Deconsolidation and consolidation of long fibre-reinforced thermoplastic composites during thermo-forming: 3D real time and in situ observations

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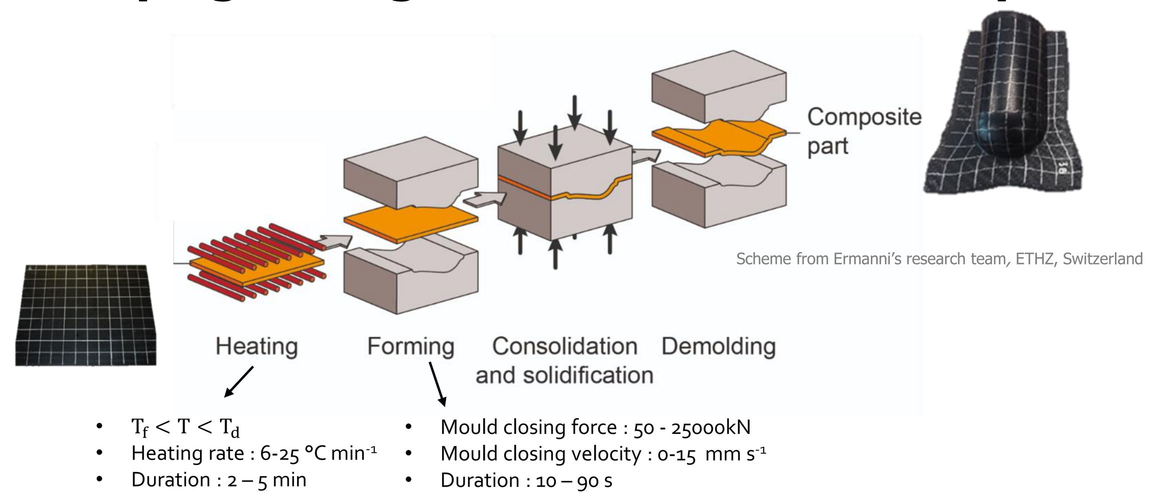
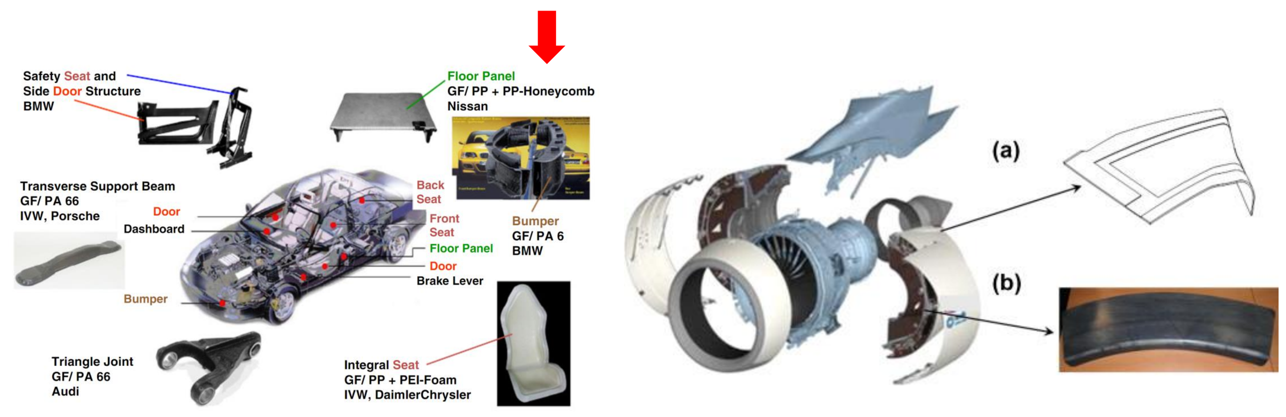
Context and objectives

Stamping of long fibre-reinforced thermoplastics



- $T_f < T < T_d$
- Heating rate : 6-25 °C min⁻¹
- Duration : 2 – 5 min

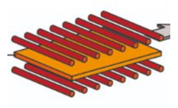
- Mould closing force : 50 - 25000kN
- Mould closing velocity : 0-15 mm s⁻¹
- Duration : 10 – 90 s



Friedrich and Almajid, *Appl Compos Mater* (2013) Vieille *et al.*, *Compos Part B* (2012)

