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


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

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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 ($\lambda_1$, $\lambda_2$, $\lambda_3$, $d$) as a function of the Steady-State Processing Conditions ($V$, $G$, $C_0$)

**Use the Above for Comparison to Limited # of DS $\mu g$ 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.

Microstructural Evaluation

\( \lambda_1 \), Primary Dendrite Arm Spacing

\( \lambda_3 \), 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

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 μg 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

Solidification Furnace with Quench (SQF) Insert

Microgravity Science Research Facility (MSRF) Aboard the ISS

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

1g 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, $V = 20\mu m \text{ s}^{-1}$, $G = 40K \text{ cm}^{-1}$

MICAST 7-1 Composite EBSD Scan
Microgravity Processing

MICAST 7-1 Ground Processed Seed Crystal
Processing in Microgravity
(Steady-State Growth Conditions)

MICAST7 – 3T (20μm s⁻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)
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\(^{-1}\) → 10μm s\(^{-1}\), G = 28K cm\(^{-1}\))
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\(^{-1}\) → 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
External Influence – Look at the Sample Crucible

- Sample Discoloration
- Reaction Surfaces
- Alumina Adhesion
External Influence – Look at the Sample Crucible

26 wt.% Si
External Influence – Sample Cross-Section Location

- MICAST7 Directionally solidified Al-7 wt% Si alloy
- Reaction
- No reaction
- Alumina crucible
- Eutectic Isotherm
- “Seed Crystal” 20μm s⁻¹
- Transition
- 10μm s⁻¹

reacted alumina pieces which could not be peeled off
External Influence – Consequences

Free Surface
- Initiate Gravity Independent TC Flow

Reaction Interface
- Porous, Gas Generation → Bubbles?

10μms⁻¹

Interdendritic Porosity
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

Cross-Section For Analysis
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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