

**National Aeronautics and
Space Administration**

**Marshall Space Flight Center
Materials and Processing Laboratory / EM31**

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26th Symposium on Gravity - Related Phenomena in Space Exploration**

Disruption of an Aligned Dendritic Network by Bubbles during Re-melting in a Microgravity Environment

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Solidification Processing of Metals and Alloys in a Microgravity Environment

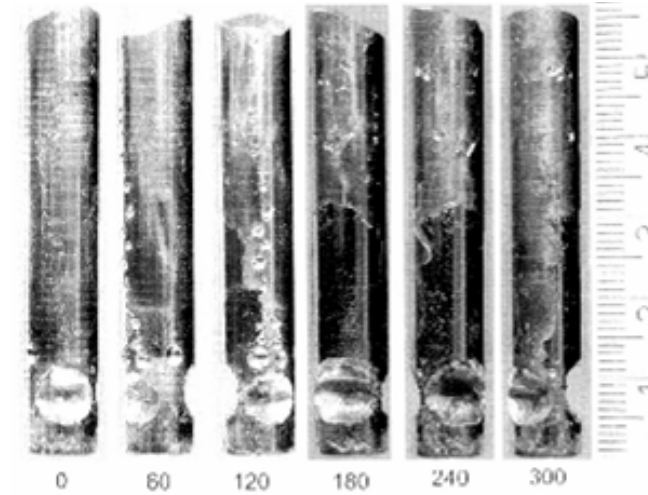
- Advantages:** **Minimize Thermo-Solutal Convection**
 Minimize Buoyancy Effects
- Intent:** **Produce Segregation Free Samples Grown Strictly**
 by Heat Transfer and Solute Diffusion
- Purpose:** **Better Understand the Relationship between**
 Processing – Microstructural Development
- Application:** **Maximize Material Properties**



Some Microgravity Processing Results



Computed Tomography Image of an Aluminum –
17.3 wt% Indium Alloy Directionally Solidified in
the AGHF During the 1996 LMS Mission

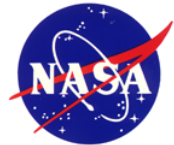


Lead-Tin-Telluride Crystal
processed on USMP-3 Mission

Pores or Bubbles Form in Microgravity

Recall: Buoyancy Minimized in μg

- Bubbles won't float away
- Compromise effective heat transfer
- Initiate *gravity-independent* thermocapillary convection



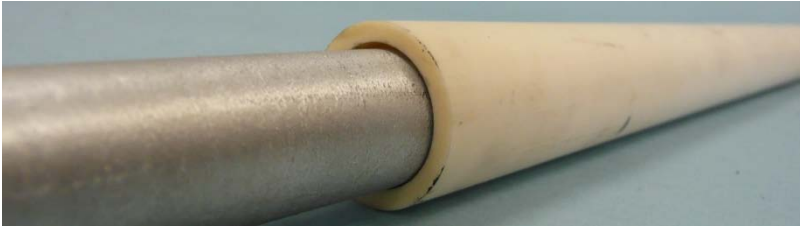
Sources of Porosity

- Evolution of dissolved gases during solidification is well documented (C.H. Tonamy, 1915)
- Gas generation from reactions of the melt with the crucible wall



Al – 7wt% Si in
Alumina Crucible

- Coalescence of “empty” space between the crucible wall and inserted sample



Crucible ID = 19.00 mm
Sample OD = 18.90 mm
For 200 mm long sample
~ 600 mm³ void volume



Effect of Bubbles during Solidification Processing in a Microgravity Environment

→ Begin Pore Formation and Mobility Investigation (PFMI)

Sample Ampoule



Flight sample PFMI-2
Pure SCN with Added N₂ Bubbles
~18 cm of Sample Length, 1 cm ID

