Rapid Solidification of Al-Cu, Al-Fe and Ni-Al particles under diffusion-limited conditions

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QUAECUMQUE VERA

CSA Workshop on Suborbital Platforms and Nanosatellites
April 14-16, 2010

Outline

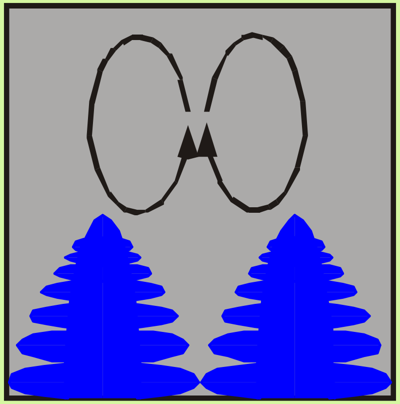
- Apparatus/Infrastruccture/Payloads
- Why research in space?
- Drop tube system
- On-line instrumentation
- Powder characterization
- EML on parabolic flights and sub-orbital rocket.
- Undercooling of primary phase and 2^{nd} phase of eutectic
- The future?

Solidification

Industry
Casting

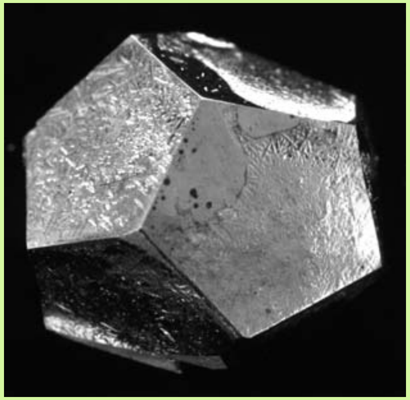
More than 90% of materials are produced from the liquid as their parent phase

Microgravity
Convection control



The conditions of solidification control the quality of the as cast material

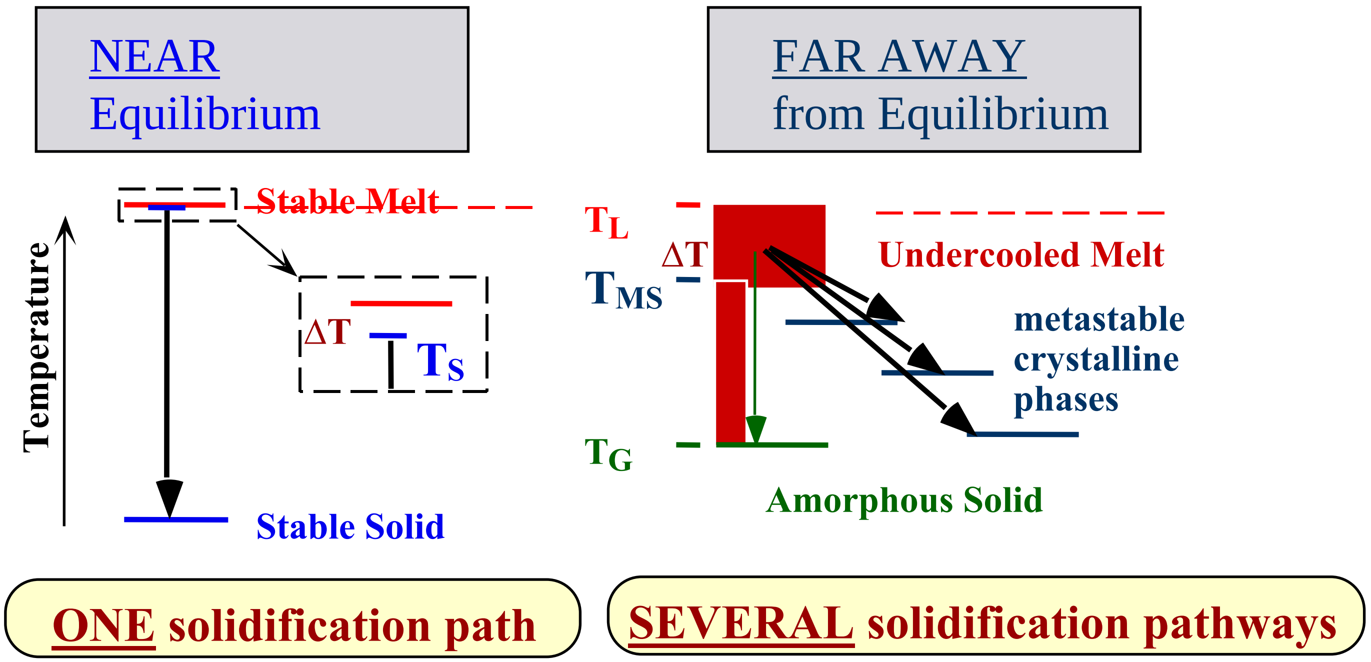
Undercooling
Novel Materials



Large number of degrees of freedom leads to a broad spectrum of materials classes.

D. Herlach, 2007

Why Undercooling?



Great Variety of Metastable Solids from Undercooled Melts

D. Herlach, 2007

Objectives of the Project

To formulate an understanding of the relationship between undercooling and microstructure.

We must be able to vary many parameters, such as:

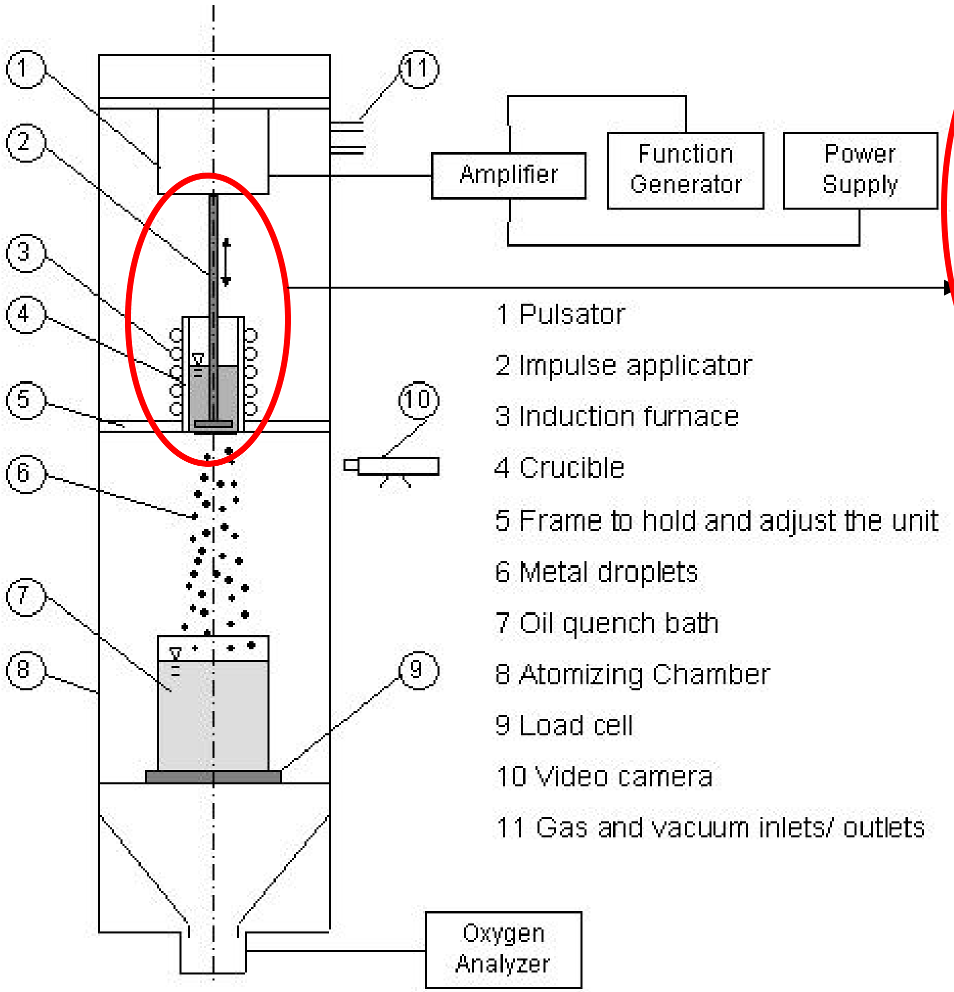
- Container or containerless solidification
- Liquid temperature
- Gas atmosphere
- Alloy system

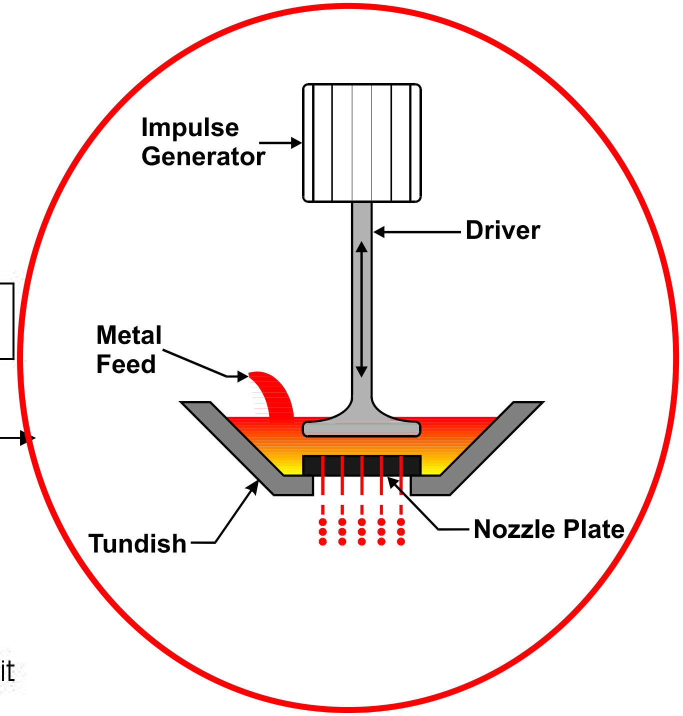
With varying amounts of convection.

Terrestrial
1g

Free Fall, Parabolic flights, …,Space
Low g

Drop tube-Impulse System: description





Two modes of operation:

- Impulse mode
- Impulse-gravity mode

D. Yuan et al, 1997

Three atomization towers

Drop tube-Impulse System: description

<u>Common Utilities and diagnostics</u>

- 20 kW induction furnace
- Oxygen analysis

<u>Tower 1:</u>

- Controlled atmosphere to ~100 ppm.
- Melt capacity of 1 litre
- Atomizing chamber is 0.5m D x 4m H
- Equiped for conducting high pressure gas atomization
- Equiped for carrying out spray forming of ingots

<u>Tower 2</u>:

- Controlled atmosphere to ~2000 ppm.
- Melt capacity of 10 litre
- Atomizing chamber is 1.5m D x 5m H
- Equiped for conducting high pressure gas atomization and low pressure (high flowrate) air atomization
- Equiped for carrying out spray forming of strip 5cm W x 1m L

<u>Tower 3:</u>

- Controlled atmosphere to ~100 ppm and vacuum operation.
- Melt capacity of 1 litre
- Atomizing chanber is 0.5m W x 2m T x 2m H

