Action Nationale de Formation
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Semi-solid Alloys

Michel SUERY

Grenoble INP, SIMaP, Saint-Martin d'Hères, France

Michel.Suery@simap.grenoble-inp.fr

Grenoble INP

SIMaP

CNRS CENTRE NATIONAL DE LA RECHERCHE SCIENTIFIQUE

OUTLINE

- Basic considerations
- Two important aspects

  Microstructural characterisation

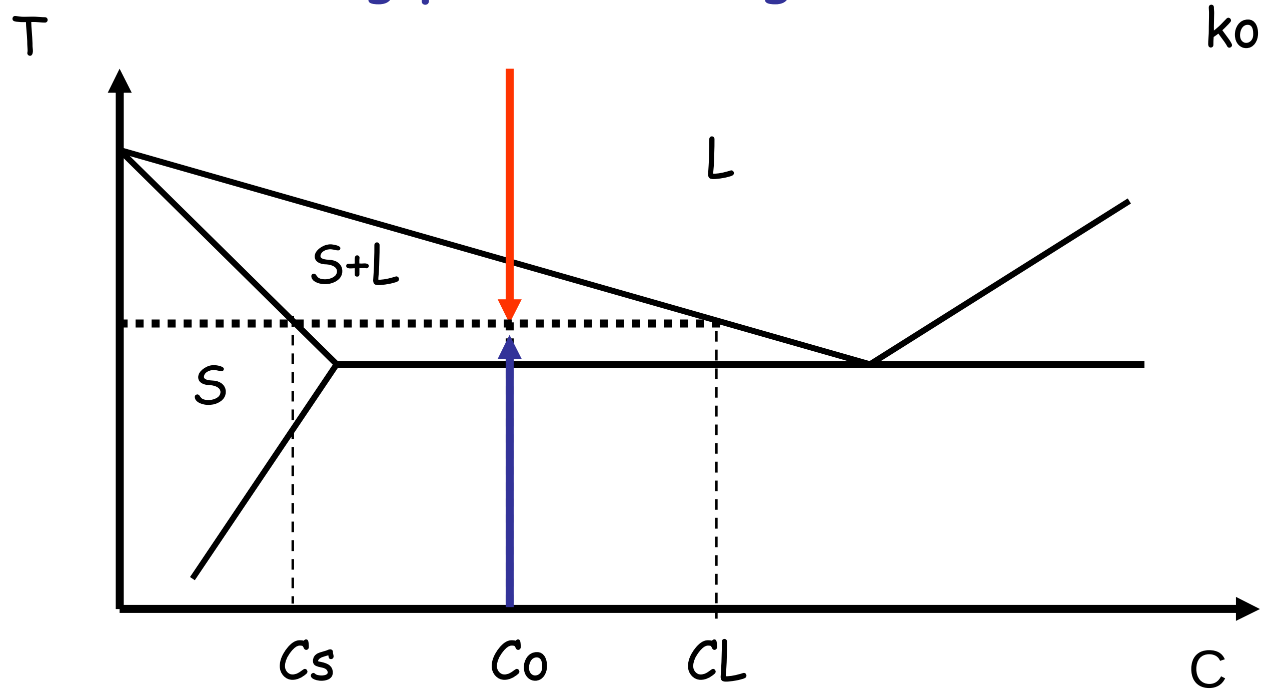
  Mechanical characterisation
- Key problems

Basic considerations

Partial melts in alloys

- ☞ During solidification
- ☞ During partial melting

Partition ratio
$k_o = C_s/C_L$



Main parameter = solid fraction fs

☞ Equilibrium: lever rule $$fs = (C_L - Co)/(C_L - Cs)$$

☞ No solid diffusion: Scheil equation $$Cs = ko.Co\ (1-fs)^{(ko-1)}$$

Other parameters for the solid

- Size
- Morphology
- Solid-liquid interface area
- Connectivity of the solid particles
- …

Characterisation of the semi-solid state important:

⇨ For a better understanding of solidification mechanisms

⇨ For a better understanding of liquid phase sintering

⇨ For a better understanding of the hot tearing phenomenon that occurs in various processes (casting, welding, …)

⇨ For the development of forming processes (intermediate between solid forming processes (forging,…) and liquid forming processes (casting, …)

Two important aspects

Microstructural characterisation

Usually carried out

- after quenching of the specimens to room temperature
- on polished sections (in 2D)

Questions :

1. Is the solidified microstructure representative of the microstructure in the S/L region?

2. Are observations on 2D sections representative of the 3D microstructure?

Question 1 : Is the solidified microstructure representative of the microstructure in the S/L region?

- alloy is just at the eutectic temperature

  no problem : (liquid $\longrightarrow$ eutectic mixture)

- alloy above the eutectic temperature

  $\longrightarrow$ problem : deposition of solid on the existing solid and then eutectic transformation

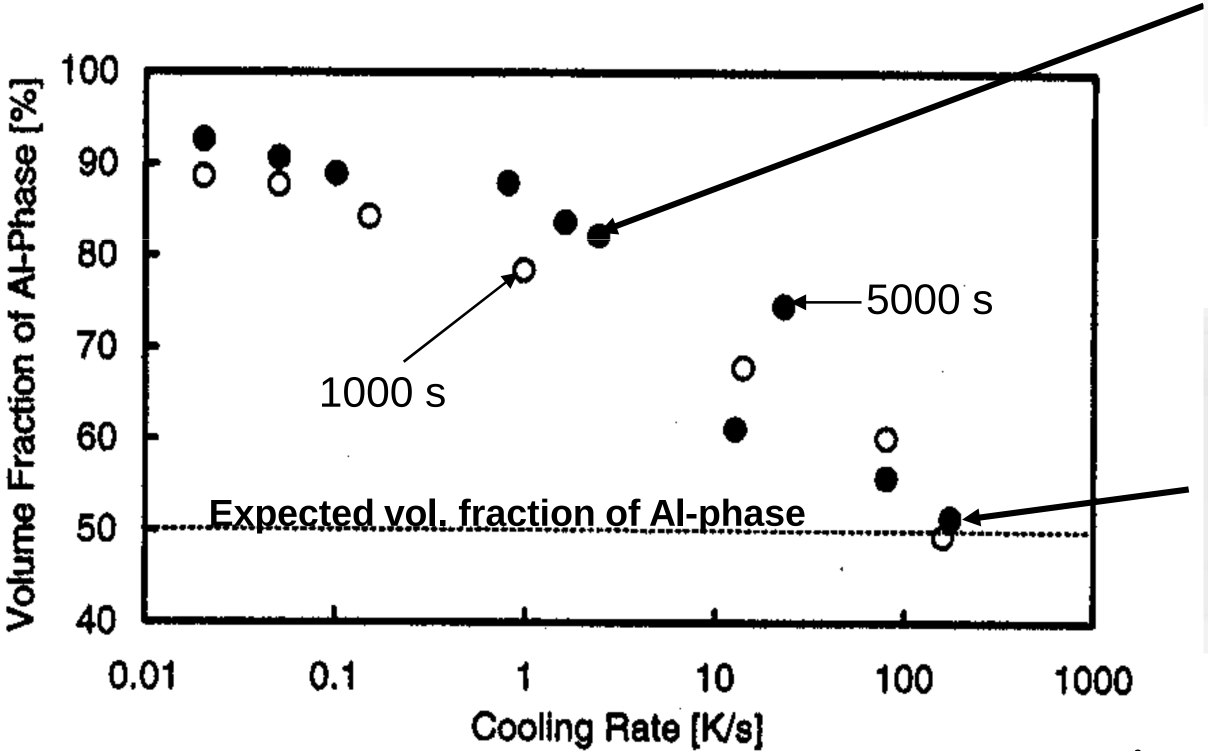
  $\longrightarrow$ solid fraction overestimated

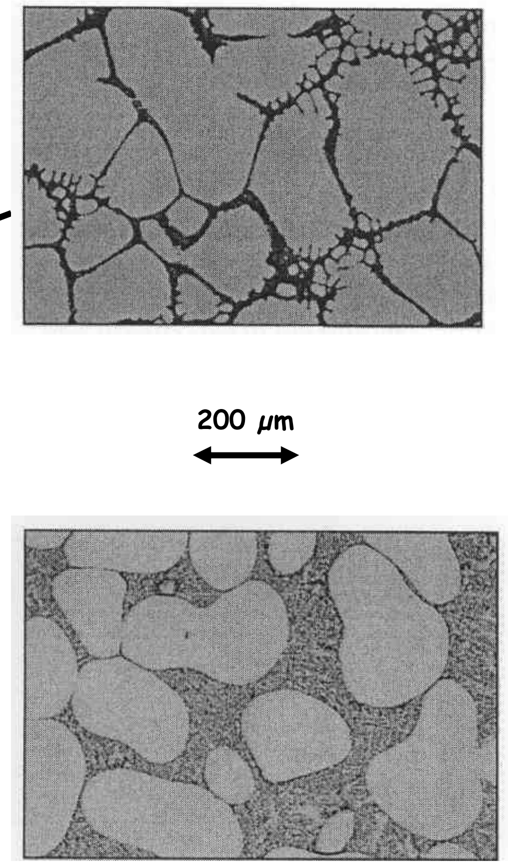
Sometimes etching allows to distinguish the two primary solids (preexisting and that formed during quenching)

O. Pompe & M. Rettenmayr

J. Crystal Growth, 1998, 192, 300-306

Al-6.8wt%Cu held at 628℃
and cooled at various rates





Cooling rate ≥ 200°C/s

Question 2 : Are observations on 2D sections representative of the 3D microstructure?

