Materials Research in Microgravity & Hypergravity

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Why?

Materials Research in Microgravity & Hypergravity

Microgravity	= zero g
Earth (terrestrial)	= 1 g
Hypergravity	> 1 g

Materials in Space

Why?

Gravity affects any material containing a fluid (gas or liquid).

Many manufacturing processes (e.g. casting) involve manipulating a liquid.

Liquid flow is affected by gravity.

The properties and quality of the manufactured part are affected.

The best way to assess the effects of gravity is to **remove it and see what happens**.

Materials in Space

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The best way to assess the effects of gravity is to **remove it and see what happens**.

We can also increase gravity levels to see what happens (in hypergravity)

Gravity – the Basic Maths

$$F = m \times a$$

$$F = m \times g \downarrow$$

Newton's 2nd Law of Motion

F is force (N)
M is mass of body (kg)
a is acceleration (m/s^2)
g is acceleration due to gravity = 9.81 on Earth

Planet	Acceleration due to gravity, "g" [m/s^2]
Mercury	3.59
Venus	8.87
Earth	9.81
Moon	1.62
Mars	3.77
Jupiter	25.95
Saturn	11.08
Uranus	10.67
Neptune	14.07
Pluto	0.42

kaiserscience.wordpress.com

$$m = \rho \times V$$

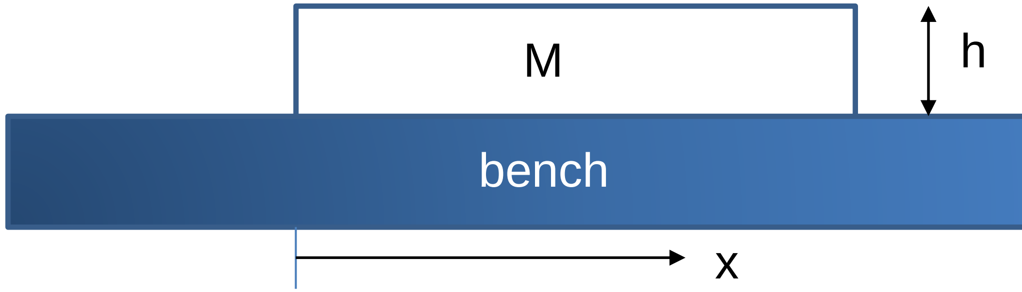
ρ is density of body (kg/m^3)

V is volume of body (m^3)

Gravity and Natural Convection

$$F = \rho \times V \times g \quad \downarrow$$

Consider a container height h of material M sitting on a laboratory bench, or bolted down on a spacecraft



M can be solid, liquid, gas or a mixture of such phases.

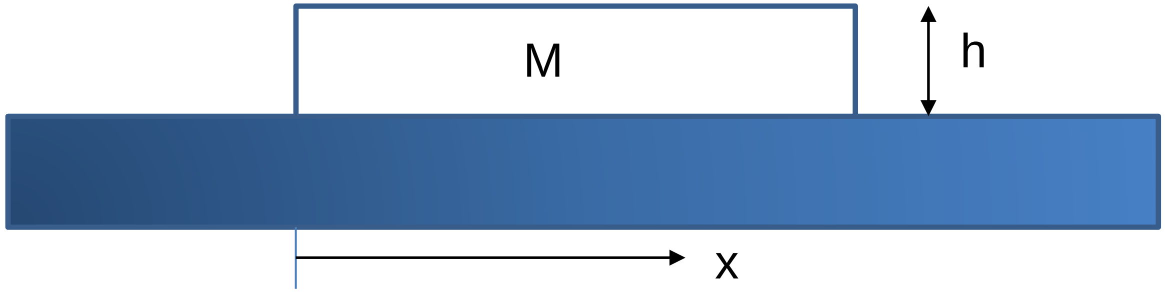
Solid: ρ does not normally vary within the material.
Liquid: ρ may vary within the material
Gas: ρ may vary within the material
} Fluid

For a fluid, ρ may vary in any direction.
Consider ρ varying with x i.e. $\rho = \rho(x)$

Gravity and Natural Convection *contd.*

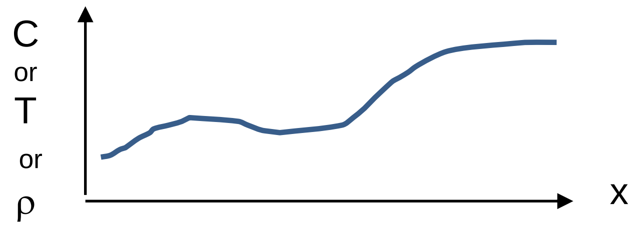
$$F = \rho \times V \times g \downarrow \qquad \rho = \rho(x)$$

Consider a container height h of fluid sitting on a laboratory bench



M contains some liquid, gas or a mixture of the two.

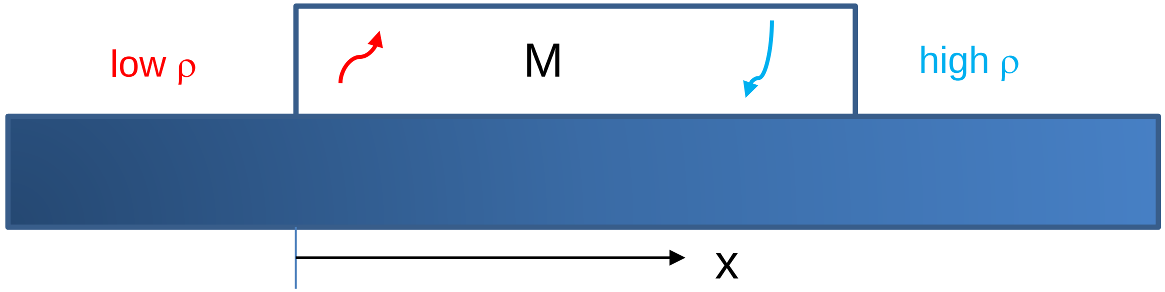
The density ρ will vary if either the temperature T or the composition C vary with x i.e. T = T(x) or C = C(x).



Gravity and Natural Convection *contd.*

$$F = \rho \times V \times g \downarrow \qquad \qquad \rho = \rho(x)$$

Denser fluid will sink and lighter fluid will rise, resulting in natural convection currents in the container



Such variations in T occurs when, for example, you're cooling a liquid metal in a mold in a casting process.

Such variations in C occur when, for example, you're cooling a liquid alloy in a mold in a casting process. Here the solid formed normally has a different composition (of solute) than average, resulting in compositional variations in the liquid.

Hence thermo-solutal convection.

Manufacturing Processes affected by Gravity

Process	Affected?	Reason - Material
Machining	✗	Solid
Forming e.g. forging or rolling	✗	Solid
Casting	✓	Liquid phase
Welding	✓	Liquid phase
Additive Manufacturing, 3D printing	✓	Liquid phase, loose powders
Ceramic Sintering	✓	Loose powders
Powder Metallurgy	✓	Loose powders
Injection moulding	✓	Liquid phase
Extrusion	✗	Solid

Microgravity

Microgravity (μg) is an environment where the gravity level is almost zero.

It is achieved when the experiment is in free-fall. The whole experiment is falling towards earth so, relative to the experimental laboratory or container, there are no net gravitational forces.

This can be achieved in:

- Drop towers
- Parabolic flights
- Sounding rockets
- Orbiting spacecraft
- Space Station

MICROGRAVITY PLATFORMS

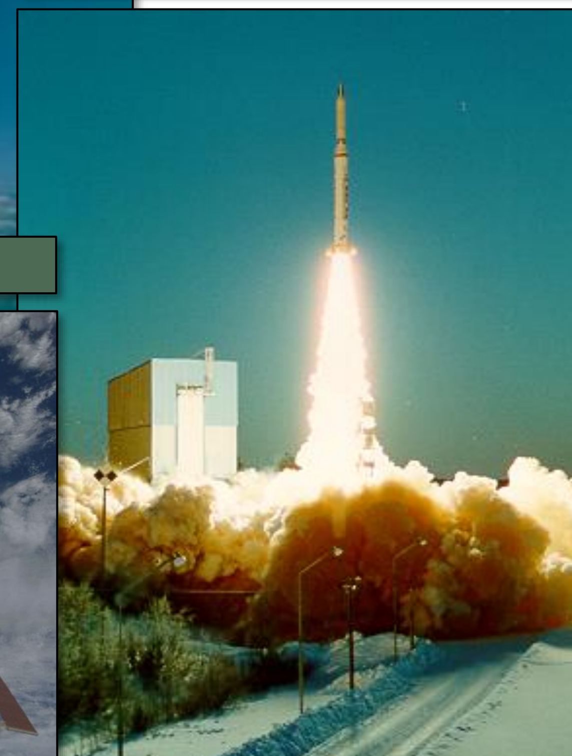
Slide courtesy: Dr Wim Sillekens, ESA

The means

Parabolic Flights

Drop Towers

Sounding Rockets



International Space Station

MICROGRAVITY PLATFORMS

Slide courtesy: Dr Wim Sillekens, ESA

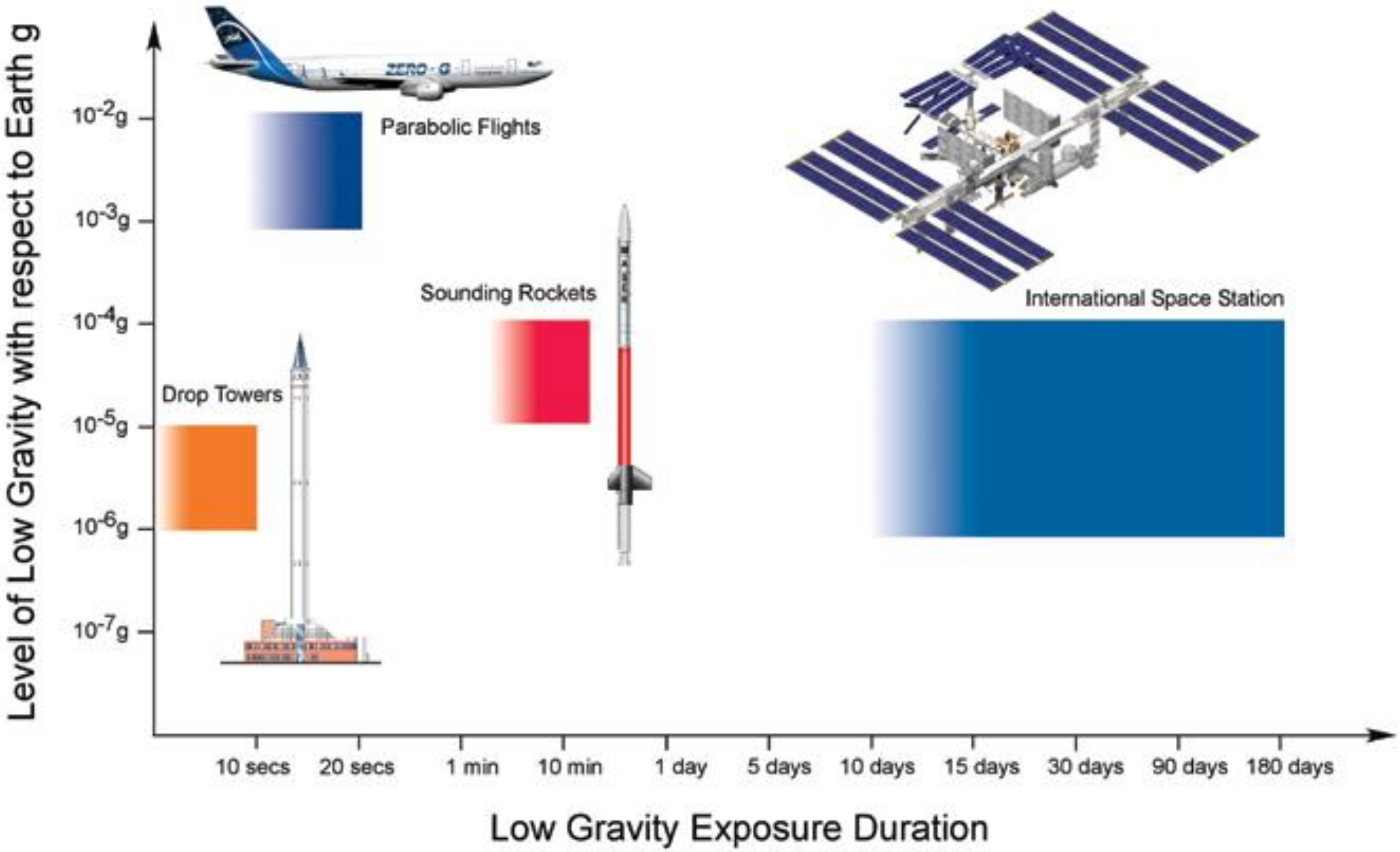
Characteristics

	DROP TOWERS (ZARM)	PARABOLIC FLIGHTS (Airbus A310)	SOUNDING ROCKETS (TEXUS/MASER, MAXUS)	INTERNATIONAL SPACE STATION (Columbus, ...)
Duration	4.7–9.3 s	22 s	6–13 min	continuous
Microgravity level	10^{-6}–10^{-5} g	10^{-3}–10^{-2} g	10^{-5}–10^{-4} g	10^{-6}–10^{-4} g
Payload	264 kg		260–480 kg	
Frequency	~20 experiments/year (~400 drops/year)	~15 experiments/ campaign (93 parabolas/ campaign)	2 campaign / 3 years (3–4 experiments/ campaign)	
Development & integration time	~2–6 months	~4–6 months	~12–24 months	~6–60 months
Overall dimensions	146 m tall shaft		13–16.2 m length	108×73×20 m (~450,000 kg)
Height	120 m internal height	6,000–8,500 m	Apogee 260–705 km	350–450 km
Other		0 (2) g, 0.17 g, 0.38 g		

MICROGRAVITY PLATFORMS

Slide courtesy: Dr Wim Sillekens, ESA

Gravity magnitude and duration[1)]

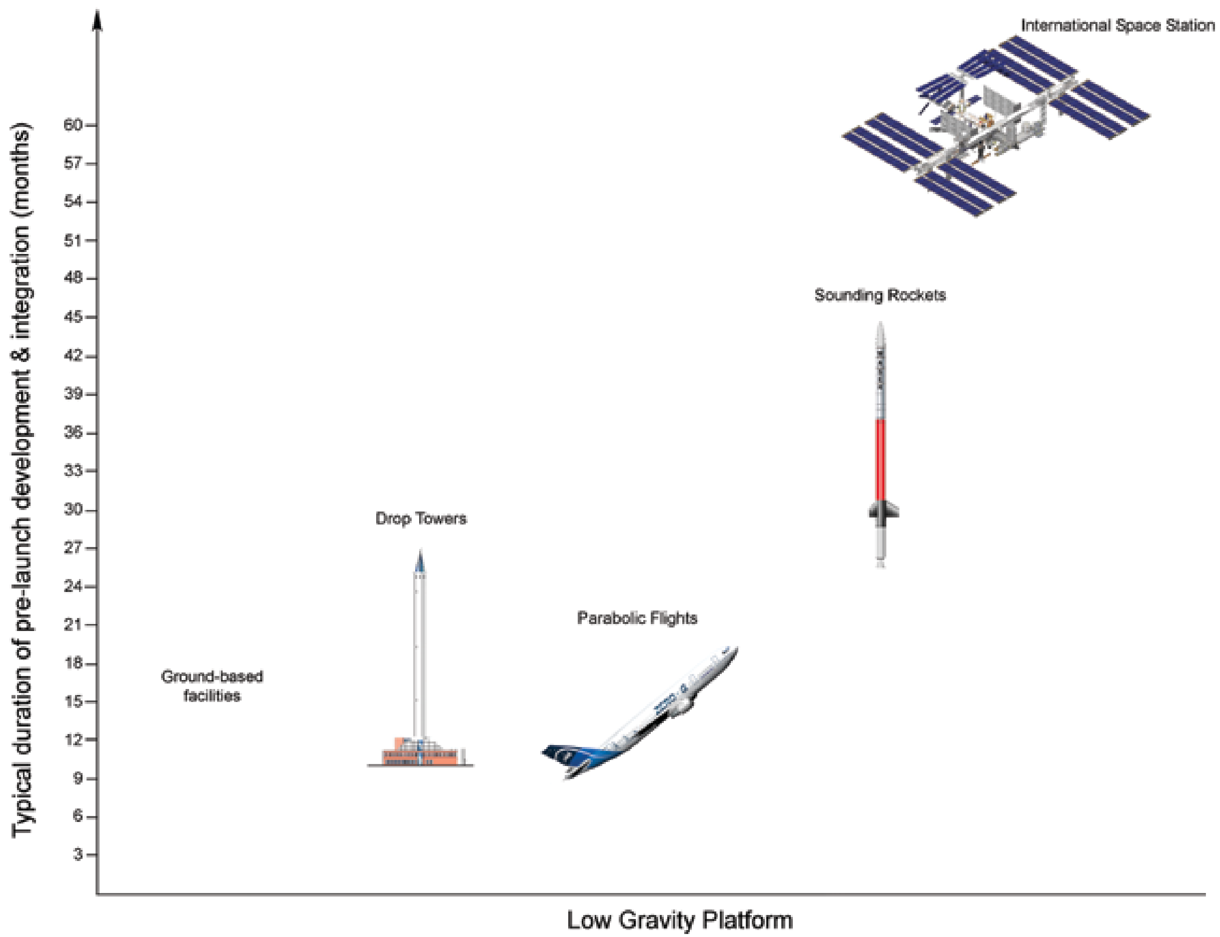


1) http://www.esa.int/Our_Activities/Human_Spaceflight/Research/European_user_guide_to_low_gravity_platforms

MICROGRAVITY PLATFORMS

Slide courtesy: Dr Wim Sillekens, ESA

Payload development & integration times[1)]



1) http://www.esa.int/Our_Activities/Human_Spaceflight/Research/European_user_guide_to_low_gravity_platforms

Some Microgravity Research Topics

Solidification of Alloys

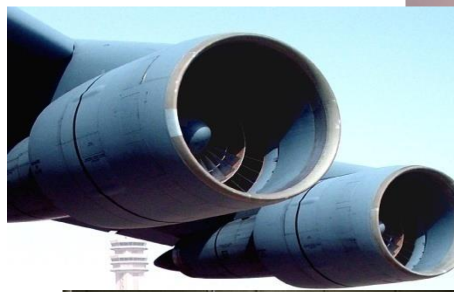
Metal Foaming

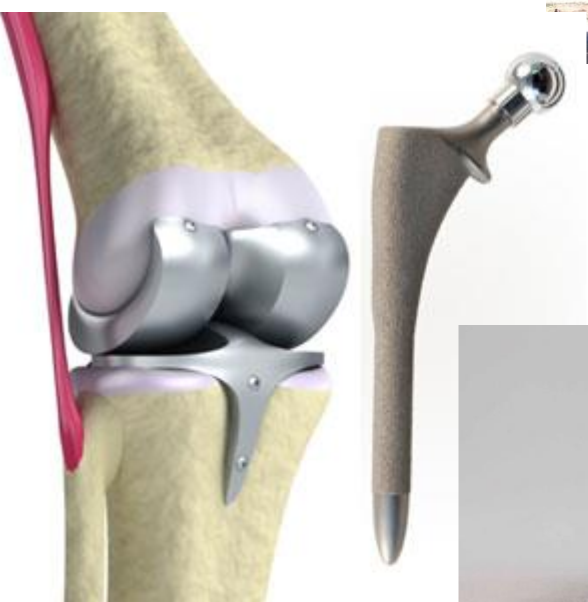
Measurement of Thermophysical Properties of Liquid Metals

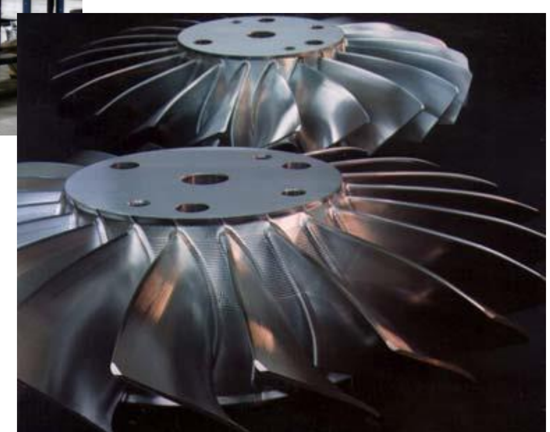
Transparent Analogues (to Metals)

Diffusion in Liquid Metals

Alloy Solidification – why?