- Thermoplastic polymer matrix: PA, PP, PEEK…
- Non-woven/woven long fibre reinforcements: carbon, glass, bio-based fibres
- Growing use in aeronautic and automotive industries for structural parts

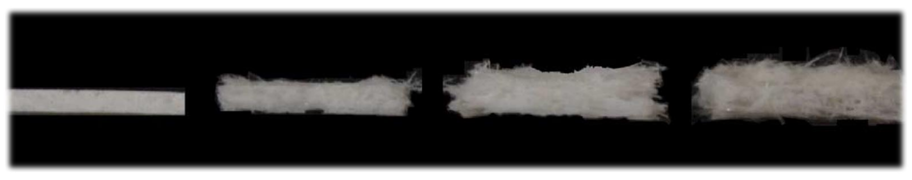
A multistage forming process with many uncontrolled deformation mechanisms and defects



Heating

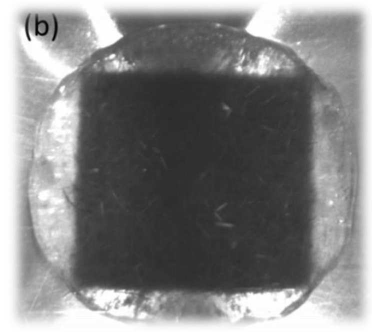
- Decrease of the matrix viscoelastic properties due to temperature increase
- Uncontrolled deformation of the fibrous reinforcement: anisotropic deconsolidation
- Uncontrolled increase of the porosity

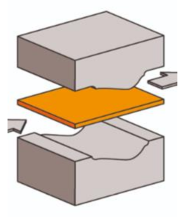
Ex.: anisotropic deconsolidation of the fibrous reinforcement in GMT



Wolfrath *et al.* Compos Part A (2005)

Ex.: matrix migration during compression of flax-fibre-mat-reinforced thermoplastic composites

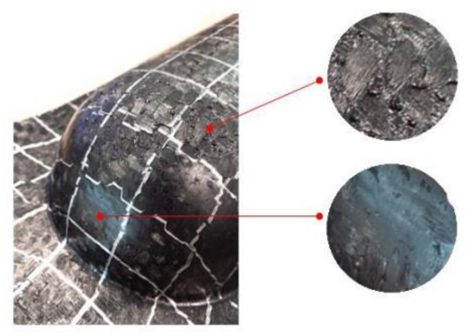




Forming

- Uncontrolled deformation of the fibrous reinforcement: anisotropic consolidation, shear, bending, biaxial tension…
- Uncontrolled/uncomplete pore closure, matrix migration

Uncomplete pore closure



M. Gassoumi, *PhD Thesis*, France (2018)

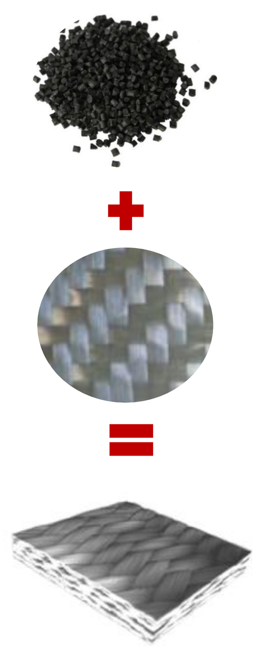
Objective of the study

To propose a method to characterize in 3D and real time:

- **(De)consolidation of the fibrous reinforcement at the fibre bundle scale**
- **Related pore growth and matrix migration mechanisms at this scale**

Materials & methods

Material: thermoplastic matrix reinforced with woven fabrics



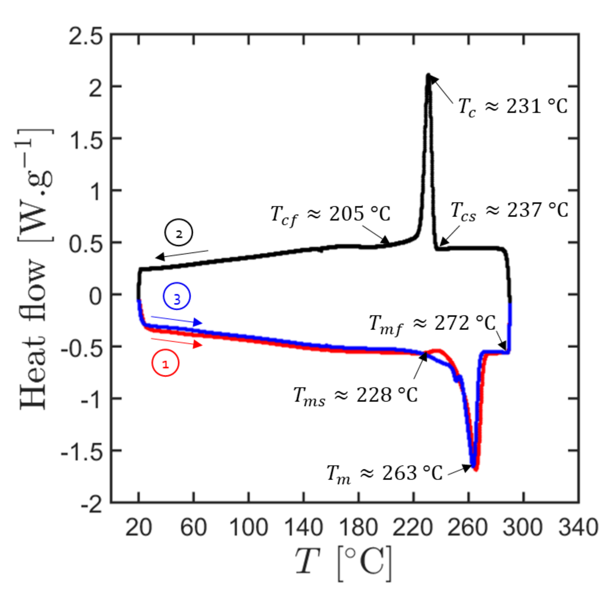
Thermoplastic polymer matrix (Solvay): polyamide 6.6 (PA 6.6)

