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Chemical-etching-based observation on spherulitic and interfacial morphologies of fiber reinforced thermoplastics

- How cooling rate affects the interlaminar bond strength of UD GF/PP composites

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FLANDERS INNOVATION & ENTREPRENEURSHIP SIM Clusters for Growth

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Presentation overview

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Background: Bond strength development

CHAPTER 2
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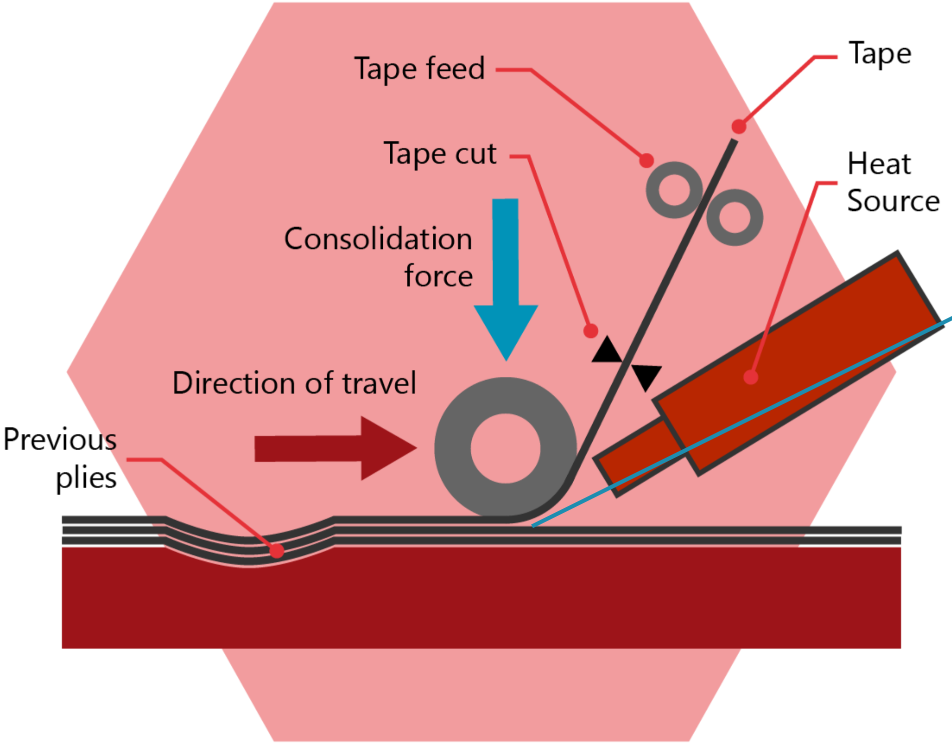
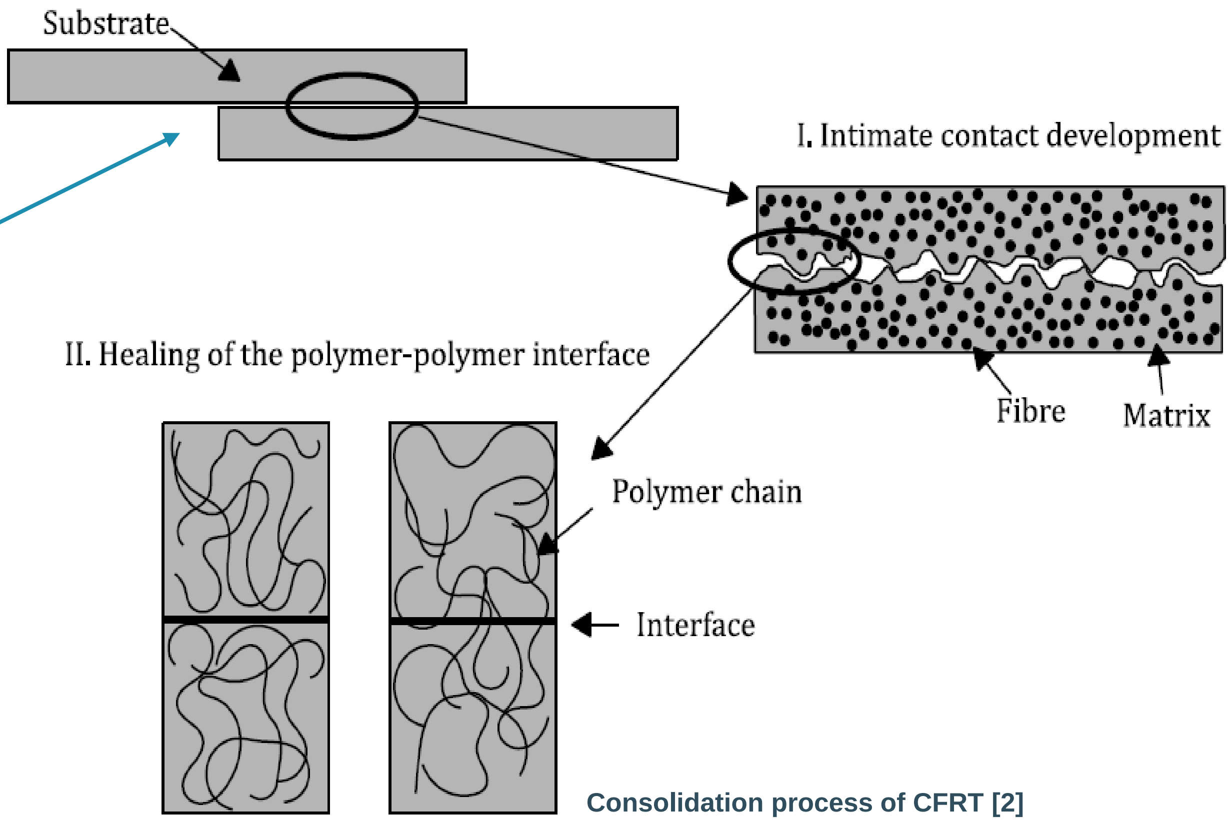
Bond strength development



