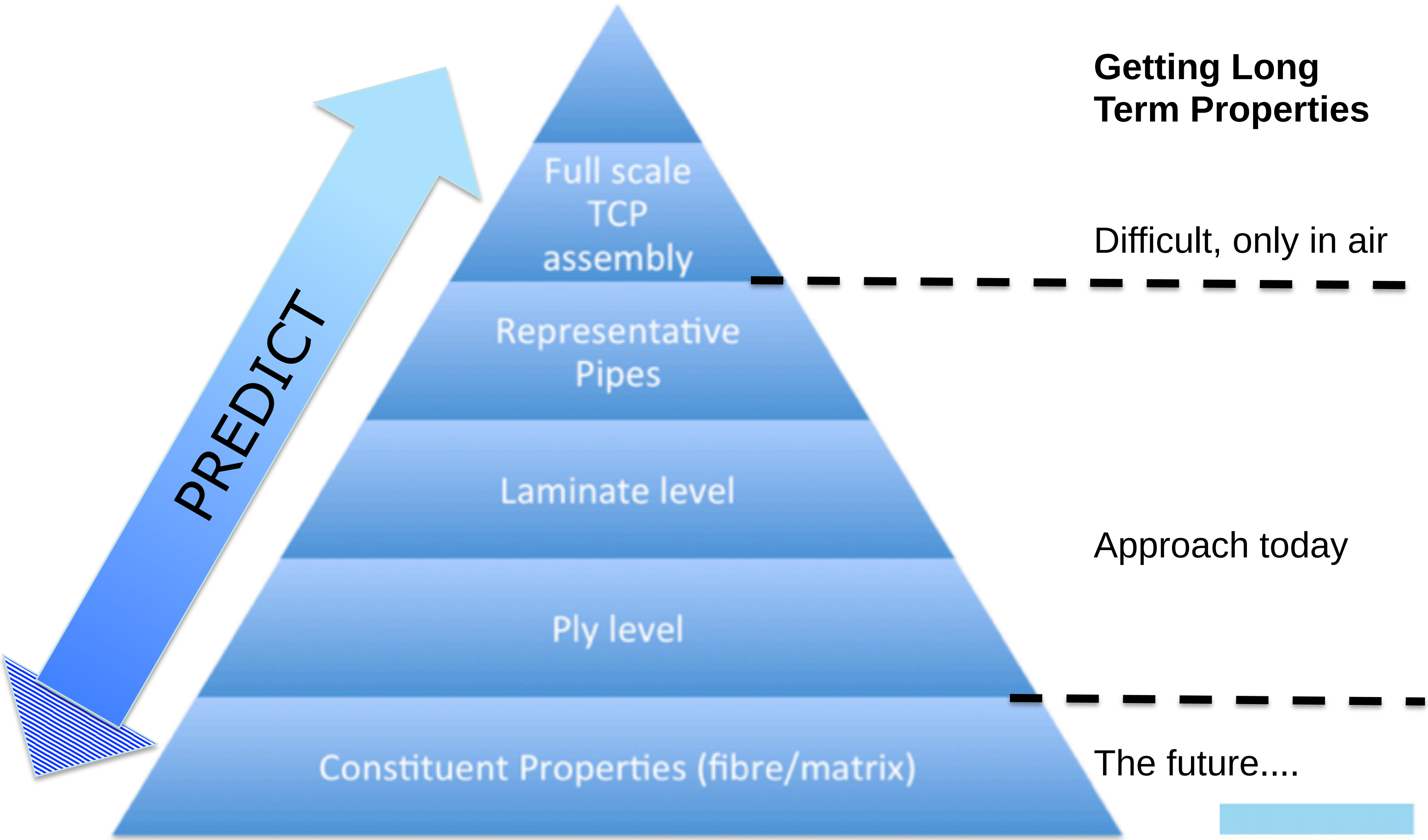
Many tests.

Testing in environments is difficult and time consuming.

This has stopped several good ideas or projects for composite applications.

But is has worked.

TEST PYRAMID in DNVGL-ST-F119



Getting Long Term Properties

Difficult, only in air

Approach today

The future....

Materials in this presentation

Glass fibers

- 3B

Epoxy

- Hexion RIMR135 resin
- RIMH137 hardener

Exposure to water (saturation)

(other systems are currently being tested)

Long-term Properties “Degradation, Aging”

Reversible:

Swelling

Irreversible:

Chemical – break or rearrange bonds

Mechanical – break bonds (cracks, yield)

Strategy:

Check whether irreversible effects can happen (usually avoid or effect should be small), then look at mechanical properties

CONTENTS

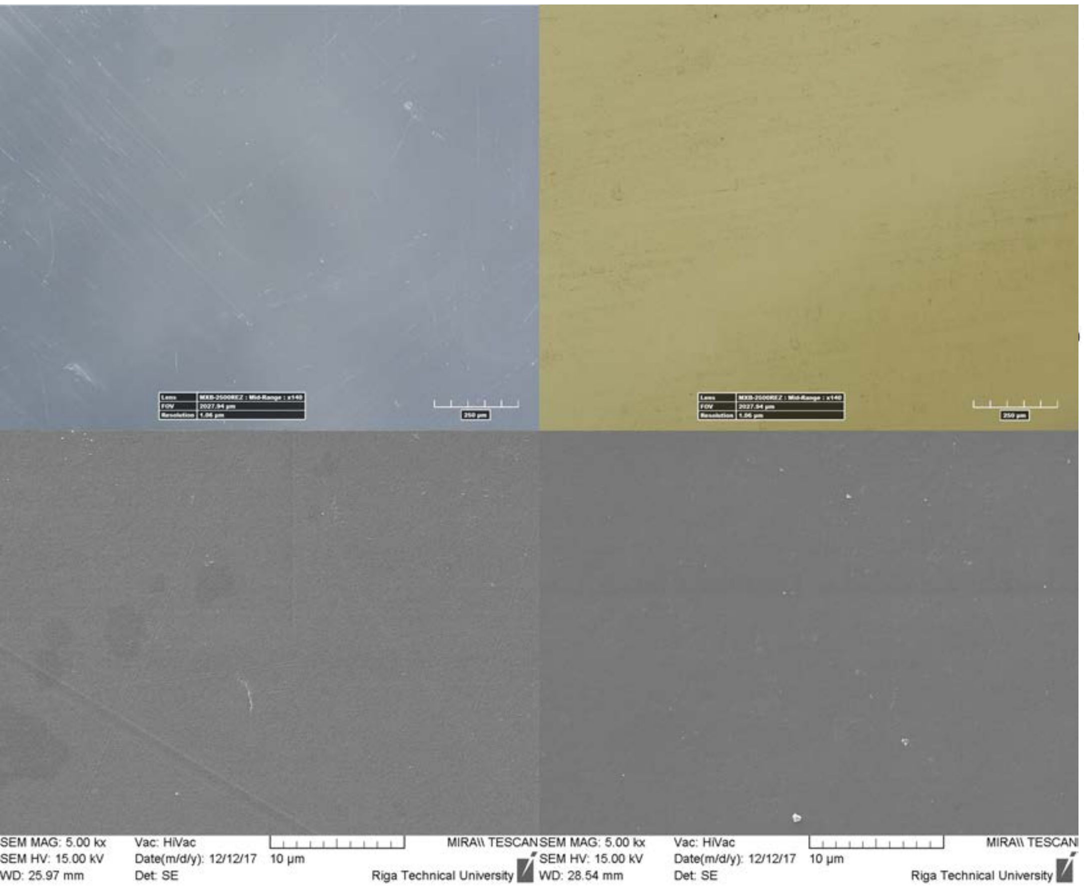
Constituents:

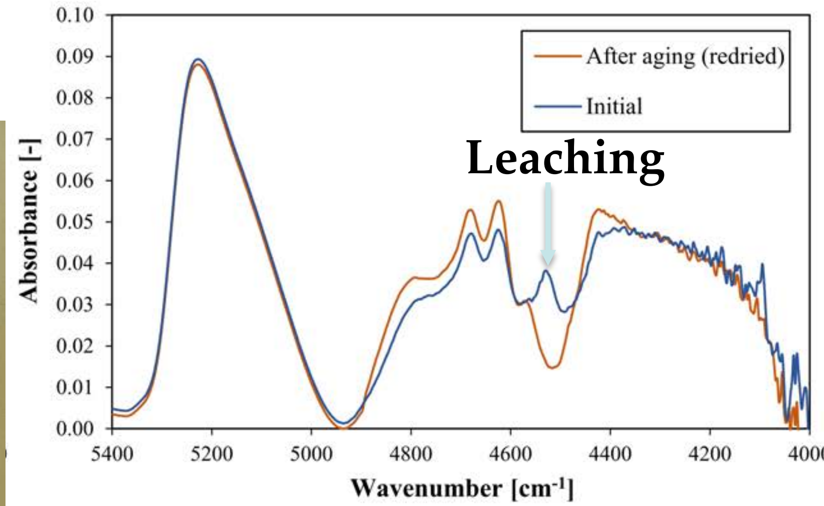
- Matrix
- Fibers
- Interphase / Sizing

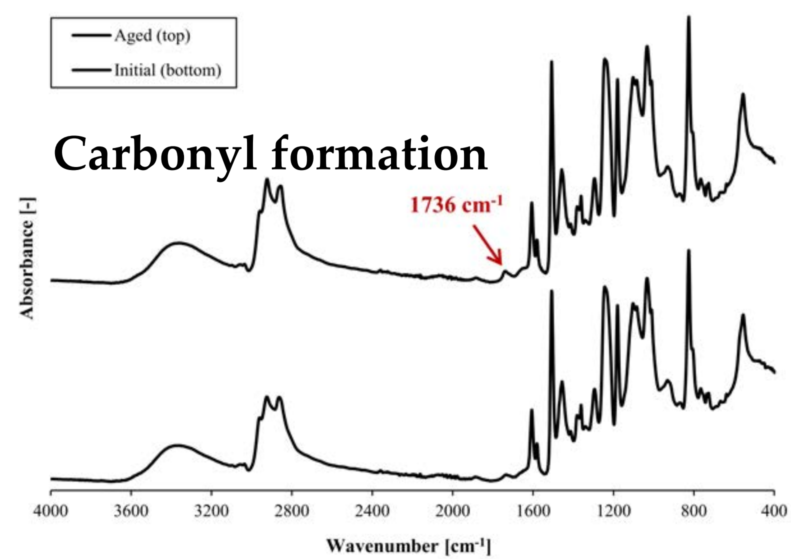
Composite Ply Properties:

- Matrix dominated
- Fiber dominated
- Interlaminar Shear

Amine Epoxy







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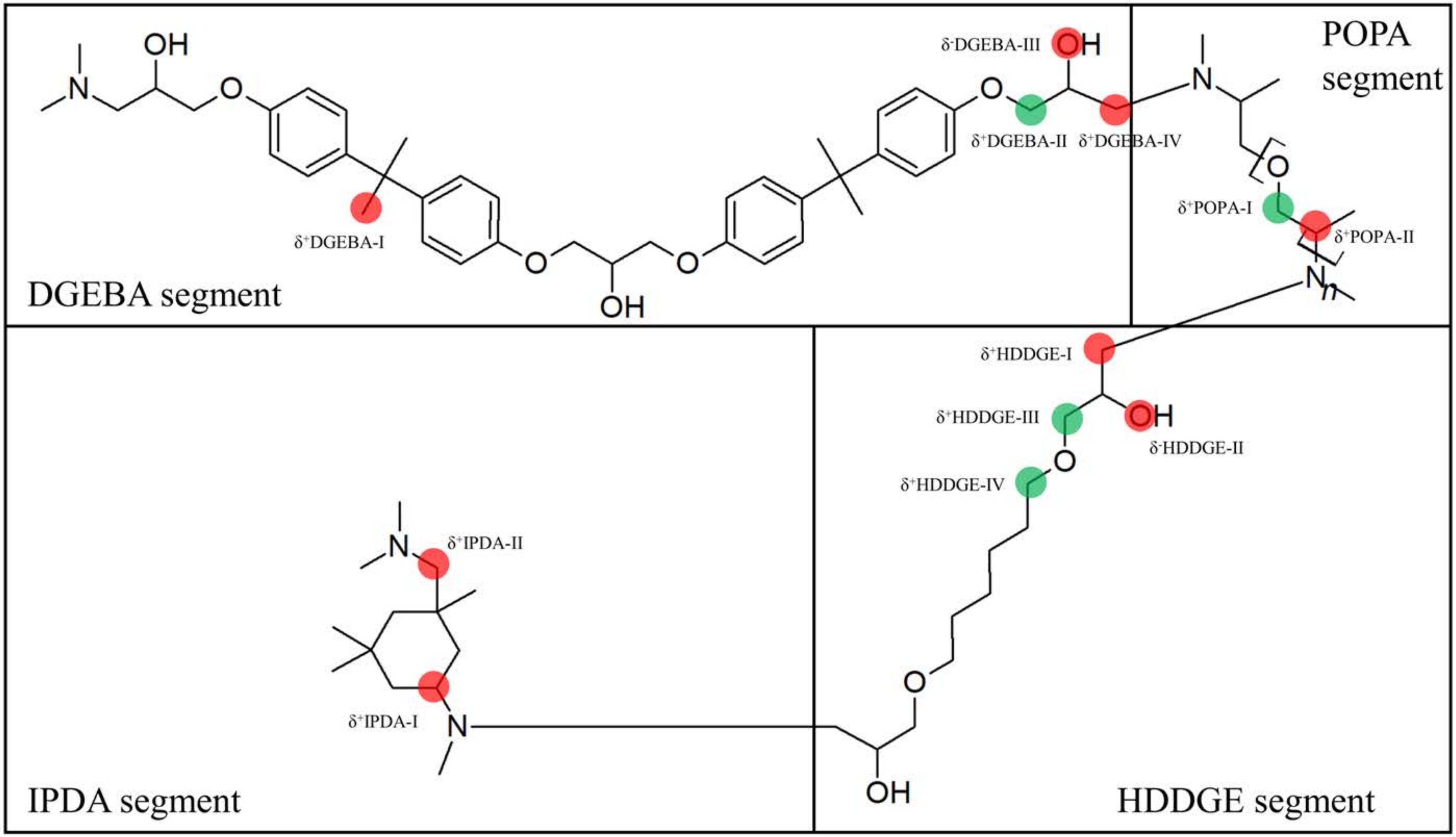
Krauklis, A.; Echtermeyer, A. Mechanism of Yellowing: Carbonyl Formation during Hygrothermal Aging in a Common Amine Epoxy. Polymers 2018, 10, 1017–1031, doi:10.3390/polym10091017

Weak Points in the Structure



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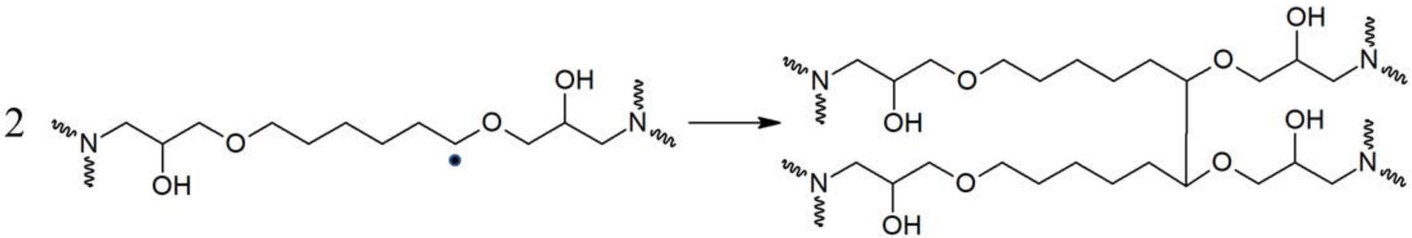
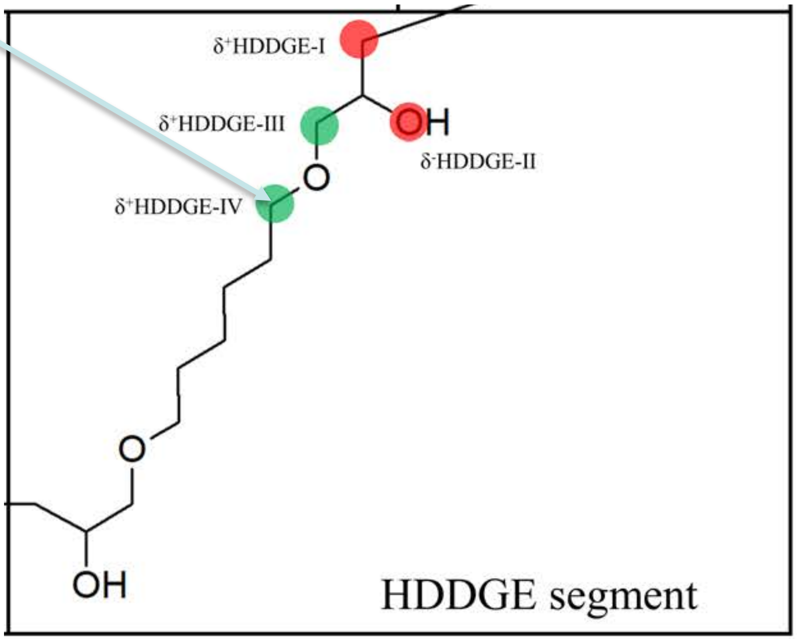
Krauklis, A.; Echtermeyer, A. Mechanism of Yellowing: Carbonyl Formation during Hygrothermal Aging in a Common Amine Epoxy. Polymers 2018, 10, 1017–1031, doi:10.3390/polym10091017

Weak Points: Crosslinking



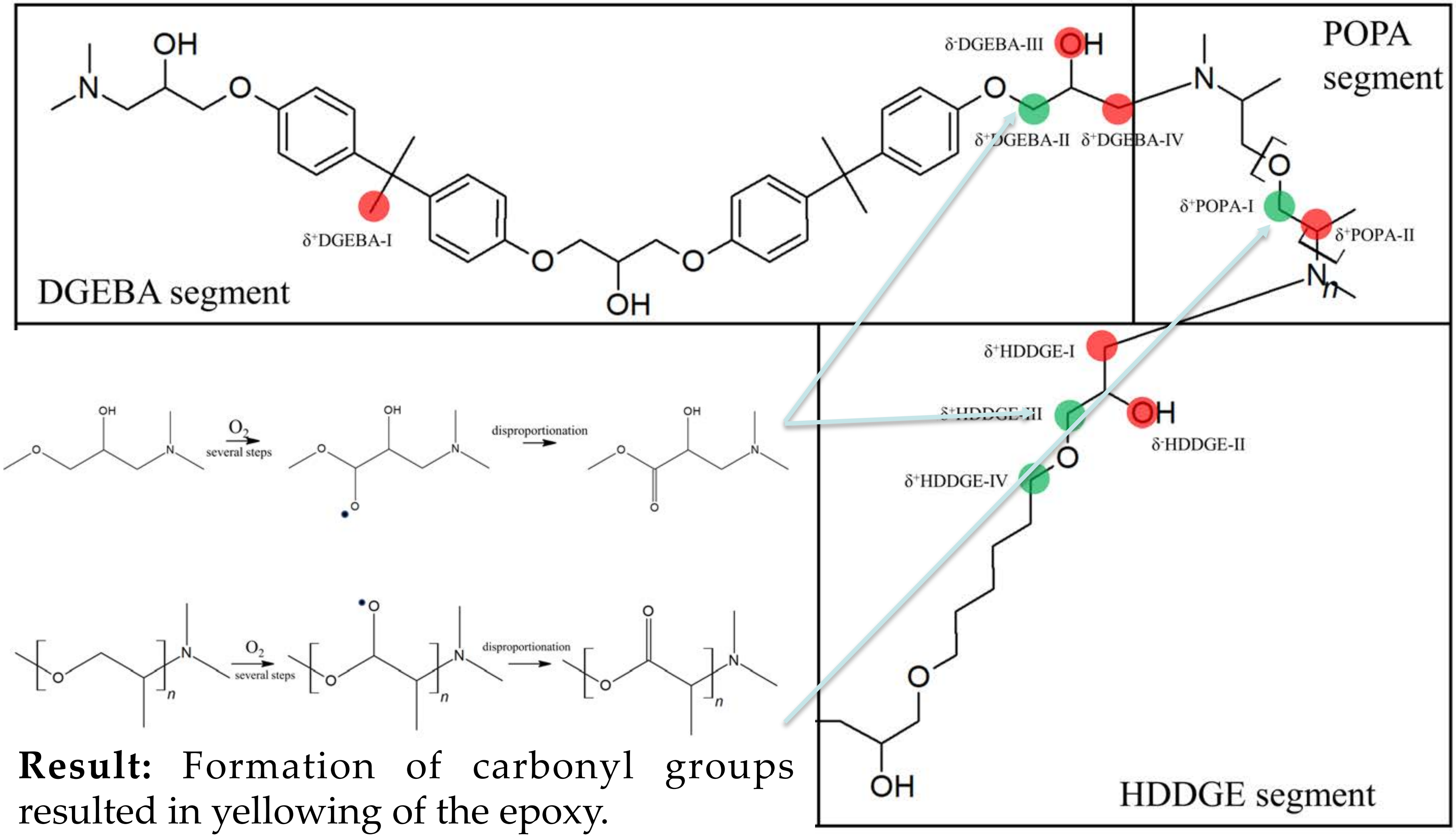
Result: Slightly increased T_g of the redried epoxy.



