MATERIAIS 2022

Exploring a Better Future

Controlling morphological development during Additive Manufacturing: a route to mapping of properties

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SPM SOCIEDADE PORTUGUESA DE MATERIAIS

POLITÉCNICO DE LEIRIA | CDRSP CENTRE FOR RAPID AND SUSTAINABLE PRODUCT DEVELOPMENT

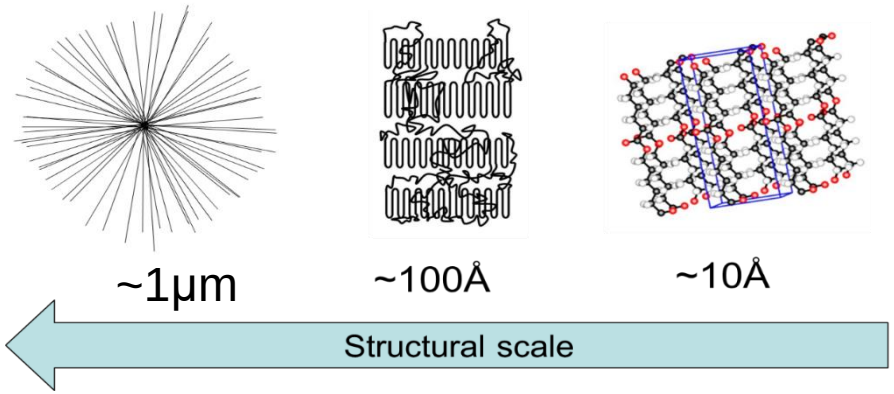
SUMMARY

- Polymer morphology
- Morphology in extrusion
- 3D Printing – FDM
- Experiment description
- Results
- Conclusion
- Future Work

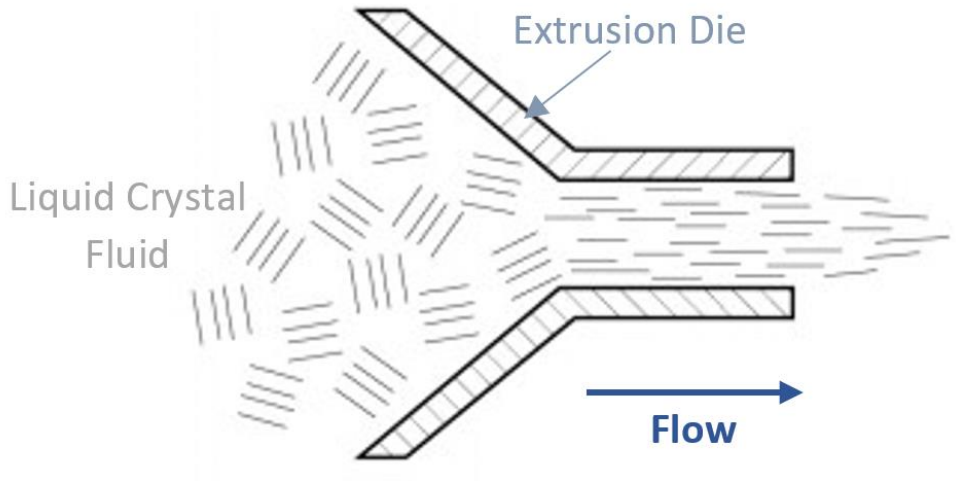
INTRODUCTION

Polymer morphology - Semi-crystalline thermoplastics

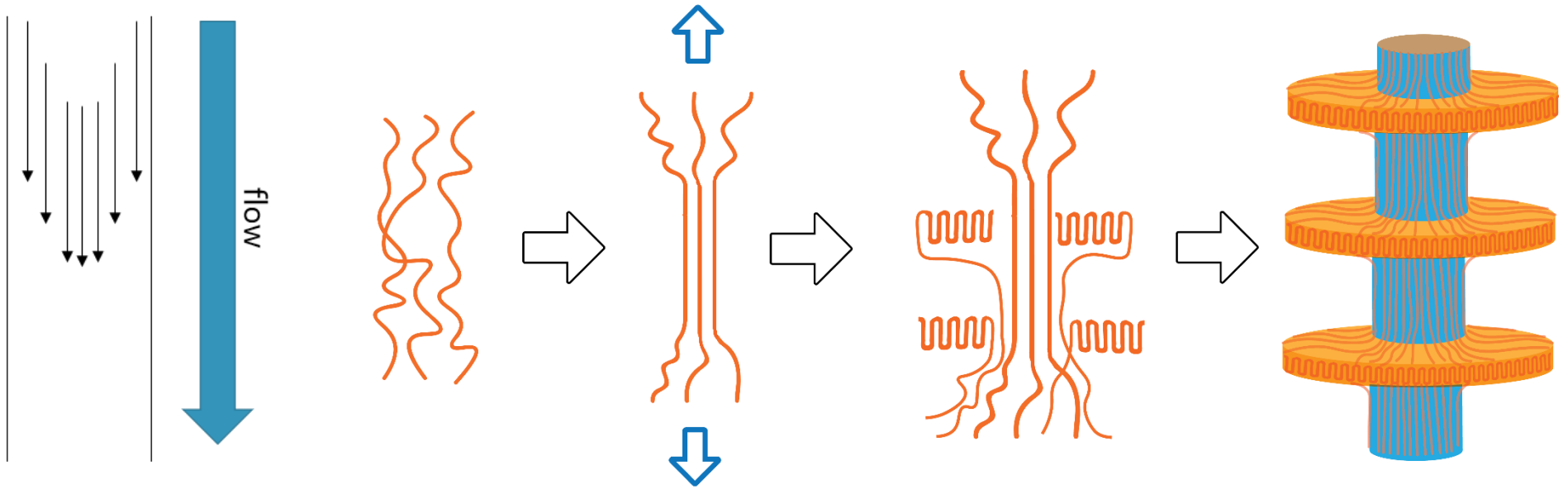
- Spherulite are assemblies of chain folded lamella that grow radially from a point in space;
- Under flow, polymer chains may create linear nuclei parallel to flow direction;
- Shish kebab structures result from lamella growing perpendicular to a nucleating surface.



