

"Phase Transformation in Materials"

11. 06. 2023 Eun Soo Park

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Equilibrium Shape and Interface Structure on an Atomic Scale



Apply thermodynamics to this fact and derive more information.

Enthalpy-dominant strong bonding energy weak bonding energy stable at low T stable at high T

Thermal Roughening

singular (smooth) interface

rough interface



Enthalpy-dominant

Entropy-dominant

Heating up to the roughening transition.

Kinetic Roughening

Rough interface - Ideal Growth \rightarrow diffusion-controlled \rightarrow dendritic growth

Smooth interface - Growth by Screw Dislocation Growth by 2-D Nucleation

Small $\Delta T \rightarrow$ "feather" type of growth \clubsuit Large $\Delta T \rightarrow$ cellular/dendritic growth

Growth rate,

The growth rate of the singular interface cannot be higher than ideal growth rate.

When the growth rate of the singular Interface is high enough, it follows the ideal growth rate like a rough interface.

 \rightarrow kinetic roughening



Interface undercooling, ΔT_i Fig.4.14 The influence of interface undercooling (ΔT_i) on growth rate

Q: Heat Flow and Interface Stability

1) Superheated liquid

: Extraction of latent heat by conduction in the crystal

2) Supercooled liquid

: Conduction of latent heat into the liquid

→ Development of Thermal Dendrite

4.2.3 Heat Flow and Interface Stability - Planar interface

1) Superheated liquid

Consider the solidification front with heat flow from L to S.



dT/dx in the liquid ahead of the protrusion will increase more positively. $T_L^{\uparrow} \& T_S^{\downarrow}$

More heat to the protrusion \rightarrow melt away

v of protrusion \downarrow to match other *v* in planar region

mould walls 6

"Removal of latent heat" → Heat Flow and Interface Stability



Heat Flow and Interface Stability - Planar interface

2) Solid growing into supercooled liquid



- heat flow from solid = the protrusion grows preferentially.



4 Fold Symmetric Dendrite Array

Development of Thermal Dendrite

cf) constitutional supercooling

When does heat flow into liquid?

- \rightarrow Liquid should be supercooled below $T_{\rm m}$.
- \rightarrow Nucleation at impurity particles in the bulk of the liquid



Fig. 4.17 The development of thermal dendrites: (a) a spherical nucleus; (b) the interface becomes unstable; (c) primary arms develop in crystallographic directions (<100> in cubic crystals); (d) secondary and tertiary arms develop 10

Q: How to calculate the growth rate (v) in the tip of a growing dendrite?

Closer look at the tip of a growing dendrite

different from a planar interface because heat can be conducted away from the tip in three dimensions.

Fig.4.18



$$T_{L}'(negative) \cong \frac{\Delta T_{C}}{r} \quad \Delta T_{C} = T_{i} - T_{\infty}$$
$$v = \frac{-K_{L}T_{L}'}{L_{V}} \cong \frac{K_{L}}{L_{V}} \cdot \frac{\Delta T_{C}}{r} \qquad v \propto \frac{1}{r}$$

Thermodynamics at the tip?

Gibbs-Thomson effect: melting point depression



Minimum possible radius (r)?



"Removal of latent heat" → Heat Flow and Interface Stability



Closer look at the tip of a growing dendrite

different from a planar interface because heat can be conducted away from the tip in three dimensions.

Assume the solid is isothermal $(T'_{S} = 0)$ From $K_{S}T'_{S} = K_{L}T'_{L} + vL_{V}$ If $T'_{S} = 0$, $v = \frac{-K_{L}T'_{L}}{L_{V}}$ A solution to the heat-flow equation for a hemispherical tip: $T'_{L}(negative) \cong \frac{\Delta T_{C}}{r} \quad \Delta T_{C} = T_{i} - T_{\infty}$ $K T'_{L} = K_{L} \quad \Delta T_{C} = 1$

$$V = \frac{-K_L I_L}{L_V} \cong \frac{K_L}{L_V} \cdot \frac{\Delta I_C}{r} \qquad V \propto \frac{1}{r}$$

Thermodynamics at the tip?

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However, ΔI also depends on How?

$$\Delta G = \frac{L_V}{T_m} \Delta T_r = \frac{2\gamma}{r} \qquad \Delta T_r = \frac{2\gamma T_m}{L_V r}$$

Minimum possible radius (r)?