T_g of dry, wet and redried epoxy was 81.7, 59.1 and 84.7 °C, respectively.

Krauklis, A.; Echtermeyer, A. Mechanism of Yellowing: Carbonyl Formation during Hygrothermal Aging in a Common Amine Epoxy. Polymers 2018, 10, 1017–1031, doi:10.3390/polym10091017

Weak Points: Carbonyl Formation



Result: Formation of carbonyl groups resulted in yellowing of the epoxy.

Krauklis, A.; Echtermeyer, A. Mechanism of Yellowing: Carbonyl Formation during Hygrothermal Aging in a Common Amine Epoxy. Polymers 2018, 10, 1017–1031, doi:10.3390/polym10091017

Chemical degradation

No significant chemical degradation.

Properties should be affected by swelling

Mechanical property changes should be reversible

Static Stress-Strain Curve

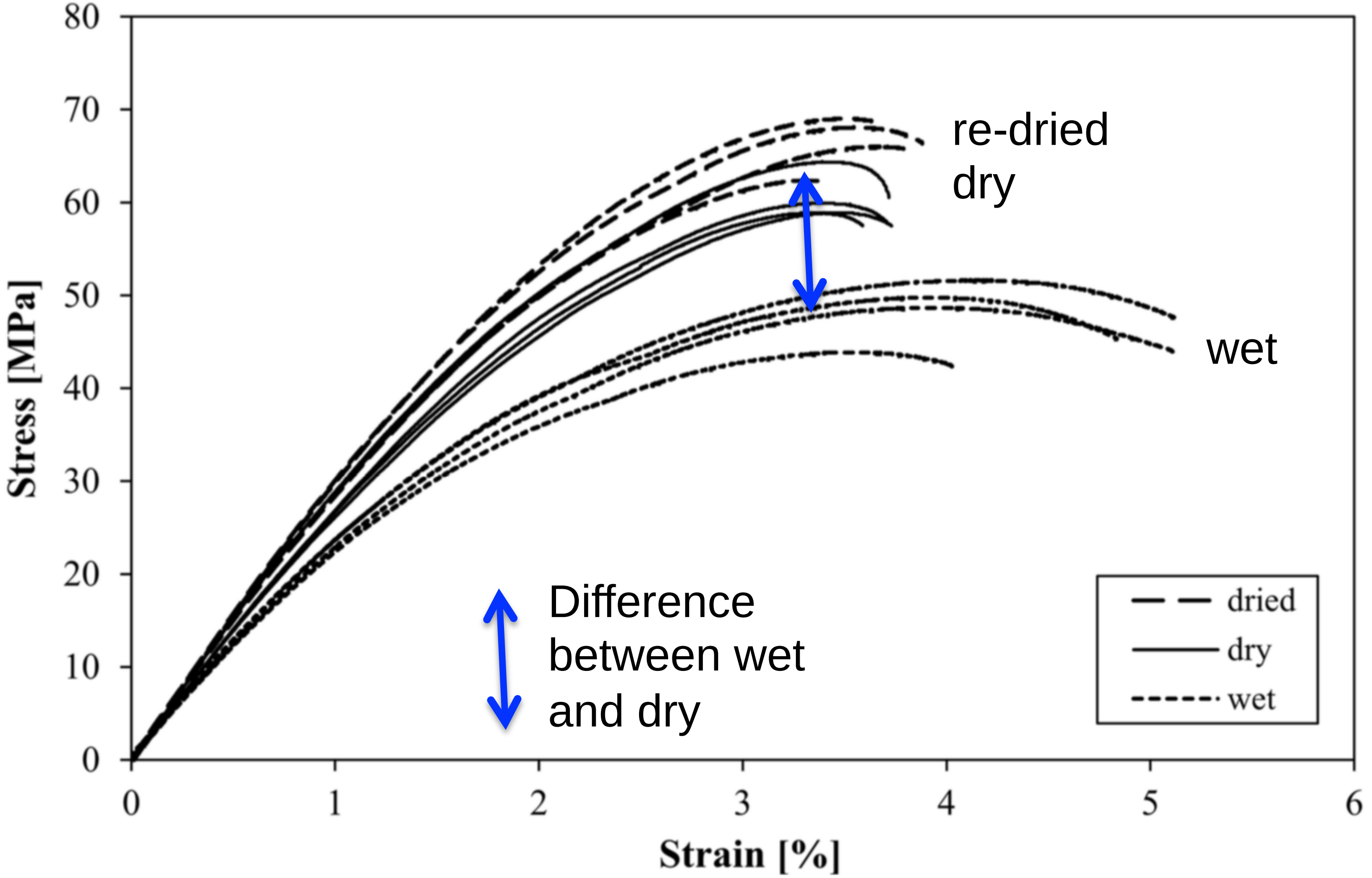
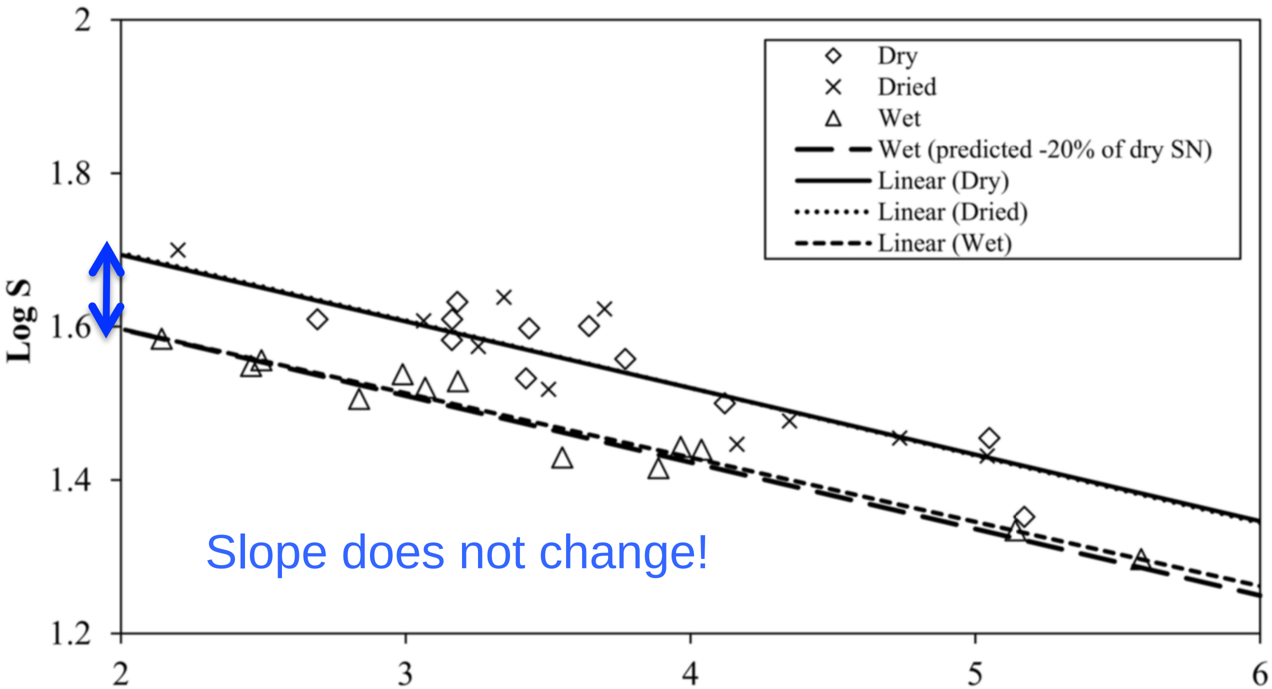


Figure 3: Stress-strain curves of dry, conditioned and dried epoxy specimens.

Krauklis, A.E.; Gagani, A.I.; Echtermeyer, A.T. Hygrothermal Aging of Amine Epoxy: Reversible Static and Fatigue Properties. Open Eng. 2018, 8, 447–454, doi:10.1515/eng-2018-0050

SN curves of the epoxy



Reversible changes. Slope remains the same.
Ideally: measure one SN curve, shift the curve based on static strength changes.

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Krauklis, A.E.; Gagani, A.I.; Echtermeyer, A.T. Hygrothermal Aging of Amine Epoxy: Reversible Static and Fatigue Properties. Open Eng. 2018, 8, 447–454, doi:10.1515/eng-2018-0050

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Constituents:

- Matrix
- Fibers
- Interphase

Composite:

- Matrix dominated
- Fiber dominated
- Interlaminar Shear

Hygrothermal Aging of R glass

Experimental:

Fiber bundles in 60 ± 1 °C water bath.

Samples in closed vessels with distilled water.

High resolution inductively coupled plasma mass spectrometry (HR-ICP-MS) to determine dissolution kinetics.

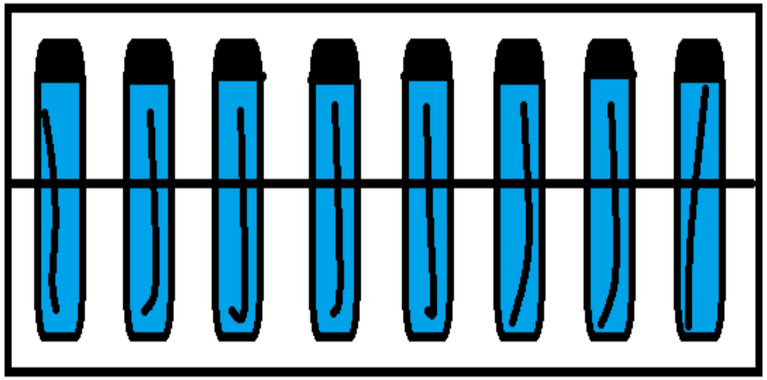
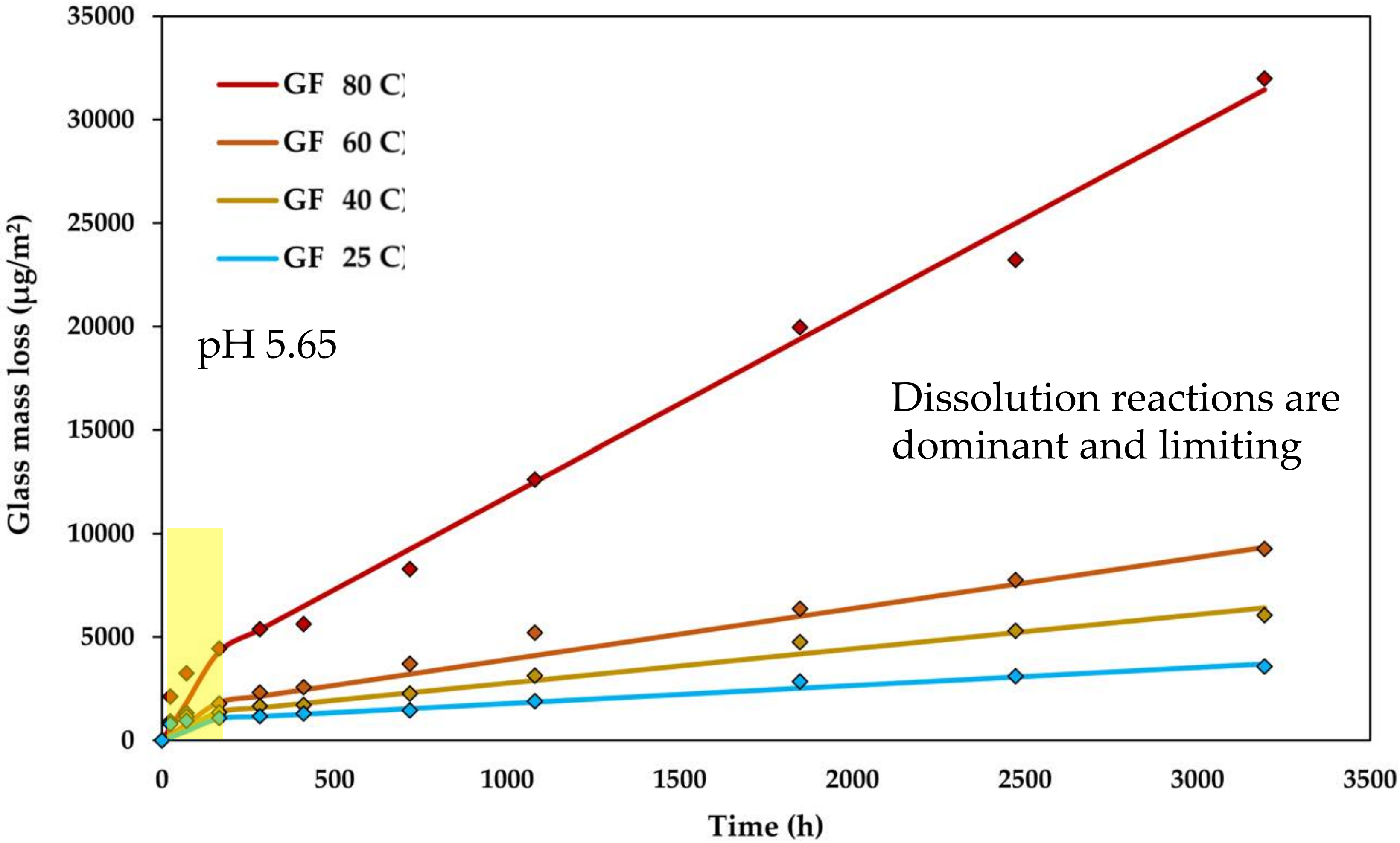


Figure: Water conditioning of glas fiber bundles and composite plates

Effect of Temperature – Arrhenius



pH 5.65

Dissolution reactions are dominant and limiting

Krauklis, A.E.; Gagani, A.I.; Vegere, K.; Kalnina, I.; Klavins, M.; Echtermeyer, A.T. Dissolution Kinetics of R-Glass Fibres: Influence of Water Acidity, Temperature and Stress Corrosion. Fibers 2019, 7(3), 22-40, doi:10.3390/fib7030022

Sized Glass Fibers in Water

Short-term non-steady state:

- Ion exchange reactions
- Gel formation
- Dissolution reactions
- Other mineral phase formation

Process is very complex (on the surface) and ion release is not linear with time. It is possible sizing also plays a role in this region.

Long-term steady state:

Dissolution reactions become dominant and limiting.