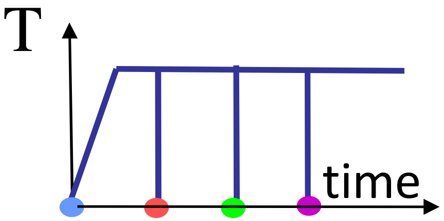
→ Yes for some parameters (solid volume fraction, size)

→ No for other parameters (connectivity)

Solutions :

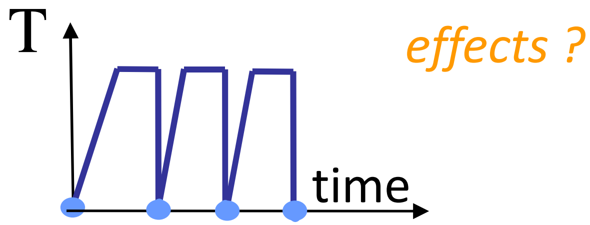
- Serial sectioning (polishing) and 3D reconstruction
    - destructive
    - low resolution usually
    - but no specific equipement
- X-Ray tomography and 3D reconstruction (computed tomography)
    - non destructive
    - high resolution (synchrotron)
    - but specific equipment

Post mortem



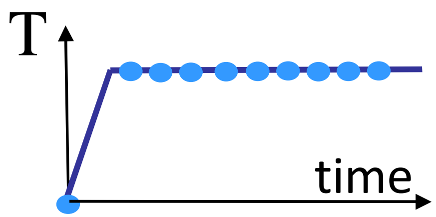
Several samples
Scans at RT

Ex situ



One sample
Furnace out of tomograph

continuous in situ



One sample
Furnace mounted
on the tomograph

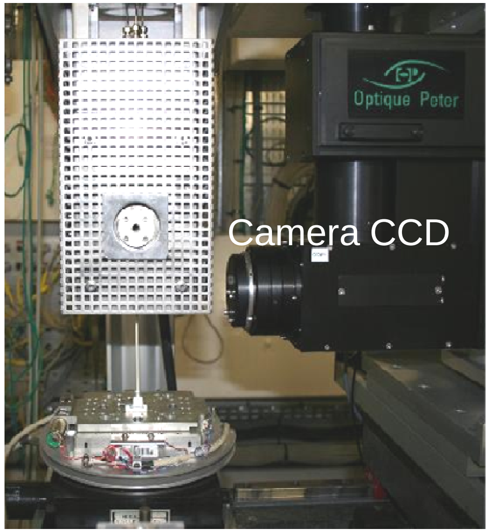
Solution
- to avoid quenching
- for 3D characterisation

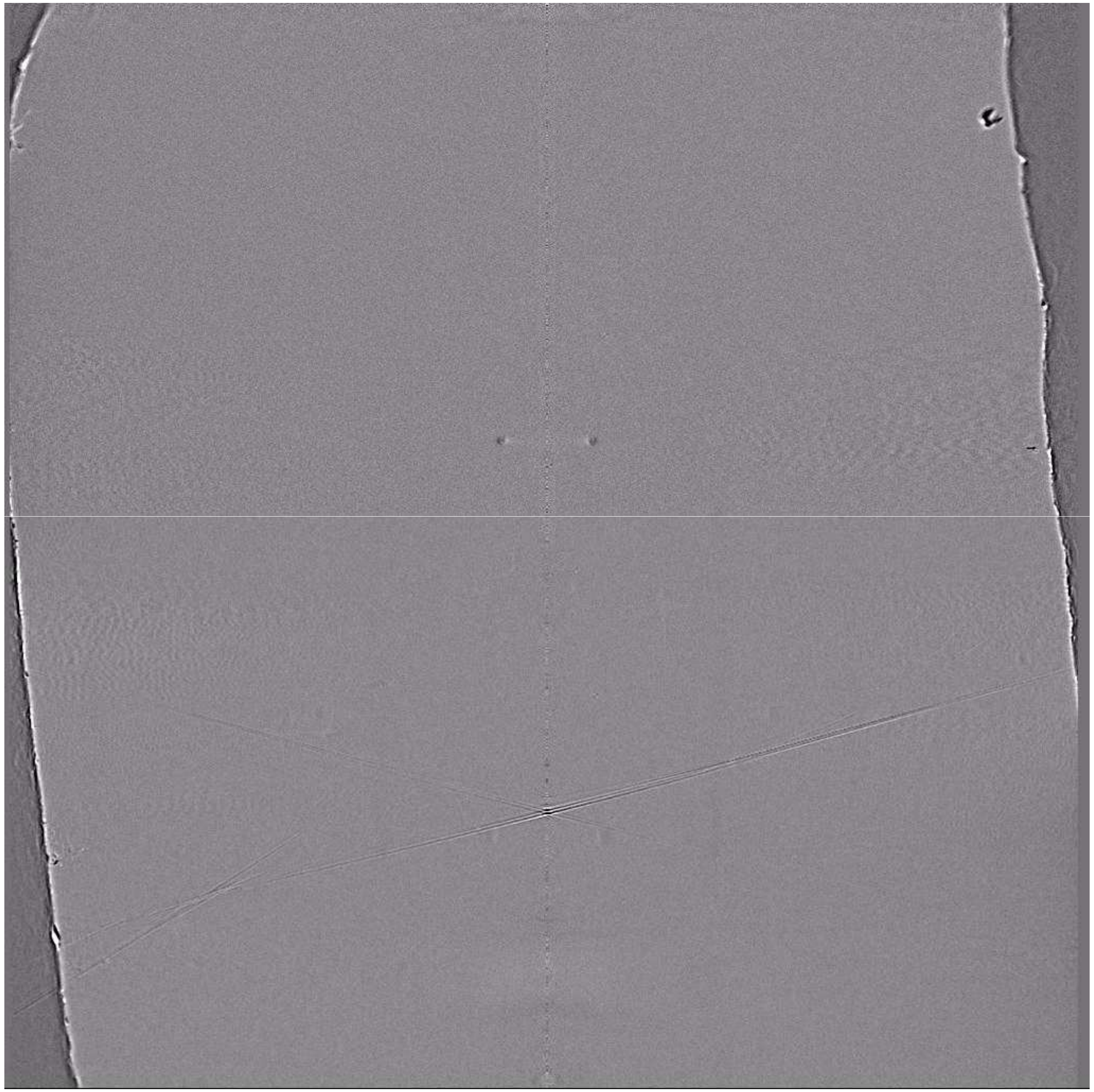
Limitations:
- Good absorption contrast between solid and liquid (Al-Cu)
- Sufficient spatial and temporal resolution

Semi-solid alloys

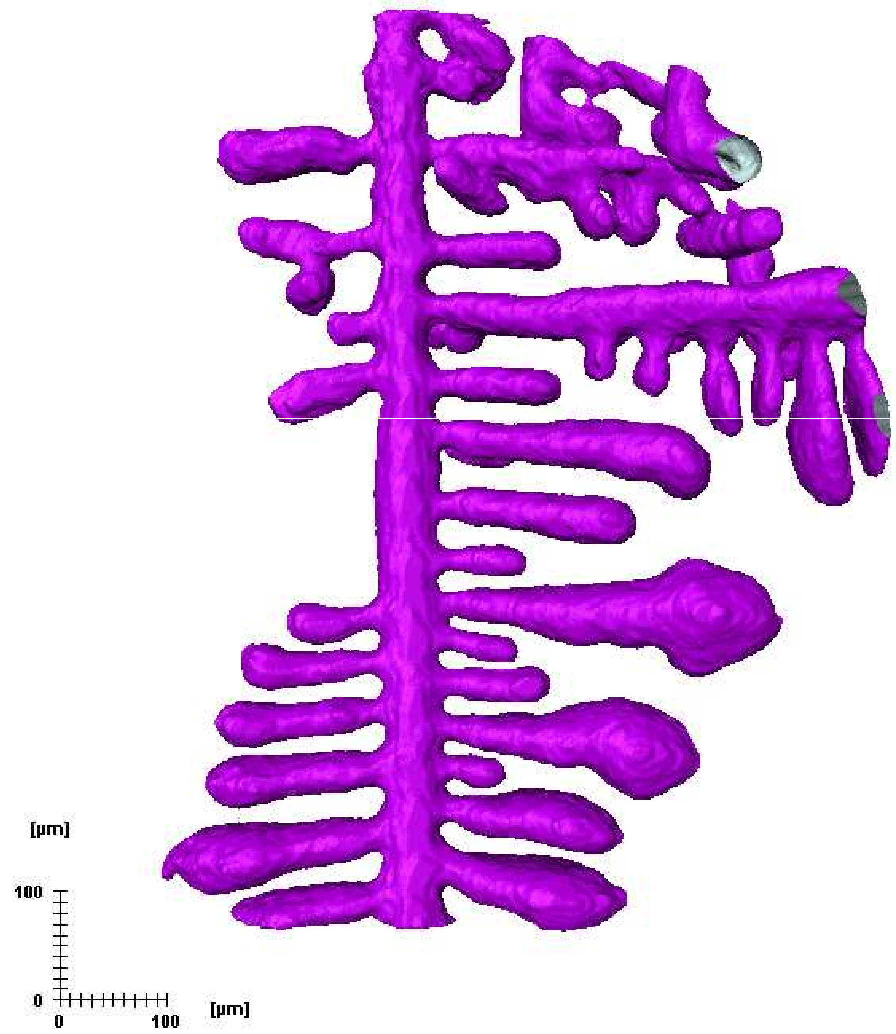
Al-8%Cu
Solidification at 3°C.min^{-1}
1.4 μm, 600 images, ~60 s
ESRF ID19

