Detachment of Tertiary Dendrite Arms during Controlled Directional Solidification in Aluminum – 7 wt% Silicon Alloys: Observations from Ground-based and Microgravity Processed Samples

R.N. Grugel, R. Erdmann, J.R. Van Hoose, S.N. Tewari, and D.R. Poirier

Electron Back Scattered Diffraction results from cross-sections of directionally solidified aluminum – 7wt% silicon alloys unexpectedly revealed tertiary dendrite arms that were detached and mis-oriented from their parent arm. More surprisingly, the same phenomenon was observed in a sample similarly processed in the quiescent microgravity environment aboard the International Space Station (ISS) in support of the joint US-European MICAST investigation. The work presented here includes a brief introduction to MICAST and the directional solidification facilities, and their capabilities, available aboard the ISS. Results from the ground-based and microgravity processed samples are compared and possible mechanisms for the observed tertiary arm detachment are suggested.

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Detachment of Tertiary Dendrite Arms during Controlled Directional Solidification in Aluminum – 7 wt% Silicon Alloys: Observations from Ground-based and Microgravity Processed Samples

Richard N. Grugel – Marshall Space Flight Center
Robert Erdman – University of Arizona
James R. Van Hoose – Siemens Corporation
Surendra Tewari – Cleveland State University
David Poirier – University of Arizona

In View of Work Subsequent to Abstract Submission, the New title is:

Spurious Dendrite Arm Orientations during Controlled Directional Solidification in Aluminum – 7 wt% Silicon Alloys: Comparison of Ground-based and Microgravity Processed Samples

This Investigation is a Collaborative Effort with the European Space Agency (ESA) Program:

Microstructure Formation in Castings of Technical Alloys under Diffusive and Magnetically Controlled Convective Conditions (MICAST)

The MICAST Microgravity Research Program Focuses on:

- A systematic analysis of the effect of convection on the microstructural evolution in cast Al-alloys.
- Experiments that are carried out under well defined processing conditions.
- Sample analysis using advanced diagnostics and theoretical modeling.

→The MICAST team investigates binary, ternary and commercial alloys based on the Al-Si system.

Intent

Conduct a Thorough Ground-based Investigation

- Utilize Aluminum – 7wt. % Silicon Alloys
  - ♦ Directionally Solidify Samples having an Initial Aligned Dendritic Array
  - ♦ Evaluate the Dendritic Microstructure (λ_1, λ_2, λ_3, d) as a function of the Steady-State Processing Conditions (V, G, C_o)

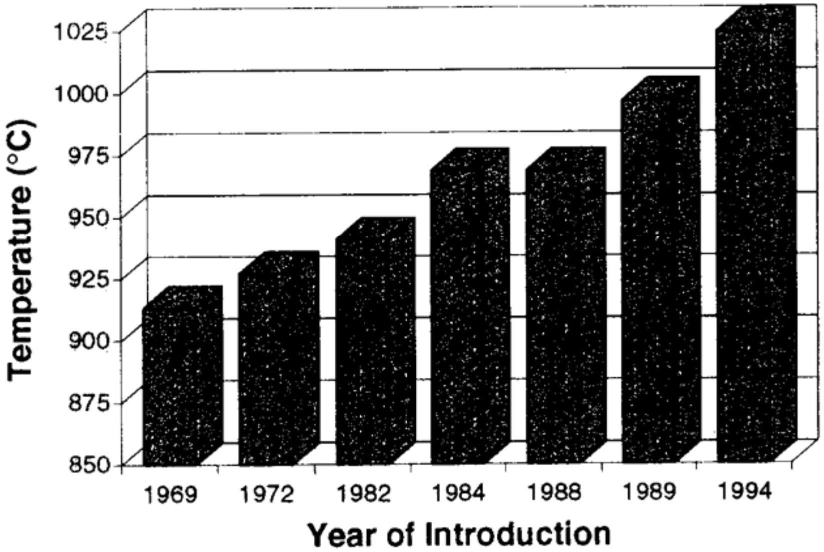
Use the Above for Comparison to Limited # of DS μ*g* Samples

- Investigate the Role of Gravity on
  - ♦ Microstructural Development, Spacing
  - ♦ Macrosegregation, Defect Generation

Outline

- Expectations
- Ground-based Results
- Microgravity Results
- Comparative Comments

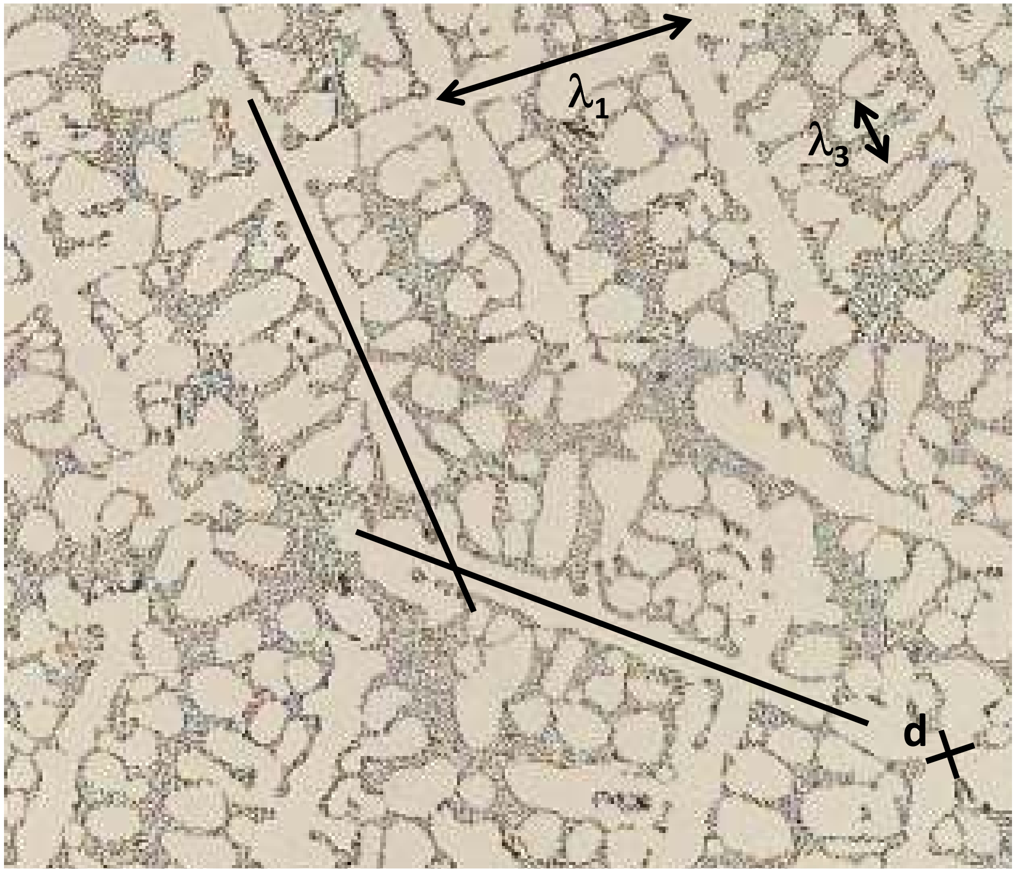
Why Directional Solidification?



Bar chart showing the changes in temperature capability of cast turbine blade alloys as a function of time. The first three alloys in the series are equiaxed, conventional cast. The next one is a monocrystal alloy. The next is a directionally solidified alloy with comparable performance at lower cost. The last two are monocrystal alloys.

J.C. Williams: Phil. Trans. R. Soc. Lond. A (1995) 351, p. 435.

Microstructural Evaluation



λ_1, Primary Dendrite Arm Spacing

λ_3, Tertiary Dendrite Arm Spacing

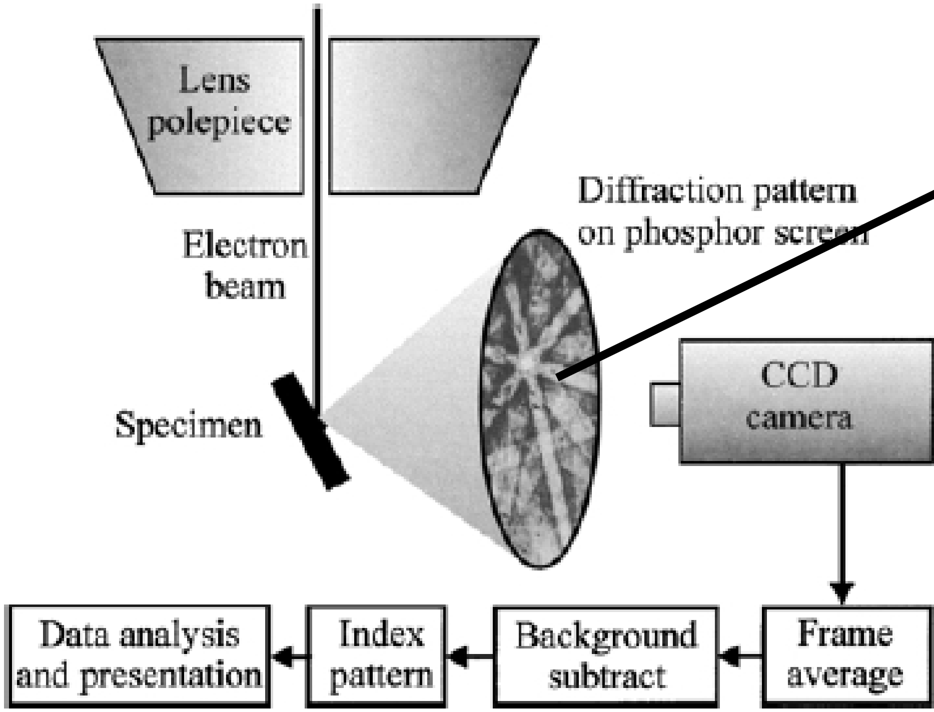
d, Primary Dendrite Trunk Diameter

Relative Dendrite Grain Orientation

Statistically Compile and Relate to Solidification Processing Conditions of:

- Growth Velocity (V)
- Temperature Gradient (G)
- Alloy Composition (C_o)

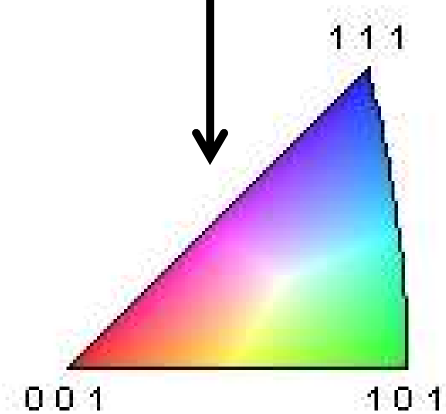
Electron Backscattered Diffraction (EBSD) as an Analysis Technique



Schematic of a typical EBSD set-up

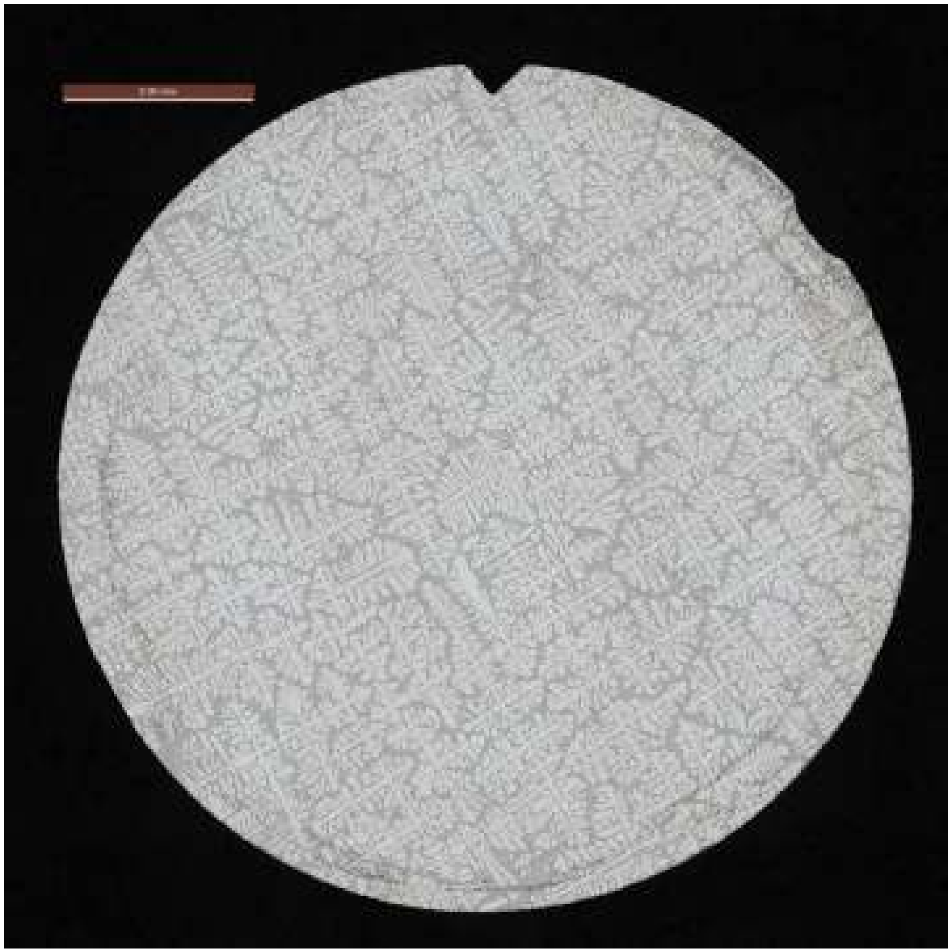
F. J. HUMPHREYS: Journal Of Materials Science 36 (2001) 3833 – 3854