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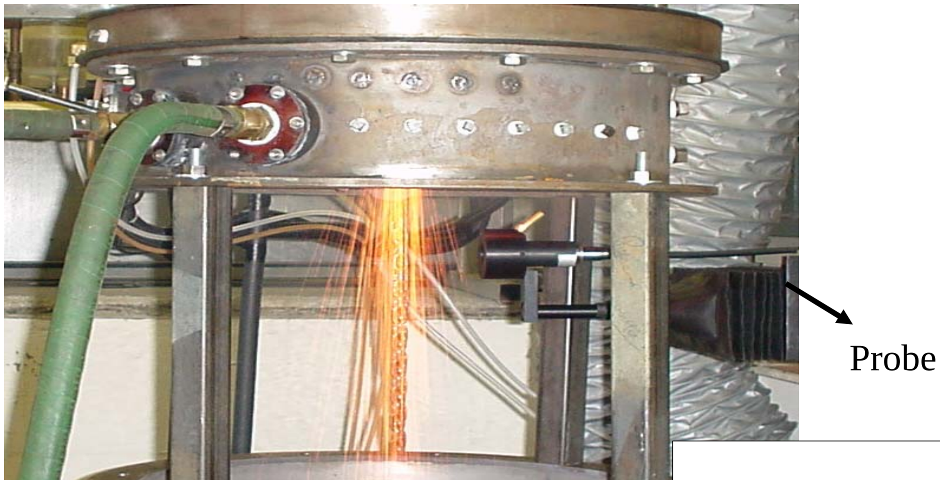
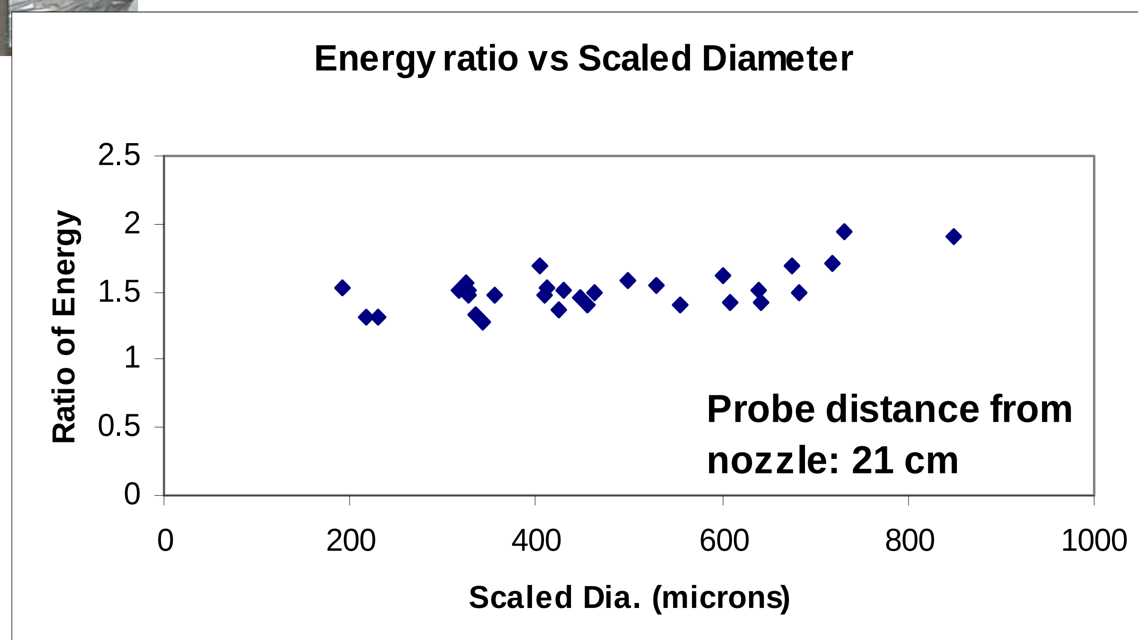
on-line Instrumentation

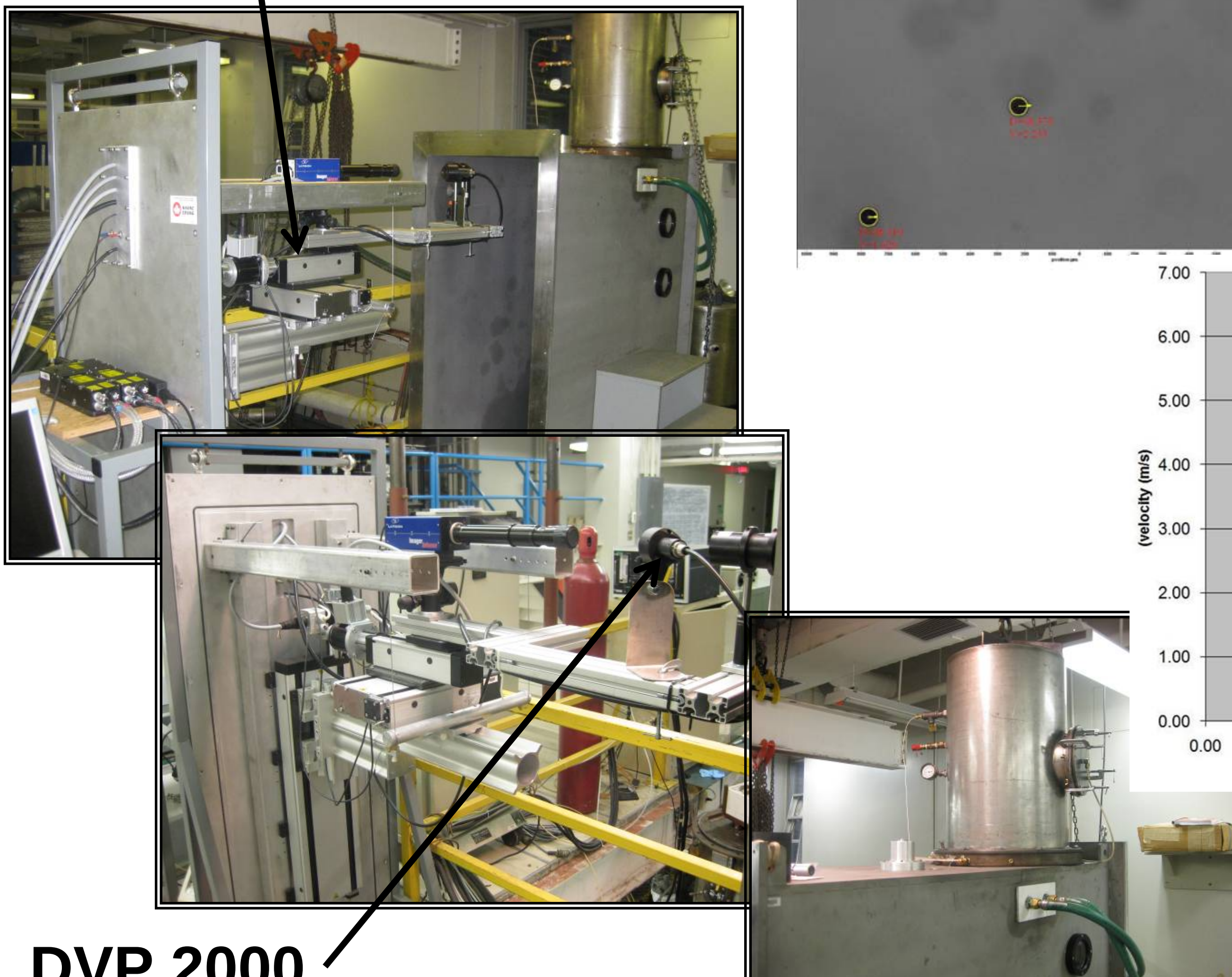
DVP 2000: for in-situ measurement of droplet velocity and temperature

