Politecnico di Torino
Dipartimento di Scienza Applicata e Tecnologia

DiSAT scienza e ingegneria

AIMAT 2021
XVI CONVEGNO NAZIONALE
CAGLIARI

OPTIMIZATION OF SELECTIVE LASER SINTERING PROCESS PARAMETERS FROM POWDER PROPERTIES: POLYAMIDE12/CARBON FIBER CASE STUDY

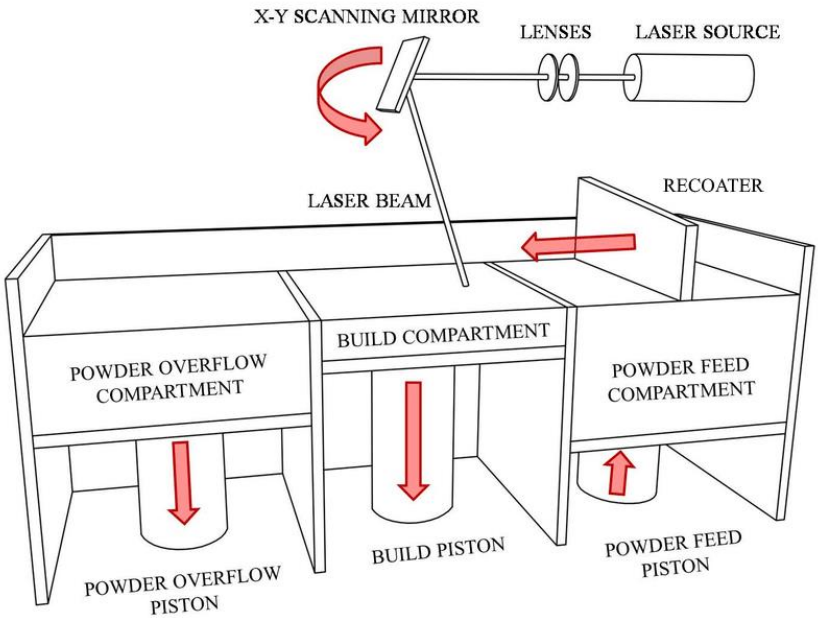
F. Lupone, E. Padovano, C. Badini*

Department of Applied Science and Technology – DISAT, Politecnico di Torino, Corso Duca degli Abruzzi 24, Torino

**federico.lupone@polito.it*

Selective Laser Sintering (SLS)

Laser powder bed fusion AM process for polymer and polymer based composites

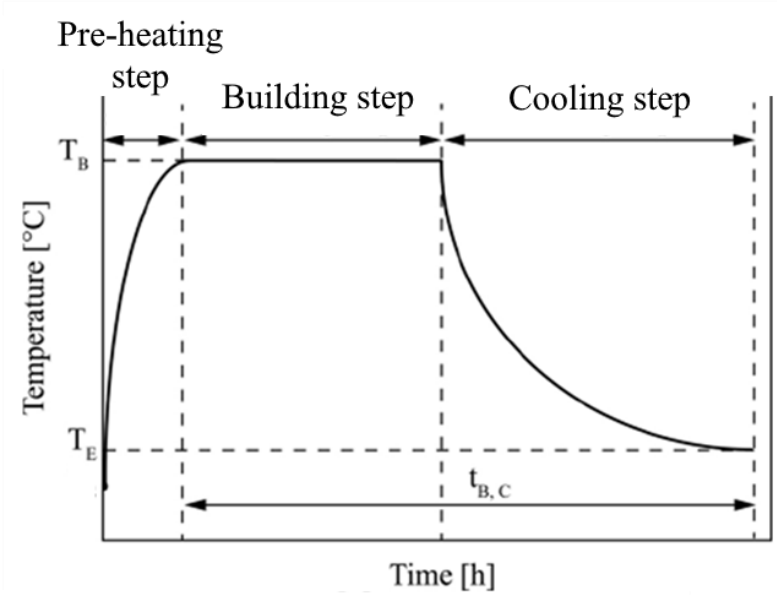


CO_2 laser exposure

Parts with complex geometry without support structures

Introduction & Motivation

Temperature profile of SLS process



Optimization of powder bed temperature (T_b) and laser parameters using a **trial and error approach**

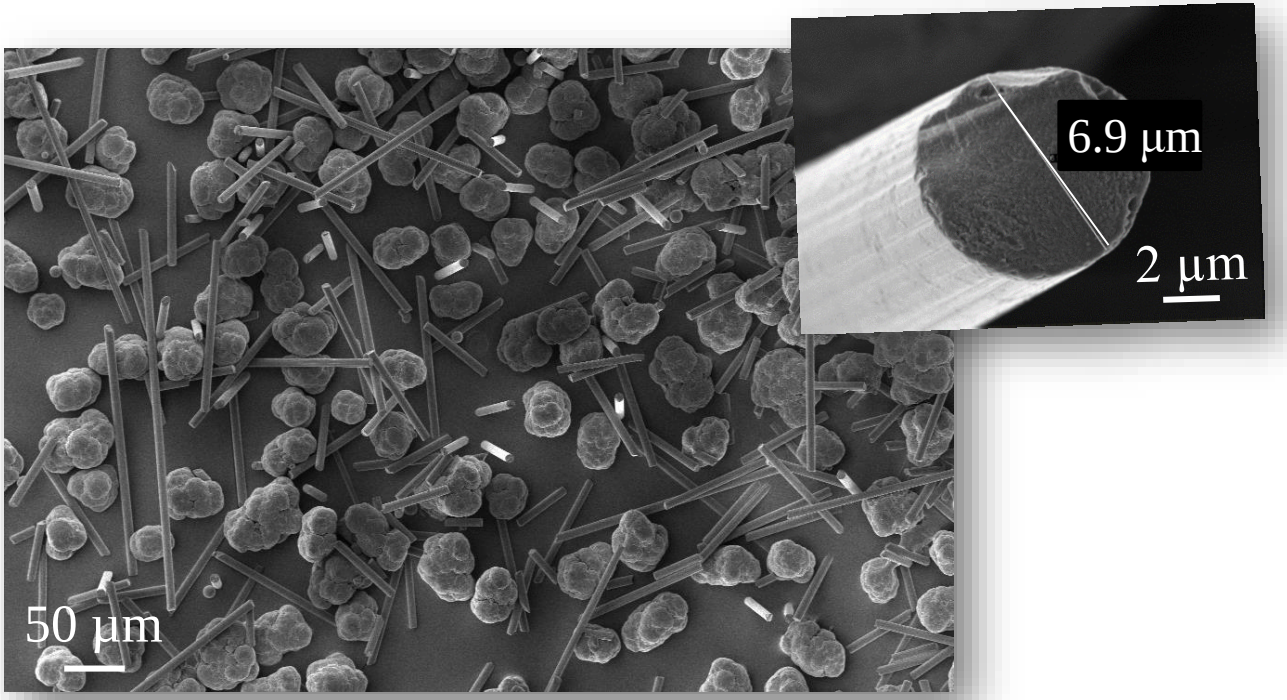
Time consuming and high cost

Do not consider material properties

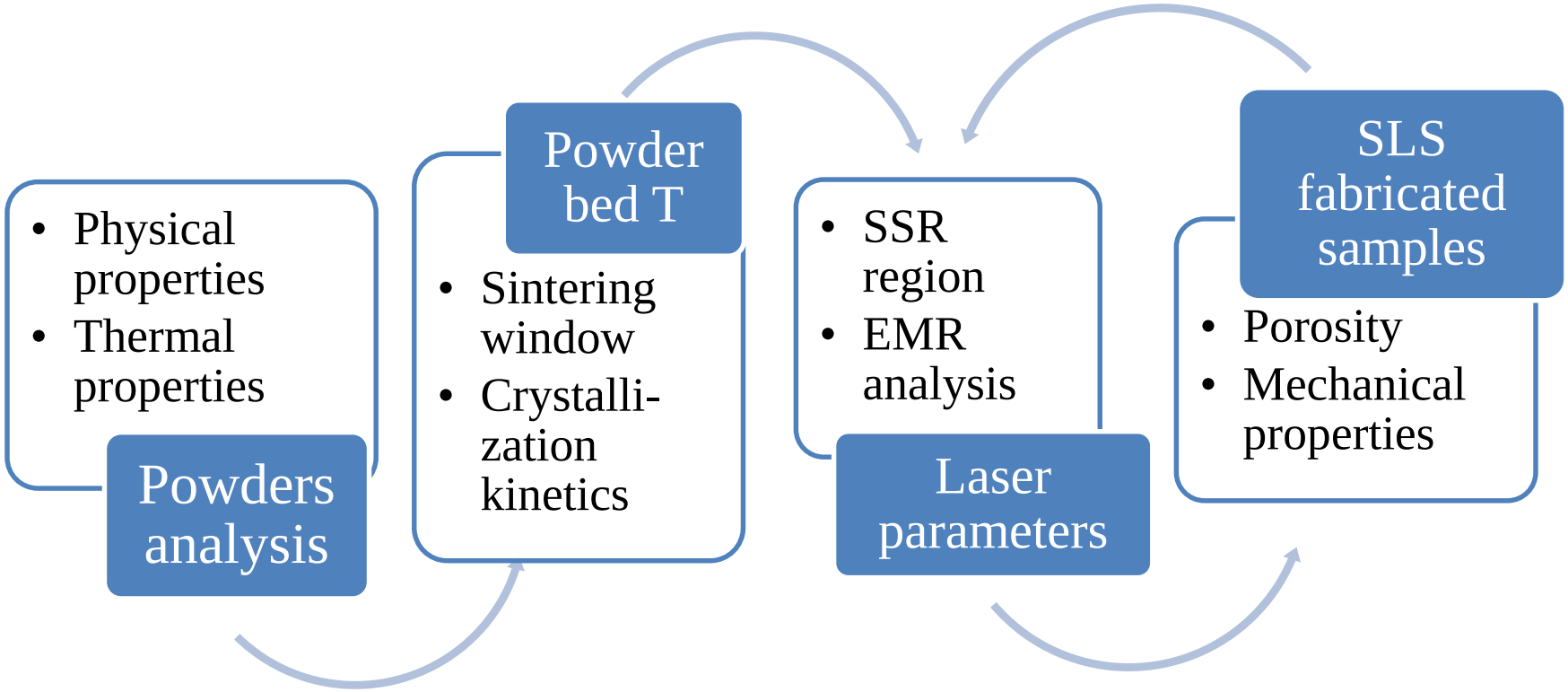
Materials & Methods

Powder-based characterization approach to optimize SLS process parameters

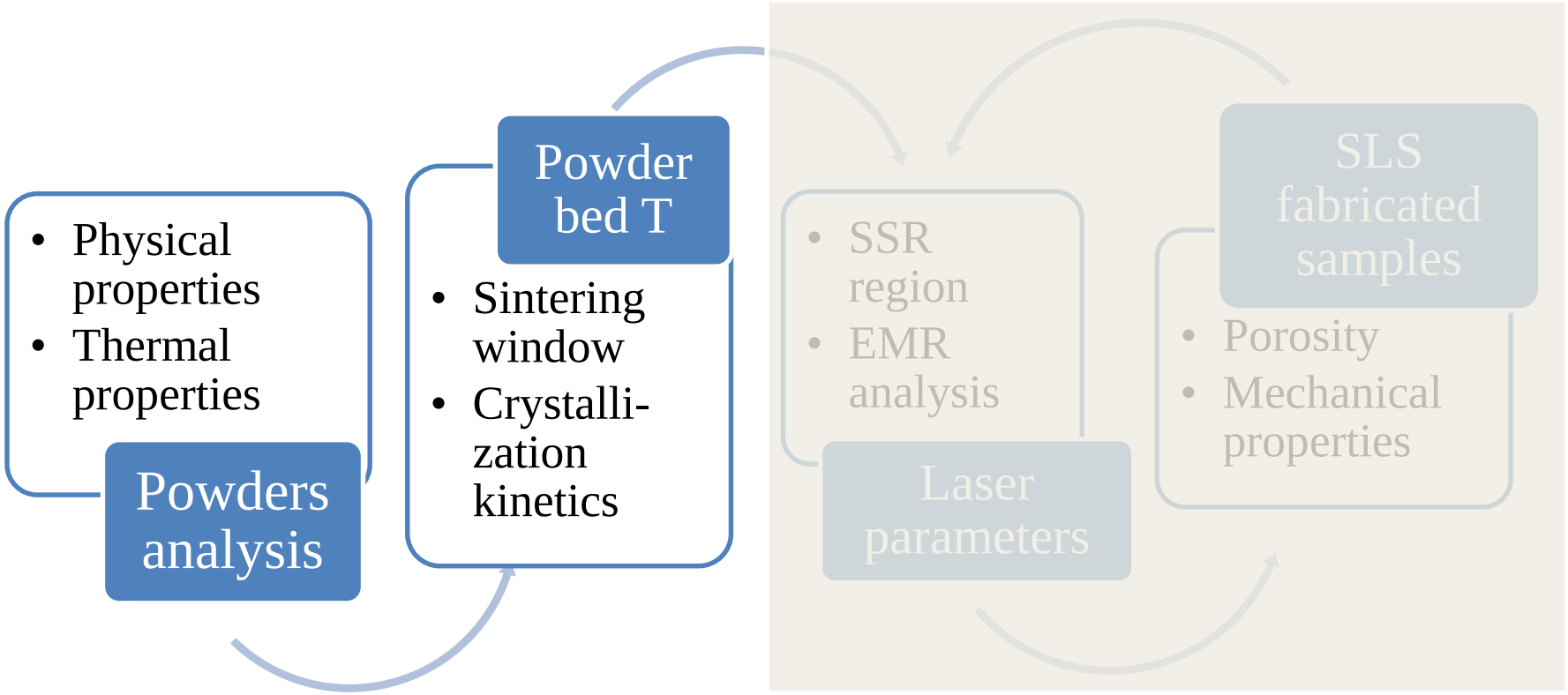
PA12/CF composites (20 wt% short CF)



