Correlations between microstructure and rheological behaviour of partial melt alloys

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Outline

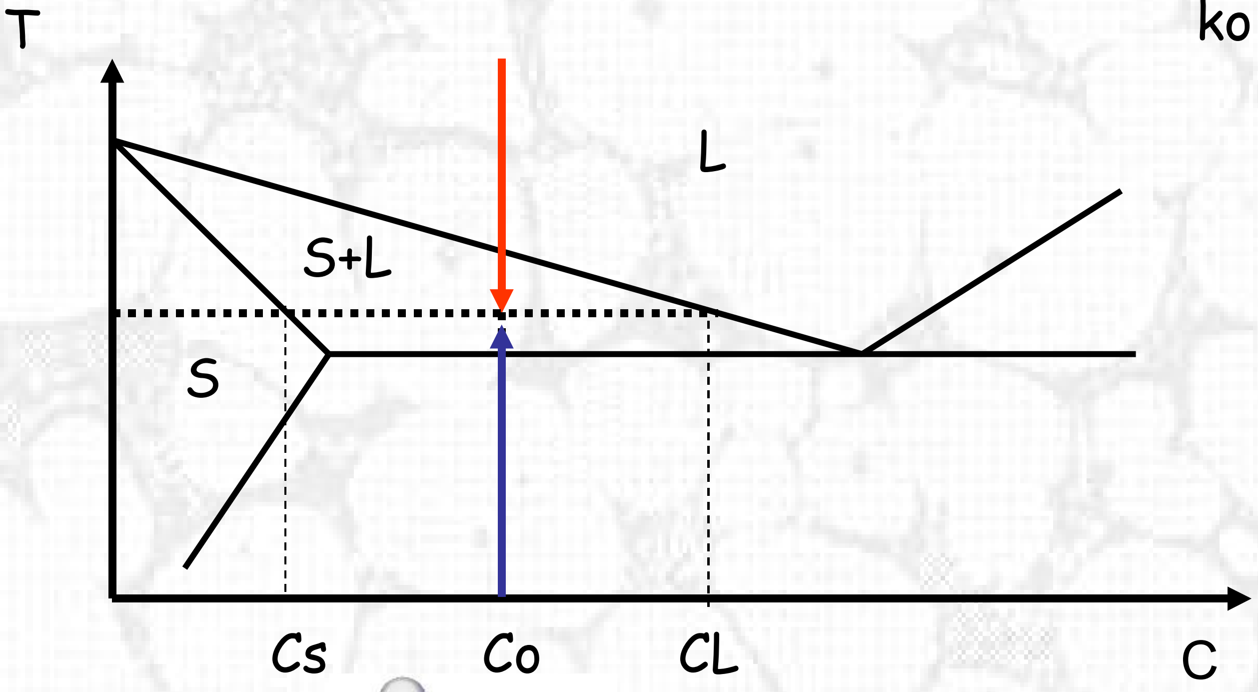
- Introduction
- Phenomenology of the behaviour of globular structures
- Phenomenology of the behaviour of dendritic structures
- Modelling
- Conclusion

Introduction

Partial melts in alloys

- ☞ **During solidification**
- ☞ **During partial melting**

Partition ratio
$k_o = C_s/C_L$



Main parameter = volume fraction of solid 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
…

Generally

For semi-solid alloys at low solid fractions (suspensions),

shear viscosity is used to characterise the behaviour which is assumed to be homogeneous (no phase separation)

Viscosity = F (liquid viscosity, shear rate, time, solid characteristics)

Higher solid fractions

segregation phenomena can occur

→ Two-phase viscoplastic models

Very high solid fractions

No liquid flow → Porous solid with liquid pockets or films

Two main situations:

Forming in the semi-solid state ⇨
- globular structures
- $0.2 < fs < 0.6$

☞ Suspension models usually sufficient

☞ Two-phase models at high solid fractions for prediction of phase separation (sponge effect)

Conventional solidification ⇨
- dendritic structures
- $0 < fs < 1$

☞ Suspension models are of no use

☞ Two-phase models are required for prediction of defects (segregation, pores, hot tears…)

Phenomenology of the behaviour of globular structures

Effect of solid fraction

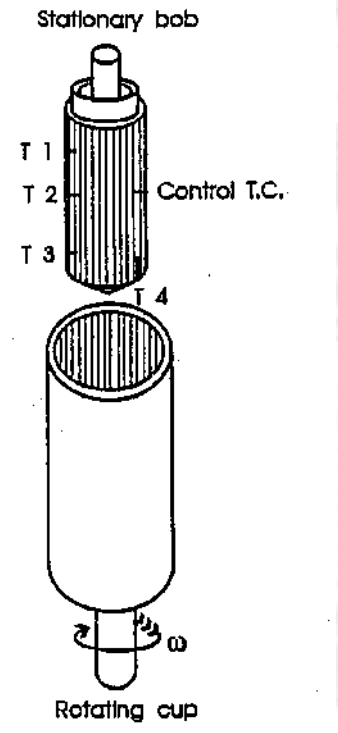
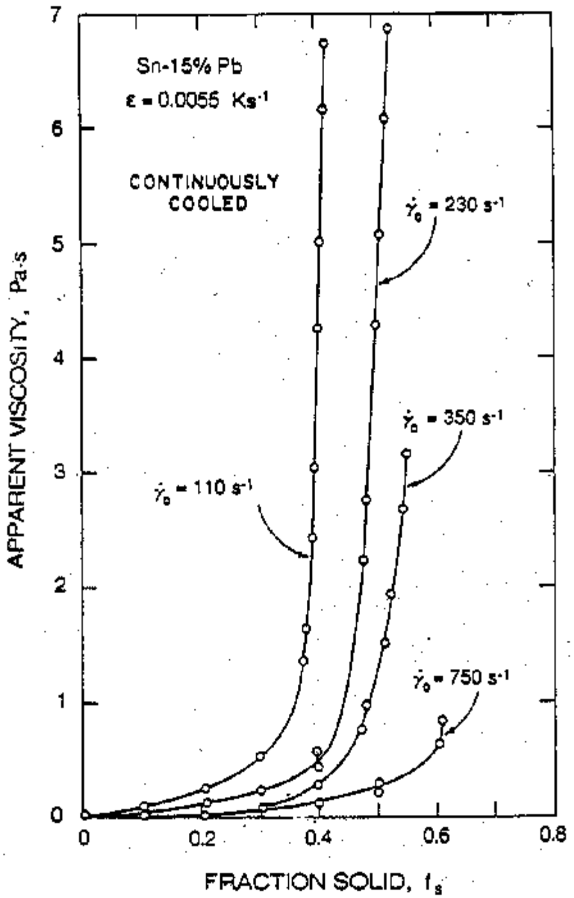
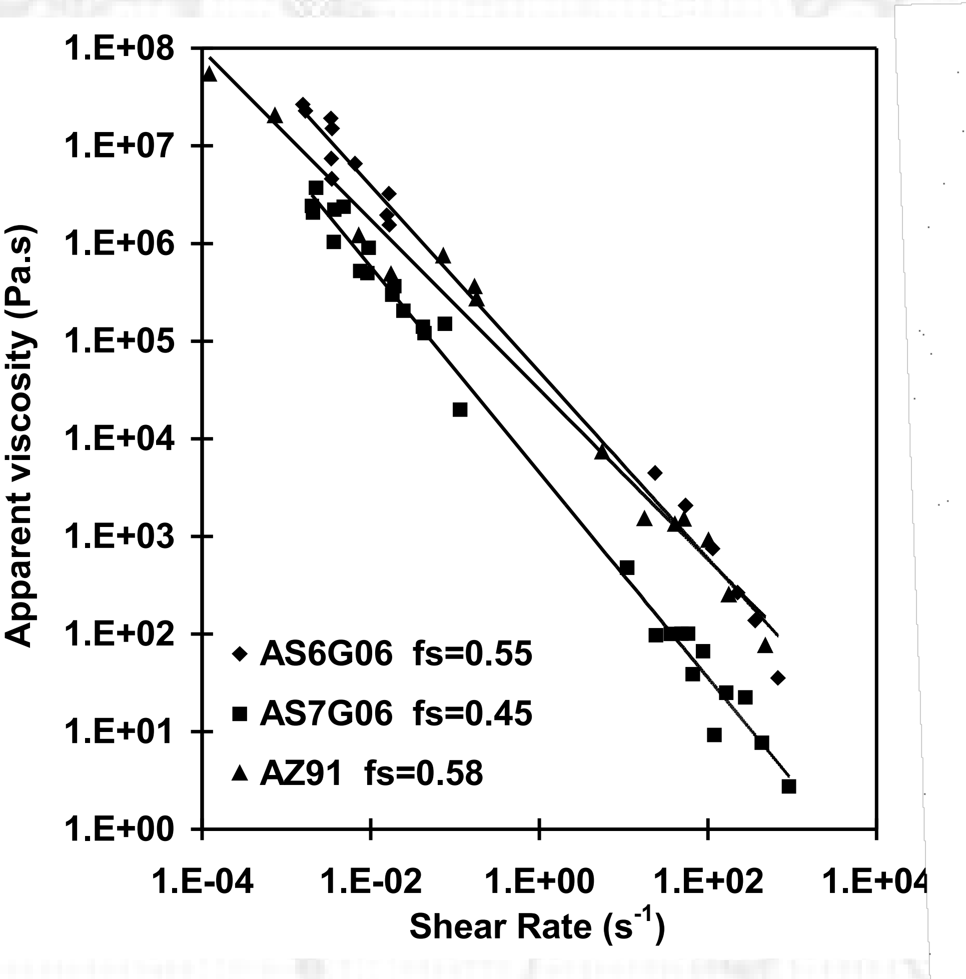


Figure 3: Schematic of graphite rotating cup and stationary bob.



Effect of shear rate: shear thinning behaviour



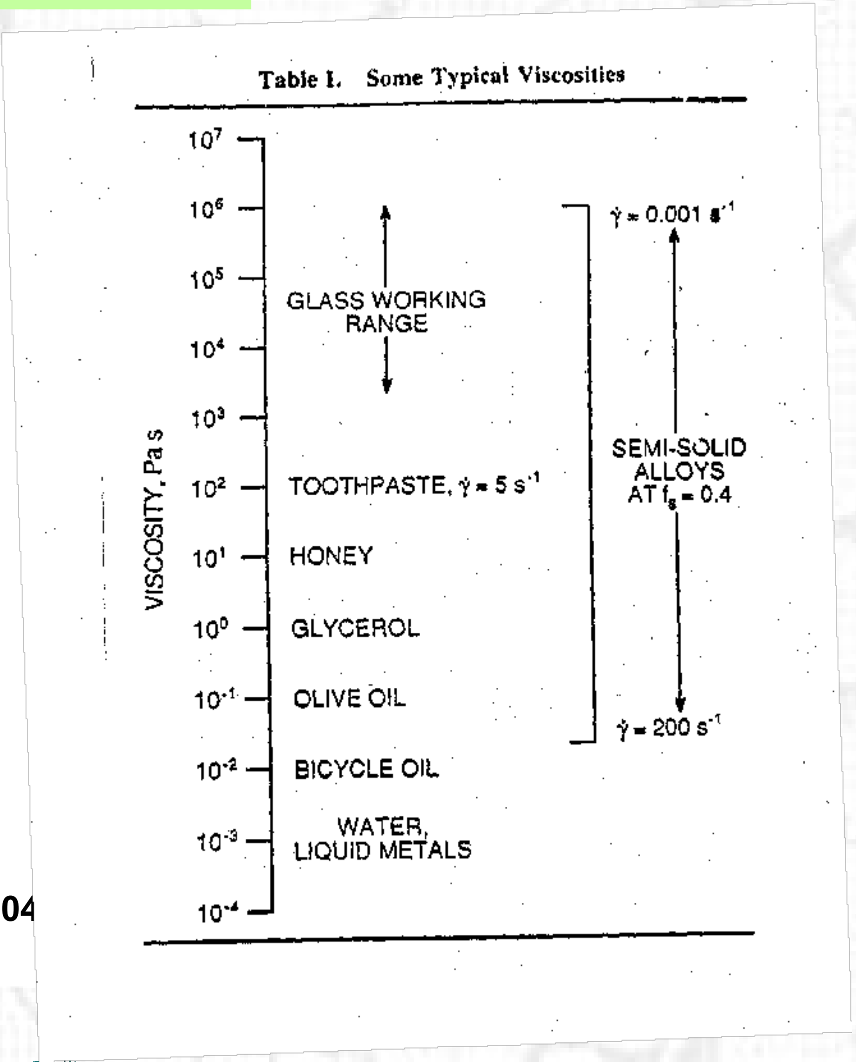
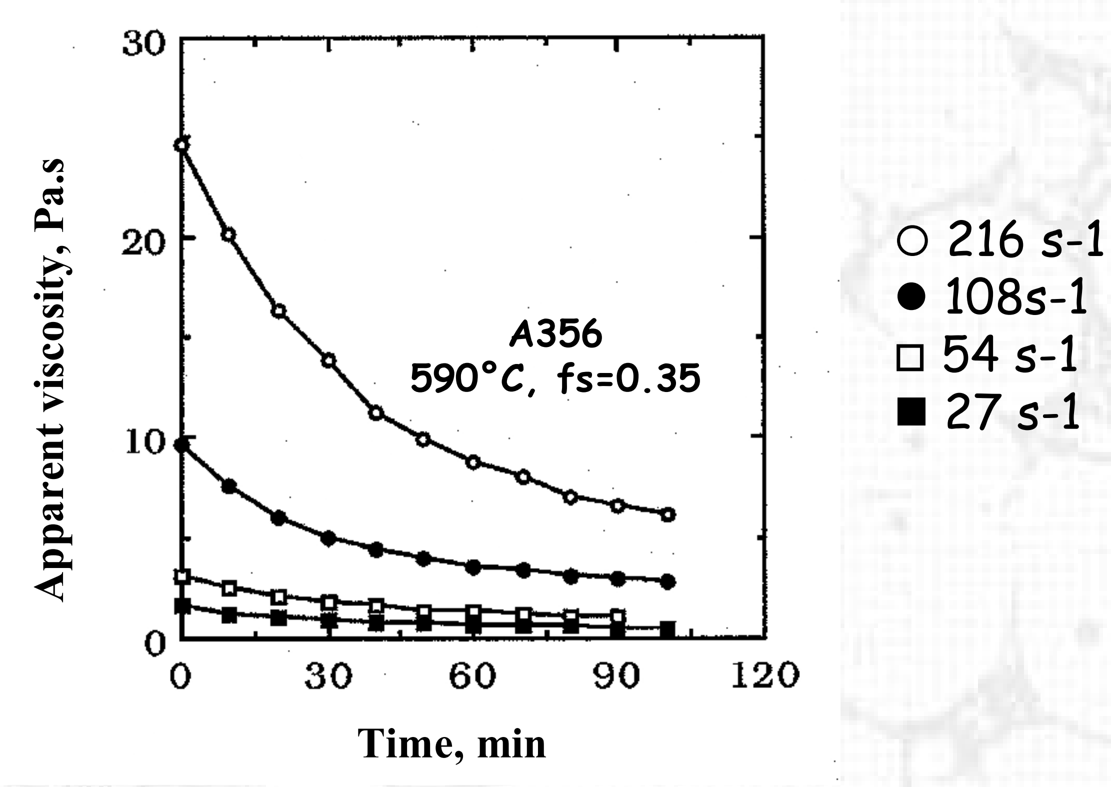


Table I. Some Typical Viscosities

VISCOSITY, Pa s		
10^7		
10^6		$\dot{\gamma} = 0.001\ s^{-1}$
10^5	GLASS WORKING RANGE	
10^4		
10^3		SEMI-SOLID ALLOYS AT $f_s = 0.4$
10^2	TOOTHPASTE, $\dot{\gamma} = 5\ s^{-1}$	
10^1	HONEY	
10^0	GLYCEROL	
10^{-1}	OLIVE OIL	$\dot{\gamma} = 200\ s^{-1}$
10^{-2}	BICYCLE OIL	
10^{-3}	WATER, LIQUID METALS	
10^{-4}		