Pattern Corresponds to a Crystal Orientation



Orientation can be Represented as a Color

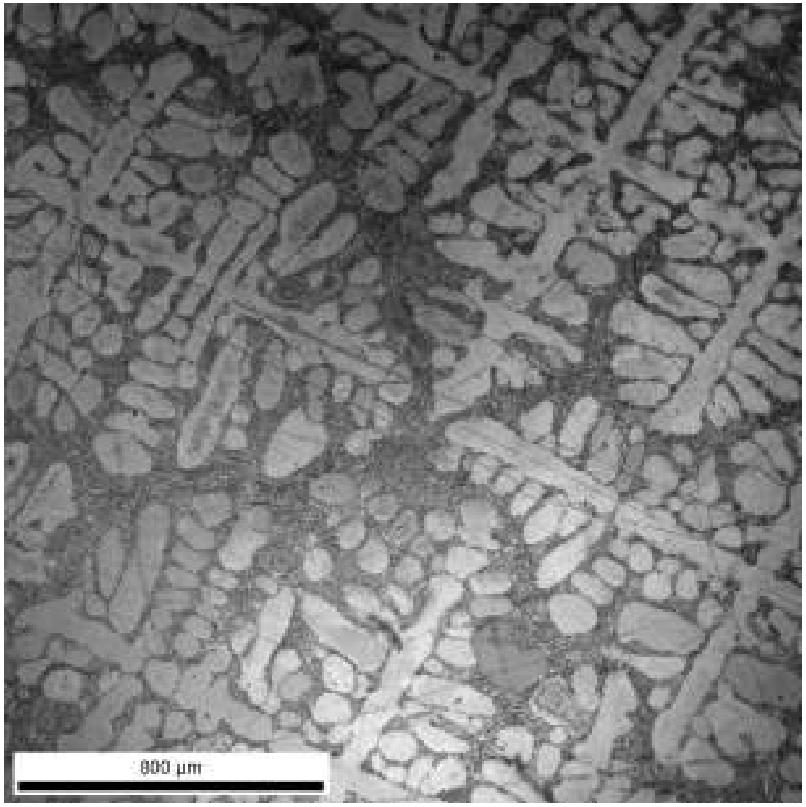
Ground-based Results

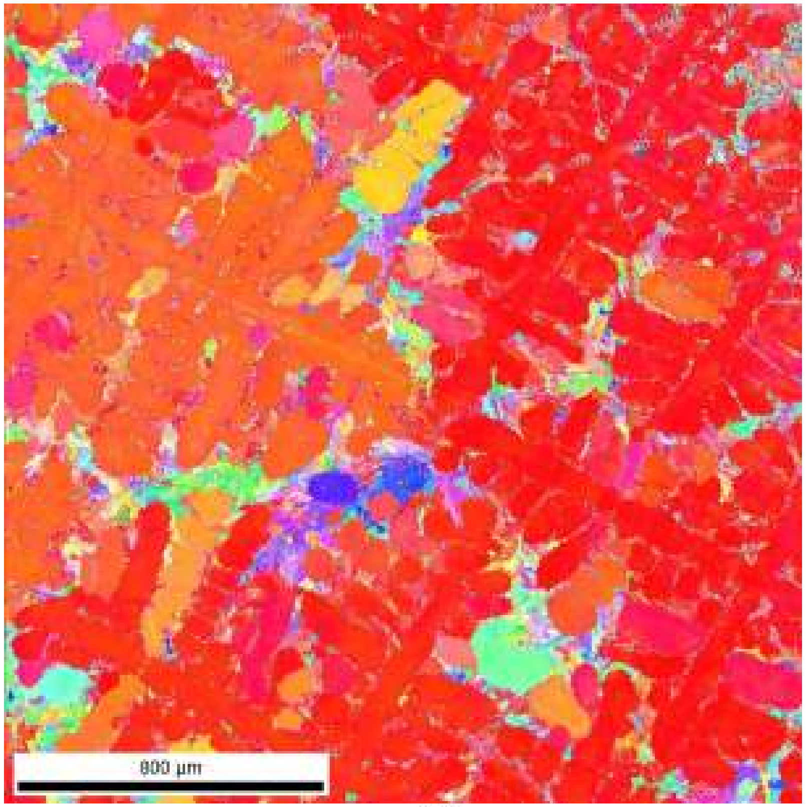


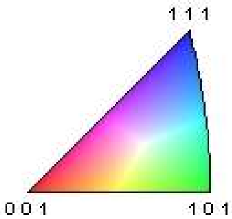
Aluminum – 7wt. % Si
Growth Velocity = 31μm s^{-1}
Temperature Gradient = 40K cm^{-1}

1) **Build up a Data Base**
   - **Establish Spacing Relationships/Trends**
   - **Compare to Microgravity Results**
2) **Use as Seed Crystals for μ*g* Samples**

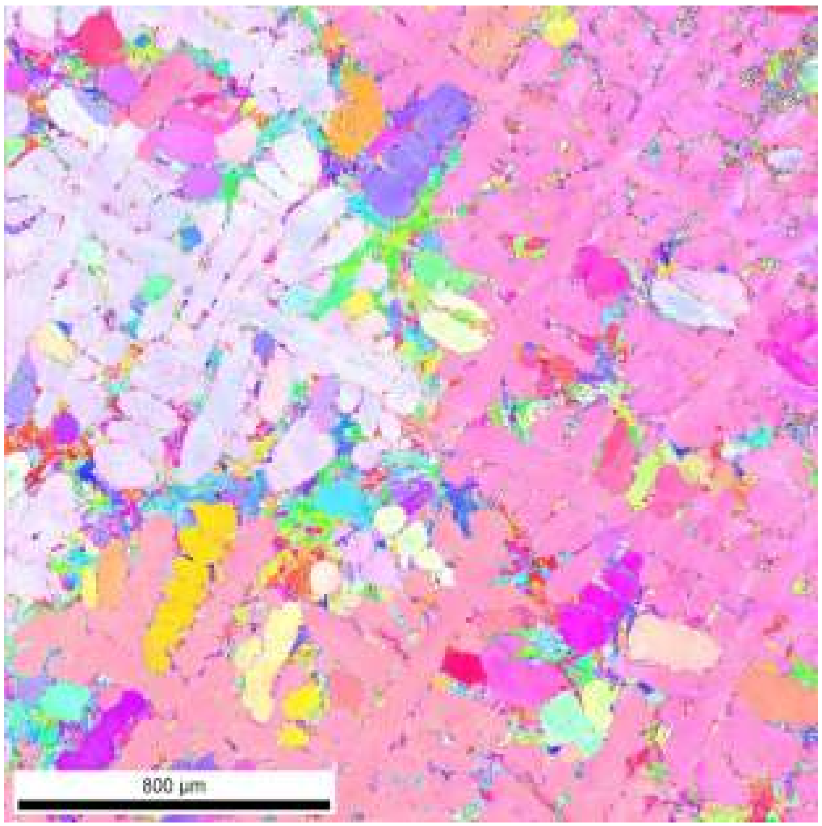
Ground-based Results







Ground-based Results



Observations

● Primary Dendrites not All Aligned in <100> Direction

● Many Tertiary Arms have "Spurious" Orientations

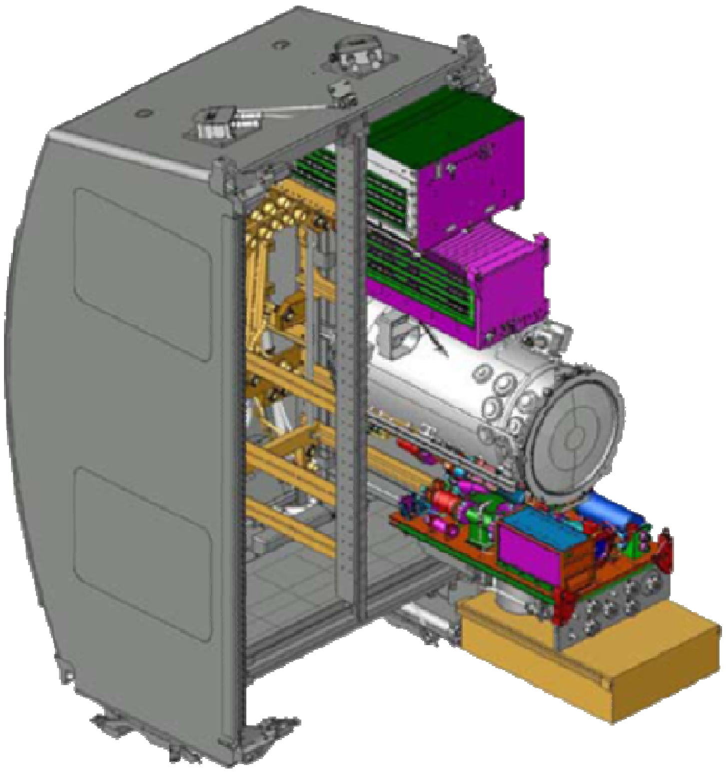
Rationalization

● Tough to get a Single <100> Dendritic Array

● Tertiary Arms Dissociated (Maybe Deformed) From and Rotated with Respect to Secondary Branches due to Local Convection

- ♦ Well Documented in the Literature
- ♦ Eliminated in Microgravity

Microgravity Processing



Microgravity Science Research Facility (MSRF) Aboard the ISS

Solidification Furnace with Quench (SQF) Insert

Sample Cartridge

Solidification Processing of Dendritic Alloys in a Microgravity Environment

Expectations

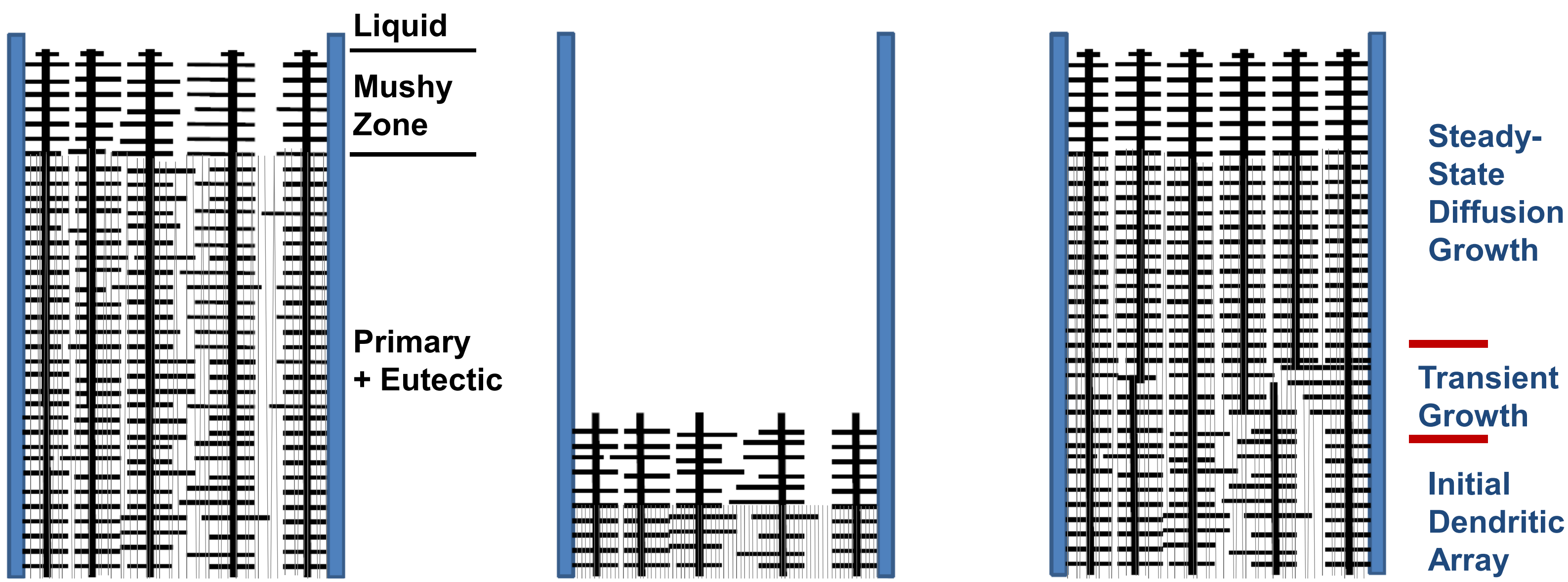
Advantages: Minimize Thermo-Solutal Convection
Minimize Buoyancy Effects