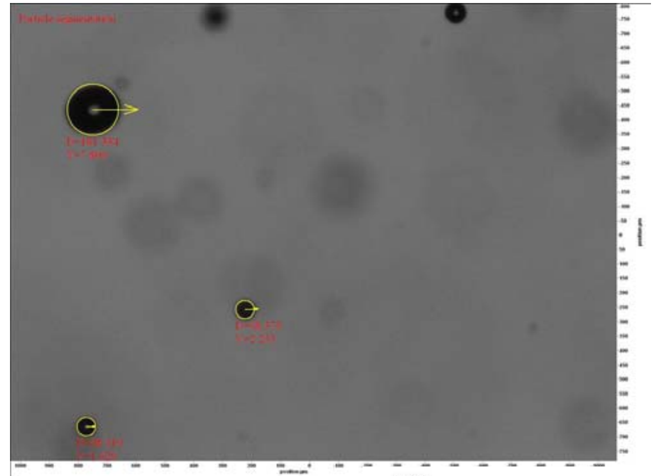


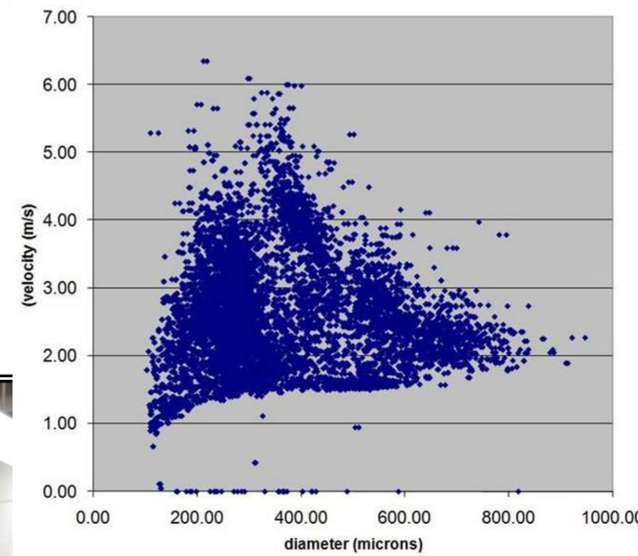


IA Instrumentation

Shadowgraph





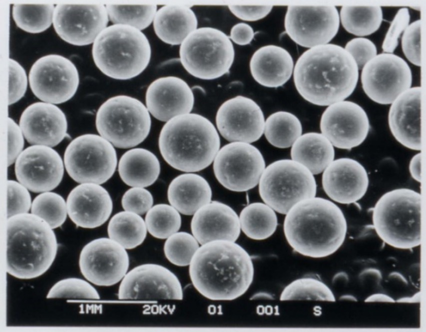


DVP 2000

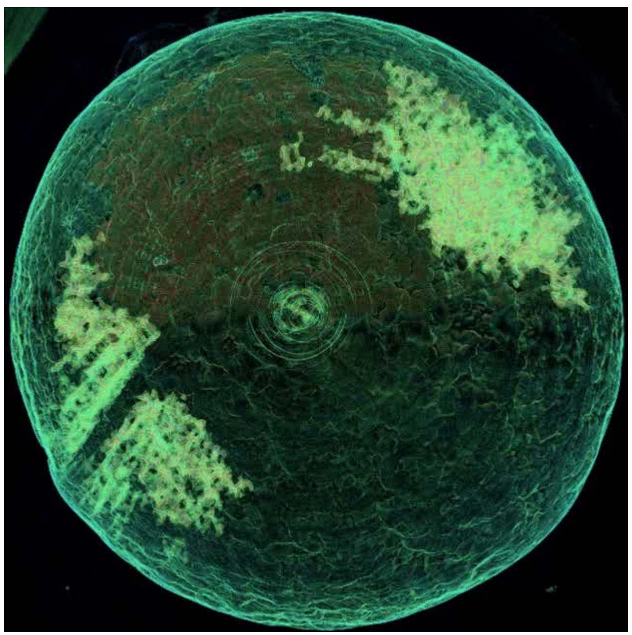
Powder Characterization

<u>powder characterization methods:</u>

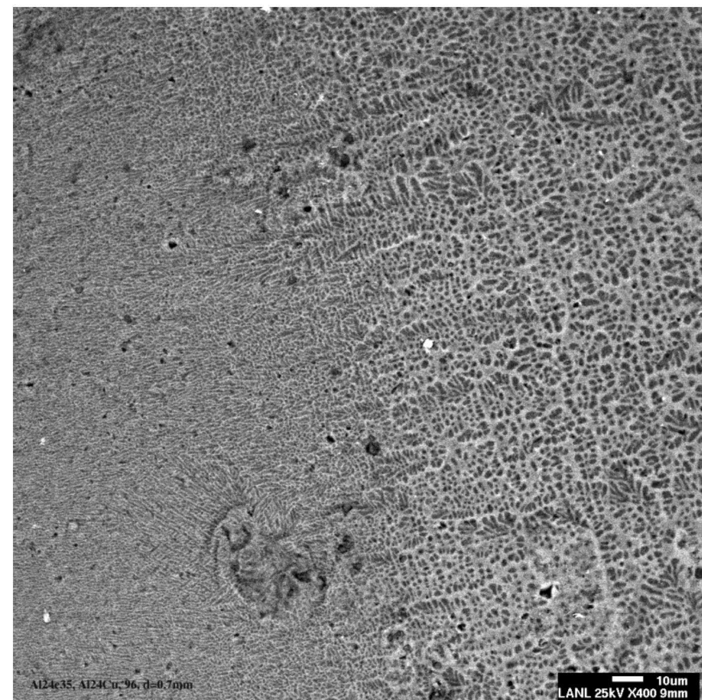
- SEM
- TEM
- Neutron diffraction
- X-Ray micro-tomography



SEM image of unsieved Copper powder produced by IAP at 1200°C using Nozzle B (run # 060793). Atmosphere 10ppm O_2 in N_2.



Micro-tomography of 500 μm Al-0.6 Fe atomized in N_2 showing eutectic and porosity.



