Pr. Thomas PARDOEN
Pr. Bernard NYSTEN
Dr. Jérémy CHEVALIER

INFLUENCE OF CRYSTALLIZATION CONDITIONS ON THE NANO-/MICRO-BEHAVIOR OF CARBON FIBER-REINFORCED PEEK COMPOSITE

we fund your future

Sophie VANPEE

CONTEXT

Thermoset → Thermoplastic

ECONOMIC

Easier production of complex parts at higher rate

PERFORMANCE

Higher toughness

ENVIRONMENTAL

CONTEXT

Thermoset → Thermoplastic

ECONOMIC

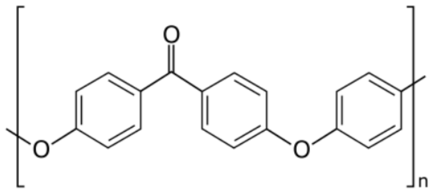
Easier production of complex parts at higher rate

PERFORMANCE

Higher toughness

ENVIRONMENTAL

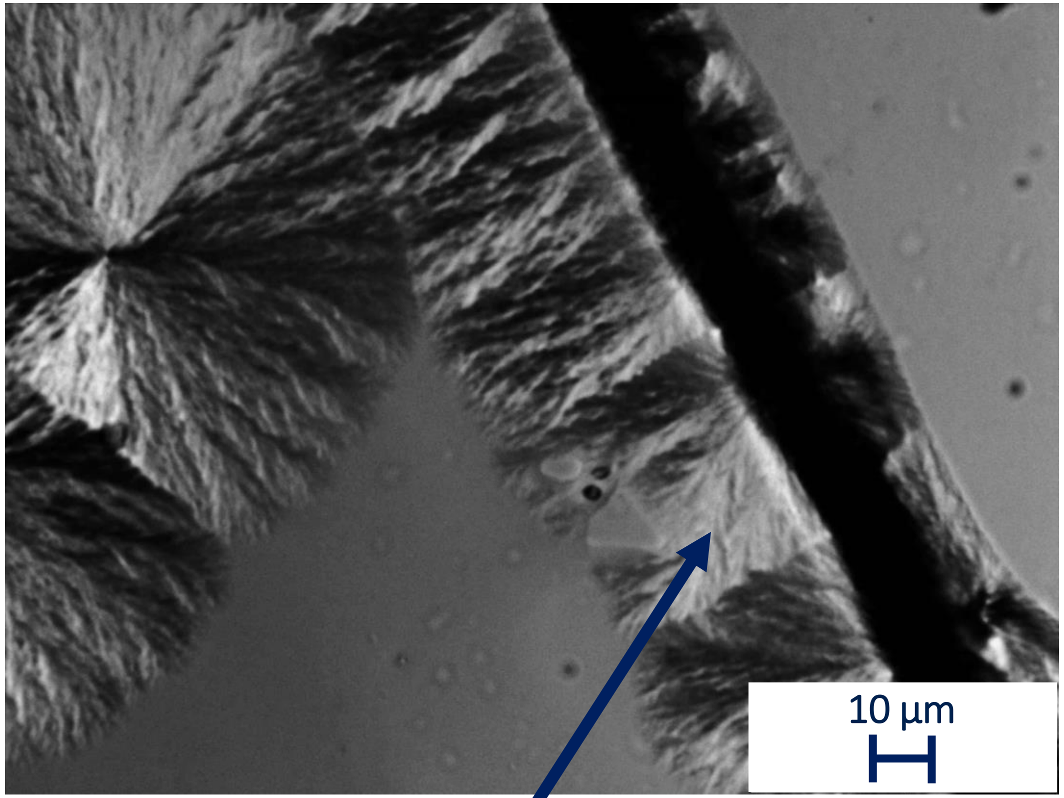
PEEK/C fiber
(Unidirectional, 66 fiber wt. %)



$T_g \approx 143°C$
$T_m \approx 343°C$

SEMI-CRYSTALLINE THERMOPLASTIC COMPOSITE: LIMITATION & QUESTIONS

Processing conditions ⟷ Performances of thermoplastic composites



Transcrystallization (TC)

In the literature

- ✓ Influence of processing conditions (time and temperature) on TC ?
- ✓ Influence of TC on macro properties (σ_0, E and K_{IC})?
- ✗ Impact of TC on mechanical behavior in the interfiber zones?

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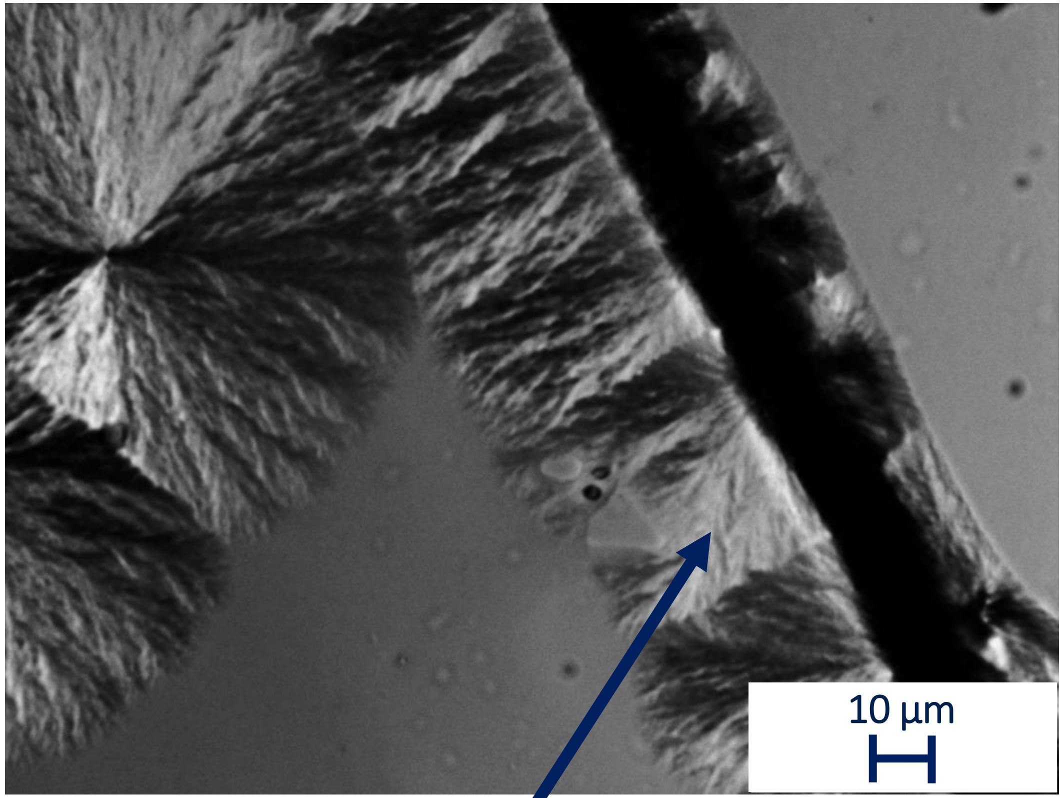

Transcrystallization (TC)

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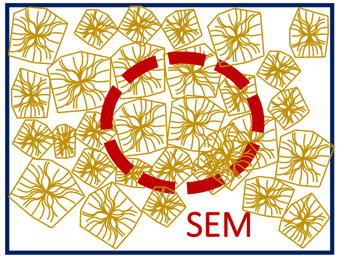
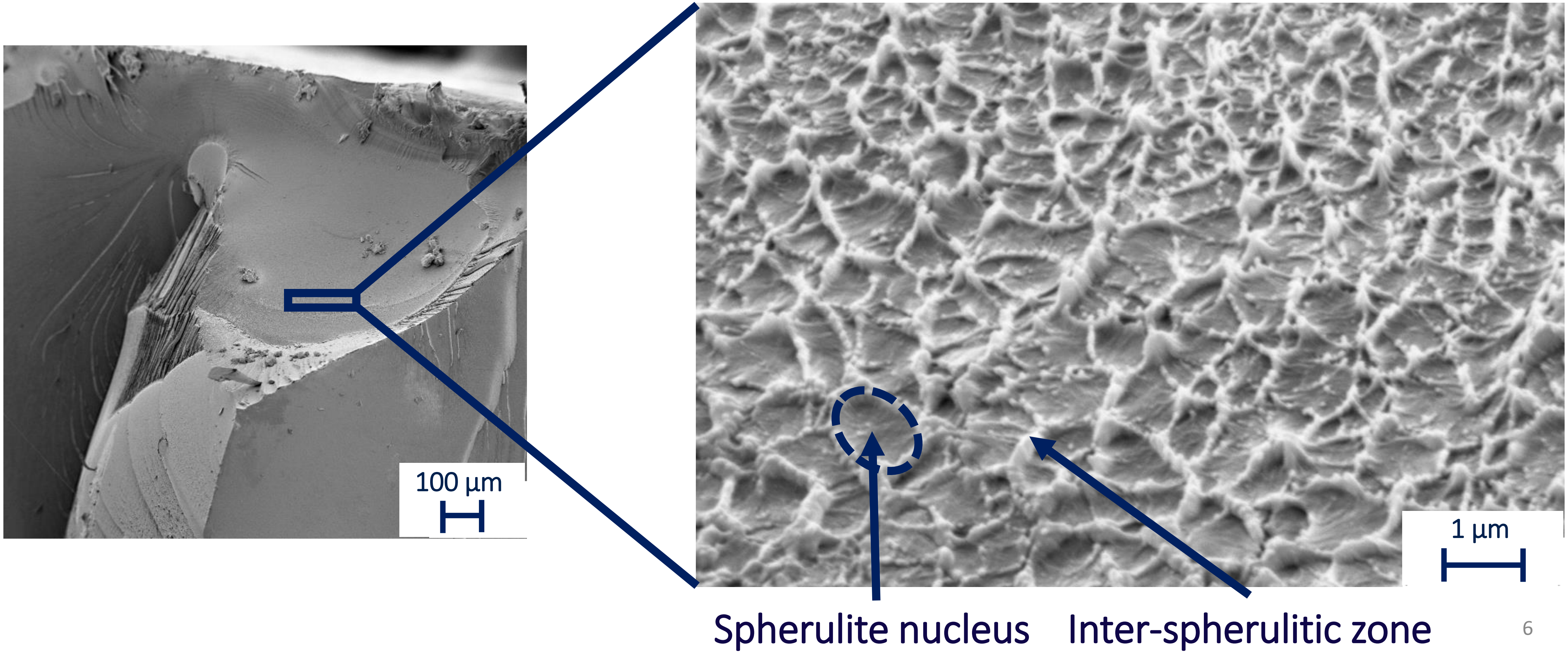
CHARACTERIZATION OF SPHERULITES – Liq. N_2 fracture + SEM