Intent: Produce Segregation Free Samples Grown Strictly by Heat Transfer and Solute Diffusion

Purpose: Better Understand the Relationship between Processing – Microstructural Development

Application: Maximize Material Properties

Ideal Schematic Microgravity Processing Scenario



1*g* Directionally Solidified Dendritic "Seed" Crystal

- ↑ Single Orientation Dendritic Array
- ↓ Non-Uniform Arm Spacing
- ↓ Segregation

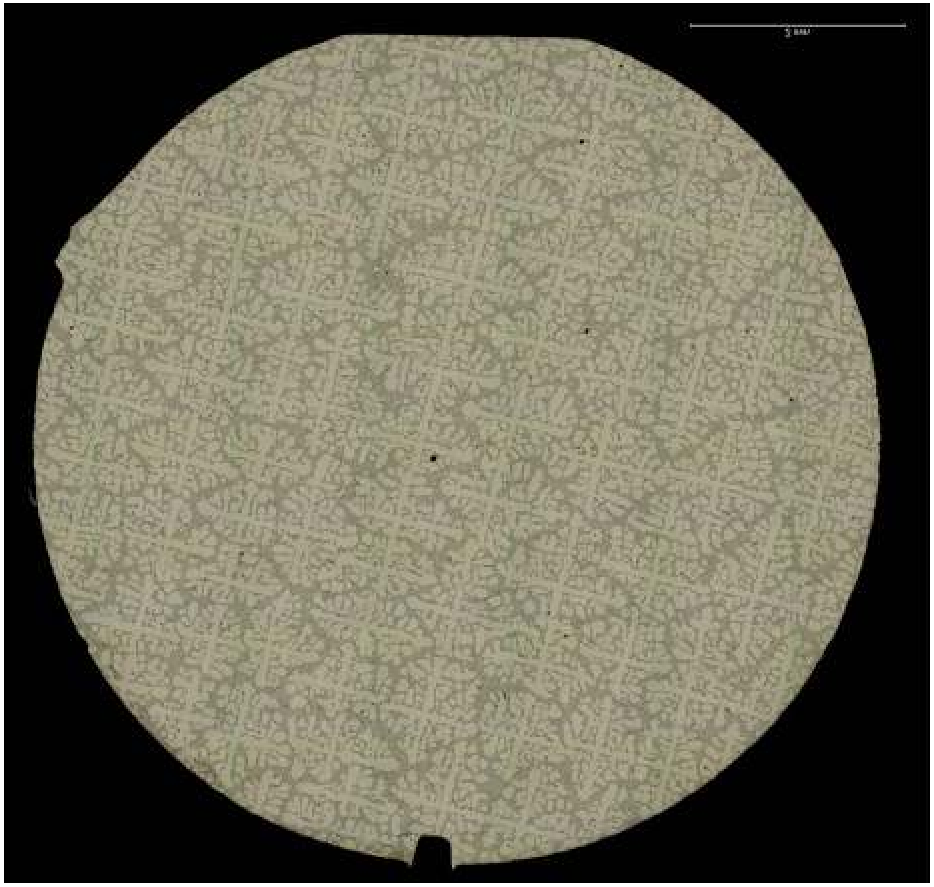
Melt Back of Dendritic Array In Microgravity (Prior to initiating controlled directional solidification)

Directional Solidification in Microgravity

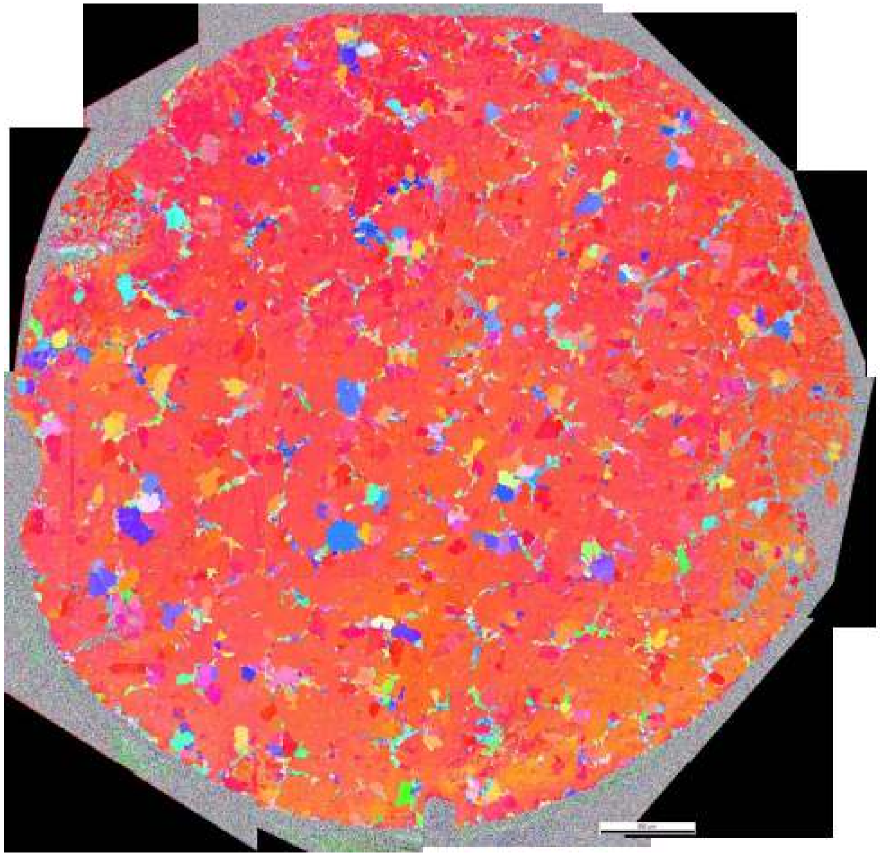
- ↑ Single Orientation Dendritic Array
- ↑ Uniform Dendrite Arm Spacing
- ↑ No Segregation

Steady State Results Meet Expectations

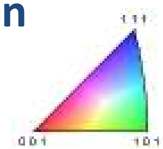
Microgravity Processing



MICAST 7-1 Ground Processed Seed Crystal
Al – 7wt. % Si, V = 20μm s^{-1} , G = 40K cm^{-1}

