IMT Nord Europe
École Mines-Télécom
IMT-Université de Lille

Région Hauts-de-France

Université de Lille

EDSMRE
Ecole Doctorale - 104
Sciences de la Matière, du Rayonnement et de l'Environnement
Université Lille Nord de France

Multi-modal imaging for porosity quantification in partially-impregnated UD woven glass fibre/ polypropylene composites

ICCM 23
BELFAST 2023

Mr. S. Sidlipura*, Dr. A. Ayadi*, and Dr. M. Lagardère-Deleglise*

IMT Nord Europe, Institut Mines-Télécom,

Centre for Materials and Processes, F-59000 Lille, France

(*) Correspondence: abderrahmane.ayadi@imt-nord-europe.fr

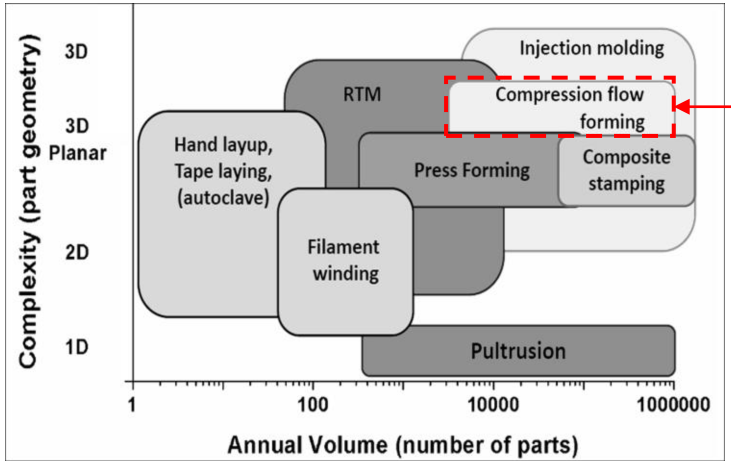
#IMTNordEurope

Table of Contents

IMT Nord Europe
École Mines-Télécom
IMT-Université de Lille

Industrial Context
Porosity in polymer-matrix composites

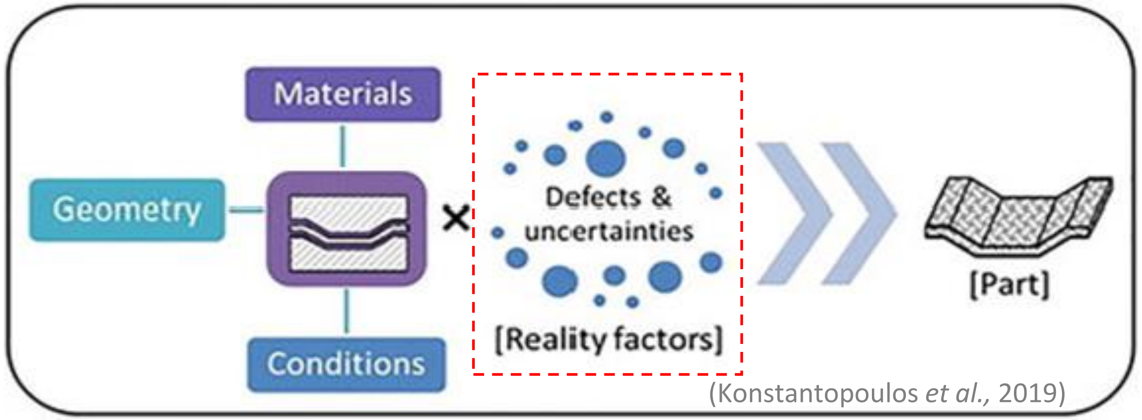
1. Classification of composite manufacturing processes



Current Study

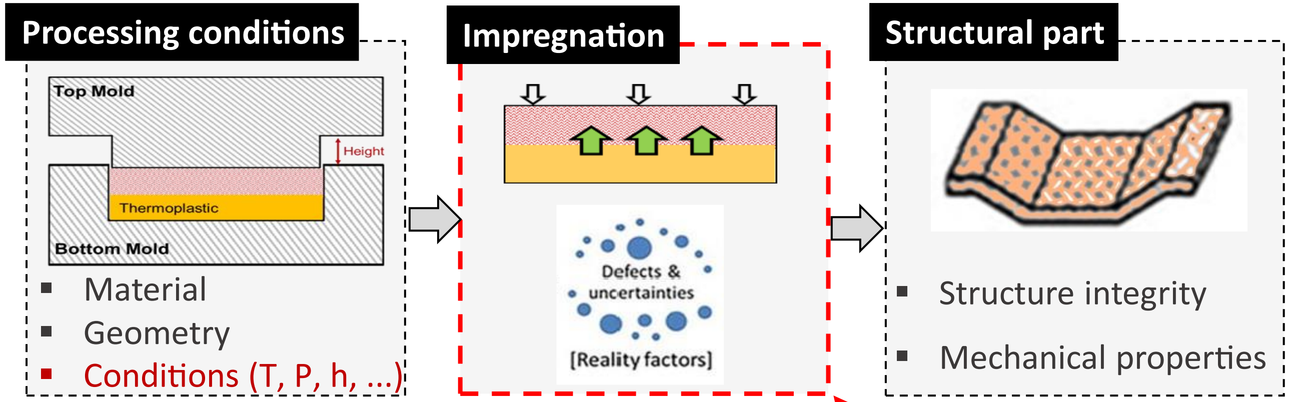
(Gupta S.K)

2. Process-induced defects



(Konstantopoulos *et al.*, 2019)

3. Compression flow forming: Case of thermoplastic (TP) matrix



Current Study

4. Residual porosity: a potential defect

Porosity can refer to one of the following flaws:

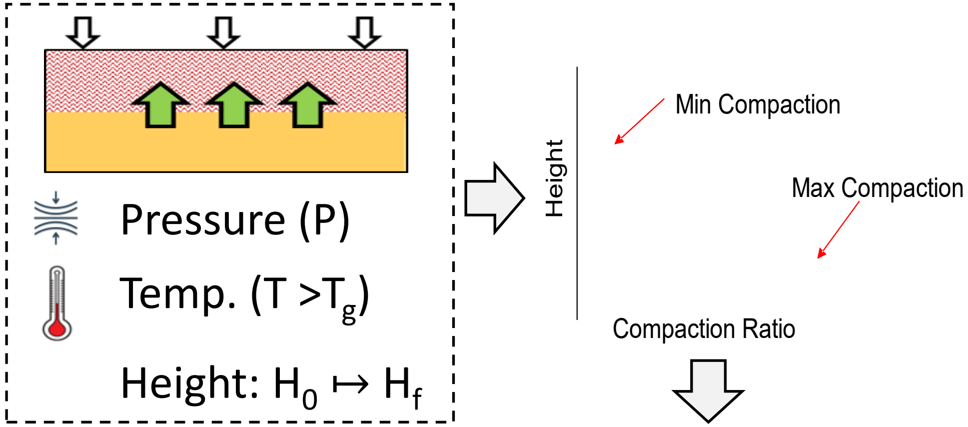
- **Unfilled reinforcement regions;**
- **Trapped air bubbles (void) ;**
- Cavities caused by the shrinkage of TP matrix

(Zuhudi *et al.*, 2021)
(Mehdikhani *et al.*, 2019)

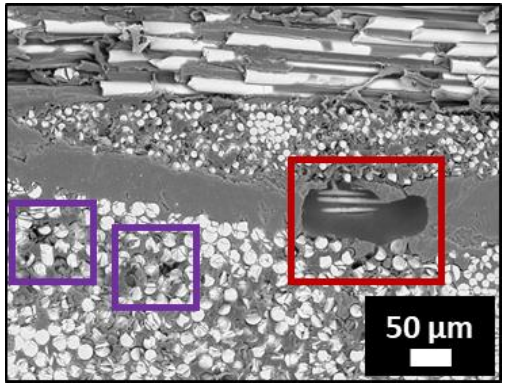
Scientific Context

Coupled thermo-mechanical physics at multiple scales

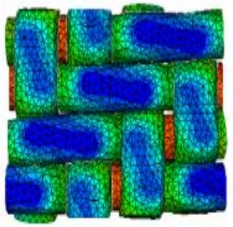
Compression Flow Modeling Process



- SEM micrograph: Porosity (trapped air bubbles/void)
  - **Inter-bundle**
  - **Inter-fibre**