Scheme of automated tape placement[1]



Consolidation process of CFRT [2]

- Consolidation is a function of temperature, pressure, and time, thus highly depends on **thermal history**;
- Bond strength is built up with the development of interdiffusion and entanglement of polymer chains, hence dominated by **polymer matrix**!

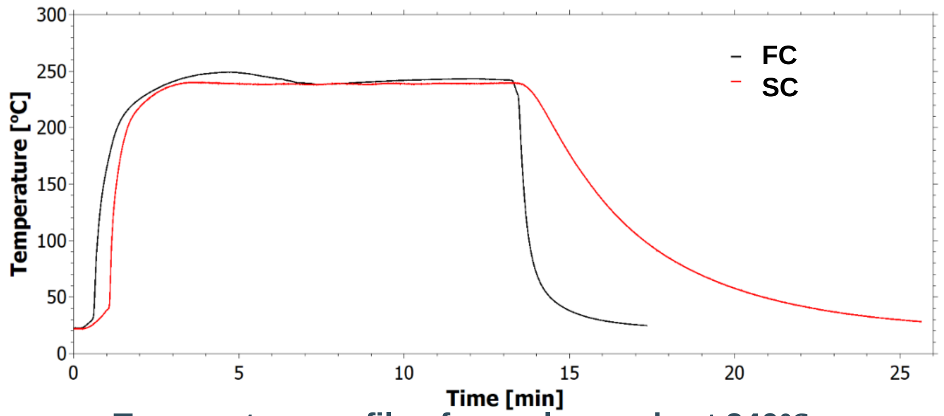
Cooling rate!

[1] https://compositesevolution.com/resources/what-is-automated-fibre-placement-afp/
[2] Sacchetti, F. R. (2017). Interlaminar toughness of fusion bonded thermoplastic composites. University of Twente.