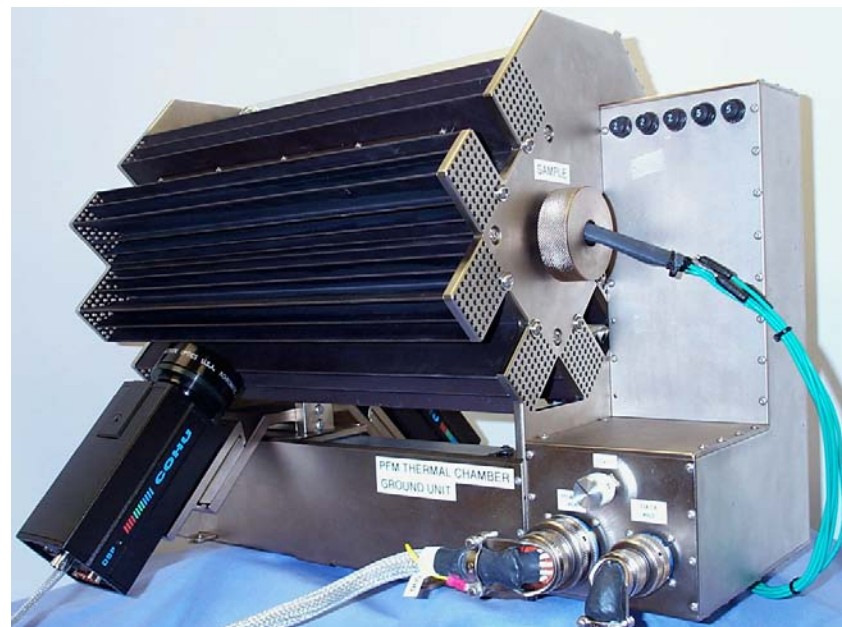
- Utilizes Succinonitrile (SCN)
 - Transparent
 - Solidifies in a manner analogous to metals
 - Low melting temperature
 - Well understood material properties
 - Previously used in microgravity experiments



Experimental Hardware

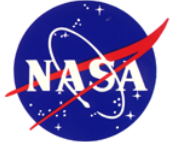


**Internal View of PFMI Chamber
Showing Sample**



PFMI Thermal Chamber

Enables Controlled, Steady-State Directional Solidification (Melting) Processing



On-Board the International Space Station



PFMI Hardware in the Microgravity Science
Glovebox



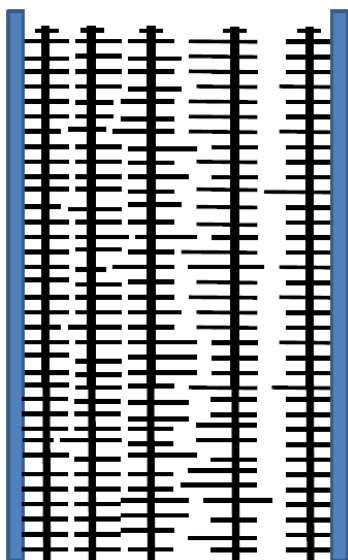
Direct Observation of Succinonitrile Dendrites
Growing through an Imposed Temperature
Gradient. Interdendritic Porosity Develops
Behind the Interface as N_2 Comes Out of Solution



Directional Solidification in a Microgravity Environment – Initial Re-melting of the Sample

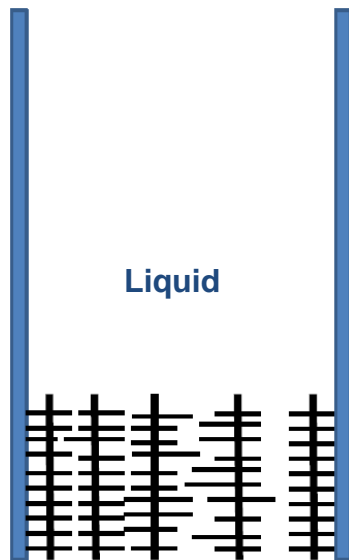
(Is this a Concern??)

Ideal Schematic Microgravity Processing Scenario

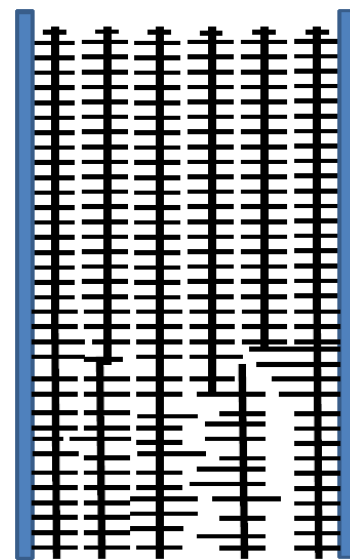


1g Directionally Solidified Dendritic “Seed” Crystal

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



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



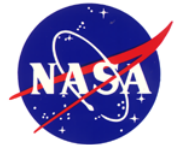
Directional Solidification in Microgravity