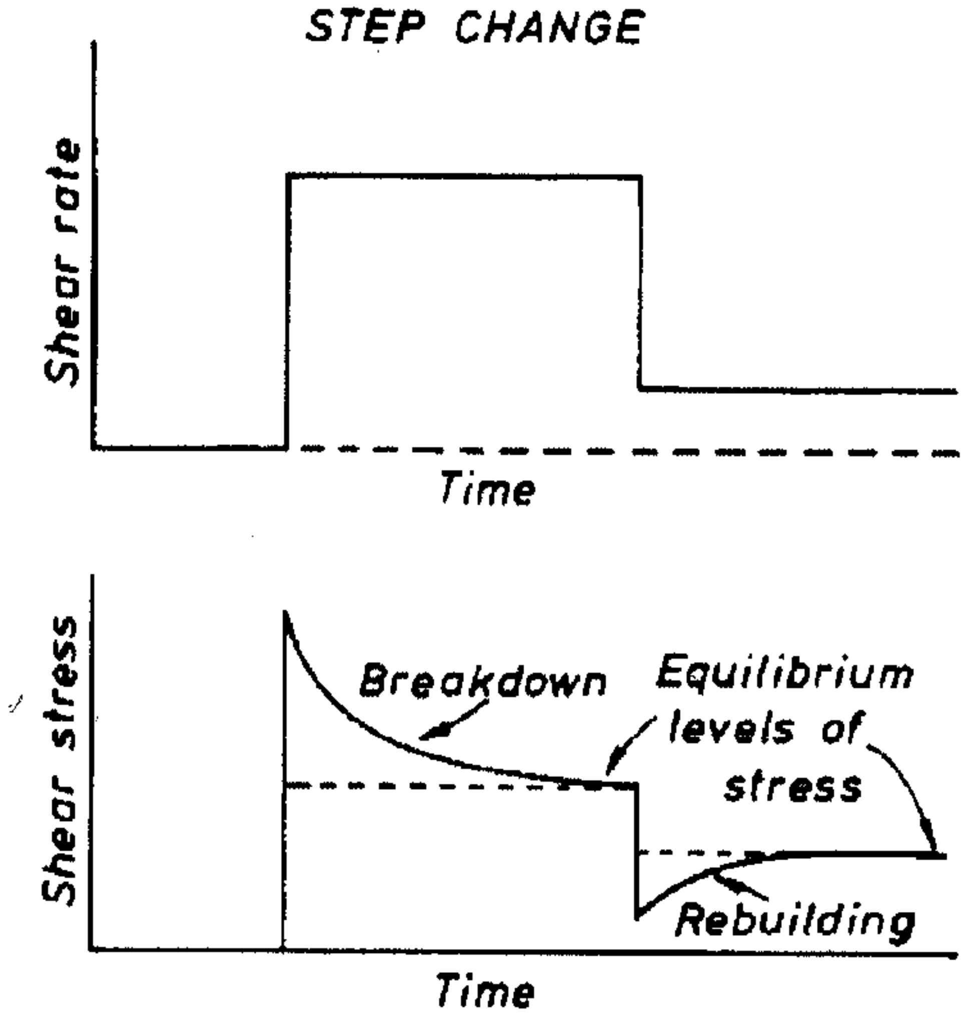
In addition, strong influence of time on viscosity (or shear stress)

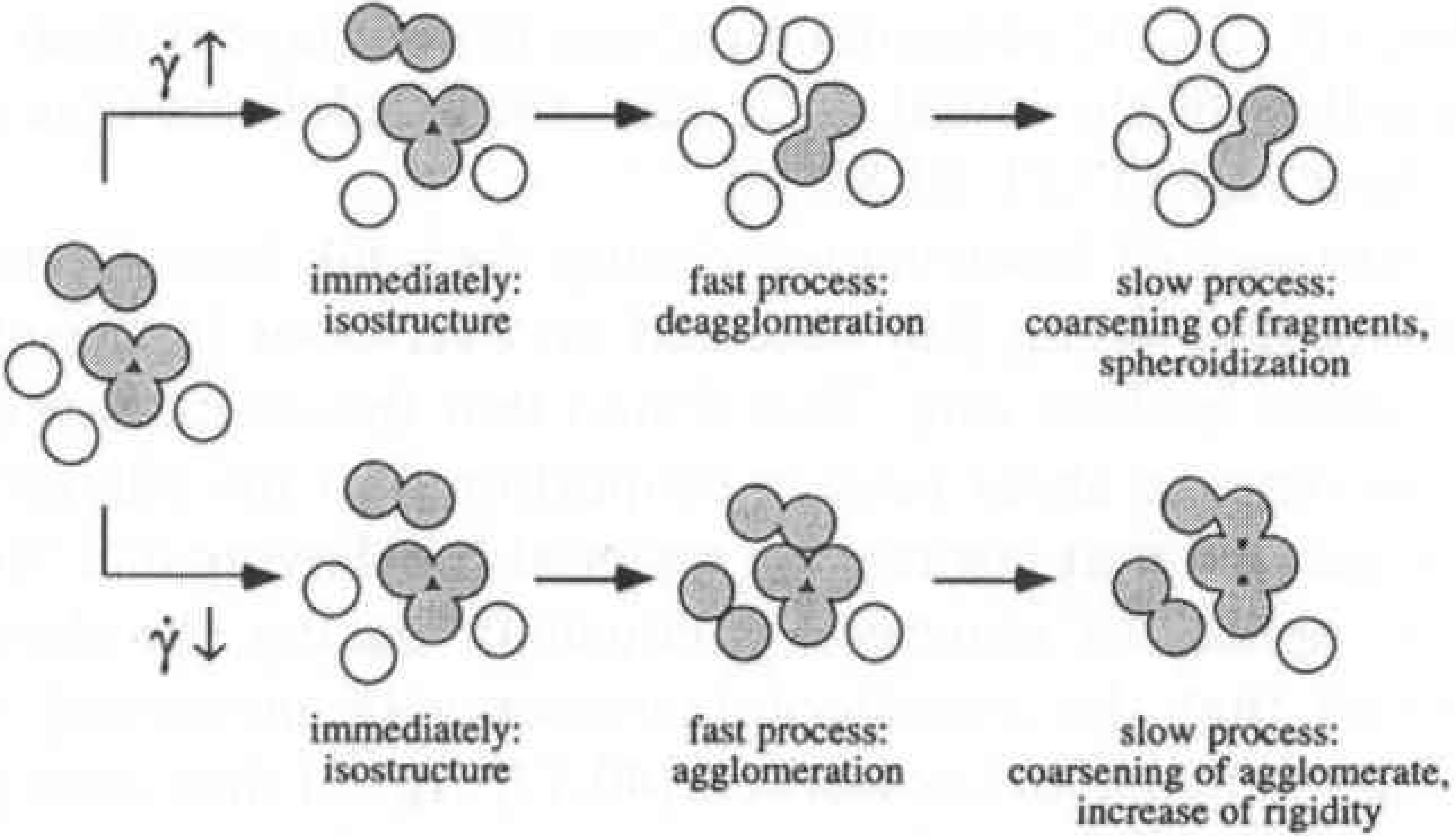


A356
590°C, fs=0.35

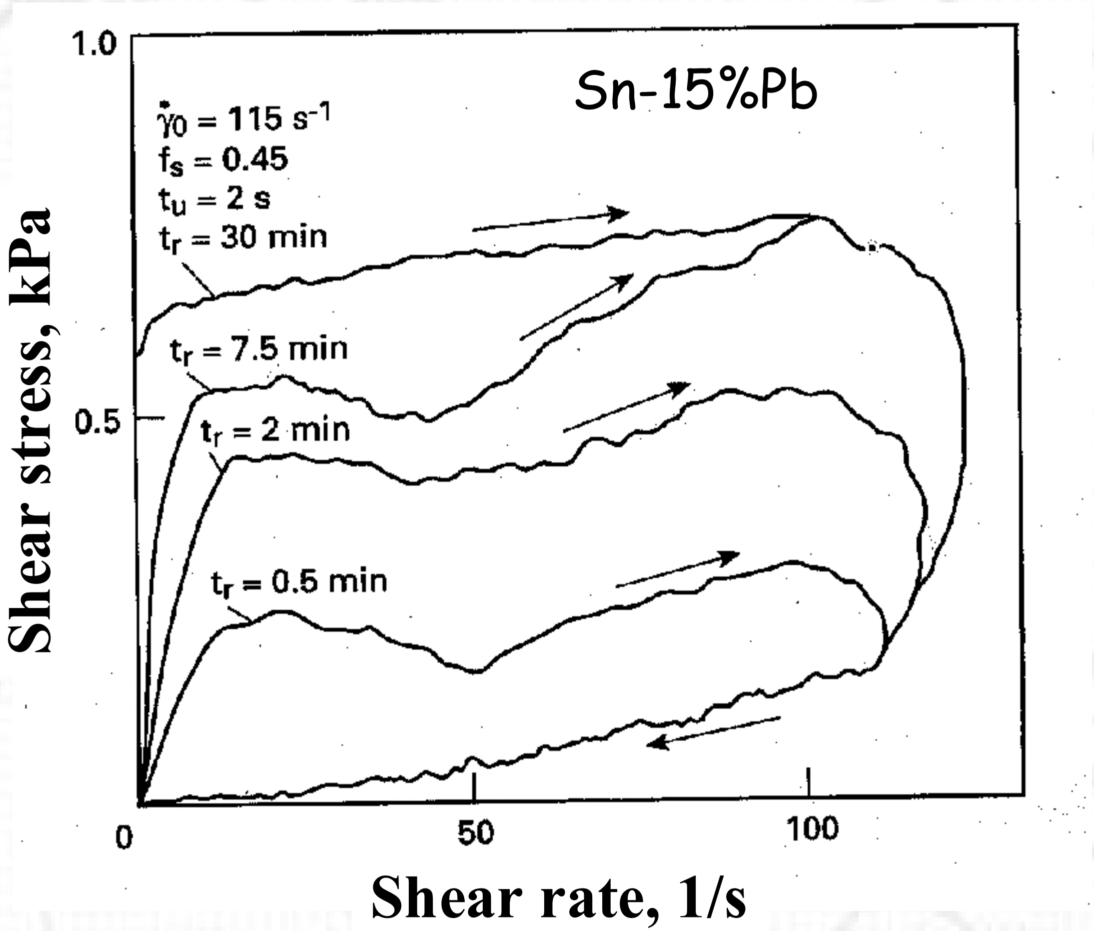
○ 216 s-1
● 108s-1
□ 54 s-1
■ 27 s-1

= **thixotropy** interpreted by agglomeration and deagglomeration phenomena of globules which depend on time and shear rate


STEP CHANGE
Shear rate
Time
Shear stress
Breakdown
Equilibrium levels of stress
Rebuilding
Time


$\dot{\gamma}\uparrow$
immediately:
isostructure
fast process:
deagglomeration
slow process:
coarsening of fragments,
spheroidization
$\dot{\gamma}\downarrow$
immediately:
isostructure
fast process:
agglomeration
slow process:
coarsening of agglomerate,
increase of rigidity

Effect of rest time

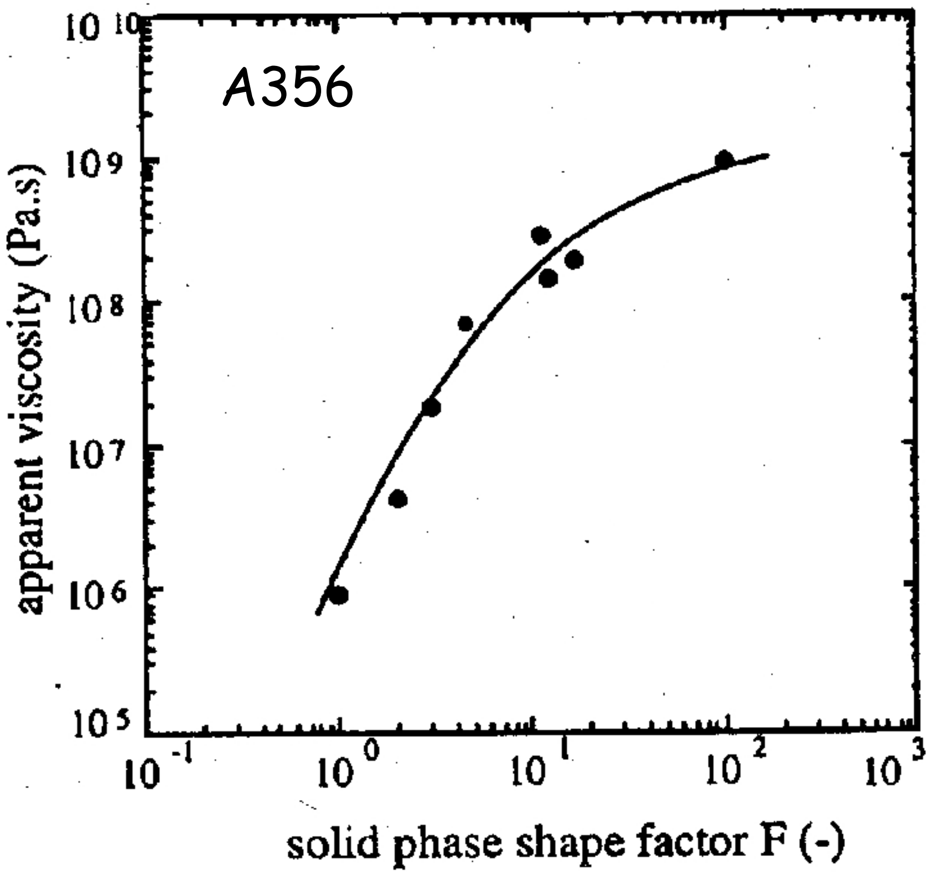


t_r = rest time

t_u = time required to increase the shear rate to its max. value

Effect of shape factor

$$F = 1/6\pi\, f_s.(S_v^2/N_A)$$



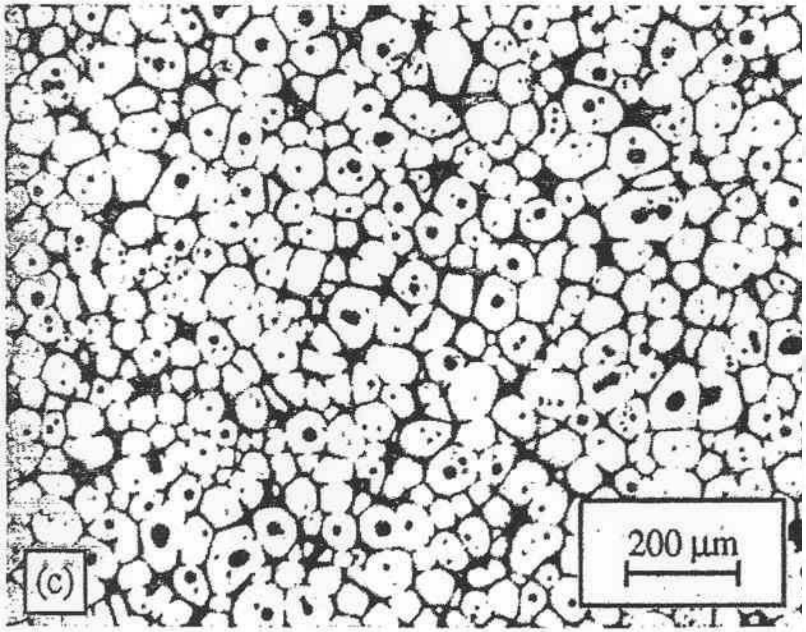
Usual definition of shape factor:

$$F = 4A/P^2$$

Effect of entrapped liquid

Semi-solid structures obtained by partial remelting

A357 alloy, 30s at 580°C



→ Liquid entrapped inside the solid phase which does not participate to the deformation

↓

Apparent solid fraction larger than the real one

Effect of solid connectivity

Solid connectivity defined by the contiguity parameter

$Cs = 2Ass/(2Ass + Asl)$

Ass = contact area between solid particles
Asl = interface area between solid and liquid

