



# Kinetic analysis of self-healing in vitrimers based on the recovery of mode I fracture toughness

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# Contents

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Introduction

Experimental work

Results and discussion

Conclusion and future work





# Introduction

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# Thousands of Composite Parts End Up in Landfills or Incinerated! Green Energy is not so Green!

🏠 Today, there is a general awareness on the environment and climate change being one of the major threats for future generations.

🏠 Composite materials are expected to make a crucial contribution to achieve the "Green Deal" launched by the European Union with the ambition to make Europe climate neutral by 2050 (HOW?)

## Matrix of composite materials:

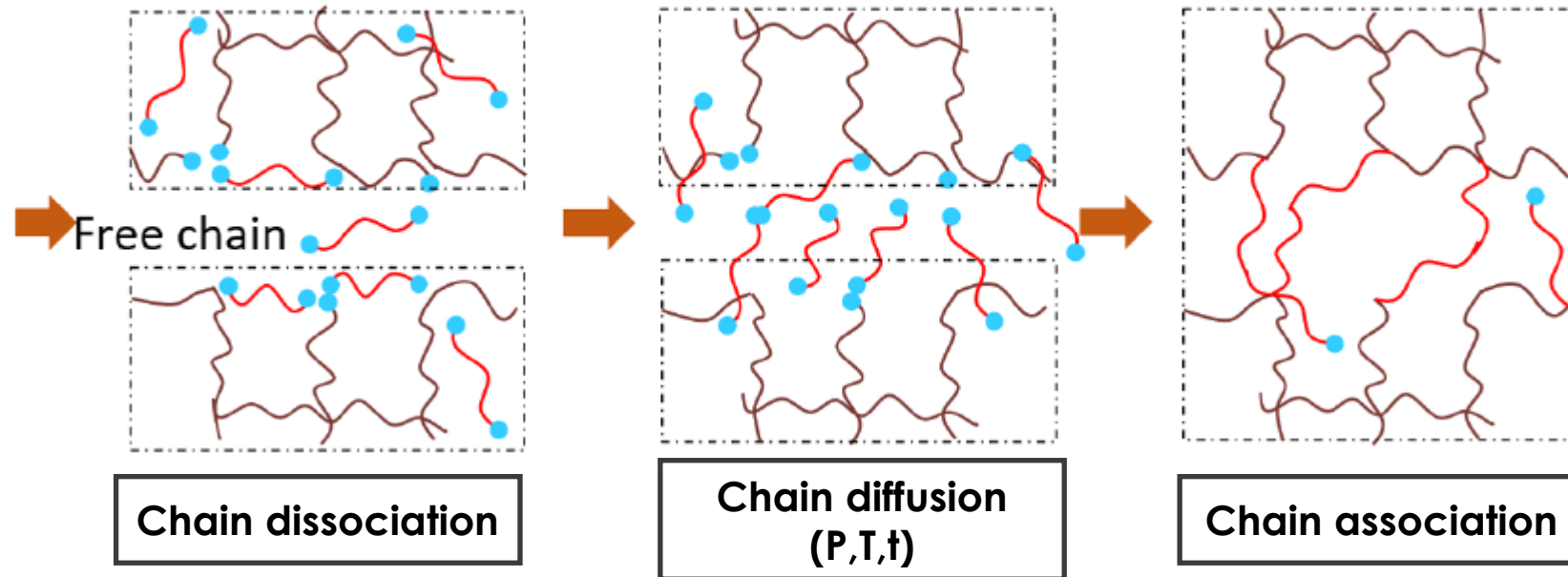
- 70-80% of the matrix in composite materials are thermoset, provide the highest performance characteristics

**But, Limited Recyclability, Difficult to Repair, Complex Manufacturing, and Disposal Issues**

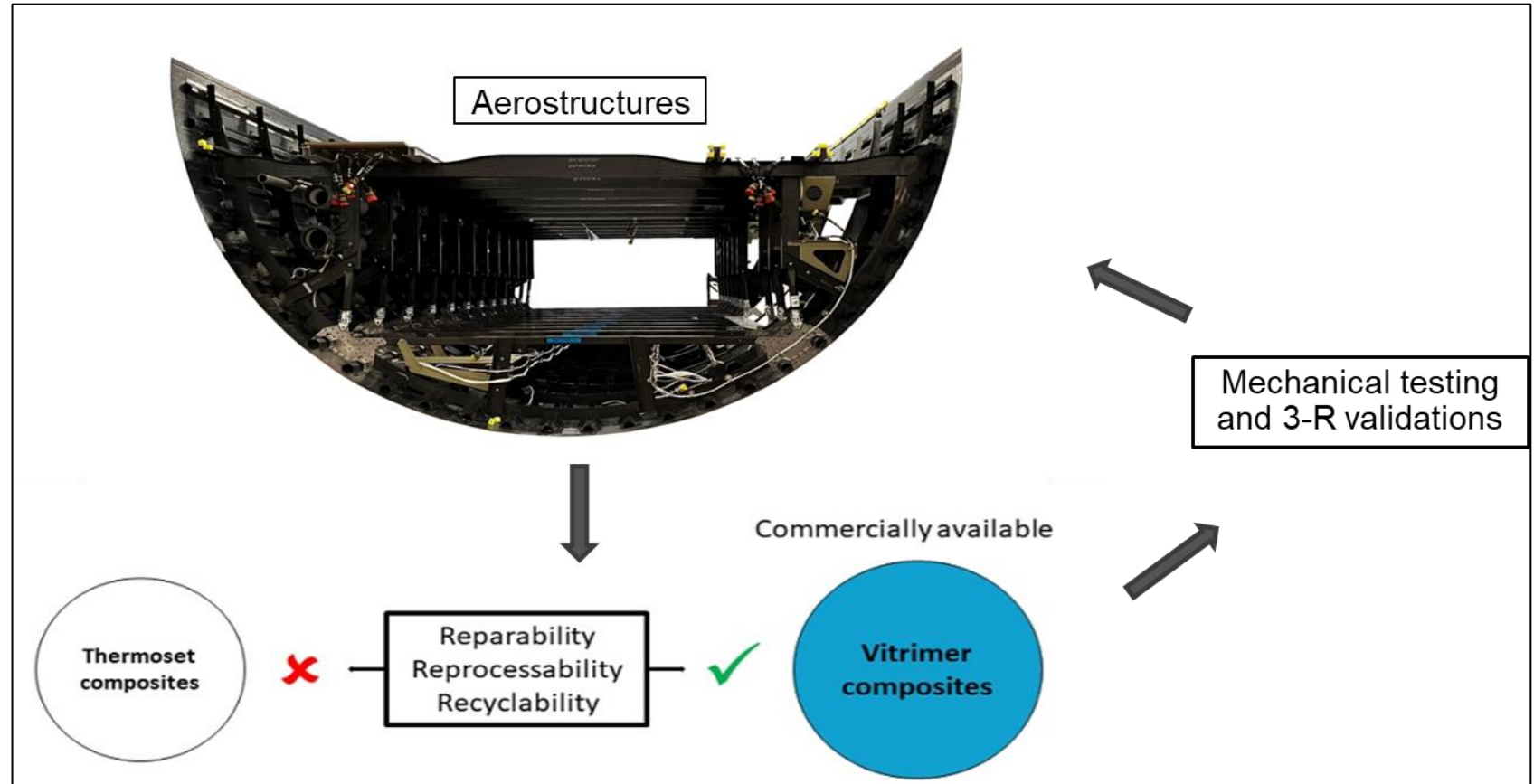


# Vitrimers – The miracle polymer materials

- 🏠 Innovations led to an advanced thermoset polymers with 3R properties.
- 🏠 Montarnal et al. (2011) introduced covalent adaptative networks (CANs), so called vitrimers,
- 🏠 The dynamic covalent bonds of vitrimers when exposed to a certain stimulus, usually heat, making the network of the vitrimers adaptable, healable, and recyclable.



