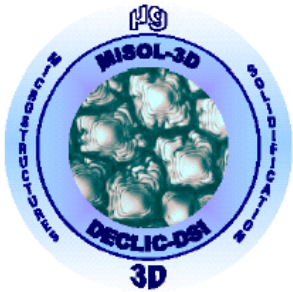


3D pattern adjustment during directional solidification of a transparent alloy conducted on DECLIC-DSI



F.L.Mota, J.Pereda, N.Bergeon
*IM2NP, CNRS
Aix-Marseille Université
Marseille, France*



R.Trivedi
*Dpt of Materials Science & Engineering
Iowa State University, Ames, USA*

L.L.Strutzenberg
*Marshall Space Flight Center,
Huntsville, USA*

K.Ji, Y.Song, A.Karma
*Physics Department
Northeastern University, Boston, USA*

D. Turret
*IMDEA Materials Institute,
Madrid, Spain*



Northeastern University

Introduction

- **DECLIC-DSI instrument**
- **Experiments in the DECLIC-DSI**

Effect of sub-boundaries on Primary spacing

- **Experimental results**
- **Phase field simulations**

Conclusion and perspectives

To study the microstructure formation during directional solidification

Transparent systems →

In situ and real time observation of interfacial microstructure

Large cylindrical crucible →

extended patterns

Onboard the ISS from 2009 to 2011 →

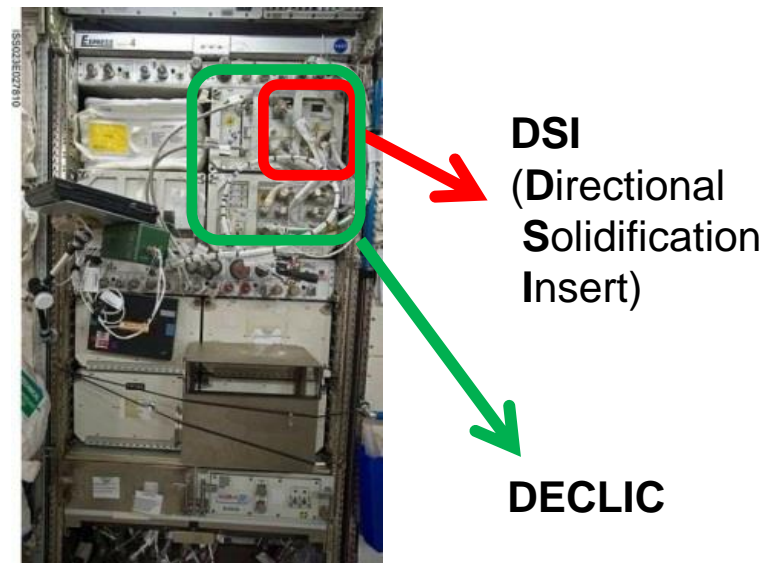
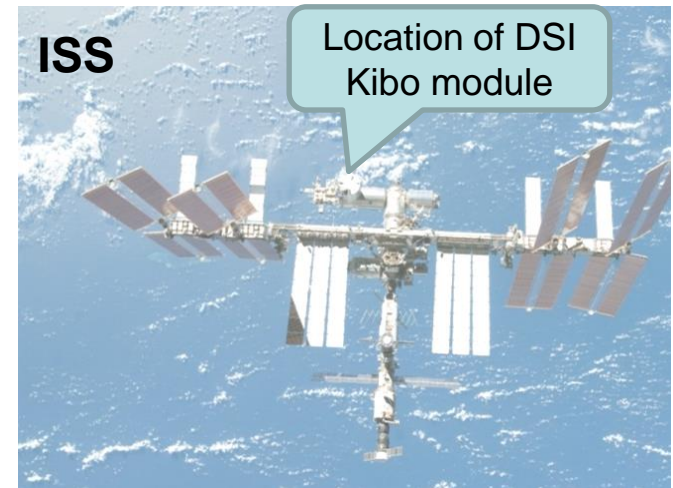
microgravity experiments dedicated to cellular regime

Experiments on ground →

understand the effect of convection

Onboard the ISS from 2017 to 2018 →

microgravity experiments dedicated to dendritic regime



Cylindrical crucible

Diameter: 1 cm

Solidification length: 10 cm

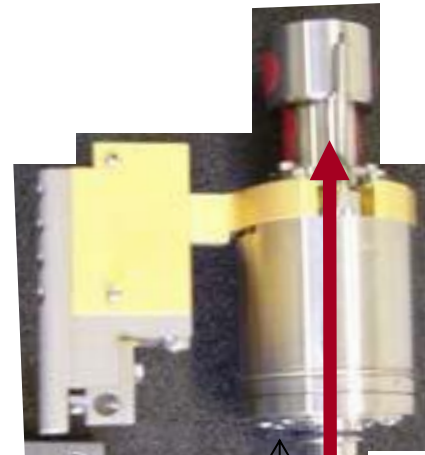
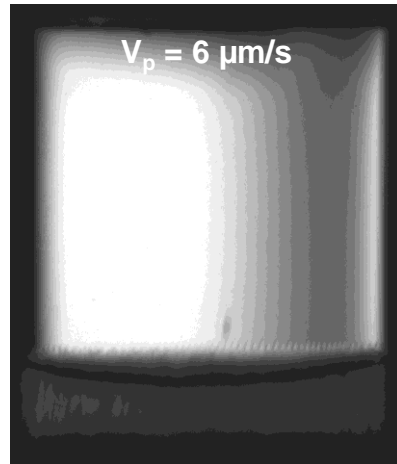
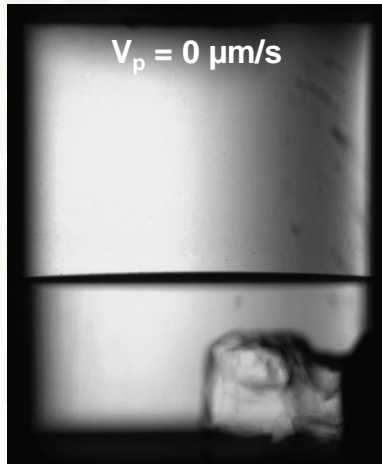
SCN – 0.24 wt% camphor