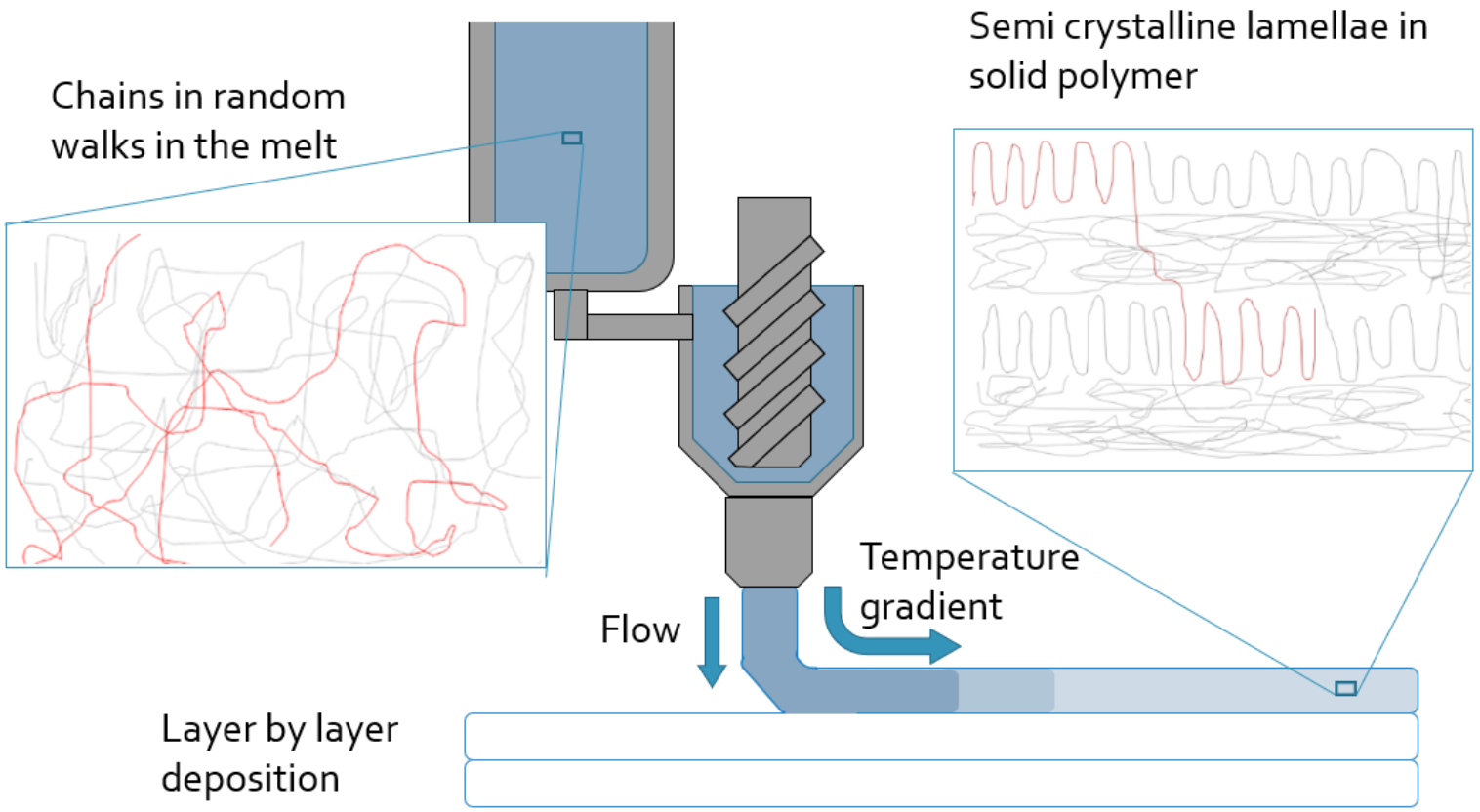
Morphology in extrusion



Morphology in extrusion



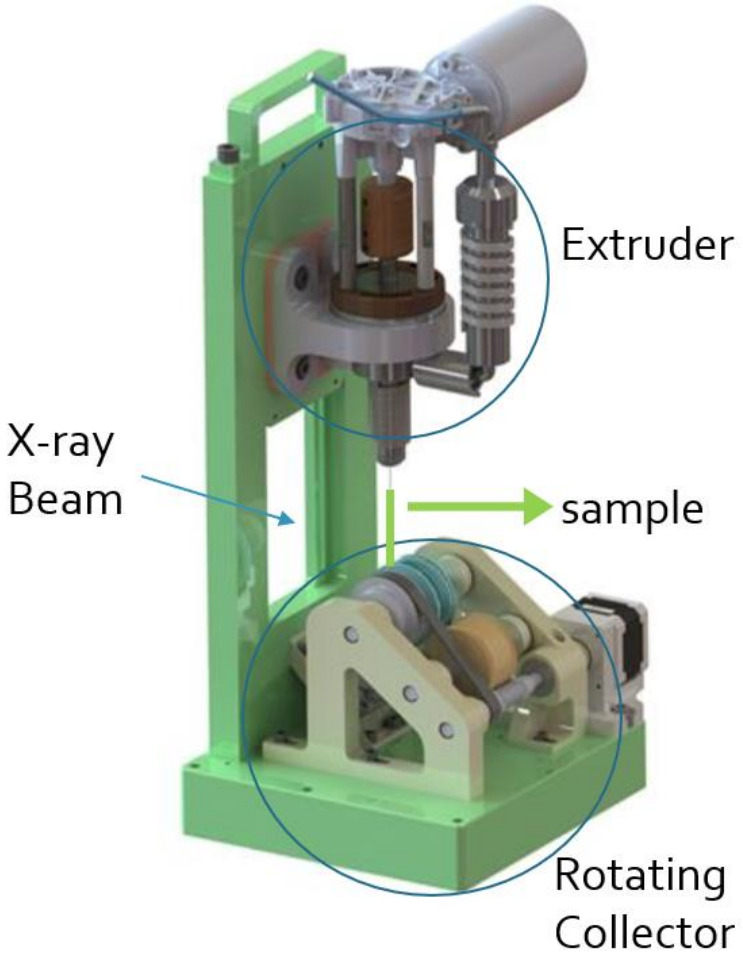
3d Printing – Fused Deposition Modelling