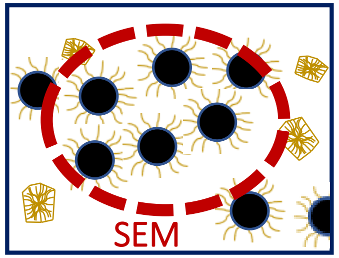
→ Spherulites ~ 1µm diameter

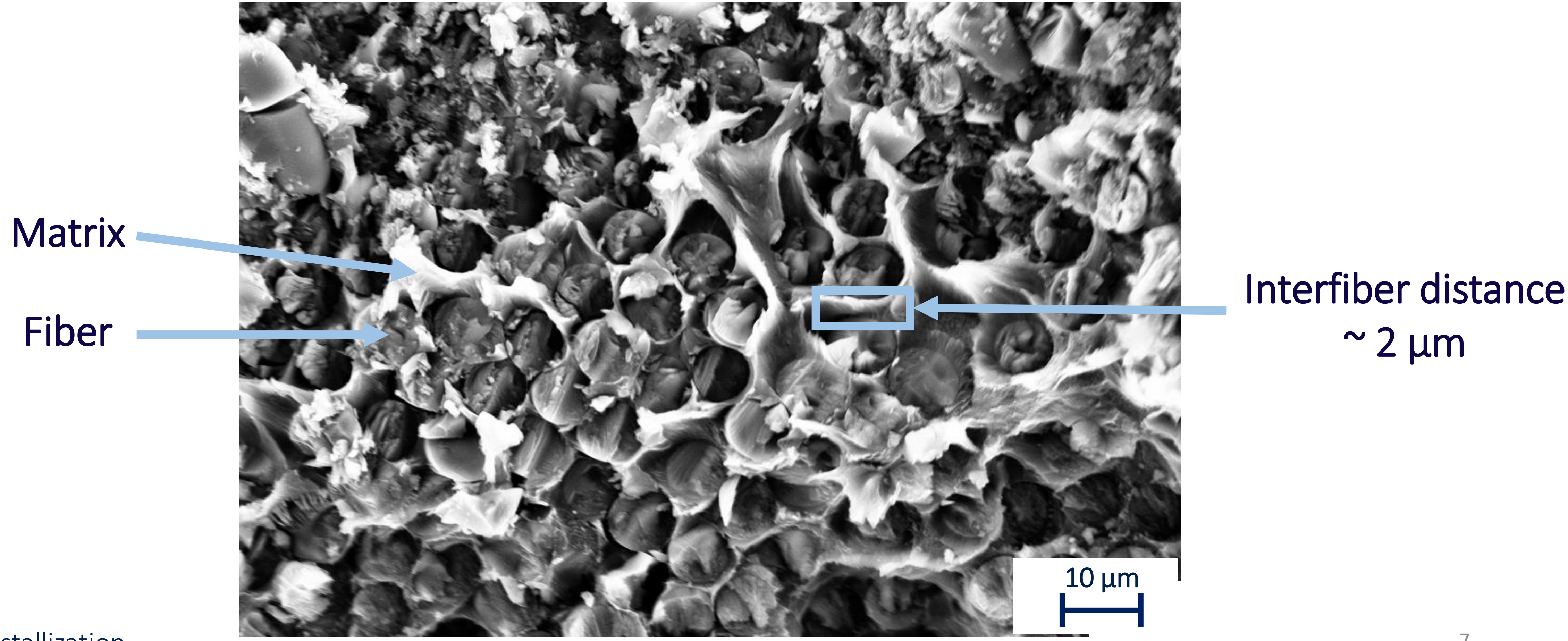




CHARACTERIZATION OF TC – Liq. N_2 fracture + SEM

→ Complicated in composite due to high V_{fiber}

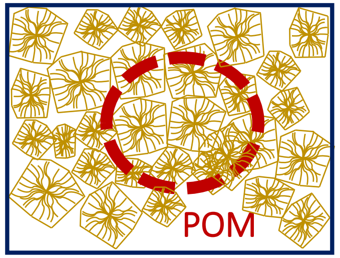


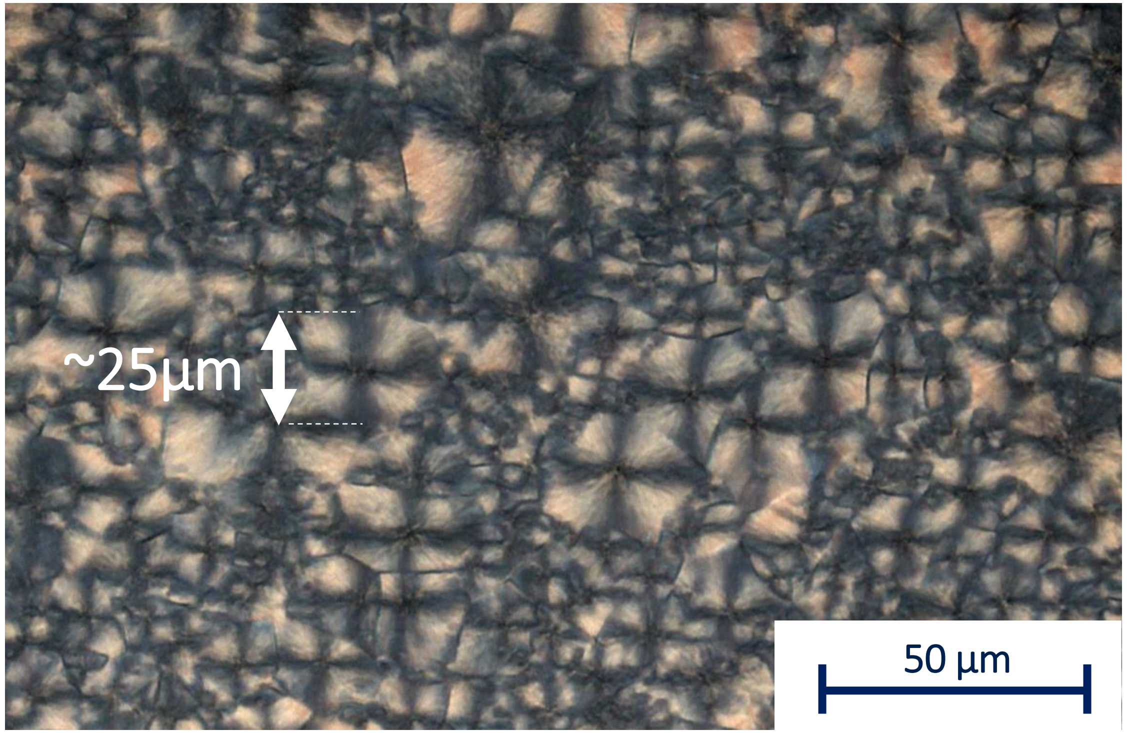


TC: transcrystallization

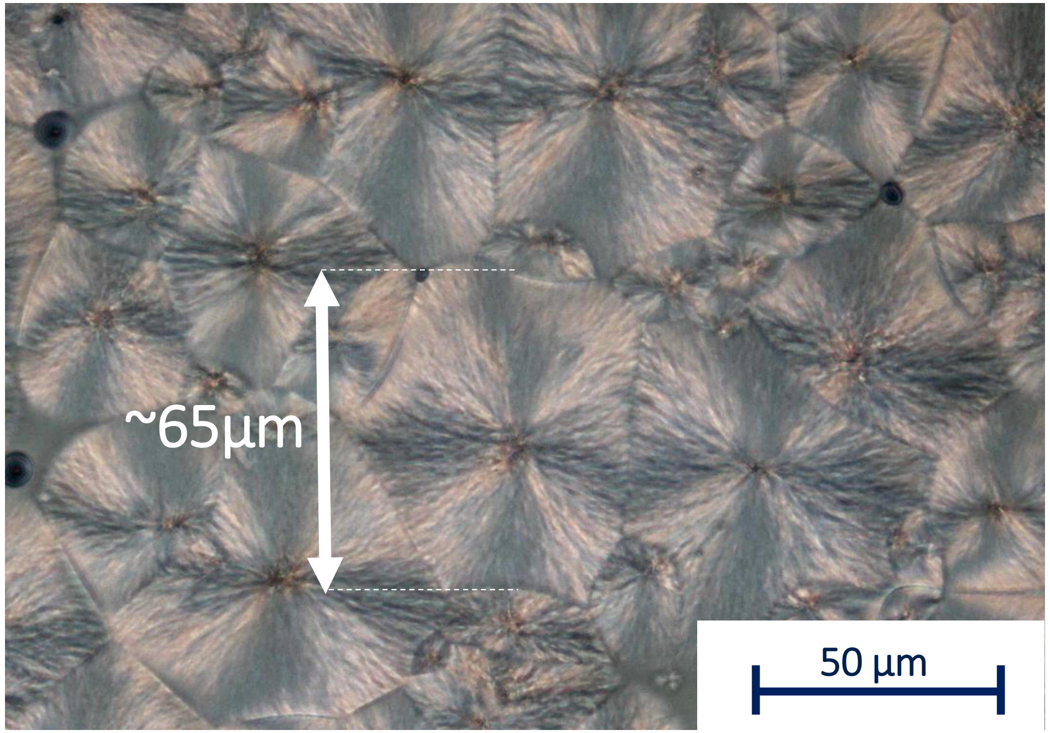
CHARACTERIZATION OF SPHERULITES – POM

→ Suitable technique for spherulites observation





$T_{isoth.\,crist.}$ = 200°C

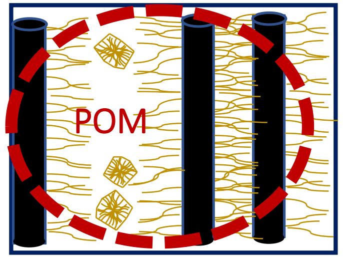


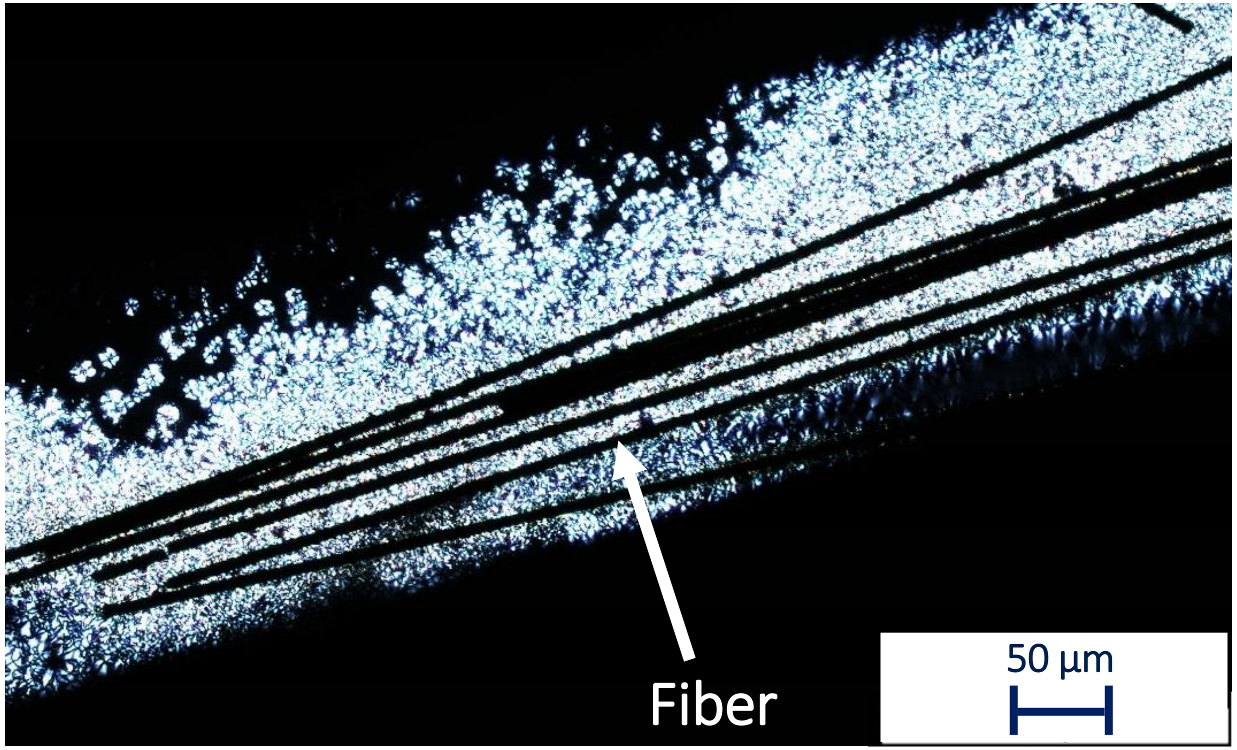
$T_{isoth.\,crist.}$ = 300°C

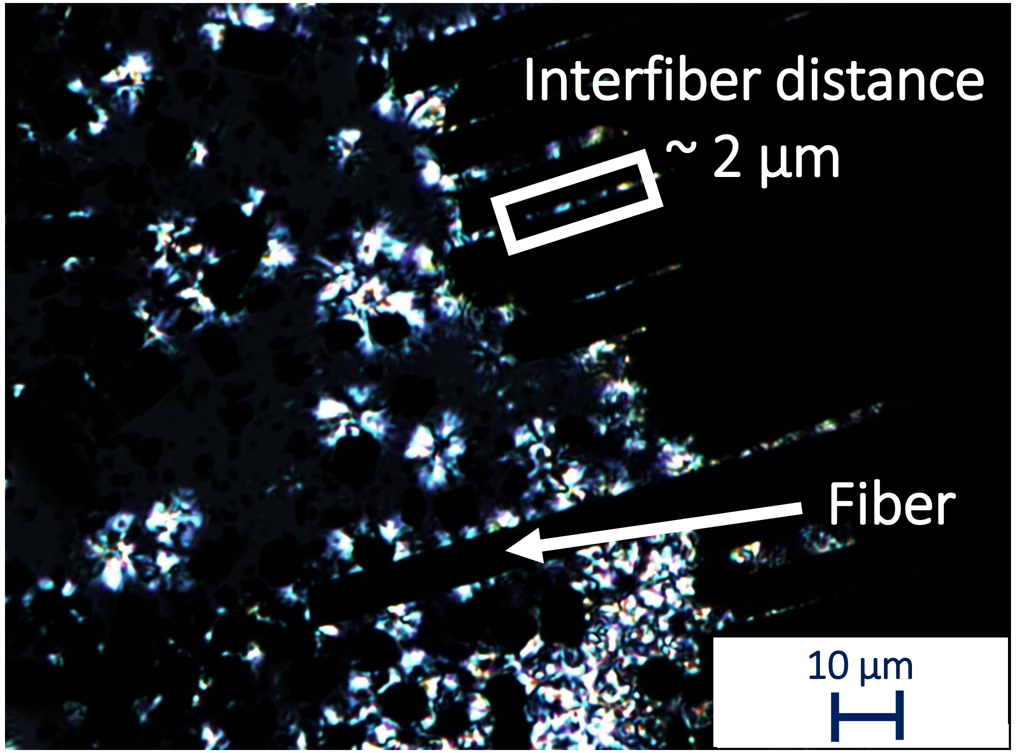
POM: polarized optical microscopy

CHARACTERIZATION OF TC – POM

Complicated in composite due to high V_{fiber}





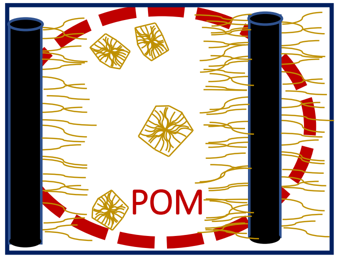


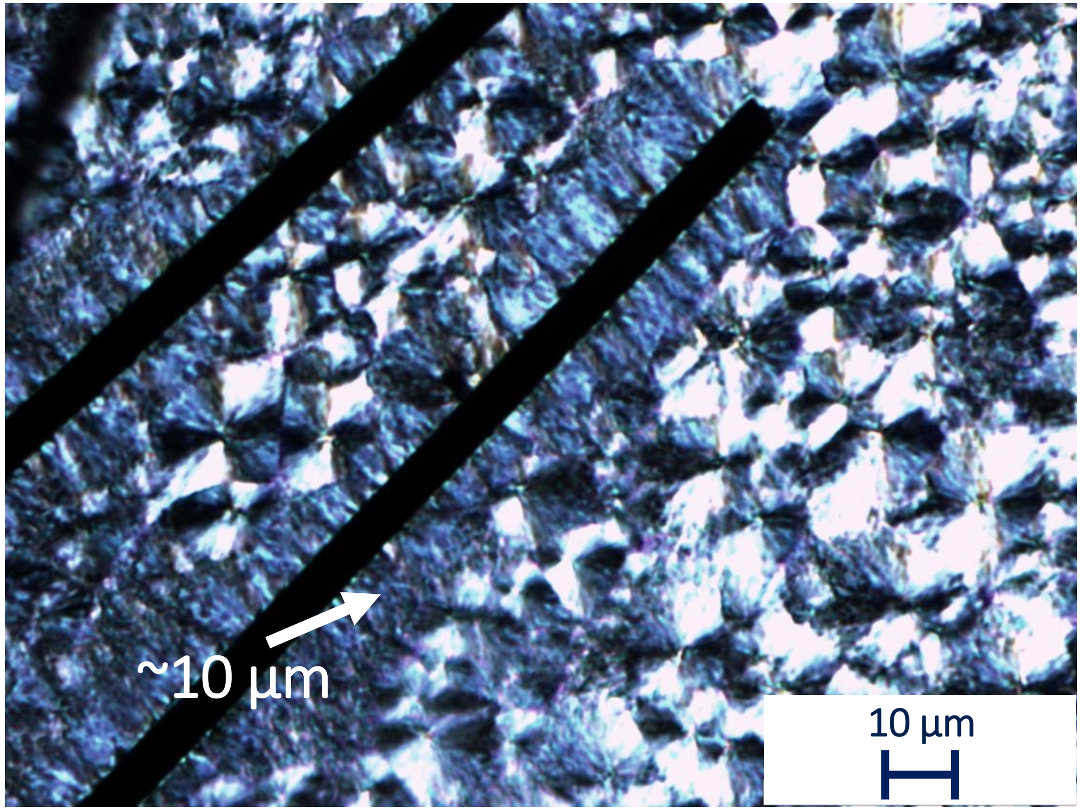
$T_{isoth.\,crist.}$ = 315°C

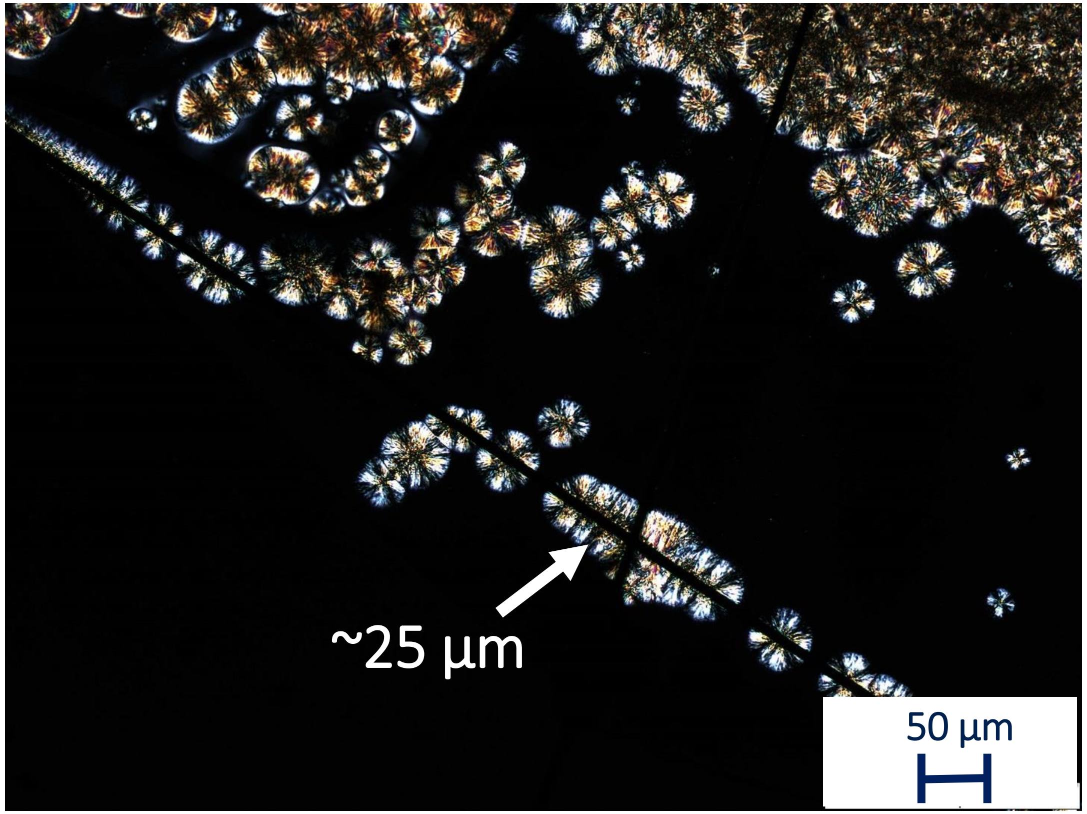
POM: polarized optical microscopy

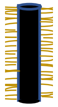
CHARACTERIZATION OF TC – POM

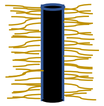
→ Easier in model samples with few fibers







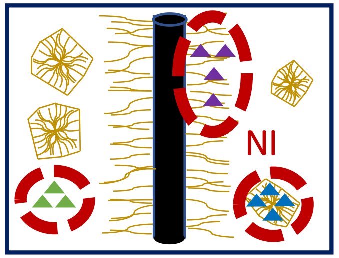
$T_{isoth.\,crist.}$ = 200°C 

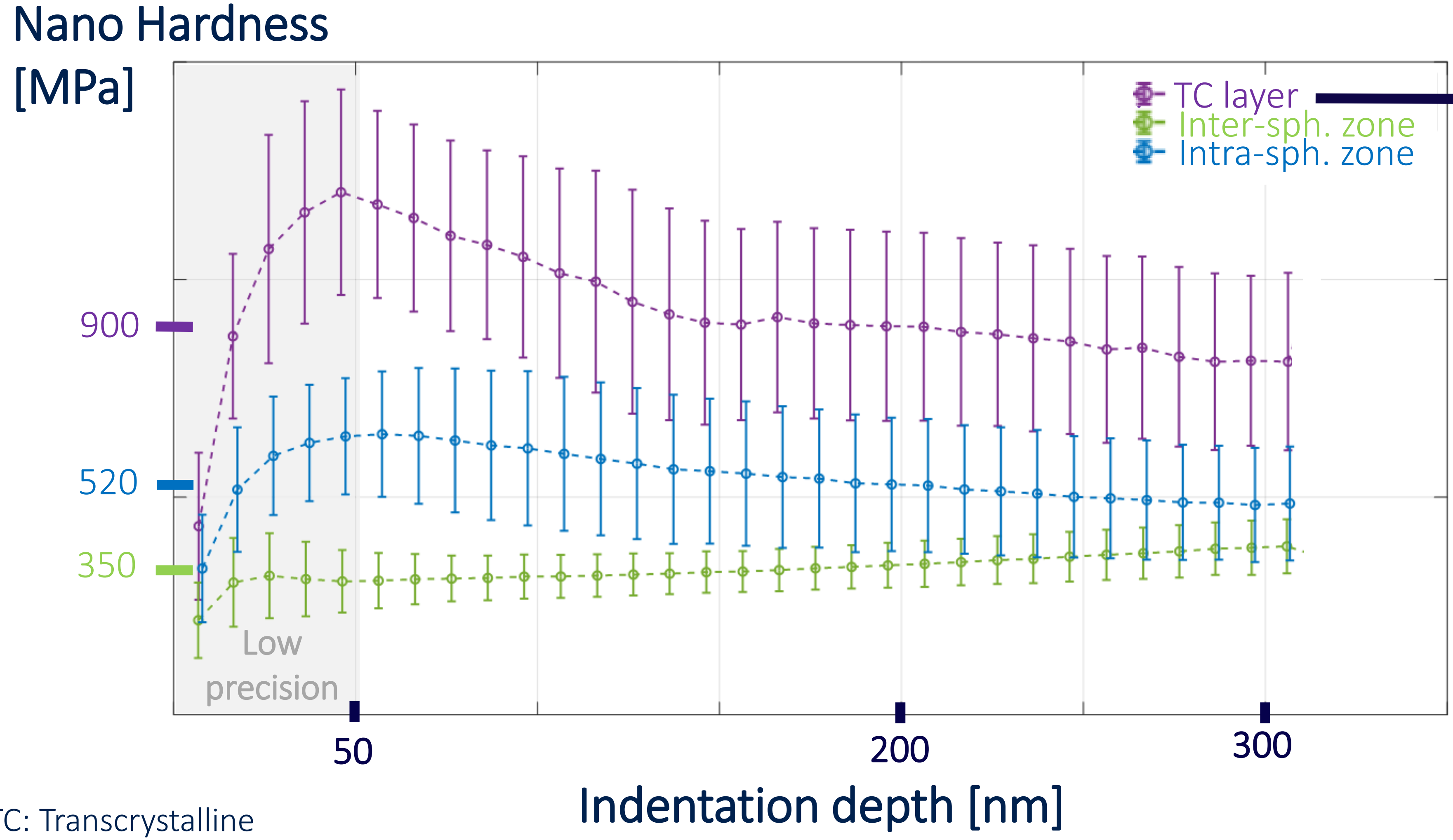
$T_{isoth.\,crist.}$ = 315°C 

POM: polarized optical microscopy

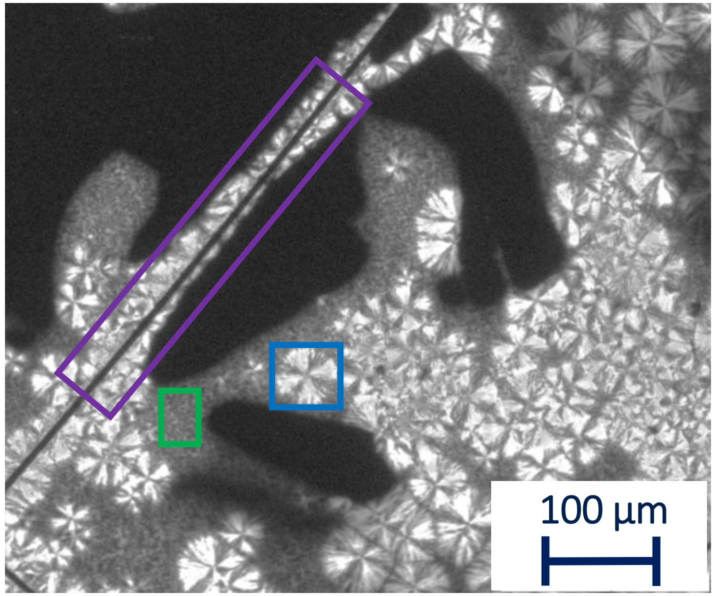
VARIATION OF THE LOCAL PEEK HARDNESS - NANOINDENTATION

Hardness in intra-sph. zone and TC layer ~ 50 % and 150 % resp. higher than in inter-sph. zone (depth ~ 200 nm)