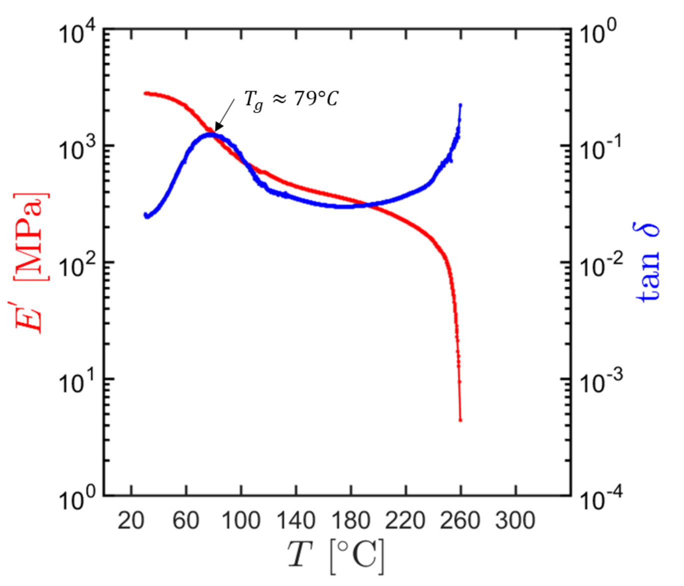
Fibrous reinforcement: Twill 2×2 made of glass fibre bundles (12k, $d_{fil} = 14$ µm)

Prepegs (Solvay): large plates with 4 plies $[0°, 90°, 0°, 90°]$, thickness ≈ 2 mm

Cylindrical samples obtained using water jet cutting

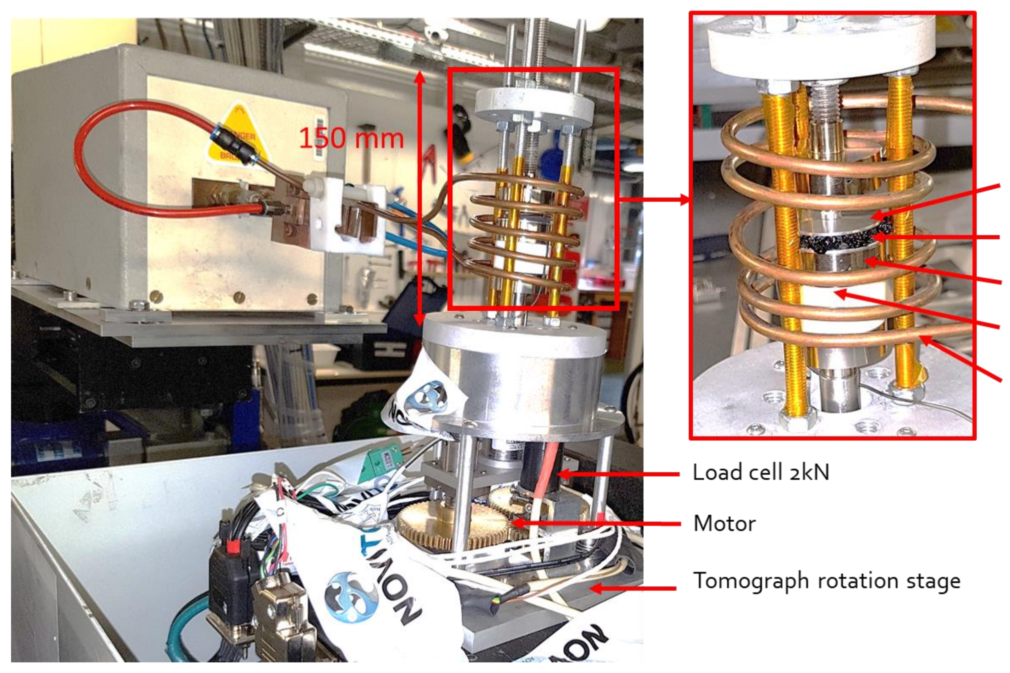
Thermophysical properties of PA 6.6 matrix