Materials and experiments

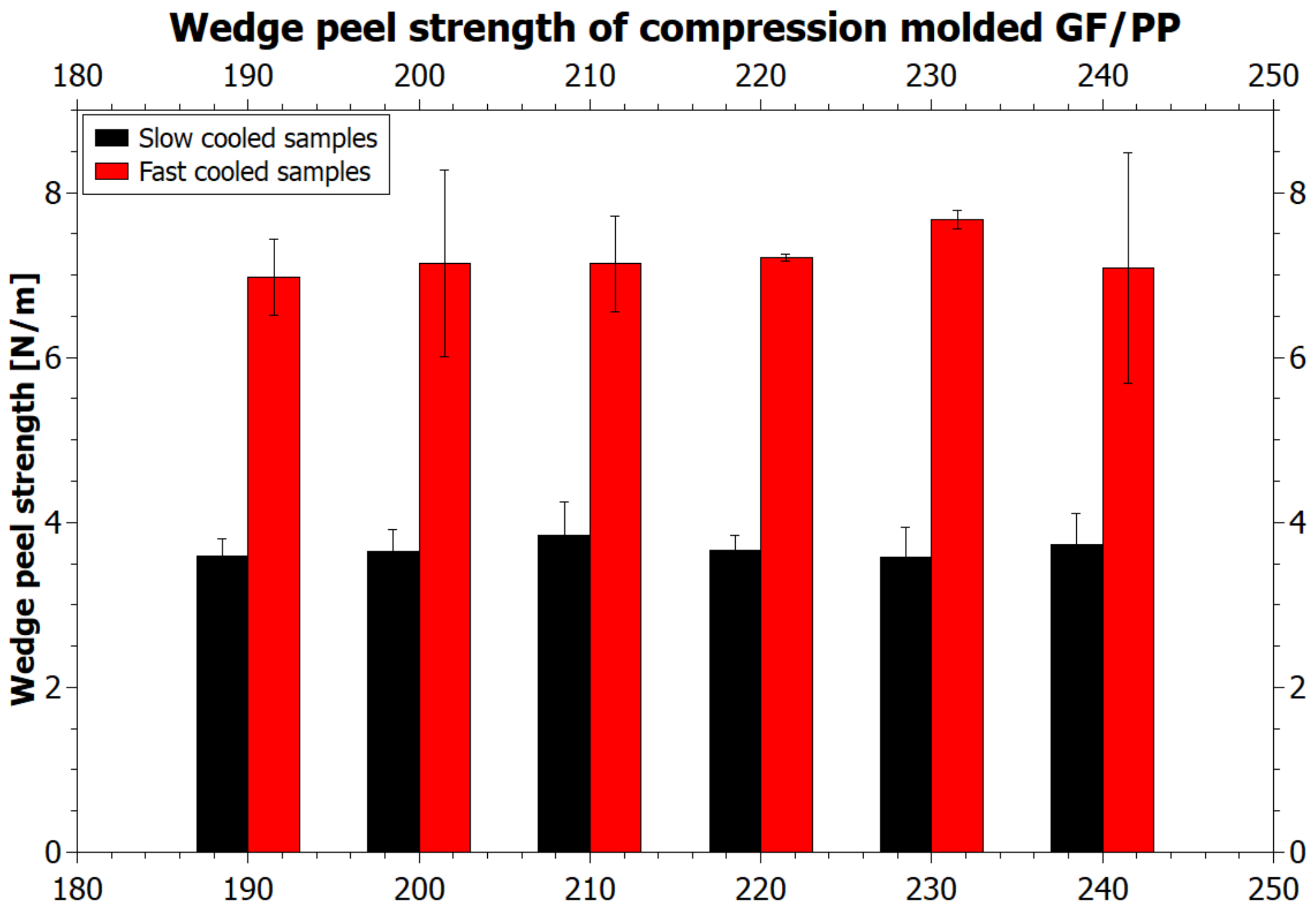
Materials:
- Glass fiber reinforced polypropylene (**GF/PP**) UD laminates $[\mathbf{0}°]_{\mathbf{4}}$ by compression molding

