

Lata Pasupulety

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Module 3- Phase equilibria

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Module content:

- Phase equilibria
- Gibb's phase rule
- Phase diagram of 1-component system
- Phase diagram of 2-component system
- Fe-C phase diagram

Module 3- Phase equilibria

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Class content:

- Free energy in Phase equilibria
- Chemical potential
- Phase equilibria
 - Phase
 - Component
 - Degree of freedom

Module 3- Phase equilibria

Free energy in Phase equilibria

- All substances have tendency to minimize their Gibbs energy at constant temperature and pressure to attain stable state
- Phase transformations from one phase to another occur to reduce free energy of the system
- Gibb's energy is an extensive property

Chemical potential

• Chemical potential is defined as the partial molar Gibb's energy for a component i in a mixture , and is denoted by μ

$$\mu_i = \left(\frac{\partial G}{\partial n_i}\right)_{p,T,n_j \neq i}$$



Module 3- Phase equilibria

Phase equilibria

 Phase equilibria between phases exist when chemical potential of a component is equal in all the phases in equilibrium

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e.g. for water at triple point
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Solid \rightleftharpoons liquid \rightleftharpoons vapour

The chemical potential of water will be equal in all the three phases

- For systems not at equilibrium, the chemical potential will point to the direction in which the system can move in order to achieve equilibrium viz., the system moves from higher chemical potential to lower chemical potential
- When various phases are in equilibrium with one another in a heterogeneous system, there can be no transfer of energy or mass from one phase to another.
- For a system at equilibrium, the various phases must have the same temperature and pressure and their respective compositions must remain constant all along



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Phase

A phase is defined as any homogeneous and physically distinct part of a system bounded by a surface and is mechanically separable from other parts of the system. It is denoted by P

- Gaseous state: P = 1 gases are completely miscible
- Liquid state: P = No. of layers when liquids are immiscible P = 1 when liquids are completely miscible
- Solid state: Each solid constitutes a separate phase
 Each polymorphic form constitutes a separate phase
 P = 1 for solid solution



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Counting the number of phases

- 1) Solid \rightleftharpoons liquid \rightleftharpoons vapour; P = 3
- Ice in the system is a single phase even if it is present as a number of pieces.

2) Calcium carbonate undergoes thermal decomposition

$$CaCO_3(s) \rightleftharpoons CaO(s) + CO_2(g)$$

 P= 3; 2 solid phases, CaCO₃ and CaO and one gaseous phase, that of CO₂

3) Ammonium chloride undergoes thermal decomposition

$$NH4Cl(s) \rightleftharpoons NH_3(g) + HCl(g)$$

P = 2; one solid, NH₄Cl and one gaseous, a mixture of NH₃ and HCl



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Components

- A component is defined as the smallest number of independently varying chemical constituents using which the composition of each and every phase in the system can be expressed
- When no reaction is taking place in a system, the number of components is the same as the number of constituents
- While expressing in terms of constituents zero and negative values are allowed



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Counting the number of components:



- 2-component system
 - Salt hydrate system

e.g.
$$Na_2SO_4.10H_2O$$
; **C=2**

$$Na_2SO_4.10H_2O = Na_2SO_4 + 10H_2O$$

$$Na_2SO_4.7H_2O = Na_2SO_4 + 7H_2O$$

$$Na_2SO_4 = Na_2SO_4 + OH_2O$$

$$H_2O$$
 = $0 Na_2SO_4 + H_2O$

All the different phases can be expressed in terms of the single constituent water

The composition of all the phases can be expressed in terms of 2 components



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Thermal decomposition of solid CaCO₃ in a closed container; C = 2

$$CaCO_3(s) \rightleftharpoons CaO(s) + CO_2(g)$$

- Though there are 3 species present, the number of components is only two
- Phases are: CaCO₃(s), CaO(s) and CO₂(g)
- Any two of the three constituents may be chosen as the components
- If CaO and CO₂ are chosen,

$$CaCO_3(s) = CaO + CO_2$$

 $CaO(s) = CaO + 0 CO_2$
 $CO_2(g) = 0 CaO + CO_2$

If CaCO₃ and CO₂ are chosen,

$$CaCO_3(s) = CaCO_3 + 0 CO_2$$

 $CaO(s) = CaCO_3 - CO_2$
 $CO_2(g) = 0 CaCO_3 + CO_2$



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Thermal decomposition of ammonium chloride in a closed system ;C = 1

$$NH_4Cl(s) \rightleftharpoons NH_3(g) + HCl(g)$$

 Phases are: solid phase of NH₄Cl(s) and gaseous phase of NH₃(g) and HCl(g)

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solid: NH_4Cl(s) = NH_4Cl
gas: NH_3(g) + HCl(g) = NH_4Cl
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- The composition of both the solid and gaseous phase can be expressed in terms of NH₄Cl. Hence the number of components is one; C=1
- If additional HCl (or NH_3) were added to the system, then C = 2
- The decomposition of NH₄Cl would not give the correct composition of the gas phase
- A second component, HCl (or NH₃) would be needed to describe the gas phase, therefore C=2



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Degrees of freedom (or variance)

The degrees of freedom or variance of a system is defined as the minimum number of intensive variables such as temperature, pressure, concentration, which must be fixed in order to define the system completely; it is denoted by F

OR

The degree of freedom of a system may also be defined as the number of variables, such as temperature, pressure and concentration that can be varied independently without altering the number of phases.