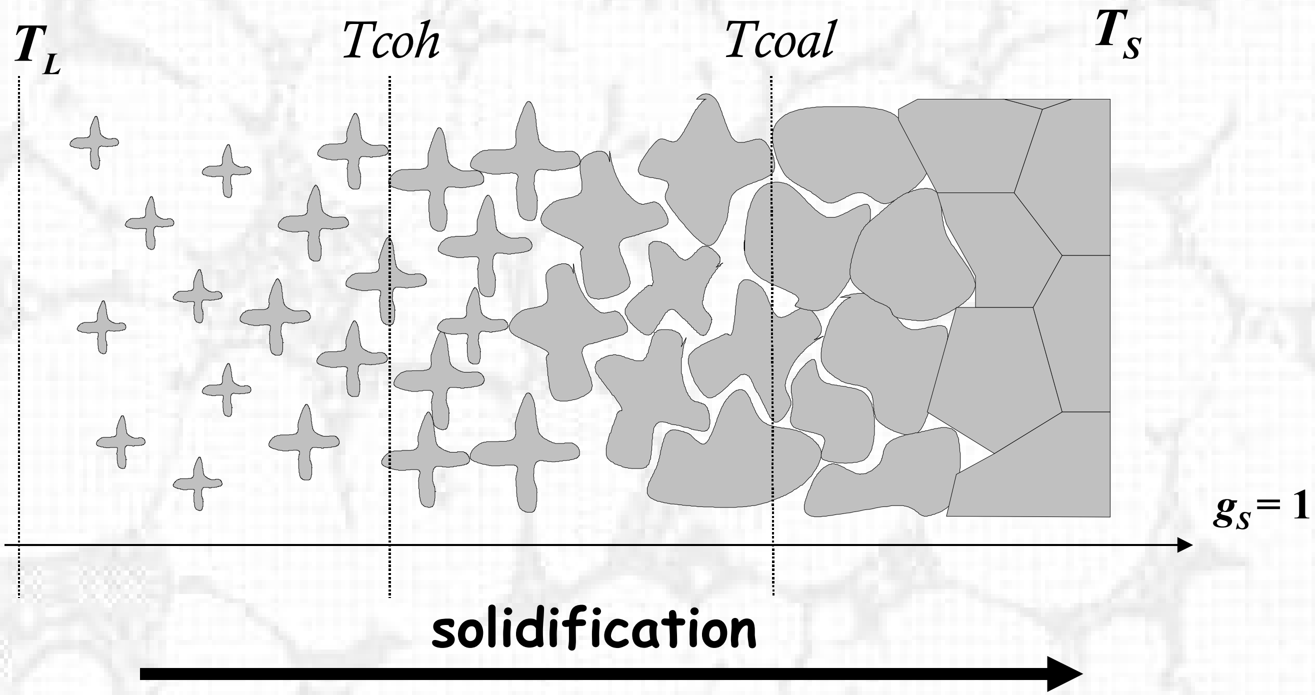
Contiguity volume = Cs.fs

For homogeneous deformation under low stresses (AA 6082)

$\boxed{Cs.fs < 0.30}$

Gullo, Steinhoff, Uggowitzer
S2P06, Torino, 2000

Phenomenology of the behaviour of dendritic structures



☞ Between TL and Tcoh: solid dendrites are free to move

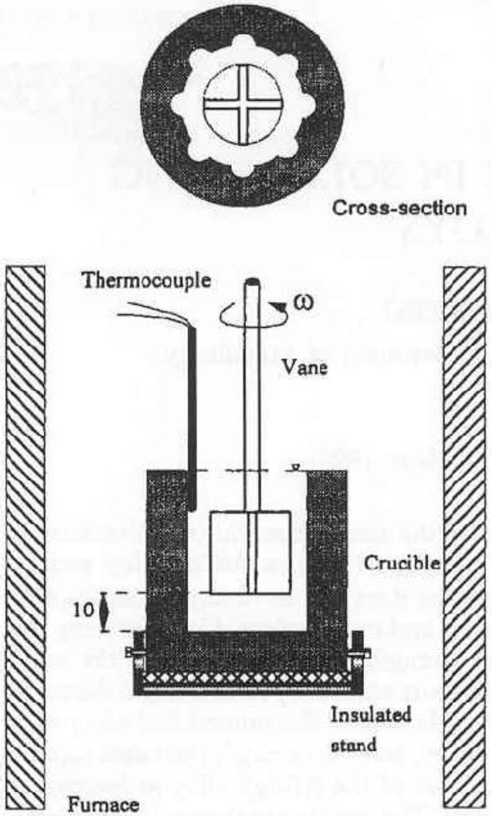
→ Similar behaviour as globular structures at low fs

→ Low viscosity

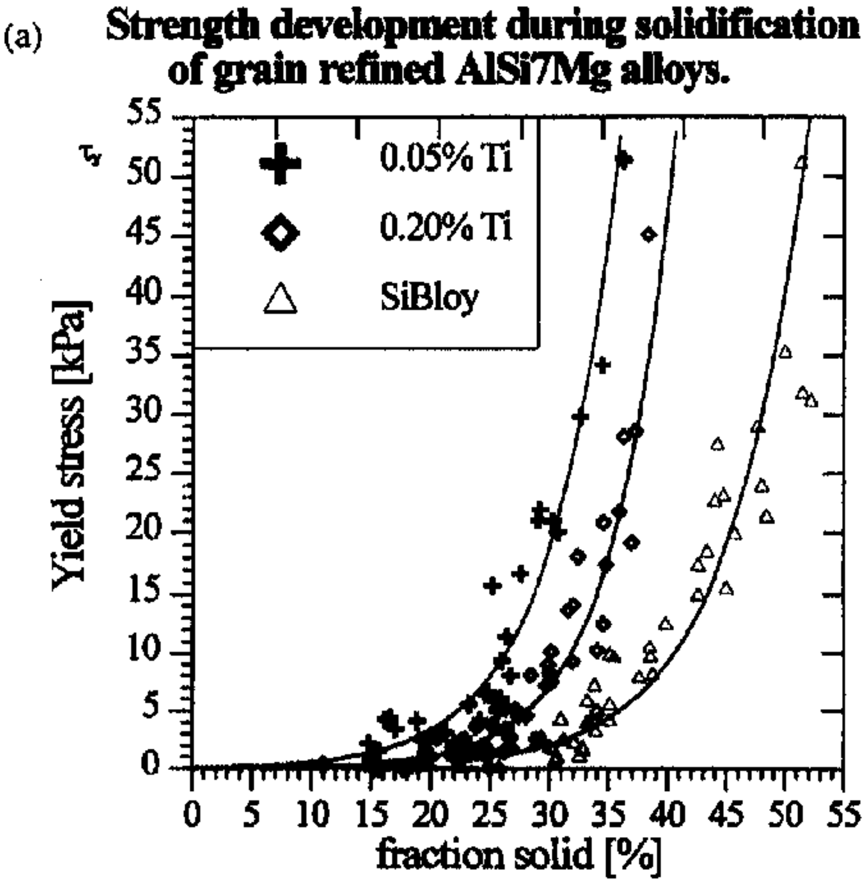
☞ Beyond Tcoh: dendrites start to interact

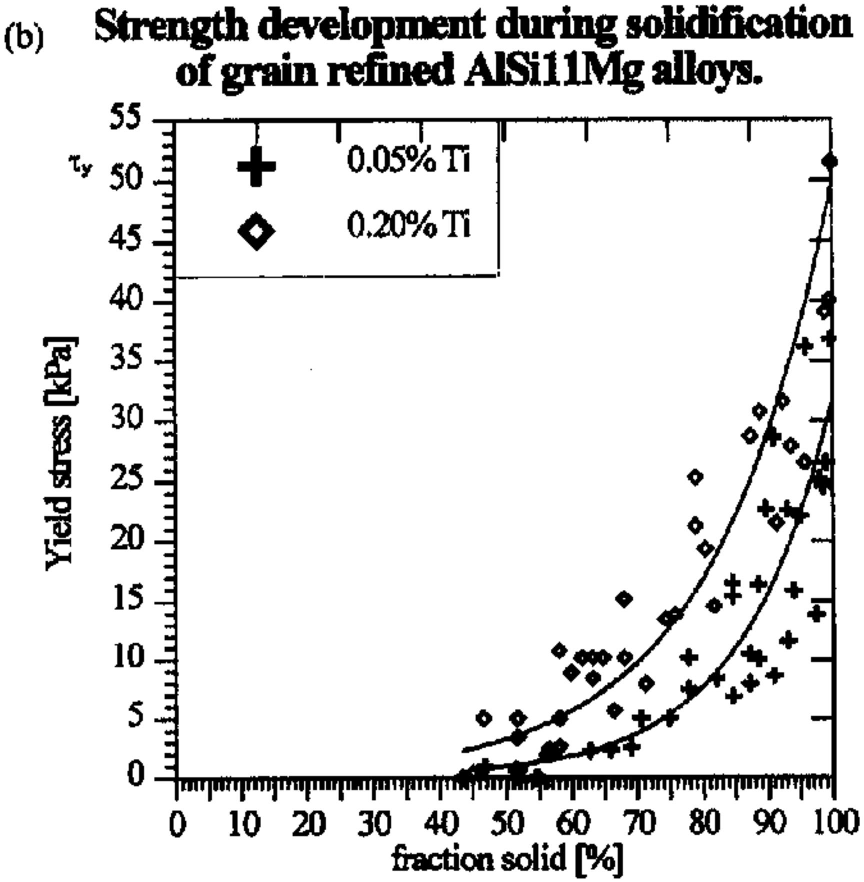
→ Shear strength becomes non negligible

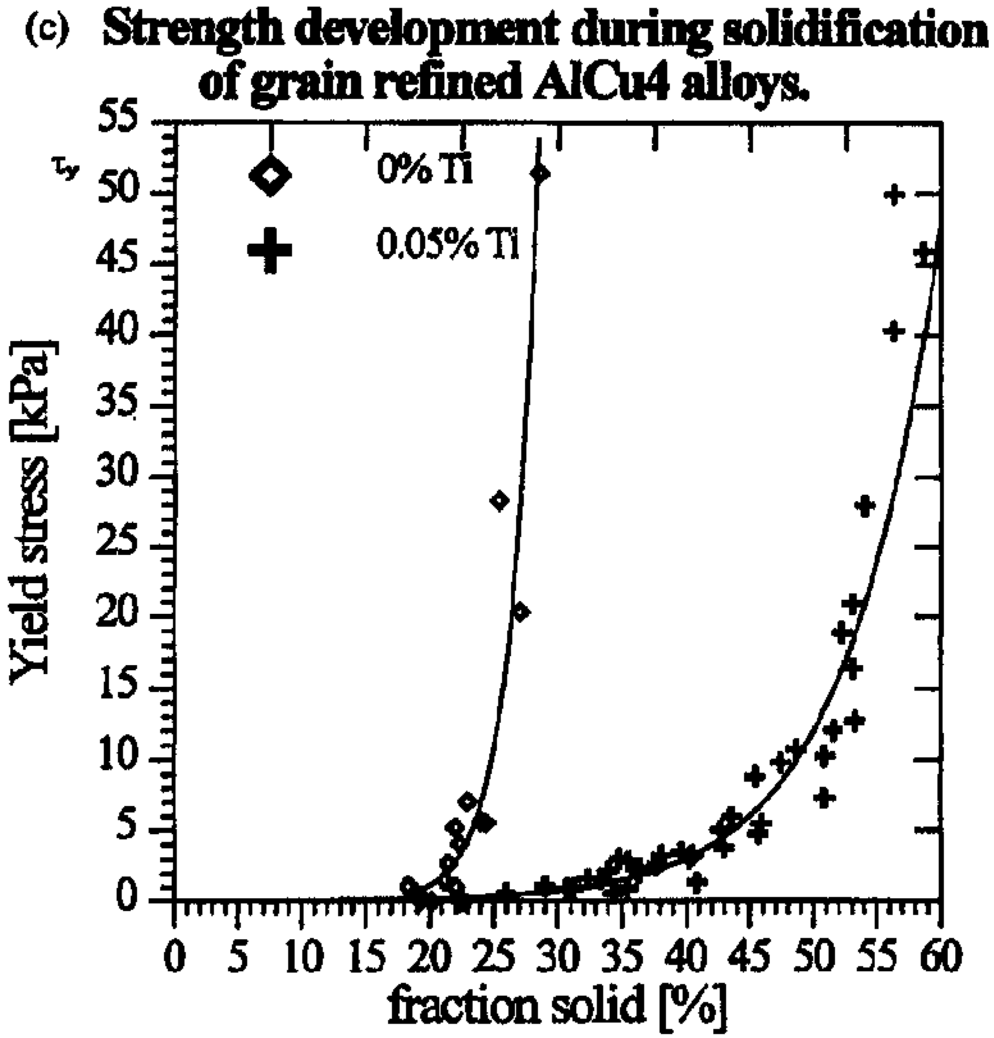
Ex: shear strength measured with a vane rheometer



A.K. Dahle, L. Arnberg
Acta Mater. 1997


(a) **Strength development during solidification of grain refined AlSi7Mg alloys.**

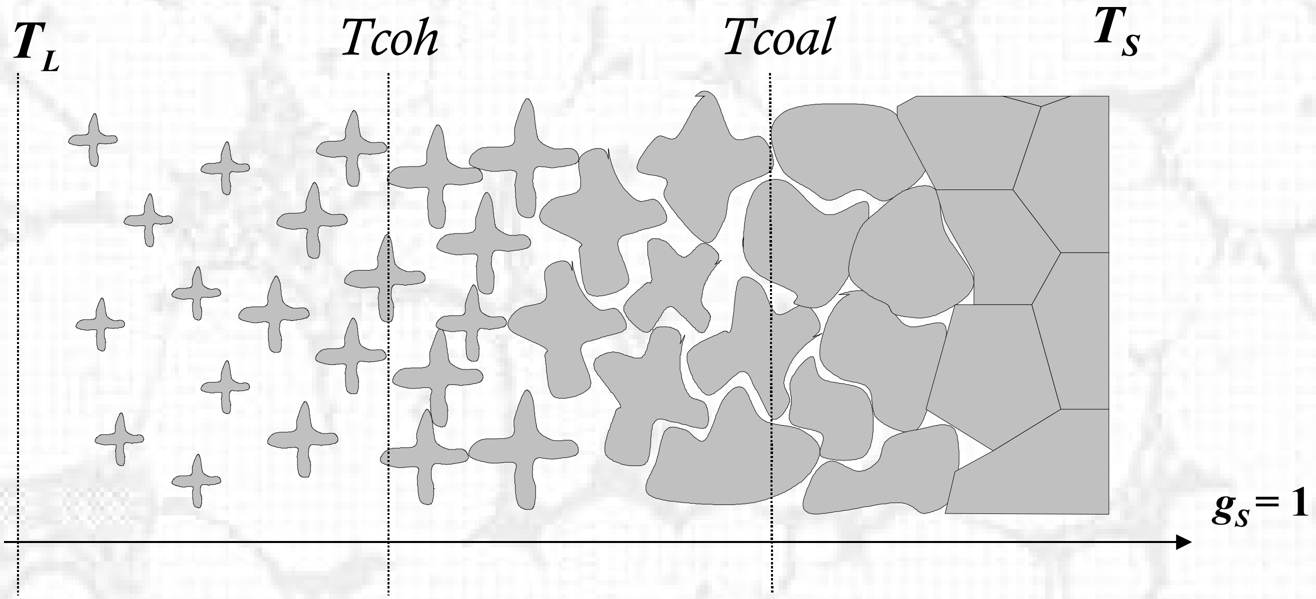

(b) **Strength development during solidification of grain refined AlSi11Mg alloys.**



