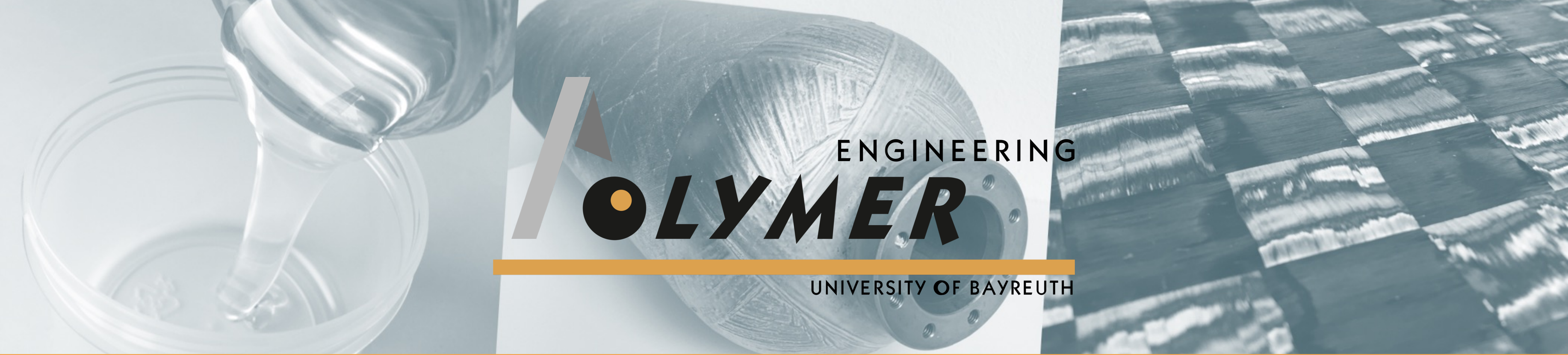
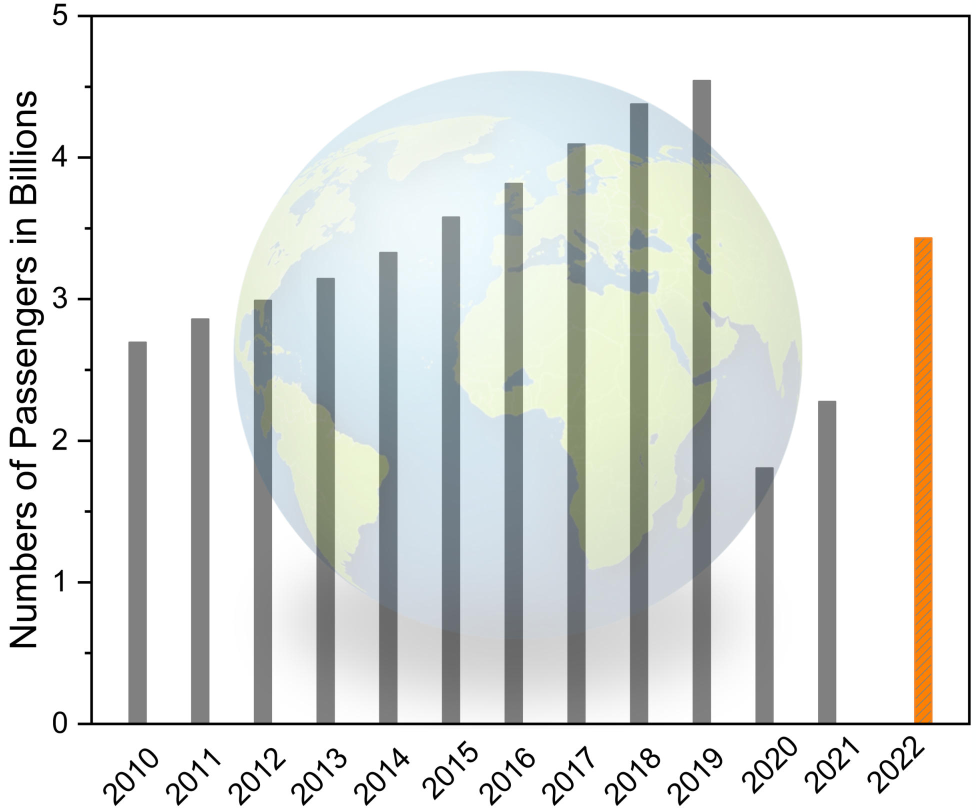


INFLUENCE OF ADDITIVES AND FIBER-LAYUP ON THE AGING BEHAVIOR OF HIGH-TEMPERATURE EPOXY RESIN PREPREG SYSTEMS

ICCM 23 | 30 July – 04 August | **Martin Demleitner**, Bastian Treiber, Quirin Niederauer, Holger Ruckdäschel

Air Travel is back! – But Strong Impact on Climate Change



Statista

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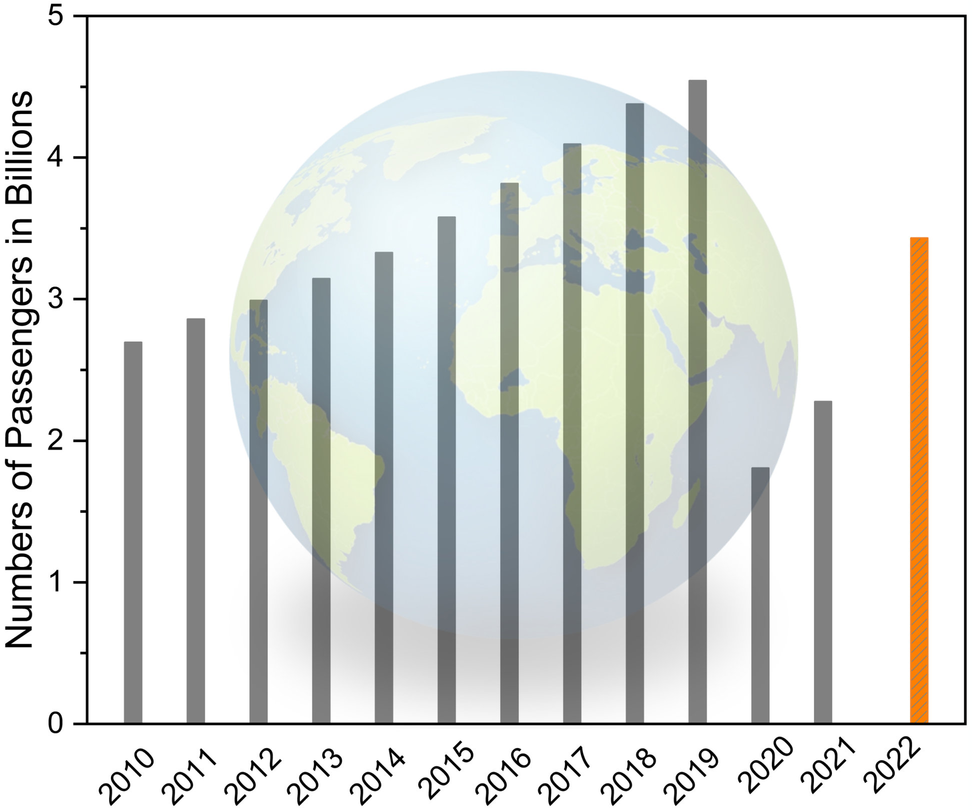
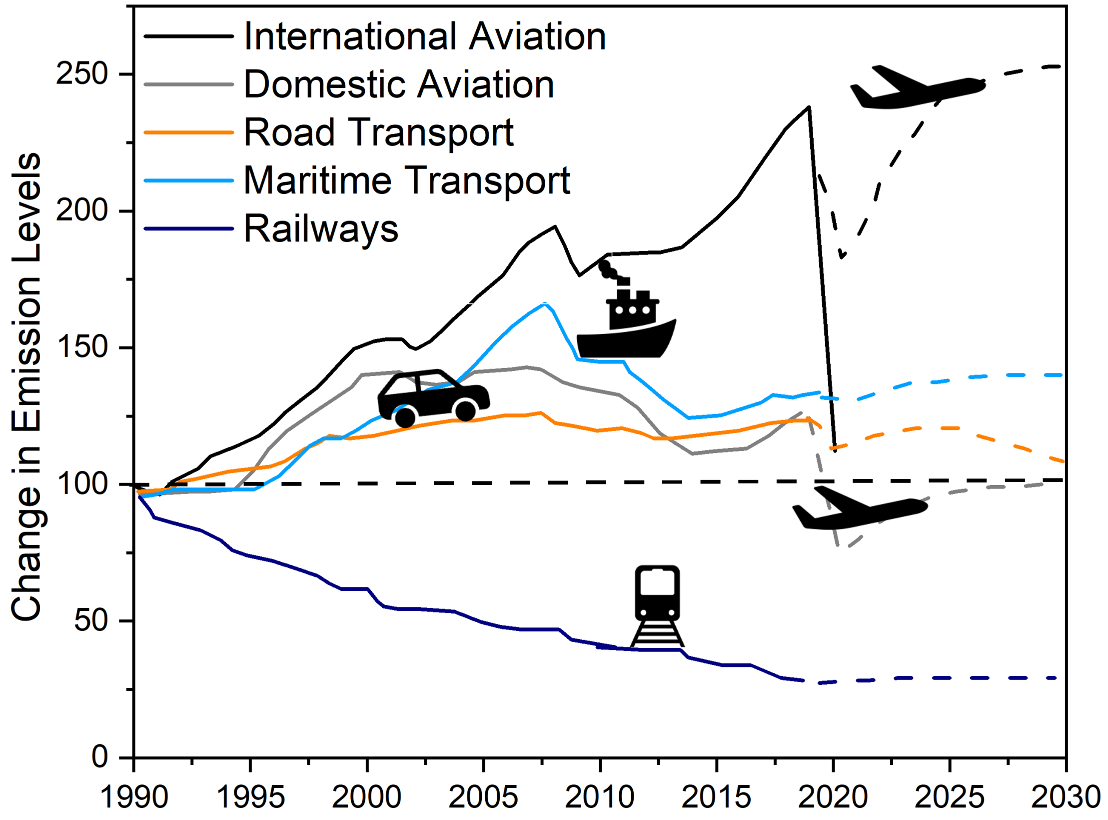


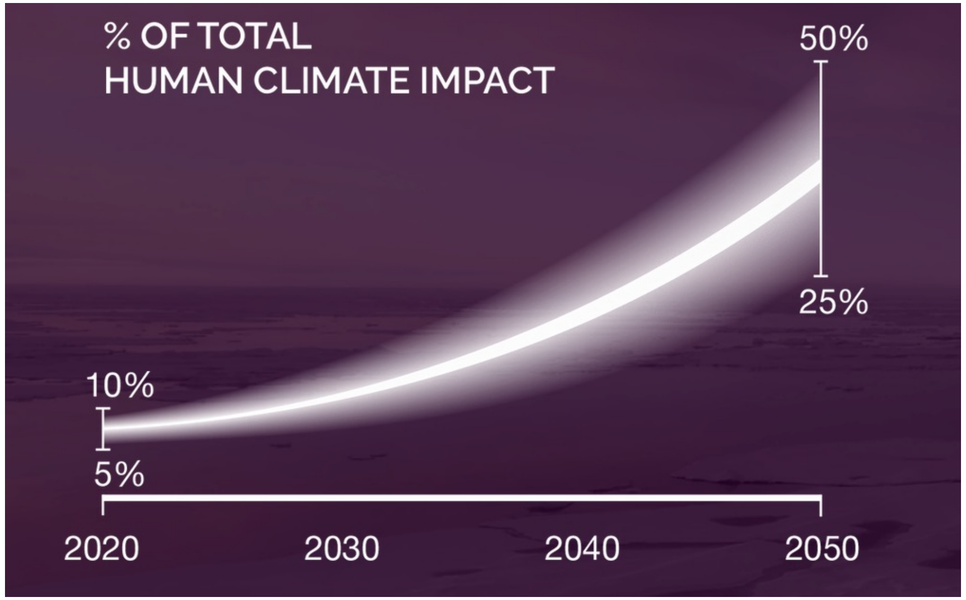


Trend towards lower CO_2 emissions for rail and car traffic
Aviation expected to grow to **Pre-Covid level** latest **2024**

Statista
https://www.eea.europa.eu/data-and-maps/indicators/transport-emissions-of greenhouse-gases/transport-emissions-of-greenhouse-gases-12

Aviation is fastest-growing Source of Greenhouse Emissions

Air traffic threatens to become the **largest CO_2 polluter** by 2050



Solutions:

- Flying less
- New propulsion systems
- Biobased fuels
- Lightweight design and material

Emissions at high altitude have **2-4x times** greater impact than comparable ground emissions

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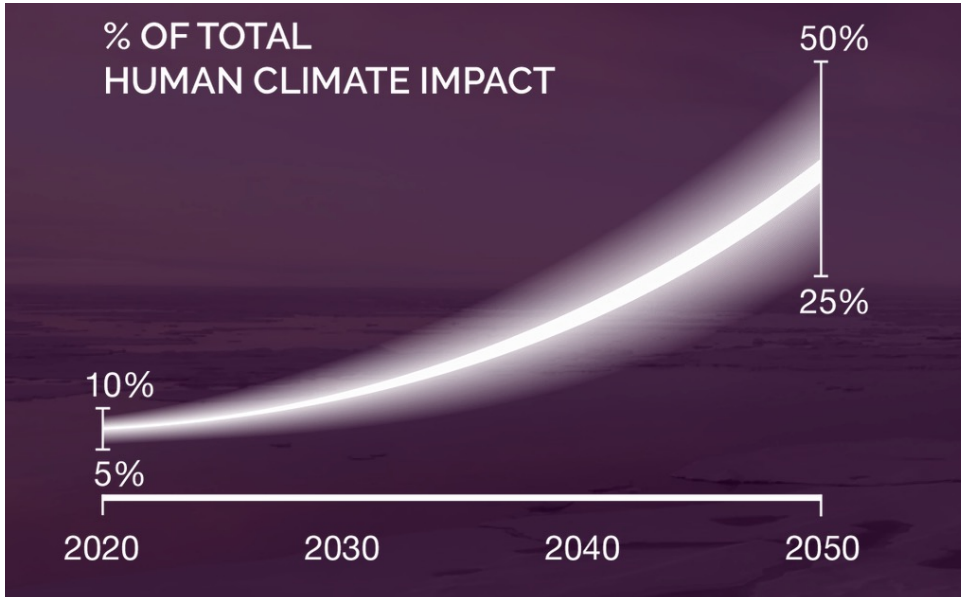

Solutions:

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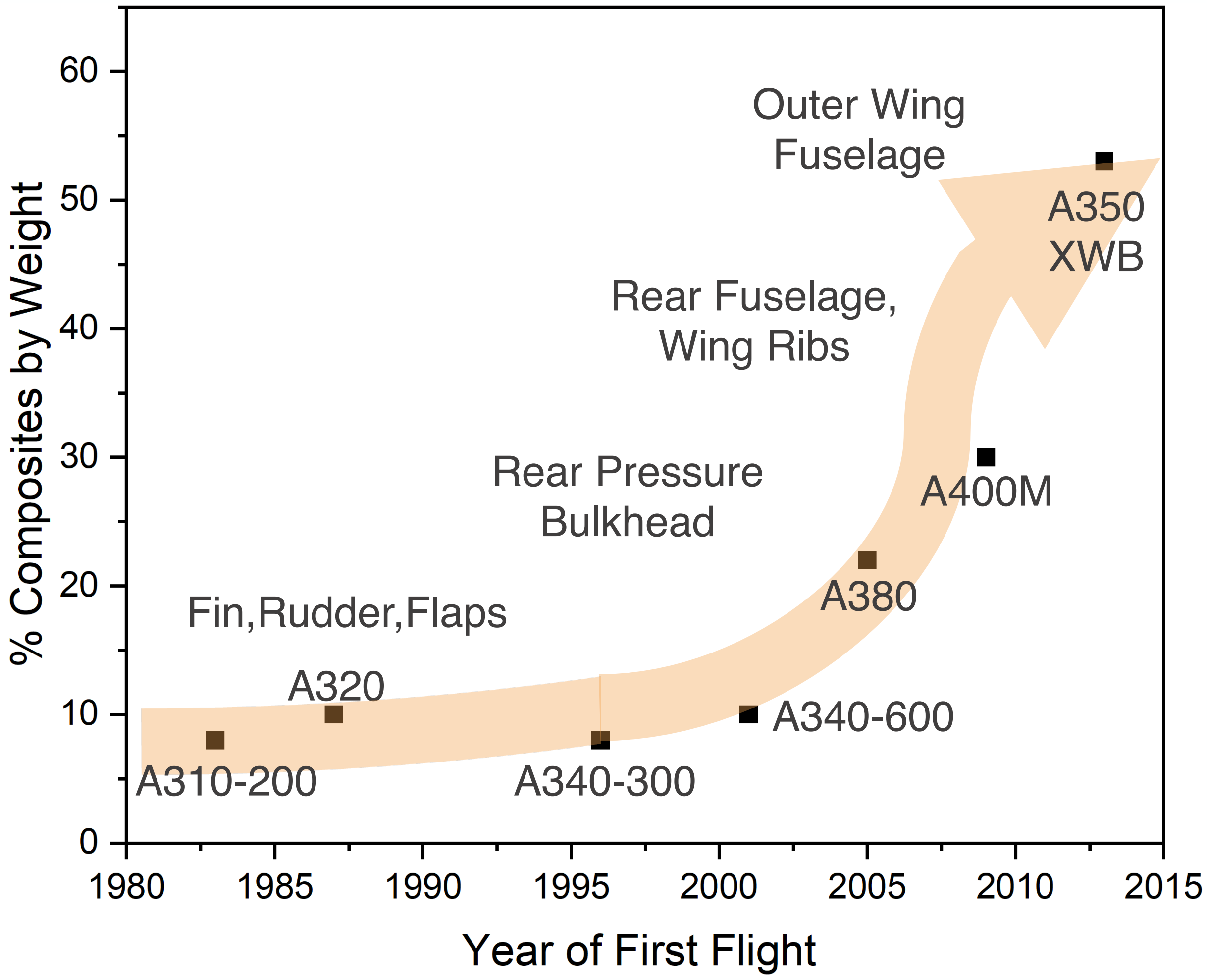
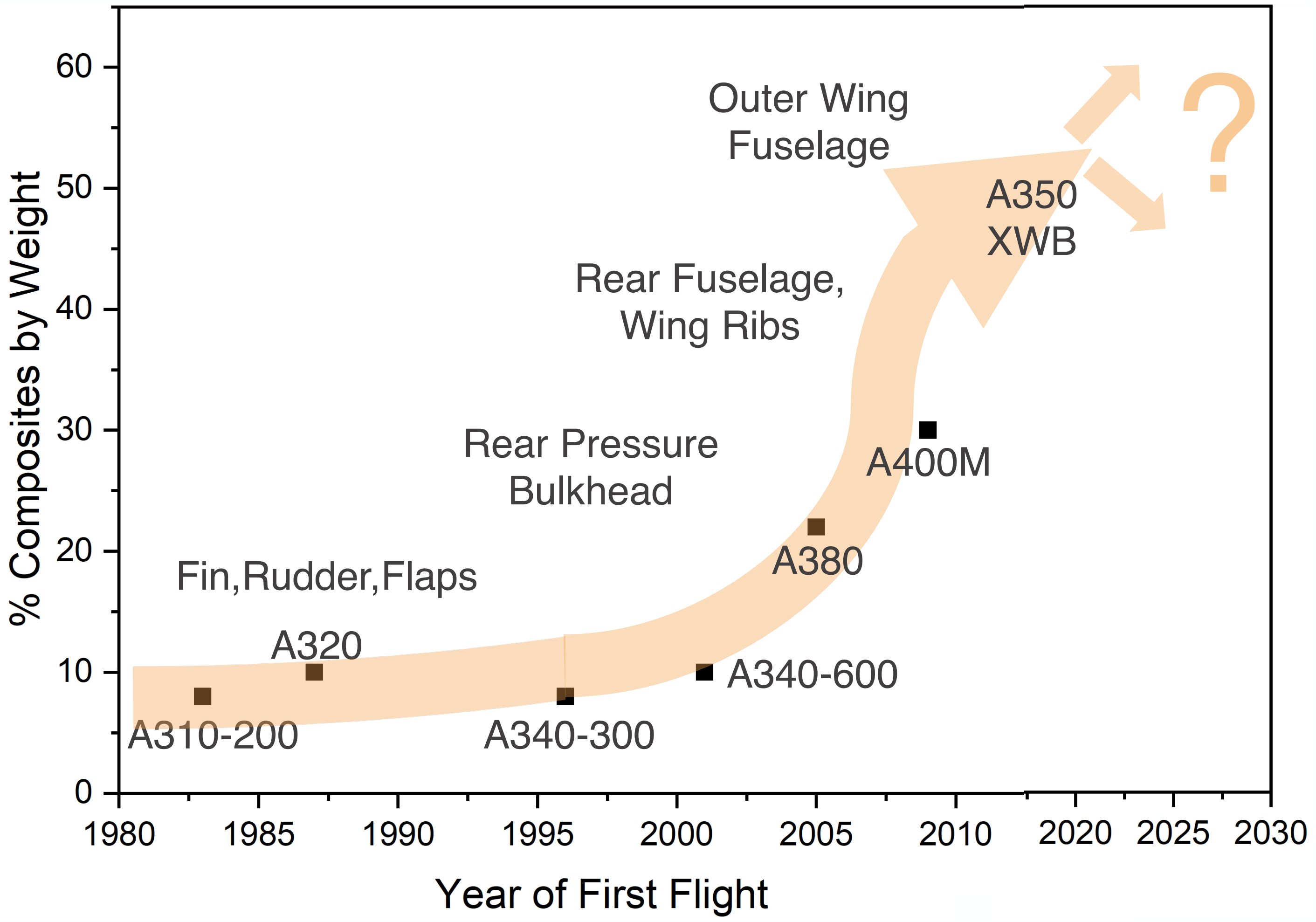
Development of FRP in Aviation



T. Trzepiencinski, Journal of Composite Science, **2021**

Development of FRP in Aviation – Quo Vadis?



Further increase through replacement of aluminium, titanium alloys by

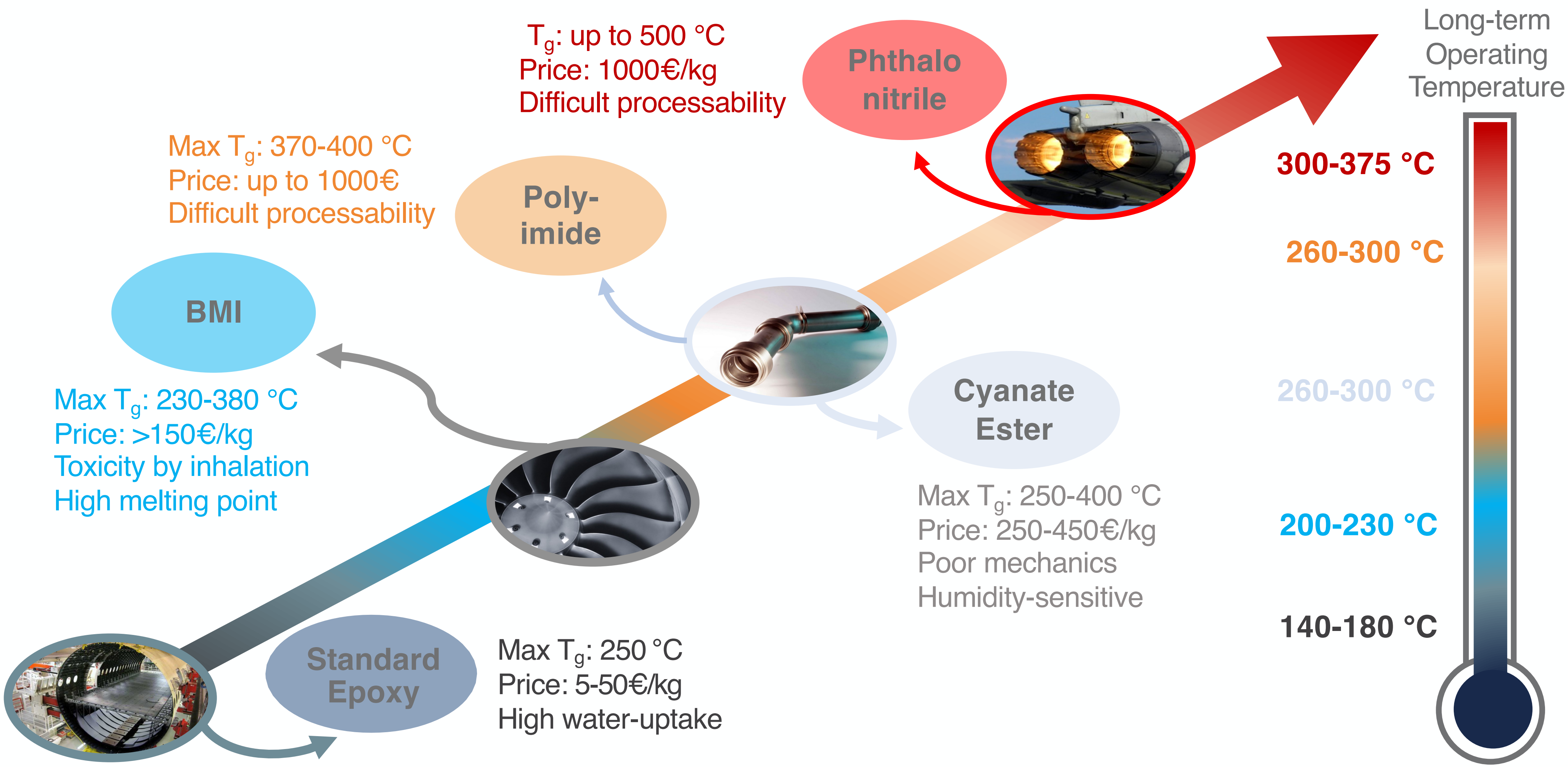
high-temperature resistant polymer composites

for

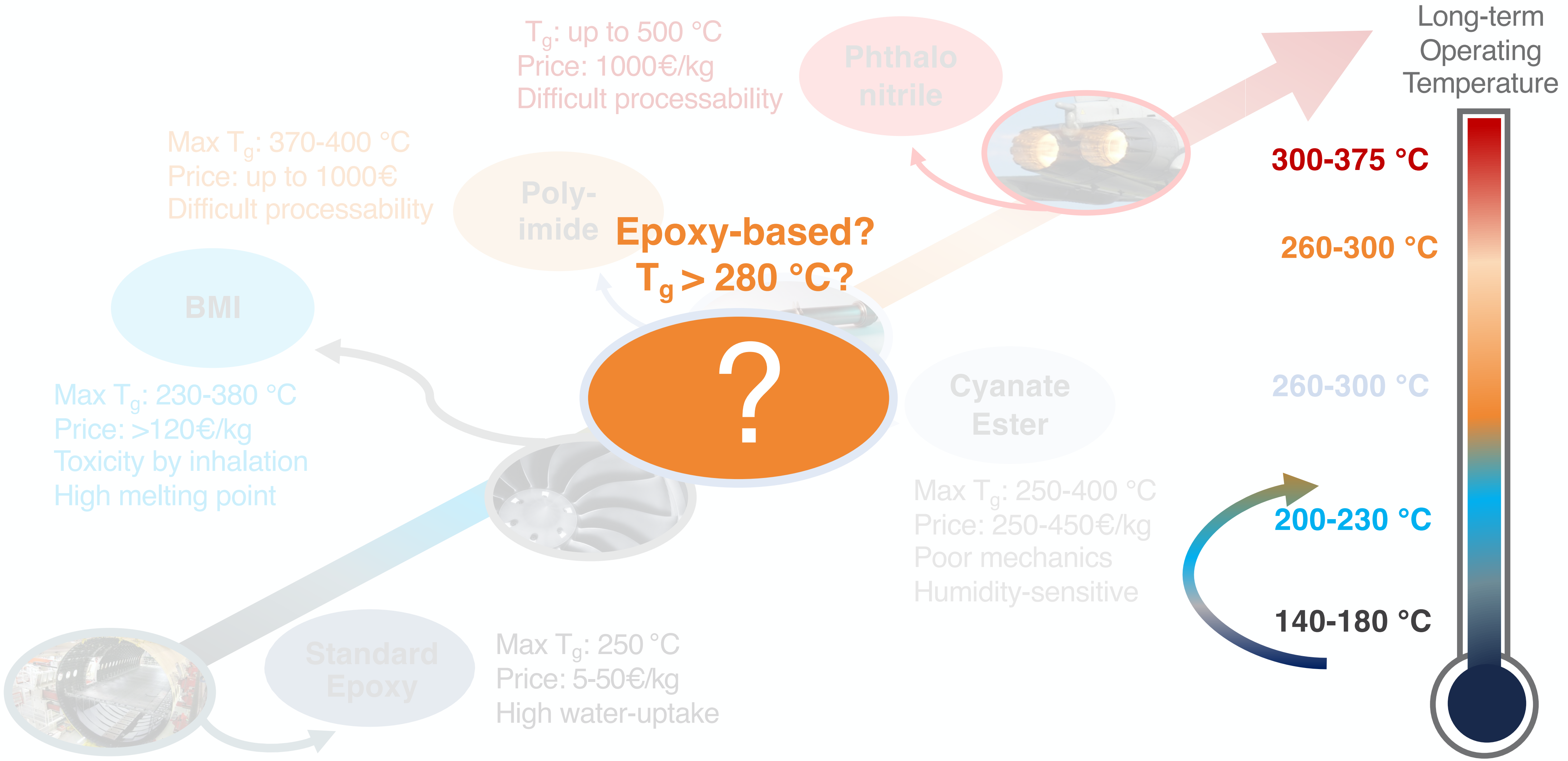
- Engine fairings
- Air ducts
- Guide vanes
- etc.

