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EFFECT OF POST CURING ON THE MECHANICAL PROPERTIES OF A UD FLAX FIBER REINFORCED EPOXY COMPOSITE

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ICNF 2017

3rd International Conference
on Natural Fibers

Advanced Materials for a Greener World



1. Introduction

- ✓ Epoxy composite reinforced with glass or carbon fibers used in many applications
- ✓ Alternative to synthetic fibers = Flax fibers (Renewable source, biodegradable, specific properties similar to glass fibers, ...)

Synthetic fiber reinforced composite :

- ✓ Glass or carbon fibers not sensitive to temperature
- ✓ Composite process temperatures $\geq 150^{\circ}\text{C}$ for a 100% cured resin

Problem : natural fibers sensitivity to high temperatures

- ✓ Exposure at 120°C \longrightarrow loss of moisture and degradation of waxes [1]
- ✓ Rigidity and fatigue behavior affected by thermal treatment beyond 150°C [2]
- ✓ \searrow (44%) of strength after 14h at 105°C after drying [3]

\rightarrow Which temperature to cure/post-cure the natural fiber reinforced composite ?

[1] Müssig J, Haag K. The use of flax fibres as reinforcements in composites. *Biofiber Reinf. Compos. Mater.*, 2015, p. 35–85

[2] Placet V. Characterization of the thermo-mechanical behaviour of Hemp fibres intended for the manufacturing of high performance composites. *Compos Part A Appl Sci Manuf* 2009;40:1111–8

[3] Baley C, Le Duigou A, Bourmaud A, Davies P. Influence of drying on the mechanical behaviour of flax fibres and their unidirectional composites. *Compos Part A Appl Sci Manuf* 2012;43:1226–33

1. Introduction

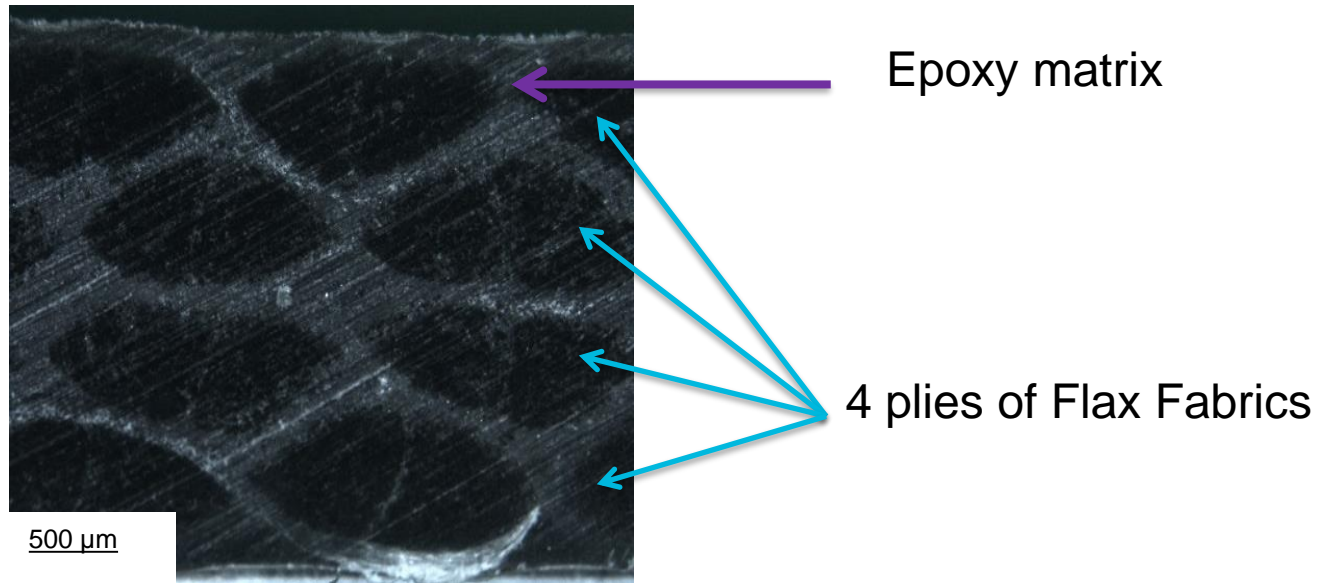
Question : Is it possible to have a well crosslinked composite with optimal mechanical properties ?