Chemical Reactions for Glass

$$(\equiv Si - ONa) + H_2O \rightarrow (\equiv Si - OH) + OH^- + Na^+ \tag{2}$$

$$(\equiv Si - OK) + H_2O \rightarrow (\equiv Si - OH) + OH^- + K^+ \tag{3}$$

$$(\equiv Si - O)_2Ca + H_2O \rightarrow 2(\equiv Si - OH) + 2OH^- + Ca^{2+} \tag{4}$$

$$(\equiv Si - O)_2Mg + H_2O \rightarrow 2(\equiv Si - OH) + 2OH^- + Mg^{2+} \tag{5}$$

$$(\equiv Si - O - Al =) + H_2O \leftrightarrow (\equiv Si - OH) + (= Al - OH) \tag{6}$$

$$(\equiv Si - O)_2Fe + H_2O \rightarrow 2(\equiv Si - OH) + 2OH^- + Fe^{2+} \tag{7}$$

$$(\equiv Si - O)_3Fe + H_2O \rightarrow 3(\equiv Si - OH) + 3OH^- + Fe^{3+} \tag{8}$$

$$(\equiv Si - O - Si \equiv) + OH^- \rightarrow (\equiv Si - OH) + (\equiv Si - O^-) \tag{9}$$

$$(\equiv Si - O^-) + H_2O \rightarrow (\equiv Si - OH) + OH^- \tag{10}$$

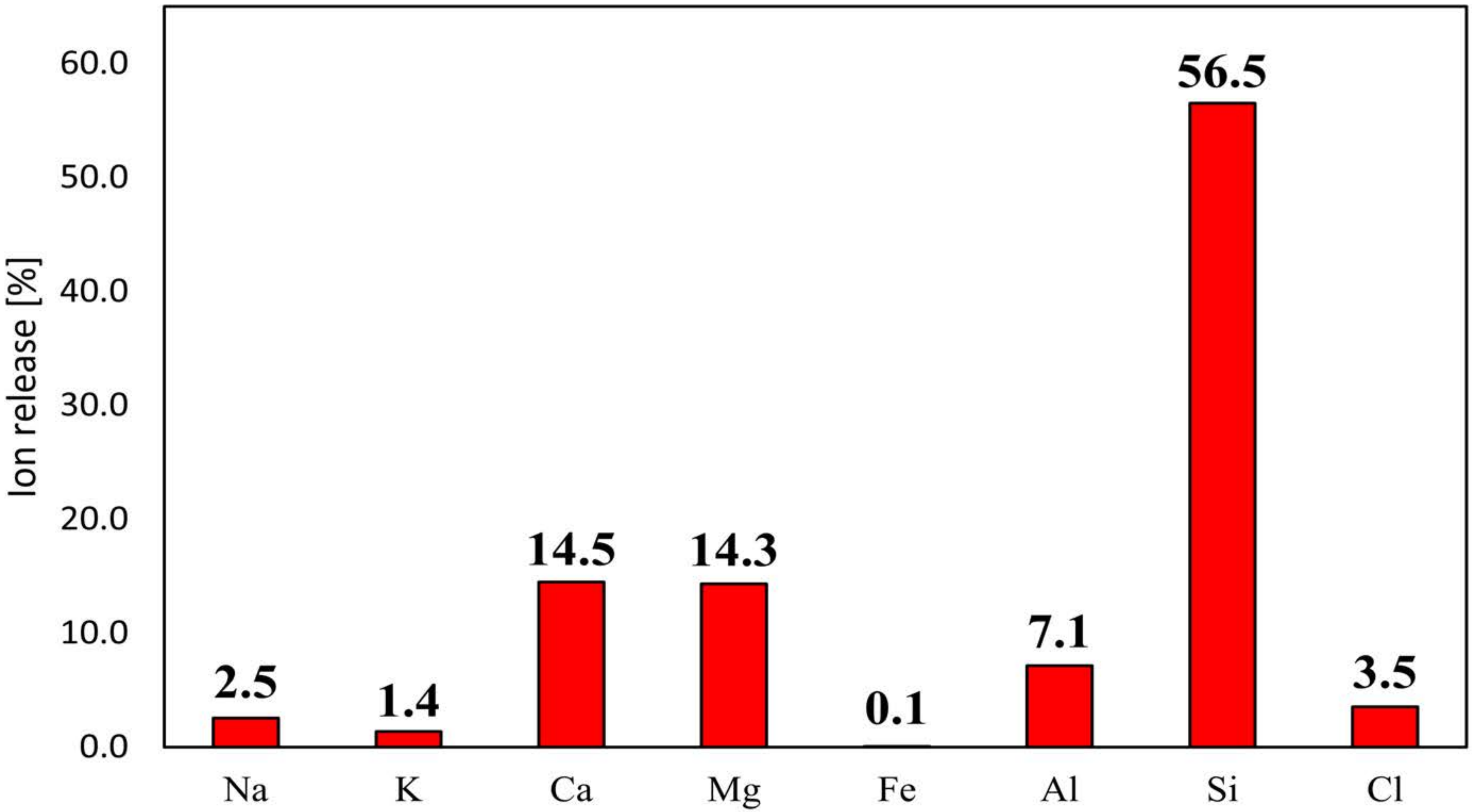
$$(\equiv Si - O - Si \equiv) + H_2O \leftrightarrow 2(\equiv Si - OH) \tag{11}$$

$$SiO_2 + 2H_2O \rightarrow H_4SiO_4 \tag{12}$$

$$MeCl_x \xrightarrow{H_2O} (Me^{x+}) + xCl^- \tag{13}$$

Krauklis, A.E.; Gagani, A.I.; Vegere, K.; Kalnina, I.; Klavins, M.; Echtermeyer, A.T. Dissolution Kinetics of R-Glass Fibres: Influence of Water Acidity, Temperature and Stress Corrosion. Fibers 2019, 7(3), 22-40, doi:10.3390/fib7030022

Ions identified: ion release rates



Krauklis, A.E.; Echtermeyer, A.T. Long-Term Dissolution of Glass Fibers in Water Described by Dissolving Cylinder Zero-Order Kinetic Model: Mass Loss and Radius Reduction. Open Chem. 2018, 16(1), 1189-1199, doi:10.1515/chem-2018-0133

Dissolution Kinetics

First order equation $\frac{\partial m}{\partial t} = K_0 S$ & consider that:

- Sizing slows down the reaction rate (add a factor), m and S are correlated by geometry

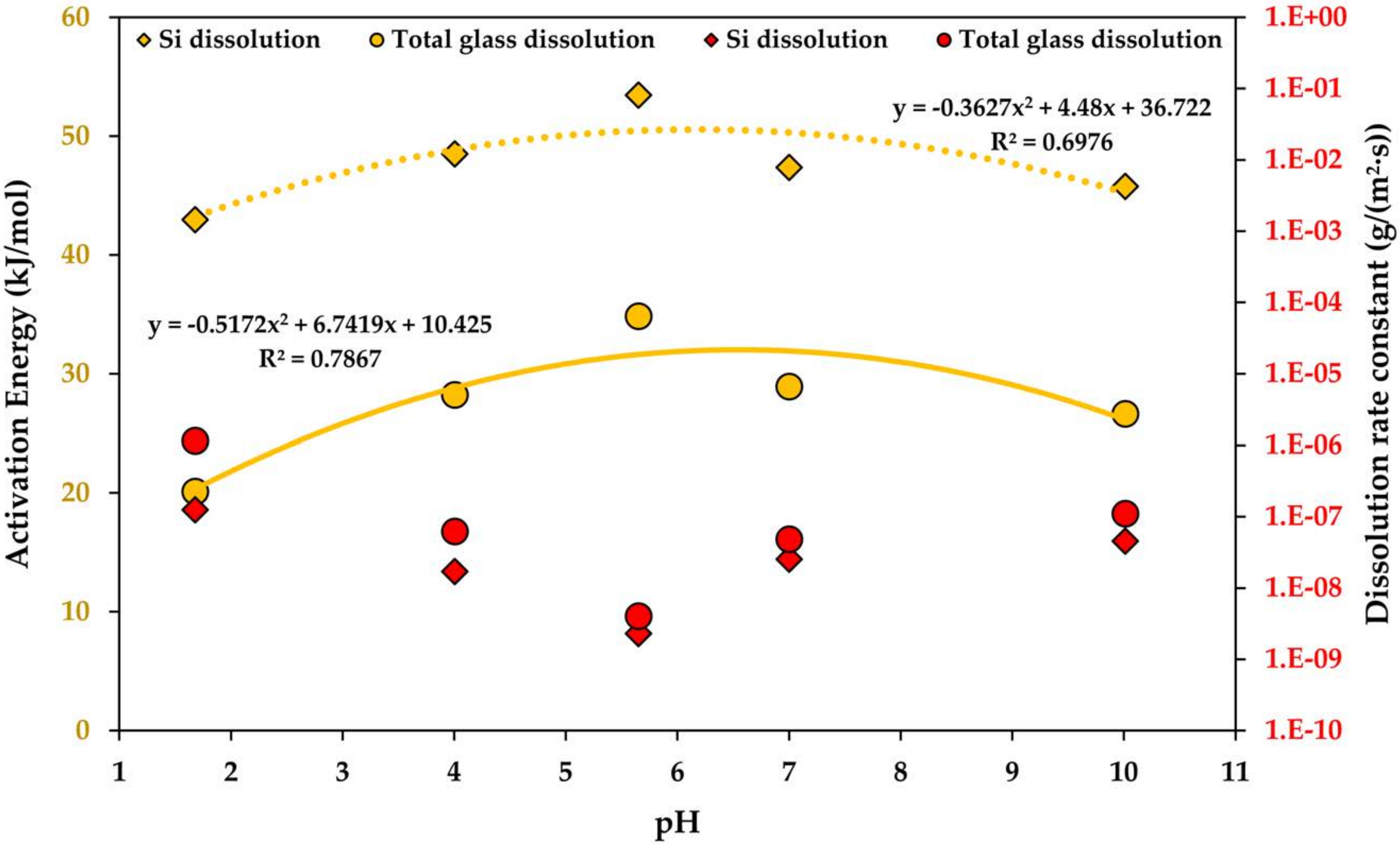
$$\frac{\partial m}{\partial t} = 2n\pi l\left(r_0 K_0 \xi_{sizing} - \frac{K_0^2 \xi_{sizing}^2}{\rho_{glass}} t\right)$$

- pH, temperature and stress corrosion affect the dissolution rate constants: $K_0 = Ae^{-\frac{E_A(pH,\sigma)}{RT}}$

Final equation:

$$\frac{\partial m}{\partial t} = 2n\pi l\left(r_0 K_0 \xi_{sizing} - \frac{(K_0 \xi_{sizing})^2}{\rho_{glass}} t\right) = 2n\pi l\left(r_0 Ae^{-\frac{E_A(pH,\sigma)}{RT}} \xi_{sizing} - \frac{\left(Ae^{-\frac{E_A(pH,\sigma)}{RT}} \xi_{sizing}\right)^2}{\rho_{glass}} t\right)$$

Effect of Acidity

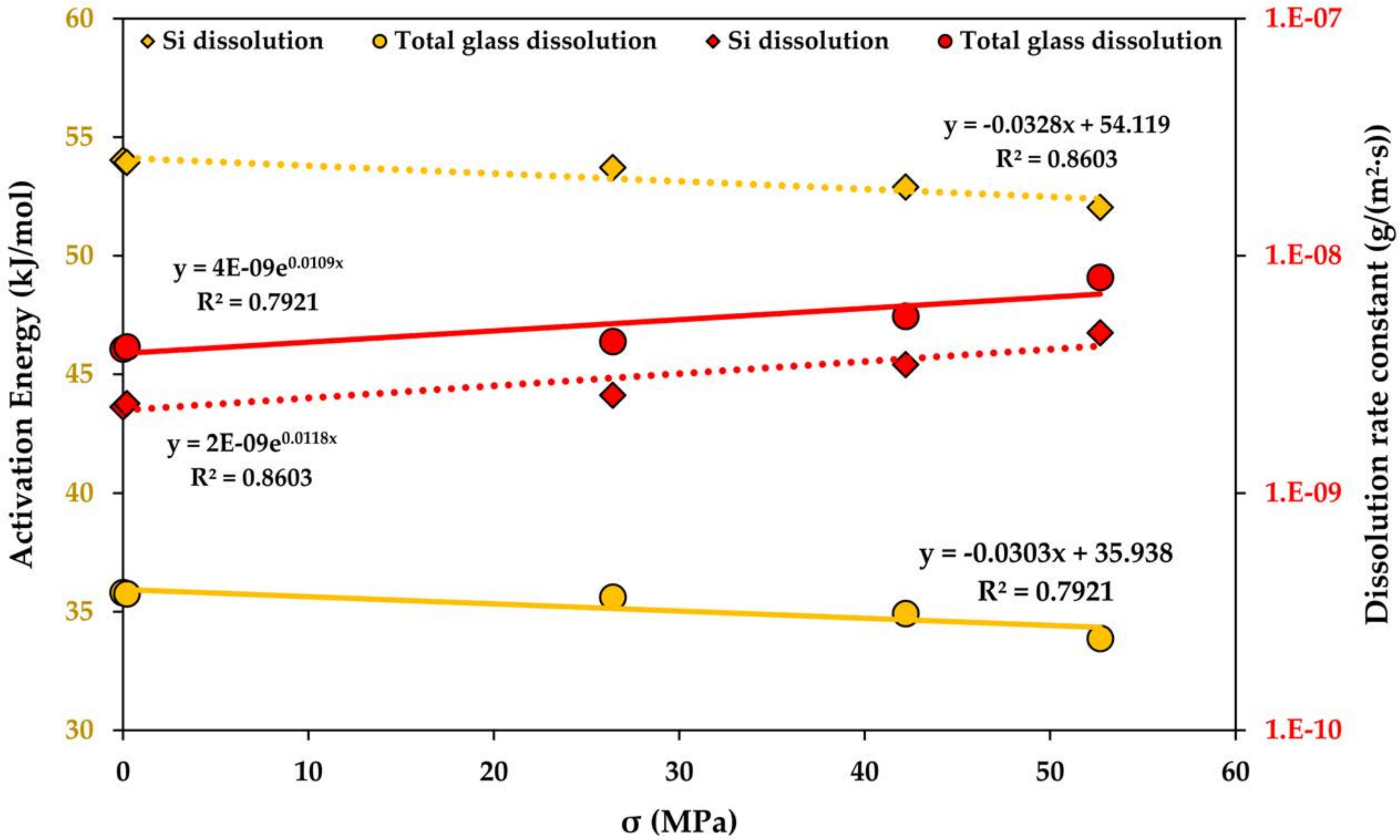


Krauklis, A.E.; Gagani, A.I.; Vegere, K.; Kalnina, I.; Klavins, M.; Echtermeyer, A.T. Dissolution Kinetics of R-Glass Fibres: Influence of Water Acidity, Temperature and Stress Corrosion. Fibers 2019, 7(3), 22-40, doi:10.3390/fib7030022

Effect of Stress

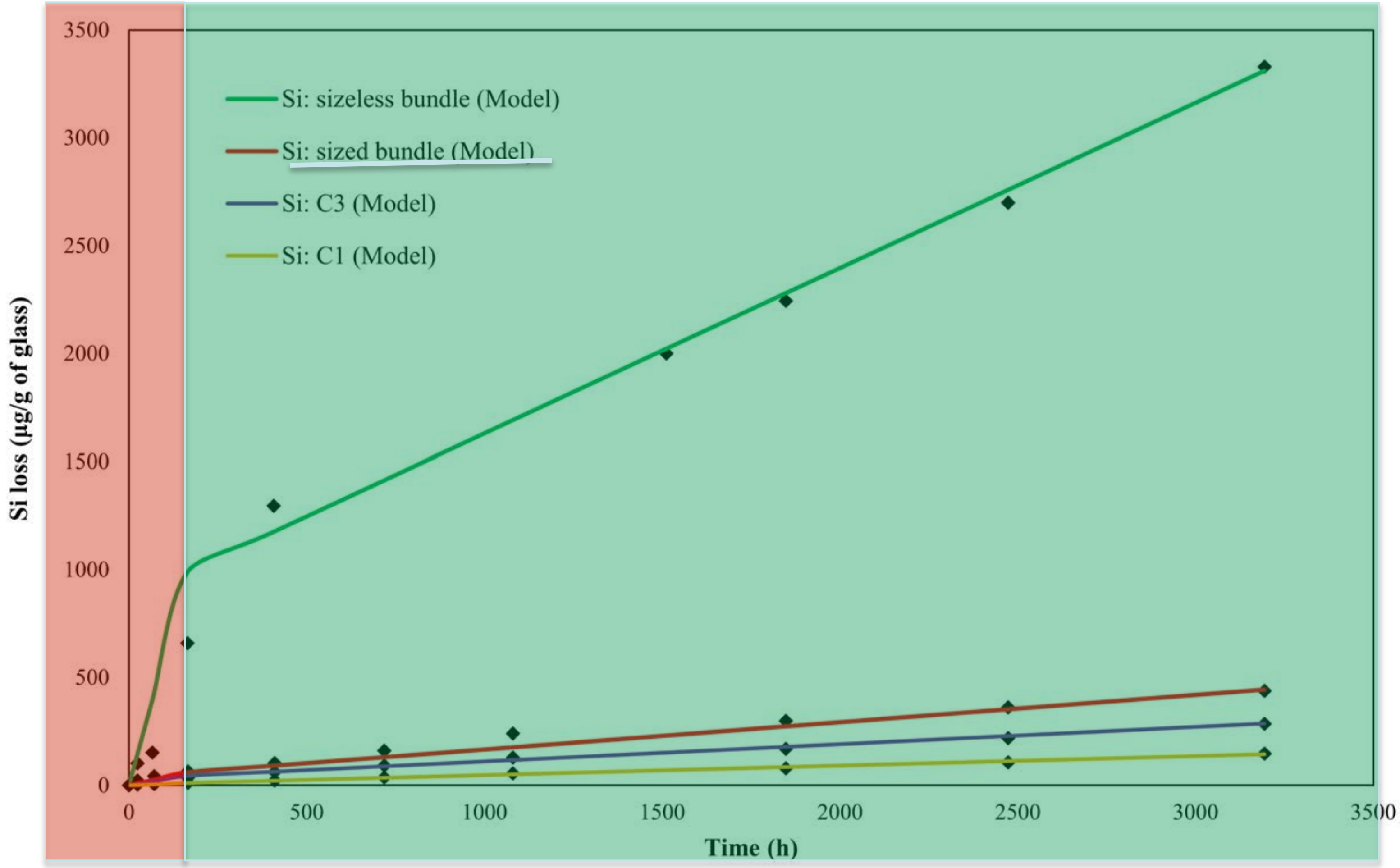
Krauklis, A.E.; Gagani, A.I.; Vegere, K.; Kalnina, I.; Klavins, M.; Echtermeyer, A.T. Dissolution Kinetics of R-Glass Fibres: Influence of Water Acidity, Temperature and Stress Corrosion. Fibers 2019, 7(3), 22-40, in a Special Issue: Advances in Glass Fibers, doi:10.3390/fib7030022

Effect of Stress



Krauklis, A.E.; Gagani, A.I.; Vegere, K.; Kalnina, I.; Klavins, M.; Echtermeyer, A.T. Dissolution Kinetics of R-Glass Fibres: Influence of Water Acidity, Temperature and Stress Corrosion. Fibers 2019, 7(3), 22-40, doi:10.3390/fib7030022

Dissolution – Sized and bare



Short-term non-steady state

Long-term steady state

Krauklis, A.E.; Echtermeyer, A.T. Dissolving Cylinder Zero-Order Kinetic Model for Predicting Hygrothermal Aging of Glass Fiber Bundles and Fiber-Reinforced Composites. In 4th International Glass Fiber Symposium; Gries, Th.; Pico, D.; Lüking, A.; Becker, Th., Eds.; Mainz, G (Verlag): Aachen, Germany, 2018; pp. 66–72, ISBN:978-3-95886-249-4.

Glass fiber chemical degradation

An analytical model is developed that predicts long-term dissolution of sized glass from fiber bundles at various pH, T and stress.

The sizing protects glass fibers from dissolution by almost 6 times.

Linking mass loss to strength loss is in progress.

Effects are slowed down further inside a composite ply.

