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CUBIC
NOVEL BIOBASED MATERIALS TO IMPROVE CIRCULARITY

Thermoforming of epoxy vitrimer thermosets

08/10/2025

Virginie Boucher

Circular Bio-based Europe Joint Undertaking
Bio-based Industries Consortium
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Outline

1. *Thermoforming process*
2. *Thermoplastic and thermoset materials*
3. *Covalent adaptable networks*
4. *Recycling of 3R composites through thermoforming*

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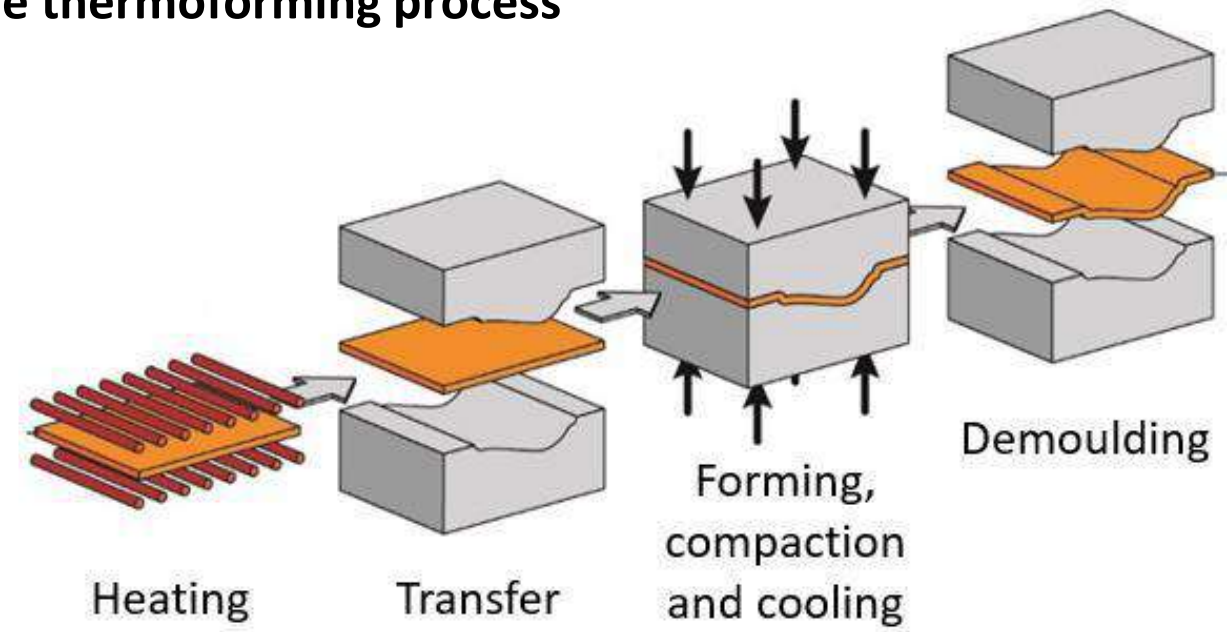
What is thermoforming?

Schematic of the thermoforming process

Carbon fiber composite laminates



Example of thermoformed carbon fiber composite part

Process:

1. Heat the composite laminate
2. Transfer the hot laminate to a press with the tool mounted
3. Close the press to form the hot laminate to the tool geometry
4. Apply pressure and wait for the formed part to cool down to a temperature low enough to freeze the material.
5. Open the press and demold the part

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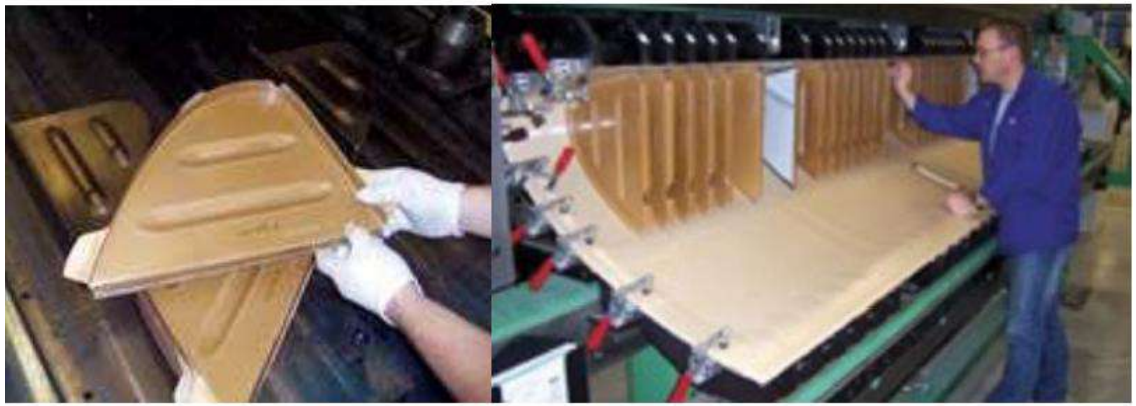
What is thermoforming?

Advantages: fast cycle times, design flexibility, lightweight parts, and potential for large-scale/automated production.

Applications: aerospace interior panels, automotive body panels, protective equipment, consumer goods.

Thermoformed brackets (left) connecting the fuselage frame and skins in the A350 XWB (right)



Thermoformed rib (left) being welded to the leading edge skin (right) of the A380

Materials: limited to thermoplastic composites

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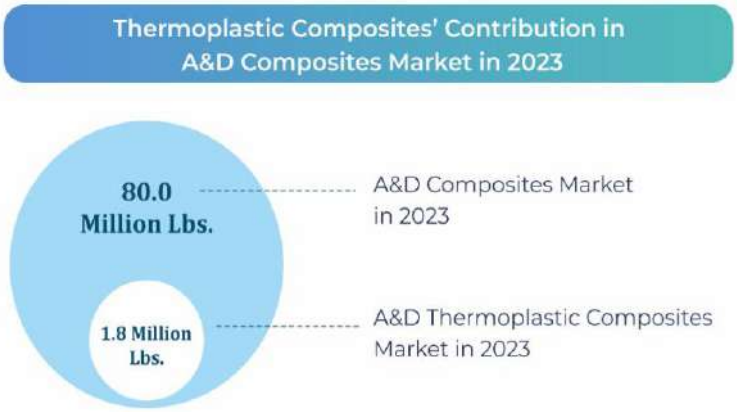
Thermoset composite materials

Thermoset composites

Market

Used in the **majority of advanced composites applications**
Over 90% of the carbon fiber reinforced polymer (CFRP) market



https://www.composights.com/article/Thermoplastic-Composites-Taking-off-into-a-Clearer-Future

Strengths

Excellent dimensional stability
High temperature resistance
Low viscosity during processing
Good fiber wet-out
Established certification history

Processing drawbacks

Longer processing times (manual or automated layup of individual plies, followed by resin transfer (if not using prepreg), and eventually the curing of the resin.