An, L., Shi, Q., Jin, C., Zhao, W. and Wang, T.J., 2022. Chain diffusion based framework for modeling the welding of vitrimers. *Journal of the Mechanics and Physics of Solids*, 164, p.104883.



Vitrimers polymer can be used in replacement of ordinary thermosets

# SUBHYCO – Particular objectives

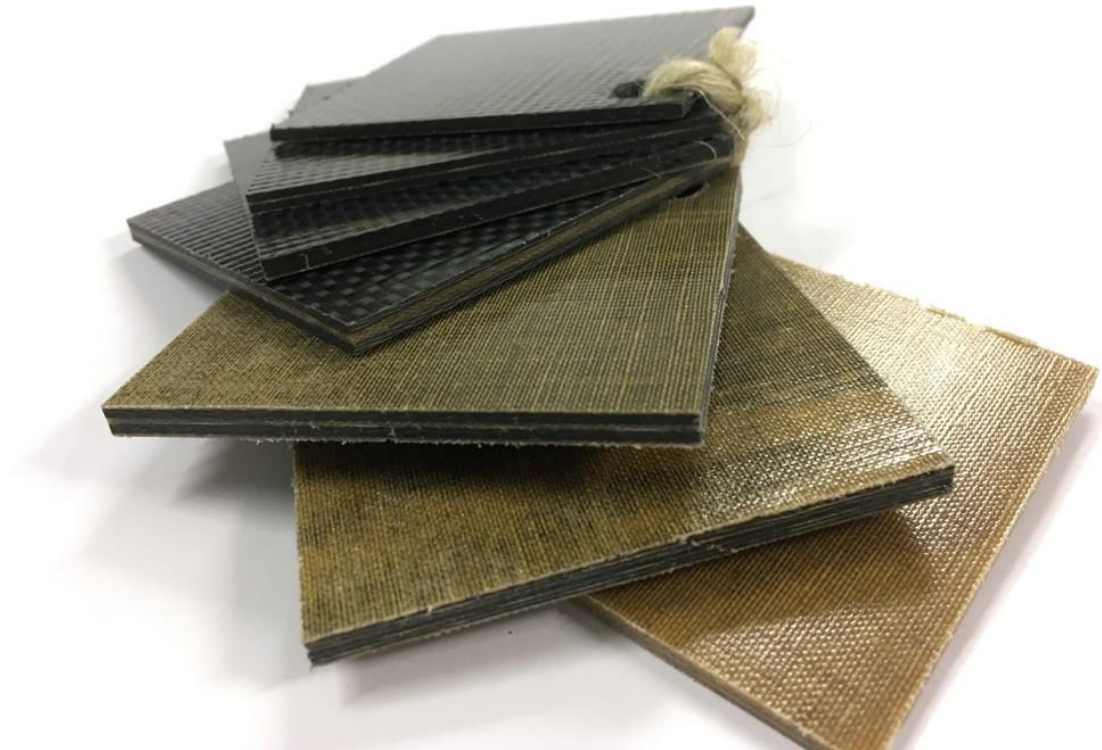
- 🐘 Definition of a toughness – based approach to **assess the self-healing** properties of claimed self-healing vitrimer polymers. We propose a methodology based on fracture tests (DCB, SENB) to assess **quantitatively** self-healing.
- 🐘 Characterization of sustainable composites reinforced with **natural fibres**.





# SUBHYCO – Particular objectives

- Development of a **highly damping hybrid** composite with glass and natural fibre reinforcements for wind turbines and the automotive sector.
- Development of hybrid composite with **recycled carbon fibres** and natural fibres.





# Experimental work

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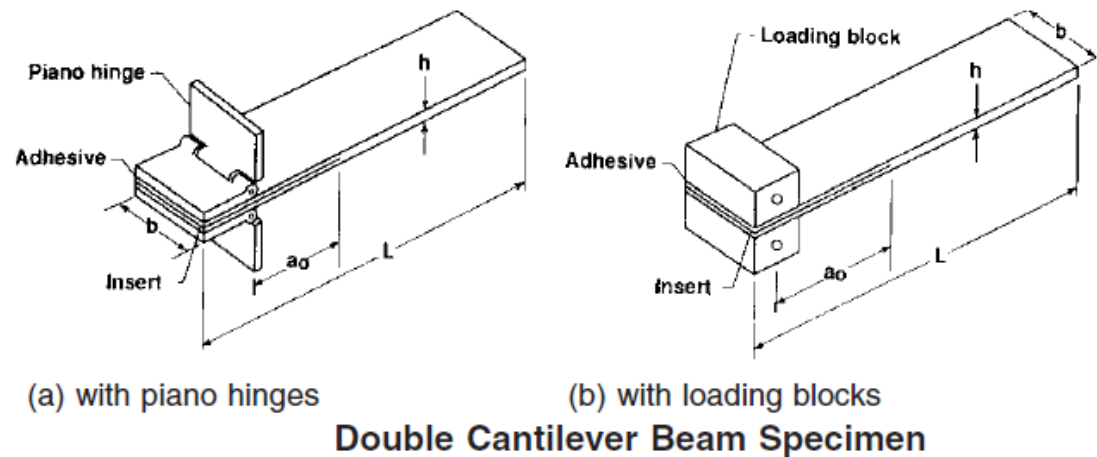
# Self Healing of vitrimers

- Up to date, there isn't a standard method to implement or assess self healing property of vitrimers
- This involves measuring the fraction or percentage of mechanical properties that have been restored by the self-healing under (Static, Fatigue , and/or Impact Damage).
- This fraction or percentage called the repair/healing efficiency concept.

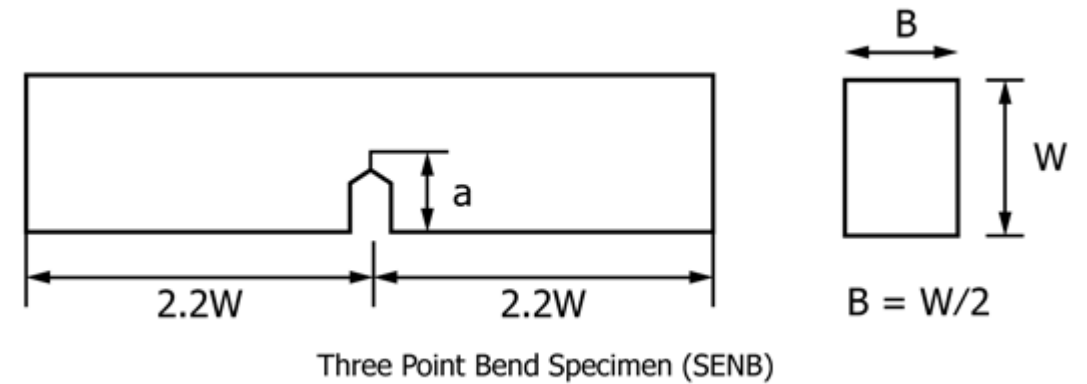
$$\text{Healing efficiency} = 100 \times \frac{\text{Property value}_{\text{healed}}}{\text{Property value}_{\text{initial}}}$$

# In our work:

- Time, temperature, and pressure dependence of self-healing in vitrimers. Based on the recovery of **mode I fracture toughness** using DCB and SENB.
- For this purpose, we have designed and manufacturing a steel mold for samples healing.