Mechanical deformation (Structural mechanics)



(TexGen -MICRA)

→ Decrease in permeability 'K'

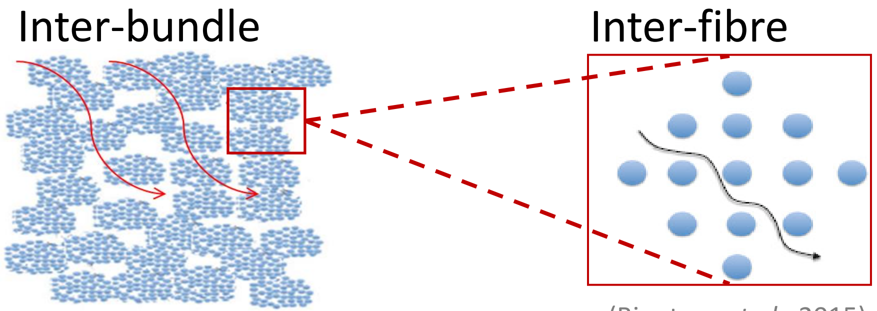
→ Mesoscale (preform permeability)

→ Microscale (Bundle permeability)

Melt TP matrix (Fluid mechanics)

Darcy's Law: (1D transverse flow)

$$\vec{v} = -\left(\frac{K}{\mu}\nabla P\right)$$



(Binetruy *et al.*, 2015)

Objective: To identify a correlation between formation of **residual porosity** with the **compaction ratio** as a processing control parameter?

Non-extensive state of the art: context of composite materials

Existing Imaging Methods and Techniques

Type	Imaging Tech.	Structural Analysis & Scale							
		Macro (plate)		Meso (yarn)			Micro (single fiber)		
		Fiber Bed	Porosity	Yarns	Porosity	Degree of Imp.	Single fiber	Porosity	Degree of Imp.
2D (+ polishing)	OM	[7] CF/epoxy prepreg	[7, 8] CF prepreg; GF/epoxy (Size & Shape, A_f)	[9] CF/epoxy (Shape)	[10] GF/epoxy (V_f)	[11] Woven CF/PA6	[12] UD CF/PEEK prepreg (Avg. diameter)	[13] CGSC/SiNC (V_f)	[11] Woven CF/PA6
	SEM	[14]CF/epoxy prepreg	[15] CMCs (V_f)	[16] 3DAW-CF/Al	[17]CF/PA66 (A_f)	[18] AF/PLA	[19] SiCf/SiC (Avg. diameter)	[20] Plain Weave CF /PA66 (V_f)	[20] CF Plain Weave/PA66
	FM		[8] GF/epoxy (Size & Shape, A_f)						
3D NDT	µ-CT	[21] Cross-ply GF/epoxy	[7, 22] CF/epoxy; PEEK prepreg (V_f; 3D localization)	[23] Woven CF/epoxy	[23] Woven CF/epoxy (V_f)	[18] AF/PLA		[13, 24] CGSC/SiNC; SiCf/SiC (V_f)	[25] Knitted GF/PP

[*] : Ref. reporting the use of multiple imaging techniques

- There is no explicit protocol for combining multi modal imaging for TP matrix composites
- In this study, priority is given to combining FM and SEM (µ-CT technique as a perspective)

(Little et al. 2012); (Abdelal and Donaldson 2017); (Khaled et al. 2021); (Gagani et al. 2018); (Ishida et al. 2020); (Oromiehie et al. 2019); (Santhosh et al. 2018); (Breister et al. 2020); (Purslow 1984); (WANG et al. 2021); (Ekoi et al. 2021); (Zou et al. 2022); (Tatlisu et al. 2011); (Liu et al. 2015); (Sabuncuoglu et al. 2020); (Amedewovo et al. 2023); (Mehdikhani et al. 2018); (Gao et al. 2022); (Ayadi et al. 2019)

IMT Nord Europe
École Mines-Télécom
IMT-Université de Lille

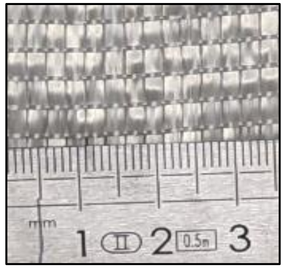
Experimental Work
Manufacturing protocol of UD Woven GF /PP Composites

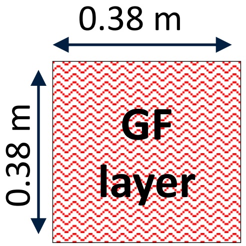
ICCM 23 BELFAST 2023

❑ Material

❑ Reinforcement

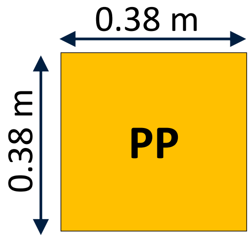
- UD NCF GF – Chomarat Ruban 8089HM
- Areal weight – 1054 g.m^{-2}
- Density – 2.55 x 10^{6} g.m^{-3}



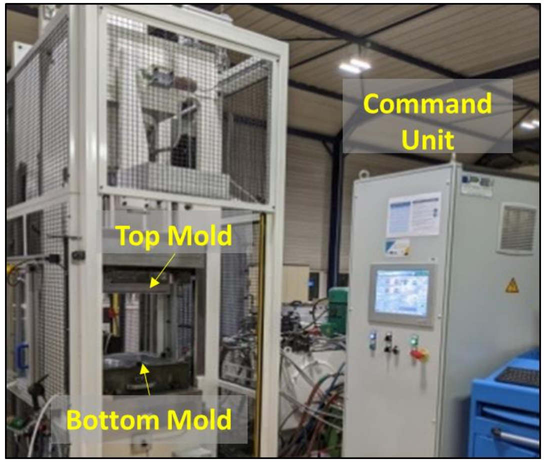


❑ Thermoplastic Films

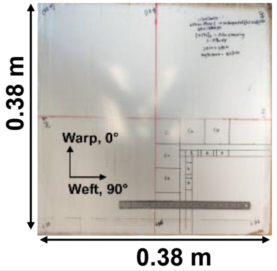
- Polypropylene – Total, PPC 13442
- Density – 0.905 x 10^{6} g.m^{-3}
- Melt Flow Index – 100g/ 10 min
- Melting Temperature – 165 °C



❑ Compression Flow Forming: Pinette Press



- 120 KN Press
- Disp. precision (0.1 mm)
- Mold : 0.38 m x 0.38 m

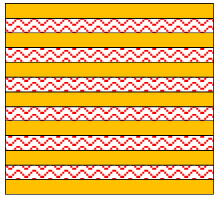


❑ Plate Manufacturing

- No. of plies – **6 GF + 7 PP Films**
- Stacking – **[0/90]$_3$**
- Size – 0.38 m x 0.38 m

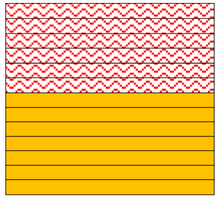
➢ Through thickness flow
➢ Deformation of fiber bed

Config. 1



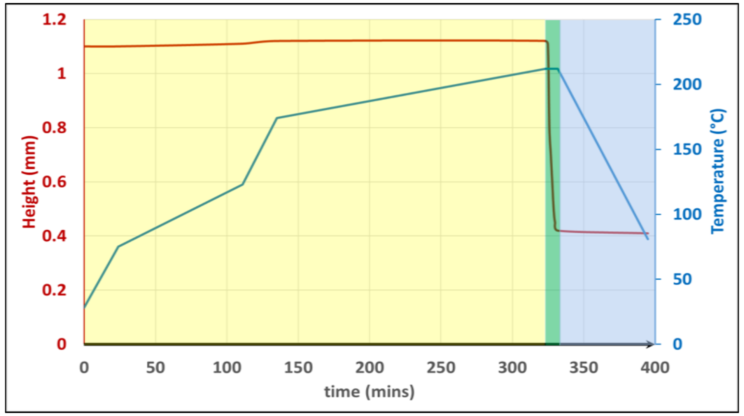
- Ref. plate
- Open mould

Config. 2

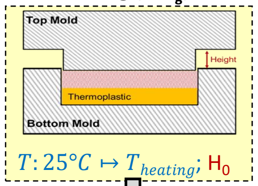


- Other plates
- Height ≠ cst

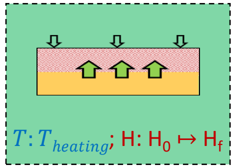
❑ Displacement-controlled manufacturing of plates