Objective of this study : Evaluating the impact of post curing on the mechanical properties of a UD flax fiber reinforced epoxy composite and identify between its components (resin, fibers or interface) which one is responsible for the obtained results

Table of Contents

- 1. Introduction**
- 2. Materials**
- 3. Influence of the post-curing on the composite**
 - 3.1 Glass transition temperature**
 - 3.2 Mechanical properties**
- 4. Influence of the post-curing on the components**
 - 4.1 Resin**
 - 4.2 Flax fibers**
 - 4.3 Interface**
- 5. Conclusion**

2. Materials



SEM image of composite
(x16)

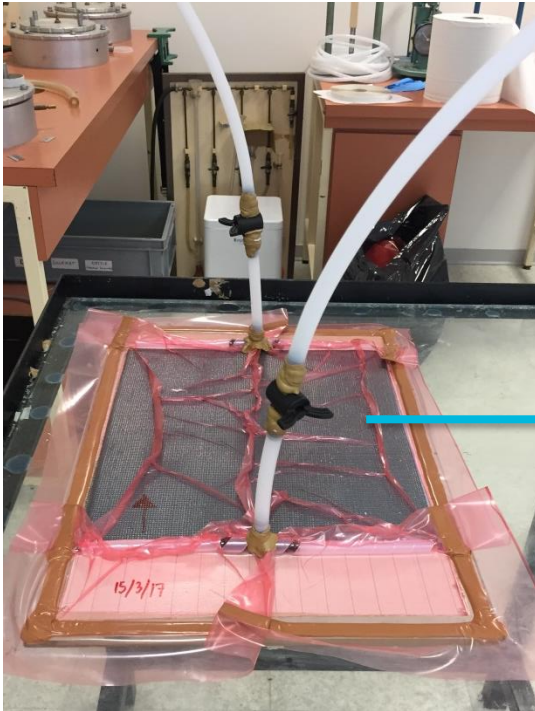
Epoxy matrix

- ❑ 80%wt DER 332 (DGEBA)
- ❑ 20%wt IPDA (hardener)

Flax Fabrics

- ❑ UD 360 (360g/m²)
- ❑ Quasi UD (weft : 330g/m² and warp : 30g/m²)

Process and post-cure



Processing : Vacuum infusion
Resin at 40°C (Viscosity of 120P/s)
→ 300x300x3mm² plates

- Cured 24hours at 80°C = NoPC
- 24hours at 80°C + 2h at 100°C = PC100
- 24hours at 80°C + 2h at 120°C = PC120
- 24hours at 80°C + 2h at 150°C = PC150

Post curing conditions reproduced :

- On resin → R-NoPC, R-PC100, R-PC120 and R-PC150
- On flax fabrics → F-NoPC, F-PC100, F-PC120 and F-PC150

Methods

→ Various series of tests to evaluate the impact of post-curing

For Composites :

- DSC (10-15mg samples, 20-200°C, 10°C/min under Nitrogen 20L/min)
- Tensile test (250 x 25 x 3mm³ samples, 100kN load cell, 1mm/min)
- ILSS (30 x 15 x 3mm³ samples, 2,5kN load cell, 1mm/min)
- SEM (FEI Quanta 200, 15kV, 100 to 5000 magnification)

For the resin :

- Tensile test (110 x 13 x 4mm³ samples, 100kN load cell, 1mm/min)

For the fibers :