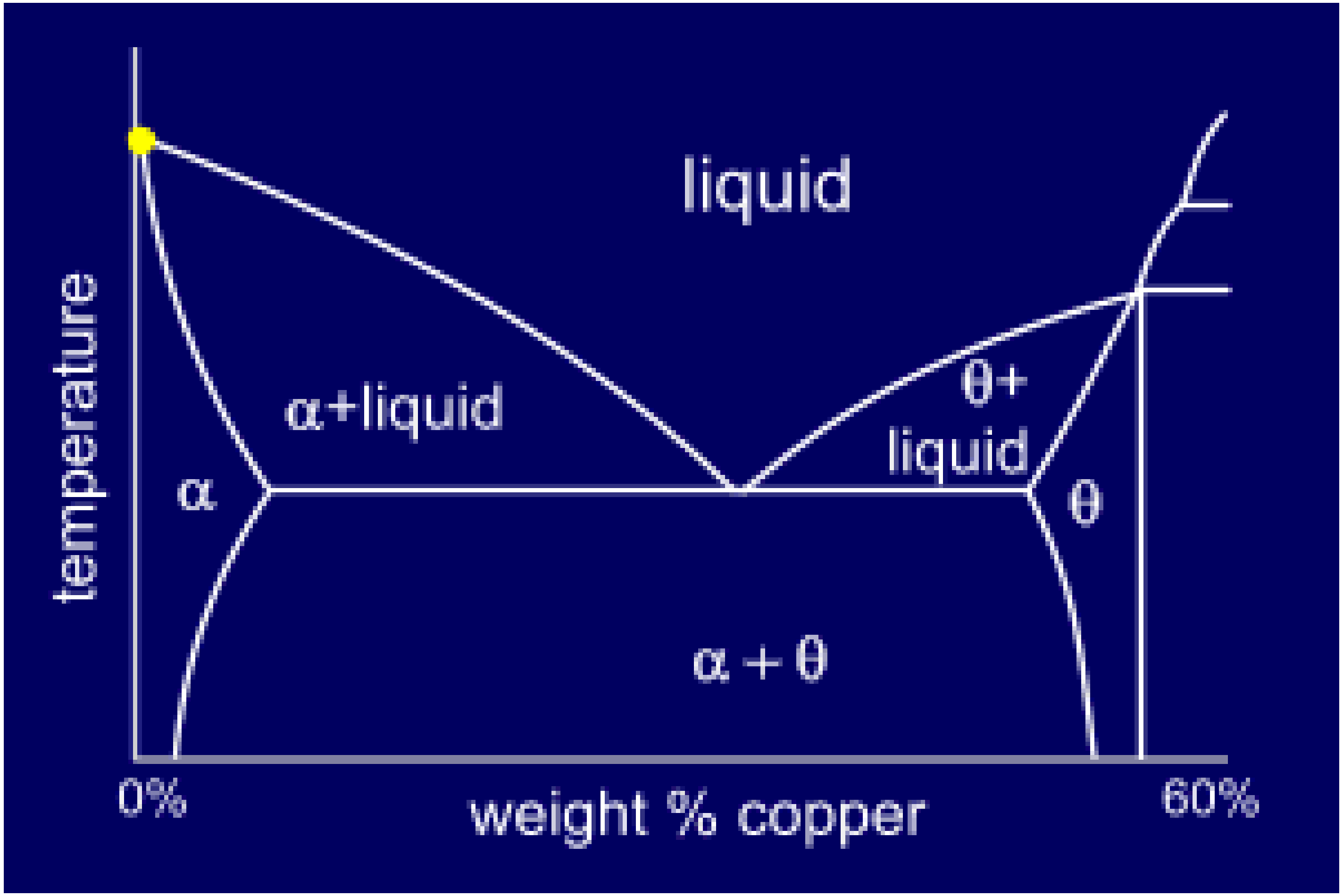




Alloy solidification – my journey into space

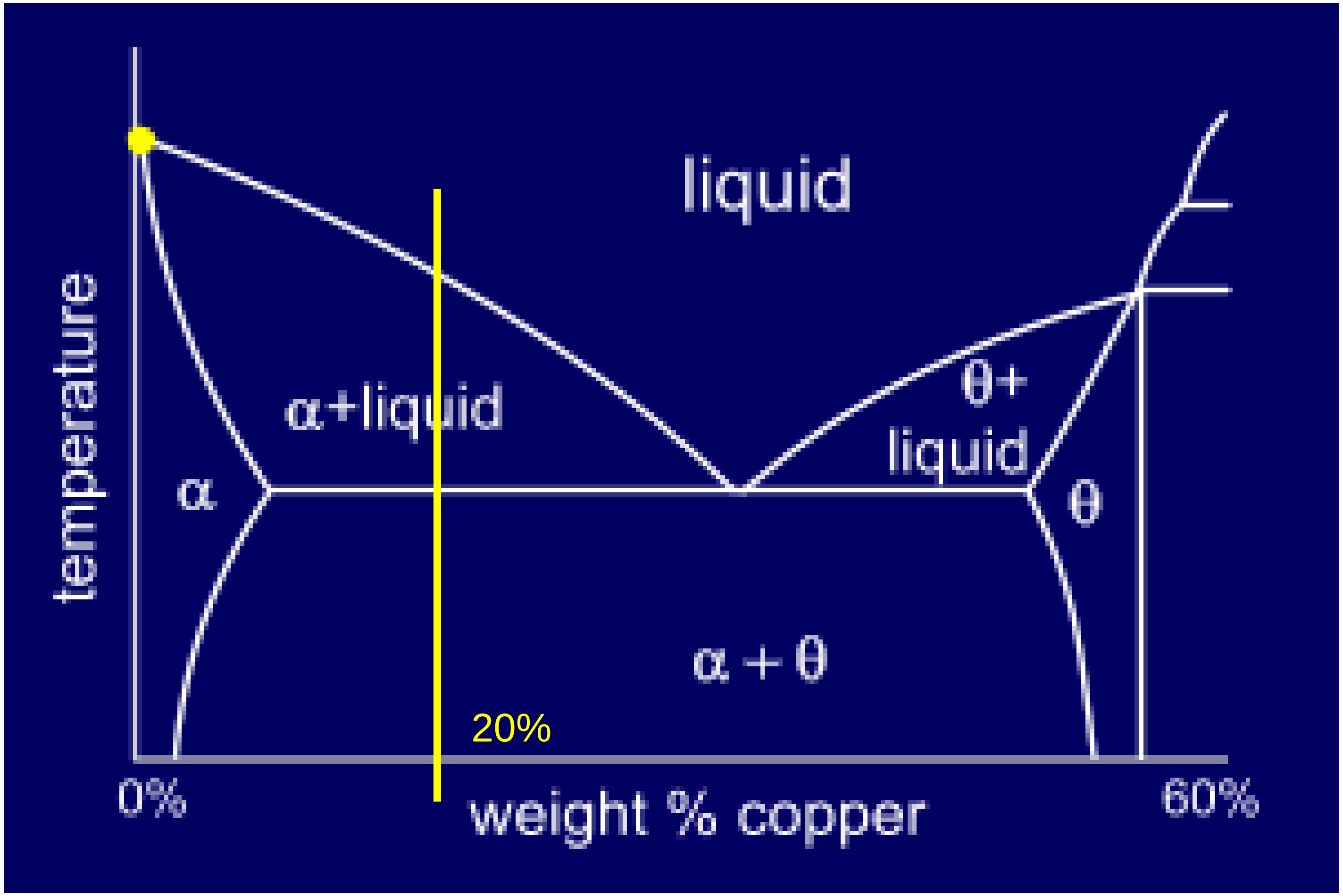
1984	undergrad project on casting ductile irons
1985 – 1987	process engineer in foundry, California and Ireland
1987 – 1990	research on twin roll casting
1990 –	PTRG
1997 – 2001	research on microstructural evolution during solidification
2000	introduced to ESA research consortium on microgravity solidification, by Prof. John Hunt, Oxford University.

Binary phase diagram



Al-Cu system

Solidification of Al – 20% Cu



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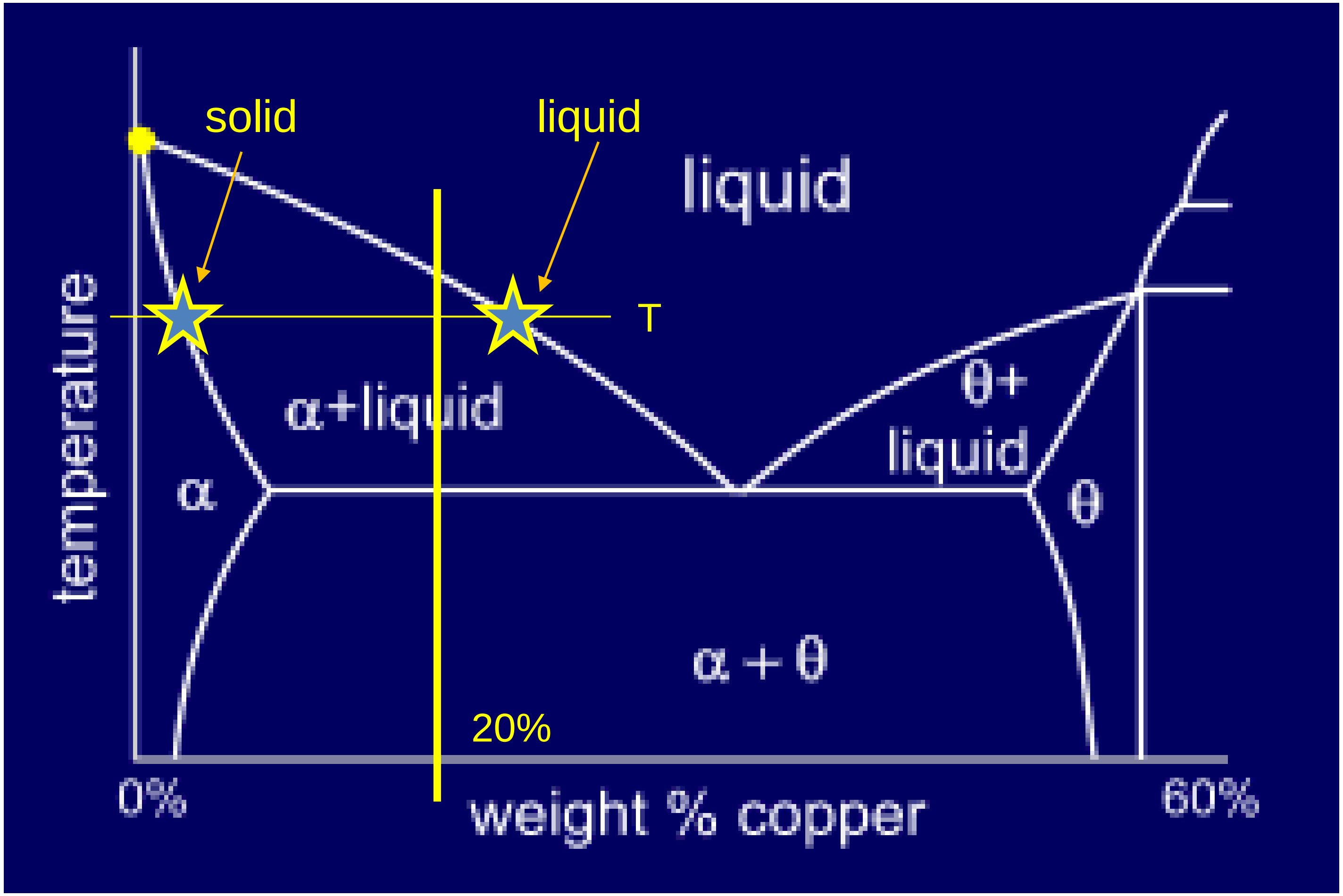
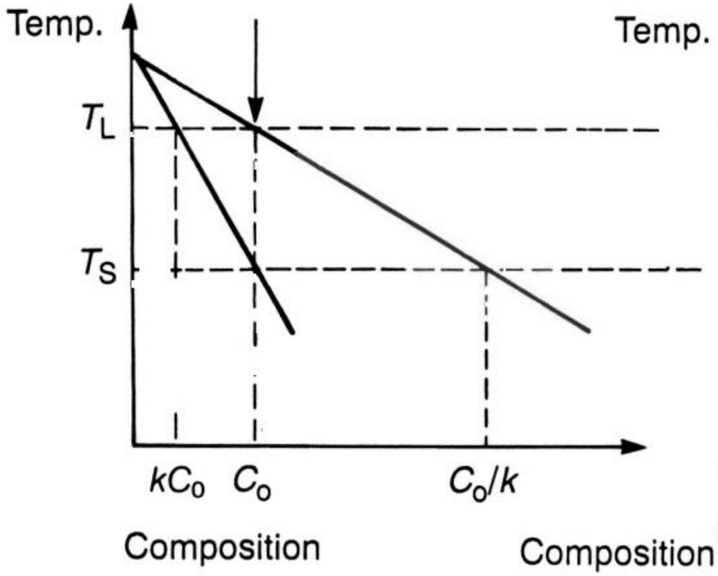
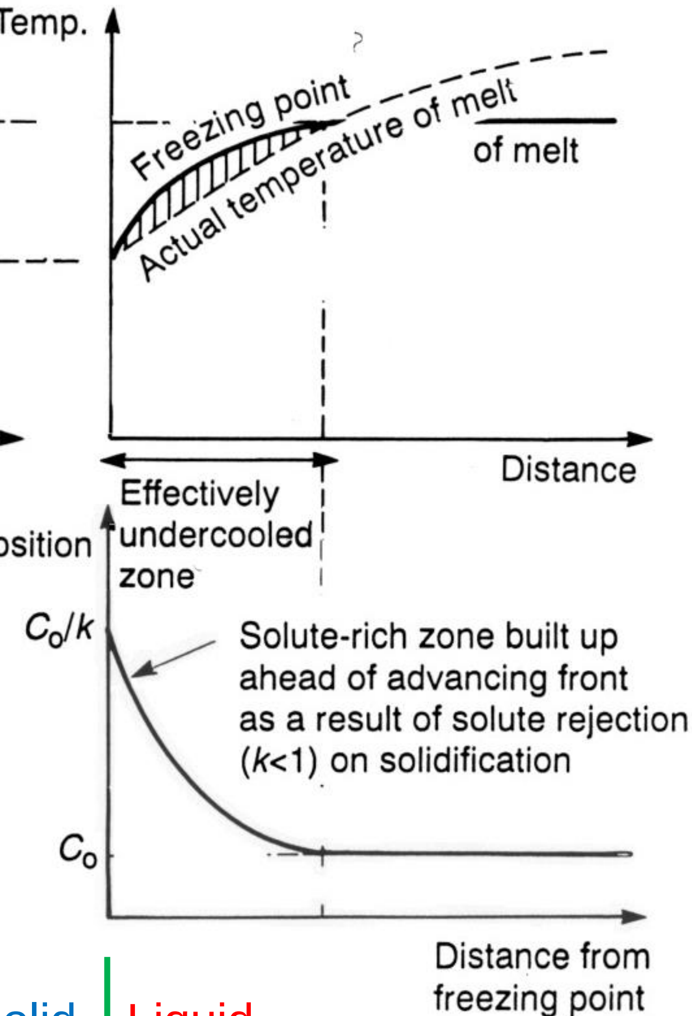


Al-Cu system

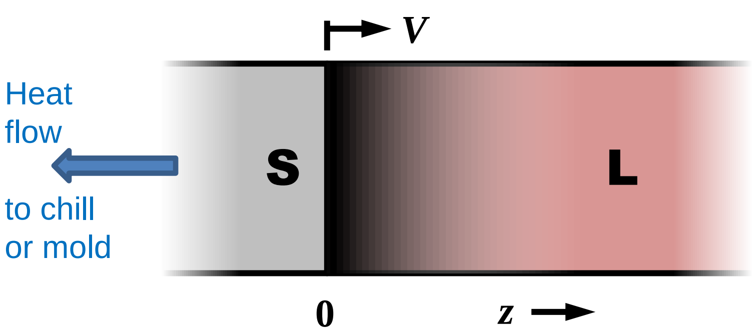
Phase diagram and solute rejection during solidification

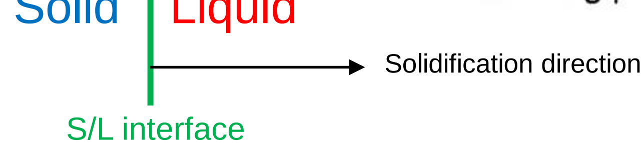
Campbell, J., *Castings*, Butterworth Heinemann, Oxford, 1991.



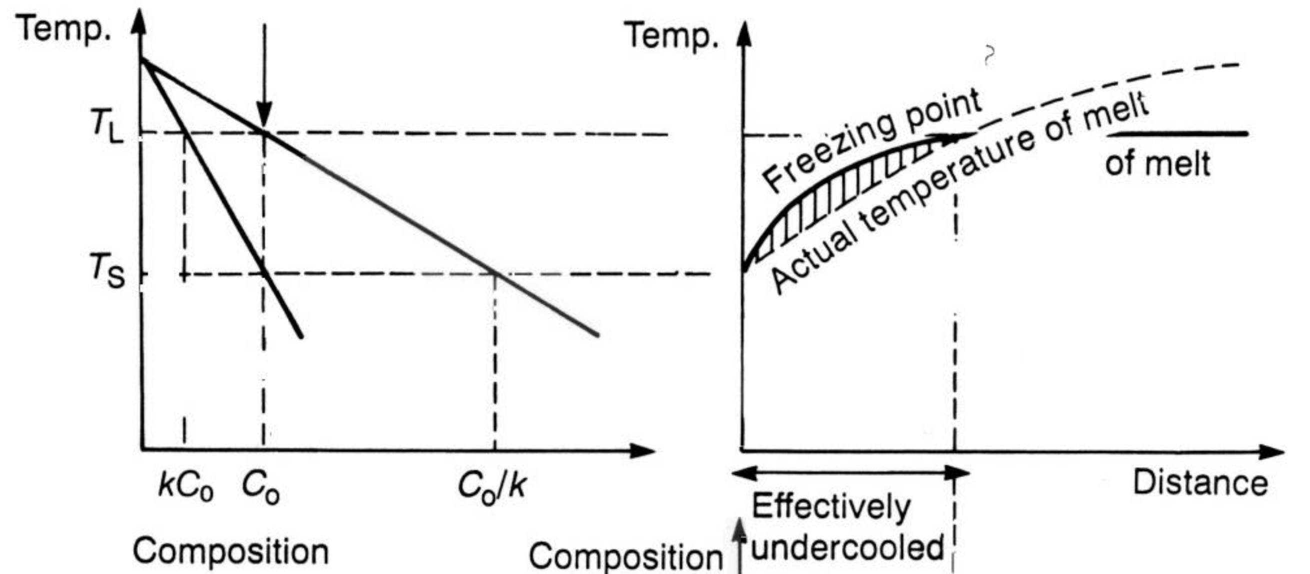


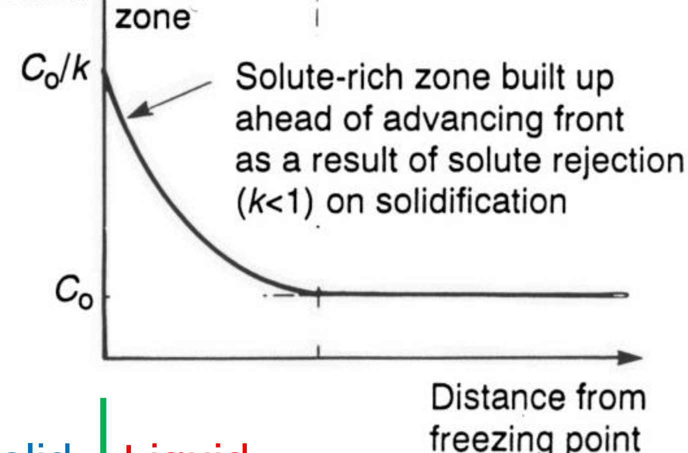
the link between the phase diagram for a binary alloy and undercooling



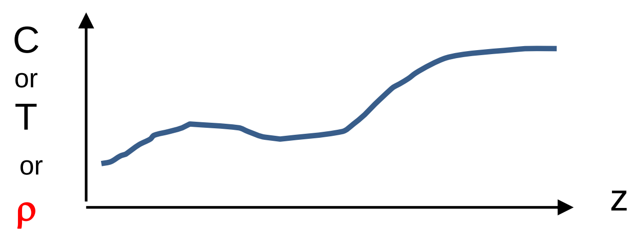


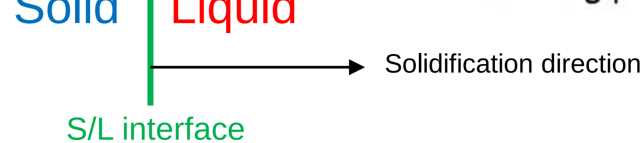
Phase diagram and solute rejection during solidification





recall:





Metallic alloy microstructure

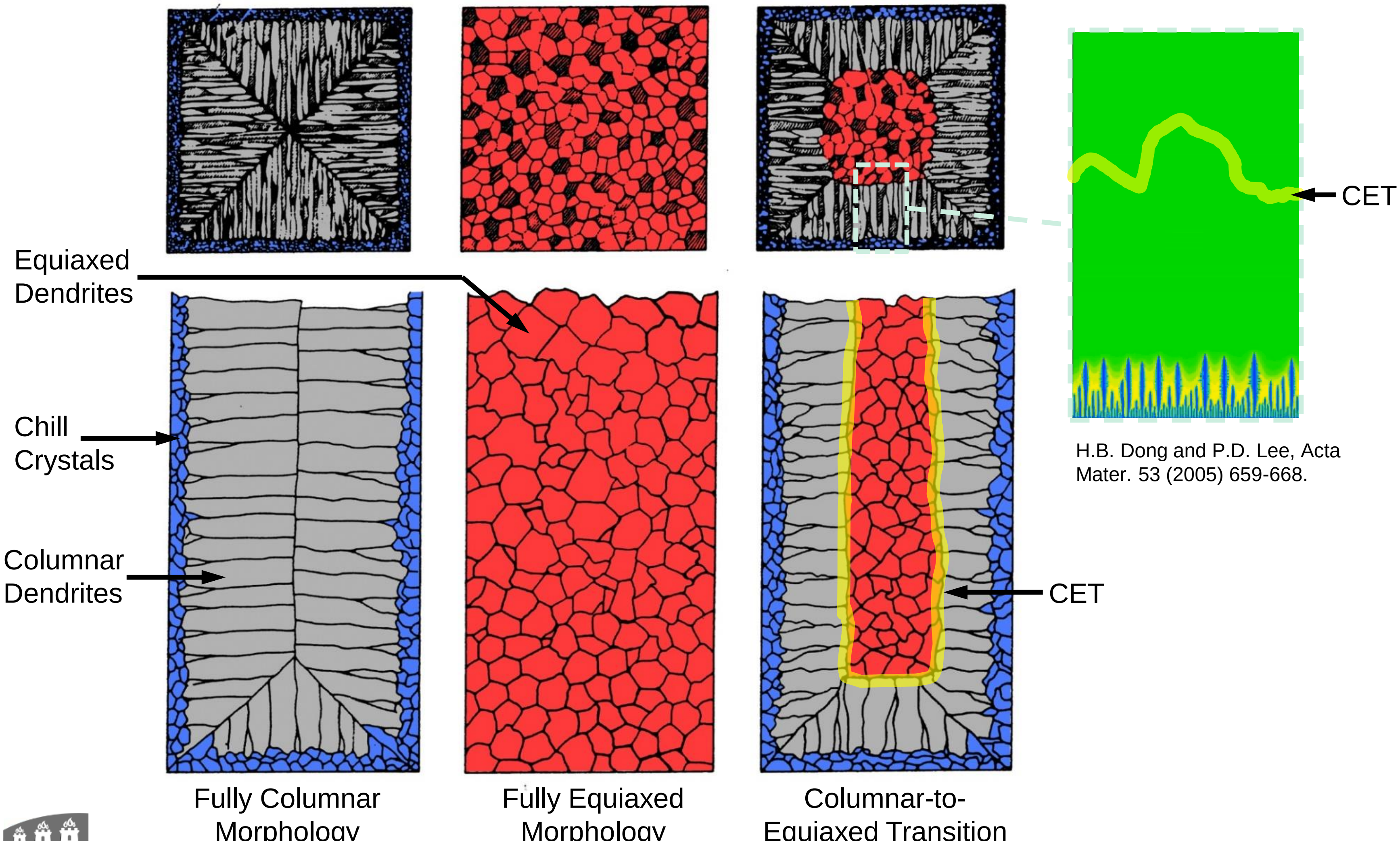
Columnar

Al-7%Si

Equiaxed

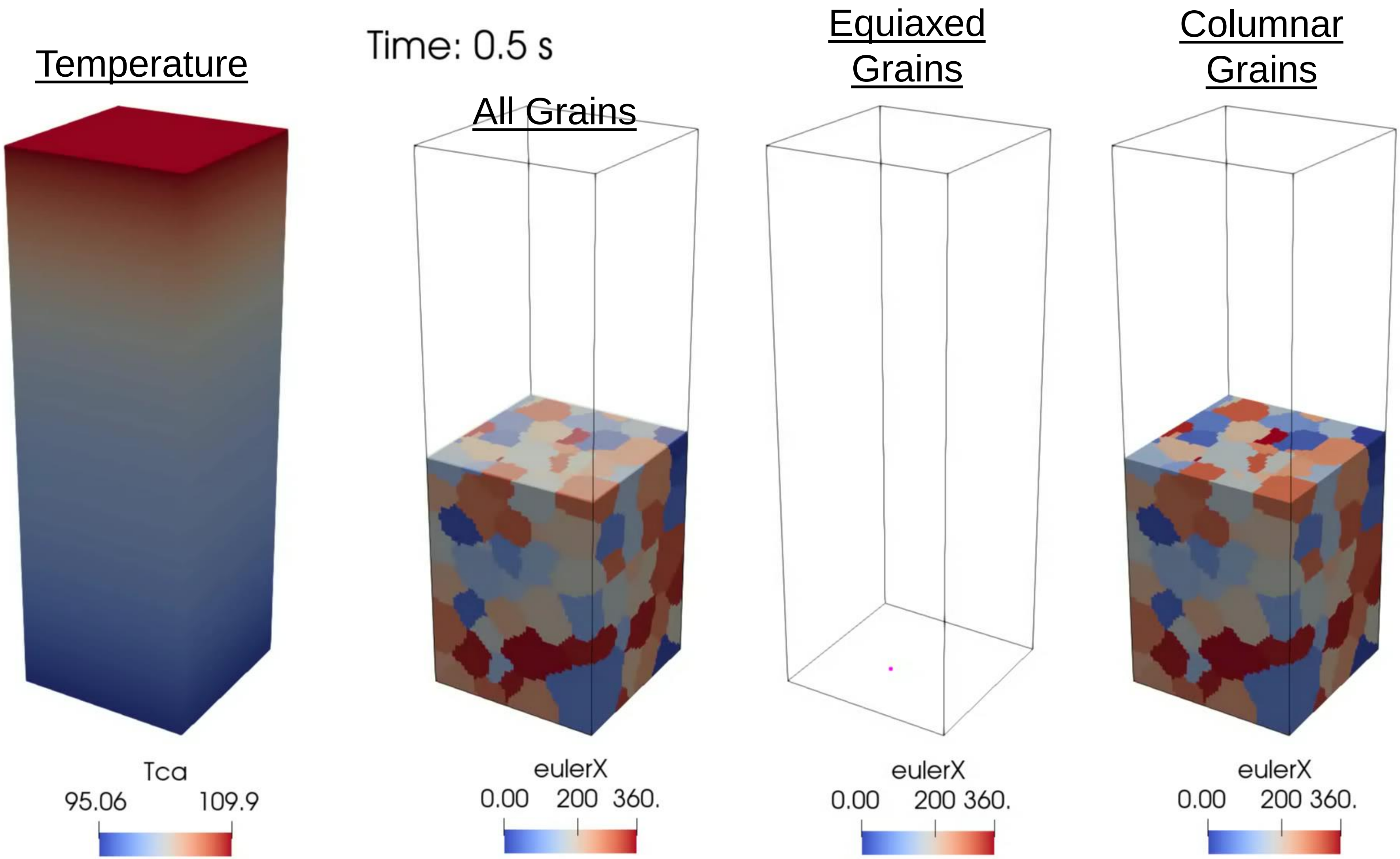
Columnar to Equiaxed Transition (CET)

Columnar-to-Equiaxed Transition (CET)



H.B. Dong and P.D. Lee, Acta Mater. 53 (2005) 659-668.

Direct Modelling of CET @ UCD



UCD DUBLIN

Computational Method: Cellular Automata
Courtesy: Daniel Dreelan, UCD PhD student

Science Foundation Ireland sfi For what's next

I-Form Advanced Manufacturing Research Centre

Microstructure Formation
- Physical Phenomena -

- Nucleation
- Dendrite growth
- Heat flow
- Fluid flow – Convection
- Sedimentation of solid dendrites
- Solute redistribution

etc..

Solidification Modelling - Challenges

- Process complexities
  - Nucleation, Dendrite growth
  - Fluid flow – natural thermo-solutal convection*
  - Sedimentation of solid dendrites*
  - Solute redistribution
- Scales of Modeling
  - Computational resources and time
- Fundamental knowledge
  - Dendrite kinetics

*The physics (and modelling) can be simplified by removing gravity.

Project: XRMON

XRMON:
In Situ **X-R**ay **Mon**itoring of Advanced Alloy Solidification Processes under Microgravity and Terrestrial Conditions

Overall Objectives

- To generate new knowledge on solidification and mass diffusion in liquid metallic alloys by in-situ and real-time radiography.

- To develop a compact experimental environment for such in-situ monitoring.

- **By use of microgravity platforms, to assess the effects of gravity on such solidification and chemical diffusion: parabolic flights, sounding rockets and ISS.**

- To produce benchmark data on gravity-free metallurgical processes, for use as validation material for computational modelling.

Current XRMON partners

IM2NP/Unversite Aix-Marseille, France

Federal Institute for Materials Research and Testing (BAM), Berlin Germany

German Aerospace Centre (DLR), Koln, Germany

University College Dublin, Ireland

NTNU – Trondheim
Norwegian University of Science and Technology

Norwegian University of Science and Technology (NTNU), Trondheim, Norway

ACCESS e.V, Aachen,Germany

esa

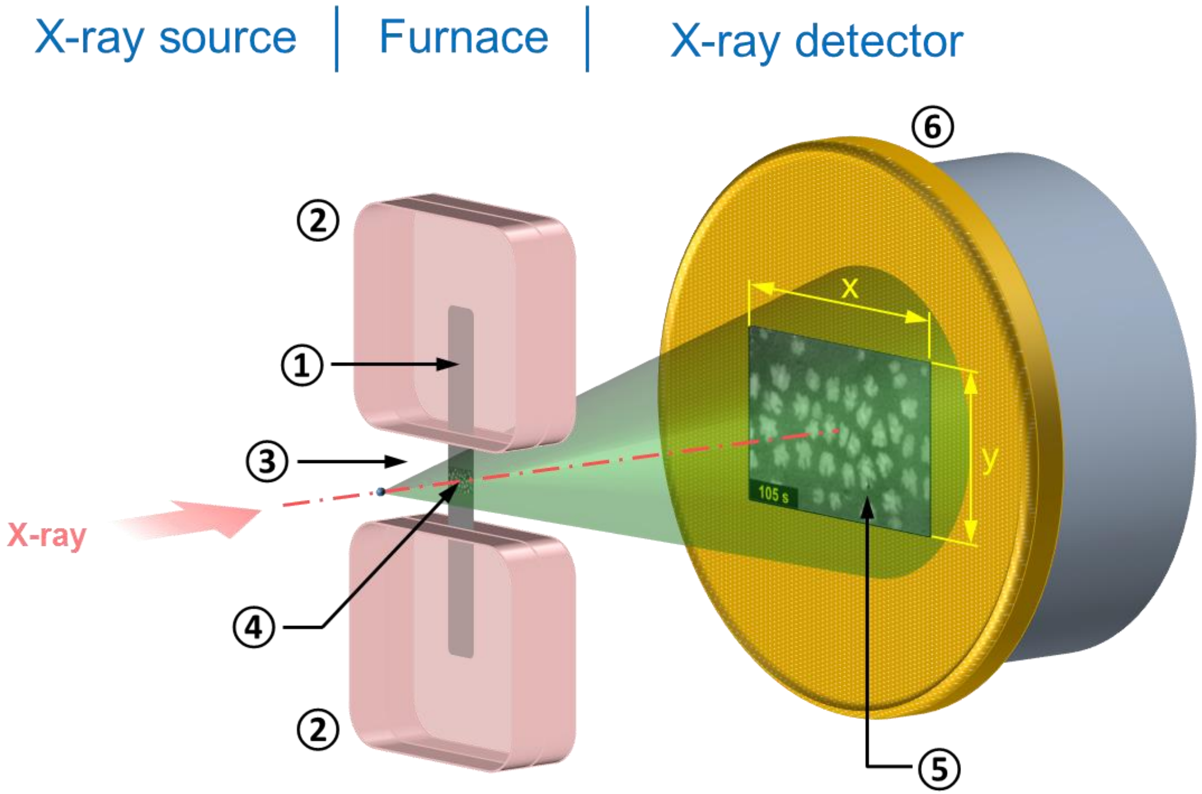
Thermo-Calc Software AB, Stockhom, Sweden

Innoval Technology Ltd., Banbury, UK

Scientific co-operation:
A. Karma (Northeastern University, Boston USA);
C. Beckermann (University of Iowa, Iowa City, USA)

Slide courtesy: Dr Wim Sillekens, ESA

X-ray monitoring of materials processes: principle[1)]



1) Nguyen-Thi H., Reinhart G., Salloum Abou Jaoude G., Mathiesen R.H., Zimmermann G., Houltz Y., Voss D., Verga A., Browne D.J., Murphy A.G.; "XRMON-GF: A novel facility for solidification of metallic alloys with in situ and time-resolved X-ray radiographic characterization in microgravity conditions"; *Journal of Crystal Growth* 374 (2013): 23–30

Achievements

Enabling technology has been developed:

Compact hardware (X-ray source, furnace, and detector) suitable for use on parabolic flights and sounding rockets. Three variants of furnace – gradient (GF), isothermal (SOL) and diffusion (DIFF).

Image enhancement and analysis software.

Currently designing a module for the ISS.

Multiple terrestrial experiments have been inspired by the XRMON work.

Multiple microgravity experiments have been performed.

Many results have been published.

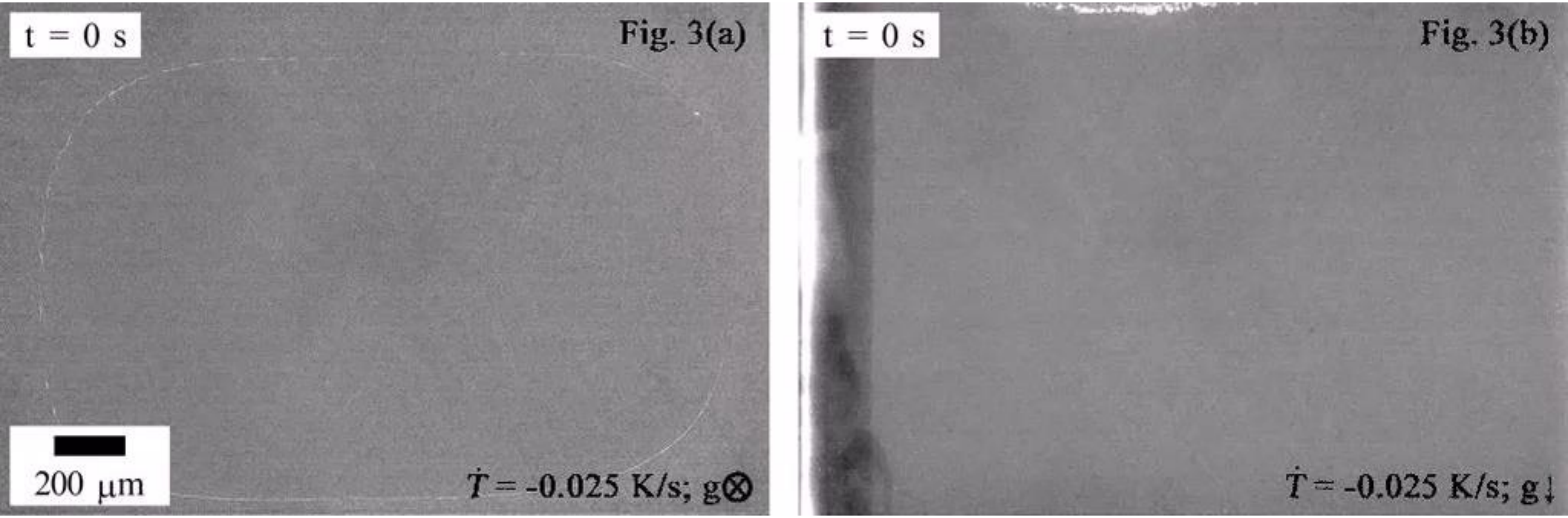
Terrestrial experiments

Equiaxed solidification of thin Al-Cu samples

NTNU – Trondheim
Norwegian University of Science and Technology

Horizontal sample

Vertical sample



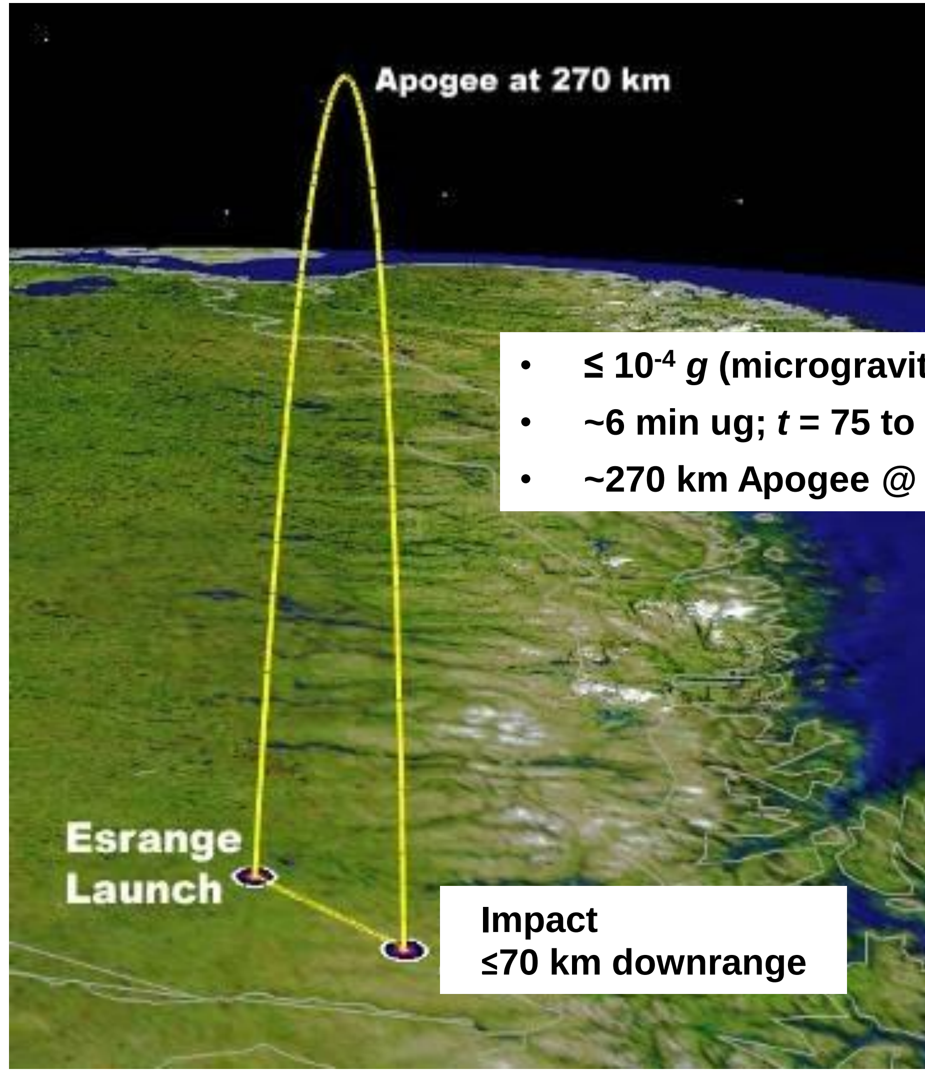
Al-rich solid dendrites float due to their lower density

Furnace: XRMON-GF, as reported in

Murphy, A.G., Mirihanage, W.U., Browne, D.J., Mathiesen, R.H., "Equiaxed dendritic solidification and grain refiner potency characterised through in situ X-radiography", *Acta Materialia* **95**, 2015, pp. 83-89

Sounding Rockets:
MASER = Materials Science Experimental Rocket

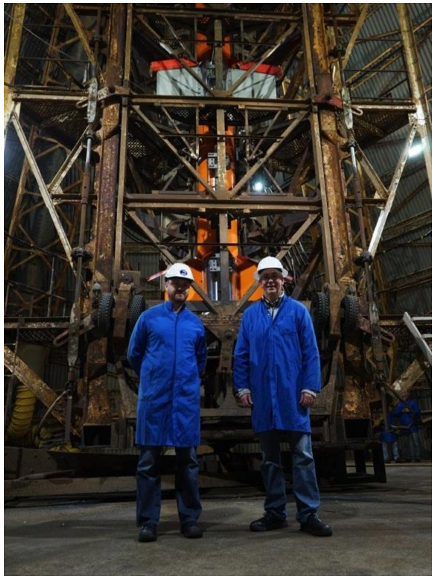
*MASER 11



- ≤ 10^{-4} *g* (microgravity)
- ~6 min ug; *t* = 75 to 435 s
- ~270 km Apogee @ *t* = 260 s

Impact
≤70 km downrange

MASER 13 Team, November 2015



MASER 13
XRMON-SOL | BIM-3 | MEDI | CDIC-3

Key experiments completed

Sounding rocket flights (microgravity):

MASER: MAterials Science Experimental Rocket; Swedish Space Corporation, Esrange launch range, Lapland, Sweden. 6 minutes microgravity.

