# Microsegregation

## Partition coeff.

k=Cs/CL at constant temp. and press.

- if mL < 0 k < 1
- if mL > 0 k > 1



Fig. 4.1 (a) Portion of a phase diagram in which the distribution coefficient  $k_0 < 1$ . (b) Portion of a phase diagram in which the distribution coefficient  $k_0 > 1$ .

#### Introduction

Microsegregation is a distribution of solute elements in dendritic or cellular cast structures and can affect mechanical and chemical properties and phase transformations in alloys, such as homogenization and solution heat treatments, pearlite-ferrite banding structure, martensitic transformation and corrosion.



Figure 1 Dimensional form of microsegregation volume element from the centre of side arm to the centre of the liquid pool



Magnification: 800xAlloy: 356.00 Primary dendritic microsegregation: primary  $\alpha$ -Al, orangegreen fields; interdendritic eutectic  $\alpha$ -Al + Si, blue fields Time of attack: 5 s

Fig. 37 Phase constituent identification in alloy 356.0 with reagent 13m (Table 4). Time of attack: 5 s. Orange-green fields are primary dendritic microsegregation regions with  $\alpha$ -Al (solid solution). Blue fields are interdendritic eutectic ( $\alpha$ -Al + Si). 800×. Compare with Table 18. (Małgorzata Warmuzek, Foundry Research Institute, Kraków, Poland)



Fig. 7.3 Isoconcentration surfaces in a columnar dendrite grown in a low-alloy steel melt (Kattamis and Flemings<sup>9</sup>).



Fig. 16 Copper and magnesium microsegregation in a direct-chill semicontinuous cast (610  $\times$  1372 mm, or 24  $\times$  54 in.) 2124 alloy ingot. (a) Dendrite cells at midthickness location in ingot and enrichment of copper and magnesium at the cell boundaries. When observed in conjunction with the electron probe microanalysis, the gradual increase in solute concentration across the dendrite cell is readily apparent. (b) Microprobe traverse across dendrites



Fig. 17 (a) The aluminum-rich end of the aluminum-copper phase diagram. (b) Solid composition ( $C_5$ ) versus fraction solid ( $f_s$ ) for Al-4.5Cu. L, liquid;  $\alpha$ , aluminum solid solution. (Ref 3)



Fig. 4.11 Concentration-distance profiles for a bar solidified under conditions of complete solute mixing in the liquid.

# Models for Microsegregation

- 1 no mixing in liquid
- 2. partial mixing in liquid
- 3. complete mixing in liquid
- 4. complete mixing in liquid
- 5. complete mixing in liquid
- 6. complete mixing in liquid

no diffusion in solid no coarsening no diffusion in solid no coarsening no diffusion in solid no coarsening diffusion in solid no coarsening diffusion in solid coarsening complete diffusion in solid

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# (6 Model). Eq solidification (Level Rule)



#### Lever Rule

The simplest case to which a mass balance can be applied is the case of equilibrium solidification (no concentration gradient in the solid or liquid). This can be expressed by the relations:

$$D_{L} \gg D_{S} \gg LV$$
 [A11.2]

Since L is the length of the solidifying system (figure All.1), equation All.2 states that the diffusion boundary layer,  $\delta_c = 2D/V$ , is much larger than the maximum distance, L, over which either solid or liquid state diffusion can occur. Similarly, it can be supposed that, if the growth rate, V, of the solid is constant and equal to  $L/t_f$ , the length of the specimen must be less than the characteristic diffusion length:

$$L \ll \sqrt{D_{s} t_{f}}$$
 [A11.3]

Under these conditions:

$$\frac{\partial C}{\partial z'} = \frac{\partial C}{\partial z'} = 0$$

Taking the differential form of equation All.1,  $C_s = kC_l$ ,  $dC_s = kdC_l$ ,  $f_s = 1-f_l$ , and  $df_s = -df_l$  can be substituted and integrations performed:

$$\int_{c_0}^{c_1} \frac{dC_{\iota}}{pC_{\iota}} = \int_{0}^{f_s} \frac{df_s}{1 - f_s p}$$

$$\frac{C_{l}}{C_{0}} = \frac{1}{1 - pf_{s}} \qquad p=1-k$$



Fraction solidified

Fig. 4.13 Solute distributions in a solid bar frozen from liquid of initial concentration C<sub>0</sub>, for : (a) equilibrium freezing; (b) solute mixing in the liquid by diffusion only; (c) complete solute mixing in the liquid; (d) partial solute mixing in the liquid.