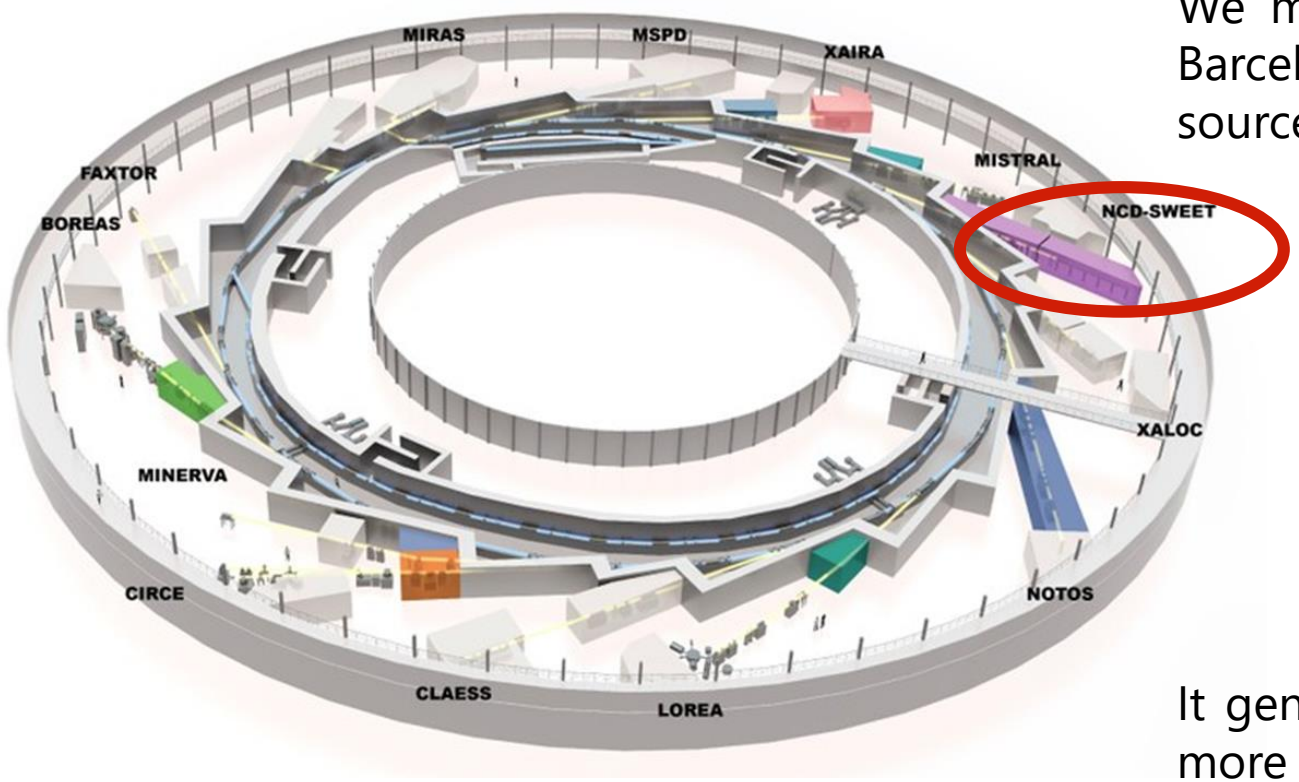
THE EXPERIMENT

Concept

- In-situ extrusion;
- Observe crystallization as is happening;
- Control of parameters while processing;
- Time resolved SAXS/WAXS analysis.