Example: water system

- Only 1 phase (solid, liquid or gas)
 - Both temperature and pressure need to be mentioned in order to define the system; F = 2



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2 phases in equilibrium,

- Only one variable, either temperature or pressure need to be specified in order to define the system; F =1
- solid water

 iquid water
- If the pressure on the system is maintained at 1 atm, then the temperature of the system gets automatically fixed at 0°C, the normal melting point of ice

3 phases in equilibrium,

- No variable can be changed
- temperature and pressure are fixed, F = 0
- solid water

 liquid water

 water vapour
- Three phases, ice, water, vapour can coexist in equilibrium at triple point of water at 0.0098°C and 4.6mm of Hg pressure only





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Module 3- Phase equilibria



Class content:

- Gibb's Phase rule
- Derivation of Gibb's Phase rule
- Phase diagram of a 1-component system

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Phase rule

- It was given by Williams Gibbs in 1874
- Statement of Gibb's phase rule

Provided the equilibrium in a heterogeneous system is not influenced by external forces (gravity, electrical or magnetic forces), the number of degrees of freedom (F) of the system is related to number of components (C) and number of phases (P) existing at equilibrium to one another by the equation

$$F = C - P + 2$$



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Derivation of the phase rule

- A system at equilibrium satisfies the following conditions:
 - Thermal equilibrium Temperature is constant
 - Mechanical equilibrium Pressure is constant
 - Chemical or material equilibrium Chemical potential of a substance is same in all the phases
- Mathematically, $\mu_i{}^{\alpha} = \mu_i{}^{\beta} = \mu_i{}^{\gamma} =$
- The system considered is: All C components distributed between P phases



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 Total number of intensive variables that need to be ascertained to describe the system:

Temperature 2 Pressure 1

Composition mole fraction of each component in every phase

• For each phase, the sum of mole fractions equals unity

$$\chi_1^{\alpha} + \chi_2^{\alpha} + \chi_3^{\alpha} + \dots + \chi_c^{\alpha} = 1$$
 (C-1)

$$\chi_1^{\beta} + \chi_2^{\beta} + \chi_3^{\beta} + \dots + \chi_c^{\beta} = 1$$
 (C-1)

$$\chi_1^{\gamma} + \chi_2^{\gamma} + \chi_3^{\gamma} + \dots + \chi_c^{\gamma} = 1$$
 (C-1)

•

•

$$\chi_1^P + \chi_2^P + \chi_3^P + \dots + \chi_c^P = 1$$
 (C-1)

- In each phase (C-1) mole fraction terms need to be defined
- Number of phases : P
- Number of composition variables = P(C-1)
- Total number of intensive variables = P(C-1) + 2



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Total number of equations(constraints):

• At equilibrium the chemical potential of particular component is same in every phase in a system

$$\mu_1^{\alpha} = \mu_1^{\beta} = \mu_1^{\gamma} = \dots$$
 $\mu_2^{\alpha} = \mu_2^{\beta} = \mu_2^{\gamma} = \dots$
 $\mu_3^{\alpha} = \mu_3^{\beta} = \mu_3^{\gamma} = \dots$
(P-1)
(P-1)

$$\mu_{c}{}^{\alpha} = \mu_{c}{}^{\beta} = \mu_{c}{}^{\gamma} = \dots$$
 (P-1)

- For C components
 C(P-1)
- Total number of equations or constraints =C(P-1)

F = Total number of variables – total number of equations

$$F = P(C-1) + 2 - \{C(P-1)\}\$$

 $F=C-P+2$

which is the Gibb's phase rule



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Application of Gibb's phase rule F = C-P+2 to 1-component system:



Water system

When only 1 phase is present:

C = 1, P = 1; F = 2; Temperature and Pressure can be varied independently **Bivariant system**

When 2 phases are in equilibrium:

C = 1, P = 2; F = 1; Temperature or Pressure can be varied independently **Univariant system**

When all 3 phases are in equilibrium:

C = 1, P = 3; F = 0; Neither Temperature nor Pressure can be varied **Invariant system**

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Phase diagram

A diagram which represents the **conditions** under which a substance exists in **different phases** in a system