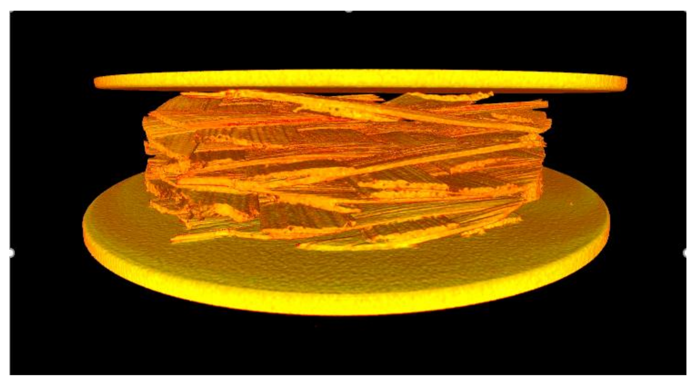


(De)consolidation setup and 3D imaging using X-ray synchrotron microtomography

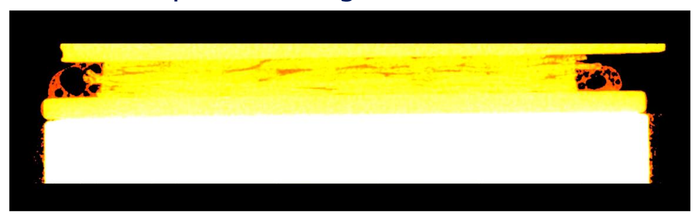
Experimental setup – ESRF ID19 beamline



Heating stage - Deconsolidation



Compression stage - Consolidation



Testing procedure

- **Deconsolidation phase: heating up to 300°C then down to the testing temperature (280°C, 290°C and 300°C)**
- **Consolidation phase: compression at various strain rates (5×10^{-4} s⁻¹ < $\dot{\varepsilon}_0$ < 5×10^{-3} s⁻¹)**

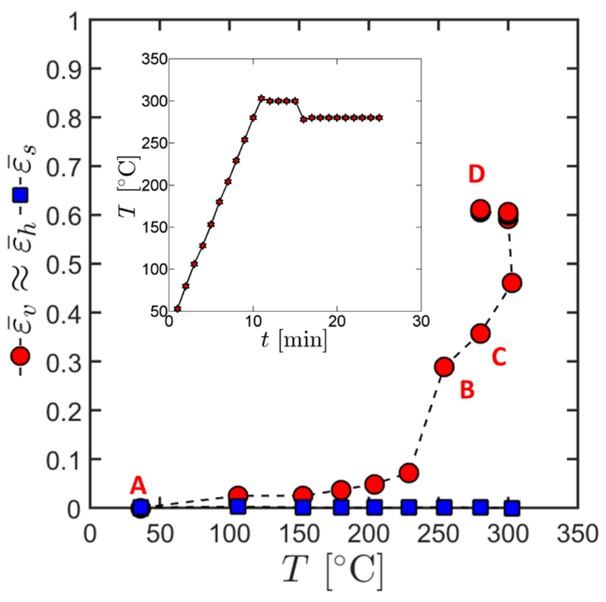
3D imaging

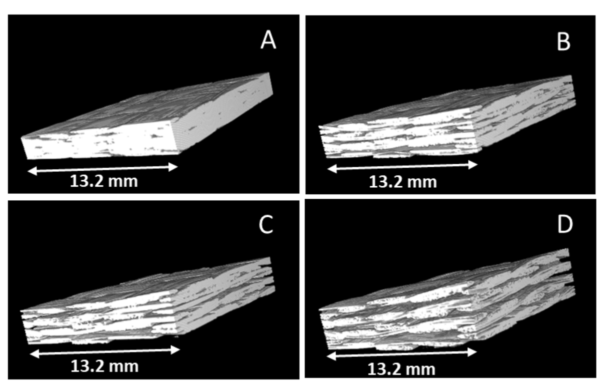
- **Setup mounted on the X-ray tomograph of the ID19 beamline (ESRF, Grenoble, France)**
- **Spatial resolution: voxel size 22³ µm³**
- **Large field of view: 2000² pixels**
- **Contrast enhancement : Paganin imaging mode**
- **Fast imaging mode: 2000 X-ray projections for a scanning time of 2 s !**