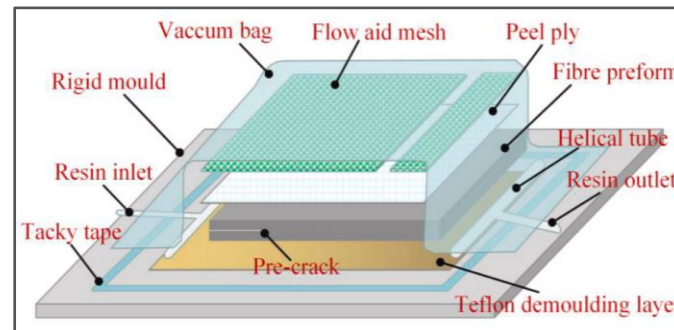
ASTM D 5528



ASTM D 5045

# Preparation of DCB specimens of vGFRP

Panel manufacturing using vacuum infusion process



The assembly was cured in an oven at 130°C for 1 hour and 150°C for 30 minutes



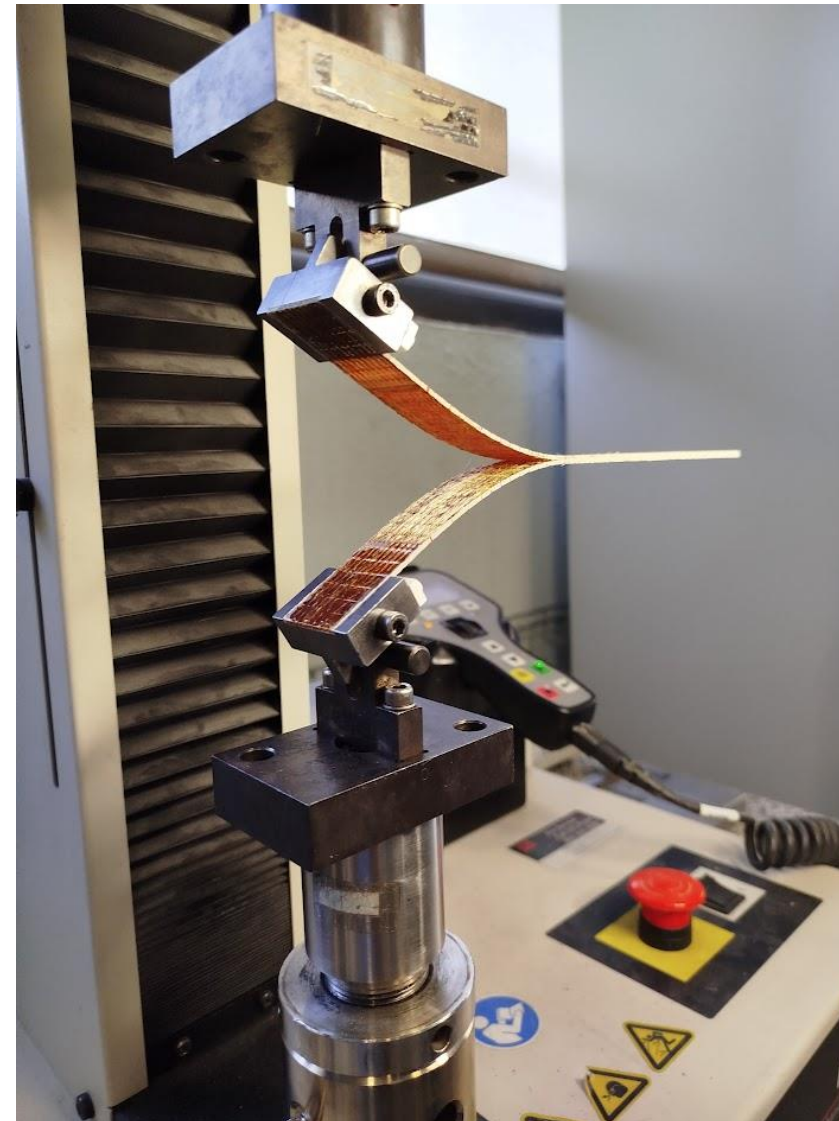
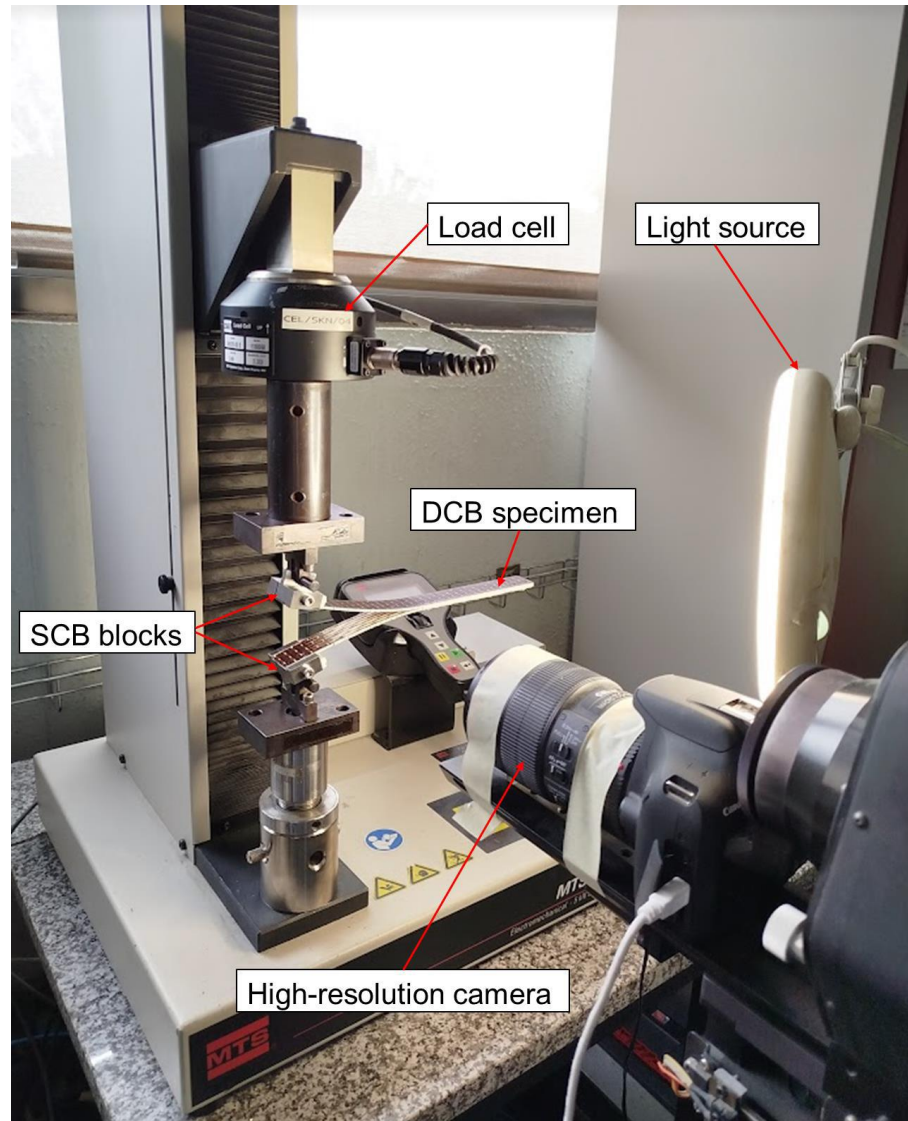
Finally, the laminates were cut into DCB specimens



The vitrimer is a disulfide-containing epoxy produced by Cidetec (Donostia-San Sebastian, Spain), which holds the patent for it.

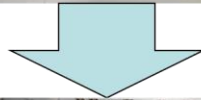
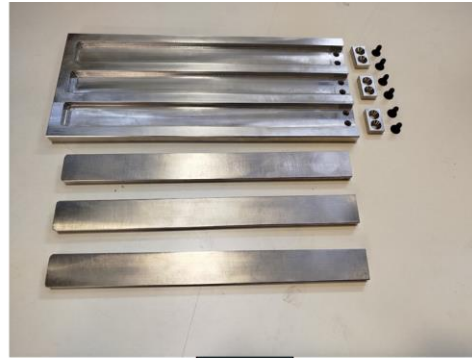
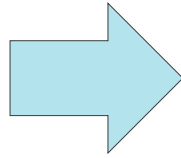
# Measurement of interlaminar fracture toughness

- The calculations of interlaminar fracture toughness for the pristine and healed samples was performed According to standard ASTM D 5528.