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

Steady-State Diffusion Growth

Transient Growth

Initial Dendritic Array



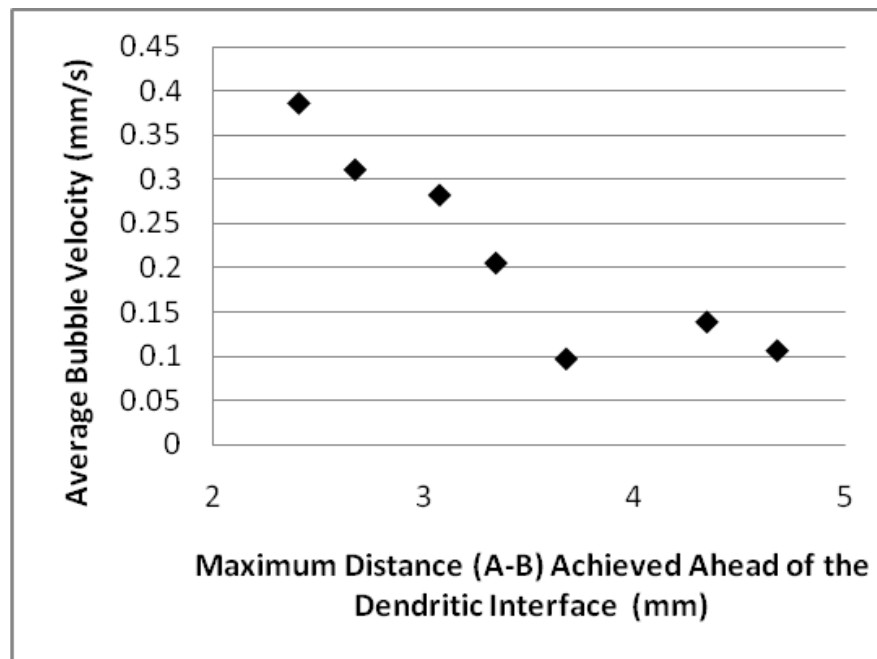
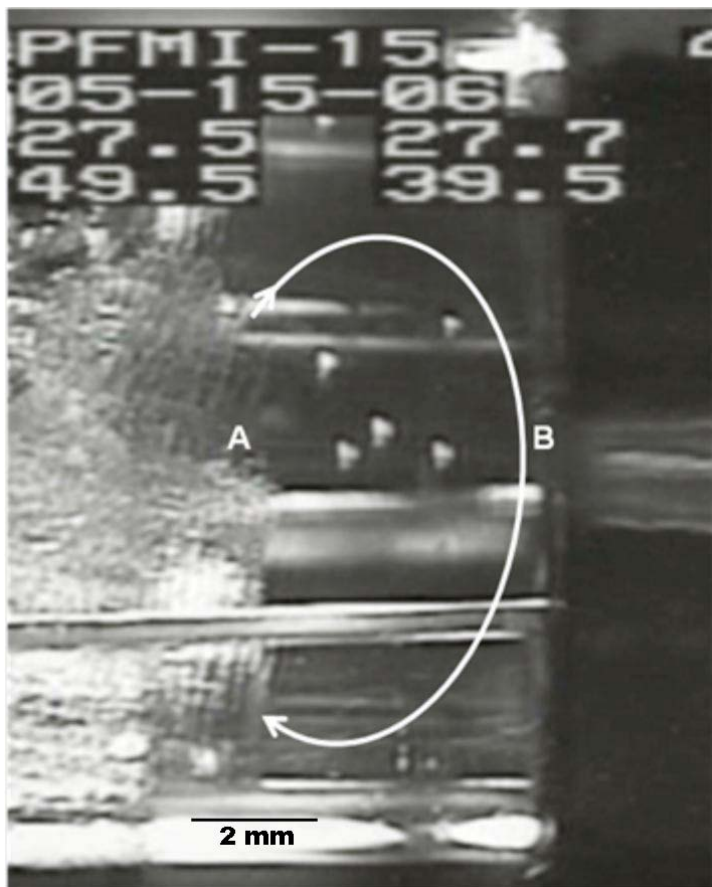
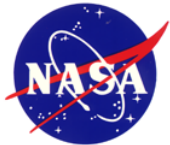
Re-melting in a Microgravity Environment (In the Presence of Bubbles)

