2021 Fall

"Phase Transformation *in* Materials"

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- Binary System mixture/ solution / compound

- Gibbs Free Energy in Binary System

$$G_1 = X_A G_A + X_B G_B \quad J/mol$$

$$G_2 = G_1 + \Delta G_{mix} \quad J/mol$$

Ideal solution ($\Delta H_{mix}=0$) $\Delta G^{mix} = RT(X_A \ln X_A + X_B \ln X_B)$

$$G = X_A G_A + X_B G_B + RT(X_A \ln X_A + X_B \ln X_B)$$

Regular solution $\Delta H_{mix} = P_{AB}\varepsilon$ where $\varepsilon = \varepsilon_{AB} - \dfrac{1}{2}(\varepsilon_{AA} + \varepsilon_{BB})$

$$G = X_A G_A + X_B G_B + \Omega X_A X_B + RT(X_A \ln X_A + X_B \ln X_B)$$

- Chemical potential and Activity

$$\mu_A = \left(\frac{\partial G'}{\partial n_A} \right)_{T, P, n_B}$$

• $\mu_A = G_A + RT\ln a_A$ $\ln\left(\dfrac{a_A}{X_A}\right) = \dfrac{\Omega}{RT}(1 - X_A)^2$

$\dfrac{a_A}{X_A} = \gamma_A = \text{activity coefficient}$
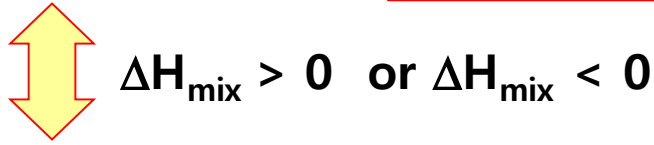
μ는 조성에 의해 결정되기 때문에 dn_A가 매우 작아서 조성변화 없어야

2

- Binary System mixture/ solution / compound

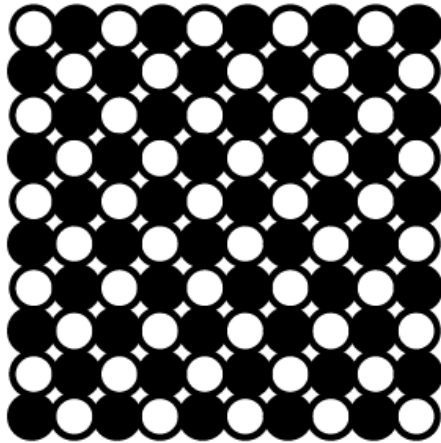**Ideal solution** $(\Delta H_{mix}=0)$ **Random distribution**

Regular solution $\boxed{\Delta H_{mix} = P_{AB}\varepsilon}$ where $\varepsilon = \varepsilon_{AB} - \dfrac{1}{2}(\varepsilon_{AA} + \varepsilon_{BB})$ $\boxed{\varepsilon \approx 0}$

⇕ $\Delta H_{mix} > 0$ or $\Delta H_{mix} < 0$

Real solution   **Ordered structure**

(a) $\varepsilon < 0$, $\Delta H_{mix} < 0$ (b) $\varepsilon > 0$, $\Delta H_{mix} > 0$ (c) when the size difference is large

Ordered alloys   **Clustering**   strain effect

P_{AB} ↑ ⟶ **Internal E** ↓ P_{AA}, P_{BB} ↑   **Interstitial solution**

3
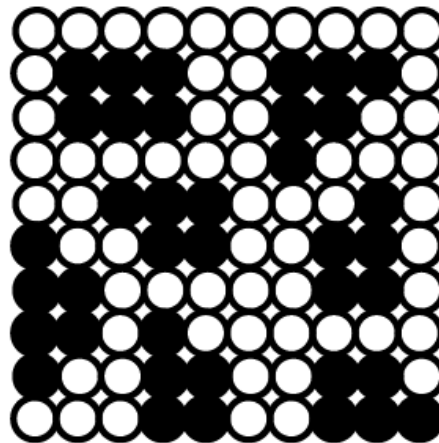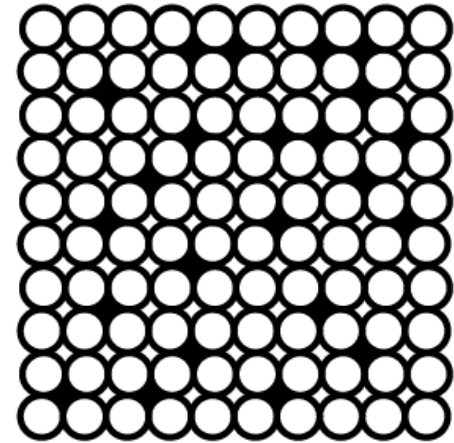
Q9: Ordered phase II:

"Long range order (LRO)"

(①superlattice, ②intermediate phase, ③intermetallic compound)
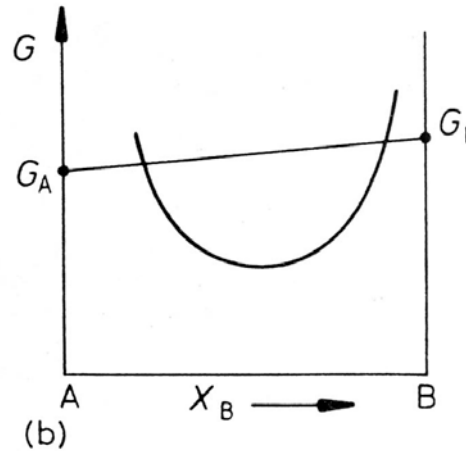
* Solid solution → ordered phase
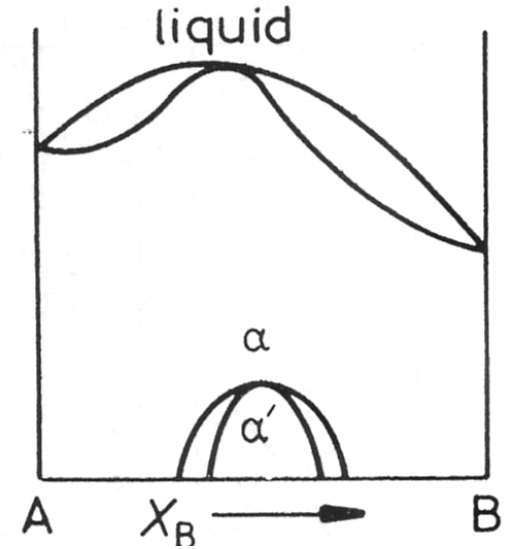
→ **random mixing**
→ **entropy ↑**
   negative enthalpy ↓

$$\Delta H_{mix}^{S} < 0$$

Large composition range
   ➔ **G ↓**

diate phases: (a) for an intermetallic com-
(b) for an intermediate phase with a wide

* Compound : AB, A_2B…

→ **entropy↓**
→ **covalent, ionic contribution.**
→ **enthalpy more negative ↓**

$$\Delta H_{mix}^{S} << 0$$

Small composition range
   ➔ **G ↓**
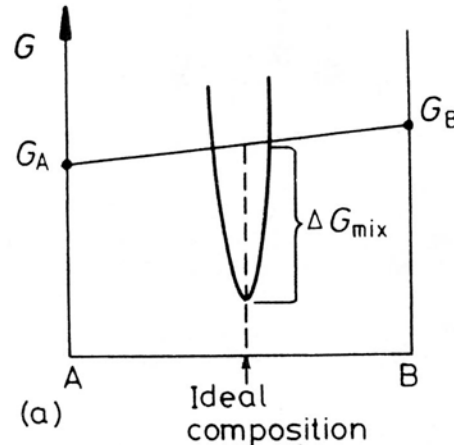
Fig. 1.23 Free energy curves for intermed
pound with a very narrow stability range, (
stability range.
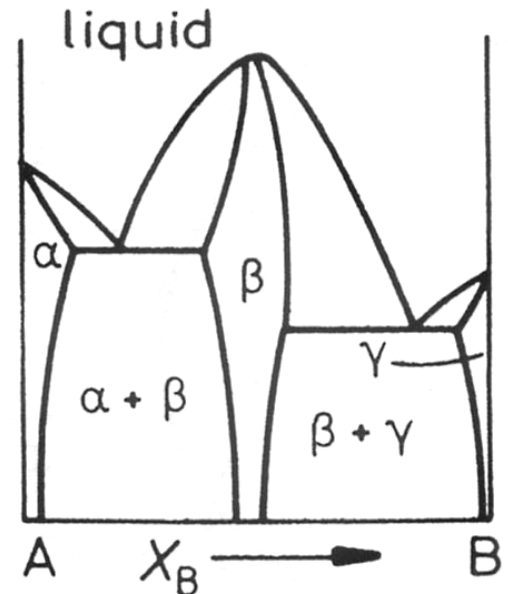
Superlattice formation: order-disorder transformation

- $\varepsilon < 0$, $\Delta H_{mix} < 0$
- **between dissimilar atoms** than between similar atoms
- **Large electrochemical factor**: tendency for the solute atoms to avoid each other
  and to associate with the solvent atoms
- **Size factor just within the favorable limit:** lead to atomic rearrangement
 so as to relieve the lattice distortion imposed by the solute atoms

(a)L2$_0$:

CuZn/FeCo/NiAl/CoAl/

FeAl/AgMg/AuCd/NiZn

Cu

Zn
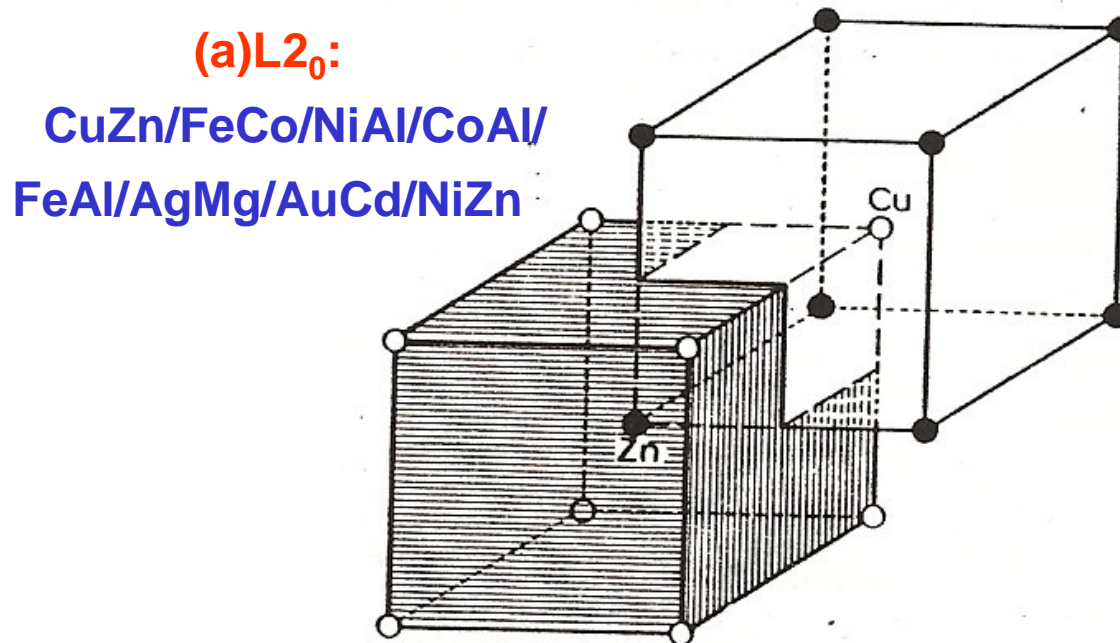
β brass superlattice viewed as two inter-penetrating cubic lattices

$$\Delta G_{mix} = \Delta H_{mix} - T\Delta S_{mix}$$

Ordered phase
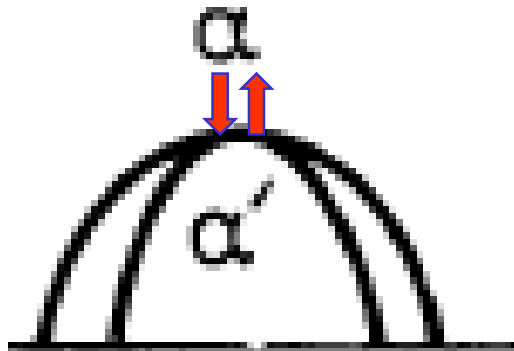
$\varepsilon < 0, \ \Delta H_{mix} < 0$

Fig. 1.21. Part of the Cu-Au phase diagram showing the regions where the Cu$_3$Au and CuAu superlattices are stable.

$\Delta S_{mix} \sim small$

At high Temp.,

$\Delta G_{mix} \sim large\ (-)$

Vacancy/another atoms
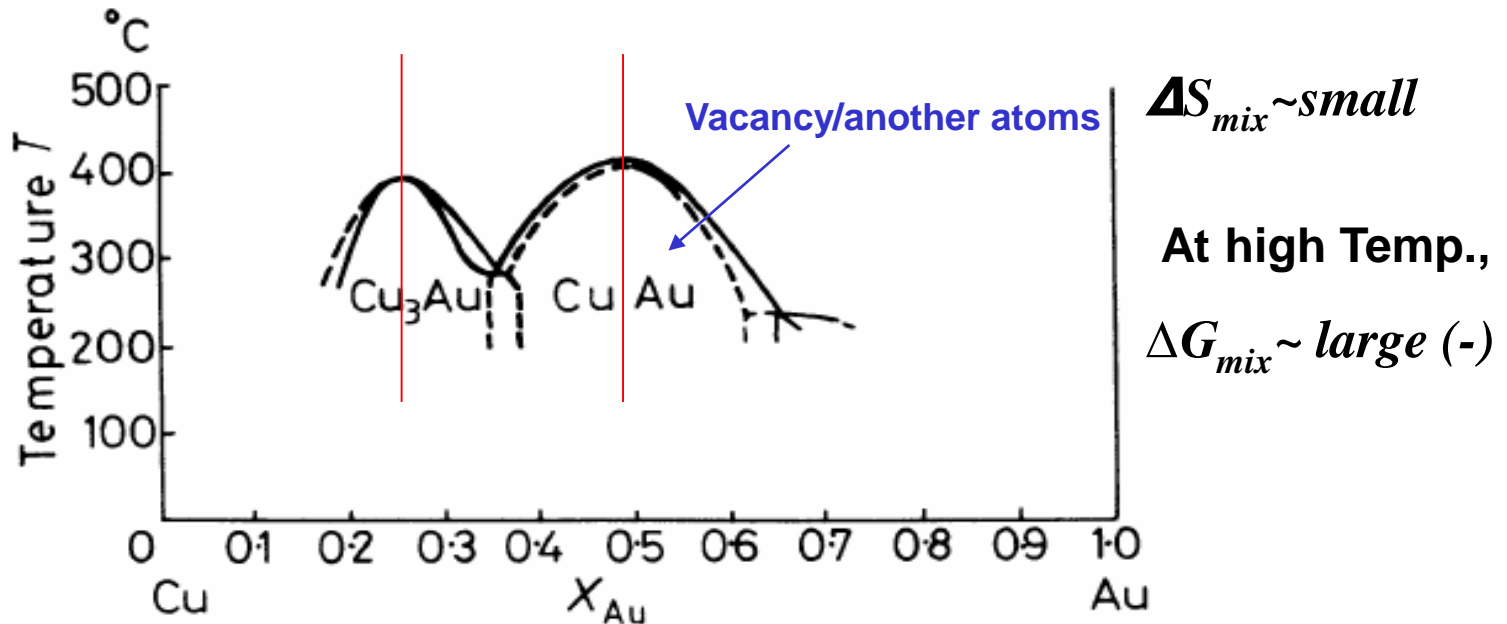
- **The entropy of mixing of structure with LRO is extremely small and the degree of order decrease with increasing temperature until above some critical temperature there is no LRO at all.**

- **This temperature is a maximum when the composition is the ideal required for the superlattice.**

- **The critical temperature for loss of LRO increases with increasing Ω or ΔH$_{mix}$, and in many systems the ordered phase is stable up to the melting point.**
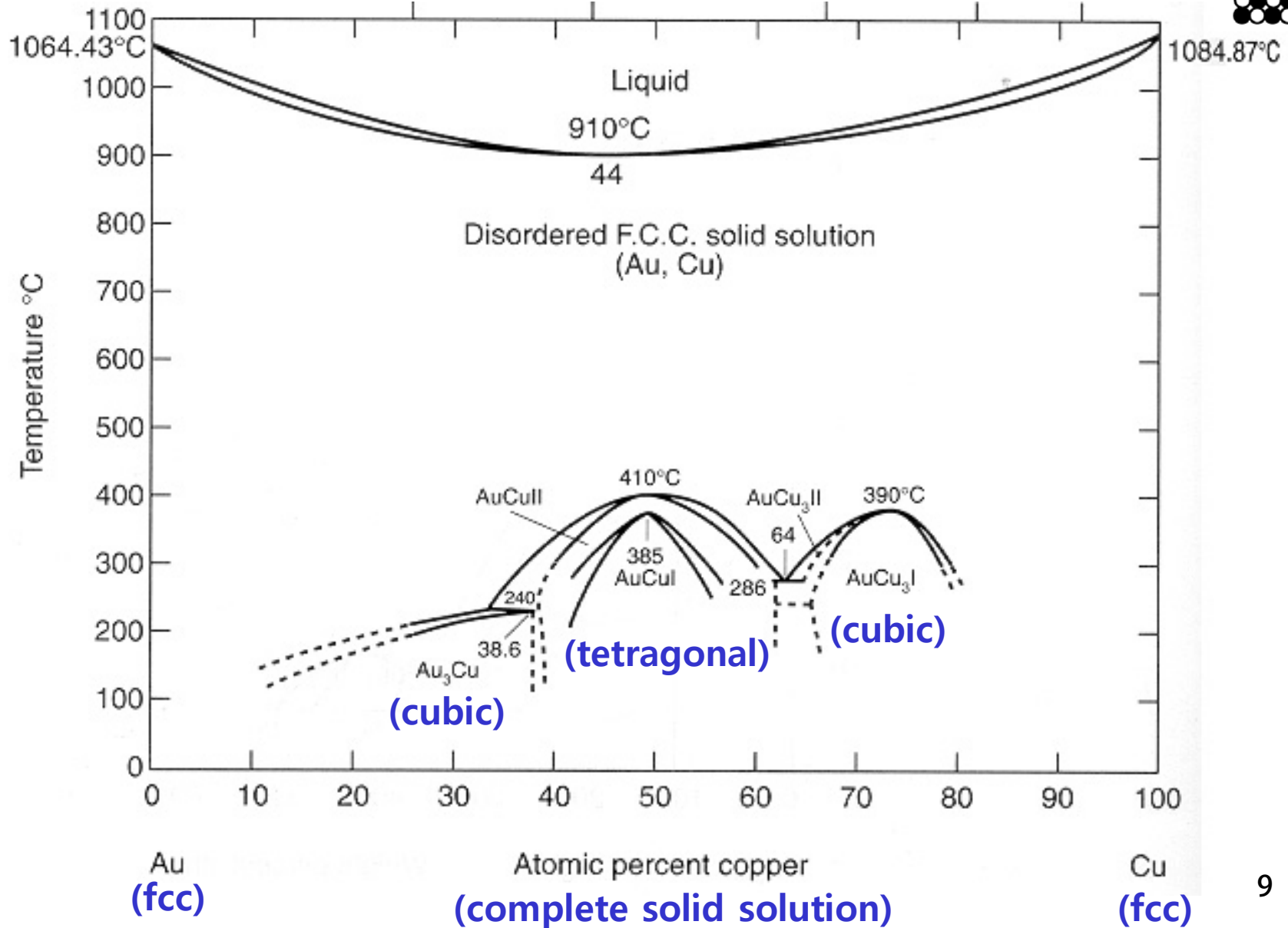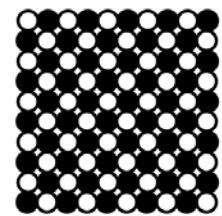
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Order-disorder phase transformation

- **Not classical phase change=~not depend on diffusion process**

- **change of temperature allowed a continuous re-arrangement of atoms without changing the phase = "2nd order transition"**

- **boundary: ordered lattice & disordered lattice/phase rule could not applied**

 there are cases in which an ordered phase of one composition exists in equilibrium with a disordered phase of a different composition.