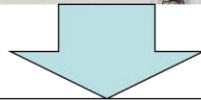
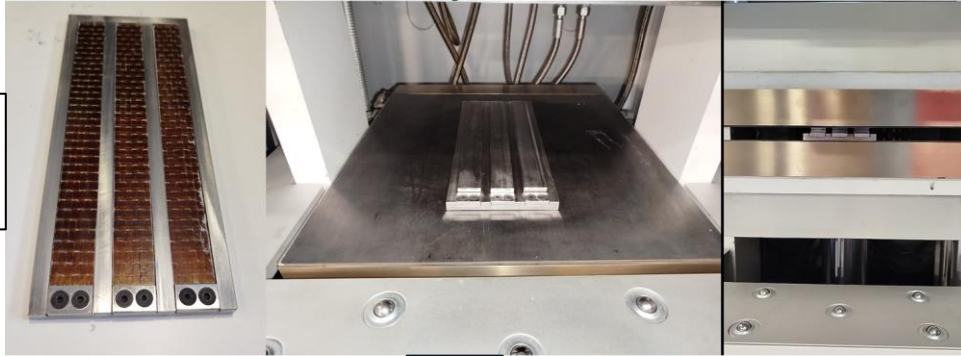


# Delamination healing

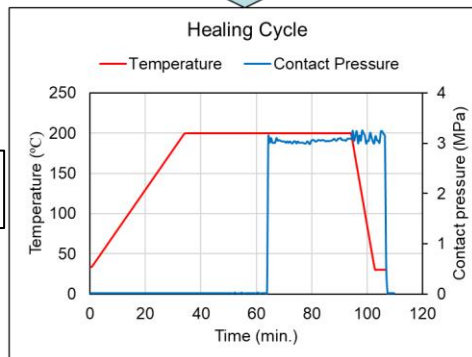
Preparation



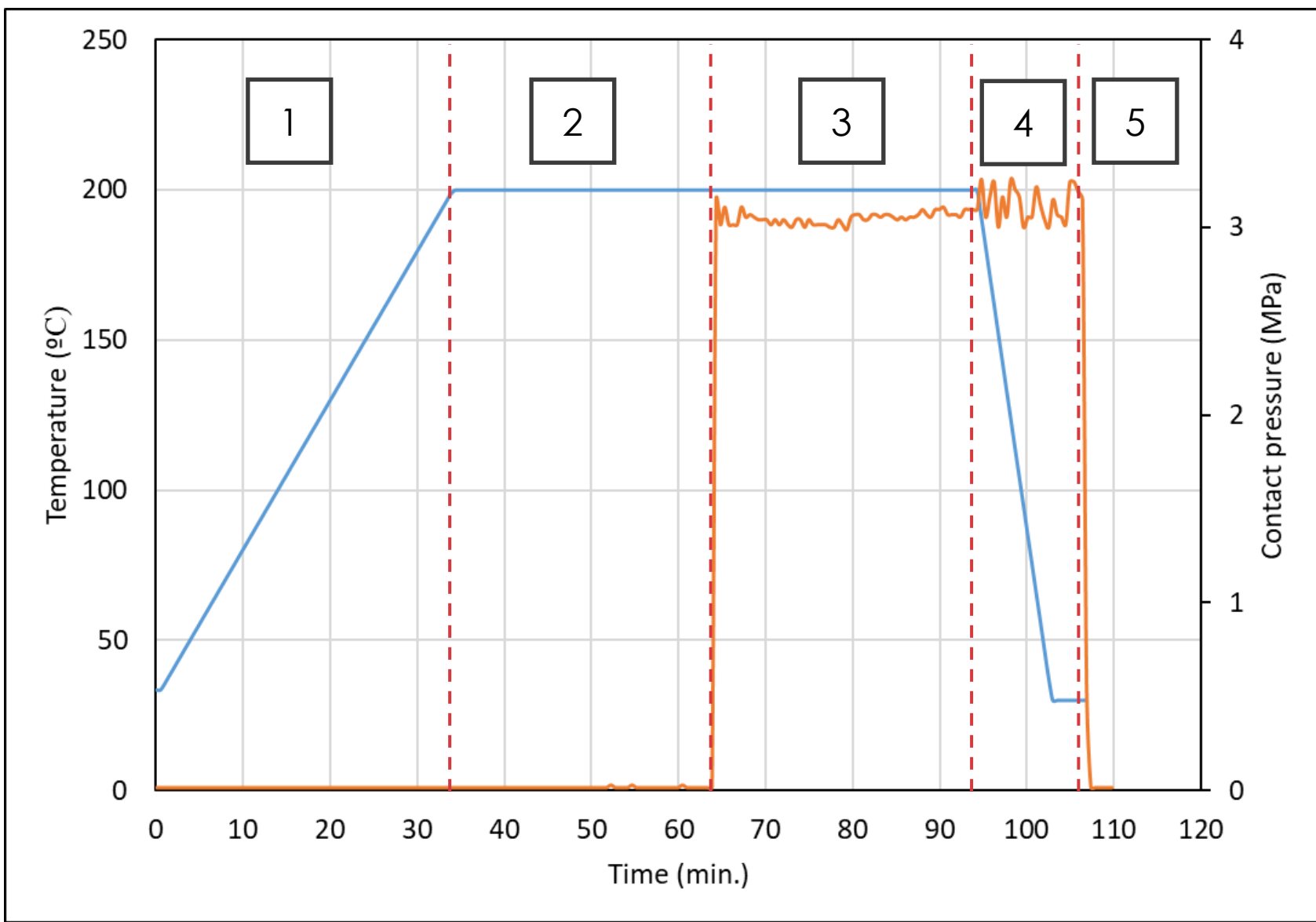
Specimen Placement



Healing Cycle

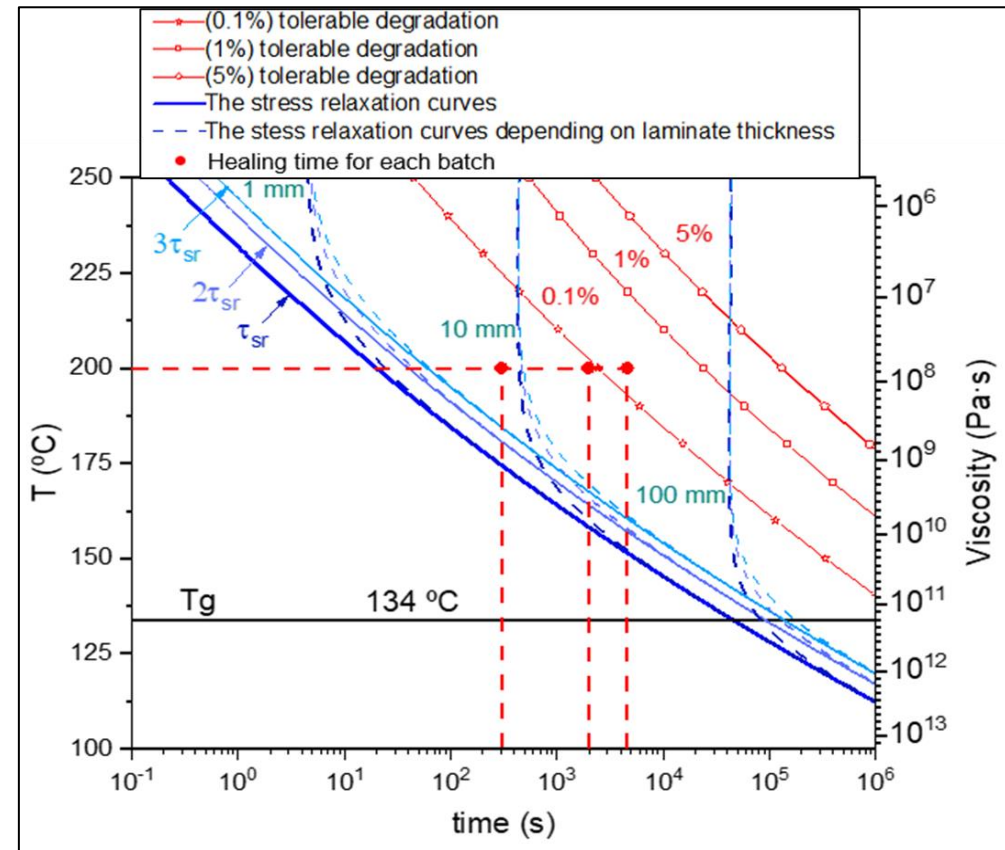


# Healing Cycle





- The healing conditions were selected with the support of the reprocessing map developed by Sánchez-Rodríguez et al. 2023.
- Temperature was fixed at 200 °C and Pressure was fixed at 3 MPa, while the times explored were 300, 2000, and 4500 sec. At least three specimens per batch were tested.