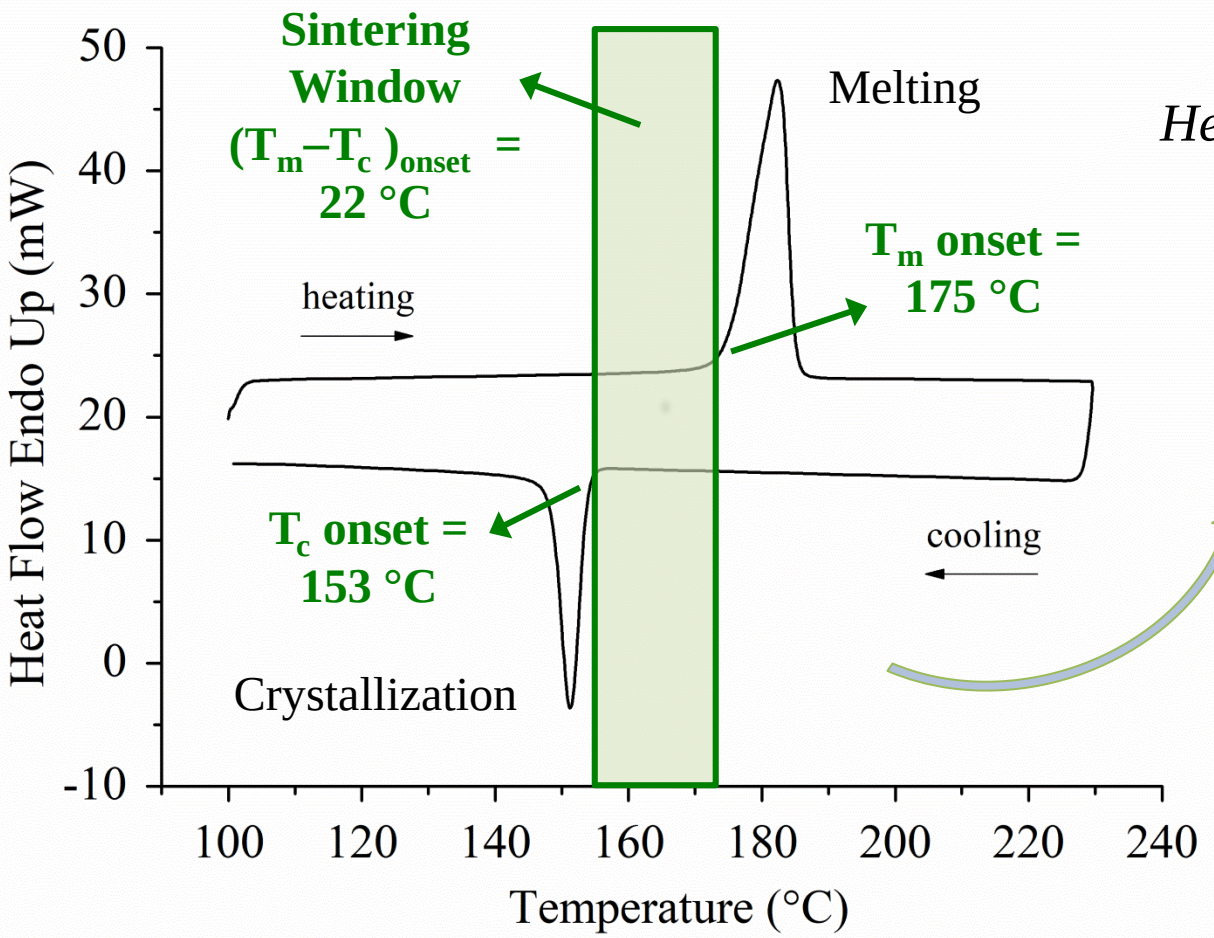
Materials & Methods



Materials & Methods



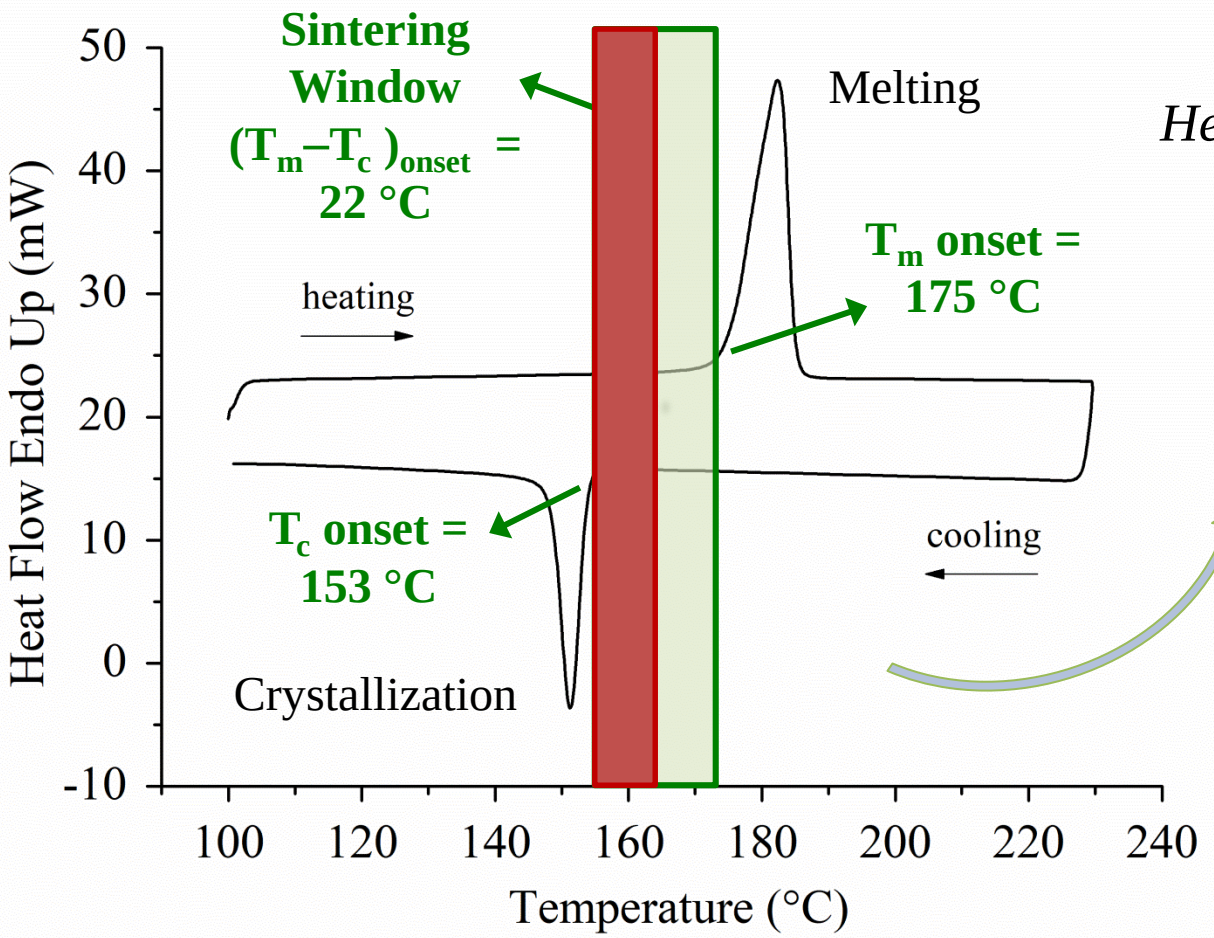
Powder bed temperature Differential Scanning Calorimetry



Heating / cooling rate 10 °C/min

T_b within the Sintering Window

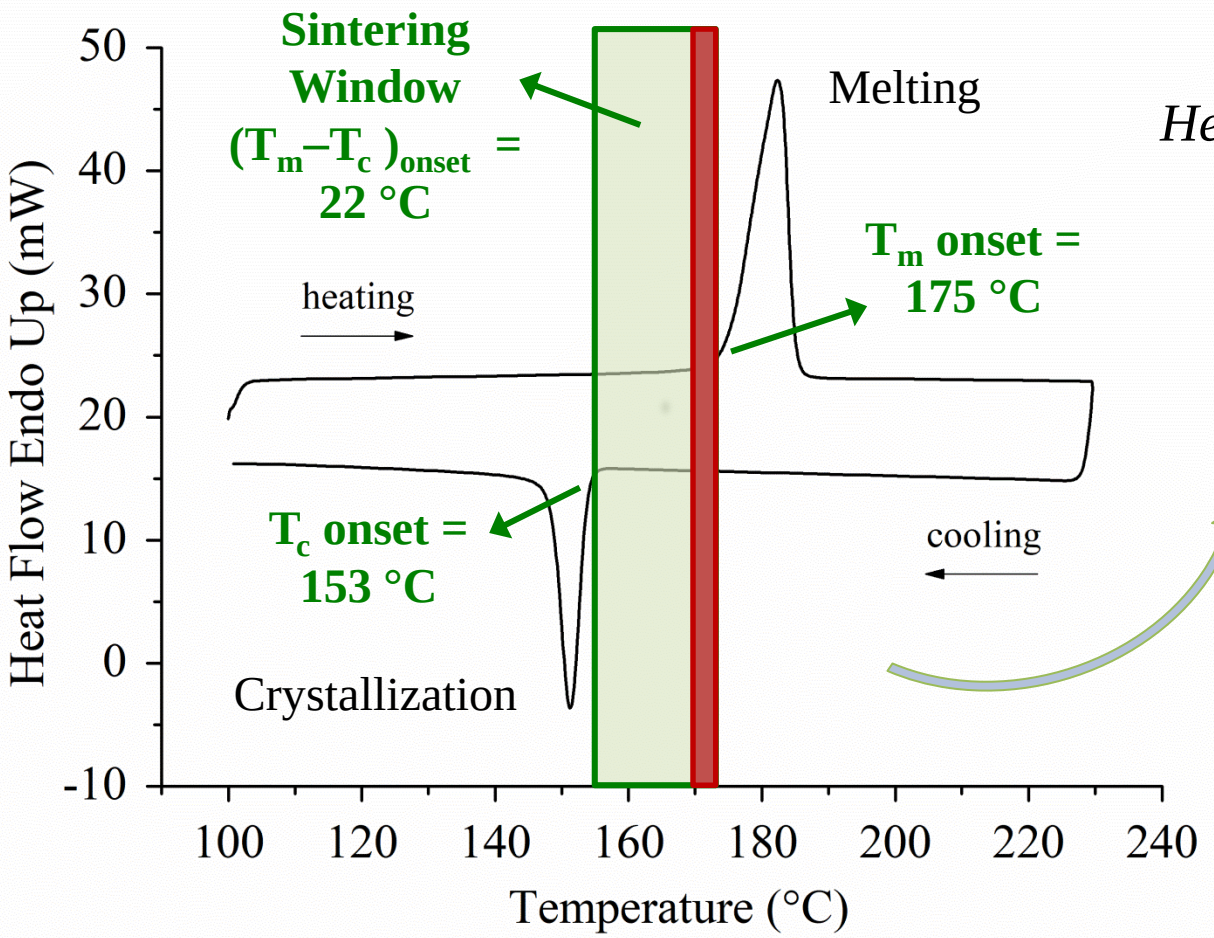
Powder bed temperature Differential Scanning Calorimetry



Heating / cooling rate 10 °C/min

T_b within the Sintering Window

Powder bed temperature Differential Scanning Calorimetry



Heating / cooling rate 10 °C/min

T_b within the Sintering Window

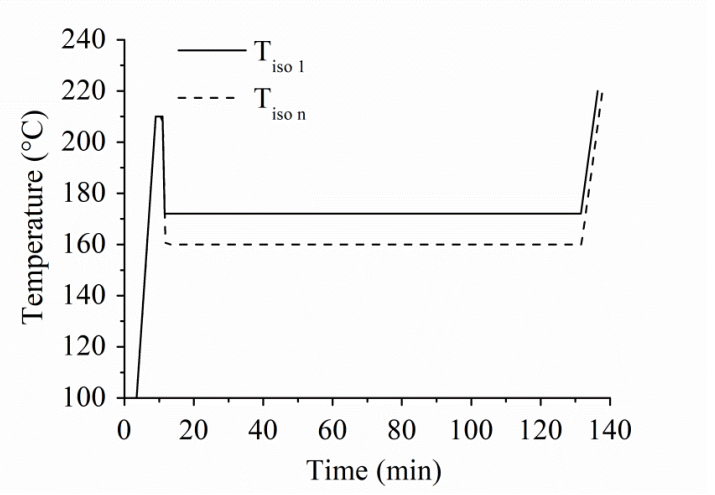
Quasi-isothermal laser sintering model

SLS as a quasi-isothermal process at the temperature of the powder bed

OPTIMIZATION STRATEGY

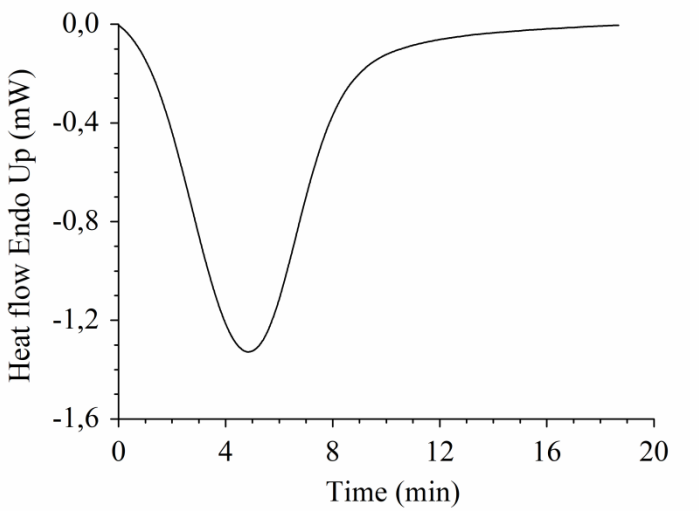
Powder	Part	T range
Isothermal crystallization kinetics (DSC)	Curl height (SLS)	160 ÷ 172 * * within SW