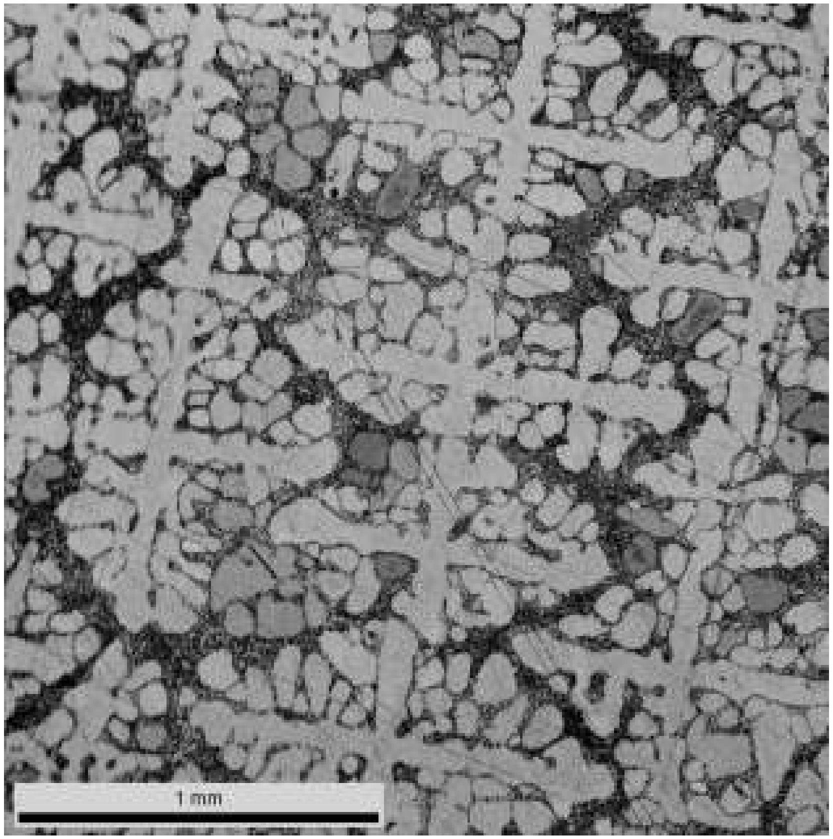


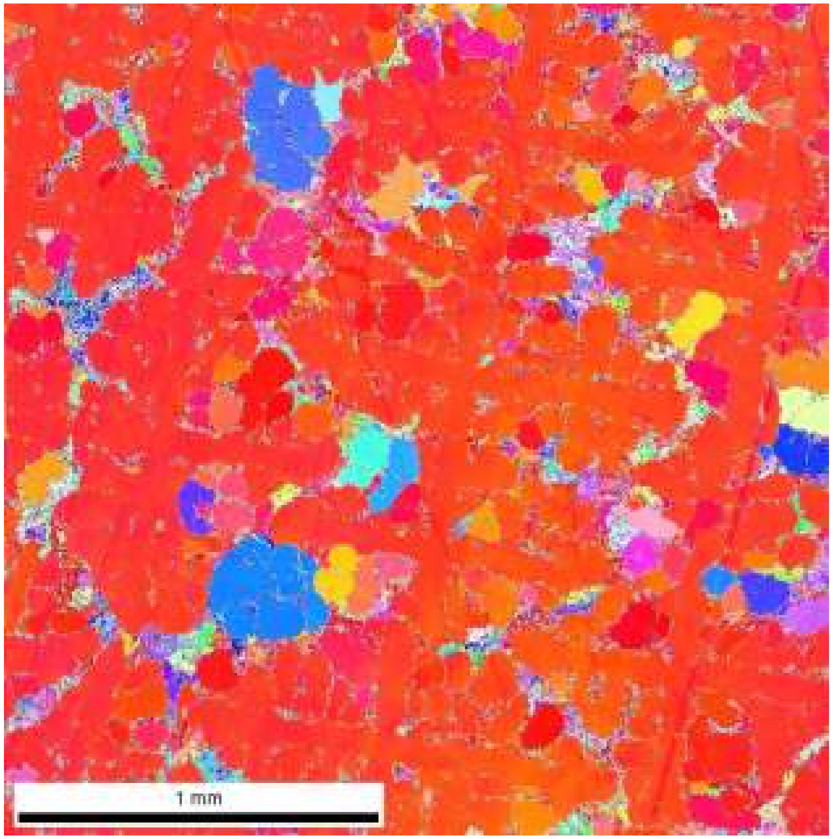
MICAST 7-1 Composite EBSD Scan



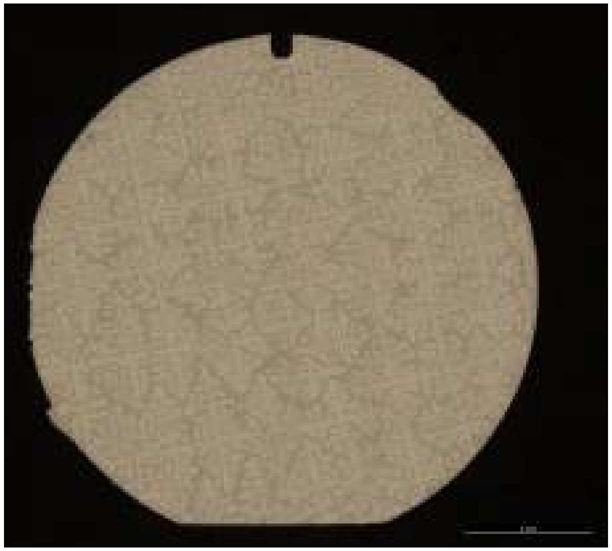
Microgravity Processing

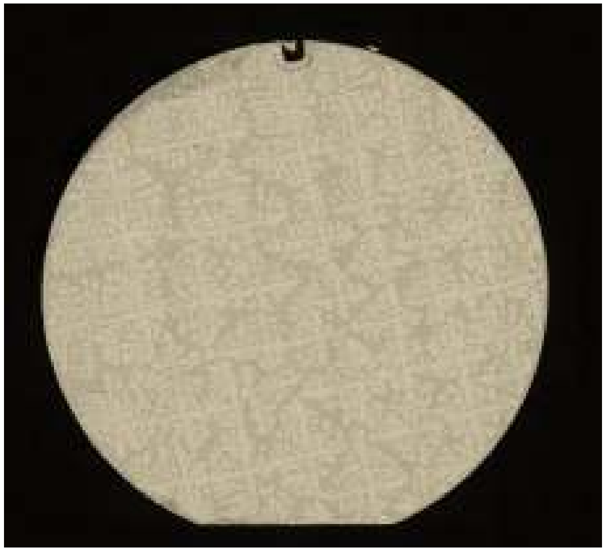
MICAST 7-1 Ground Processed Seed Crystal

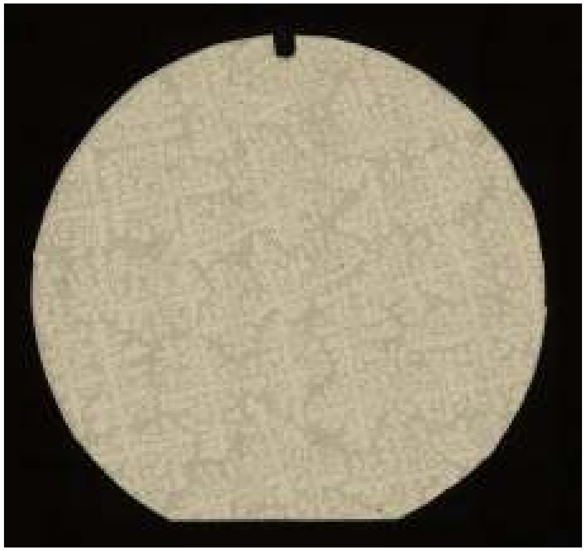




Processing in Microgravity
(Steady-State Growth Conditions)

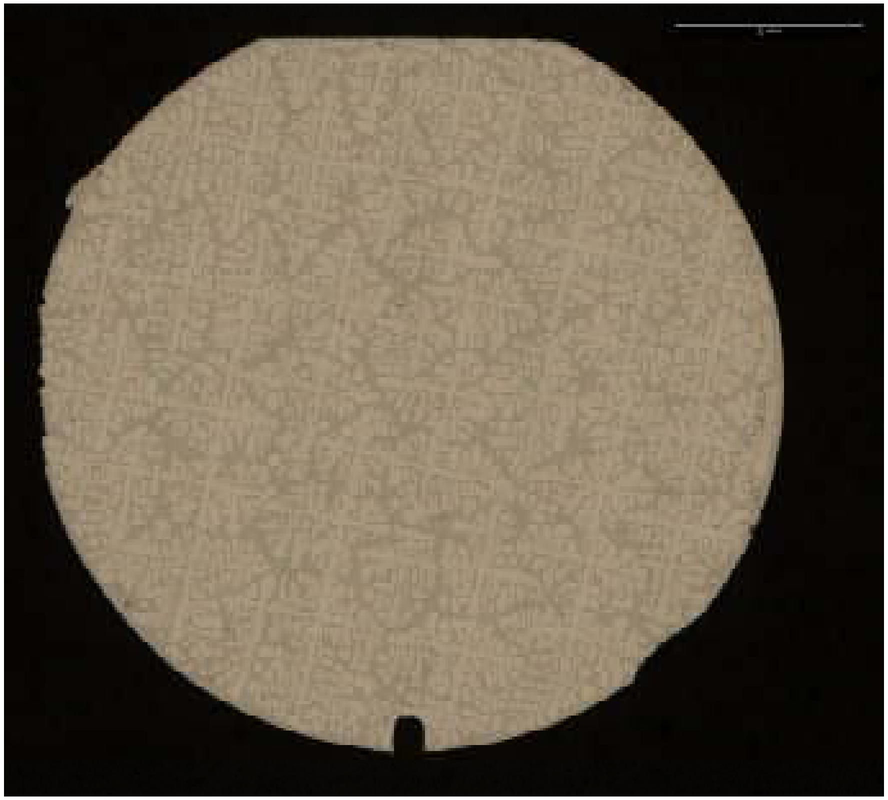

MICAST7 – 3T (20µms^{-1}, G = 28K cm^{-1})

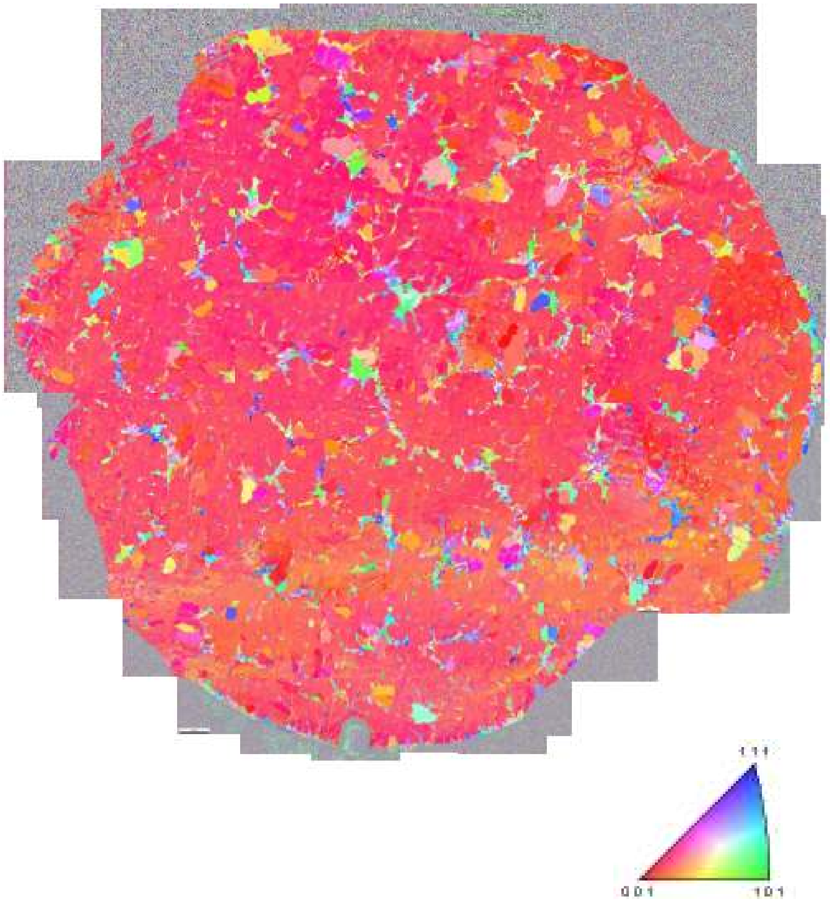

MICAST7 – 4T (20µm s^{-1} → 10µm s^{-1})


MICAST7 – 5T (10µm s^{-1})

Processing in Microgravity

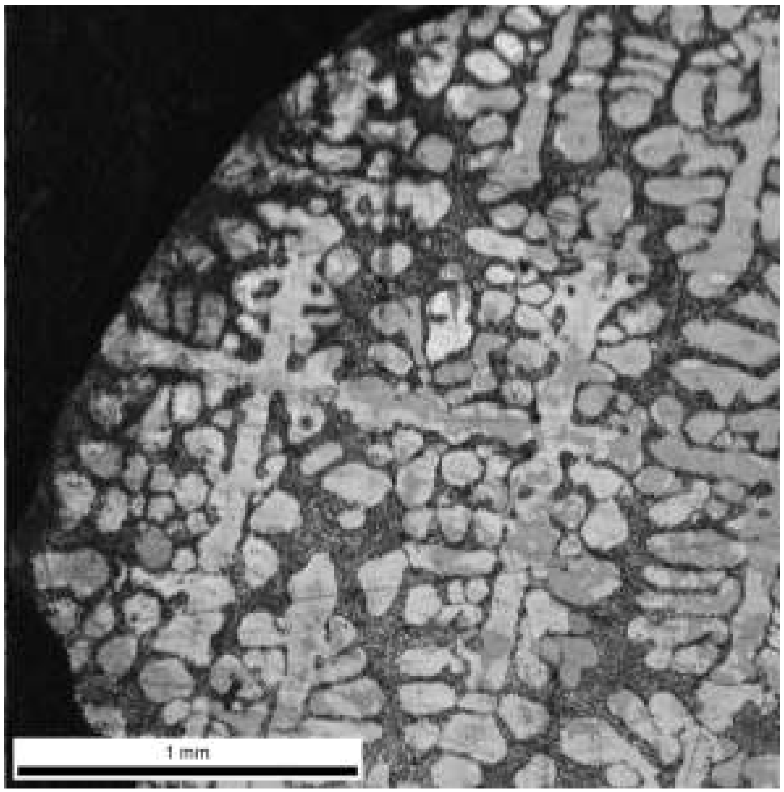
MICAST7 – 3T (20µm s^{-1}, G = 28K cm^{-1})

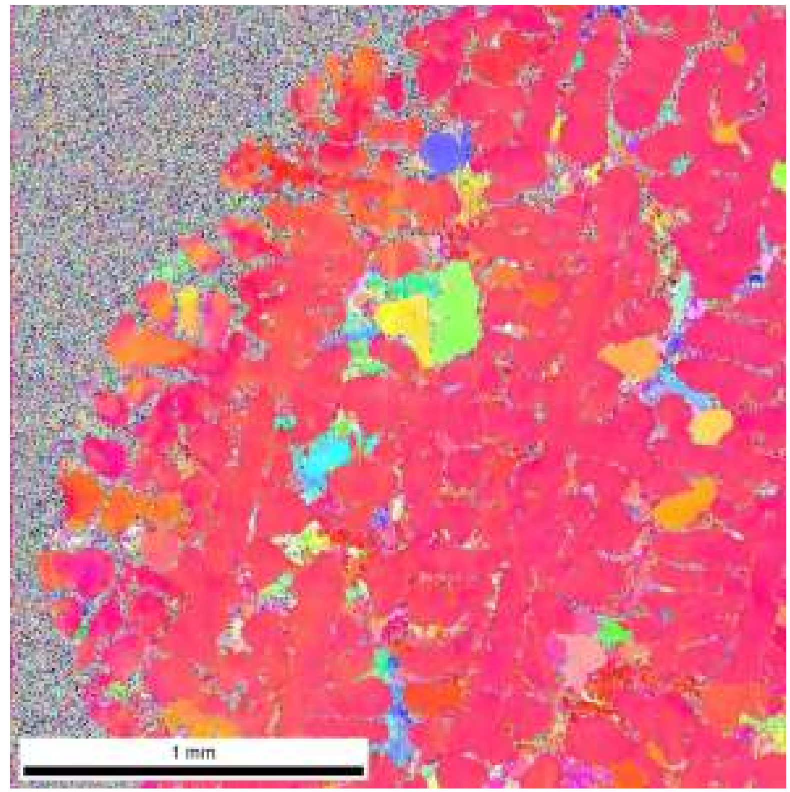




Processing in Microgravity

MICAST7 – 3T (20µm s^{-1}, G = 28K cm^{-1})