ALBA Synchrotron light source



https://www.cells.es/en/beamlines/bl11-ncd

We make use of the ALBA Synchrotron Light Source in Barcelona which is a 3rd generation synchrotron light source

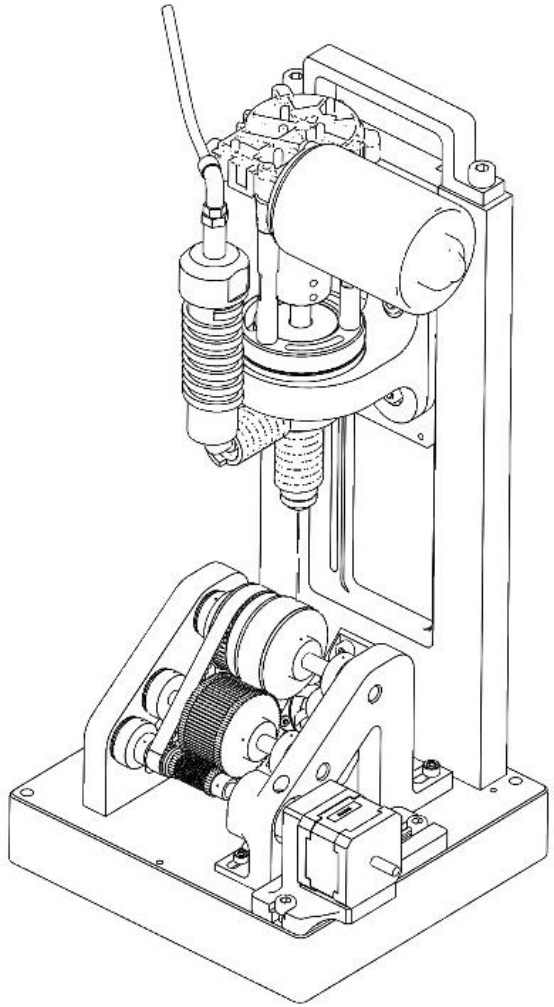
It is essential for

- ✓ Time –resolving studies
- ✓ Small samples
- ✓ Spatial mapping
- ✓ Weak Scatterers

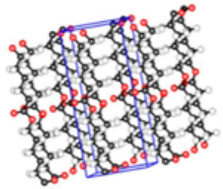
It generates intense beams of x-rays and other radiation, more than a million times as bright as a laboratory source

Developed equipment

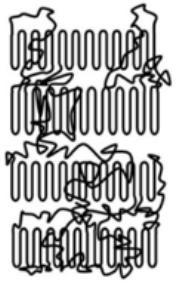
- Positionning accuracy of ± 100 µm;
- Range up to 55 degrees on WAXS;
- Extrusion and collector velocity control;
- Vertical range of 50 mm.