RX

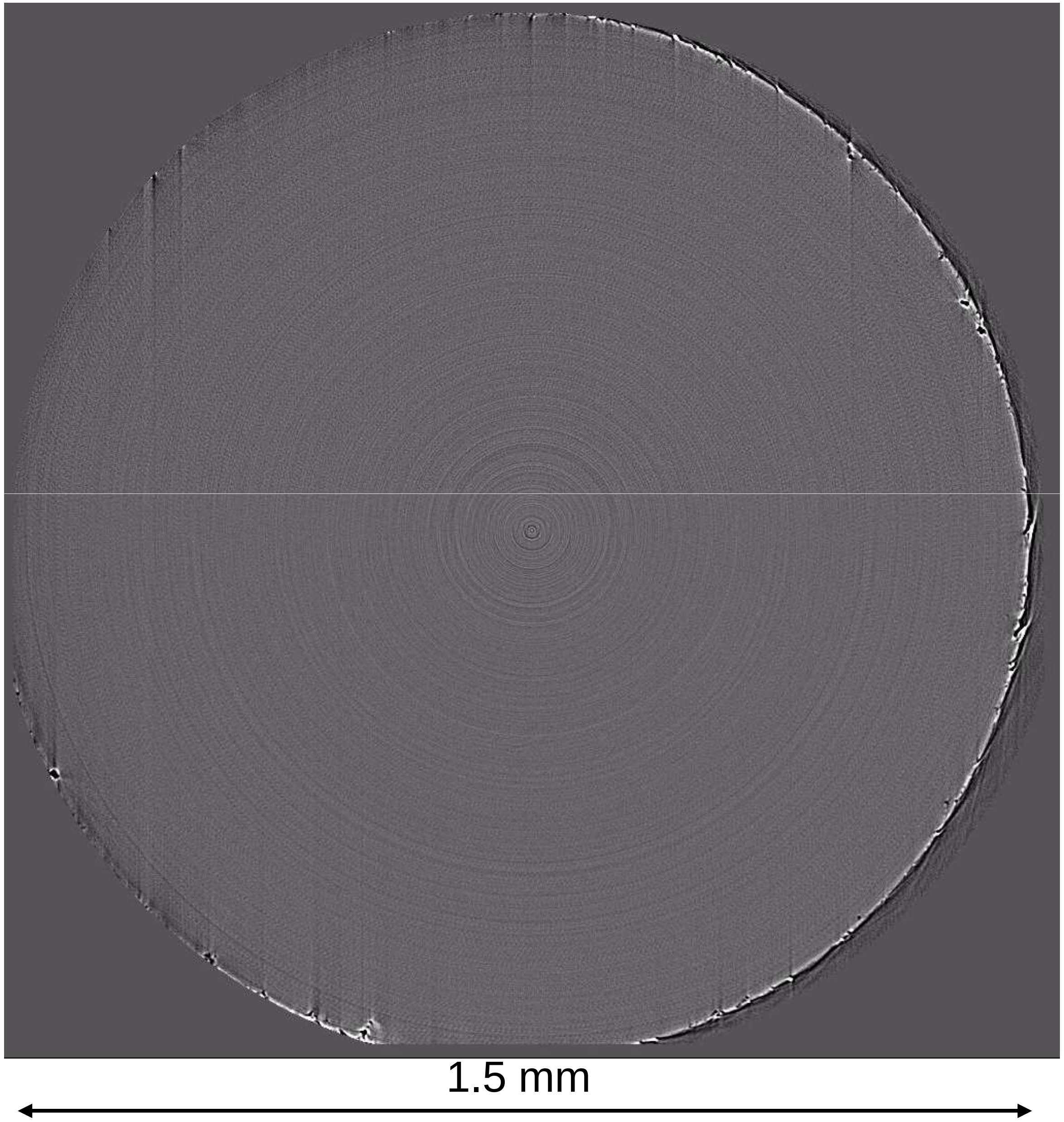




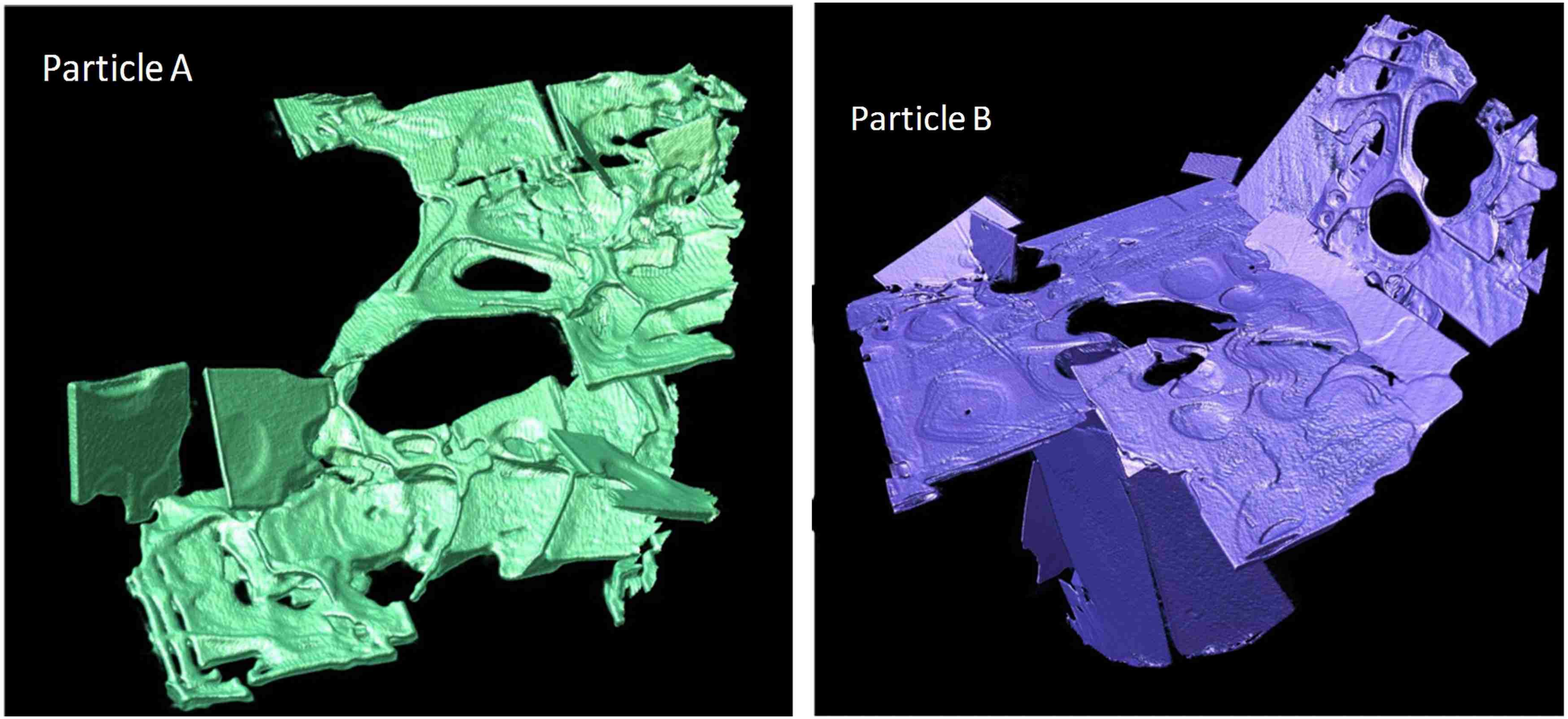
1.5 mm


[µm]
100
0
0
100
[µm]

Al-8%Si-4%Cu-0.8%Fe
Solidification at 1.4°C.min^{-1}
1.4 μm, 600 images, ~36 s
ESRF ID19



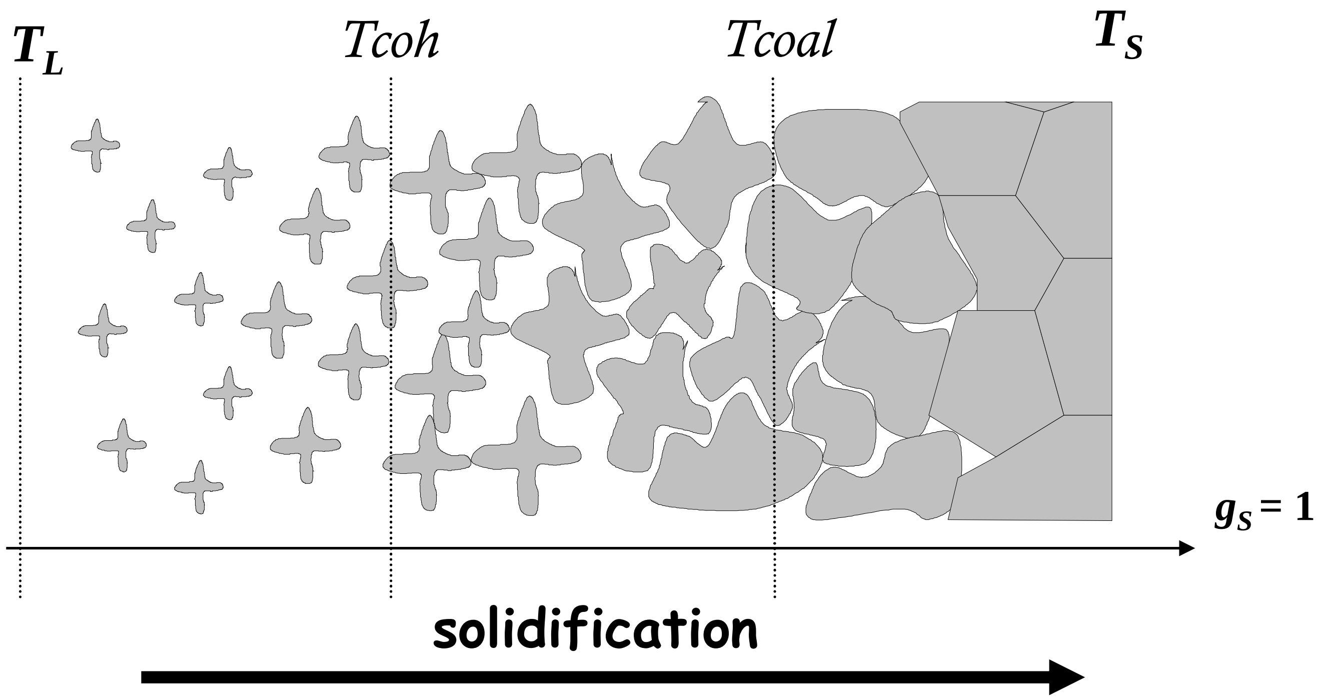
β-Al_5FeSi particles



200µm

Terzi et al. Acta Mat. 58 (2010) 5370

Mechanical characterisation

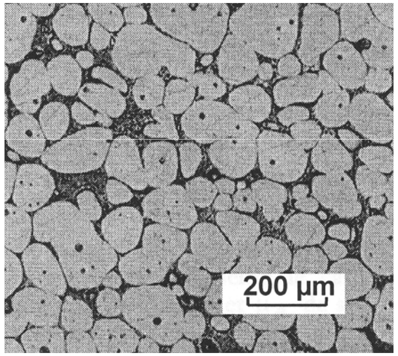


Two ranges of solid fractions are interesting:

☞ intermediate solid fractions (~ 40 à 60 %)

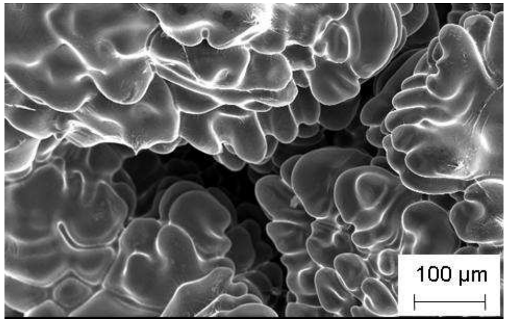
For semi-solid metal forming (rheoforming, thixoforming)

⇨ globular solid morphology



☞ large solid fractions (~ 80 à 95 %) for a better understanding of

hot tearing phenomena



⇨ **For modelling semi-solid metal forming (fs ~ 50%)**

Various viscometers: Couette, Searle, cone and plate...

⟶ $\eta = \eta\ (\eta_L$, Fs, shear rate, ...)

Problem: usually determination carried out at steady state

Reality: injection of semi-solid alloy in a mold takes less than 1 s

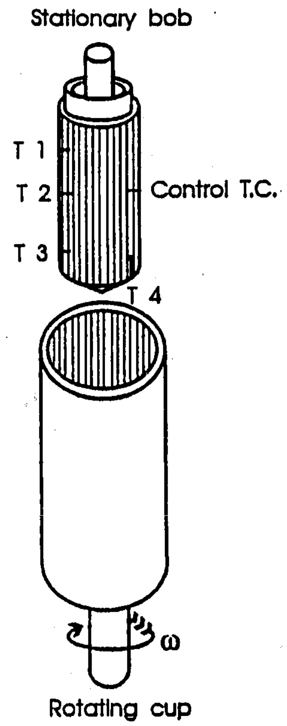


Figure 3: Schematic of graphite rotating cup and stationary bob.

(Couette)

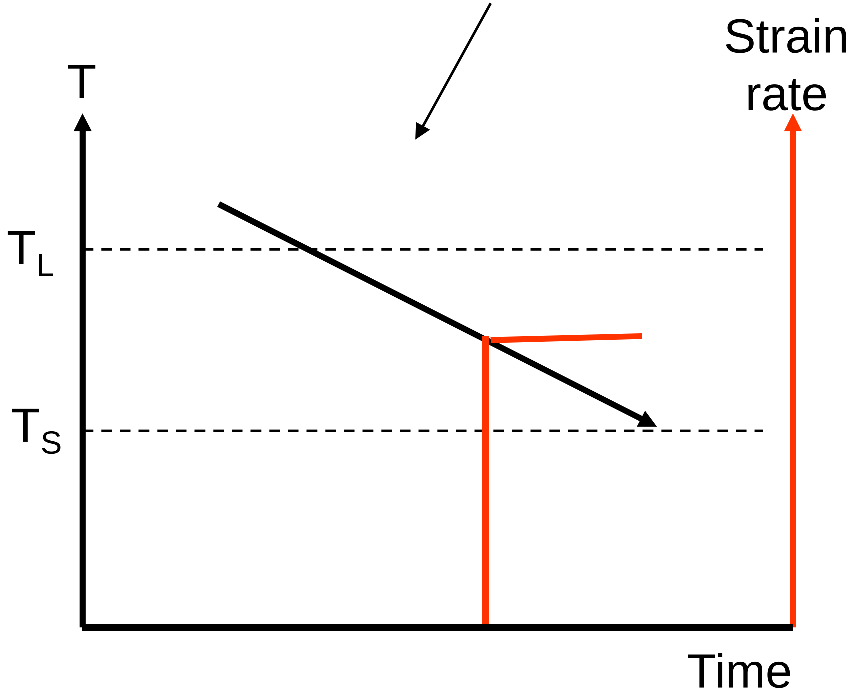
⇨ For understanding hot tearing (Fs ~ 90%)

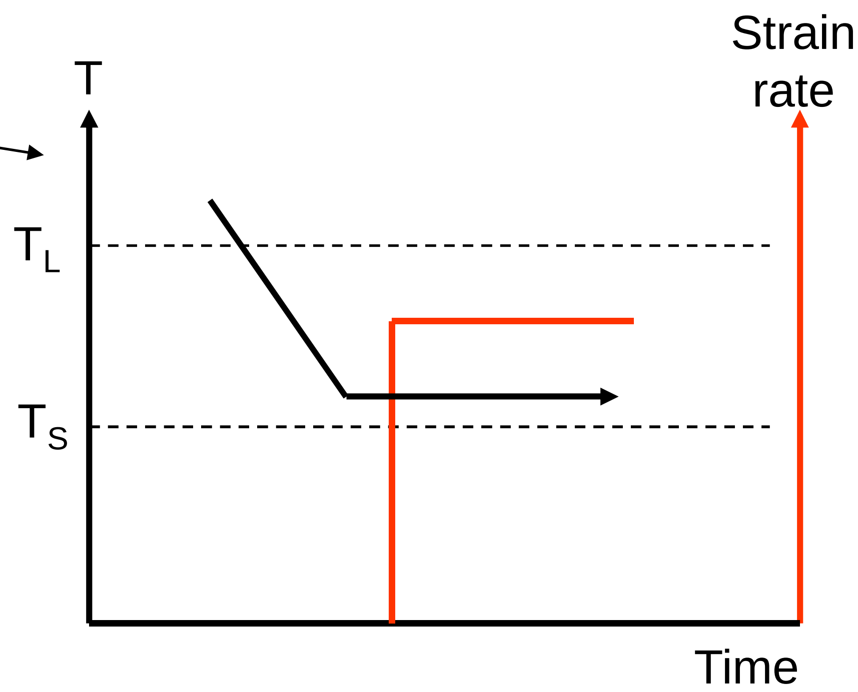
Mechanical tests during solidification with cooling rates similar to those corresponding to processes

- a few °C/s for casting
- several 100°C/s for welding (quite impossible)

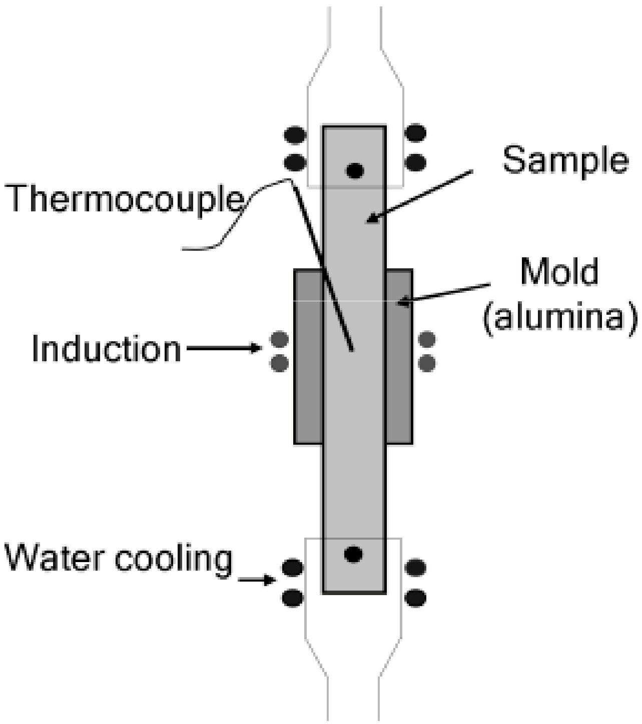
Two types of tests:

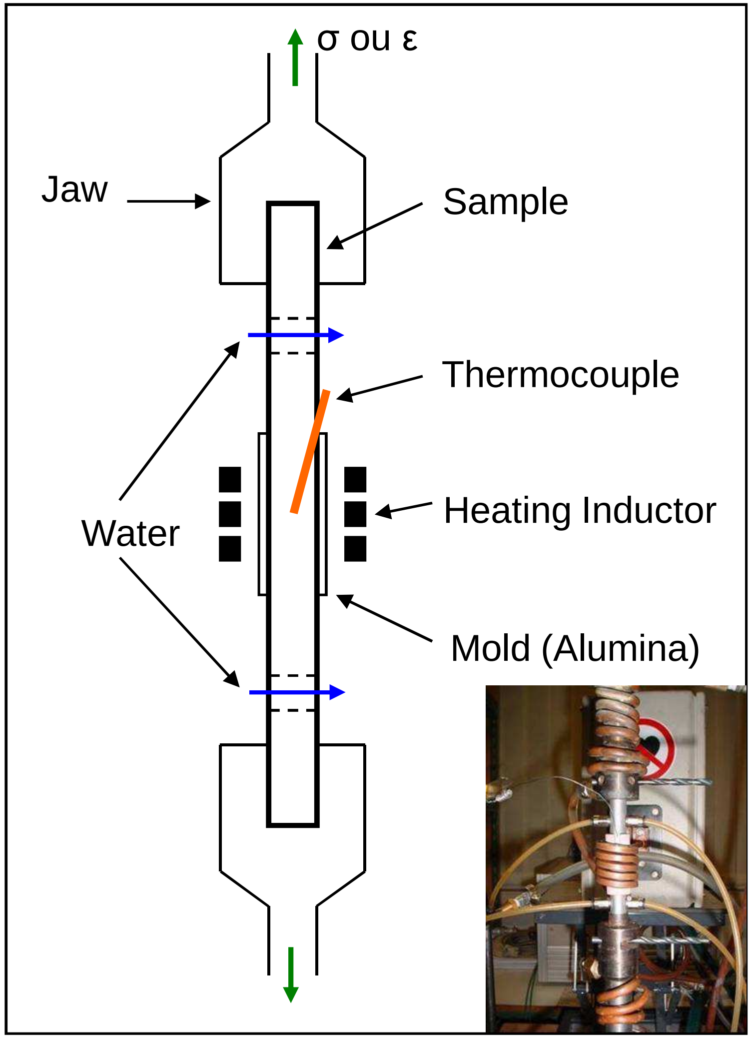
- isothermal
- non isothermal





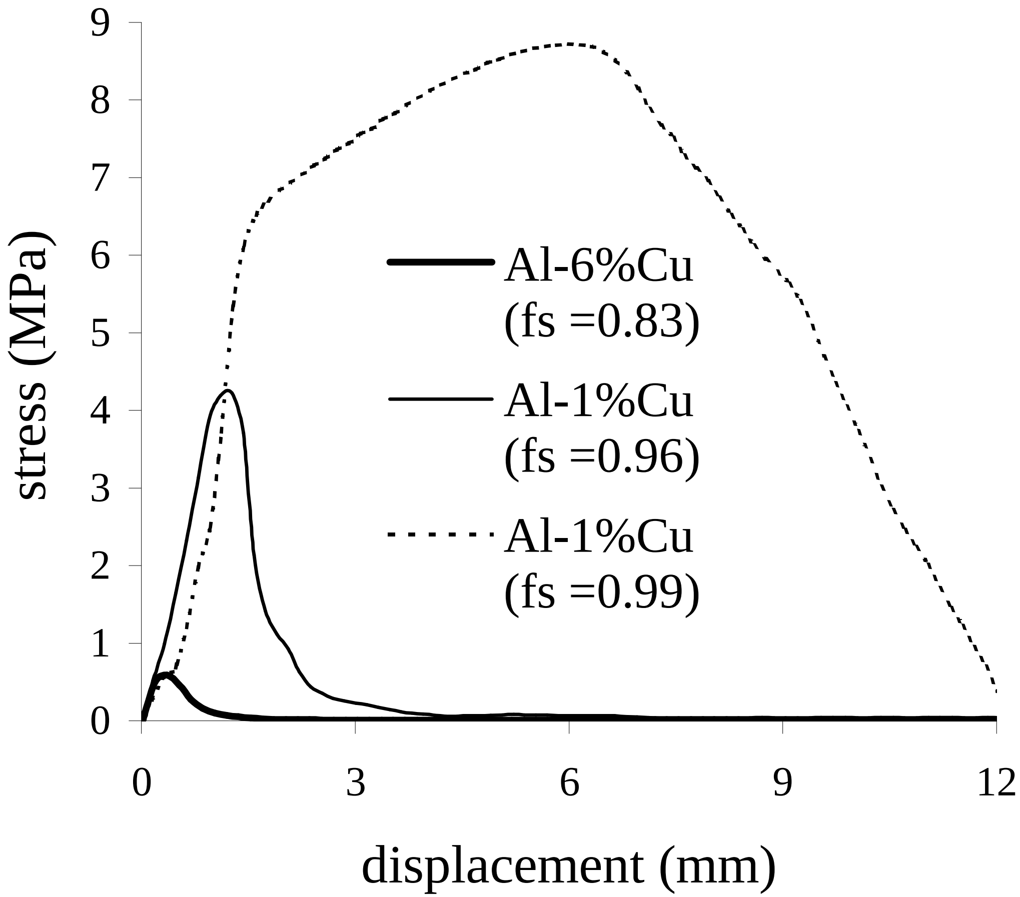
Tensile experiments on Al-Cu and 6061 aluminium alloy