Cannot be remelted or reshaped after curing -> not thermoconformable

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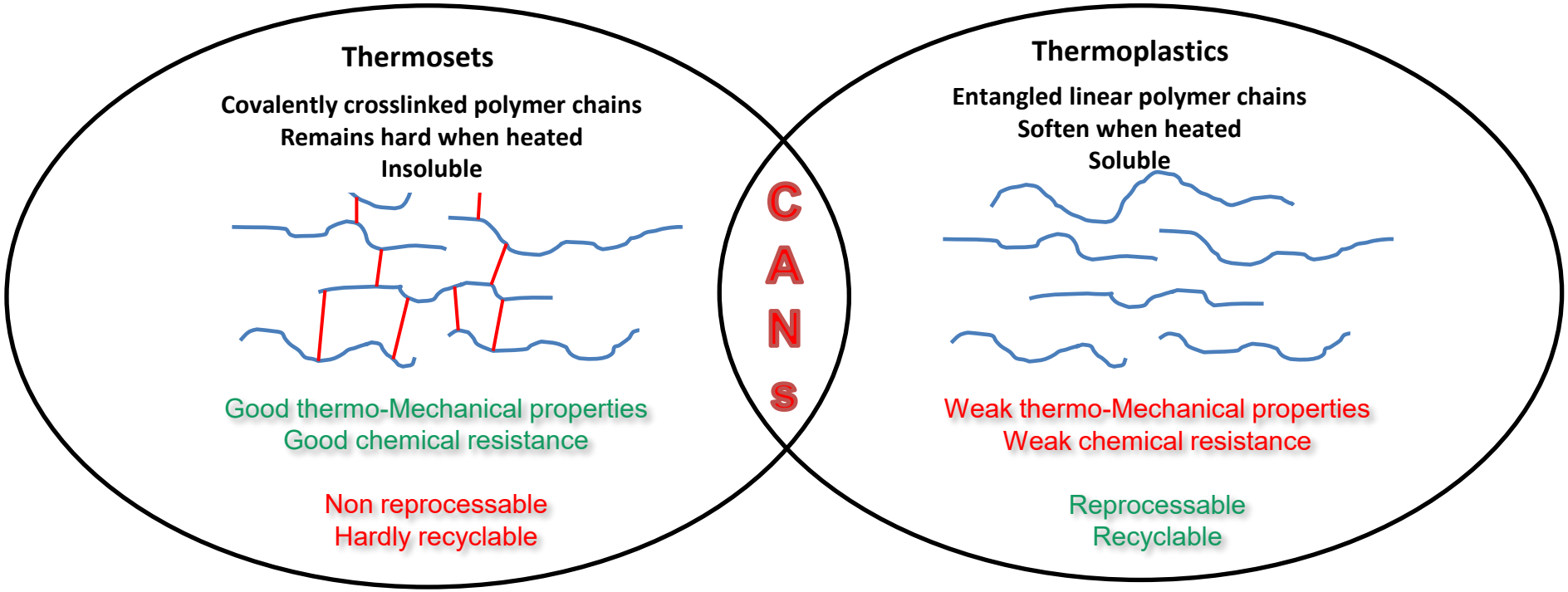
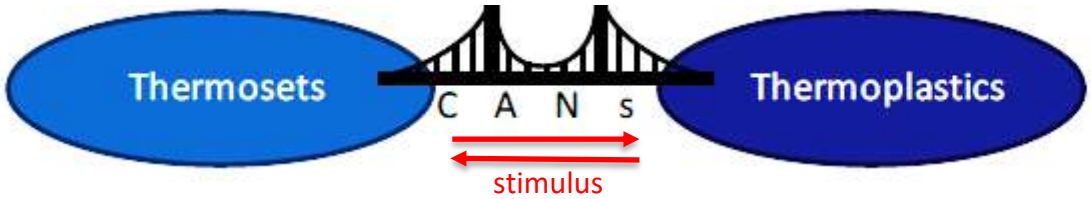
Thermosets versus thermoplastics





CANs = Covalent Adaptable Networks

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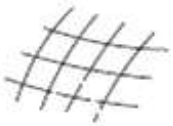
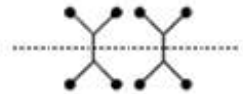
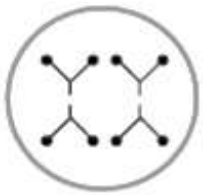
Covalent Adaptable Networks

Conventional thermosets

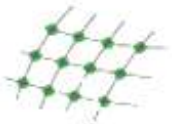


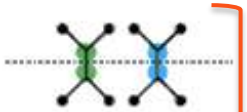


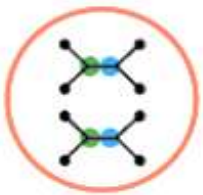


Permanent crosslinks

CANs







*Example of epoxy vitrimers based on **aromatic disulfide exchange***

Reprocessing of cured composite laminates

Reprocessable

Easy repair of delamination damages applying local heat and pressure.

Repairable

Mechanical recycling applying heat and pressure to obtain second generation parts.

Easy matrix disruption for high quality fiber recovery.

Recyclable

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Thermoforming of 3R composite materials

MC4 – Multi-level Circular Process Chain for Carbon and Glass fiber Composites

April 2022 → March 2025

Funded by the European Union

MC4 gathered **16 partners from 9 different European countries**, covering the whole value chains: process developers, material manufacturers, end users manufacturing the composite parts. The consortium was led by Profactor (Austria).

To establish a **multi-level circular process for carbon and glass fiber composites**	To develop **performant and economically realistic processes** that are adapted to the specificities of the two value chains	To give to the European industry the means to **master and own its patented manufacturing processes** of recycled materials

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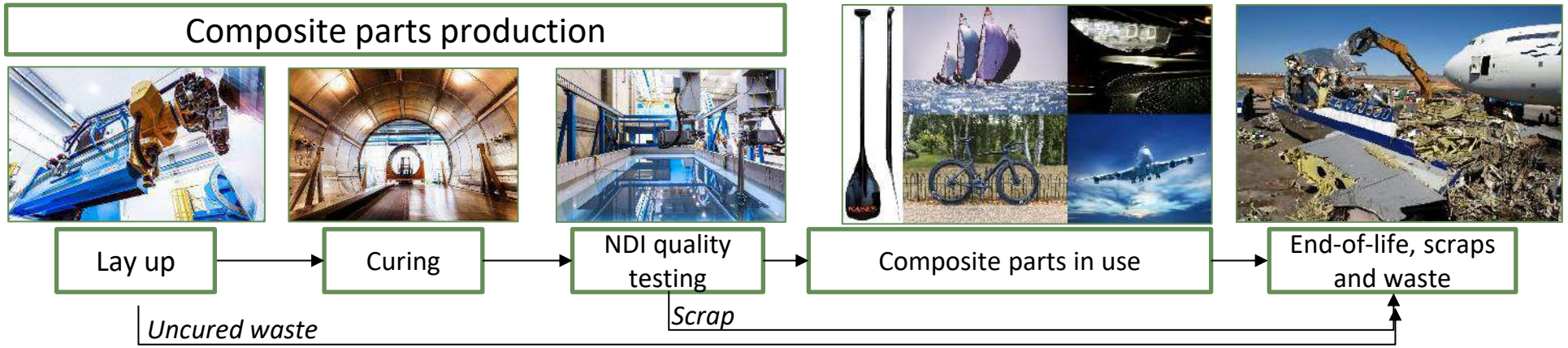
Thermoforming of 3R composite materials

Today's typical composites value chain:



MC4's pathways for recycling:

CF

- The re-use of uncured carbon fiber scrap material directly in the production line to reduce the amount of scrap generated during manufacturing as a short-term solution — *Short term*
- Chemical recycling as a long-term pathway to regain valuable carbon fiber from end-of life parts and convert them into yarns, fabric and nonwoven material for new parts — *Long term*

GF

- The shredding of glass fiber composites and the re-used in new parts, which will in the short term reduce the amount of virgin material needed for such parts. — *Short term*
- The use of a vitrimer resin, which enables the reshaping of parts at their end of life and provides a long-term solution for glass fiber end-of-life parts — *Long term*

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Thermoforming of 3R composite materials

Approach: use of a new reshapeable resin for easier recycling

- Formulation of a 3R epoxy resin
- Manufacturing and characterization of GF reinforced 3R epoxy composites
- Lab-scale optimization of the reshaping process
- Transfer of the process to end-user toward manufacturing trials

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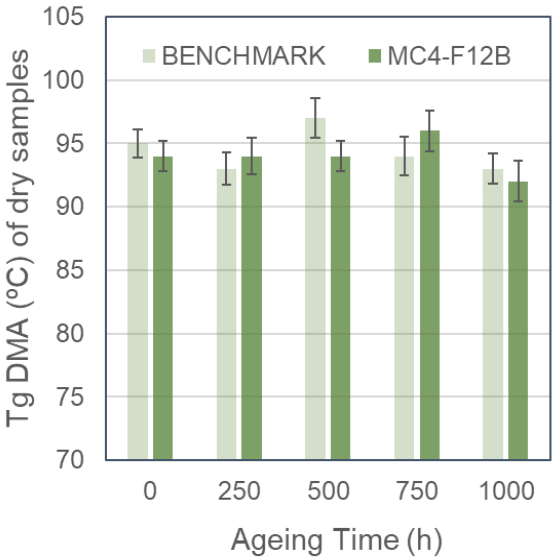
Thermoforming of 3R composite materials