Convection Effects from “Large” Bubbles

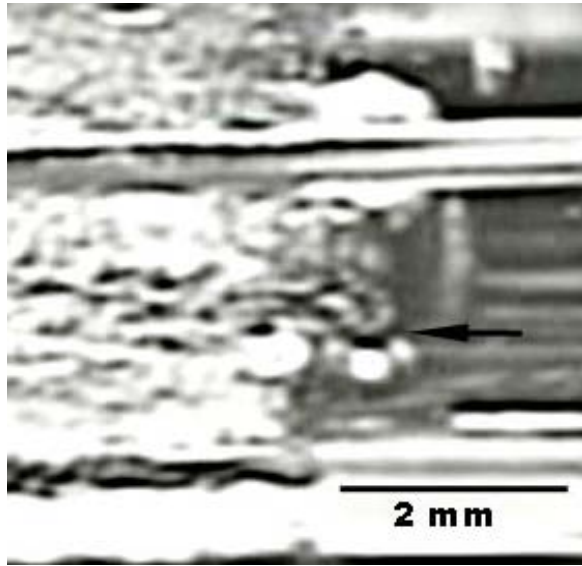
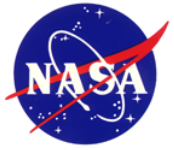




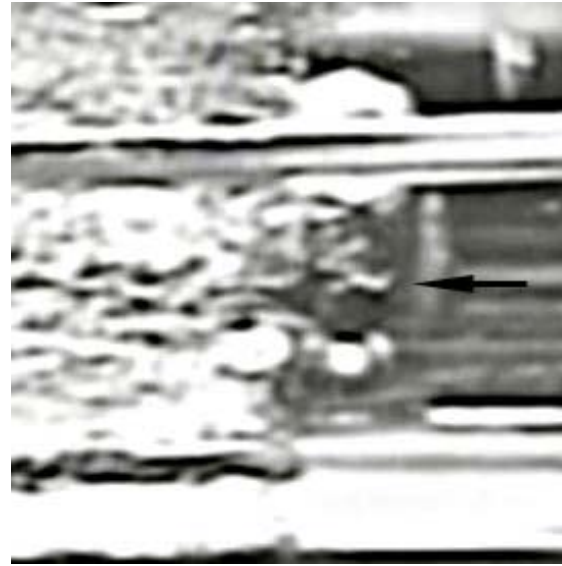
**Observation and Extent of Convection via Tracer Bubbles
(time x5)**



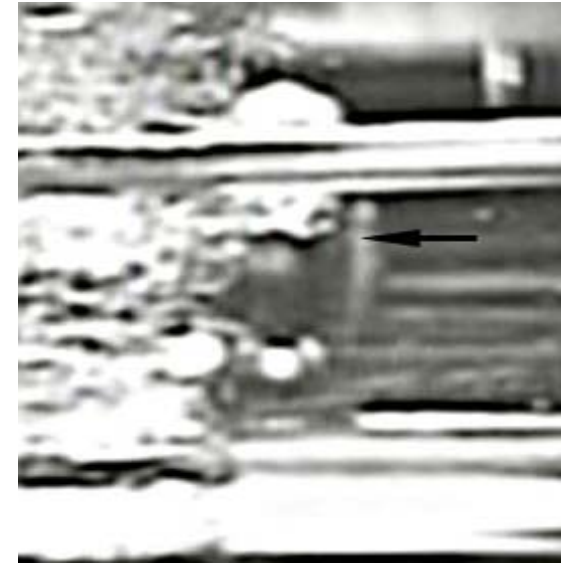
Thermocapillary flow field set up by a large bubble at the interface; velocities as established by the miniature tracer bubbles.