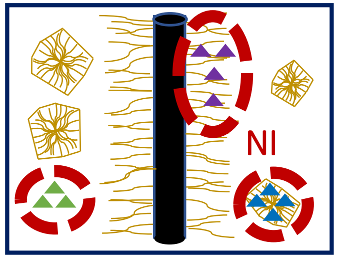


Indents at ~ 20 µm from the fiber

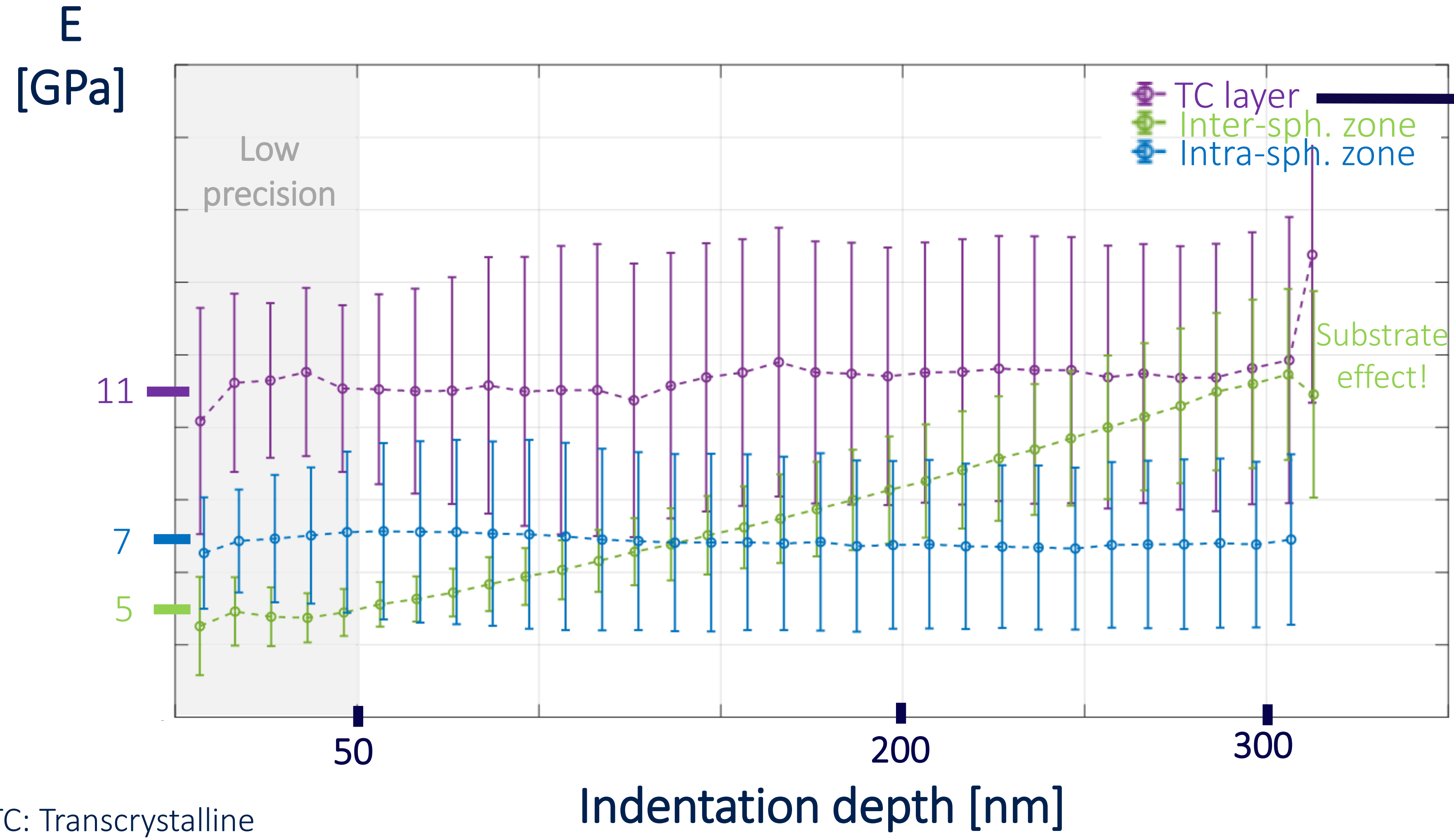


TC: Transcrystalline

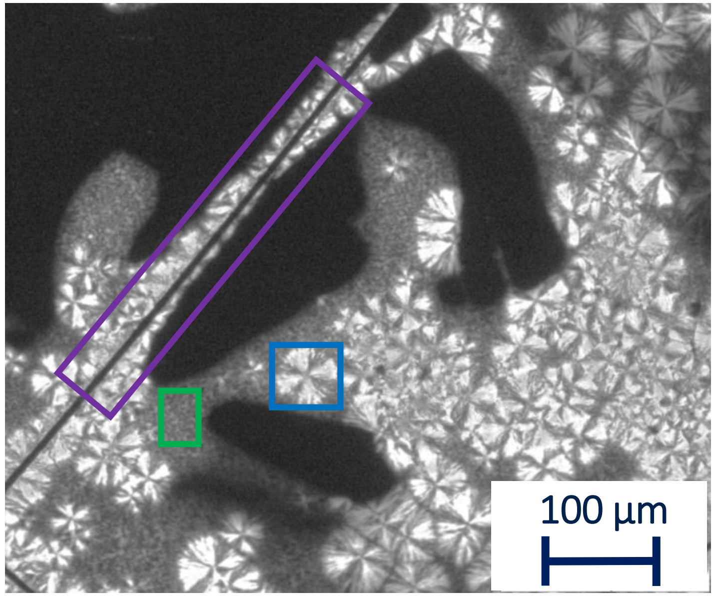
VARIATION OF THE LOCAL PEEK YOUNG'S MODULUS - NANOINDENTATION



E in TC layer ~ 70 % higher than in intra-sph. zone (depth ~ 200 nm)



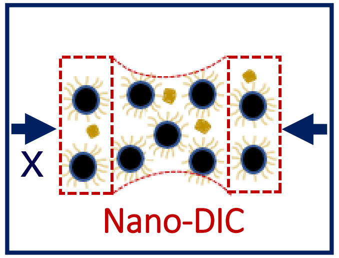
Indents at ~ 20 µm from the fiber



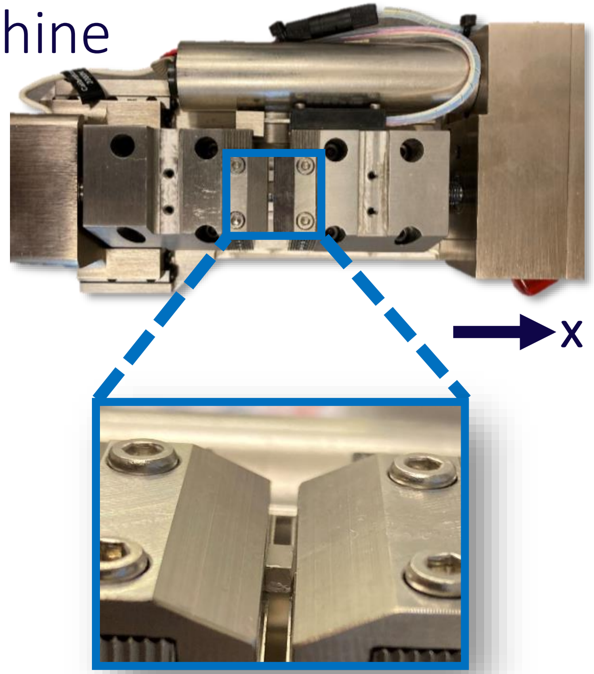
TC: Transcrystalline

TRANSVERSE COMPRESSION TESTS IN SEM + NANO-DIC

→ Access to local strain field in the interfiber zones showing intense plastic localization

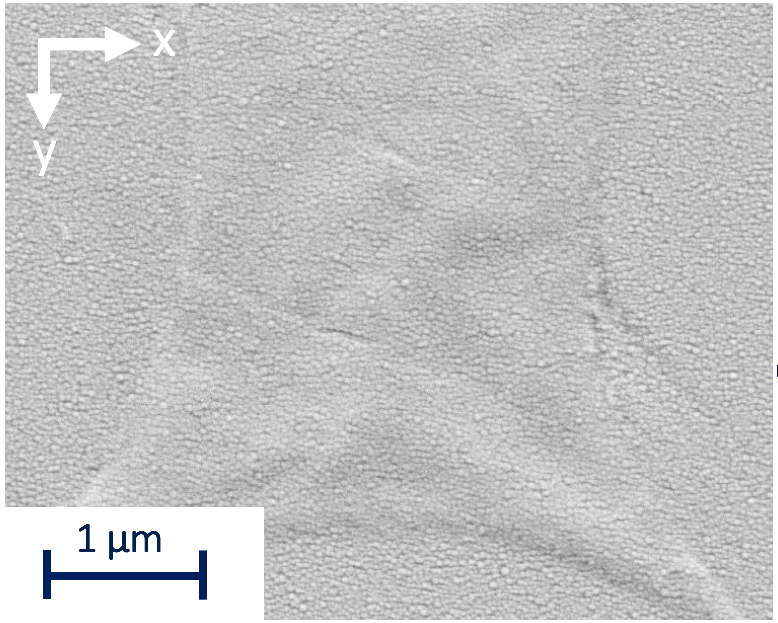


Mini compression machine

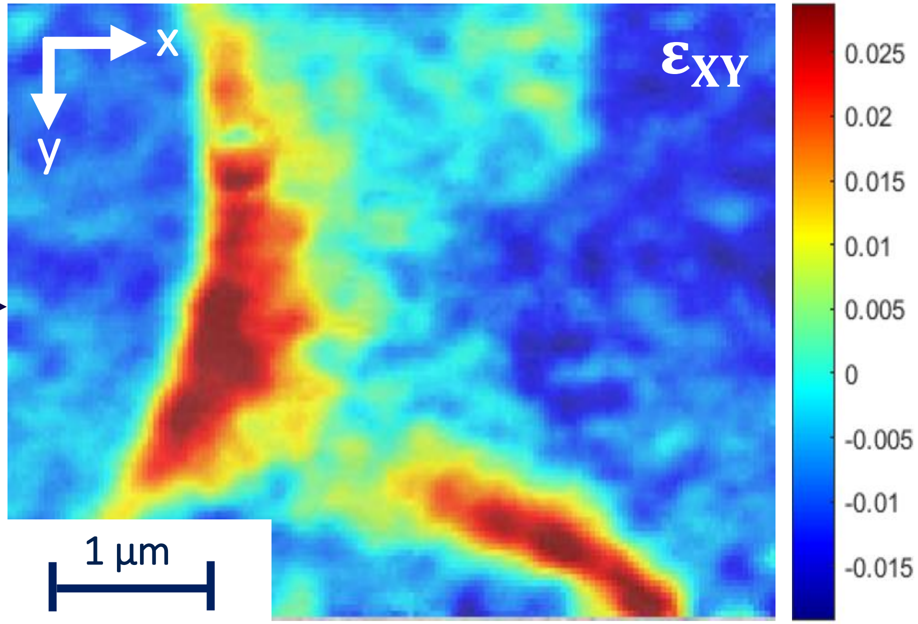


Unidirectional composite with nanometric speckle pattern

N. Klavzer, S. Gayot et al. (2023)



SEM imaging after each load increment



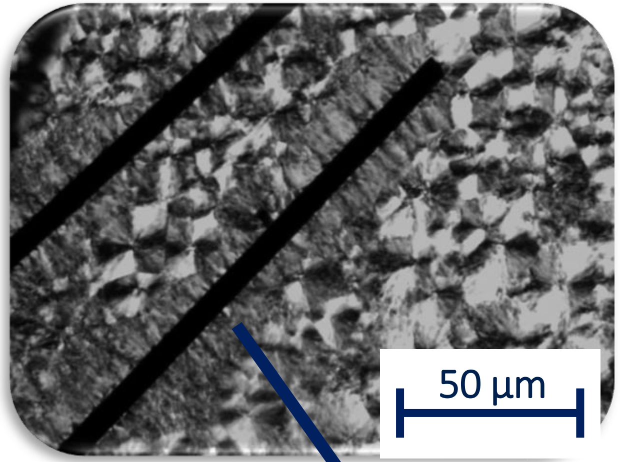
Micro-scale strain mapping from DIC

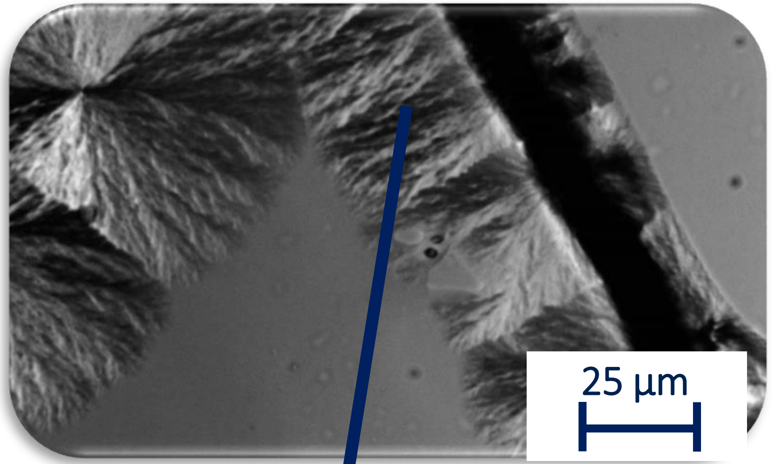
DIC: Digital image correlation

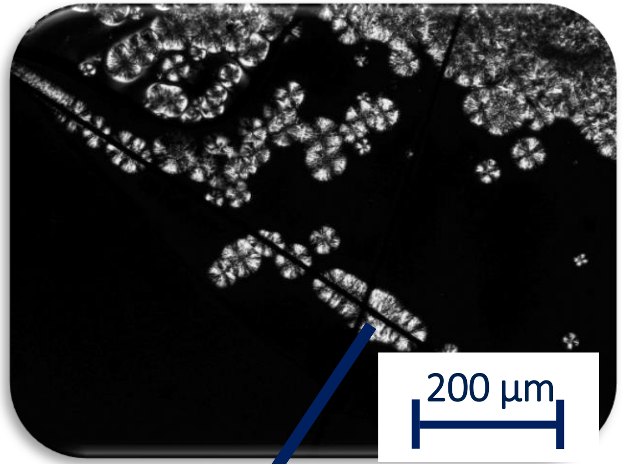
TAKE-HOME MESSAGES

PROCESSING CONDITIONS ⟷ PERFORMANCES

Semi-crystalline thermoplastic composite





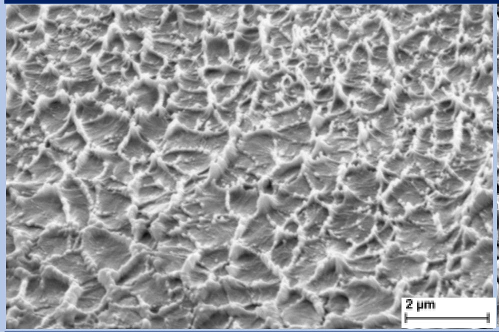


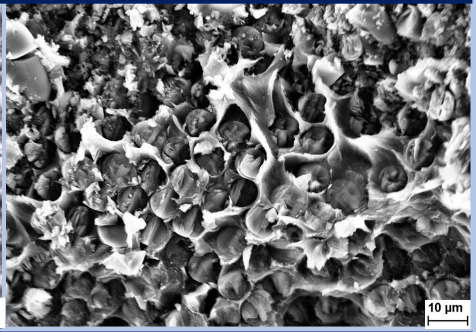
Impact on mechanical behavior in the interfiber zones ?

TAKE-HOME MESSAGES

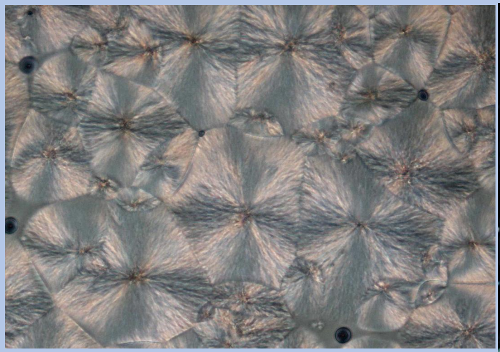
MICROSTRUCTURE

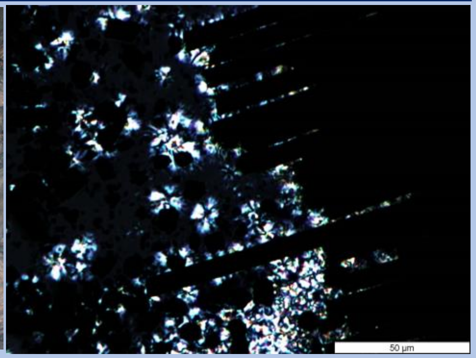
Liq. N_2 fracture + SEM





POM





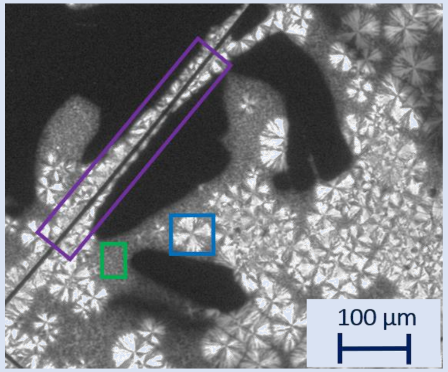
Characterization of PEEK microstructure in composite more complicated than in the neat PEEK due to small interfiber distances (~ 2 µm)

↓

Characterization on model PEEK/C fiber samples

NANO- /MICRO- MECHANICS

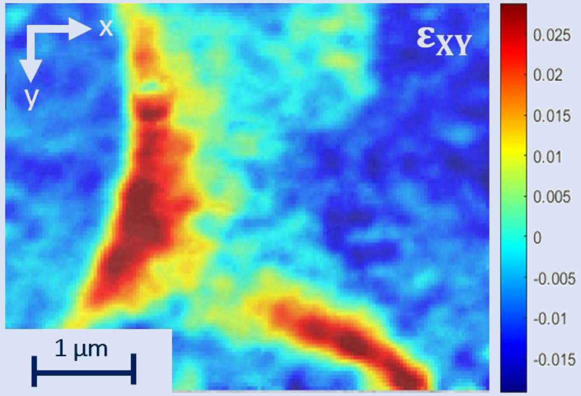
Nanoindentation



H in intra-sph.zone and TC layer ~ 50 % and 150 % resp. higher than in inter-sph. zone

E in TC layer ~ 70 % higher than in intra-sph. zone

Transverse compression in SEM + Nano-DIC



Access to µm-scale strain field in the interfiber zones sophie.vanpee@uclouvain.be

Back-up slides

Composites: Part A 168 (2023) 107455

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Composites Part A

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Nanoscale digital image correlation at elementary fibre/matrix level in polymer–based composites