Phase diagram of a 1-component system

F = C - P + 2For a 1-component system F = 3 - P

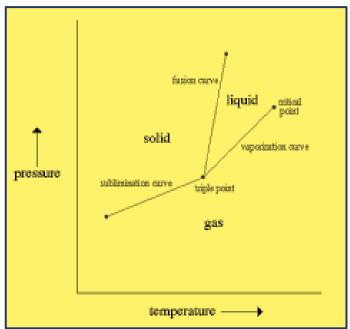
Single phase: F = 2; Area in a diagram

Two phases in equilibrium : F = 1; line in a diagram

Three phases in equilibrium : F = 0 ; point in a diagram



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High pressure, low temperature: solid phase High temperature, low pressure: vapour phase In between: liquid phase

```
solid ⇌ liquid liquid ⇌ vapour N vapour ⇌ solid E
```

Solid ≠ liquid ≠ vapour

POINT

Source:http://abyss.uoregon.edu/~js/glossary/triple_point.html





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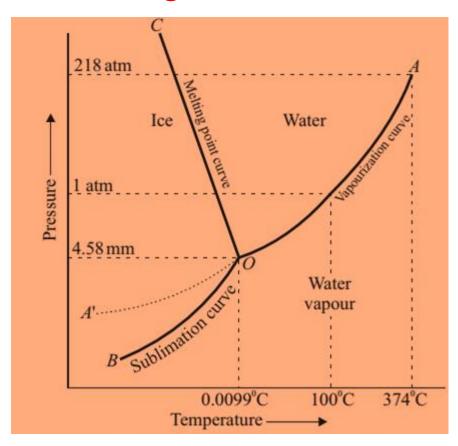
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Class content:

• Phase diagram of water

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Phase diagram of water



OC: Melting point curve

OA: Vaporisation curve

OB: Sublimation curve

O: Triple Point

A: Critical point

OA': Metastable equilibrium



Source: https://imbooz.com/engineering-chemistry/phase-diagram-for-water-system/

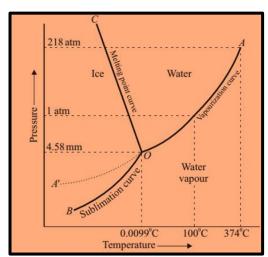
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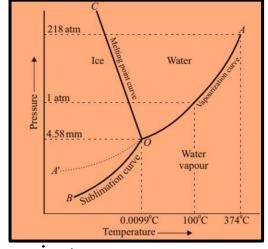
Equilibrium between solid and liquid (fusion curve OC)

- •F=1, monovariant system
- variation of melting point of ice with pressure
- •slope is negative; as ice melts its volume decreases or density increases
- Clausius-Clapeyron equation

$$\frac{dp}{dT} = \frac{\Delta H_{Fusion}}{T\Delta V} = negative$$

Where,





 ΔV = decrease in volume as ice melts is -ve; $\Delta H_{fusion} = endothermic, +ve$

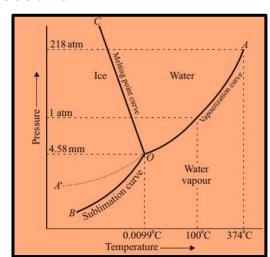
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Equilibrium between solid and vapour (sublimation curve OB)

- ice
 ⇒ water vapour
- F=1, monovariant system
- variation of sublimation temperature of ice with pressure
- slope is positive
- Clausius Clapeyron equation

$$\frac{dp}{dT} = \frac{\Delta H_{sub}}{T\Delta V} = positive$$

Where,



 ΔV = Increase in volume as ice sublimates ,+ve ; $\Delta Hsublimation$ = endothermic reaction, +ve



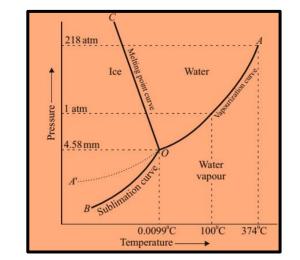
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Equilibrium between liquid and vapour (vaporization curve OA)

- F=1, monovariant system
- variation of boiling temperature of water with pressure
- slope is positive
- Clausius Clapeyron equation

$$\frac{dp}{dT} = \frac{\Delta H_{Vapourisation}}{T\Delta V} = positive$$

Where,



 ΔV = Increase in volume as liquid water vapourises,+ve; $\Delta H_{Vapourisation}$

= endothermic reaction, +ve



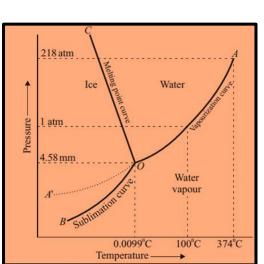
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Triple point "O":

- Represents equilibrium between liquid, vapour and solid water (ice)
- All three phases are present together
- F = 0, invariant system
- Triple point for water lies at 0.0098 °C and 4.58 mmHg

Critical point "A":

- the interface between liquid water and water vapour vanishes
- a point above which water does not exist in liquid state
- Critical point lies at 374°C and 220 atm pressure

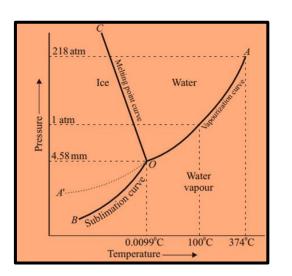




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Metastable equilibrium (OA'):