Main results

Heating stage - Deconsolidation

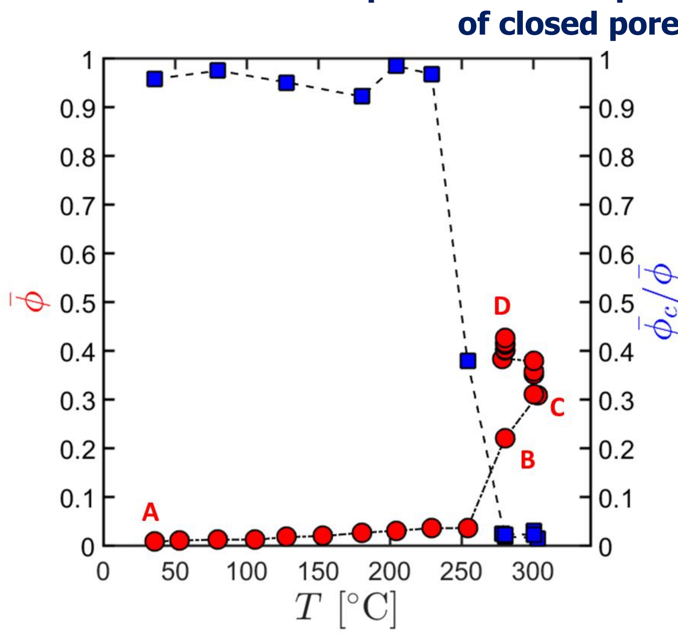
Evolution with the temperature of thickness $\bar{\varepsilon}_h$, plane $\bar{\varepsilon}_s$ and volume $\bar{\varepsilon}_v$ strains



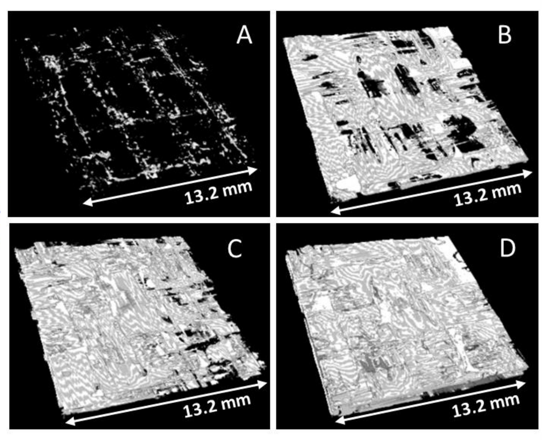


- Significant increase of the out-of-plane strain $\bar{\varepsilon}_h$ up to 0.6
- Negligible surface strain $\bar{\varepsilon}_s$ due to the highly anisotropic and in-plane fibrous architecture

Evolution with the temperature of the porosity $\bar{\phi}$ and relative volume fraction of closed pores $\bar{\phi}_c/\bar{\phi}$