Consolidation parameters:
- Isothermal temperature: 190 ℃ ~ 240 ℃;
- Holding time: 10 min;
- Pressure: 0.7 MPa;
- Different cooling rate



Temperature profile of samples made at 240℃

	at 200 °C	at 166 °C	at 102 °C
Slow cooled samples (**SC**)	51 K/min	42 K/min	23 K/min
Fast cooled samples (**FC**)	604 K/min	484 K/min	206 K/min



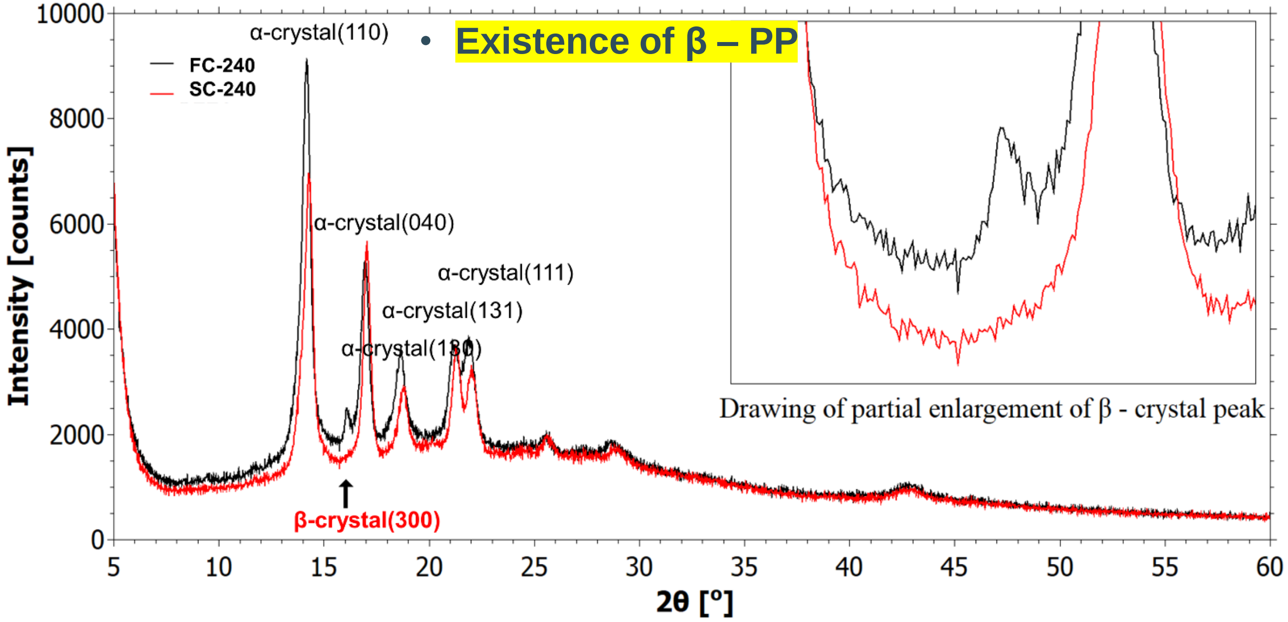
Remark:
- For semi-crystalline GF/PP, the higher the cooling rate, the higher the bond strength