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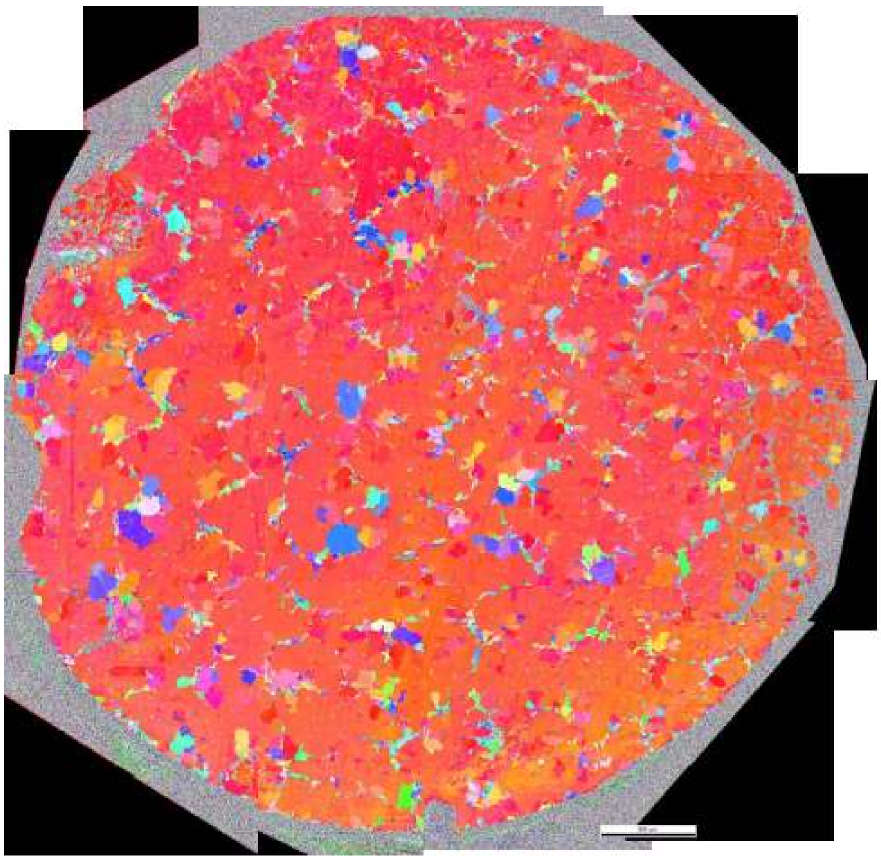
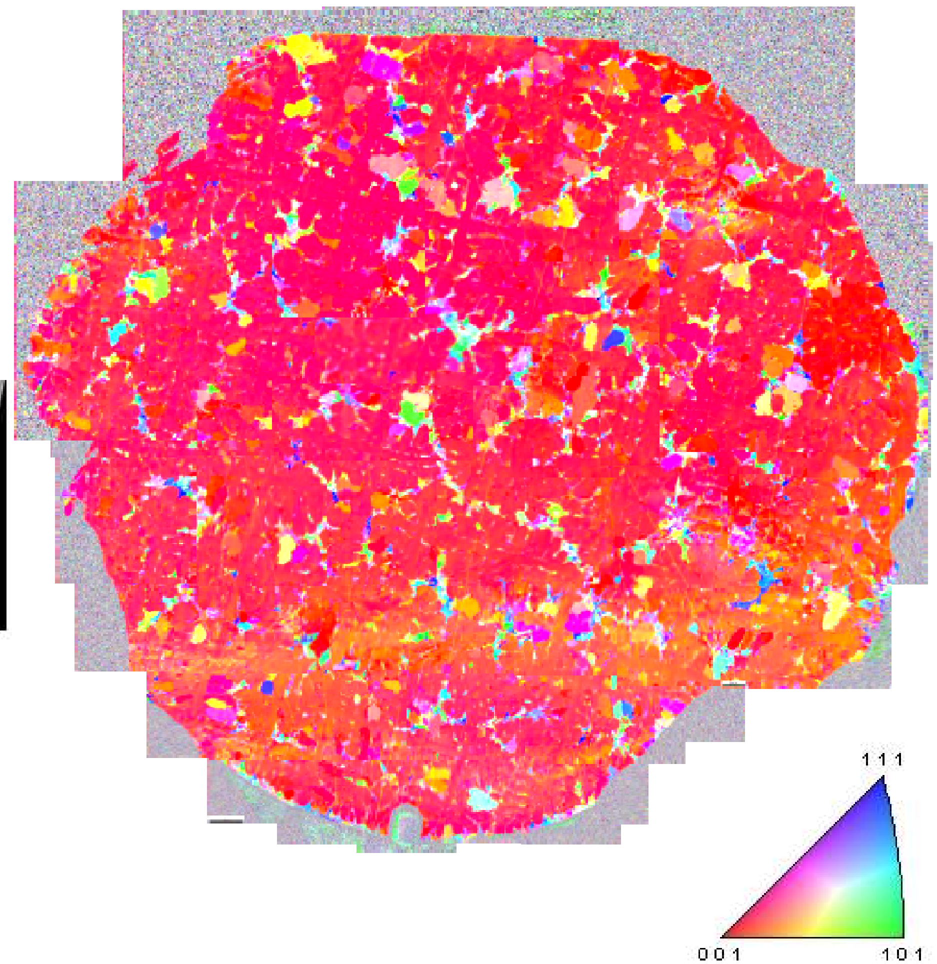
MICAST 7-1 Ground Processed Seed Crystal
Al – 7wt. % Si, V = 20μm s^{-1} , G = 40K cm^{-1}

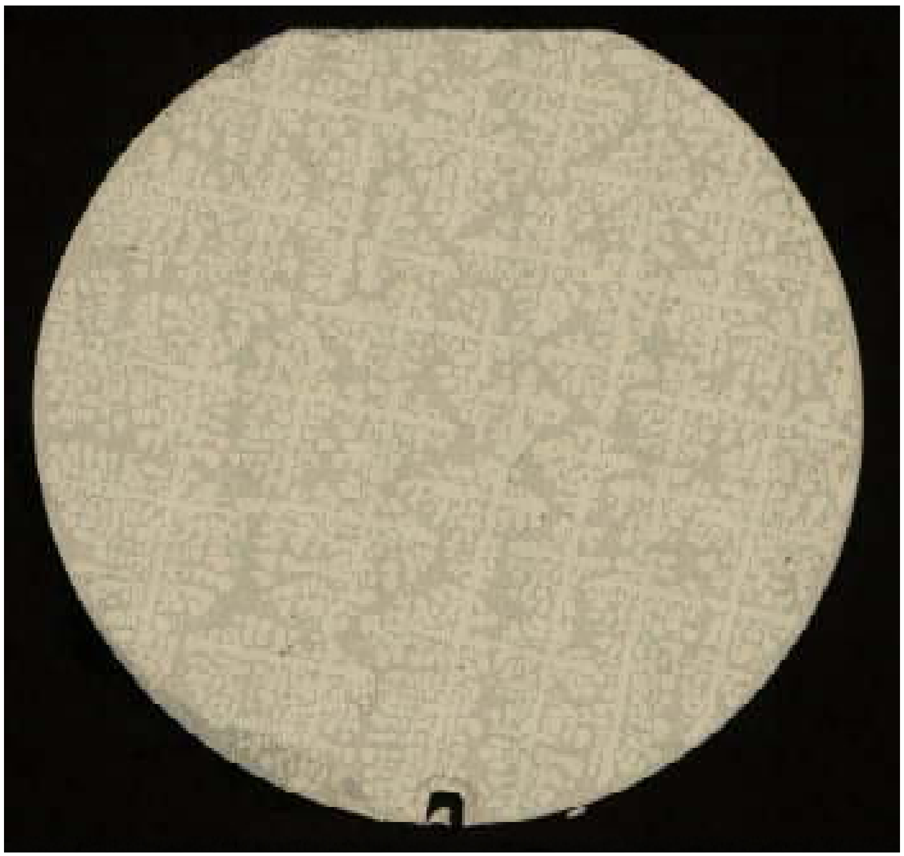


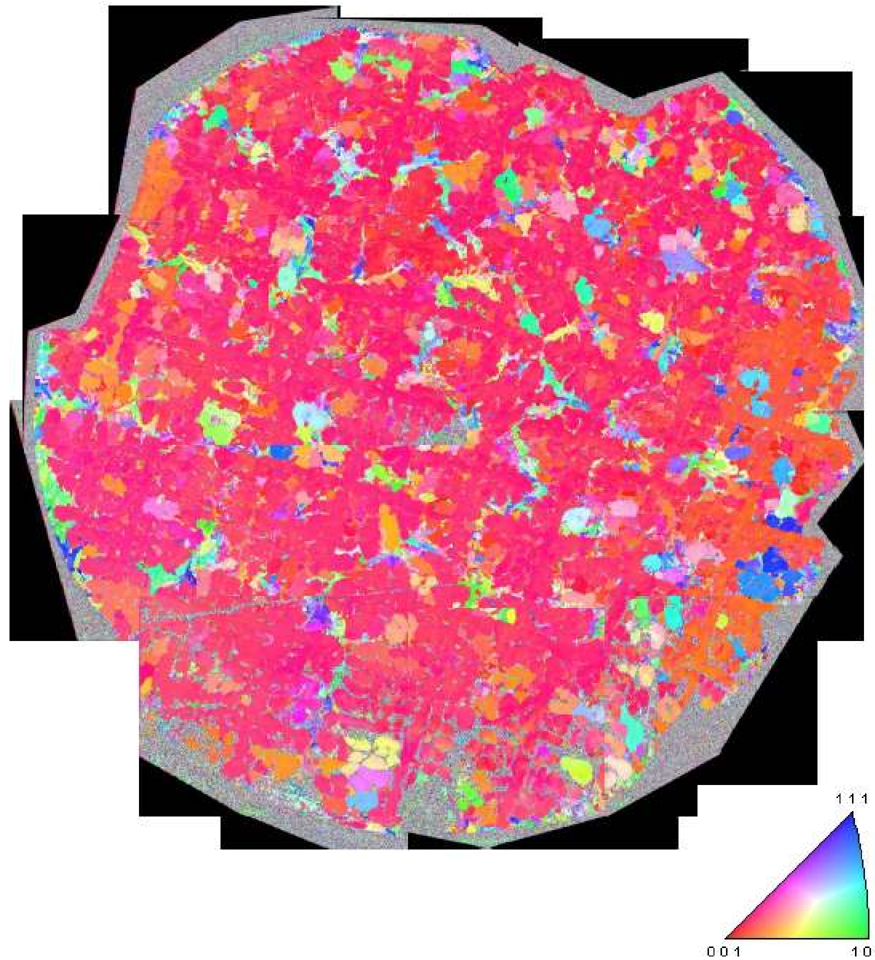
MICAST7 – 3T
(20μm s^{-1}, G = 28K cm^{-1})



Processing in Microgravity

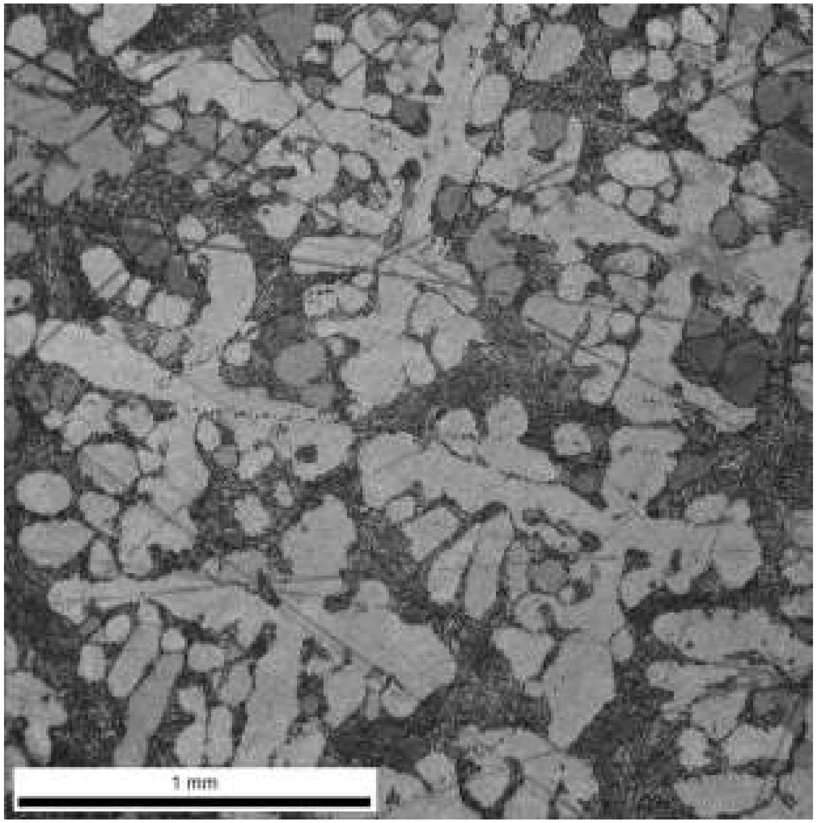
MICAST7 – 4T (20μm s^{-1} → 10μm s^{-1}, G = 28K cm^{-1})