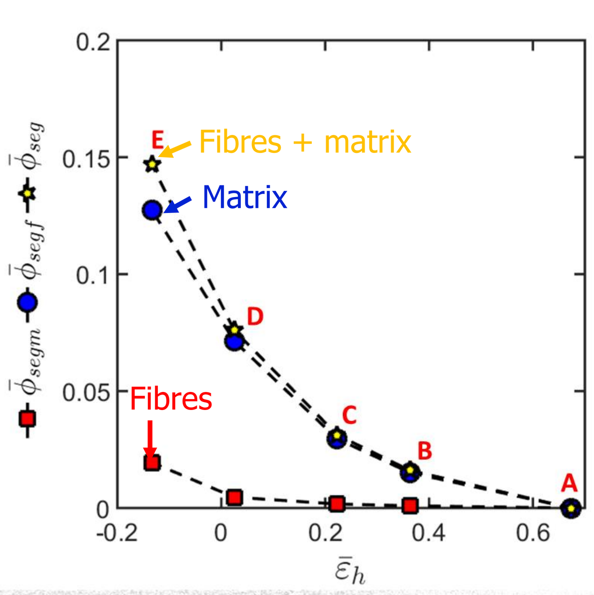
3D observations of pore growth

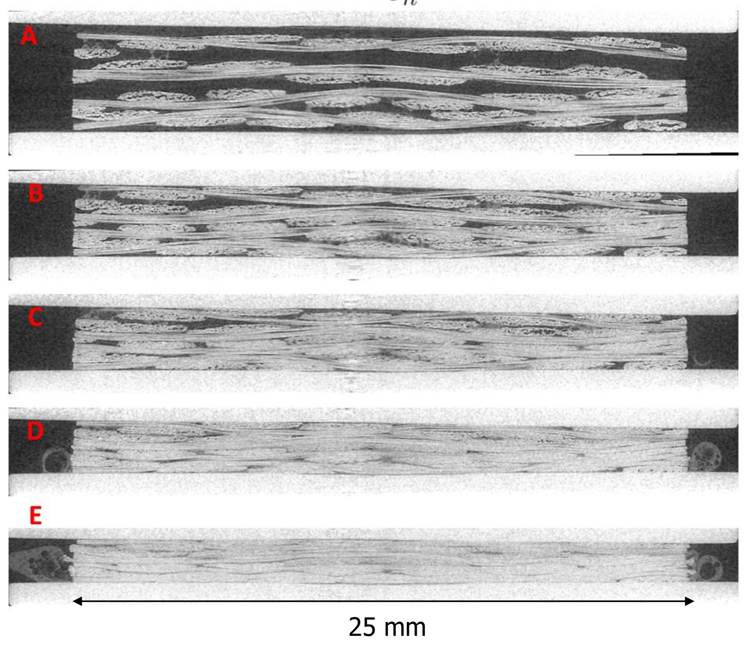


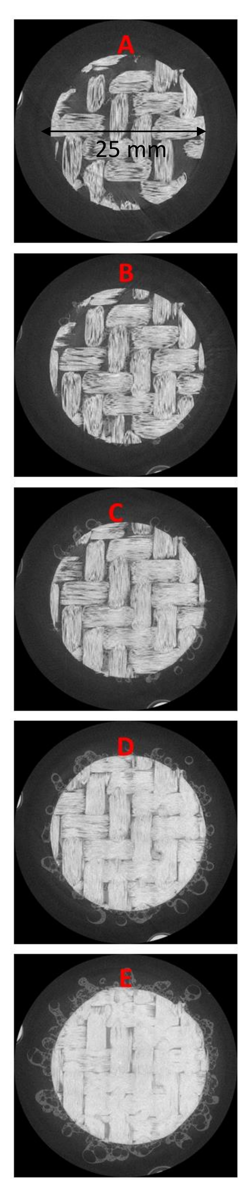
- The swelling of the fibrous network induced a pronounced increase of the porosity from 0.008 up to 0.45
- The kinetics of porosity growth exhibited sharp increase from 230°C to T_{mf}
- The initial amount of closed pores practically corresponded to the initial porosity

Compression stage - consolidation

Evolution with the macroscale compression strain of the volume fraction of segregated fibre and matrix phases