- **Simple composition of the type AB or AB$_3$ can the transformation (i.e. at the temperature maximum) be considered diffusionless.**
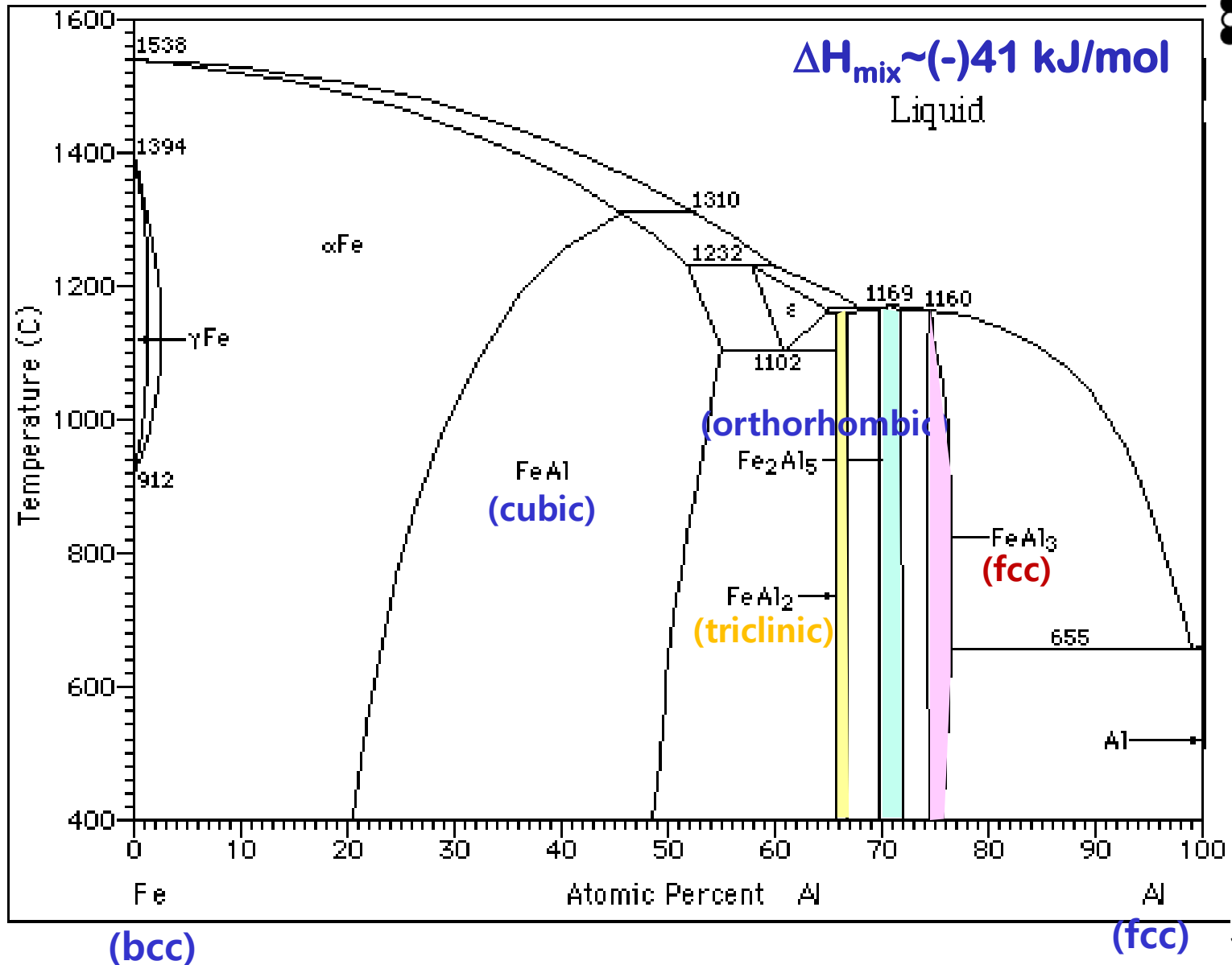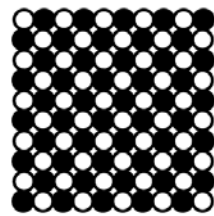
Ordered Phase

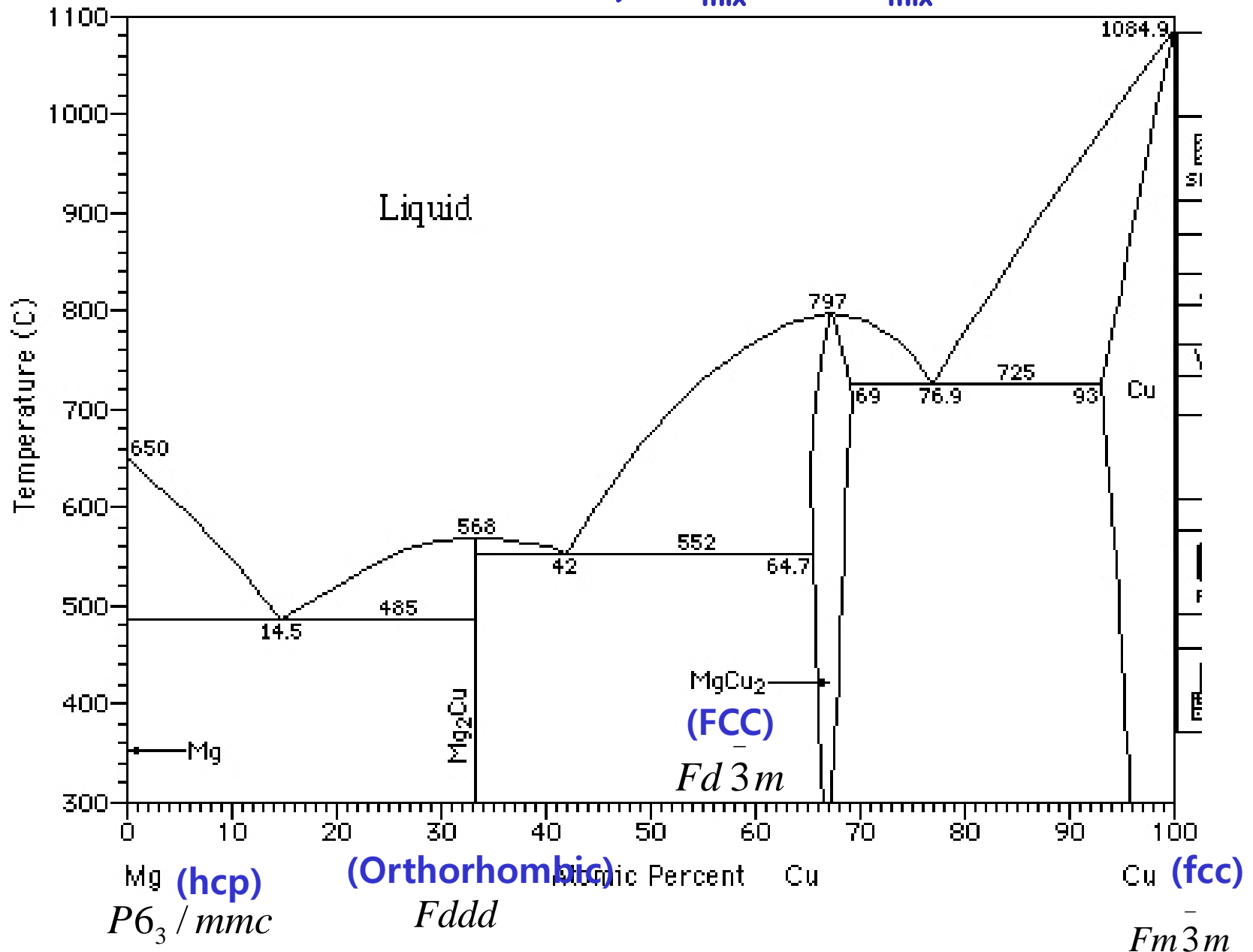$\varepsilon < 0, \quad \Delta H_{mix} < 0 \ / \ \Delta H_{mix} \sim -20 \ kJ/mol$

Intermediate Phase

$\Delta H_{mix} \sim (-)41\ kJ/mol$

Fe–Al phase diagram with Liquid, αFe, γFe, FeAl (cubic), Fe$_2$Al$_5$ (orthorhombic), FeAl$_2$ (triclinic), FeAl$_3$ (fcc) phases. Temperatures marked: 1538, 1394, 912, 1310, 1232, 1169, 1160, 1102, 655. (bcc) at Fe side, (fcc) at Al side.

Intermediate Phase

$\varepsilon < 0, \ \Delta H_{mix} < 0 \ / \ \Delta H_{mix} \sim -38 \text{ kJ/mol}$

(FCC)

$Fd\bar{3}m$

(Orthorhombic)

Mg **(hcp)**

$P6_3 / mmc$

$Fddd$

Cu **(fcc)**

$Fm\bar{3}m$
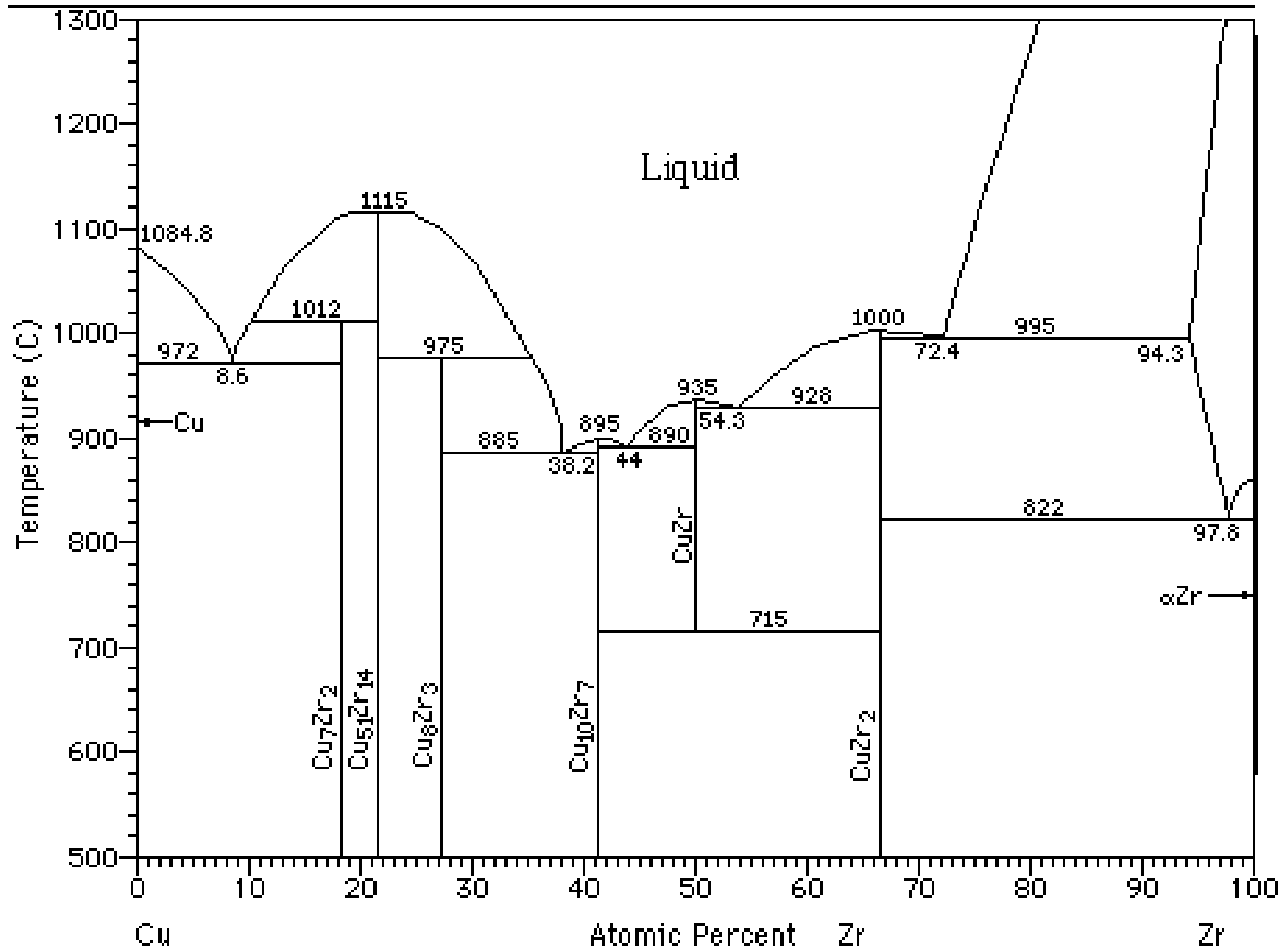
Intermediate Phase

Intermediate Phase
_"different crystal structure as either of the pure component"

3 main factors
determining <u>the structure of Intermediate phase</u> ?

1) Relative atomic size

- **Laves phase** (**size difference: 1.1~1.6** ex: MgCu$_2$)

  <u>**fill space most efficiently ~ stable**</u>

- **Interstitial compound**: MX, M$_2$X, MX$_2$, M$_6$X

  M= Cubic or HCP ex: Zr, Ti, V, Cr, etc, X= H, B, C, and N
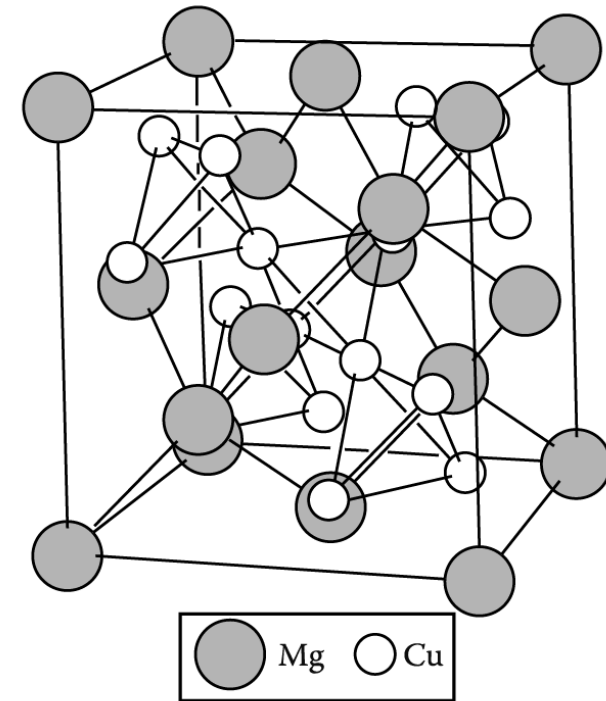
2) Relative valency electron

- **electron phases** ex_α & β brass

  # of valency electrons per unit cell
  → <u>depending on compositional change</u>

3) Electronegativity

- <u>**very different electronegativites**</u> → **ionic bond_normal valency compounds**
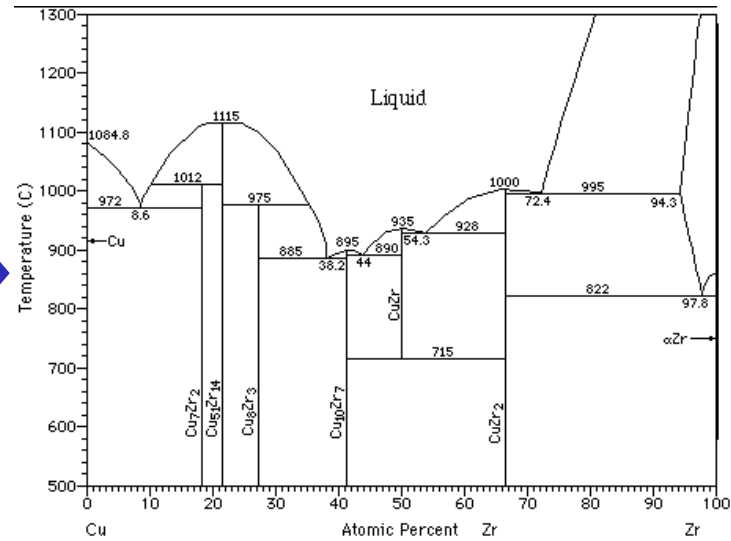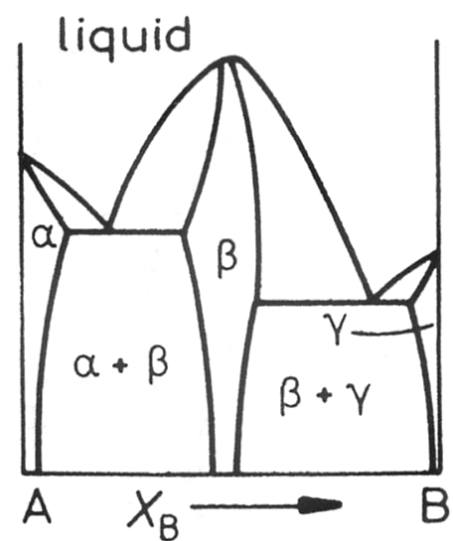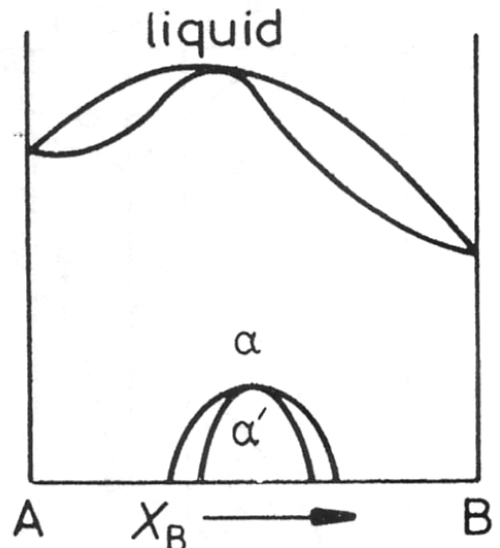 **ex Mg$_2$Sn**

◯ Mg ◯ Cu

MgCu$_2$ (A Laves phase)

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$\Delta H_{mix}^{S} > 0$: **Solid solution → solid state phase separation (two solid solutions)**

$\Delta H_{mix}^{S} >> 0$: **liquid state phase separation (up to two liquid solutions)**

metastable miscibility gap

stable miscibility gap
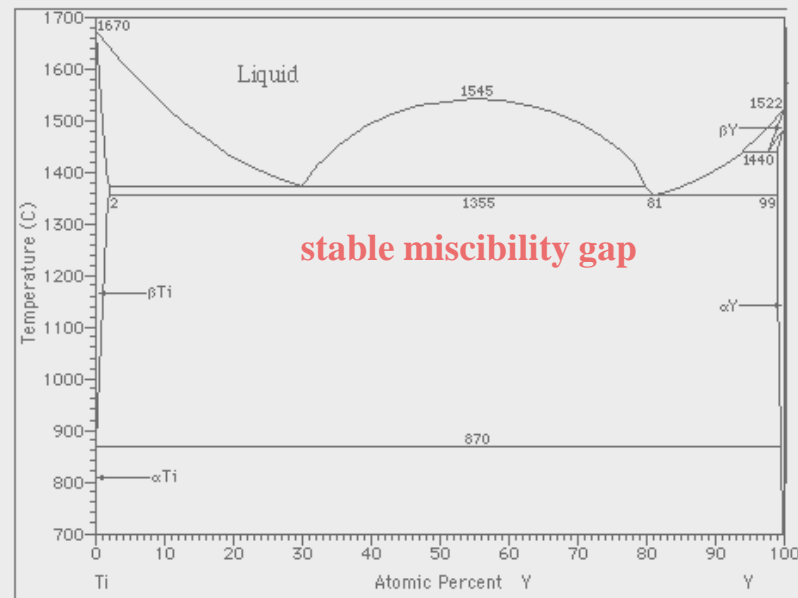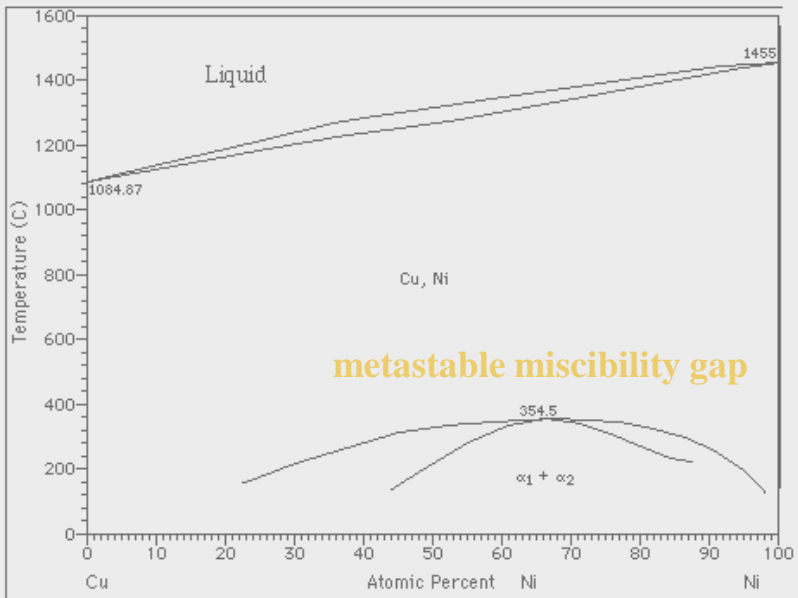
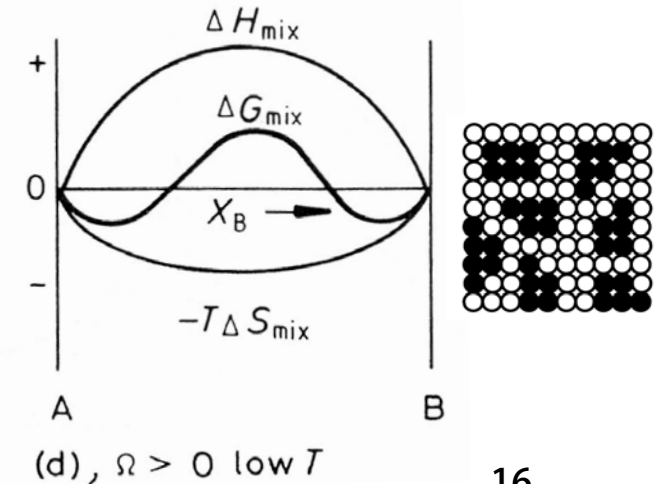$\Delta H_{mix}^{S} < 0$: **Solid solution → ordered phase**
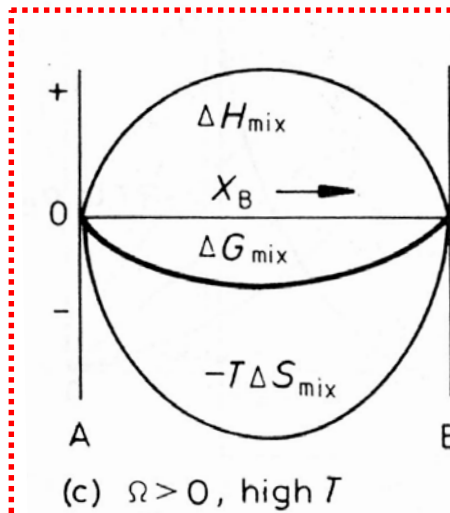
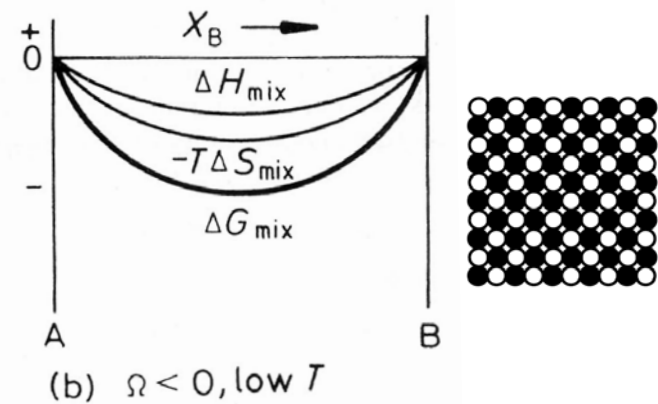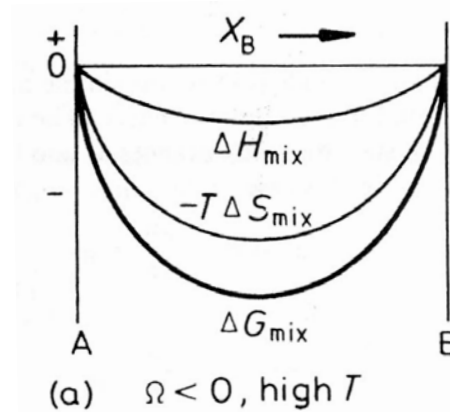$\Delta H_{mix}^{S} << 0$: **Compound : AB, A₂B…**
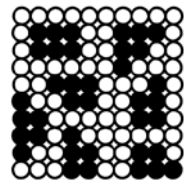
Q1: "Clustering"? → Phase separation