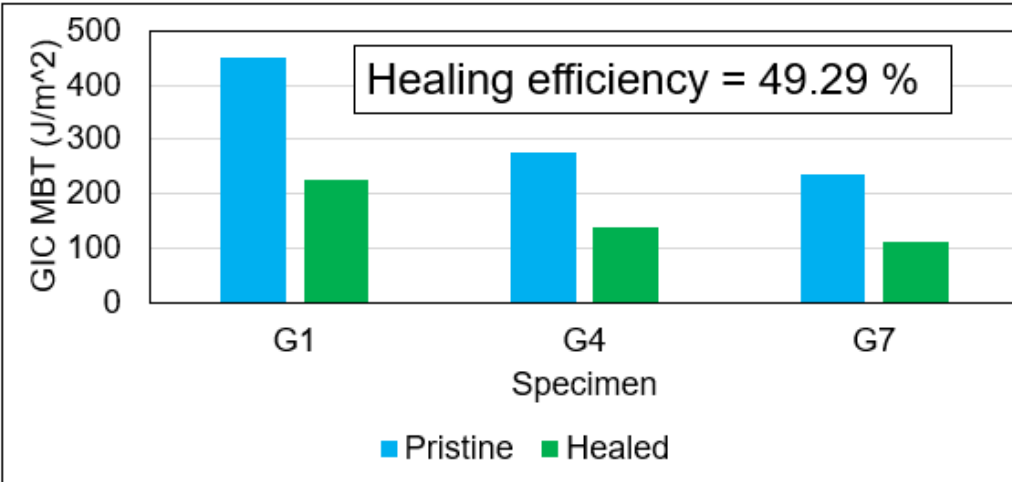
# Results and discussion

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# For DCB test

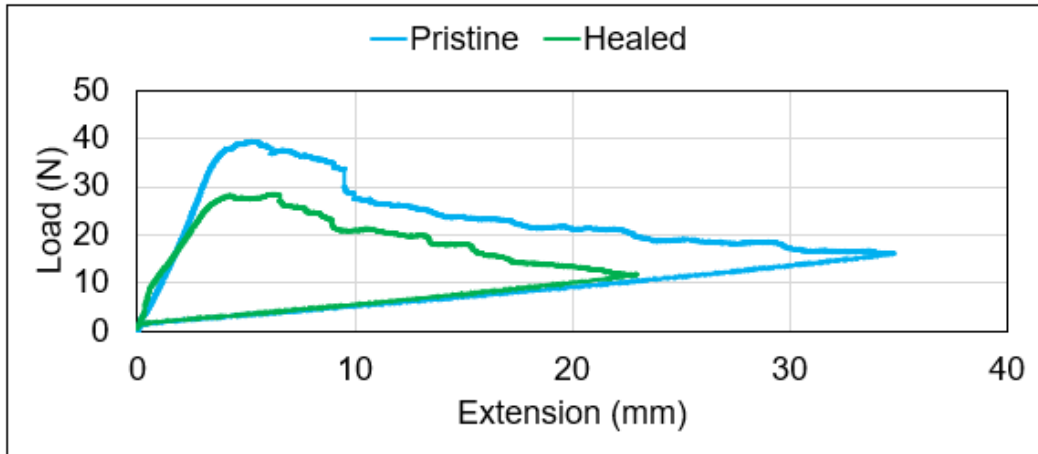
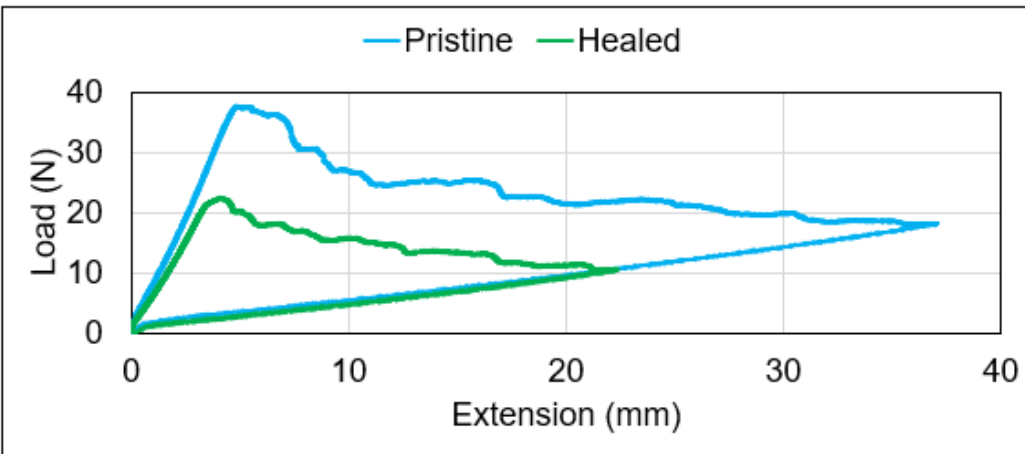
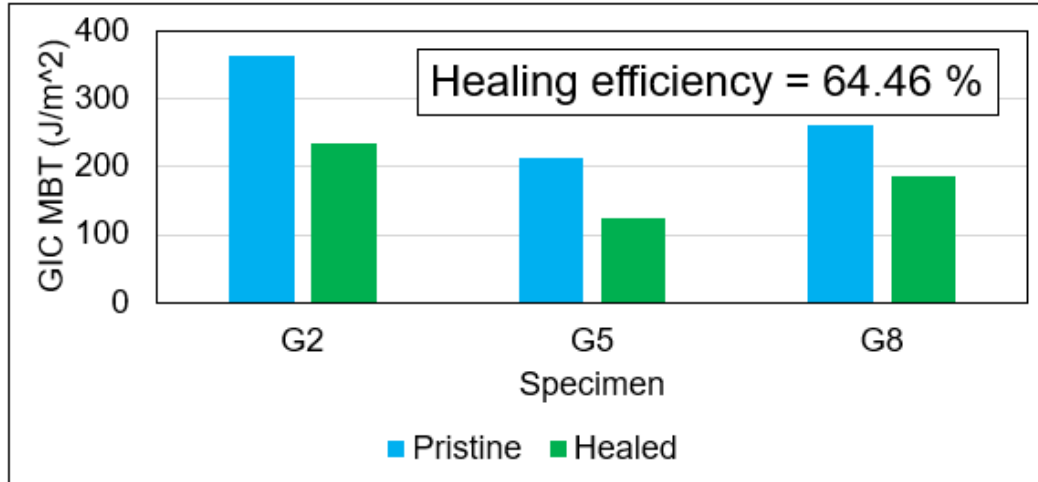
## First batch