Figure 6.4: SEGREGATION CURVES 1N THE PRESENCE OF BACK DIFFUSION. The composition of the liquid (assumed to be homogeneous as in figure 6.3) increases at the end of the specimen. Under lever-rule conditions, the increase is from  $C_0$  to  $C_0/k$ , while the Scheil equation predicts an increase from  $C_0$  to infinity. All of the intermediate cases can be described by one relationship (equation 6.9) which contains a modified  $\alpha$ -parameter,  $\alpha$ ', which can take values between 0 and 0.5. Note that the curve represents the path of the interface concentration,  $C_t^*$ , as a function of  $f_s$ . The final solute distribution profile in the solid cannot be determined in this way because it changes with time when  $\alpha > 0$ . Therefore, only the end concentration ( $f_s = 1$ ) represents a measurable value.



Figure 6.5: RELATIONSHIP OF THE SEGREGATE FREEZING POINT TO THE PHASE DIAGRAM. An increasing concentration (for distribution coefficients less than unity) is associated with a decreasing liquidus temperature since the slope, m, is then less than zero. Using the curves of figure 6.4, the temperature of the liquid as a function of volume fraction solidified can be derived. The use of realistic diffusion coefficients shows that, for small systems (such as interdendritic regions - figure 6.6) interstitial C in delta- and gamma-Fe will behave according to the lever-rule. Hence, the last liquid of a binary Fe-C melt will solidify at a temperature close to the solidus while substitutional alloys, such as Al-Cu, which typically have much smaller solid-state diffusion coefficients will usually contain eutectic material in the last (interdendritic) regions to solidify even when the overall composition is less than the solubility limit at  $T_e$ .



Figure 6.7: MICROSEGREGATION. The equilibrium melting range,  $\Delta T_0$  does not, except for the lever rule case, correspond to the range,  $\Delta T'$ , over which the mushy zone develops. The dendrite tips need a certain undercooling which is determined by the stability of the tip. The dendrite roots will usually have much higher concentrations than  $C_0/k$ , due to non-equilibrium solidification. This often leads to interdendritic precipitation of eutectic phases of volume fraction,  $f_e$ , even if the composition is not on the eutectic tie-line. In the columnar zone of a casting, as shown here, the volume fraction of solid,  $f_s$ , will follow an S-shaped curve like that in the uppermost diagram.

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## 1 model



I = 4.12 The effect of the mixing conditions on the nature of the solute layer at the interface: (a) no mixing, diffusion only; (b) partial mixing; (c) complete mixing.







Fig. 4.7 The solute profile ahead of the interface during steady-state solidification with solute redistribution by diffusion only (k<sub>0</sub> < 1).





Fig. 4.10 Concentration-distance profiles for a bar solidified under condition. where solute transport in the liquid is by diffusion only: (a)  $k_0 < 1$ ; (b)  $k_0 > 1$ .



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no coarsening

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## 2 model



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the effective distribution coefficient is given by  $k_{\rm E} = \frac{k_0}{k_0 + (1 - k_0) \exp[-(R\delta/D)]}$ 





Fraction solidified

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Figure 1 Dimensional form of microsegregation volume element from the centre of side arm to the centre of the liquid pool In literature, many attempts have been done in order to analyze solute distribution during solidification of a binary or a multicomponent alloy. The first and simplest analytical model for a binary alloy came from Gulliver-Scheil, considering a constant partition coefficient at the interface during solidification [1,2]. In addition to this, they assumed that there is:

1. No difference in the density between solid and liquid

- 2. Constant physical properties
- 3. Complete mixing in the liquid
- 4. No effect of macrosegregation
- 5. Plate-like morphology at the solid -liquid interface
- 6. No back diffusion in the solid during solidification
- 7. Constant volume element, i.e. no dendrite arm coarsening,
- 8. No undercooling at the tip of dendrite at the beginning of solidification
- 9. No effect of temperature gradient zone melting (TGZM) on solute distribution.