Heating (T>T$_g$)



Impregnation



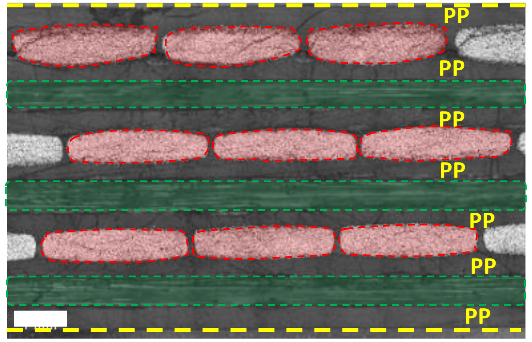
IMT Nord Europe
École Mines-Télécom
IMT-Université de Lille

Experimental Work

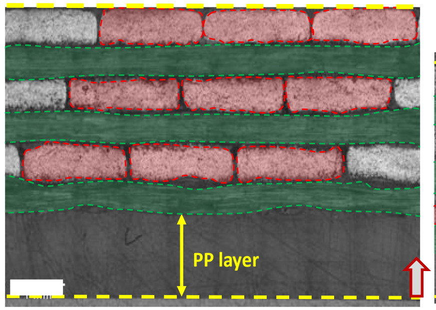
Manufacturing of partially-impregnated UD Woven GF /PP composite plates

❑ Optical microscopy observations

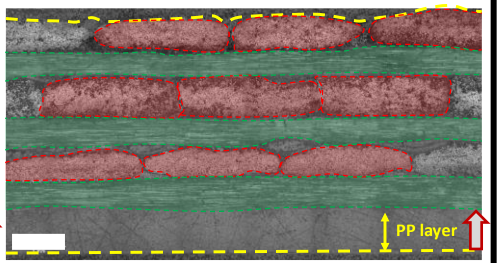
- **Ref. plate (Cr=0%)**

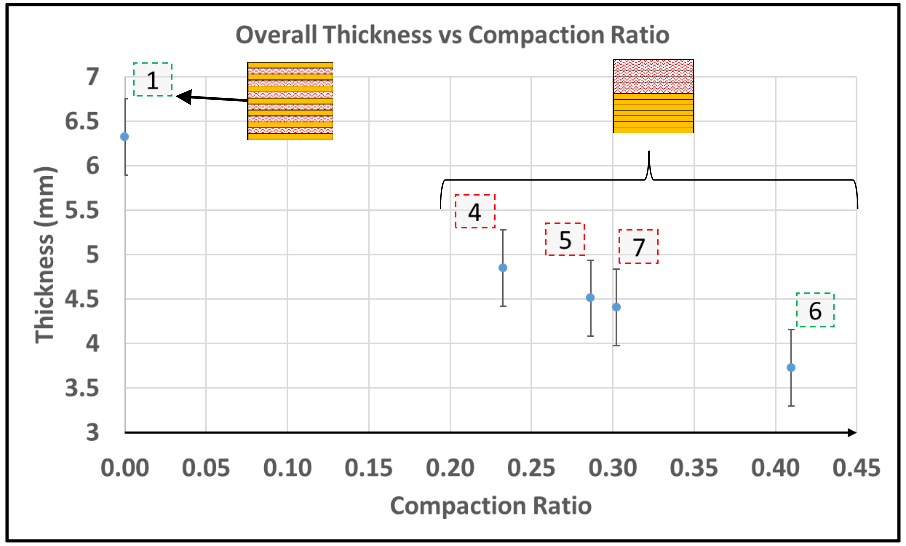


- **Plate 4 (Cr=23%)**

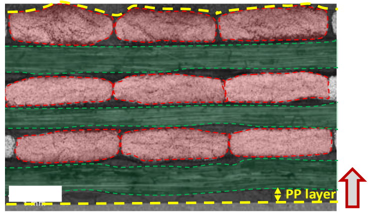


- **Plate 7 (Cr=30%)**





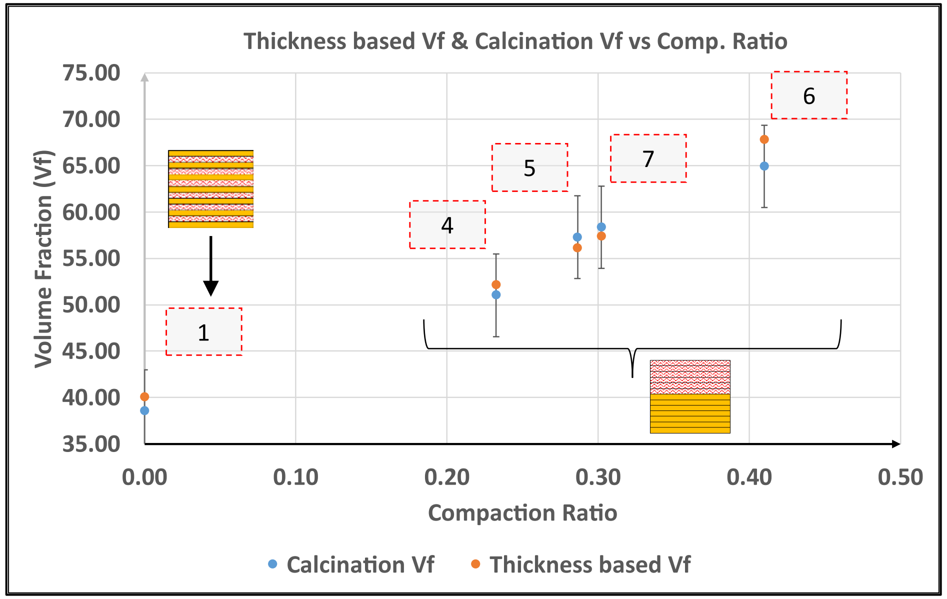
- **Plate 6 (Cr=41%)**



⇧ Transverse flow direction

1 mm Scale

❑ Calcination tests: ASTM D3171



❑ Macro-scale observations

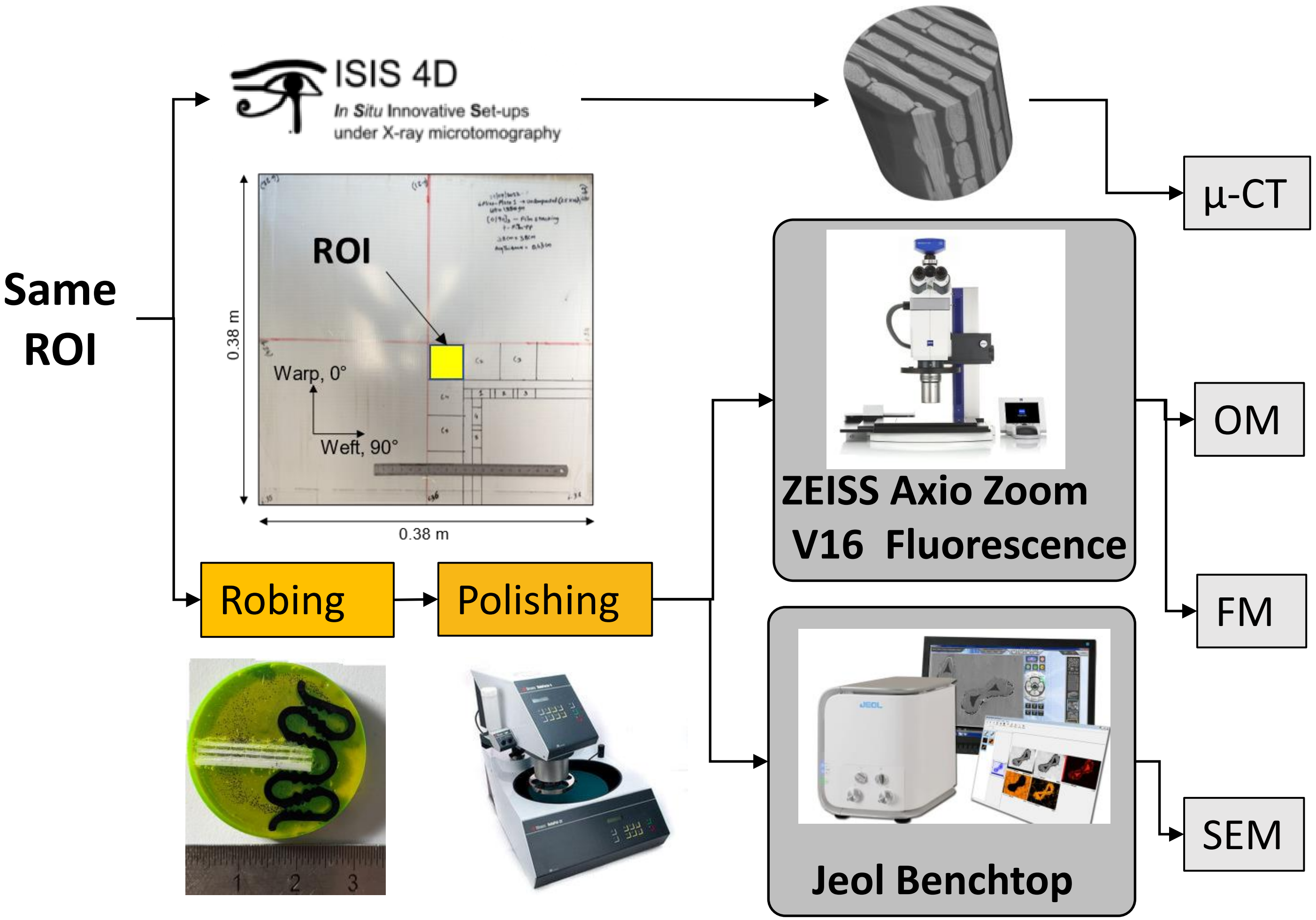
The change of Cr % can be used to manufacture **partially-impregnated** plates of different:

- ➢ V_f
- ➢ Bundle shapes
- ➢ Residual thickness of PP layer

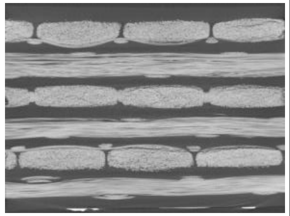
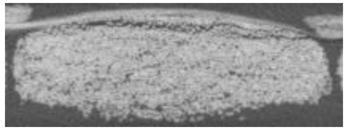
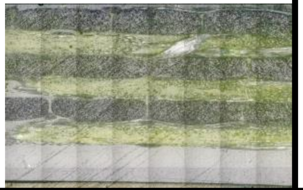
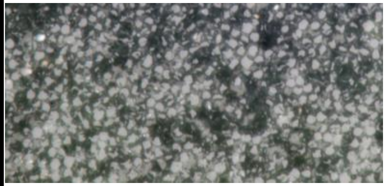
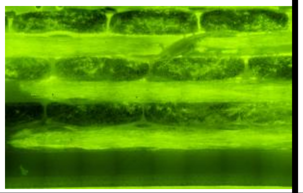
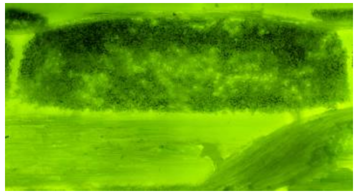
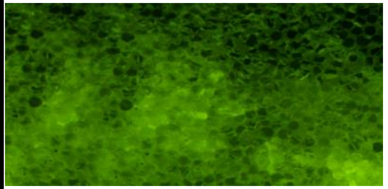
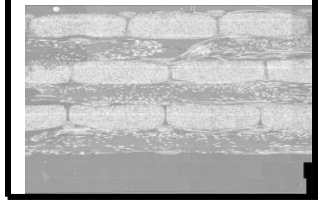
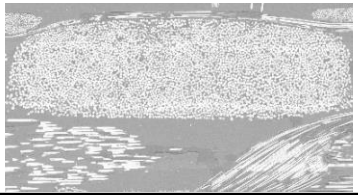
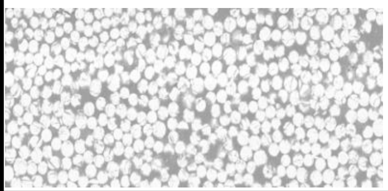
Preparation for Microstructure Analyses

Image based analysis for porosity quantification

❑ Microstructure Analyses: Image-based

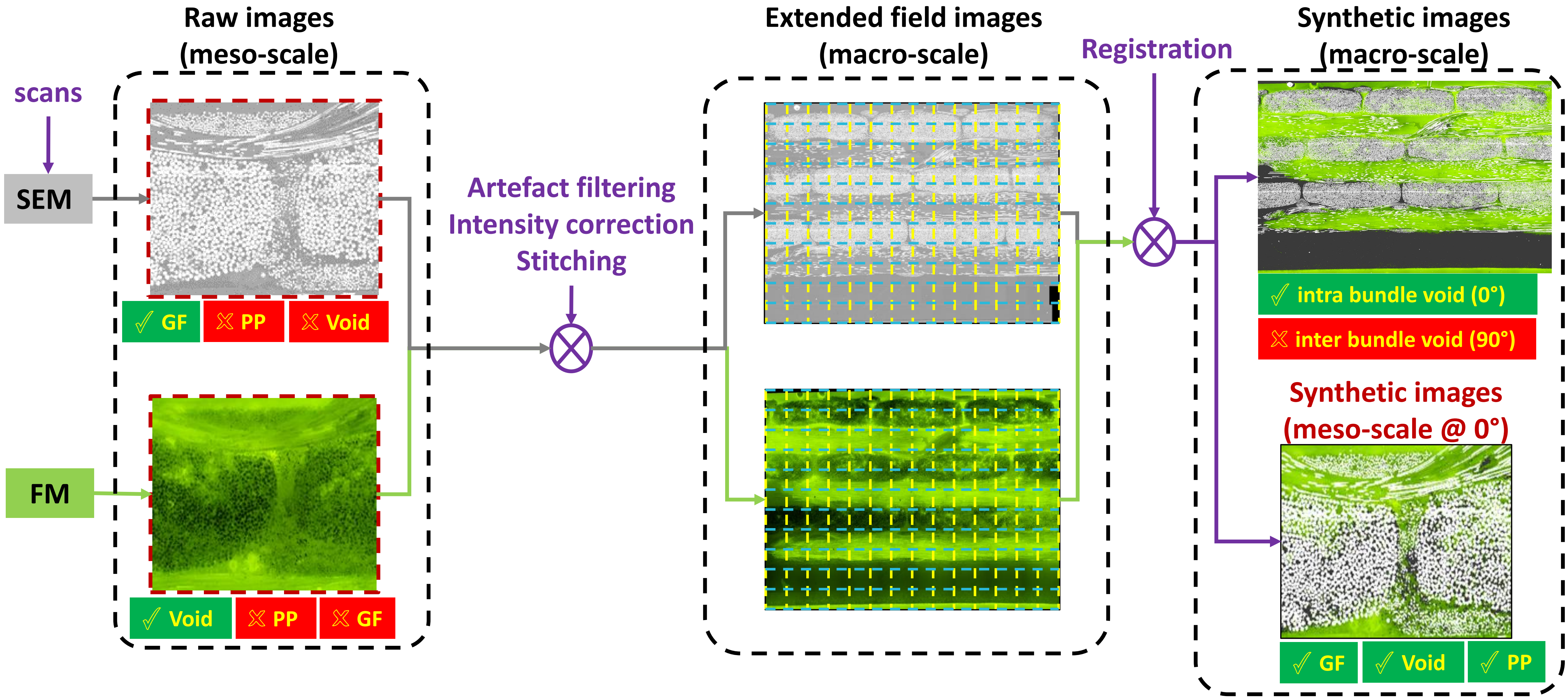


❑ Example of output images

Macro Scale (Plate thickness)	Meso Scale (Yarn Cross-section)	Micro Scale (Single fiber)
		
		
		
		