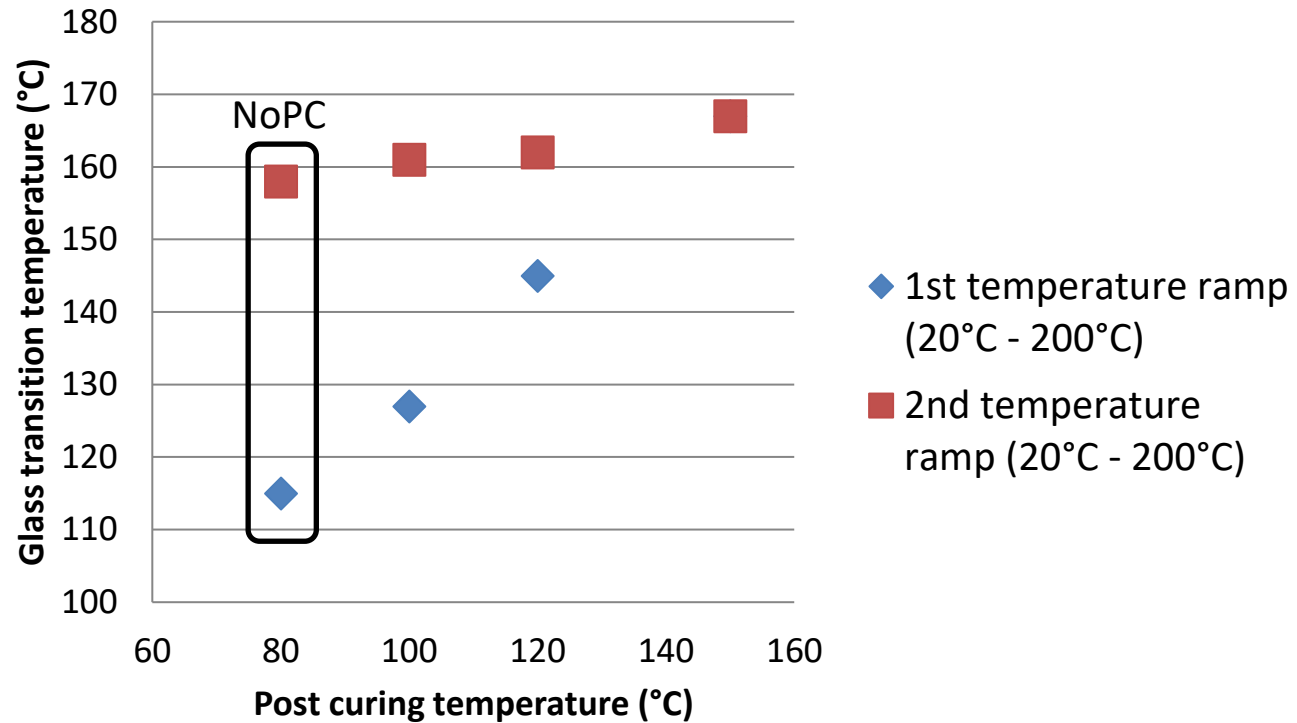
- Tensile test (10kN load cell, 1mm/min)
 - ✓ 50mm large sample
 - ✓ Gage length : 10, 25, 50,100 and 200mm