V: 0.1 – 30 $\mu\text{m/s}$

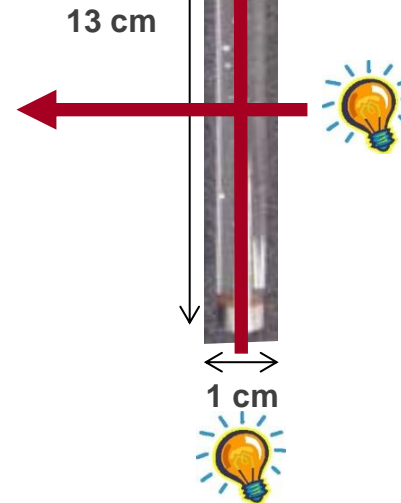
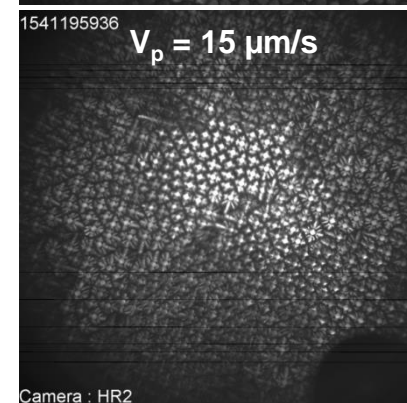
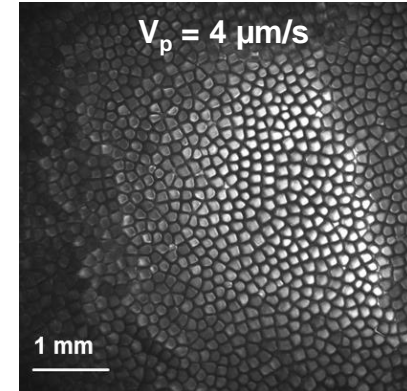
G: 12 and 19 K/cm

Solid seed: single crystal of selected orientation; kept during the whole flight campaign