Healing condition: 200°C, 3 MPa, 300 sec

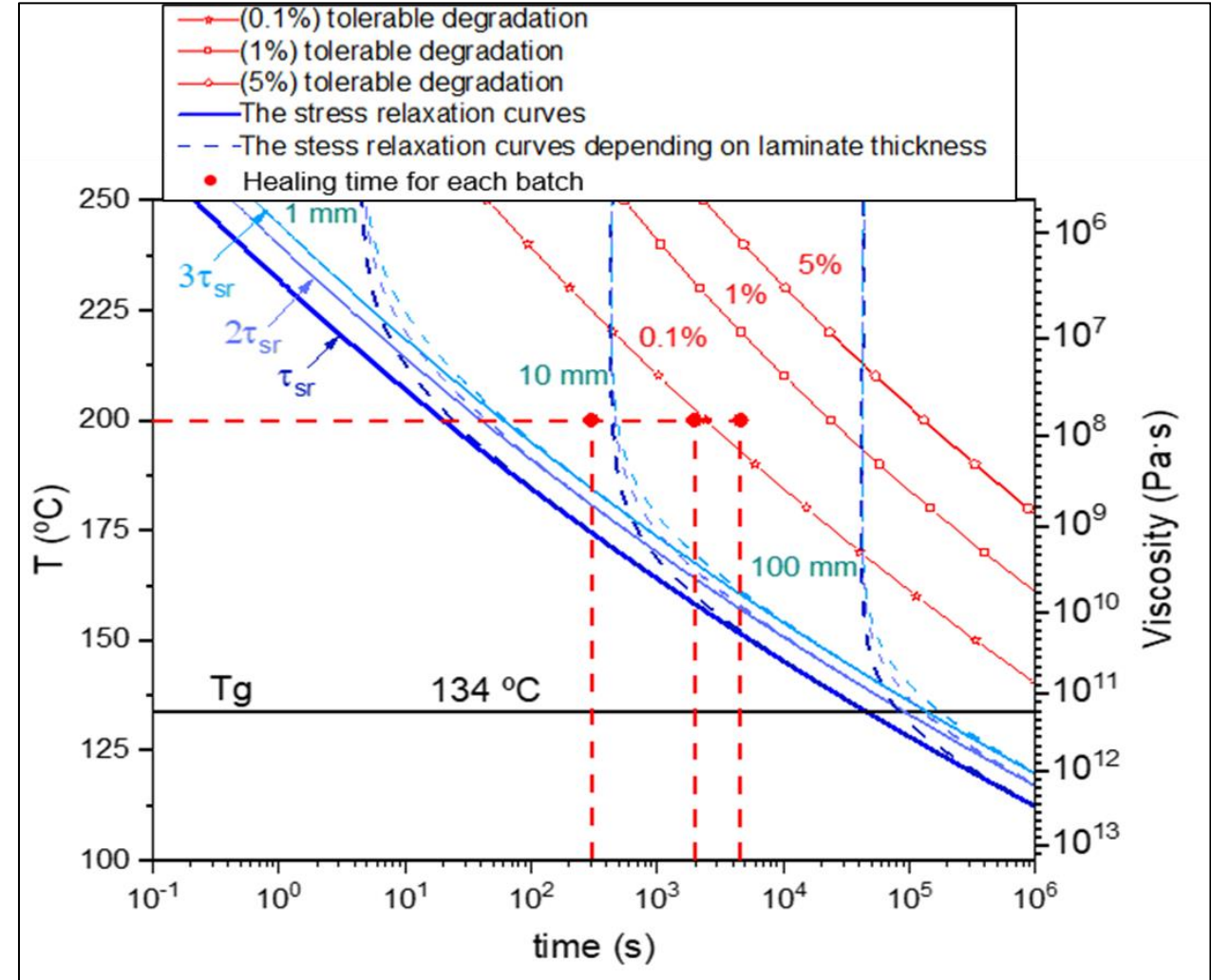
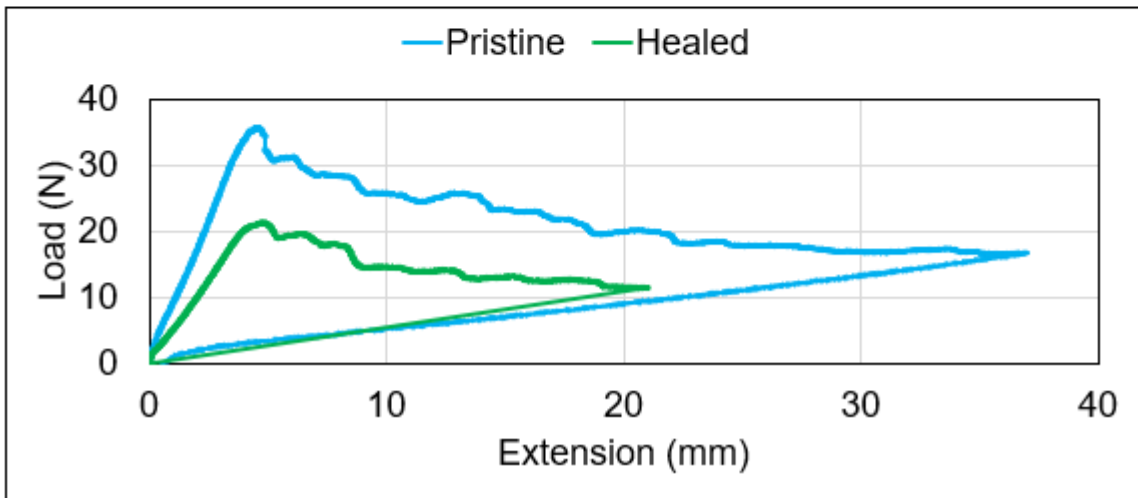
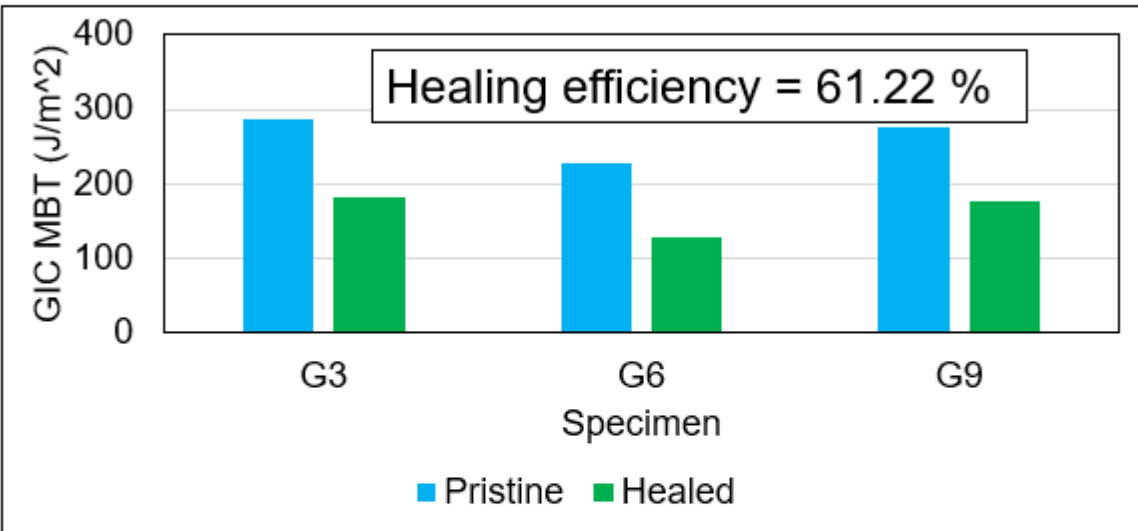