3. Influence of the post-curing on the composite

3.1 Glass transition temperature

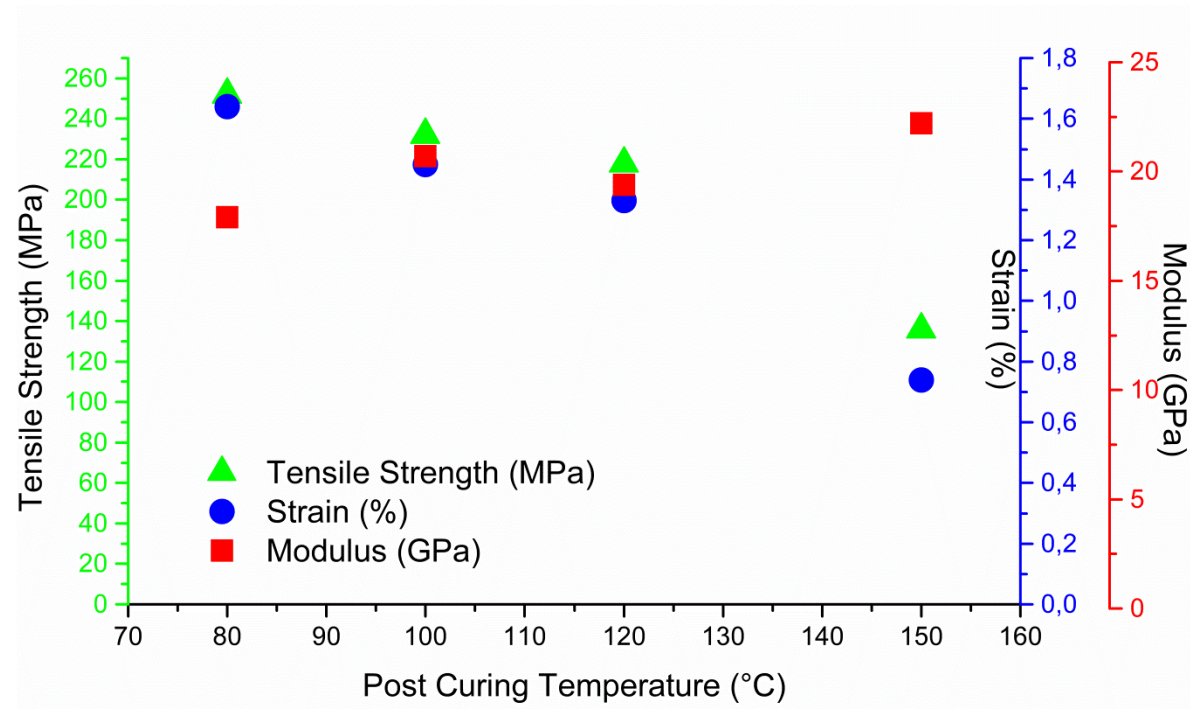
3.2 Mechanical properties

3.1 Glass transition temperature



- ✓ Determined with DSC curves
- ✓ The higher the PC temperature the higher the Tg
- ← Increase of the crosslinking rate (incomplete except for PC150)

3.2 Mechanical properties



- ✓ ↘ of the tensile strength (46% for PC150) and the elongation at break (55% for PC150)
- ✓ Determination of the modulus at 2 different strain zones (0,05-0,25% and 0,5-0,8%)
- ✓ ↗ of the Modulus when the PC temperature increases

Table of Contents

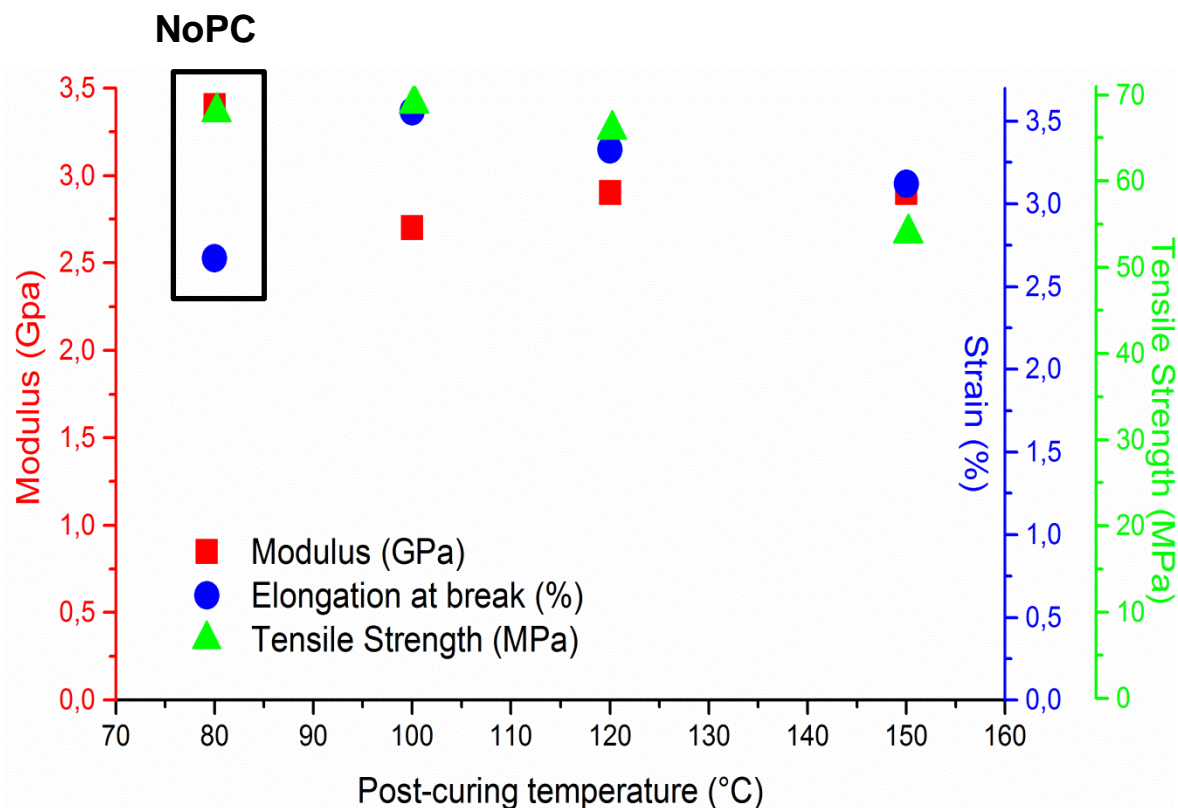
4. Influence of the post-curing on the components