CONTENTS

Constituents:

- Matrix
- Fibers
- Interphase

Composite:

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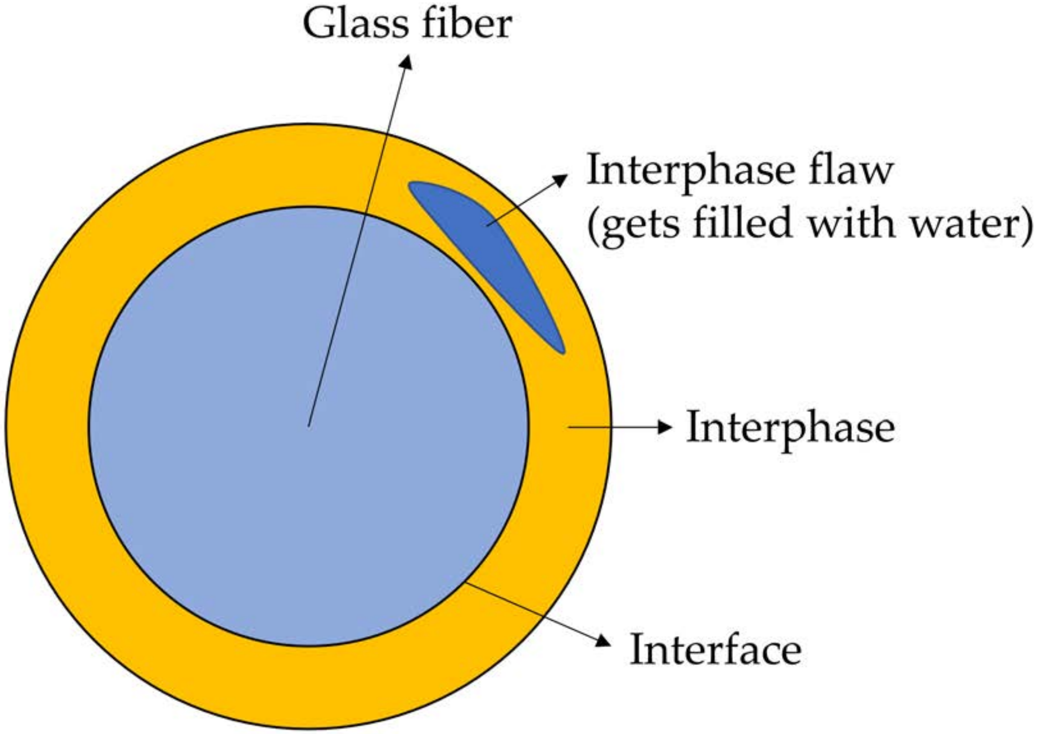
Hydrolysis of the Interphase



Krauklis, A.E.; Gagani, A.I.; Echtermeyer, A.T. Long-Term Hydrolytic Degradation of the Sizing-Rich Composite Interphase. Coatings 2019, 9(4), 263-286, doi:10.3390/coatings9040263

Experimental Program for Diffusion testing

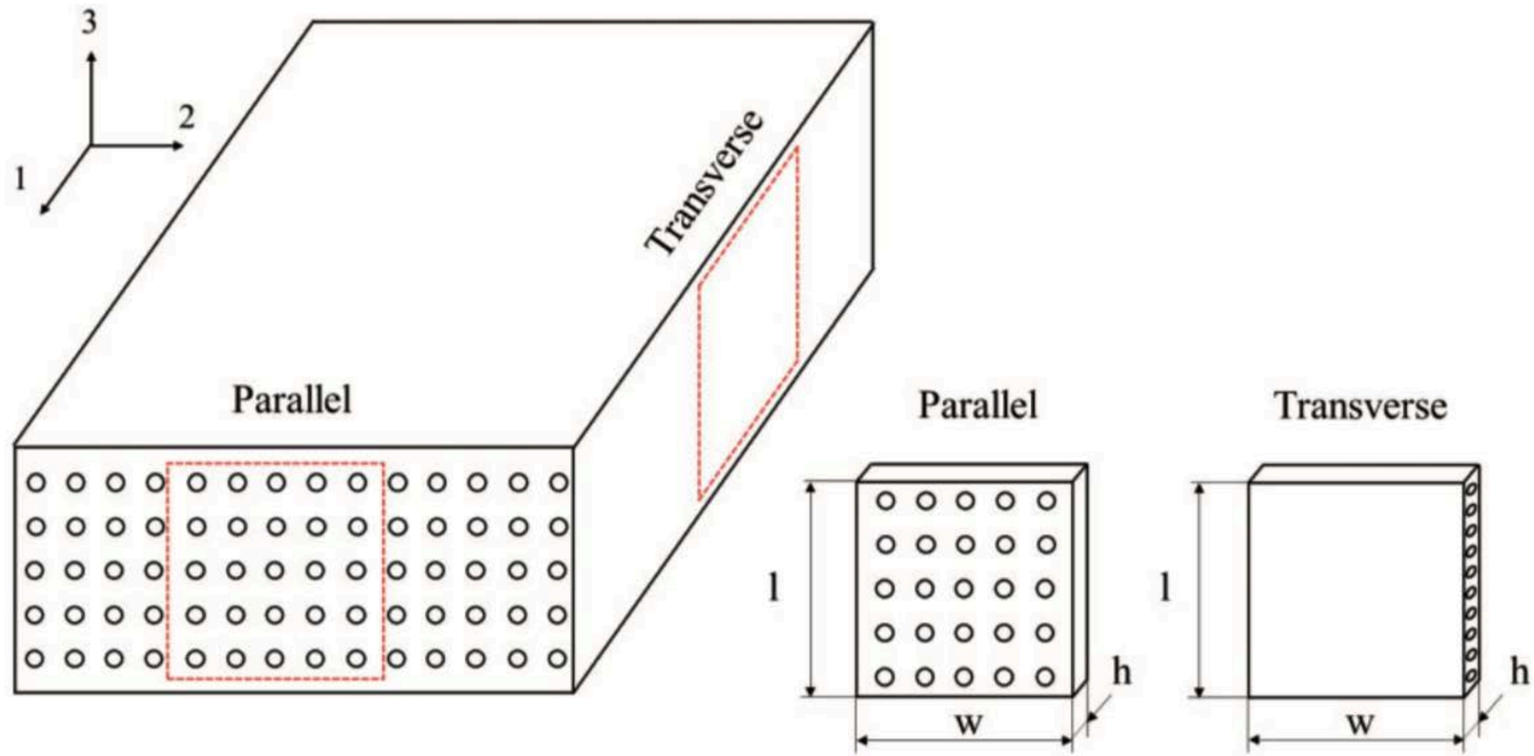
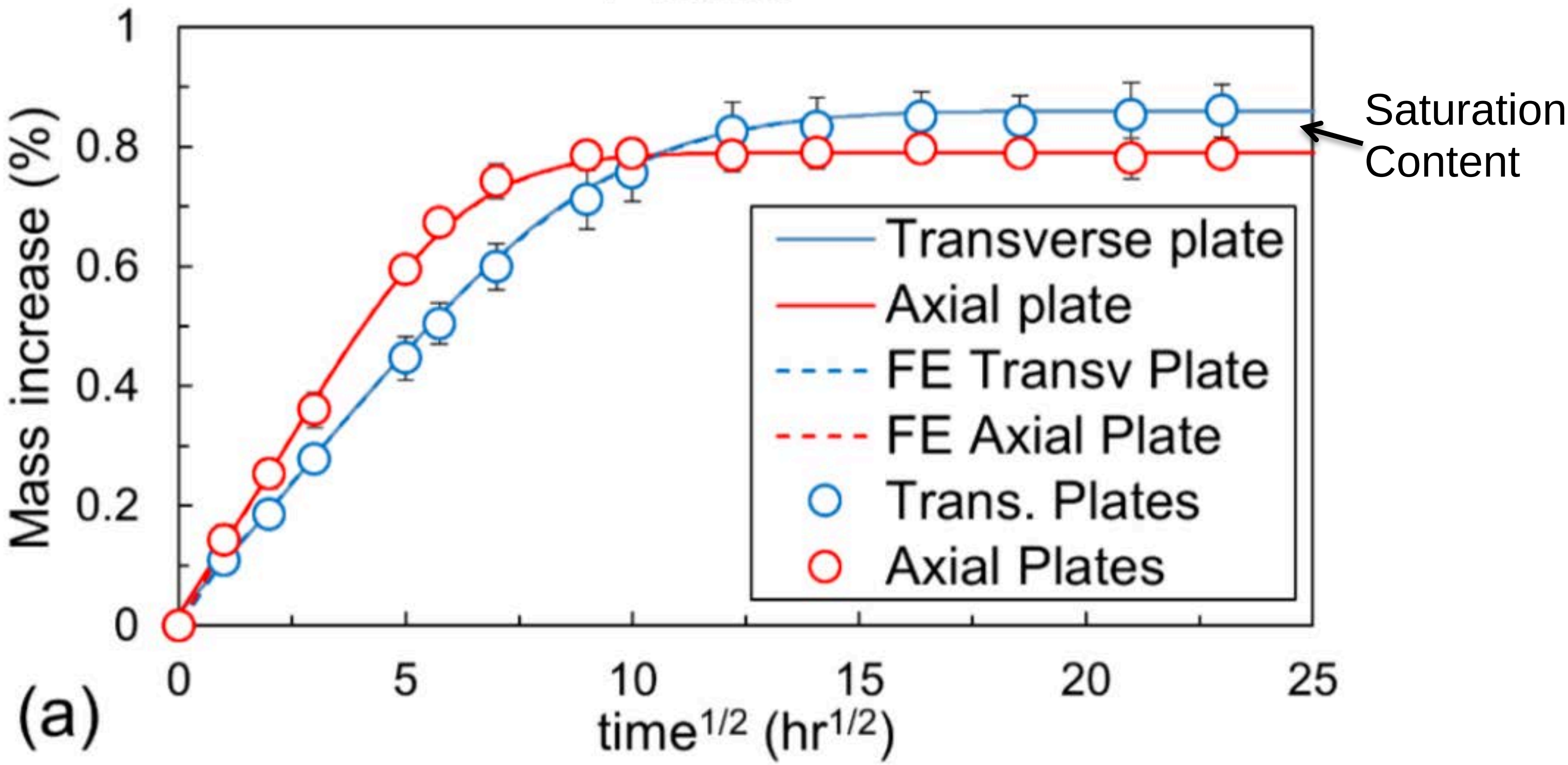


Figure 1. Composite samples configurations. Dimensions are 50 mm × 50 mm × 1.5 mm.

Gagani, Abedin; Fan, Yiming; Muliana, Anastasia H; Echtermeyer, Andreas, Micromechanical modelling of anisotropic water diffusion in glass fiber epoxy reinforced composites. Journal of composite materials 2018

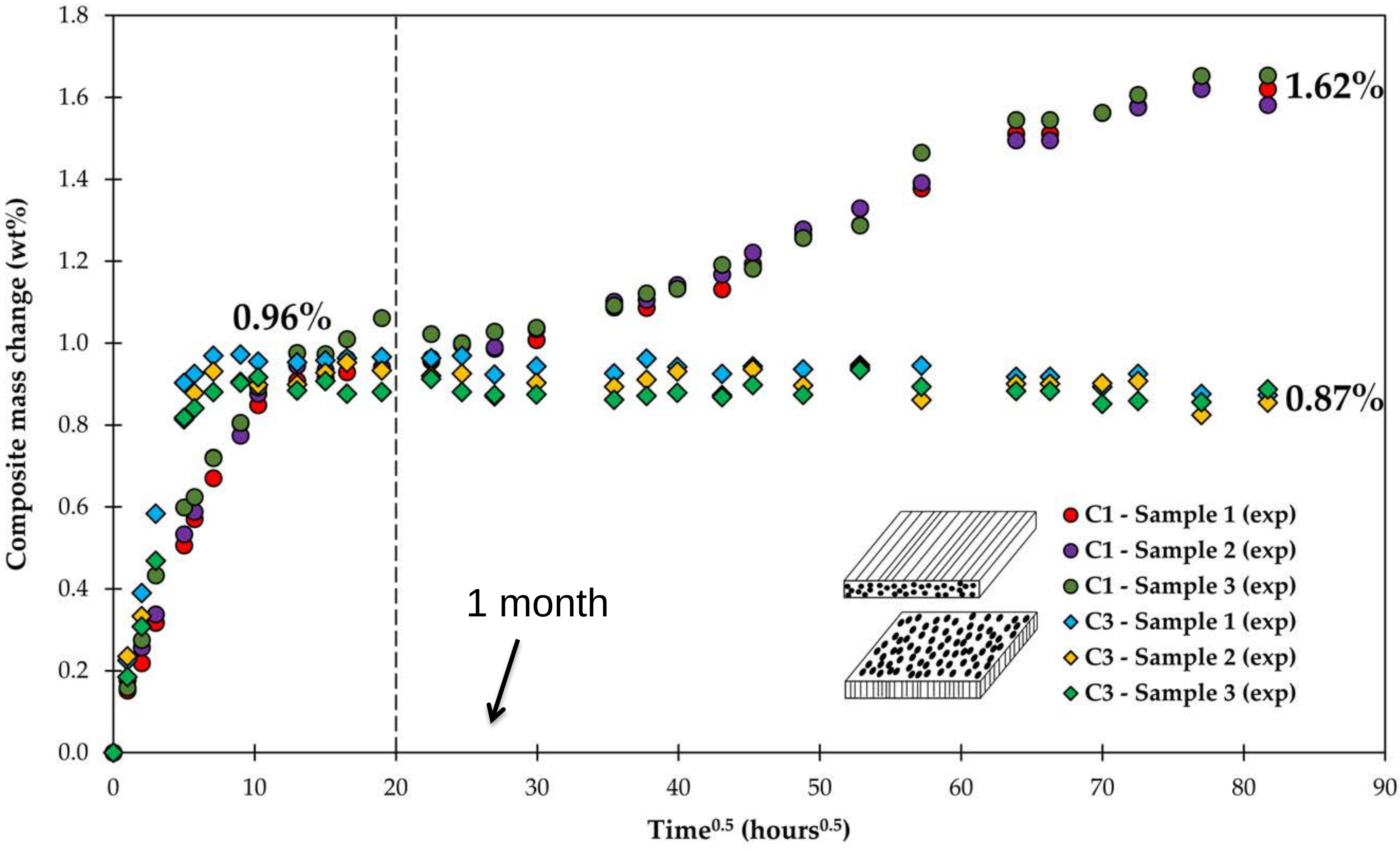
Diffusivity Measurements



$$M(t) = M_{eq}\left[1-\left(\frac{8}{\pi^2}\right)^3 \sum_{i=1}^{\infty}\sum_{j=1}^{\infty}\sum_{k=1}^{\infty}\frac{1}{(2i-1)^2}e^{-(2i-1)^2\left(\frac{\pi}{l}\right)^2 D_{\perp}t}\cdot\frac{1}{(2j-1)^2}e^{-(2j-1)^2\left(\frac{\pi}{w}\right)^2 D_{\perp}t}\cdot\frac{1}{(2k-1)^2}e^{-(2k-1)^2\left(\frac{\pi}{h}\right)^2 D_{\parallel}t}\right] \quad (18)$$

Gagani, Abedin; Krauklis, Andrey; Echtermeyer, Andreas, (2018) Orthotropic fluid diffusion in composite marine structures. Experimental procedure, analytical and numerical modelling of plates, rods and pipes. Composites Part A: Applied Science and Manufacturing, Vol. 115, pp. 196-205

Gravimetric data and mass balance



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Krauklis, A.E.; Gagani, A.I.; Echtermeyer, A.T. Long-Term Hydrolytic Degradation of the Sizing-Rich Composite Interphase. Coatings 2019, 9(4), 263-286, doi:10.3390/coatings9040263

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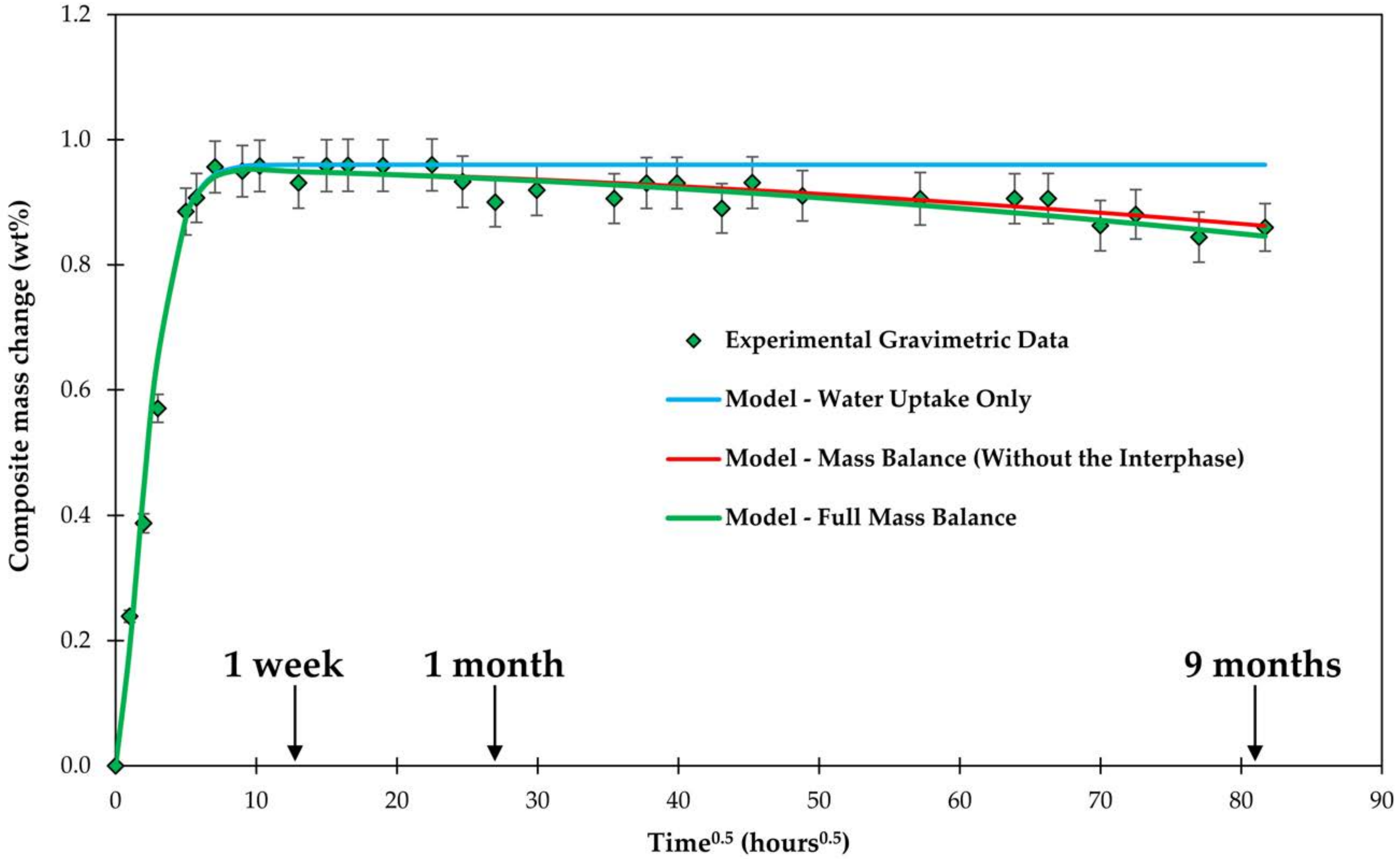