- Ice fails to form at the triple point and water continues to exist in liquid phase
- The vapour pressure of the liquid continues along OA'
- This is called <u>super cooled water</u> and represents metastable equilibrium involving liquid and vapour phases.
- Any disturbance will cause the system to go back to stable equlilibrium (OB)
- The vapour pressure of the system in the metastable region is more than that of the stable system ice at the same temperature







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Class content:

- Phase diagram for CO₂ system
- Reduced phase rule for a 2-component system

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AB: Sublimation curve

BD: Melting point curve

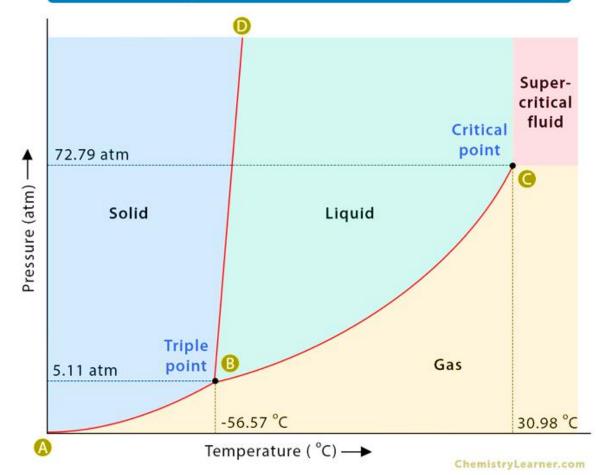
BC: Vaporization curve

B: Triple Point

C: Critical point

Beyond C: Supercritical Fluid

Phase Diagram of Carbon Dioxide (CO₂)



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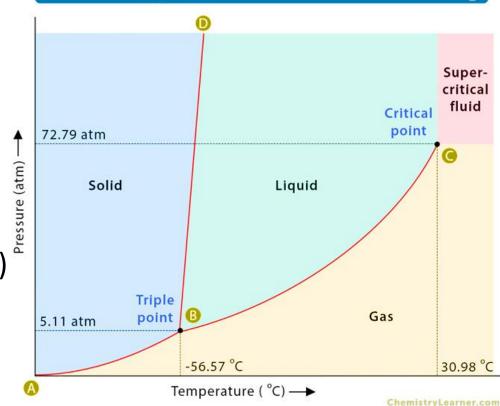


Equilibrium between gas and solid (Sublimation curve AB)

- solid $CO_2 \rightleftharpoons gaseous CO_2$
- •F=1, mono variant system
- •variation of sublimation temperature of solid ${\rm CO_2}$ with pressure
- slope is positive
- •Clausius Clapeyron equation: $\frac{dP}{dT} = \frac{\Delta H_{sub}}{T\Delta V}$ (+ve slope)

 ΔV = Increase in volume as solid CO₂ sublimes, +ve; $\Delta H_{sublimation}$ = endothermic reaction,+ve

Phase Diagram of Carbon Dioxide (CO₂)



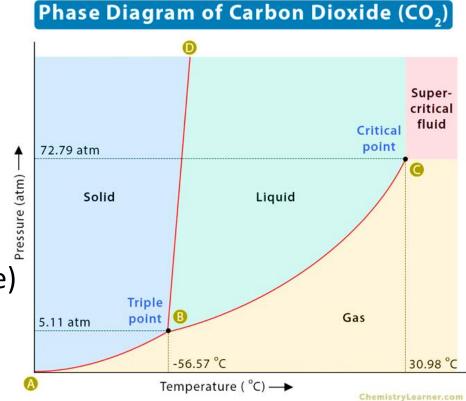
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Equilibrium between solid and liquid (melting/fusion curve BD)

- solid $CO_2 \rightleftharpoons liquid CO_2$
- •F=1, mono variant system
- •variation of melting/fusion temperature of solid CO₂ with pressure
- slope is positive
- •Clausius Clapeyron equation: $\frac{dP}{dT} = \frac{\Delta H_{fusion}}{T\Delta V}$ (+ve slope

 ΔV = Increase in volume as solid CO₂ liquefies, +ve; ΔH_{fusion} = endothermic reaction,+ve



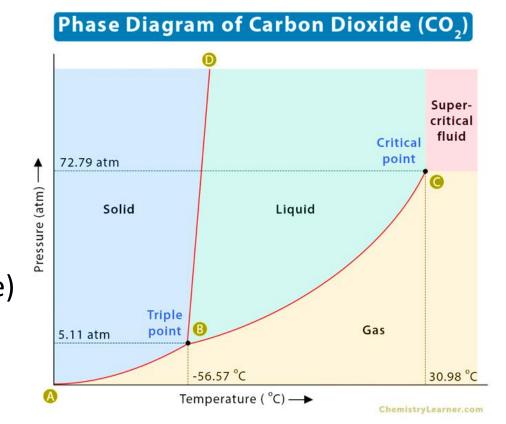
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Equilibrium between liquid and gas (vaporization curve BC)

