Is the matrix of a polymer composite really like a bulk sample of the same polymer ? Almost.

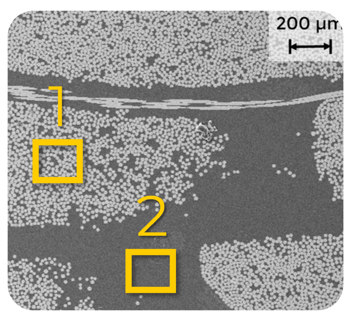
Micromechanical analysis of interphase and matrix in a glass fiber-reinforced thermoplastic composite

INTRODUCTION

- Fiber-scale models are used to predict the mechanical behavior of polymer composites
- The matrix is generally attributed the constitutive properties of the bulk polymer, though the presence of fibers and the formation of an interphase may influence its mechanical properties
- In this work, in-situ micromechanical analysis of a methacrylic matrix (Elium) is performed on glass fiber reinforced composites. Intra- (1) and inter-tow (2) matrix stiffness is compared to bulk values (3), for various levels of physical ageing.

METHODS

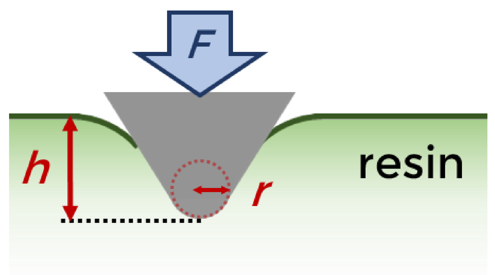
Elium/glass fiber composite



Bulk resin

t_0
Thermal rejuvenation at $T \approx T_g$

t_0, $t_n = t_0 + \Delta t$
Measurement of matrix stiffness by indentation at t_0 and after n months