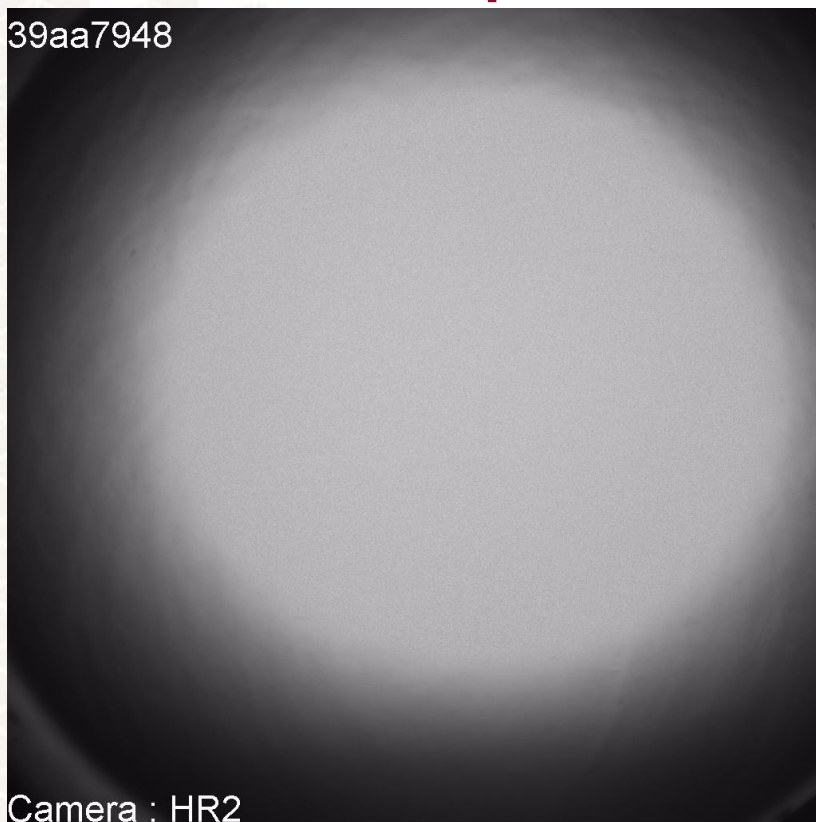
Side view



Axial view



Reference experiment

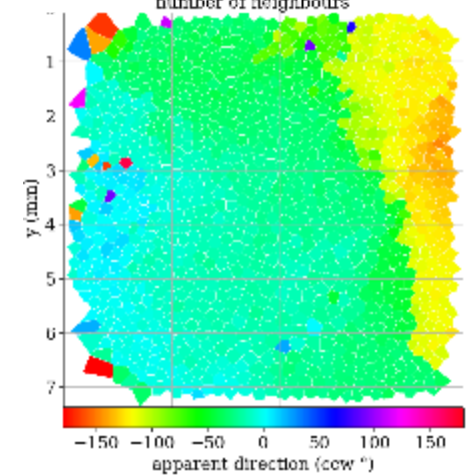
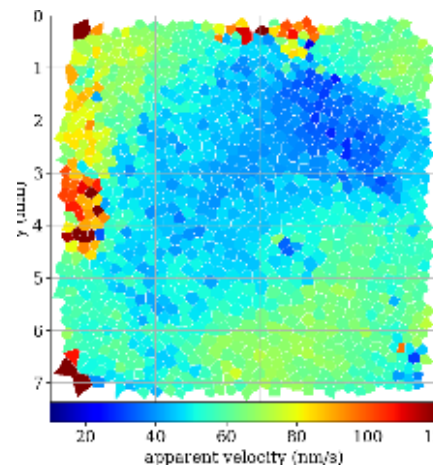
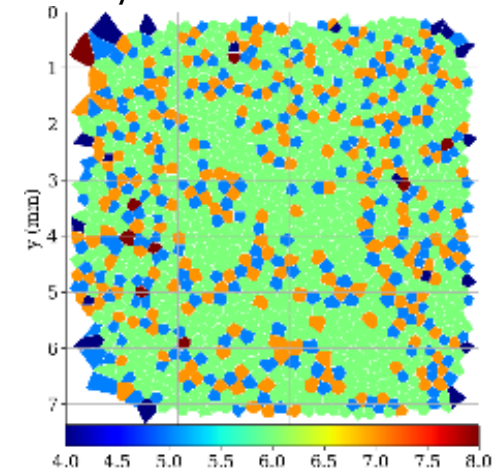
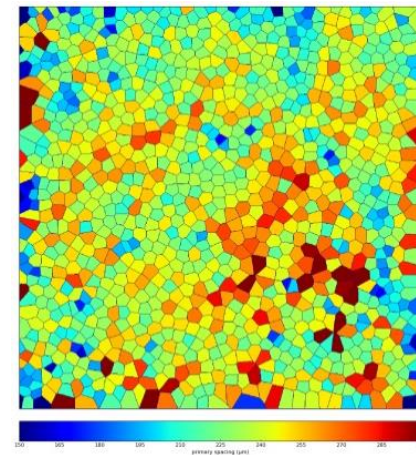


Pattern analysis

5

In-house software to follow each cell in time:

- Primary spacing
- Number of 1st neighbors
- Trajectories (V, direction)



7.37 x 7.37 mm² Real duration: 9h
 $V_p = 2 \mu\text{m/s}$ $G = 19 \text{ K/cm}$

- Pattern sliding
- Very few tip-splittings
- Areas of high elimination

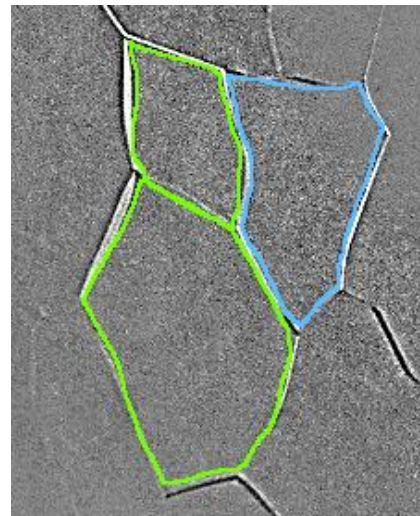
$V = 1 \mu\text{m/s}$ $G = 19 \text{ K/cm}$

Evidence of Sub-boundaries

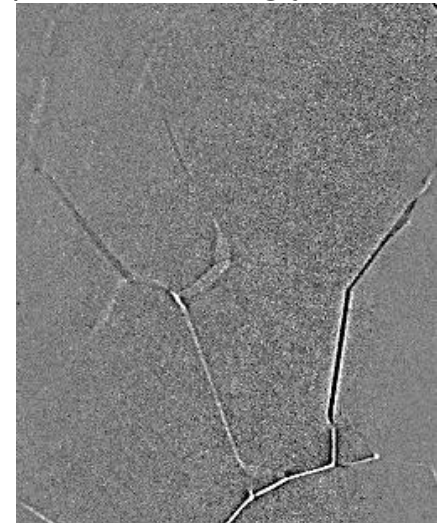
6

- All boundaries are moving due to non-negligible surface tension anisotropy
 - Confirms the sub-boundary nature
 - Even at rest, some SB are moving at very low velocity – SB coarsening
- Motion associated to numerous phenomena → **dynamical reorganization**

Elimination of SG



Nucleation of a new SG (on pre-existing junctions)