Solidification: Liquid ----> Solid

< Nucleation >

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Nucleation in Pure Metals

- < Growth >
- Equilibrium Shape and Interface Structure on an Atomic Scale
- Growth of a pure solid
- Heat Flow and Interface Stability

4.3 Alloy solidification

- Solidification of single-phase alloys
- Eutectic solidification
- Off-eutectic alloys
- Peritectic solidification

Solidification: Liquid ----- Solid

1) Pure Metals: Nucleation and Growth

a) homogeneous Nucleation or Heterogeneous Nucleation • Undercooling ΔT



Q: Alloy solidification?

- 1. Solidification of single-phase alloys
- Three limiting cases

1) Equilibrium Solidification: perfect mixing in solid and liquid

2) No Diffusion in Solid, Perfect Mixing in Liquid

3) No Diffusion on Solid, Diffusional Mixing in the Liquid

- Planar S/L interface \rightarrow unidirectional solidification



- Constitutional supercooling

1. Solidification of single-phase alloys



k : partition coefficient X: mole fraction of solute

Planar S/L interface

In this phase diagram of straight solidus and liquidus, *k* is const. (independent of *T*).

Fig. 4.19 A hypothetical phase diagram. $k = X_s/X_l$ is constant. T_1 $X_{\rm L}$ T_2 X_{S} Liquidus T_3 $T_{\rm E}$ X_{\max} kX_0 X_0 $\frac{X_0}{k}$ $X_{\rm E}$ X_{Solute} Heat Solid Liquid \rightarrow unidirectional solidification (a) x -

1. Solidification of single-phase alloys

Three limiting cases

- 1) Equilibrium Solidification (perfect mixing in solid & liquid)
- 2) No Diffusion in Solid, Perfect Mixing in Liquid
- 3) No Diffusion on Solid, Diffusional Mixing in the Liquid



- Sufficient time for diffusion in solid & liquid
- Relative amount of solid and liquid : lever rule
- Solidification starts at $T_1 (X_s = kX_0)$ and ends at $T_3 (X_L = X_0/k)$.

Composition vs x at T_2



Fig. 4.20 Unidirectional solidification of alloy X_0 in Fig. 4.19. (a) A planar S/L interface and axial heat flow. (b) Corresponding composition profile at T_2 assuming complete equilibrium. Conservation of solute requires the two shaded areas to be equal. $A_s = A_L$

1) Equilibrium Solidification : perfect mixing in solid and liquid



2) Non-equilibrium Solidification: No Diffusion in Solid, Perfect Mixing in Liquid

- : high cooling rate, efficient stirring
- Separate layers of solid retain their original compositions mean comp. of the solid $(\overline{X_s}) < X_s$
- Liquid become richer than $X_0/K \rightarrow X_E$ at the last part of solidification.
- Variation of X_s : solute rejected to the liquid \rightarrow solute increase in the liquid



(d) 0

Distance along bar

(a) As Fig. 4.19, but including the mean composition of the solid. (b) Composition profile just under T_1 . (c) Composition profile at T_2 (compare with the profile and fraction solidified in Fig.4.20b) (d) Composition profile at the eutectic temperature and below.



Mass balance: non-equilibrium lever rule (coring structure)

 T_1 T_2

 T_3

 kX_0

 X_0

When cooled by ΔT from any arbitrary T, determine the followings.

: solute ejected into the liquid = ? \rightarrow solute increase in the liquid

Ignore the difference in molar volume $T_{\rm E}$ between the solid and liquid.

f_s: volume fraction solidified



$$\begin{array}{c} df_{s} & (X_{L} - X_{S}) \\ (1-f_{s}) & dX_{L} \end{array}$$

 $X_{\rm F}$

X_{Solute}

$$(X_L - X_S)df_S = (1 - f_S)dX_L$$

when
$$f_s = 0 \rightarrow X_s$$
, X_L ?

Solve this equation.

 $X_{\rm L}$

Liquidus

$$X_{S} = kX_{0}$$
 and $X_{L} = X_{0}$

Initial conditions



"If k<1: predicts that if no diff. in solid, some eutectic always exist to solidify." $(X_s < X_L)$

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3) No Diffusion on Solid, Diffusional Mixing in the Liquid

: high cooling rate, no stirring \rightarrow diffusion



local equil. at S/L interface



Composition profile at $T_2 < T_{S/L} < T_3$?

Steady-state profile at T_3 ? at T_E or below?