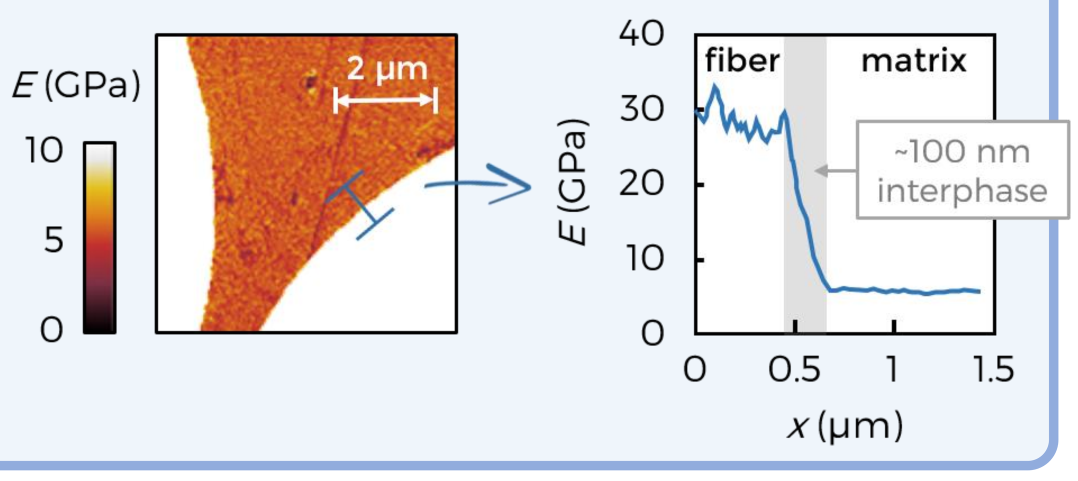


NANOINDENTATION
$h \leq 500$ nm
Resolution ± 0.1 GPa

AFM
$h \leq 3$ nm
Resolution ± 0.5 GPa

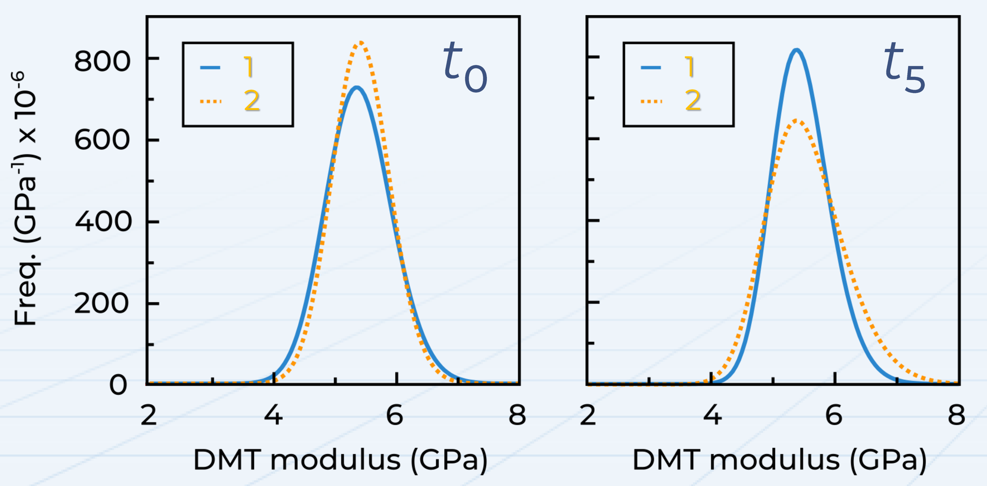
RESULT #1

Presence of a ~100-nm STIFF INTERPHASE in the composite.



RESULT #2

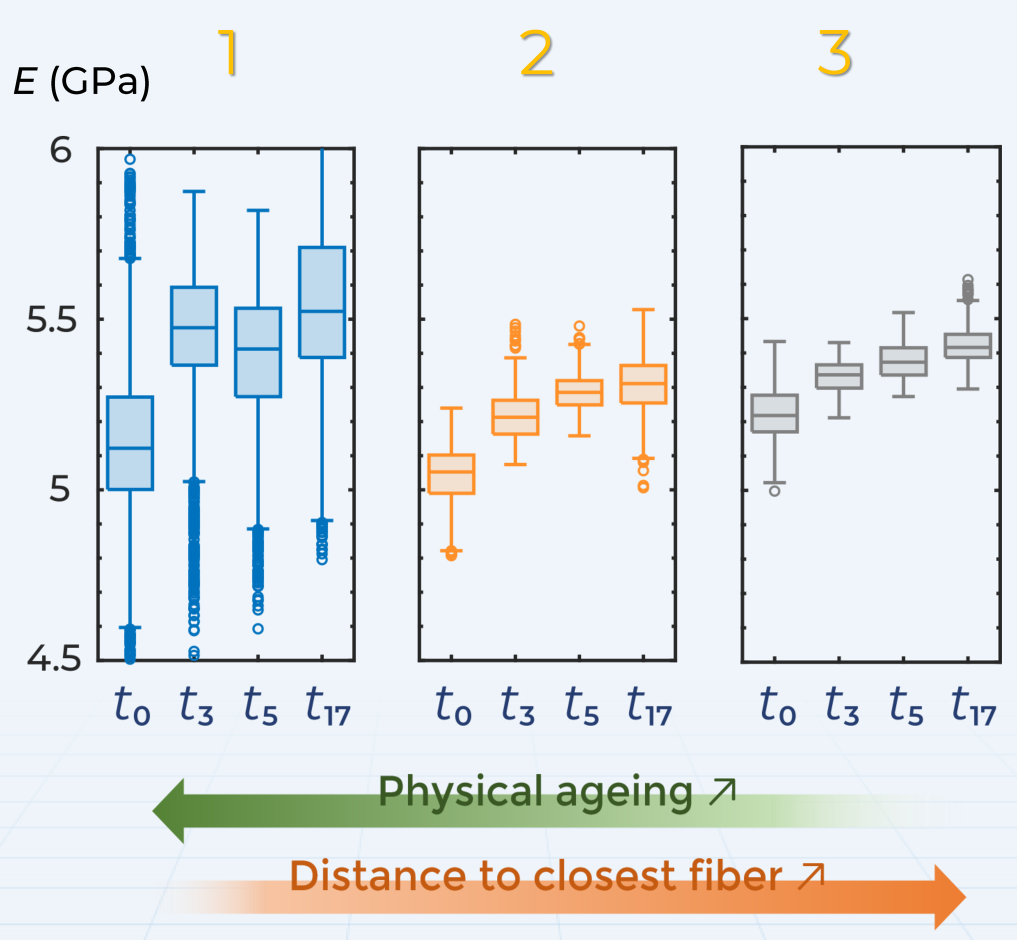
NO SIGNIFICANT DIFFERENCE (>10%) in resin stiffness E between conditions 1, 2 & 3, AT SAME AGEING TIME.