Maser-11:	Foaming experiments (2008)
Maser-12:	Columnar Solidification (2012)
Maser-13:	Equiaxed Solidification (2015)
Maser-14:	Columnar-to-Equiaxed Transition (2019)

Parabolic flights (varying g):

ESA-Parabolic Flight (PF) campaigns; Novespace, Bordeaux, France (2013-)

Sounding Rocket Experiments

Maser-11 Foaming experiments (2008)

Courtesy of Prof. Francisco García-Moreno
Technische Universität Berlin

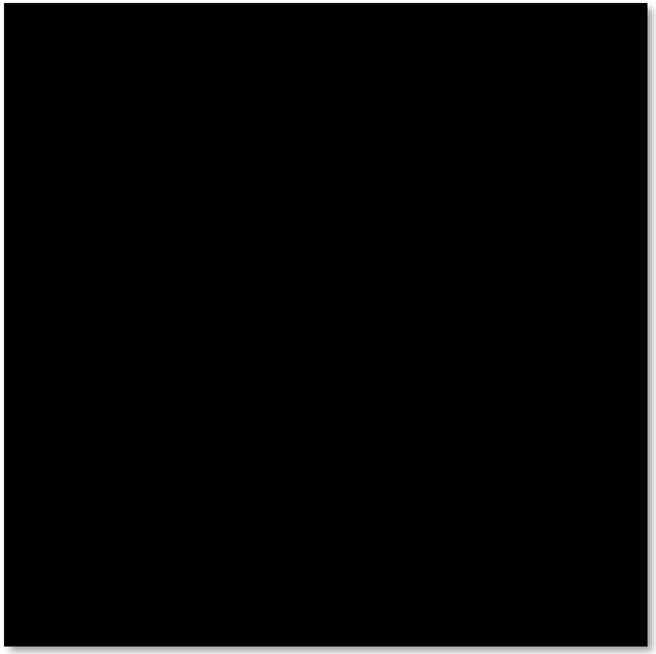
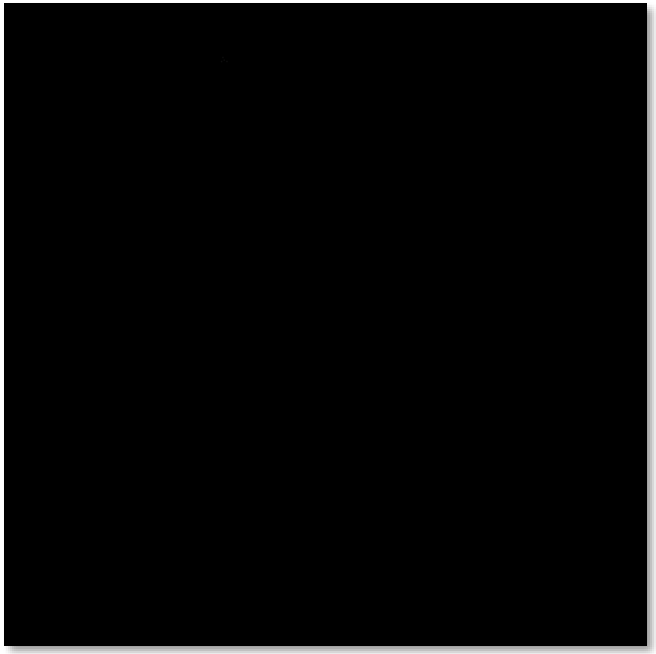
ADVANCED MATERIALS PROCESSING

Slide courtesy: Dr Wim Sillekens, ESA

in-situ observation of metallic foam formation on sounding rocket MASER 11

($AlSi6Cu4+TiH_2$)

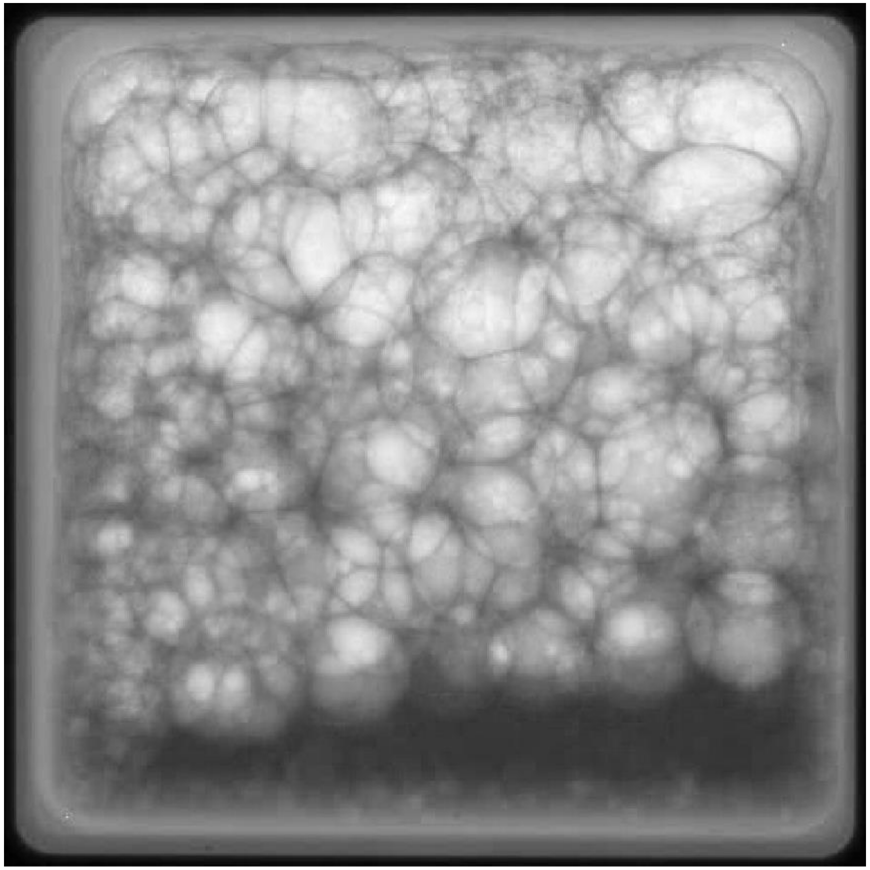
10 mm

Ground (vertical arrangement)	Flight
	

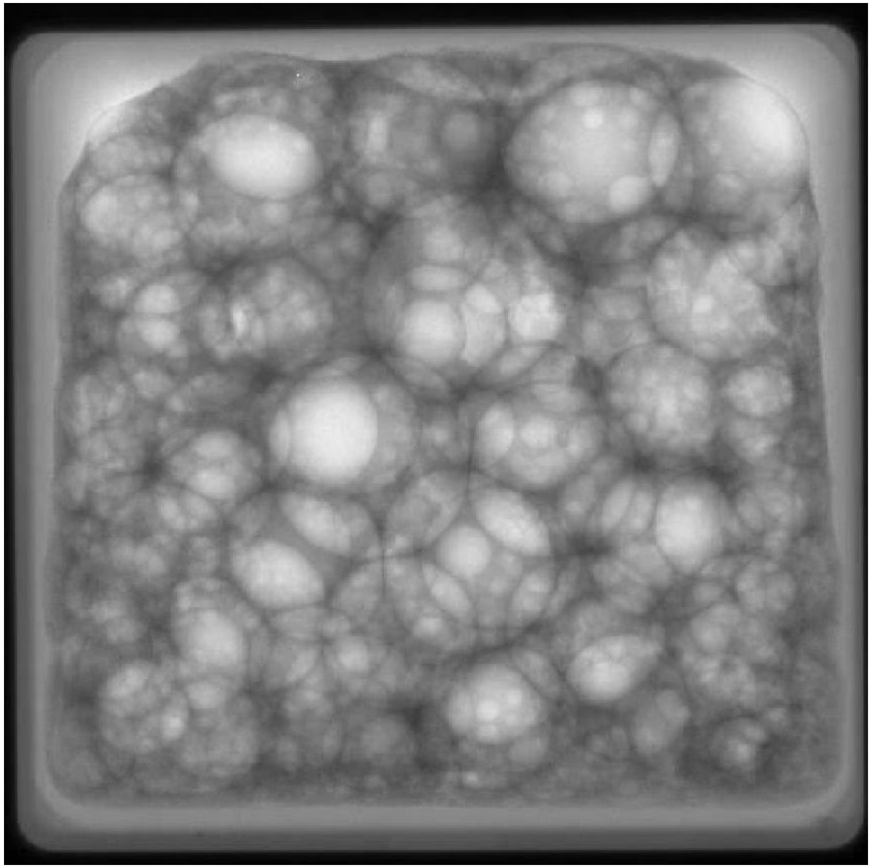
Garcia-Moreno F., Mukherjee M., Jimenez C., Banhart J.; “X-ray radioscopy of liquid metal foams under microgravity”; *Transactions of the Indian Institute of Metals* 62/4–5 (2009): 451–454

MASER 11 – X-ray radiographic analysis

gravity



microgravity



10 mm

Videos x25

MASER 11

Thixo AlSi6Cu4 + 0.6wt% TiH_2

Sounding Rocket Experiments

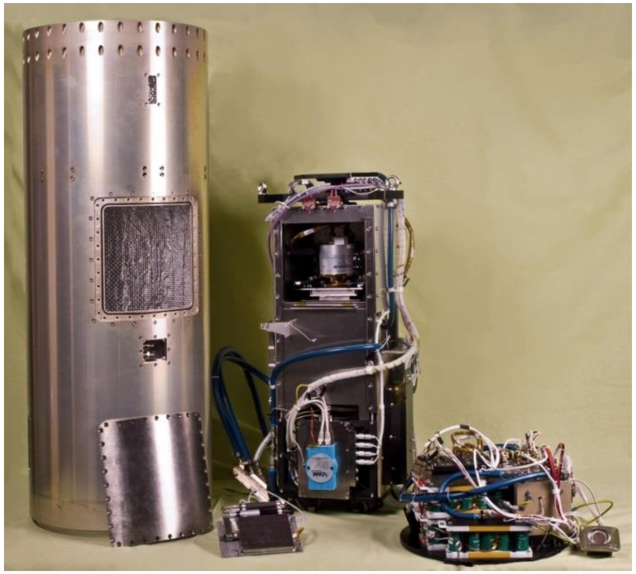
Maser-12 Columnar Solidification (2012)

Courtesy of Prof. Henri-Nguyen-Thi
Universite d'Aix Marseille

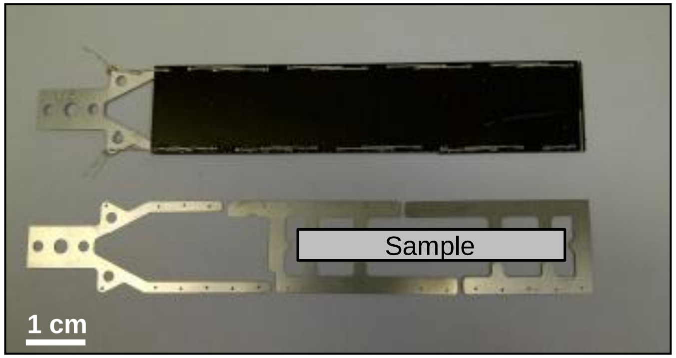
MASER-12 module and XRMON-GF

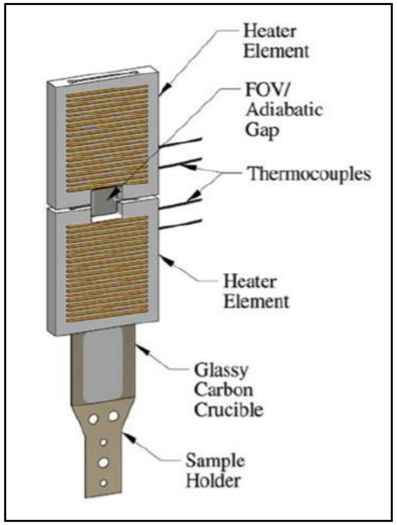
- XRMON activity (2010-2013) :
  - → Development of a dedicated apparatus (XRMON-GF) to perform the first ever **metal alloy solidification in microgravity, with *in situ* observation**

XRMON-GF experiment module on MASER 12



- **Sample size:**
  50 mm x 5 mm x **~ 0.2 mm**
- **Crucible:**
  - Flexible glassy carbon sheets
  - Stainless steel frame





MASER-12 campaign (Esrange, Kiruna, Sweden)

- MASER 12 was launched on 13 February 2012.
- ≈ 6.5 minutes of good microgravity ($10^{-5/-4}$g)
- The experiment was executed according to the pre-programmed sequence and no operator interaction was needed.
- Landing was smooth and payload returned in good condition to Esrange after approx. 2 hours.
- Flight temperature data was down-loaded on flight day and images the day after as soon as support equipment could be retrieved from launch tower.

Im2np Aix Marseille université cnrs

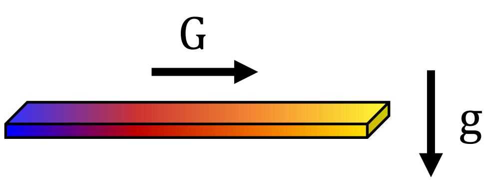
Directional solidification experiments

- 1 experiment in **µg** conditions (MASER-12)

Duration ≈ 5 min around ≈ 3 min of solidification in the FoV

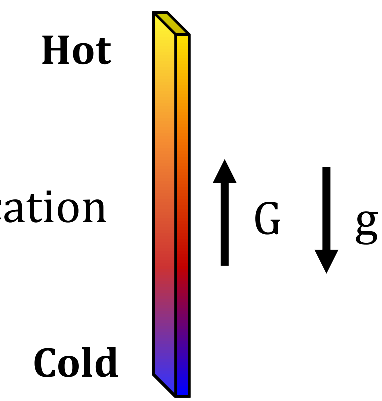
H. Nguyen-Thi et al., J. Cryst. Growth., 374(2013)

- 2 references at **1g** (identical thermal profile)



❖ Horizontal solidification

❖ Upward solidification



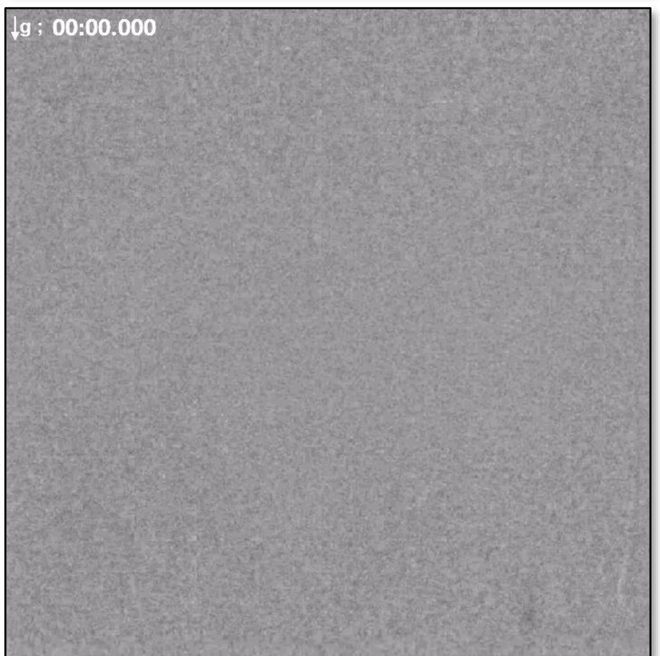
Nguyen-Thi, H., *et al.*, *Microgravity Sci. Technol.* **26**, 2014, 37-50.

Slide courtesy: Dr Wim Sillekens, ESA

in-situ observation of columnar solidification on sounding rocket MASER 12 Al-20wt.%Cu alloy

thermal gradient=15 K/mm, cooling rate =-0.15 K/s, Field of View=5×5 mm)[1)]

Ground (vertical arrangement)



hot side

cool side

Flight

1) Nguyen-Thi H., Browne D.J., Zimmermann G., Reinhart G., Murphy A., Salloum-Abou-Jaoude G., Abou-Khalil L., Mathiesen R., Sillekens W.; "Overview of in-situ x-ray studies of metal alloy solidification in microgravity conditions: The XRMON project"; *Proceedings of the 6th Decennial International Conference on Solidification Processing (SP17)*; Brunel University, London UK (2017): 292–295

Maser-12: Conclusions

- **MASER-12 / XRMON-GF:**

  First experiment using a device combining X-ray radiography and a gradient furnace for solidification study in microgravity

- **Key results were obtained on:**
  - Growth rates vs sample orientation
  - Fragmentation of columnar dendrites in both µg and 1g experiments
    - In 1g-Upward fragments floated due to buoyancy
    - In µg fragments moved to the cold side due to shrinkage

Sounding Rocket Experiments

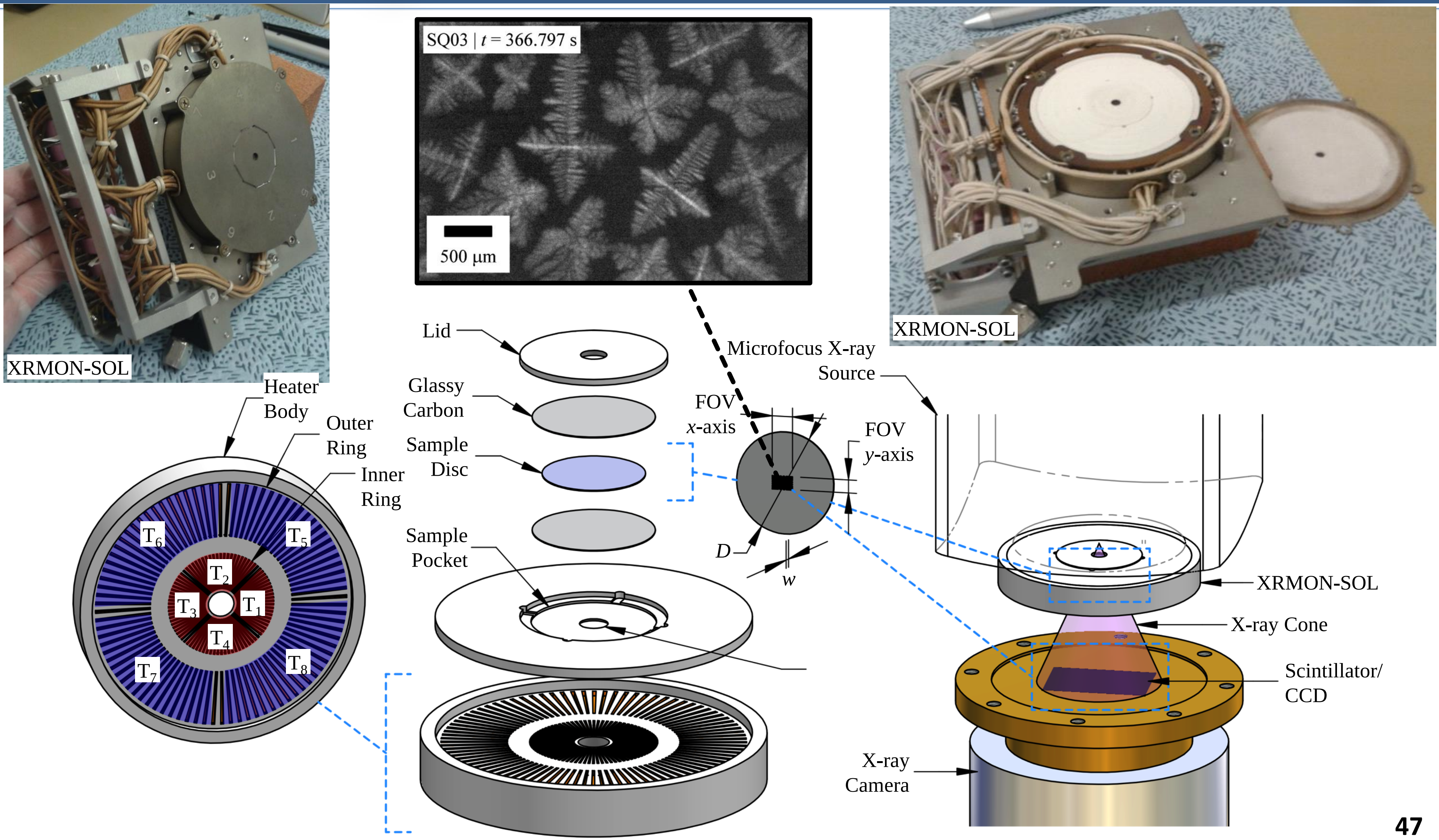
Maser-13 Equiaxed Solidification (2015)