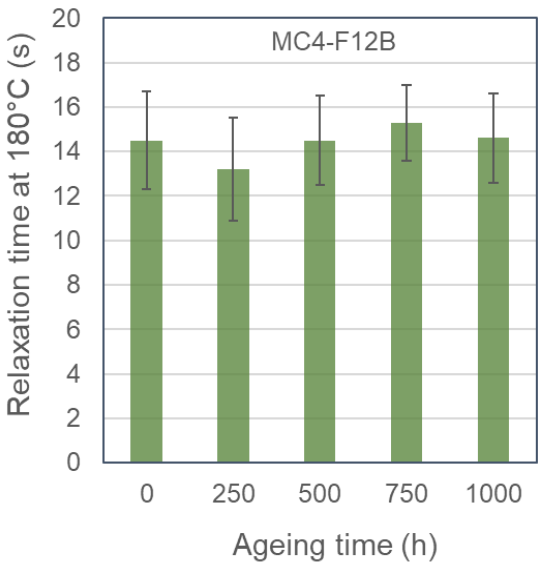
Formulation & Selection of the 3R resin with best-balanced overall performance

MC4

Requirements			MC4-F12B	Benchmark resin
SUITABLE FOR INFUSION	Viscosity (mPa.s)	at 25ºC	988	598
		at 40ºC	218	80
		at 60ºC	54	23
	Working time (min), at 80ºC	G'=G'' (gel point)	-	38
		1000 mPa.s	237	-
		300 mPa.s	195	-
	Infusion temperature (ºC)		60	35
	Curing cycle		1.5h 130ºC + 1h 150ºC	1.5h 80ºC + 5h 100ºC
SUITABLE FOR KAYAK APPLICATION	Tg (ºC)	DSC/DMA	81/94	86/91
	Tensile	Strength (MPa)	80.4	70.3
		Elongation at break (%)	6.15	6.4
	Flexural	Strength (MPa)	138	114
		Elongation at break (%)	6.1	6.25
RESHAPEABLE VIA THERMOFORMING	Stress relaxation (1% strain)	at 180ºC	13''	No relaxation
		at 160ºC	62''	
		at 140ºC	308''	

Retention of key properties over time
(weathering acc. to ISO-4892-2 standard)





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Thermoforming of 3R composite materials

Mechanical properties of 3R GF composite laminates

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Manufactured by Resin Transfer Molding (RTM) with:

- GF fabric and 3R resin;
- orientation of GF fabric as per standard tests methods.

Validation of kayak and paddle demonstrators' designs by the end-user.

Group of properties	Testing standard	Properties	Unit	Value	Standard deviation
Tensile Properties (0º)	ISO 527-4	Modulus	GPa	21.6	0.7
		Ultimate stress	MPa	436.0	22.8
		Ultimate strain	%	1.7	0.1
		Poisson ratio	-	0.122	0.01
Tensile Properties (90º)	ISO 527-4	Modulus	GPa	20.8	0.4
		Ultimate stress	MPa	369.0	14.8
		Ultimate strain	%	1.7	0.1
		Poisson ratio	-	0.137	0.03
Compression Properties (0º)	ASTM D 3410	Modulus	GPa	27.4	1.0
		Ultimate stress	MPa	238.0	24.4
		Ultimate strain	%	8.2	0.9
Compression Properties (90º)	ASTM D 3410	Modulus	GPa	24.8	0.2
		Ultimate stress	MPa	248.1	11.2
		Ultimate strain	%	7.1	0.6
In Plane Shear	ISO 14129	Modulus	GPa	3.2	0.1
		Strength at 5 % elongation	MPa	48.3	1.5
Interlaminar Shear Strength (0º)	ISO 14130	Ultimate stress	MPa	40.4	0.9
Interlaminar Shear Strength (90º)	ISO 14130	Ultimate stress	MPa	43.0	1.2

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3R material reshaping possibilities– Flattening of the recycled parts

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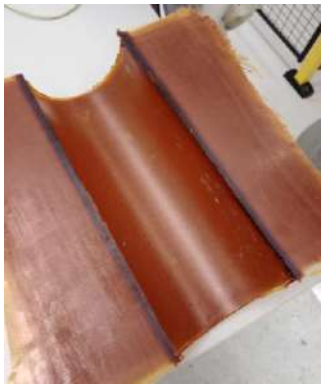
Vacuum Infusion of curved 3R composite laminates

Infusion trials realized on curved surfaces to get curved composite laminates to check the possibility to obtain flat laminates from curved laminates such as the recycled ones that will be used to manufacture the paddle through thermoforming.

After curing of 3R resin infusion

3R resin/Glass fiber curved laminate



Flat and curved laminate samples, after trimming

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Evaluation of the mechanical properties of 3R glass fiber composite after weathering acc. to ISO4892-2 A1:2004-plastics

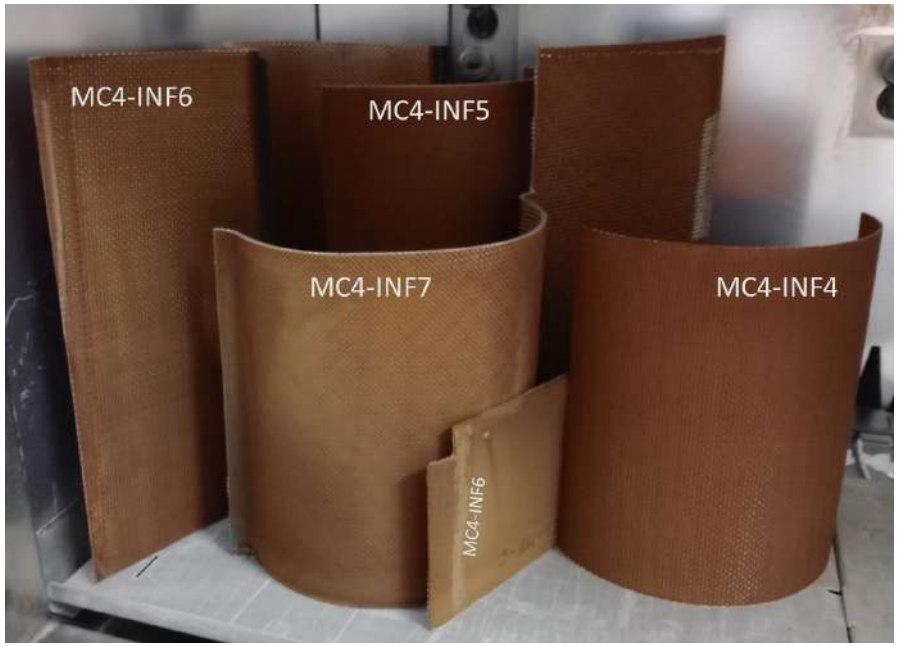
MC4

- Weathering conditions:

Wet aging equipment	CI 4000, Atlas
Dry aging equipment	Suntest XLS+, Atlas
UV source	Xenon, 60 W/m2, between 300 and 400 nm
Wet/dry cycles	18 min/102 min (ISO 4892-2 Table 3)
Temperature	65ºC, at blackbody sensor

- Position of the samples changed every 250 h for homogeneous UV and rain exposure.

- Duration: 1000 hrs.