DSC program for isothermal crystallization tests



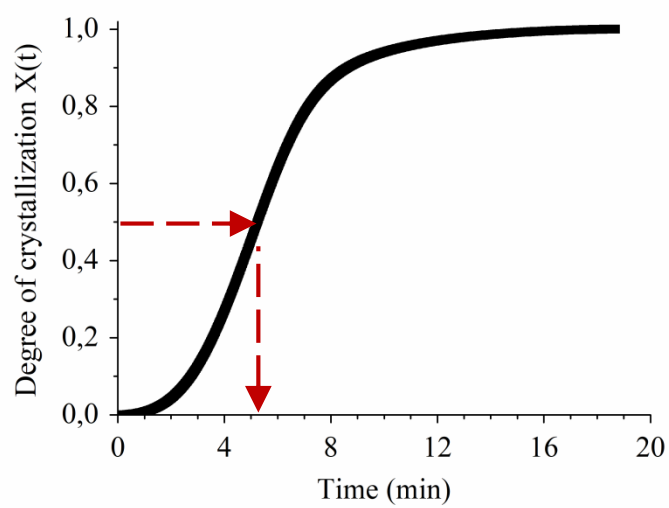
PA12/CF Crystallization kinetics

Isothermal DSC curve



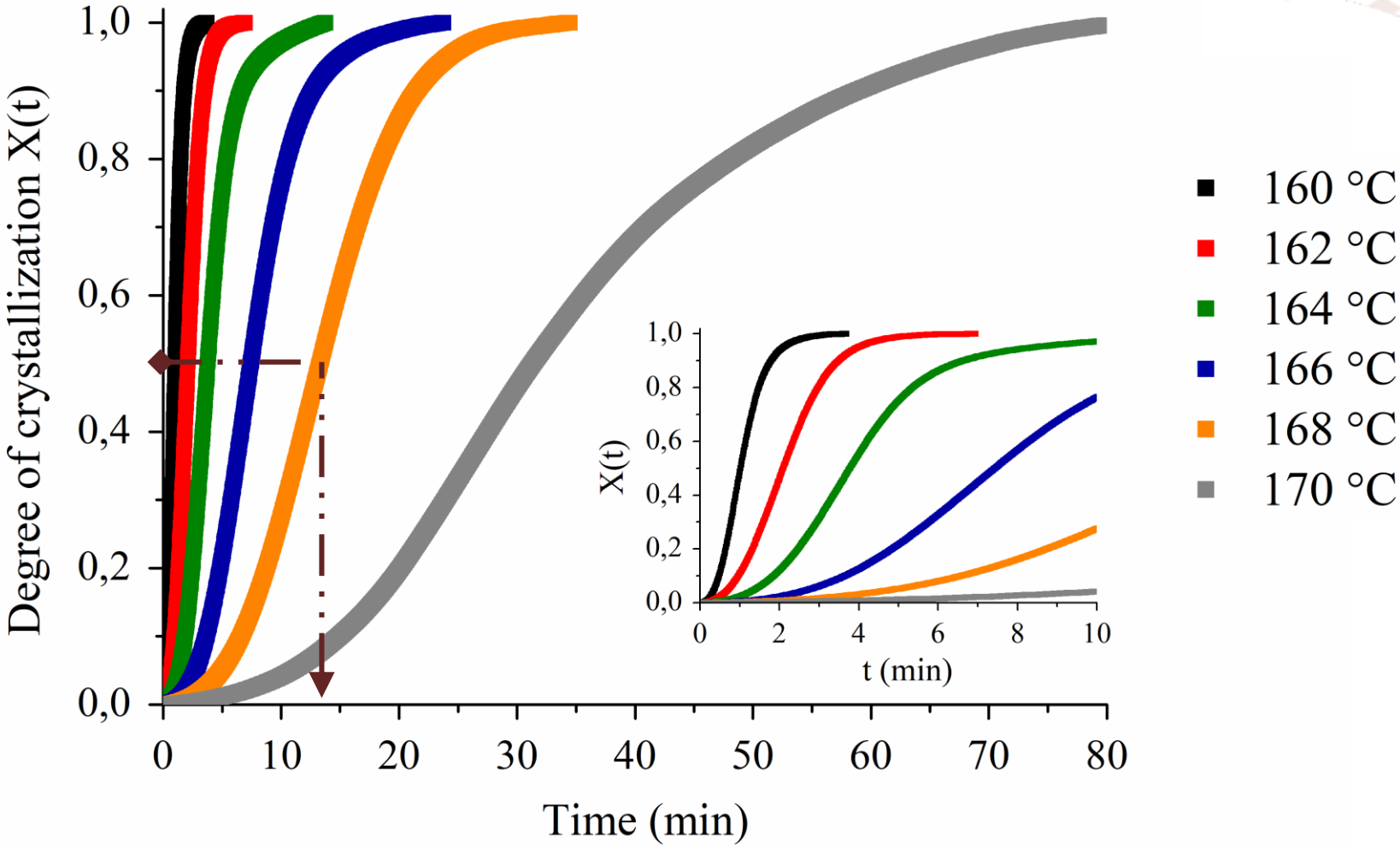
Evolution of the degree of crystallization

$$X(t) = \frac{\int_{t_0}^{t} \frac{dH_c(t)}{dt} dt}{\int_{t_0}^{t_\infty} \frac{dH_c(t)}{dt} dt}$$



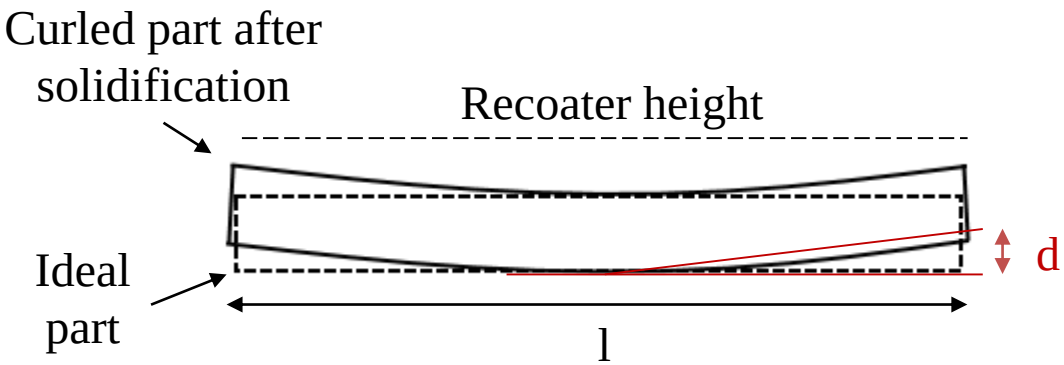
Crystallization halftime $t_{1/2}$ = $t_{50\%}$ conversion

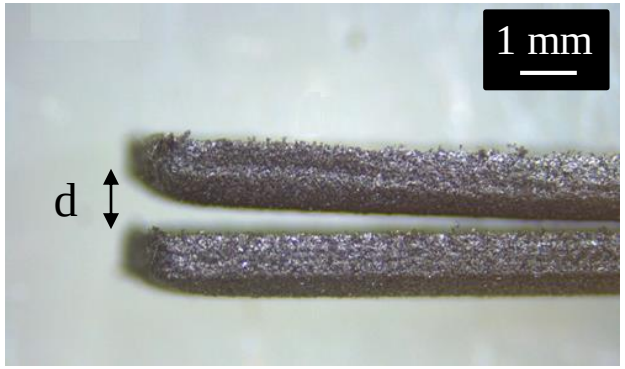
PA12/CF Crystallization kinetics



PA12/CF Curling evaluation

Side view of the part





Curling measurements

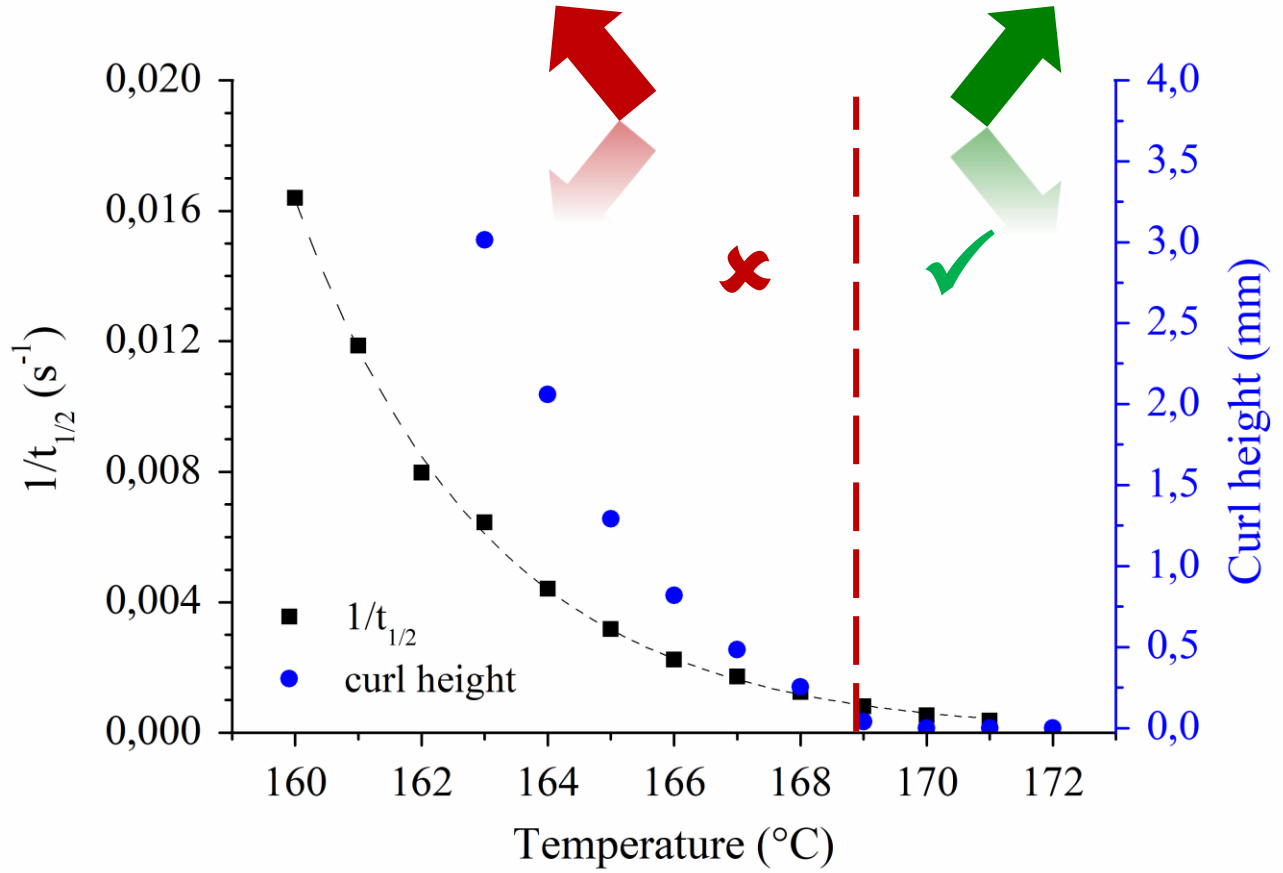
T_{iso} (°C)	d (mm)
170	0
168	0.25
167	0.48
166	0.82
165	1.29
164	2.06
163	3.02
162	failed *
160	failed *