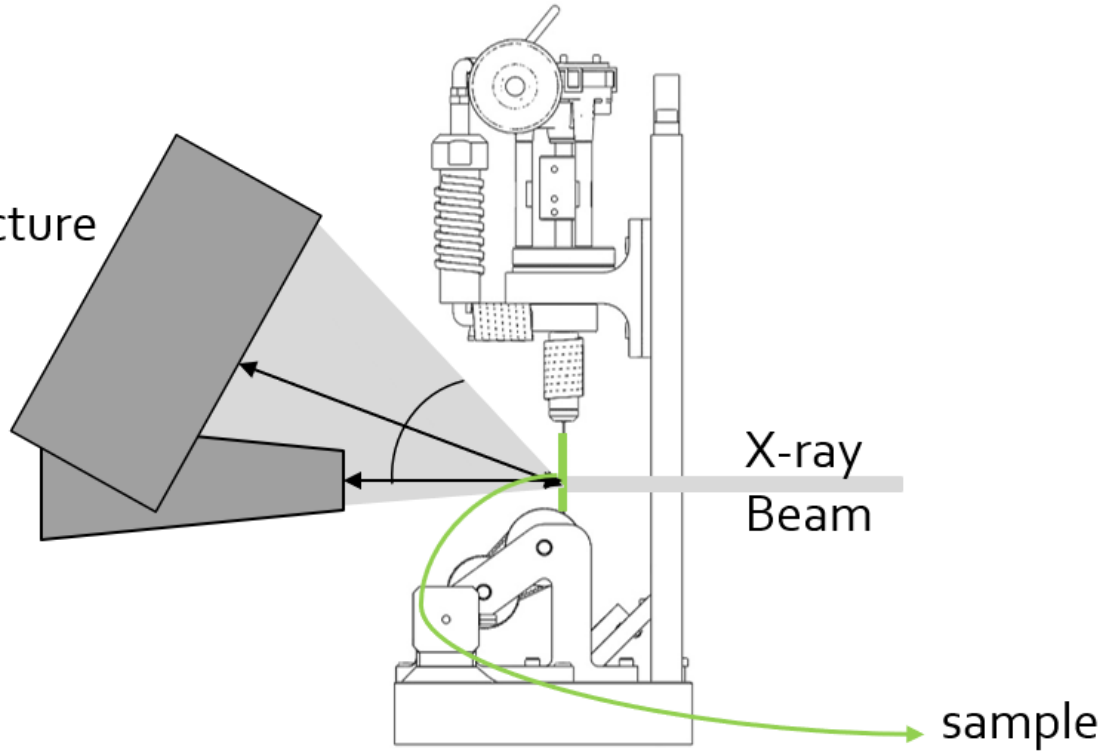
Experimental Assembly



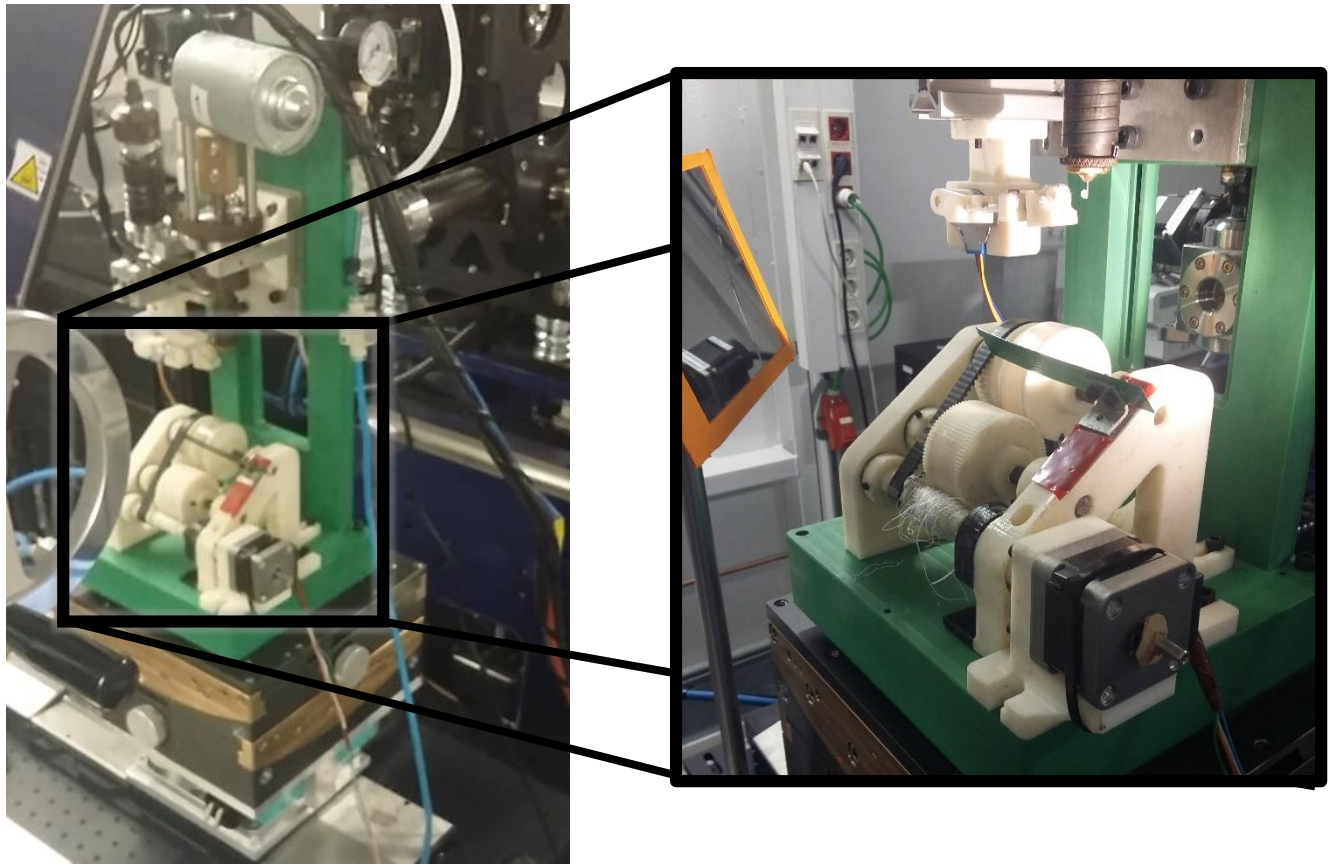
WAXS detector
Crystalline structure



SAXS detector
Lamellae
morphology



Experimental Assembly

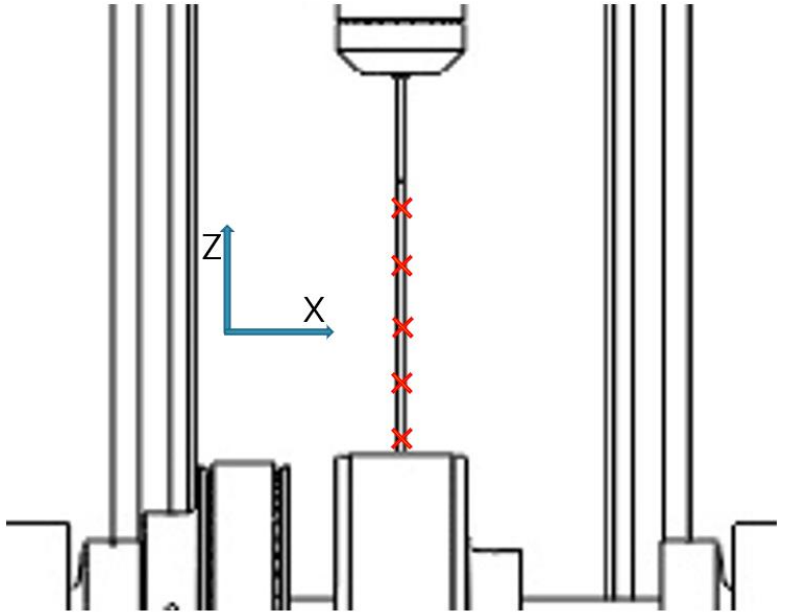


Experimental Assembly