The partial differential equation of Gulliver-Scheil model for these conditions can be written due to fig.1 as follows:

$$A_2 = A_3$$
$$(C_L - C_S) \frac{dx_i}{dt} = (L - X_i) \frac{dC_L}{dt}.$$

(1)

The analytical solution of this differential equation is:

 $C_{\rm S} = k C_{\rm O} \big(1-f_{\rm S}\big)^{(k-1)}\,. \label{eq:C_S}$ 

$$CL=Co(1-fs)^{k-1}$$
  
 $CL=Cofl^{k-1}$   
 $Cs=kCofl^{k-1}$ 

$$f_{s} = \frac{1}{1 - 2\alpha' k} \left[ 1 - \frac{T_{f} - T}{T_{f} - T_{l}} \right] \frac{\frac{1 - 2\alpha' k}{k - 1}}{\frac{1 - 2\alpha' k}{k - 1}}$$



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Figure 6.5: RELATIONSHIP OF THE SEGREGATE FREEZING POINT TO THE PHASE DIAGRAM. An increasing concentration (for distribution coefficients less than unity) is associated with a decreasing liquidus temperature since the slope, m, is then less than zero. Using the curves of figure 6.4, the temperature of the liquid as a function of volume fraction solidified can be derived. The use of realistic diffusion coefficients shows that, for small systems (such as interdendritic regions - figure 6.6) interstitial C in delta- and gamma-Fe will behave according to the lever-rule. Hence, the last liquid of a binary Fe-C melt will solidify at a temperature close to the solidus while substitutional alloys, such as Al-Cu, which typically have much smaller solid-state diffusion coefficients will usually contain eutectic material in the last (interdendritic) regions to solidify even when the overall composition is less than the solubility limit at  $T_e$ .



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Co=4.5



fs



fs

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Later, it was seen that the assumption of no solid-state back diffusion during solidification was not true. Especially, this becomes important near to the end of solidification due to the steep concentration profile and also important in the solidification of body centered cubic structures, such as  $\delta$ -Fe in steels. Brody-Flemings proposed an approximate analytical model, taking into account one dimensional diffusion of solute in solid during solidification in plate-like dendrite arms [3]. The other assumptions of the Gulliver-Scheil model were kept the same. From fig.1, the partial differential equation of Brody-Flemings model can be written as follows:

$$A_2 = A_3 + A_1$$

$$(C_L - C_S)\frac{dx_i}{dt} = D_S \left(\frac{dC}{dx}\right)_{X_i} + (L - X_i)\frac{dC_L}{dt}.$$
(3)

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Figure 1 Dimensional form of microsegregation volume element from the centre of side arm to the centre of the liquid pool The analytical solution of this differential equation for the parabolic growth rate is:

$$C_{s} = kC_{o}\left(1 - f_{s}(1 - 2k\alpha)\right)^{\left(\frac{k-1}{1-2\alpha k}\right)}.$$

#### Cs=kCo(1-(1-k\*beta)\*fs)^((k-1)/(1-k\*beta))

The dimensionless parameter  $\alpha = (D_{st_f})/L^2$  determines the extent of diffusion in the solid phase. Several authors criticized  $\alpha$  dimensionless parameter for being not considered true back diffusion in solid, especially for rapid solid-state diffusion cases,  $\alpha > 0.1$ . For the parabolic growth rate, Clyne-Kurz replaced  $\alpha$  parameter with  $\Omega$  parameter which is also a function of  $\alpha$  and but behave correctly over the whole range of  $\alpha$  values for plate-like morphology [4].

$$\Omega(\alpha) = \alpha \left( 1 - \exp\left(-\frac{1}{\alpha}\right) \right) - \frac{1}{2} \exp\left(-\frac{1}{2\alpha}\right).$$
(5)

Kirkwood modified the Brody-Flemings model for the dendrite arm coarsening situation [5]. He added the last term to the partial differential equation of Brody-Flemings This represents the

### Beta=2\*alpha/(1+2\*alpha) Ohnaka Beta=2\*alpha/(1+2\*alpha+1/2k)



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#### Beta=2\*alpha/(1+2\*alpha) Ohnaka

dilution of liquid due to the increase in the size of dendrite arm. In this model, the other assumptions are same as Brody-Flemings model.