T. Trzepiencinski, Journal of Composite Science, **2021**

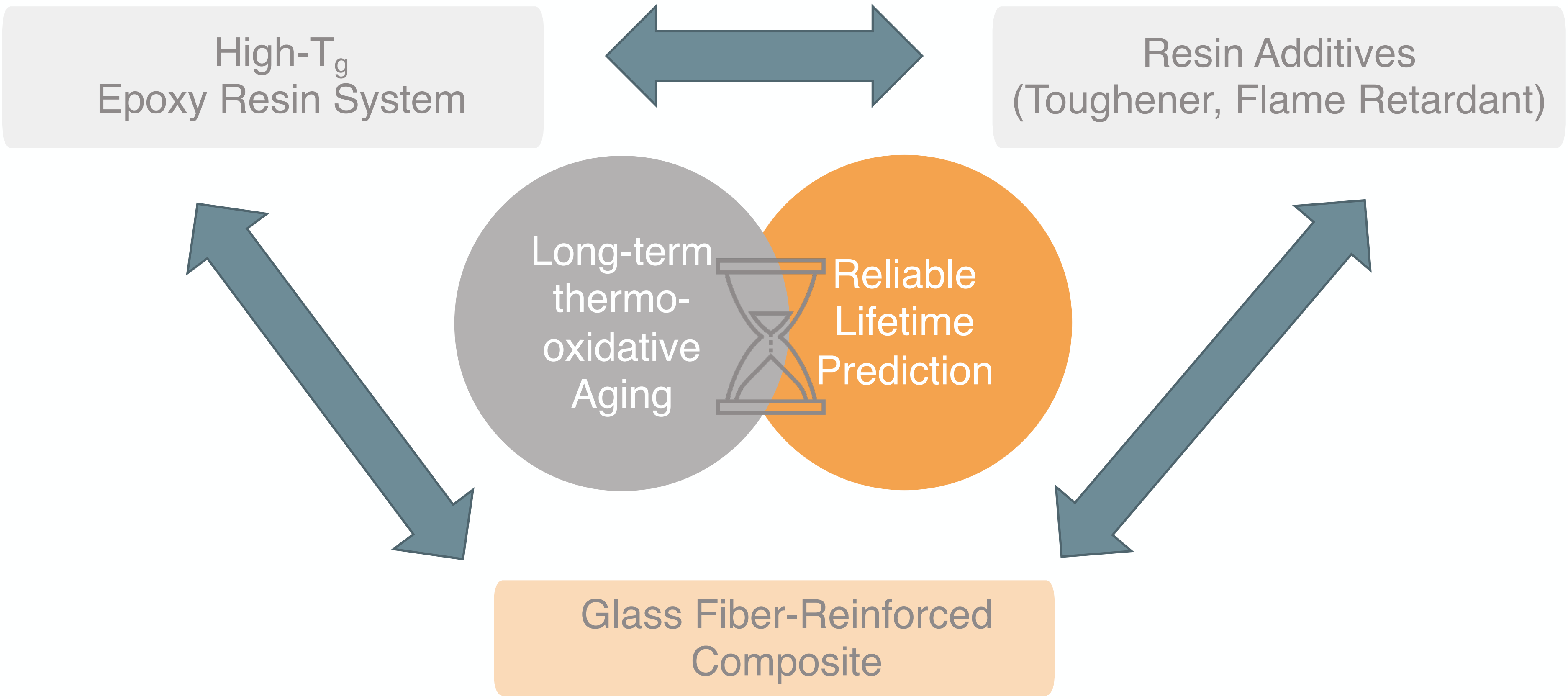
High-Temperature Resin Systems



High-Temperature Resin Systems



Motivation for the Research



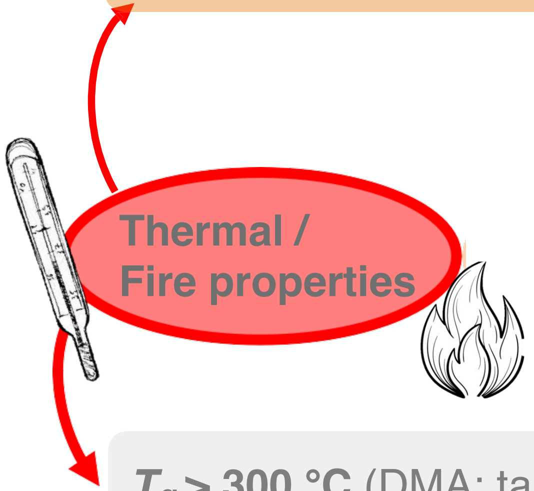
Prepreg/Composite

Neat Resin

High-T_g Epoxy Resin System – Technical Aims

(P)HHR < 65 kW/m²

TSP: < 20 m²



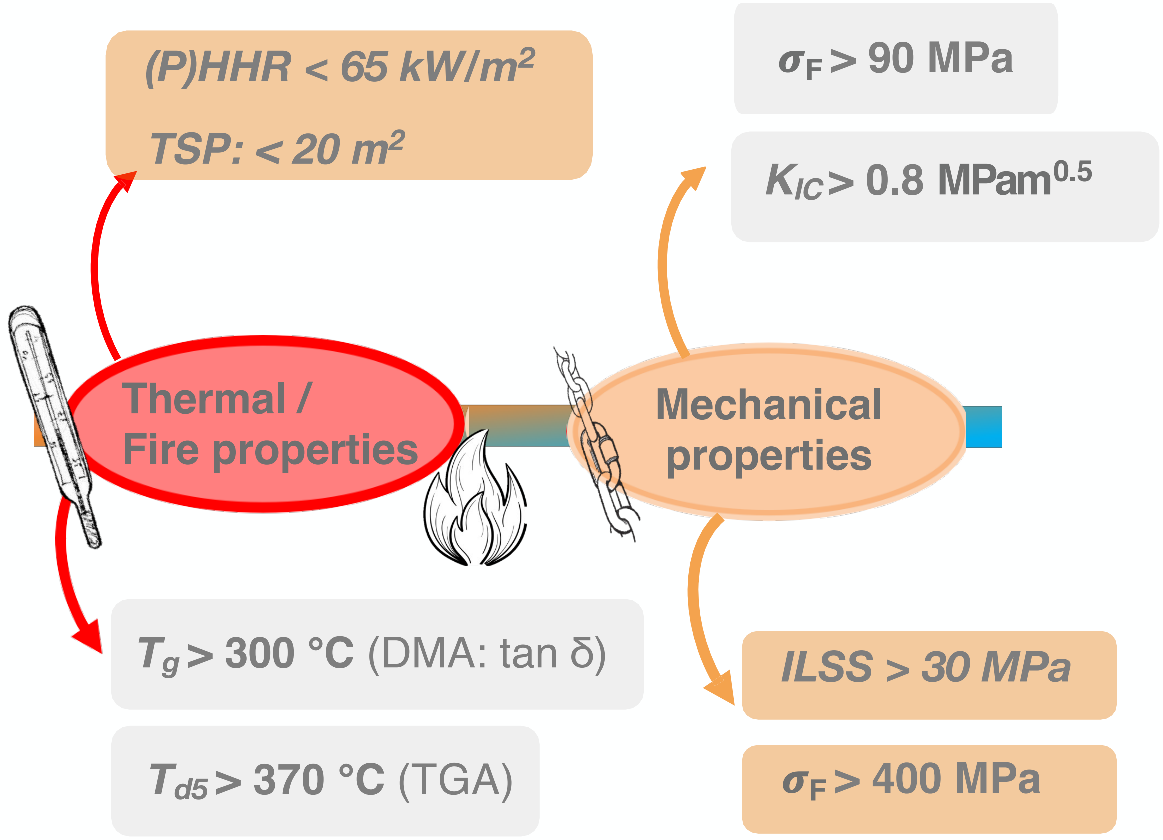
T_g **> 300 °C** (DMA: tan δ)

T_{d5} **> 370 °C** (TGA)

High-T_g Epoxy Resin System – Technical Aims

Prepreg/Composite

Neat Resin



Prepreg/Composite

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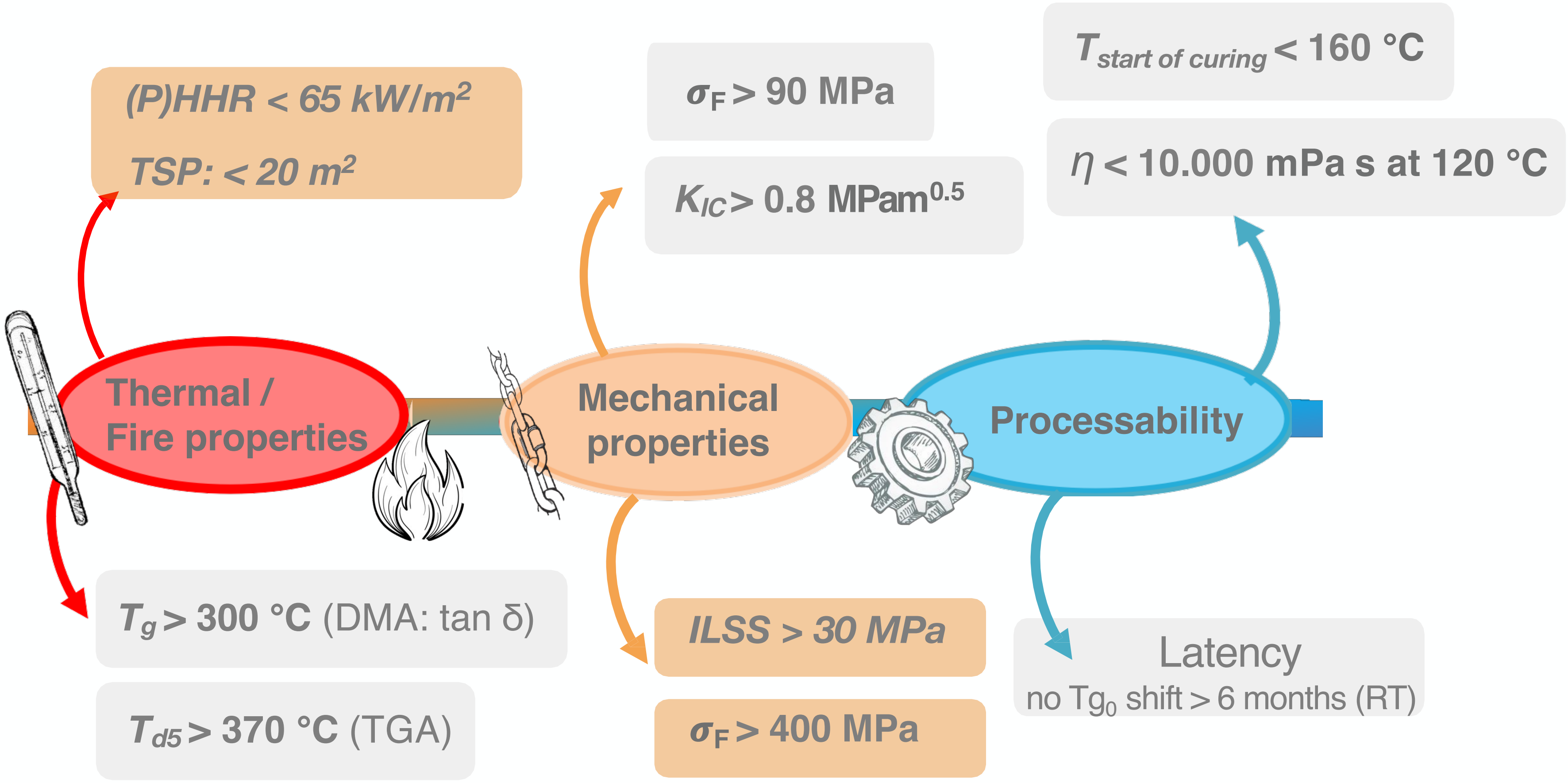

Prepreg/Composite

Neat Resin

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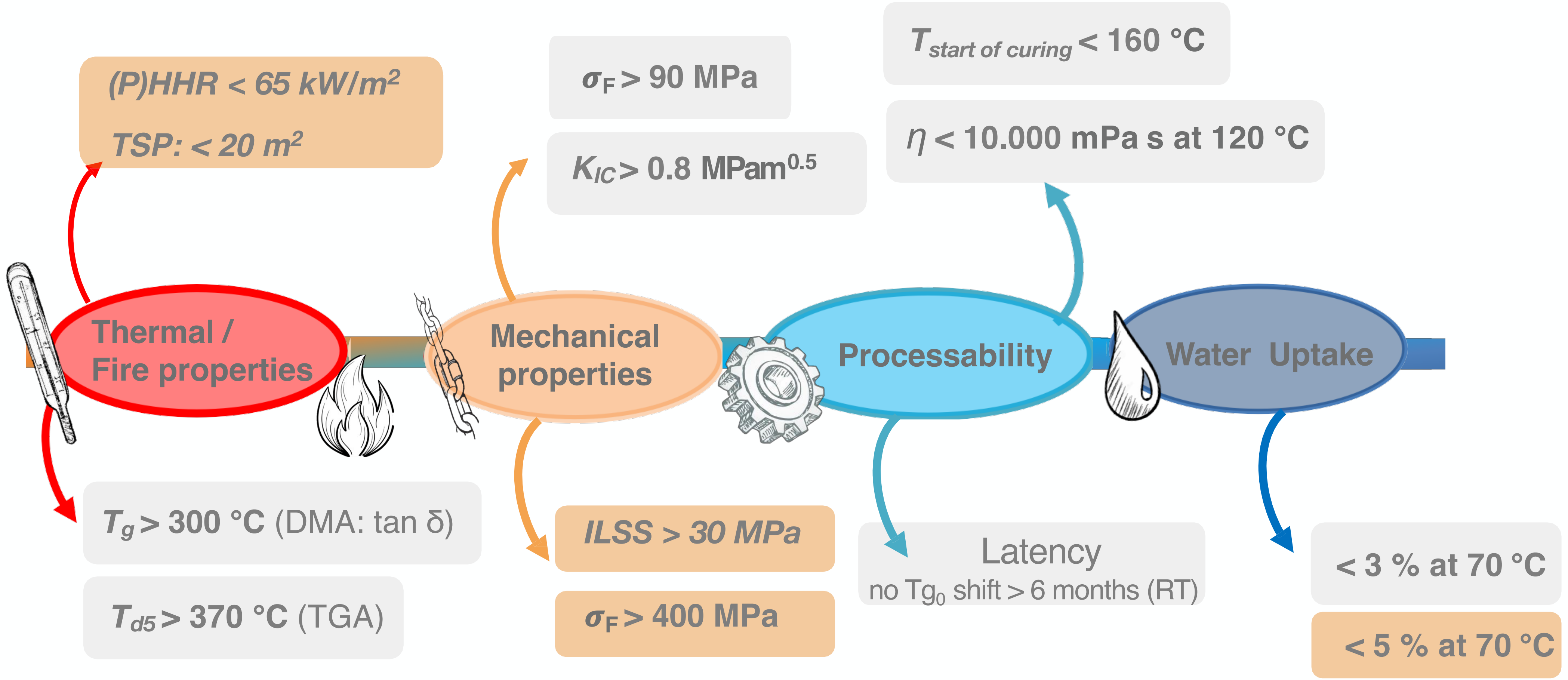
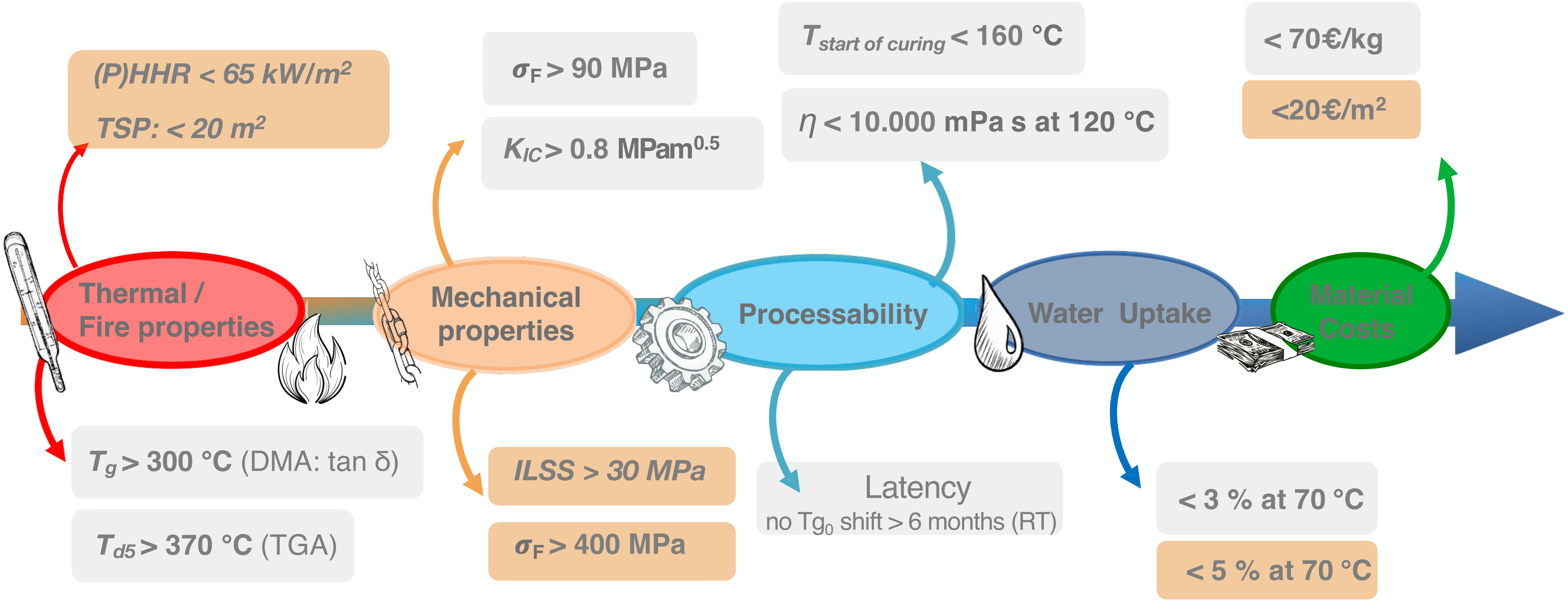
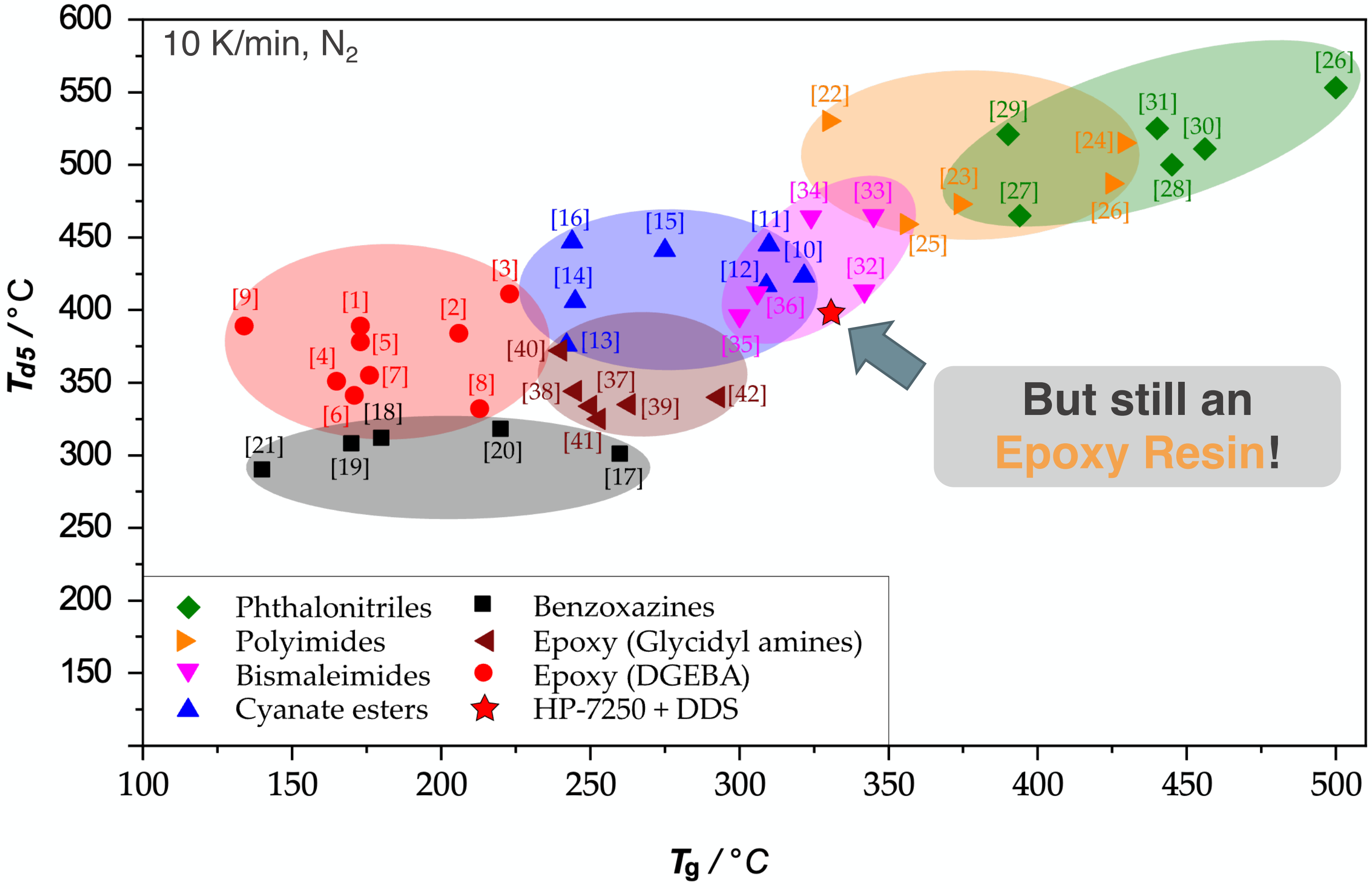