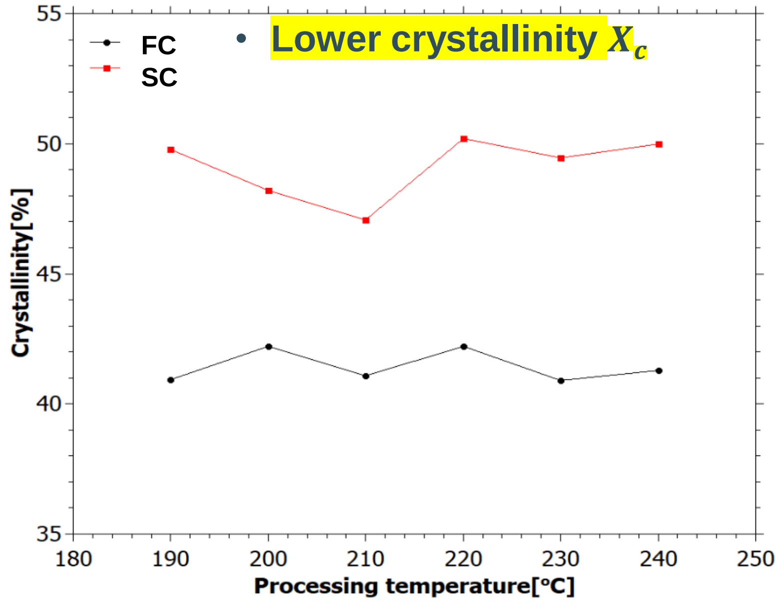
[1] Schäfer, P. M. (2017). Consolidation of carbon fiber reinforced polyamide 6 tapes using laser-assisted tape placement (Doctoral dissertation, Technische Universität München).

Materials: Crystallization

PP: semi-crystalline (α, β, γ, δ ……)

XRD and DSC analysis:





• Smaller crystallite size X_s

	Average crystallite size X_s [Å] of α – PP crystals at different 2θ				
	14,14°	16,87°	18,58°	21,11°	21,86°
SC samples	165,17	221,83	195	185,83	168,33
FC samples	167,67	207,17	162,17	144,17	145,33

➢ From the perspective of materials, more amorphous PP, fine α – PP, and β – PP exhibit higher ductility, significantly enhancing the bond strength of fast cooled GF/PP

The underlying and direct causes?

Performance determined by the **microstructure!**

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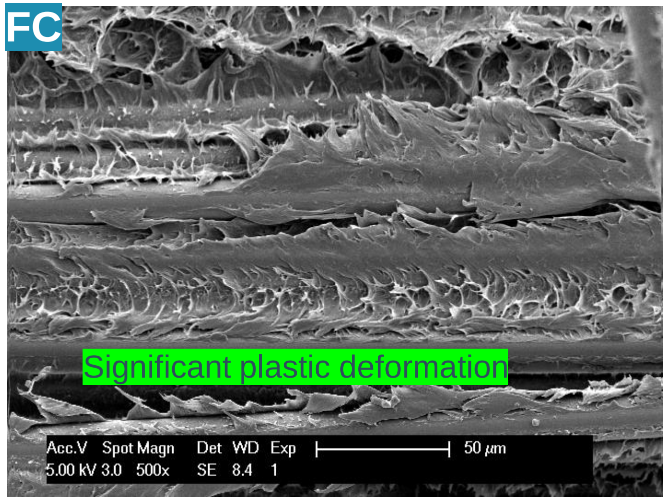
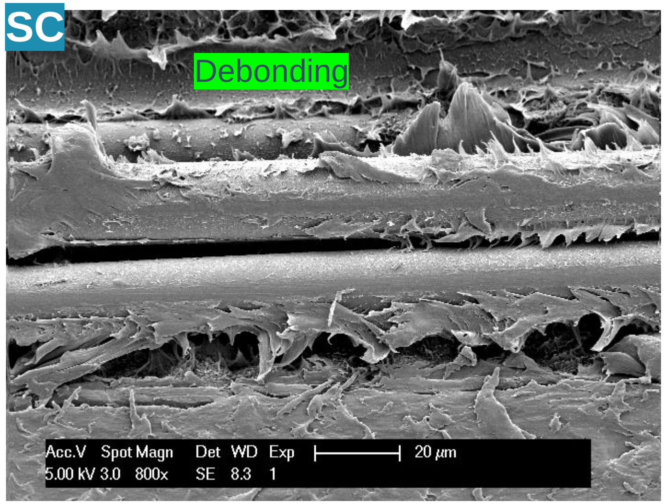
Microstructures: chemical etching

Delaminated surfaces:





(Near GF surface) Matrix failure!