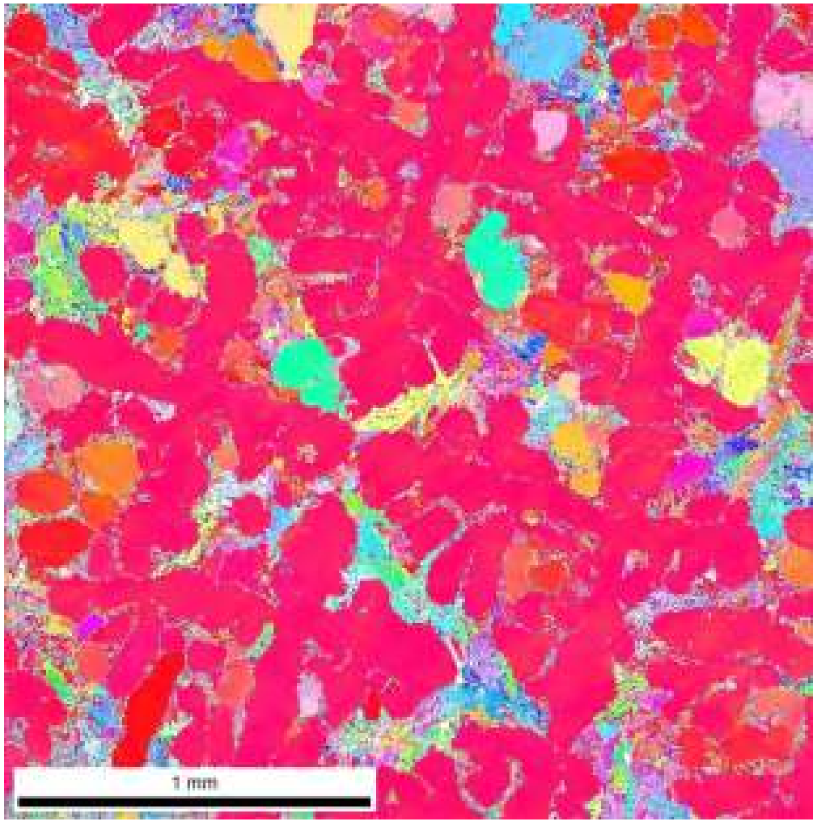




Processing in Microgravity

MICAST7 – 4T (20μm $s^{-1} \rightarrow$ 10μm s^{-1}, G = 28K cm^{-1})





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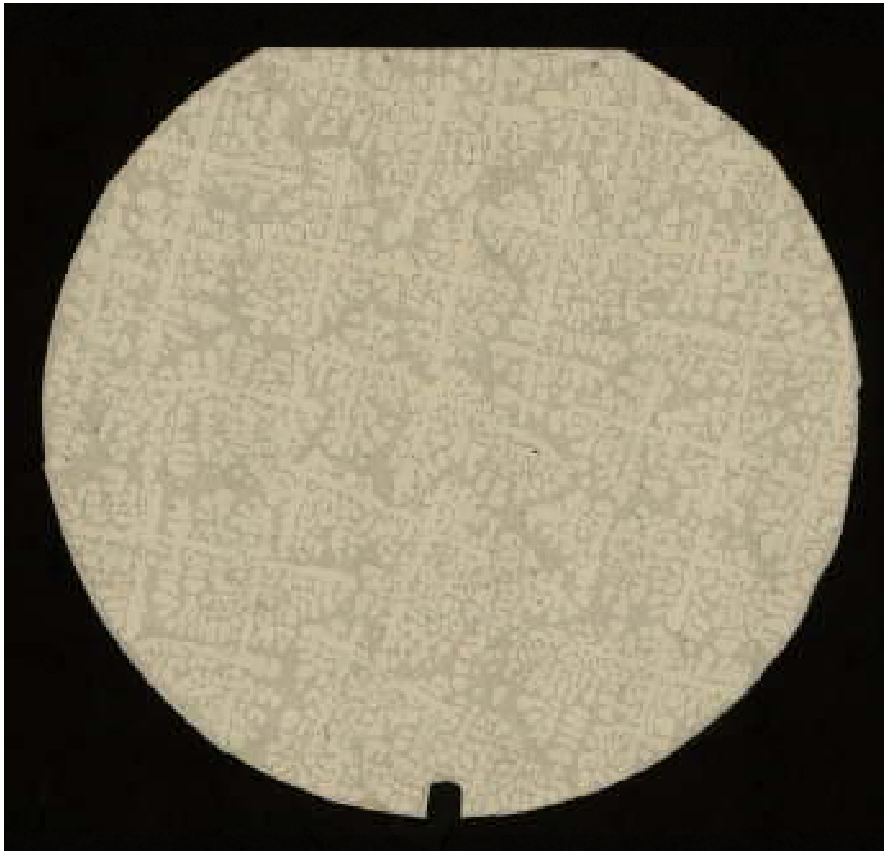
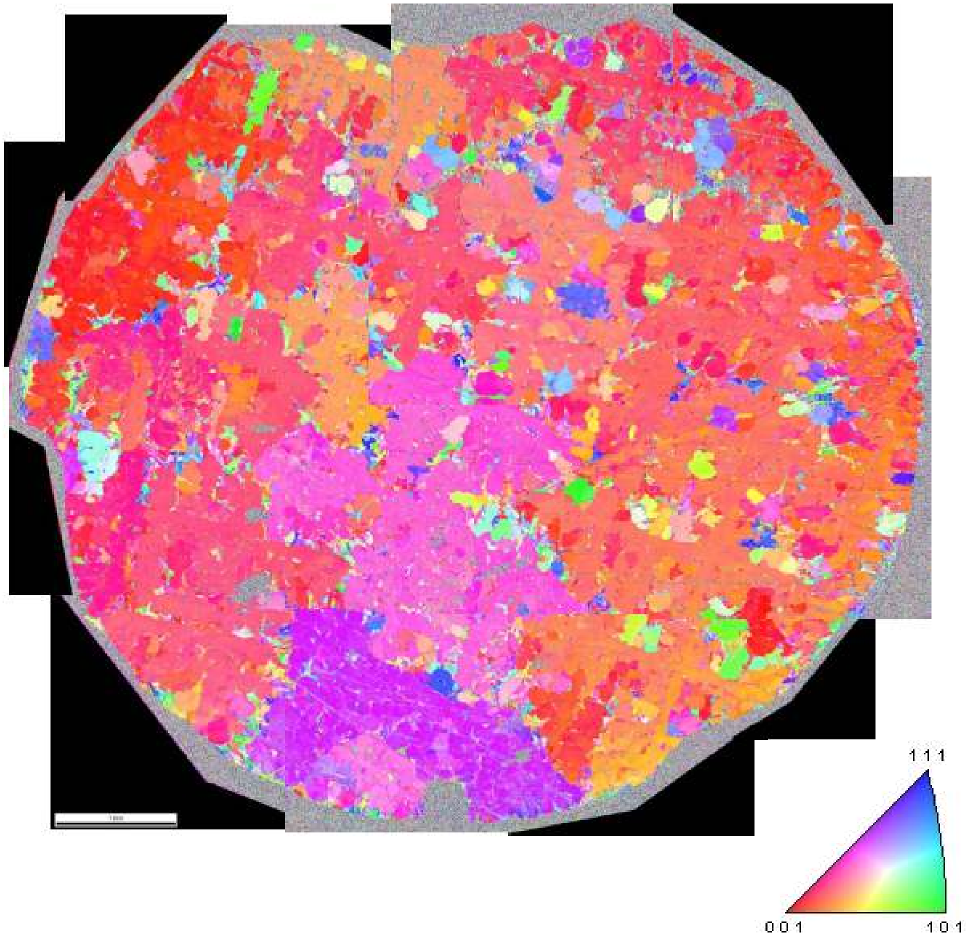
Processing in Microgravity

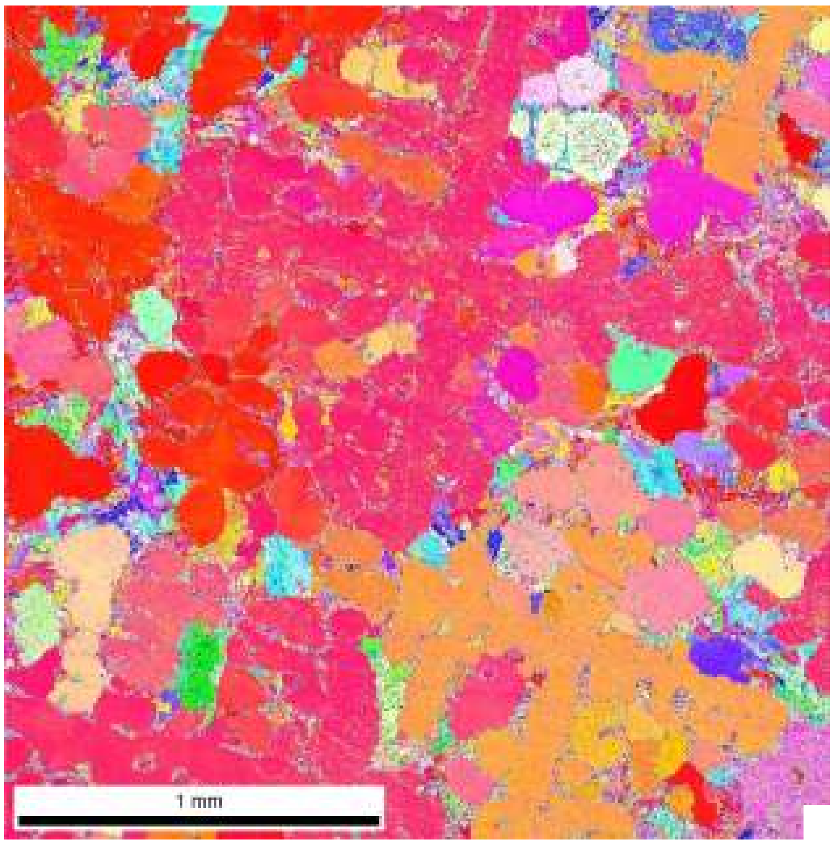
MICAST7 – 5T (10µm s^{-1}, G = 28K cm^{-1})

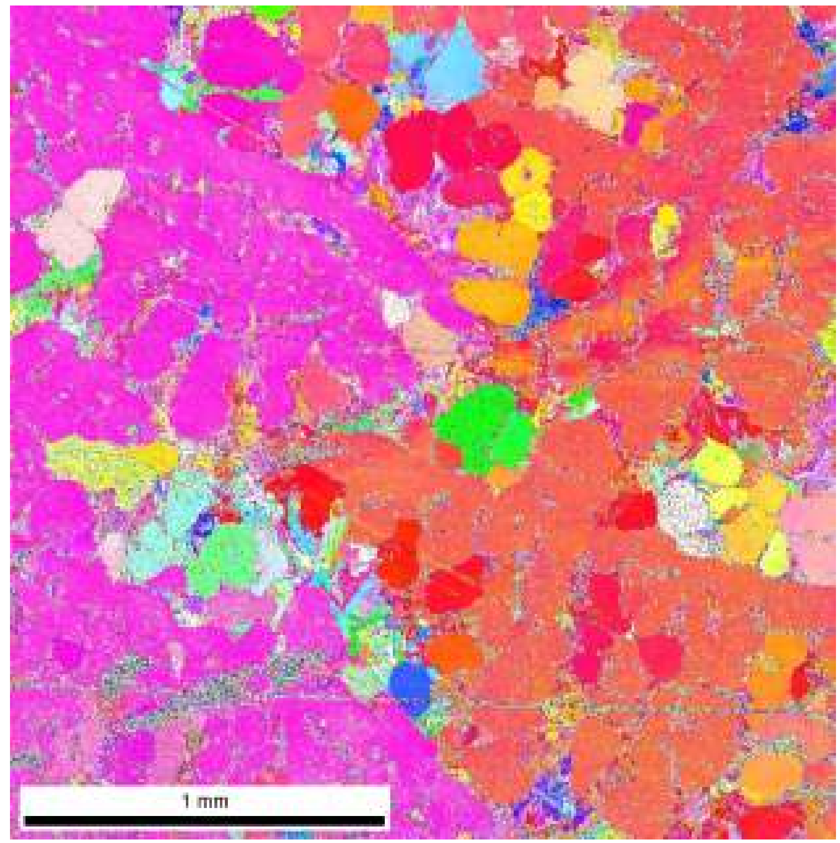


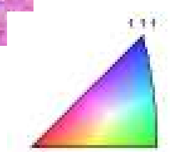


Processing in Microgravity

MICAST7 – 5T (10µm s^{-1}, G = 28K cm^{-1})