Metastable vs Stable miscibility gap

* The degree of ordering or clustering will decrease as temp. increases due to the increasing importance of entropy.
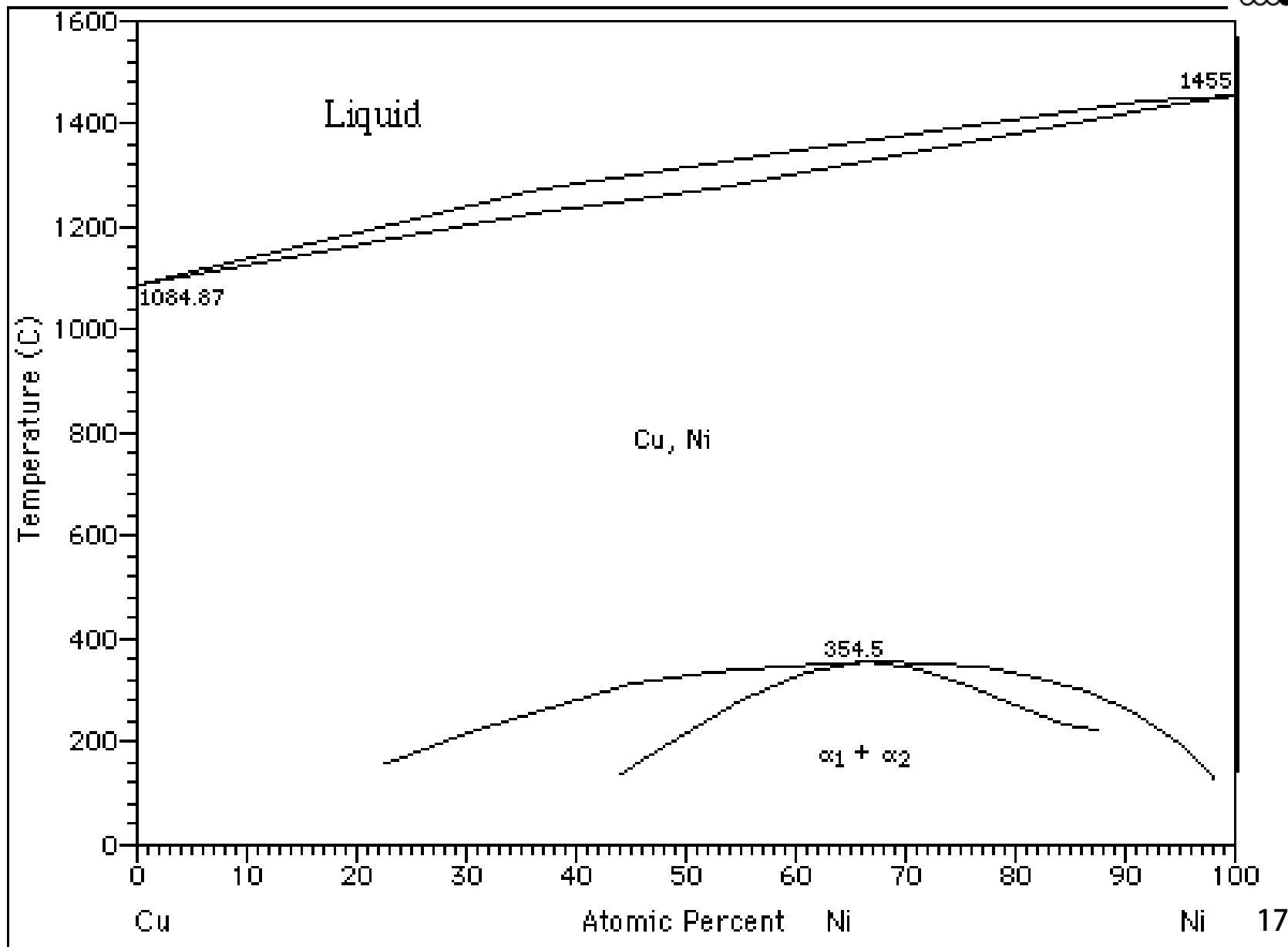
High temp. $\longrightarrow$ Entropy effect $\Uparrow$ $\longrightarrow$ Solution stability $\Uparrow$

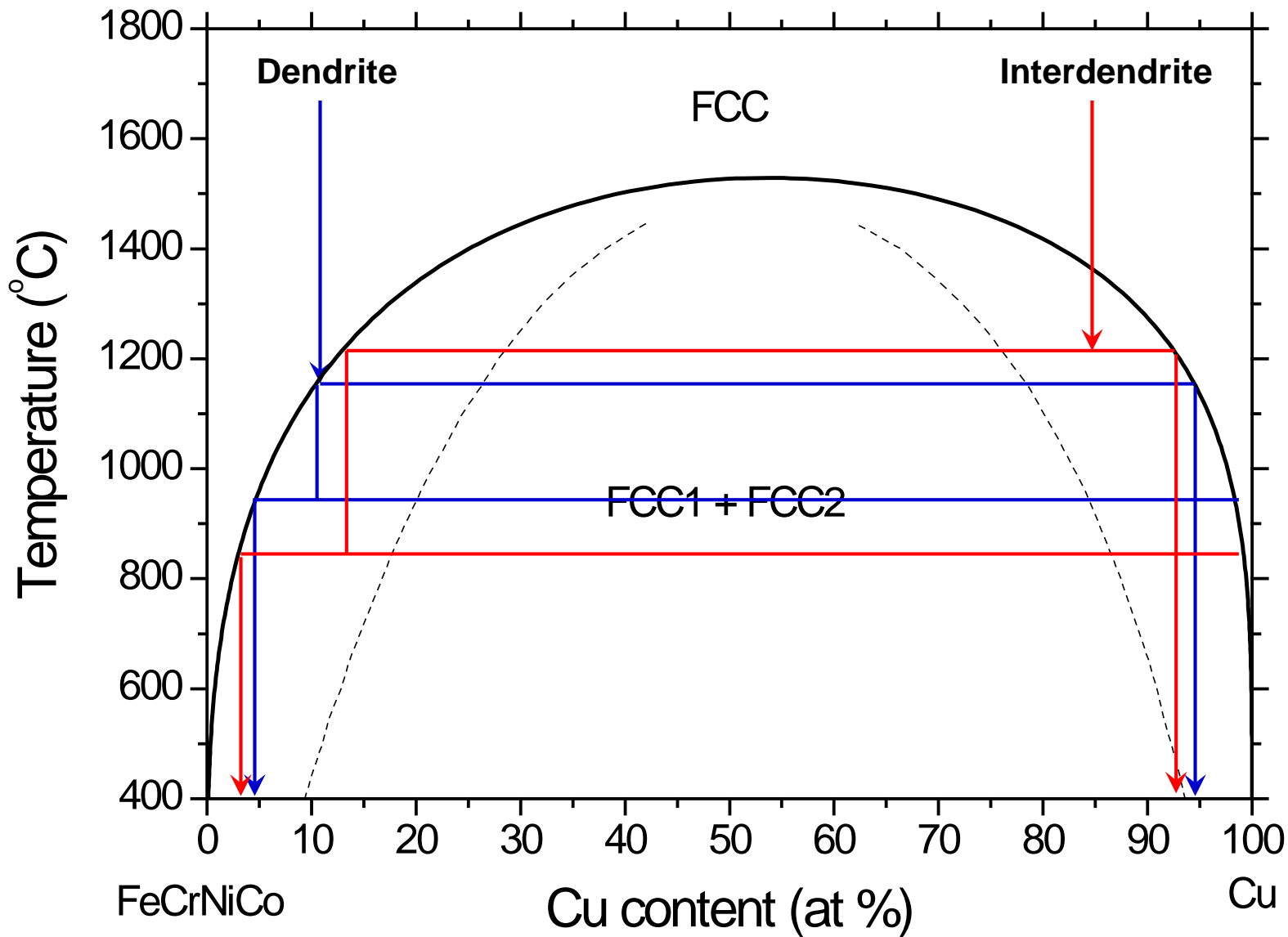$$\Delta G_{mix} = \Delta H_{mix} - T\Delta S_{mix}$$

(a) $\Omega < 0$, high T

(b) $\Omega < 0$, low T

(c) $\Omega > 0$, high T

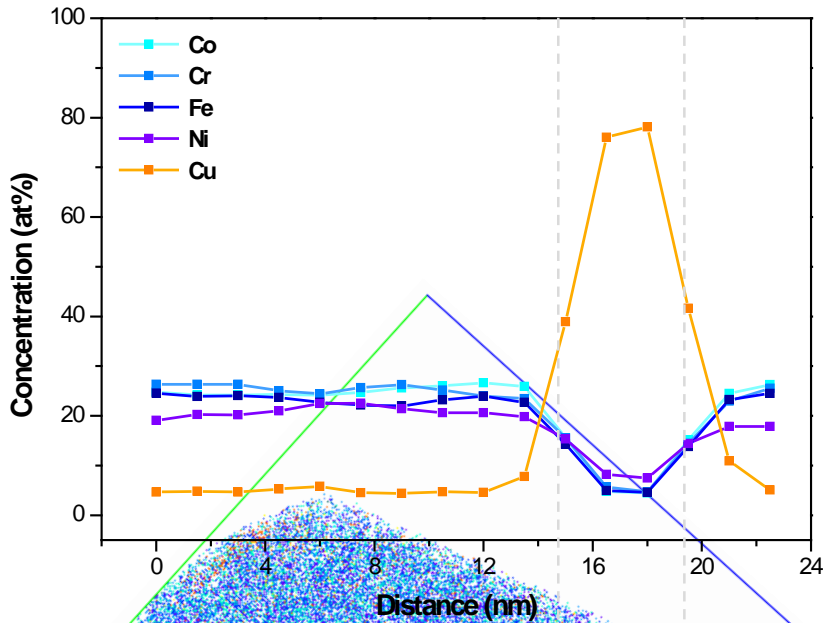(d), $\Omega > 0$ low T

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Cooling process in the miscibility gap

Dendrite / interdendrite formation

Compositional analysis of as-cast CoCrFeNi/Cu HEA (dendrite)

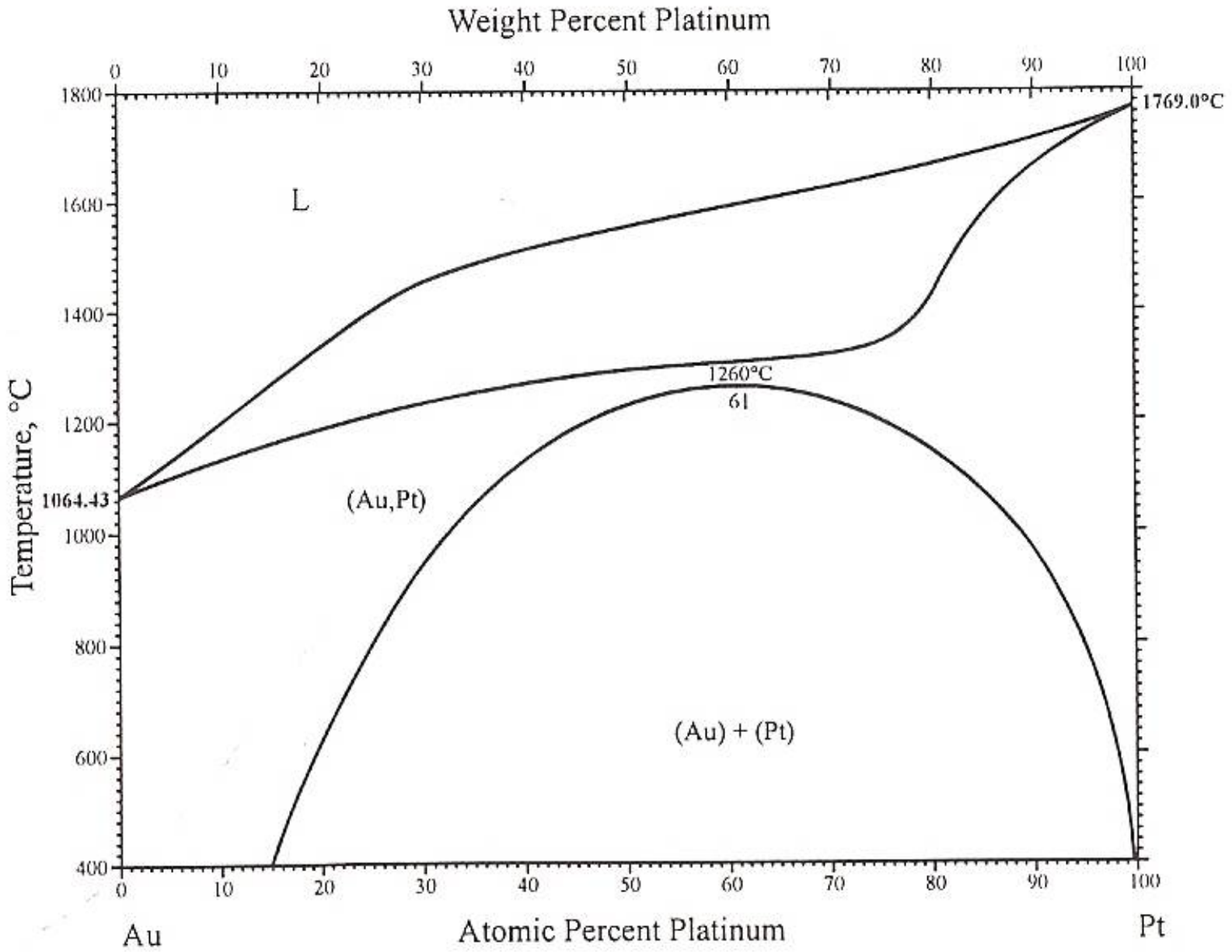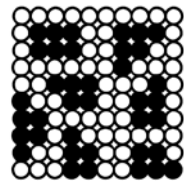ROI 1, 2 : 1.4 nm x 2 nm x 2 nm

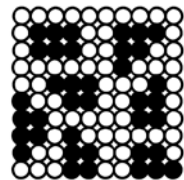ROI 3   : 1.2 nm x 2 nm x 23 nm

(1D concentration profile)

1	at%	2	at%
Co	26.19	Co	0.33
Cr	24.15	Cr	0.46
Fe	24.59	Fe	0.39
Ni	19.59	Ni	5.00
Cu	4.74	Cu	93.56

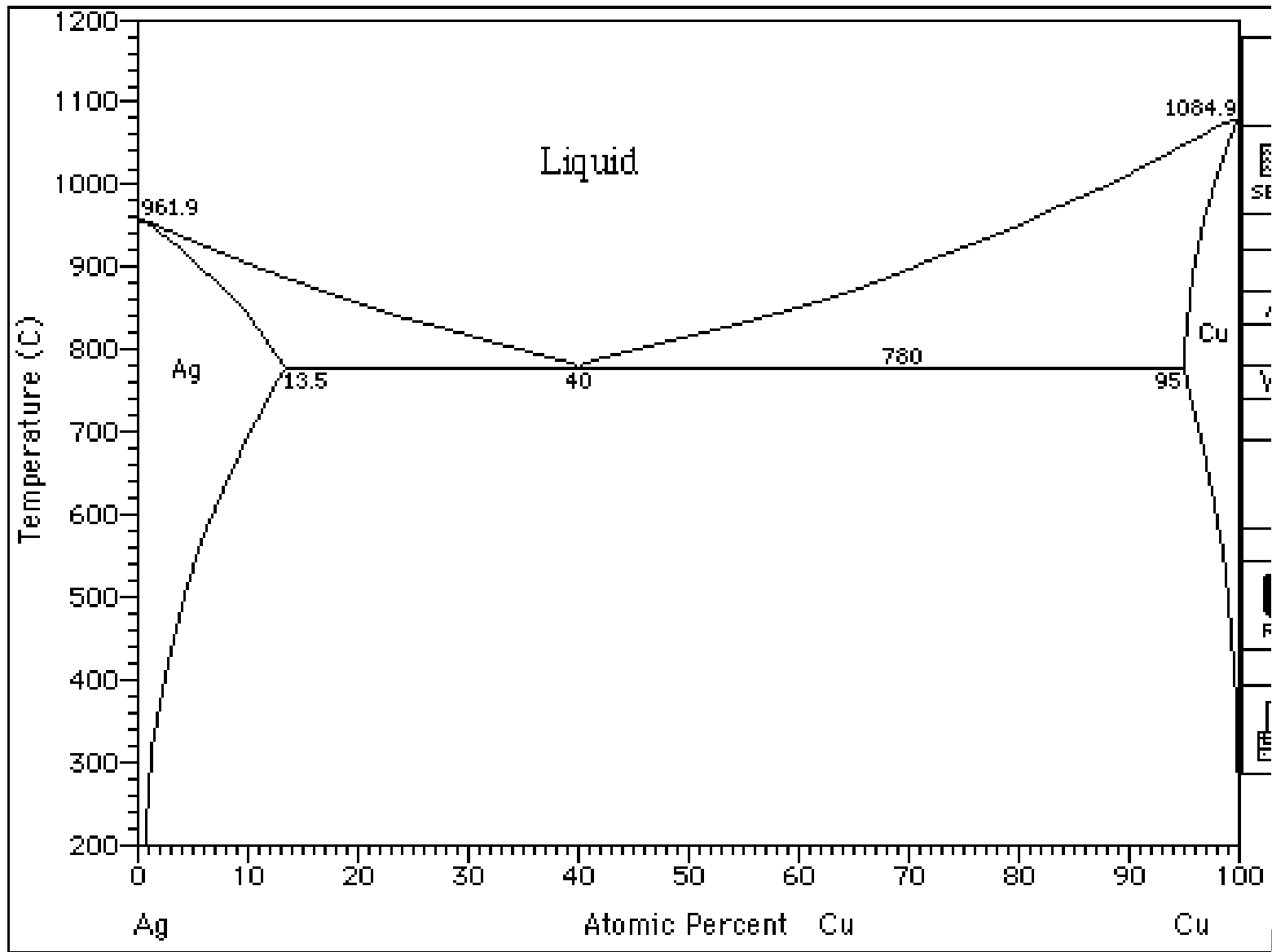| 3-1'	at%	3-2'	at%
Co	25.29	Co	2.01
Cr	25.63	Cr	3.35
Fe	23.63	Fe	2.56
Ni	20.66	Ni	6.90
Cu	4.42	Cu	84.92

- **Dendrite region: matrix (4.74 at%Cu) + 2nd phase (93.56 at%Cu)**

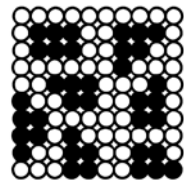- **No segregation at the interface between Matrix and 2nd phase**