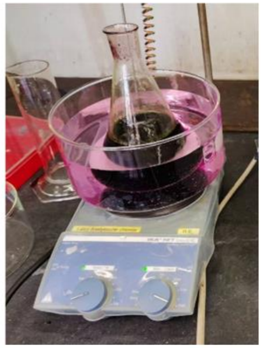
→ How to visualize the (near GF surface) morphology?

CE

Chemical etching (CE):
The **permanganic acid etchant** preferentially etches the amorphous part of the polymer in the spherulites, in such a way that the lamellae then clearly appear [1]

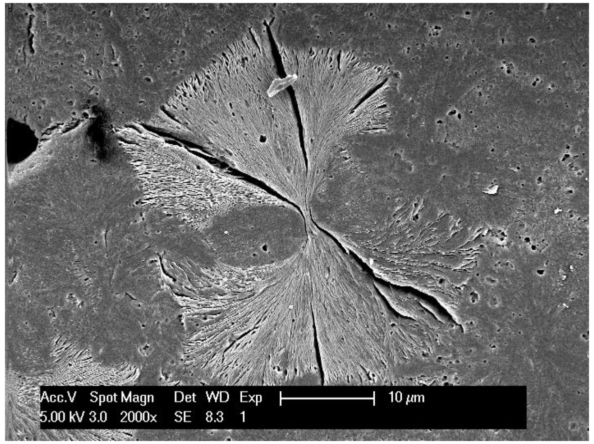
(Polished epoxy embedded) samples

Permanganic acid etchant



Rinse and drying

Sputtering, SEM



Revealed PP spherulite by CE

[1] Lu, Z., & Zhang, K. F. (2009). Morphology and mechanical properties of polypropylene micro-arrays by micro-injection molding. The International Journal of Advanced Manufacturing Technology, 40(5), 490-496.