Development of Multi-modal Imaging Technique

Same microstructure / different imaging modes: SEM & Fluorescence Microscopy

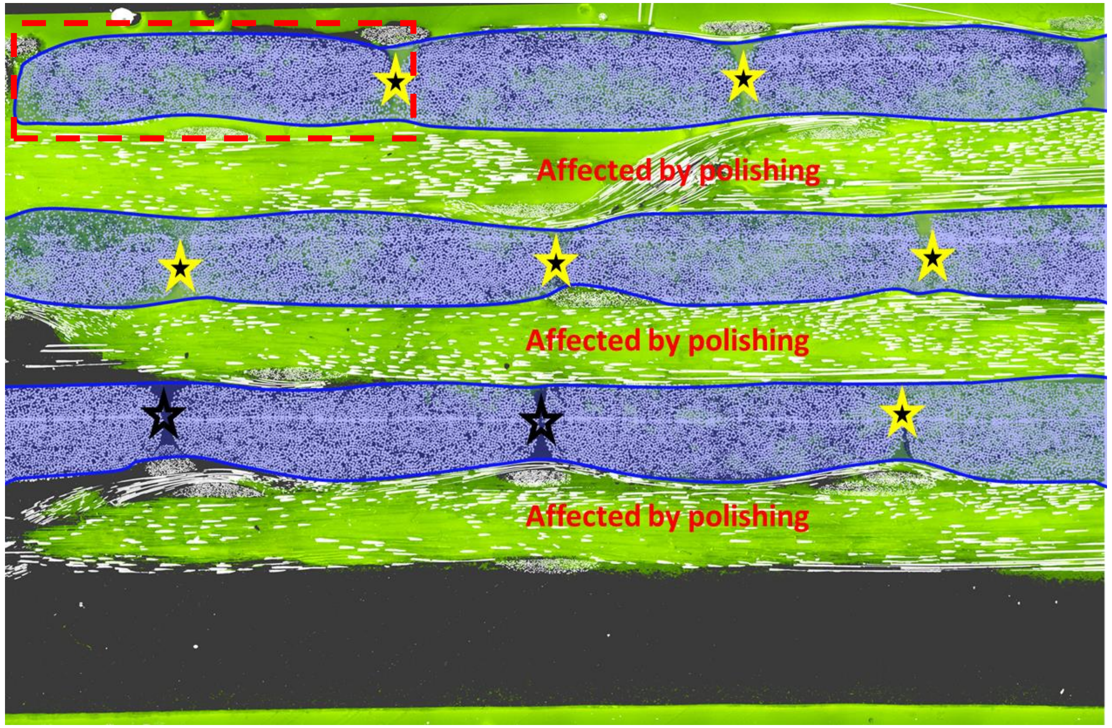


Qualitative Analyses of combined FM-SEM images

Advantages and limitations of the suggested FM-SEM based protocol

❑ Macro Scale

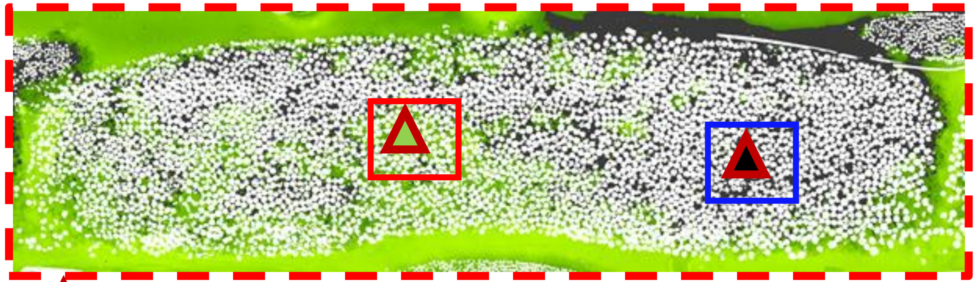
- ✓ Layer 1 (0°)
- ✗ Layer 2 (90°)
- ✓ Layer 3 (0°)
- ✗ Layer 4 (90°)
- ✓ Layer 5 (0°)
- ✗ Layer 6 (90°)
- ✓ PP layer



★ Inter bundle porosity

☆ PP impregnated inter bundle channels

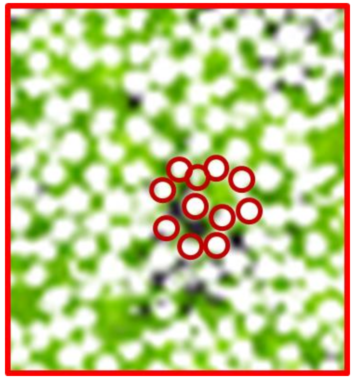
❑ Meso Scale

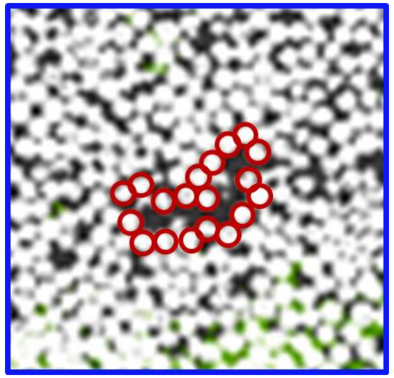


△ Intra bundle porosity

▲ PP impregnated single fibers

❑ Micro Scale



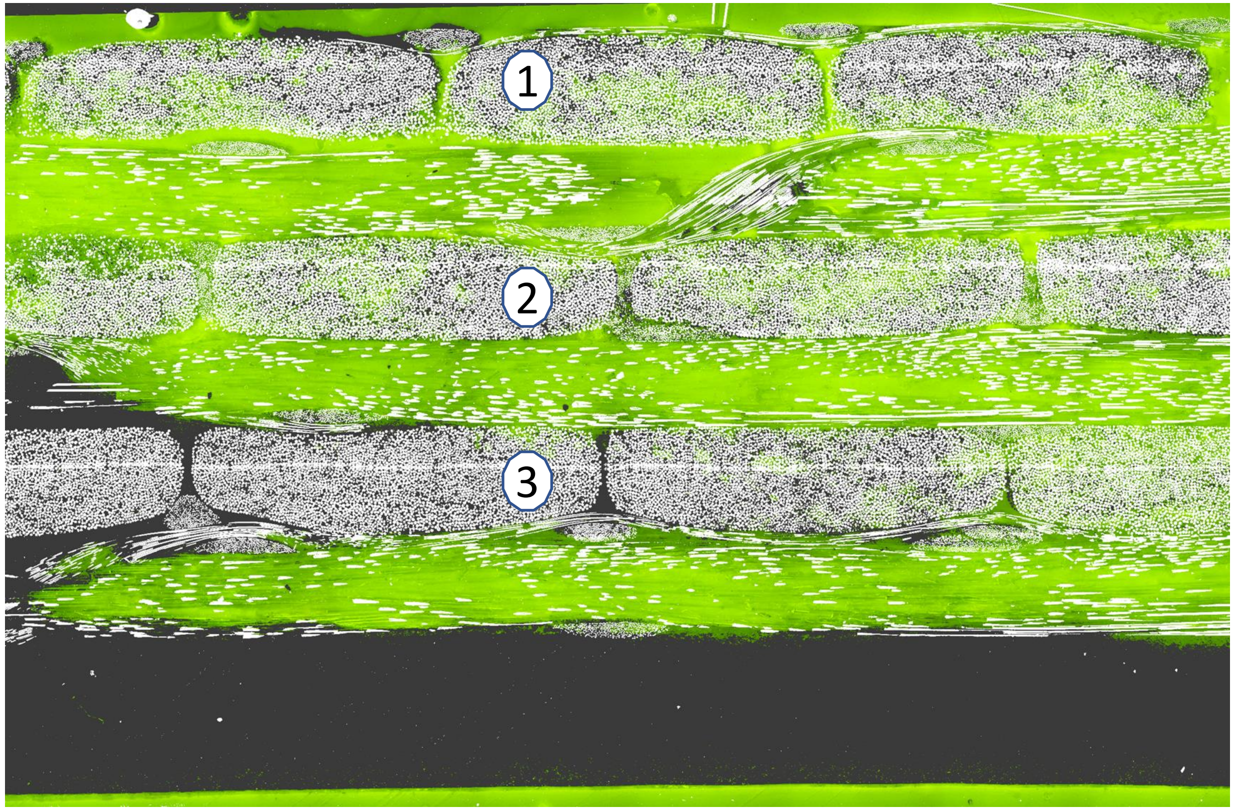


○ Single GF ■ PP ■ Porosity

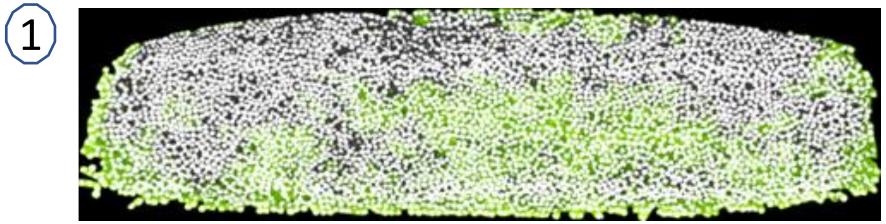
IMT Nord Europe
École Mines-Télécom
IMT-Université de Lille