 $A_2 = A_3 + A_1 + A_4$ 

$$(C_{L} - C_{S})\frac{dx_{i}}{dt} = D_{S}\left(\frac{dC}{dx}\right)_{X_{i}} + (L - X_{i})\frac{dC_{L}}{dt} + (C_{L} - C_{O})\frac{dL}{dt}.$$
(6)

$$C_{S} = kC_{O} \left( 1 - \frac{f_{S}}{1+b} \right)^{(k-1)}.$$
 (16)

Similarly, it can be easily shown that the analytical solution of Eq.6 for the parabolic growth rate becomes:

$$C_{s} = kC_{o} \left( 1 - f_{s} \left( \frac{1 - 2k\Omega}{1 + b} \right) \right)^{\left( \frac{k - 1}{1 - 2k\Omega} \right)}.$$
(17)

M can be calculated by using a fitting program between the exact solution and this analytical model. For constant cooling, M is computed as a function of n and k by comparing the exact numerical model of Kirkwood [6] with Eq.16 whereas, for parabolic solid growth, M is obtained by comparing the Voller's parabolic solid growth model [7] with the Eq.16.

where 
$$b = \frac{n}{M} = \frac{n}{\left(\frac{1.2 + 2.4n^{0.7}}{k}\right)}$$
. for constant cooling (18)  
 $b = \frac{n}{M} = \frac{n}{\left(\frac{-41.12 + 51.59n^{0.12}}{k}\right)}$ . for solidification controlled by parabolic solid growth

(19)

M is constant and depends on partition coefficient, k, and coarsening exponent, n. It is also assumed that dendrite arms coarsen with time due to the power law equation:

 $L = At^n \,. \tag{8}$ 



Figure 2 Comparisons between Mortensen exact model (solid line)[8] and approximate analytical model (dashed line) for constant cooling rate



Figure 3 Comparisons between Voller model (solid line) and approximate analytical model (dashed line) for parabolic solid growth for different k values when  $\alpha=0$ 



Figure 4 Comparisons between Voller's model (solid line) and approximate analytical model for parabolic solid growth for different k values as a function of Fourier number. (o for  $\Omega$  and  $\bullet$  for modified  $\Omega$ , i.e. 0.75 $\Omega$ )

# Solidification in Ternary System

#### Solidification Path in Ternary Systems

In general, the path of solidification (trace of liquid or solid composition as a function of  $f_S$ ) can be obtained in the case of solidification by relating the composition to  $f_t$ , which must be the same for all of the elements). Using equation All.10 and All.18:

$$\begin{pmatrix} C_{\iota} \\ C_{o} \end{pmatrix}_{B} = (1 - u_{B}f_{S})^{-\frac{PB}{UB}}$$
[A11.25]
$$\begin{pmatrix} C_{\iota} \\ C_{o} \end{pmatrix}_{C} = (1 - u_{C}f_{S})^{-\frac{PC}{UC}}$$

where  $u_i = 1 - 2\alpha_i k_i$  and  $p_i = 1 - k_i$ . Eliminating  $f_s$ :

$$\begin{pmatrix} C_{\iota} \\ C_{o} \end{pmatrix}_{B} = \left\{ 1 - \frac{u_{B}}{u_{C}} \left[ 1 - \left( \frac{C_{\iota}}{C_{o}} \right)_{C} \right]^{-\frac{u_{C}}{p_{C}}} \right\}^{-\frac{p_{B}}{u_{B}}}$$
[A11.26]

# Solidification in Al-Cu-Mg Alloys

## 7050 Plate

Focus on one alloy (7050) and product (thick hot rolled plate)



Components machined from 7050 alloy thick plate are widely used in load bearing applications e.g. wing spars