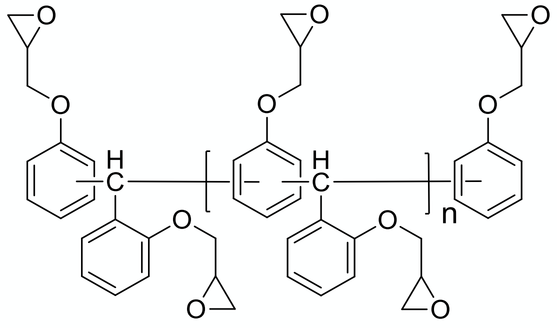
Prepreg/Composite
Neat Resin

High-T_g Epoxy Resin System – Technical Aims

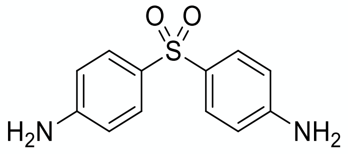


State of the Art





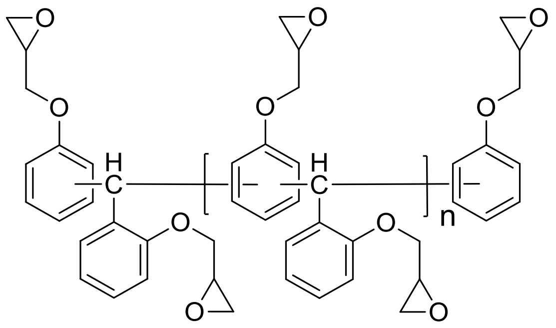
HP-7250



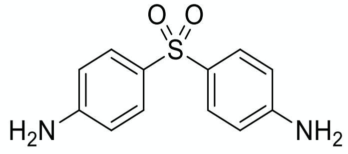
4,4´-Diaminodiphenyl sulfone

[1]-[42]: See Master Thesis: Influence of Toughener and Flame Retardant Additives on the Aging Behavior of a High-Tg Epoxy Resin System by Bastian Treiber for literature

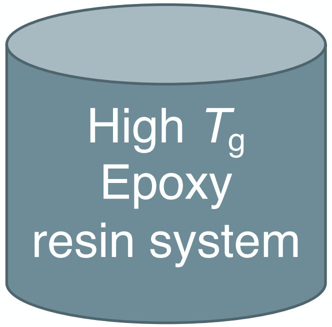
Overview of Chosen Material System

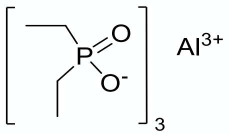


HP-7250
Functionality: 5.0
by DIC Corporation (Japan)



4,4´-Diaminodiphenyl sulfone
by ACCI Specialty Materials (USA)

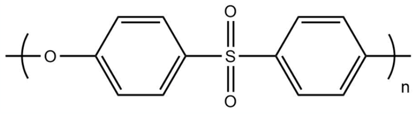




Exolit OP935
Particle size D50: 2μm
Content: 10 wt.%
by Clariant AG (Germany)

FR

Toughener



Sumikaexcel 5003P
Hydroxy-terminated PES powder
Particle size 20 μm
Content: 20 wt.%
By Sumitomo Chemicals (Japan)

Experimental Design

Sample Preparation

N$_2$

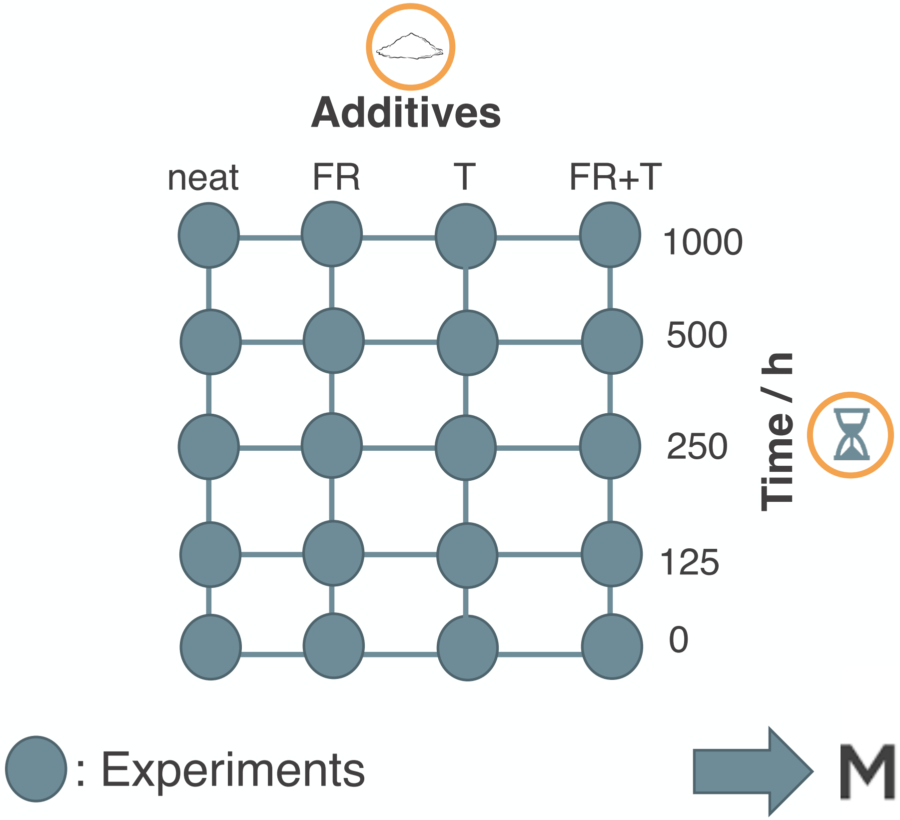
Testing

Factors: Time, Temperature, Additives, Atmosphere

Outputs: Dimensional, Thermal, Mechanical, Fire related

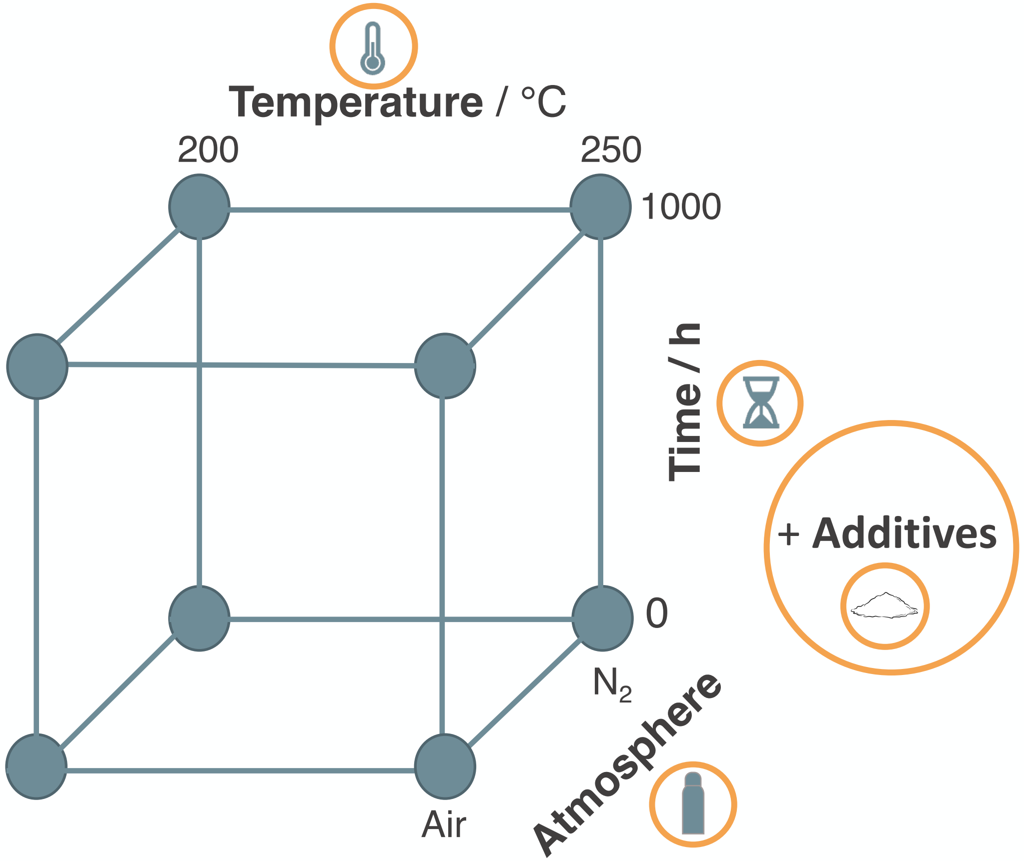
Methods – Design of Experiments (DoE)

Full factorial design
two factors at 225 °C + air → 2D:

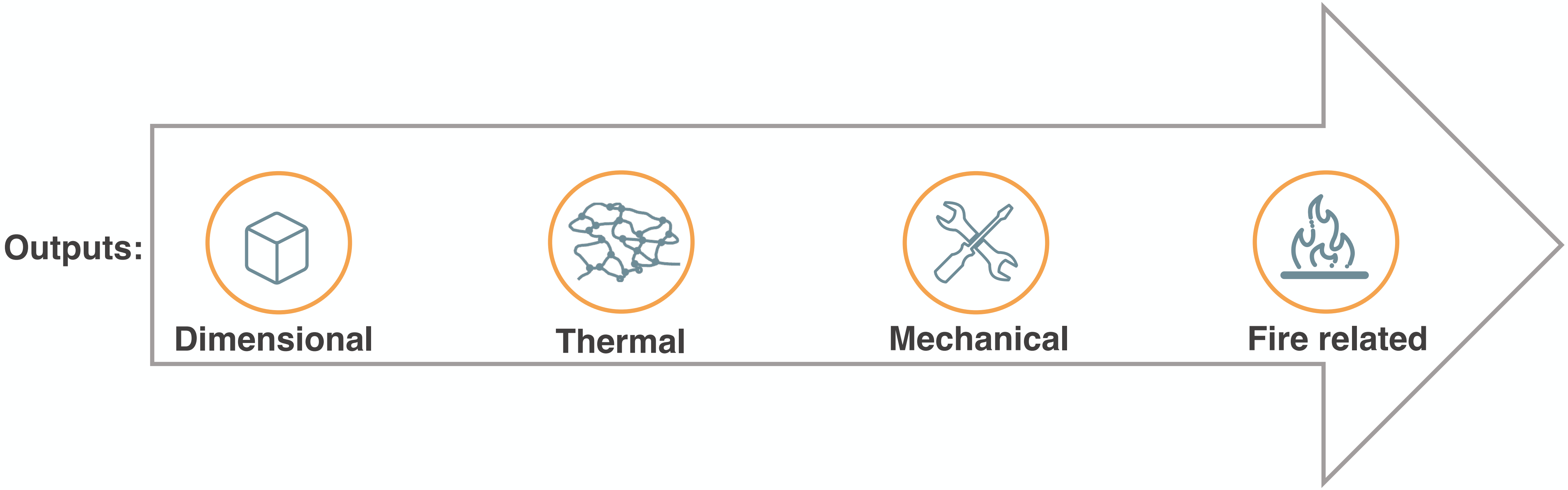


→ Minitab

Partial factorial design (Taguchi)
four factors → 4D:



Properties



Outputs

Outputs:

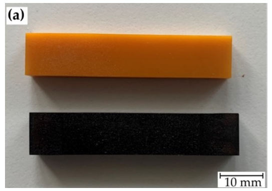
Dimensional

Thermal

Mechanical

Fire related

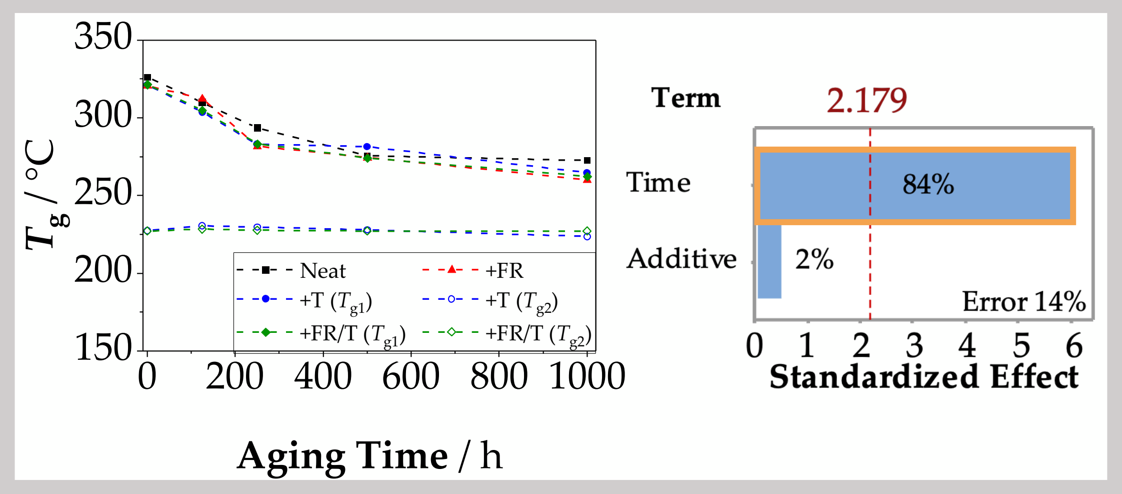
T_g



DMA specimens

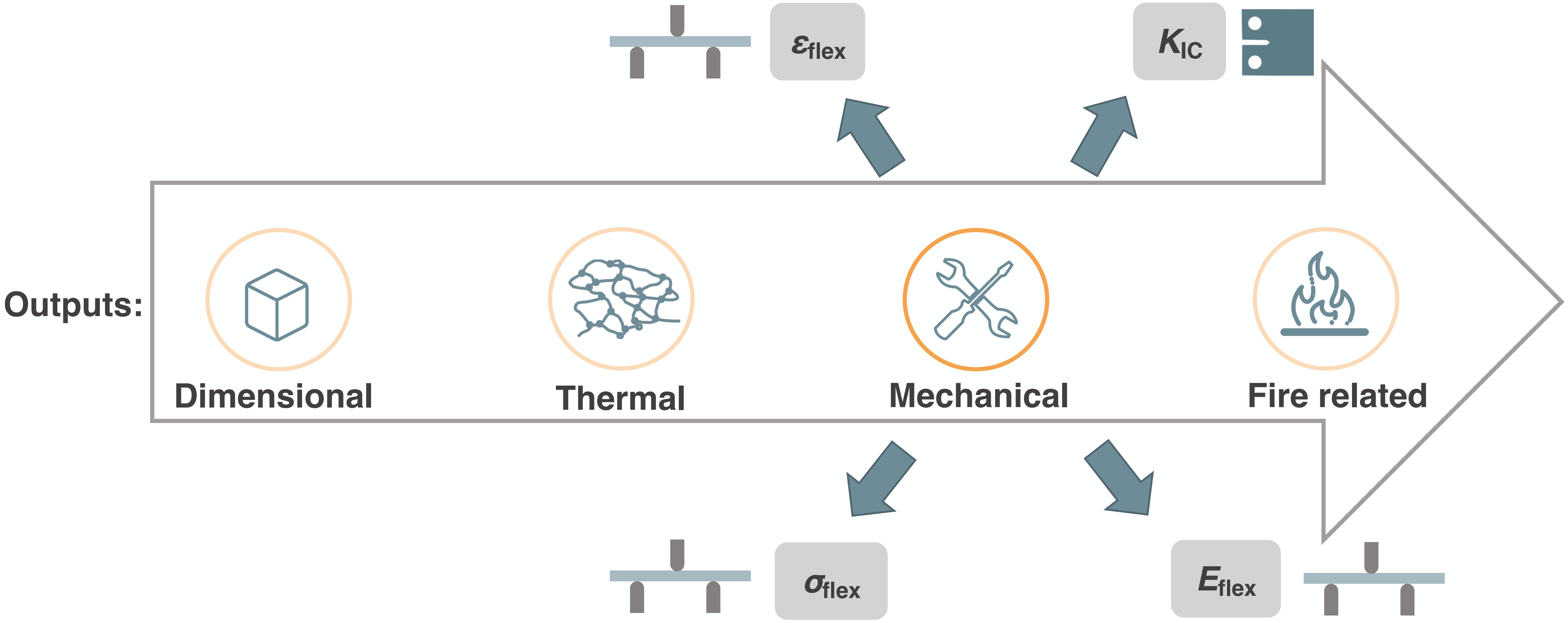
FWHM

Full Factorial DoE– Thermal Properties: DMA



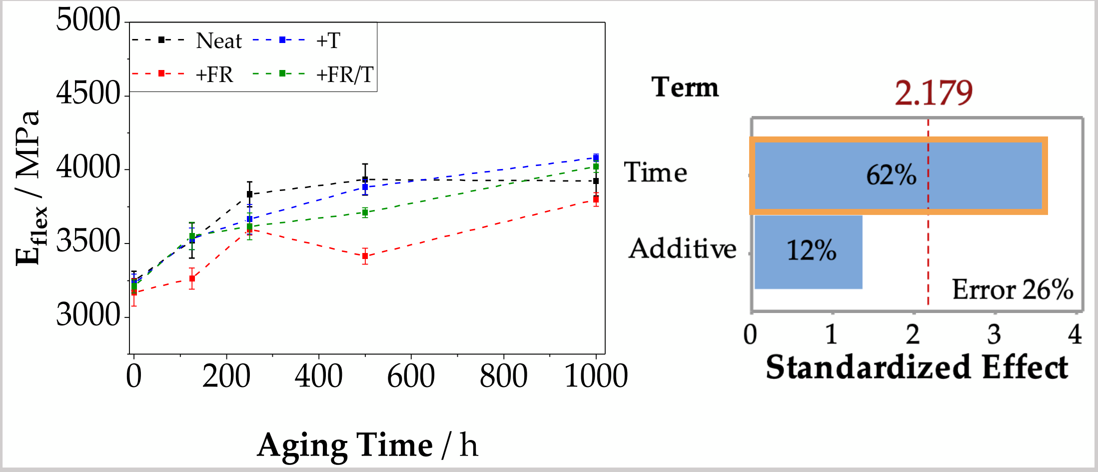
- Converging trends ~270 °C
- Significant factor: time
- No impact on T_g of the PES phase

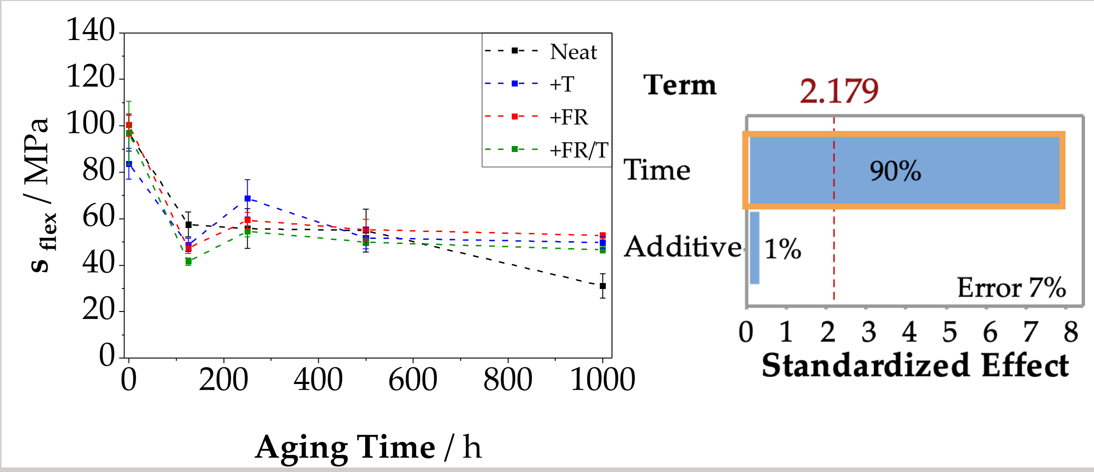
Outputs



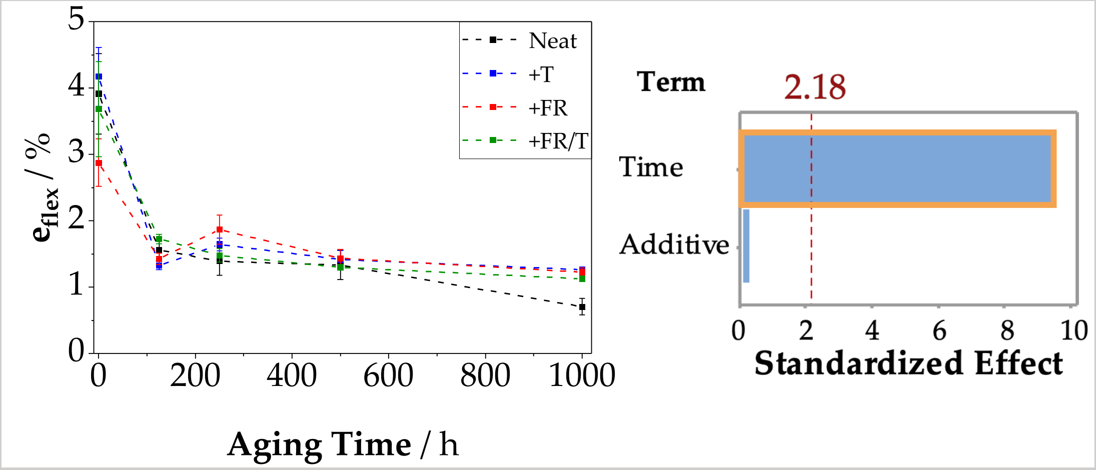
ε_{flex}
K_{IC}
Outputs:
Dimensional
Thermal
Mechanical
Fire related
σ_{flex}
E_{flex}

Full Factorial DoE– Mechanical Properties: 3-PBb





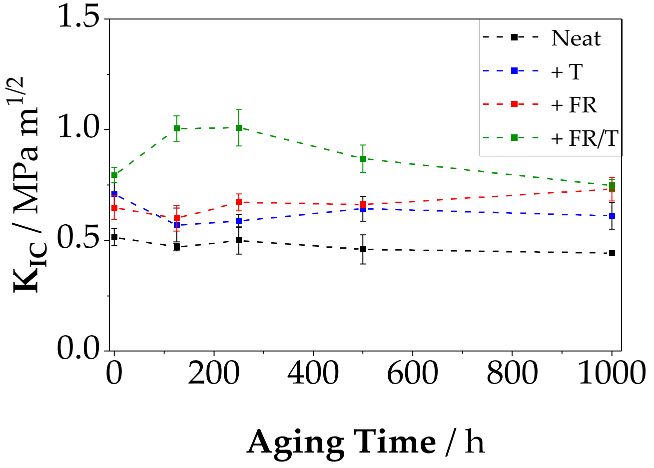
- Converging trends to ~4000 MPa
- Significant factor: time

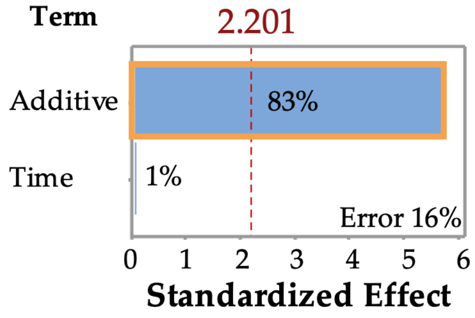


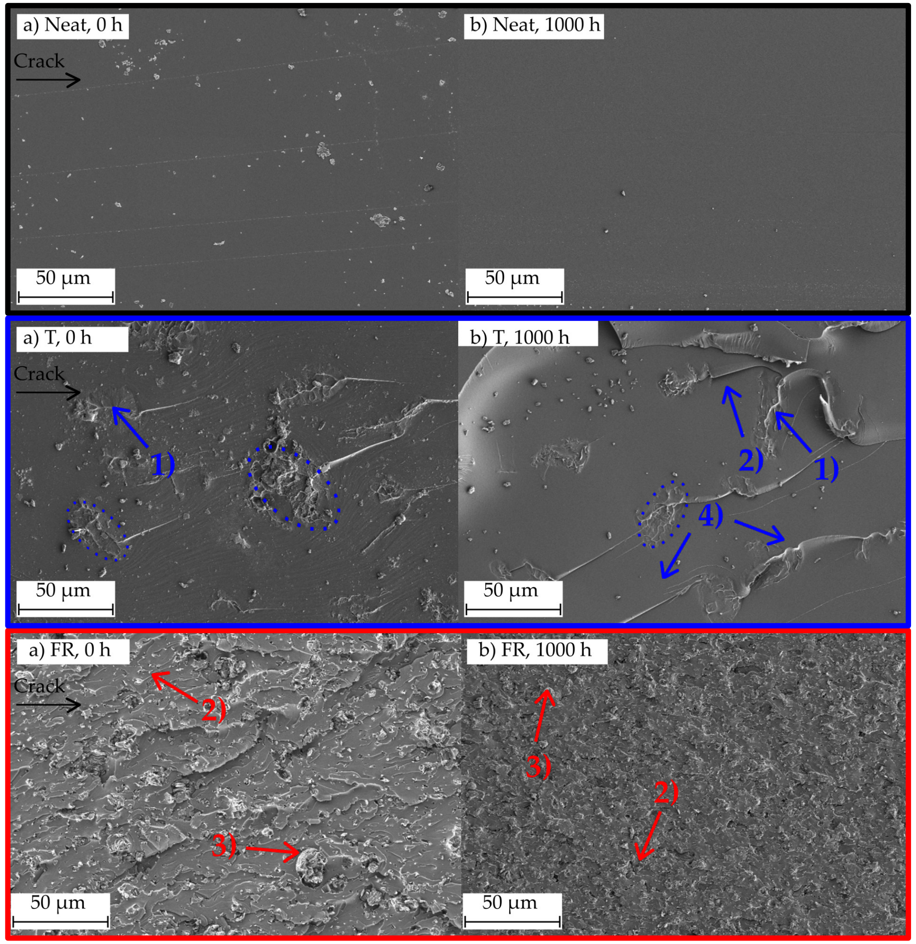
- Strong drop after 125 h
- Significant factor: time

- Strong drop at 125 h
- Significant factor: time

Full Factorial DoE– Mechanical Properties: CT



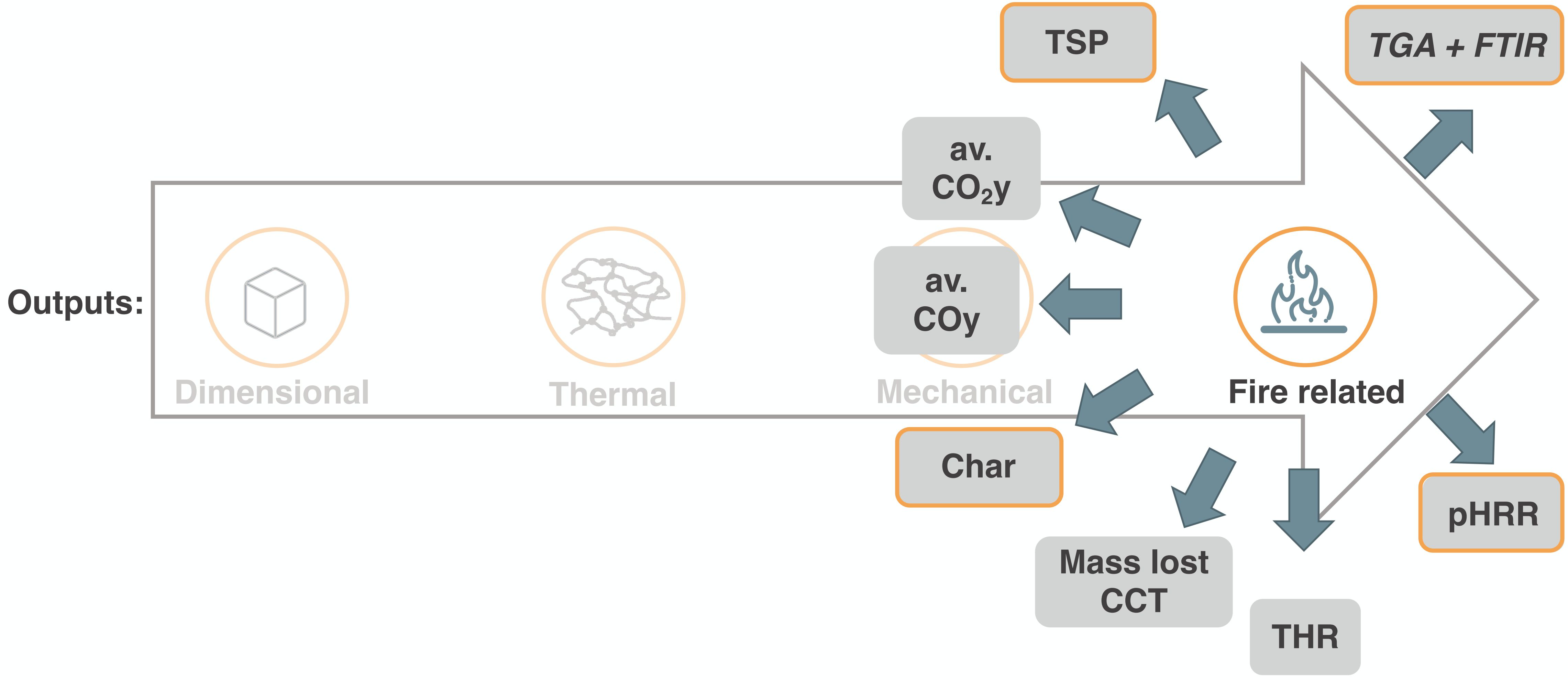




1) Crack-bridging
2) Crack-pinning
3) Crack path deflection
4) Particle-induced shear banding