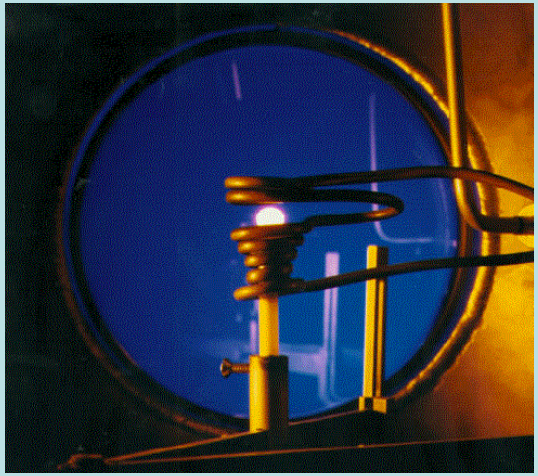
Dr. Ke Han,
Los Alamos National Labs

FESEM Image of 700 μm Al-24Cu Atomized in He

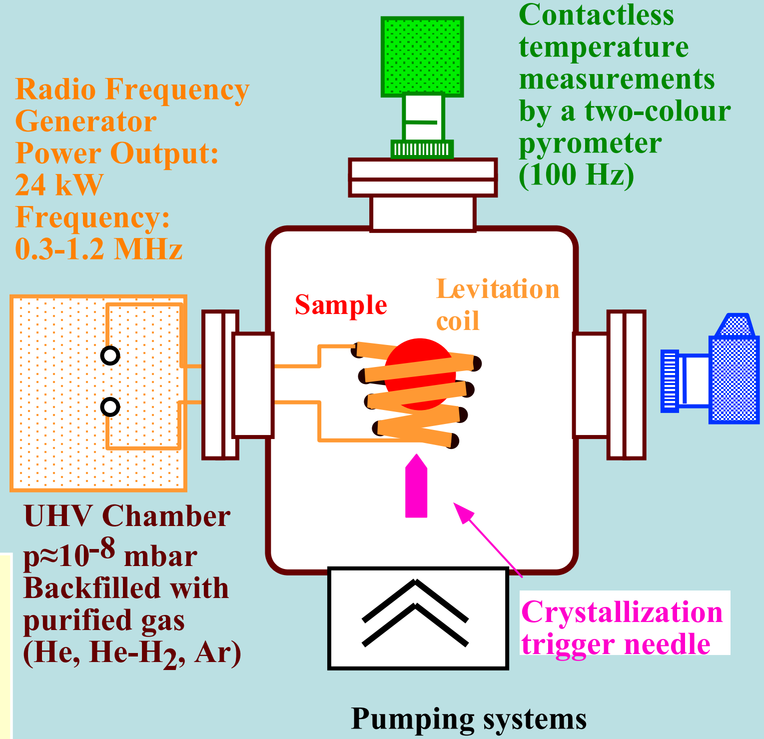
DLR Germany

9 international projects

Each with an international team of 4 to 6 researchers from different countries

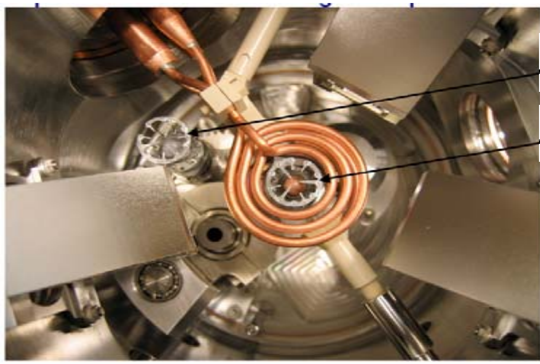


- Bulk samples: 5 mm - 1 cm
- T-range 1000 K < T < 2500 K
- Investigations of:
  - Solidification
  - Thermophysics
- Limitations on Earth:
  - Temperature range
  - Environment
  - El.-magn. stirring
  - Deformation of sample shape

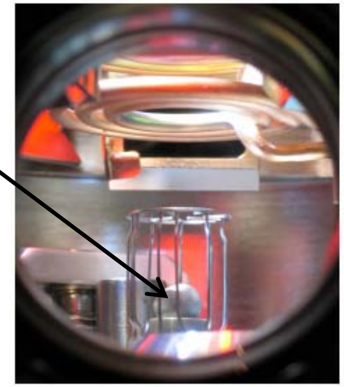


TEMPUS: DARA/DLR
EML for ISS: DLR/ESA

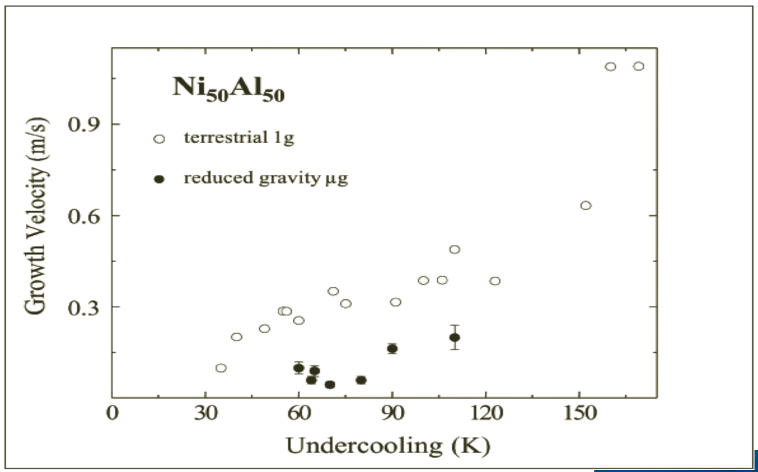
<u>Successfully tested in Space:</u>
NASA Spacelab Missions:
IML-2 (1994)
MSL 1 (1997)
MSL 1R (1997)



Ni-Al sample in its sample cage prior to insertion into the levitation field.