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Correlation Among Crystalline Morphology of PEEK, Interface Bond Strength, and In-Plane Mechanical Properties of Carbon/PEEK Composites

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Department of Mechanical Engineering, Hong Kong University of Science and Technology, Clear Water Bay, Hong Kong

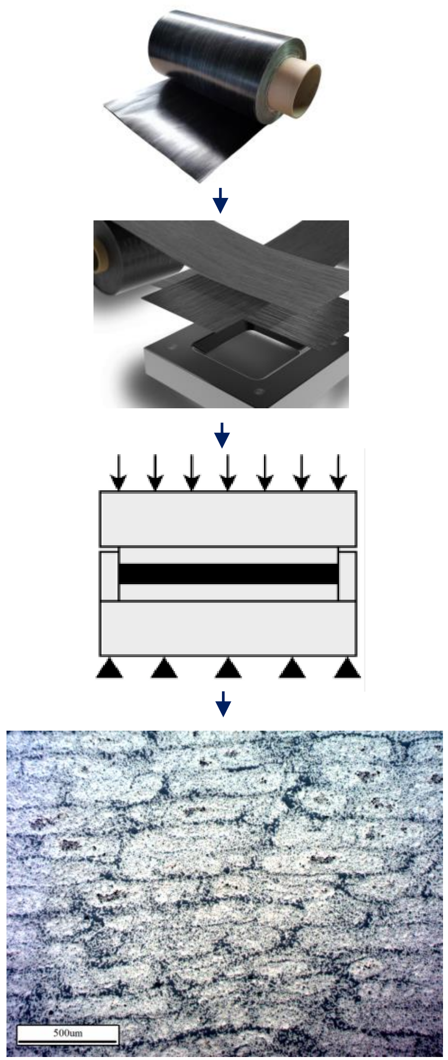
Received 26 March 2001; accepted 29 May 2001

Table I Summary of the Tensile Test Results and Crystallinity of Neat PEEK Resin

	Cooling Rate (°C/min)					
	1	80	160	600	1000	2000
Property	Mean ± SD	Mean ± SD	Mean ± SD	Mean ± SD	Mean ± SD	Mean ± SD
Tensile strength (MPa)	108.5 ± 5.0	93.8 ± 9.0	92.5 ± 3.5	71.7 ± 10.4	57.5 ± 3.5	53.8 ± 3.3
Tensile modulus (GPa)	4.6 ± 0.5	—	4.0 ± 0.3	3.7 ± 0.1	3.3 ± 0.2	3.0 ± 0.2
Failure strain (%)	3.0 ± 1.4	9.8 ± 1.4	7.9 ± 3.0	—	115 ± 7.1	183.3 ± 5.8
Crystallinity (%)	38	30	28	26	19	17

MATERIALS

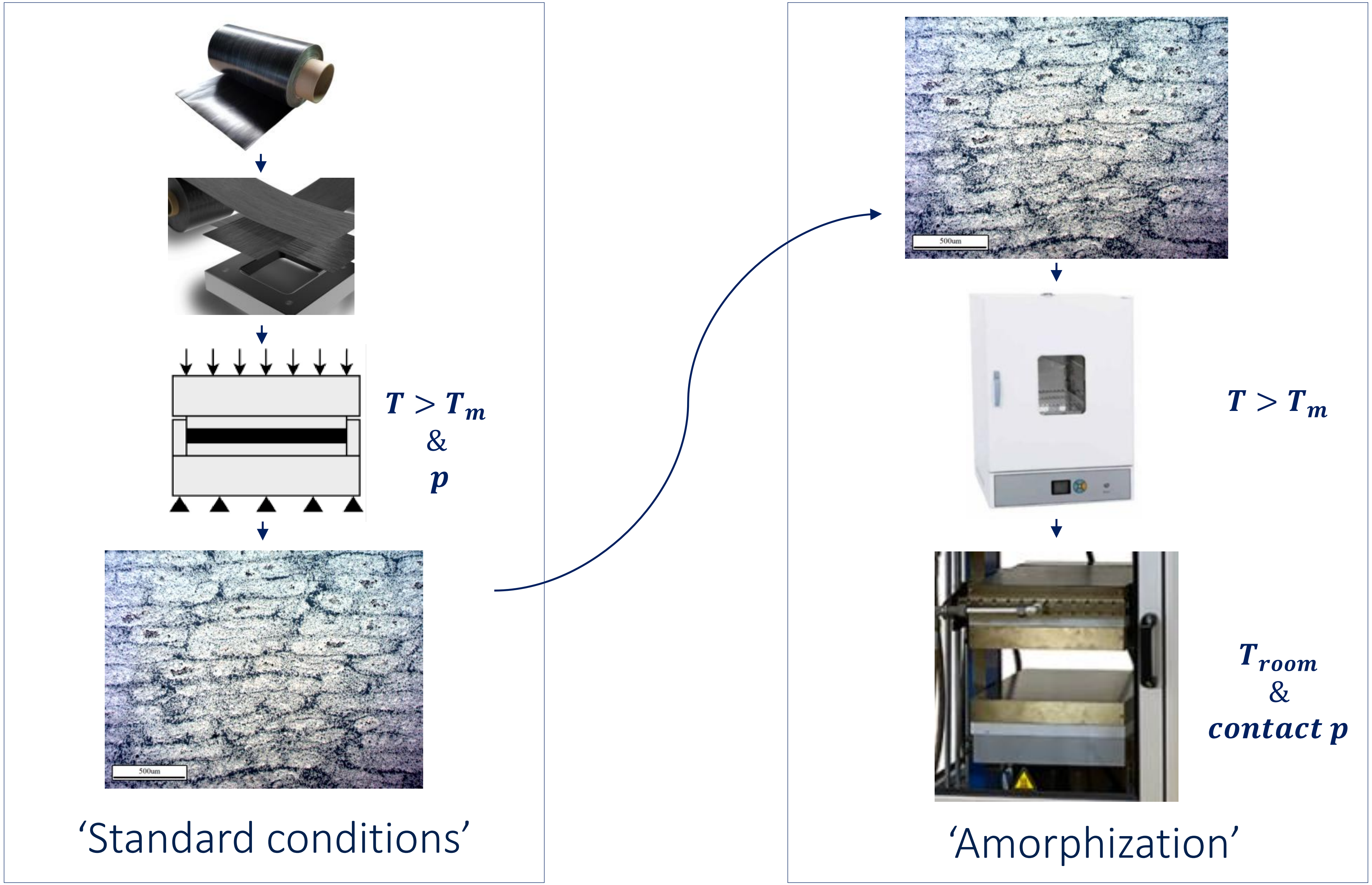
Semi-crystalline thermoplastic



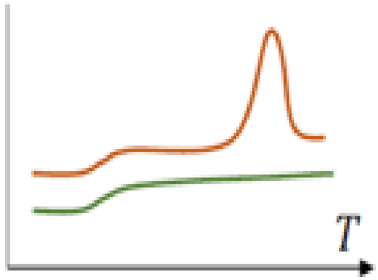
Unidirectional (UD) composite

‘Model’ samples

UD COMPOSITE

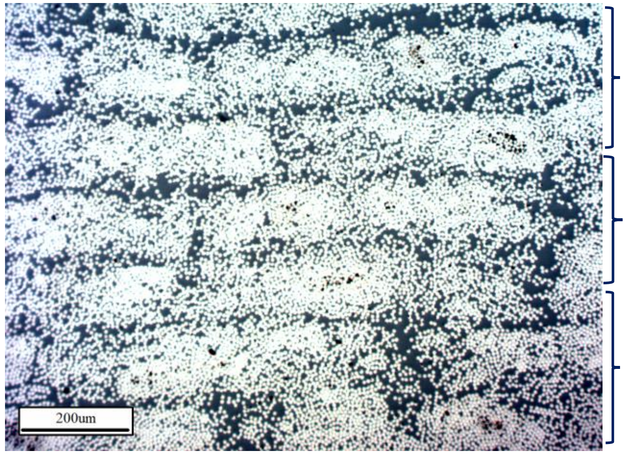


DEGREE OF CRYSTALLINITY - χ_c



DSC

'Standard conditions'

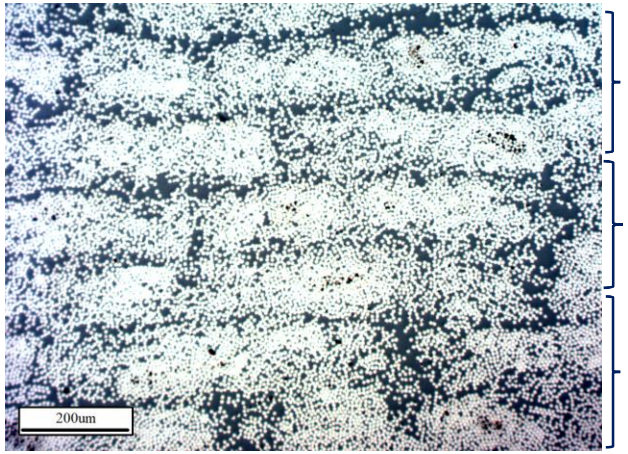


χ_c ~ 34 %

χ_c ~ 36 %

χ_c ~ 28 %

'Amorphization'



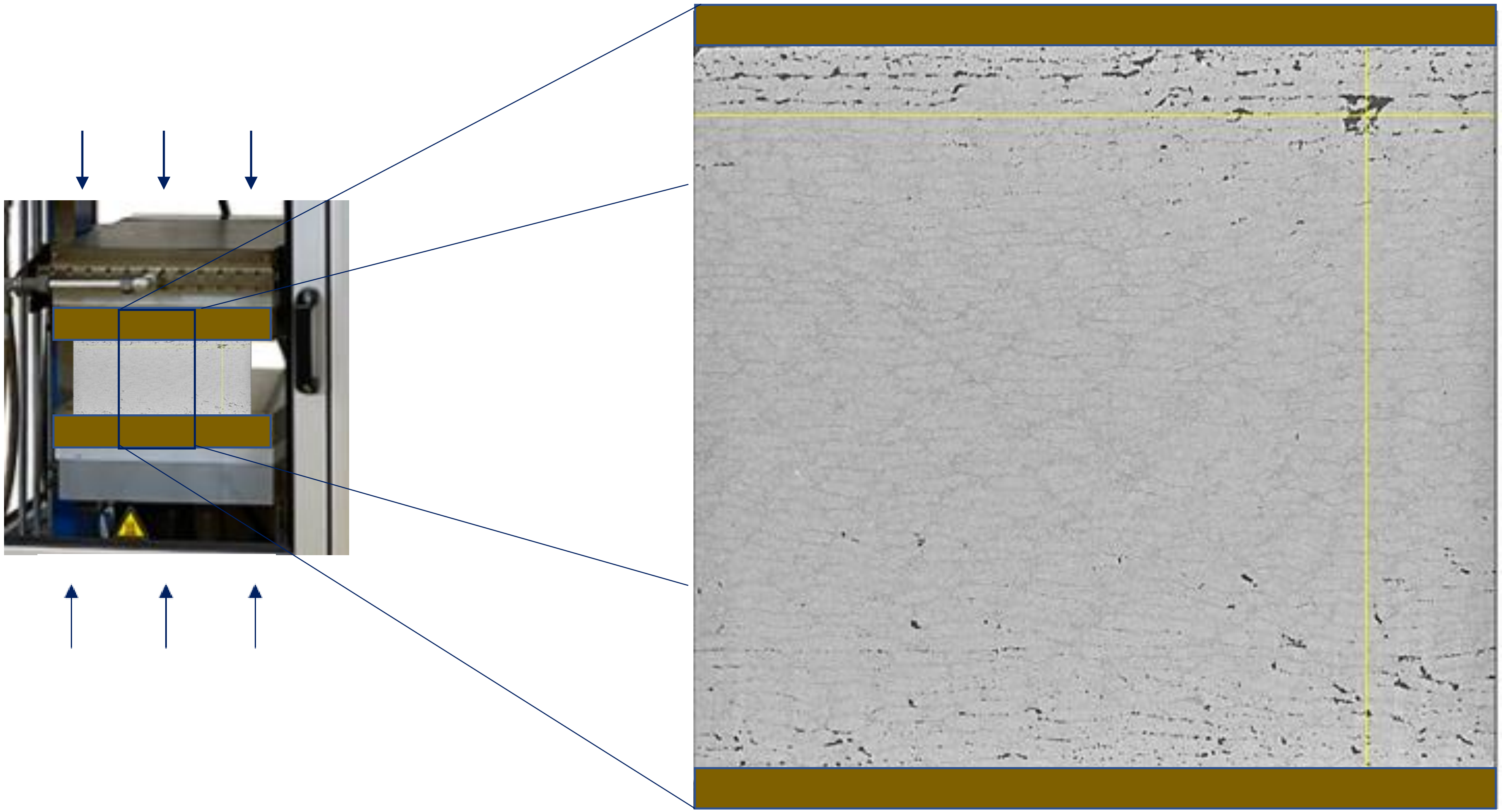
χ_c ~ 15 %

χ_c ~ 28 %

χ_c ~ 18 %

DSC: differential scanning calorimetry

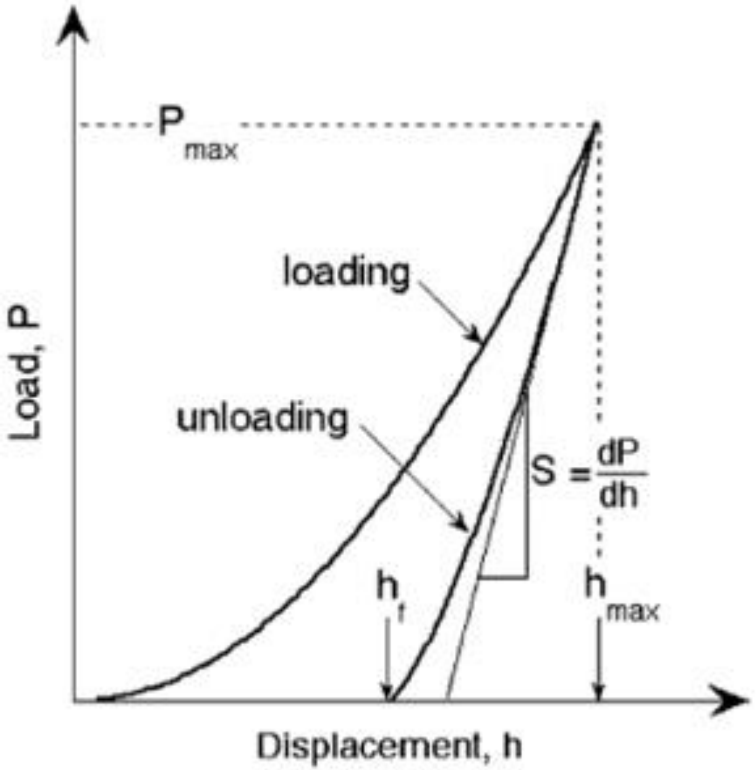
DEFECTS DETECTION – TOMOGRAPHY



PRINCIPLE OF NANOINDENTATION

Instrumented nanoindentation: *indentation depth* of the tip and the *force* are measured continuously during the test

Direct calculation of *Young modulus* and *hardness* from the *“force* vs *depth”* curve *



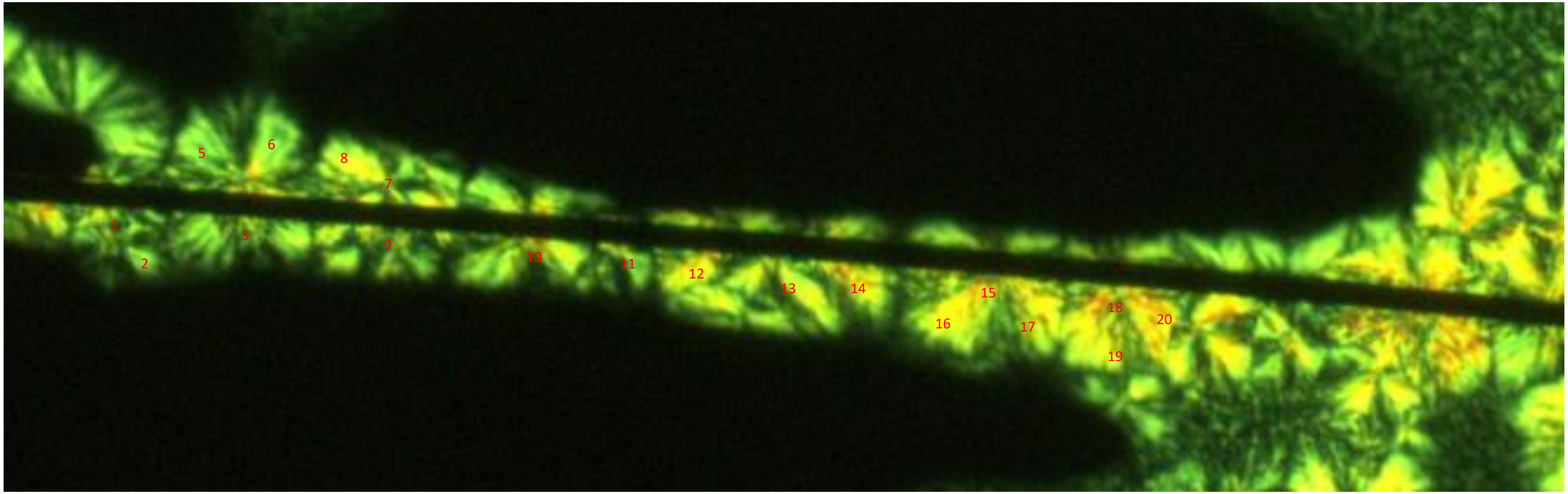
$$H = \frac{P_{\max}}{A}$$

$$S = \beta \frac{2}{\sqrt{\pi}} E_{\text{eff}} \sqrt{A}$$

$$\frac{1}{E_{\text{eff}}} = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i}$$