## Second batch

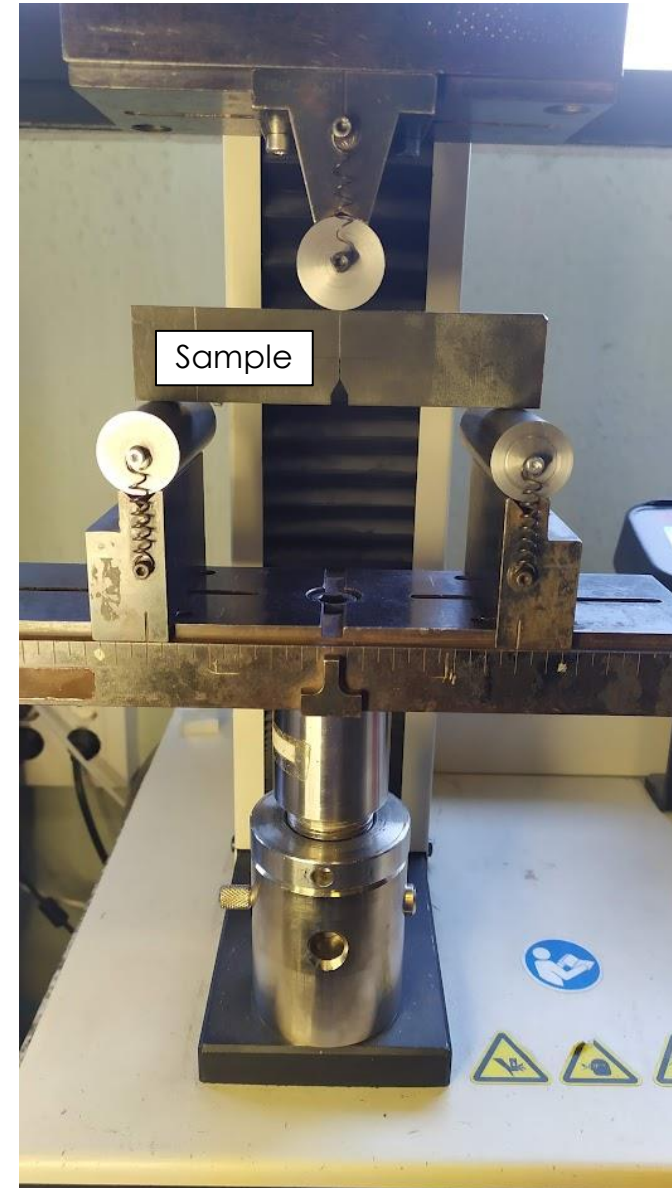
Healing condition: 200°C, 3 MPa, 2000 sec



**Third batch**  
**Healing condition: 200°C, 3 MPa, 4500 sec**

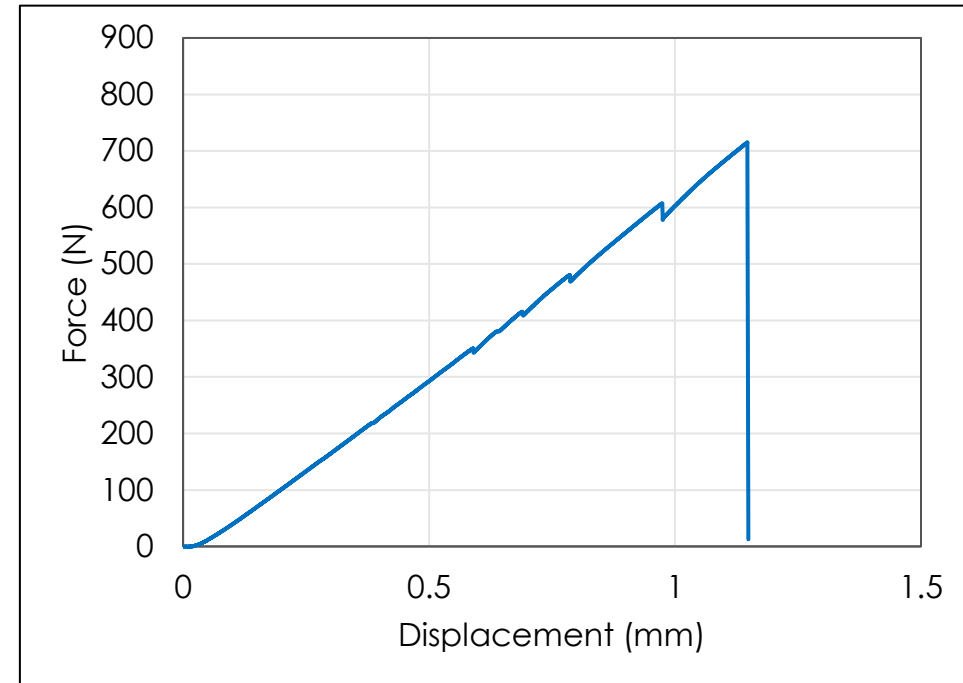
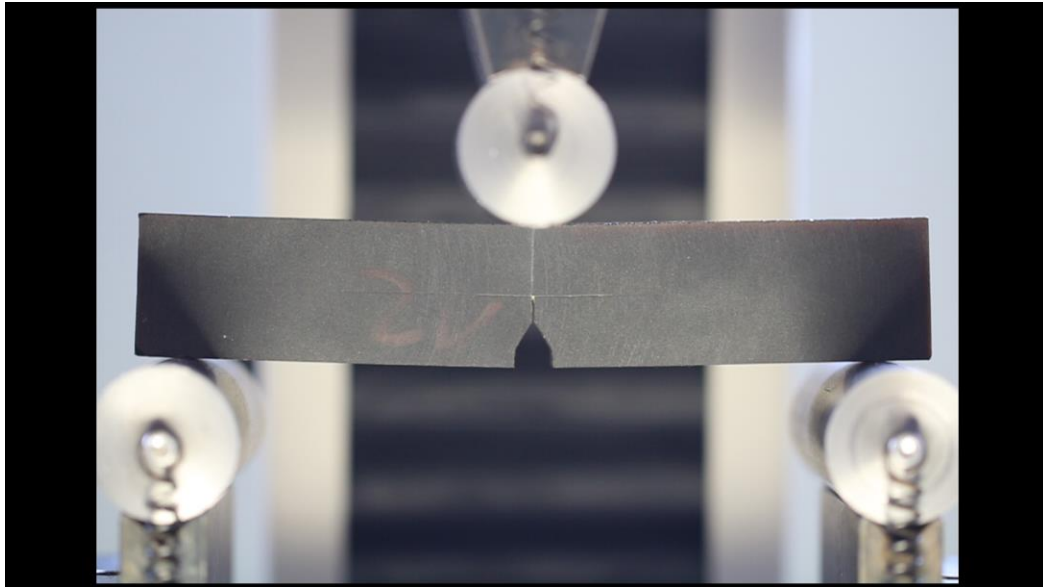


- On the other hand, for assessing the self-healing of plain vitrimers, fracture toughness was measured using single-edge notched bending (SENB) according to ASTM D 5045