Prof. David J. Browne
University College Dublin (PI)

Maser-13 Sounding Rocket Mission: Equiaxed Solidification

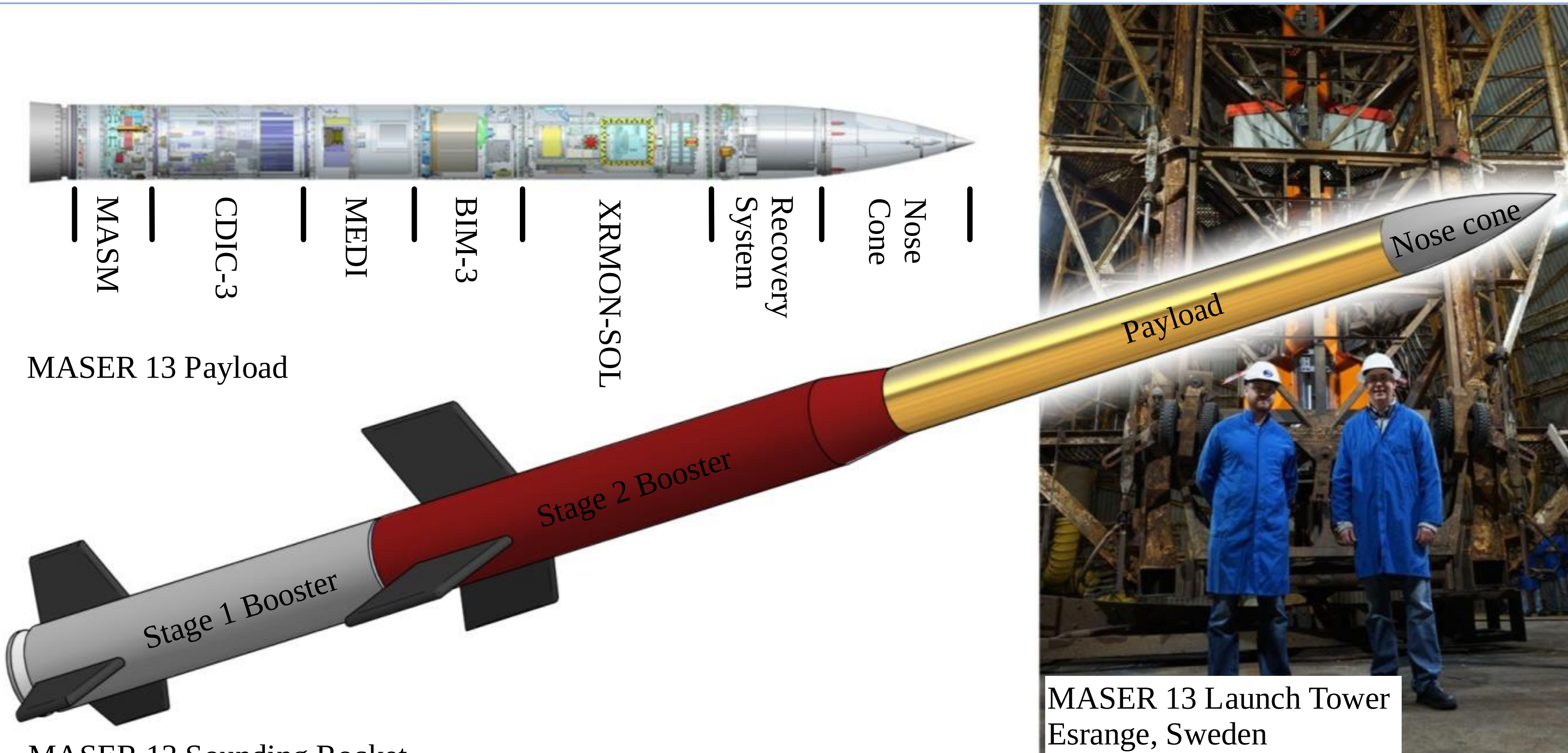
NTNU – Trondheim
Norwegian University of Science and Technology

Furnace for equiaxed solidification: XRMON-SOL



Murphy, A.G., *et al.*,
J. Cryst. Growth, **440**, 2016, 38-46

MASER 13: Payload



MASER 13 Payload

MASER 13 Sounding Rocket

MASER 13 Launch Tower
Esrange, Sweden

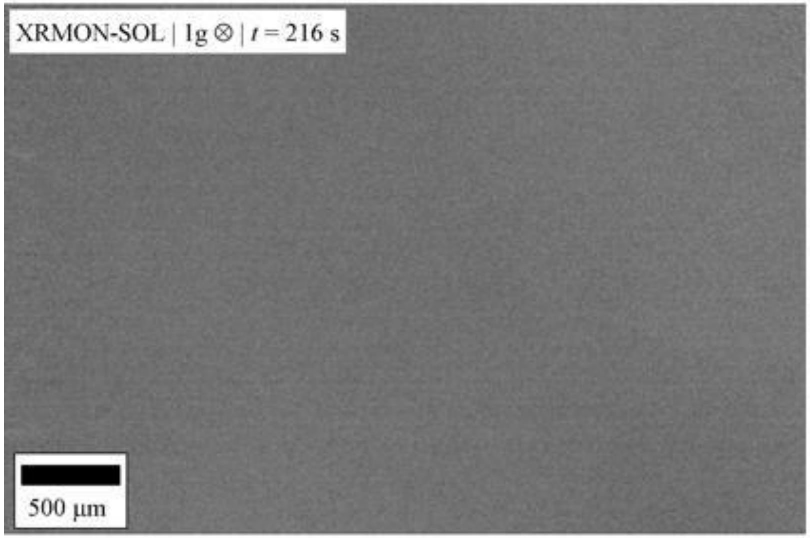
esa

Maser 13 Blast-Off

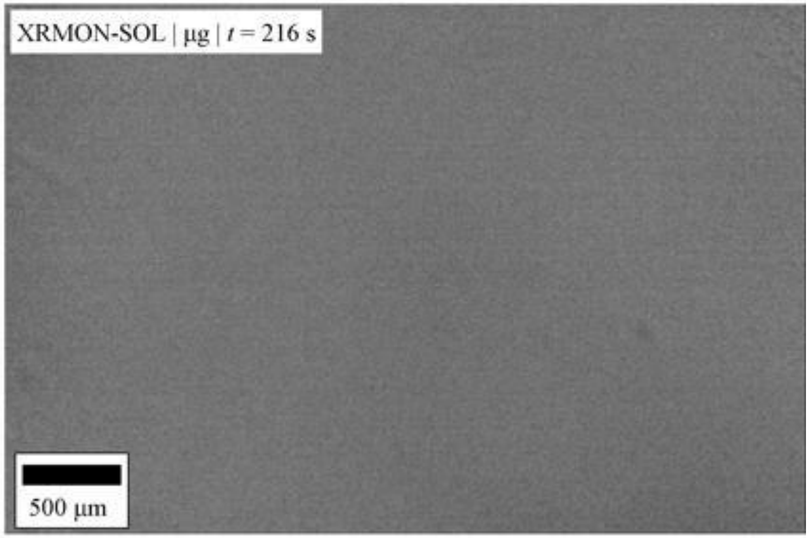
Esrange, Sweden, 06:00, 1 December 2015

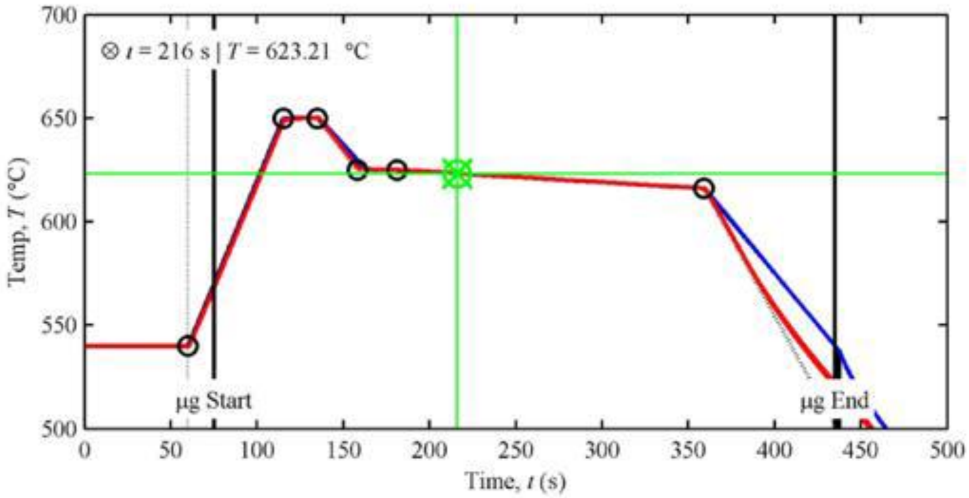
XRMON-SOL: Terrestrial & µg Results

Ground reference test (sample horizontal)



Microgravity experiment (telemetry)

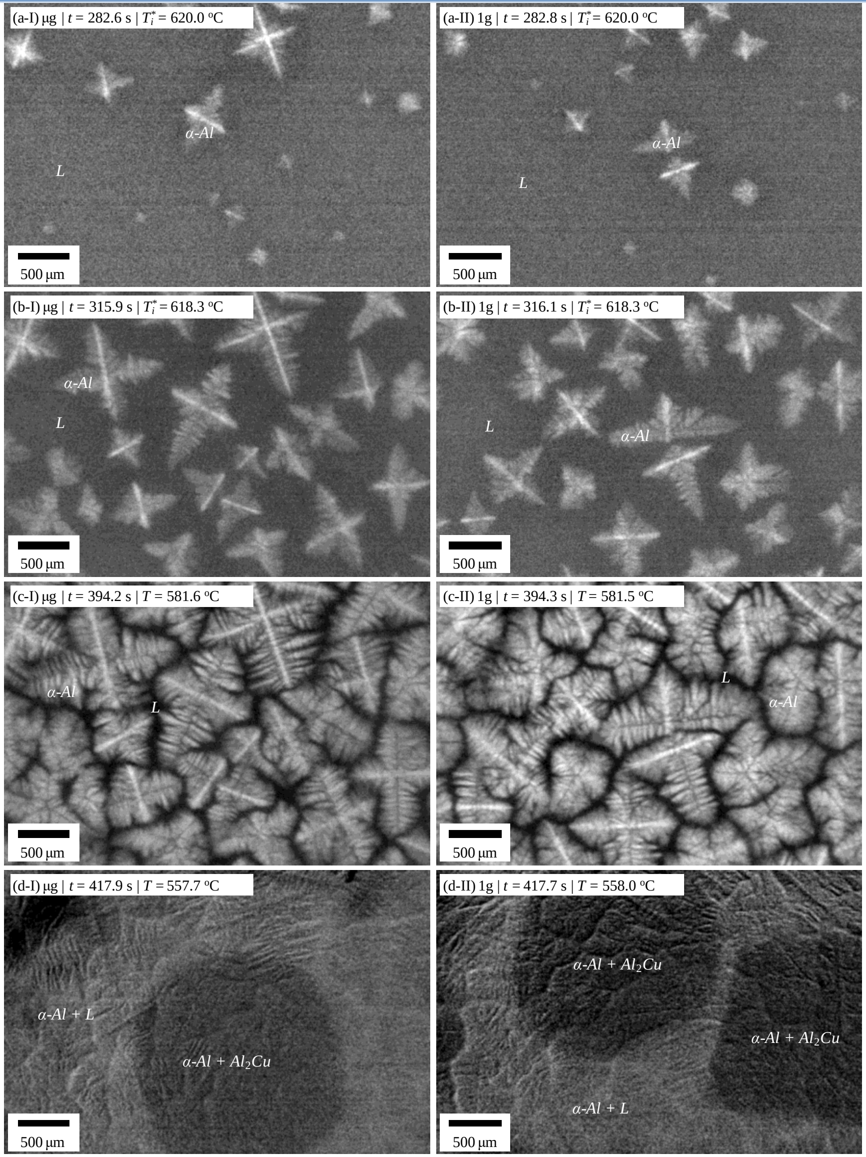




Al-20wt%.%Cu
grain-refined

microgravity terrestrial

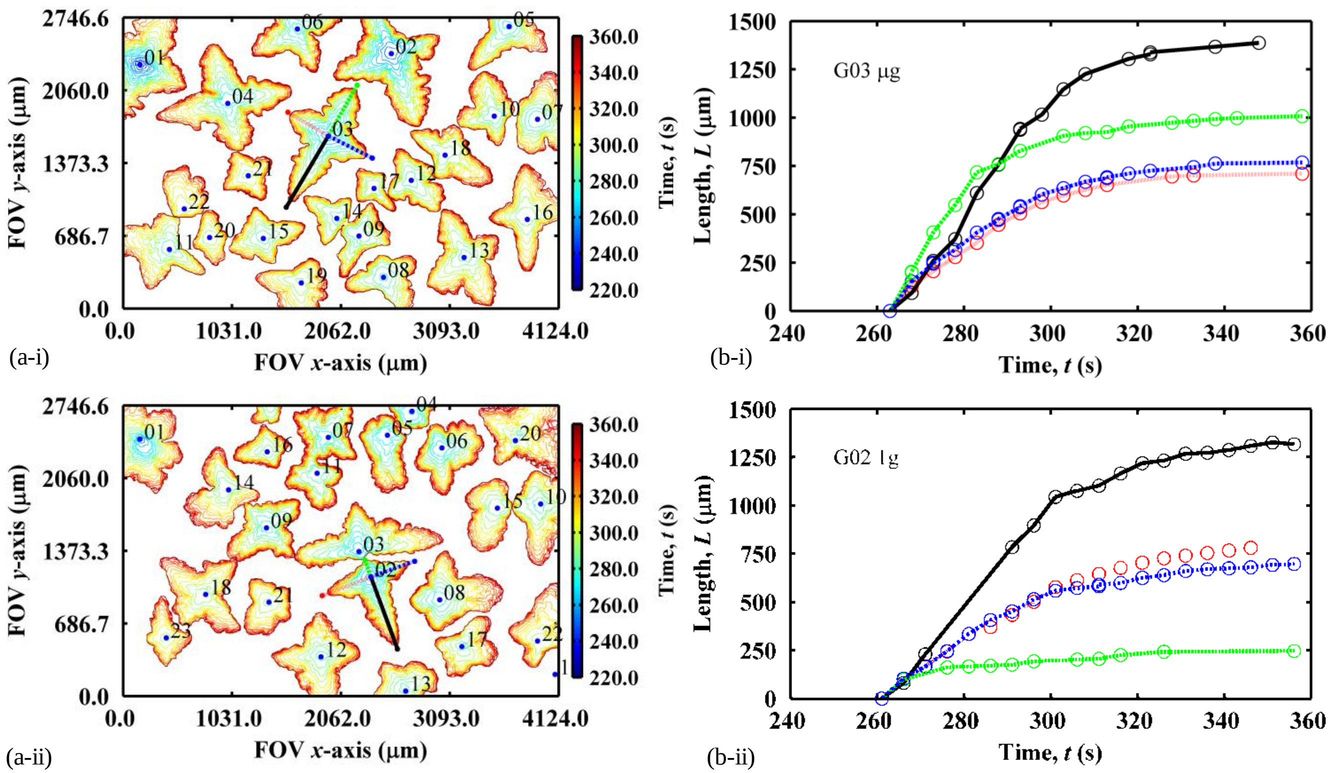
Maser 13: analysis



Maser 13: grain growth

grain envelope evolution recorded as a function of time

primary arm growth as a function of time (t) for selected grains



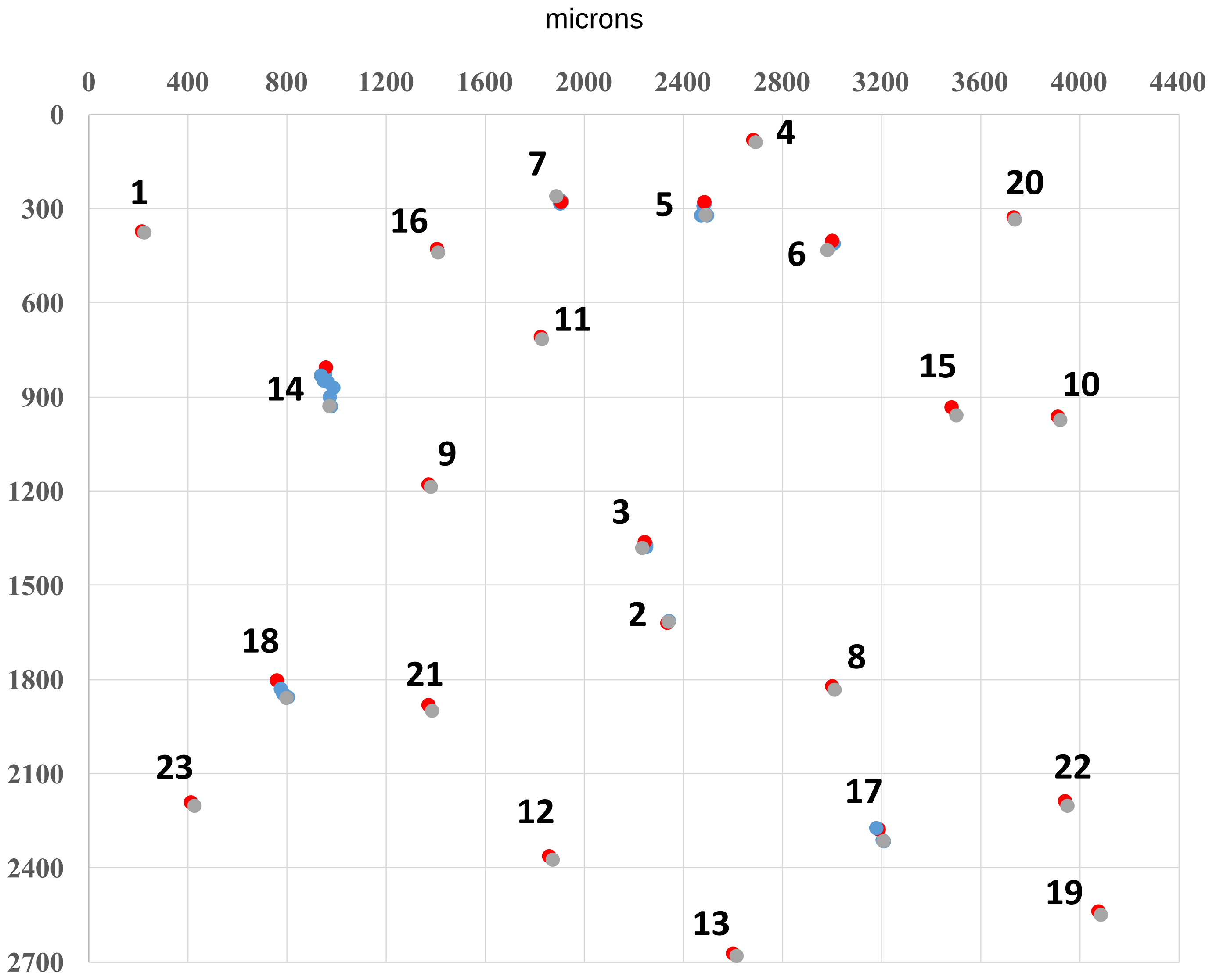
microgravity

terrestrial

Murphy, A.G., *et al.*,
J. Cryst. Growth, **454**, 2016, pp. 96-104

In addition to grain growth, equaxed grain motion has now been quantified …

1g grain motion analysis



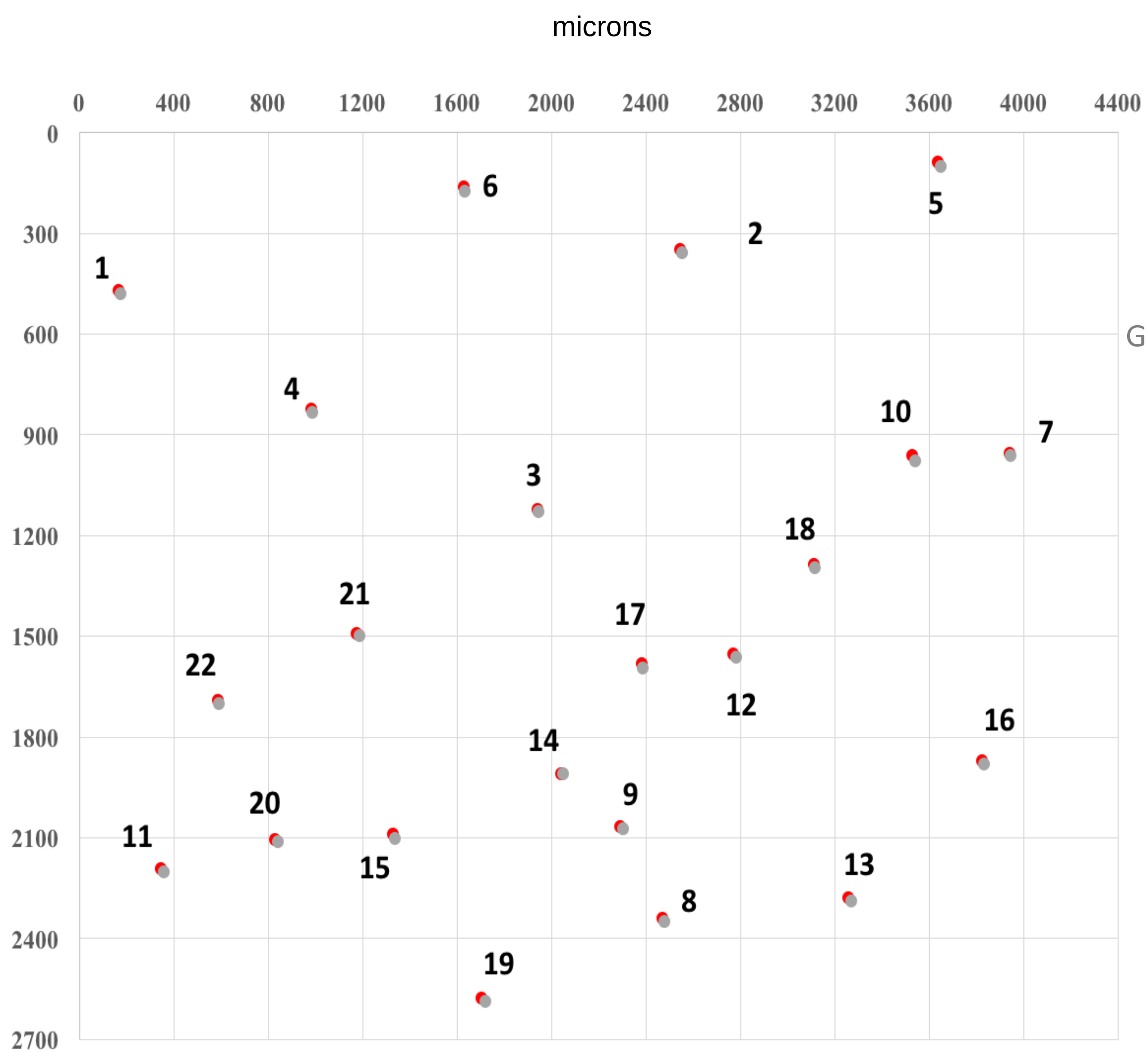
RED: nucleus

BLUE: grain motion (260 - 360 s)