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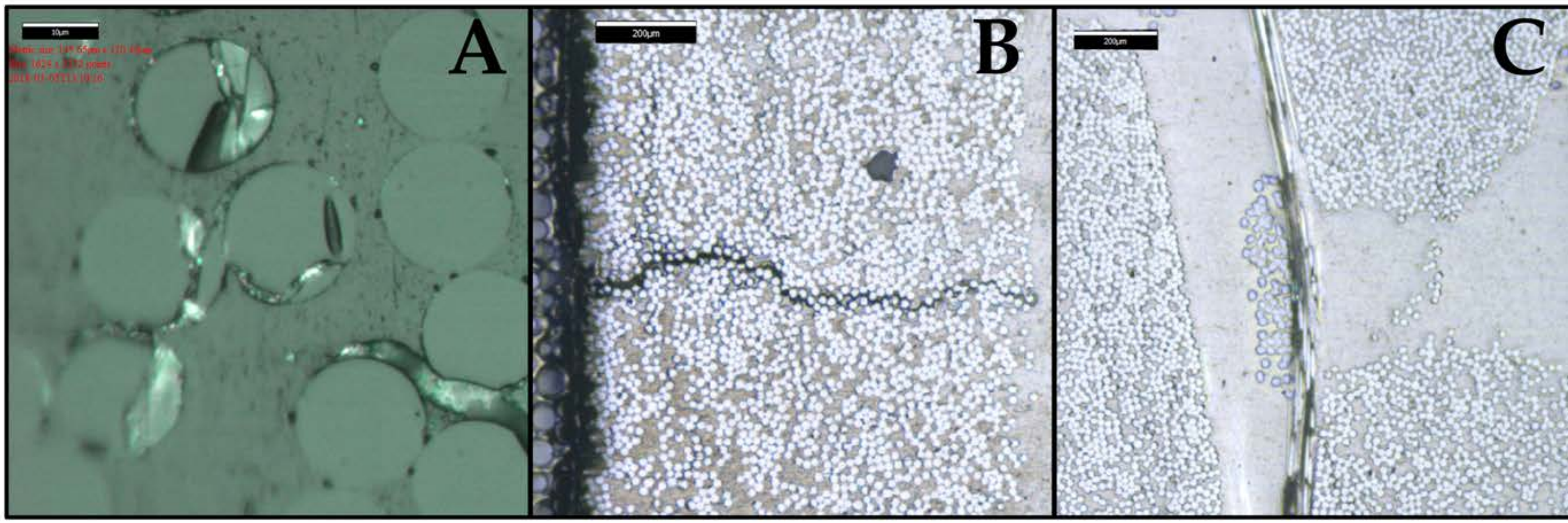
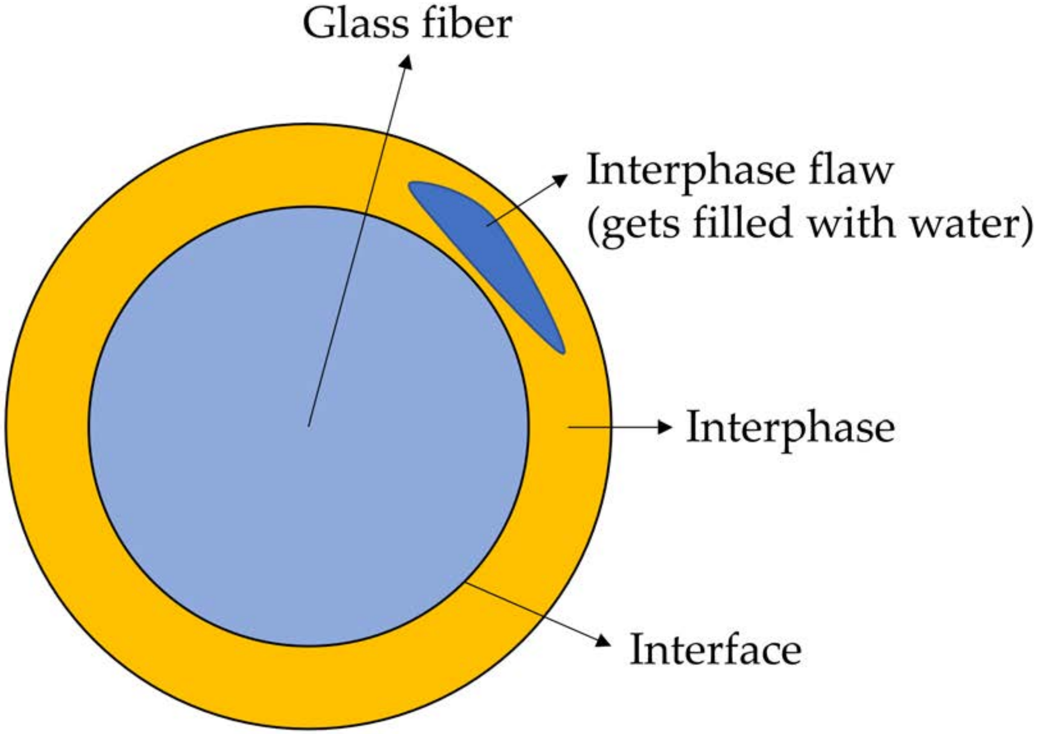
Hydrolysis of the Interphase



Micrograph of a composite sample exposed to water for 6673 h at 60 °C. The micrograph indicates the (**A**) fiber/matrix debondings; (**B**) matrix transverse cracks; (**C**) splitting along the fibers.

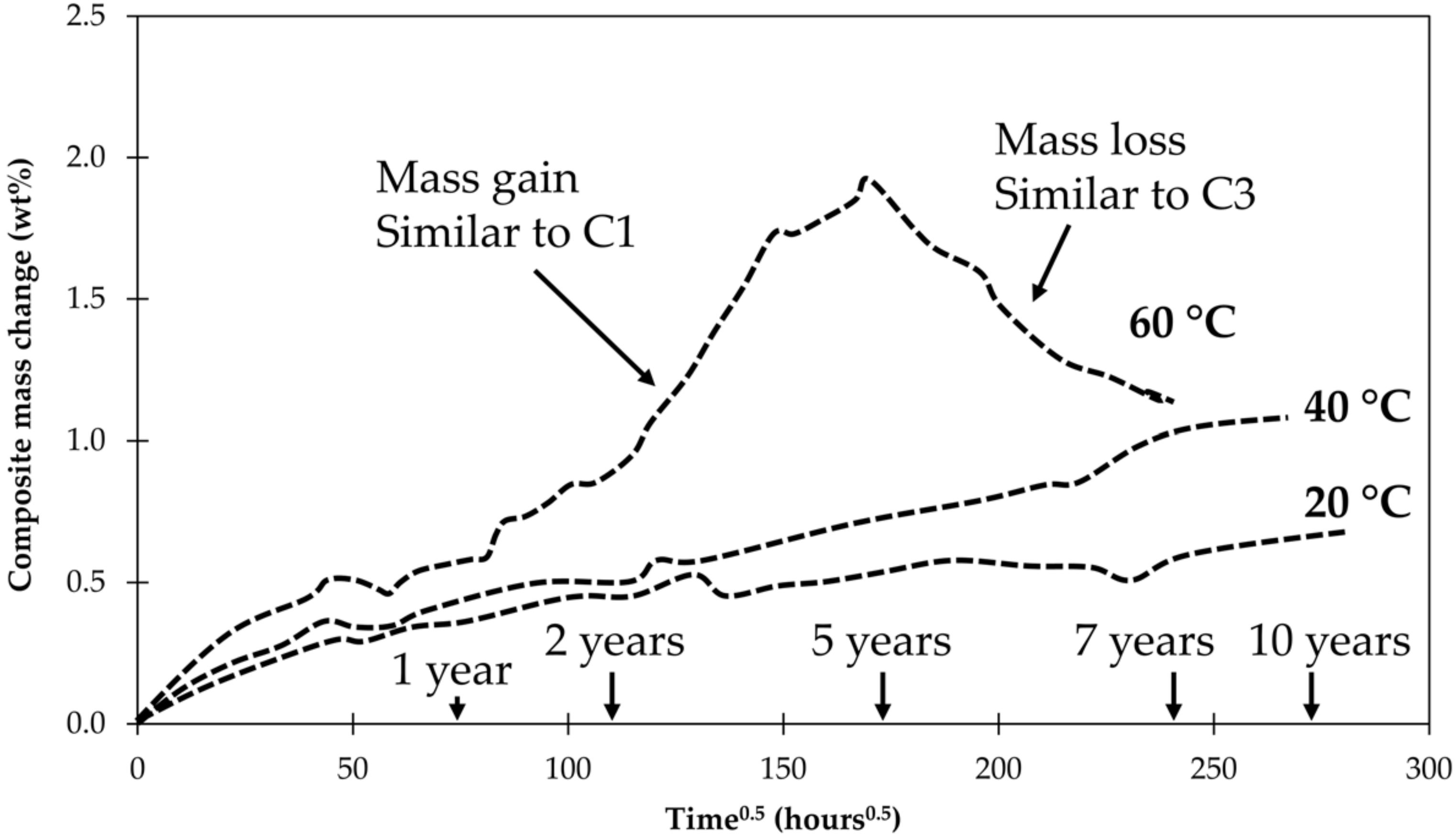
Krauklis, A.E.; Gagani, A.I.; Echtermeyer, A.T. Long-Term Hydrolytic Degradation of the Sizing-Rich Composite Interphase. Coatings 2019, 9(4), 263-286, doi:10.3390/coatings9040263

Hydrolysis of the Interphase



Krauklis, A.E.; Gagani, A.I.; Echtermeyer, A.T. Long-Term Hydrolytic Degradation of the Sizing-Rich Composite Interphase. Coatings 2019, 9(4), 263-286, doi:10.3390/coatings9040263

Long-Term Water Uptake



Data from Perreux, D.; Choqueuse, D.; Davies, P. Anomalies in moisture absorption of glass fibre reinforced epoxy tubes. Compos. Part A 2002, 33, 147–154.

Krauklis, A.E.; Gagani, A.I.; Echtermeyer, A.T. Long-Term Hydrolytic Degradation of the Sizing-Rich Composite Interphase. Coatings 2019, 9(4), 263-286, doi:10.3390/coatings9040263

Hydrolysis Kinetics of Interphase (speculative)

Table 4. Systematized scenarios of the interphase dissolution kinetics.

	$K_i^0 S_i^0$ (g/h)	Sizing coverage (%)	δ_i (nm)	S_i^0 (m²)	K_i^0 (g/(m²·h))	Time to total dissolution (years)
Scenario 1	$1.80 \cdot 10^{-7}$	100	65	$4.43 \cdot 10^{-5}$	$4.06 \cdot 10^{-3}$	22.7
Scenario 2	$1.80 \cdot 10^{-7}$	100	65	1.01	$1.78 \cdot 10^{-7}$	30.5
Scenario 3	$1.80 \cdot 10^{-7}$	90, after [39]	72	0.91	$1.98 \cdot 10^{-7}$	30.5

Krauklis, A.E.; Gagani, A.I.; Echtermeyer, A.T. Long-Term Hydrolytic Degradation of the Sizing-Rich Composite Interphase. Coatings 2019, 9(4), 263-286, doi:10.3390/coatings9040263

Interphase Results

Fiber-matrix interphase degradation was observed after the matrix was fully saturated with water and typical water absorption tests according to ASTM D5229 were stopped.

Thus, ASTM D5229 should involve longer water uptake times.

Due to water-induced dissolution or swelling stresses, fiber-matrix interphase flaws were formed, which then lead to increased water uptake.

When a interpenetrated network of flaws is formed, a mass loss occurs.

<u>Speculatively</u>, for the SMALL composite laminate samples studied here with a saturated epoxy matrix, the fiber matrix interphase is predicted to be fully degraded after 22 to 30 years.

CONTENTS

Constituents:

- Matrix
- Fibers
- Interphase

Composite Ply Properties:

- Matrix dominated
- Fiber dominated
- Interlaminar Shear

Matrix dominated ply properties

Matrix dominated properties (Matrix cracking) was not investigated in this project, since most composites used offshore for pipes and pressure vessels have liners and matrix cracking is "acceptable".

For other applications matrix cracking may be critical.

Fiber dominated ply properties

Sized fiber bundles loose mass 6 times slower than bare fiber bundles.

Fiber´s mass loss inside a composite was inconclusive so far, but slower. Bundle loss could be taken as a conservative value. Relative changes might remain the same?

We are working on a link between mass loss and strength loss allowing use of the kinetic models developed for mass loss.

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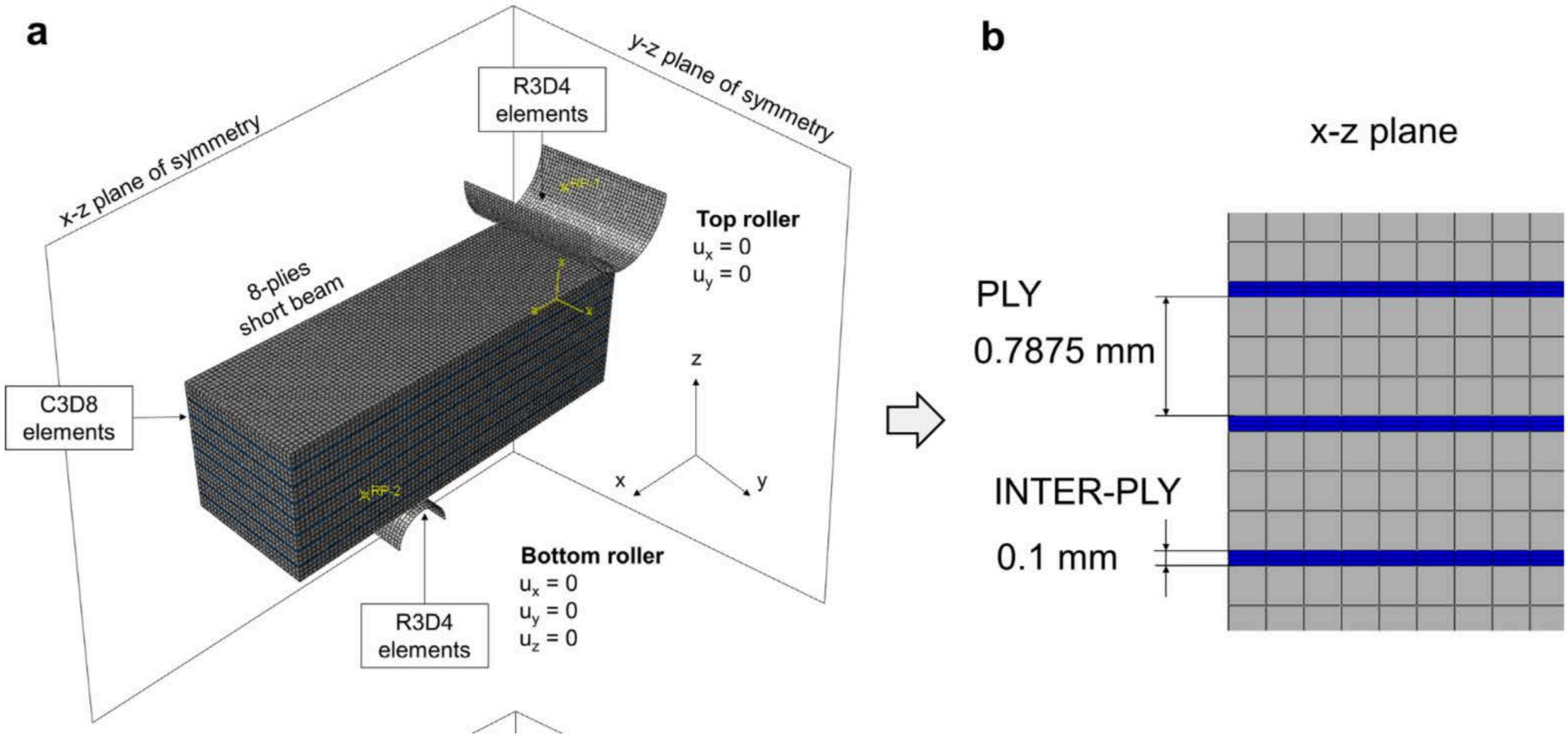
Interlaminar shear

Can this through thickness property be predicted by matrix properties?

From a practical point of view it is not sufficient, because the interface´s properties between plies is somewhat dependent on the production process, especially for thermoplastics.