No Diffusion on Solid, Diffusional Mixing in the Liquid







- Fig. 4.22 Planar front solidification of alloy X_0 in Fig. 4.19 assuming no diffusion in solid and no stirring in the liquid.
- (a) Composition profile when S/L temperature is between T_2 and T_3 in Fig. 4.19.
- X_{ma} (b) Steady-state at T₃. The composition solidifying equals the composition of liquid far ahead of the solid (X_0) .
 - Composition profile at T_E and below, showing (C) the final transient.

"Alloy solidification" - Solidification of single-phase alloys

* No Diffusion on Solid. Diffusional Mixina in the Liquid



When the solid/liquid interface is within ~D/v of the end of the bar the bow-wave of solute is compressed into a very small volume and the interface composition rises rapidly leading to a final transient and eutectic formation.

No Diffusion on Solid, Diffusional Mixing in the Liquid

During steady-state growth,

(Interface \rightarrow liquid: Diffusion rate)

Rate at which solute diffuses down the concentration gradient away from the interface = Rate at which solute is rejected from the solidifying liquid

(Solid→Interface: solute rejecting rate)

Set up the equation.

$$J = DC_{L}' = v(C_{L} - C_{S})$$
$$J = -D\frac{\partial X_{L}}{\partial x} = v(X_{L} - X_{S})$$

(Solidification rate of alloy: excess solute control)

$$\mathbf{K}_{S}T'_{S} = \mathbf{K}_{L}T'_{L} + \mathbf{V}L_{V}$$

(Solidification rate of pure metal: latent heat control, 10⁴ times faster than that of alloy)

Solve this equation.

 $X_s = X_0 \quad for \quad all \quad x \ge 0$

$$\frac{dX_L}{X_L - X_0} = -\frac{v}{D}dx$$

$$\ln(X_L - X_0) = -\frac{v}{D}x + c$$

$$x = 0; X_L = X_0 / k$$
 steady-state

$$c = \ln(\frac{X_0}{k} - X_0)$$



- ($X_{\underline{l}}$ decreases exponentially from $X_{\underline{0}}/k$ at x=0, the interface, to $X_{\underline{0}}$ at large distances from the interface. The concentration profile has a characteristic width of D/v.)
- The concentration gradient in liquid in contact with the solid :

$$J = -DX'_{L} = v(X_{L} - X_{S}) \qquad X'_{L} = -\frac{X_{L} - X_{S}}{D/v}$$



Q: Cellular and Dendritic Solidification by "constitutional supercooling" in alloy

2. Cellular and Dendritic Solidification

Fast Solute diffusion similar to the conduction of latent heat in pure metal, possible to break up the planar front into dendrites.

 \rightarrow complicated, however, by the possibility of temp. gradients in the liquid.


* Constitutional Supercooling

No Diffusion on Solid, Diffusional Mixing in the Liquid



T_{equil.}

Fig.4.23 Solid Liquid $\frac{X_0}{k}$ Χ $X_{\rm L}$ X_0 (a) $\frac{D}{v}$ Critical gradient T_1 T_{e} (b) TConstitutional supercooling T_3 At the interface, Solid \rightarrow Liquid $T_L = T_{equil.} (not T_E) = T_3$



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 $T_L' > (T_1 - T_3)/(D/v)$: the protrusion melts back \rightarrow Planar interface: stable $T_L' / v < (T_1 - T_3)/D$: Constitutional supercooling \rightarrow cellular/ dendritic growth

Q: Planer → Cell structure → Dendrite?

by constitutional supercooling in superheated liquid

If temperature gradient ahead of an initially planar interface is gradually reduced below the critical value, (constitutional supercooling at solid/liquid interface)



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Tips of the cells grow into the hottest liquid and therefore contain the least solute.







(b)

Fig.4.26 Cellular microstructures

Note that each cell has virtually the same orientation as its neighbors and together they form a single grain.

(a) A decanted interface of a cellularly solidified Pb-Sn alloy (x 120)

(after J.W. Rutter in Liquid Metals and Solidification, American Society for Metals, 1958, p. 243).

(b) Longitudinal view of cells in carbon tetrabromide (x 100) (after K.A. Jackson and J.D. Hunt, Acta Metallurgica 13 (1965) 1212).



The change in morphology from cells to dendrites

- * Cellular microstructures are only stable for a certain range of temp. gradients.
- → Sufficiently low temp. gradients → Creation of constitutional supercooling in the liquid between the cells causing interface instabilities in the transverse direction (although, No temp. gradient perpendicular to the growth direction)
- → Develop arms, i.e. dendrites form & Change in the direction of the primary arms away from the direction of heat flow into the crystallographically preferred directions i.e. (100) for cubic metals.



