DYNAMIC SEM STUDIES AND ANALYSIS OF HEAT STORAGE MATERIALS PHASE CHANGE TRANSITIONS

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MOTIVATION OF SEM DEVICE USES

Observations of **various materials**, at **microscale**

***Ex situ* qualitative:**

- **Morphology** images
- Chemical **composition**/distribution images

***In situ* & *real time* quantitative results:**

- Materials behaviour during **thermal** &/or **mechanical contraints**
- **Phase transitions** characterisations: alloys synthesis **mechanisms**…
- Fronts propagation **monitoring**…

DEVICES & FEATURES

SEM: Zeiss Evo LaB6

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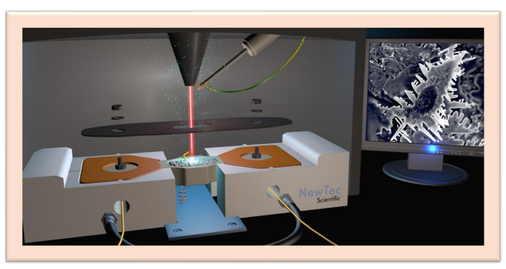

Hot stage: MT1000 NewTec Scientific

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SEM features

- High & low vacuum
- High speed imaging
- From 5 to 30 KV
- From 100x to 10,000x

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Hot stage features

- 2 ovens with a cooling system
- 5 thermocouples
- From 20°C to 800°C
- Traction device
- High & low heating/cooling speed