Interlaminar Shear Strength

New method to analyze SBS test



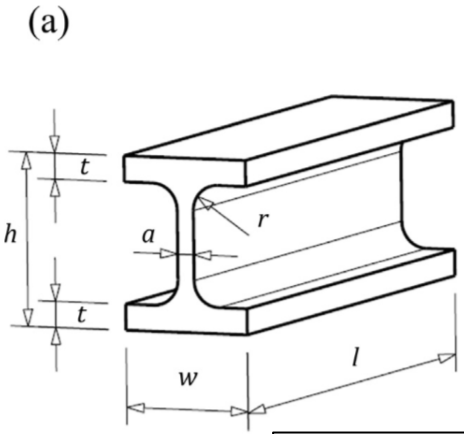
Gagani, Abedin; Krauklis, Andrey; Vedvik, Nils Petter, Echtermeyer, Andreas, (2019) A novel method for testing and determining ILSS for marine and offshore composites. Composite Structures, Vol. 220, pp. 431-440

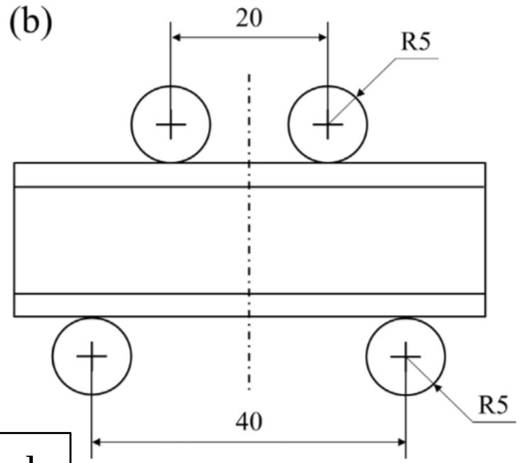
Test composite with I-beam

Replace SBS Test with I beam

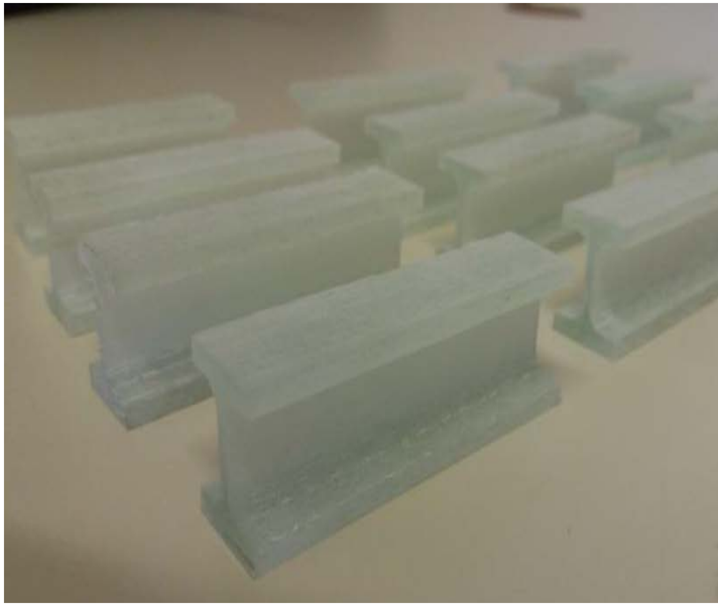
Conditioning time reduced from 8 to 2 months

Still possible to observe shear failure



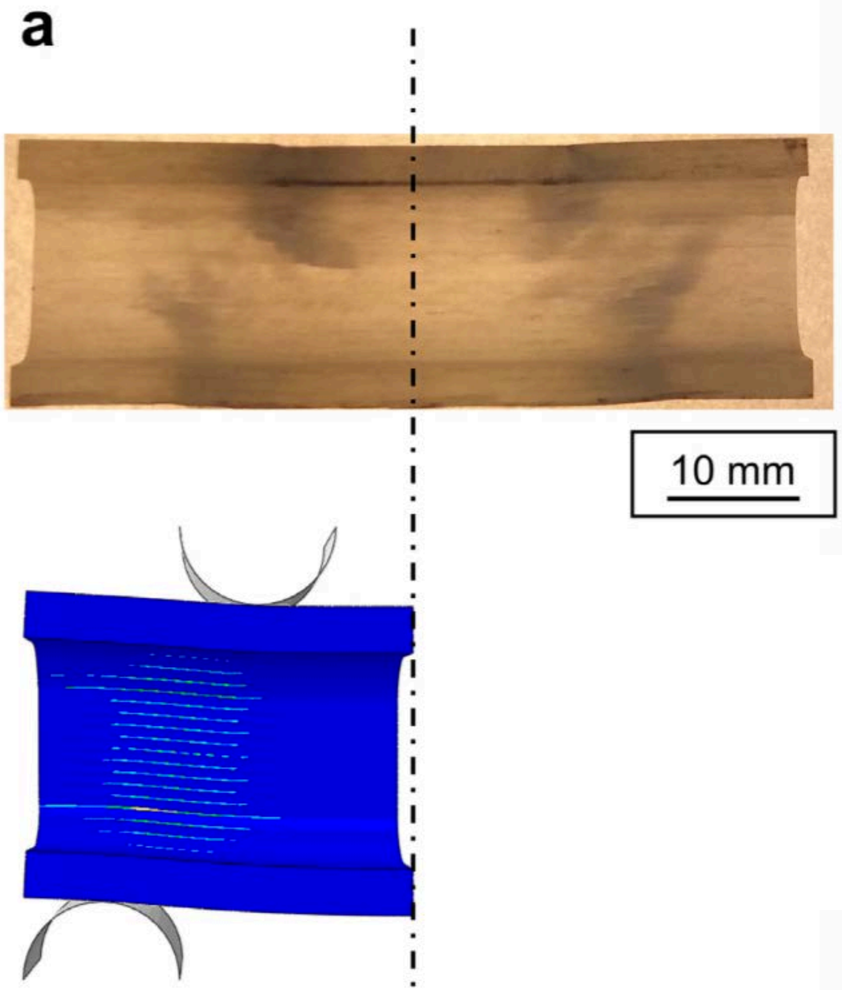


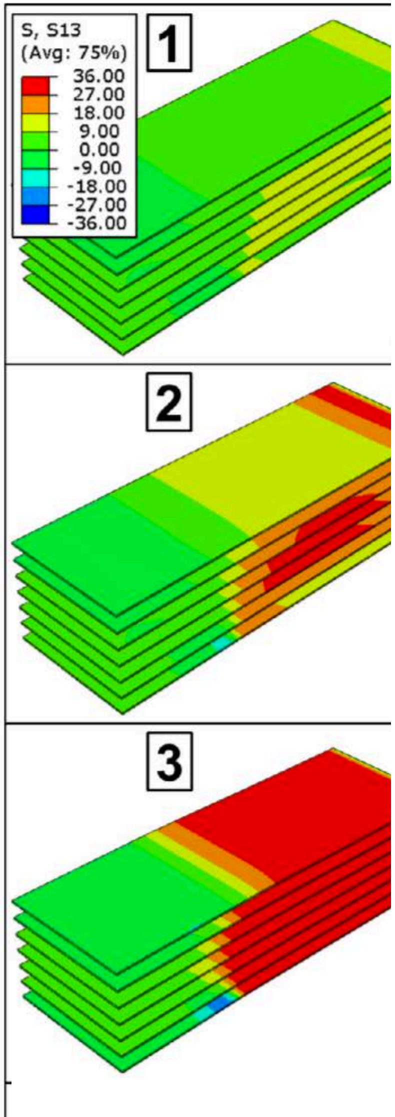
Samples obtained from 20 mm thick laminates



Gagani, Abedin; Mialon, Emeric; Echtermeyer, Andreas. (2019) Immersed interlaminar fatigue of glass fiber epoxy composites using the I-beam method. International Journal of Fatigue. vol. 119.

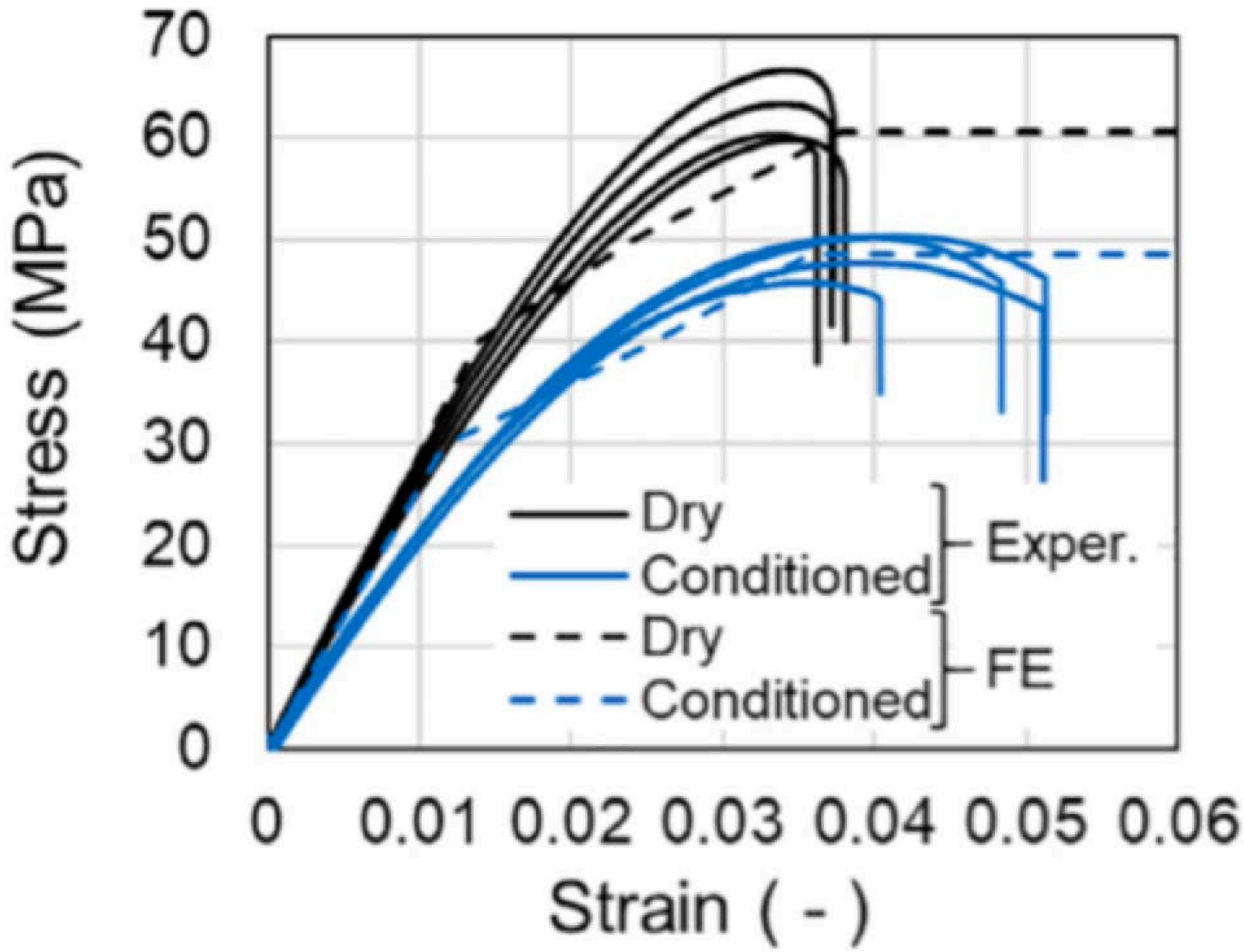
Damage development





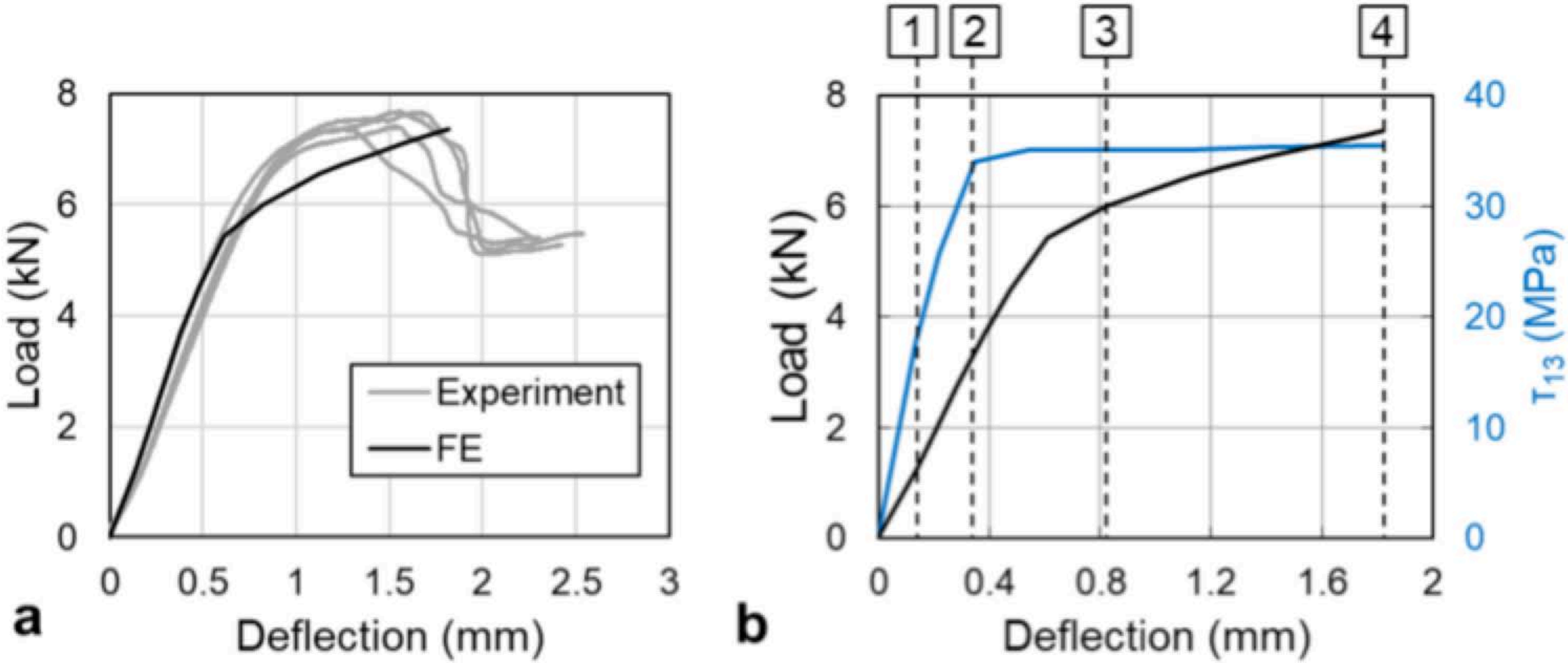
Gagani, Abedin; Krauklis, Andrey; Vedvik, Nils Petter, Echtermeyer, Andreas, (2019) A novel method for testing and determining ILSS for marine and offshore composites. Composite Structures, Vol. 220, pp. 431-440

Epoxy Material Data



Abedin I. Gagani, Andrey E. Krauklis, Erik Sæter, Nils Petter Vedvik, Andreas T. Echtermeyer
A Novel Method for Testing and Determining ILSS for Marine and Offshore Composites, Composite Structures, 220, 2019

SBS test interpretation



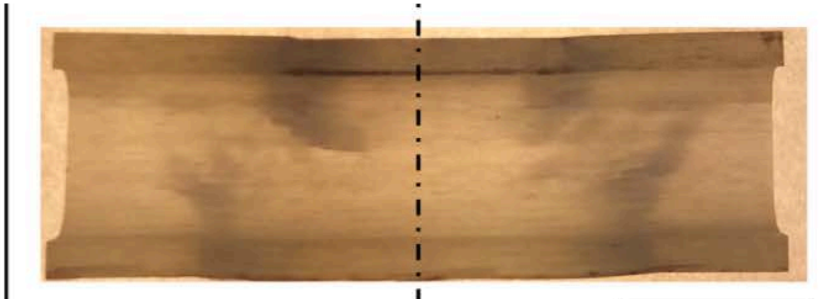
This analysis method gives the same shear properties for ILSS and matrix alone

This is the “ideal” cases for the theory development.

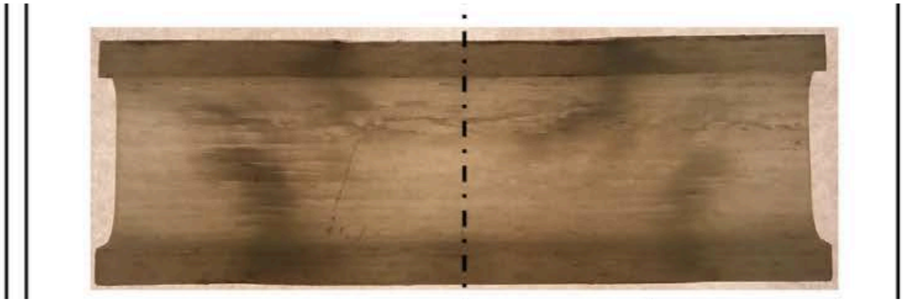
For qualification we need to test ILSS to catch production related changes in through thickness properties + possible damage from swelling/chemicals

Abedin I. Gagani, Andrey E. Krauklis, Erik Sæter, Nils Petter Vedvik, Andreas T. Echtermeyer
A Novel Method for Testing and Determining ILSS for Marine and Offshore Composites, Composite Structures, 220, 2019

Damage dry - wet



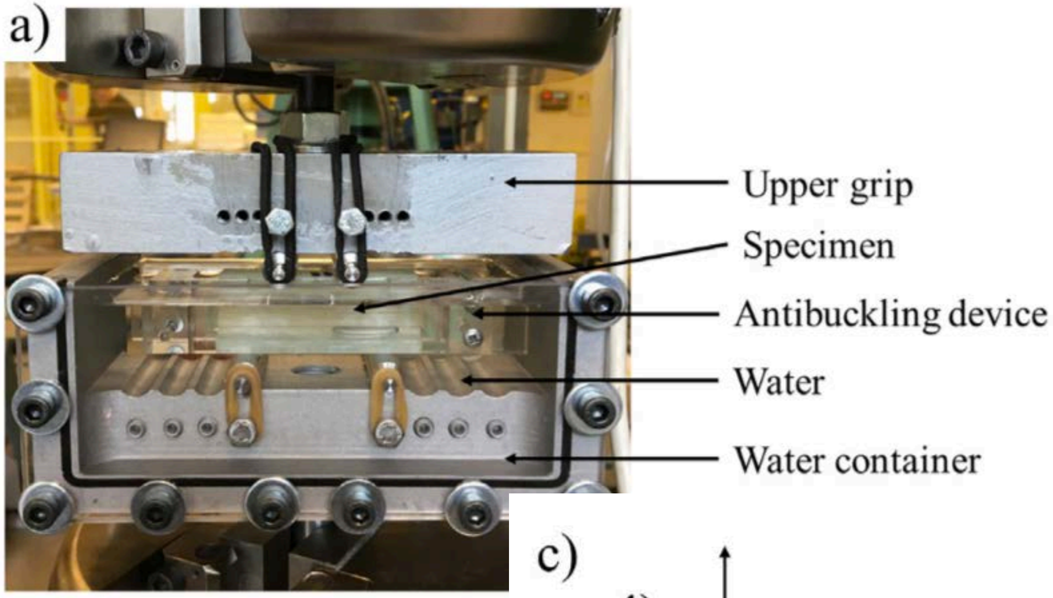
Dry I beam

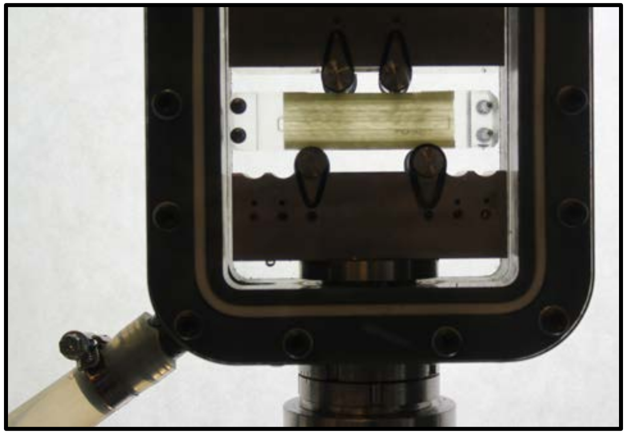


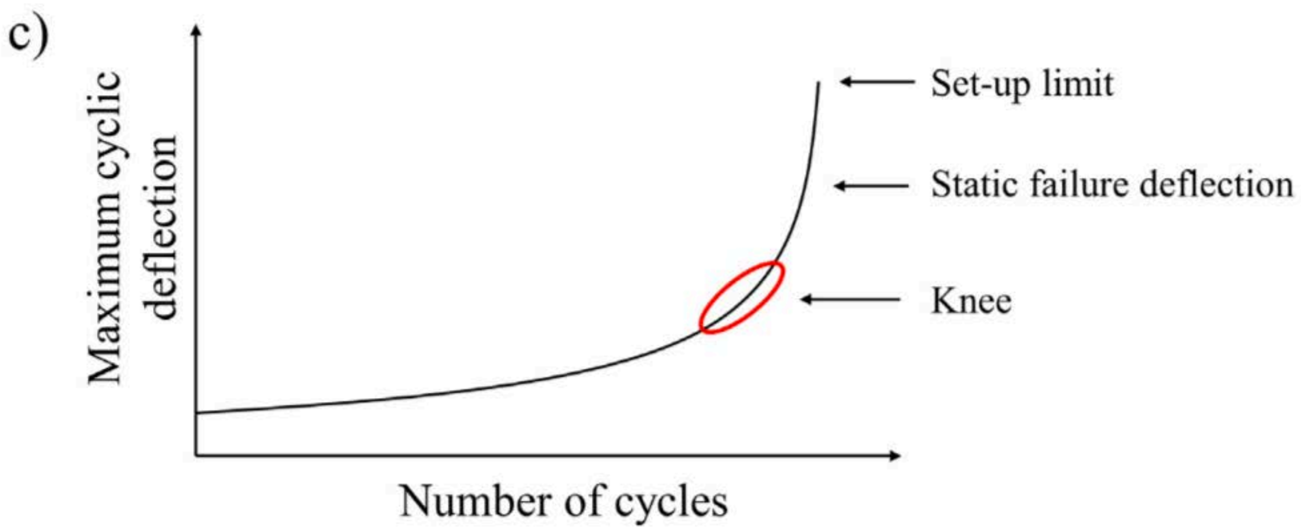
Wet I beam

Gagani, Abedin; Krauklis, Andrey; Vedvik, Nils Petter, Echtermeyer, Andreas, (2019) A novel method for testing and determining ILSS for marine and offshore composites. Composite Structures, Vol. 220, pp. 431-440