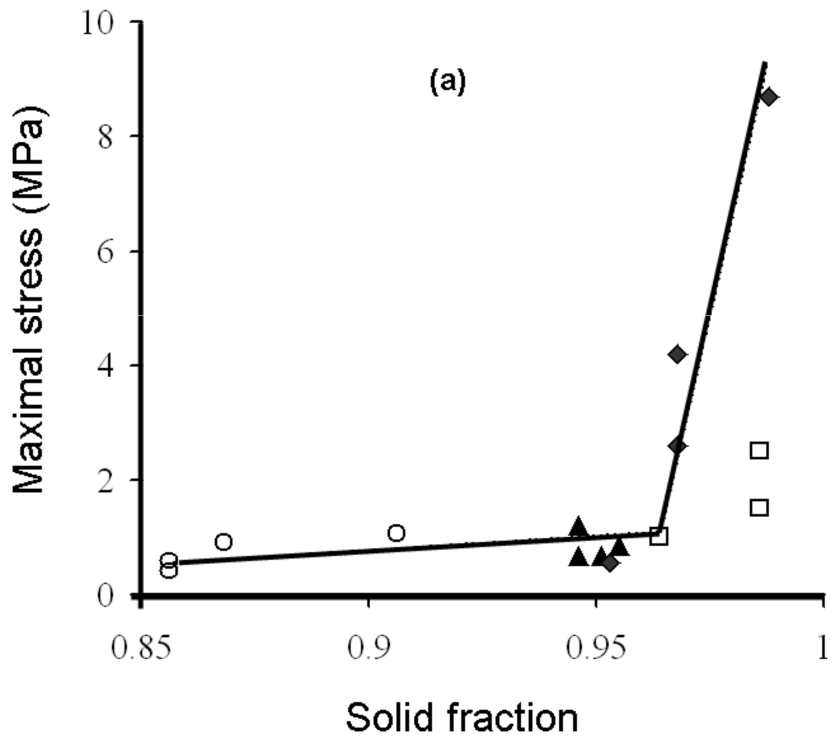


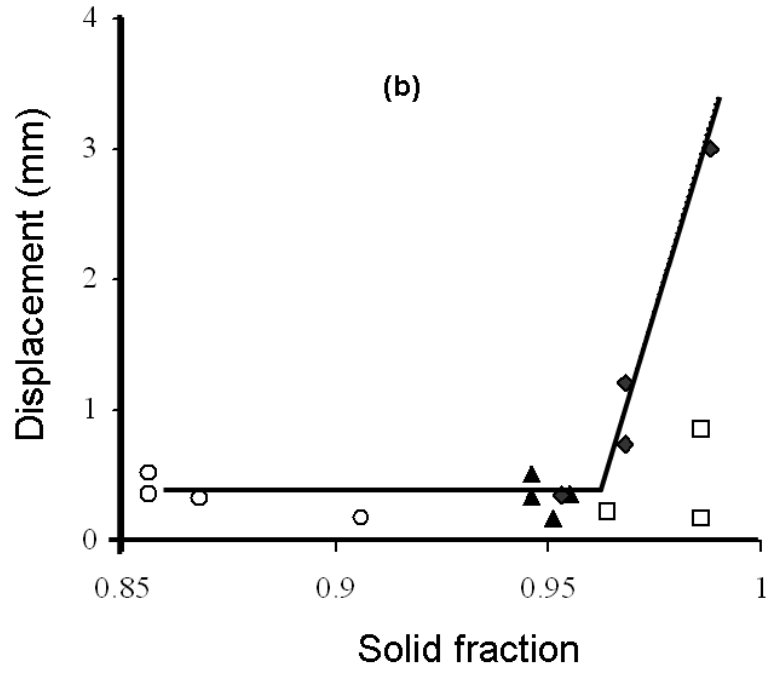
Length ~ 12 cm
Diameter ~ 10 mm

Isothermal tests on Al-Cu alloys



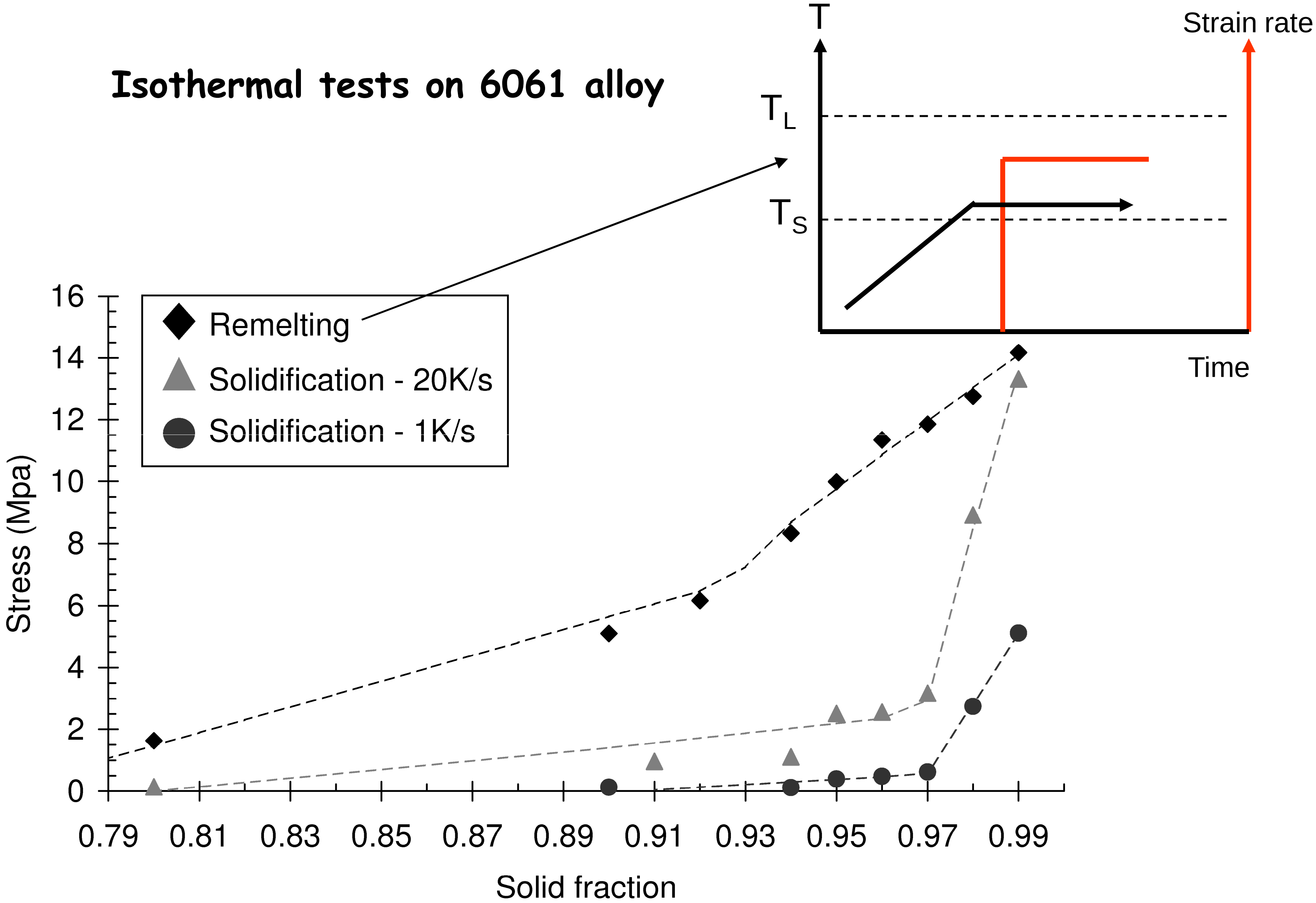
○ Al-6wt.% Cu; ▲ Al-4wt.% Cu; □ Al-2wt.% Cu; + Al-1wt.% Cu
Cooling rate = 60K/min, displacement rate = 100mm/min



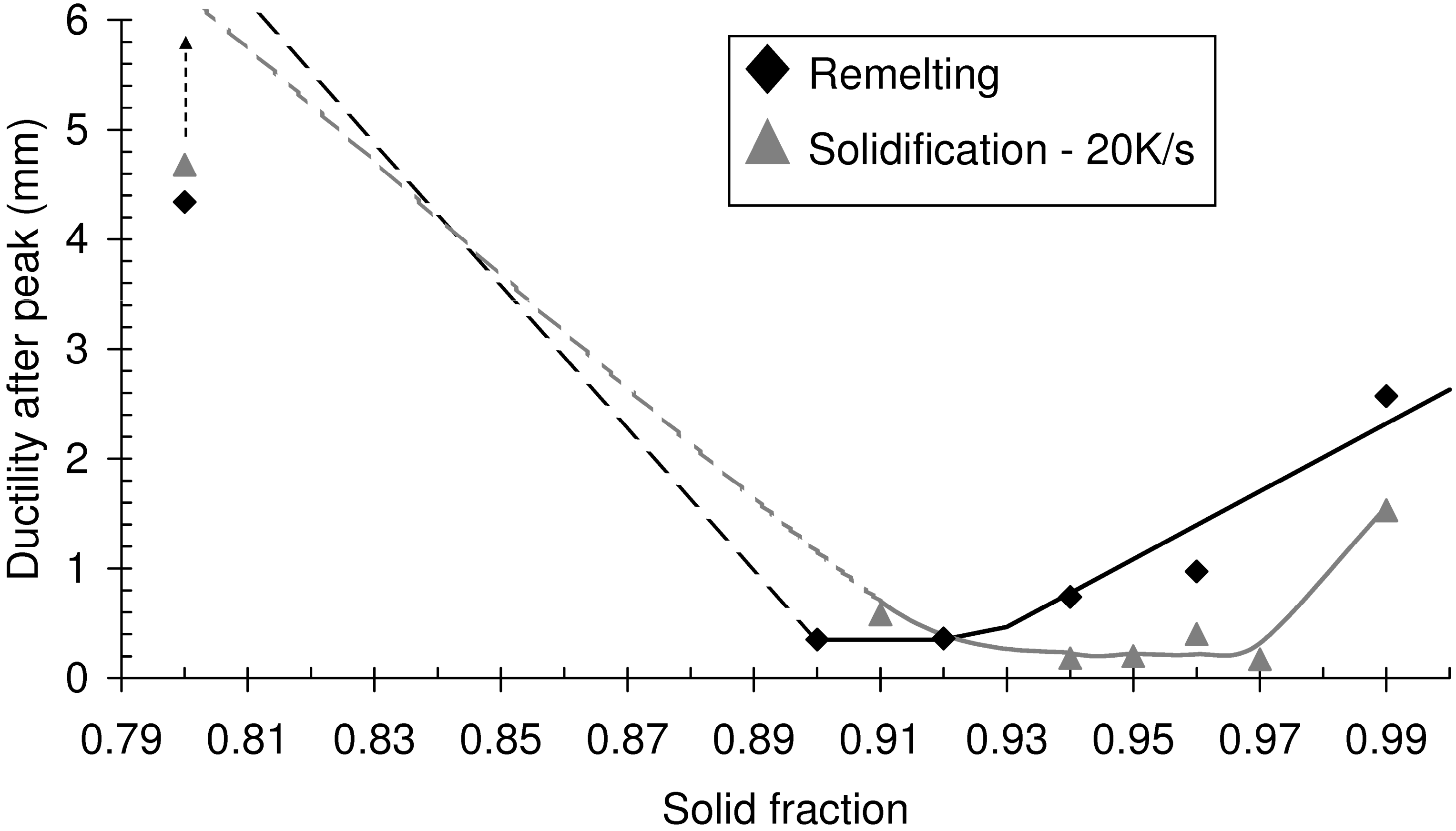


M. Suéry, C.L. Martin, M. Braccini, Y. Bréchet,
Adv. Eng. Mat., 3, (2001), 589-593

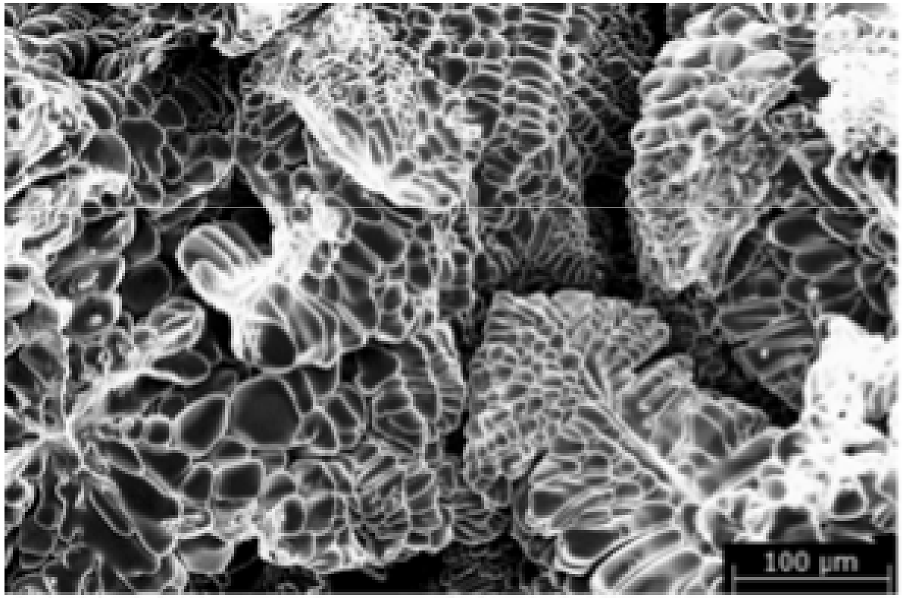
Isothermal tests on 6061 alloy



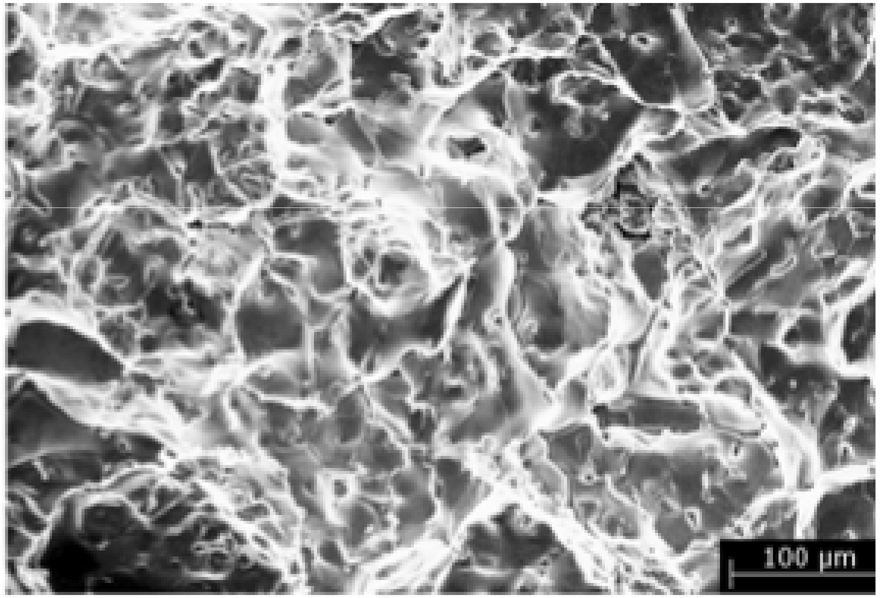
E. Giraud, M. Suéry, M. Coret
Met. Mat. Trans., 41A (2010) 2257-2268