- fscoh ~ 15 to 20 % for dendritic structures

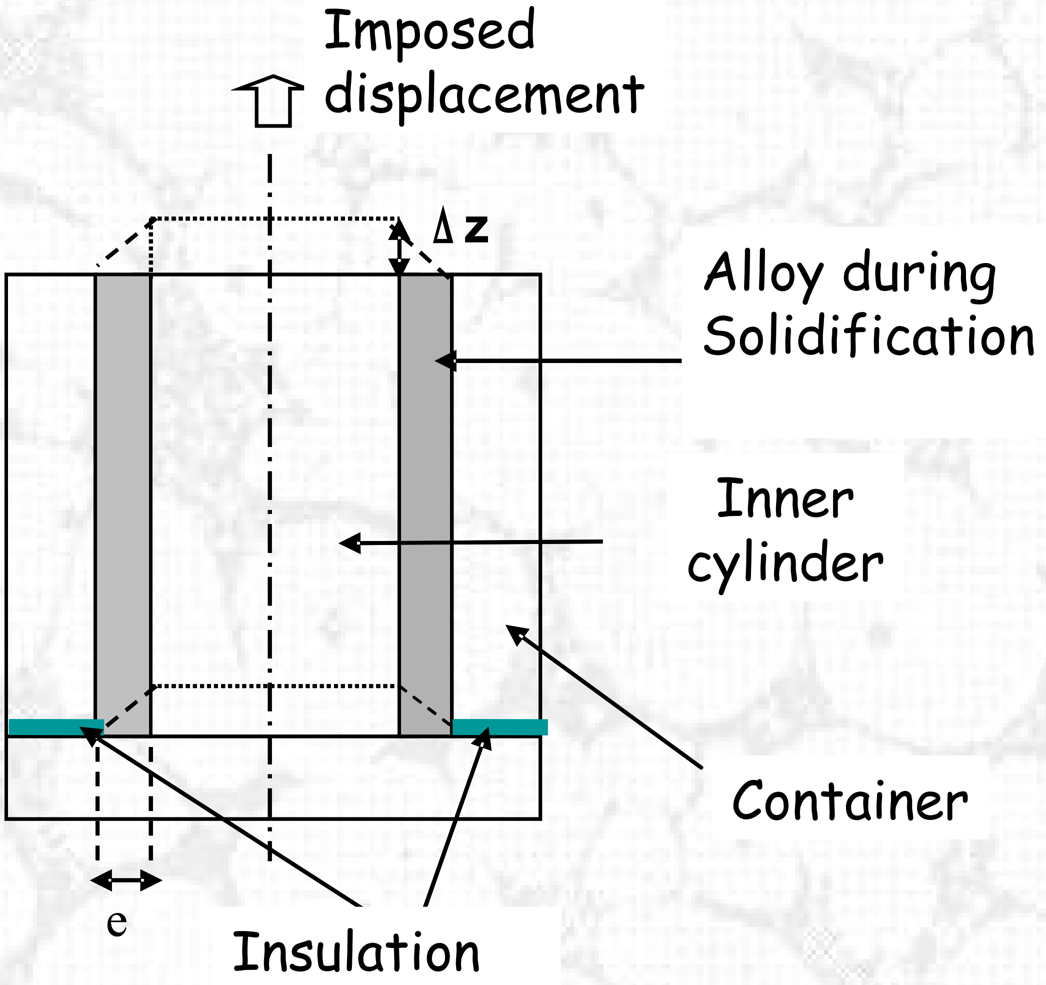
- grain refinement increases fscoh

☞ Around Tcoal : tensile stresses become important but liquid films are still present



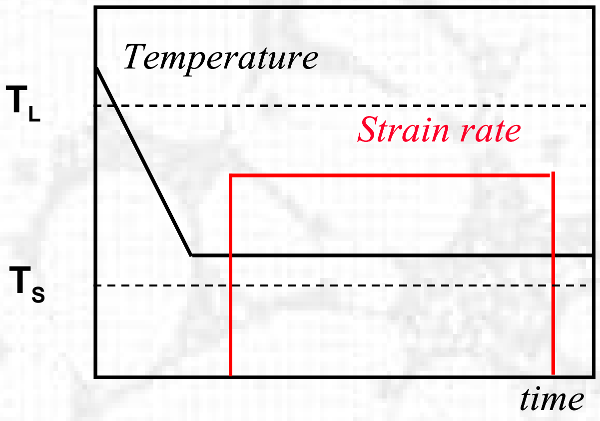
solidification

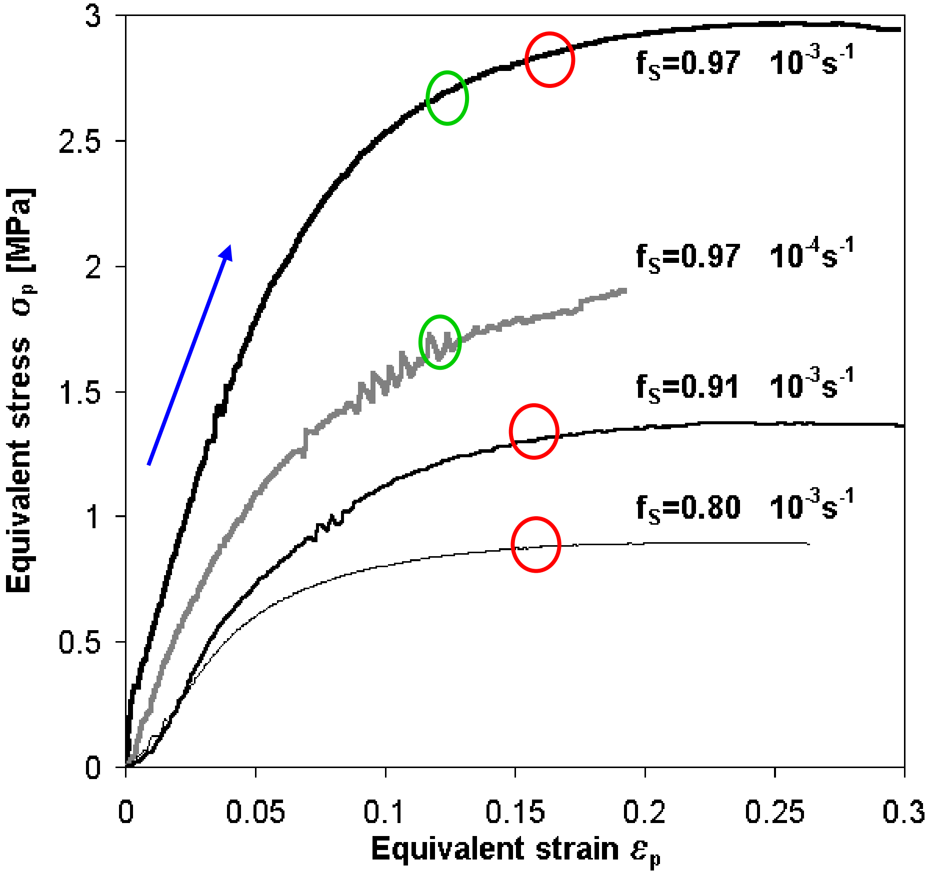
Shear deformation



Isothermal tests after partial solidification

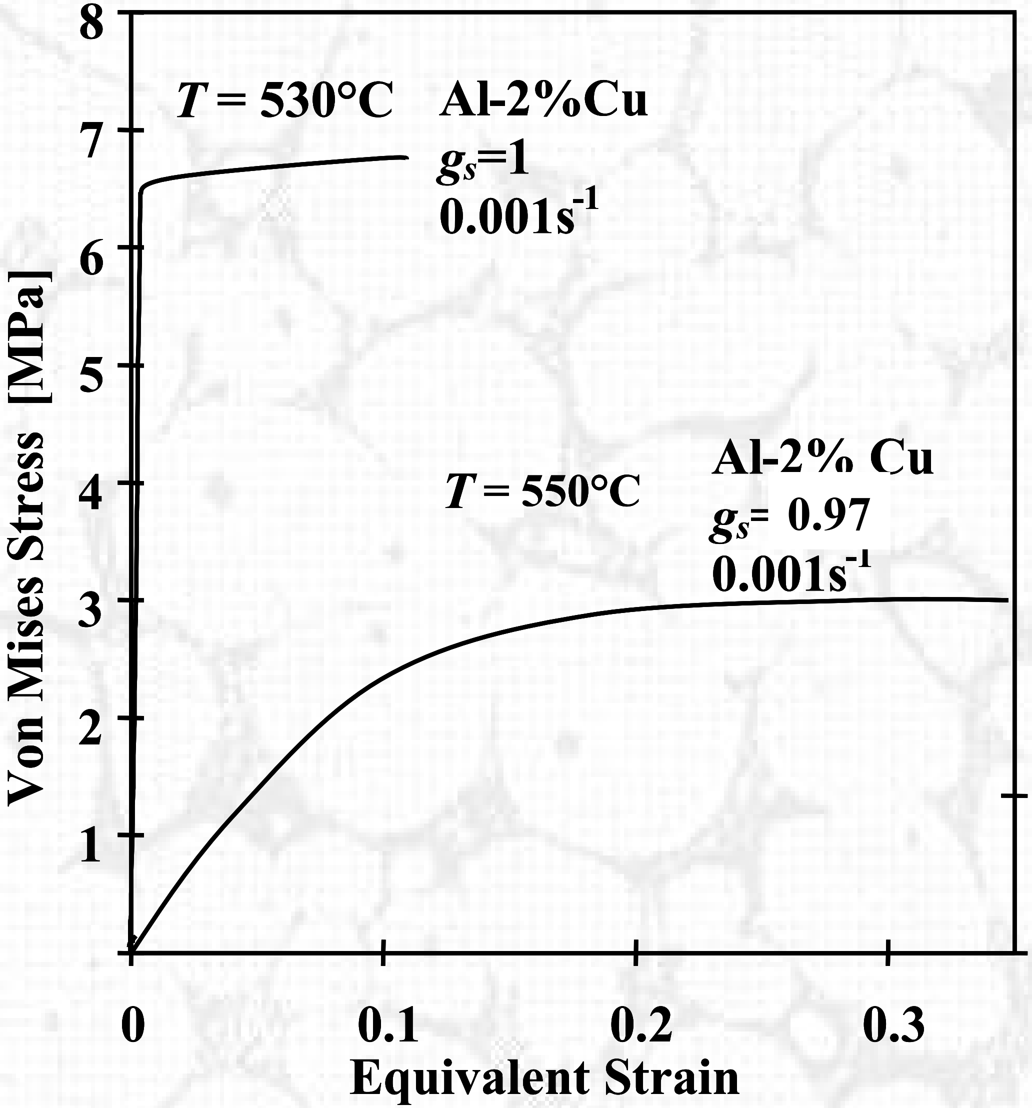
Al-2%Cu





- **Effect of solid fraction**
- **Effect of shear rate**
- **Effect of strain**

Comparison with the fully solid alloy



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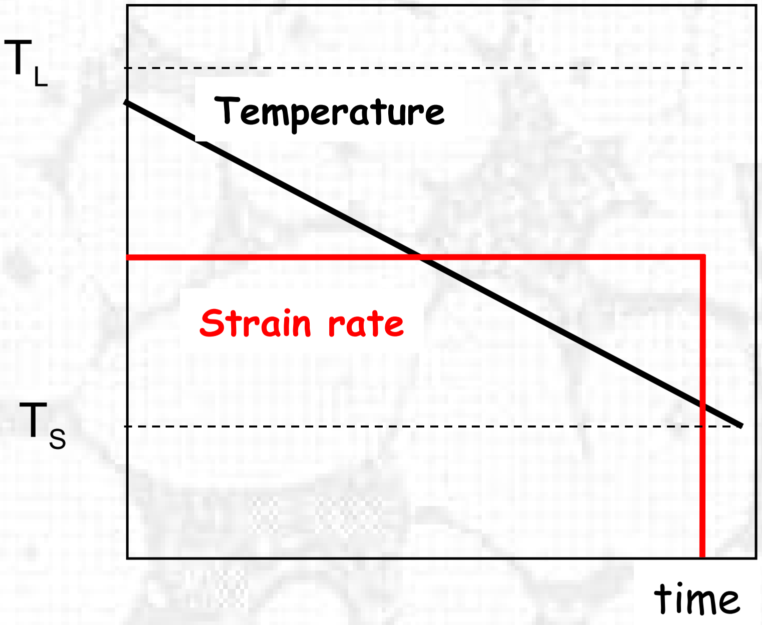
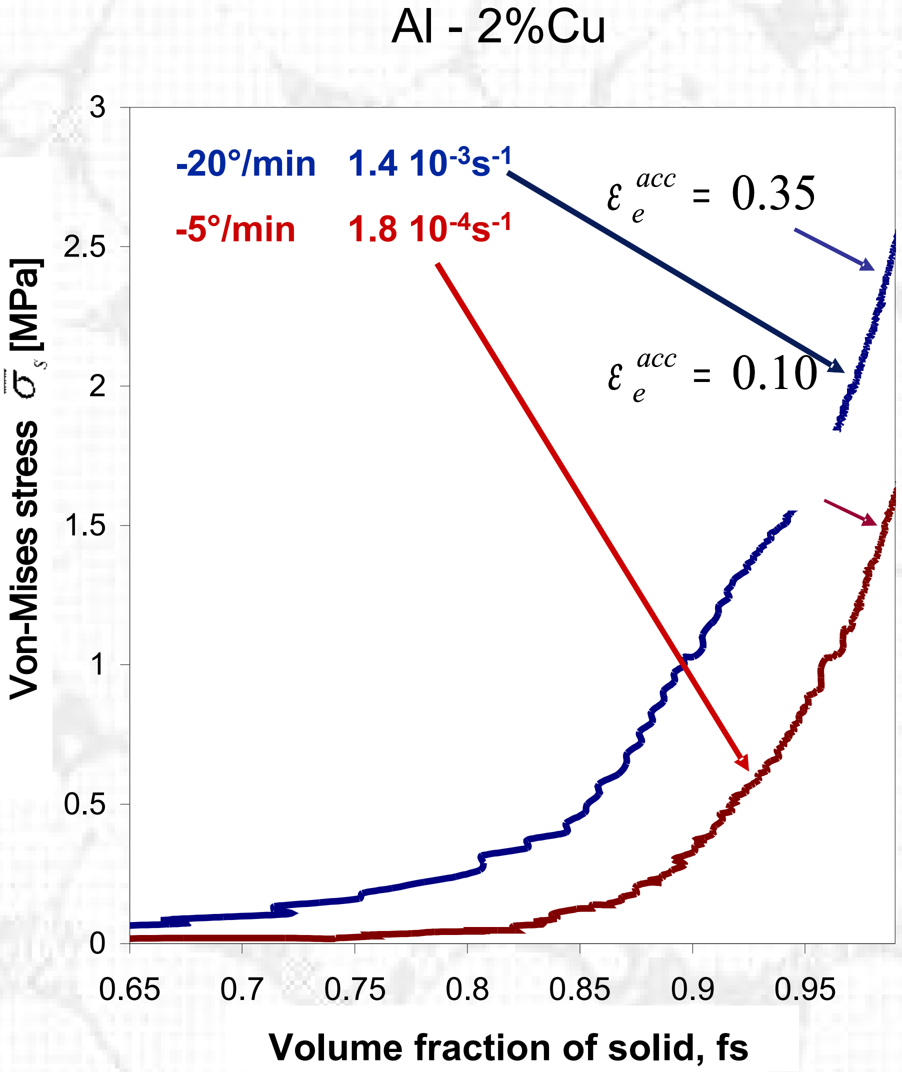
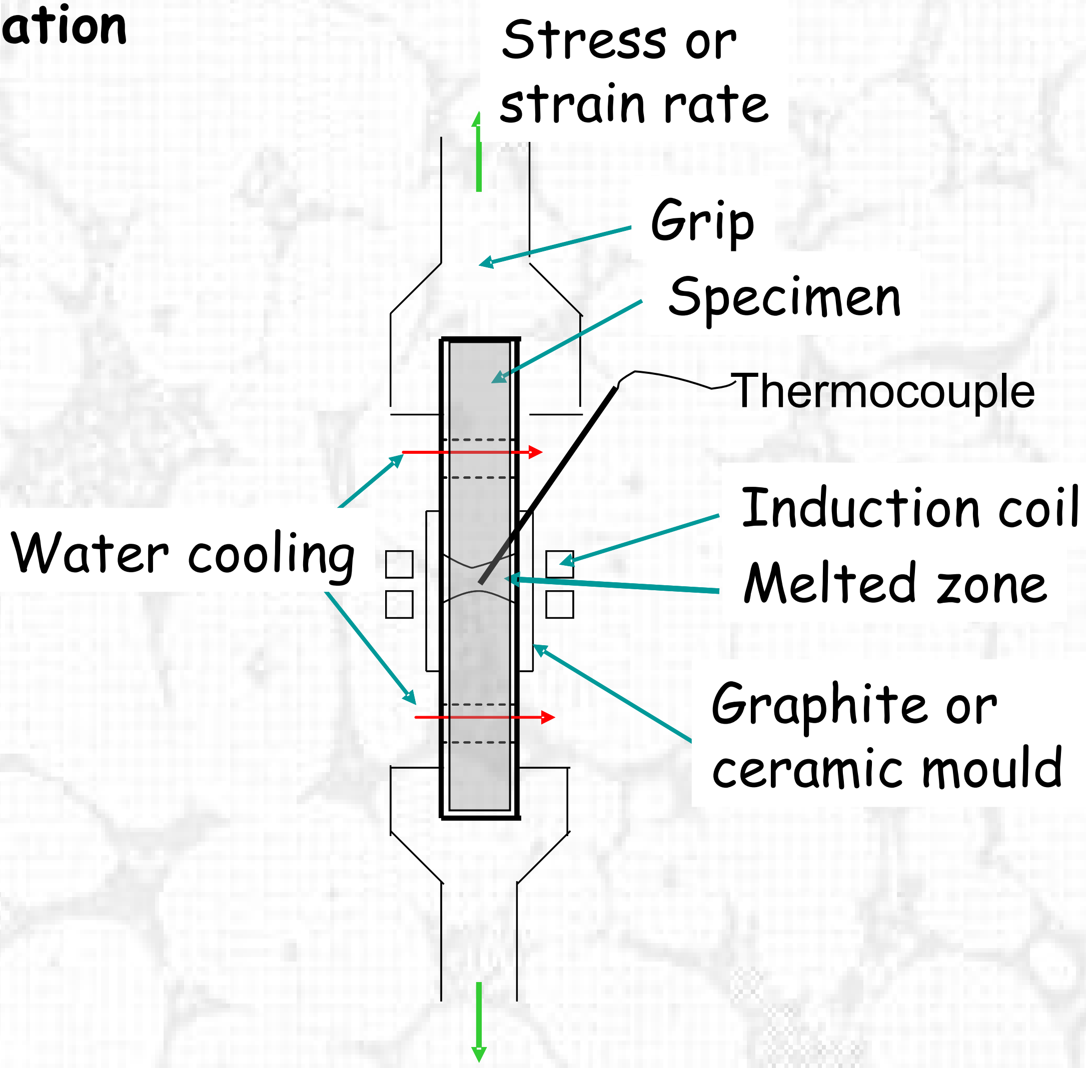
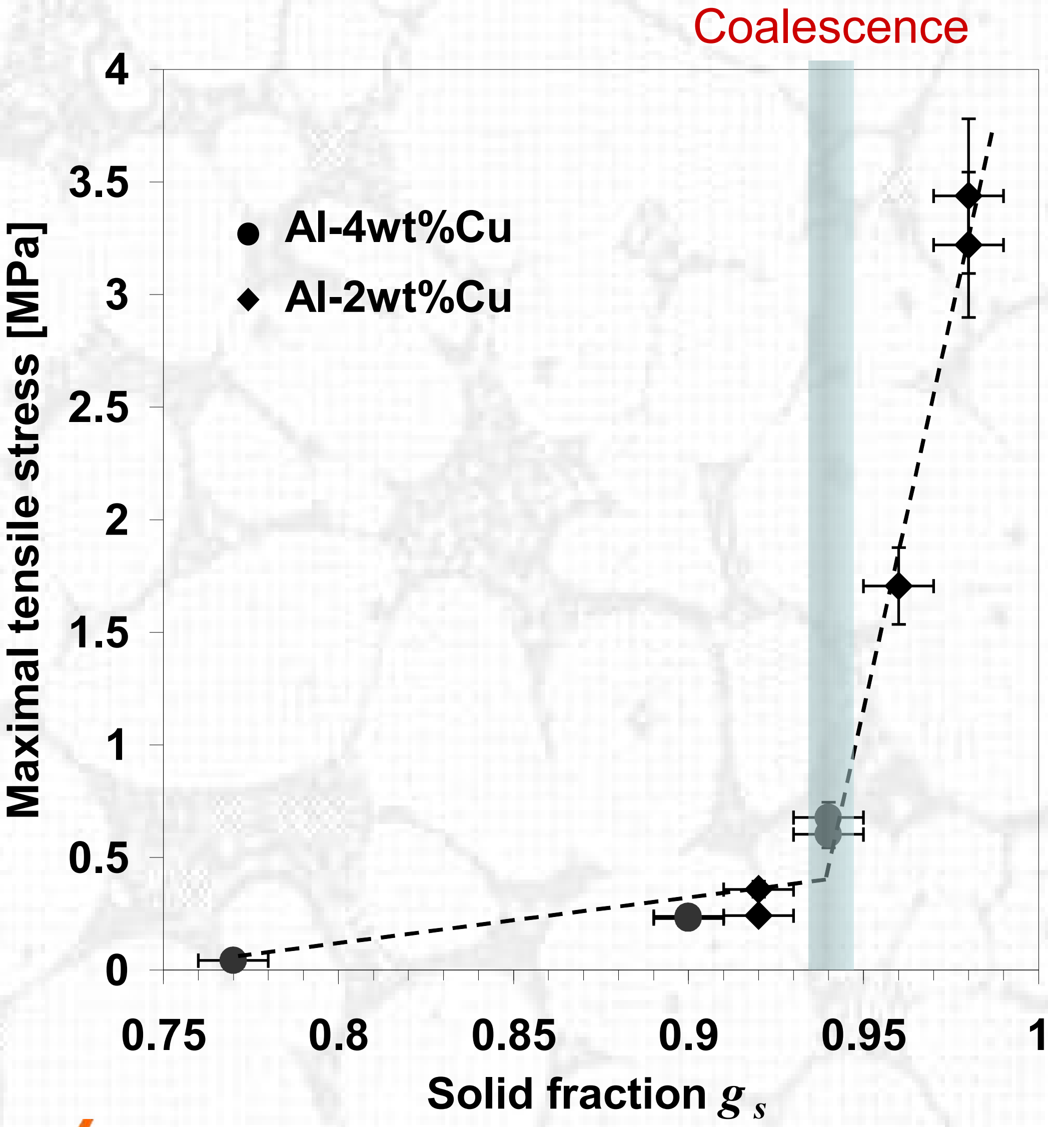
Tests during solidification