Fatigue – Failure Point





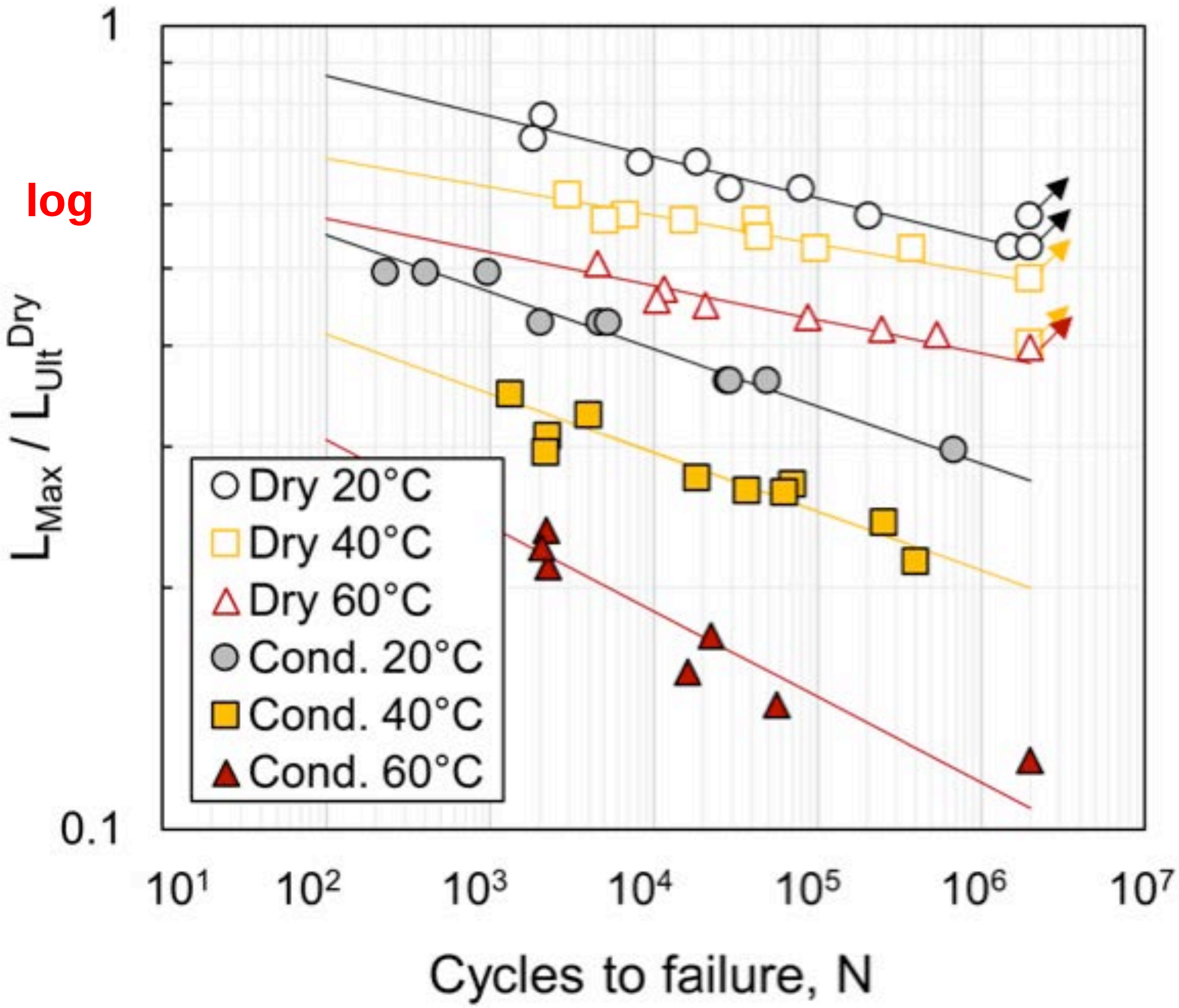


NTNU

Gagani, Abedin; Mialon, Emeric; Echtermeyer, Andreas. (2019) Immersed interlaminar fatigue of glass fiber epoxy composites using the I-beam method. International Journal of Fatigue. vol. 119.

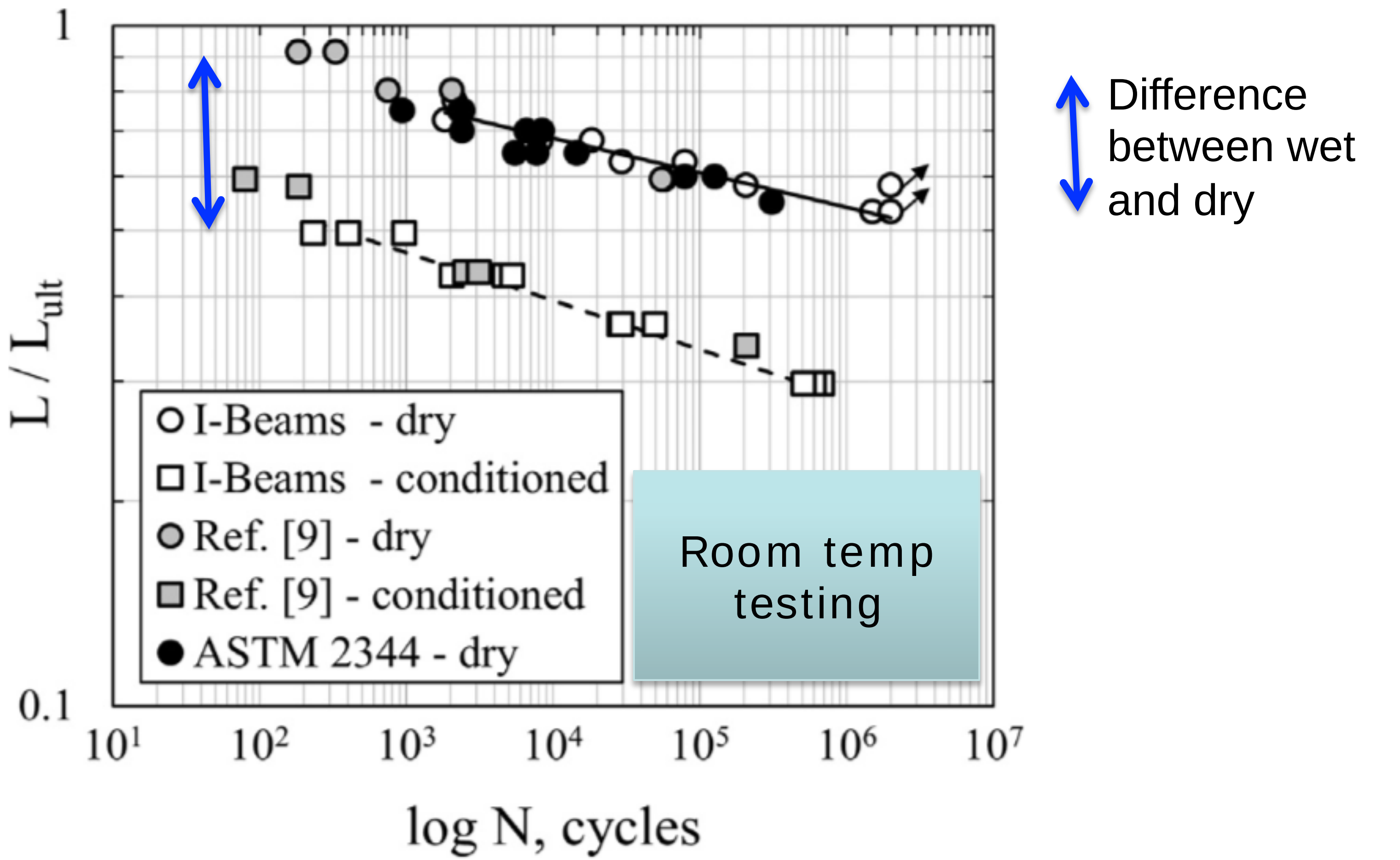
Dry-Wet SN curves

log



A. T. Echtermeyer, A. I. Gagani, A. E. Krauklis and R. Moslemian, Lomg Term Fatigue Dedradation– Superposition of Dry and Wet Properties, 22nd International Conference on composite materials (ICCM22)

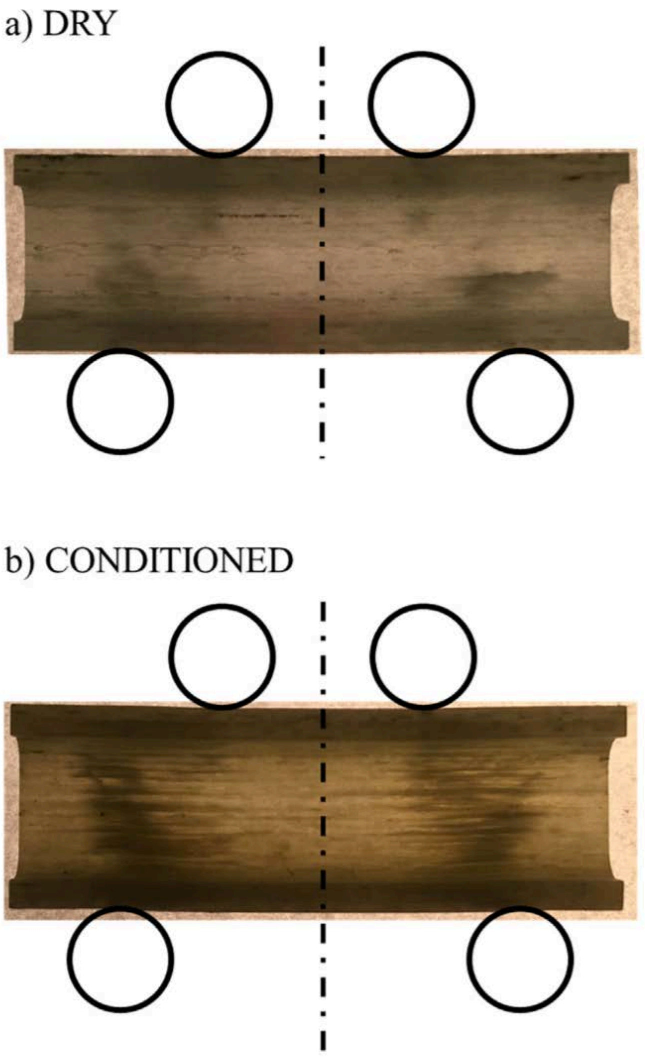
Compare with standard SBS

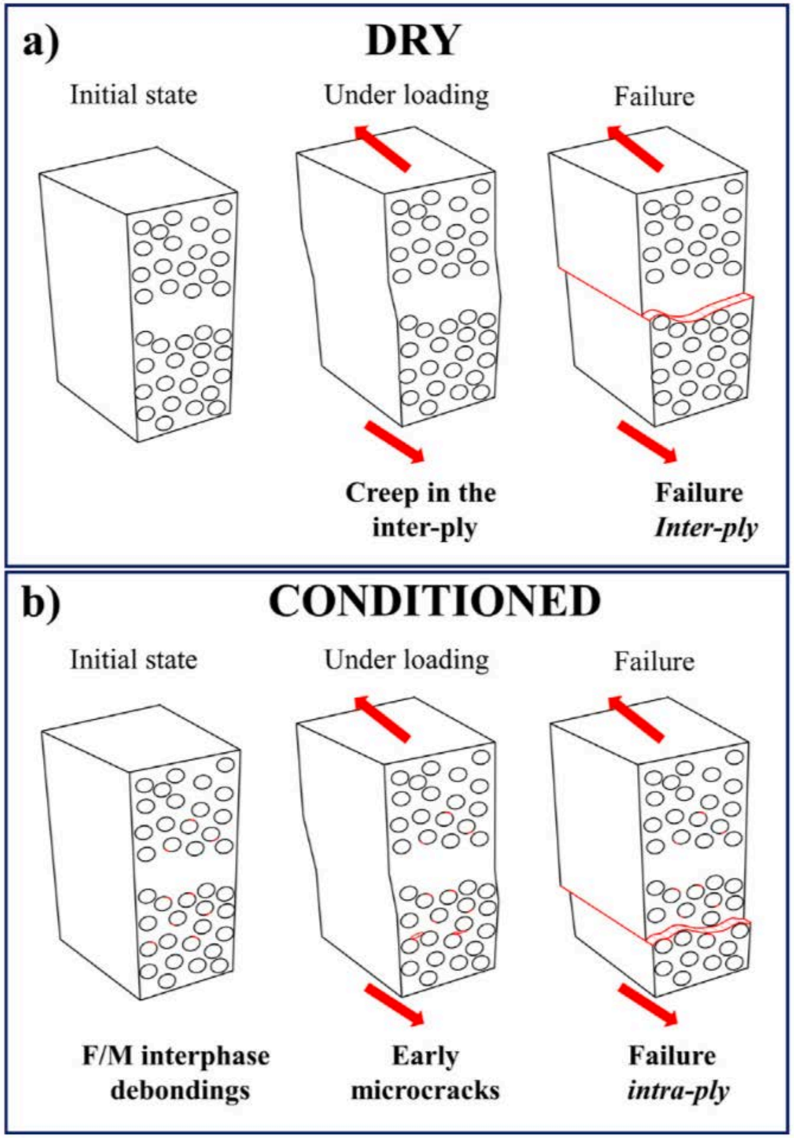


Gagani, Abedin; Mialon, Emeric; Echtermeyer, Andreas. (2019) Immersed interlaminar fatigue of glass fiber epoxy composites using the I-beam method. International Journal of Fatigue. vol. 119.

I.B.C.M. Rocha, S. Raijmaekers, R.P.L. Nijssen, F.P. van der Meer, L.J. Sluys, Hygrothermal ageing behavior of a glass/epoxy composite used in wind turbine blades, Composite Structures,Volume 174, 2017, Pages 110-122, ISSN 0263-8223, https://doi.org/10.1016/j.compstruct.2017.04.028.