Microstructures: F/M morphology

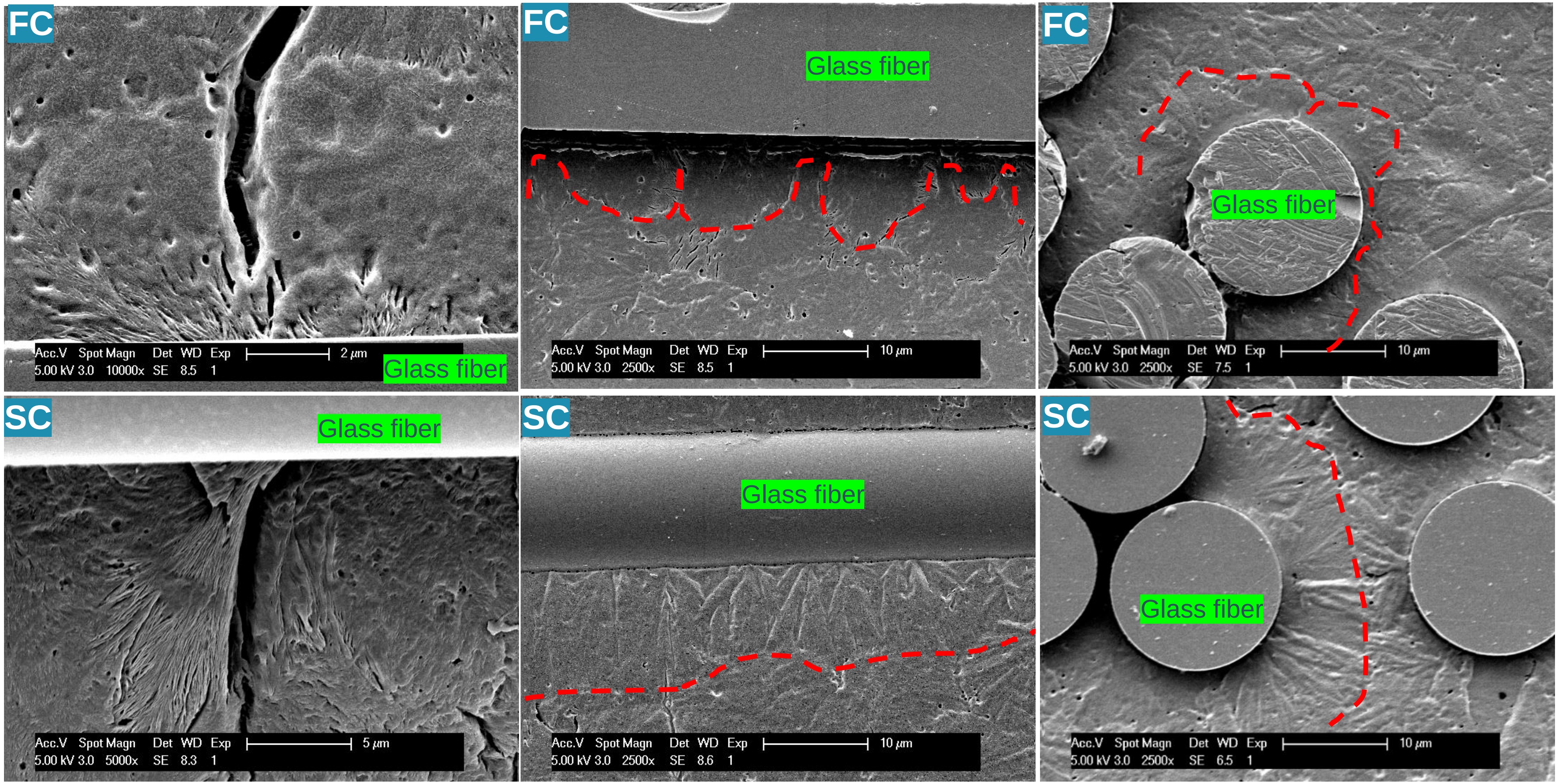
As-manufactured laminates, after epoxy embedding, polishing, etching:

- Fiber nucleation;
- Different growth tendencies;
- Different F/M morphology.

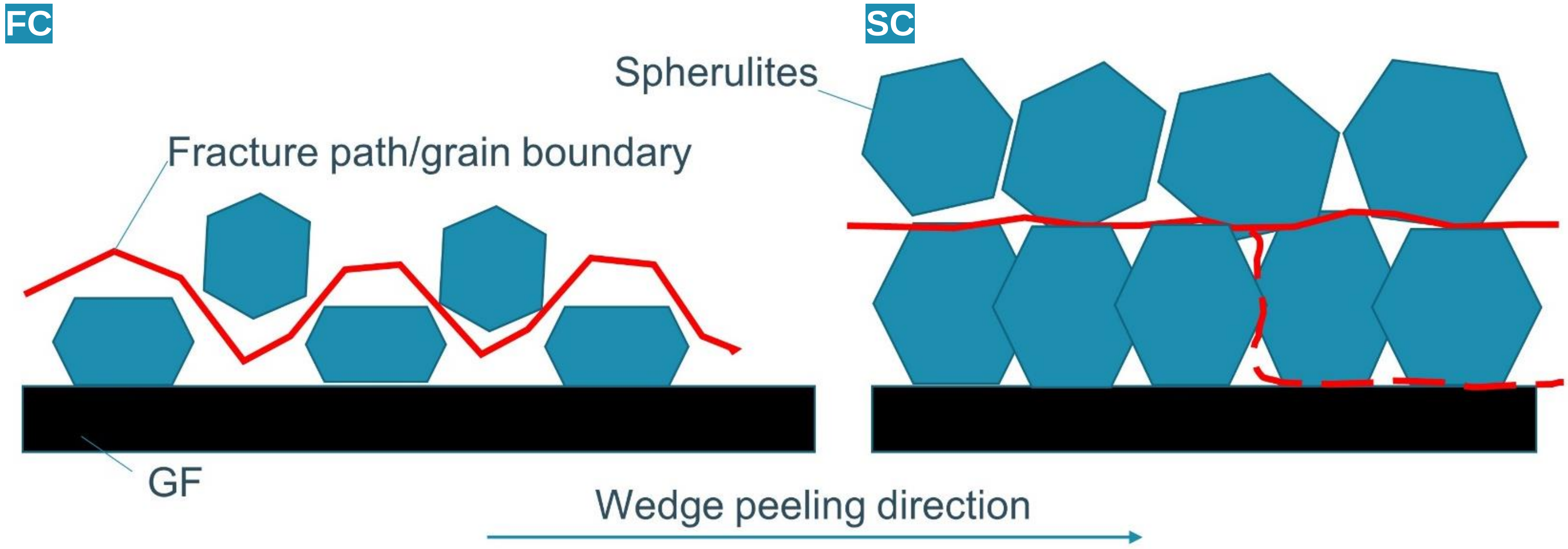
Crack propagates along the grain boundary