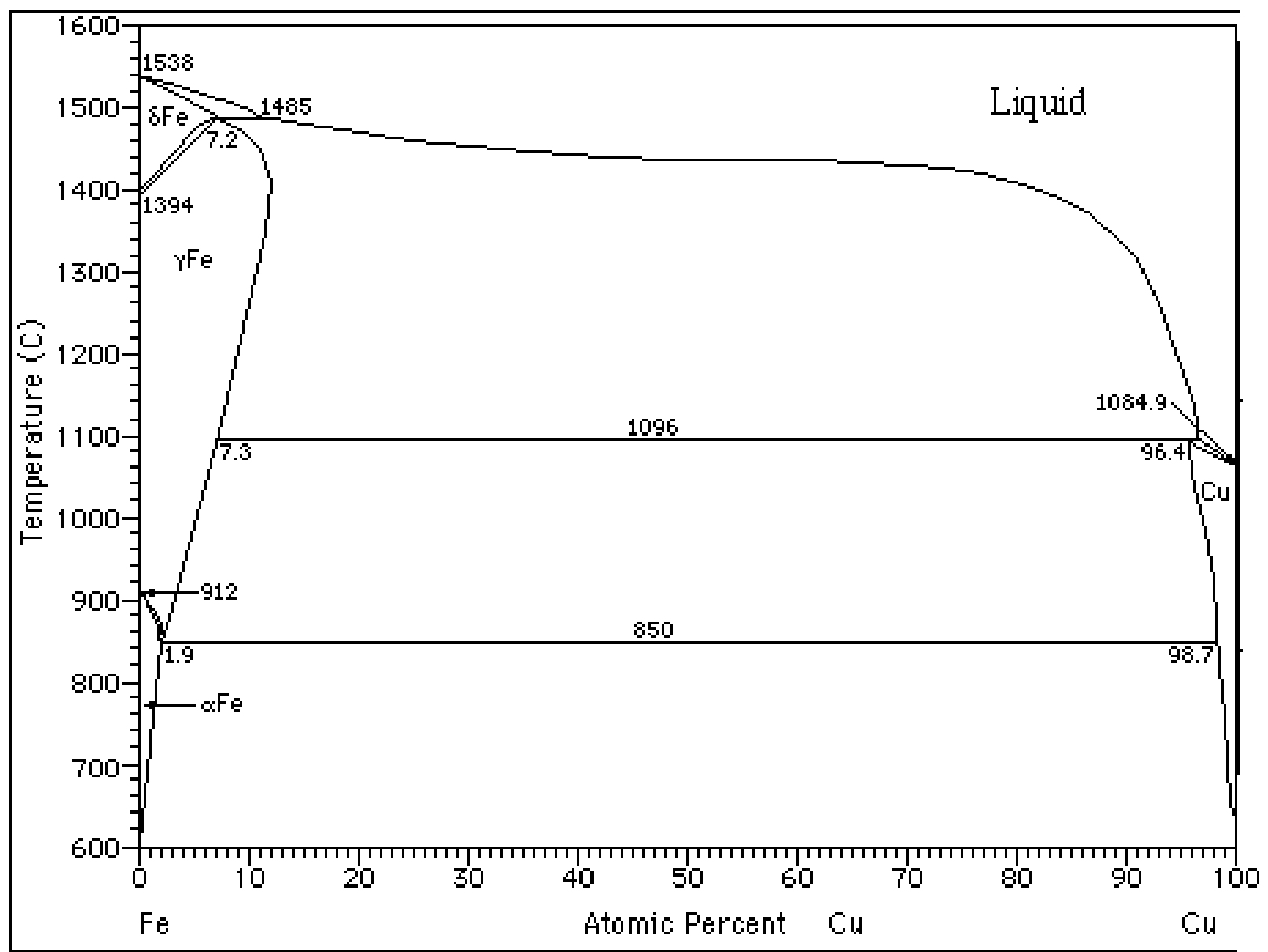$\varepsilon > 0, \ \Delta H_{mix} > 0 \ / \quad \Delta H_{mix} \sim \textbf{+17} \ \textbf{kJ/mol}$
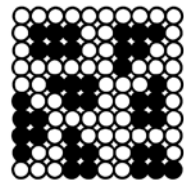
Weight Percent Platinum

Temperature, °C

1769.0°C

L

1260°C
61

(Au,Pt)

1064.43

(Au) + (Pt)

Atomic Percent Platinum

Au

Pt

20

$\epsilon > 0, \quad \Delta H_{mix} > 0 \, / \, \Delta H_{mix} \sim +5 \text{ kJ/mol}$

ε >> 0, ΔH$_{mix}$ >> 0 / ΔH$_{mix}$ ~ +60 kJ/mol

ε >> 0, ΔH$_{mix}$ >> 0 / ΔH$_{mix}$ ~ +58 kJ/mol

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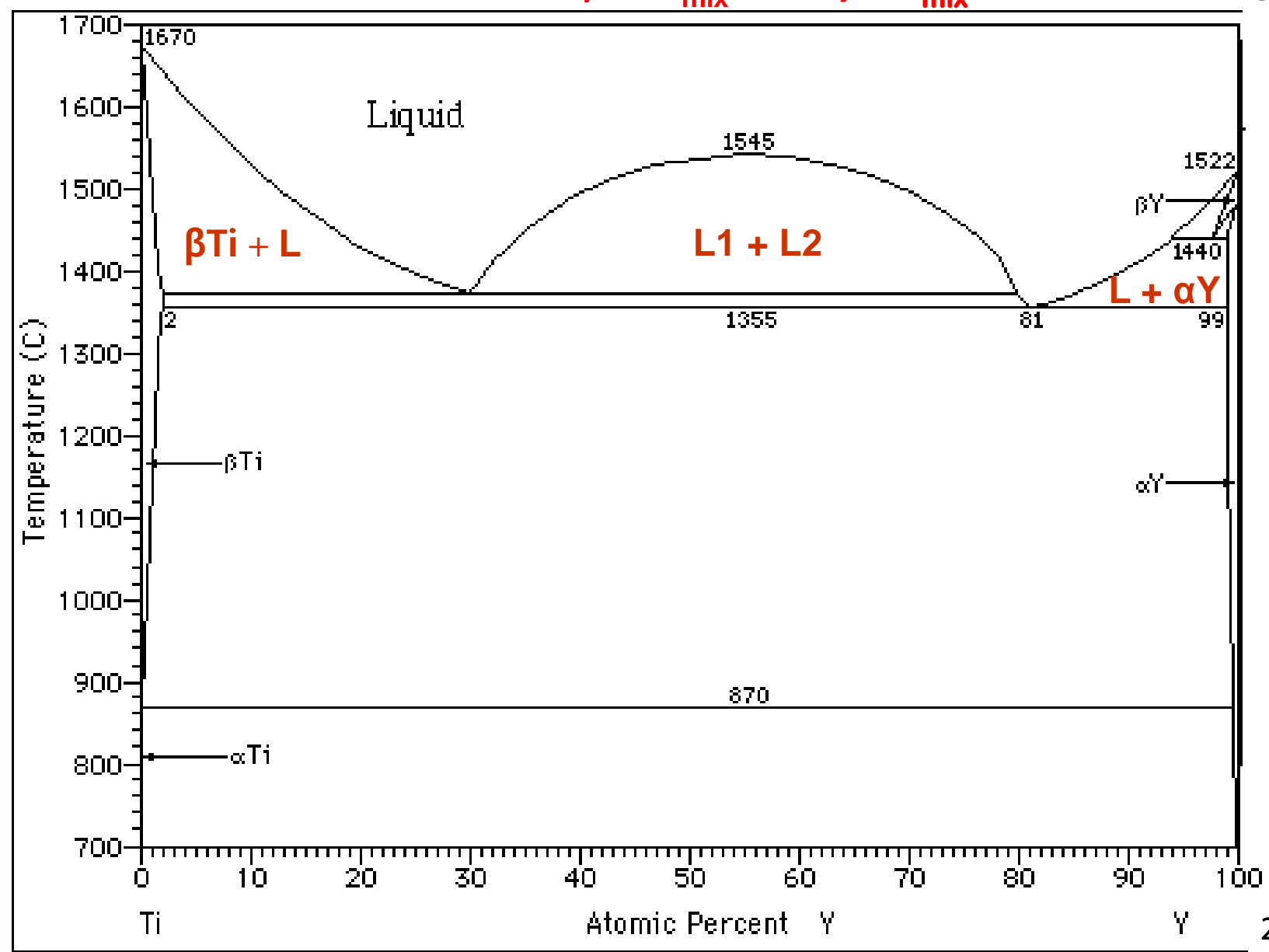
Positive heat of mixing relation among constituent elements

▷ **Alloy design considering heat of mixing relation among constituent elements**
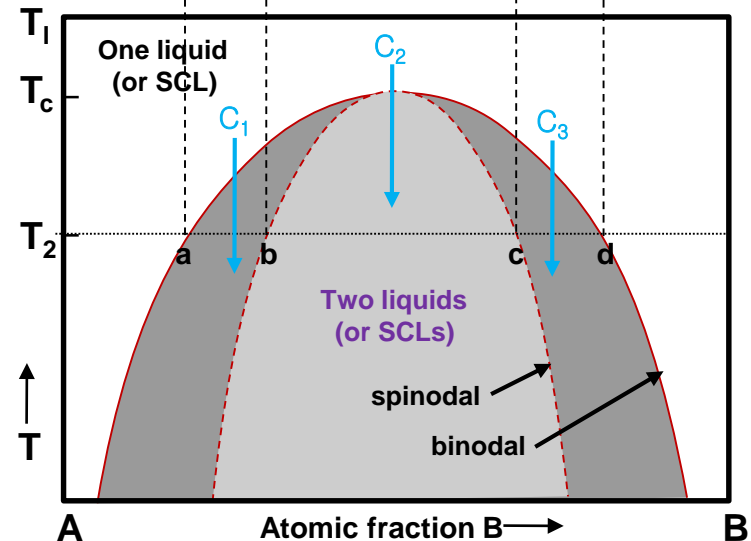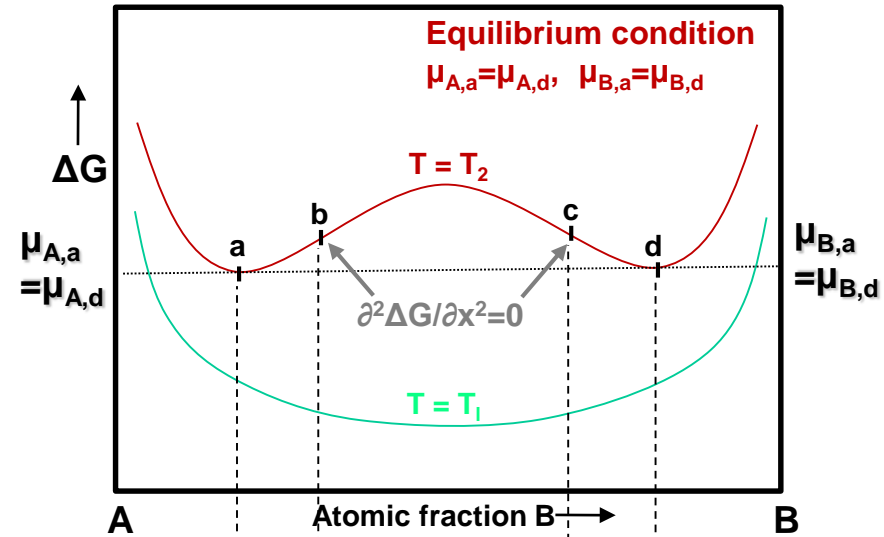
$$\Delta H_{mix} \gg 0 \text{ between A \& B}$$

⬇
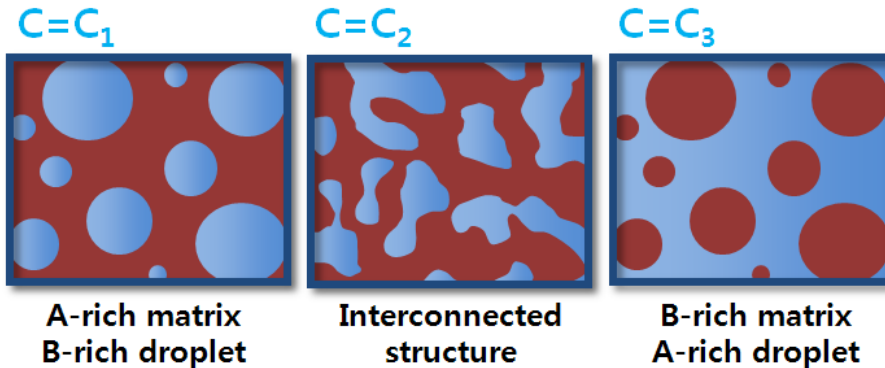
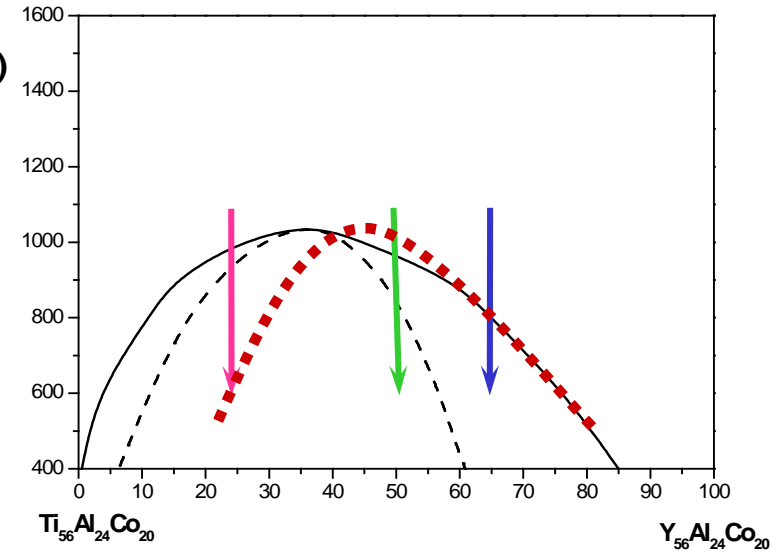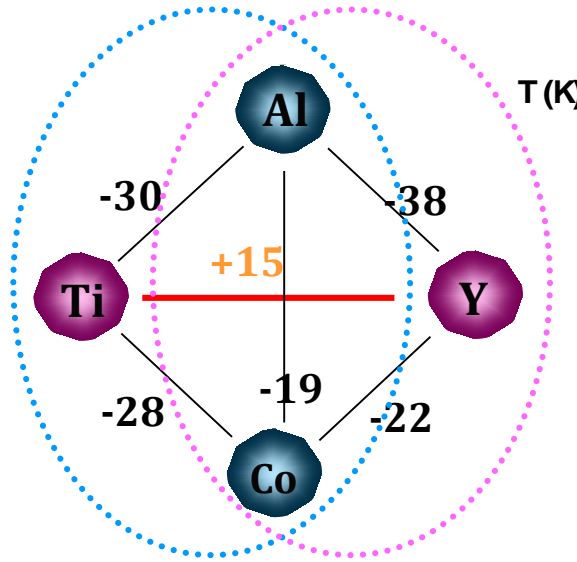**creates (meta)stable miscibility gap in limited composition range**

⬇

Phase separation to A-rich & B-rich phase

▷ **Different two-phase structure by initial composition before phase separation**

C=C₁   C=C₂   C=C₃

A-rich matrix B-rich droplet   **Interconnected structure**   **B-rich matrix A-rich droplet**

Equilibrium condition $\mu_{A,a}=\mu_{A,d}, \ \mu_{B,a}=\mu_{B,d}$

ΔG

$T = T_2$

b   a   c   d

$\mu_{A,a}=\mu_{A,d}$ $\mu_{B,a}=\mu_{B,d}$

$\partial^2\Delta G/\partial x^2=0$

$T = T_l$

A   Atomic fraction B⟶   B

T_l T_c T_2 T

One liquid (or SCL) C_2 C_1 C_3

a   b   c   d

Two liquids (or SCLs)

spinodal

binodal

A   Atomic fraction B⟶   B

Nucleation and growth ↔ Spinodal decomposition without any barrier to the nucleation process

* Ti-Y-Al-Co system

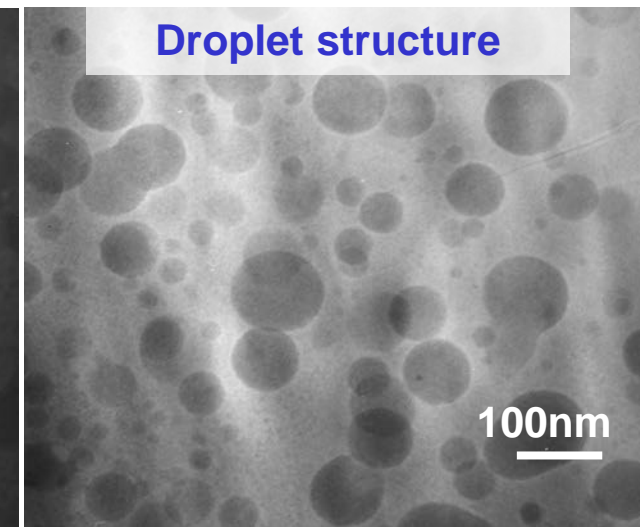$(Y_{56}Al_{24}Co_{20})_{25}(Ti_{56}Al_{24}Co_{20})_{75}$

$(Y_{56}Al_{24}Co_{20})_{50}(Ti_{56}Al_{24}Co_{20})_{50}$

$(Y_{56}Al_{24}Co_{20})_{65}(Ti_{56}Al_{24}Co_{20})_{35}$

* La-Zr-Al-Cu-Ni system

FIGURE 5.17
Schematic of the miscibility gap and the sequence of phase formation during cooling in the La–Zr–Al–Cu–Ni system. The positions of letters (a) to (d) in the diagram on the left correspond to the schematic microstructures (a) to (d) on the right. (Reprinted from Kündig, A.A. et al., *Acta Mater.*, 52, 2441, 2004. With permission.)

Phase separation in metallic glasses
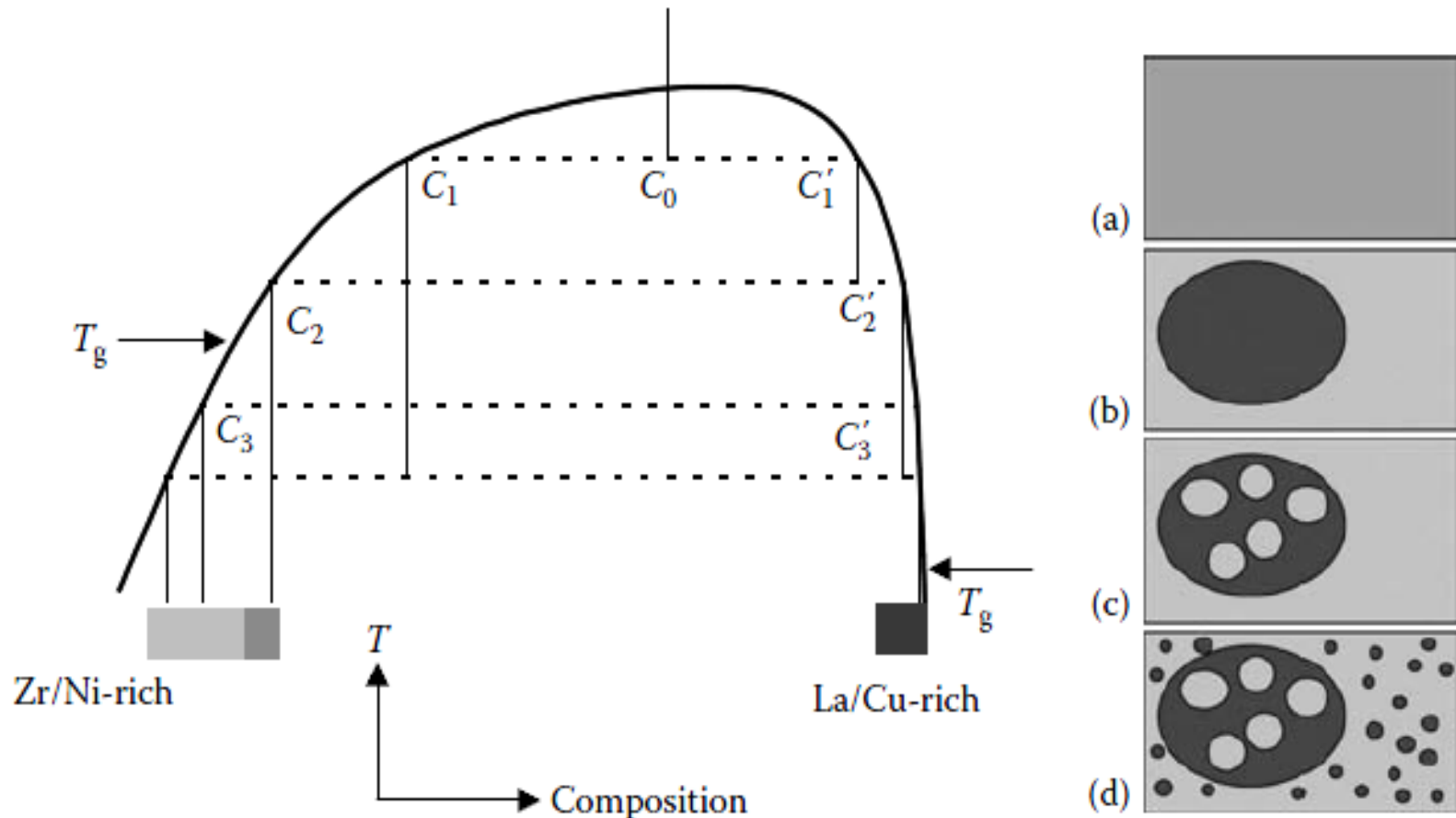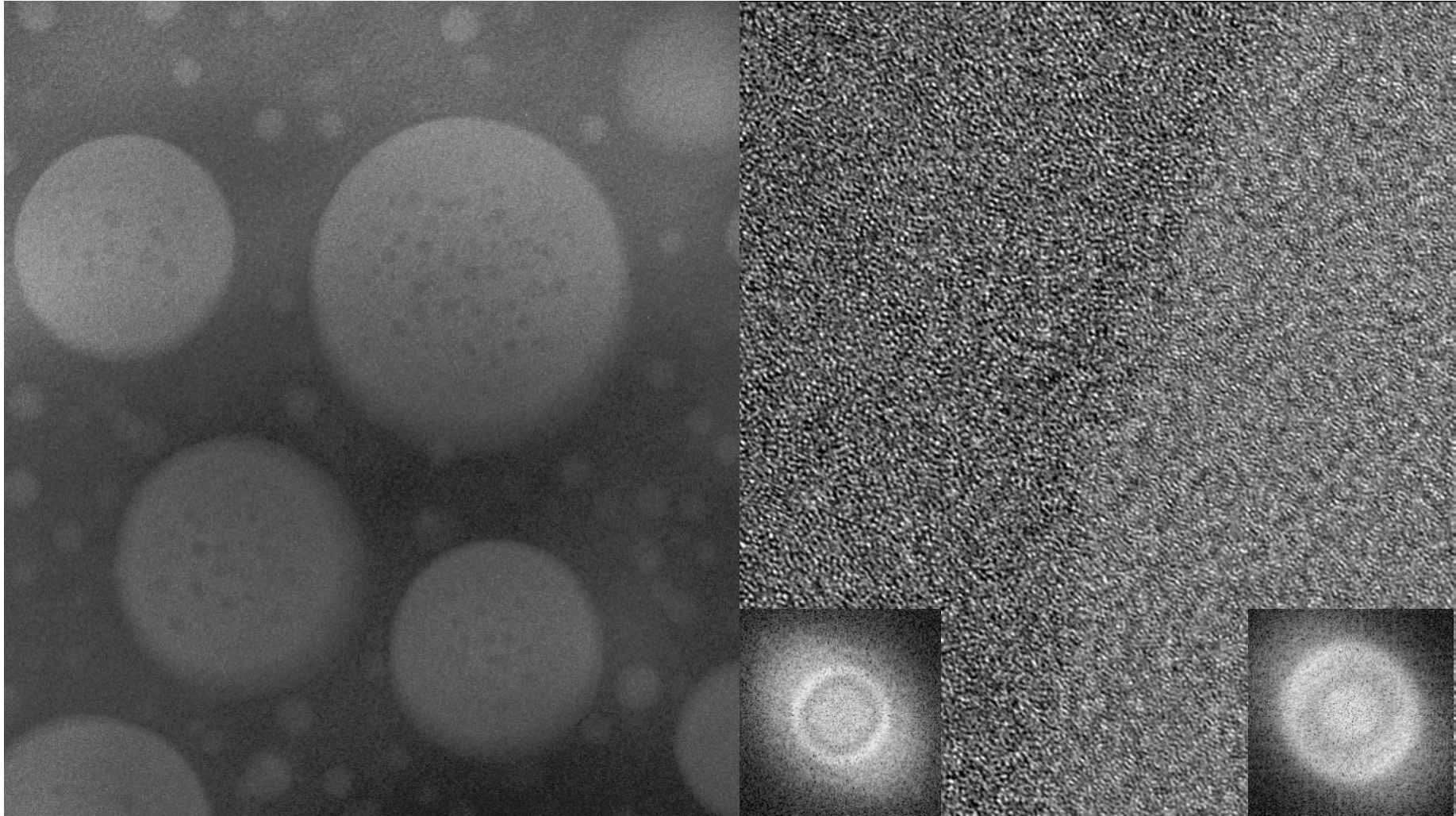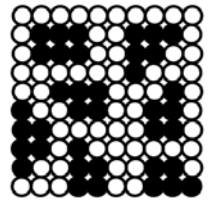
- **Binary System** mixture/ solution / compound