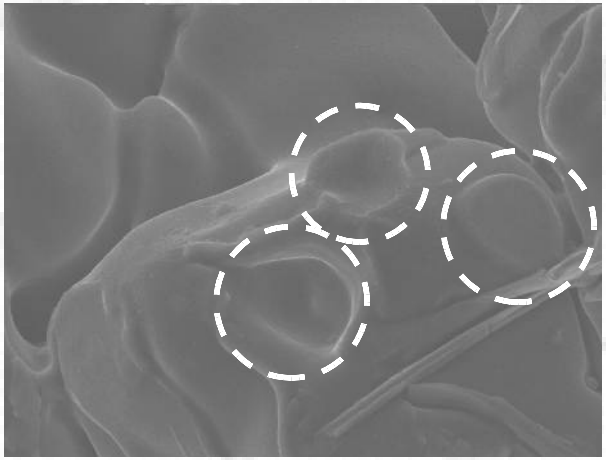


Tensile deformation





$g_s = 0.96$

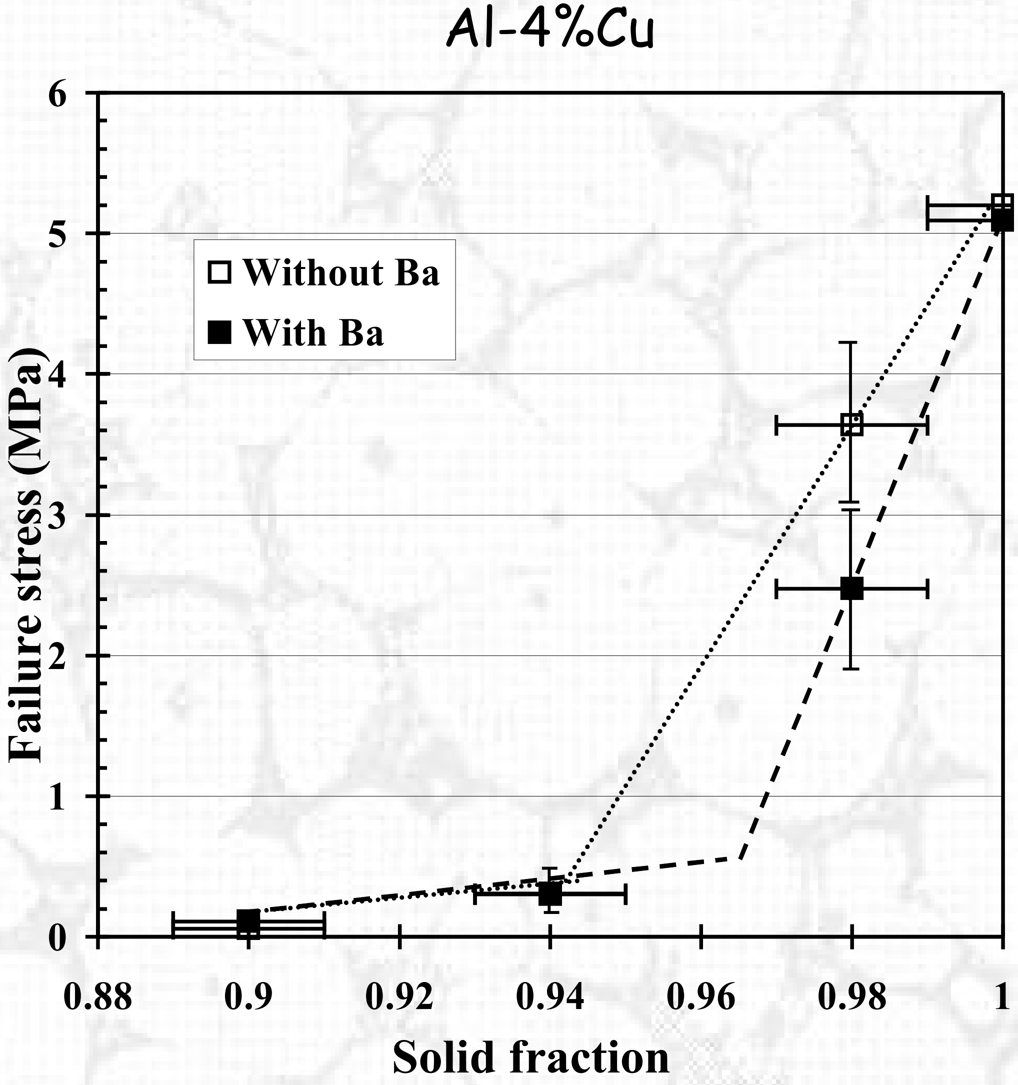


Ductile fracture of solid bridges between dendrites

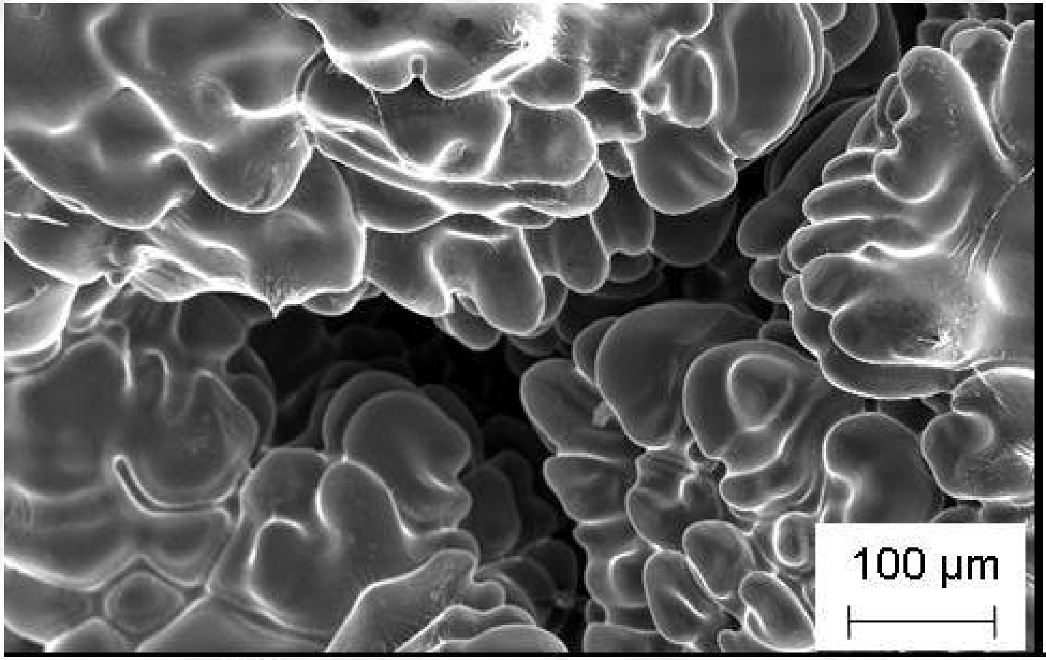
Effect of Ba addition (300 ppm)

↓

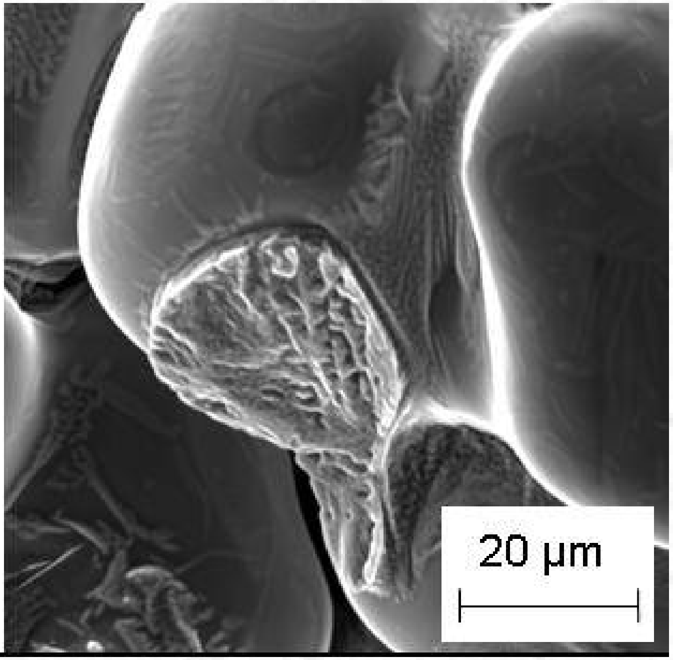
Coalescence is delayed because Ba lowers γ_{sl}