T = 0 seconds



T = 23 seconds



T = 56 seconds

A time sequence showing dendrite fragment transportation at the solid-liquid interface

Consequence of “Large” Bubbles

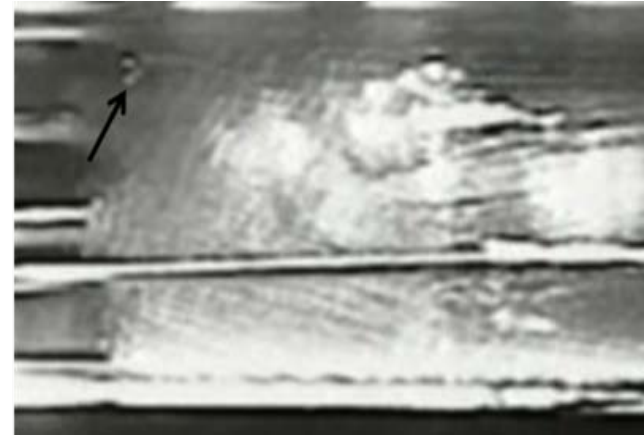
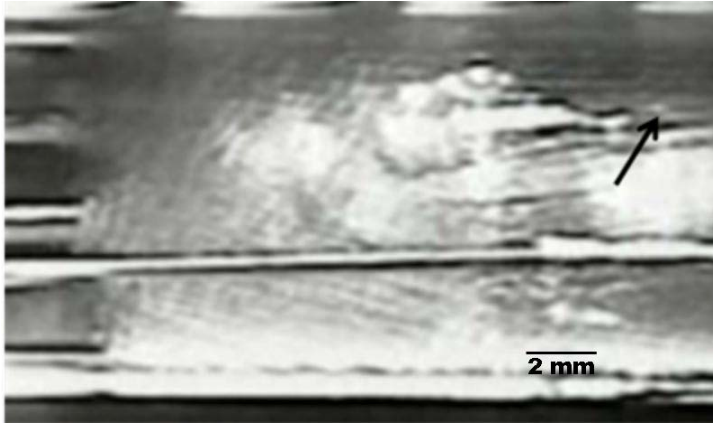
- Induce Thermocapillary Flow
 - ◆ Raze aligned dendritic arrays
 - ◆ Transport dendrite fragments
 - ◆ Bring warm, “bulk” liquid to the interface

→ Disrupt the desired interface alignment



“Small” Bubble Implications

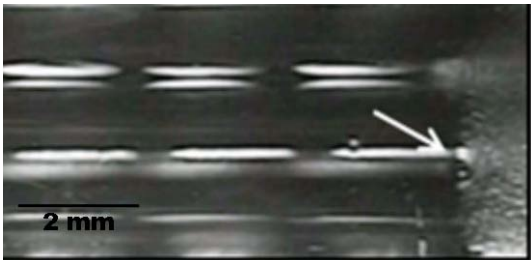
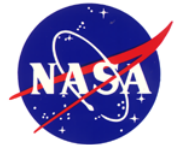




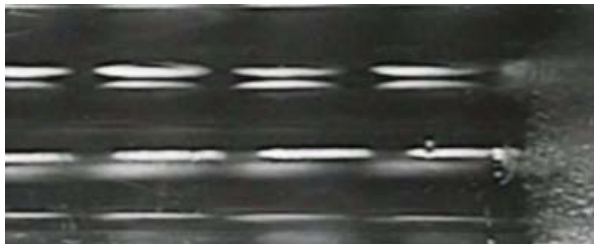
**Movement of a small bubble through the mushy zone.
Note the faint trail that developed as it passed through the mushy zone**

The distance in the plane of the photograph is ~9 mm, time of “flight” ~0.2 seconds

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



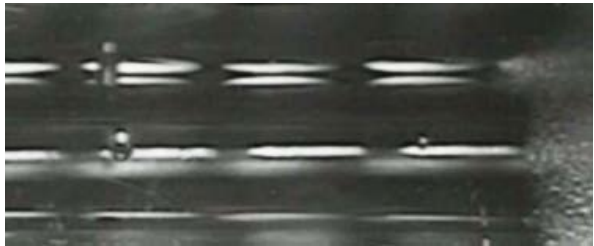
(a)



(b)



(c)



(d)

Series of photographs showing a small bubble moving up the imposed temperature gradient at a velocity of $\sim 0.7 \text{ mms}^{-1}$.
(Succinonitrile – 0.24 wt% water, $G = 0.12 \text{ Kmm}^{-1}$)
 ~ 15 minutes after (d) bubble has dissolved