Extrudate scanning

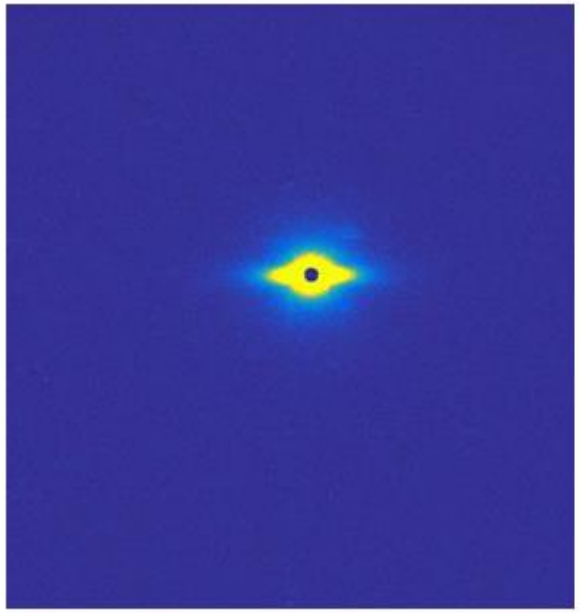
- Point measurements on the Z direction, observing the polymer scattering **as it crystallizes**;

- Static extrusion means that measurements along the Z axis relate to the **evolution in time**.