4.1 Resin

4.2 Flax fibers

4.3 Interface

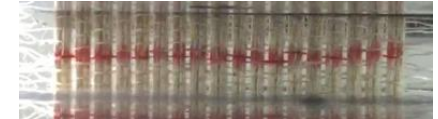
4.1 Resin



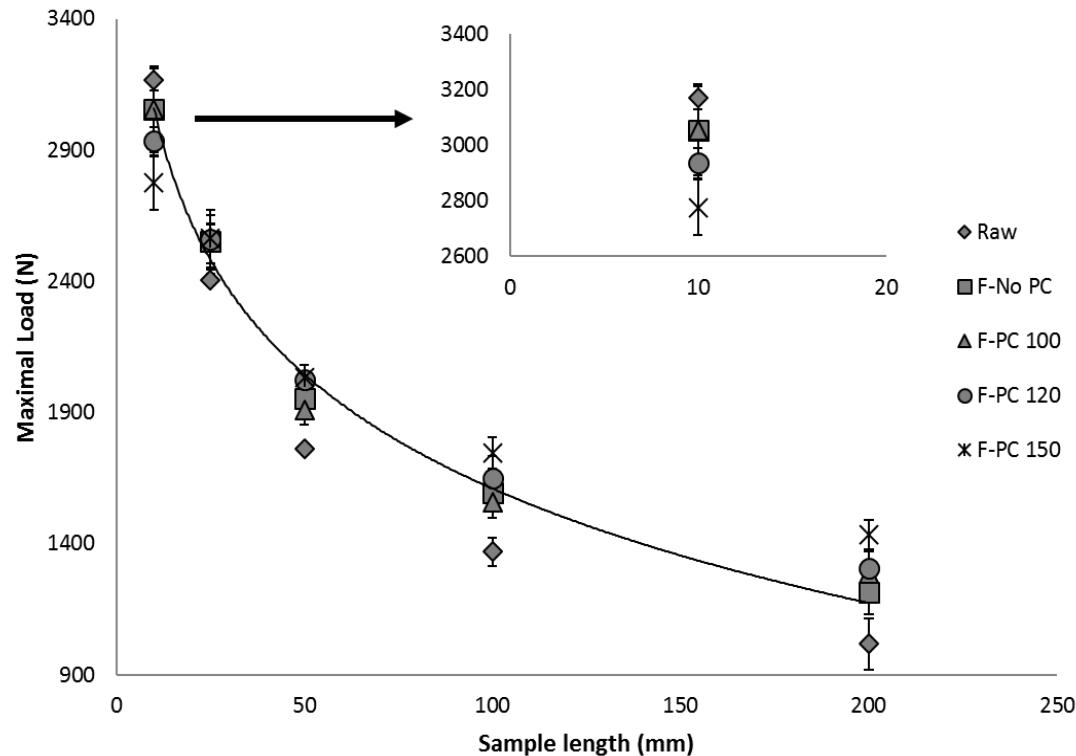
- ✓ Modulus with a 15% drop for R-PC150
- ✓ 20% of the tensile strength for R-PC150 (change of coloration = early oxidation)
- ✓ of elongation at break with post curing
- ✓ of elongation at break when $T_{\text{post curing}}$

4.2 Flax Fibers

- ✓ Tensile tests at different gage lengths 10-200mm
- ✓ Breaking mode = Mix of fiber breaking + disentanglement
- ✓ At short length → Mainly fiber breaking (10mm)
- ✓ At longer length → Mainly disentanglement (200mm)