Fracture surfaces

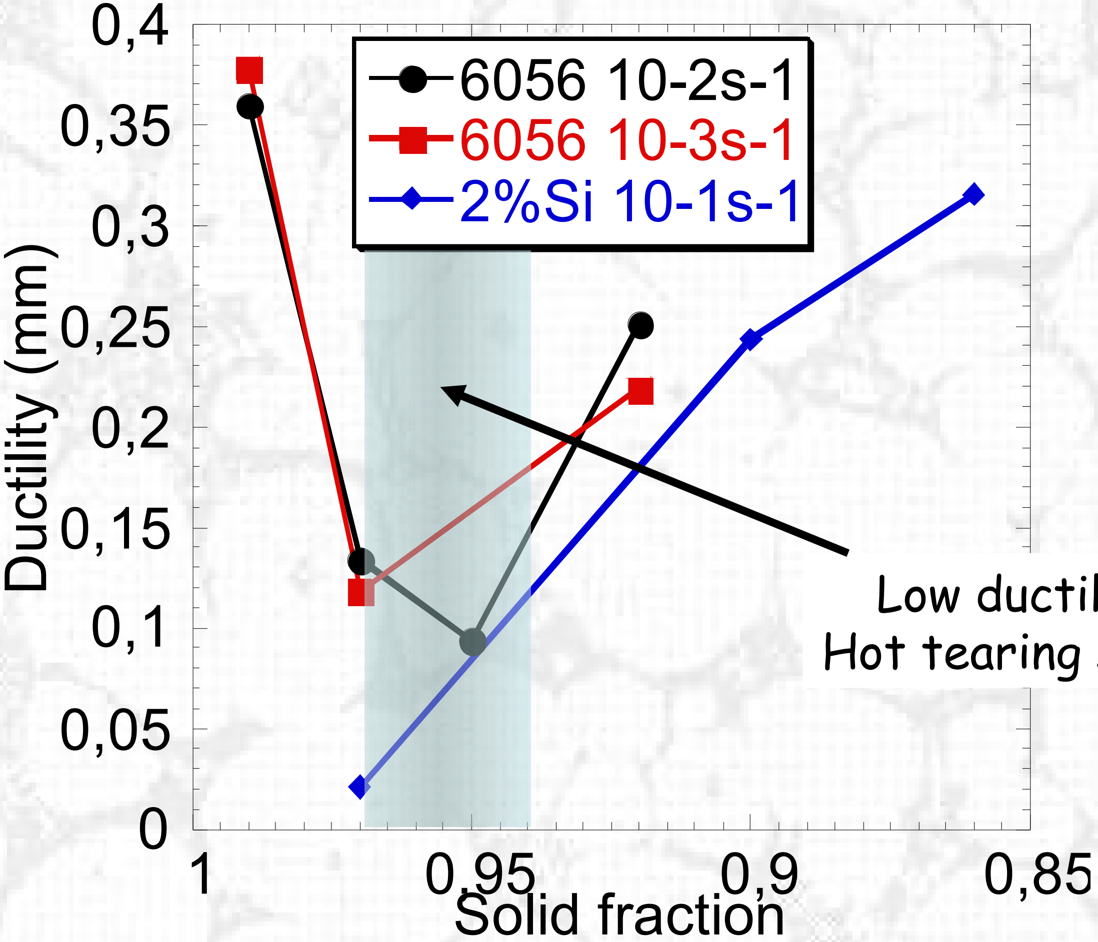


$f_s = 0.90$



$f_s = 0.98$

Other results in terms of ductility



Low ductility range =
Hot tearing susceptibility

Modelling

Suspension models

- Very low solid fractions: Einstein law

$$\eta = \eta_L\ (1 + 2.5\ fs)$$

- Higher solid fractions: interaction between solid globules

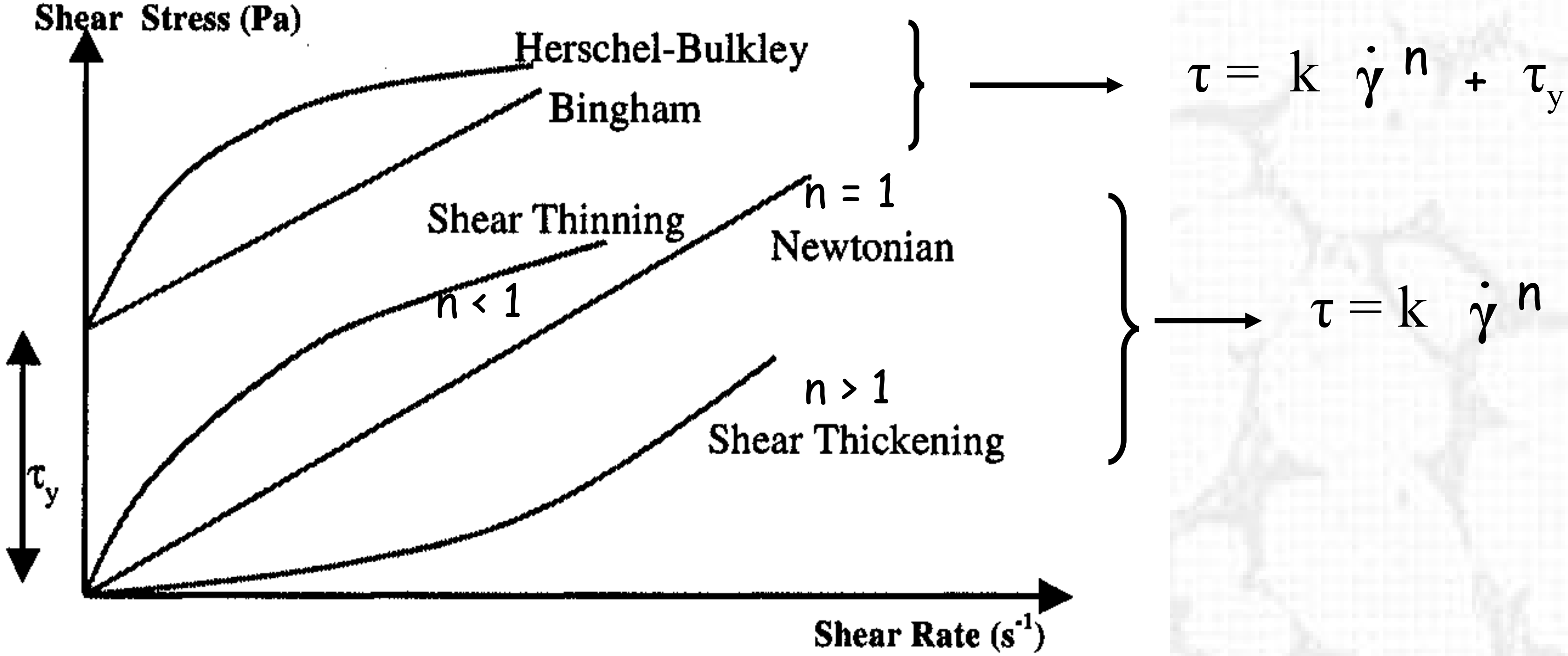
$$\eta = \eta_L\ (1 - \Phi_{eff} / \Phi_M)^{-2.5\ \Phi_M}$$

Krieger & Dougherty eq.
Trans. Soc. Rheol. 1959

Φeff = effective solid fraction taking into account entrapped liquid

Φ_M = maximum packing fraction of the solid

Influence of shear rate

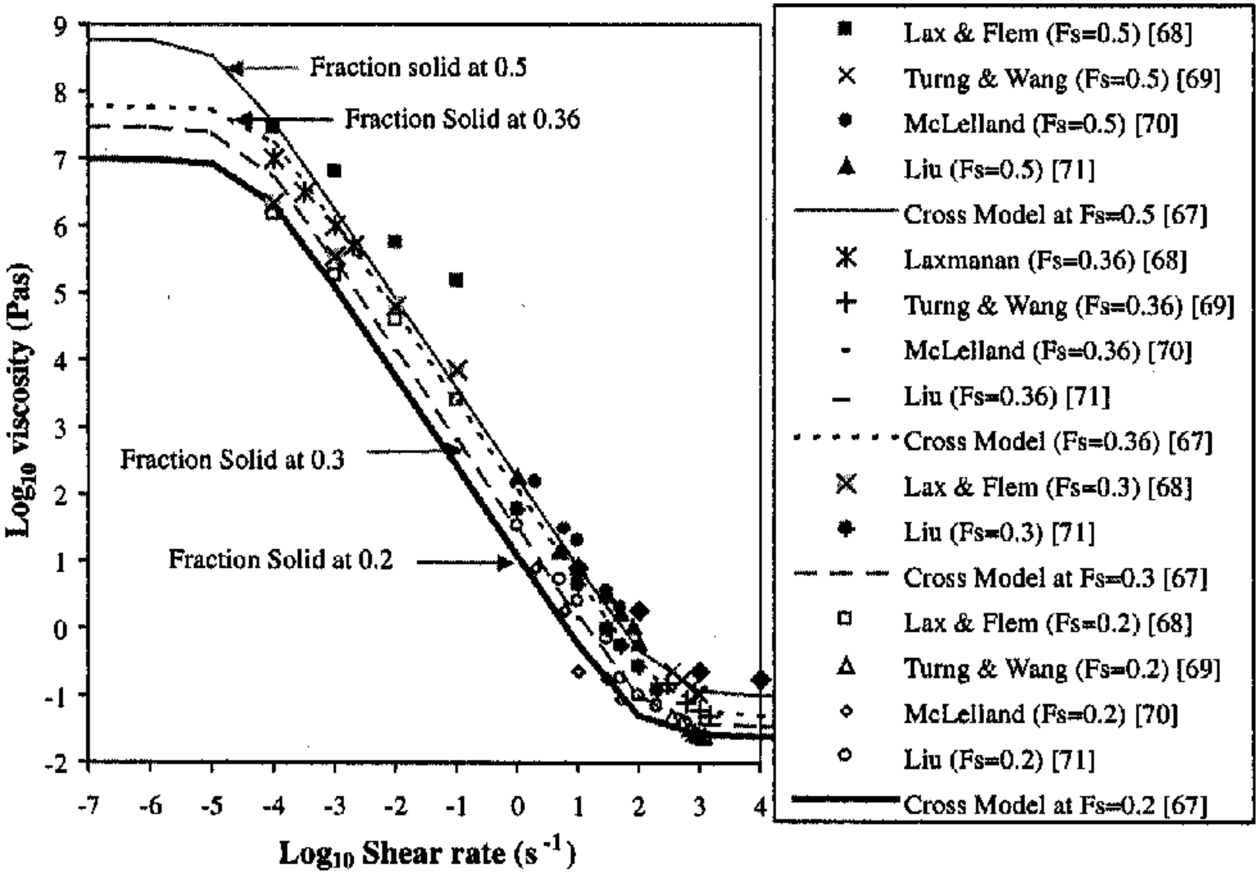


$$\tau = k\,\dot{\gamma}^{n} + \tau_y$$

$$\tau = k\,\dot{\gamma}^{n}$$

Cross model

$$\eta = \eta_\infty + \left[\frac{\eta_0 - \eta_\infty}{1 + k\dot{\gamma}^n} \right]$$



Models incorporating a structural parameter

$$\boxed{\eta = \eta\,(fs, \dot{\gamma}\ , \lambda)}$$

λ = structural parameter

→ 1 for a completely built structure

→ 0 for a completely broken down structure

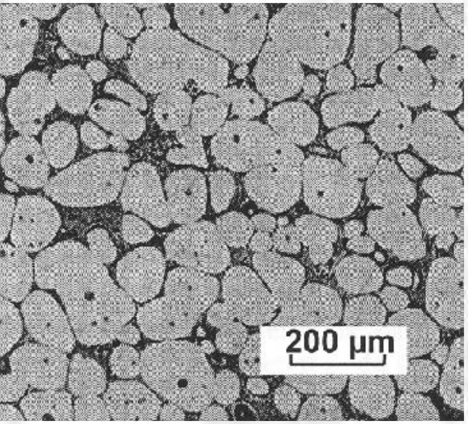
$$\boxed{\frac{d\lambda}{dt} = a(1-\lambda)^b - c\lambda\dot{\gamma}^d}$$

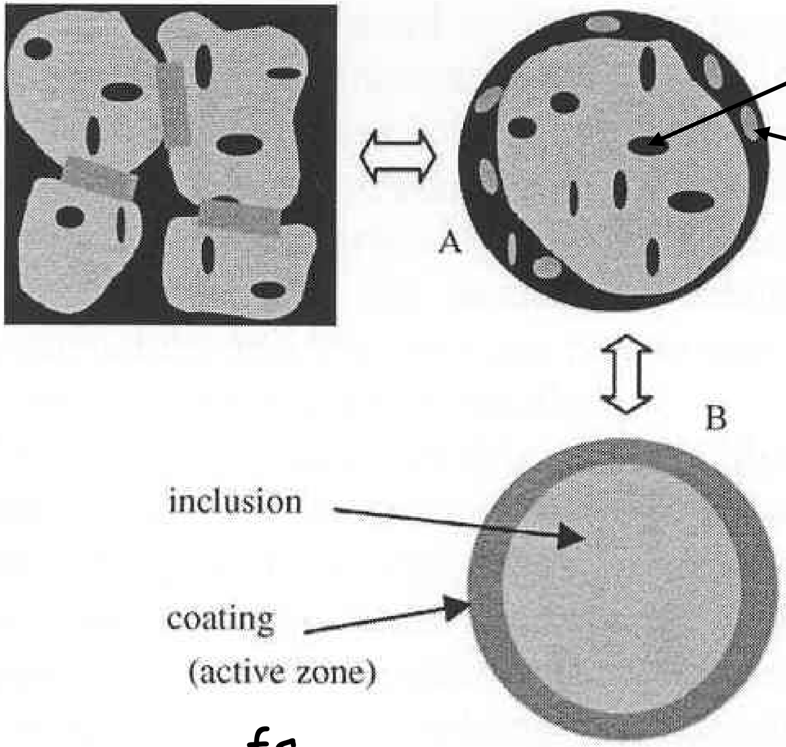
Build-up

Breakdown

Micro-macro modelling approach

**V. Favier & al.
Int. J. Forming Processes, 2004**



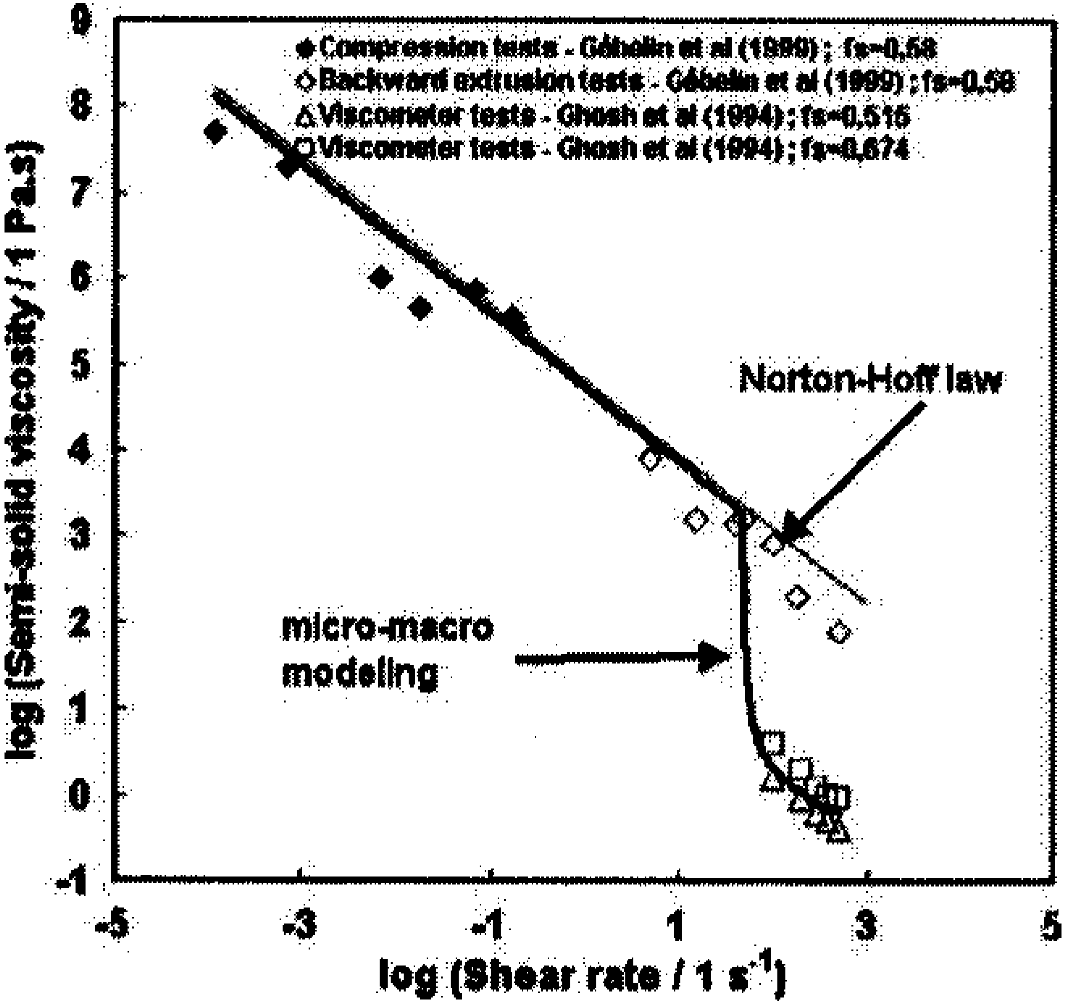


Interglobular liquid

Solid contacts

fs_A

fs_A depends on shear rate and solid fraction


◆ Compression tests - Gébelin et al (1999) ; fs=0,58
◇ Backward extrusion tests - Gébelin et al (1999) ; fs=0,58
△ Viscometer tests - Ghosh et al (1994) ; fs=0,516
□ Viscometer tests - Ghosh et al (1994) ; fs=0,674
Norton-Hoff law
micro-macro modeling
log (Semi-solid viscosity / 1 Pa.s)
log (Shear rate / 1 s-1)
9
8
7
6
5
4
3
2
1
0
-1
-5
-3
-1
1
3
5