Quantitative Analysis

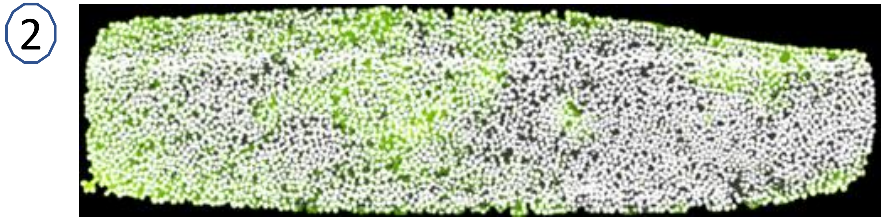
Micro Scale ROI



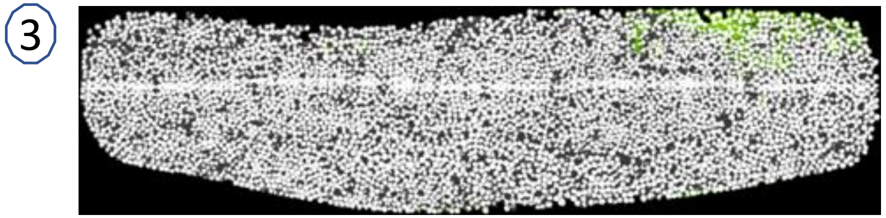
- Extracted ROI



GF 64.17% Void 19.03%



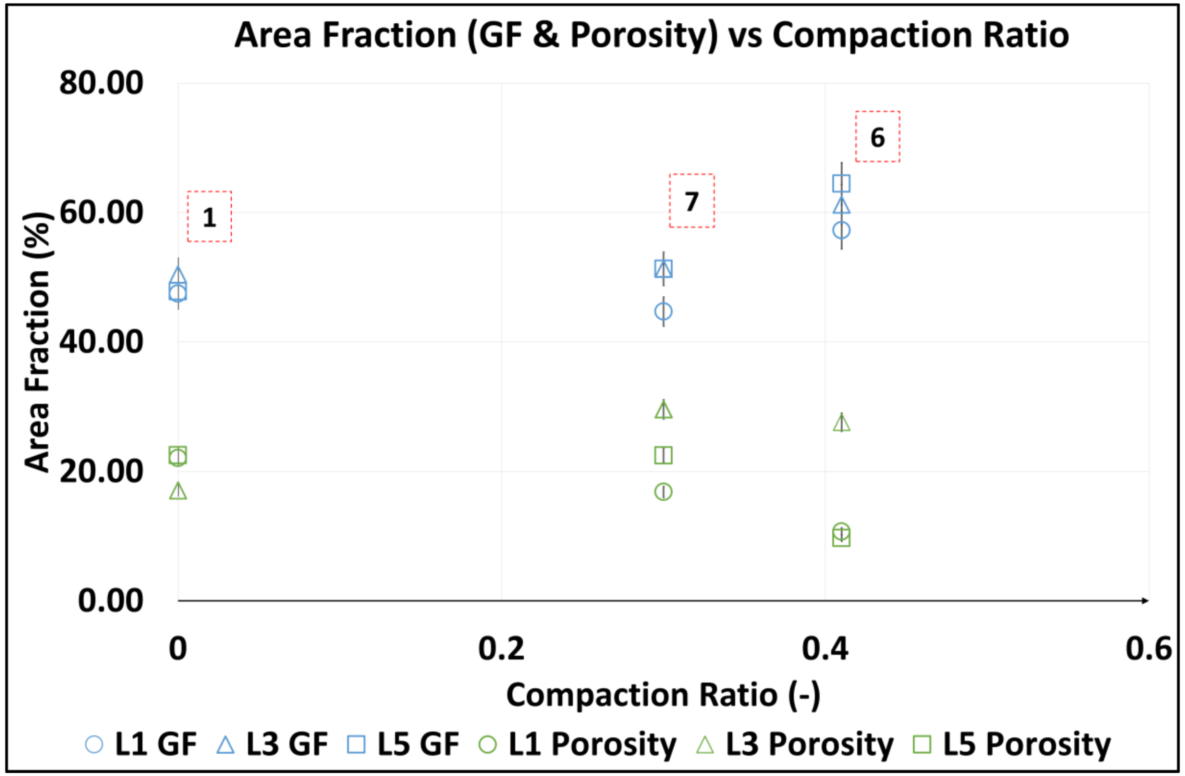
GF 66% Void 17.92%

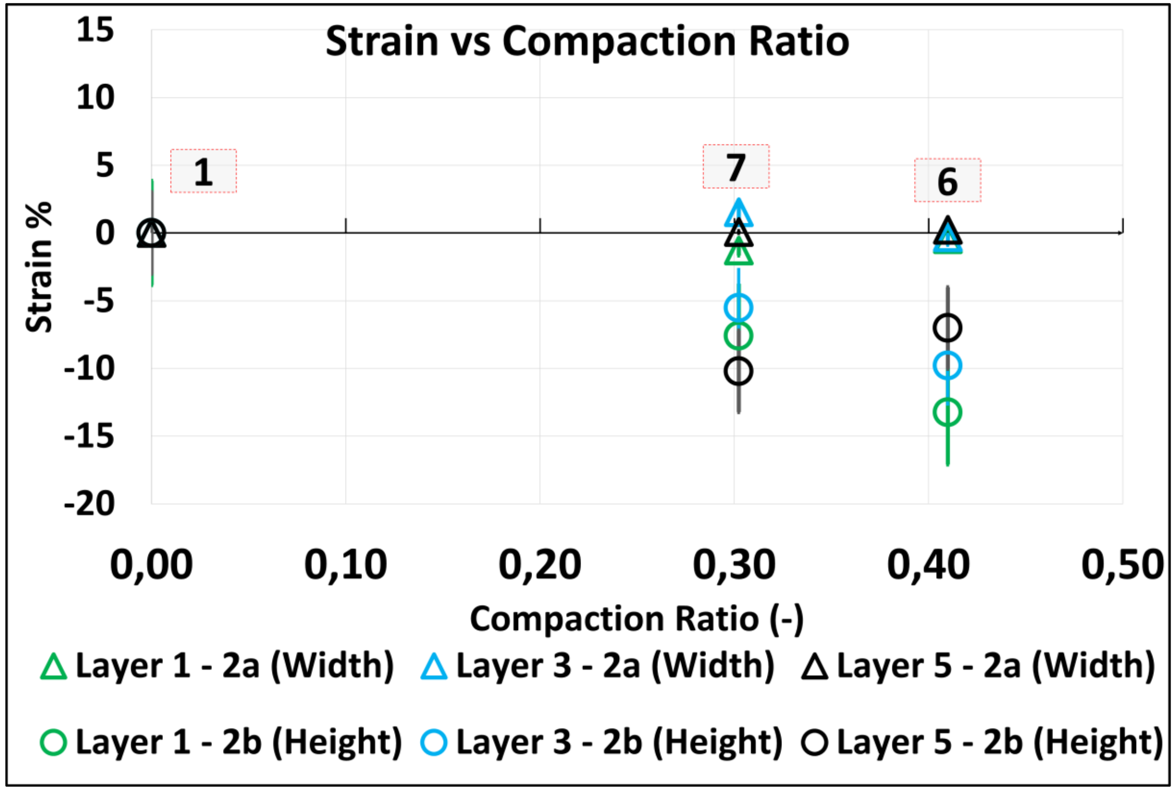


GF 63.71% Void 2.06%

Quantitative Analysis

Micro Scale ROI





Conclusions & Perspectives

- From the multi-modal imaging approach, it was demonstrated that the quantitative analyses at the micro scale of mesh deformation, degree of impregnation and porosity percentage can be conducted.
- From the combined FM-SEM image, we can depict that the variation in degree of impregnation in the mesh as the matrix flow front progressed from layer 6 to layer 1.
- The Space homogeneity of porosity in mesh belonging to each layer and within the layer is not same. Thus, high standard deviation from the average porosity. This is due to non homogenous flow front of PP matrix when pressure is applied.
- From the strain plot, it is observed that the layer 5 is more compacted than layer 1 & 3, which results in decreased mesh permeability. However, the porosity levels in layer 5 is low due to proximity to PP matrix in the lay-up.