* collision between recoater and part

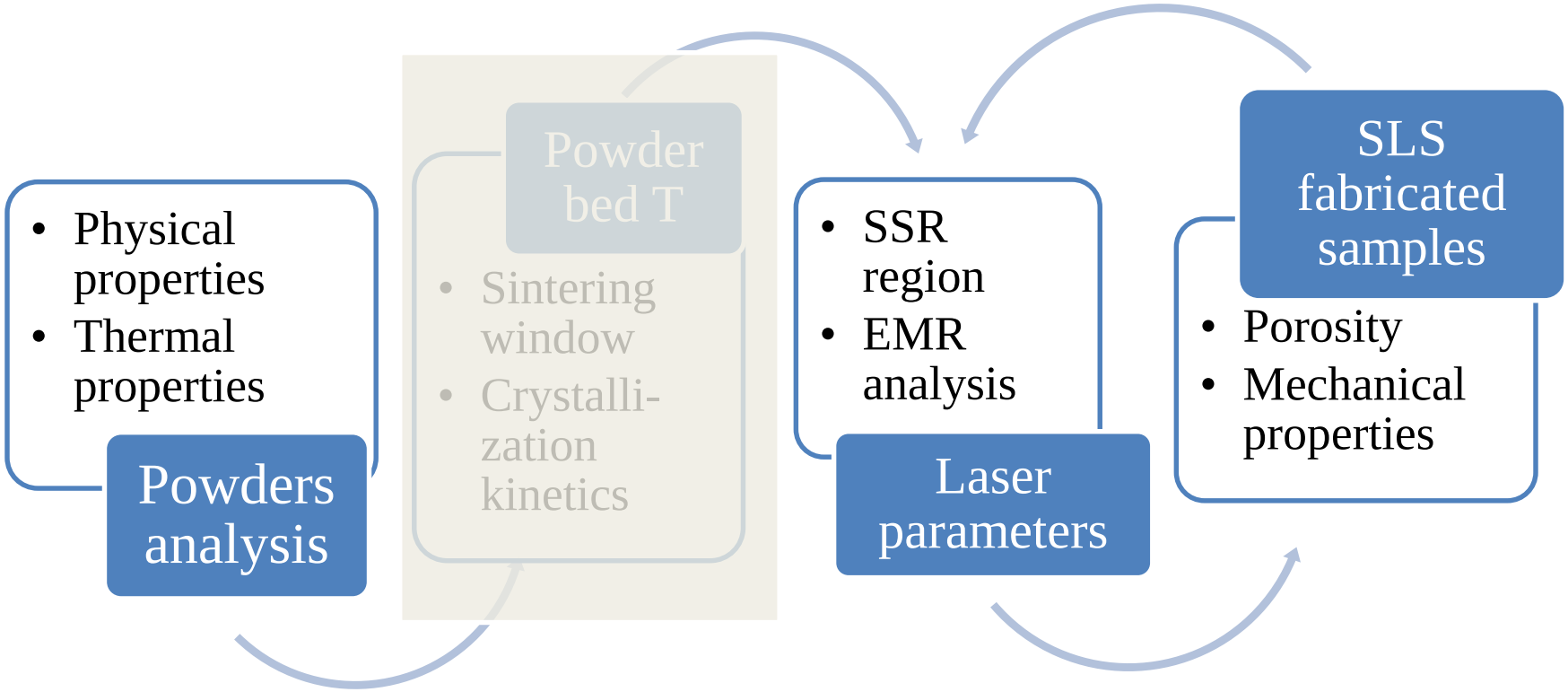
PA12/CF Crystallization vs curling

Part distortion due to early crystallization

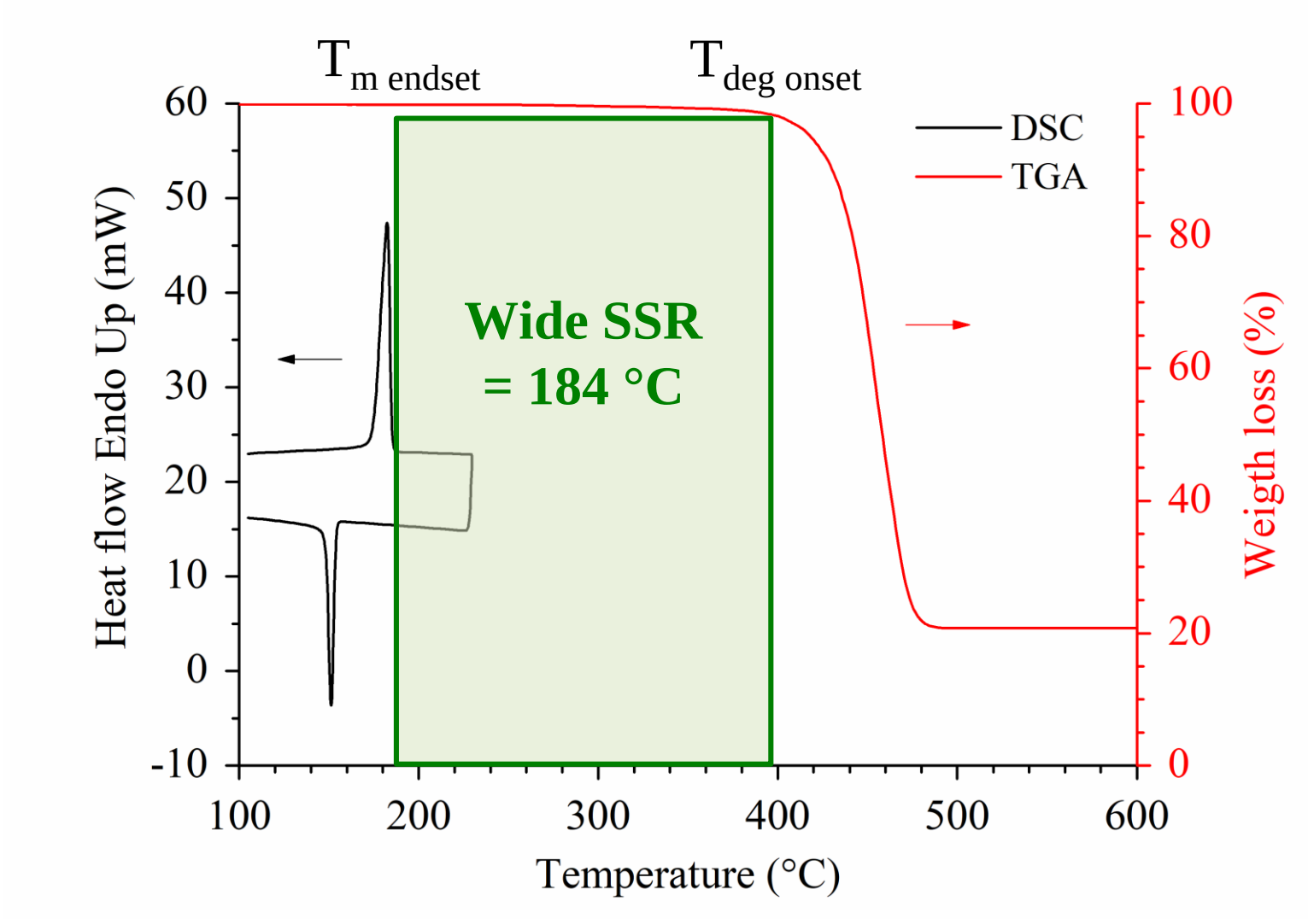
No curling for $T_b > 169$ °C



Materials & Methods



PA12/CF Stable Sintering Region (SSR)



PA12/CF SSR approach

Energy to melt a layer (J/mm^3)

$$E_m = \left[C_p^{\,p} \left(T_m - T_b \right) + h_f \right] \rho \Phi$$

Energy for degradation (J/mm^3)

$$E_{deg} = \left[C_p^{\,m} \left(T_{degonset} - T_m \right) \right] \rho$$

Parameters for E_m and E_{deg}	Value	Technique
Powder bed temperature T_b (°C)	170	
Melting temperature T_m (°C)	182.3	DSC
Powder specific heat $C_p^{\,p}$ (J/g °K)	1.99	DSC
Melting enthalpy h_f (J/g)	81.4	DSC
Packing factor Φ	0.38	tap density
Density ρ (g/cm^3)	1.15	pycnometry
Onset degradation $T_{deg\ onset}$ (°C)	369	TGA
Melt specific heat $C_p^{\,m}$ (J/g °K)	1.81	DSC
Activation energy E_A (J/g)	361.1	TGA
Molecular weight M_W (g/mol)	18080 [1]	GPC

[1] Dupin, S. et al. Microstructural origin of physical and mechanical properties of polyamide 12 processed by laser sintering. *Eur. Polym. J.* **2012**, *48*, 1611–1621.

PA12/CF SSR approach

Laser energy density per unit volume (J/mm^3)

$$E_V = \frac{P}{v \cdot H \cdot d_{layer}}$$

Energy to melt ratio (EMR)

$$EMR = \frac{E_v}{E_m}$$

PROCESSING WINDOW

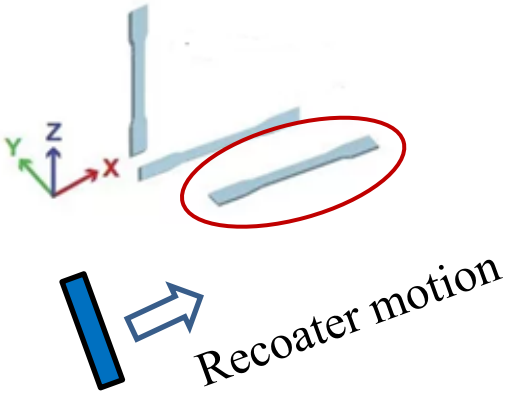
E_m (J/mm^3)	E_{deg} (J/mm^3)	EMR
0,046	0,416	1 ÷ 8.9

predict the proper process parameters that could induce **sufficient energy for melting** whilst **keeping the temperature within the stable sintering region**

PA12/CF SLS sample production

Validation of EMR approach

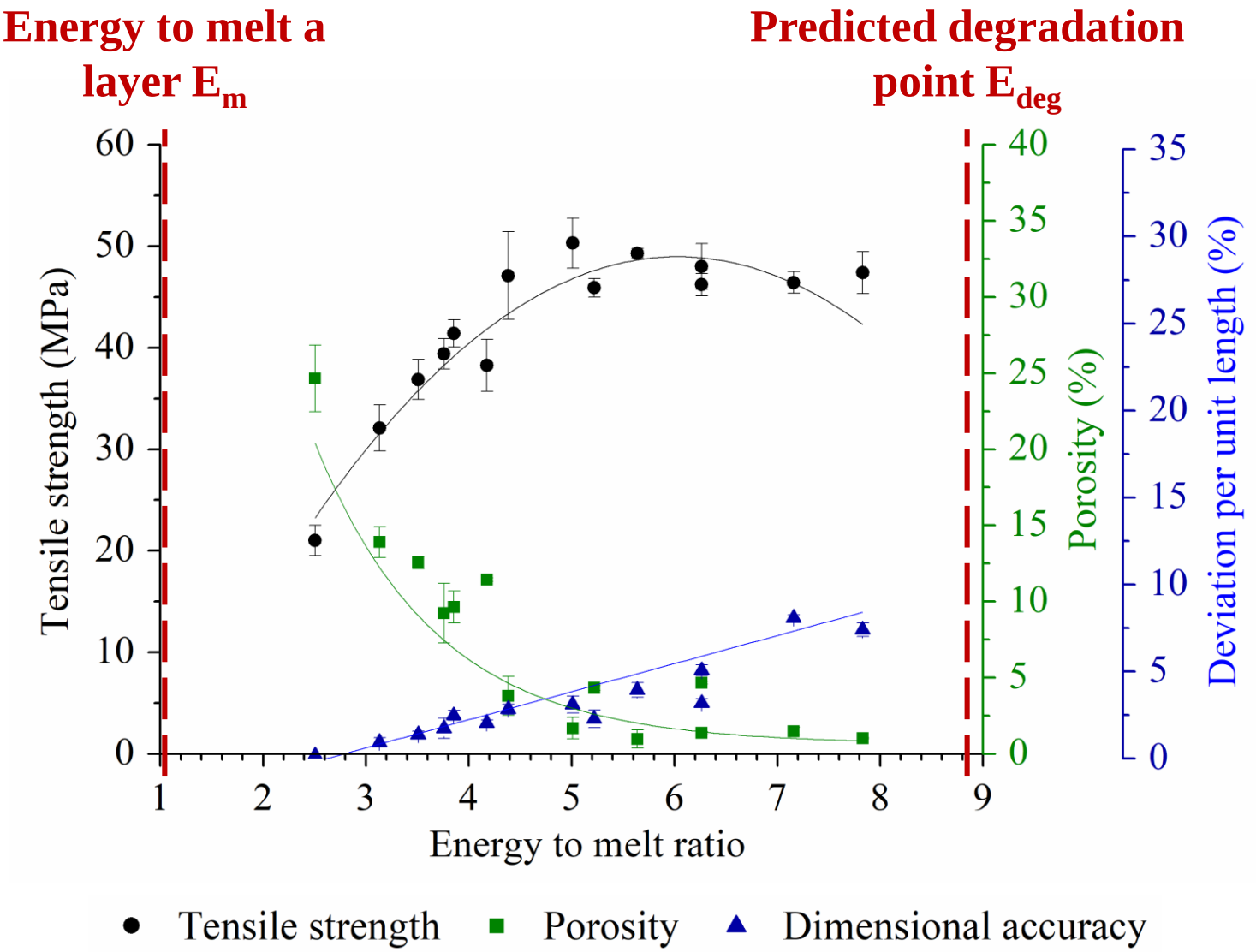
Samples built on xy plane in Sharebot SnowWhite printer



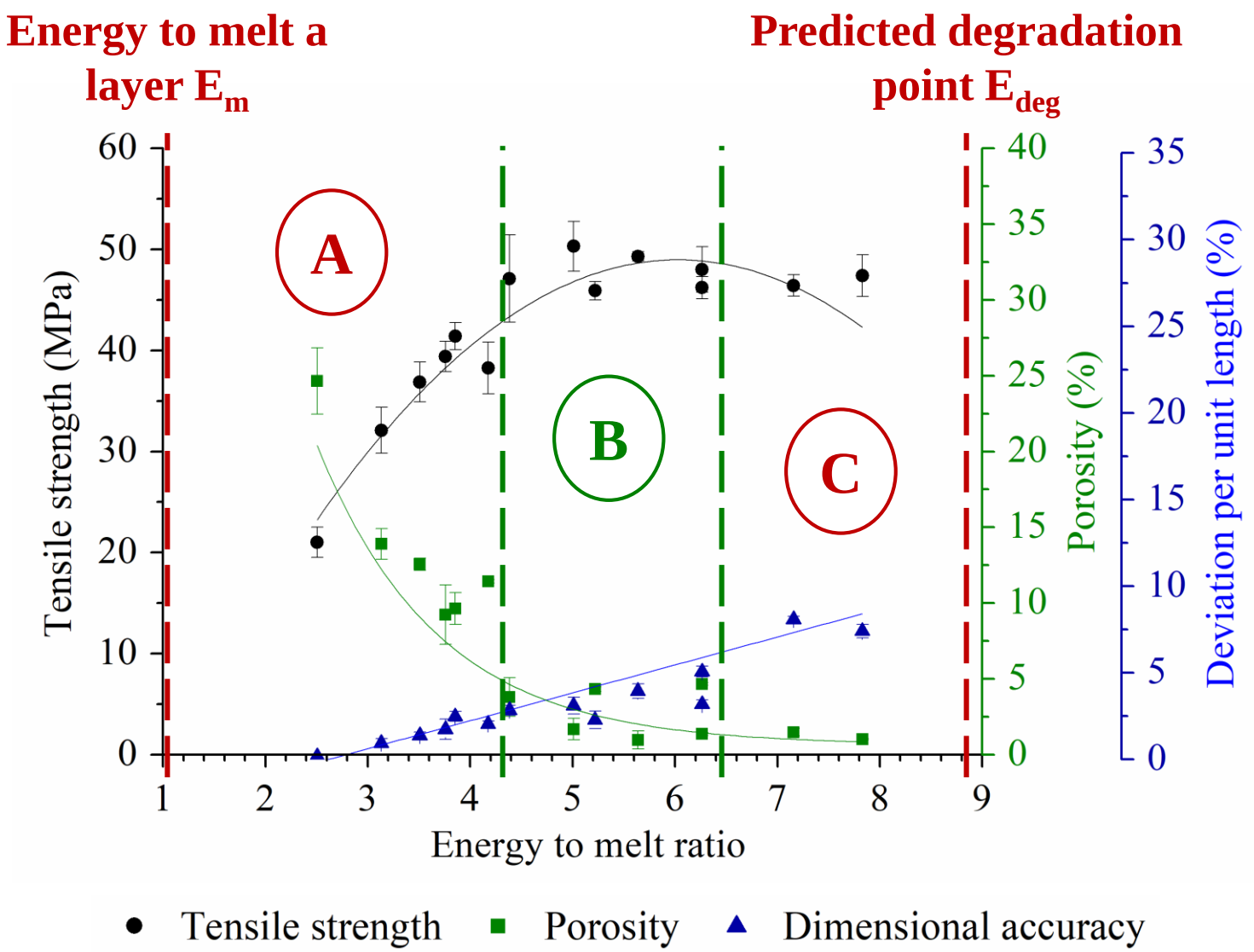
PROCESS PARAMETERS

Power (W)	Scan speed (mm/s)	Hatch spacing (µm)	Layer height (µm)	EMR
20 ÷ 50	960 ÷ 3100	100	100	2,5 ÷ 7,8