- liquid $CO_2 \rightleftharpoons gaseous CO_2$
- •F=1, mono variant system
- •variation of boiling temperature of liquid CO₂ with pressure
- slope is positive
- •Clausius Clapeyron equation: $\frac{dP}{dT} = \frac{\Delta H_{vap}}{T\Delta V}$ (+ve slope)

 ΔV = Increase in volume as liquid CO₂ vaporizes, +ve; $\Delta H_{vaporization}$ = endothermic reaction,+ve



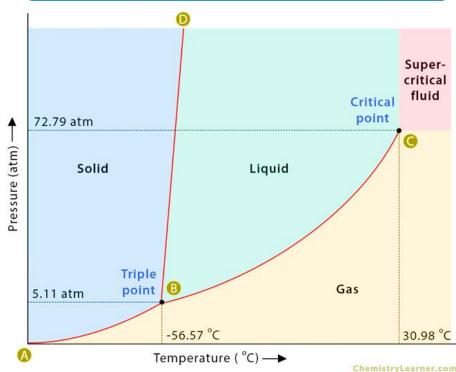
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Triple Point (B)

- •Represents equilibrium between liquid, vapor and solid CO₂ (dry ice)
- All three phases co-exist simultaneously
- •F=0,invariant system
- •Triple point for CO₂ lies at -56.6° C and 5.11 atm
- i.e. liquid CO₂ cannot exist at pressures lower than 5.11 atm.



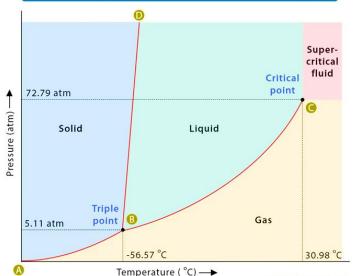


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- At 1 atm, solid CO_2 sublimes directly to vapor instead of melting into a liquid, whilst preserving a temperature of -78.5°C.
- Thus, solid CO₂ is also called "dry ice". It is a cold solid with no liquid phase observed when it is warmed.
- This property makes it a useful coolant without using water (generally required for chilling purposes).

Phase Diagram of Carbon Dioxide (CO,





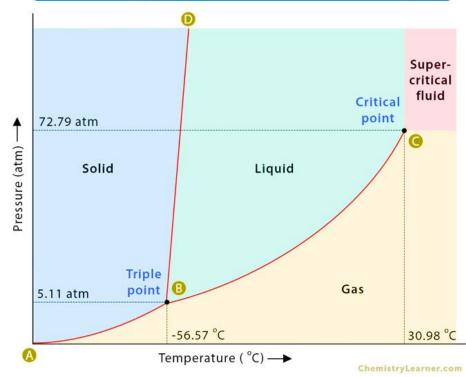
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Critical Point (C)

- Represents the maximum temperature and pressure beyond which liquid and vapor phases become indistinguishable, i.e. the interface between liquid CO₂ and CO₂ (gas) vanishes.
- At such high temperatures, the gas cannot condense back to liquid, no matter how much pressure is applied.
- Critical point lies at 30.98 °C and 72.79 atm.
- Beyond the critical point, the substance becomes a supercritical fluid.

Phase Diagram of Carbon Dioxide (CO₂)



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Supercritical Fluid

- Supercritical carbon dioxide is widely used as a solvent for industrial extraction processes.
- It is widely used for decaffeinating coffee.
- It is widely used as a dry-cleaning solvent.
- It is also used as a natural refrigerant, making it a low carbon (more environmentally friendly) solution for domestic heat pumps.

https://makeagif.com/gif/supercritical-fluids-aQxehz



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Phase diagram of a two-component system

- The phase rule equation is F = C P + 2
- For a 2 component system the ordinary phase rule cannot be used
- For a two component system, C = 2 then F = 2 P + 2 = 4 P
- The minimum number of phase is 1; F = 4 1 = 3
- This requires 3 dimensional space which cannot be explained on the plane of paper
- One of the three variables is kept constant
- Measurements in these systems are generally carried out at atmospheric pressure
- Pressure may be considered constant; degrees of freedom is reduced by 1,

•
$$F = C - P + 2 - 1$$

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- The phase rule takes the form F = C P + 1 and is known as the reduced phase rule
- Equilibria such as solid-liquid equilibria are such systems in which the gas phase is absent and hence are hardly affected by small changes in pressure
- Systems in which the gas phase is absent are called condensed systems
- F = C P + 1 is also known as condensed phase rule



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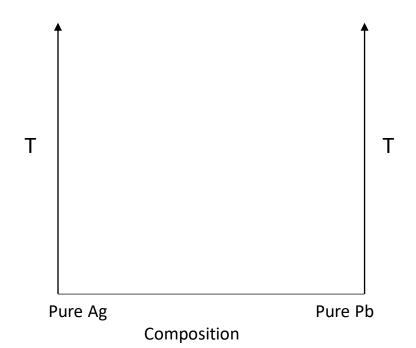
Class content:

- Phase diagram of Pb-Ag system
- Determination of solid-liquid equilibria
- Pattinson's process

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Phase diagram of Pb-Ag system

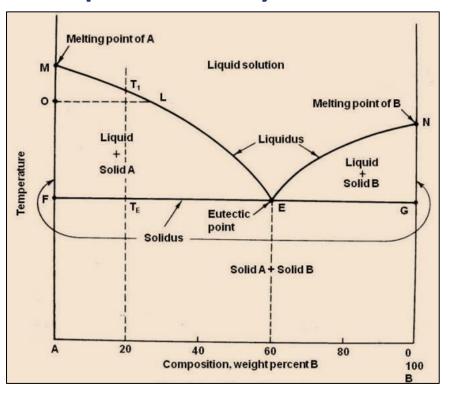
- Phase rule for a 2- component system: F = C-P+1
- Pressure is constant
- Plot is between Temperature and Composition
- Pb and Ag are miscible in all proportions in the liquid (molten) state
- In solid state they are completely immiscible





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Simple eutectic system



When a mixture of the 2 components is heated till the mixture melts and then cooled:

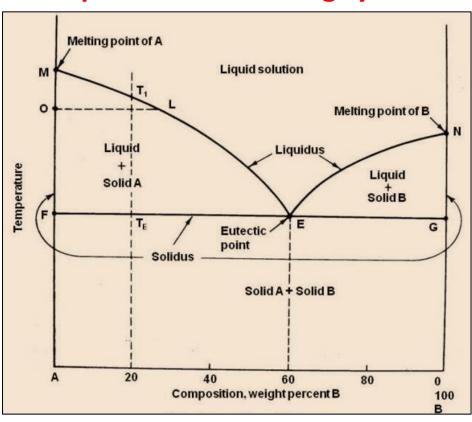
- First crystal of A if formed when f.pt.
 of A in the mixture is reached
- On further cooling more solid A precipitates and liquid melt becomes richer in B
- With decrease in temperature more and more solid A separates and the liquid melt moves along curve ME
- Finally when temperature reaches F, solid B also starts precipitating
- Three phases are present at E: solid
 A, solid B and the liquid melt
- Further cooling will just result in cooling of solid A & B



Source:https://in.pinterest.com/pin/818107088531422918/

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Simple eutectic: Pb-Ag system



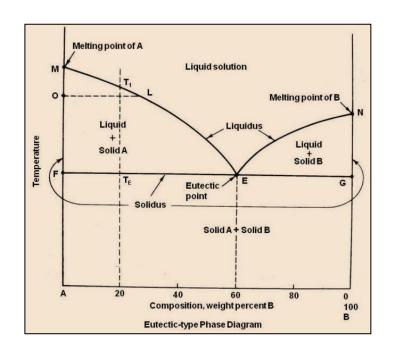
Pb-Ag system

- A: 100% Ag, M: 961°C
- B: 100%Pb, N: 327°C
- Area above MEN: Liquid melt
- Area MEF : Solid A + Liquid melt
- Area NEG : Solid B + Liquid melt
- Below FEG : Solid A + Solid B
- Eutectic point "E"
- Curves : ME and NE



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- Eutectic mixture: A mixture of two components which has the lowest freezing point of all the possible mixtures of the components
- It has a definite composition and a sharp melting point
- Number of phases at eutectic point = 3
- F = C-P+1; C =2, P = 3,so **F = 0**; invariant point,
- Eutectic temperature: 303°C
- Eutectic composition: 97.4 % Pb and2.6 % Ag





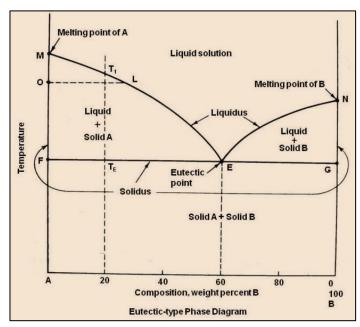
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CURVE ME: freezing point curve of Ag

- It shows decrease in freezing point / melting point of Ag due to the addition of Pb to Ag
- Solid Ag is in equilibrium with liquid melt of Pb in Ag
- Here C = 2 and P = 2, then the reduced phase rule is F = C P + 1 = 2 2 + 1 = 1
- Hence the system is univariant.