Ideal solution $(\Delta H_{mix} = 0)$ **Random distribution**

Regular solution $\boxed{\Delta H_{mix} = P_{AB}\varepsilon}$ where $\varepsilon = \varepsilon_{AB} - \dfrac{1}{2}(\varepsilon_{AA} + \varepsilon_{BB})$ $\boxed{\varepsilon \approx 0}$

⇕ $\Delta H_{mix} > 0$ or $\Delta H_{mix} < 0$

Real solution   **Ordered structure**

(a) $\varepsilon < 0$, $\Delta H_{mix} < 0$ (b) $\varepsilon > 0$, $\Delta H_{mix} > 0$ (c) **when the size difference is large**

Ordered alloys   **Clustering**   **strain effect**

$P_{AB} \uparrow \longrightarrow$ **Internal E** $\downarrow$ $P_{AA},\ P_{BB} \uparrow$ **Interstitial solution**

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$\Delta H_{mix}^{S} > 0$: **Solid solution → solid state phase separation (two solid solutions)**

$\Delta H_{mix}^{S} \gg 0$: **liquid state phase separation (up to two liquid solutions)**

metastable miscibility gap

stable miscibility gap
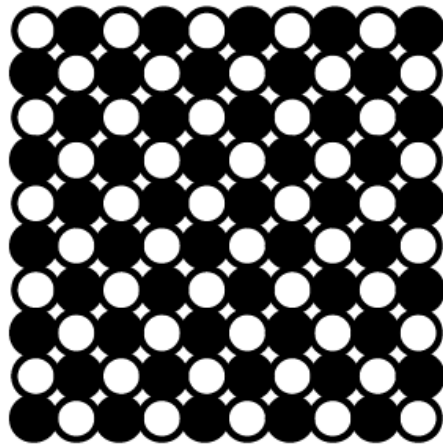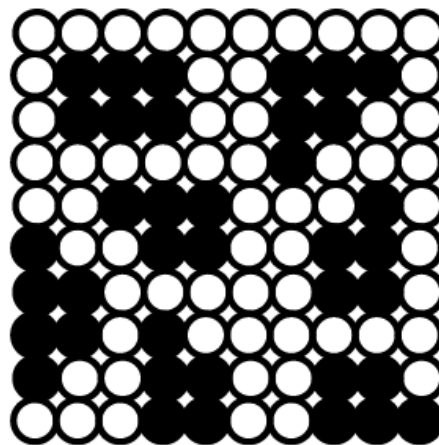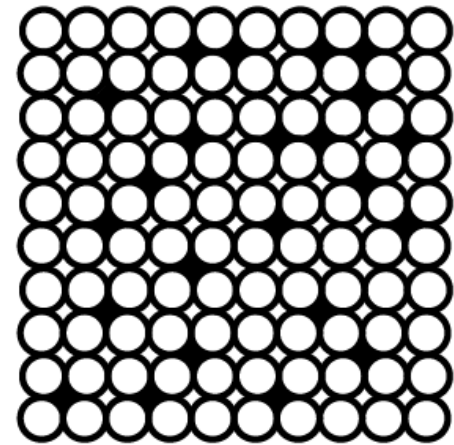
$\Delta H_{mix}^{S} < 0$: **Solid solution → ordered phase**

$\Delta H_{mix}^{S} \ll 0$: **Compound : AB, A$_2$B…**

Q2: How can we define equilibrium in heterogeneous systems?

Equilibrium in Heterogeneous Systems

A, B different crystal structure → two free energy curves must be drawn, one for each structure.

We have dealt with the case where the components A and B have the same crystal structure.

$$G = X_A G_A + X_B G_B$$
$$+ \Omega X_A X_B + RT(X_A \ln X_A + X_B \ln X_B)$$

What would happen when the components A and B have a different crystal structure?
→ heterogeneous system

(a)

(b)

Ex (fcc)   Ex (bcc)

31
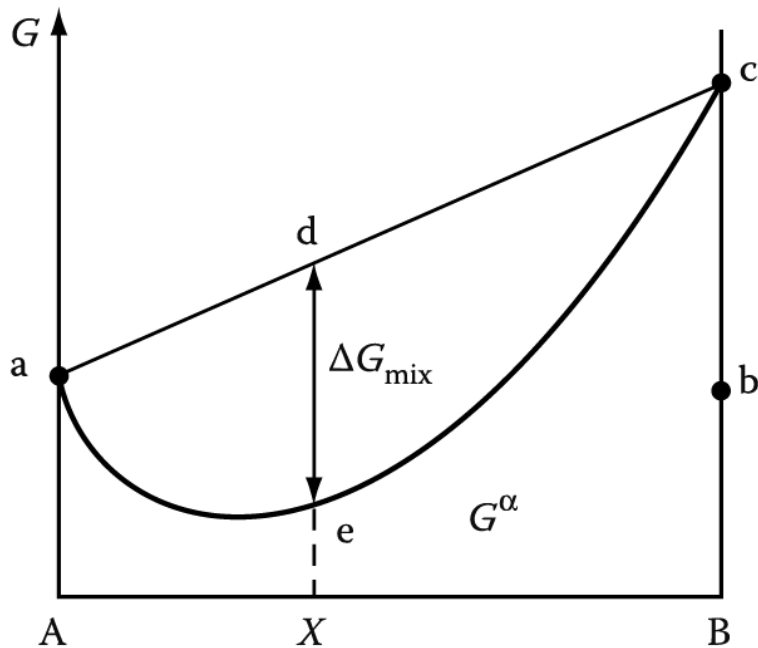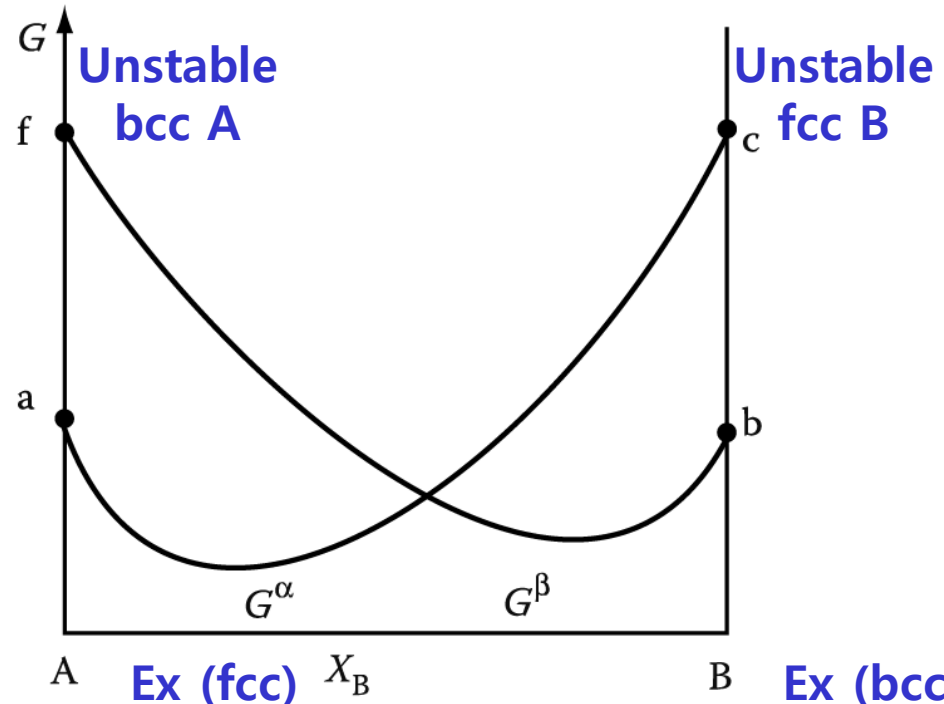
Equilibrium in Heterogeneous Systems

If $G^{\alpha}(X_B^{\alpha})$ and $G^{\beta}(X_B^{\beta})$ are given,
what would be $G(\alpha + \beta)$ *at* $X_B^o = ?$

- bcg & acd, deg & dfc
  : similar triangles

- Lever rule

- 1 mole (α+β)
  : bc/ac mol of α + ab/ac mol of β
  → bg + ge = be_total G of 1 mol
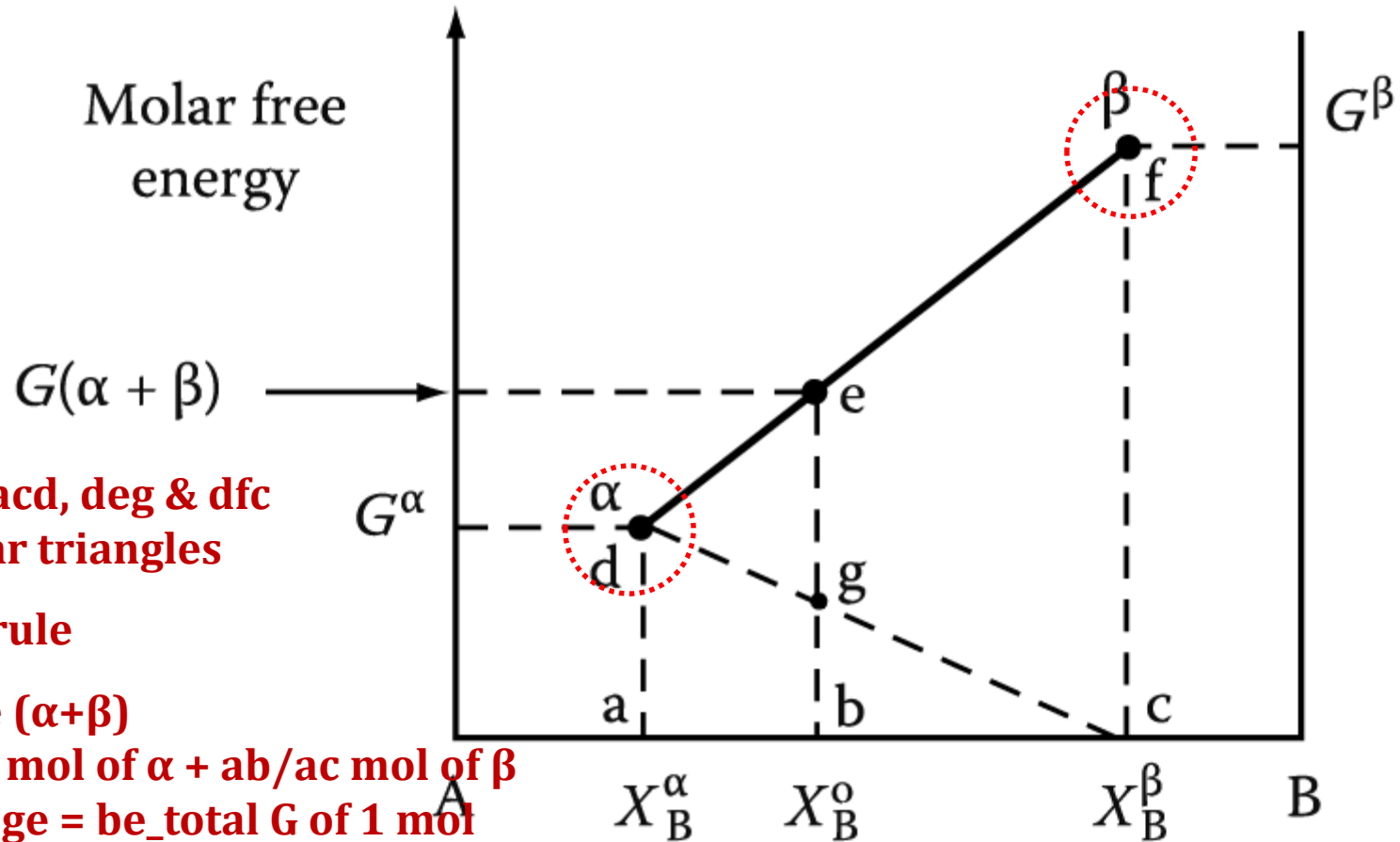
Fig. 1.26 The molar free energy of a two-phase mixture (α+β)

Lever rule

A geometric interpretation:
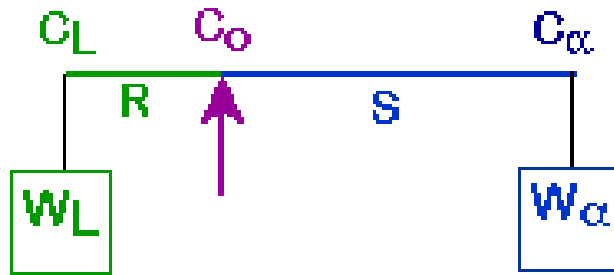
moment equilibrium:

$$W_L R = W_\alpha S$$

$$1 - W_\alpha$$

solving gives Lever Rule

Sum of weight fractions: $W_L + W_\alpha = 1$

Conservation of mass (Ni): $C_O = W_L C_L + W_\alpha C_\alpha$

Combine above equations:

$$W_L = \frac{C_\alpha - C_O}{C_\alpha - C_L} = \frac{S}{R + S}$$

$$W_\alpha = \frac{C_O - C_L}{C_\alpha - C_L} = \frac{R}{R + S}$$
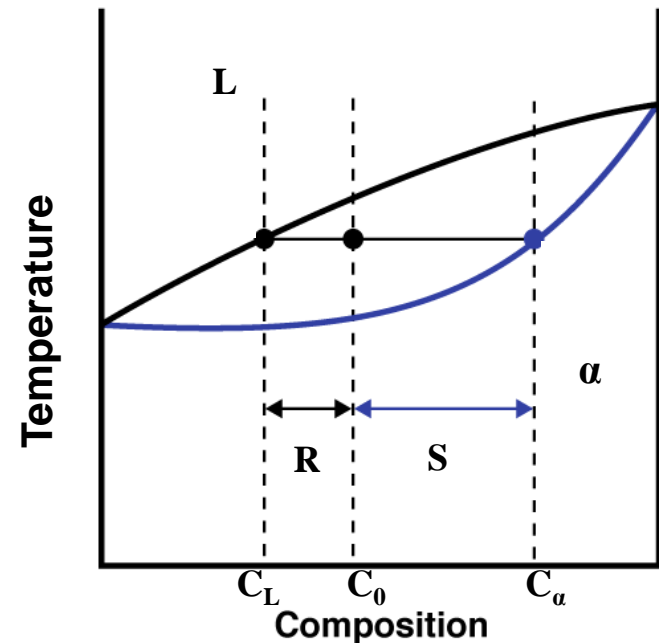
Equilibrium in Heterogeneous Systems

In X^0, $G_0^\beta > G_0^\alpha > G_1$

Exchange of A and B atoms

➡ **α + β phase separation**

Chemical Equilibrium (μ, a)
→ multiphase and multicomponent
($\mu_i^\alpha = \mu_i^\beta = \mu_i^\gamma = \ldots$), ($a_i^\alpha = a_i^\beta = a_i^\gamma = \ldots$)

$$\mu_A^\alpha = \mu_A^\beta \qquad \mu_B^\alpha = \mu_B^\beta$$

Unified Chemical potential of two phases

(a)

(b)

34
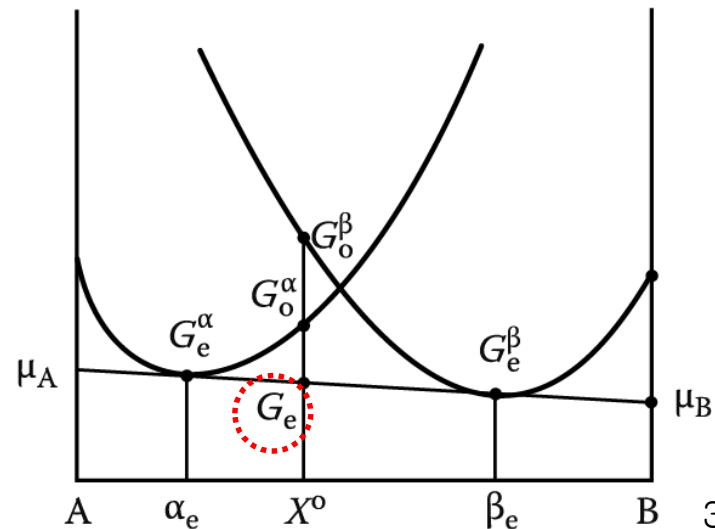
Variation of activity with composition

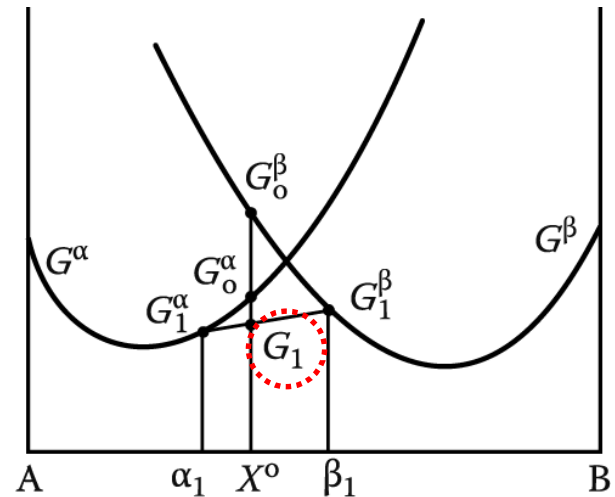The most stable state, with the lowest free energy, is usually defined as the state in which the pure component has unit activity of A in pure α.

when $X_A = 1 \rightarrow a_A^\alpha = 1$

when $X_B = 1 \rightarrow a_B^\beta = 1$

when α and β in equil.

$$a_A^\alpha = a_A^\beta$$

$$a_B^\alpha = a_B^\beta$$

Unified activity of two phase
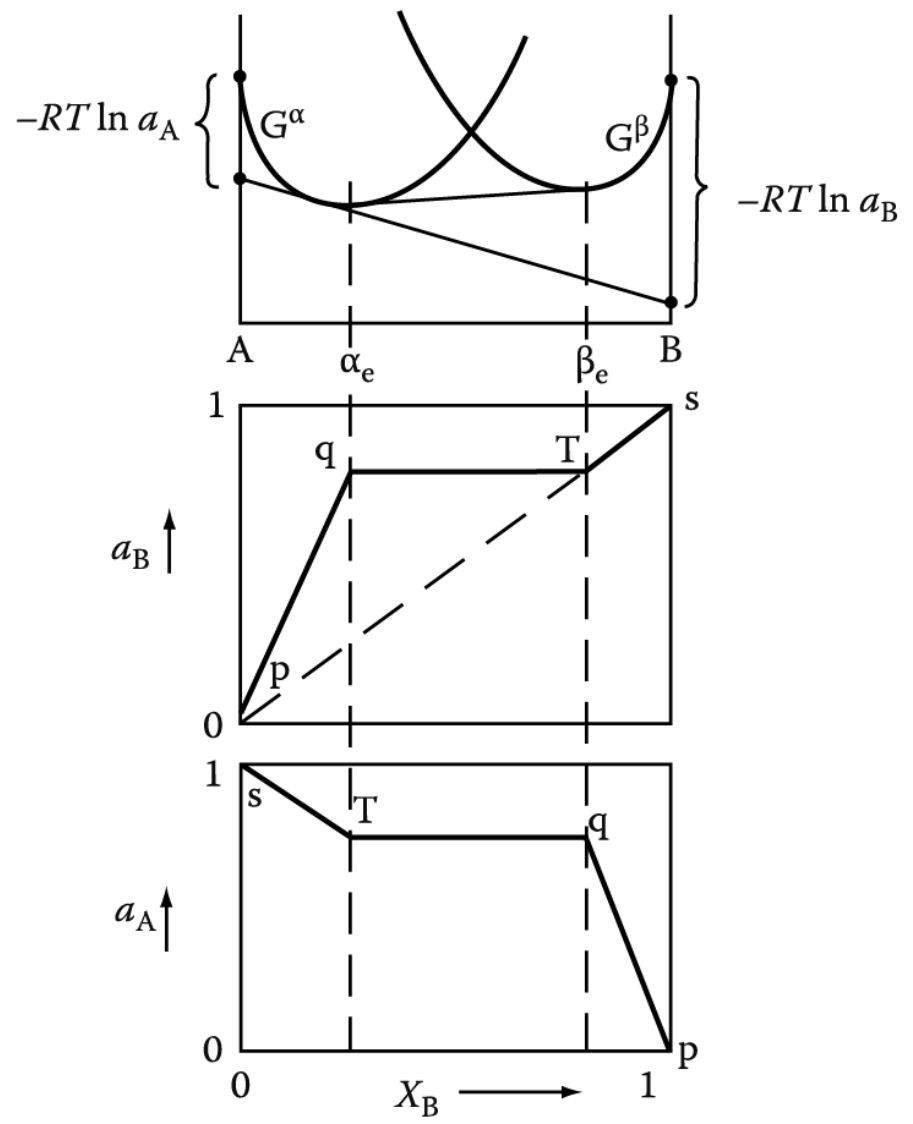
Fig. 1.28 The variation of a_A and a_B with composition for a binary system containing two ideal solutions, α and β