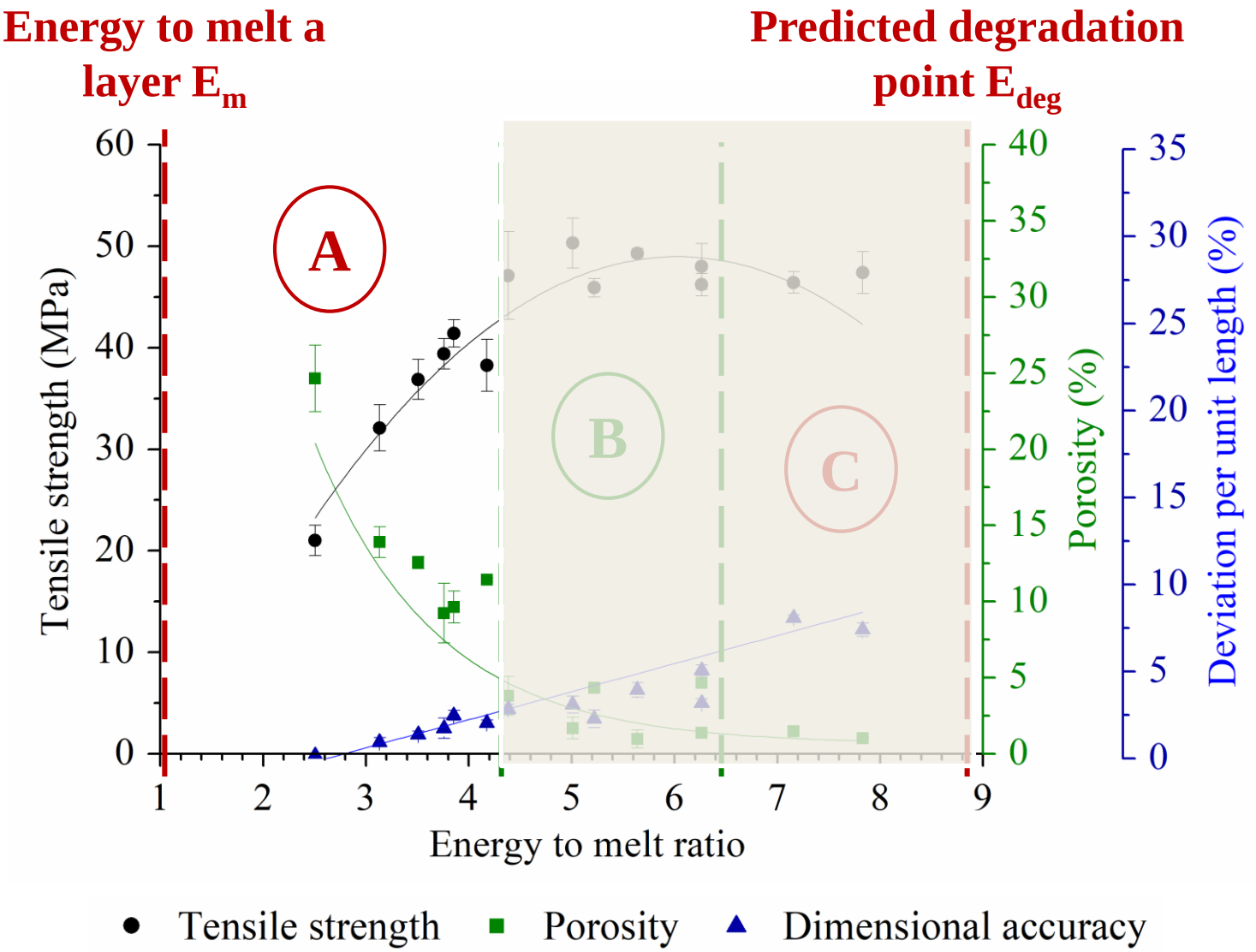
PA12/CF SSR analysis



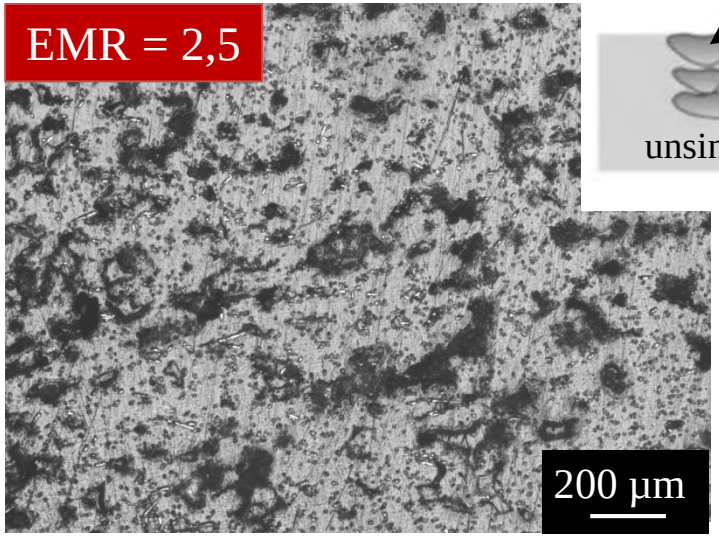
PA12/CF SSR analysis

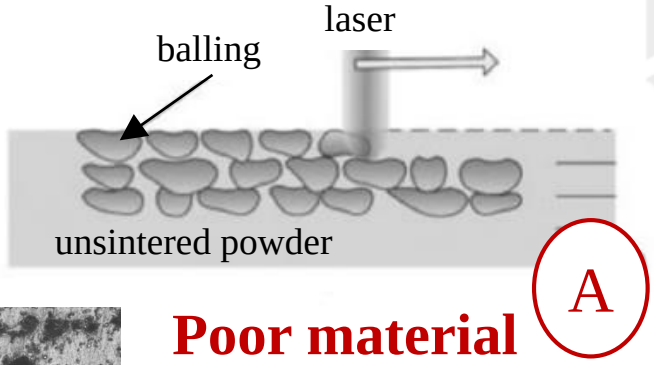


PA12/CF Region “A”



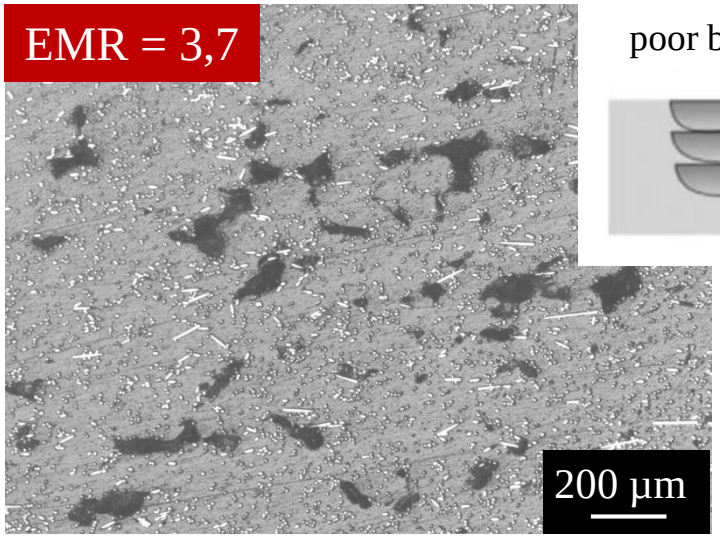
PA12/CF Region “A”

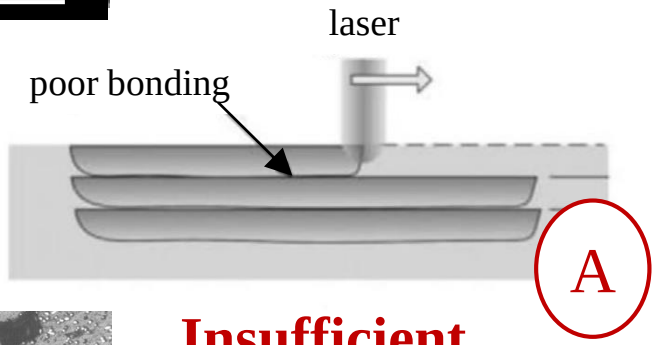




Poor material fusion

Porosity = 24,6%

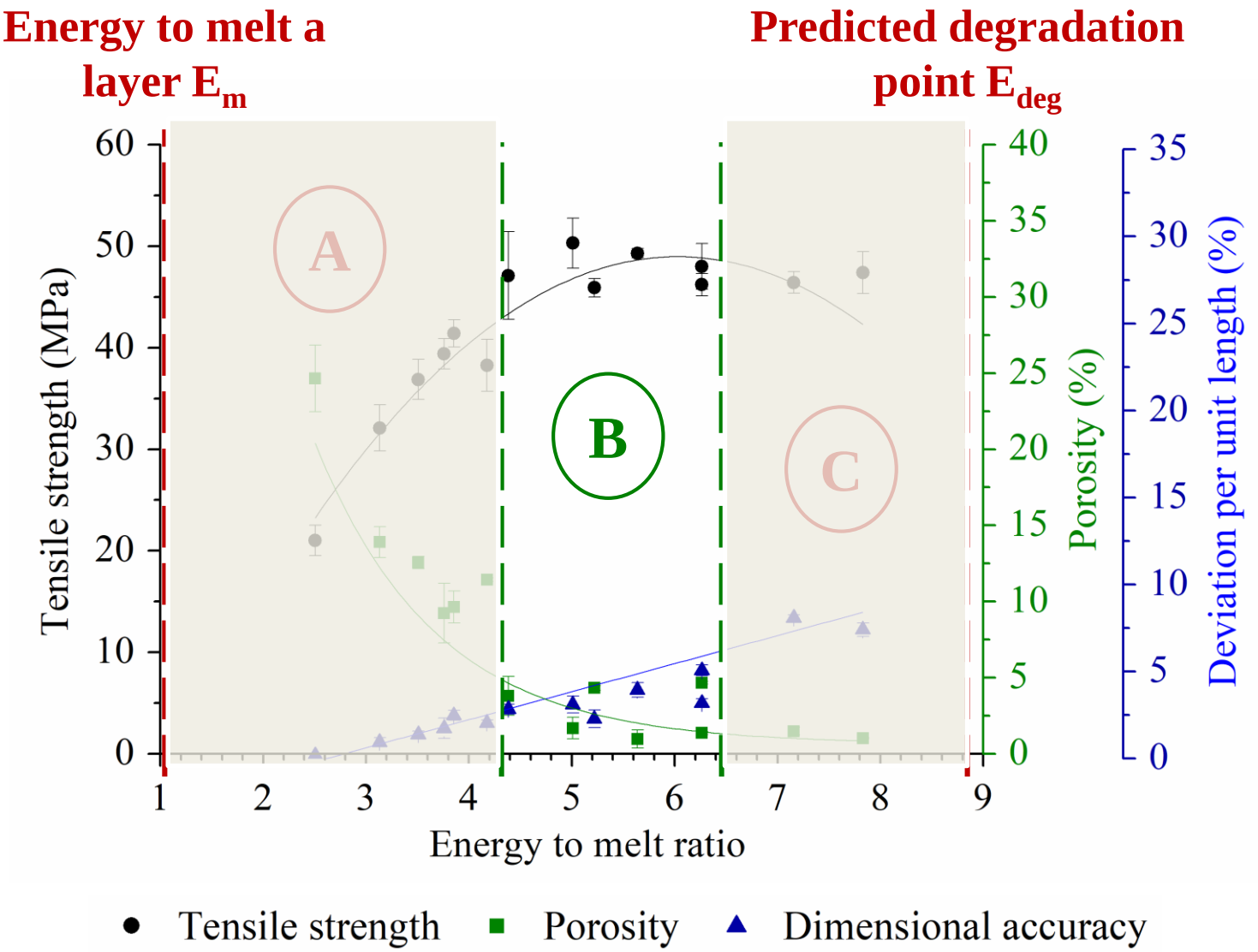




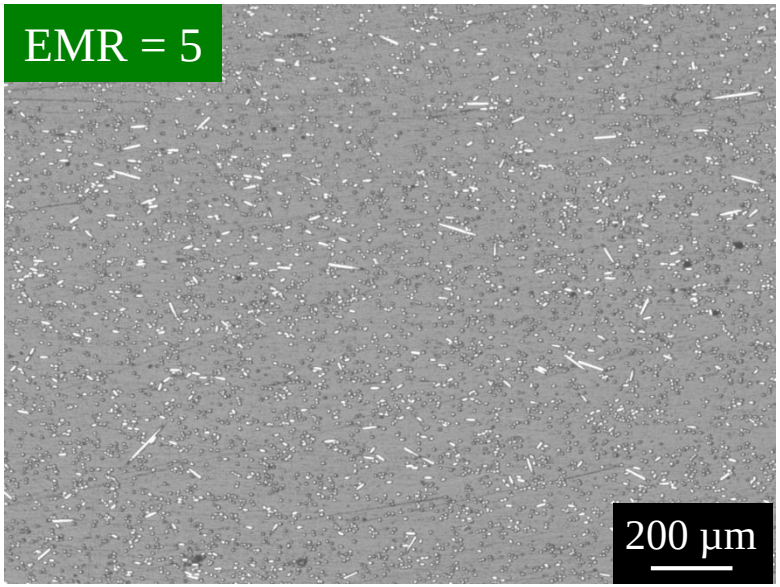
Insufficient melting

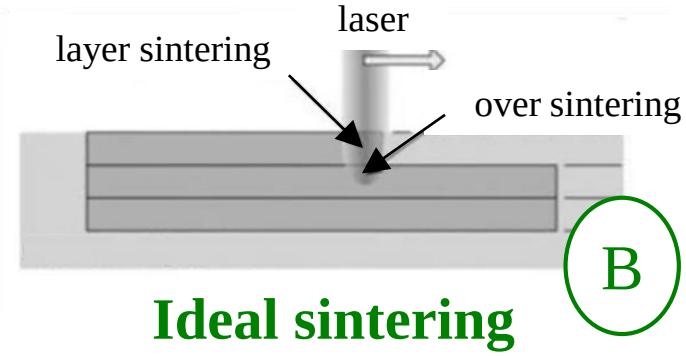
Porosity = 9,2%

PA12/CF Region “B”



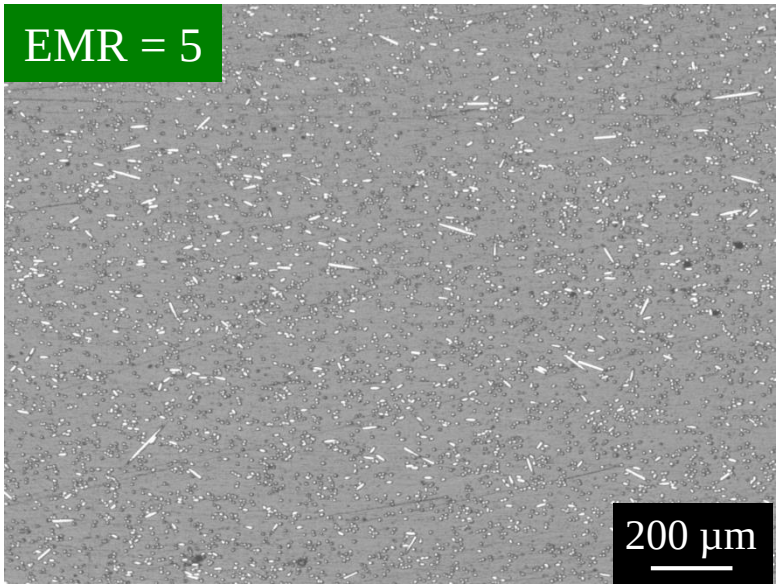
PA12/CF Region “B”

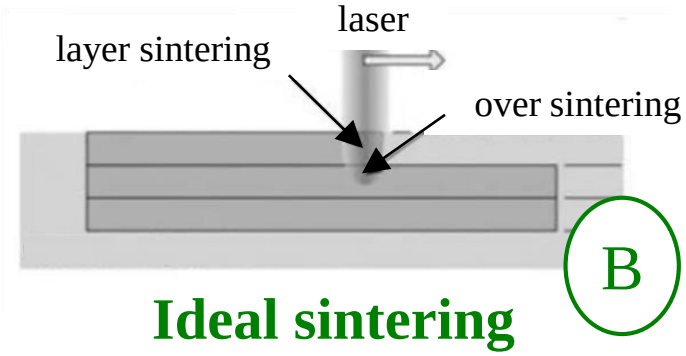




E (GPa)	σ (Mpa)	ε (%)	void (%)
3.2 ± 0.1	50.3 ± 2.5	4.9 ± 0.3	1

PA12/CF Region “B”





E_v is 4 – 6 times higher than E_m

?