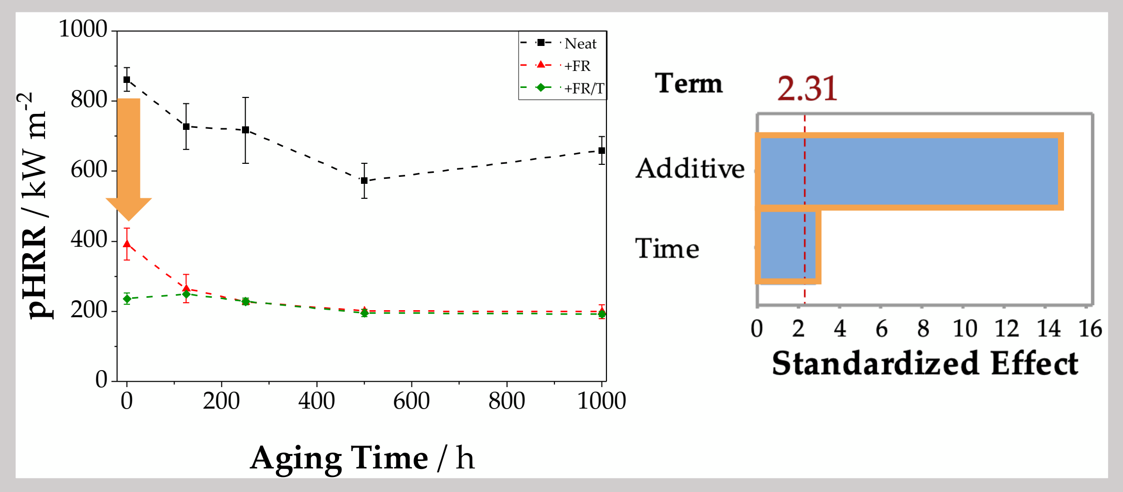
R. A. Pearson, A. F. Yee, "Toughening mechanisms in thermoplastic-modified epoxies: 1. Modification using poly(phenylene oxide)", Polymer 1993, 34, 3658–3670

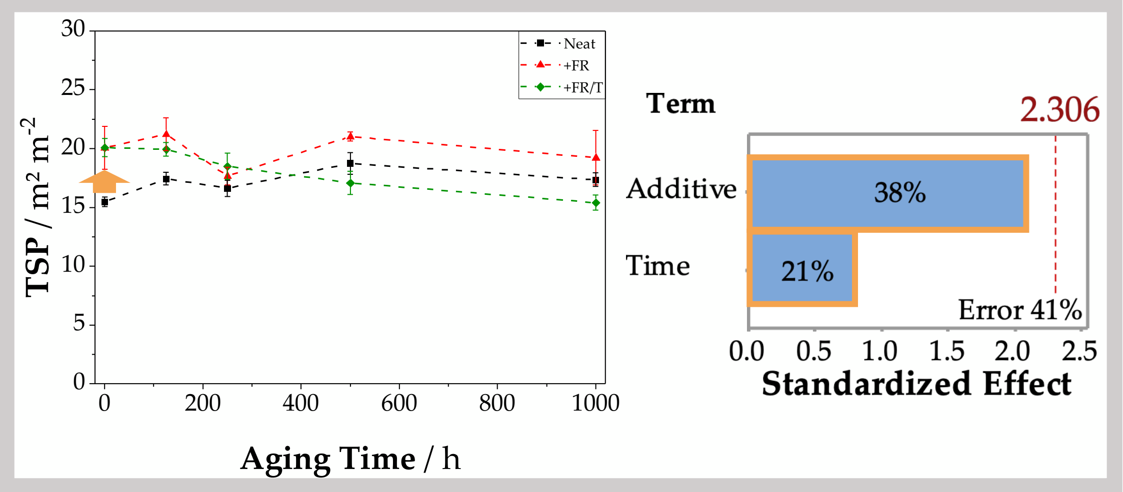
Outputs



Outputs:
Dimensional
Thermal
Mechanical
Fire related
TSP
TGA + FTIR
av. CO_2y
av. COy
Char
Mass lost CCT
THR
pHRR

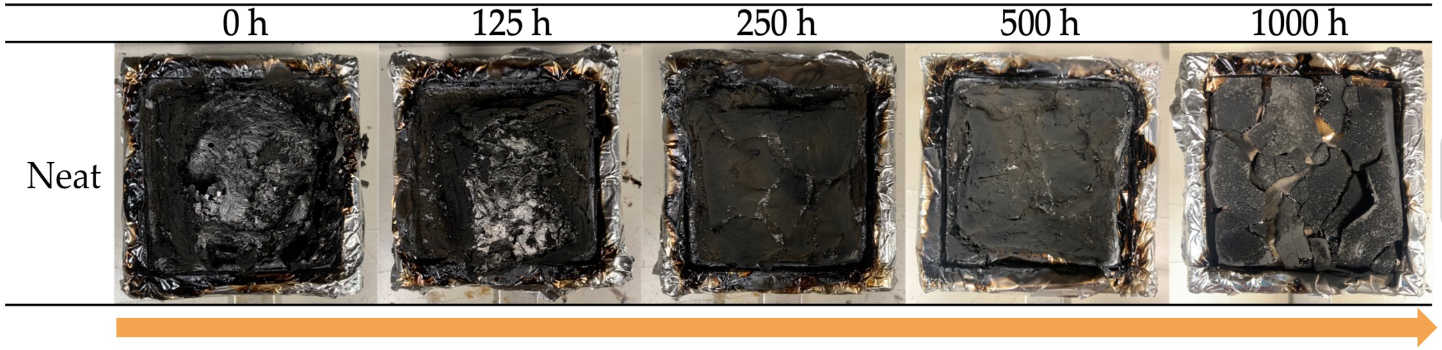
Full Factorial DoE – Fire Properties: CCT





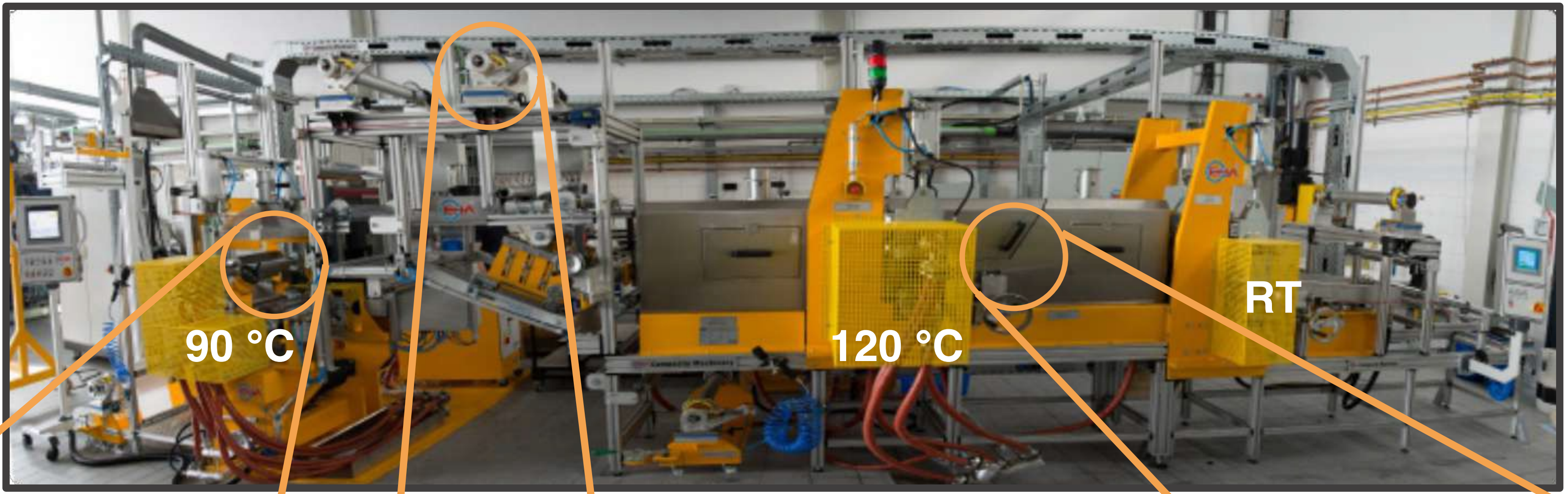
- Strong drop by adding FR and FR/T
- Significant factor: additive and time

- Enhanced smoking at 0 h
- No significant factors, large model error



- Lower intumescence

Prepreg Processing



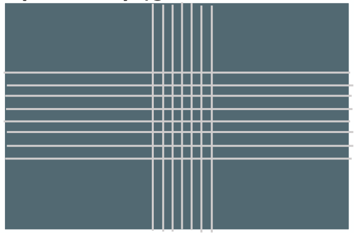
Style 7781 Glass-Fabric
satin 8H weave pattern
Weight 295 g/m²

Source (Helvetica, 8pt)

Laminate Manufacturing

Crossply Laminate

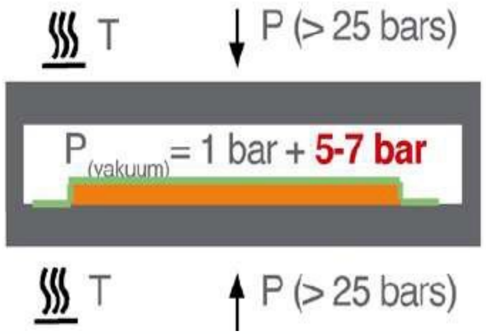
$(0/90)_{4s}$



Lay-up on heating table (50% FVC)

- for shear-loaded parts
- for pipes/ducts

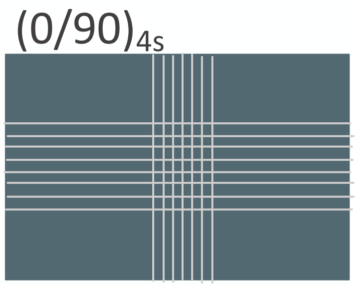
Curing Process



Curing Cycle:
1h 120 °C/3h 180
Free-Standing Tempering 1h at 250 °C

Laminate Manufacturing

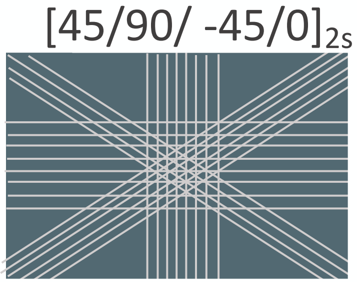
Crossply Laminate



- for shear-loaded parts
- for pipes/ducts

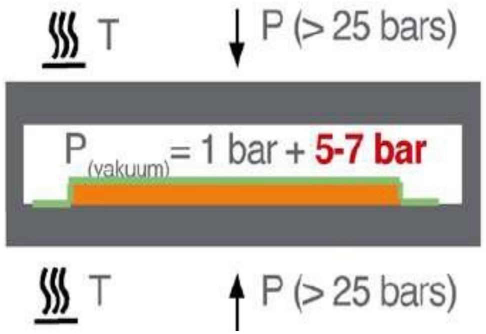
Lay-up on heating table (50% FVC)