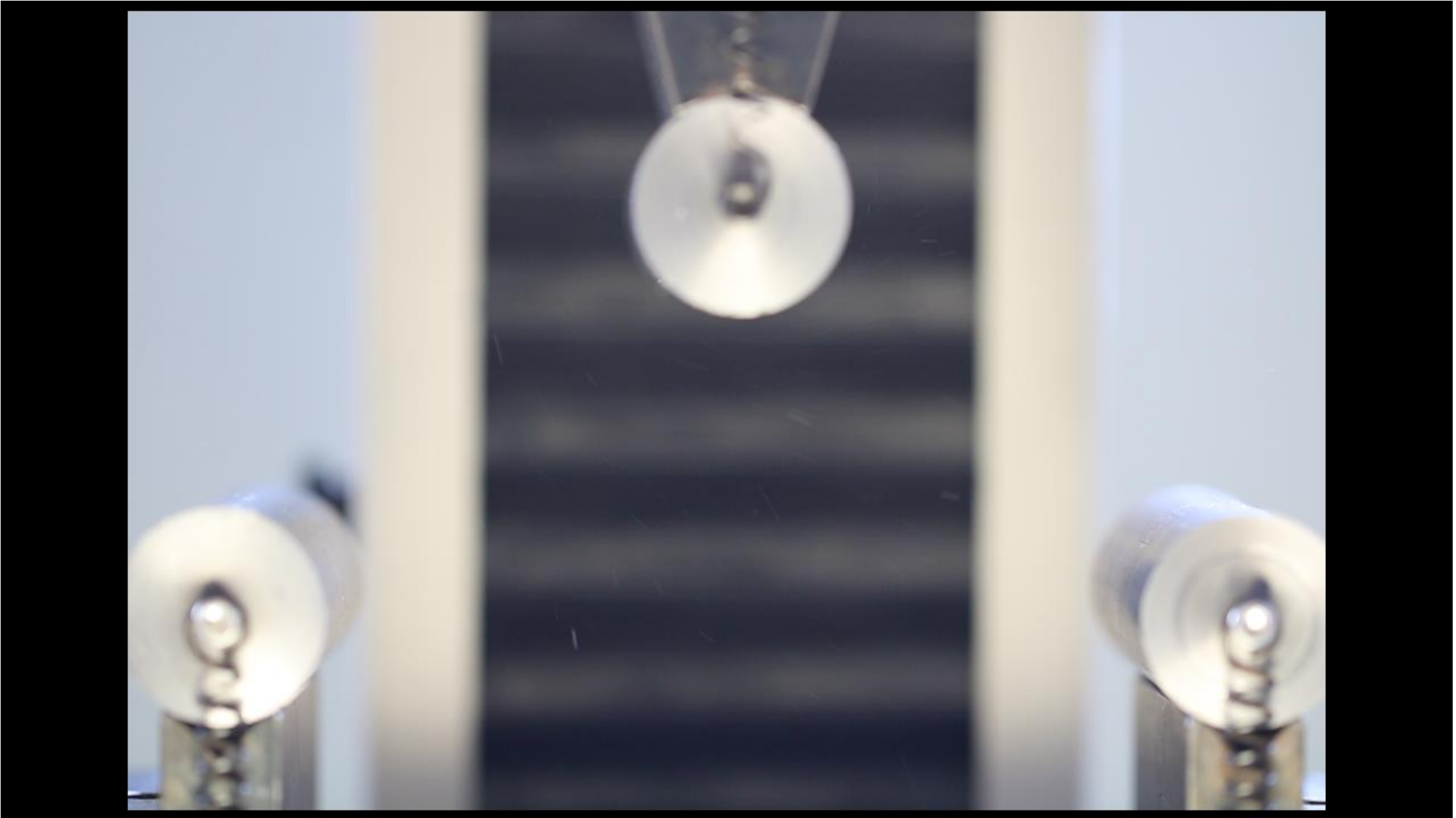
# For SENB test

According to ASTM D5045

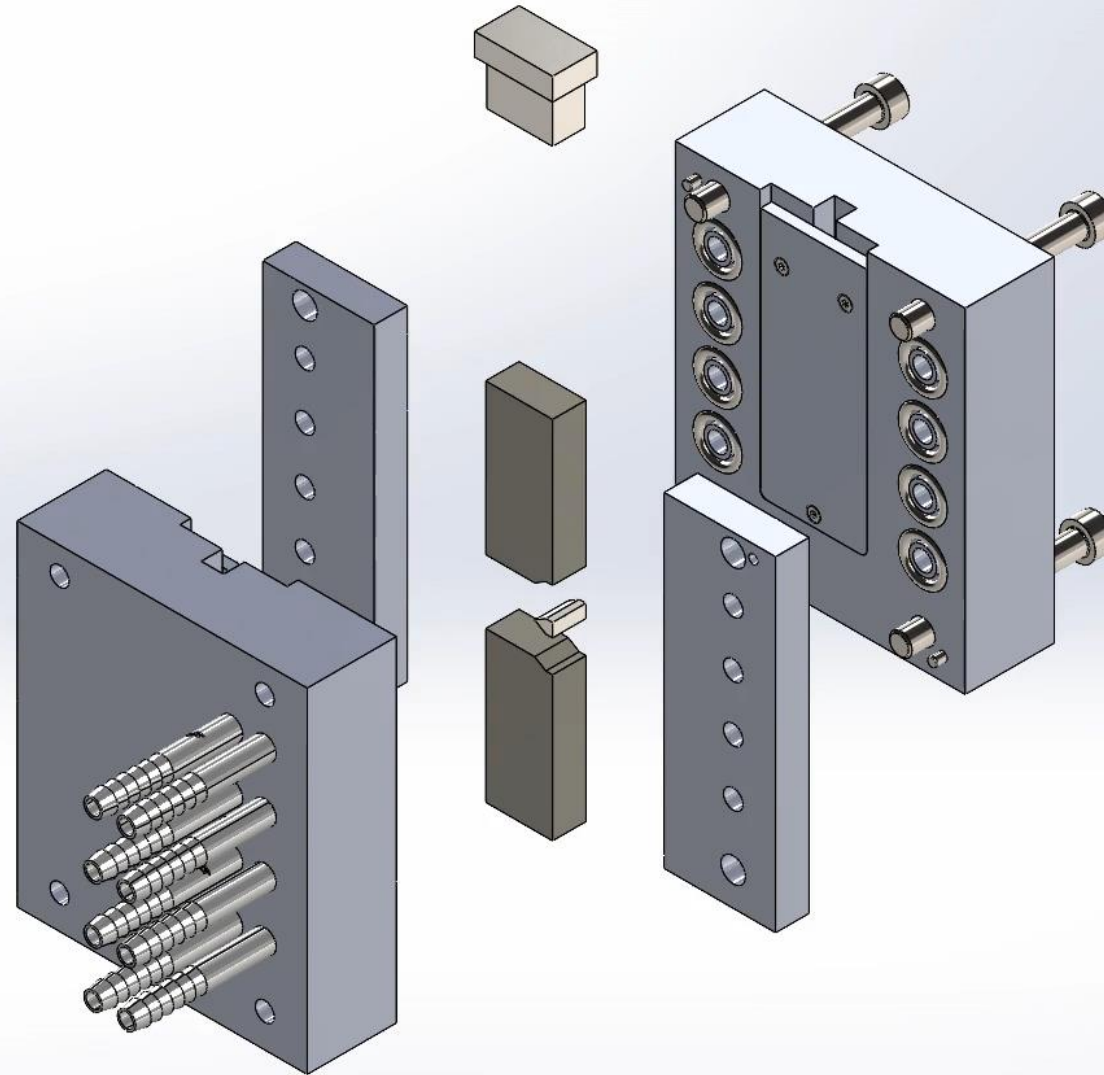


$K_{Ic}$ (MPa. m <sup>(1/2)</sup> )	3.34
$G_{Ic}$ (kJ. m <sup>-2</sup> )	6.37

The Plane-strain fracture toughness ( $K_{Ic}$ ) and fracture energy ( $G_{Ic}$ ) were determined.



# Healing mold for SENB Samples







# Conclusion and future work

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# Conclusion

- The healing efficiency for the healed DCB specimens healed for 2000 and 4500 seconds were more than 60%.
- Increasing the time allows higher chain diffusion and improves healability at the delamination surface.
- This work will deepen the understanding of the self-healing phenomena and will guide the definition of suitable thermal stories for the repair of plain/pure vitrimers, or vitrimer-reinforced composite.

# Future work

- Try to reach a stable crack propagation with the SENB test, through apply a natural crack and change specimen dimensions.
- Characterization of sustainable composites reinforced with UD **natural fibres**.
- Development of a **hybrid** composite with **glass** and **natural fibre** reinforcements for **wind turbines and the automotive sector**.
- Development of hybrid composite with **recycled carbon fibres** and natural fibres.



# Thank you

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