Samples in the weathering chamber, after 1000 hrs weathering

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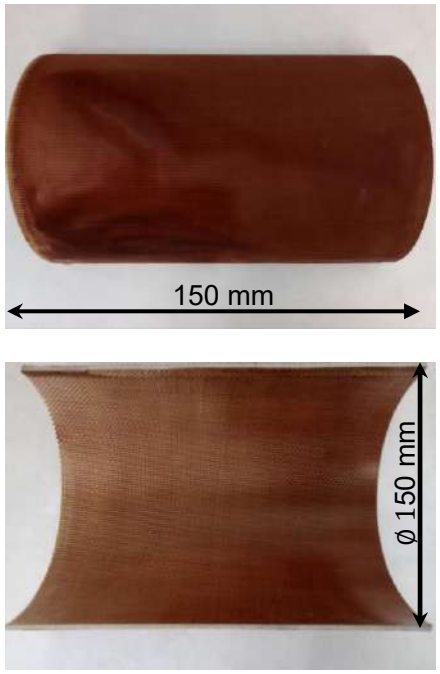
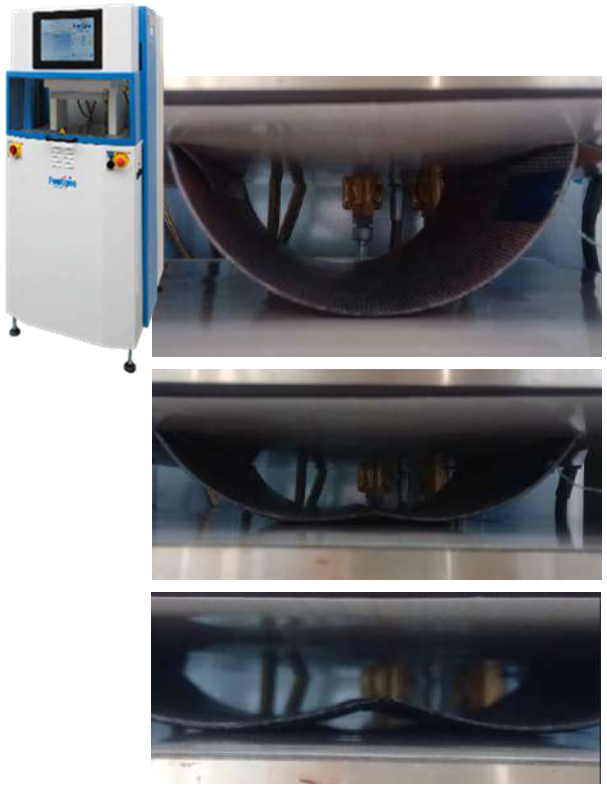
Thermoforming of 3R composite materials

Preliminar flattening of 3R glass fiber composite after weathering acc. to ISO4892-2 A1:2004-plastics

MC4



Initial curved laminate, after aging



During flattening in hot plates press

200ºC, 40 bars, 50 mm/min

Aged and flattened laminate

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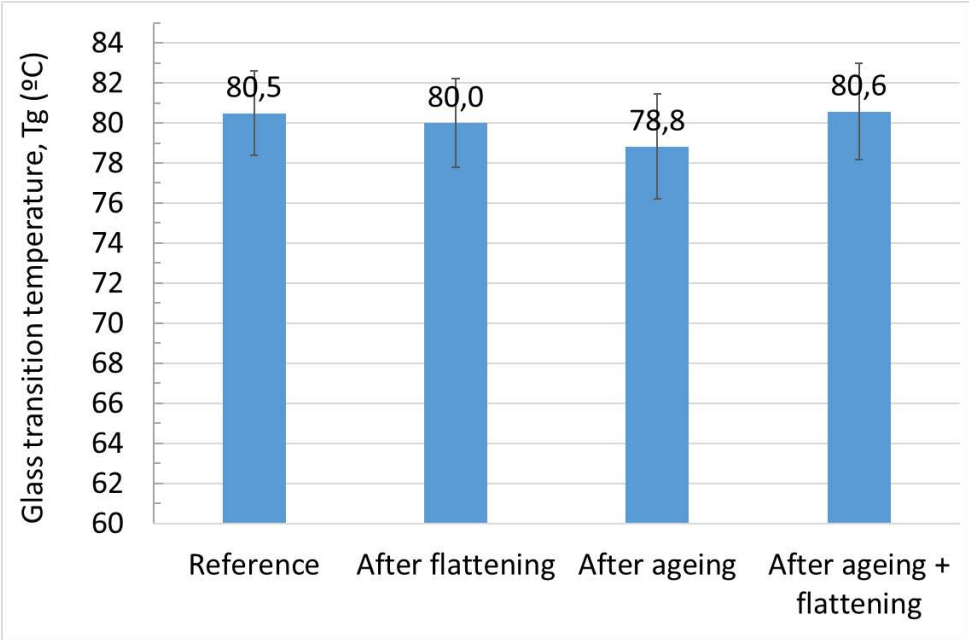
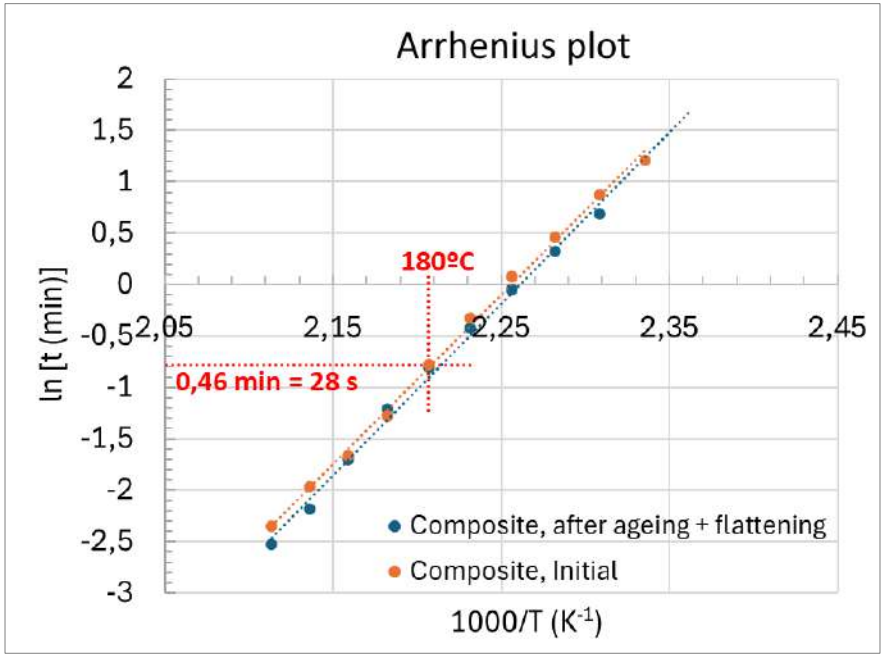
Evaluation of the properties of 3R glass fiber composite after weathering & flattening

MC4

Glass transition, DSC



Stress relaxation, DMA



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Thermoforming of 3R composite materials

Evaluation of the properties of 3R glass fiber composite after weathering & flattening of 0° laminates (3 mm thick)

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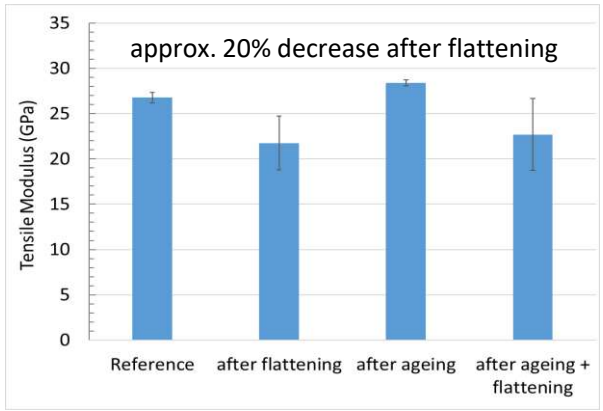
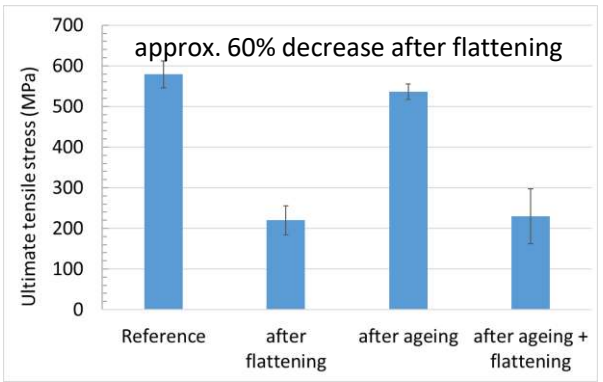
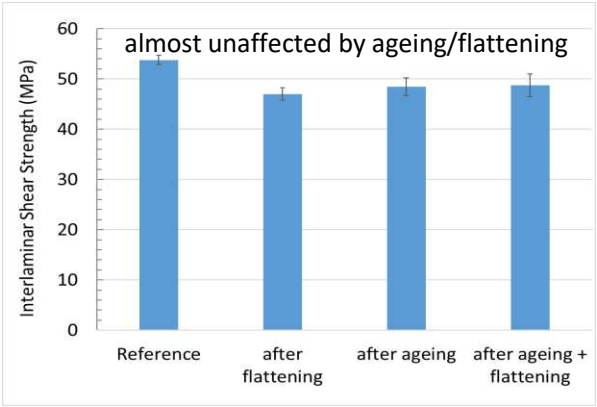
0º Aged and flattened laminate