Quasi-Isotropric Laminate



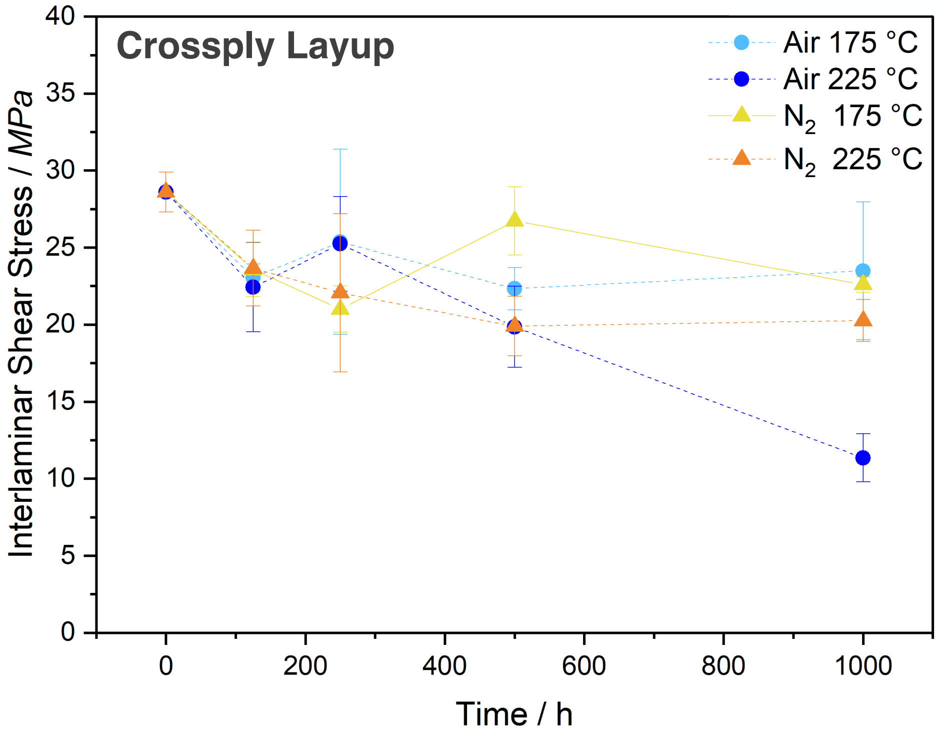
- Standard lay-up

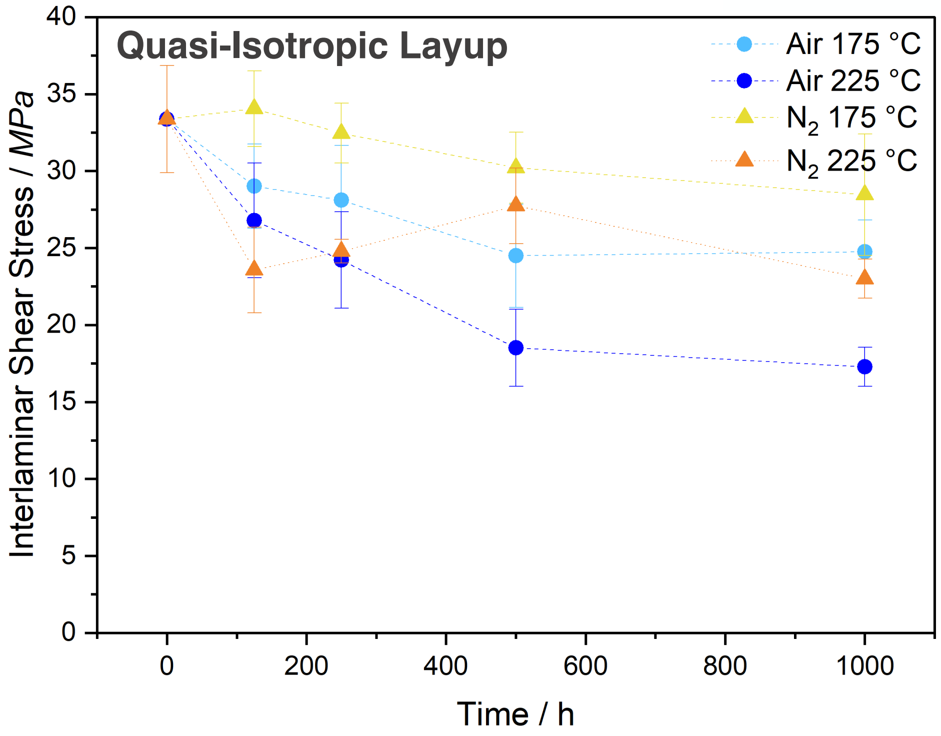
Curing Process



Curing Cycle:
1h 120 °C/3h 180
Free-Standing Tempering 1h at 250 °C

Influence of Aging on Interlaminar Shear Stress



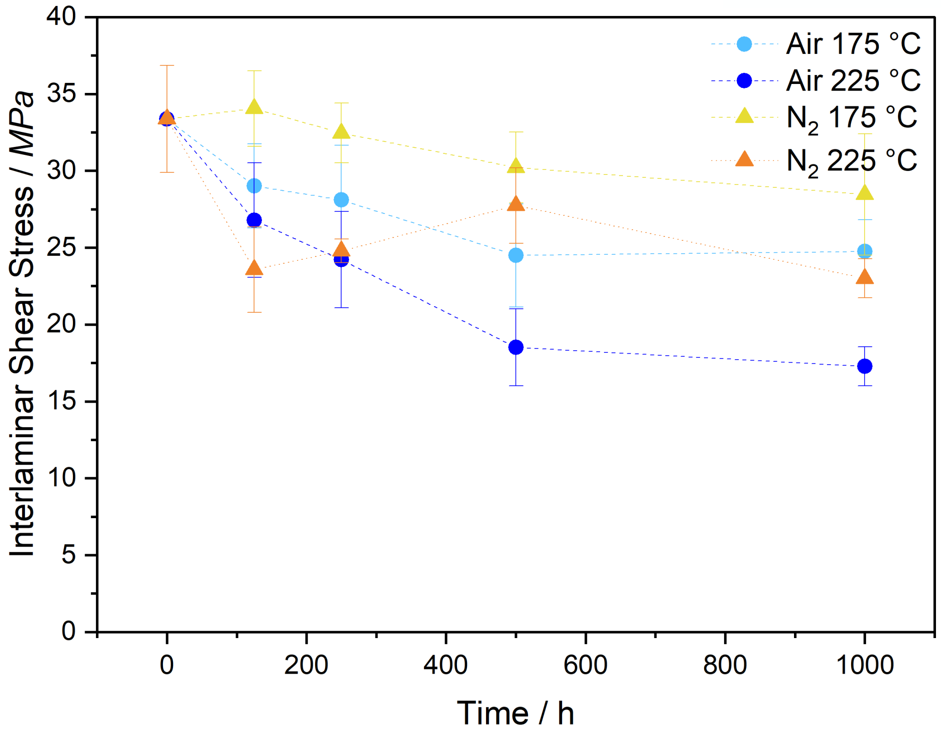


Stronger drop in crossply laminate due to higher thermal stress in 0/90 direction

Influence of Aging on Interlaminar Shear Stress

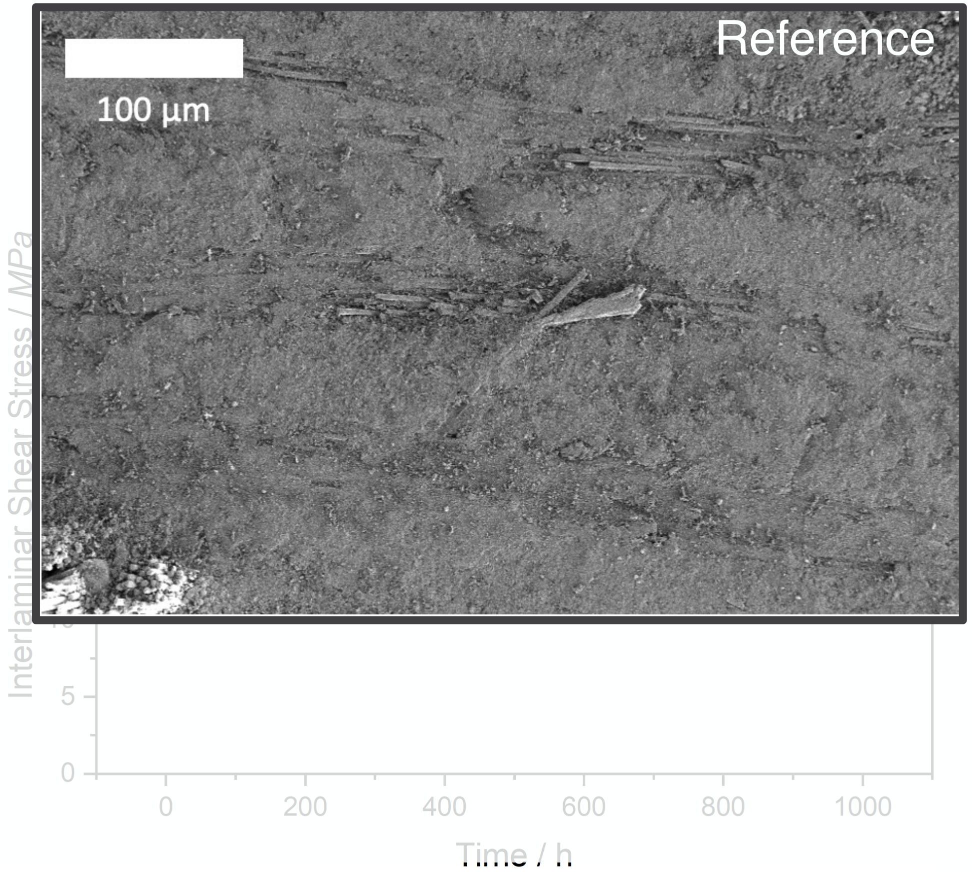
Crossply Layup

Quasi-Isotropic Layup

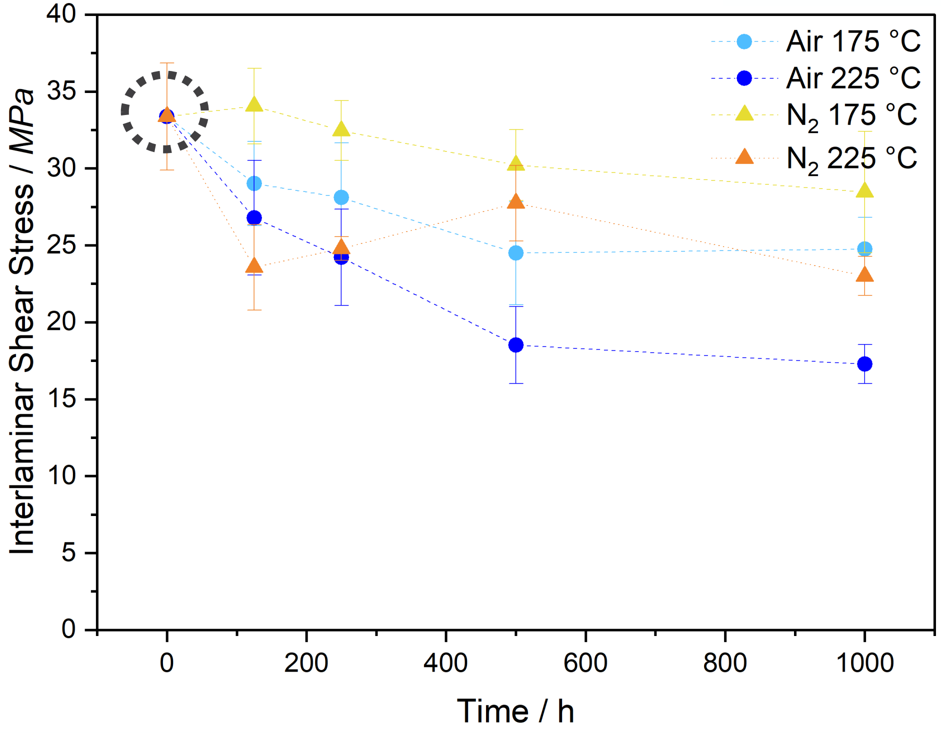


Stronger drop in crossply laminate due to higher thermal stress in 0/90 direction

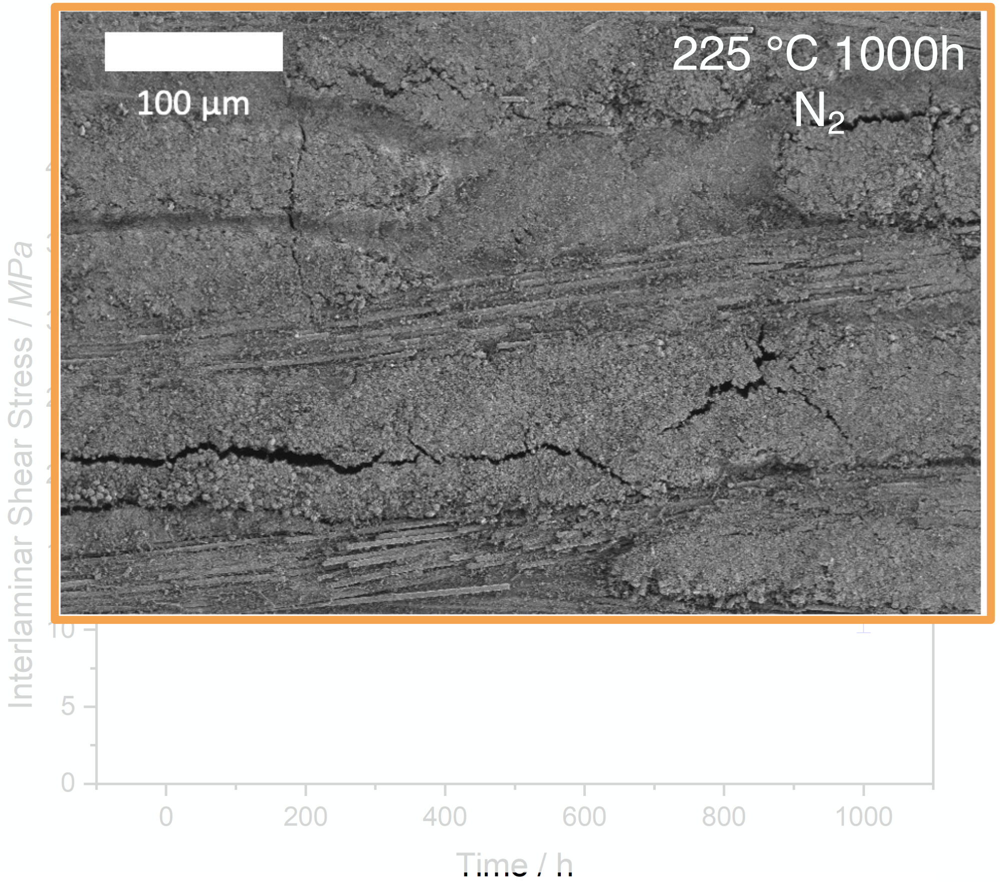
Influence of Aging on Interlaminar Shear Stress