Fiber breaking : 10mm



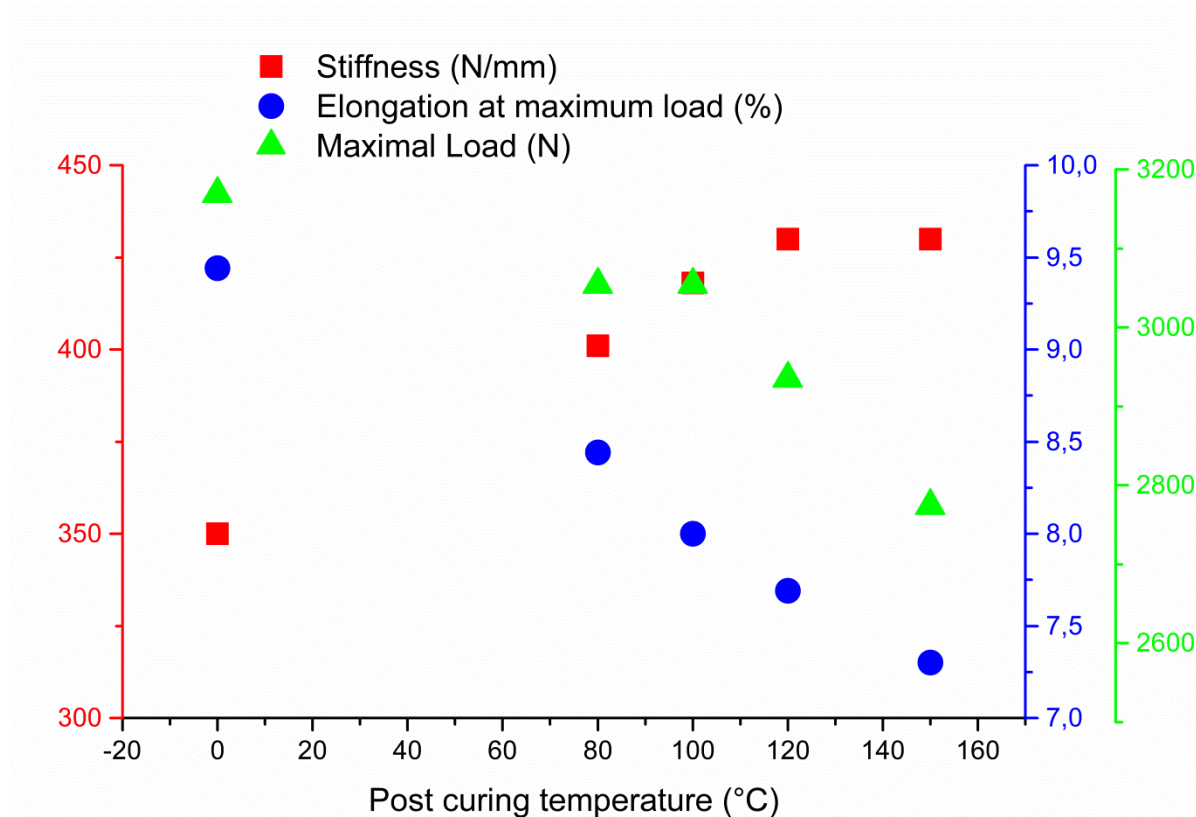
Disentanglement : 200mm

Composite tensile tests → Mode of breaking = Neat breaking → 10mm samples

4.2 Flax Fibers

Focus on 10mm samples

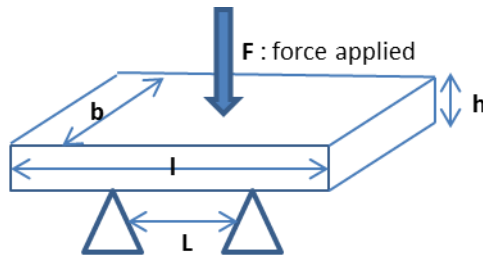
↗ of the stiffness with post cure
↘ of elongation or maximal load