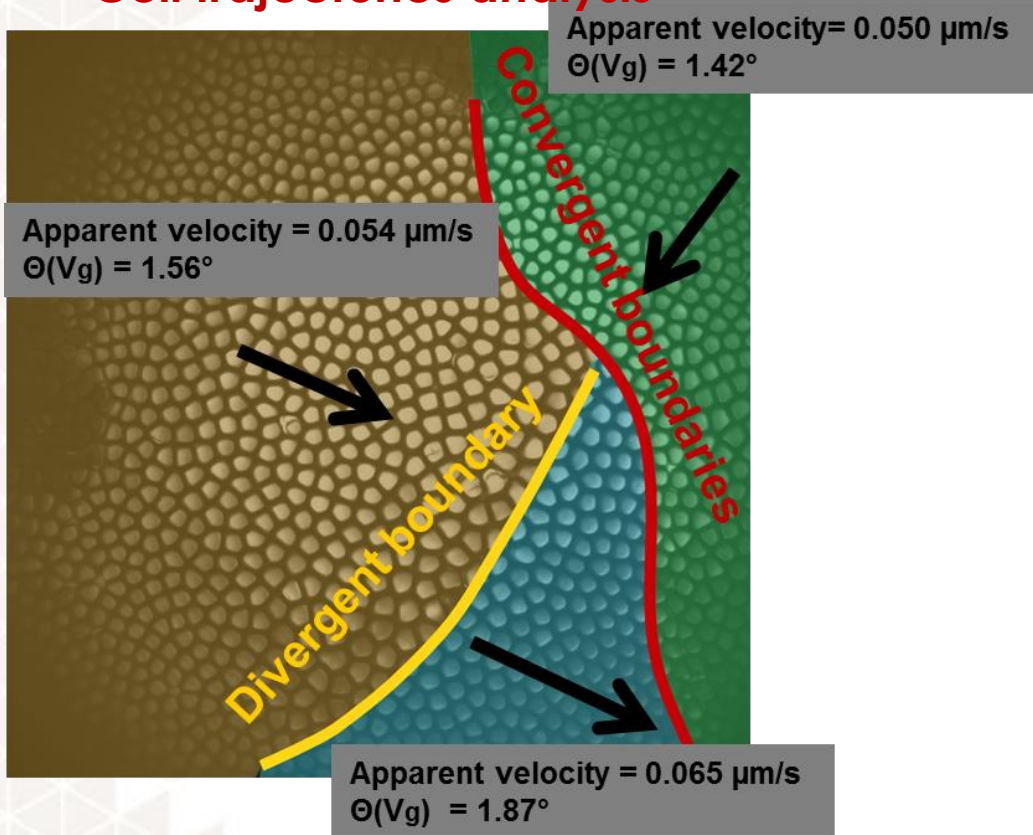
- **All the Sub-grain boundaries are fixed when morphological instability triggers**

500 μm

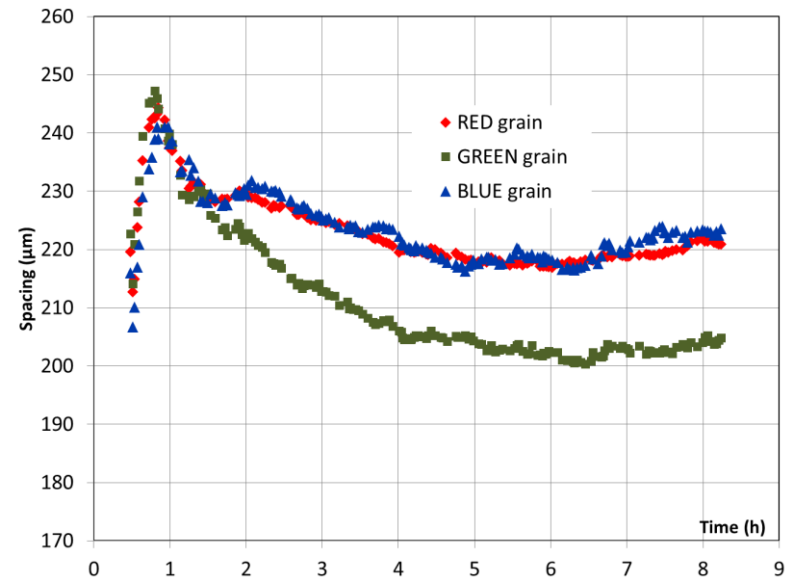
S. Bottin-Rousseau et al. PRB 66 (2012) 4102
G. Faivre et al. CR Phys 14 (2013) 149

Effect of sub-grain on the dynamics of primary spacing

Cell trajectories analysis

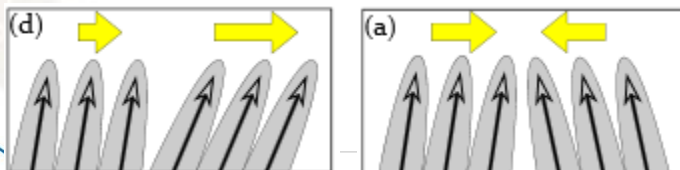


Primary spacing evolution by sub-grain



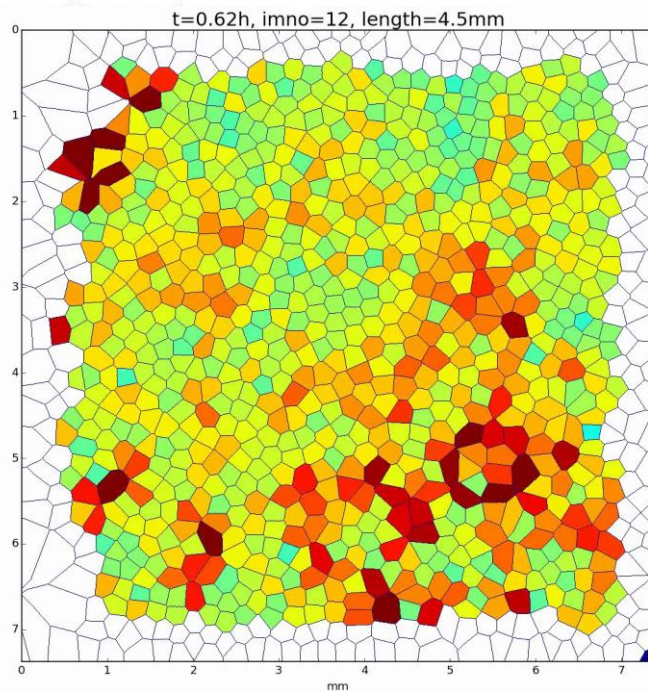
- 3 areas of $\neq V_g$ and $\neq \theta \Rightarrow$ 3 sub-grains
- Divergent and convergent GB

