Semi-solid Alloys

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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

\[ k_o = \frac{C_S}{C_L} \]
Main parameter = solid fraction $f_s$

Equilibrium: lever rule  
$$f_s = \frac{(C_L-Co)}{(C_L-Cs)}$$

No solid diffusion: Scheil equation  
$$C_s = ko \cdot Co \cdot (1-fs)^{(ko-1)}$$

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 $\rightarrow$ eutectic mixture)

- Alloy above the eutectic temperature
  problem: deposition of solid on the existing solid
  and then eutectic transformation
  $\rightarrow$ solid fraction overestimated

Sometimes etching allows to distinguish the two primary solids (preexisting and that formed during quenching)
Al-6.8wt%Cu held at 628°C and cooled at various rates

Expected vol. fraction of Al-phase

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 equipment

• X-Ray tomography and 3D reconstruction (computed tomography)
  • non destructive
  • high resolution (synchrotron)
  • but specific equipment
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**Post mortem**

- Several samples
- Scans at RT

**Ex situ**

- One sample
- Furnace out of tomograph

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**Solution**
- to avoid quenching
- for 3D characterisation

**Limitations:**
- Good absorption contrast between solid and liquid (Al-Cu)
- Sufficient spatial and temporal resolution
Al-8%Cu
Solidification at 3°C.min⁻¹
1.4 µm, 600 images, ~60 s
ESRF ID19

RX
Camera CCD

1.5 mm
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$_5$FeSi particles

Mechanical characterisation

\[ T_L \quad T_{coh} \quad T_{coal} \quad T_S \]

\[ g_s = 1 \]

solidification
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...

\[ \eta = \eta (\eta_L, Fs, \text{shear rate}, \ldots) \]

Problem: usually determination carried out at steady state

Reality: injection of semi-solid alloy in a mold takes less than 1 s
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-6%Cu** (fs =0.83)
- **Al-1%Cu** (fs =0.96)
- **Al-1%Cu** (fs =0.99)
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- ○ Al-6wt.% Cu;
- ▲ Al-4wt.% Cu;
- □ Al-2wt.% Cu;
- + Al-1wt.% Cu

Cooling rate = 60K/min, displacement rate = 100mm/min

Isothermal tests on 6061 alloy

- **Remelting**
- **Solidification - 20K/s**
- **Solidification - 1K/s**

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

![Graph showing ductility after peak vs solid fraction for remelting and solidification at 20K/s.](image-url)
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 $\sim 0.97$

Coherency solid fraction $\sim 0.6$
X-Ray tomography experiments during solidification with tensile deformation (ESRF)

**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

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