CURVE NE: freezing point curve of Pb

- It shows decrease in freezing point of Pb due to the addition of Ag to Pb
- Solid Pb is in equilibrium with liquid melt of Ag in Pb
- Here C = 2 and P = 2, then the reduced phase rule is F = C P + 1 = 2 2 + 1 = 1
- Hence the system is univariant





Module 3- Phase equilibria

Determination of solid-liquid equilibria

 For the determination of equilibrium conditions between solid and liquid phases – Thermal Analysis

Thermal analysis:

The study of the **cooling curves** of various compositions of a system during solidification

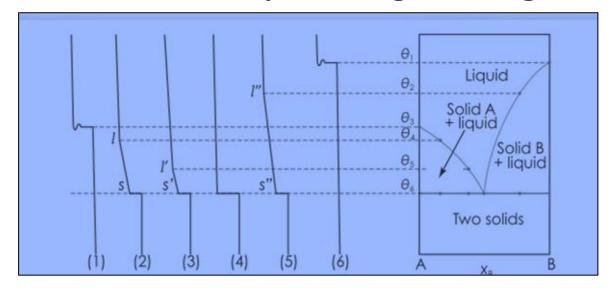
Cooling curves:

- Temperature versus time
- Freezing point and eutectic point can be determined from the cooling curves



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Construction of phase diagram using cooling curves



Source:http://www.mchmultimedia.co m/PhysicalChemistryhelp/clientstories/study-tips/digginginto-phase-diagrams-coolingcurves.html

For pure solid: When the freezing point is reached, temperature remains constant until the liquid is fully solidified.

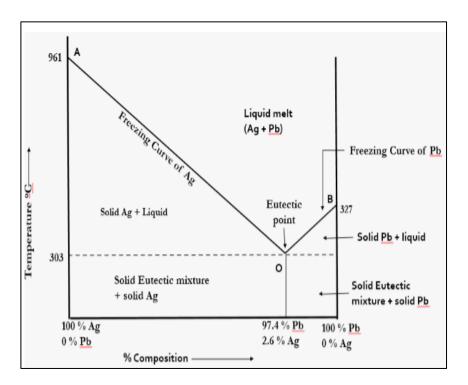
For a mixture of solids: When crystallisation of one of the components starts ,cooling curve exhibits a break. The temperature decreases continuously until the eutectic point is reached. Now the temperature remains constant, till the completion of solidification.



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Pattinson's process for the desilverisation of argentiferous lead

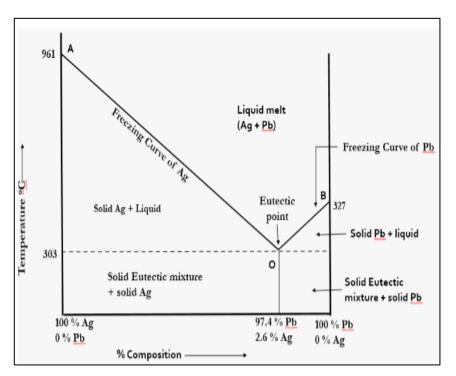
- The process of heating argentiferous lead containing a very small quantity of silver (~0.1 %) and cooling it to get pure lead and liquid richer in silver
- •Argentiferous lead is heated to a temperature above the melting point of pure lead
- •The melt is allowed to cool
- •Temperature of the melt reaches the freezing curve of Pb where solid lead starts separating
- As the system further cools, more and more lead separates and the liquid in equilibrium with the solid lead gets richer in silver





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- •The lead that separates, floats and is continuously removed by ladles
- •When the temperature of the liquid reaches the eutectic temperature, solid lead is in equilibrium with the liquid having the eutectic composition
- After removing the lead that separates, the liquid is cooled further when it solidifies to give a mixture of lead and silver having the eutectic composition of 2.6 % of silver
- This solid mixture of lead and silver is subjected to other processes for the recovery of silver







THANK YOU

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Module 3- Phase equilibria



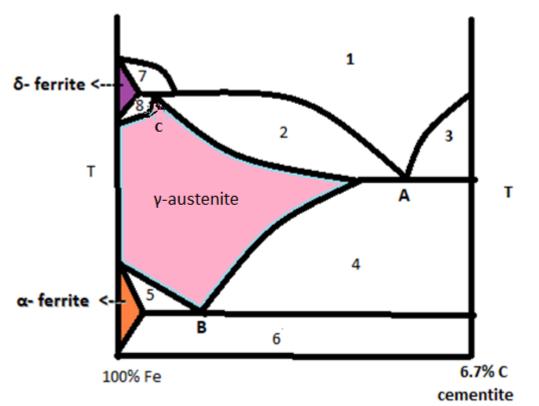
Class content:

Fe-C phase diagram

Module 3- Phase equilibria

Iron- Carbon (Fe-C) Phase Diagram

- Fe-C system is a 2-component system; F = C P + 1
- the Fe-C phase diagram is a fairly complex one
- we consider the part of the diagram, up to around 6.7% Carbon