- Noticeable differences of λ evolution depending on sub-grain
- **Cannot be attributed to differences of misorientation ($1.4^\circ < \theta(V_g) < 1.9^\circ$)**



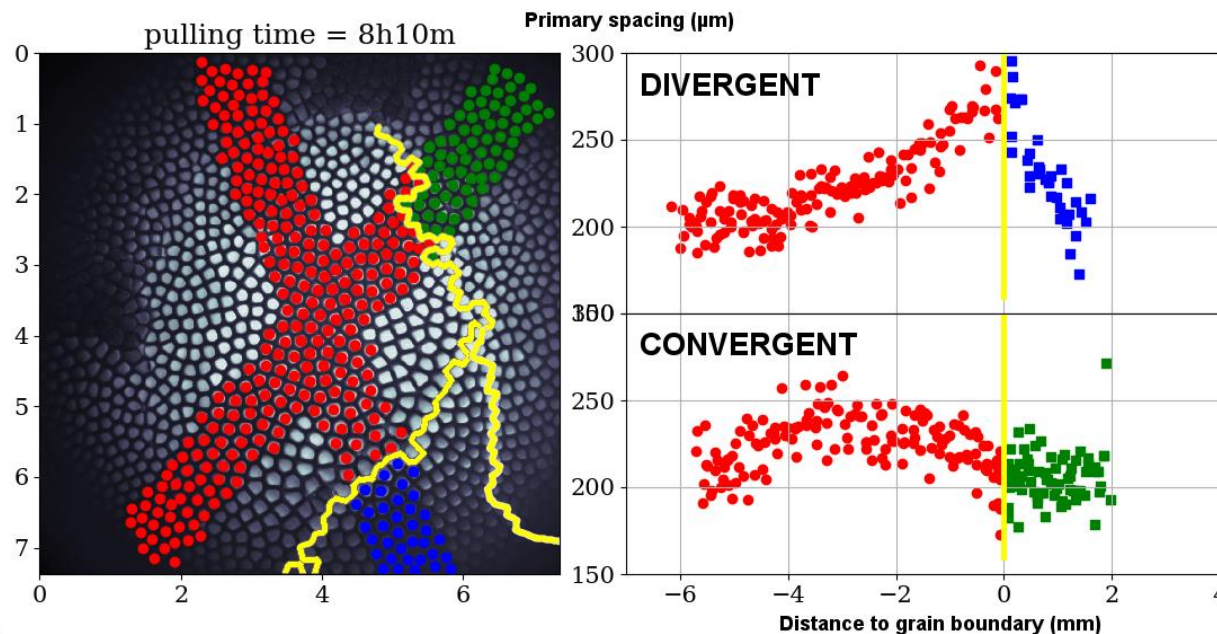
Boundaries configuration

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- **Initially:** large distribution of λ , homogeneously distributed
- **With time:** global decrease of λ except around the divergent GB

Primary spacing map
Duration: 6.5 h

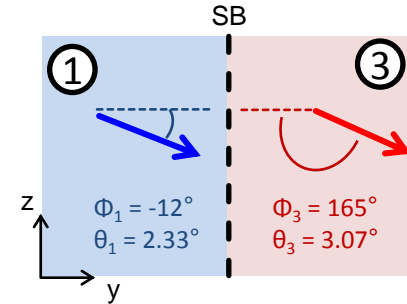
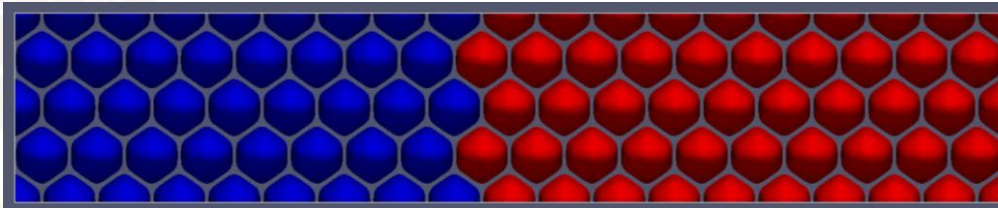


- The evolution of primary spacing strongly depends on the distance to the divergent GB (effect ≈ 20 cells)
- The effect of convergent GB is of shorter distance (≈ 5 cells)