➢ Extending the approach to include µCT images (on-going work)
➢ Extending the approach to correlate image-based quantitative analyses and process control parameters
➢ Quantifying uncertainties related to image-based analysis for quantitative porosity analyses

Acknowledgements

The authors acknowledge:

- The European Regional Development Fund FEDER, the French state and the Hauts-de-France Region council for co-funding the PhD grant of Mr. Sujith SIDLIPURA
- Dr. Vincent THIERY for his advice/training to use the 2D microscopy equipment (IMT Nord Europe, France)
- The technical personnel of the ISIS 4D platform (Lille, France) for his assistance to conduct the µCT scans. This platform has been funded by the International Campus on Safety and Inter-modality in Transportation (CISIT), the Hauts-de-France Region, the European Community and the National Center for Scientific Research (CNRS).

ISIS 4D
In Situ Innovative Set-ups
under X-ray microtomography

Thank You

IMT Nord Europe
École Mines-Télécom
IMT-Université de Lille

www.imt-nord-europe.fr

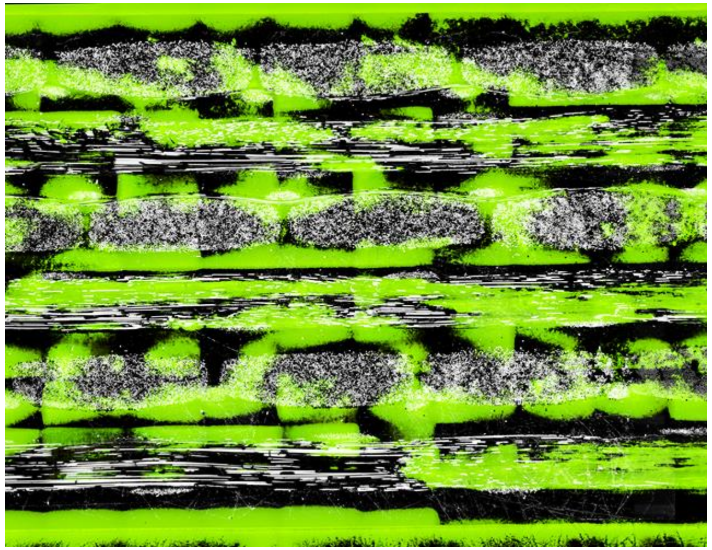
Bibliography

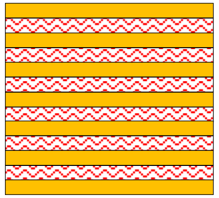
1) S. Konstantopoulos, C. Hueber, I. Antoniadis, J. Summerscales, and R. Schledjewski, "Liquid composite molding reproducibility in real-world production of fiber reinforced polymeric composites: a review of challenges and solutions," *https://doi.org/10.1080/20550340.2019.1635778*, vol. 5, no. 3, pp. 85–99, Jul. 2019, doi: 10.1080/20550340.2019.1635778.
2) Gupta,S. K., "Robotic Assistants for Composite Prepreg Sheet Layup", Center for Advanced Manufacturing, Viterbi School of Engineering University of Southern California.
3) "Voids in fiber-reinforced polymer composites: A review on their formation, characteristics, and effects on mechanical performance - Mahoor Mehdikhani, Larissa Gorbatikh, Ignaas Verpoest, Stepan V Lomov, 2019."
4) N. Z. M. Zuhudi, A. F. Zulkifli, M. Zulkifli, A. N. A. Yahaya, N. M. Nur, and K. D. M. Aris, "Void and Moisture Content of Fiber Reinforced Composites," *Journal of Advanced Research in Fluid Mechanics and Thermal Sciences*, vol. 87, no. 3, pp. 78–93, Sep. 2021, doi: 10.37934/ARFMTS.87.3.7893.
5) "TexGen available from MICRA." https://licensing.micragateway.org/product/texgen (accessed Apr. 24, 2023).
6) C. Binetruy, F. Chinesta, and R. Keunings, "Flows in Polymers, Reinforced Polymers and Composites," 2015
7) Little, J.E., Yuan, X. and Jones, M.I. (2012) 'Characterisation of voids in fibre reinforced composite materials', NDT & E International, 46(1), pp. 122–127. Available at: https://doi.org/10.1016/J.NDTEINT.2011.11.011.
8) Abdelal, Nisrin, and Steven L. Donaldson. 2017. 'Comparison of Methods for the Characterization of Voids in Glass Fiber Composites'. Https://Doi.Org/10.1177/0021998317710083 52 (4): 487–501. https://doi.org/10.1177/0021998317710083.
9) Khaled, Bilal, Loukham Shyamsunder, Josh Robbins, Yatin Parakhiya, and Subramaniam D. Rajan. 2021. 'Framework for Predicting Failure in Polymeric Unidirectional Composites through Combined Experimental and Computational Mesoscale Modeling Techniques'. Fibers 2021, Vol. 9, Page 50 9 (8): 50. https://doi.org/10.3390/FIB9080050.
10) Gagani, Abedin, Yiming Fan, Anastasia H. Muliana, and Andreas T. Echtermeyer. 2018. 'Micromechanical Modeling of Anisotropic Water Diffusion in Glass Fiber Epoxy Reinforced Composites'. Journal of Composite Materials 52 (17): 2321–35. https://doi.org/10.1177/0021998317744649.
11) Ishida, Osuke, Junichi Kitada, Katsuhiko Nunotani, and Kiyoshi Uzawa. 2020. 'Impregnation and Resin Flow Analysis during Compression Process for Thermoplastic Composite Production'. Https://Doi.Org/10.1080/09243046.2020.1752964 30 (S1): 39–58. https://doi.org/10.1080/09243046.2020.1752964.
12) Oromiehie, Ebrahim, Ulf Garbe, and B. Gangadhara Prusty. 2019. 'Porosity Analysis of Carbon Fibre-Reinforced Polymer Laminates Manufactured Using Automated Fibre Placement'. Https://Doi.Org/10.1177/0021998319875491 54 (9): 1217–31. https://doi.org/10.1177/0021998319875491.
13) Santhosh, Unni, Yasser Gowayed, Greg Ojard, Imelda Smyth, Sujith Kalarikkal, and George Jefferson. 2018. 'Quantification of Porosity in Ceramic Matrix Composites Using Thermography'. Journal of Nondestructive Evaluation 37 (2): 1–14. https://doi.org/10.1007/S10921-018-0487-Z/METRICS.
14) Breister, Adam M., Muhammad A. Imam, Zhichao Zhou, Karthik Anantharaman, and Pavana Prabhakar. 2020. 'Microbial Dark Matter Driven Degradation of Carbon Fiber Polymer Composites'. BioRxiv, April, 2020.04.05.024463. https://doi.org/10.1101/2020.04.05.024463.
15) Purslow, D. 1984. 'On the Optical Assessment of the Void Content in Composite Materials'. Composites 15 (3): 207–10. https://doi.org/10.1016/0010-4361(84)90276-3.
16) WANG, Zhenjun, Siyuan YANG, Shiping SUN, Yingfeng ZHANG, Changchun CAI, Bowen XIONG, Wei YANG, Zhifeng XU, and Huan YU. 2021. 'Multiscale Modeling of Mechanical Behavior and Failure Mechanism of 3D Angle-Interlock Woven Aluminum Composites Subjected to Warp/Weft Directional Tension Loading'. Chinese Journal of Aeronautics 34 (8): 202–17. https://doi.org/10.1016/J.CJA.2020.09.016.
17) Ekoi, Emmanuel J., Andrew N. Dickson, and Denis P. Dowling. 2021. 'Investigating the Fatigue and Mechanical Behaviour of 3D Printed Woven and Nonwoven Continuous Carbon Fibre Reinforced Polymer (CFRP) Composites'. Composites Part B: Engineering 212 (May). https://doi.org/10.1016/J.COMPOSITESB.2021.108704.
18) Zou, Ailing, Zhongde Shan, Shaozong Wang, Xiaojun Liu, Xueya Ma, Dongming Zou, and Xibin Jiang. 2022. 'Study on Porosity of Aramid Fiber Reinforced Composites Prepared by Additive Manufacturing'. Https://Doi.Org/10.1177/26349833221121831 31 (October): 263498332211218. https://doi.org/10.1177/26349833221121831.
19) Tatlisu, H., V. Balek, I. N. Beckman, N. Kardjilov, A. Hilger, and H. Rauch. 2011. 'Imaging and Diffusion Structural Diagnostics of Silicon Carbide-Based Composites and Fibers'. Journal of Thermal Analysis and Calorimetry 107 (2): 447–52. https://doi.org/10.1007/S10973-011-1856-3.
20) Liu, Bing, Anchang Xu, and Limin Bao. 2015. 'Preparation of Carbon Fiber-Reinforced Thermoplastics with High Fiber Volume Fraction and High Heat-Resistant Properties'. Http://Dx.Doi.Org/10.1177/0892705715610408 30 (5): 724–37. https://doi.org/10.1177/0892705715610408.
21) Sabuncuoglu, Baris, Hamed Tanabi, Jeroen Soete, and Stepan V. Lomov. 2020. 'Micro-CT Analysis of Deviations in Fiber Orientation and Composite Stiffness near the Microvascular Channels Embedded in Glass-Fiber Reinforced Composites'. Composite Structures 237 (April): 111896. https://doi.org/10.1016/J.COMPSTRUCT.2020.111896.
22) Amedewovo, Luc, Arthur Levy, Basile de Parscau du Plessix, Julien Aubril, Arnaud Arrive, Laurent Orgéas, and Steven Le Corre. 2023. 'A Methodology for Online Characterization of the Deconsolidation of Fiber-Reinforced Thermoplastic Composite Laminates'. Composites Part A: Applied Science and Manufacturing 167 (April): 107412. https://doi.org/10.1016/J.COMPOSITESA.2022.107412.
23) Mehdikhani, Mahoor, Larissa Gorbatikh, Ignaas Verpoest, and Stepan V. Lomov. 2019. 'Voids in Fiber-Reinforced Polymer Composites: A Review on Their Formation, Characteristics, and Effects on Mechanical Performance'. Journal of Composite Materials 53 (12): 1579–1669. https://doi.org/10.1177/0021998318772152/ASSET/IMAGES/LARGE/10.1177_0021998318772152-FIG20.JPEG.
24) Gao, Yantao, Wenfeng Hu, Sanfa Xin, and Lijuan Sun. 2022. 'A Review of Applications of CT Imaging on Fiber Reinforced Composites'. Journal of Composite Materials 56 (1): 133–64. https://doi.org/10.1177/00219983211050705/ASSET/IMAGES/LARGE/10.1177_00219983211050705-FIG20.JPEG.
25) Ayadi, Abderrahmane, Mylène Deléglise-Lagardère, Chung Hae Park, and Patricia Krawczak. 2019. 'Analysis of Impregnation Mechanism of Weft-Knitted Commingled Yarn Composites by Staged Consolidation and Laboratory X-Ray Computed Tomography'. Frontiers in Materials 6 (October): 255. https://doi.org/10.3389/FMATS.2019.00255/BIBTEX.