Change in Failure Mechanism





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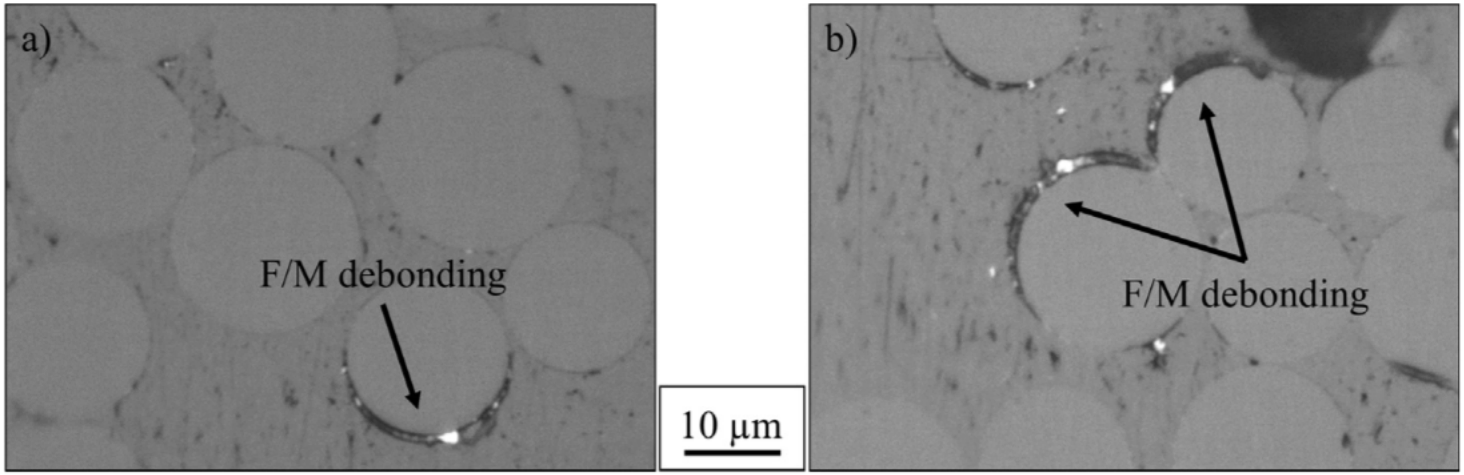
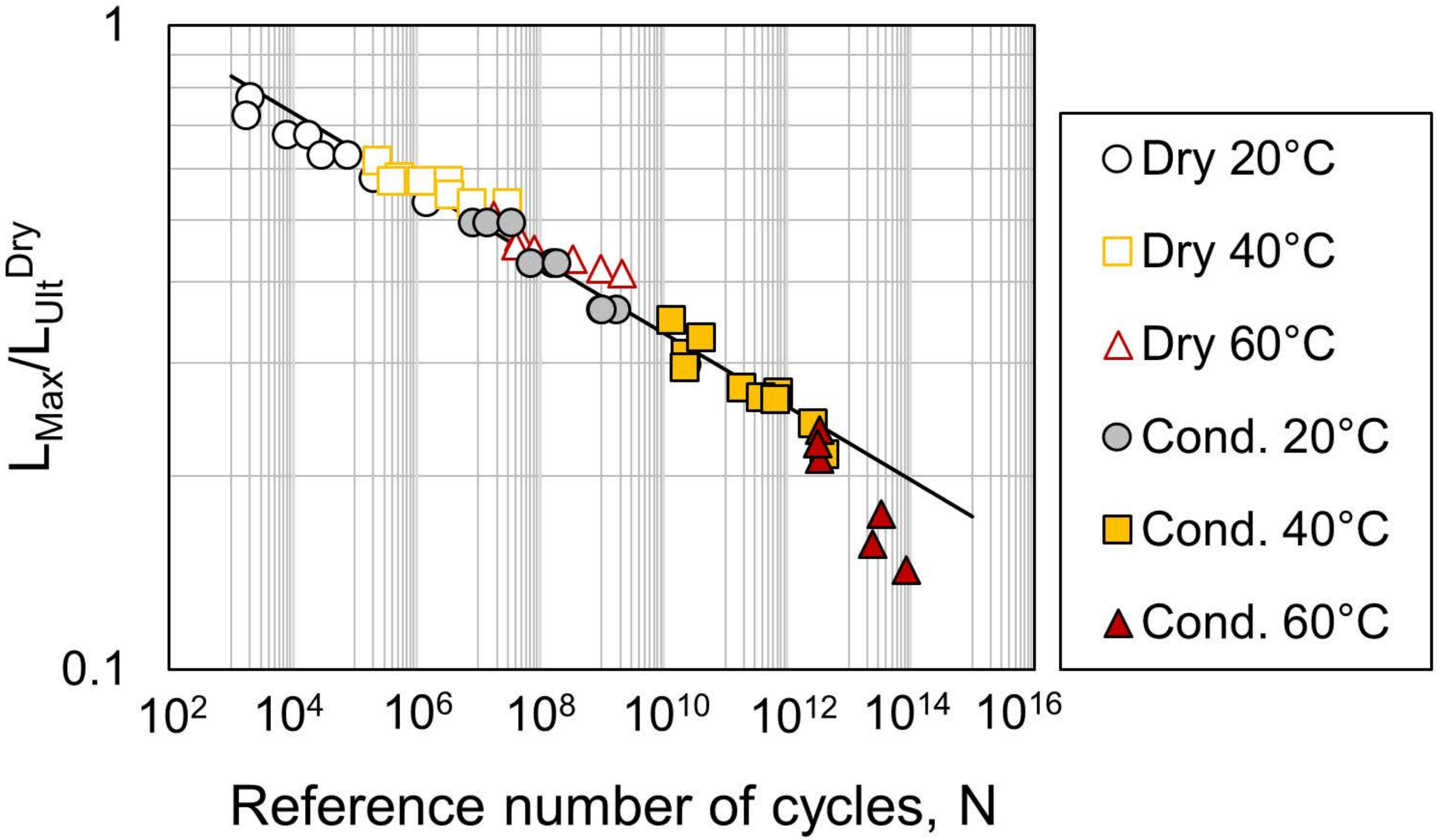
Fiber-Matrix debonding for wet conditioned specimens



Debonding was observed after conditioning before mechanical testing

Gagani, Abedin; Mialon, Emeric; Echtermeyer, Andreas. (2019) Immersed interlaminar fatigue of glass fiber epoxy composites using the I-beam method. International Journal of Fatigue. vol. 119.

Fatigue Master Curve



A. T. Echtermeyer, A. I. Gagani, A. E. Krauklis and R. Moslemian, Lomg Term Fatigue Dedradation– Superposition of Dry and Wet Properties, 22nd International Conference on composite materials (ICCM22)

Arrhenius Approach

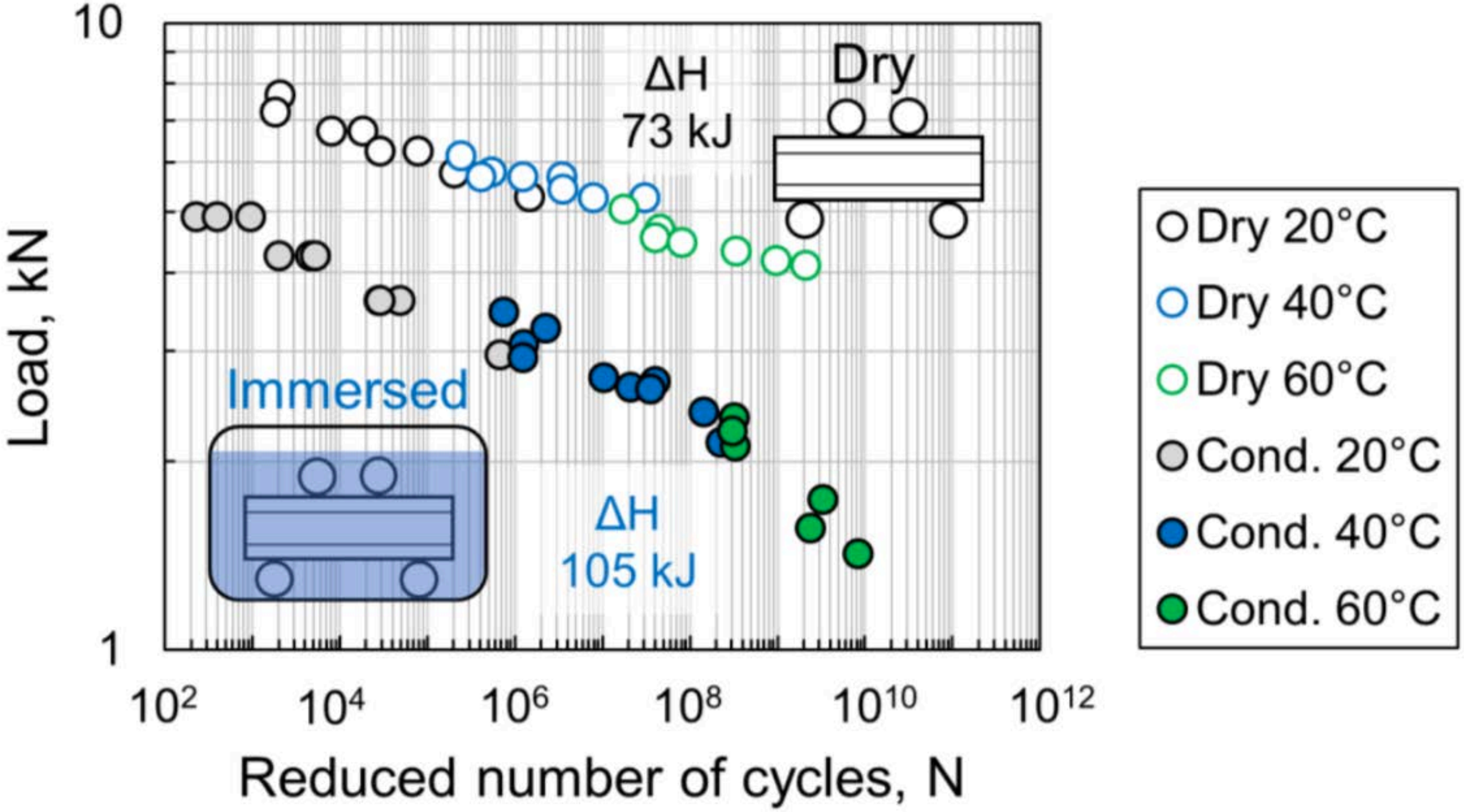
$$\log a_T = log\left(\frac{t_0}{t_1}\right) = \log t_0 - \log t_1 = \frac{-\Delta H}{2.3R}\left(\frac{1}{T_1} - \frac{1}{T_0}\right)$$

$$\log a_N = log\left(\frac{N_0}{N_1}\right) = \log N_0 - \log N_1 = \frac{-\Delta H}{2.3R}\left(\frac{1}{T_1} - \frac{1}{T_0}\right)$$

Zhurkov, S.N. and Korsukov, V.E. *Atomic mechanism of fracture of solid polymers.* Journal of Polymer Science, 1974. 12: p. 385-398.

Nakada, M. and Y. Miyano, Accelerated testing for long-term fatigue strength of various FRP laminates for marine use. Composites Science and Technology, 2009. 69(6): p. 805-813.

Master Curves for Dry and Wet



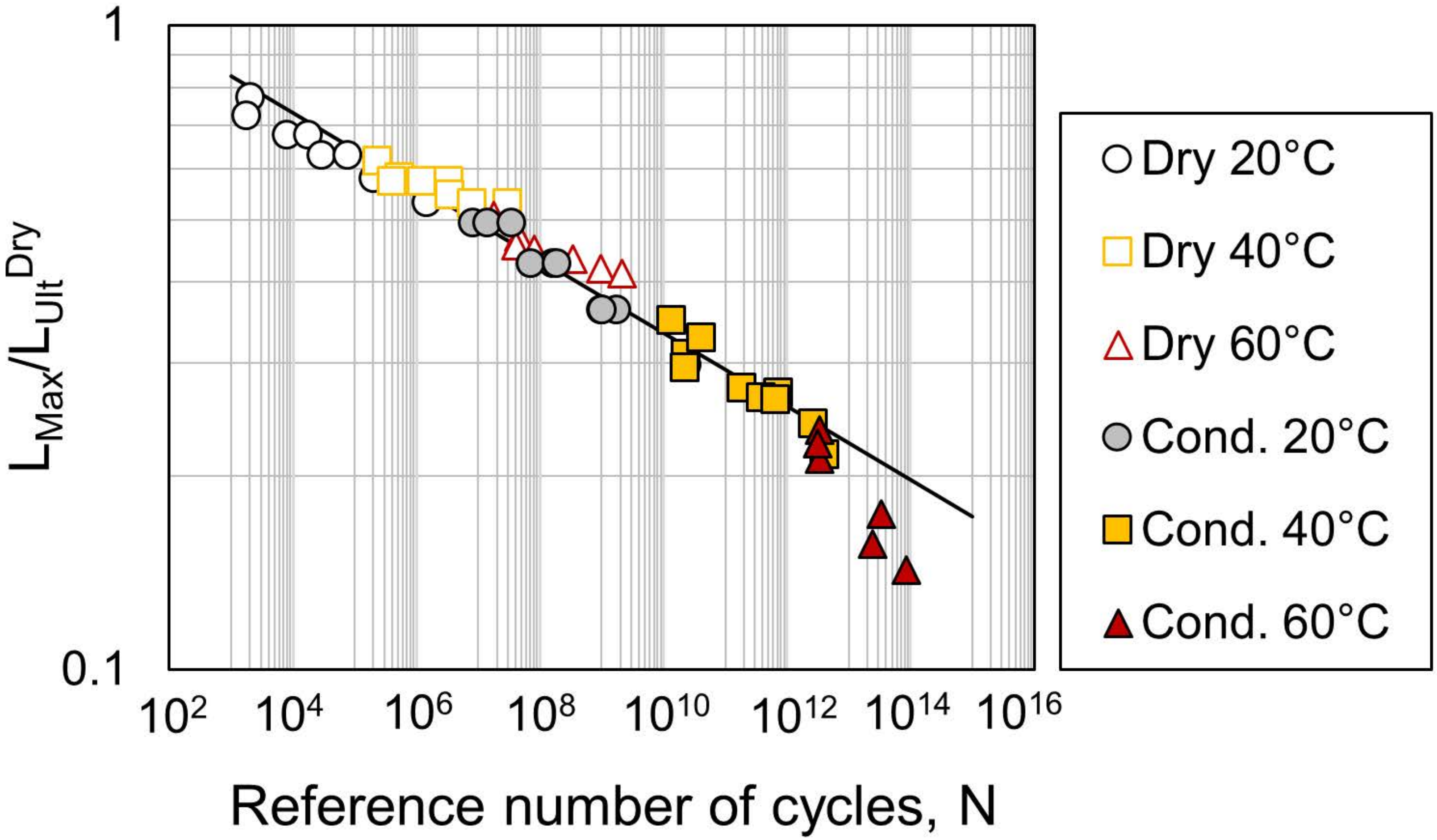
Gagani, Abedin; Mialon, Emeric; Echtermeyer, Andreas. (2019) Immersed interlaminar fatigue of glass fiber epoxy composites using the I-beam method. International Journal of Fatigue. vol. 119.

Equivalent Temperature

$$T^* = T \frac{T_g^{dry}}{T_g^{cond}}$$

$$\log N - \log N_R = \frac{-\Delta H}{2.303R}\left(\frac{1}{T_R} - \frac{1}{T^*}\right)$$

Fatigue Master Curve



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Interlaminar Shear

Time Temp Superposition works for Interlaminar Shear Fatigue, if we do not cross Tg

(seems to work for thermoplastics too)

A change of failure mechanism gives a new slope of the SN curve → new activation enthalpy.

Dry and Wet data can be superimposed by an equivalent temperature related to Tg

Summary

Simplifications Matrix (Idealistic)

Fatigue of Matrix exposed to water can be described by one static dry SN curve and shift of static strength. No need to measure many SN curves. (The same seems to apply to temperature changes).

Simplifications Glass Fibers (Idealistic)

Sized Glass Fiber mass loss can be described by Arrhenius with an activation energy dependent on pH and stress. This gives the advantages of measuring a few cases and calculating the rest.

The link of mass loss to strength change is in progress.

Composite fiber dominated tensile ply strength can “probably” be scaled in the same way.

Interlaminar Shear (Idealistic)

No fiber matrix debonding when wet:

Measure SN curve at two temperatures, get activation enthalpy.

Measure only Tg on saturated sample.

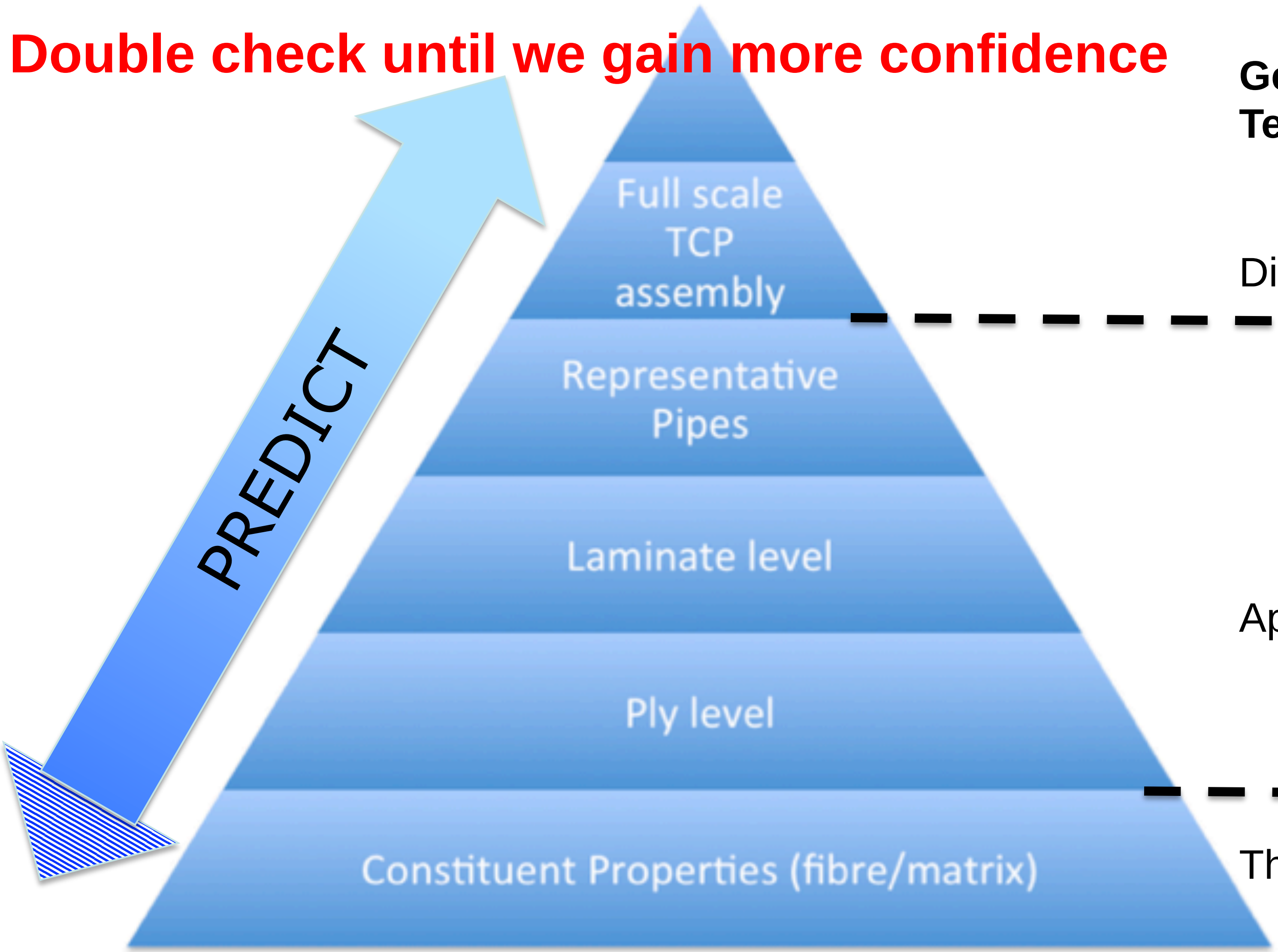
Calculate all other properties

If fiber matrix debonding, measure SN curve at two temperatures, to get "saturated" activation enthalpy

Calculate all other properties

Simplification (realistic)

Double check until we gain more confidence



Getting Long Term Properties

Difficult, only in air

Approach today

The future..NOW..

CONCLUSIONS

Long term degradation is critical and current test methods are sufficient but costly.

This program has increased the understanding of long term degradation

Substantial savings in testing are possible, especially for interpolations.

Following the approach taken here more simplifications can be established.

Acceptance and full utilization will take time, but a framework for achieving this exists now.