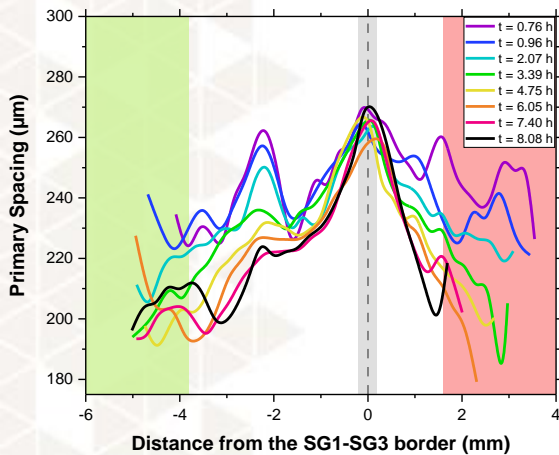
Critical role of boundaries

Divergent SB: comparison experiment and PF

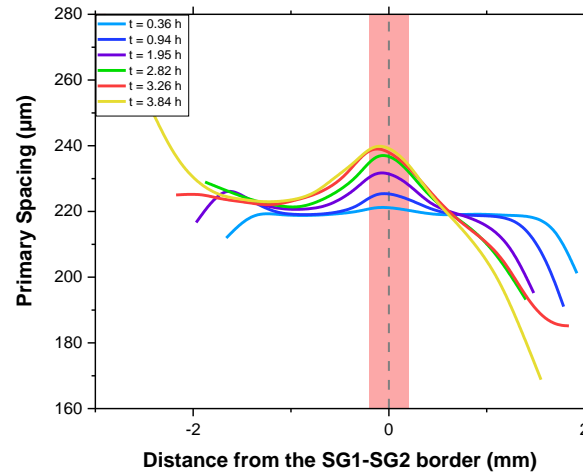
- Target condition: $V_p = 2 \mu\text{m/s}$, $G = 19 \text{ K/cm}$
- Regular hexagonal array ($\lambda = 220 \mu\text{m}$)
- Simulation box: $3960.74 \times 760.14 \mu\text{m}^2$



EXPERIMENT



3D SIMULATION



→ The **plateau λ** on the left is decreasing in experiment, while increasing in the 3D PF simulation

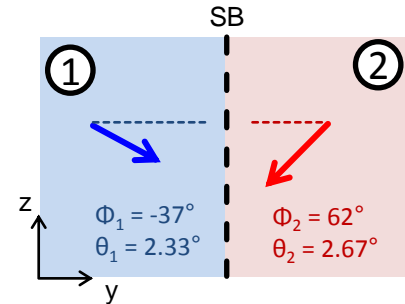
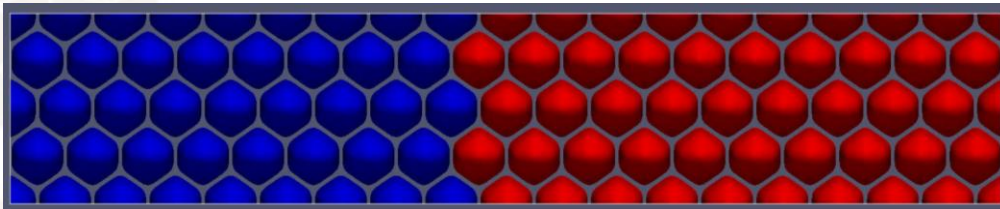
- **Exp:** the source produces cells with smaller λ
- **PF:** the source produces cells with larger λ

→ The **shape of the λ peak** extends in simulation while becomes narrower in experiment

Long distance
effect of sources?

Convergent SB: comparison experiment and PF

- Target condition: $V_p = 2 \mu\text{m/s}$, $G = 19 \text{ K/cm}$
- Regular hexagonal array ($\lambda = 220 \mu\text{m}$)
- Simulation box: $3960.74 \times 760.14 \mu\text{m}^2$



→ Mechanisms along SB

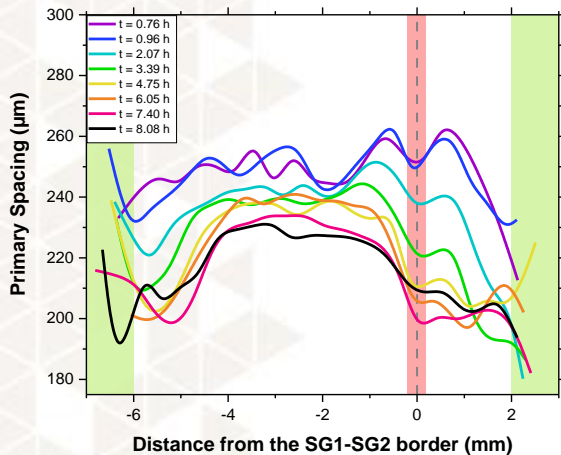
- **Exp:** SB \approx stable, numerous eliminations
- **PF:** incursions

→ The λ at the SB keeps decreasing until reaches the lowest point

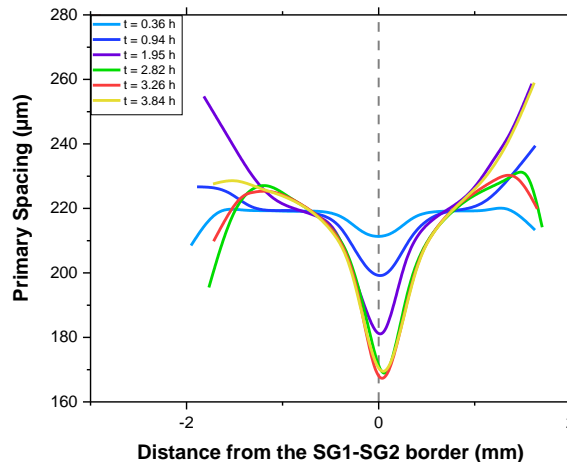
→ The plateau λ on the right side of SB is decreasing in experiment, while increasing in the 3D PF simulation