Interim Summary

1) Seed Crystal: Very Good Alignment, Some Spurious Grains/Arms

2) 20µm s^{-1}: Very Good (Better) Alignment, Less Spurious Grains

3) Transition, 20µm $s^{-1} \rightarrow$ 10µm s^{-1}: Dendrites Coarsening, Still Good Alignment, Increased Spurious Grains, Explainable

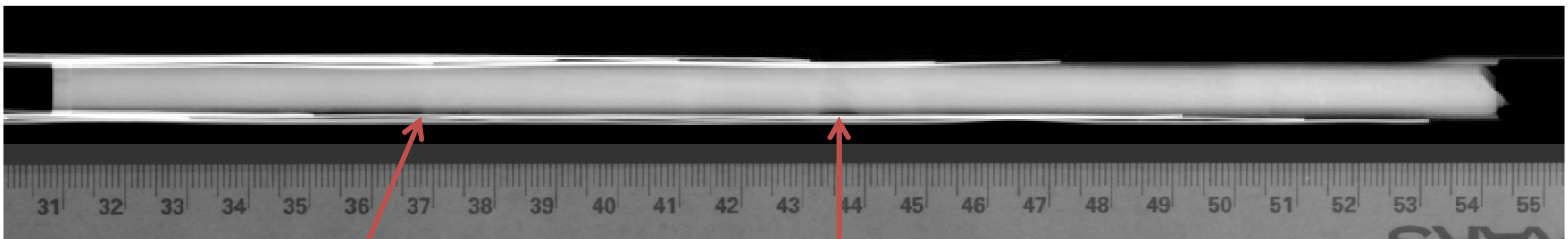
4) 10µm s^{-1}: Very Poor Alignment, Very Many Spurious Grains

4) WHY?

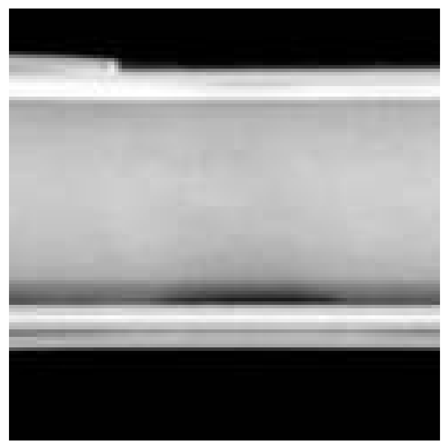
- Consequence of the Transition not Reaching Steady-State
- Locally Induced Solute Concentration Effects
- External Influence

External Influence – Look at the Sample Assembly

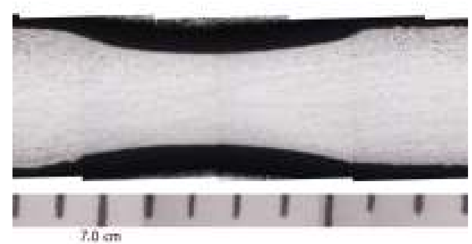
X-ray Image



Eutectic Melt Back / Isotherm



Circumferential
Detached Free Surface



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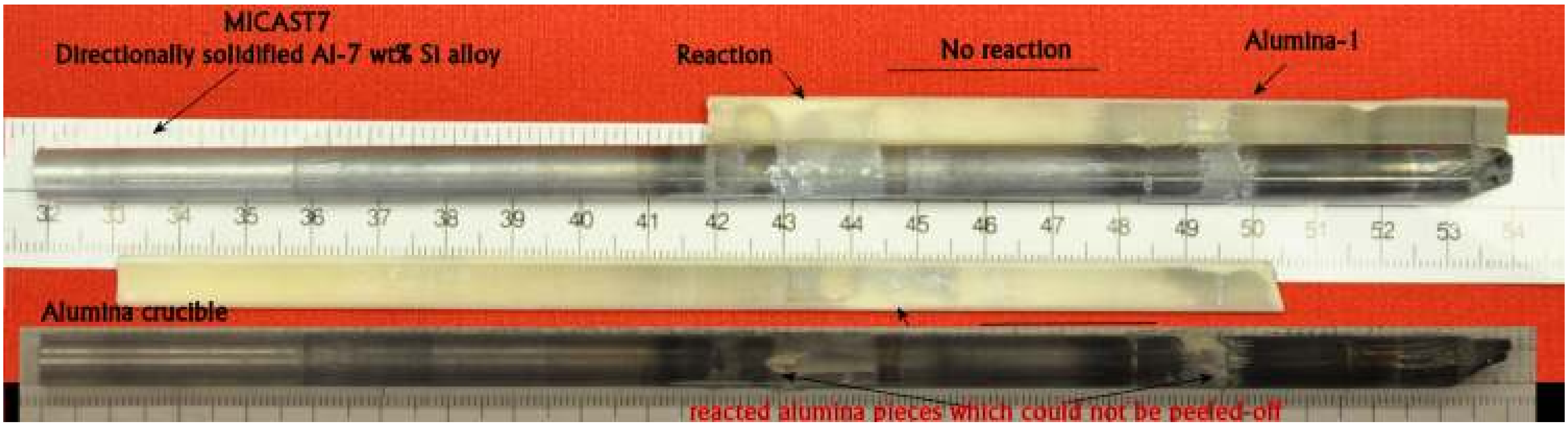
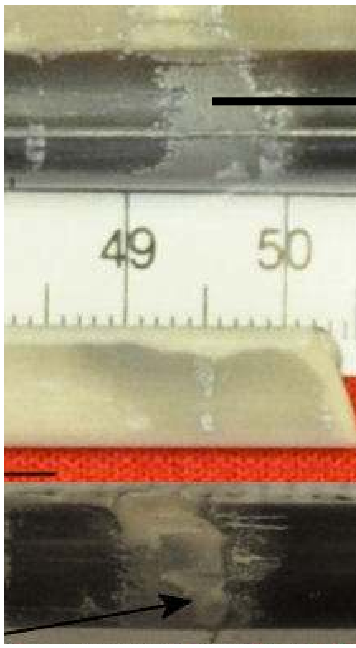
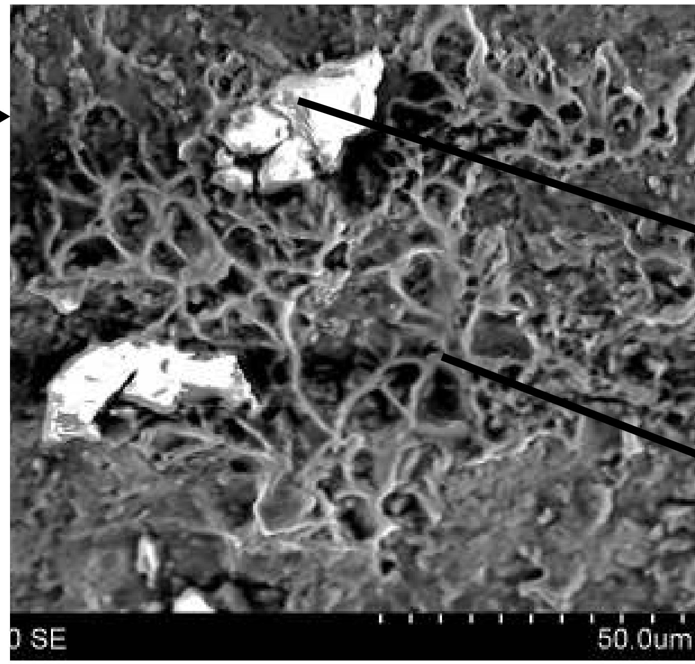
External Influence – Look at the Sample Crucible

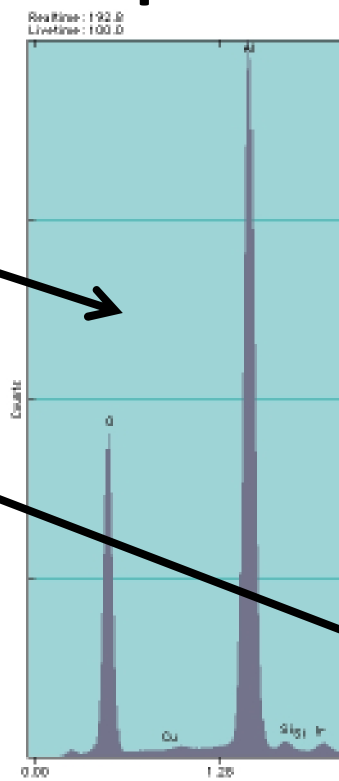


- Sample Discoloration
- Reaction Surfaces
- Alumina Adhesion

External Influence – Look at the Sample Crucible

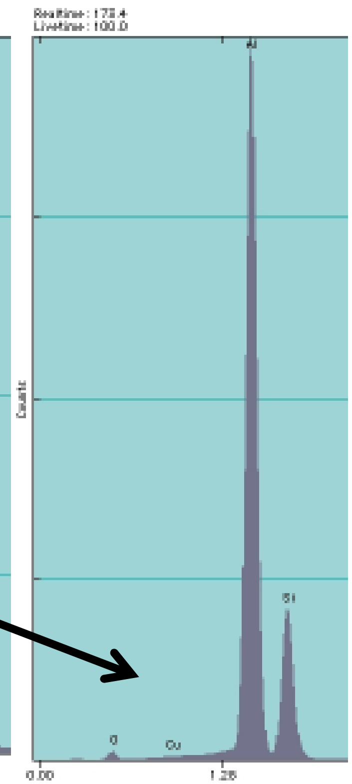






Quantitative Results for Spectrum1
Analysis: Bulk Method: Standardless
Acquired 15-Sep-2011, 15.0 KeV @10 eV/channel

Element	Weight %	Std. Dev.	Atomic %
O	41.40	0.87	54.97
Al	55.84	1.15	43.96
Si	1.18	0.38	0.89
Cu	0.00	0.00	0.00
Ir	1.58	0.49	0.17
Total	100.00		

