MME 345
Lecture **B:06**

Solidification and Crystallisation

5. Formation and control of granular structure

Ref:

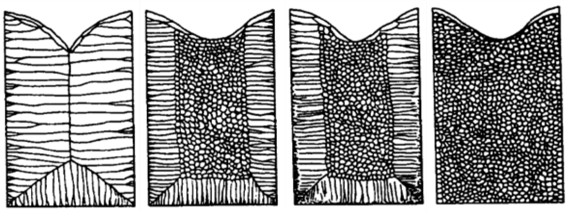
[1] A. Ohno, The Solidification of Metals, Chijin Shokan Co. Ltd., 1976
[2] P. Beeley, Foundry Technology, Butterworth-Heinemann, 2001

Topics to discuss today ….

1. The ingot structure
2. Control of grain structure

1. The ingot structure

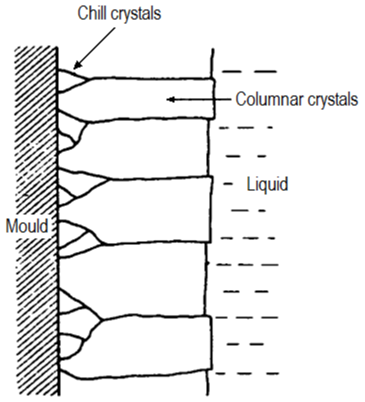
- ❑ Various types of solidified structures are obtained depending on
  - **the amount and properties of the solutes contained in the metal,**
  - **the properties, size, and shape of the mould used, and**
  - **the pouring temperature and technique**


(a) **(b)** **(c)** **(d)**

(a) columnar grains only, (b) columnar grains & equiaxed grains at the centre,
(c) columnar grains & equiaxed grains at the centre, (d) columnar grains & equiaxed grains at the centre

1.1 Formation of chill and columnar zones

- ❑ A large number of fine crystals nucleated randomly at the mould wall immediately after pouring due to very high cooling rate



transition from chill zone to columnar zone

- ❑ These crystals then start growing in competition with each other

  only the most favourably oriented crystals grow further

- ❑ The outer layer of castings where randomly oriented crystals were growing is called the **chill zone**, while the structure consisting of crystals that grow inwards opposite to the direction of the heat flow is called the **columnar zone**

- For a **too high liquid temperature**, the chill zone becomes indistinct because of delayed nucleation and re-melting of nucleated crystals

  even when the temperature of liquid is decreased at later stage, chill zone will not develop due to the reduction in cooling rate by an increase in mould temperature

- For a **high cooling rate**, the chill layer becomes thinner and microscopically invisible

- During the growth of columnar zone, the number of crystals decreases and a preferred orientation develops by the survival of those crystals which are favourably oriented

  this favourable orientation of growth is always having the dendritic growth direction (for example <100> for fcc crystals)

- The interface between the columnar crystals and the liquid may be smooth, cellular or dendritic according to the composition of the alloy and the rate of solidification

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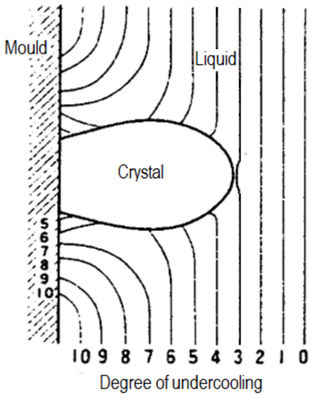
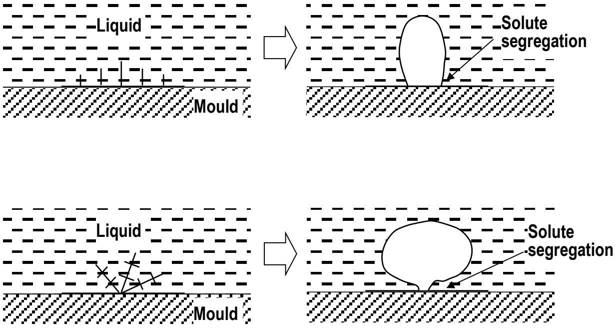
1.2 Formation of equiaxed crystals



undercooling at a solid/liquid interface which is growing at the mould wall

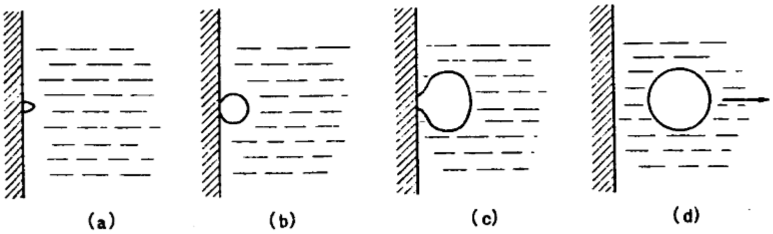
- Segregation of solute occurs at the root of the crystals nucleated at the mould, which depresses the growth of crystal at its root.
- Head of crystal grows at a much faster rate than its root.
- The side of crystal grow preferentially than its root and the crystal begins to have a **necked** or **granular** shape form.