Qualitative Analyses of Combined FM-SEM images

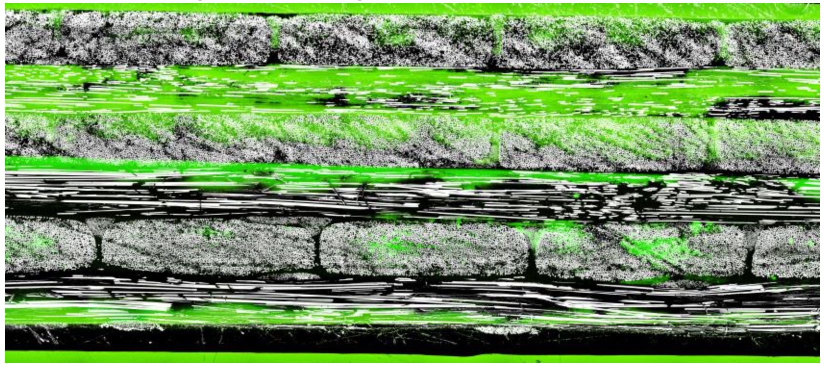
Advantages and limitations of the suggested SEM-FM based protocol

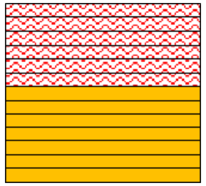
- **Ref. plate (Cr=0%)**



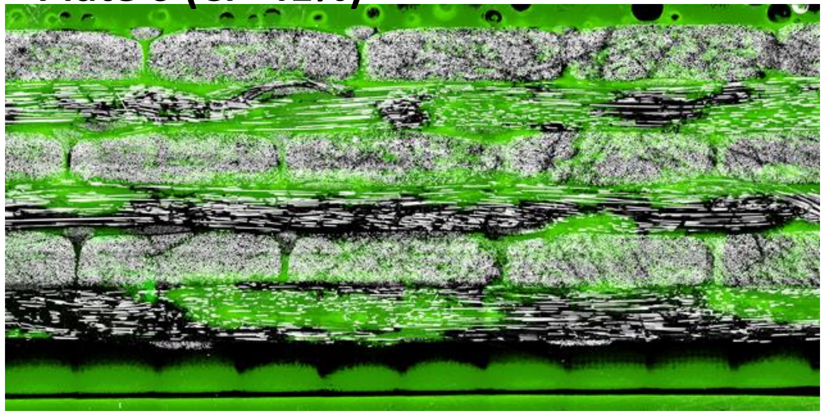


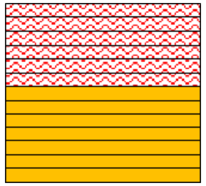
- **Plate 7 (Cr=30%)**





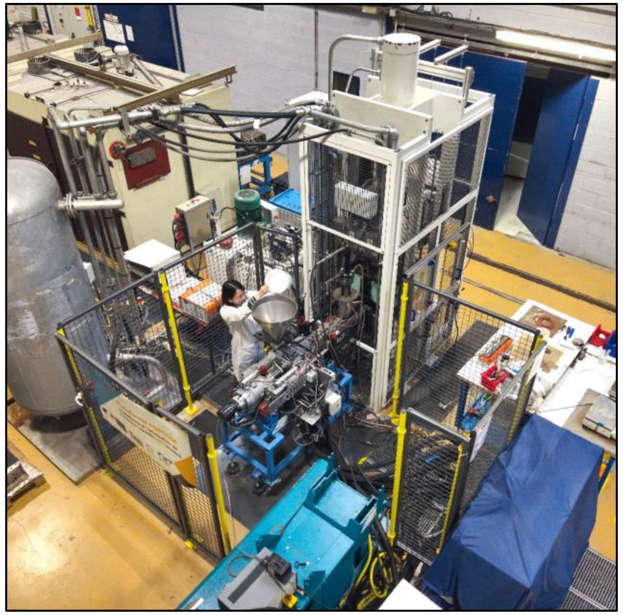
- **Plate 6 (Cr=41%)**





RESEARCH CENTER FOR ACADEMIC AND INDUSTRIAL COLLABORATIVE PROJECTS

- **Advanced Composites Manufacturing: POPCOM Platform**
  - All Composites materials, including bio-based materials and thermoplastic matrix composites process



- **Composites Additive Manufacturing, Hybrid Processes**
- **Virtual Engineering, Process, Part and Process Simulation**
- **Non-Destructive Testing, Performance Assessment**
- **Structural Mechanics – Large Scale Fatigue Test Unit**