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Fig. 4.28 Columnar dendrites in a transparent organic alloy.

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Cellular and Dendritic Solidification

At the interface, $T_L = T_e$ (not T_E) = $T_3 \rightarrow T_{L, \text{ liquid}} = T_1 : T' = T_1 - T_3$ (superheating)

• Criterion for the stable planar interface:

 $T_{L}' > (T_{1}-T_{3})/(D/v)$: the protrusion melts back_steeper than the critical gradient $T_{L}'/v > (T_{1}-T_{3})/D$ $(T_{1}-T_{3}: Equilibrium freezing range of alloy)$

 \longrightarrow Large solidification range of T₁-T₃ or high *v* promotes protrusions.

need to well-controlled experimental conditions (temp. gradient & growth rate)

Constitutional supercooling:

Formation of Cell and Dendrites Structures

Solute effect : addition of a very small fraction of a percent solute with

very small k ($k = \frac{X_s}{X_l}$) \rightarrow (T₁-T₃) \uparrow promotes dendrites.

Cooling rate effect : <u>Higher cooling rate</u> allow less time for lateral diffusion of the rejected solute and therefore require <u>smaller cell or dendrite arm</u> <u>spacings</u> to avoid constitutional supercooling.

Solidification of Pure Metal

: Thermal gradient dominant



Solidification of single phase alloy: Solute redistribution dominant

a) Constitutional supercooling

Planar \rightarrow Cellular growth \rightarrow cellular dendritic growth \rightarrow Free dendritic growth

응고계면에 조성적 과냉의 thin zone 형성에 의함 Dome 형태 선단 / 주변에 hexagonal array T↓→조성적 과냉영역 증가 Cell 선단의 피라미드형상/가지 들의 square array/Dendrite 성장방향쪽으로 성장방향 변화 성장하는 crystal로 부터 발생한 <u>좌</u> <u>열을 과냉각 액상쪽으로 방출</u>함에 의해 형성 Dendrite 성장 방향/ Branched rod-type dendrite

→ "Nucleation of new crystal in liquid" 성장이 일어나는 interface 보다 높은 온도

b) Segregation

: normal segregation, grain boundary segregation, cellular segregation, dendritic segregation, inversegregation, coring and intercrystalline segregation, gravity segregation



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Τ

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- Off-eutectic alloys
- Peritectic solidification
Q: Various different types of eutectic solidification $(L \rightarrow \alpha + \beta)$?



(e)

various

(g)

Fig. 14 Schematic representation possible in eutectic structures. (a), (b) and (c) are alloys shown in fig. 13; (d) nodular; (e) Chinese script; (f) acicular; (g) lamellar; and (h) divorced.

(f)

(h)

4.3.2 Eutectic Solidification

Various different types of eutectic solidification \rightarrow Both phases grow simultaneously.

Normal eutectic

both phases have low entropies of fusion.



Fig. 4.30 Rod-like eutectic. Al₆Fe rods in Al matrix. Transverse section. Transmission electron micrograph (x 70000).

Anomalous eutectic One of the solid phases is capable of faceting, i.e., has a high entropy or melting.



The microstructure of the Pb-61.9%Sn (eutectic) alloy presented a coupled growth of the (Pb)/ β Sn eutectic. There is a remarkable change in morphology increasing the degree of undercooling with transition from regular lamellar to anomalous eutectic.

Eutectic

Divorced Eutectic





Q: Thermodynamics and Kinetics of eutectic solidification $(L \rightarrow \alpha + \beta)$?

This section will only be concerned with normal structures, and deal mainly with lamellar morphologies.





Plot the diagram of Gibbs free energy vs. composition at T_3 and T_4 .

What is the driving force for the eutectic reaction (L $\rightarrow \alpha$ + β) at T₄ at C_{eut}?

What is the driving force for nucleation of α and β ? " ΔT "

Eutectic Solidification (Kinetics)

: $\Delta T \rightarrow$ formation of interface + solute redistribution

If α is nucleated from liquid and starts to grow, what would be the composition at the interface of α /L determined?

 \rightarrow rough interface (diffusion interface) & local equilibrium

How about at β/L ? Nature's choice? Lamellar structure



→ Gibbs-Thomson Effect