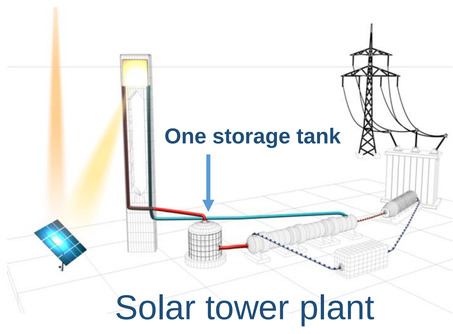
STUDIED MATERIALS

Heat Storage Materials of interest

Phase Change Materials (PCMs)
&
PCM-based composites

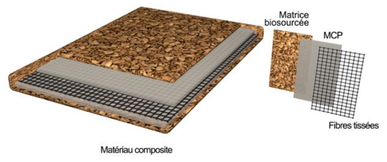
Industrial applications

THERMAL **STORAGE**



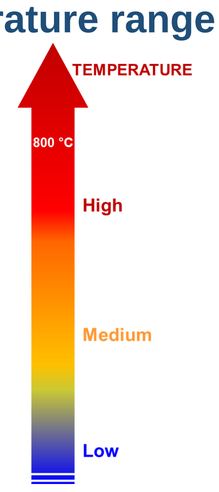
Solar tower plant

THERMAL **MANAGEMENT**



100% biosourced composite

Temperature range

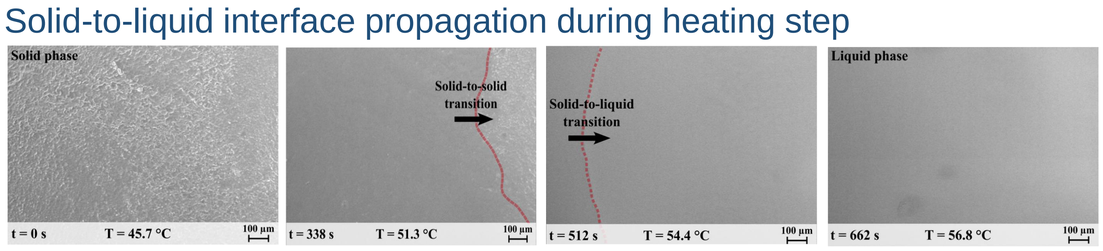


RESULTS OBTAINED FOR PURE & MULTICOMPONENTS PCMs

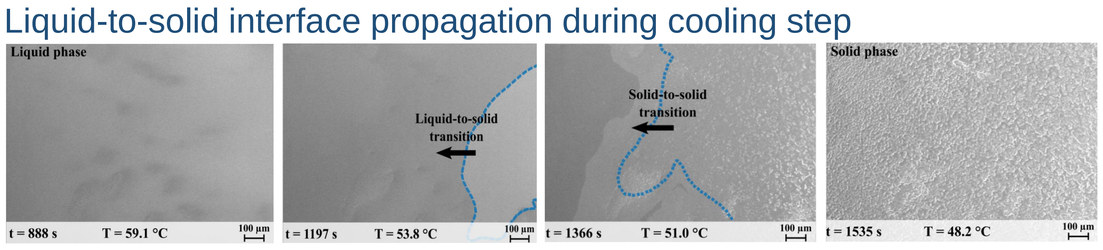
FOSSIL- OR BIOSOURCED-BASED ORGANIC TYPES

1) PARAFFIN RT60 CASE ➟ STANDARD PCM

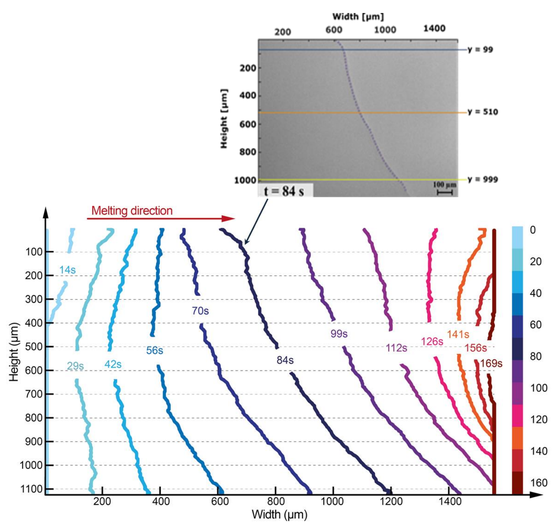
Solid-to-liquid interface propagation during heating step

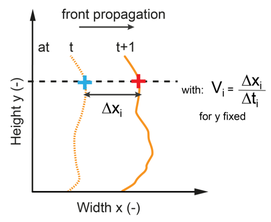


Liquid-to-solid interface propagation during cooling step



Imaging processing (Matlab software)





Front propagation average velocities assessments for:

- liquefaction:

$$\bar{v}_{s/l} \approx 10 - 14\ \mu m/s$$

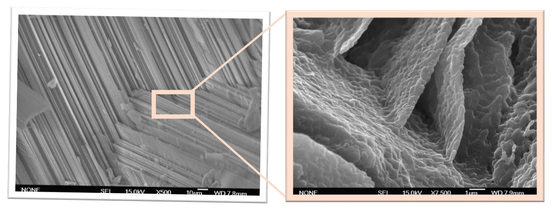
- solidification:

$$\bar{v}_{l/s} \approx 1 - 4\ \mu m/s$$

METALLIC- OR MINERAL-BASED INORGANIC TYPES

1) PURE COMPOUND: LiOH CASE ➟ STANDARD PCM

Packed lamellae structure

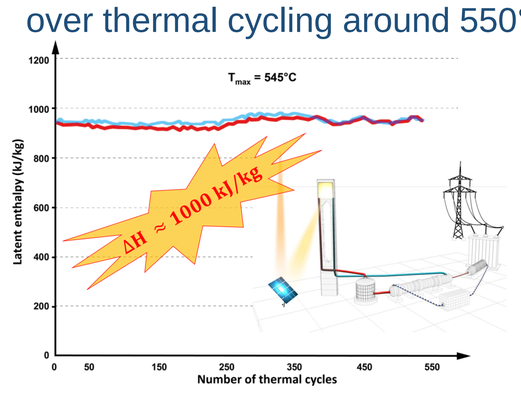


⇒ anisotropic thermal behaviour:

$$\bar{k}_{\parallel} \approx 44\ W/m.K$$

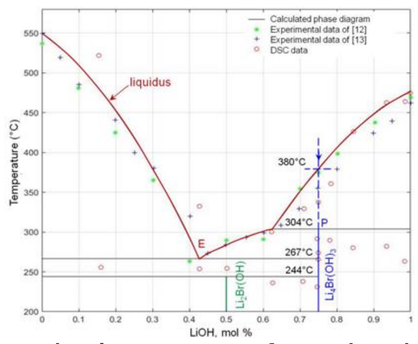
$$\bar{k}_{\perp} \approx 0.86\ W/m.K$$

Very high energy stability over thermal cycling around 550°C

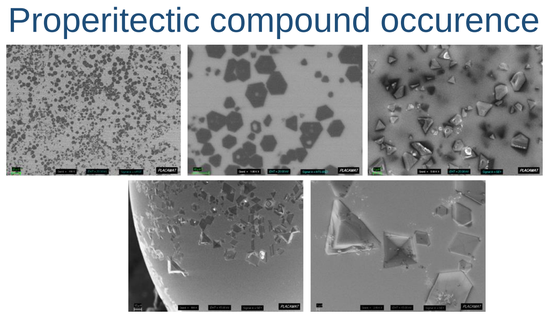


2) BINARY PERITECTIC COMPOUND: $Li_4Br(OH)_3$ CASE ➟ ADVANCED PCM

LiOH/LiBr calculated phase diagram

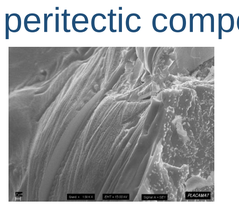


Experimental monitoring steps:
Properitectic compound occurence



Theoretical process of synthesis

Final peritectic compound



REFERENCES

- P. Legros, E. Lebraud, F. Achchaq, "*$Li_4Br(OH)_3$ microstructure monitoring over its synthesis to tackle the lithium-based salts exploitation challenges as advanced phase change materials for storage technologies*", Materials & Design, 2020. https://doi.org/10.1016/j.matdes.2020.109160
- F. Achchaq, S.-C. Moon, P. Legros, "*Unlocking the power of LiOH: Key to next-generation ultra-compact thermal energy storage system", Heliyon-Cell Press, 2024. In press*