GREY: grain motion (360 - 435 s)

Ground reference experiment – sample horizontal

µg grain motion analysis

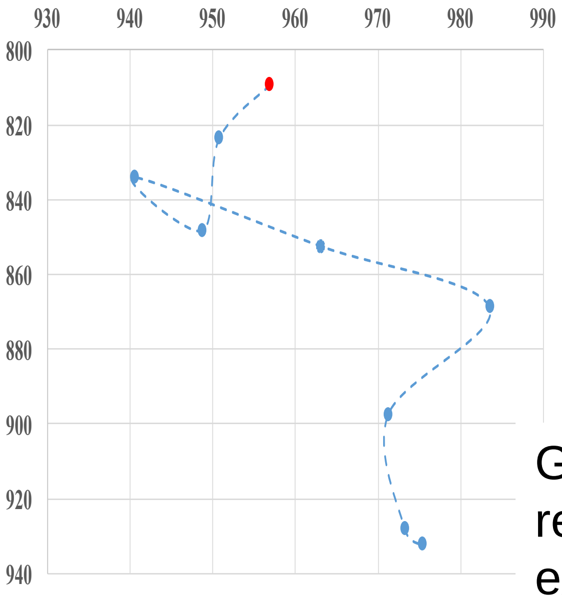


RED: nucleus

No motion prior to 360s

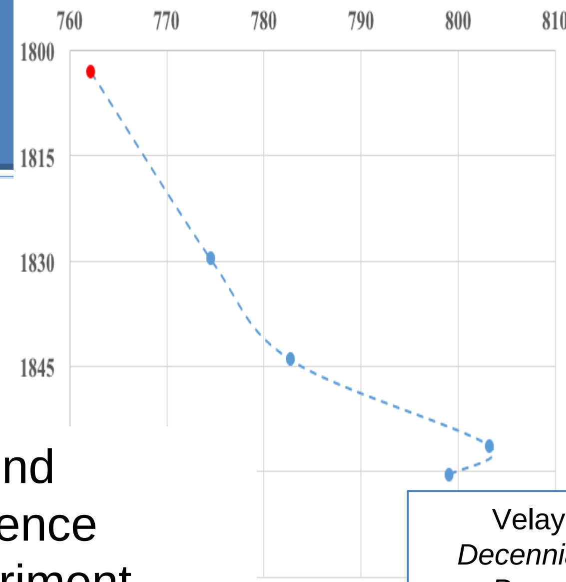
GRAY: grain motion (360-435 s)

Microgravity experiment – Maser 13

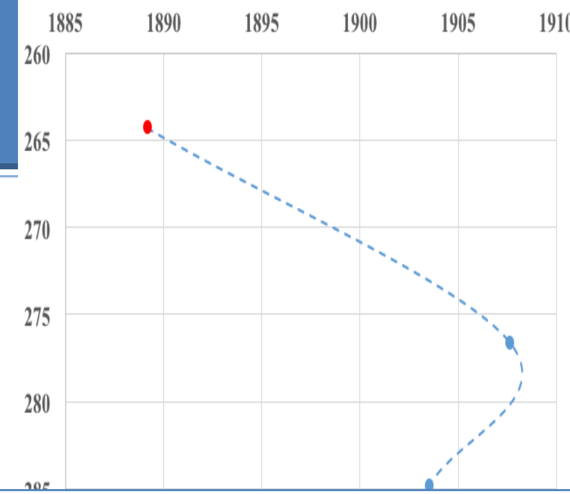


Grain 14

Ground reference experiment

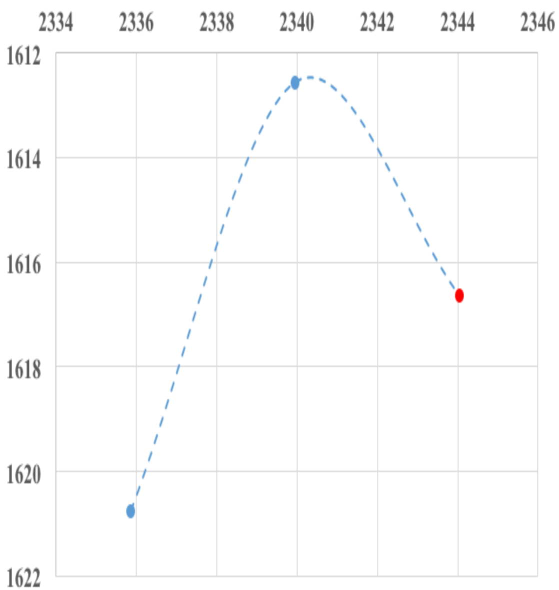


Grain 18

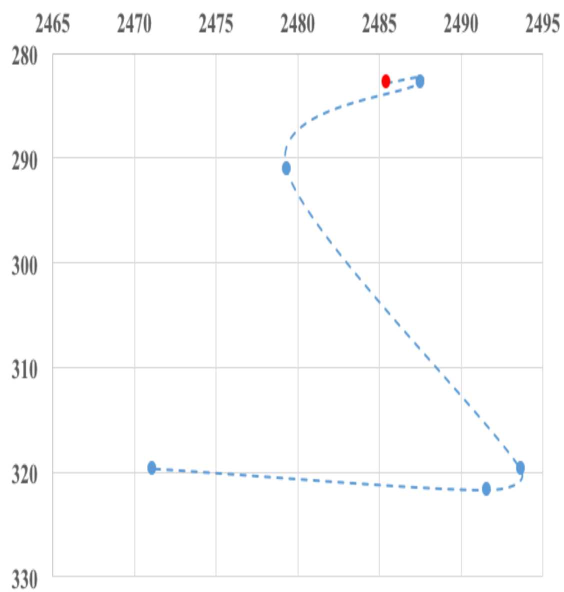


Velayutham, S., Browne, D.J., *Proc. SP17, 6th Decennial International Conference on Solidification Processing*, Old Windsor, UK, 25-28 July 2017

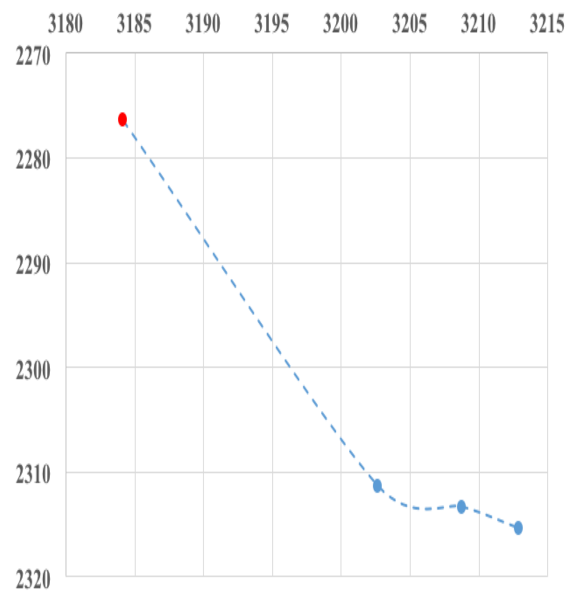
Grain 7



Grain 3



Grain 14



Grain 17

Individual grain motion; microns

Automatic dendrite recognition from Maser 13 video

Machine Learning

Jonathan Mullen, PhD student

Each dendrite is isolated digitally and becomes a separate video.

This enables measurements of growth rates, rotation and impingement to be determined automatically.

This will be applied to legacy, new, and future X-ray videos of solidification, to speed up post-experimental processing and expedite research findings.

Mullen, J., Celikin, M., Cunningham, P., Browne, D.J.,

"A comparison of terrestrial and microgravity isothermal equiaxed alloy solidification through machine learning, multi-stage thresholding and sub-dendrite-based in situ X-ray video processing",

presented at TMS Virtual Annual Meeting & Exhibition, online conference, 14-18 March 2021.

Manuscripts in progress.

An example of the consequences of 'winner take all' sub dendrite allocation, which can result in areas which rapidly switch designations early into the development of a given sub dendrite.

Observations: XRMON-SOL on Maser 13

- Grain rotation and movement clearly evident throughout terrestrial solidification.
- **Complete melting and equiaxed solidification sequence in space monitored by X-radiography.**
- Microgravity-based solidification shows no grain movement early during solidification. Later, past dendrite coherency, some grain motion is observed.
- Solidification shrinkage causing grain motion ahead of the eutectic front visible during microgravity solidification.
- The differences between 1g samples solidified with a horizontal vs. vertical orientation (earlier work) are far greater than those observed between the 1g horizontal experiment and the μg experiment.

Murphy, A.G., *et al.*,
Acta Materialia, **95**, 2015, 83-89.

- **We have isolated shrinkage-induced motion of equiaxed grains.**
- **Machine learning is now being used to interrogate the videos for quantitative data.**

Sounding Rocket Experiments

Maser-14 Columnar to Equiaxed Transition (2019)

Courtesy of
Prof. Henri-Nguyen-Thi
& Prof. Guillaume Reinhart
Universite d'Aix Marseille

Manuscript currently in preparation for journal publication.

Maser rocket studies completed

Maser-11:	Foaming experiments (2008)
Maser-12:	Columnar Solidification (2012)
Maser-13:	Equiaxed Solidification (2015)
Maser-14:	Columnar-to-Equiaxed Transition (2019)

Solidification "trilogy"

Al-20%Cu

Parabolic Flight Experiments

Slides courtesy of
Prof. Henri Nguyen-Thi
University d'Aix Marseille

Other PIs:

Dr Gerhard Zimmermann
ACCESS

Dr David J. Browne
University College Dublin

Parabolic Flight Campaigns: Directional Solidification

- **H. Nguyen-Thi & G. Reinhart**
  Aix Marseille Univ., CNRS, IM2NP, Marseille, France
- **G. Zimmermann**
  ACCESS e.V. & RWTH Aachen, Aachen, Germany

Technical support

- **J. Li** *(SSC, Sweden)*
- **A. Verga, N. Melville** (ESA)

XRMON parabolic flight campaigns

- ESA – PF 58th (4 – 6 June 2013)
- ESA – PF 60th (7 – 11 April 2014)
- ESA – PF 61st (7 – 11 September 2014)
- ESA – PF 64th (25 – 28 April 2016)

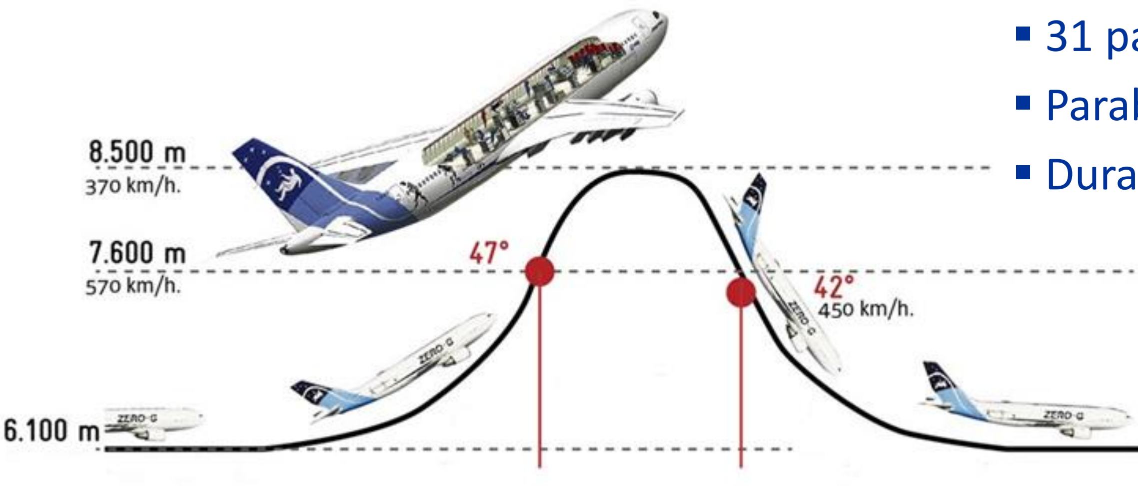
❑ Topic:

Microgravity investigation of directionally solidified columnar grain structures and the Columnar-Equiaxed Transition

Note: Browne et al. also carried out experiments on equiaxed solidification, during PF58 and PF60.

Murphy, A,G. *et al.*, *Materials Science Forum* **790-791**, 2014, 52-58.

Relevance of Parabolic Flight to Solidification

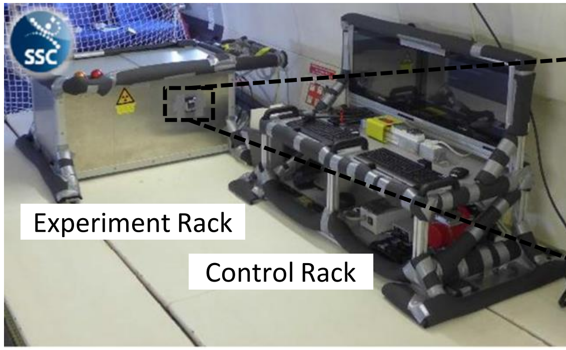


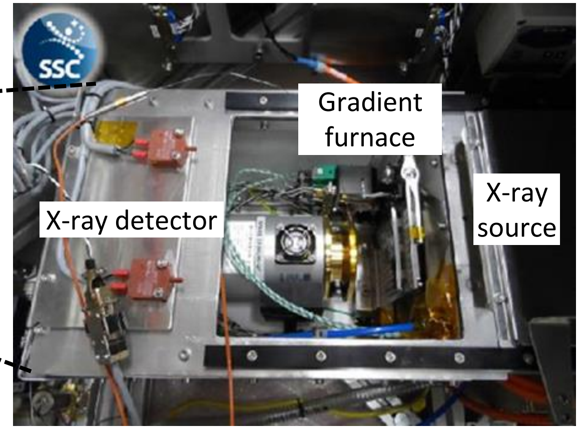
- 31 parabolas during a flight
- Parabolas are grouped in sets of 5
- Duration of the flight = 2-3 hours

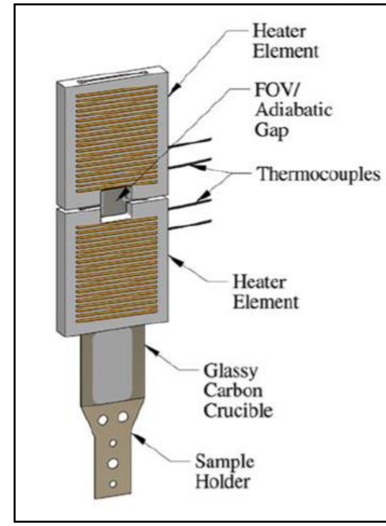
$1\ g_0$	$1.8\ g_0$	$\sim 0\ g_0$	$1.8\ g_0$	$1\ g_0$
120 s	24 s	20 s	24 s	120 s

Step-variations in gravity level during parabola
→ solidification microstructure

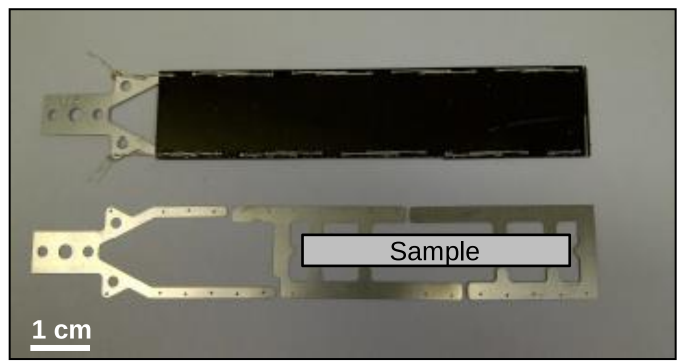
XRMON-PFF (Parabolic Flight Facility)







- **Sample size:**
  50 mm x 5 mm x ~0.2 mm
- **Crucible:**
  - Flexible glassy carbon sheets
  - Stainless steel frame



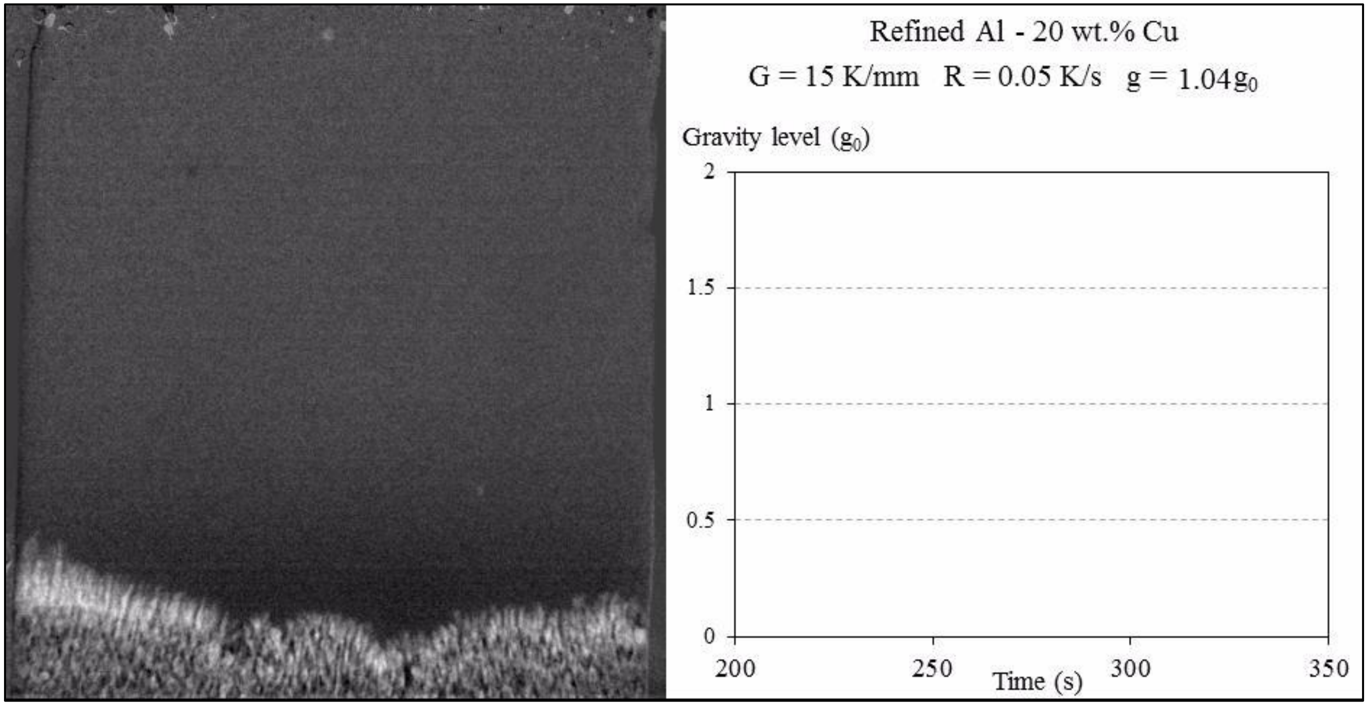
Im2np Aix*Marseille université cnrs access