- **Exp:** the source produces cells with smaller λ
- **PF:** the source produces cells with larger λ

EXPERIMENT



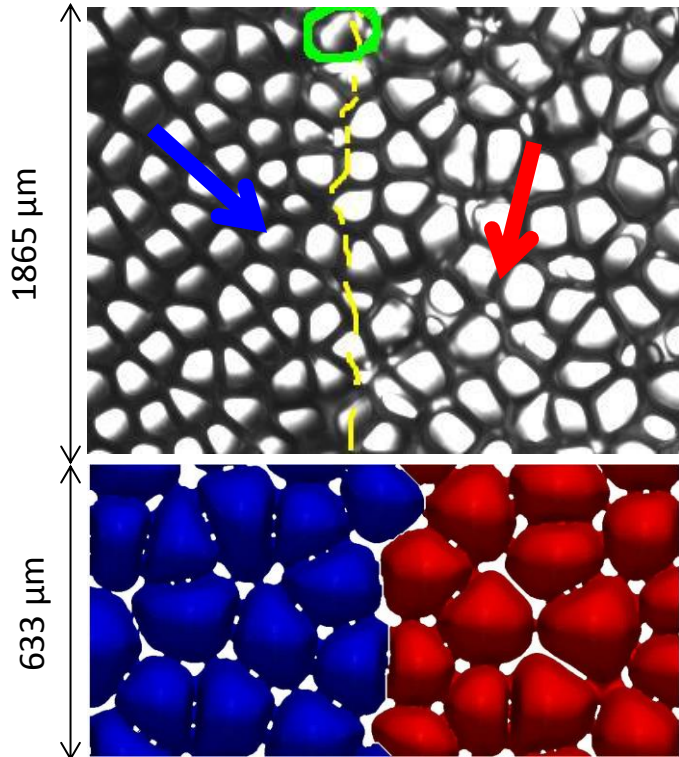
3D SIMULATION



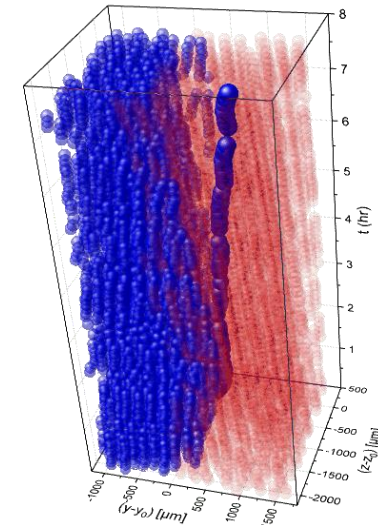
Long distance effect of sources?

Conclusion

- ⇒ **Success of μg experiments:** extended 2D patterns of cells
 - **Benchmark data in diffusive transport mode**
- ⇒ **Critical role of sub-boundaries configuration on primary spacing evolution**
 - *3D phase simulations in progress...*
- ⇒ **1st observation of solitary cells:** 3D phase field simulations
 - **Behavior maps = f(orientation angles)**