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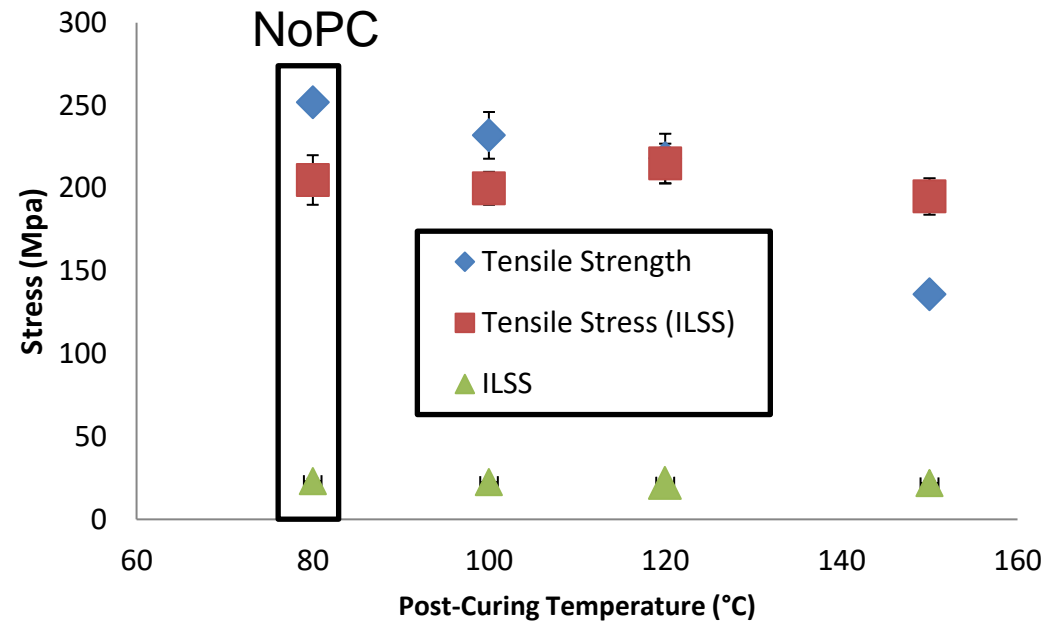
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4.3 Interface



$$\text{Tensile Stress (ILSS)} = \frac{4 F L}{3 b h^2}$$

$$\text{ILSS} = \frac{3F}{4bh}$$



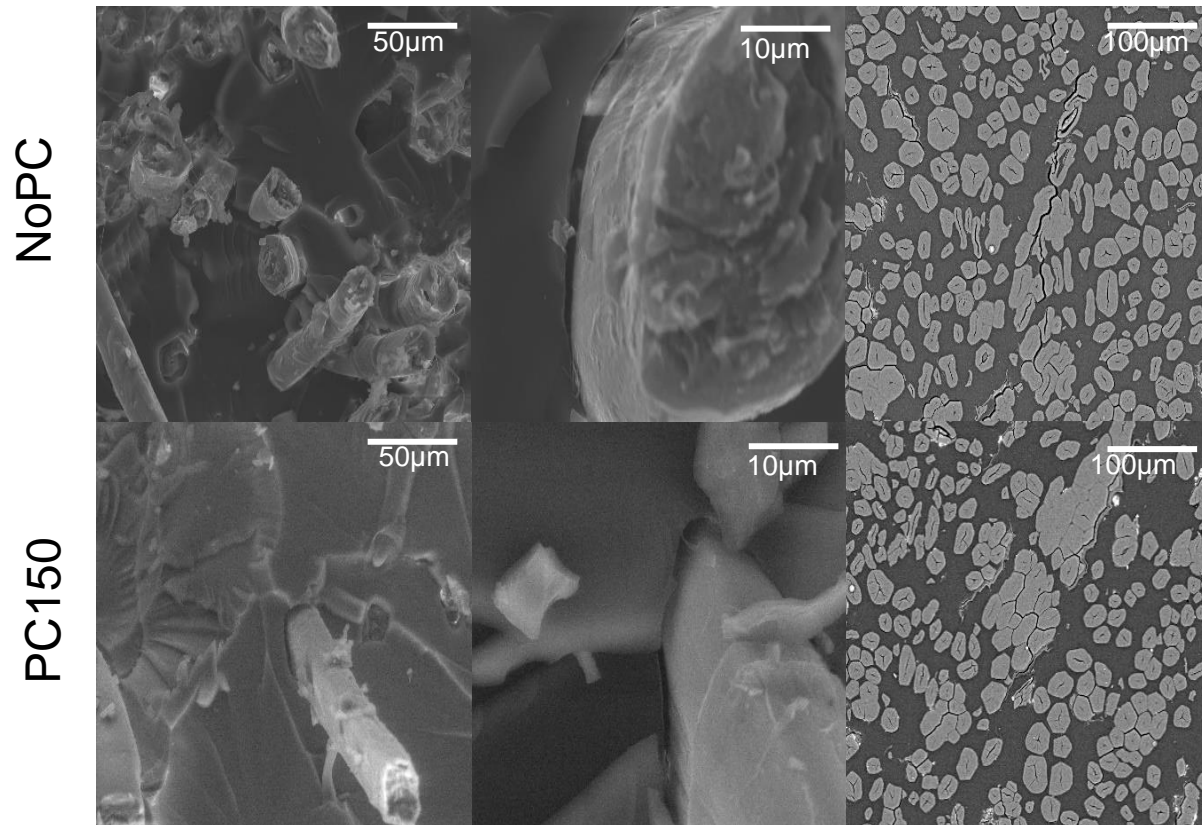
Tensile Strength > Tensile Stress (ILSS) for NoPC, PC100 and PC120