E (GPa)	σ (Mpa)	ε (%)	void (%)
3.2 ± 0.1	50.3 ± 2.5	4.9 ± 0.3	1

PA12/CF Possible explanations

- Optical absorptivity of powders at CO_2 laser wavelength (10,6 µm) < 1

IR absorptance $_{PA\ 12}$ > 0,9 [2]

- Melt pool depth too low to effectively re-melt the previous layer

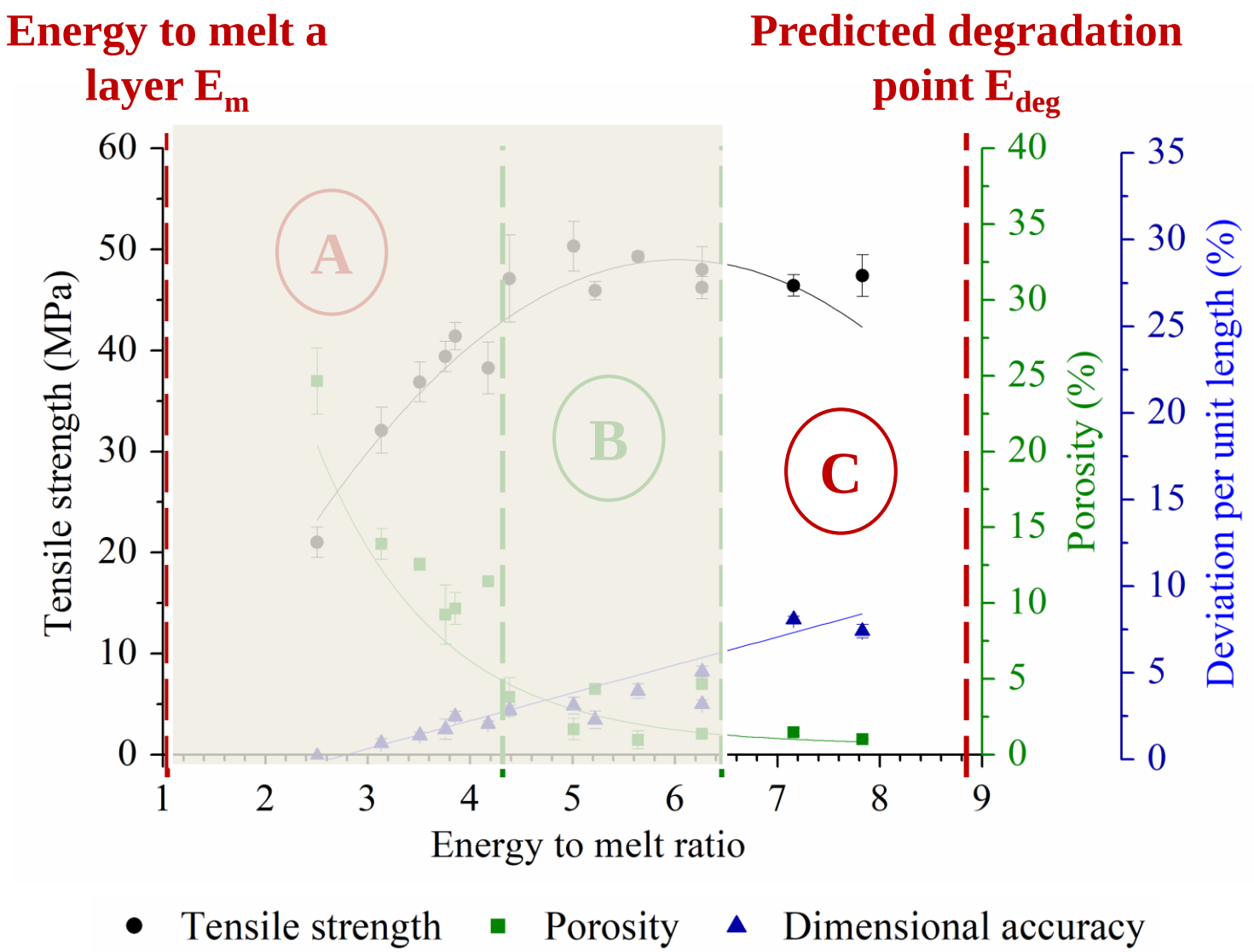
Laser attenuation coefficient

- Slow coalescence of polymer particles with respect to laser-powders interaction time (30-60 ms)

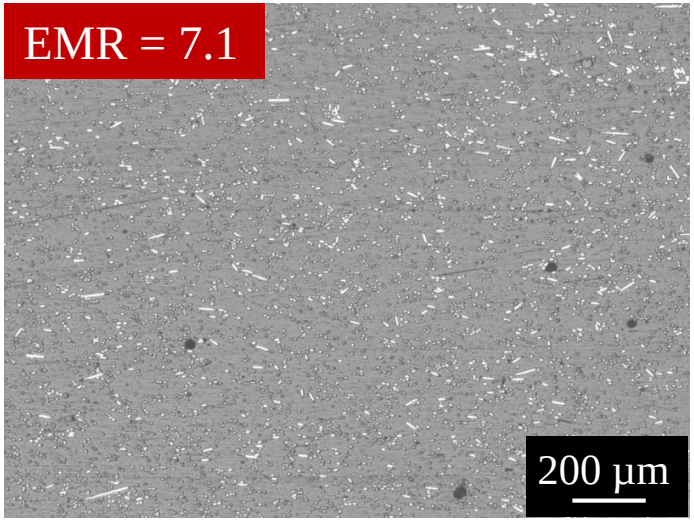
High temperature rheological properties

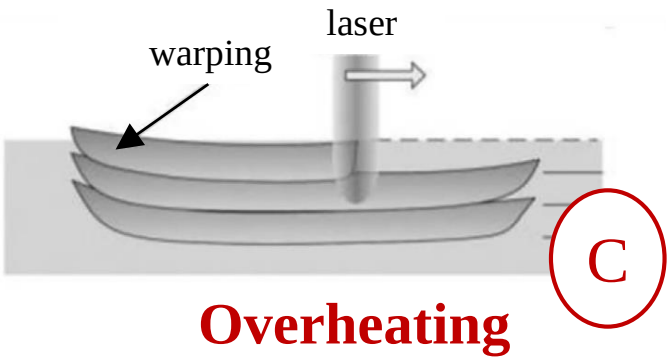
[2] Laumer, T. et al. Optical analysis of polymer powder materials for Selective Laser Sintering. *Polym. Test.* **2016**, *56*, 207–213.

PA12/CF Region "C"

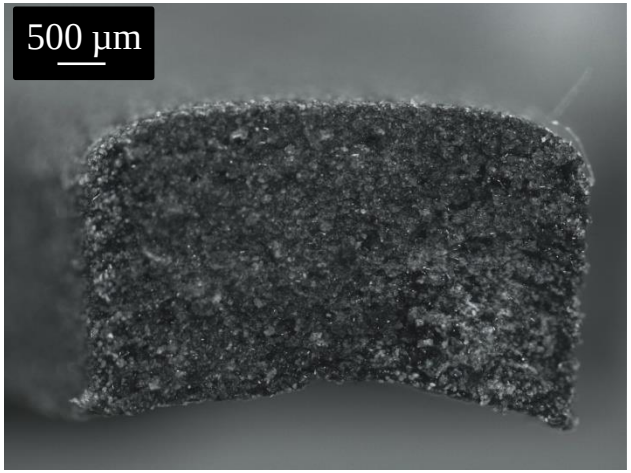


PA12/CF Region “C”

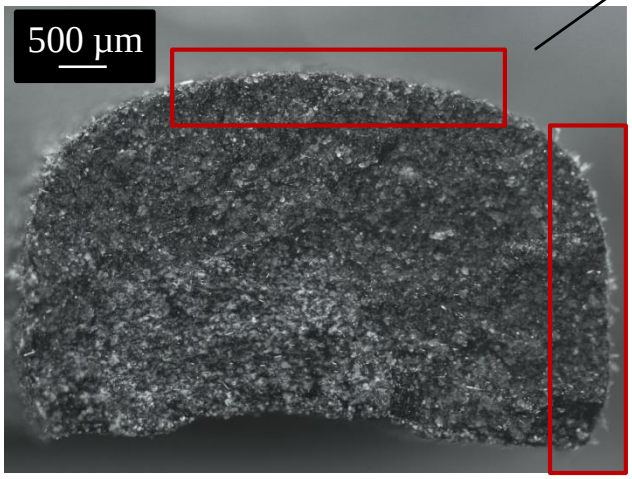




Region “B” - EMR=5



Region “C” - EMR=7.1



Warped surface

Rounded edged

Loss of dimensional accuracy

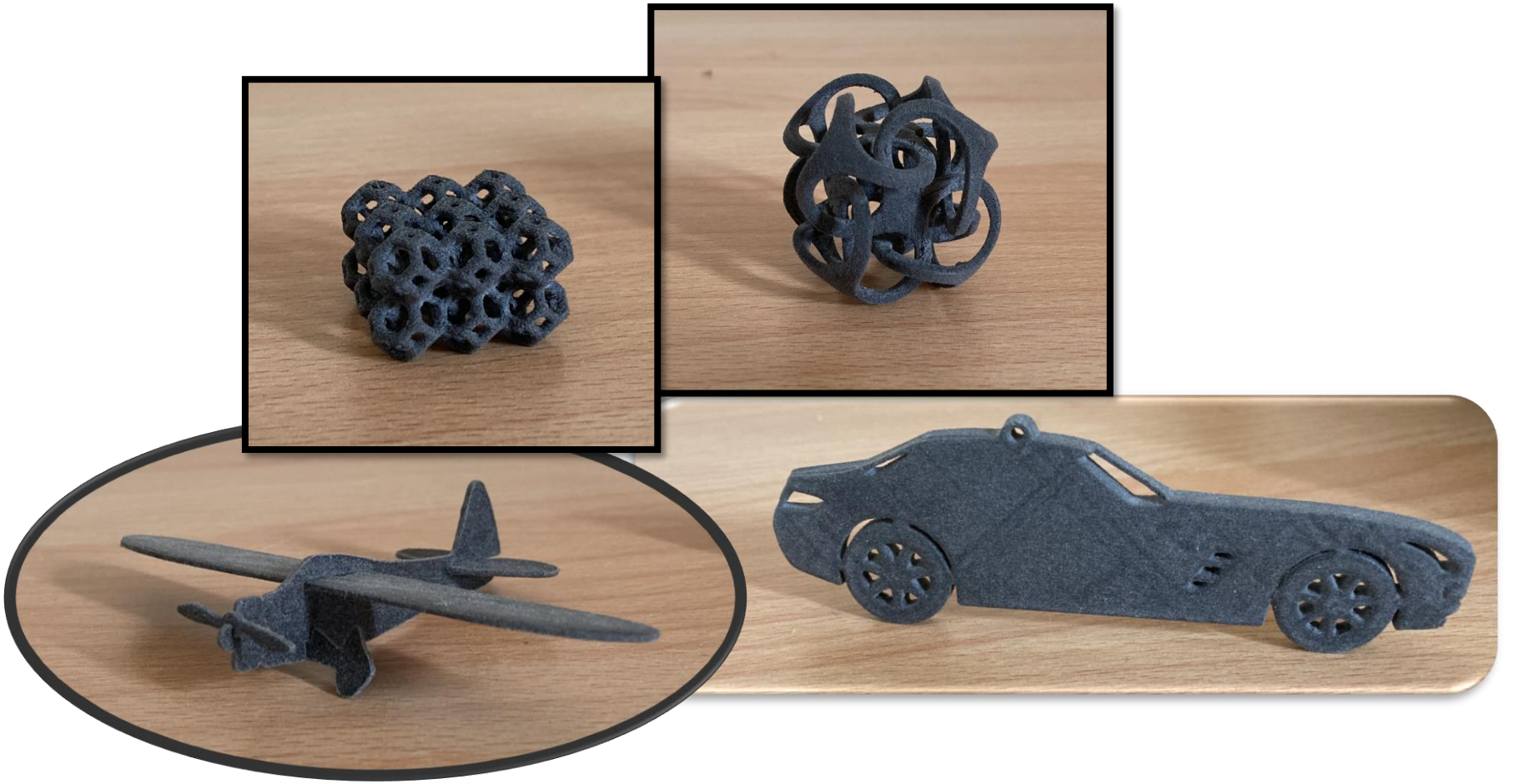
Conclusion & Perspective

Optimization of powder bed temperature and laser parameters using a **“powder-based” characterization approach**