Tensile test acc. to ISO 527-4





ILSS (0º) acc. to ISO 14130

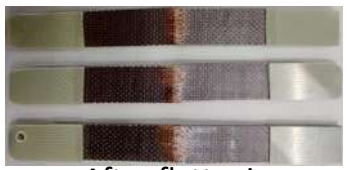


No effect of the 1000-hrs weathering

Due to the **formation of wrinkles** with breakage of GF, flattening **greatly affects tensile properties**.

Typical breakage during tensile testing :


Reference


After flattening

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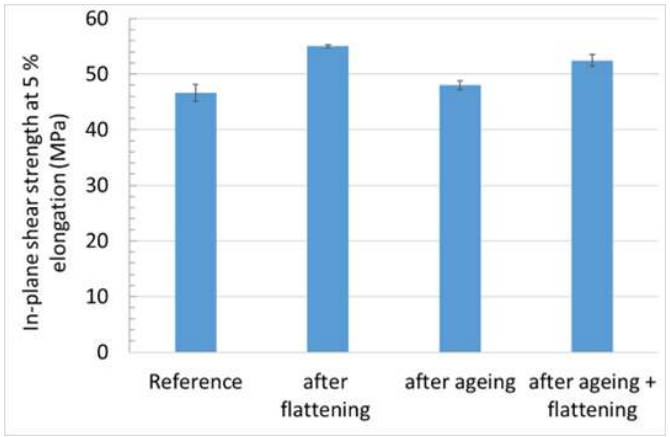
Thermoforming of 3R composite materials

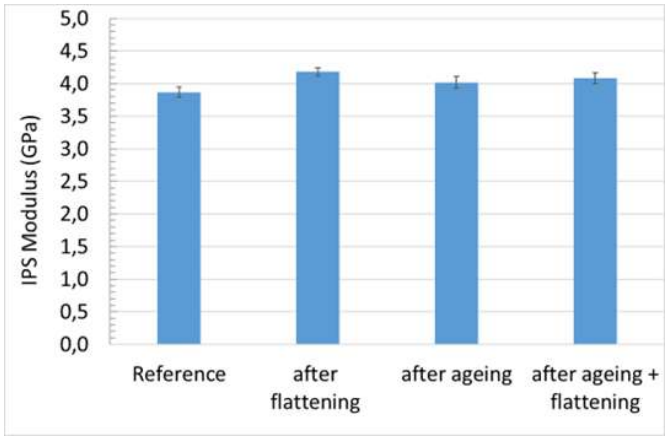
Evaluation of the properties of 3R glass fiber composite after weathering & flattening of +/-45° laminates (3mm thick)

MC4

+/- 45º Aged and flattened laminate

In-plane shear strength testing acc. to DIN EN ISO 14129





No effect of the 1000-hrs **weathering**

No effect of the **flattening**

No formation of wrinkles during the flattening process.

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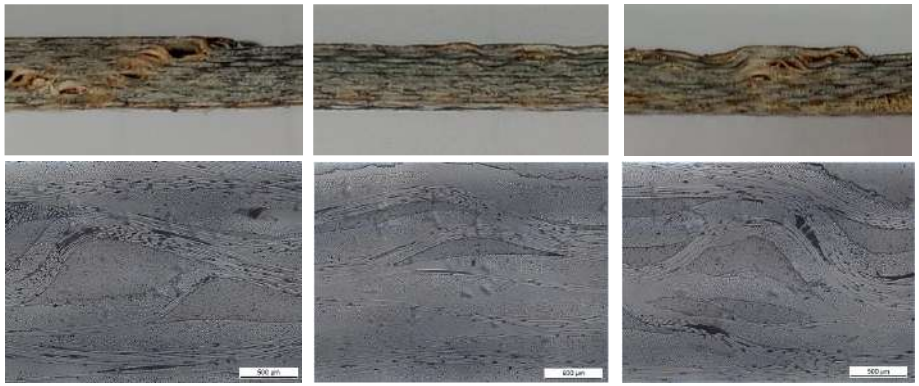
Thermoforming of 3R composite materials

Evaluation of the properties of 3R glass fiber composite after weathering & flattening

MC4

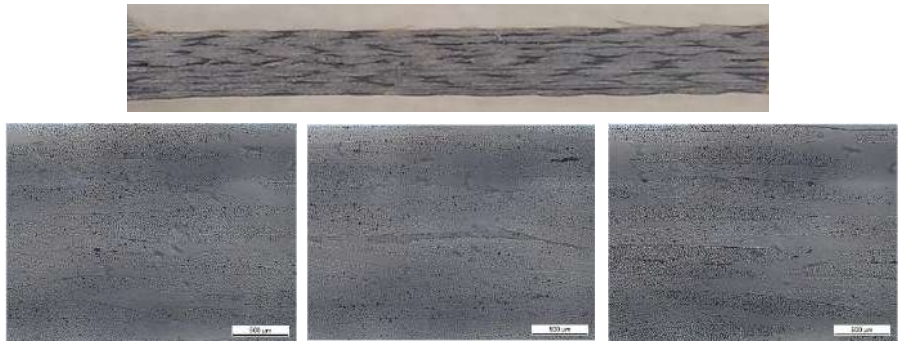
Optical microscopy of composites cross-sections

Flattened 0° laminate



Zone 1 Zone 2 Zone 3

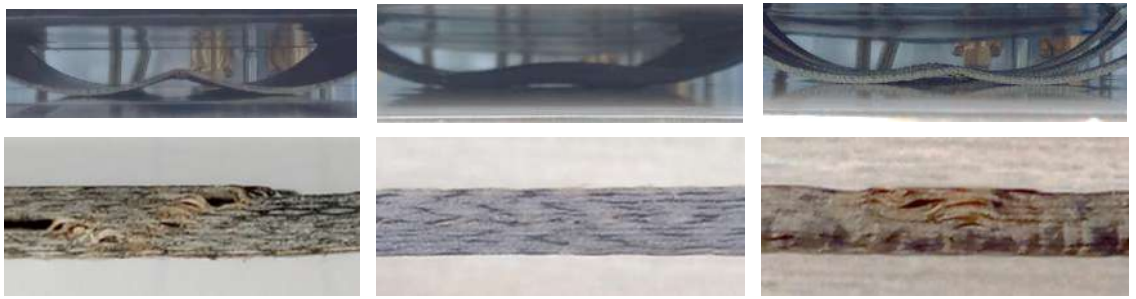
Flattened +/-45° laminate



Zone 1 Zone 2 Zone 3

Significant effect of the glass fiber fabric lay-up on the thermoforming results.

What about a combination of these orientations?