35

Equilibrium in Heterogeneous Systems

In X^0, $G_0^\beta > G_0^\alpha > G_1$ ⟹ α + β separation ⟹ unified chemical potential

$\mu_A = \mu_B$

$a_A^\alpha = a_A^\beta$

$-RT \ln a_A^\alpha$

$-RT \ln a_A^\beta$

μ_A

G^α

G_0^β

G_0^α

G_1^α

G_1^β

G_1

G_e

G^β

$a_B^\alpha = a_B^\beta$

$-RT \ln a_B^\alpha$

$-RT \ln a_B^\beta$

μ_B

A α_4 ← α_1 X^0 β_1 → β_4 B

36
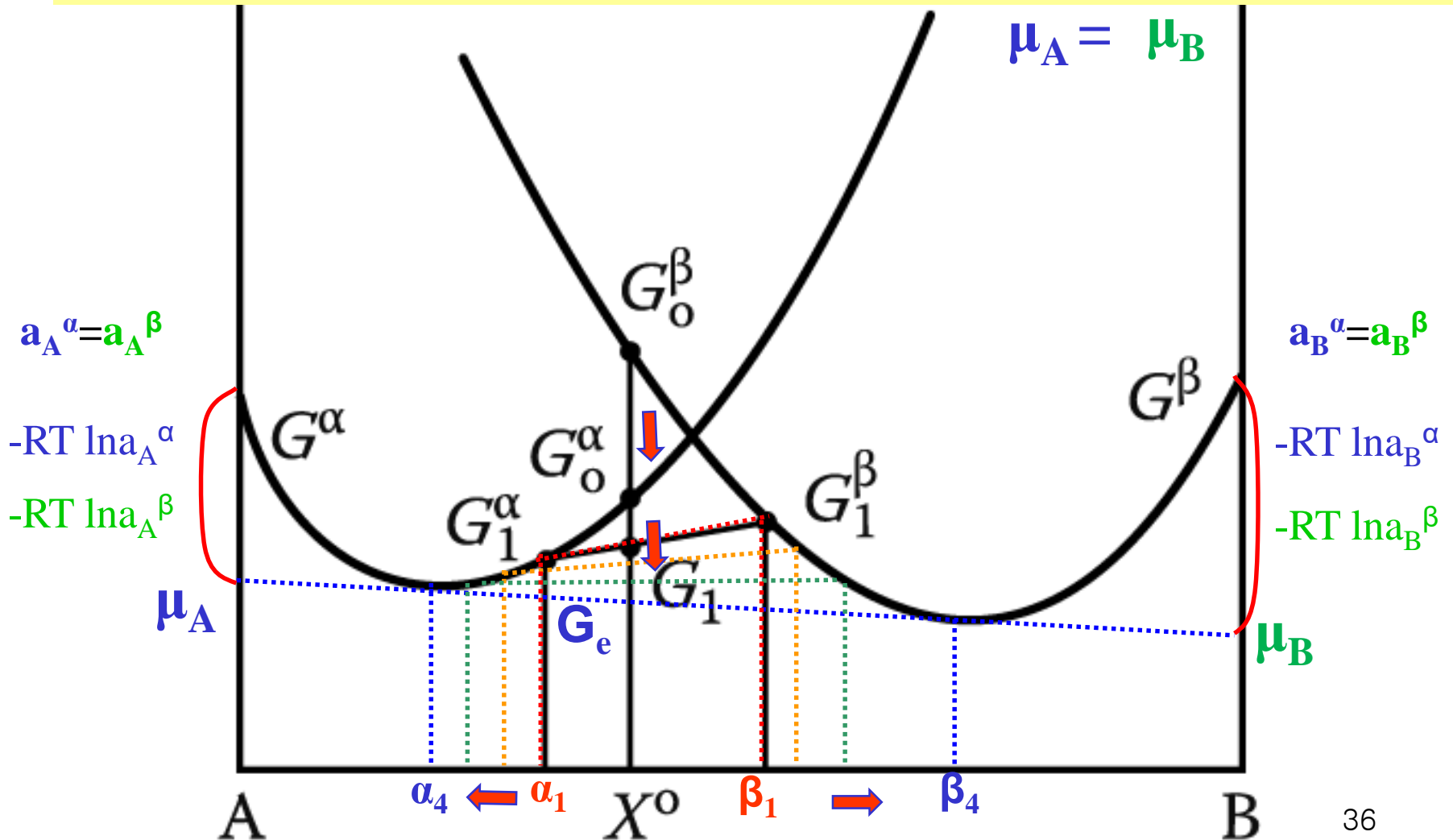
Q3: How equilibrium is affected by temperature in complete solid solution?

1) Simple Phase Diagrams

Assumption: (1) completely miscible in solid and liquid.
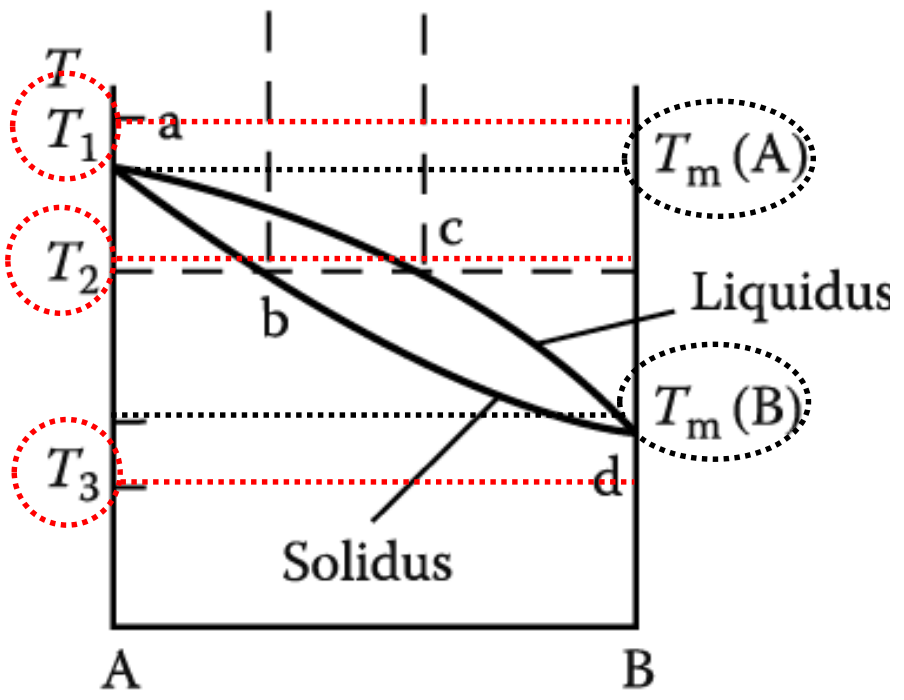**(2) Both are ideal soln.** $\Delta H^{L}_{mix} = 0 \quad \Delta H^{S}_{mix} = 0$
(3) $T_m(A) > T_m(B)$
(4) $T_1 > T_m(A) > T_2 > T_m(B) > T_3$

Draw G^L and G^S as a function of composition X_B
at T_1, $T_m(A)$, T_2, $T_m(B)$, and T_3.
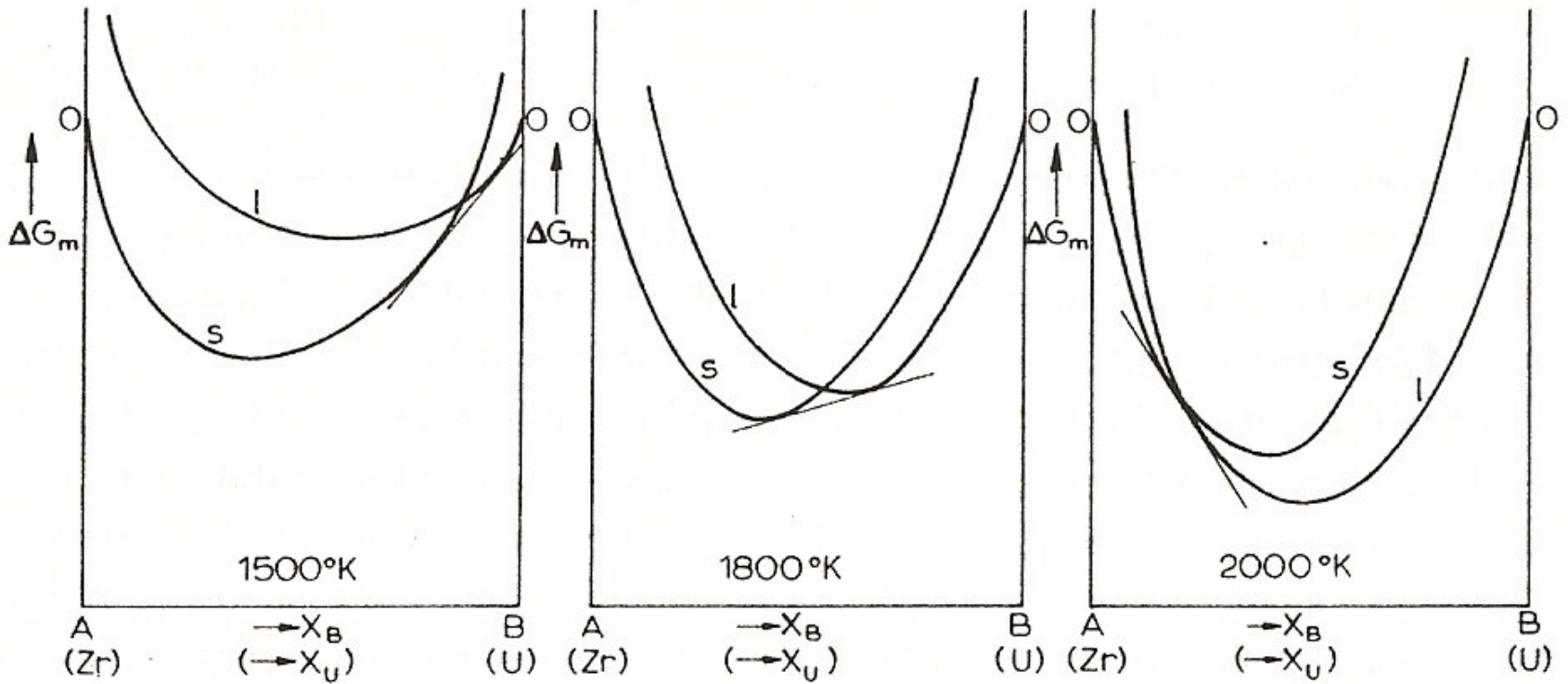

(f)

1) Simple Phase Diagrams

Fig. 26. Free energy curves for liquid and solid phases in the U–Zr system at 1500°, 1800° and 2000 °K.

1.5 Binary phase diagrams

1) Simple Phase Diagrams

1) **Variation of temp.: $G^L > G^s$**
2) **Decrease of curvature of G curve**
   (∵ decrease of $-T\Delta S_{mix}$ effect)

Assumption:

(1) completely miscible in solid and liquid.
(2) Both are ideal soln.
(3) $T_m(A) > T_m(B)$
(4) $T_1 > T_m(A) > T_2 > T_m(B) > T_3$
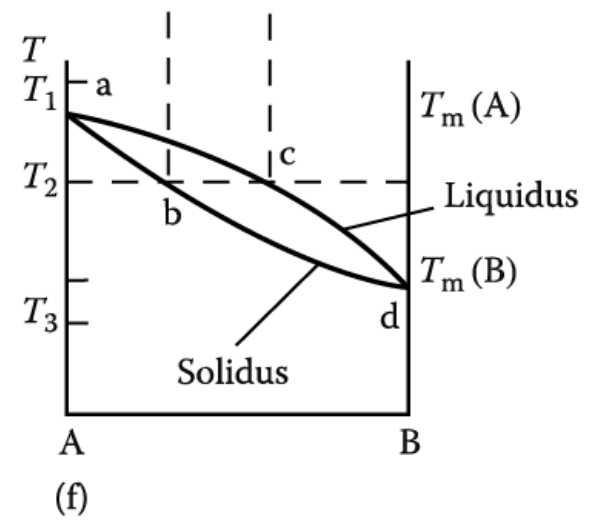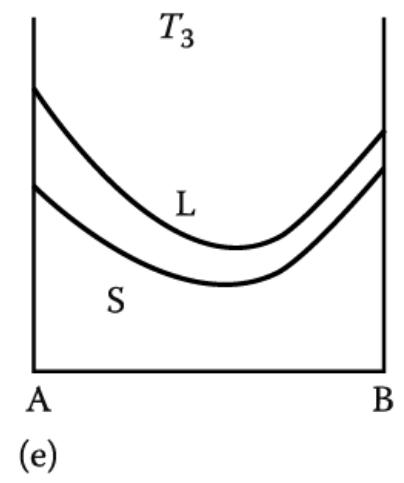
1.5 Binary phase diagrams

1) Simple Phase Diagrams

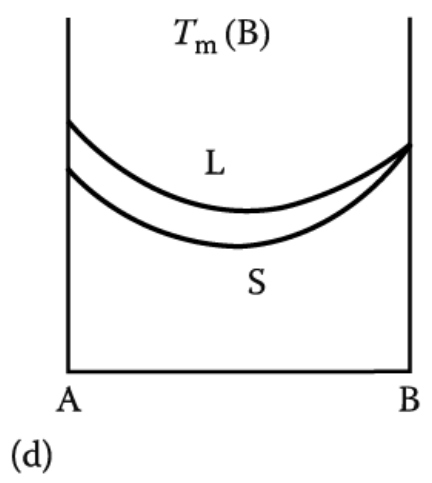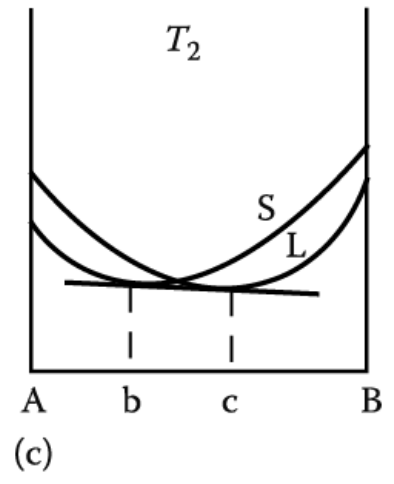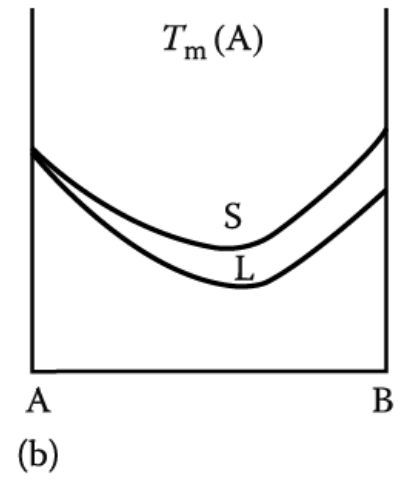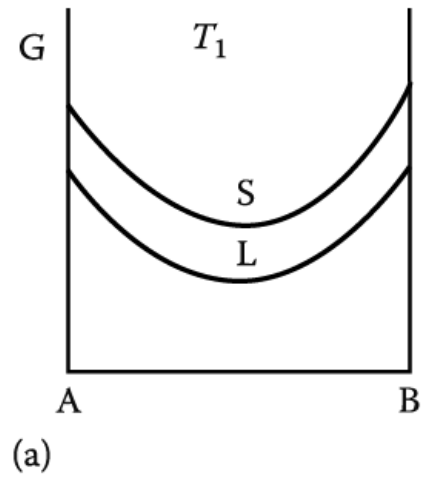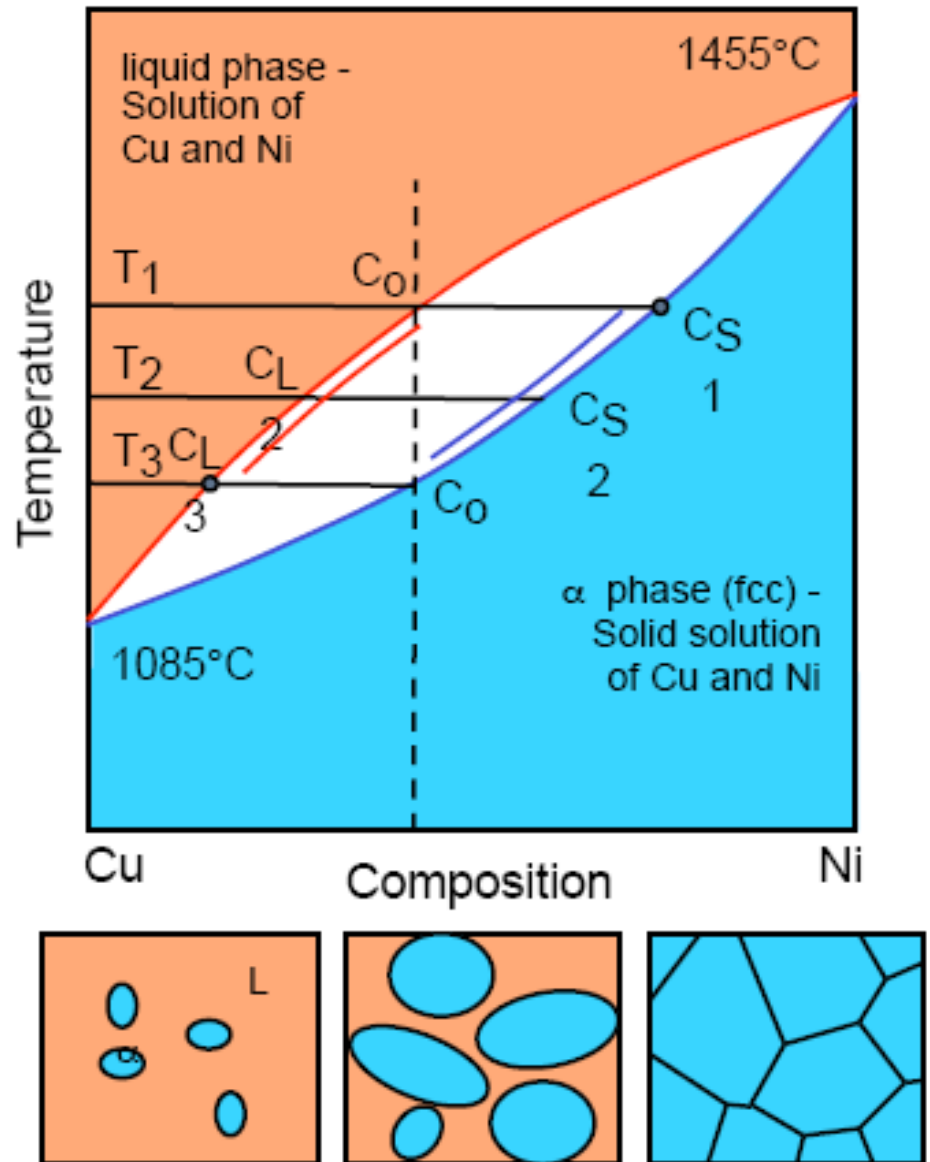The simplest type of binary phase diagrams is the isomorphous system, in which the two constituents form a continuous solid solution over the entire composition range. An example is the Ni-Cu system.