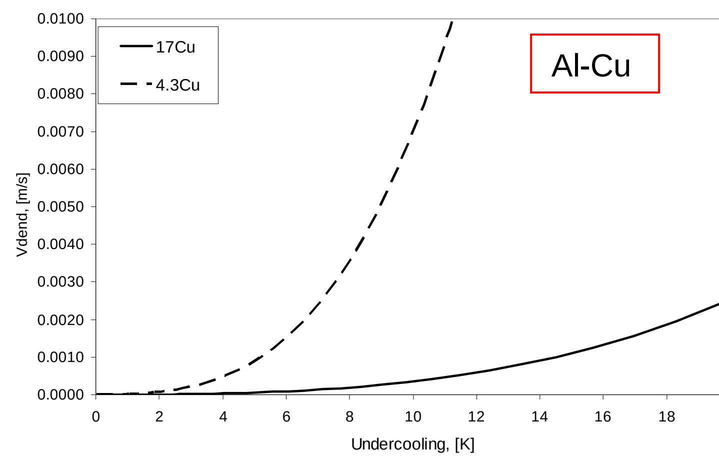
TEXUS 44 carrying EML launched February 2008 Al-Ni alloy



Dendrite growth velocity as a function of undercooling measured on intermetallic $Ni_{50}Al_{50}$ alloy both under terrestrial conditions (open circles) and in reduced gravity (closed circles).

Reutzel, Hartmann, Galenko, Schneider, Herlach, 2007

ESA PFC January 2008 Al-Fe alloy



- Composition dependence of dendrite velocity
- Velocity increases with undercooling

NEQUISOL Participants

D.M. Herlach

Ruhr-University Bochum, Faculty of Physics and Astronomie,
Institute of Materials Physics in Space, DLR,
Germany

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Spain

H. Henein

University of Alberta
Canada

U.Fritshing and V. Uhlenwinkle (NEQUISOL III)

University of Bremen
Germany

Studies using EML

CCEMLCC
COOLCOP
MAGNEPHAS
METCOMP
MULTIPHAS
NEQUISOL
RESISTIVITY
SEMITHERM
THERMOLAB

Areas of research:

- Electrical resistivity measurements of melts,
- Non-equilibrium solidification of melts,
- Thermophysical property measurements of melts.

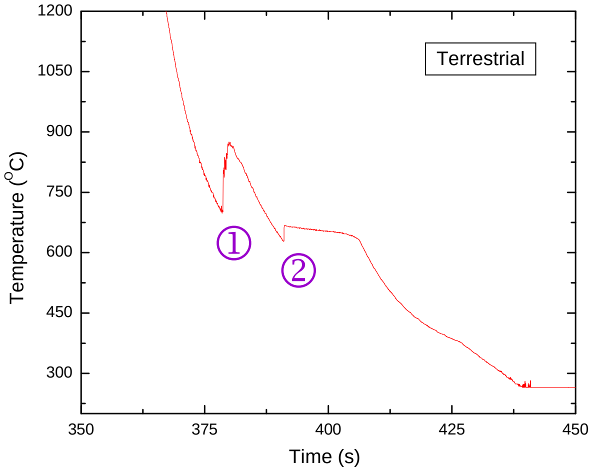
Countries participating:

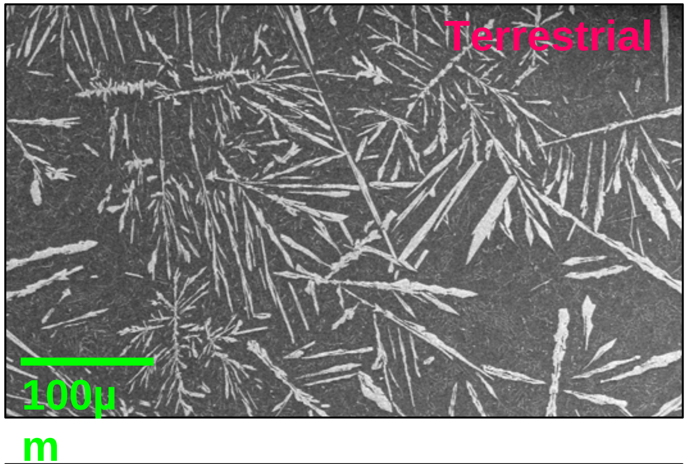
Austria
Canada
Denmark
France
Germany
Japan
Netherland
Russia
Spain

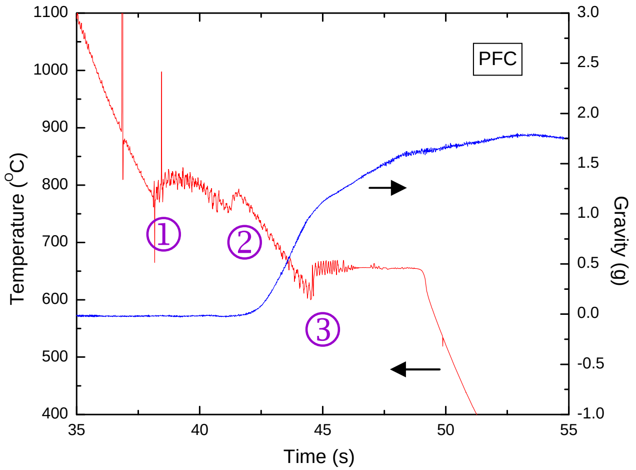
Systems under study:

- AlCu
- AlFe
- CuCo
- MMC in NiTa
- NbNi
- NiAl
- Steel alloys
- ZrNi

Al-8.0Fe ($Al_{96}Fe_4$) Terrestrial and PFC Experiments







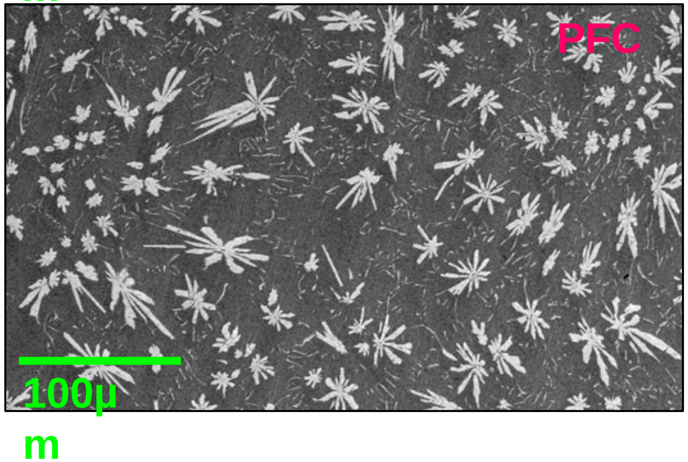


Figure 6 shows the Temperature-Time profiles (cooling part) of Terrestrial, PFC, and the corresponding SEM images.