RESULT #3

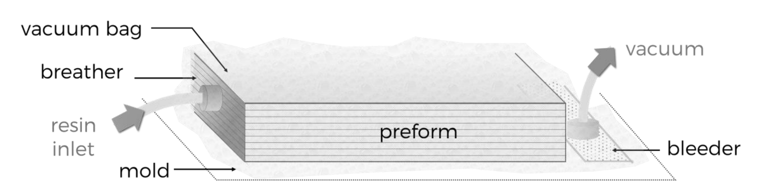
STIFFNESS INCREASES MORE IN THE FIRST 3 MONTHS OF AGEING for all conditions, before a plateau is reached.

Largest increase (+10%) in intra-tow matrix (1), suggesting MORE PHYSICAL AGEING NEAR THE FIBER-MATRIX INTERFACE.



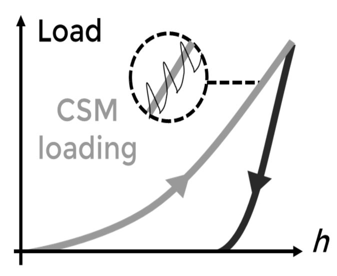
COMPOSITE MANUFACTURING

- Elium: thermoplastic methyl methacrylate-based monomer, compatible with liquid molding
- Infusion of 90-ply glass fabric layup + in-situ polymerization
- Final thickness 7.3 cm, final fiber volume fraction ~0.5
- Extraction of cuboid samples of dimensions ~1x1x1cm³
- 10-min thermal rejuvenation at T_g (100°C) + fine mechanical polishing : defines t_0
- 17-month ageing in room conditions
- Characterization at t_0, t_0 + 3 months (t_3), t_0 + 5 months (t_5) and t_0 + 17 months (t_{17})



NANOINDENTATION

- Continuous stiffness measurement mode (CSM) with Berkovich tip



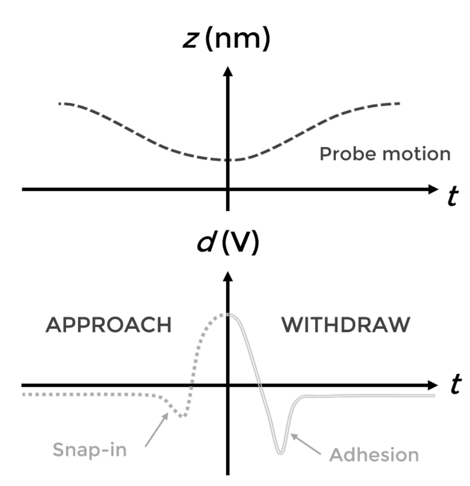
- Tip radius r ~ 100 nm, load $F \leq 5$ mN
- Oliver & Pharr model for E calculation
- For condition 1 (intra-tow), fiber constraint factor (FCF) is defined as : $FCF = x/h$ with x = distance to closest fiber and h = indentation depth.
- For $FCF > 20$, results no longer influenced by fiber proximity
- Data for condition 1 selected in range $30 < FCF < 40$

STATISTICAL ANALYSIS OF NANOINDENTATION DATA

- Random intercept linear mixed model run with SAS, with stiffness E or hardness H as response
- Fixed effects : fiber proximity (i.e. conditions 1, 2 or 3), measurement date (i.e. t_0, t_3, t_5 or t_{17})
- Random effects : indent number (several data points used for each indent), and indent batch number
- Greater data dispersion in condition 1 (intra-tow matrix pockets) than in conditions 2 and 3, so model structure accounts for data heteroscedasticity
- Same trends in H and E measurements

AFM

- PeakForce quantitative nanomechanical mapping at 2kHz



- Tip radius r ~ 10nm (RTESPA-300), load $F \leq 0.3$ mN
- Use of DMT model for E calculation
- Post-treatment of data with softwares Gwyddion and Igor Pro: elimination of fiber and interphase data points + outliers, generation of E value histograms compiling all data for a given condition (1 or 2).

S. F. Gayot[1,2], N. Klavzer[2], C. Bailly[1], T. Pardoen[2], P. Gérard[3], B. Nysten[1]

[1] Institute of Condensed Matter and Nanosciences, UCLouvain, Louvain-la-Neuve, Belgium
[2] Institute of Mechanics Materials and Civil Engineering, UCLouvain, Louvain-la-Neuve, Belgium
[3] Groupement de Recherche de Lacq, Arkema, Lacq, France

UCLouvain

ARKEMA