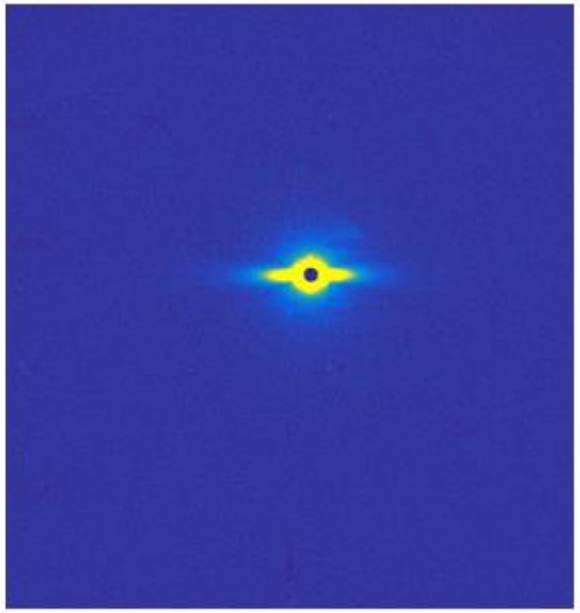


RESULTS

Filament analysis - SAXS

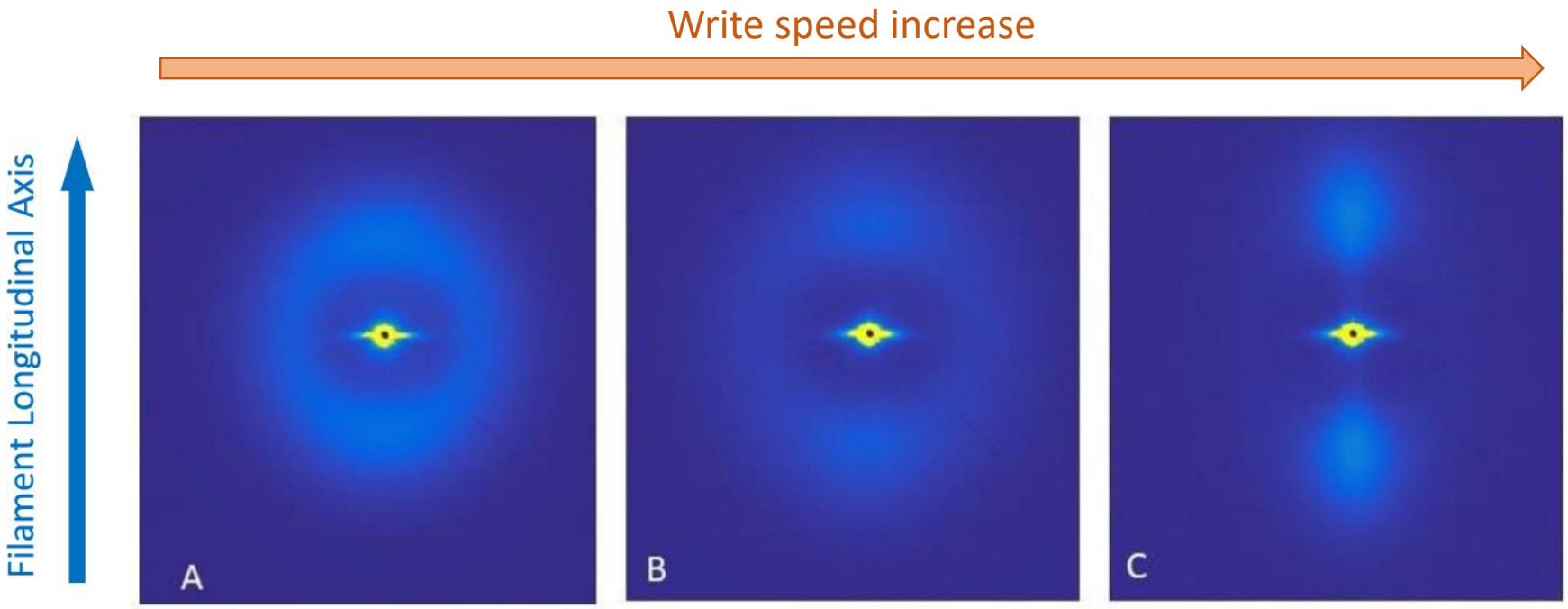


Lower collector velocity



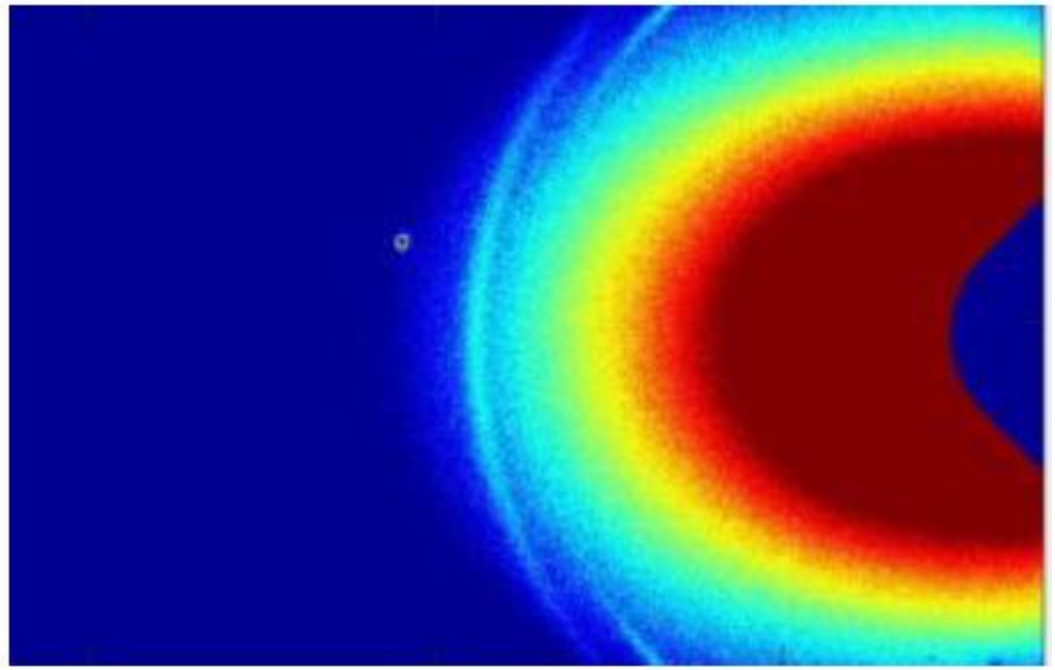
Higher Collector Velocity

in-situ Analysis - SAXS



LDPE
Extrusion Temperature = 170°C

in-situ Analysis - WAXS



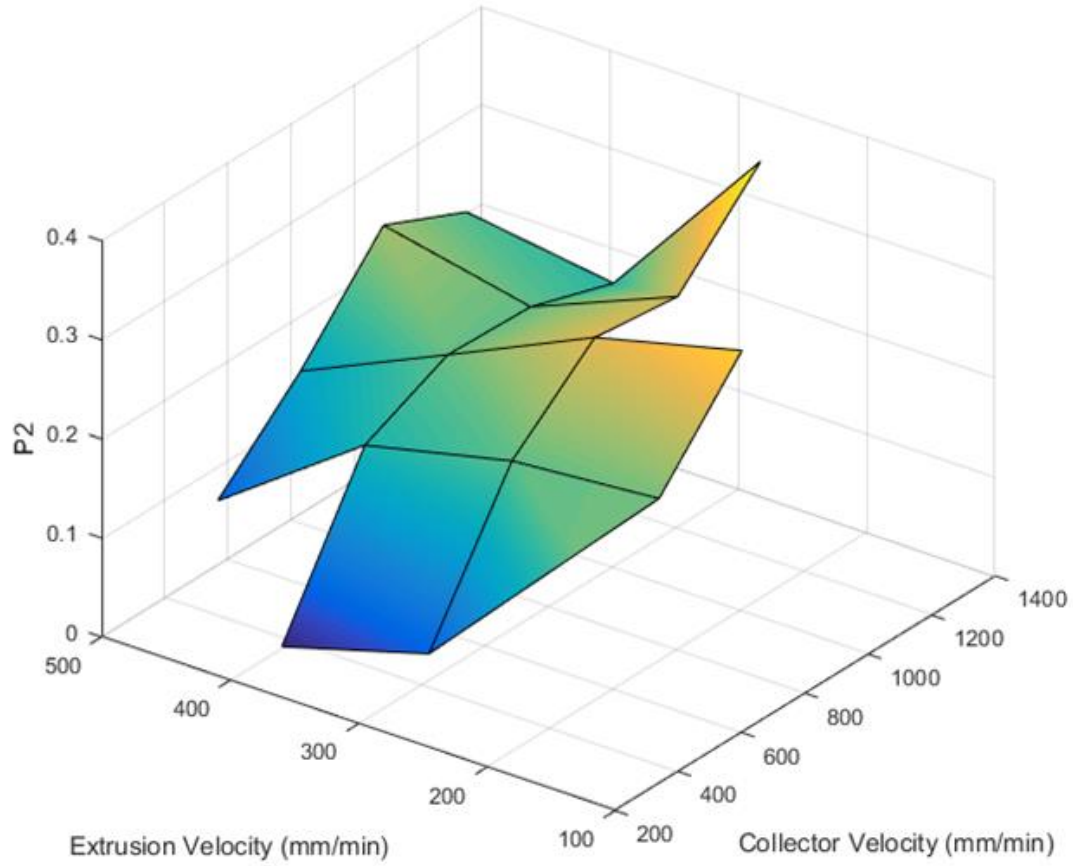
LDPE
Extrusion Temperature = 170°C

Processing and Morphology

Orientation factor:

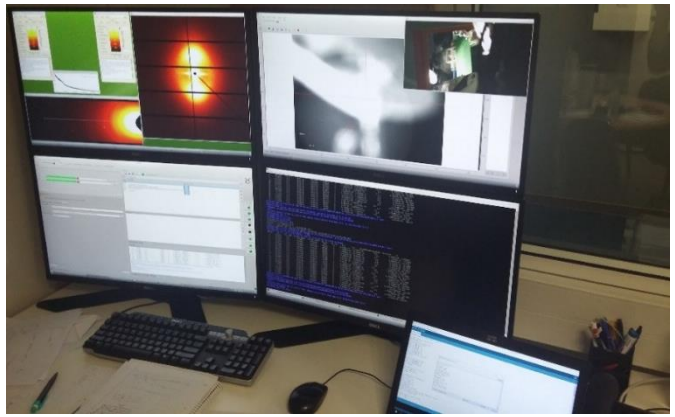
$$\langle P_2 \rangle = \frac{\int_0^{\frac{\pi}{2}} I(|\vec{Q}|,\alpha) P_2(\cos(\alpha))\sin(\alpha) d\alpha}{\int_0^{\frac{\pi}{2}} I(|\vec{Q}|,\alpha)\sin(\alpha) d\alpha}$$

P_2=1 – uniform orientation of crystals