IA Drop Tube Experiments

Undercooling of PFC, Terrestrial and Impulse Atomized Samples for Al-Fe System

Composition	PFC		Terrestrial		IA	
	Phase	ΔT (C)	Phase	ΔT (C)	Phase	ΔT (C)
Al-8wt%Fe*	$Al_{13}Fe_4$ twinning	75	$Al_{13}Fe_4$ twinning	116	Al_mFe	-
	α-Al	-	α-Al	-	$Al_{13}Fe_4$	-
	Eutectic $Al_{13}Fe_4$/α-Al	21	Eutectic $Al_{13}Fe_4$/α-Al	23	α-Al	-
					Eutectic $Al_{13}Fe_4$/α-Al	-
Al-1.90wt%Fe**	-		TBD	0	α-Al	-
			Eutectic TBD	27	Eutectic Al_mFe/α-Al	17
Al-0.61wt%Fe***	-		TBD	40	α-Al	-
			Eutectic TBD	21	Eutectic Al_mFe/α-Al	10

The future?

EML-ISS planned for 2011 or 2012

AO-99-023

- Batches 1 and 2

  NiAl and AlCu samples.

- Batches 3 and 4

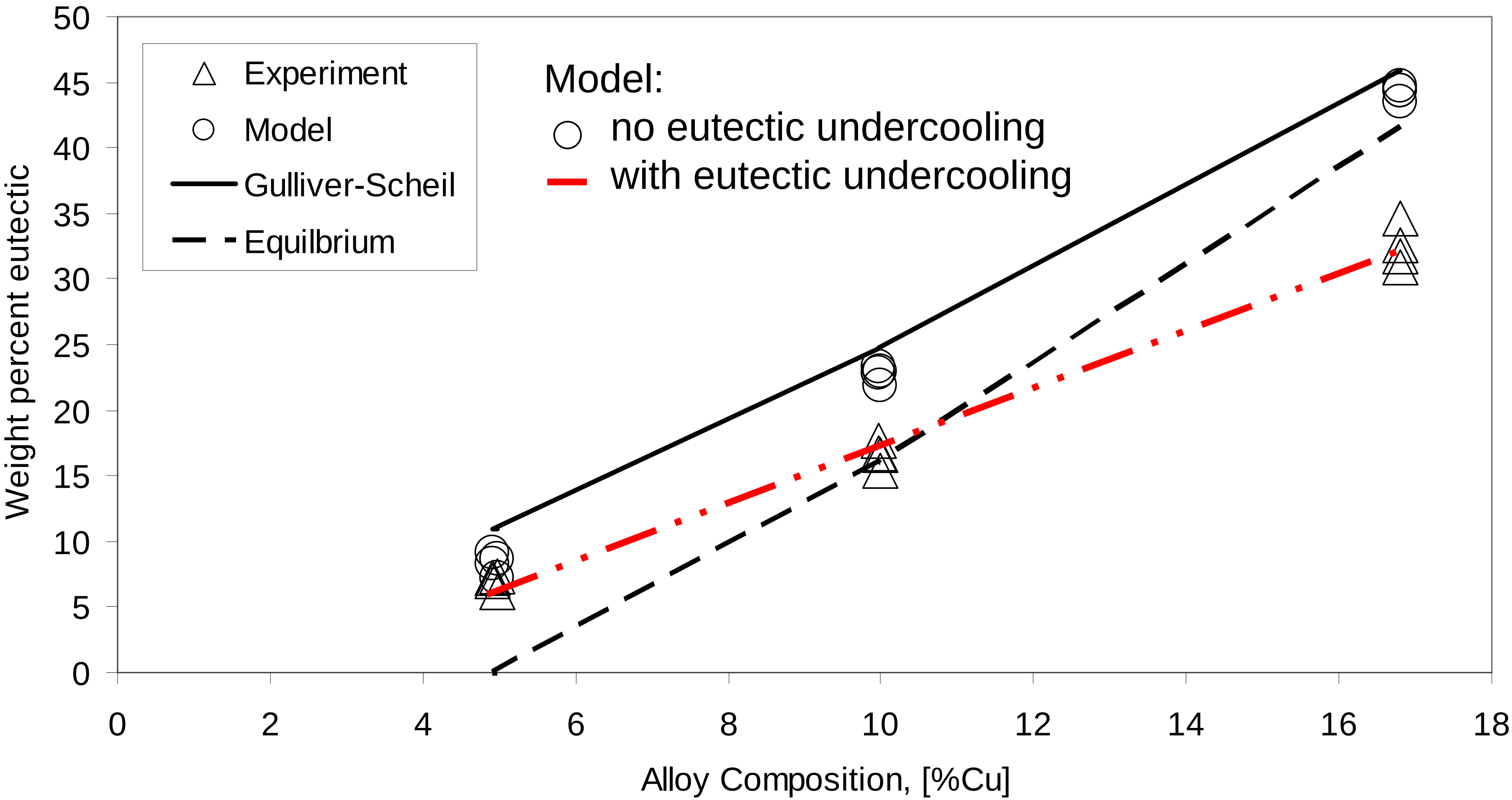
  NiAl, Tool Steel (Fe-Cr-C) and Al-Si samples.

Preparation for Batches 3 and 4 is needed using PFC's and Sub-orbital rocket.

Strategic Project under the Canada-France: Al-Cu, Al-Mg

Ecole des Mines de Paris
Universite d'Aix de Marseille
University of Alberta
Novelis Global Technologies Inc.