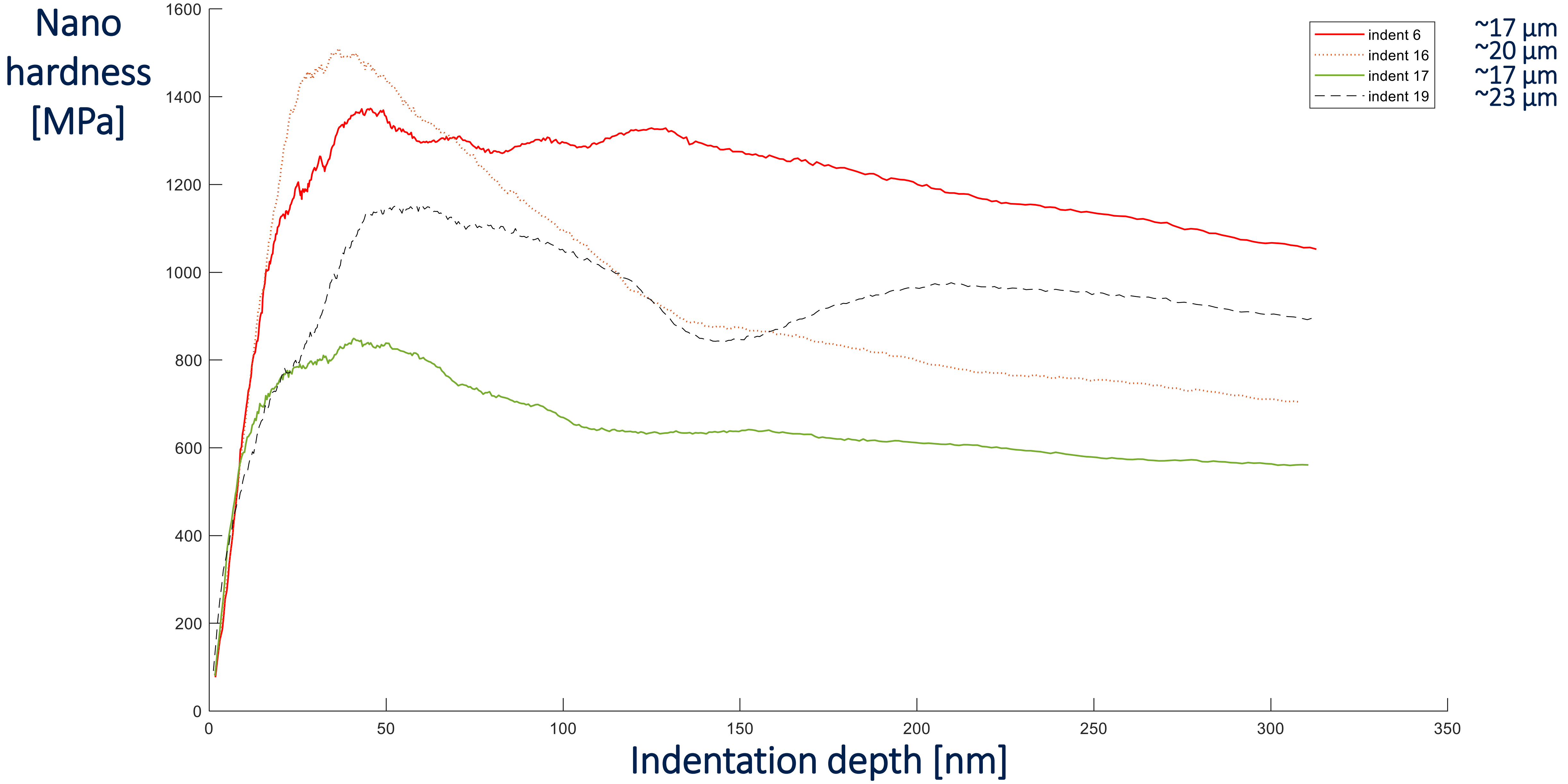
*Olivier & Pharr

VARIATION OF THE LOCAL PEEK HARDNESS - NANOINDENTATION

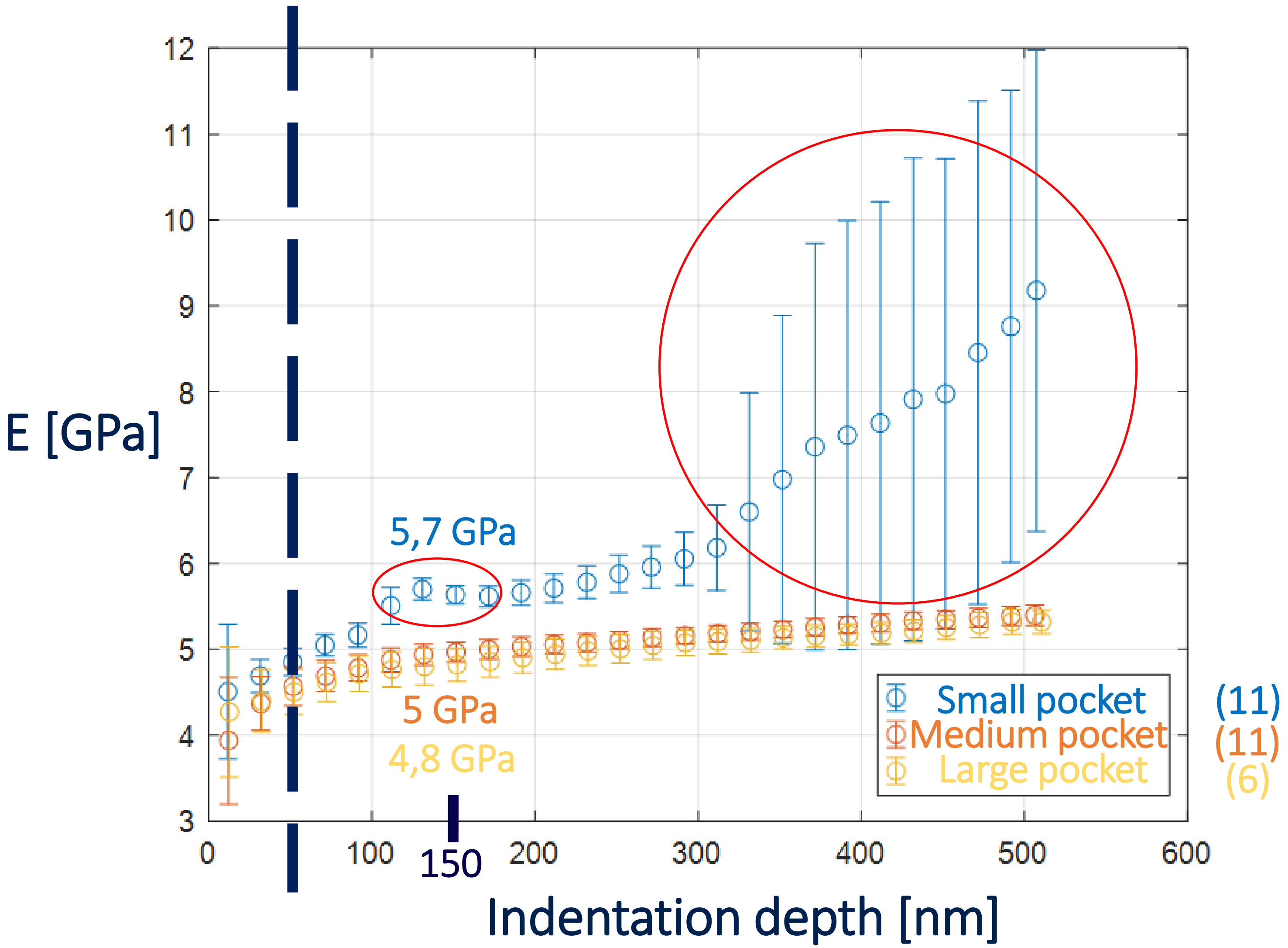


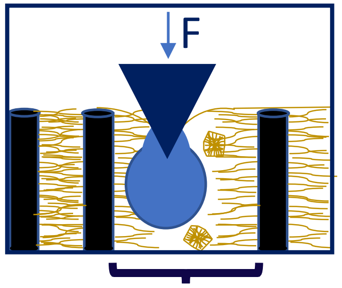
TC layer

VARIATION OF THE LOCAL PEEK HARDNESS - NANOINDENTATION



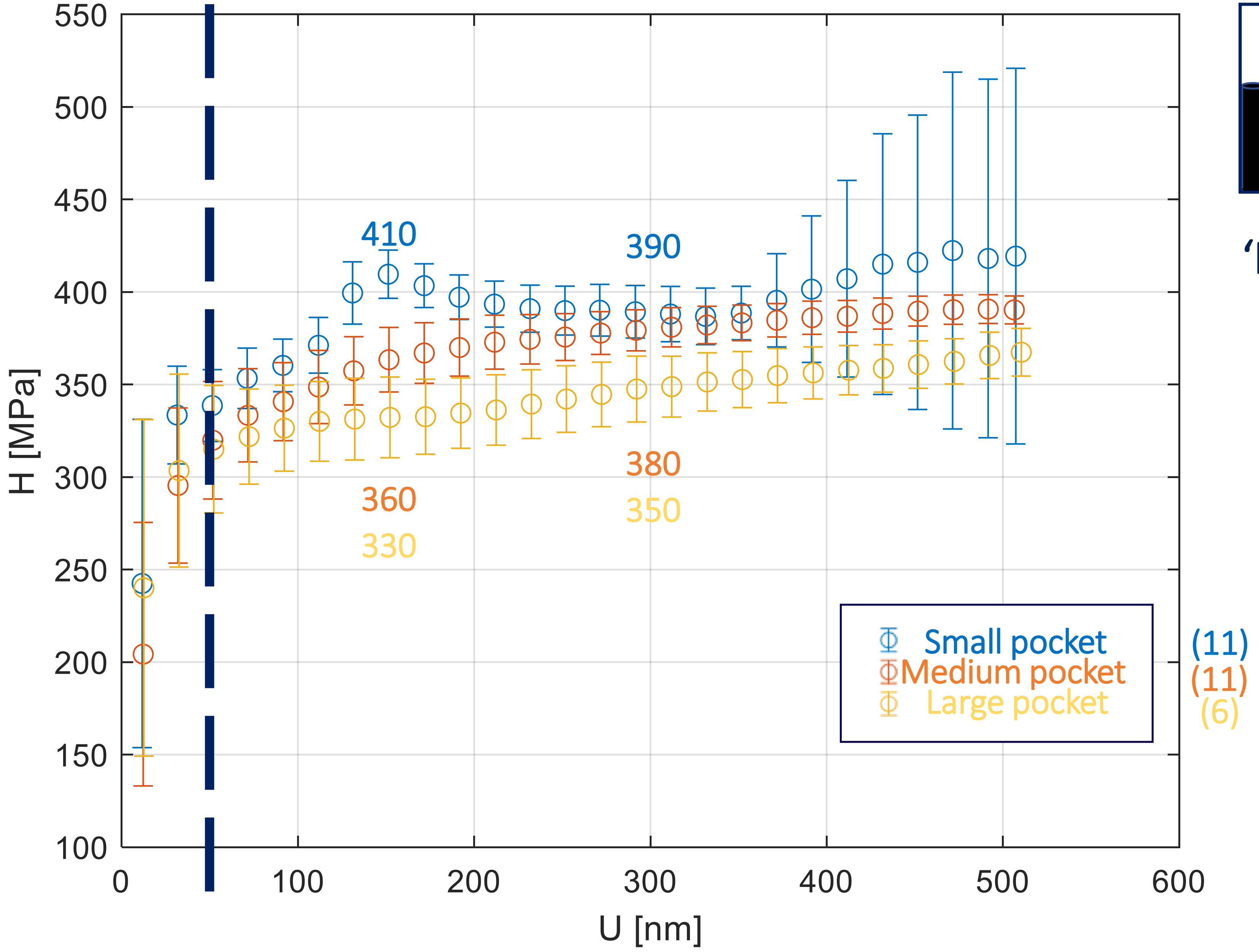
NEAR FIBER MATRIX MECHANICAL PROPERTIES - NANOINDENTATION

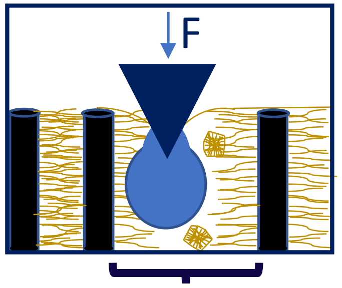




'PEEK pocket'

NEAR FIBER MATRIX MECHANICAL PROPERTIES - NANOINDENTATION

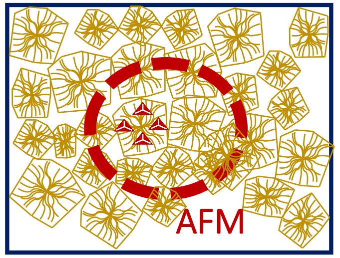


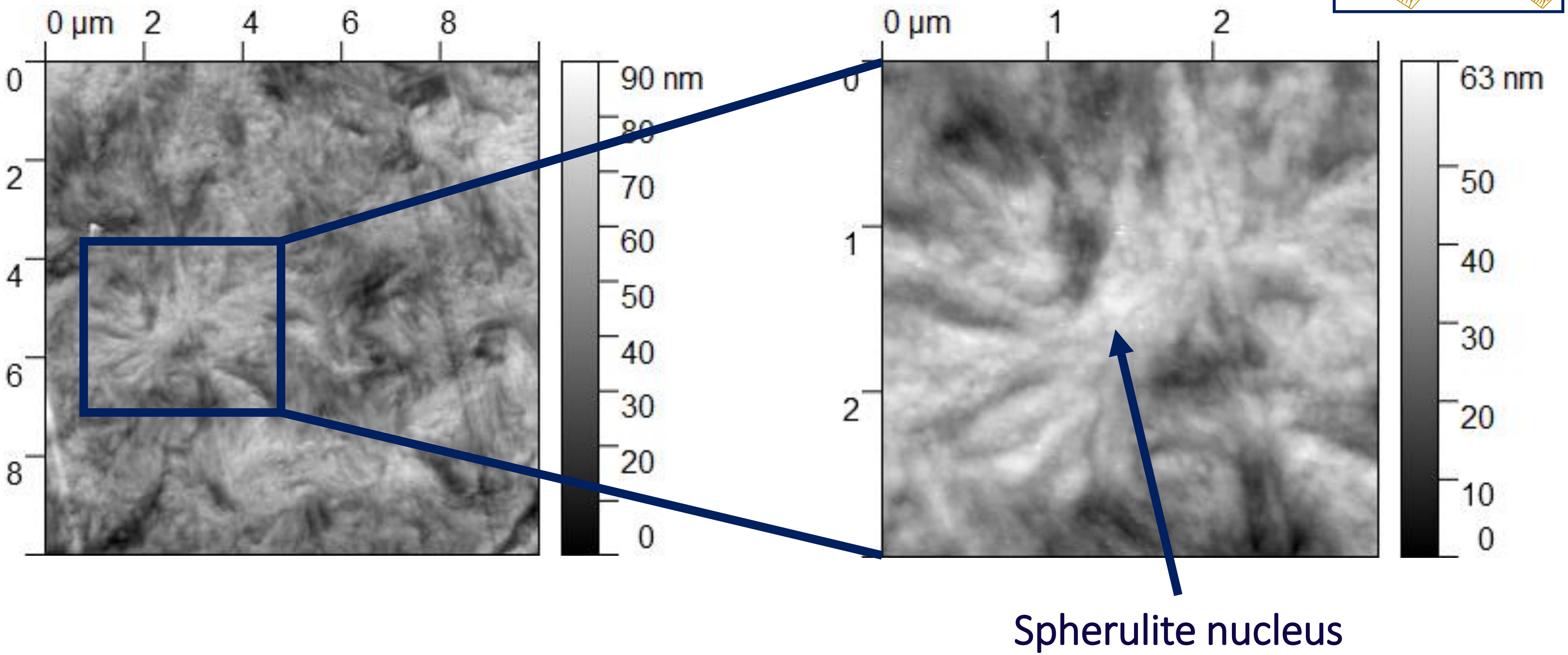


‘PEEK pocket’