0/0/0/0/0/0/0/0/0/0 (°) +45/-45/+45/-45/+45/-45/+45/-45/+45/-45 (°) 0/+45/0/-45/0/0/-45/0/+45/0 (°)

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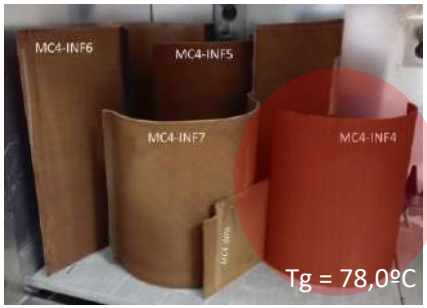
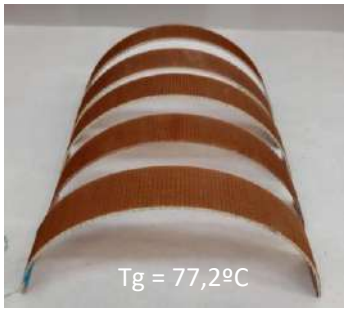
Thermoforming of 3R composite materials

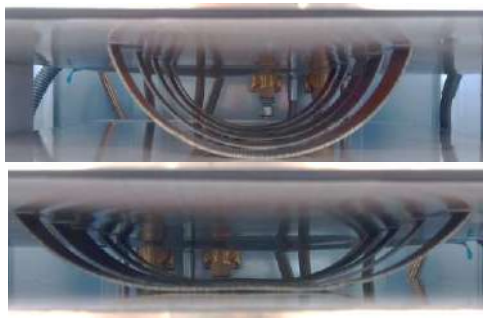
Full repurposing steps for 1,5 mm-thick composite material with kayak lay-up (0/+45/0/-45/0°)



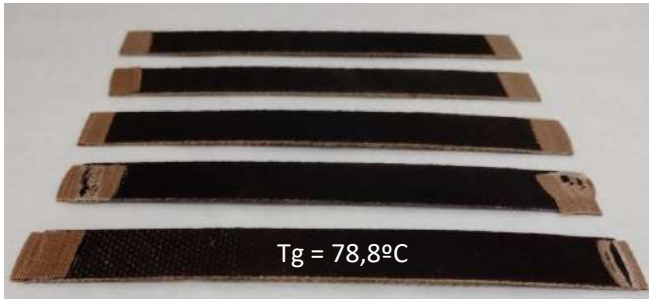
Curved laminate with kayak lay-up in the weathering chamber, after 1000 hrs weathering



Trimmed parts from "recycled" part



Flattening process of the "recycled" parts



Flattened parts, obtained from "recycled" composite part

3R adhesive placing

2 recycled laminates with 3R adhesive in between

During thermoforming of the "recycled" part

3mm-thick thermoformed sample, obtained from 1,5mm-thick recycled composite part

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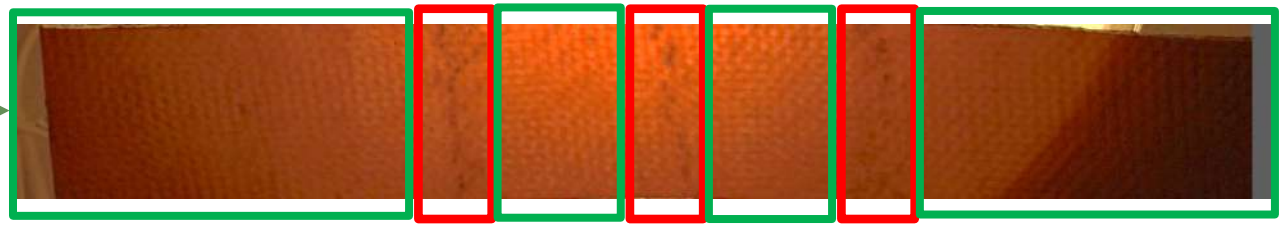
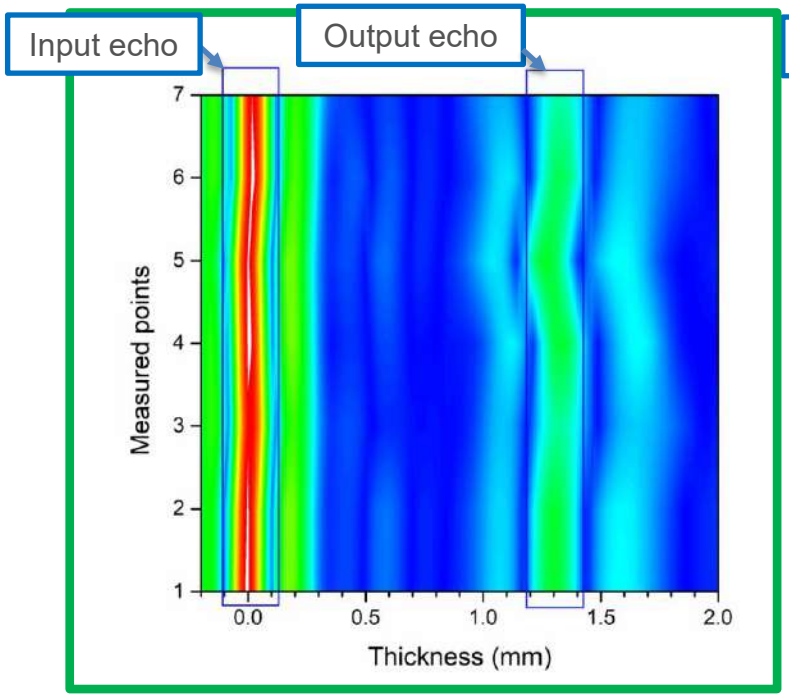
Thermoforming of 3R composite materials

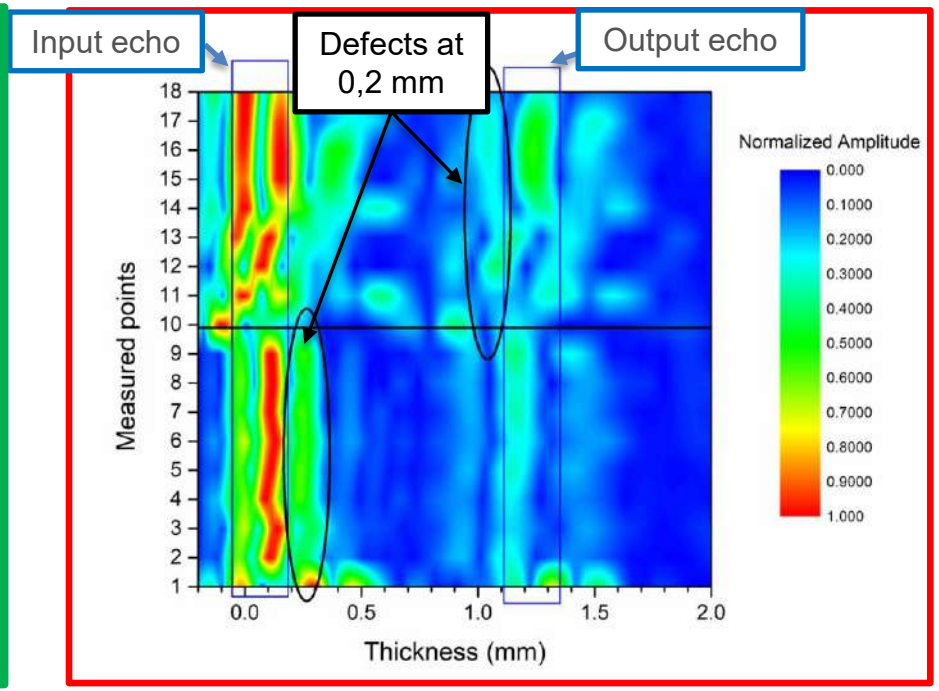
Evaluation of the properties of 3R glass fiber composite with kayak lay-up (1,5mm thick) - NDT