Two-phase models

Basic assumptions :

- partition of stress into two parts:
  - → deviatoric part of the macroscopic stress assumed to be carried exclusively by the skeleton of solid grains
  - → hydrostatic part, which is partially carried by the solid and partially by the liquid phase.

$$\mathbf{S}_{EFF} = \mathbf{S} - \delta p_L \mathbf{1}$$

or

$$\mathbf{S}_{EFF} = \mathbf{S} - (1 - f_S) p_L \mathbf{1}$$

- solid skeleton considered as a porous medium

- liquid flow described by Darcy's law

Solid skeleton

$$\mathbf{D}^{\mathrm{p}} = \frac{\partial \Phi}{\partial \Sigma_{\mathrm{EFF}}} \qquad \Phi = \hat{\Phi}(\sigma_e, \sigma_h) \qquad \begin{cases} \sigma_e = \sqrt{3/2\, \Sigma'_{\mathrm{EFF}} \cdot \Sigma'_{\mathrm{EFF}}} \\ \Sigma'_{\mathrm{EFF}} = \Sigma_{\mathrm{EFF}} - \sigma_h \mathbf{1} \\ \sigma_h = (\mathrm{tr}\Sigma_{\mathrm{EFF}})/3 \end{cases}$$

For the dense solid

$$\Phi_d = \frac{\dot{\varepsilon}_0 \sigma_0}{n+1} \left(\frac{\sigma_e}{\sigma_0} \right)^{n+1}$$

For the porous solid

$$\Phi = \frac{\dot{\varepsilon}_0}{(n+1)\sigma_0^n} F(\sigma_e, \sigma_h, f_L, c, n, \ldots)^{n+1}$$

$$\Sigma_{EFF} \bullet \mathbf{D}^{\mathrm{p}} = (1 - f_L)\sigma_{eq}\dot{\varepsilon}_{eq} \quad \text{where } \dot{\varepsilon}_{eq} = \dot{\varepsilon}_0 (\sigma_{eq}/\sigma_0)^n$$

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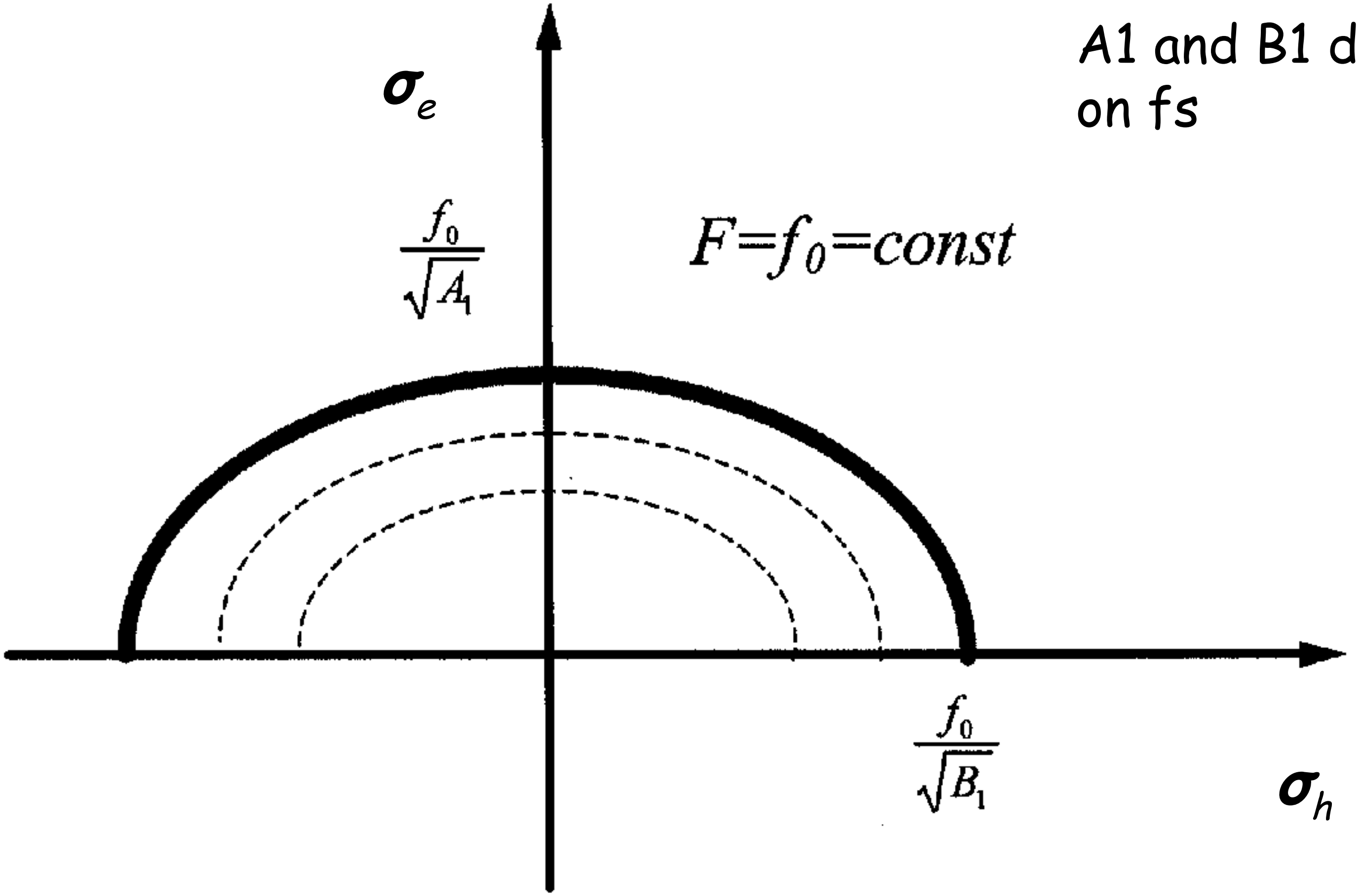
Centered ellipse

$$F(\sigma_e,\sigma_h,f_L,n)=\sqrt{A_1\sigma_e^{\,2}+B_1\sigma_h^2}$$

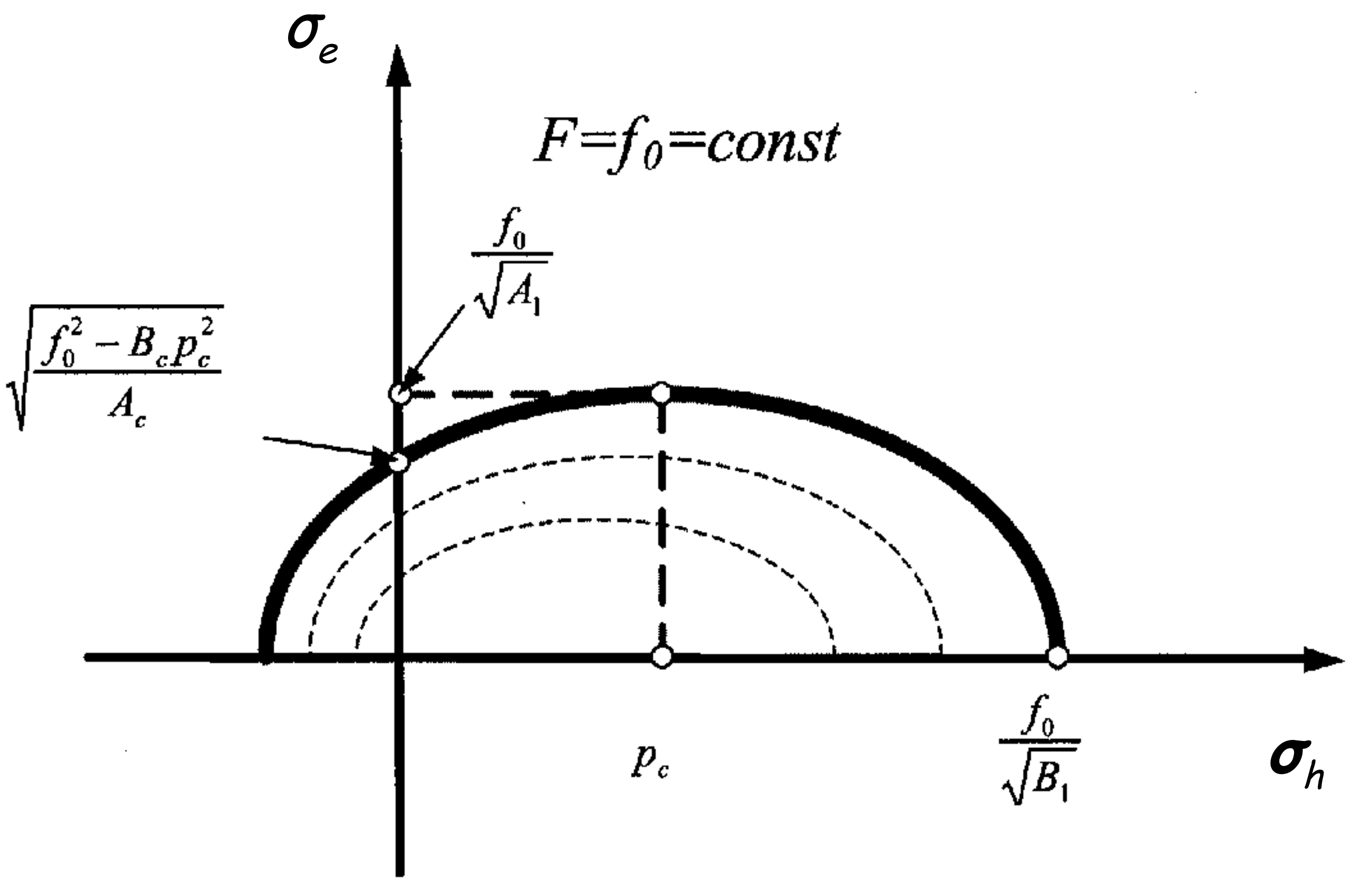


A1 and B1 depend on fs

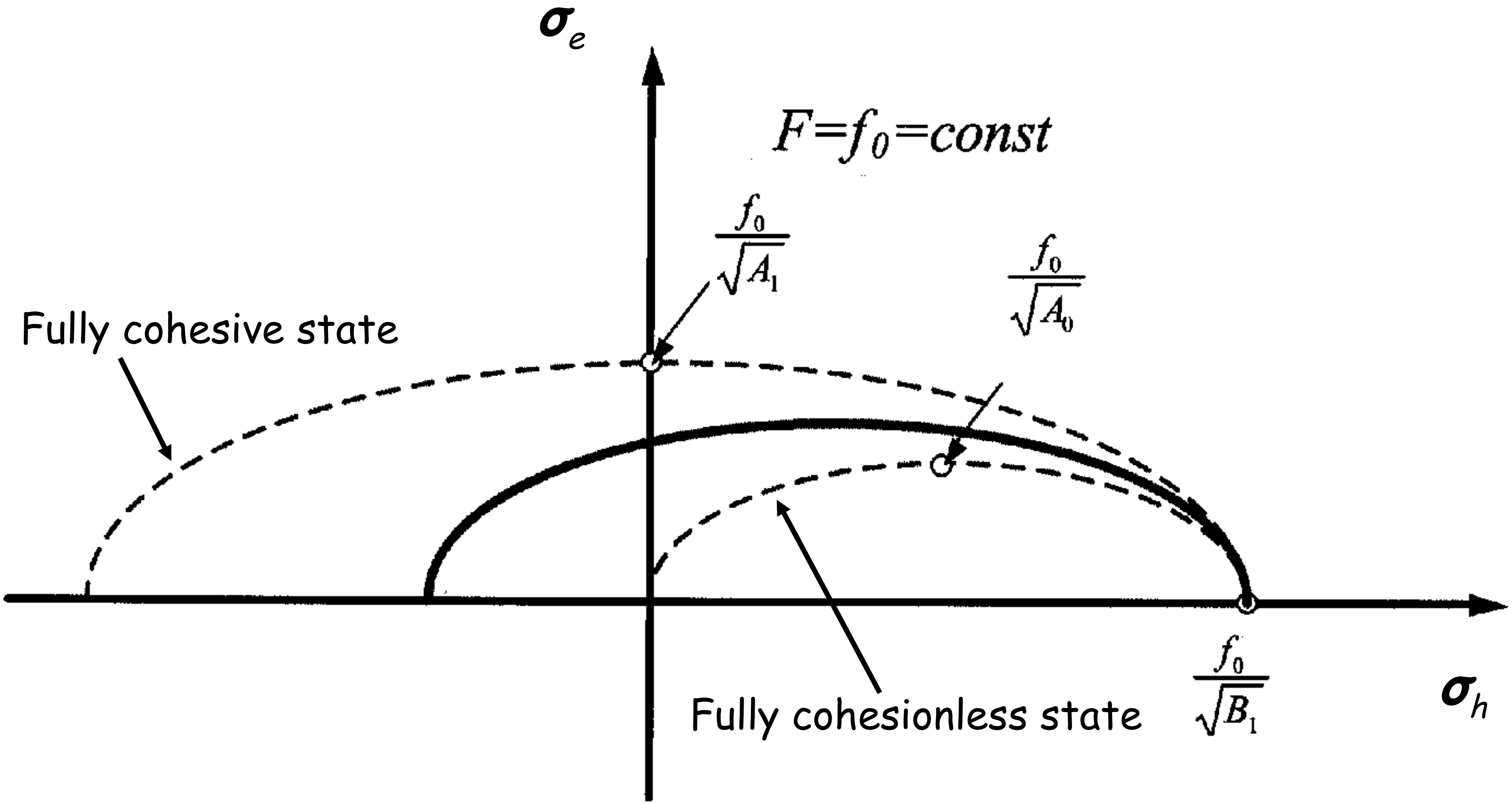
Non centered ellipse

$$F(\sigma_e,\sigma_h,f_L)=\sqrt{A_C\sigma_e^2+B_C(\sigma_h+p_C)^2}$$

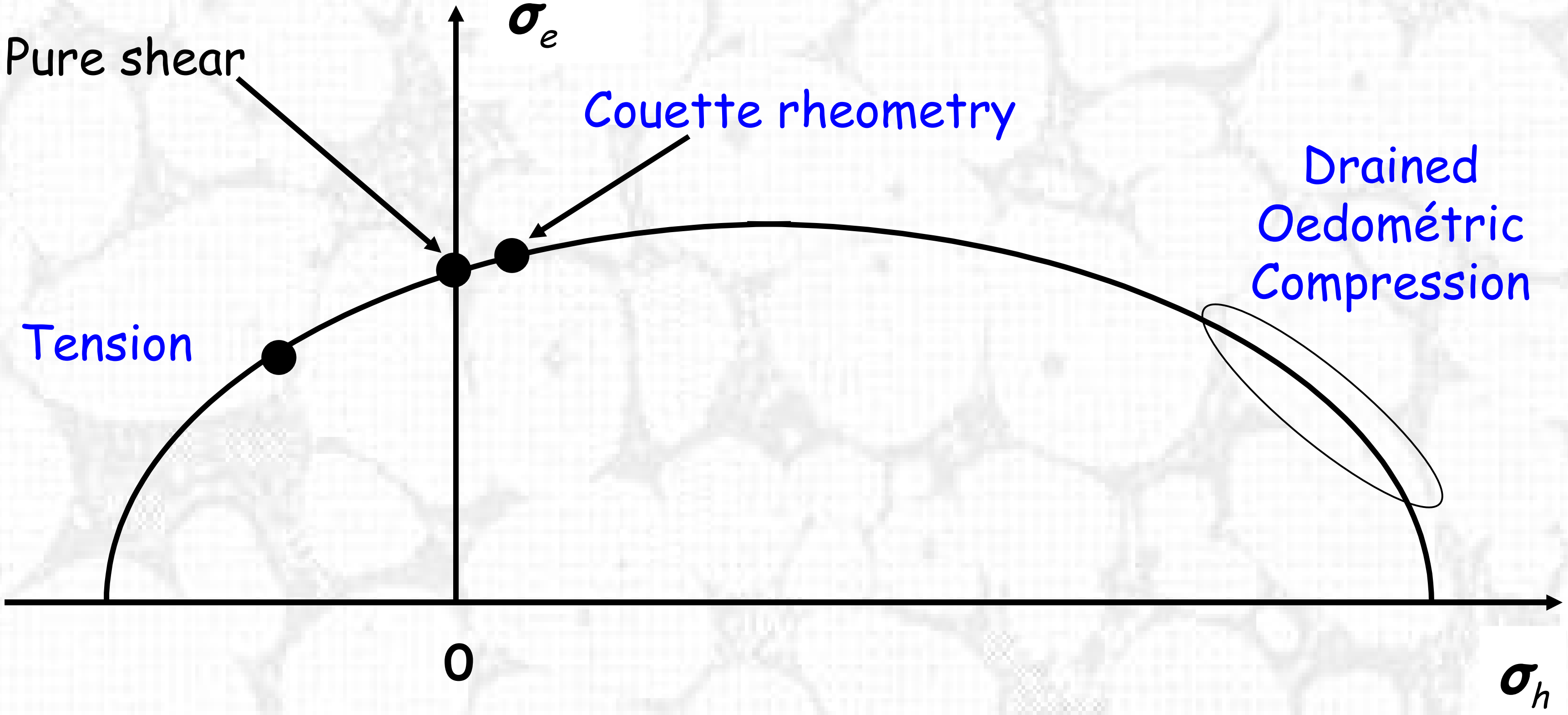
Tension $\neq$ compression



Thixotropic non centered ellipse



Experiments to be carried for identification



Liquid flow

Darcy's law

$$f_L \mathbf{v}_L = -\frac{\mathbf{K}}{\mu} \cdot (\nabla p_L - \rho_L g)$$

K: permeability tensor (anisotropic in the general case)