CHARACTERIZATION OF SPHERULITES- AFM

Topographic contrast

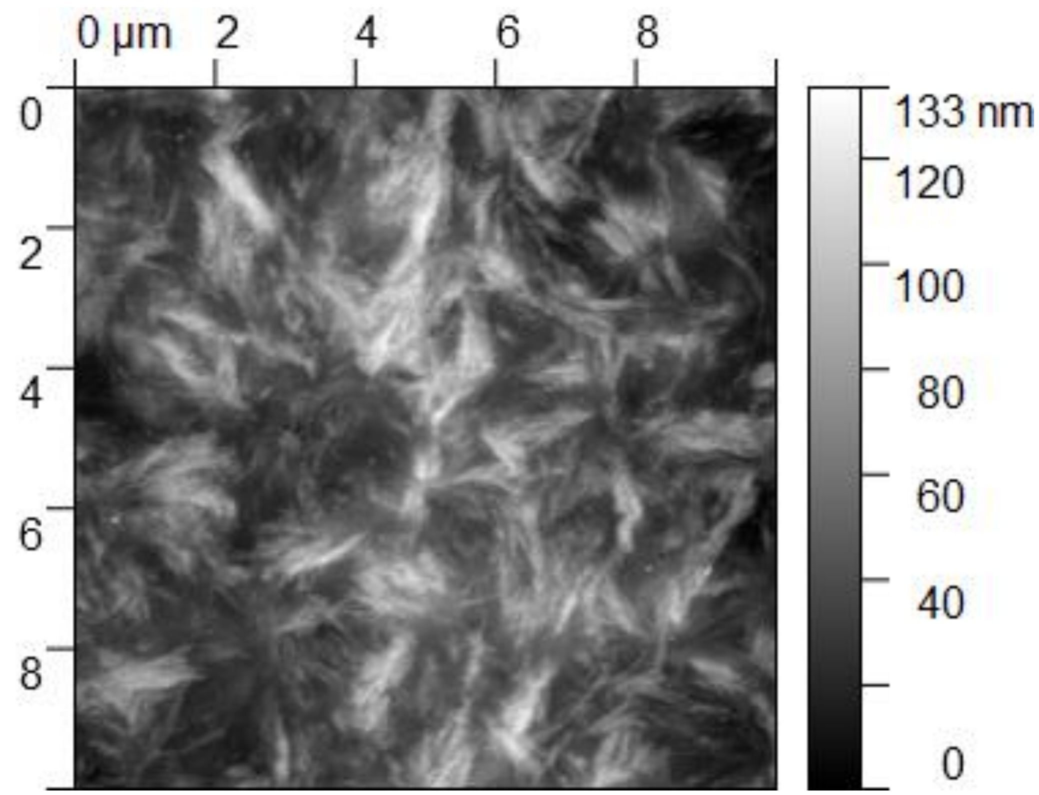


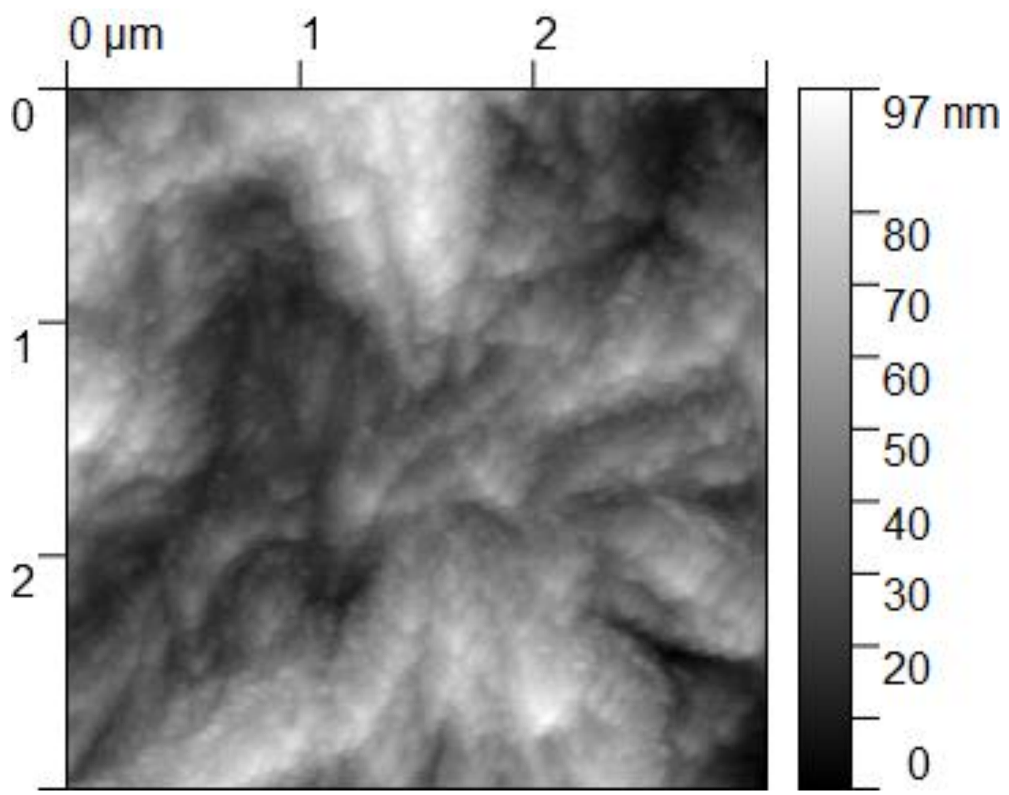


AFM: atomic force microscopy

SPHERULITES OBSERVATIONS - AFM

Topographic contrast
Etched samples

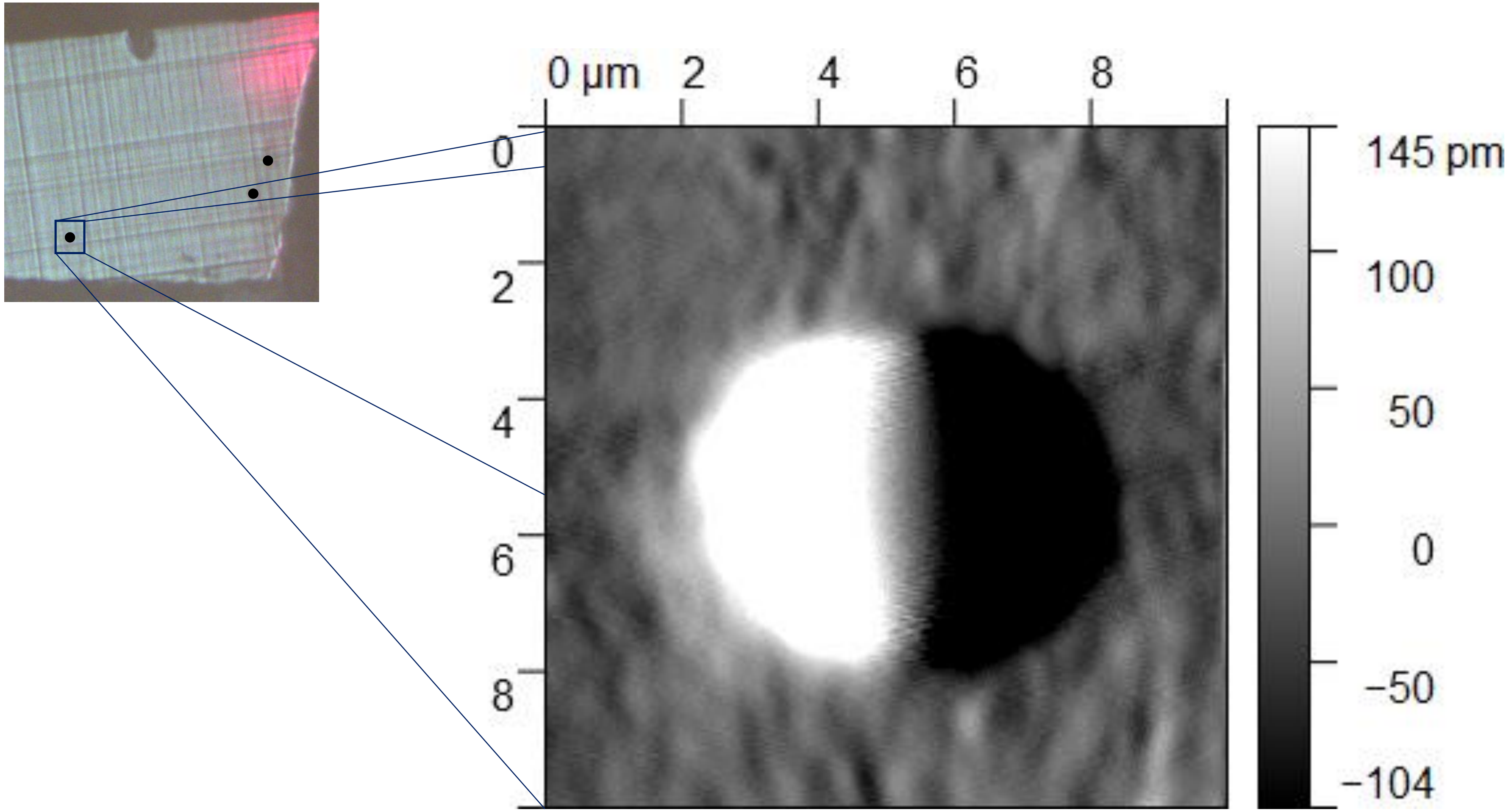




AFM: atomic force microscopy

TRANSCRYSTALIZATION OBSERVATIONS - AFM

Error image

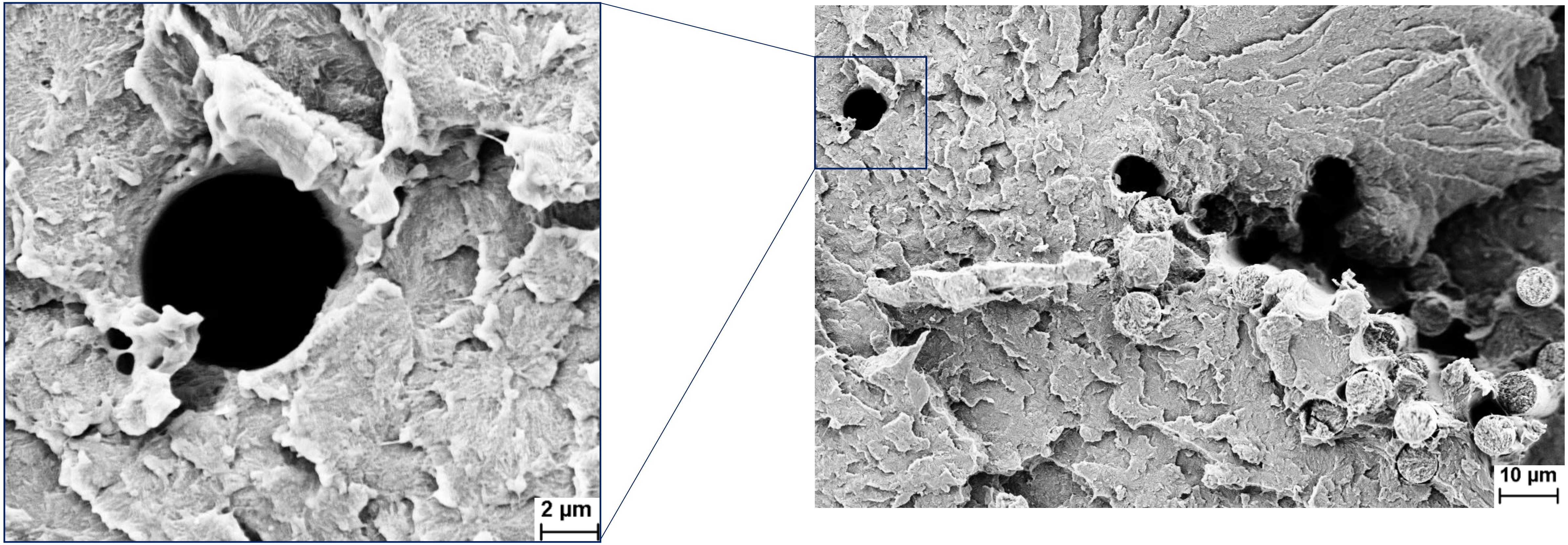


Difficulties:

- Differential erosion
- Fiber/matrix interface decohesion during sample preparation
- Presence of fibers => cutting line on the surface

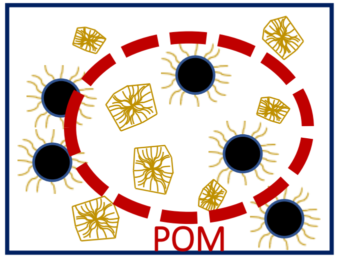
AFM: atomic force microscopy

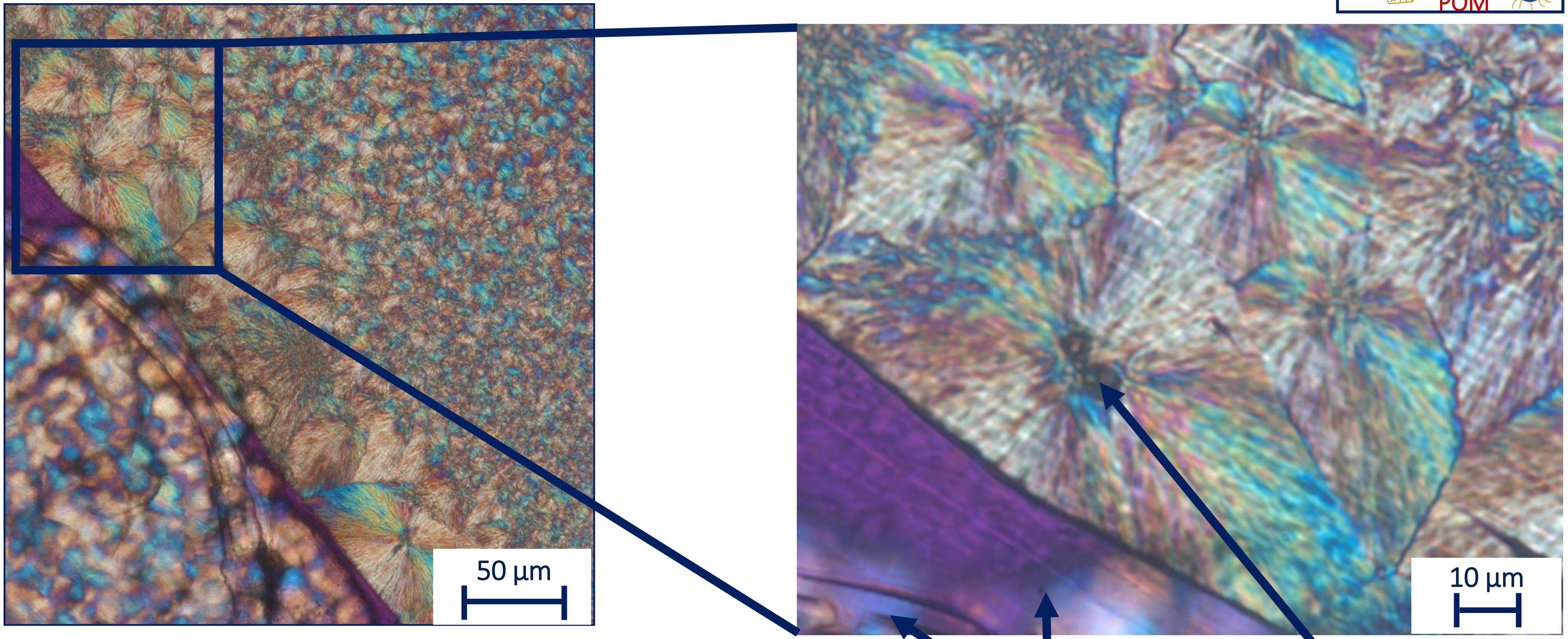
TRANSCRYSTALIZATION OBSERVATIONS - Liq. N_2 fracturing + SEM