Flattening



MC4





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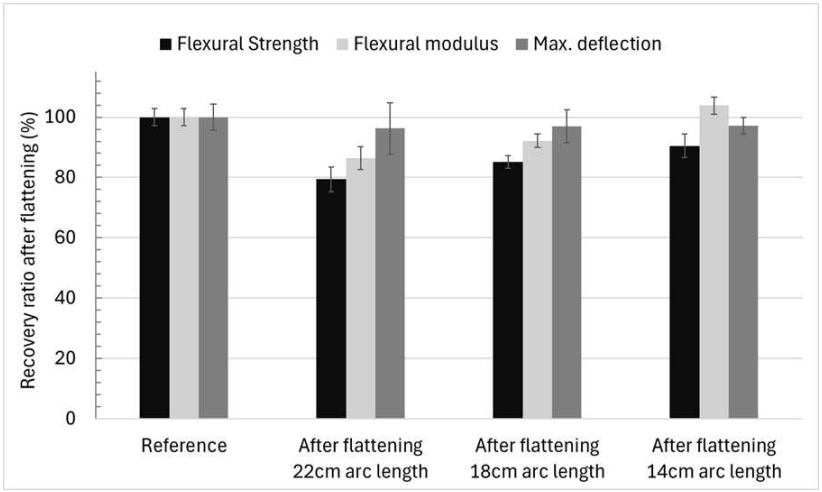
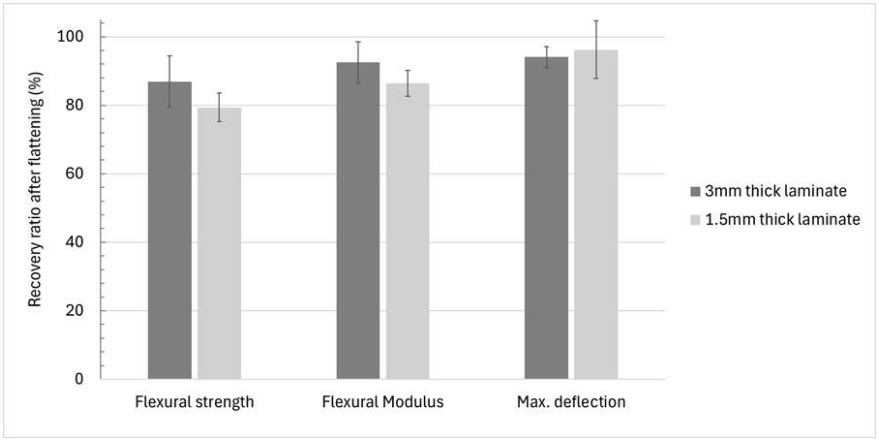
Thermoforming of 3R composite materials

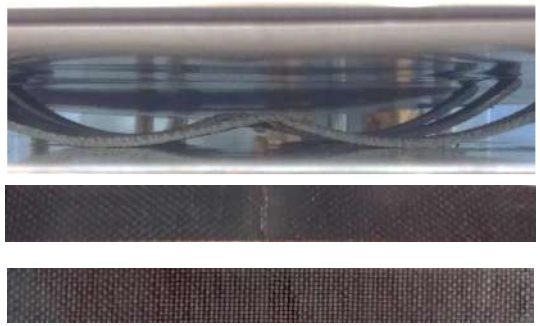
Flattening of 3R GF composite with kayak lay-up – arc beam length effect & thickness effect

22 cm arc length | 18 cm arc length | 14 cm arc length





1.5 mm thick laminate



3 mm thick laminate

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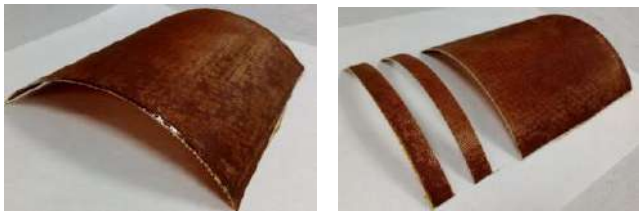
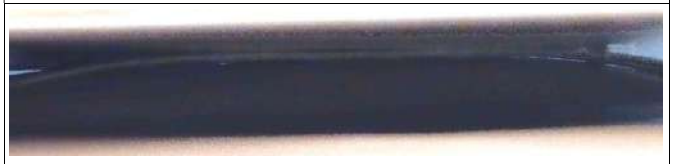
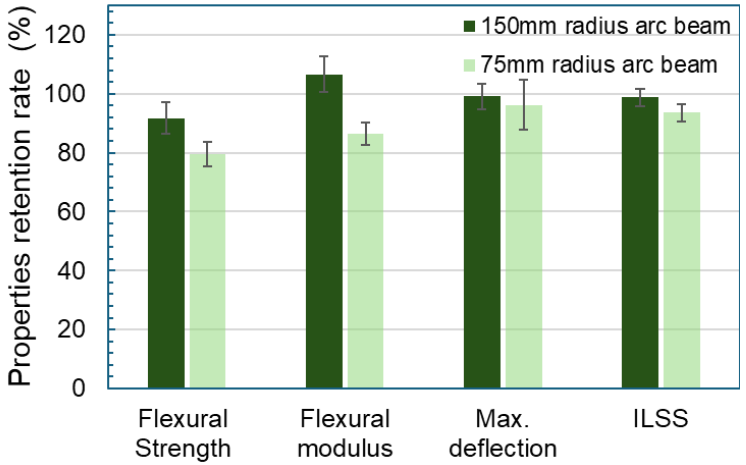
MC4

Thermoforming of 3R composite materials

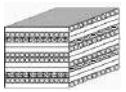
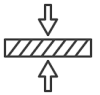
Flattening of 3R GF composite with kayak lay-up – arc beam radius effect

MC4

150 mm radius curved laminate, and trimming	
150 mm radius initial composite sample with 27 cm arc length	
Deformation of the composite sample during the flattening process	 
Flattened composite sample	
Flattened composite examined with retro diffused lighting	



Factors affecting the properties recovery ratio:

- GF fabric lay-up 
- Thickness 
- Radius of curvature 
- Arc beam length 

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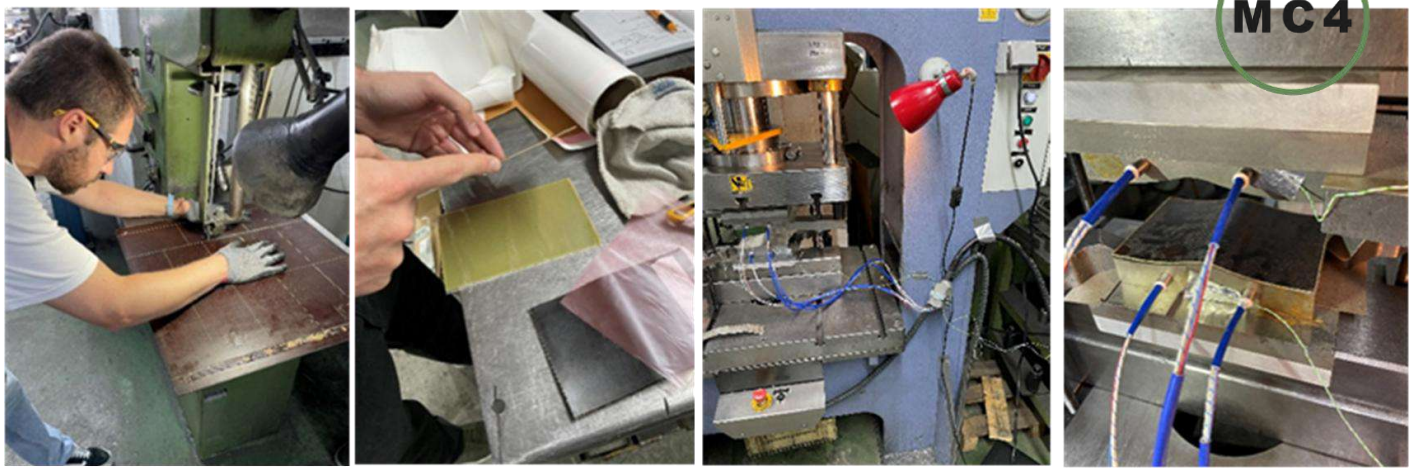
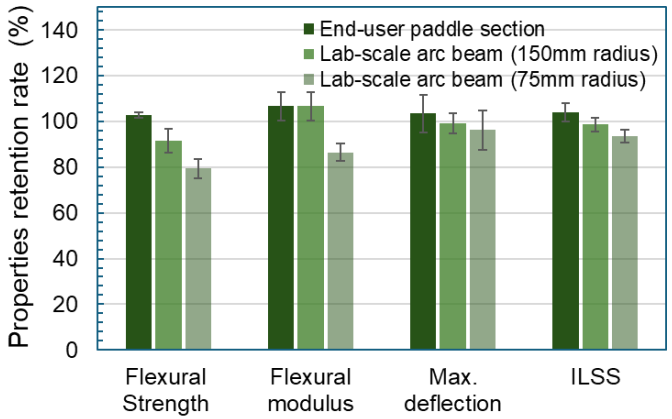
Thermoforming of 3R composite materials