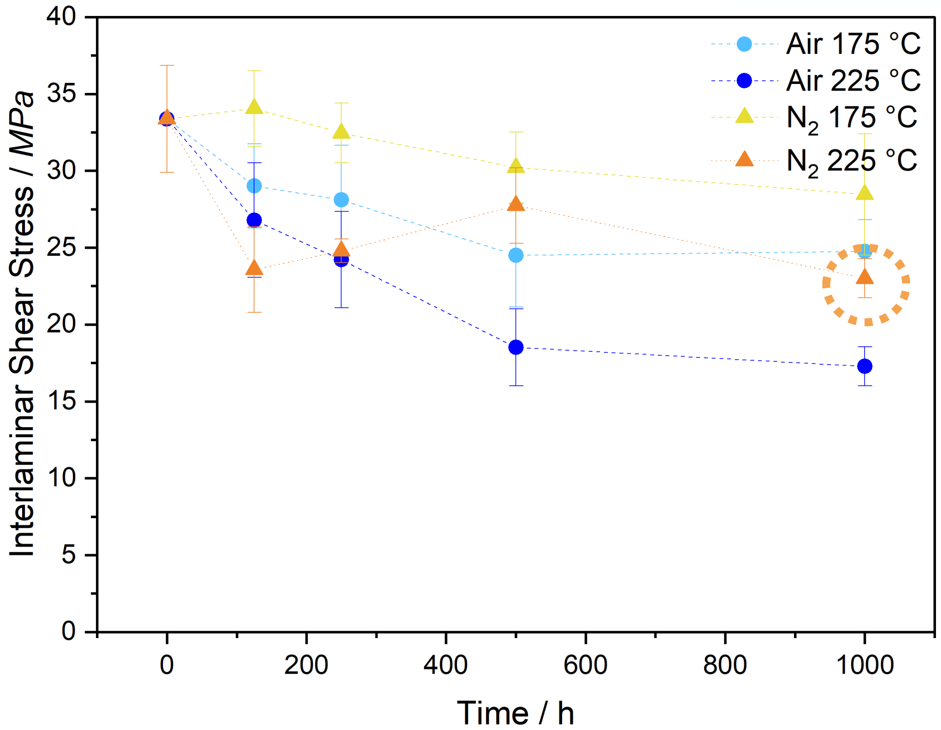
Quasi-Isotropic Layup



Influence of Aging on Interlaminar Shear Stress

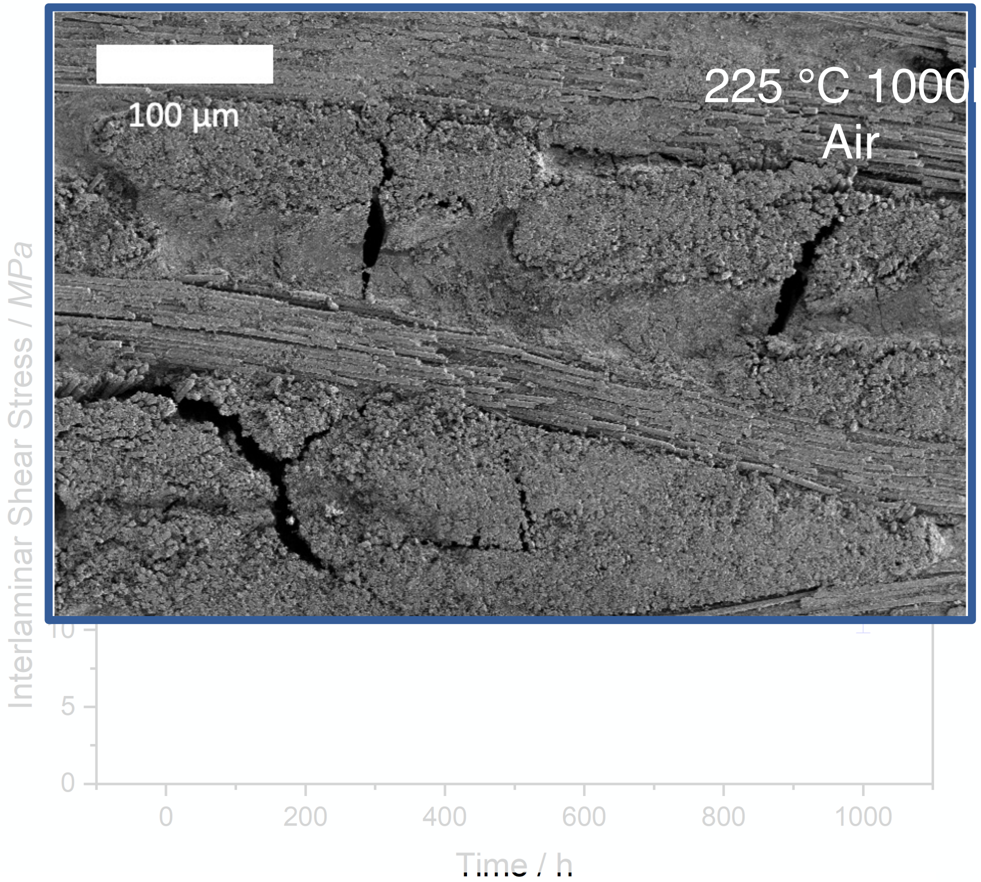


Quasi-Isotropic Layup

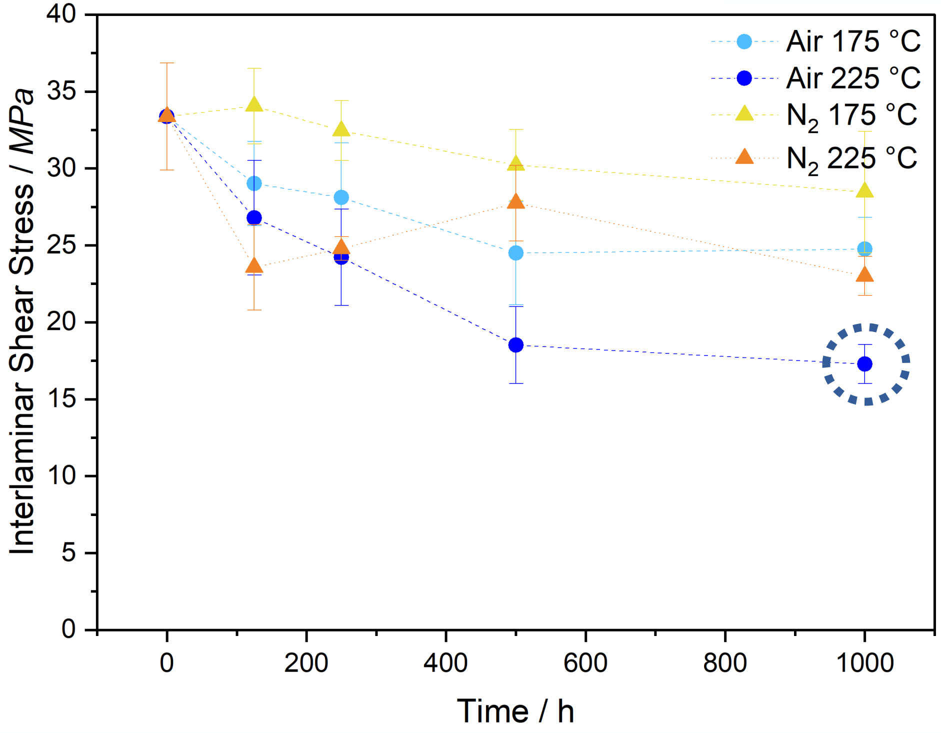


Minor cracks visible in nitrogen atmosphere

Influence of Aging on Interlaminar Shear Stress



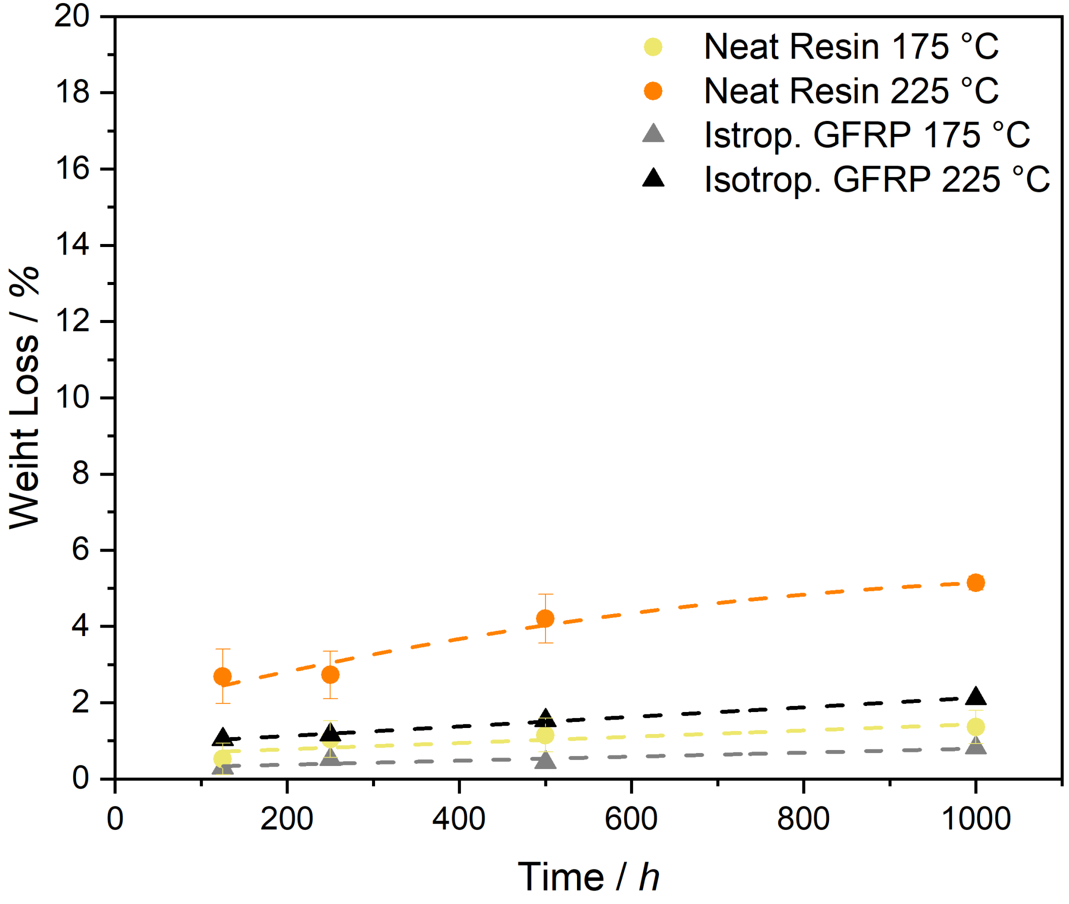
Quasi-Isotropic Layup



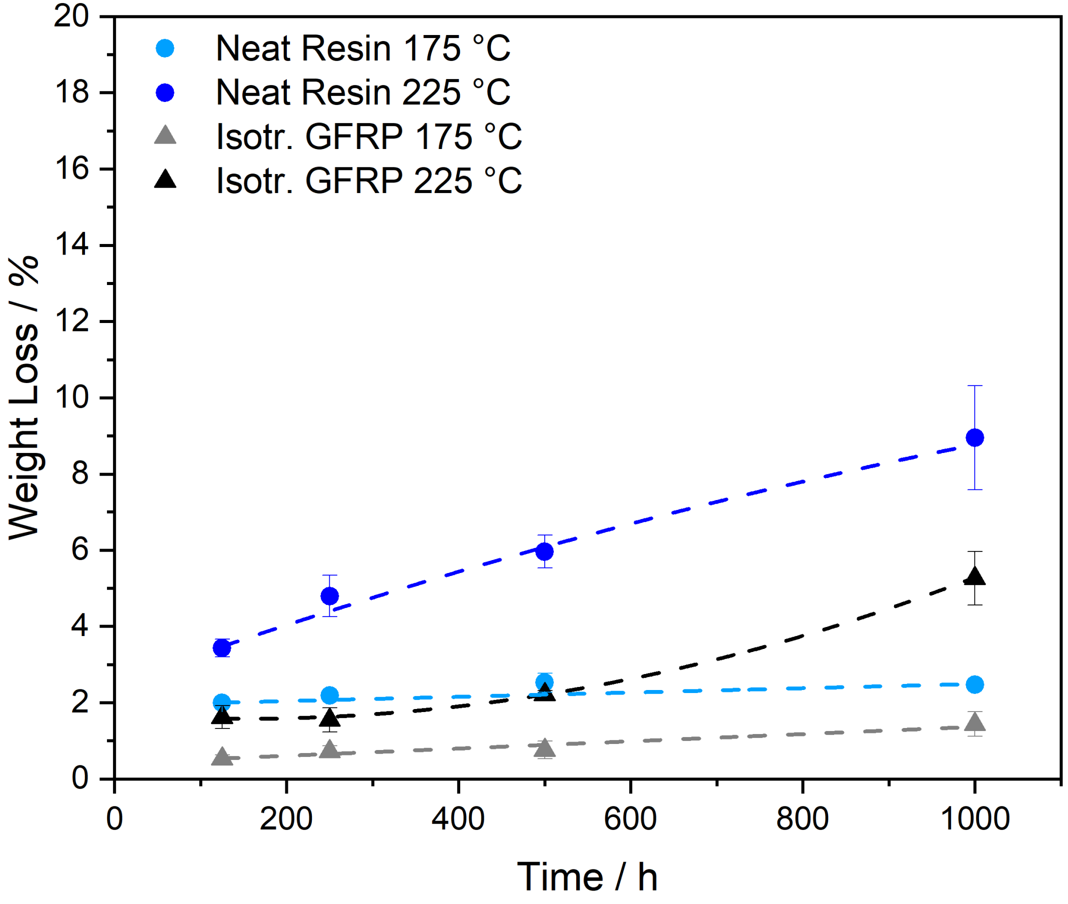
Wider cracks with higher occurence visible in air atmosphere

Influence of Fiber Reinforcement on Aging Behaviour

N_2 Atmosphere

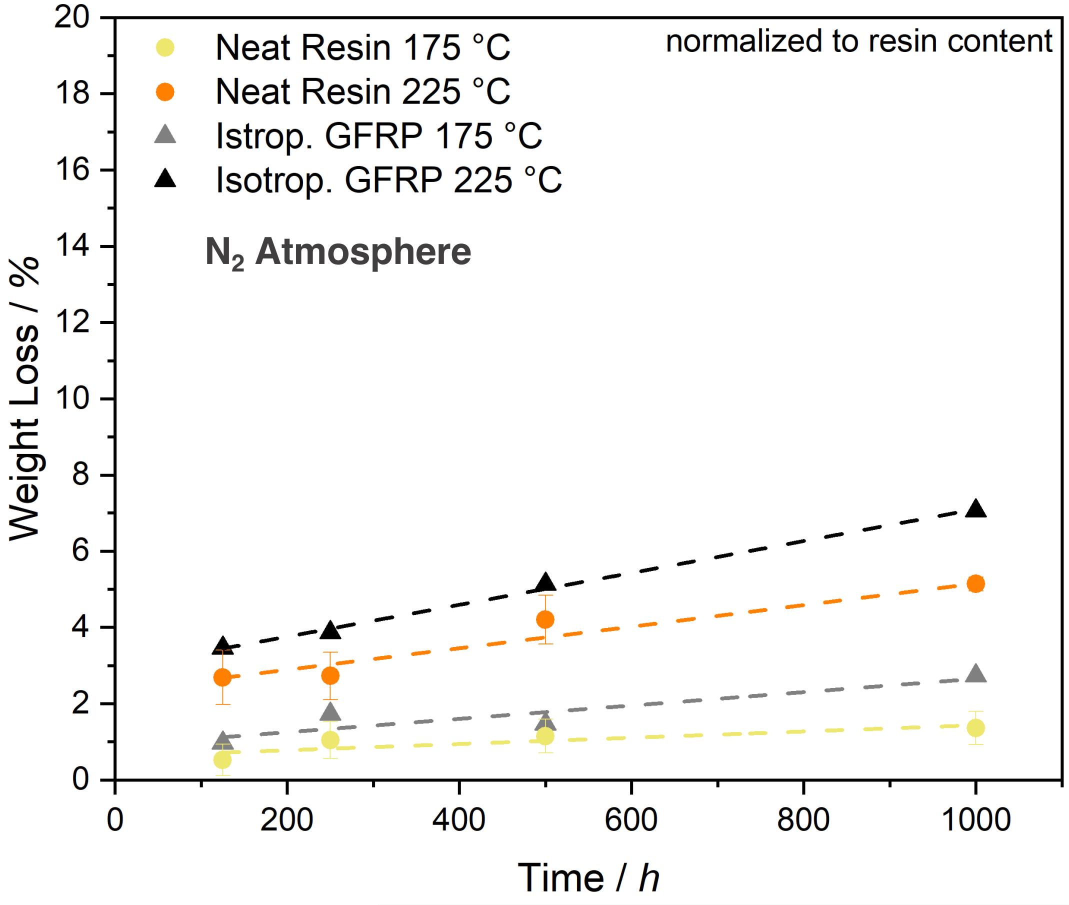


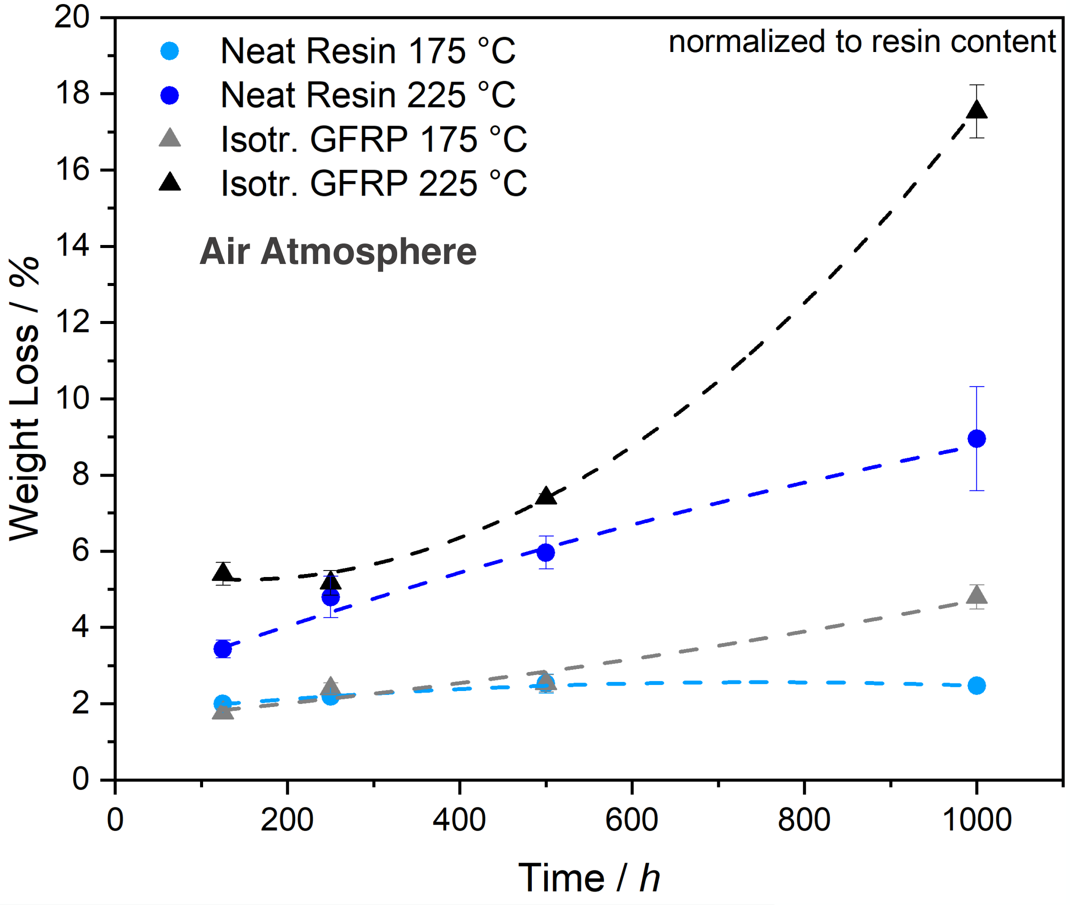
Air Atmosphere



Based on DMA specimen

Influence of Fiber Reinforcement on Aging Behaviour





Composite shows higher weight loss due to sizing degradation and pathways
Linear degradation in N_2, oxidation leads to higher weight loss in air

Based on DMA specimen

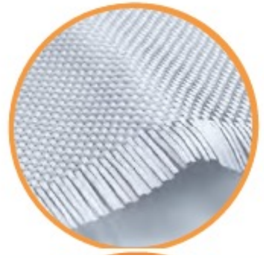
Summary of Results

I. High-T_g epoxy resin system developed with temperature-stable additives suitable for prepreg processing

II. Influence of toughener and flame-retardant on thermal degradation, mechanical properties and flame retardant properties

III Influence of fiber-reinforcement on aging behavior determined

Next Steps…



Determination of the influence of additives on the isothermal aging behavior on GFRP and CFRP composite

Determination of the influence of additives on the temperature cyclic aging behavior on GFRP and CFRP composite

Life time prediction for neat, additivated resin system and laminate with *Netzsch Kinetics Neo*

Questions?

Aging...
You can‘t avoid it.....