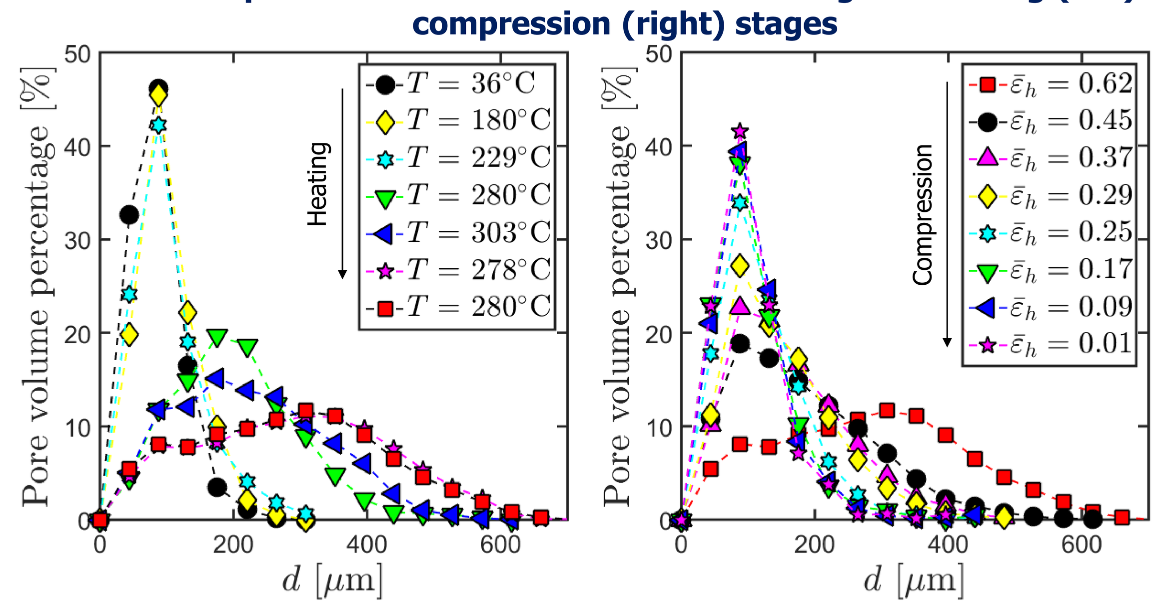




- During consolidation: heterogeneous flow conditions characterized by pronounced matrix segregation and squish of fibre bundles!

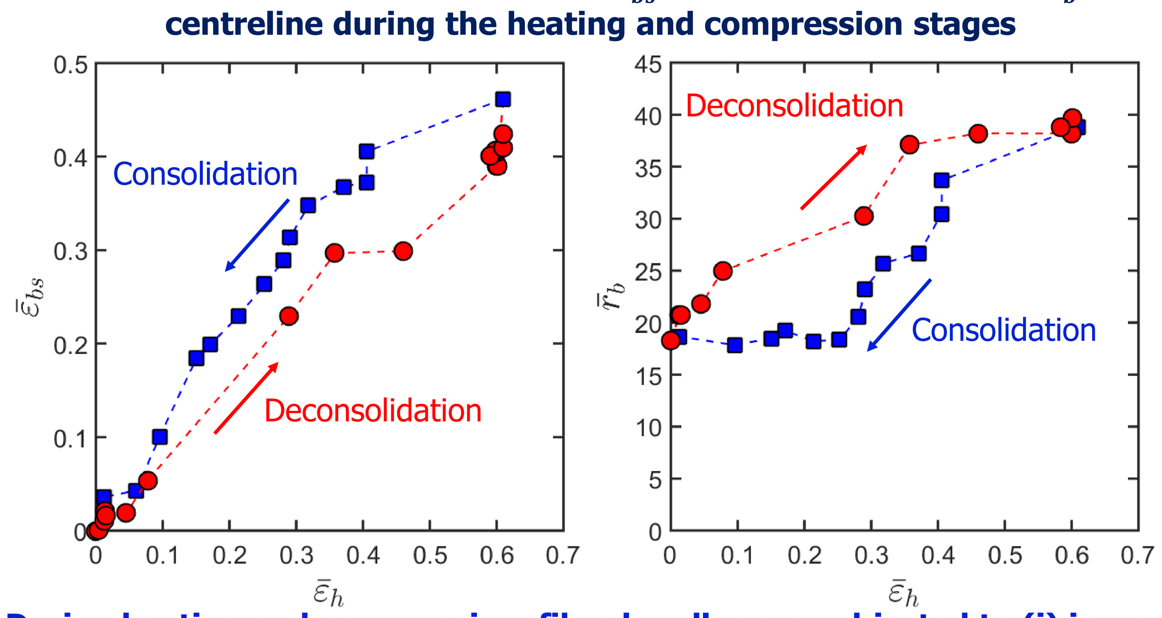
Deconsolidation vs consolidation

Evolution of pore size distributions obtained during the heating (left) and compression (right) stages



- Pore size distributions exhibited Weibull shapes
- During heating: progressive increase of the mean pore diameter $\bar{d}$ from ≈ 80 to 300 µm at 300°C.
- During compression : progressive decrease of $\bar{d}$ close to its initial value, i.e., $\bar{d}$ ≈ 100 µm at the end of the compression stage.

Evolution of bundle cross-section strain $\bar{\varepsilon}_{bs}$ and radius of curvature $\bar{r}_b$ of their centreline during the heating and compression stages



- During heating and compression, fibre bundles are subjected to (i) important swelling-/shrinkage of their cross sections and (ii) complex folding-unfolding mechanisms