PF 60th & 61st: CET triggered by step increase of g-level

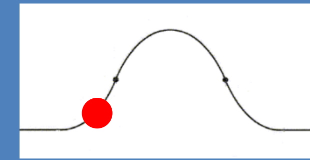
- **Sample: Refined Al – 20 wt.% Cu**
- Temperature gradient G = 15 K/mm
- Cooling rate R = 0.05 K/s (→ columnar growth at 1g)

Duration: 150s

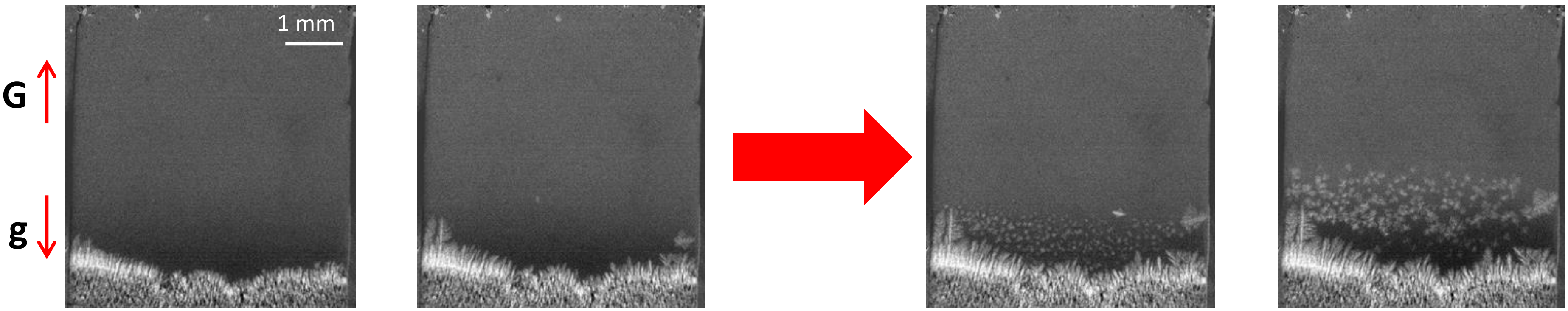
1 mm



CET triggering by increase of gravity level



$1g_0$	$1.8g_0$



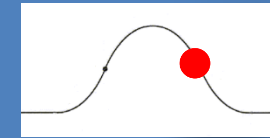
t = 202 s   t = 230 s   t = 240 s   t = 248 s

- Columnar microstructure (V ≈ 3µm/s)
- Dendrite fragmentation
- Upward dendrite fragment motion
  → Melting of the grains

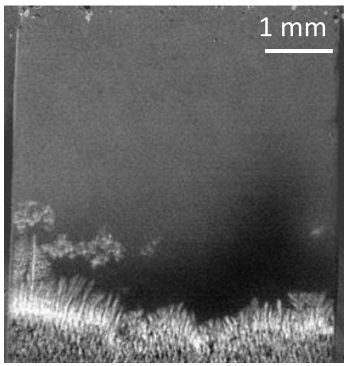
- Sudden nucleation of a large number of equiaxed grains
  → CET
- Upward motion of the grains
  → Melting of the grains

Im2np   Aix*Marseille université   cnrs   access

Second CET triggering by increase of gravity level

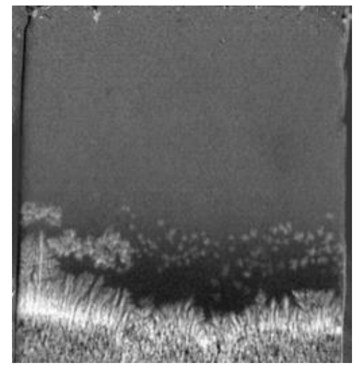


$0g_0$

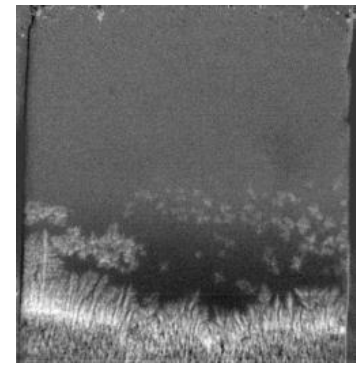


t = 280 s

$1.8g_0$



t = 295 s



t = 302 s

- No grain motion
- No fragmentations

- New nucleation of a large number of equiaxed grains
  $\rightarrow$ CET
- Upward motion of the grains

Parabolic Flights: Conclusion & Perspectives

- **Parabolic Flight Campaign is an efficient tool to study the effects of g-level variation on the microstructure formation**
  - → CET provoked in a refined Al-Cu alloy by the sharp increase of gravity
  - → Equiaxed growth in nearly isothermal furnace of Al – Cu alloys
  - → Further CET experiments, on Al-10wt.% Cu have been carried out
- **Parabolic Flight Campaign is a relatively inexpensive tool to perform preliminary studies in microgravity conditions.**

L. Abou-Khalil, G. Salloum-Abou-Jaoude, G. Reinhart, C. Pickmann, G. Zimmermann, H. Nguyen-Thi, "Influence of gravity level on columnar-to-equiaxed transition during directional solidification of Al–20 wt% Cu alloys". Acta Materialia **110**, 44–52 (2016)

UCD parabolic flight experiments

Equiaxed solidification of grain-refined alloy

See YouTube Video

https://www.youtube.com/watch?v=O3A9uhU4Cx0

Murphy, A.G., Li, J., Janson, O., Verga, A., Browne, D.J., "Microgravity and hypergravity observations of equiaxed solidification of Al-Cu alloys using in-situ X-radiography recorded in real-time on board a parabolic flight", *Materials Science Forum*, **790-791**, 2014, pp. 52-58

Transparent Alloys

Transparent Alloys:

Transparent organic substances form an analogue for metallic alloys, enabling the *in-situ* observation of solidification kinetics using visual microscopy.

These were the first materials* in which *in situ* solidification could be observed, before the advent of advanced X-ray sources and detectors for use with optically opaque metallic alloys.

For some transparent alloys in certain cases solidification is also affected by gravity. For this reason, transparent alloys have also been processed on the ISS and on sounding rockets.

*Jackson, K.A. and Hunt, J.D., Transparent compounds that freeze like metals, *Acta Met.*,**13**, 1212-1215, 1965.

Transparent Alloy Solidification

Slide courtesy: Dr Wim Sillekens, ESA

in-situ observation of equiaxed solidification on sounding rocket MASER 13 (MEDI experiment)[1)]

- Organic alloy NPG–(30 wt.%)dC, T_L=79.3 °C; cell dimensions 10×13×3 mm; Gradient=0.6 K/mm, cooling rate=-0.0125 K/s
- Overview images (FoV=13.6×10.9 mm); µg period 70–460 s, acceleration 11×)

NPG-DC: Neopentylgycol-(d)Camphor (an energy storage material)

Ground (vertical arrangement)

hot side

cool side

Flight

1) Sturz L., Hamacher M., Eiken J., Zimmermann G.; "In-situ observation of growth and interaction of equiaxed dendrites in microgravity"; *Proceedings of the 7th International Conference on Solidification and Gravity*; Miskolc-Lillafüred HU (2018): 90

2) Mooney, R.P., Sturz, L. Zimmermann, G., McFadden, S., "Thermal characterisation with modelling for a microgravity experiment into polycrystalline equiaxed dendritic solidification with in-situ observation", International Journal of Thermal Sciences **125**, 2018, pp. 283-292

Properties of Liquid Alloys

The thermophysical properties of molten alloys are necessary for analysis of many manufacturing process which involve starting via melting – casting, welding, additive manufacturing.

It can be difficult to measure some properties due to interaction with the crucible in which the liquid alloy is contained.

For this reason, containerless processing is often used, in which a sample of the molten liquid is levitated by an electromagnetic field.

This is more readily achieved in zero g conditions – in space.

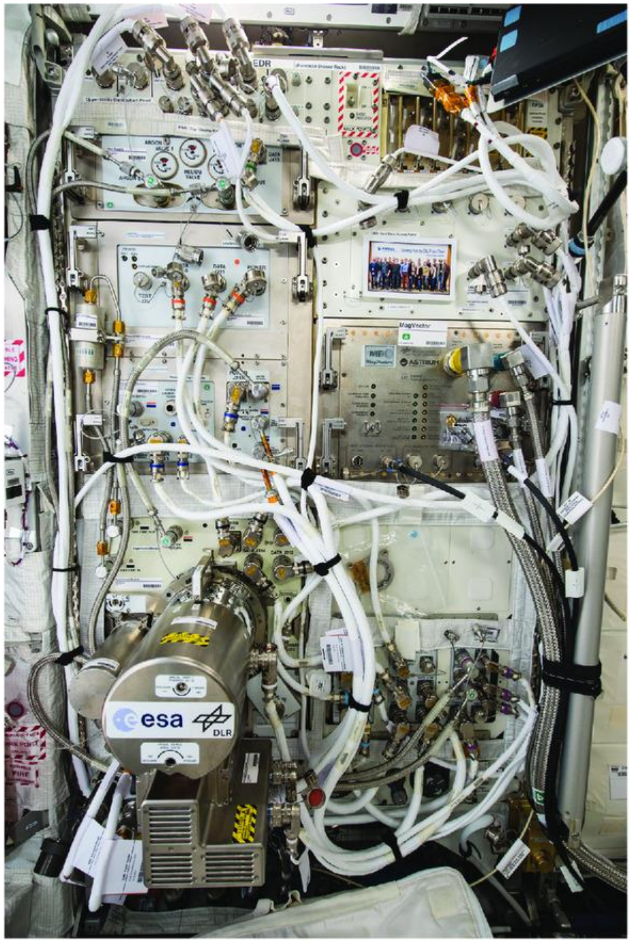


Image: Airbus Defence and Space

Thermophysical property measurement in space

Why do we do property measurement?

- **Improve quality control** and promote sustainable manufacturing development of transformative new energy efficient technologies
- **Support next-generation molten metal manufacturing capabilities** through modeling of casting, welding, single-crystal pulling and additive manufacturing operations
- The **predictive capability of a model** is only as good as the quality of the property data used to generate a simulation

Improve process modeling to achieve UN-SDG

International collaboration is important because space testing is expensive and cost **sharing through use of multi-user facilities** is an effective approach in order to leverage emergent complementary scientific investigations. In support of transnational objectives, **common manufacturing challenges** are identified though sharing of facilities in support of the UN sustainable development goals.

Why do we need microgravity?

- By levitating a molten sample during containerless processing, contamination from container walls is eliminated
- Sedimentation and buoyancy effects are eliminated in microgravity
- Without strong gravitational accelerations and with reduced levitation forces, spherical samples allow fewer deviations from theory improving the measurement accuracy
- Better control of convection results in higher measurement precision

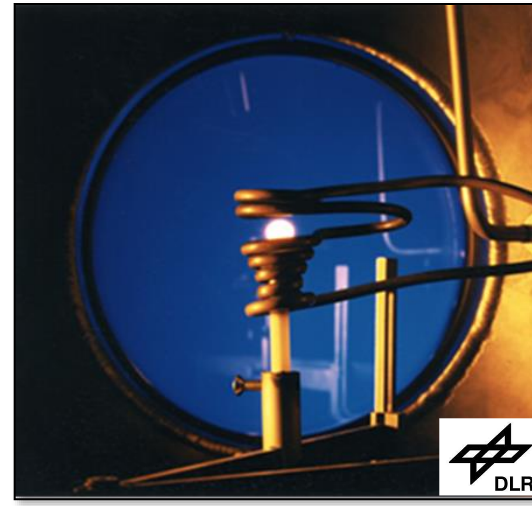
Eliminate gravity-induced systematic error

Slide: courtesy Prof. Doug Matson, Tufts University, USA

How is containerless processing in space accomplished?

Two very different levitation techniques may be employed

- ElectroStatic Levitation (ESL): JAXA Electrostatic Levitation Furnace (ELF)
- ElectroMagnetic Levitation (EML) : ESA/DLR ISS-EML facility.



Typical measurements

- Density changes are monitored by observing how volume changes with temperature using cinematography
- Surface tension tests are accomplished by observing sample oscillation frequency response
- Viscosity tests are run by observing how oscillations dampen once excitation is terminated
- Specific heat capacity measurements are conducted using modulation calorimetry

Sample temperature is monitored using radiation pyrometry
Sample behavior is monitored using high-speed video cameras

Slide: courtesy Prof. Doug Matson, Tufts University, USA

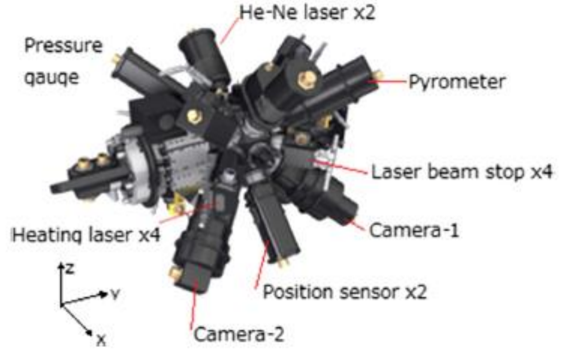
How is ESL accomplished in Space?

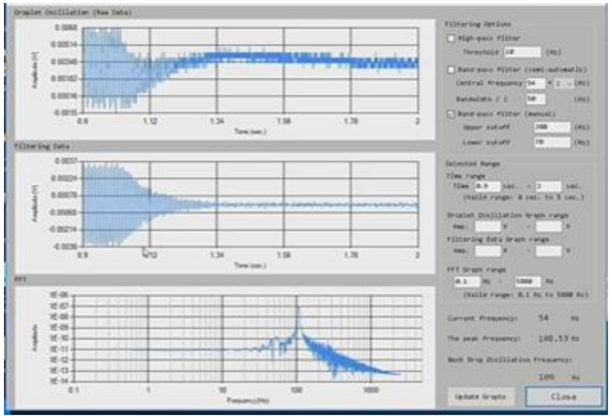
ElectroStatic Levitation
JAXA Electrostatic Levitation Furnace (ELF)

Sample is positioned by charged electrical plates and a complex laser shadow monitoring system is used to keep the sample at the central location

Sample is heated by a series of lasers directed tetrahedrally to minimize surface temperature gradients

Pulses are excited by applying a small amplitude change in the positioning field causing a deformation response at the system natural frequency





Sample deformation

Sample results

Slide: courtesy Prof. Doug Matson, Tufts University, USA

How is EML accomplished in Space?

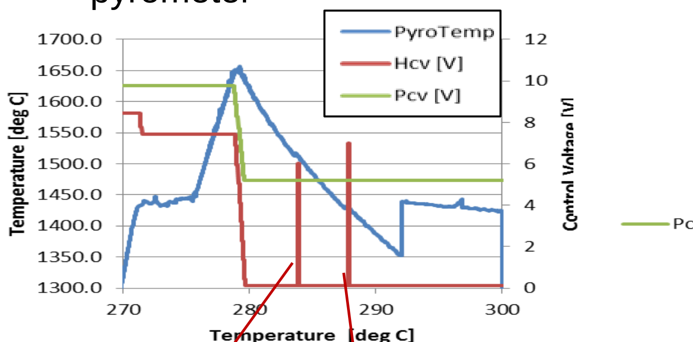
ElectroMagnetic Levitation
ESA/DLR ISS-EML facility

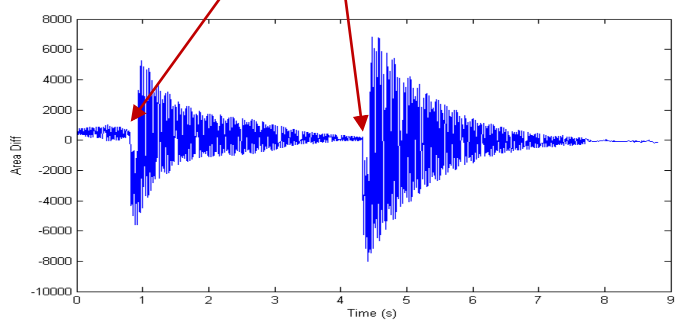
Sample is positioned by magnetic fields produced by HF electric currents passing through water cooled copper tubes

Sample is heated by inductive coupling between the field and a conductive sample

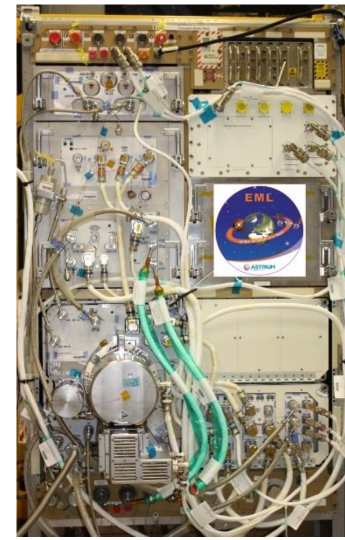
Pulses are excited by applying a sudden change to the heating field which compresses and then releases the sample causing deformation response

Temperature profile from pyrometer





Deformation from video



Slide: courtesy Prof. Doug Matson, Tufts University, USA

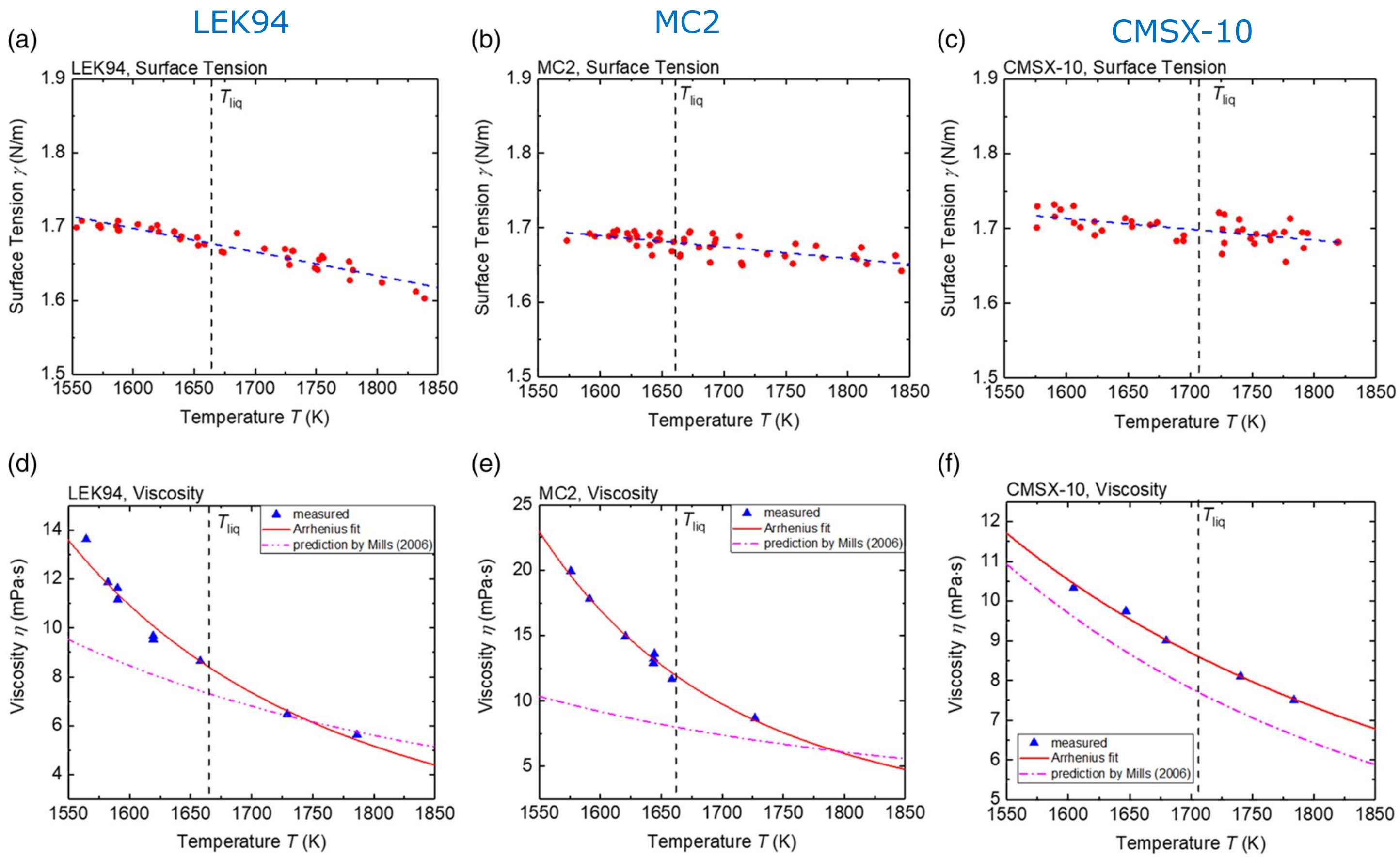
MATERIALS PROPERTIES MEAURED ON ISS

Slide courtesy: Dr Wim Sillekens, ESA

THERMOPROP project: Ni-based superalloy properties as a function of T [1)]

surface tension

viscosity



1) Mohr M., Wunderlich R., Dong Y., Furrer D., Fecht H.-J.; "Thermophysical properties of advanced Ni-based superalloys in the liquid state measured on board the International Space Station"; *Advanced Engineering Materials* (2019): 1901228 (DOI 10.1002/adem.201901228)