FC:
- Spreading growing;
- Semi spherulites;
- More amorphous PP, and fewer spherulites.

SC:
- Perpendicular growing;
- (Like) transcrystalline structures.



Microstructures: F/M morphology



Interlocked grain boundary, with more amorphous PP in between spherulites, higher fiber-matrix adhesion

Relatively flat and straight grain boundary parallel to GF surface (also wedge peeling direction), poor fiber-matrix adhesion

Conclusion

- ✓ High cooling rate induces lower crystallinity, smaller crystallite size, and β-crystals, resulting in a more ductile matrix;
- ✓ Different cooling rate makes differences in fiber-matrix morphology, resulting in different crack propagation paths and hence the different level of bond strength

Thank you!

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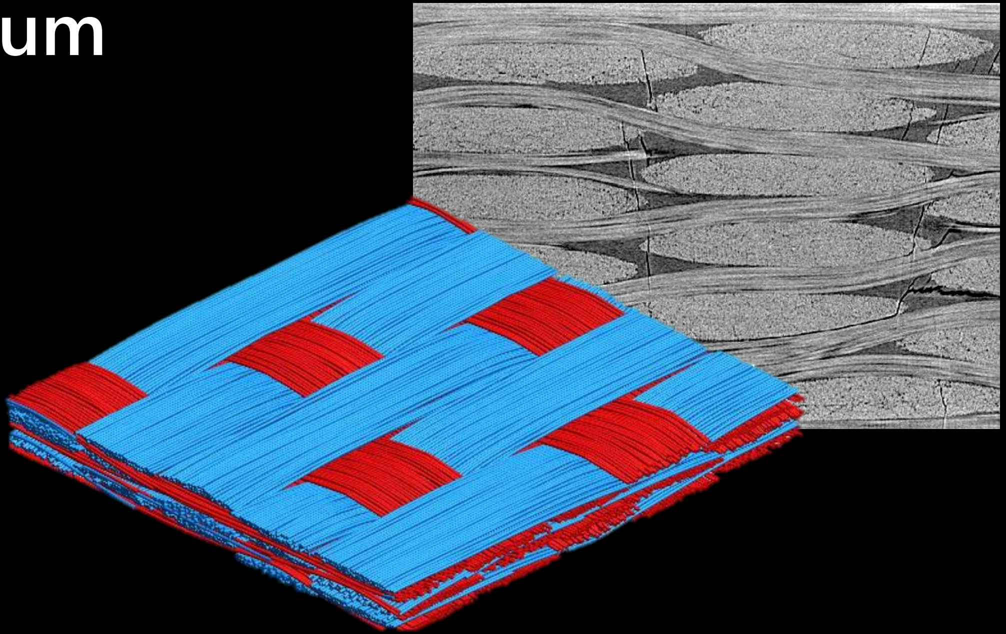
Abstract submission deadline   31 Mar 2024



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