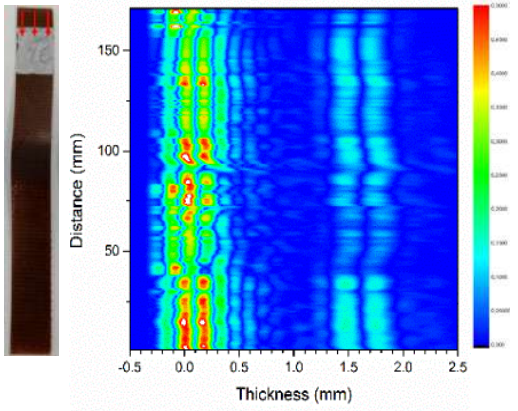
End-user manufacturing trials

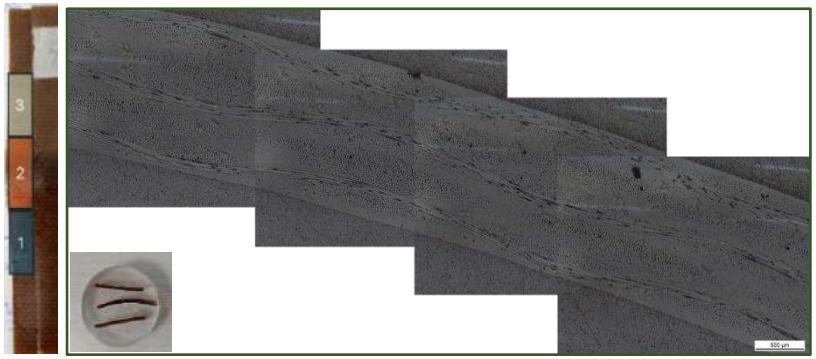
Paddle section with **very high Properties Retention Rate**



Successful transfer from lab to end-user with **good reproducibility**







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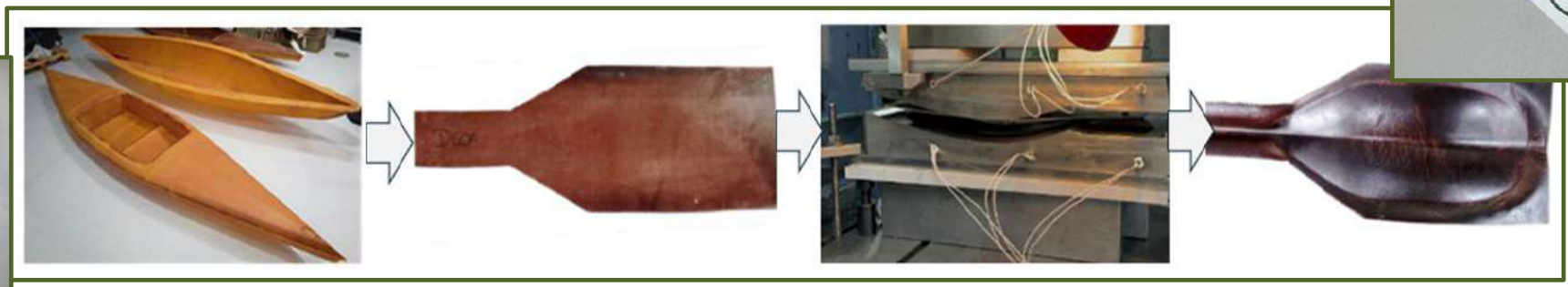
Thermoforming of 3R composite materials

Final manufacturing trials by end-user



Properties retention rates > 85% (tensile, flexural, ILSS properties).

Good visual aspect after finishing operations.

Defining **specifications based on first-use and reuse applications** is **crucial** for 3R resin formulation.

CURRENT PROCESS

- Non-recyclable materials.
- End-of-life (EoL) GF composites disposed of in landfills at a rate of 100%.

MC4 PROCESS

- Recyclable materials without requiring separation of GF from matrix
- Design for circularity
- Reuse of 94% of EoL kayak material
- GF composites lifespan extension
- High-quality recycled materials, with properties retention rates > 85%
- Good cost/performance ratio

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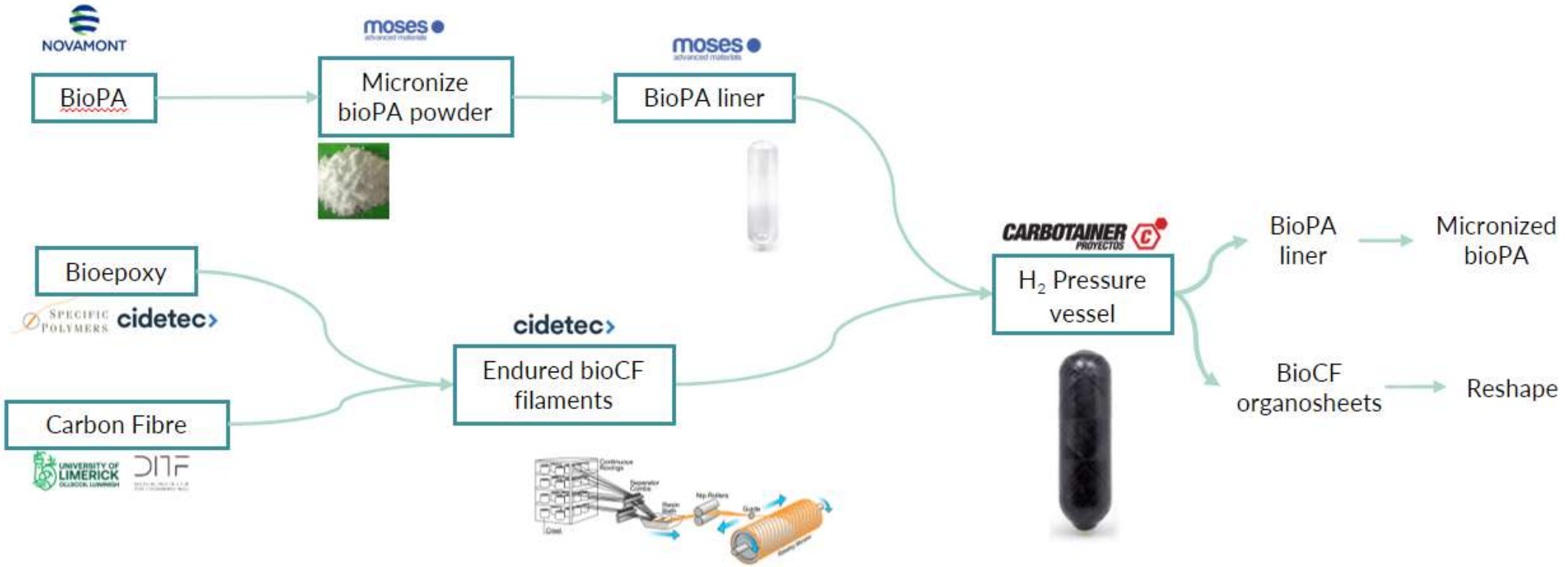
Thermoforming of 3R composites in CUBIC project



To demonstrate complex product circularity by validating their de-manufacturing process, recyclability and valorization.

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Thank you for your attention!

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Thermoforming of 3R composite materials

Camber Link, SAFEMIUM project
(Basque Business Development Agency)

Longitudinal stiffener (LS)

- Manufacturing with Discontinous Compression Moulding
- Thermoformability demonstrated but improvable internal quality (due to overheating)
- 63% cycle time reduction compared to autoclave processing

AIRPOXY

Thermoformable, repairable and bondable smart epoxy- based composites for aero structures

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