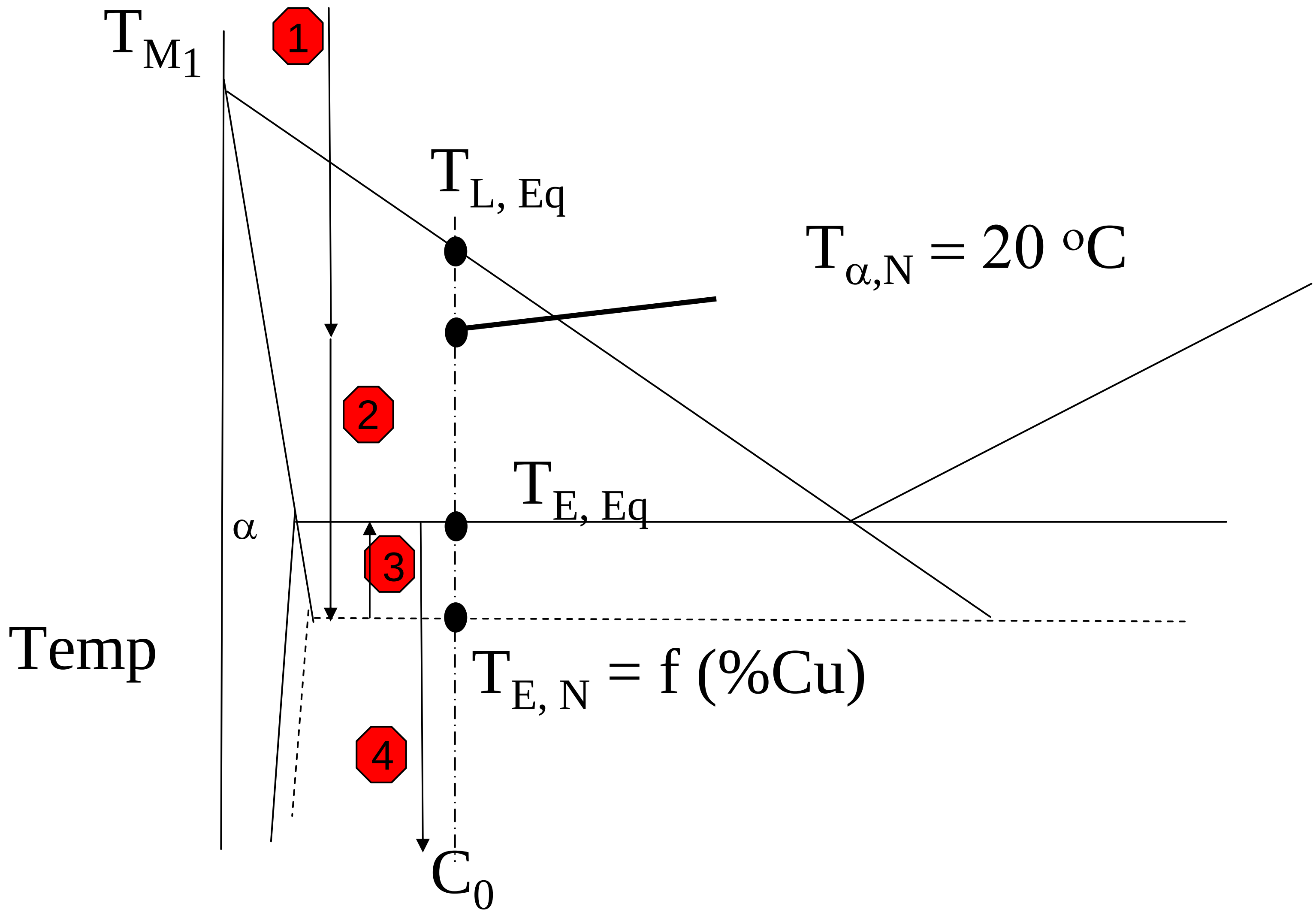
Questions?



Weight percent eutectic as a function of alloy composition. Values calculated from from measurements of weight percent $CuAl_2$ using Neutron Diffraction and volume percent eutectic using Stereology from SEM images.

Collaboration with C-A Gandin, Ecole des Mines de Paris and D. Herlach, DLR, Cologne.

Solidification path of Al-Cu droplets



General Work Plan for the Project Participants

Germany

France

Theoretical:
- Detailed microsegregation modelling of the solidification of levitated droplets.

Experimental:
- Characterization of solidified levitated droplets and comparison of model results with experiments.

Spain

Experimental:
- High pressure gas atomization of Ni-Al alloys.
- Characterization of atomized powders.

Canada

General Work Plan for the Project Participants

Germany

France

Spain

Canada

Theoretical:

- Microsegregation modelling of the solidification of drop tube generated droplets.
- Phase field modelling of drop tube generated droplets (McMaster).

Experimental:

- Generate droplets using high capacity drop tube facility.
- Make in-situ measurements of droplet temperature, velocity and size in flight.
- Characterization of solidified droplets and comparison of model results with experiments.

Impulse System: Sample spray Cu, 1200ºC, 37 orifices

Impulse System: Materials Atomized

Alloy	d50 Range (μm)
Al Alloys	
Al	310 & 2, 4, 6, 8, 10 mm
Al - 4.5Cu	250 - 550
Al - 10Cu	250 - 550
Al - 17Cu	250 - 550
Al - 22Cu	250 - 550
Al - 42Cu	unsized
AA 6061, 6111	250 - 850
Al 357	560 – 700
Al-10Sr & Al–24Sr	1000
Al-Al_2O_3 (5, 10 and 20 vol% of 35 μm Al_2O_3)	1000
Cu Alloys	
Cu	500 - 1400
Bronze	180 - 720
Cu-5Ag	unsized

Alloy	d50 Range (μm)
Lead	
Pb-10%Sn	200-1000
Pb-12%Sn	130
Mg Alloys	
Mg	2 to 4 mm
Mg - 9Al -1Zn	850 - 1000
Mg - 9Al - 1Zr	850
Nd Alloys	
NdFeB	1000
Ni Alloys	
Ni-10%Al	350

Impulse System

Alloy	d50 Range (μm)
Steels	
1040, H13, 4140, 304SS	350 - 1500
Titanium	
Ti34Cu45Zr11Ni8	350
Zinc	
Zn – 500 ppm Pb	250
Zn	150
Zn alkaline battery powder	100 and 150

1.3 ≤ d84/d50 ≤ 1.5

0.063 kg/s pilot scale unit operated for 2.5 hrs.

Mass flux is ~7300 kg/s m^2

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Impulse System: description

<u>Instrumentation</u>:

- Oxygen analysis
- Melt temperature
- Load cell for powder production rate measurement
- Applied impulse frequency and amplitude
- Applied force to the melt
- High speed single frame imaging
- Shadowgraph system for in-flight measurement of droplet size and velocity
- Two colour pyrometer for in-flight measurement of droplet temperature
- Video imaging of the spray