'Model' samples

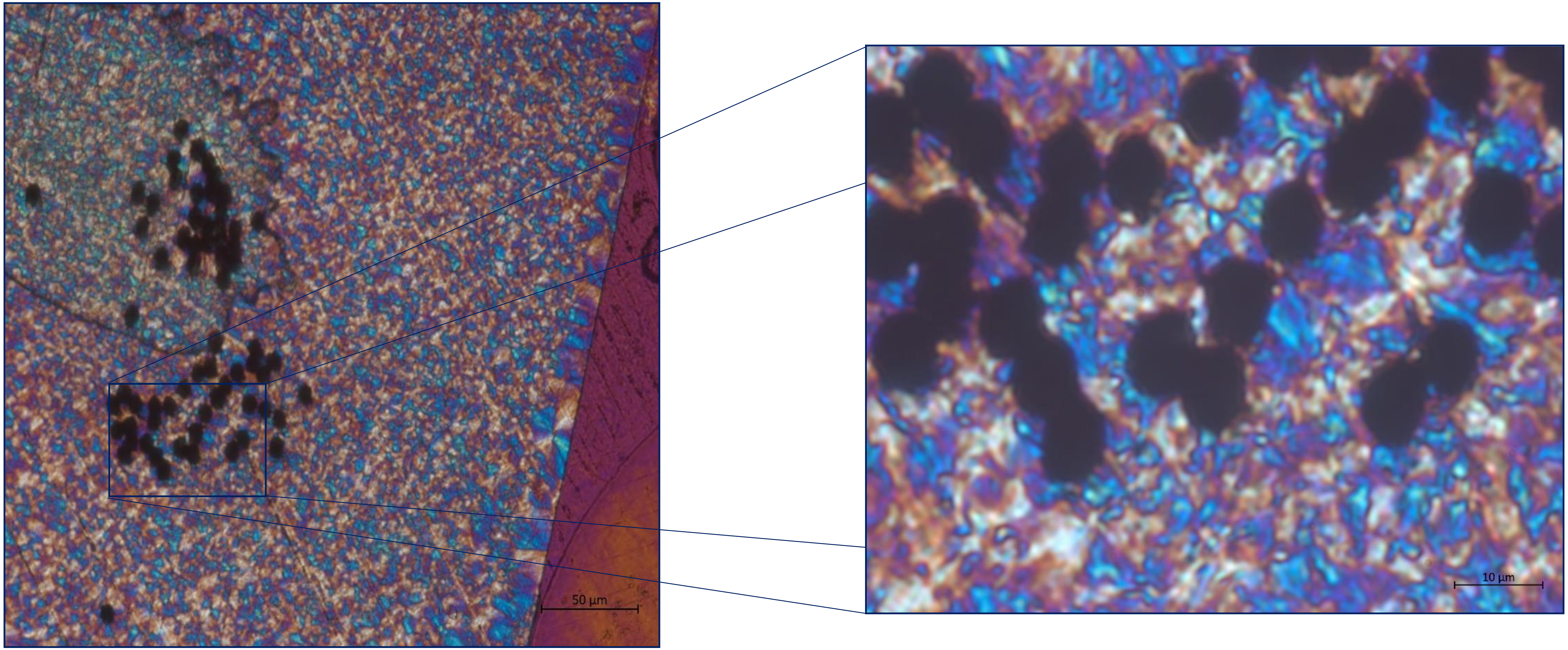
TRANSCRYSTALLIZATION OBSERVATIONS - POM





POM: polarized optical microscopy

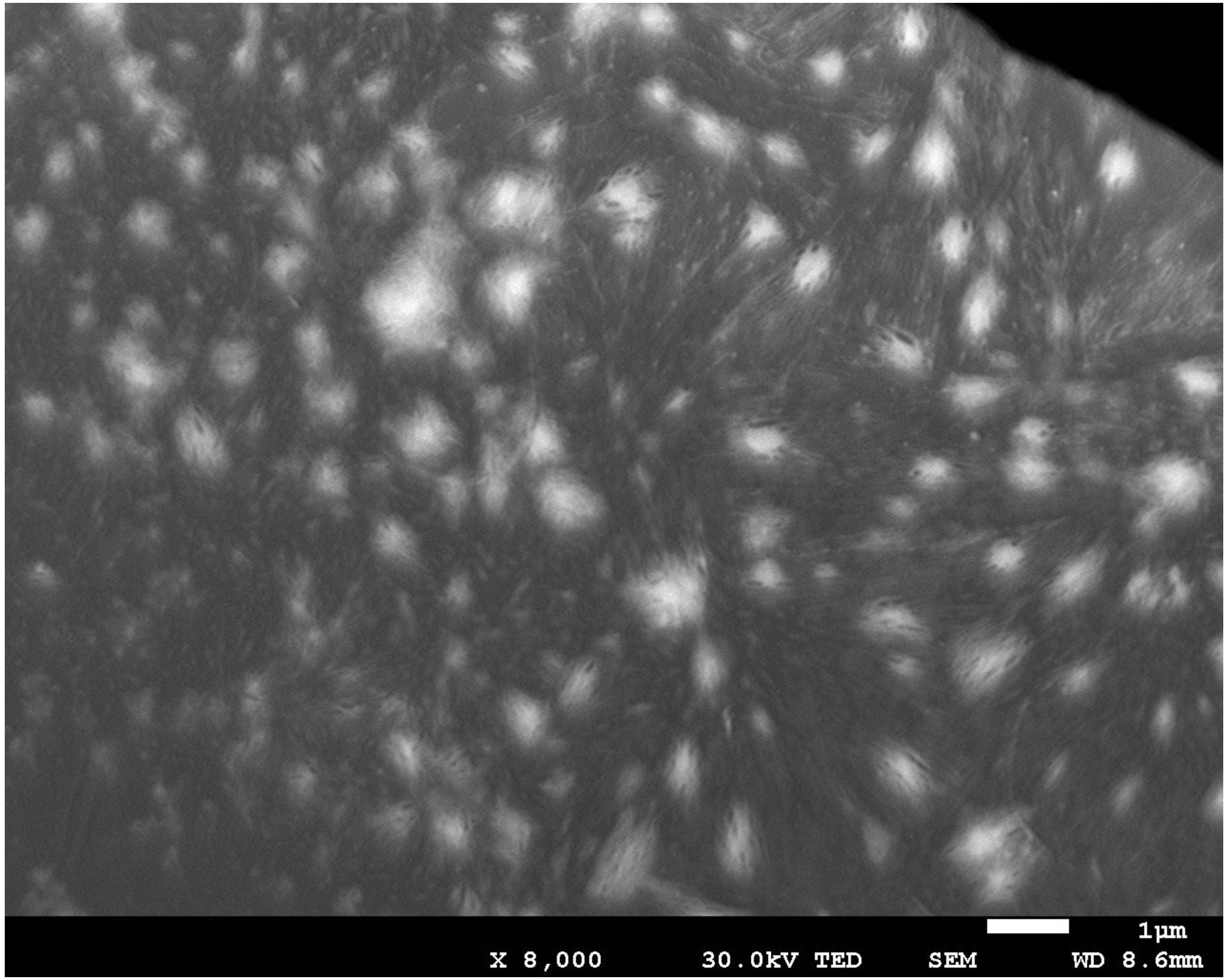
TRANSCRYSTALIZATION OBSERVATIONS - POM



‘Model’ samples

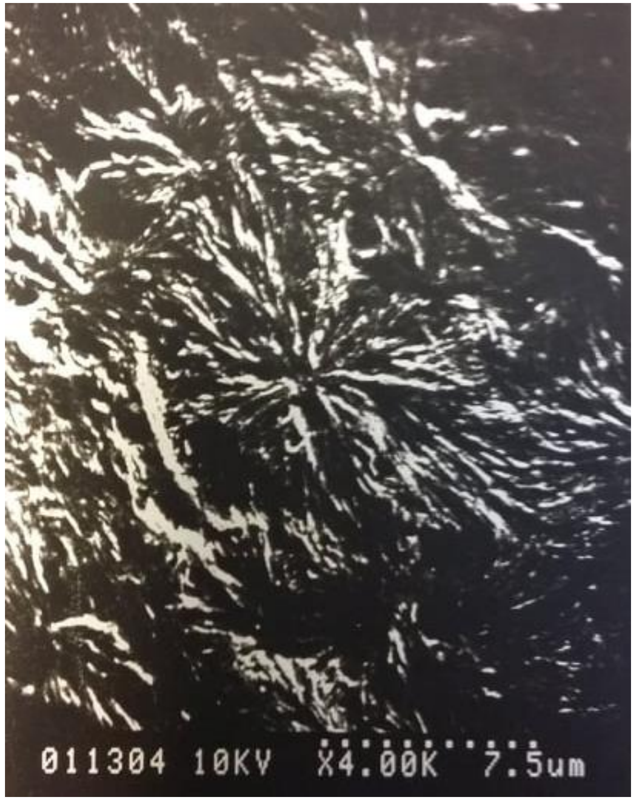
POM: polarized optical microscopy

SPHERULITES OBSERVATIONS - STEM



STEM: scanning TEM

SPHERULITES OBSERVATIONS - PERMANGANATIC ETCHING + SEM

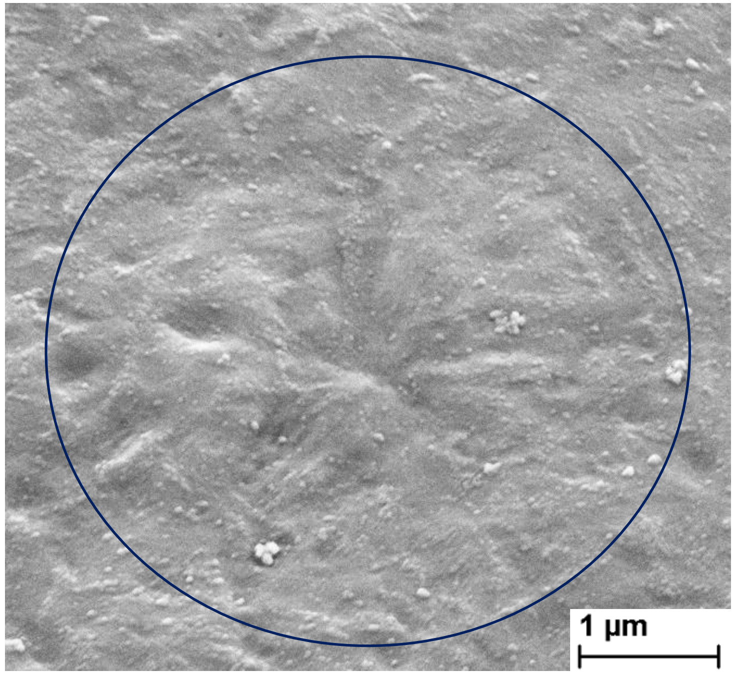


M. Debaut (1990-1991)

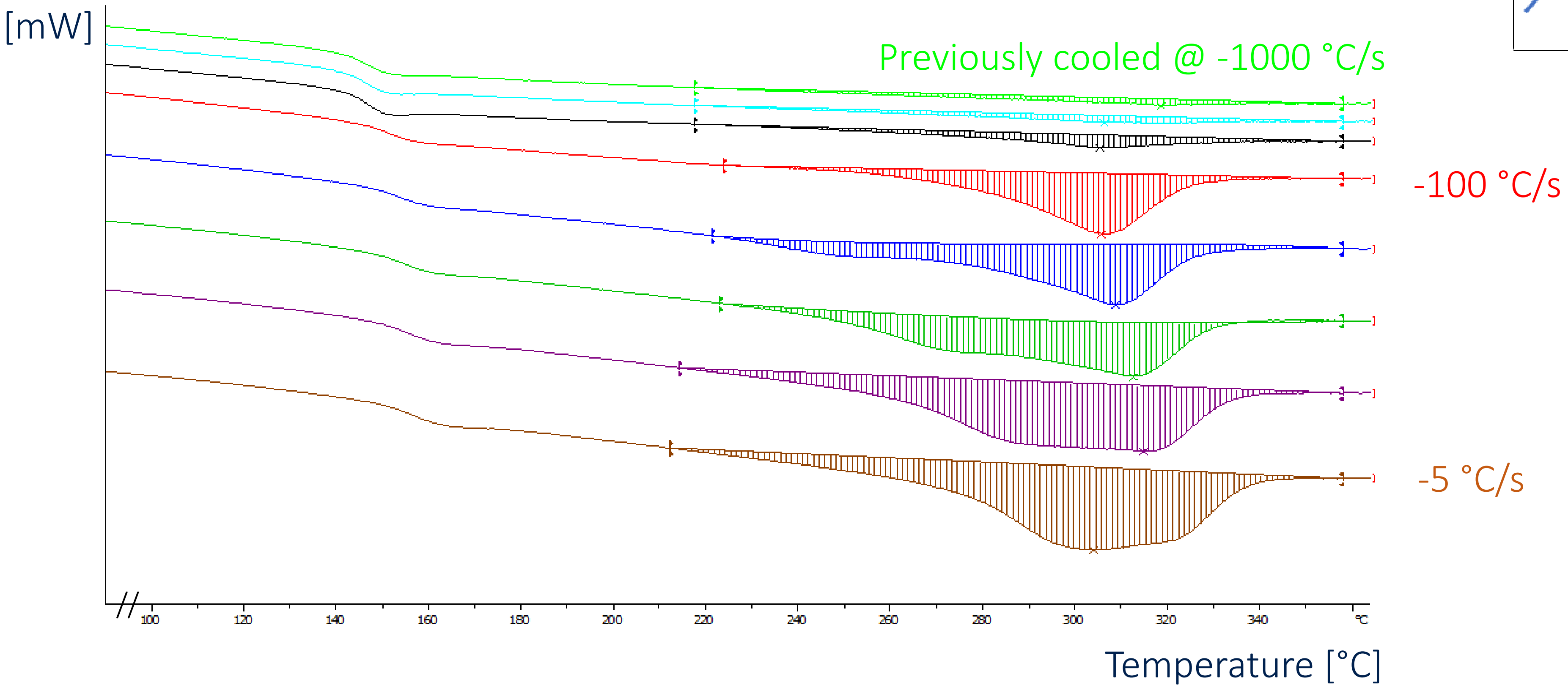
Chemical etching step (5 min):
sulfuric acid, phosphoric acid, distilled water & potassium permanganate

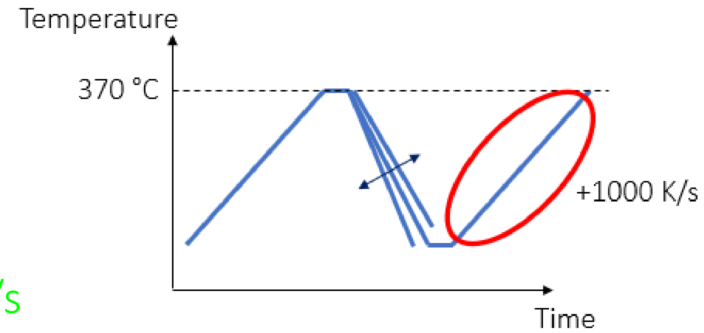
Rinsing step (1 min):
hydrogen peroxyde & water

Drying step:
Acetone



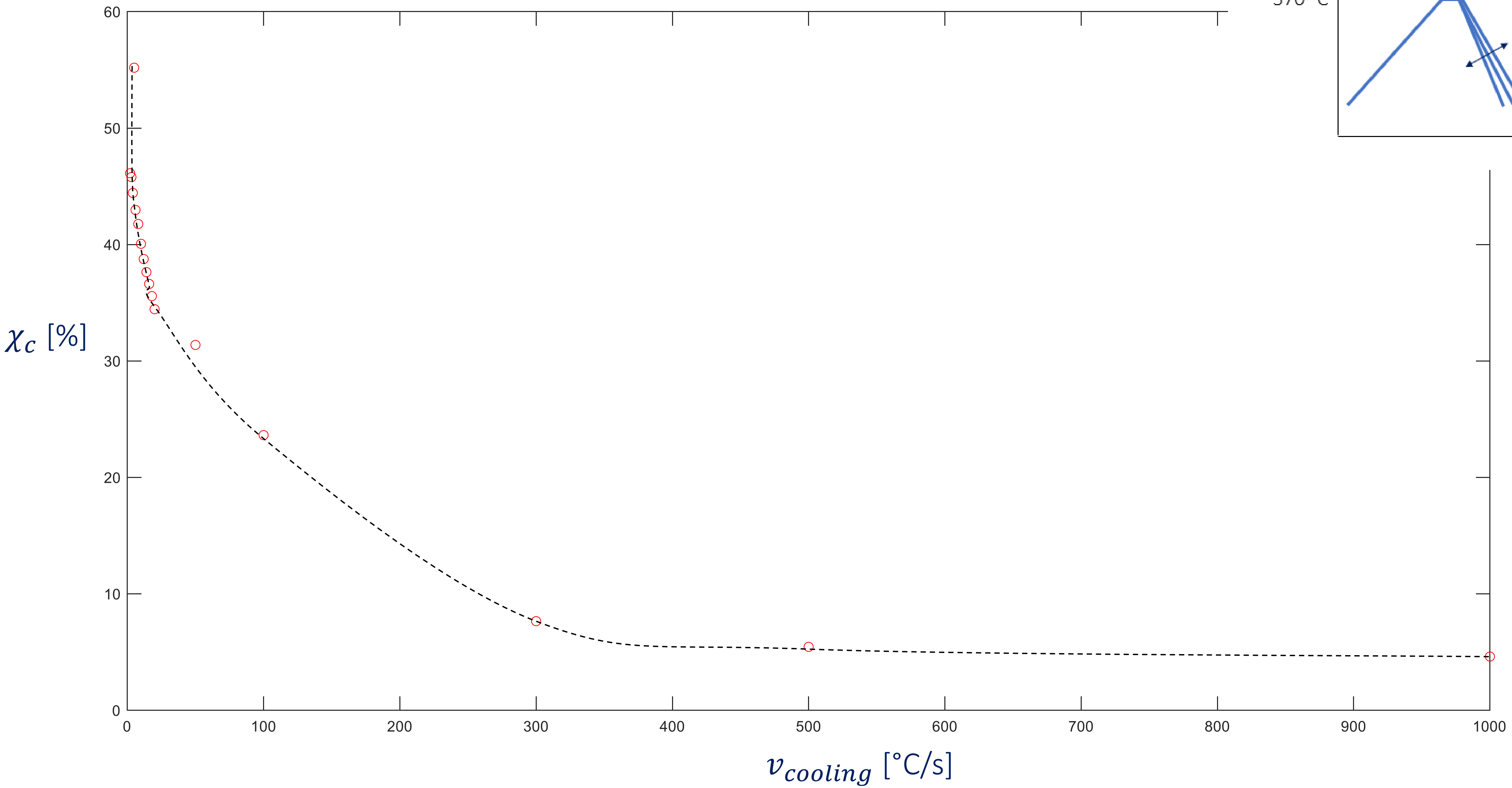
DEGREE OF CRYSTALLINITY – IMPACT OF COOLING RATE

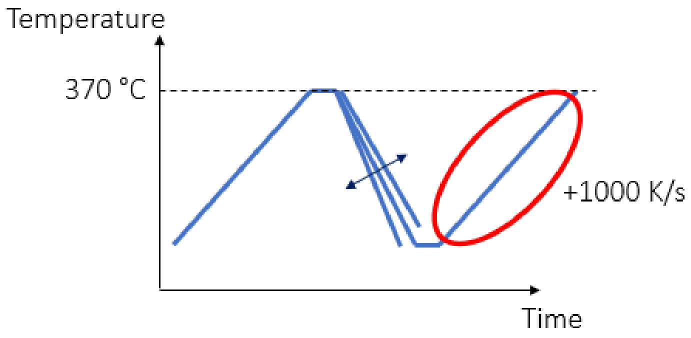




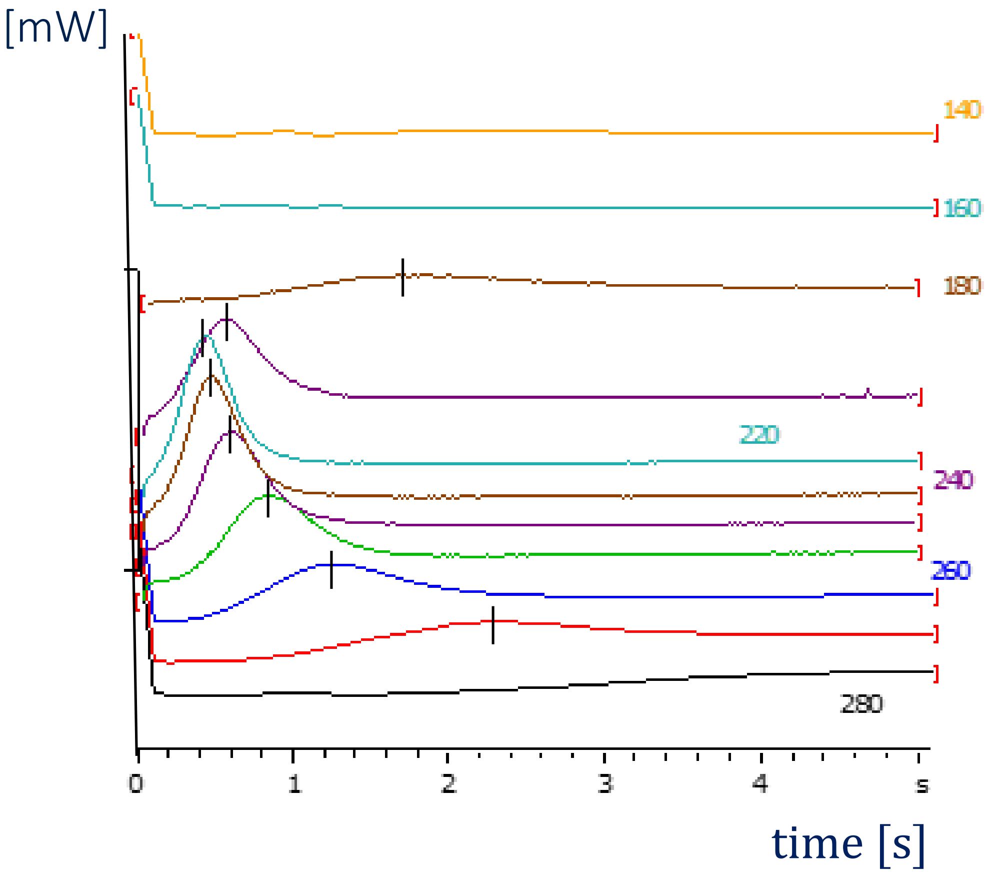
FDSC: flash differential scanning calorimetry