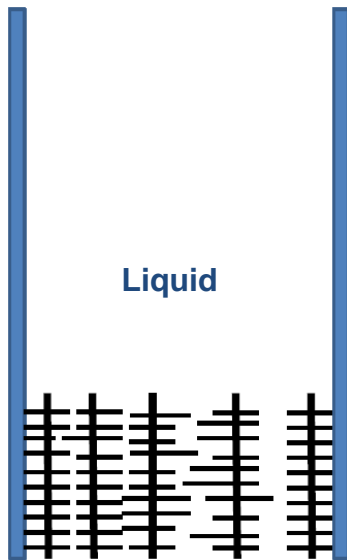
Consequence of “Small” Bubbles

- **Dynamic Effect**
 - ◆ Affects a few dendrites – reposition secondary arms
 - ◆ Potential thermocapillary flow effects while at interface

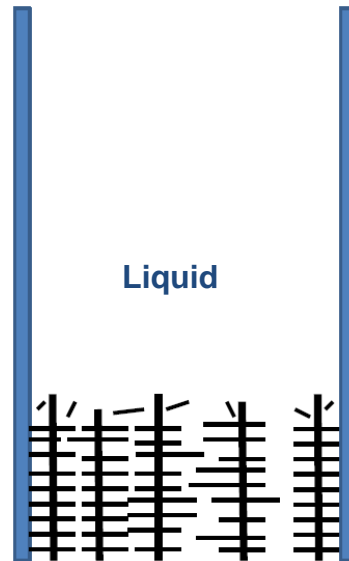
→ Disrupt the desired interface alignment



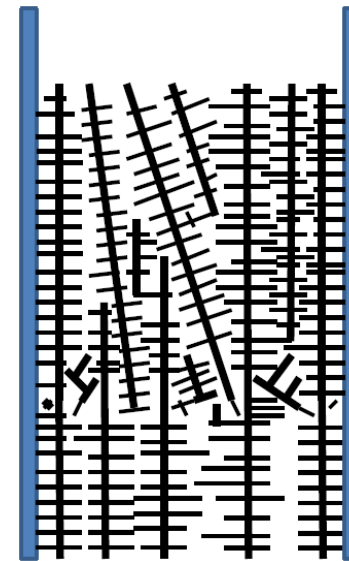
Consequence of Disrupting the Desired Dendritic Alignment



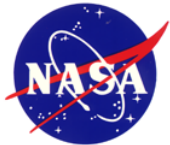
**Melt Back of Dendritic
Array In Microgravity
(Prior to initiating
controlled directional
solidification)**



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



**Subsequent Directional
Solidification In Microgravity
→ Compromised Science
→ Compromised Material
Properties**



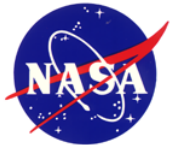
Conclusions

The quiescent Microgravity environment can be quite dynamic

Thermocapillary flow about “large” static bubbles on the order of 1mm in diameter was easily observed by following smaller tracer bubbles. The bubble induced flow was seen to disrupt a large dendritic array, effectively distributing free branches about the solid-liquid interface.

“Small” dynamic bubbles were observed to travel at fast velocities through the mushy zone with the implication of bringing/detaching/redistributing dendrite arm fragments at the solid-liquid interface.

Large and small bubbles effectively re-orient/re-distribute dendrite branches/arms/fragments at the solid liquid interface. Subsequent initiation of controlled directional solidification results in growth of dendrites having random orientations which significantly compromises the desired science.



Acknowledgment

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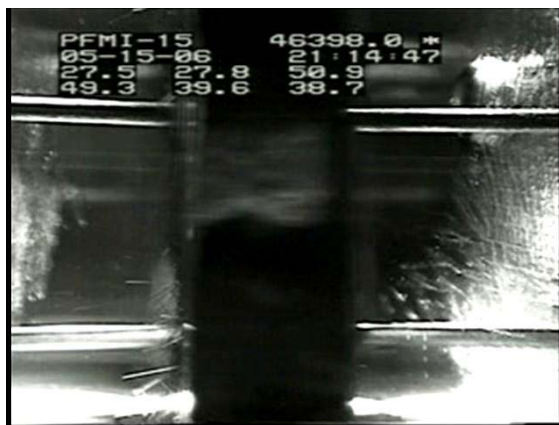
Back Up Slides



(a)



(b)



(c)



(d)

Series of photographs (a-d) over a 37 second period that show interface melt back, emergence of a bubble on the upper right side (arrow point in a), and displacement of the dendritic network at the solid-liquid interface. The molten zone is initiated by the ring in the center of the photograph; it is positioned over an in-situ thermocouple tip which is currently reading 50.9° C.



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