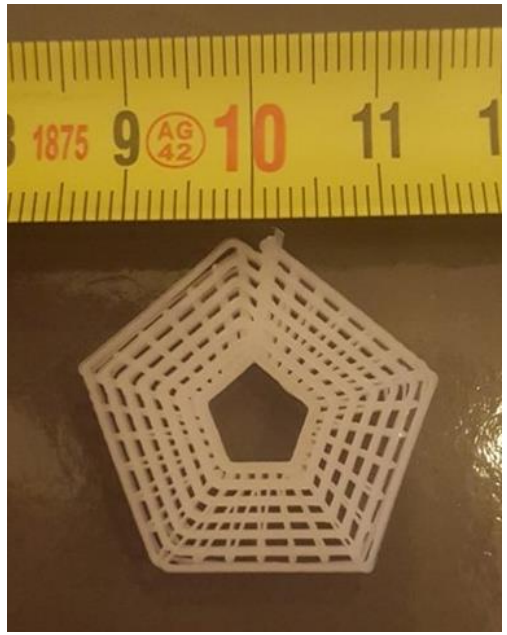
CONCLUSIONS

- **Processing and structure relationships** in extruded filaments were successfully observed at the ALBA synchrotron light source;
- Higher write speeds lead to faster cooling – **extended chains during crystallization**
- Production of 3d printed parts with targeted morphology **possible based on parameters control.**



Future Research

- Exploring the design of products to take advantage of this new approach.



THE TEAM

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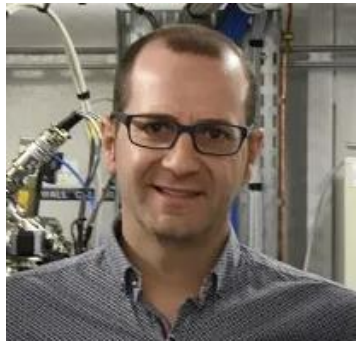
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Thanks for your attention.

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