Remelting
Solidification - 20K/s
Ductility after peak (mm)
Solid fraction
0
1
2
3
4
5
6
0.79
0.81
0.83
0.85
0.87
0.89
0.91
0.93
0.95
0.97
0.99

displacement rate = 0.1 mm/s, cooling rate = 20 K/s



Fs = 0.95

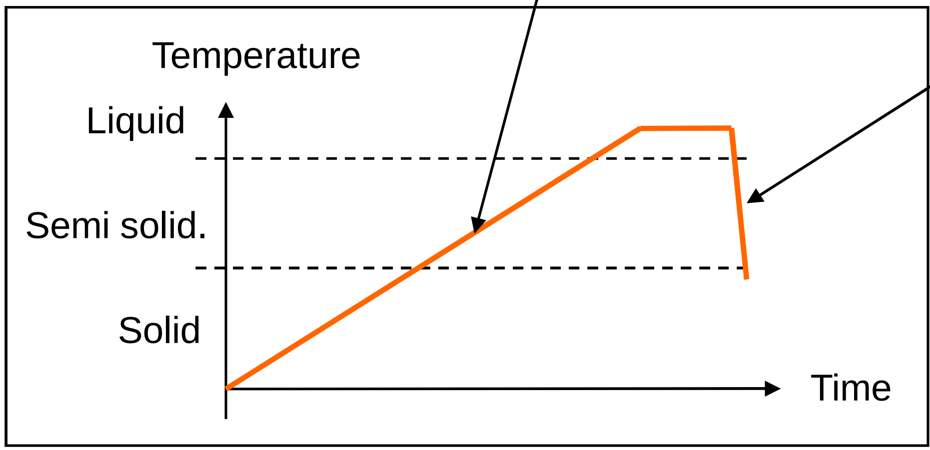


Fs = 0.99

Non isothermal test on 6061 alloy: constrained solidification

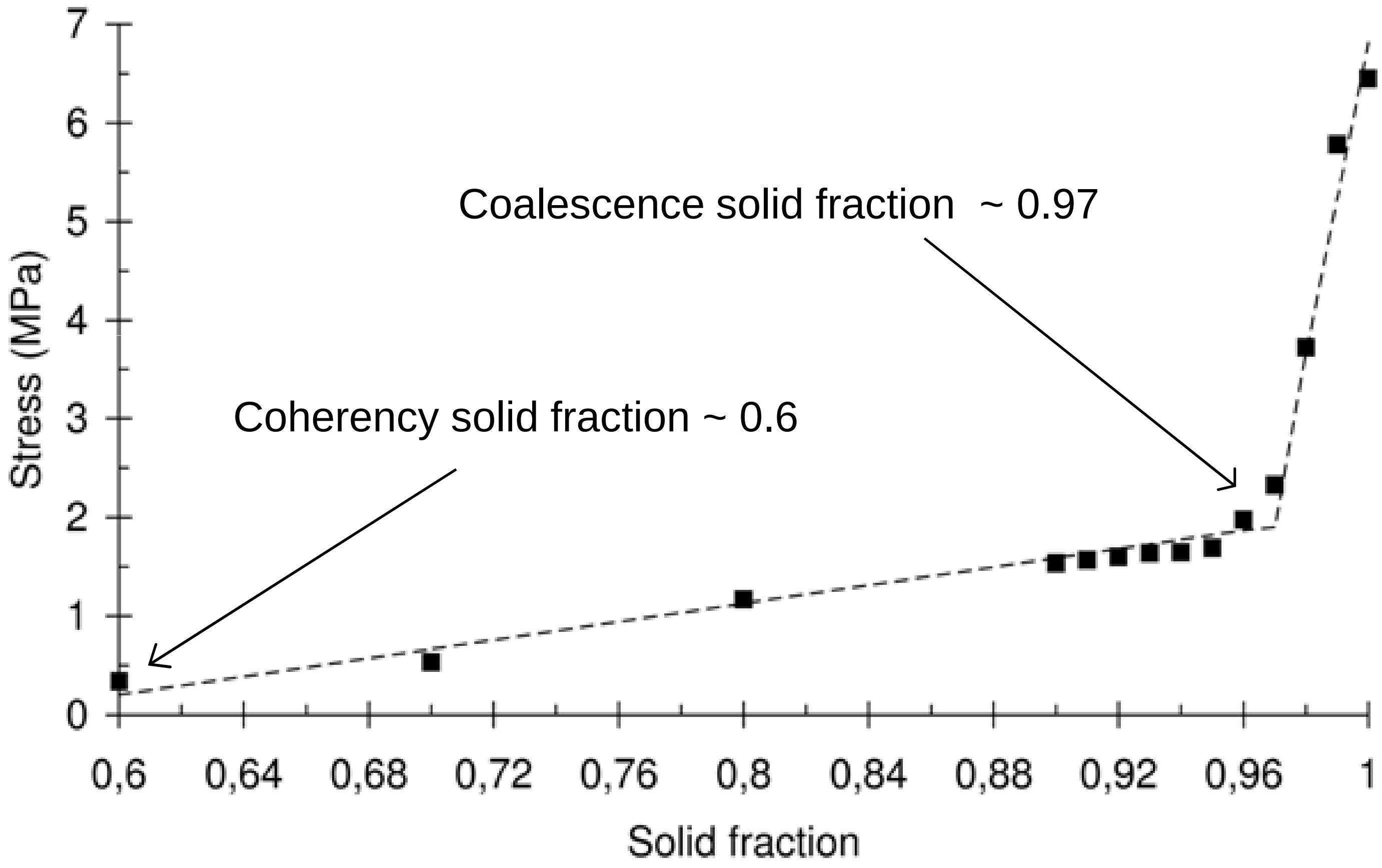
Heating rate ~ 2 K/s
Strain accommodated

Cooling rate ~ 80 K/s
Grips of the machine fixed
(constrained solidification)

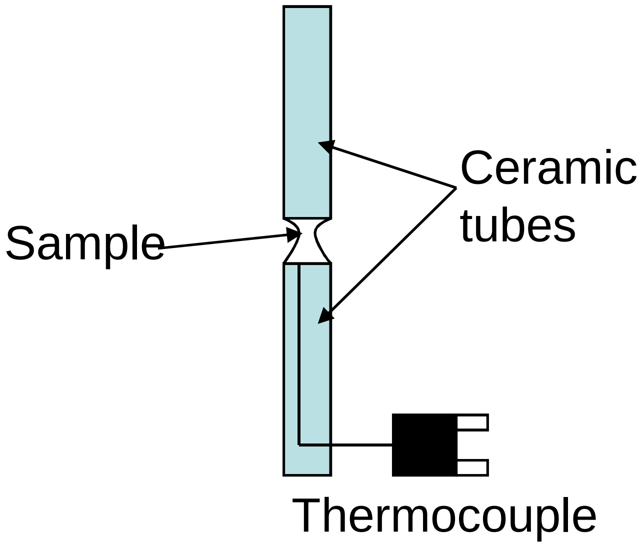


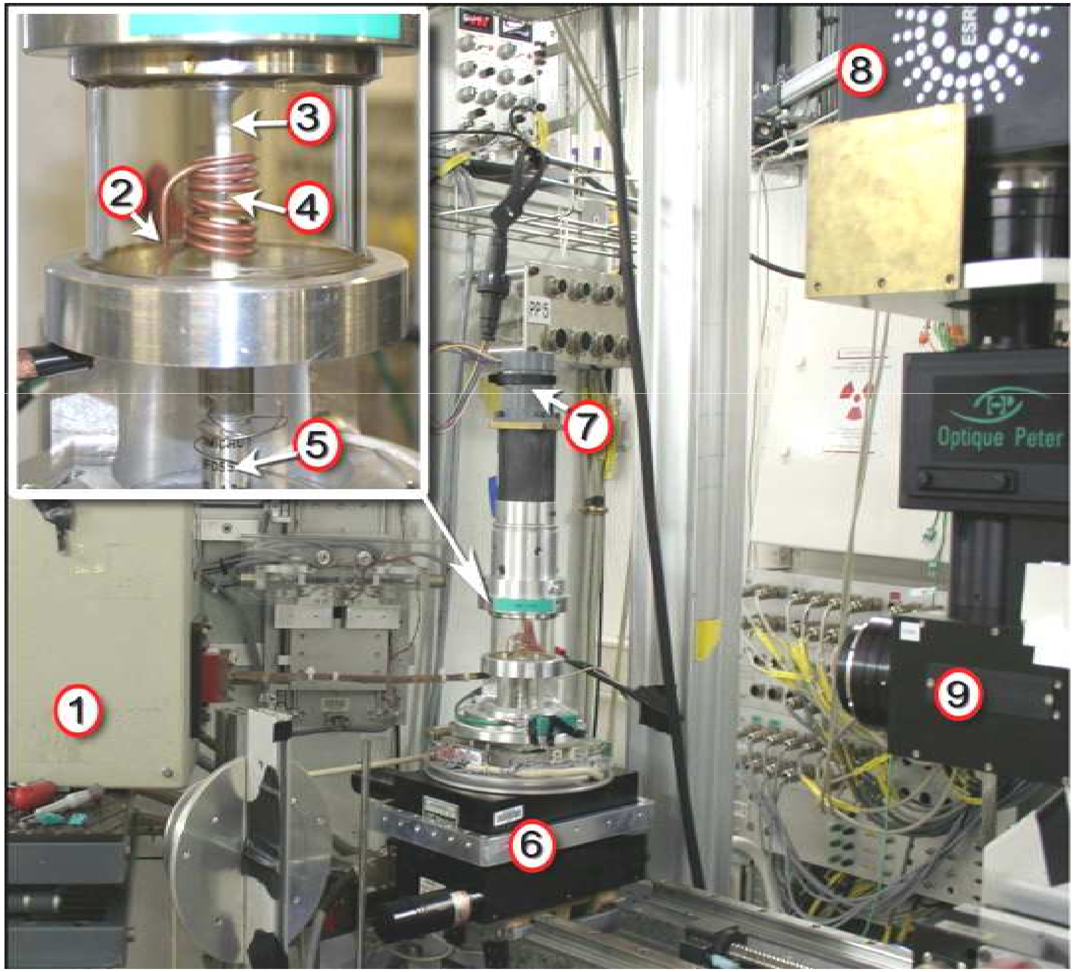
Stress as a function of temperature

E.Giraud, M. Suéry, J. Adrien, E. Maire, M. Coret
3rd International Workshop “Hot Cracking Phenomena in Welds”
15-16 Mars 2010, Columbus, USA