Solidification of alloy C_o starts on cooing at T_1. The first solid formed has a composition of C_{s1} and the liquid C_o. On further cooling the solid particles grow larger in size and change their composition to C_{s2} and then C_o, following the solidus whereas the liquid decrease in volume and changes its composition from C_o to C_{L3} following the liquidus. The solidification completes at T_3.

liquid phase - Solution of Cu and Ni

1455°C

T_1 C_o

C_S 1

T_2 C_L

C_S 2

$T_3 C_L$ 2

3

C_o

α phase (fcc) – Solid solution of Cu and Ni

1085°C

Temperature

Cu   Composition   Ni

L

Cooling Curves
determination of Phase diagrams
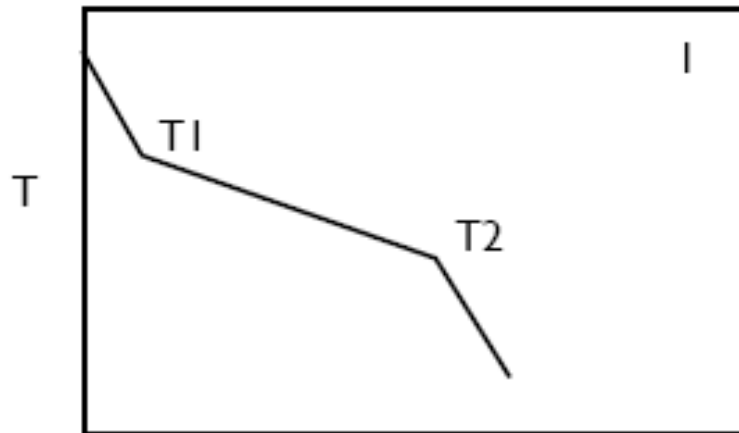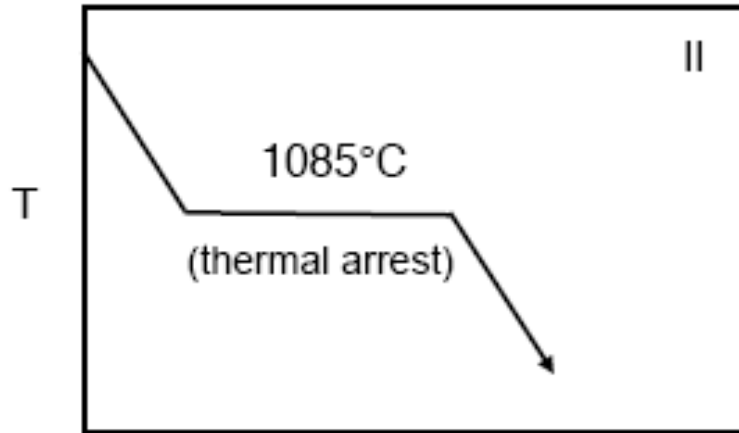
Q4: How equilibrium is affected by temperature in systems with miscibility gap?

2) Systems with miscibility gab

$$\Delta H^{L}_{mix} = 0 \qquad \Delta H^{S}_{mix} > 0$$
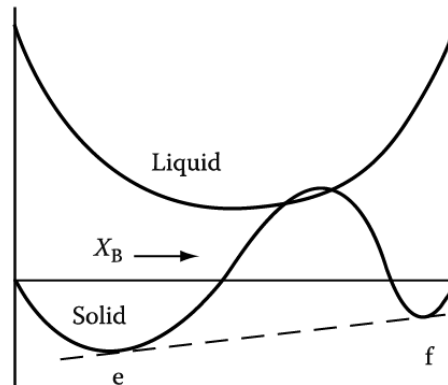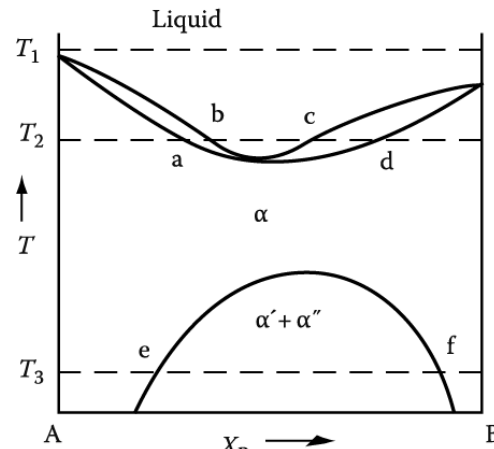
congruent minima

How to characterize Gˢ mathematically
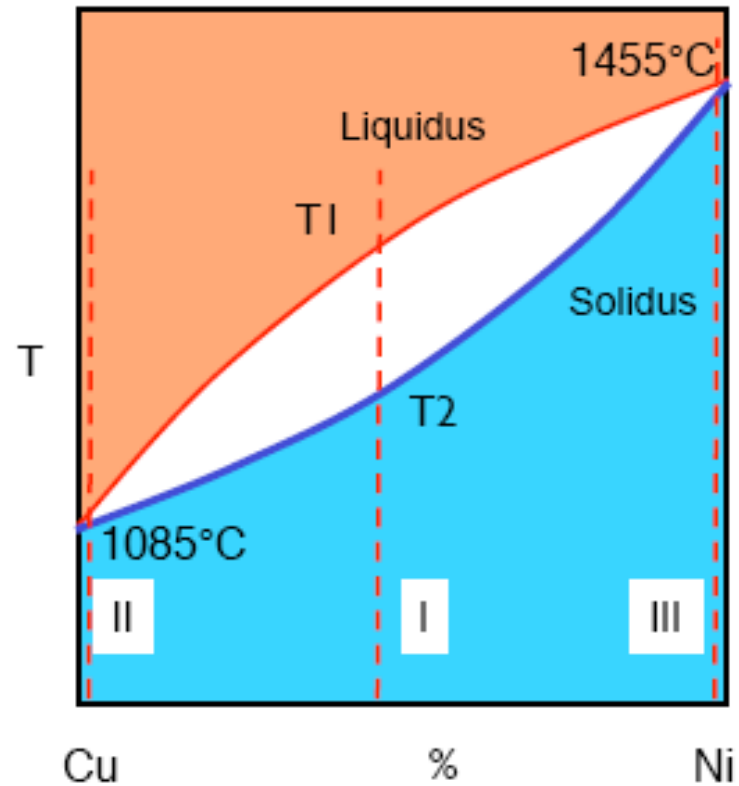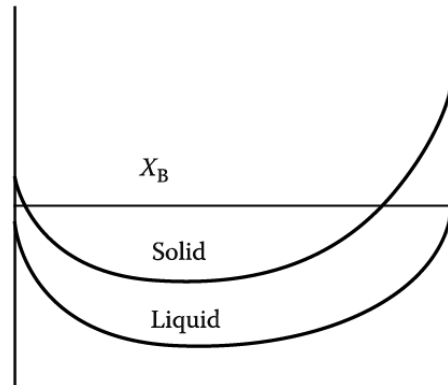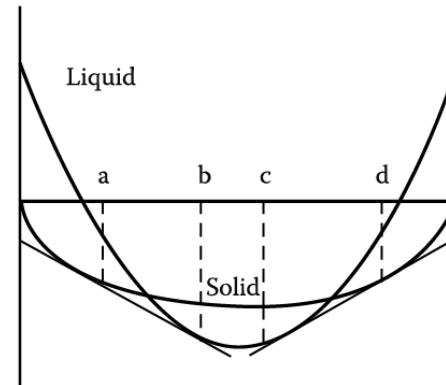**in the region of miscibility gap between e and f ?**

Ideal Solutions

$$G_2 = G_1 + \Delta G_{mix}$$

Molar free energy

Low T

ΔG_{mix}

High T

$0 \qquad X_B \longrightarrow \qquad 1$
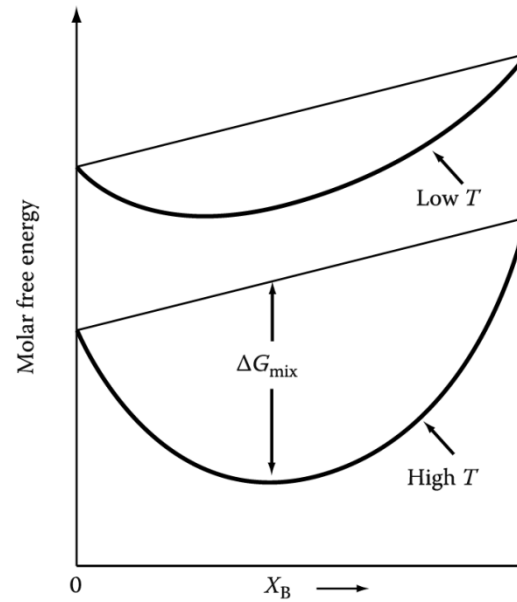
Regular Solutions

$$G = X_A G_A + X_B G_B + \underbrace{\Omega\, X_A X_B}_{\Delta H_{mix}} + \underbrace{RT\,(X_A \ln X_A + X_B \ln X_B)}_{-T\Delta S_{mix}}$$

Reference state

Pure metal $G_A^0 = G_B^0 = 0$
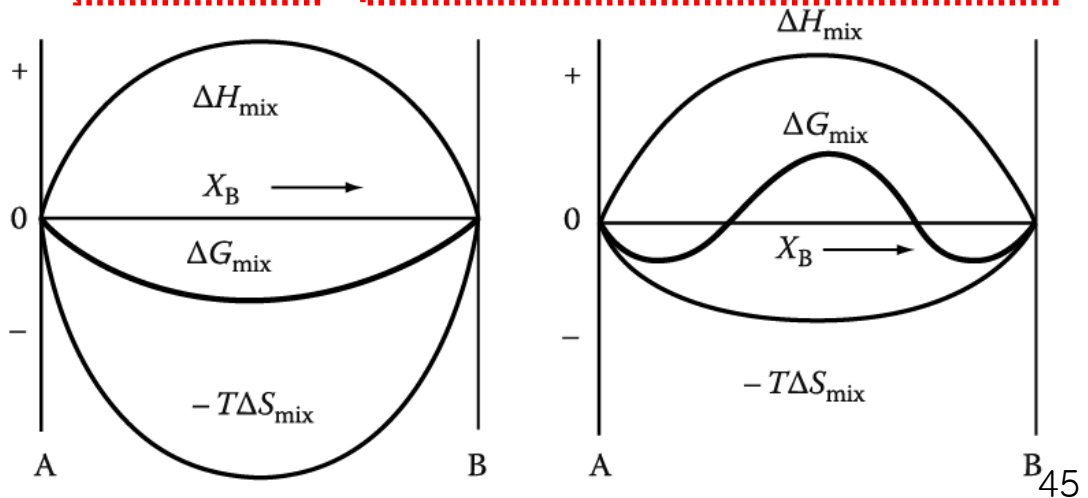
$+$

ΔH_{mix}

$X_B \longrightarrow$

0

ΔG_{mix}

$-$

$-T\Delta S_{mix}$

A

B

(c) $\Omega > O$, high T

$+$

ΔH_{mix}

ΔG_{mix}

0

$X_B \longrightarrow$

$-$

$-T\Delta S_{mix}$

A

B

(d) $\Omega > O$, low T

$$\Delta G_{mix} = \Delta H_{mix} - T\Delta S_{mix}$$

45

2) Systems with miscibility gab

$$\Delta H^{L}_{mix} = 0 \qquad \Delta H^{S}_{mix} > 0$$

- **When A and B atoms dislike each other,** $\Delta H_{mix} > 0$

- **In this case, the free energy curve at low temperature has a region of negative curvature,** $\dfrac{d^2 G}{dX_B^2} < 0$

- **This results in a 'miscibility gap' of α′ and α″ in the phase diagram**
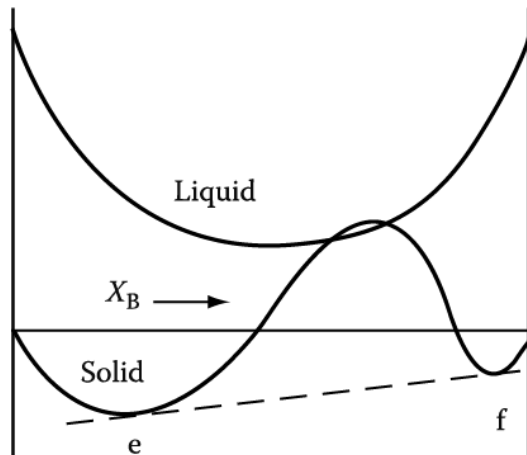
(c)
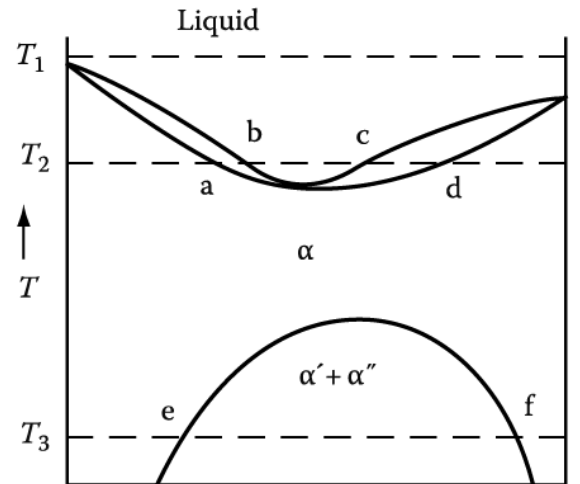
(d) **congruent minima**

46

2) Variant of the simple phase diagram

$$\Delta H_{mix} > 0 \qquad \Delta H^{\alpha}_{mix} > \Delta H^{l}_{mix}$$
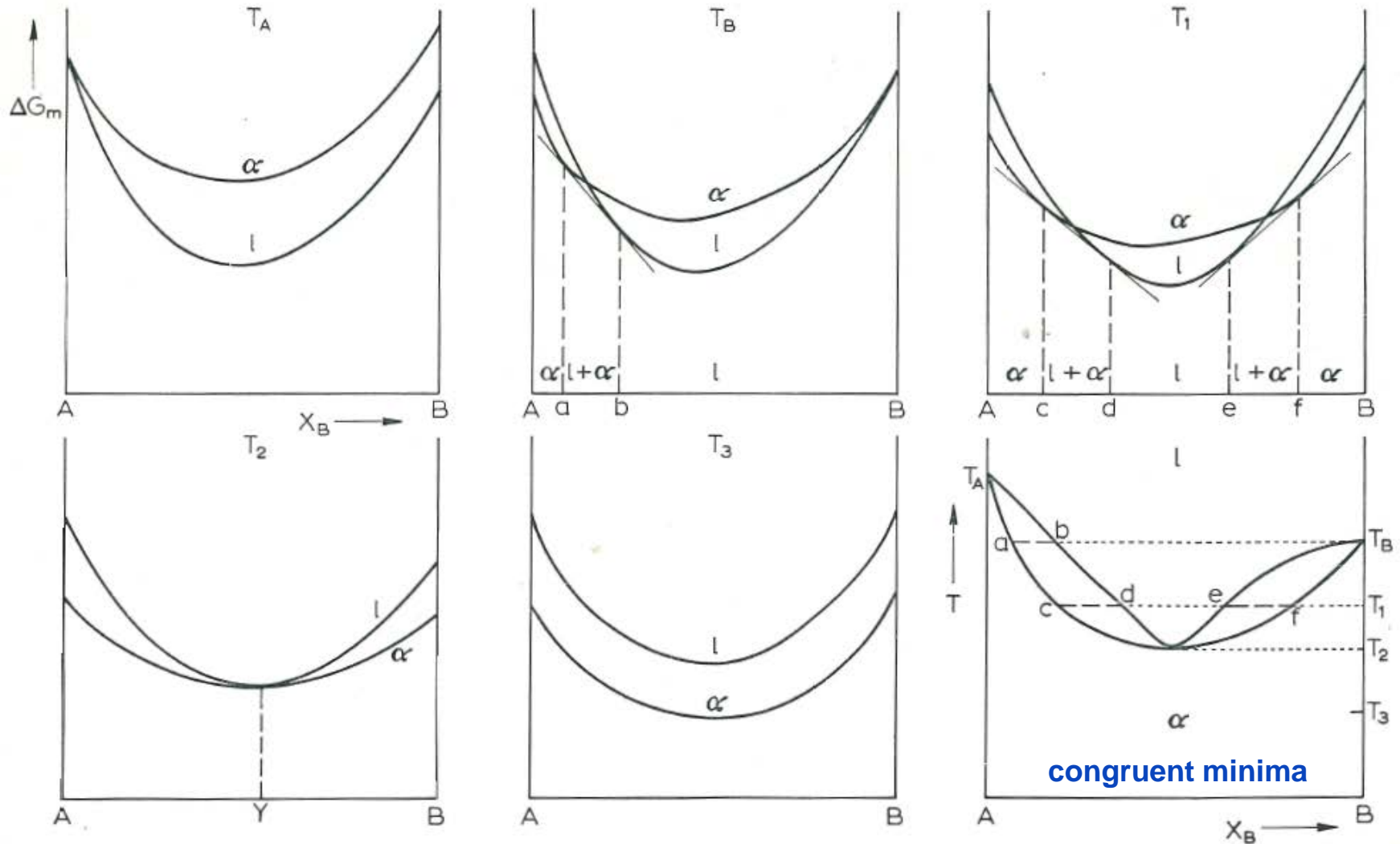
Fig. 30. Derivation of the phase diagram (Fig. 29b) from free energy curves for the liquid and solid phases. $T_A > T_B > T_1 > T_2 > T_3$.

Q5: How equilibrium is affected by temperature in simple eutectic systems?

4) Simple Eutectic Systems $\quad \Delta H_{mix}^{L} = 0 \quad \Delta H_{mix}^{S} >> 0$

- **$\Delta H_m >> 0$ and the miscibility gap extends to the melting temperature. (when both solids have the same structure.)**
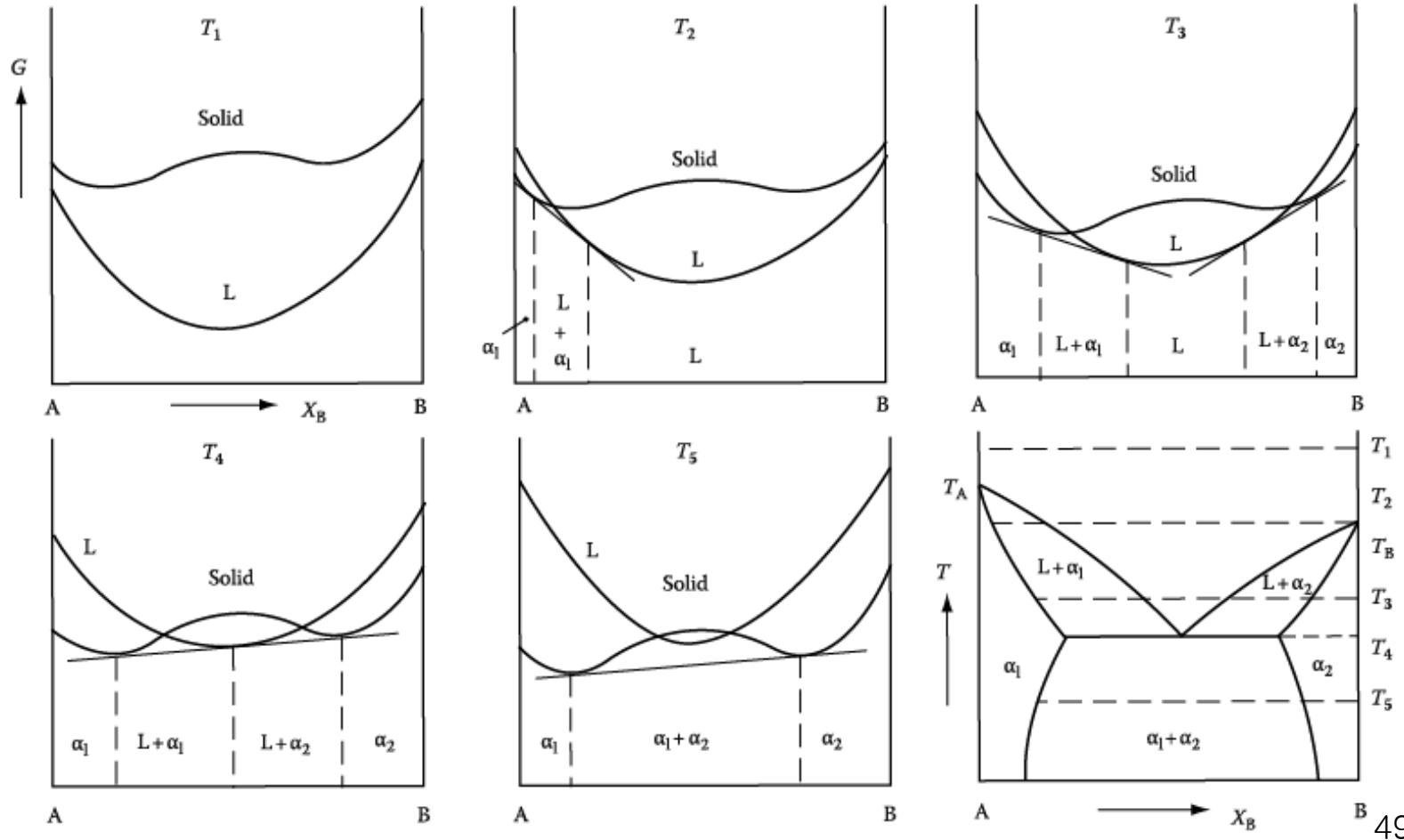
Fig. 1.32 The derivation of a eutectic phase diagram where both solid phases have the same crystal structure.

(when each solid has the different crystal structure.)