→ Broke under shear stress

PC150 : No conclusion

☐ Post Curing does not affect the ILSS while ↘ of tensile strength

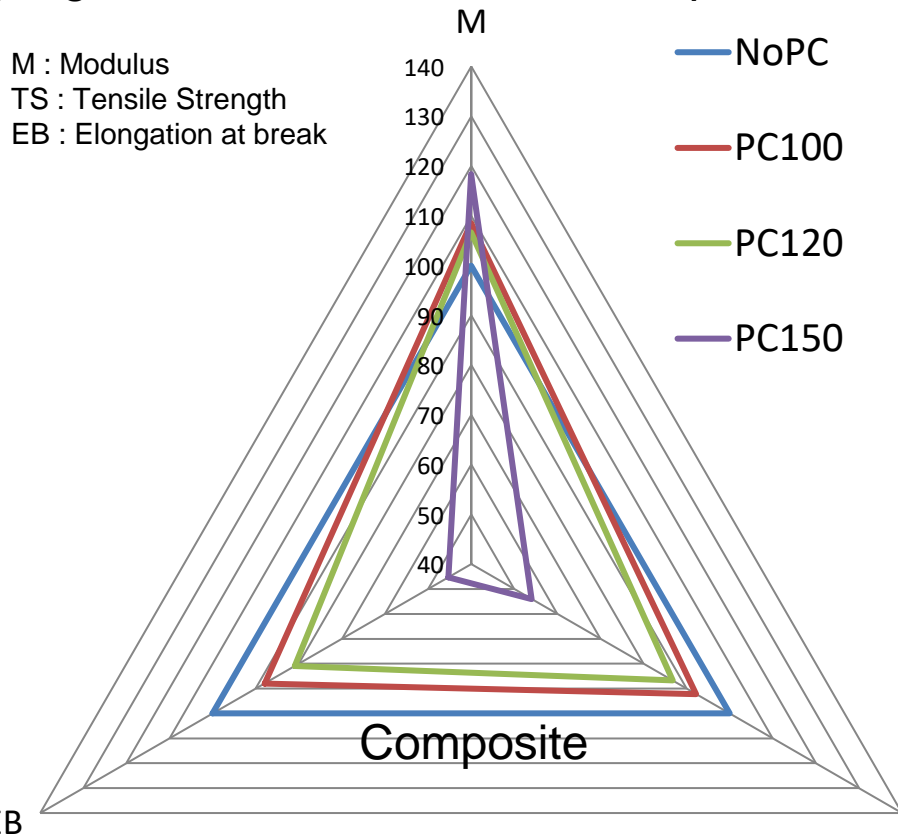
4.3 Interface



- ✓ Weak adhesion before post-curing
- ✓ No change of the interface with temperature

5. Conclusion

Objective = identify the component (fibers, resin or interface) responsible for the progressive decrease of the composite mechanical properties



up to - 20% of tensile strength + early oxidation for R-PC150

Resin

Interface

Fibers

- Weak
- Not affected by post-curing

down to - 23% of stiffness for F-PC150

- **Component responsible = fibers modified during process**
- Combining a well crosslinked resin with natural fibers = hardly possible
- Good compromise for a flax fiber reinforced epoxy composite : PC 120 → Tg of 145°C and limited drop of about 14% of the tensile strength

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Thank you for your
attention