Coalescence solid fraction ~ 0.97
Coherency solid fraction ~ 0.6
Stress (MPa)
0
1
2
3
4
5
6
7
0,6
0,64
0,68
0,72
0,76
0,8
0,84
0,88
0,92
0,96
1
Solid fraction

X-Ray tomography experiments during solidification with tensile deformation (ESRF)

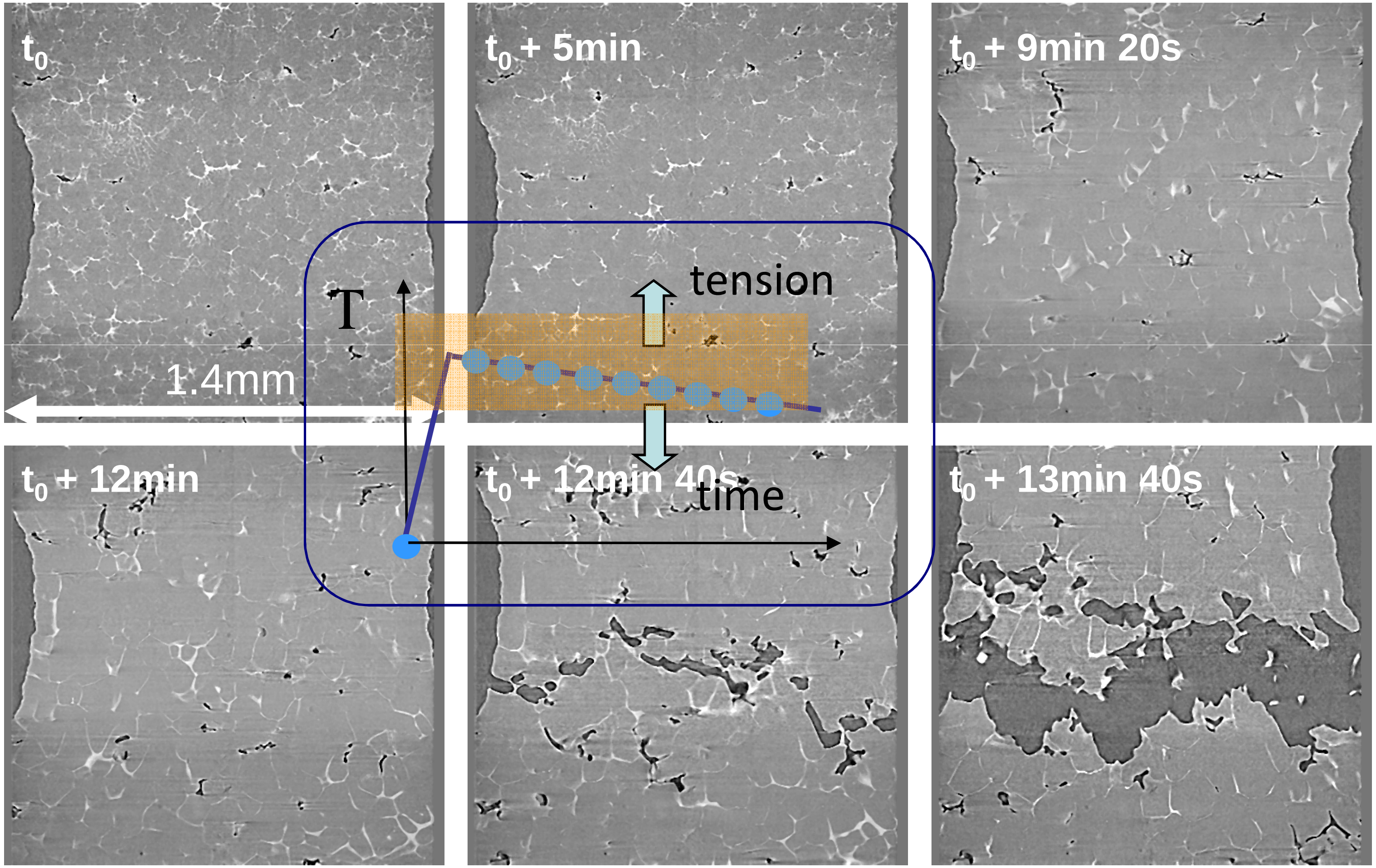




S. Terzi, L. Salvo, M. Suéry, N. Limodin, J. Adrien, E. Maire, Y. Pannier, M. Bornert, D. Bernard, M. Felberbaum, M. Rappaz, E. Boller, Scripta Materialia 61 (2009) 449–452

Al-8%Cu, cooling

Optics: 1.4 µm, initial temperature: 555°C
Scan time : 13 s, time between scans: 32 s
0.25 µm/s, 0.25°C/min



M. Suéry, J. Adrien, C. Landron, S. Terzi, E. Maire,
L. Salvo, J.-J. Blandin, Int. J. Mat. Res., 101 (2010) 1080-1088

Key problems

⇨ **Modelling of semi-solid forming in the conditions of industrial forming**

= transient situation

agglomerated microstructure ⇨ disagglomerated microstructure

⇨ **Modelling of the mechanical behaviour of semi-solid alloys at very high solid fractions ($0.9 < Fs < 1$)**

- models based on mechanics of continuous media
- granular models

⇨ **Hot tearing criterion**

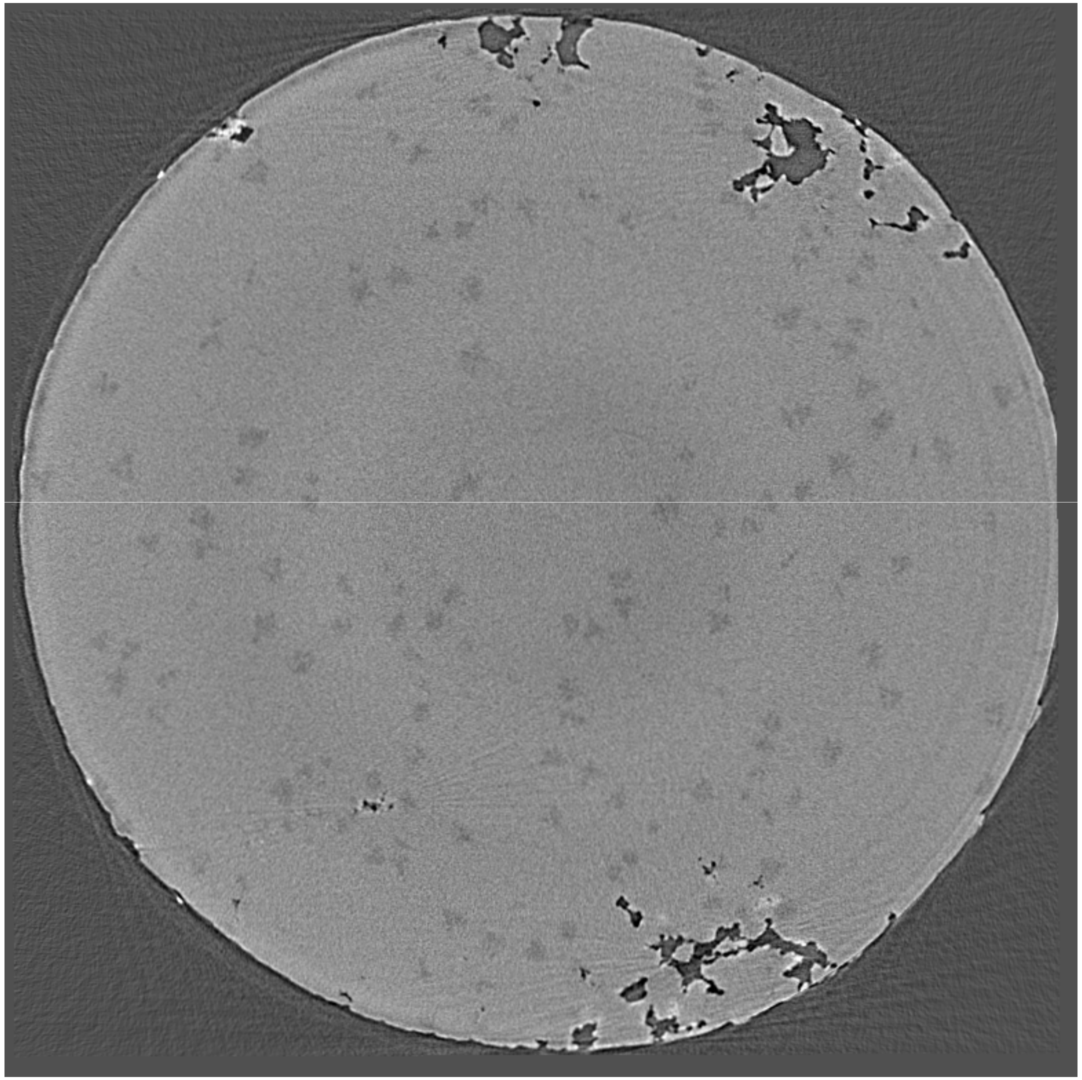
- Stress > critical stress
- Strain > critical strain
- Liquid pressure < critical value
- …

⇨ **Solidification mechanisms (complex alloys, high cooling rates,…)**

⇨ X-Ray microtomography with good temporal resolution

Al-20%Cu

- PCO DIMAX camera
- Scan duration = 0.15s allowing a cooling rate of 5°C/s
- 2 µm optics
- 80 scans during solidification



Thank you for your attention