Orientation defect

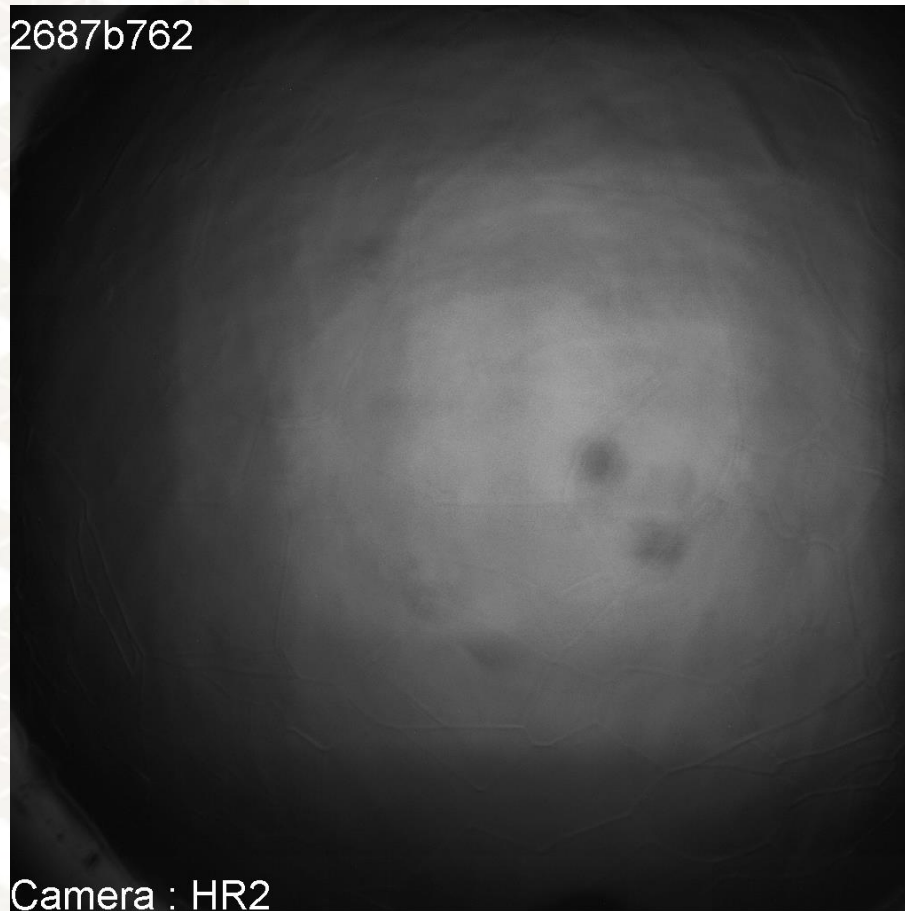


$V_p = 1.5 \mu\text{m/s}$
 $G = 19 \text{ K/cm}$

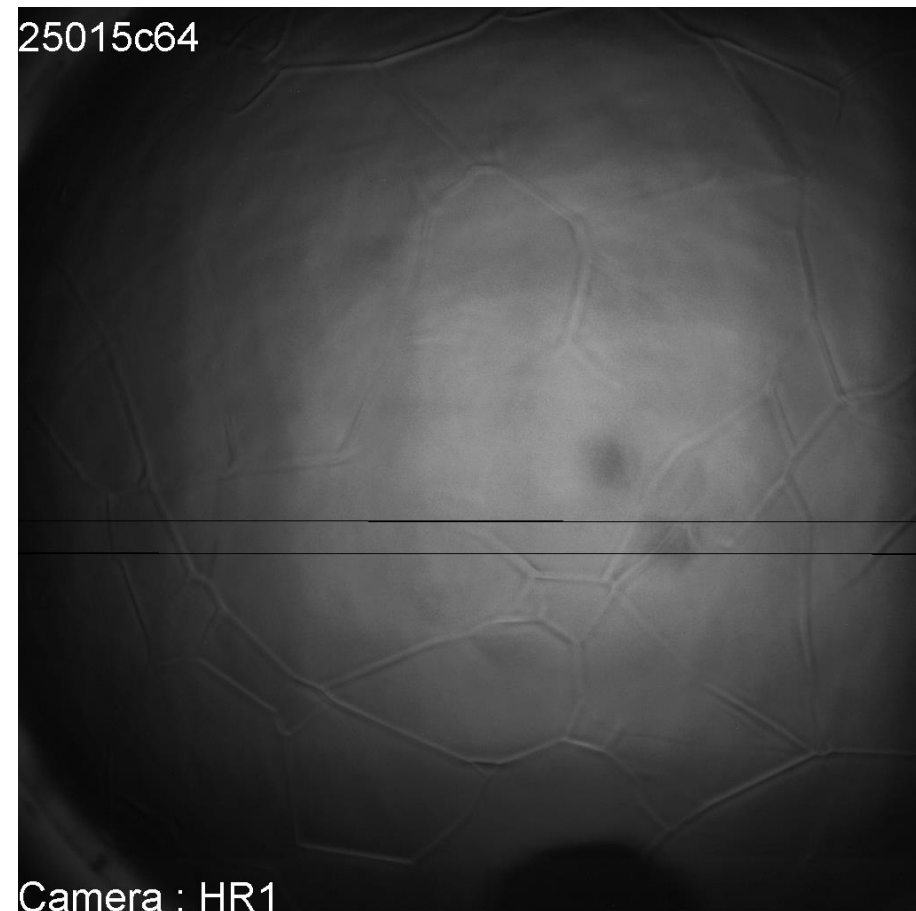
DSI-R: dedicated to the dendritic regime

12

- Increase of concentration \Rightarrow **Dendrites at lower pulling rates**
- Study of **the formation of well-developed dendritic array structures**



$V_1 = 12$ $V_2 = 3$ $\mu\text{m/s}$, $G = 12$ K/cm, 3h28min



$V_1 = 1.5$ $V_2 = 12$ $\mu\text{m/s}$, $G = 12$ K/cm, 6h15min

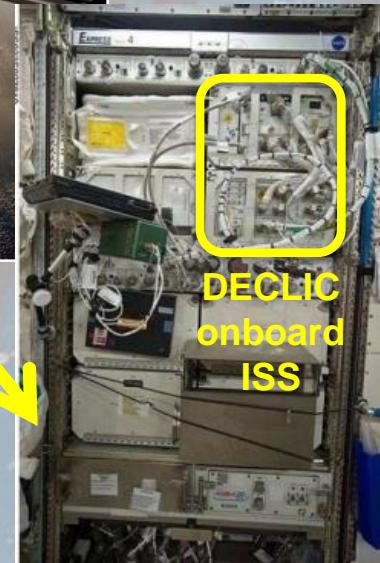
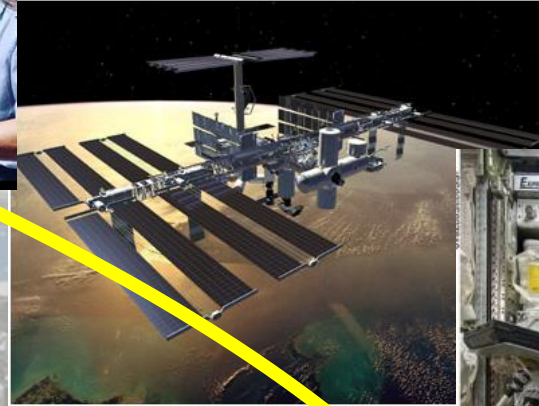
Deepest thanks to
CNES and NASA



Image IM2NP



Image CADMOS-CNES/Sébastien Rouquette



DECLIC
onboard
ISS

*Thank you for
your attention*