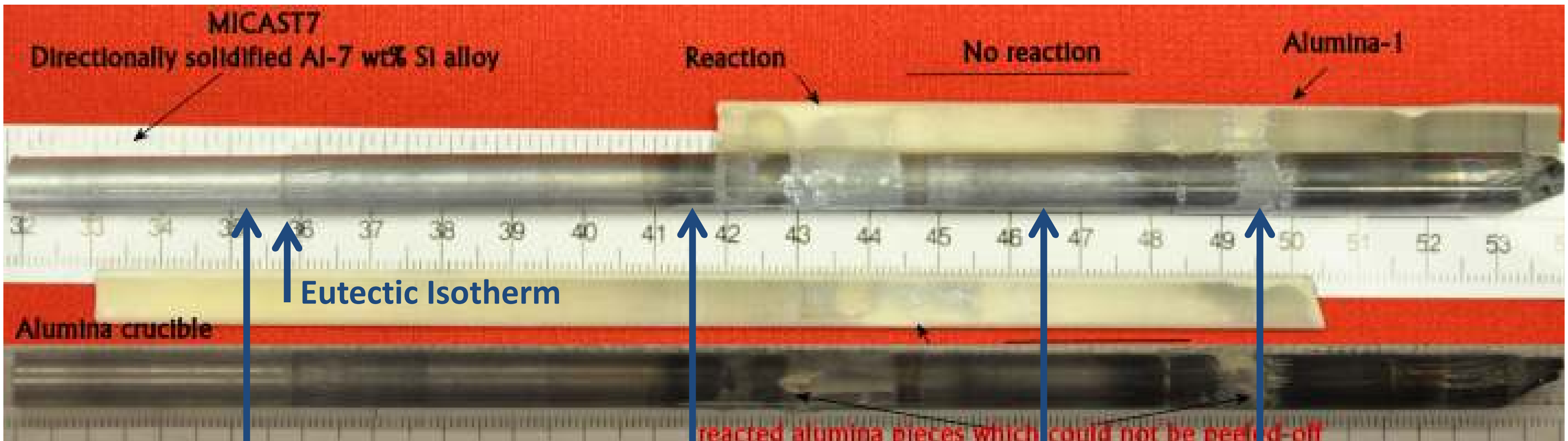


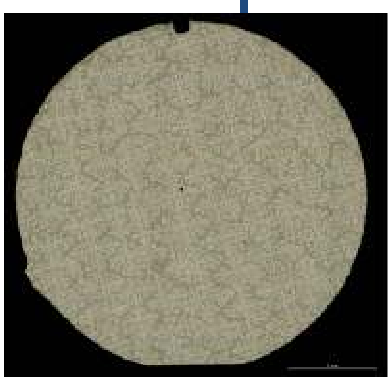
Quantitative Results for Spectrum1
Analysis: Bulk Method: Standardless
Acquired 15-Sep-2011, 15.0 KeV @10 eV/channel

Element	Weight %	Std. Dev.	Atomic %
O	2.23	0.64	3.74
Al	71.54	1.47	71.19
Si	26.24	0.92	25.08
Cu	0.00	0.00	0.00
Total	100.00		

26 wt.% Si

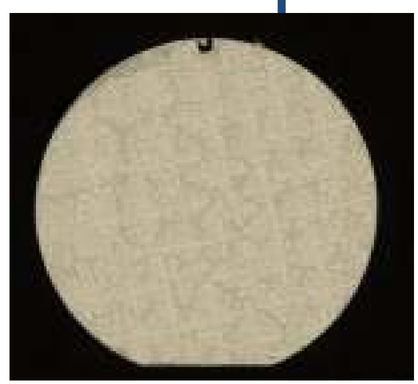
External Influence – Sample Cross-Section Location



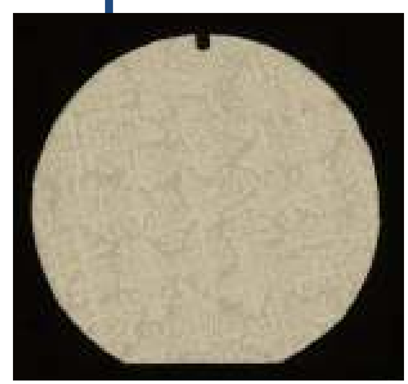


"Seed Crystal"

20µm s^{-1}

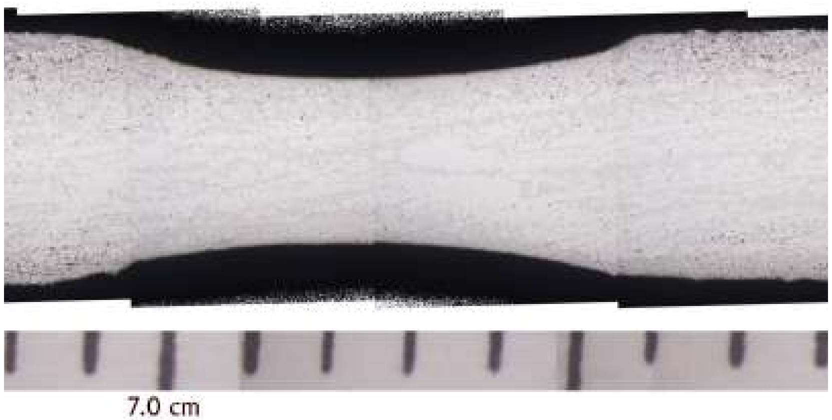


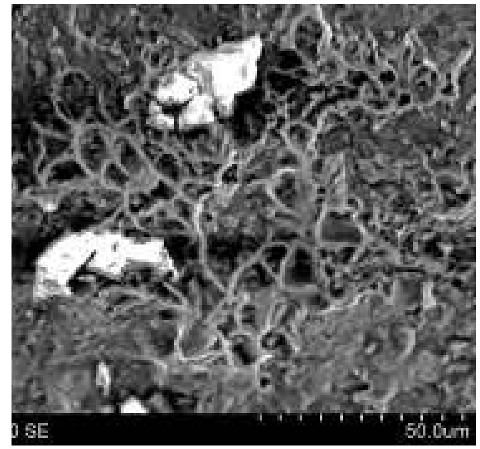
Transition



10µm s^{-1}

External Influence – Consequences



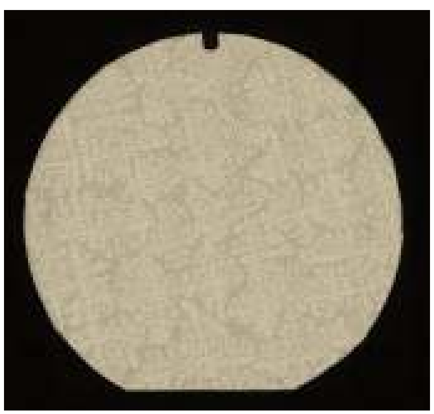


Free Surface

- Initiate Gravity Independent TC Flow

Reaction Interface

- Porous, Gas Generation → Bubbles?



$10\mu m s^{-1}$

Interdendritic Porosity

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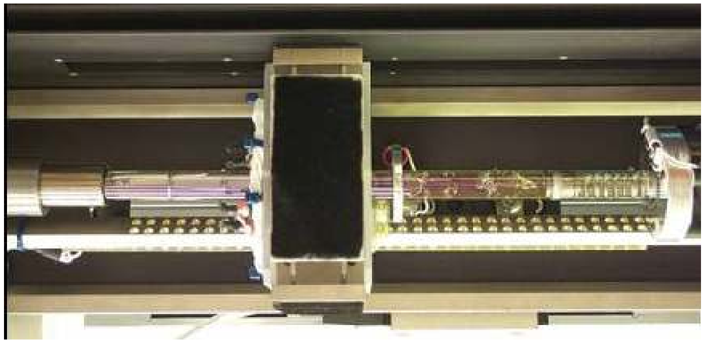
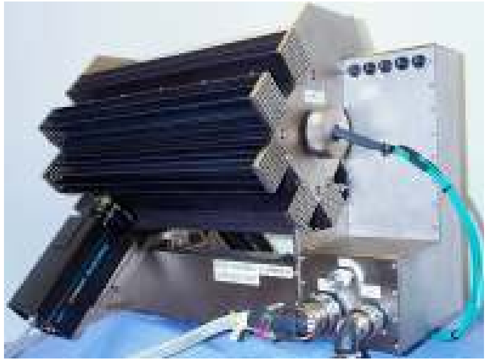
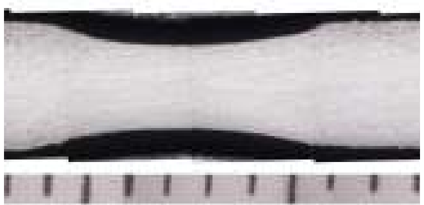
Consequences of Bubbles in Microgravity

Pore Formation and Mobility Investigation (PFMI)

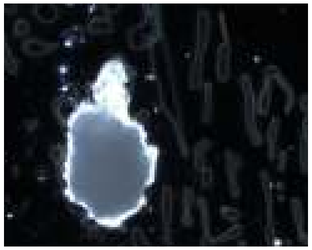






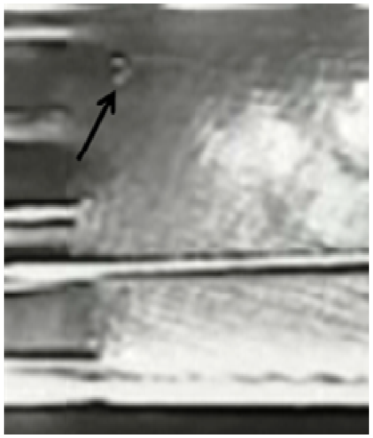
Free Surface

- **Initiate Gravity Independent TC Flow**



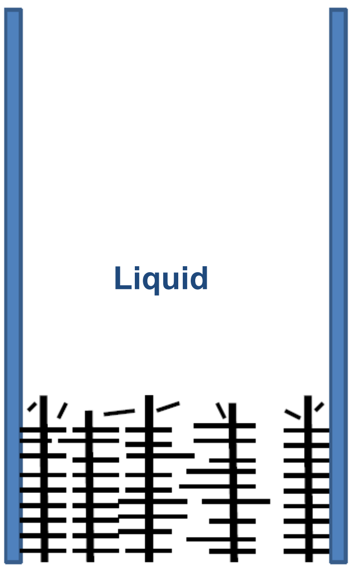
Interdendritic Porosity

- Average (minimum) bubble velocity is 45 mm/s.
- Bubble appeared to disrupt dendrite fragments just below it.

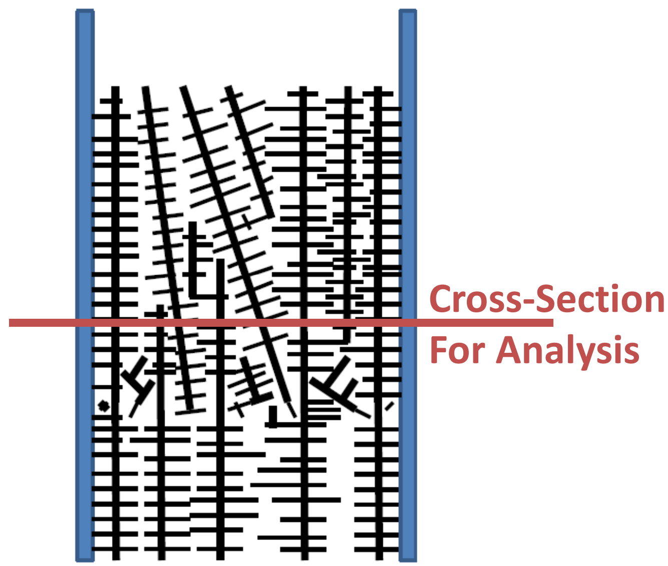


→ Disrupt the desired interface alignment

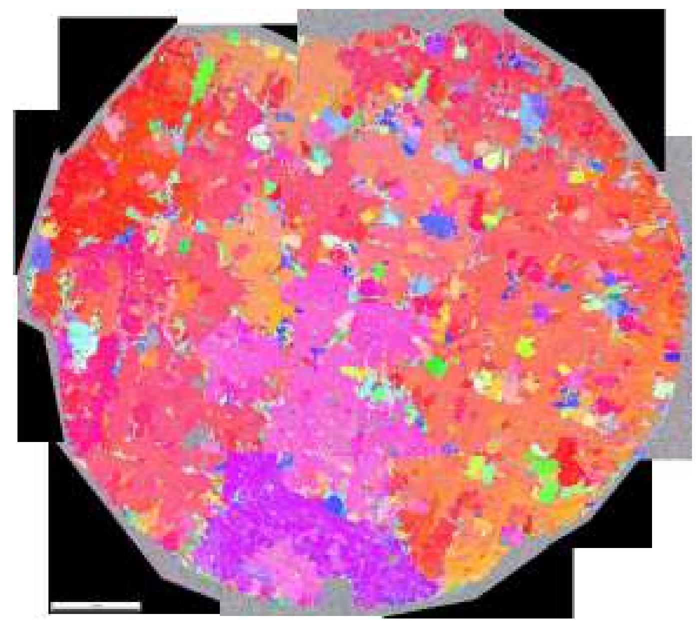
Consequence of Disrupting the Desired Dendritic Alignment



Initial Solid-Liquid Interface after Disruption by Bubbles
→ Mis-oriented Dendrite Arms/Fragments



Subsequent Directional Solidification In Microgravity



Conclusions

Dendritic Solidification in Microgravity Environment is Far from being Well Understood

Inferred that Gravity Independent Phenomena (from Bubbles) Served to Disrupt Dendritic Interfaces / Arrays

- **Can't Assume the "Quiescent" Microgravity Environment is Quiescent**

Sound Sample Preparation is Essential

Acknowledgments

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