Isotropic permeability:

$$K = \frac{d_m^2}{180} \frac{f_L^3}{(1 - f_L^2)}$$

Carman-Kozeny

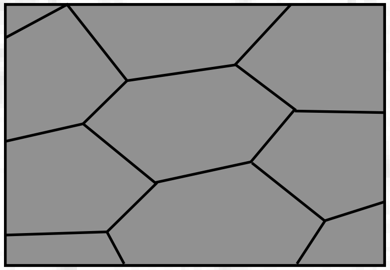
d_m = average grain size for equiaxed microstructures

Modelling at very high solid fractions

☞Between Tcoal and Ts: liquid flow no longer possible (liquid pockets)

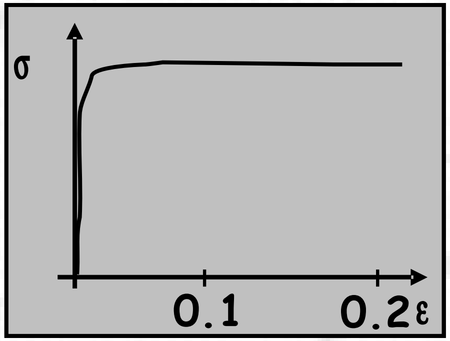
⇨ viscoplastic deformation of a porous solid saturated with liquid

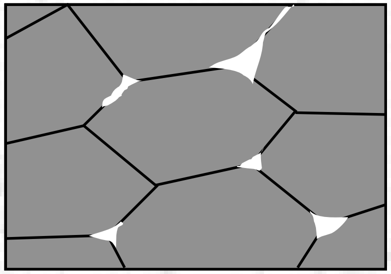
However, presence of liquid induces a partial cohesion of the solid which is evolving during deformation



$g_s=1$

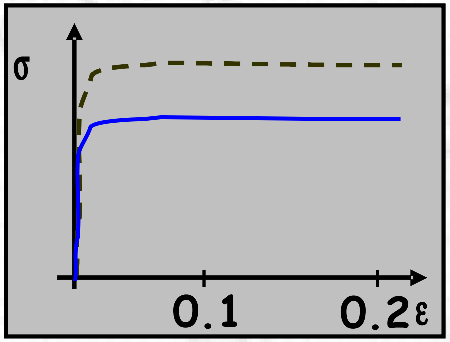
Solid alloy at high temperature

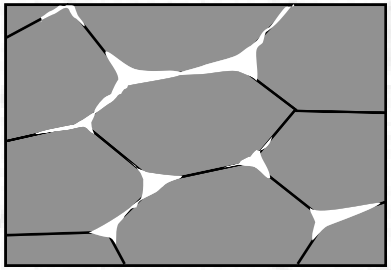




$g_s<1$

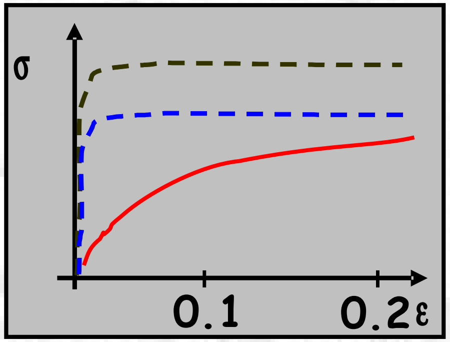
Porous alloy saturated with liquid





$g_s<1, C<1$

Alloy with Partial cohesion



$$\boxed{\dot{\boldsymbol{\varepsilon}}_s^{p} = \frac{\dot{\varepsilon}_0}{\left(Cs_0\right)^n}\left\{-\frac{A_2}{3}\bar{P}_s\mathbf{1} + \frac{3}{2}A_3\mathbf{S}_s\right\}\left\{A_2\bar{P}_s^{2} + A_3\bar{\sigma}_s^{2}\right\}^{\frac{n-1}{2}}}$$

$$\frac{\mathrm{d}C}{\mathrm{d}t} = \alpha\left(g_s, X\right)\left(1 - \frac{C}{C^*\left(g_s, X\right)}\right)\dot{\varepsilon}_e$$

C = solid cohesion

X = stress triaxiality

C^* = cohesion at saturation

$\alpha(g_s, X)$ and $C^*(g_s, X)$ must be determined experimentally

g_s evolves with solidification and strain

Example: Al-2%Cu during shear

$$\alpha\left(g_s, X = 0\right) = \alpha_0 + \alpha_1 \frac{g_s^{\frac{1}{3}}}{1 - g_s^{\frac{1}{3}}}$$

$$C^*\left(g_s, X = 0\right) = 1 - \left(1 - g_s\right)^p$$

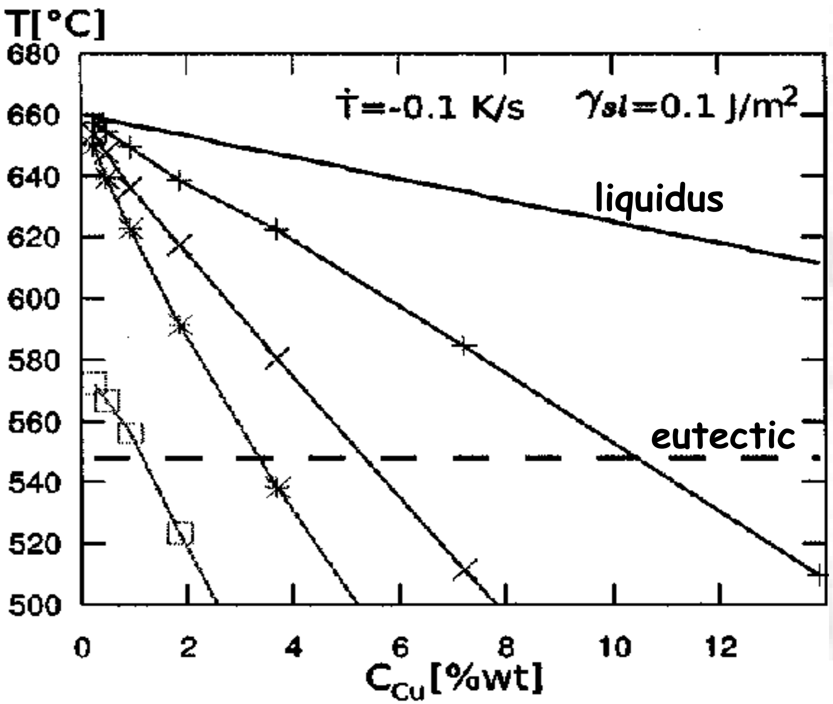
$\alpha_0 = 4.45$ $\alpha_1 = 1.07.10^{-2}$ $p = 0.11$

Another approach = granular model for equiaxed mushy zones (2D)

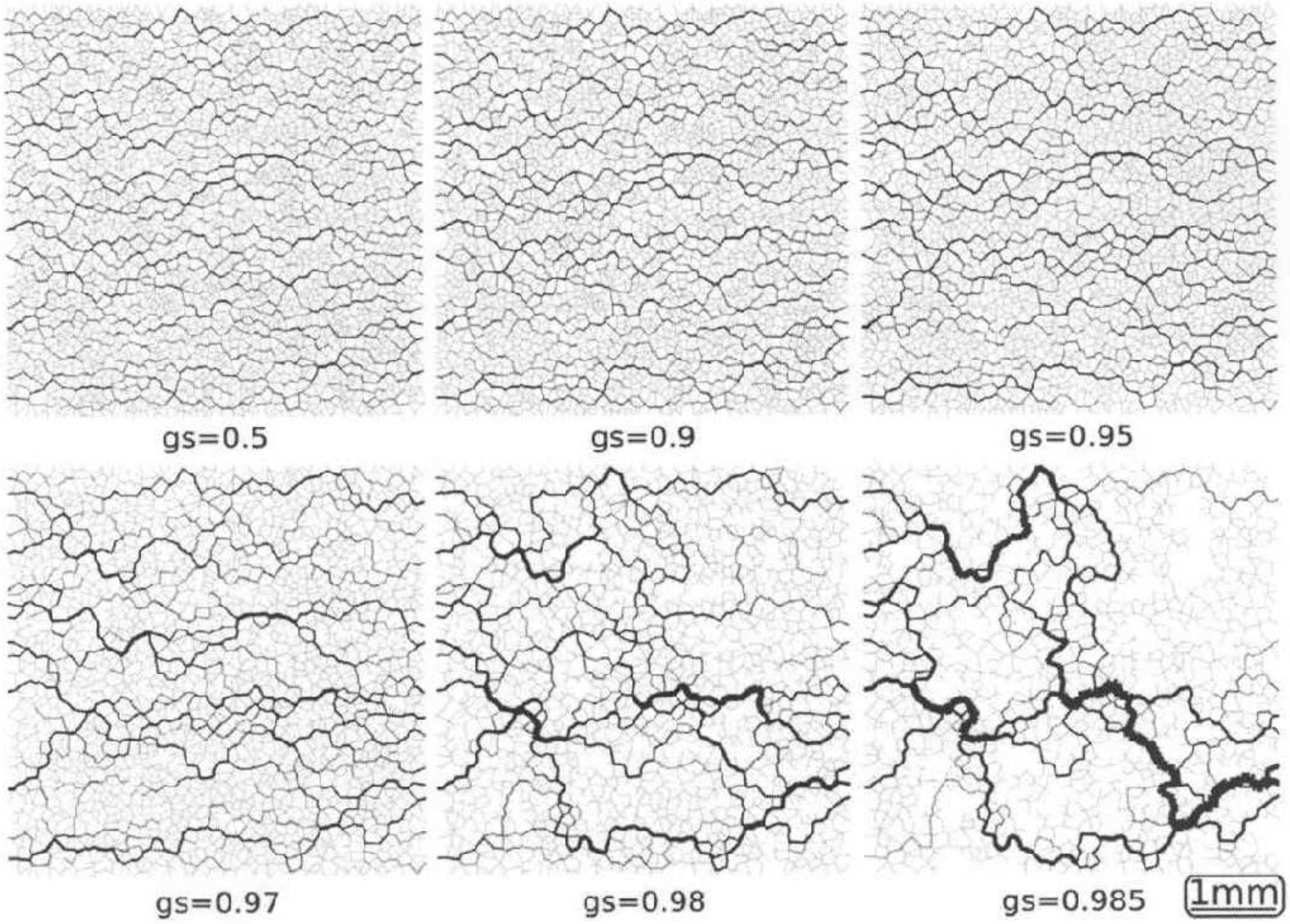
Vernede et al. Acta Mat. 2006

→ Prediction of the various solid transitions

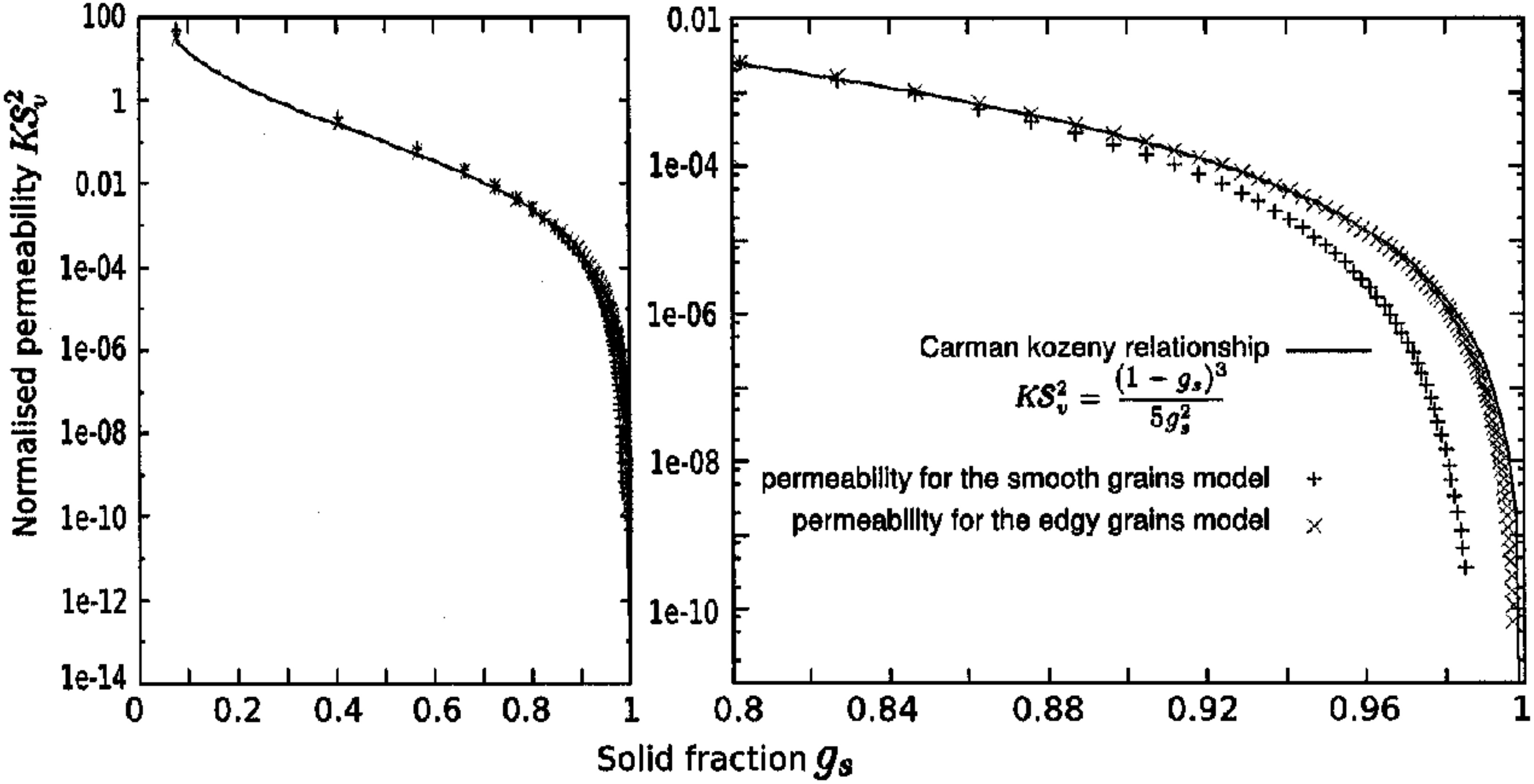
Grain contact	+
Liquid isolation	×
Percolation by contact	✳
Percolation by bridging	☐



→ Analysis of liquid flow ⇨ permeability



Permeability



Conclusion

- Rheological behaviour of partial melts is complex
  - effect of strain rate
  - effect of solid fraction (suspension, f_{coh}, f_{coal})
  - effect of time and strain
  - effect of stress state

- Modelling is also necessarily complex

- Identification of models is difficult
  - Partial remelting/solidification
  - High temperature
  - Several types of tests

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Thank you for your attention

Questions?

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