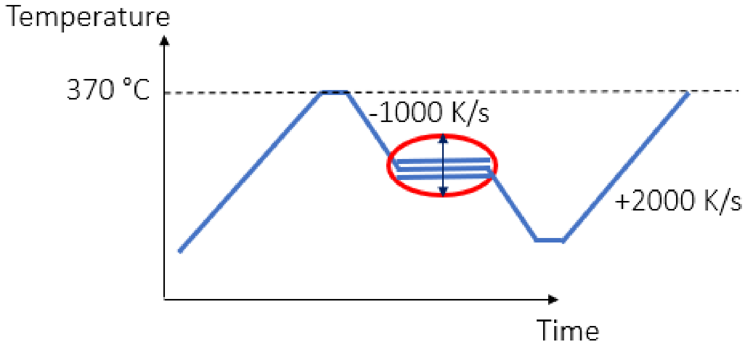
DEGREE OF CRYSTALLINITY – IMPACT OF COOLING RATE

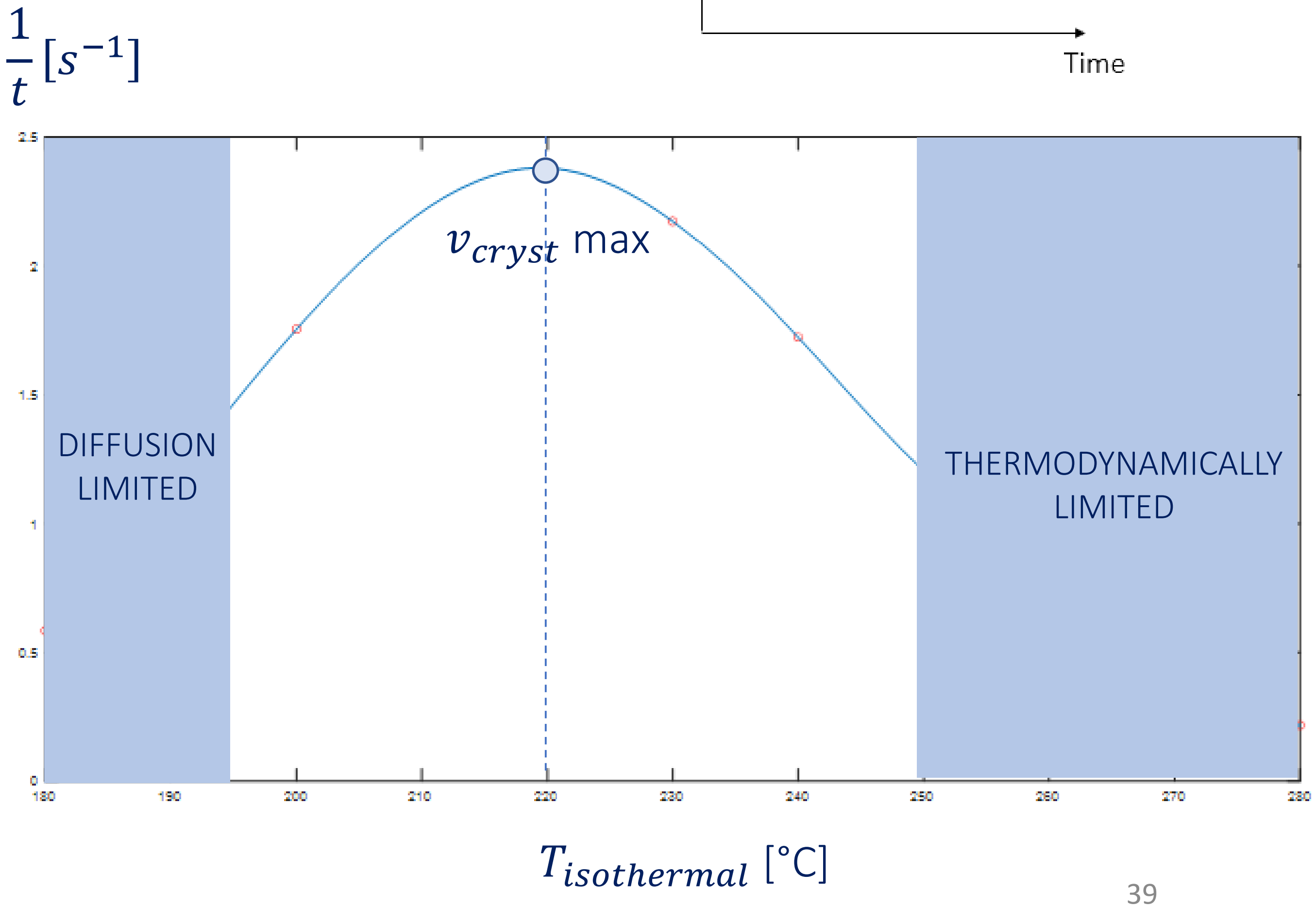




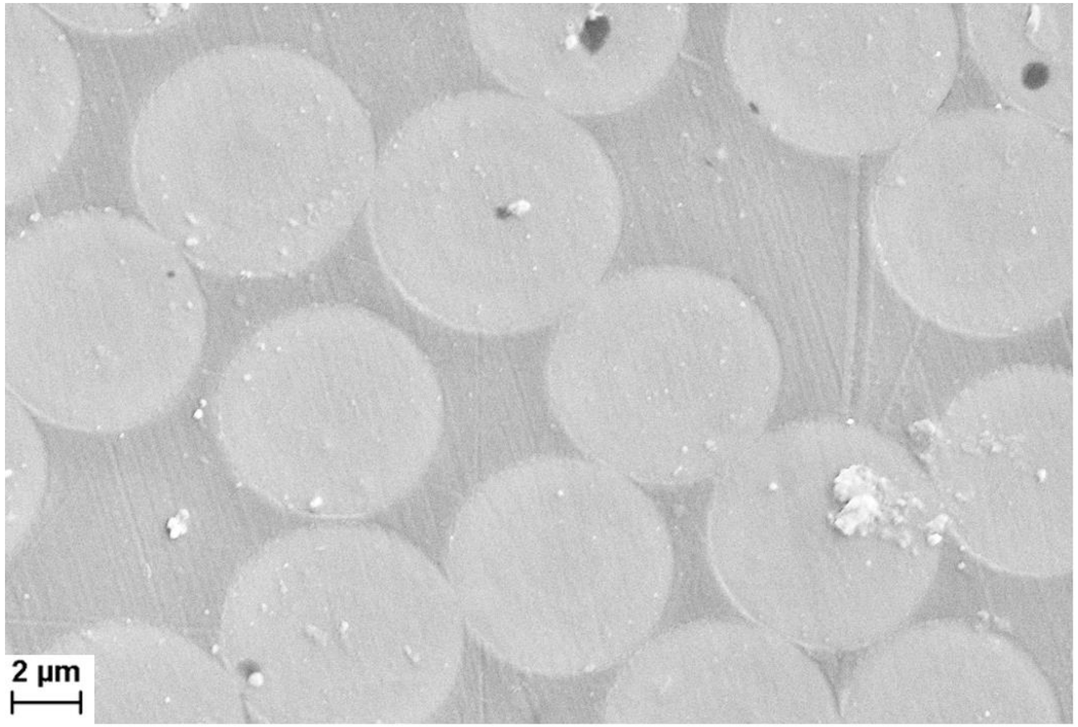
CRYSTALLISATION RATE – ISOTHERMAL CRYSTALLIZATION



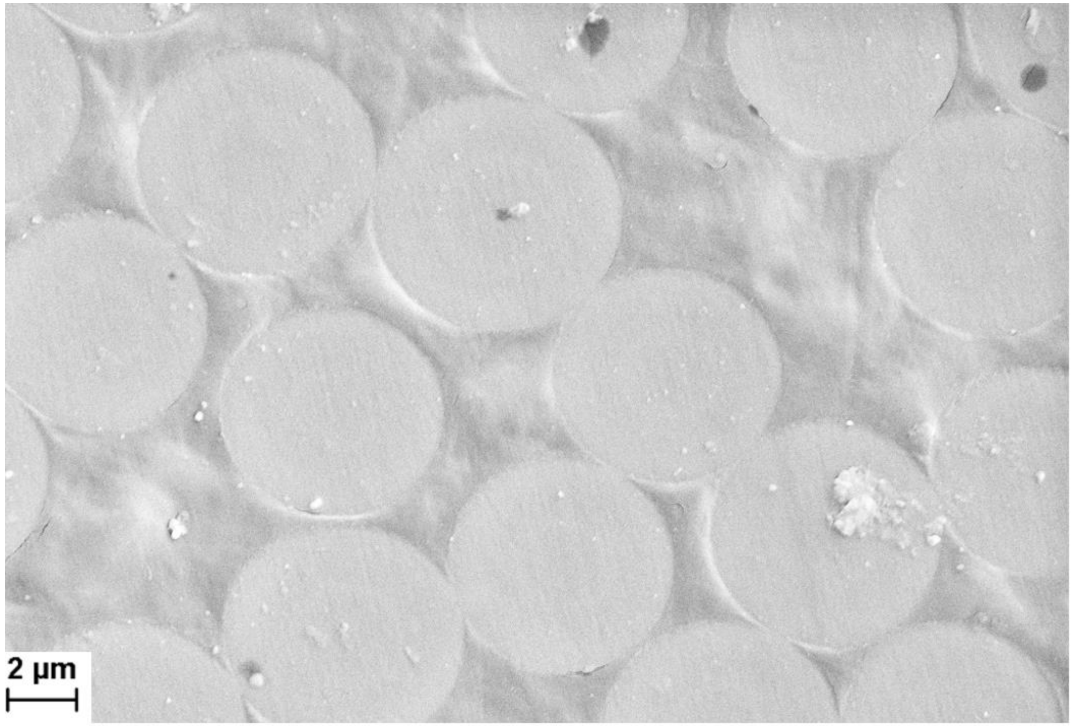




IMPACT OF ELECTRON BEAM ON THE MATRIX - SEM

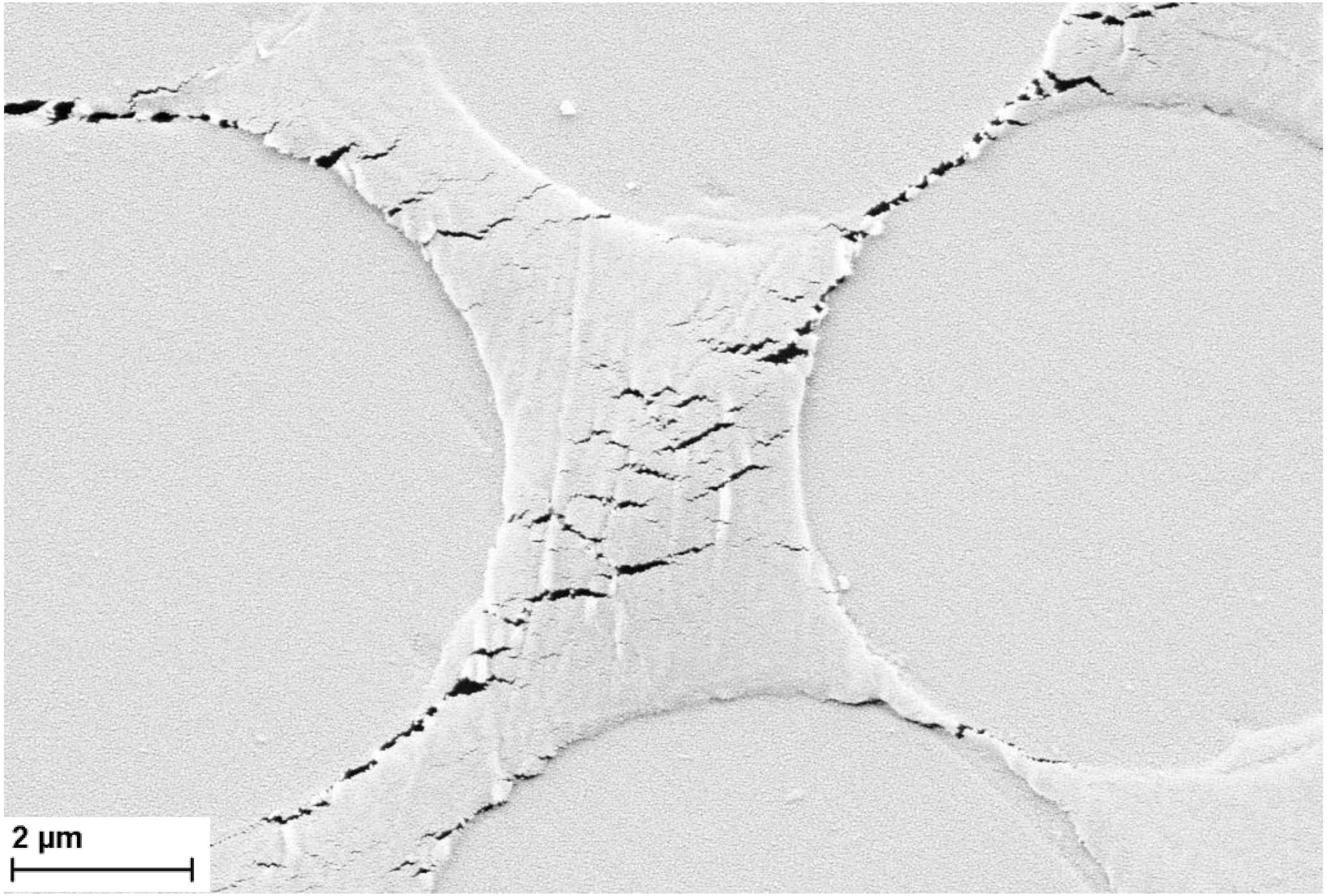


e- BEAM

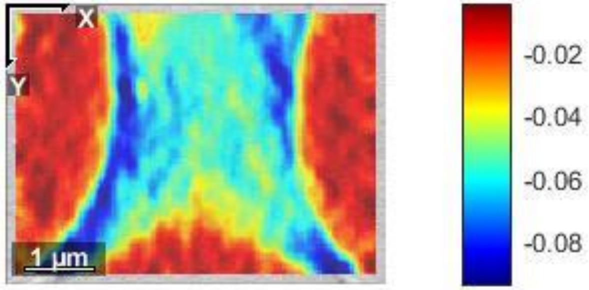


SEM + DIC (TRANSVERSE COMPRESSION TEST)

4 kV, SE2, Pt coating



SEM + DIC (TRANSVERSE COMPRESSION TEST) Ncorr



Type: exx-plot

Reference Name: Sample01StrainAnalysis090322_01.jpg

Current Name: Sample01StrainAnalysis090322_02.jpg

Analysis type: regular

RG-DIC Radius: 12 | Strain Radius: 5 | Subset Spacing: 5

Diffnorm Cutoff: 1e-06 | Iteration Cutoff: 50 | Threads: 4

Step Analysis: Disabled

RG-DIC Subset Truncation: Enabled | Strain Subset Truncatic

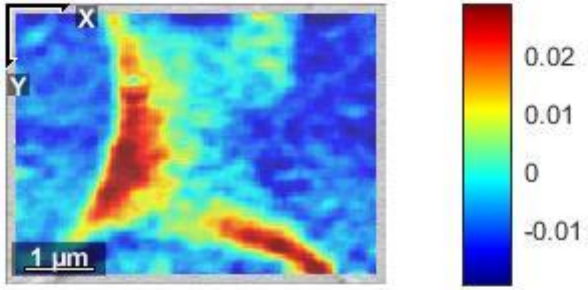
Image Correspondences: [0 1]

Units/pixels: 0.0059069 µm/pixels

Correlation Coefficient Cutoff: 1.5168

Radial Lens Distortion Coefficient: 0

Max: 0.0050 | Median: -0.0410 | Min: -0.1144



Type: exy-plot

Reference Name: Sample01StrainAnalysis090322_01.jpg

Current Name: Sample01StrainAnalysis090322_02.jpg

Analysis type: regular

RG-DIC Radius: 12 | Strain Radius: 5 | Subset Spacing: 5

Diffnorm Cutoff: 1e-06 | Iteration Cutoff: 50 | Threads: 4

Step Analysis: Disabled

RG-DIC Subset Truncation: Enabled | Strain Subset Truncatic

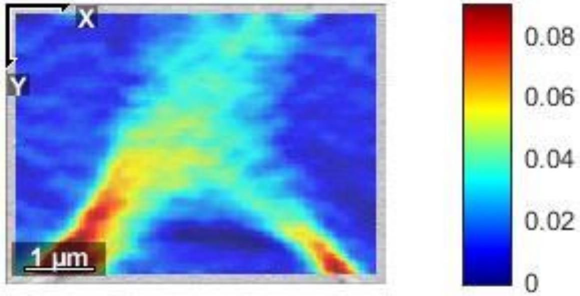
Image Correspondences: [0 1]

Units/pixels: 0.0059069 µm/pixels

Correlation Coefficient Cutoff: 1.5168

Radial Lens Distortion Coefficient: 0

Max: 0.0355 | Median: -0.0054 | Min: -0.0349



Type: eyy-plot

Reference Name: Sample01StrainAnalysis090322_01.jpg

Current Name: Sample01StrainAnalysis090322_02.jpg

Analysis type: regular

RG-DIC Radius: 12 | Strain Radius: 5 | Subset Spacing: 5

Diffnorm Cutoff: 1e-06 | Iteration Cutoff: 50 | Threads: 4

Step Analysis: Disabled

RG-DIC Subset Truncation: Enabled | Strain Subset Truncatic

Image Correspondences: [0 1]

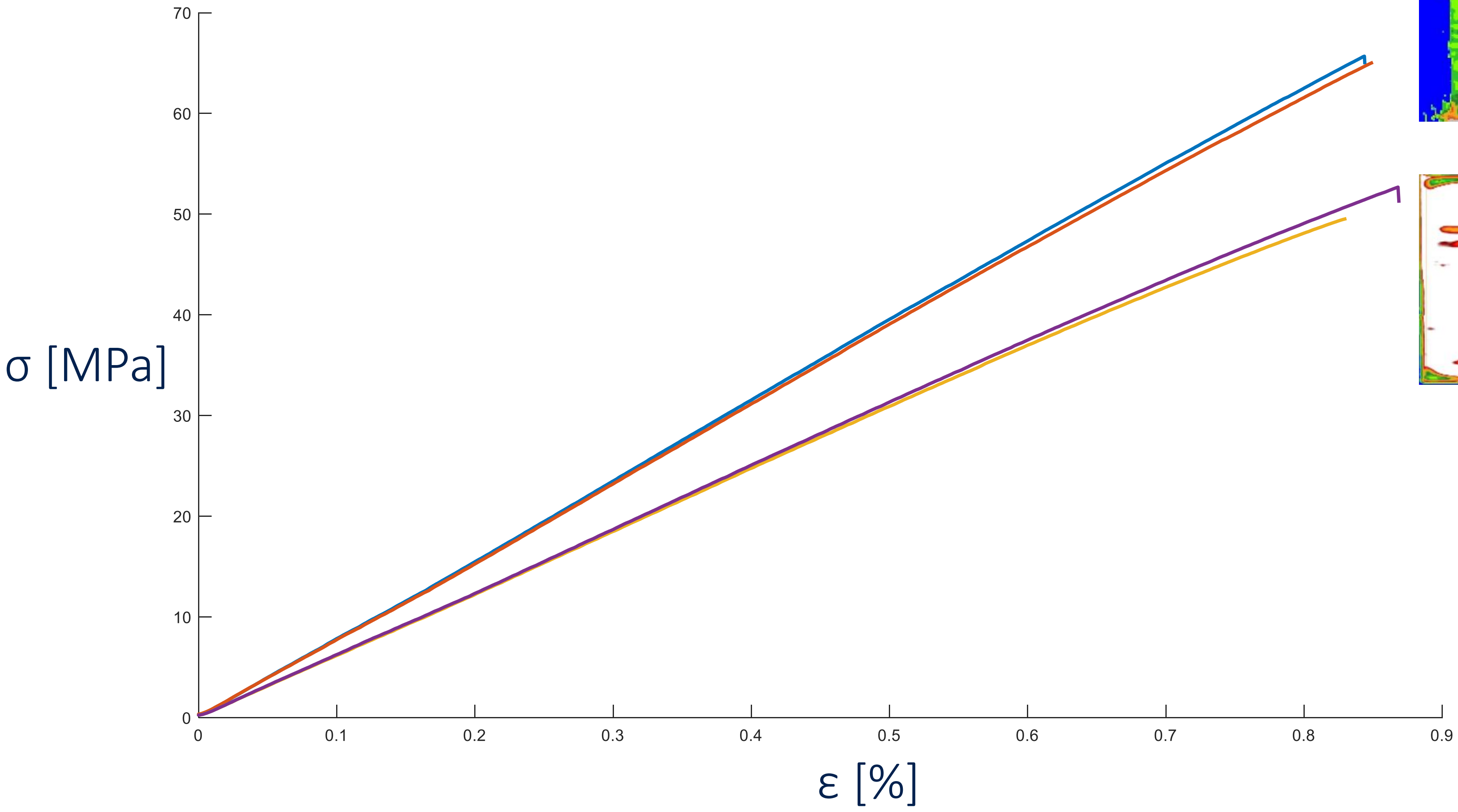
Units/pixels: 0.0059069 µm/pixels

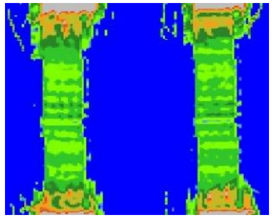
Correlation Coefficient Cutoff: 1.5168

Radial Lens Distortion Coefficient: 0

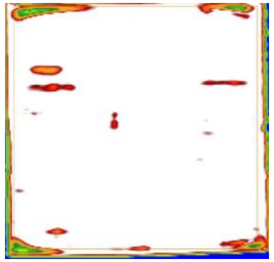
Max: 0.1171 | Median: 0.0181 | Min: -0.0121

IMPACT OF PROCESSING CONDITIONS - TRANSVERSE TENSILE TESTS





‘Standard conditions’



‘Amorphization’

IMPACT OF PROCESSING CONDITIONS - TRANSVERSE COMPRESSION TESTS