Si	Fe	Cu	Mg	Zn	Ti	Zr	Al
$0.12 \mathrm{max}$	$0.15 \max$	2.0-2.6	1.9 - 2.6	5.7 - 6.7	0.06	0.08 - 0.15	bal

7050 composition specification

#### Processing Sequence - 7050 Plate



## Microstructural Changes





Cast Homogenized Rolled

Solutionized

# Simple Phase Diagrams

Even for simple 2xxx alloy (AI-Cu-Mg), need data for 3 binaries and information about ternary phases



MTDATA predicted phase diagrams

Real, commercial Al-alloys may contain > 10 alloying elements!

Success of thermodynamic models relies on availability of sufficient, high quality, thermodynamic data

### Predictions for Binary Al-Cu Alloy



# Predictions for Ternary Al-Cu-Mg alloy Predictions for 2xxx (AI-4.5Cu-1.5wt%) Mg alloy



# Prediction of Freezing Range

To reduce tendency for solidification cracking, need to minimize absolute freezing range

Use thermodynamic model to predict freezing range for different alloy compositions



Effect of Mg content on freezing range of eutectic in Al-4.5Cu-x Mg alloy


## **Microsegregation in Manganese Steels**

• Fe-C-Mn Aloys

## Fe-C-Mn Phase Diagram

Alloy No	%С	%Mn	%5	%P	%Other
а <del>на 1976 — 1</del> 97 — 19770 — 19777 — 1977 — 1977 — 1977 — 1977 — 1977 — 1	0.10	1.57	0.003	0.005	< 0.02
2	0.21	1.60	0.003	0.005	< 0.02
3	0.40	1.58	0.003	0.005	< 0.02
4	0.75	1.59	0.003	0.005	< 0.02

Table 1: Chemical Composition of the Investigated Steels.

Table 2: Temperature Gradients in the Calibration Specimen as a function of Growth Rate for Three Series.

Series	Growth Rate (mm/s)	Temperature Gradient (K/mm) in liquid in liquid+solid		
	0.025	8.4	10.2	
9		5.5	.9	
1	0.25	4.8	7.4	
1	0.5	3.5	6.5	
5	0.025	10.7	12	
2	1	7.2	10.5	
2	0.25	5.9	9	
2	0.5	4.6	7.4	
2	0.025	13.2	15.6	
7	1	9.8	11.3	
2	0.25	7.6	9.2	
3	0.5	6.9	8.3	







Figure 1: (a) The transverse section of close packed primary arm spacing arrangement. (b) The two extreme possible arrangement of secondary arms on the longitudinal section showing concave solidification model.



Figure 2: Segregation ratio between secondary arms as a function of cooling rate and carbon content at 1200°C.



Figure 4: Compatison of predicted maximum concentration of manganese by concave computer model to the experimental results as a function of cooling rate at 1200°C (Fe-1.6%Mn-0.8%C)



Figure 3: The segregation ratio of manganese as a function of cooling rate and carbon content at 1200°C.



Figure 5: Comparison of predicted Cmax and Cmin of manganese by secondary arm coarsening model with the experimental results as a function of cooling rate for 0.4 and 0.8%C at 1200°C.



Figure 6: Variation of maximum concentration of manganese and computer results as a function of cooling rate for 0.1%C at 1485°C.



Figure 8: Variation of maximum concentration of manganese and computer results as a function of cooling rate for 0.8%C at 1200°C.



Figure 7: Variation of maximum concentration of manganese and computer results as a function of cooling rate for 0.4%C at 1200°C.



Figure 9: Average manganese segregation ratio as a function of carbon content for primary arms and secondary arms.



Figure 9: Average manganese segregation ratio as a function of carbon content for primary arms and secondary arms.



**Figure 7.41** The degree of segregation of <u>Cr</u> as a function of the <u>C</u> concentration (in wt-%) in a series of Fe–Cr–C alloys with constant <u>Cr</u> concentration. The values, which are plotted in the diagram, originate from four different and independent investigations. Reproduced with permission from the Scandinavian Journal of Metallurgy, Blackwell.