50
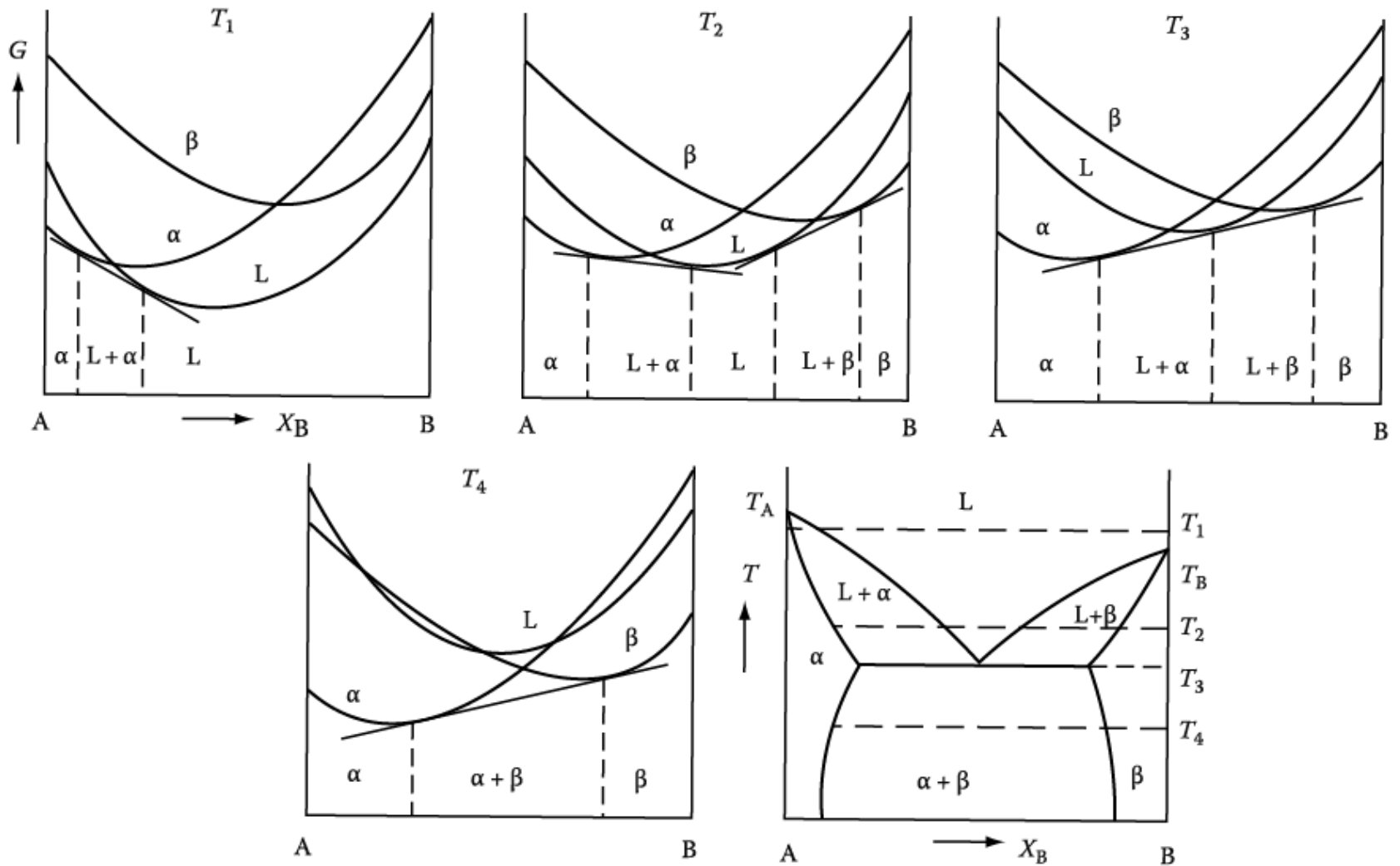
Fig. 1.32 The derivation of a eutectic phase diagram where each solid phases has a different crystal structure.

Cu-Zn Phase Diagram

Eutectic and Peritectic Reactions

peritectic:
$$\gamma + L \rightleftarrows \delta$$
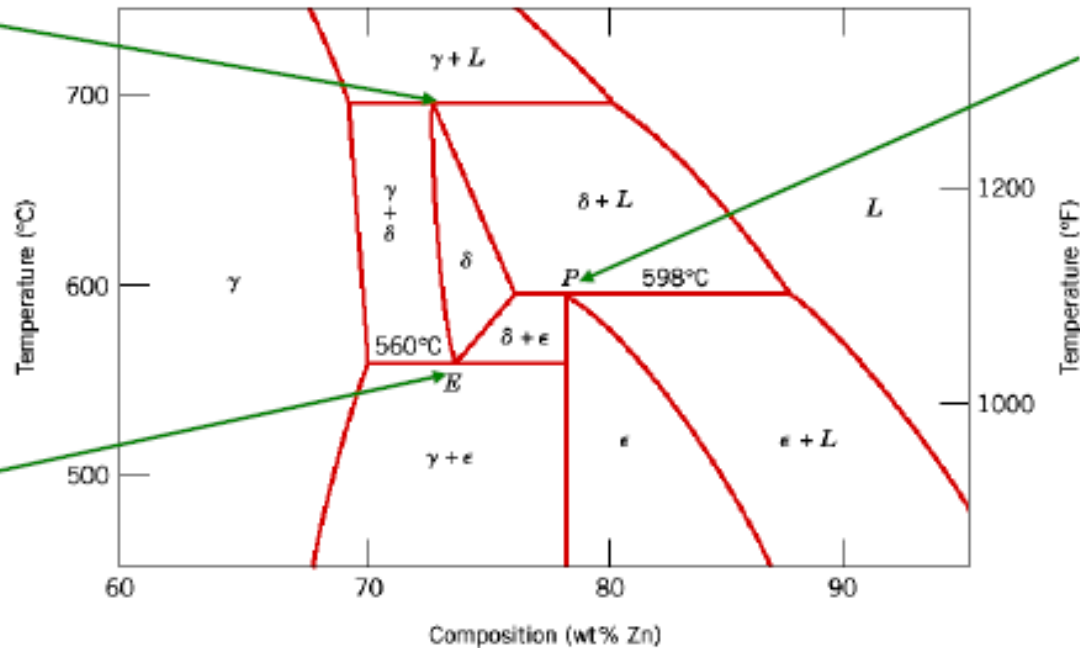
peritectic:
$$\delta + L \rightleftarrows \varepsilon$$

eutectoid:
$$\delta \rightleftarrows \gamma + \varepsilon$$

Eutectoid: **one** solid phase transforms into **two** other solid phases upon cooling

Peritectic: one solid and one liquid phase transform into another solid phase upon cooling

Peritectoid: **two other solid phases** transform into **another solid phase** upon cooling
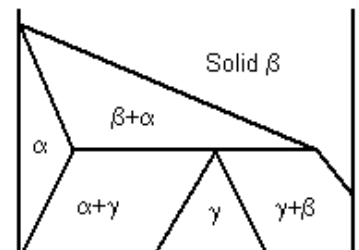
2) Variant of the simple phase diagram
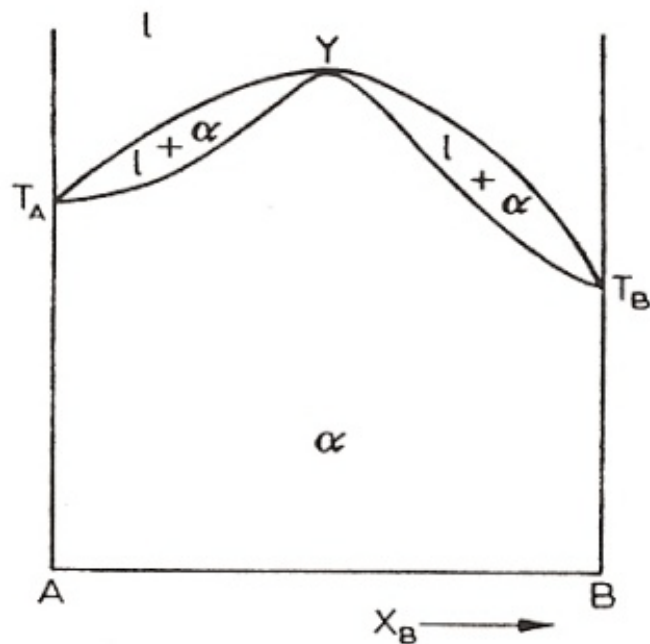
$$\Delta H_{mix} < 0 \qquad\qquad \Delta H_{mix}^{\alpha} < \Delta H_{mix}^{l} < 0$$

Fig. 32. Phase diagram with a maximum in the liquidus.
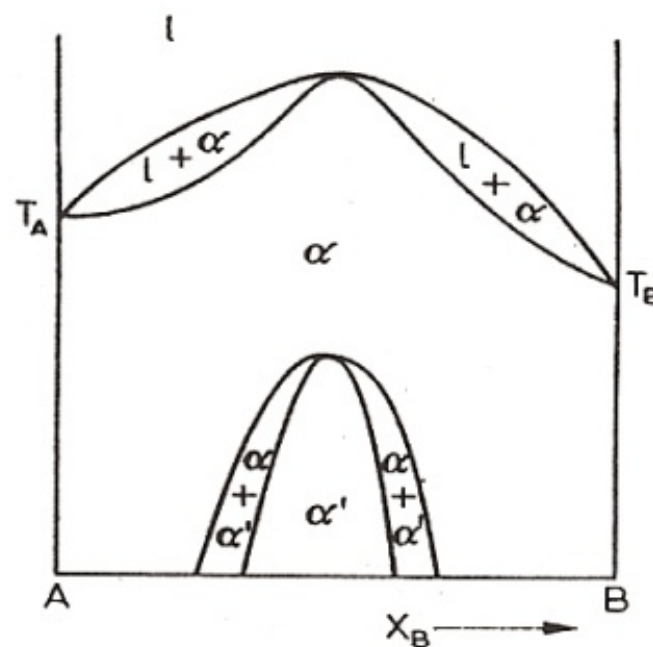
Fig. 33. Appearance of an ordered α' phase at low temperatures.

congruent maxima

5) Phase diagrams containing intermediate phases
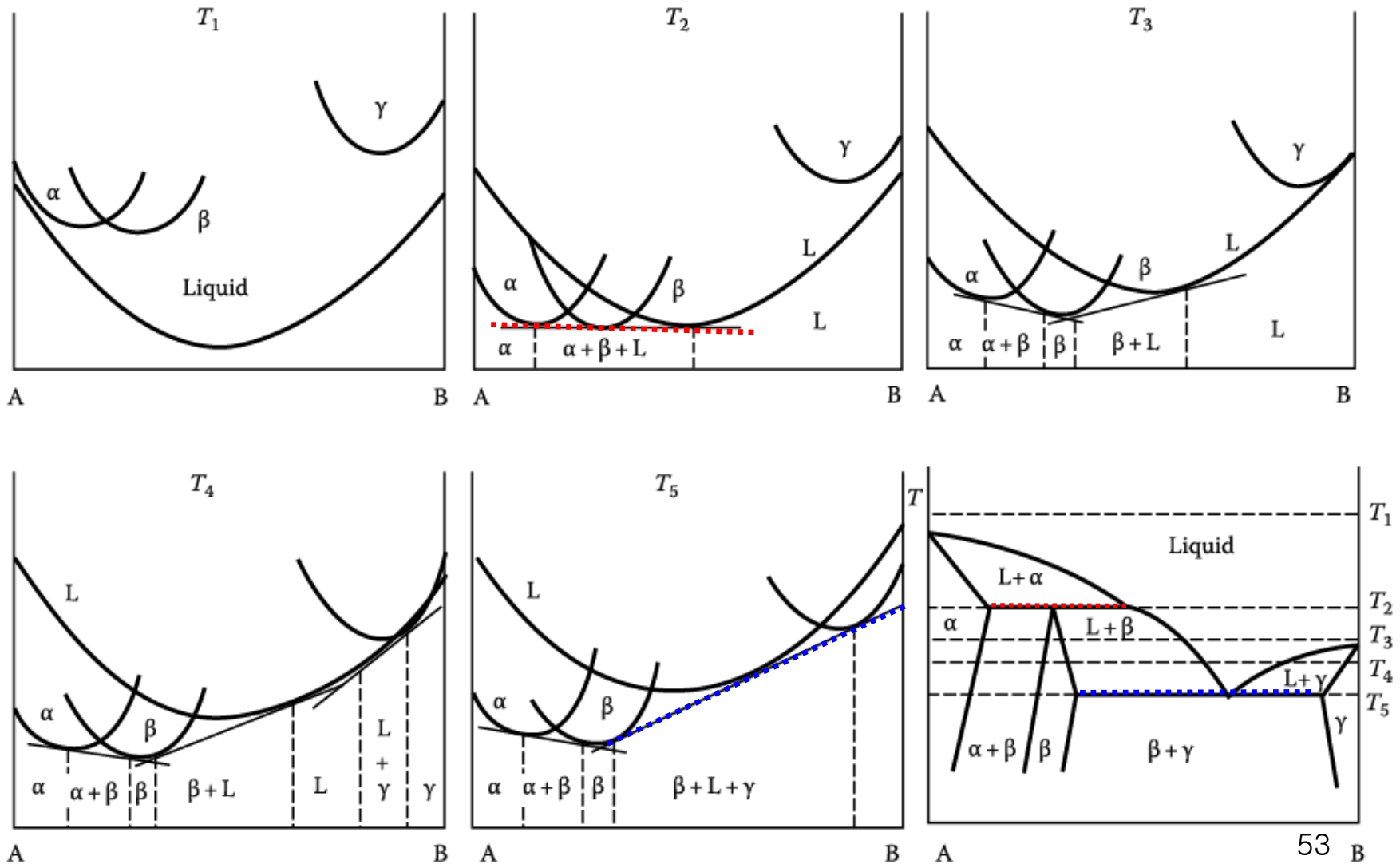
5) Phase diagrams containing intermediate phases

54
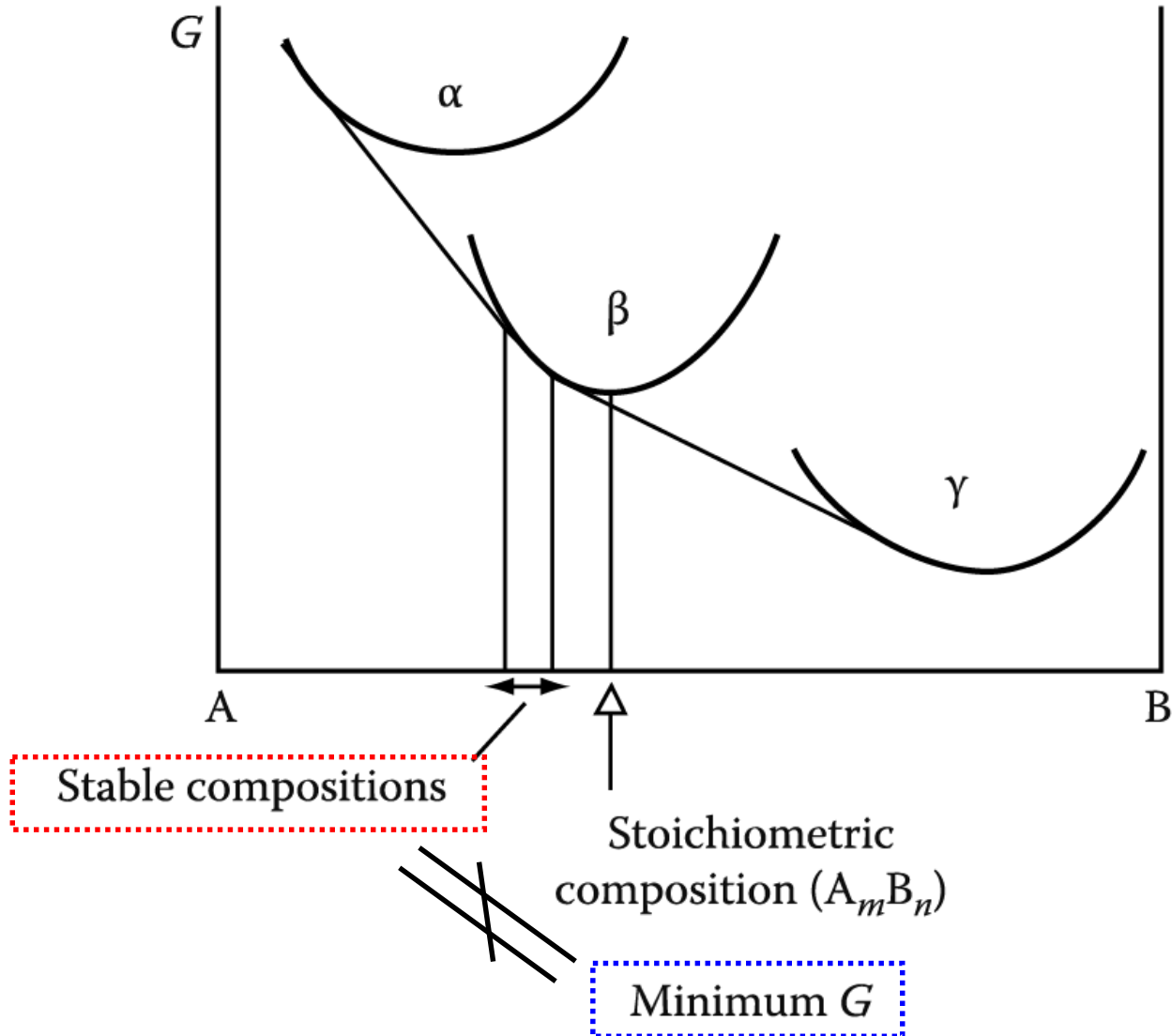
θ phase in the Cu-Al system is usually denoted as CuAl$_2$ although the composition X$_{Cu}$=1/3, X$_{Al}$=2/3 is not covered by the θ field on the phase diagram.


Al-Cu

Phase	Composition, at.% Cu	Pearson symbol	Space group	Struktur-bericht designation	Prototype
(Al)	0 to 2.48	$cF4$	$Fm\bar{3}m$	$A1$	Cu
θ	31.9 to 33.0	$tI12$	$I4/mcm$	$C16$	Al_2Cu
η_1	49.8 to 52.4	$oP16$ or $oC16$	$Pban$ or $Cmmm$	...	...
η_2	49.8 to 52.3	$mC20$	$Cm/2$	...	...
ζ_1	55.2 to 56.8	$hP42$	$P6/mmm$	...	...
ζ_2	55.2 to 56.3	m^{**}	...	...	...
ε_1	59.4 to 62.1	c^{**}	...	...	...
ε_2	55.0 to 61.1	$hP4$	$P6_3/mmc$	$B8_1$	NiAs
δ	59.3 to 61.9	hR^*	$R\bar{3}m$	...	...
γ_0	63 to 68.5	$cI52$	$I\bar{4}3m$	$D8_2$	Cu_5Zn_8
γ_1	62.5 to 68.5	$cP52$	$P\bar{4}3m$	$D8_3$	Al_4Cu_9
β	69.5 to 82	$cI2$	$Im\bar{3}m$	$A2$	W
α_2	76.5 to 78	...	...	...	...
(Cu)	80.3 to 100	$cF4$	$Fm\bar{3}m$	$A1$	Cu

J.L. Murray, *Phase Diagrams of Binary Copper Alloys*, P.R. Subramanian, D.J. Chakrabarti, and D.E. Laughlin, ed., ASM International, Materials Park, OH, 18-42 (1994)

X.L. Liu, I. Ohnuma, R. Kainuma, and K. Ishida, *J. Alloys Compds*, 264, 201-208 (1998)
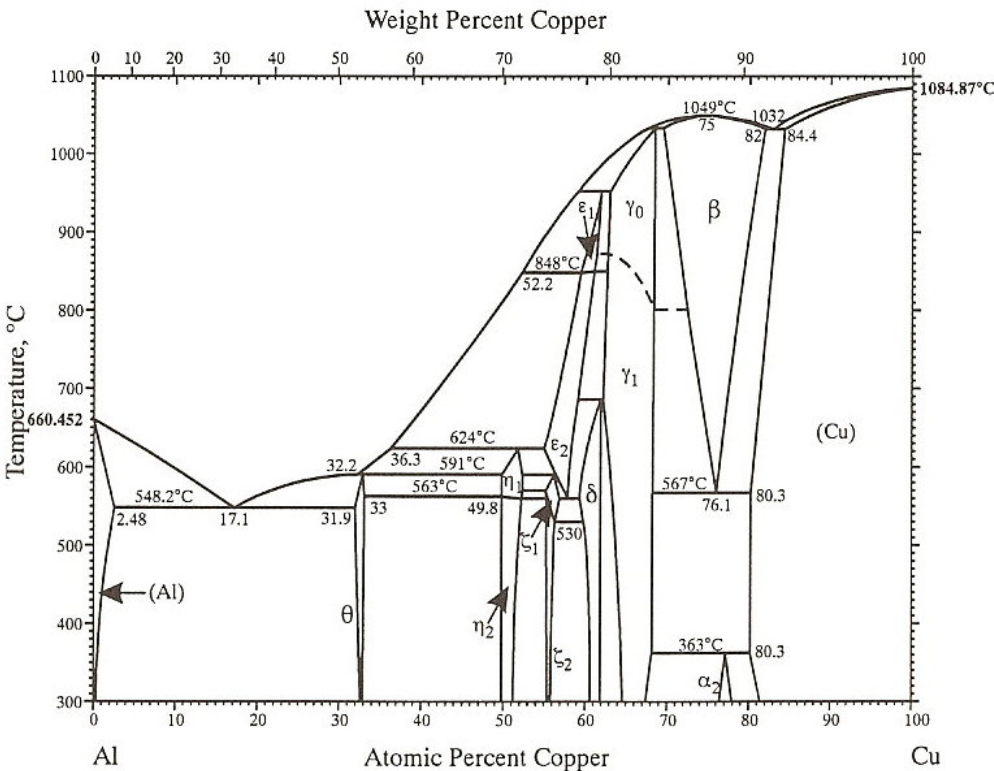
Summary I: Binary phase diagrams

1) Simple Phase Diagrams

Both are ideal soln. → 1) Variation of temp.: $G^L > G^S$ 2) Decrease of curvature of G curve

(∵ decrease of -TΔS$_{mix}$ effect)

2) Systems with miscibility gap $\Delta H_{mix}^L = 0$ $\Delta H_{mix}^S > 0$

1)Variation of temp.: $G^L > G^S$ 2)Decrease of curvature of G curve **+** Shape change of G curve by H

4) Simple Eutectic Systems $\Delta H_{mix}^L = 0$ $\Delta H_{mix}^S >> 0$

→ miscibility gap extends to the melting temperature.

3) Ordered Alloys $\Delta H_{mix}^L = 0$ $\Delta H_{mix}^S < 0$

$\Delta H_{mix} < 0 \rightarrow$ A atoms and B atoms like each other. → Ordered alloy at low T

$\Delta H_{mix} << 0 \rightarrow$ The ordered state can extend to the melting temperature.

5) Phase diagrams containing intermediate phases

Stable composition ≠ Minimum G with stoichiometric composition