- If none of the main axes of the crystal is perpendicular to the mould wall, the neck will become much narrower and more unstable.



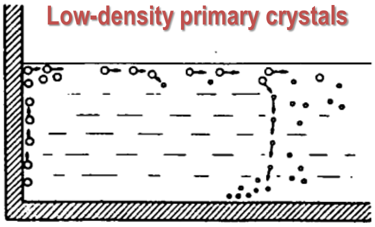
the formation of necked-shape crystals on the mould wall

- Such narrow crystals **can easily be separated** from the mould wall by a mechanical force that can be produced by stirring, thermal convection, and temperature fluctuation in the liquid.



schematic illustration of the separation of equiaxed crystals at a mould wall

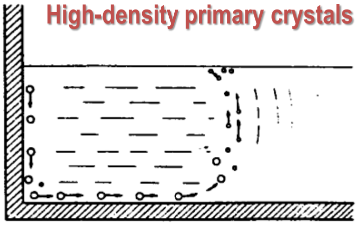
Low-density primary crystals



schematic illustration of the crystal movement during the initial stage of solidification in Sn-10Bi alloy

- The crystals separated from mould wall will float up along the wall against the downward convection if the density of the primary crystal is less than that of the liquid
- The floating crystals will promote separation of narrow necked crystals on the upper wall of the mould and pushed the crystals on the molten surface towards the centre of the casting

High-density primary crystals



schematic illustration of the crystal movement during the initial stage of solidification in Bi-5Sn alloy

- On the other hand, as in most commercially used alloys, if the density of separated crystals are higher than that of the liquid, separated crystals will be precipitated along the mould wall and promote separation of narrow necked crystals on the lower wall of the mould which then move to the centre of the casting

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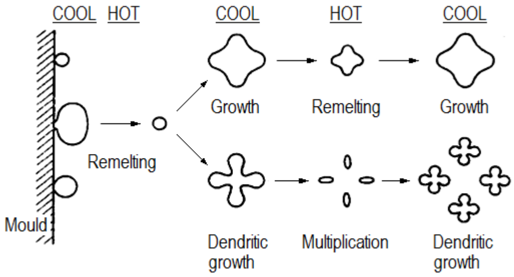
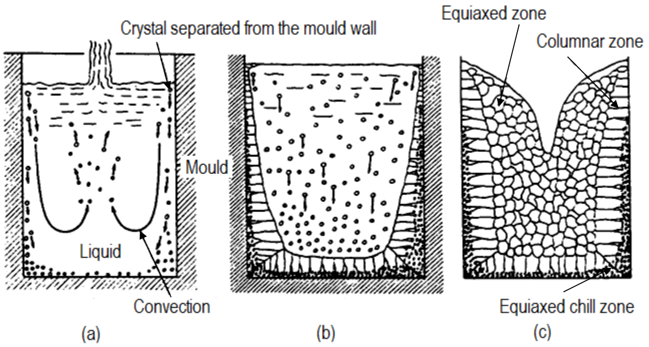
- Separation of crystals may then continuously proceed like a chain reaction in the initial stage of solidification.
- Since thermal convection in the liquid metal is the largest in the mould wall at the beginning stage of solidification, separation of equiaxed crystals from the mould wall can most easily occur at this stage.
- When separated crystals precipitate along the mould wall, some of these crystals will be trapped at the cold mould surface, particularly at the corner, and will form the **equiaxed chill zone** there.

- The crystals that escape from the cold surface of the mould are carrier to the centre of the mould by thermal convection and start to float.
  - Some of the floating crystals are re-melted off into the liquid, some of the crystals will be reduced in size by partial re-melting, and some of the crystals will be fragmented



- **Multiplication** of the separated crystals can also be expected during precipitation and floatation if the temperature is not uniform throughout the liquid.

- As the convection in the liquid decreases, the tendency of the separation of crystals from the mould wall decreases.
- The columnar crystals then start to grow both from the mould wall and from the equiaxed chill zone and continue until they are stopped by the equiaxed crystals which are floating in the central region in the mould.
- Finally an ingot with the macrostructure containing equiaxed chill zone, columnar crystals and equiaxed crystals will be produced.



formation of ingot structure: equiaxed chill zone, columnar zone, and central equiaxed zone

2. Control of grain structure

☐ The principal factors governing the final metallographic structure of a casting may now be listed. They are:

1. Constitution and thermal properties of the alloy;
2. Casting design and dimensions;
3. Thermal properties of the mould;
4. Superheat and final casting temperature;
5. Conditions for heterogeneous nucleation;
6. Conditions affecting motion during solidification;
7. Subsequent heat treatment.

☐ Thus, although the structure of a casting is in the first instance a function of **alloy composition** and **casting geometry**, it is also sensitive to measures taken in founding.