α- ferrite : 0°C – 900°C

γ- austenite : 900°C – 1400°C **δ- ferrite :** 1400°C – 1540°C

Beyond 1540°C – Fe melts

Cementite(Fe₃C): 6.7% C



Module 3- Phase equilibria

Phases in Fe-Fe₃C Phase Diagram

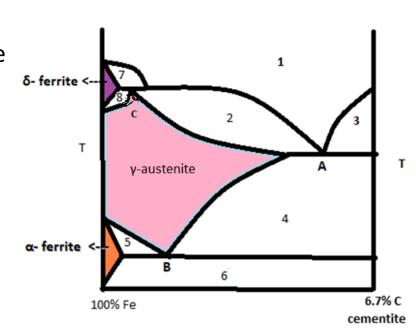
α-ferrite

- solid solution of C in BCC Fe
- Stable form of iron at room temperature
- Transforms to FCC γ-austenite at 900 °C
 γ-austenite
- solid solution of C in FCC Fe
- Transforms to BCC δ -ferrite at 1400 °C

δ-ferrite

- solid solution of C in BCC Fe
- The same structure as α-ferrite
- Stable only at high T, above 1400 °C
- Melts at 1540 °C
 - Fe₃C (iron carbide or cementite)
- This is a intermetallic compound.

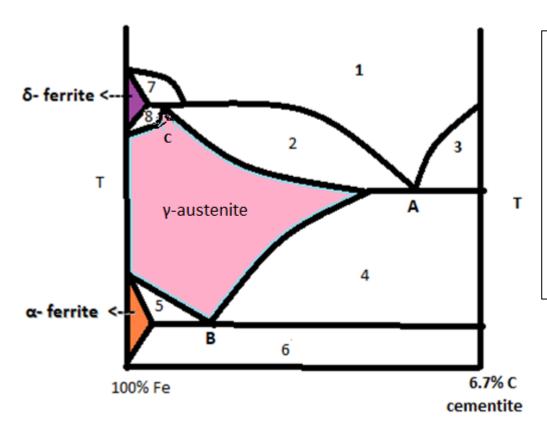
Fe-C liquid solution





Module 3- Phase equilibria

Areas in the Fe-Fe₃C phase diagram



1: liquid melt

2 : γ- austenite + liquid melt

3 : cementite + liquid melt

4 : γ- austenite + cementite

5 : α - ferrite + γ- austenite

6 : α - ferrite + cementite

7 : δ- ferrite + liquid melt

8 : δ- ferrite + γ - austenite



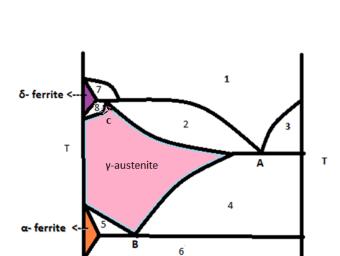
Module 3- Phase equilibria

Eutectic point A:

- liquid melt of Fe-C transforms into two different solid phases γ-austenite and cementite (Fe₃C) on cooling; L ↔ γ + Fe₃C
- corresponds to 4.3 % C, 1130 °C
- 3 phases are in equilibrium; γ-austenite, cementite and liquid melt
- F=0. The system is invariant

Eutectoid point B:

- γ -austenite phase transforms into two different solid phases α -ferrite and cementite (Fe₃C) on cooling; $\gamma \leftrightarrow \alpha + Fe_3C$
- corresponds to 0.8 % C, 723 °C
- 3 phases are in equilibrium; γ -austenite, α ferrite and cementite
- F=0. The system is **invariant**



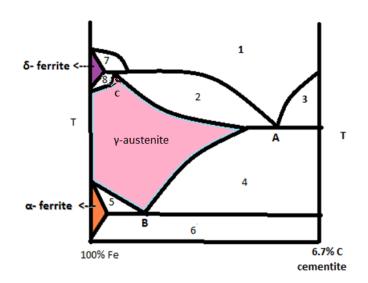
6.7% C cementite



Module 3- Phase equilibria

Peritectic point C:

- •liquid melt of Fe-C transforms into two different solid phases γ -austenite and cementite (Fe₃C) on cooling: $\delta + L \leftrightarrow \gamma$
- corresponds to 0.16 % C, 1498 °C
- 3 phases are in equilibrium; γ-austenite,
 δ- ferrite and liquid melt
- F=0. The system is **invariant**





Module 3- Phase equilibria

Three types of ferrous alloys of Fe and Carbon

Wrought Iron:

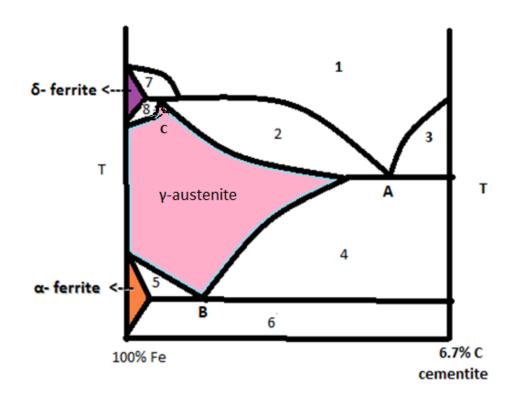
less than 0.008 % C

Steel:

• 0.008 - 2.14 % C

Cast iron:

• 2.14 - 6.7 wt % C







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