Institute of Materials
Physics in Space

Research in Space Conditions:

Diffusion in Liquid Metals

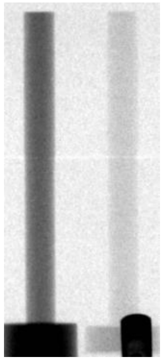
Equiaxed Solidification

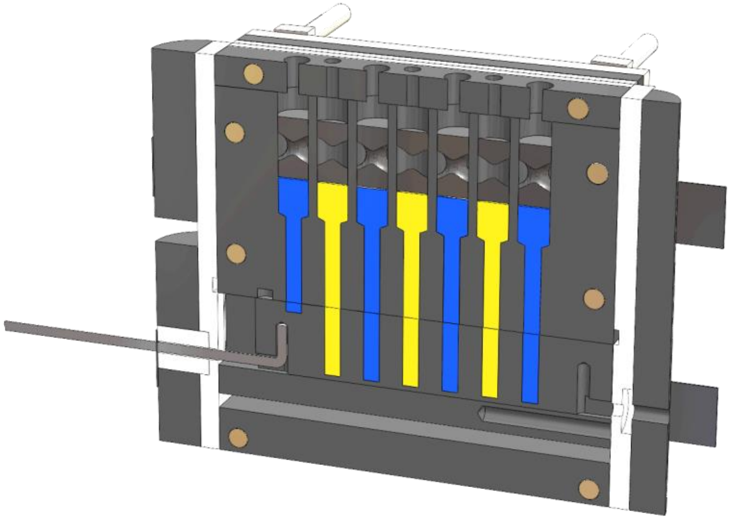
Florian Kargl

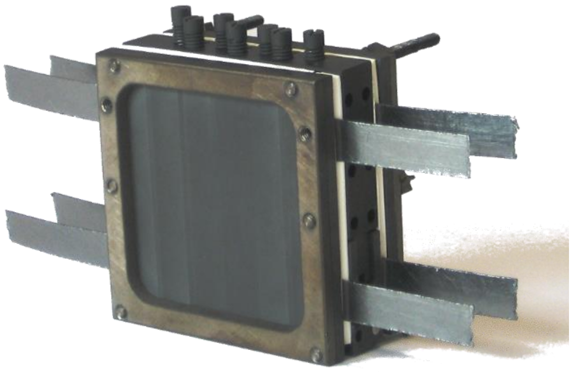
DLR

HELMHOLTZ
RESEARCH FOR GRAND CHALLENGES

Shear Cell for Diffusion Measurement Experiment



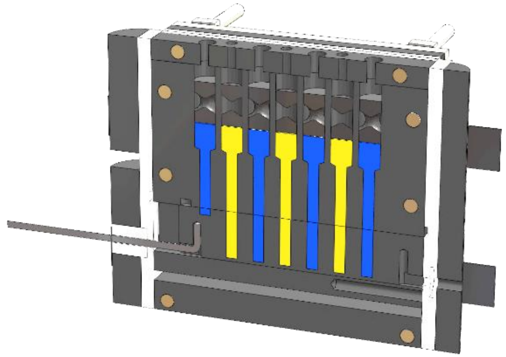




Slide: courtesy Prof. Florian Kargl, DLR

Diffusion in Liquid metal alloys

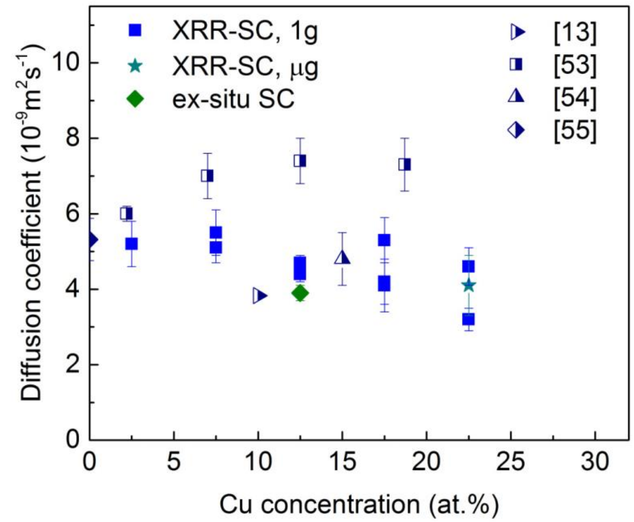
Chemical diffusion in binary in Al-Cu



Institute of Materials
Physics in Space

- In-situ observation of concentration evolution in capillary samples
- Determination of chemical diffusion coefficient in binary metal alloys

- Chemcial diffusion coefficient is required for modelling of alloy solidification.



- Long-time µg-platforms:
  - Combined self- and chemical diffusion experiments
  - Experiments on ternary alloys
- X-ray radiography for process control

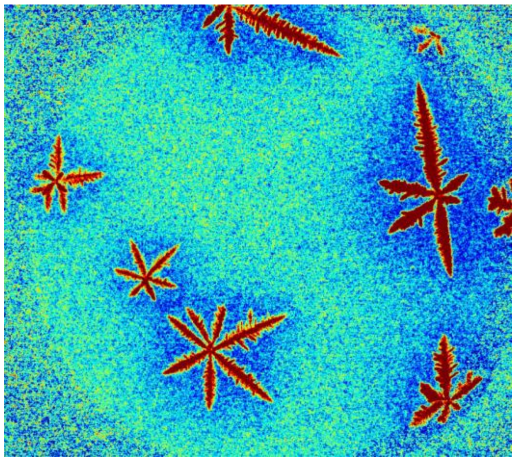
Slide: courtesy Prof. Florian Kargl, DLR

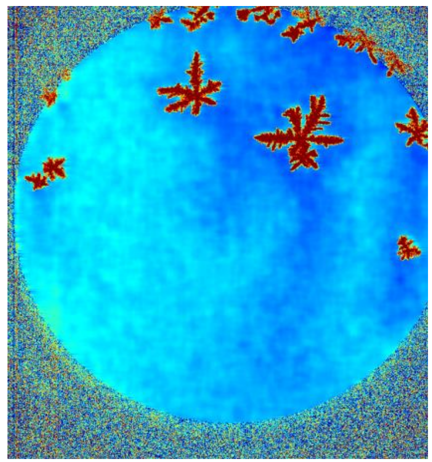
Sondermann et al. Phys. Rev. B 93, (2016) 184201; Belova et al. Scripta Mater. 143, (2018) 40; Sondermann et al. Phys. Rev. B 99, (2019) 024204

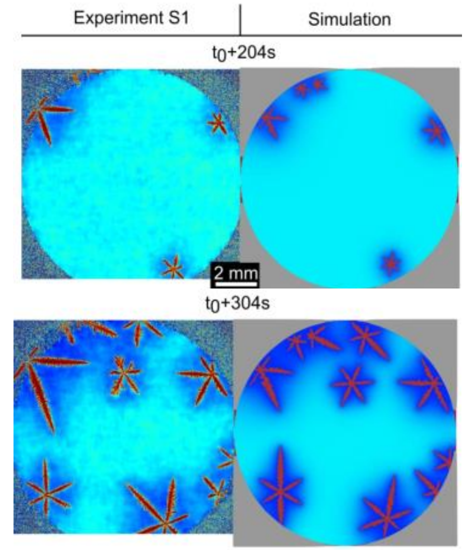
Equiaxed Alloy Solidification

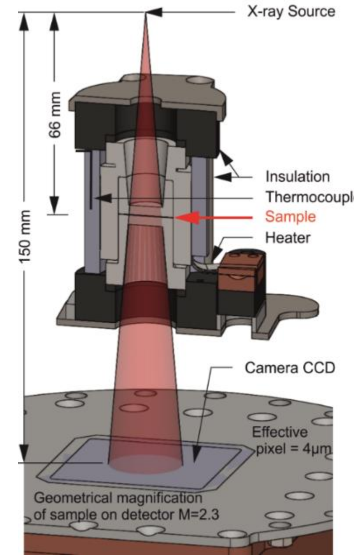
Al-Ge alloys

Institute of Materials
Physics in Space









- Free dendritic and interacted-dendrite growth
- Dendrite orientation selection and transition

→ benchmark experiments without buoyancy convection

→ statistical evaluation of growth process and for long solidification duration

Slide: courtesy Prof. Florian Kargl, DLR

Becker et al. Acta Mater. 201, (2020) 286; ibid 165, (2019) 666; Phys. Rev. M 2, (2018) 073405; Scripta Mater. 124, (2016) 34
Becker et al. Rev. Sci. Instrum. 86, (2015) 063904; Wegener et al. Rev. Sci. Instrum. 92, (2021) 035114.

What about hypergravity?

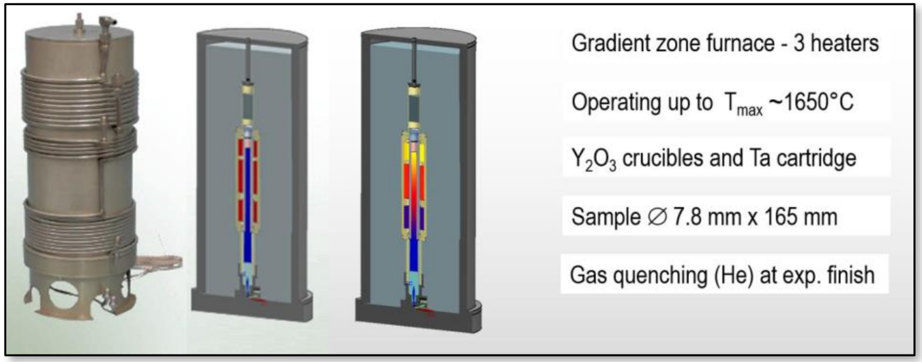
$g_{hyper} >> 1.0\ g$

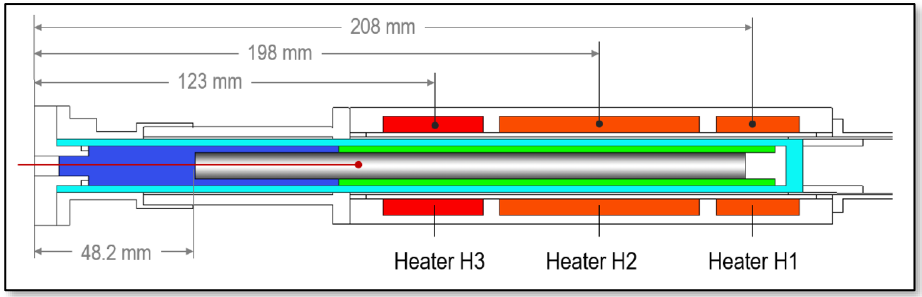
Solidification in Hypergravity

Slide courtesy: Dr Wim Sillekens, ESA

GRADECET: directional solidification of γ-titanium-based alloy GE4822 (TiAl48Cr2Nb2 at.%) – experimental set-up[1)]

- ESA's Large-Diameter Centrifuge (LDC) @ ESTEC for hypergravity
- Sounding rocket MAXUS9 for microgravity





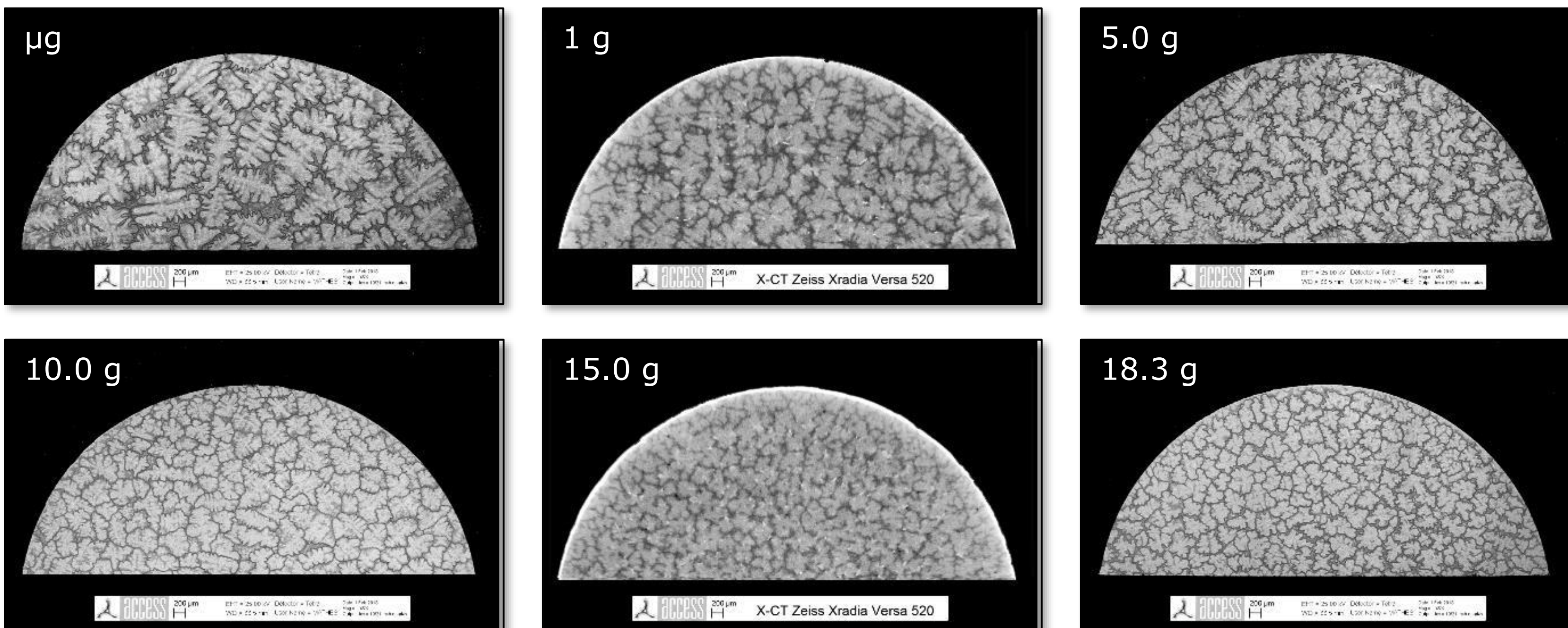
1) Hecht U., Huang C., Zollinger J., Daloz D., Založnik M., Cisternas M., Viardin A., McFadden S., Gránásy L., Lapin J., Leriche N., Kargl F.; "TiAl-based alloys under hypergravity and microgravity conditions"; *Proceedings of the 7th International Conference on Solidification and Gravity*; Miskolc-Lillafüred HU (2018): 27–36

Hypergravity Results - 1

Slide courtesy: Dr Wim Sillekens, ESA

GRADECET: directional solidification of γ-titanium-based alloy GE4822 (TiAl48Cr2Nb2 at.%) – results (1/2)[1]

- Columnar β(Ti) dendrites (transverse sections @ ~10 mm growth length)



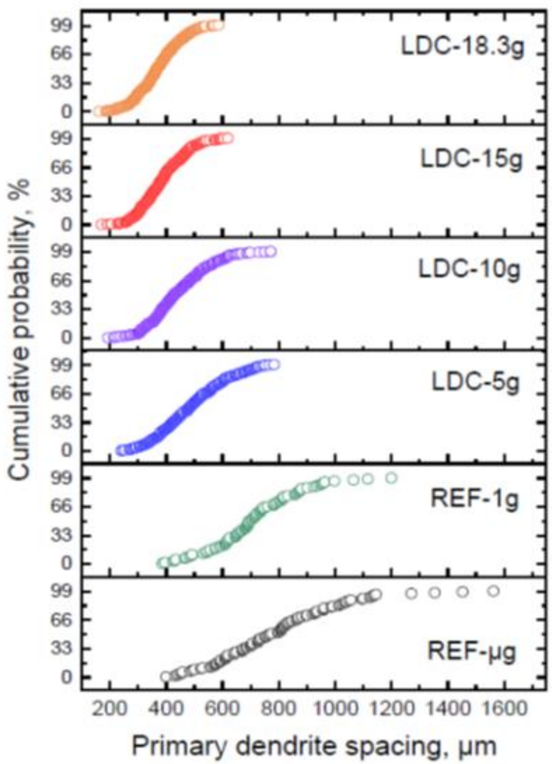
1) Hecht U., Huang C., Zollinger J., Daloz D., Založnik M., Cisternas M., Viardin A., McFadden S., Gránásy L., Lapin J., Leriche N., Kargl F.; "TiAl-based alloys under hypergravity and microgravity conditions"; *Proceedings of the 7th International Conference on Solidification and Gravity*; Miskolc-Lillafüred HU (2018): 27–36

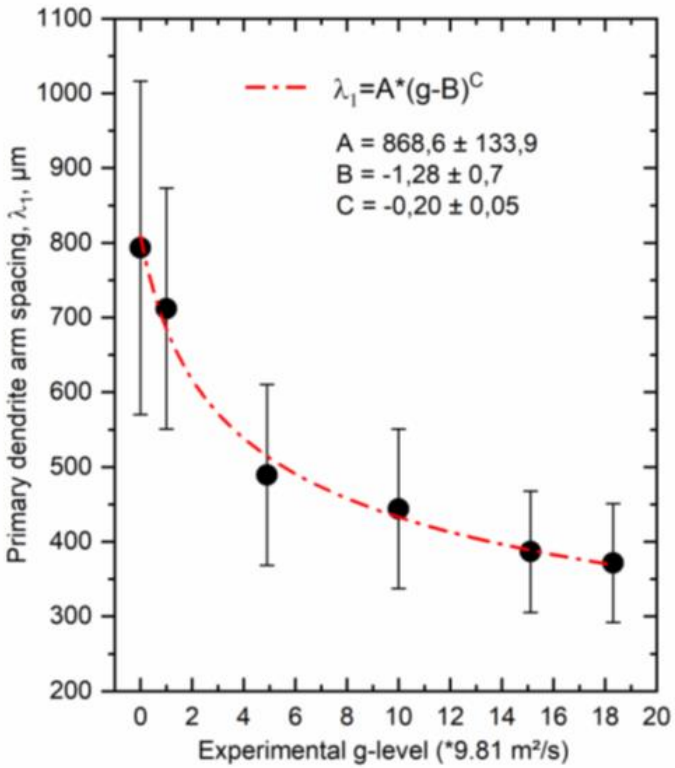
Hypergravity Results - 2

Slide courtesy: Dr Wim Sillekens, ESA

GRADECET: directional solidification of γ-titanium-based alloy GE4822 (TiAl48Cr2Nb2 at.%) – results (2/2)[1)]

- Distribution of and scaling behaviour for the mean spacing (PDAS)





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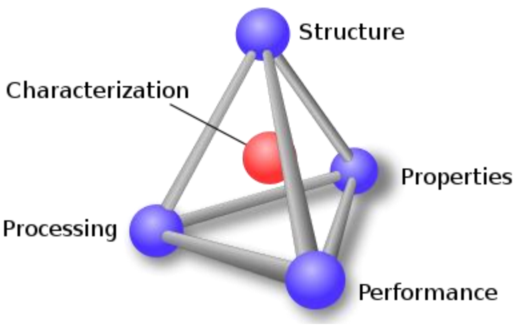
Summary

- **Gravity** affects materials during their processing.
- This in turn affects the microstructure, properties and performance of materials and components.
- The effects of gravity can be assessed if you **turn it down or turn it off**.
- This can be done in a number of ways, but mostly **in space**.



The UN Office of Outer Space Affairs is providing opportunties to all for such Access to Space.

This is in support of the UN's Sustainable Development Goals*

* https://www.unoosa.org/oosa/en/ourwork/space4sdgs/index.html

END

Thank You

UNITED NATIONS
Office for Outer Space Affairs

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Science Foundation Ireland sfi For what's next

All contributors – acknowledged on slides.

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