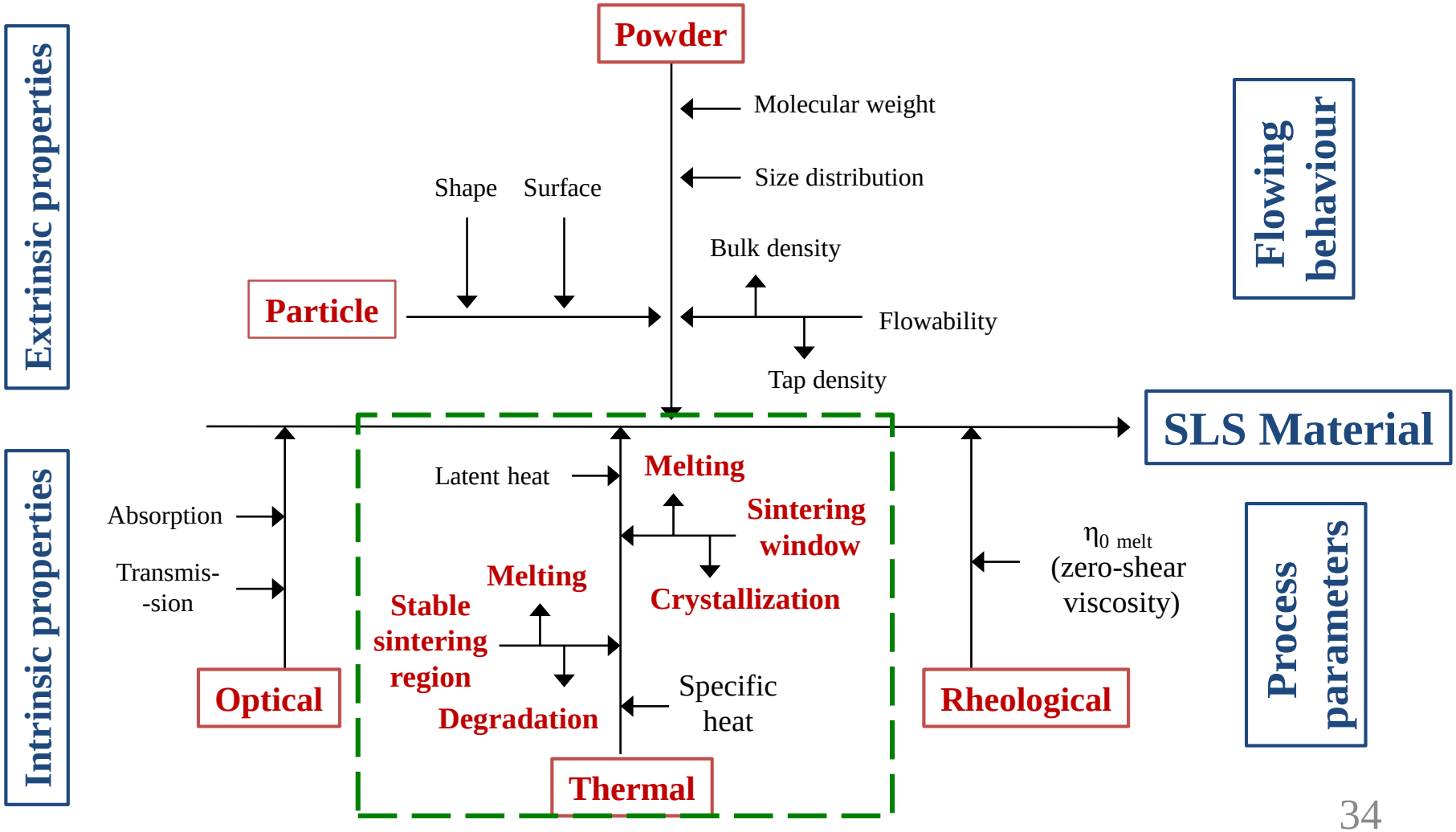
- Reduced trial and errors builds: “a priori” evaluation of optimized processing regions ✓
- Consider material properties: crystallization kinetics ($t_{1/2}$), EMR ✓

- Can be generalized to other polymer systems ?
- Can be improved (rheological and optical properties of powders) ?

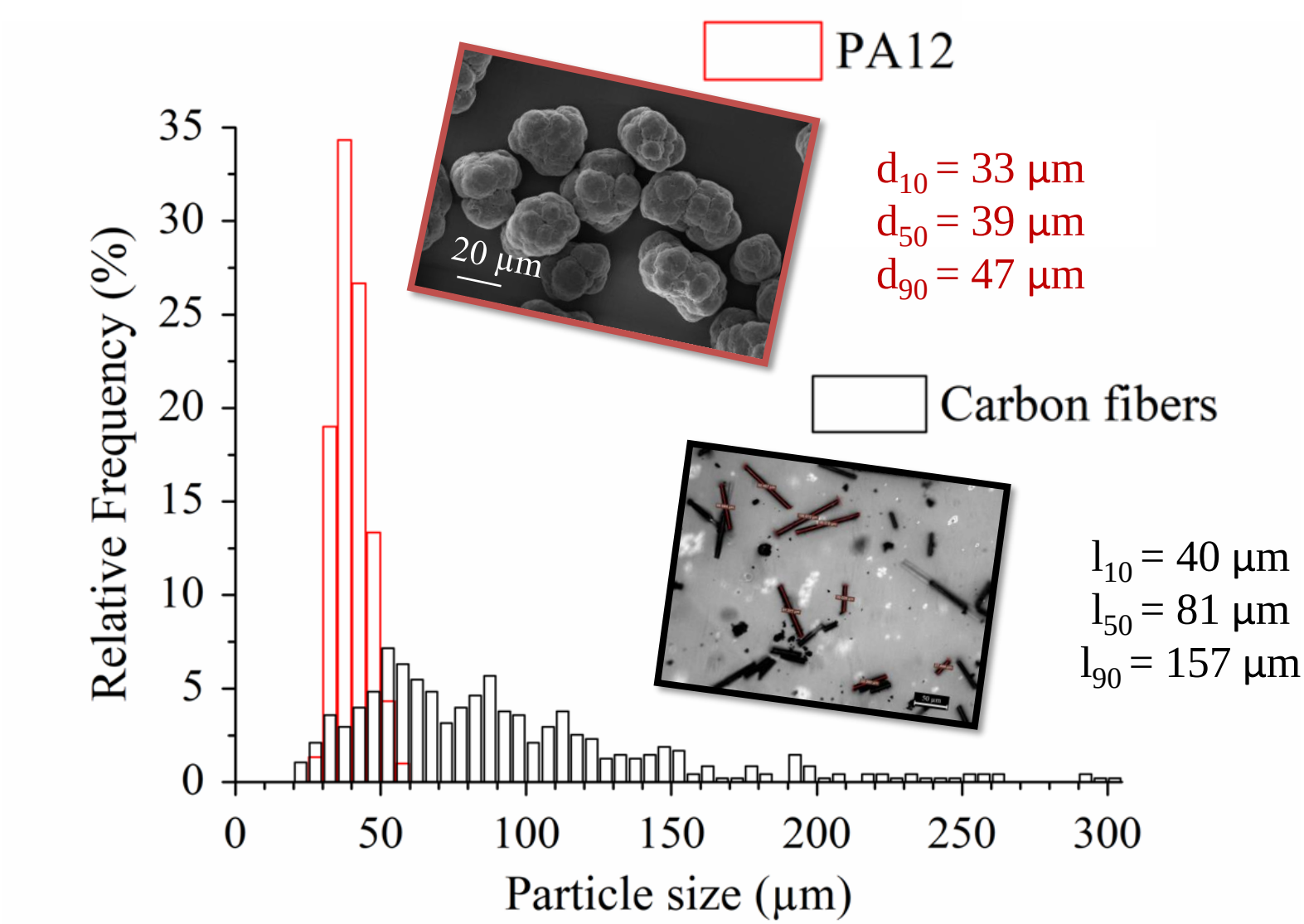
Once you've done process optimization



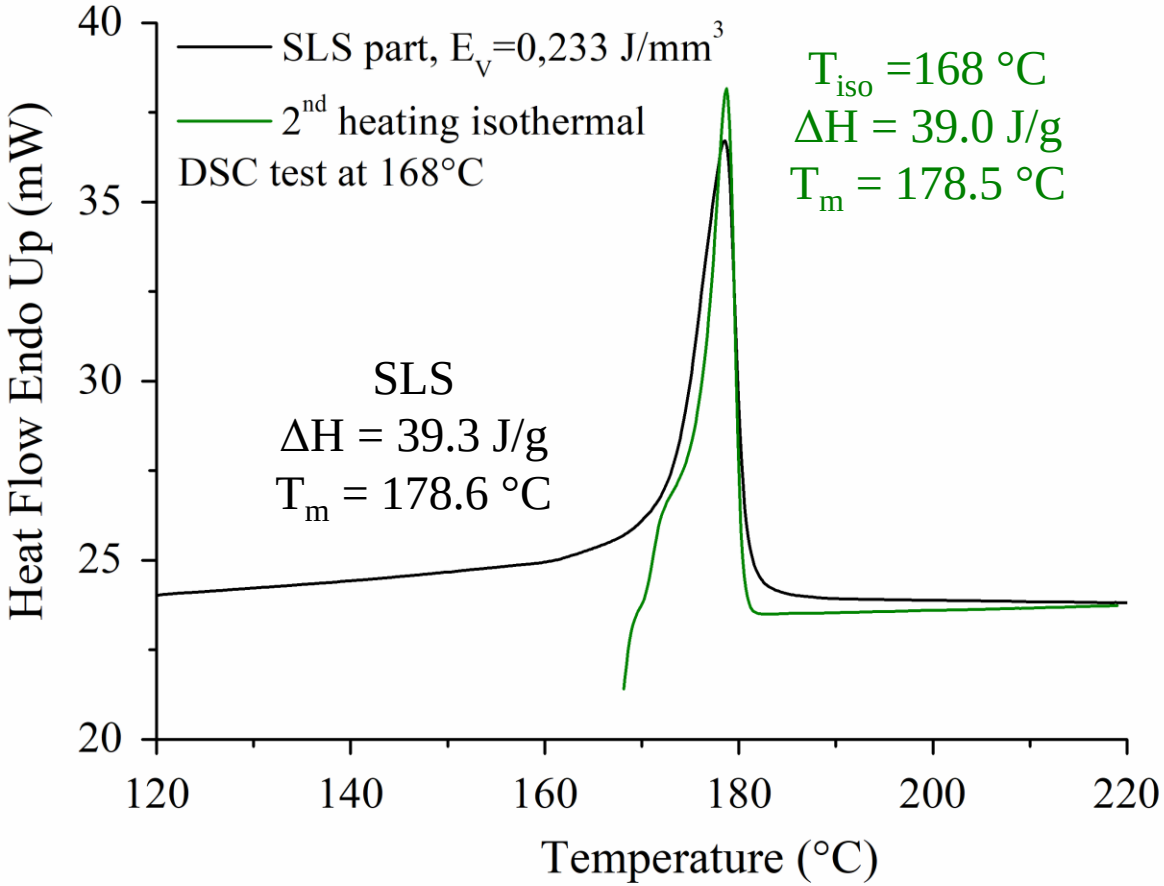
Outline of polymer powders properties significant for LS processing



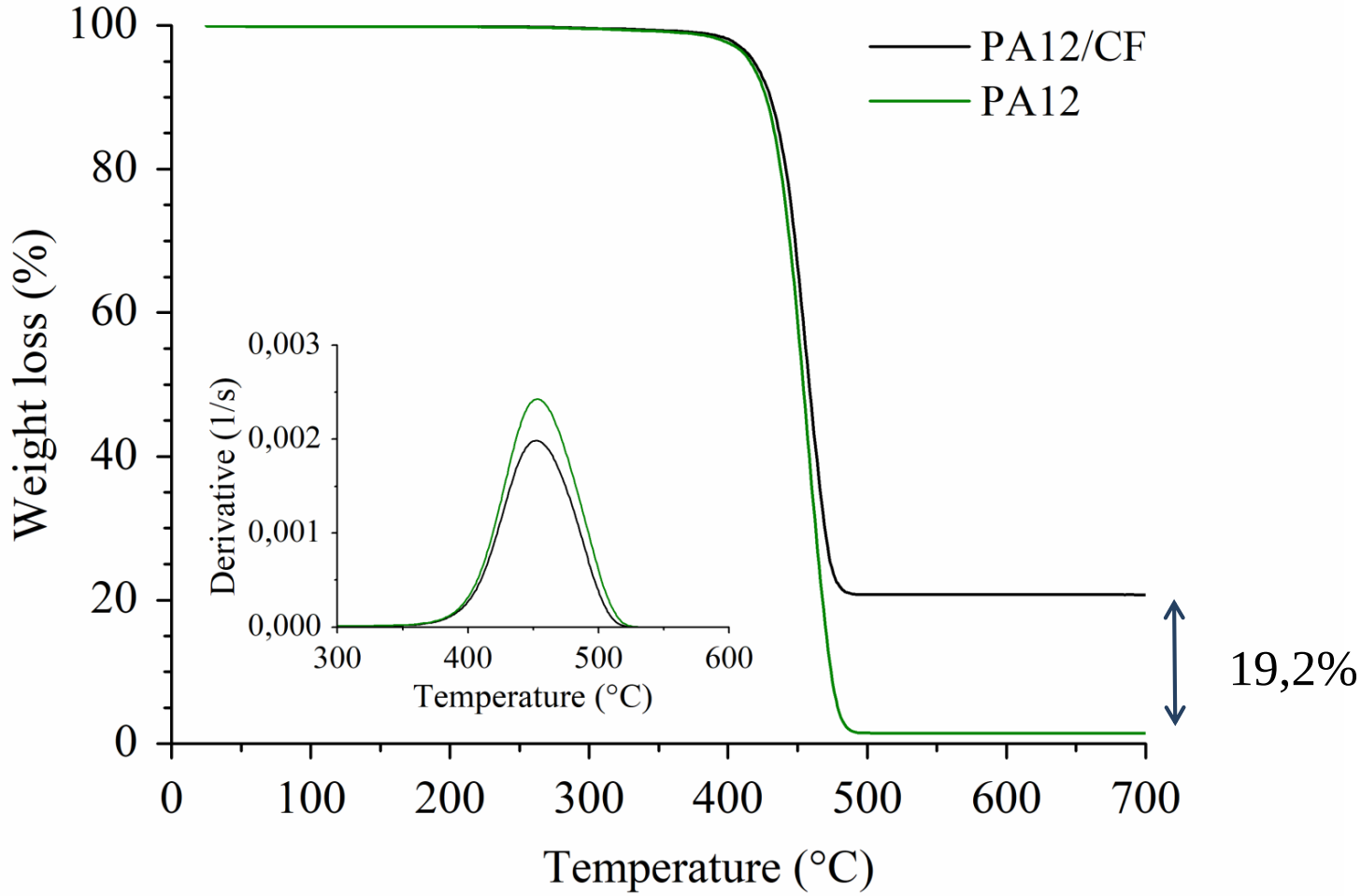
PA12/CF Particle size distribution



PA12/CF Quasi-isothermal LS model validation

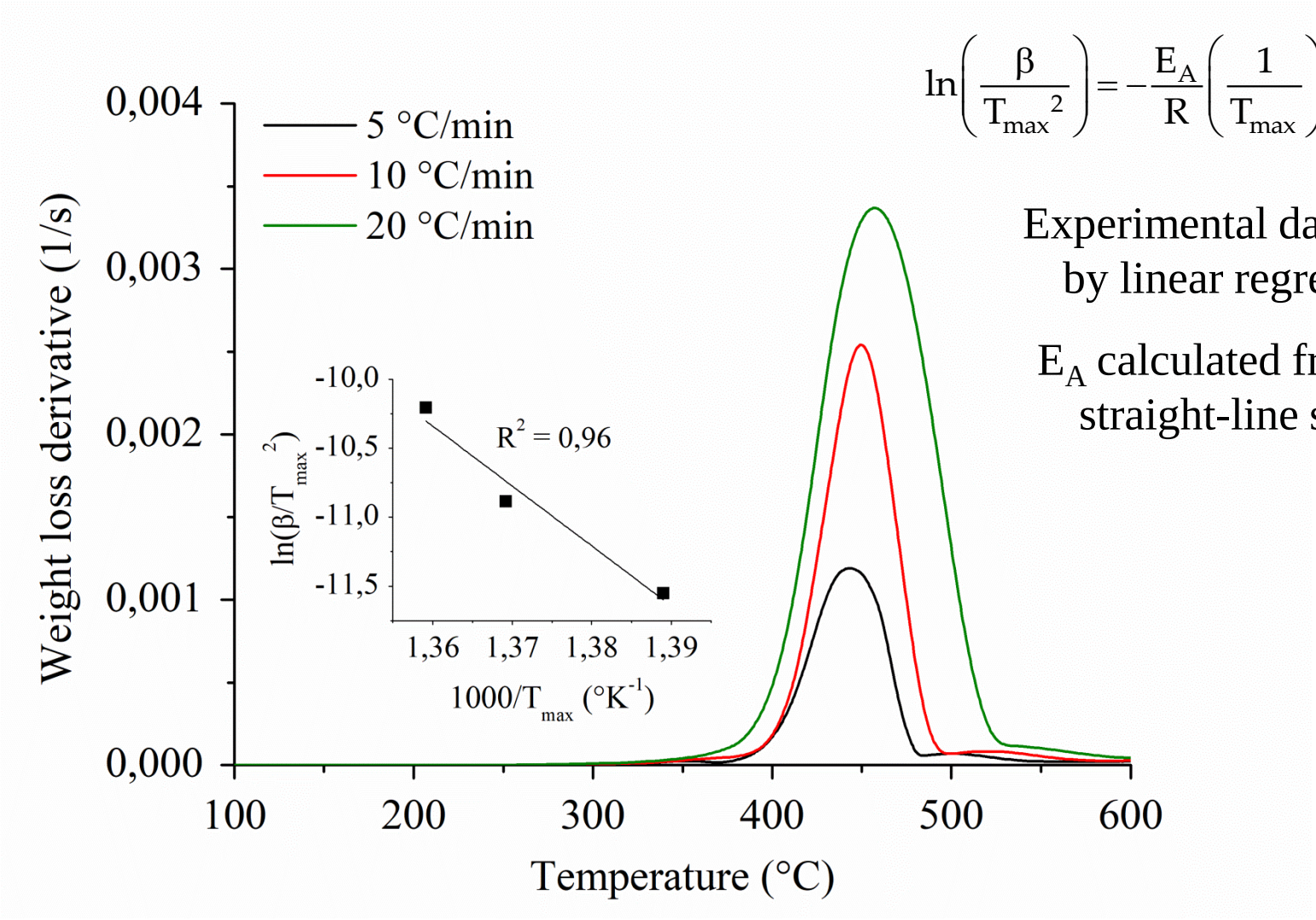


PA12/CF Thermogravimetrical analysis



PA12/CF Activation energy for degradation

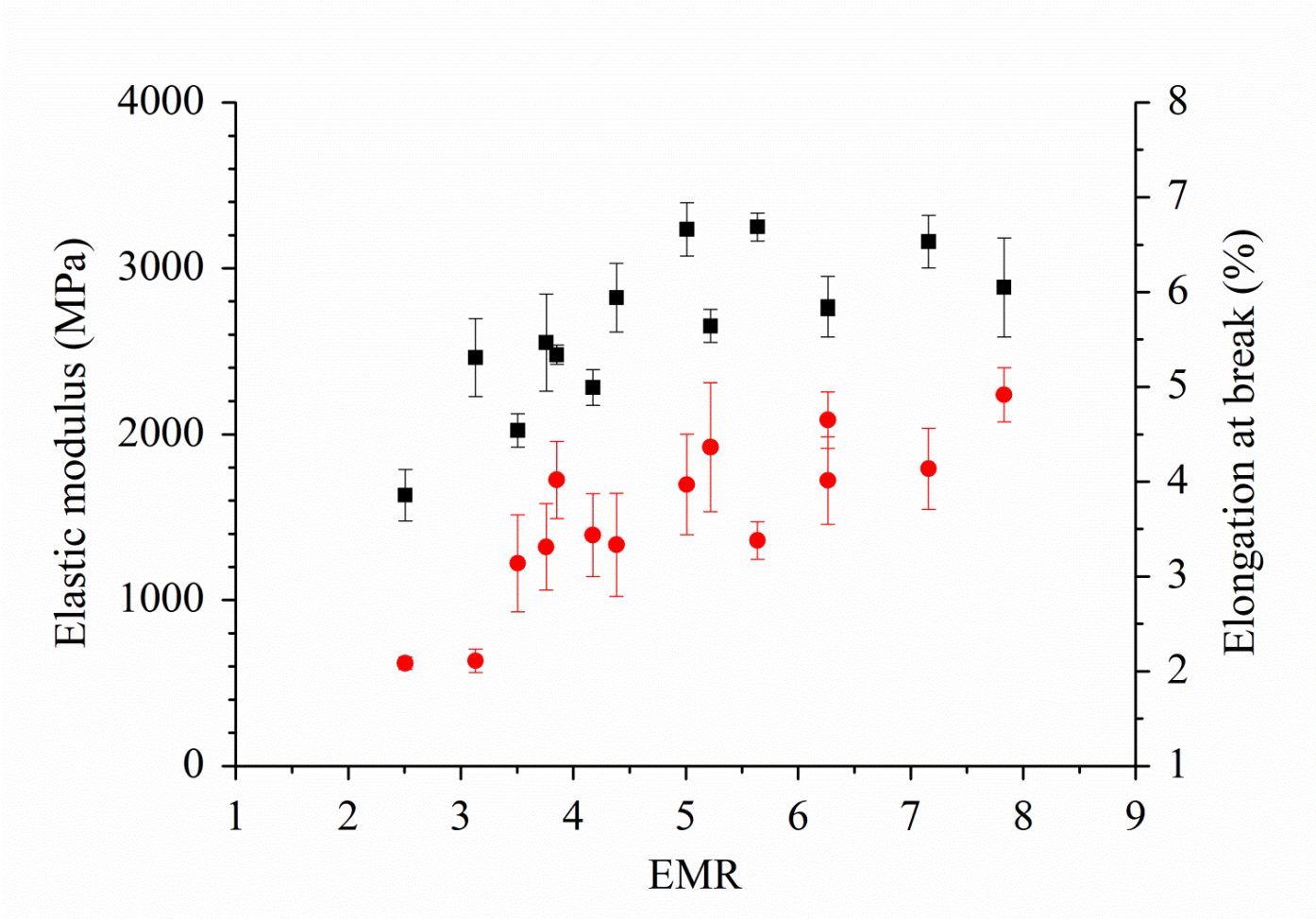
$$\ln\left(\frac{\beta}{T_{max}^{2}}\right) = -\frac{E_A}{R}\left(\frac{1}{T_{max}}\right) + \ln\left(\frac{AR}{E_A}\right)$$



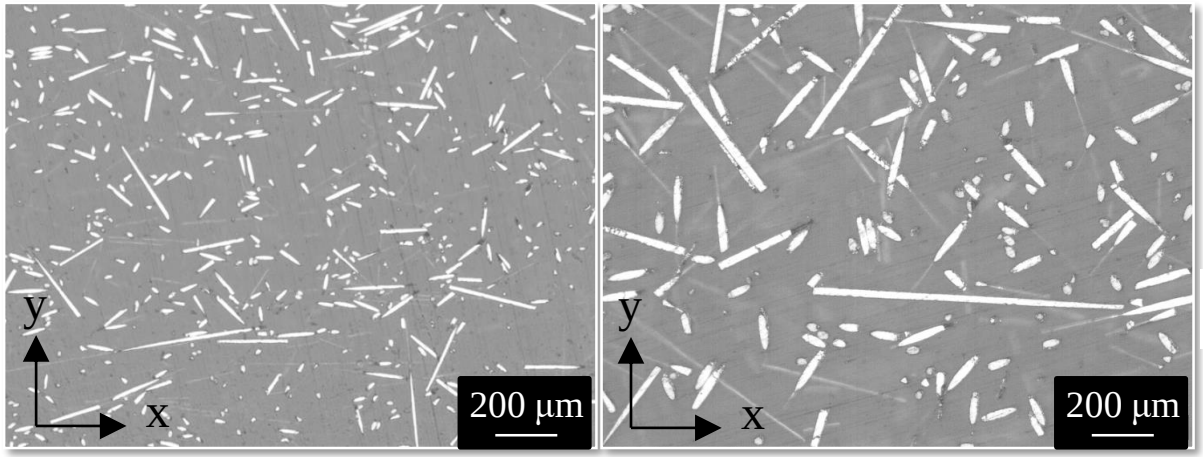
Experimental data fitted by linear regression

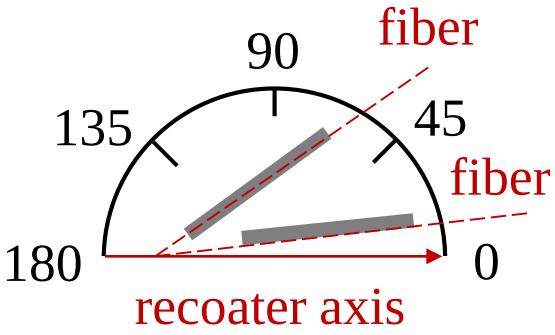
E_A calculated from the straight-line slope

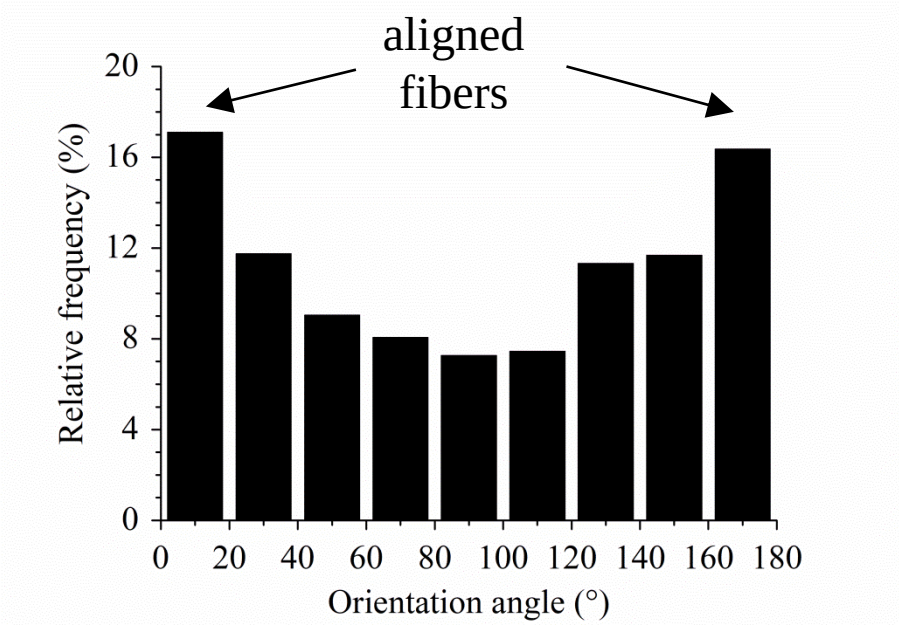
PA12/CF Elastic modulus and elongation at break

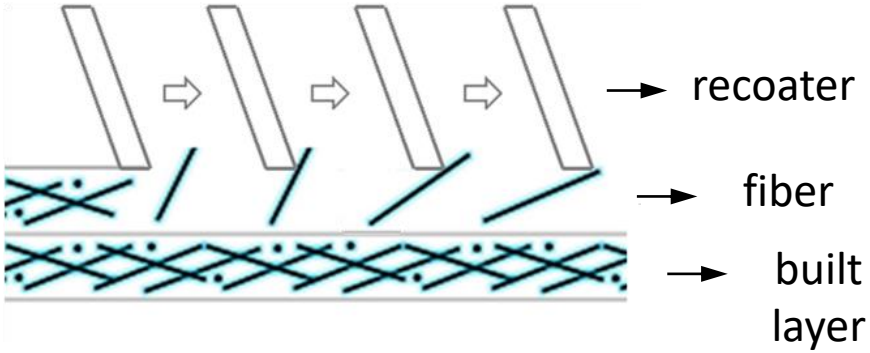


PA12/CF Fiber orientation









Effect of layer spreading on fiber orientation

PA12/CF Surface fracture