☐ These include: **(1)** **preliminary treatment** of the liquid metal, and **(2)** **variation in cooling rate** within the mould.

☐ It would, however, be true to say that manipulation of the freezing process in castings has been **more usually directed at the problems of feeding** than at structure.

☐ The importance of structure in cast alloys lies mainly in the structure sensitive properties which can be utilized in engineering.

☐ Unlike wrought materials, in which further opportunities exist for changing both structure and dislocation density, **the initial microstructure is frequently the main vehicle for the control of properties in castings**, although subsequent heat treatment plays its role in some cast alloys.

☐ In examining the critical characteristics of cast structures, it will be convenient to consider two types of metallographic feature:

1. **Grain structure** (size, shape and orientation)
2. **Sub-structure** (distribution of micro-constituents, dendrite substructure)

☐ The normal cast structure can be further complicated by the presence of **shrinkage cavities** or by the macroscopic **segregation** of alloying elements.

☐ Control of cast structure may take either of the following two directions:

1. **Refinement of grain and constituent size**

   Some of these treatments produce more drastic changes in the microstructure of light alloys and cast irons, providing beneficial influences on the morphology of individual phases.

2. **Manipulation of grain shape and orientation**

   to provide anisotropy of structure and properties by design, a principle developed with great success in the field of high temperature superalloys.

2.1 Control of grain structure during solidification

☐ For all but a few specialized applications (e.g. single crystal turbine blades, magnet alloys) fine-grained equiaxed structures are preferred in castings and ingots

these structures are isotropic and their properties are markedly superior

☐ To develop these structures requires suppression of columnar growth

this can be achieved by encouraging conditions favourable to the formation of the equiaxed nuclei

☐ To obtain the ingot that consists entirely of equiaxed crystals

1. making narrow necked crystals on the mould wall
2. preventing the formation of stable solid skin
3. promoting the separation of crystals from the mould wall
4. preventing the re-melting off of the separated crystals

☐ Two main approaches have been adopted :

1. control of nucleation by controlling the casting conditions or by using the inoculants, and

2. use of physical methods (e.g. stirring, ultrasonic vibration) to induce dynamic grain refinement by enhancing crystal separation from the mould wall at the beginning of solidification.

☐ To achieve these targets, five main variables for controlling the structure can be identified :

1. Grain refiners
2. Vibration
3. Pouring methods
4. Mould materials
5. Pouring temperature

1. Effect of grain refiners

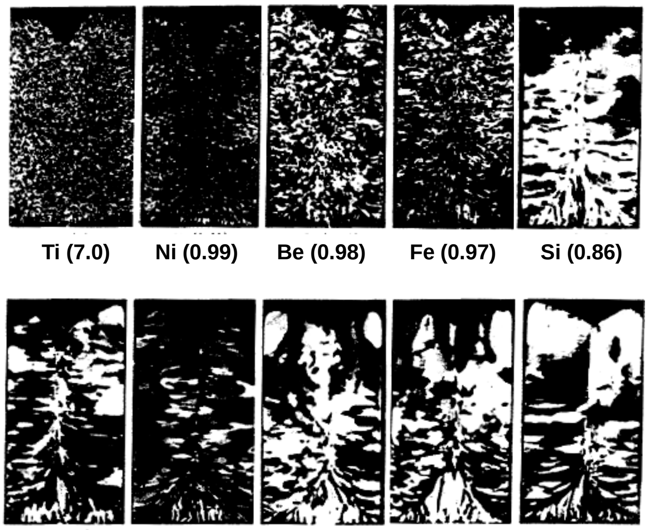
- According to the classical theory, grain refiners change the properties (e.g. wettability) of molten surface and act as nucleating site to **enhance heterogeneous nucleation**

  must be present in the solid state in the liquid

- Modern day theory believes that, besides that, grain refiners are also segregated at the root of the nucleated crystals and make the root narrower to **cause easier crystal separation**

  elements having a smaller distribution coefficient, k_0, or a higher segregation coefficient, $|1 - k_0|$, are more effective as grain refiner

Segregation coefficient in Al alloys		Segregation coefficient in steels	
Element	**$|1- k_0|$**	**Element**	**$|1- k_0|$**
Titanium	7.00	Sulphur	0.95-0.98
Nickel	0.99	Oxygen	0.90-0.98
Magnesium	0.70	Silicon	0.34-0.45
Zinc	0.56	Manganese	0.15-0.20
Manganese	0.30	Tungsten	0.05



Ti (7.0) **Ni (0.99)** **Be (0.98)** **Fe (0.97)** **Si (0.86)**

Cu (0.83) **Cr (0.80)** **Mg (0.70)** **Zn (0.56)** **Mn (0.30)**

effect of segregation coefficient $|1-k_0|$ on macrostructure of aluminium ingot with 0.1% addition of the grain refiners

2. Effect of vibration

☐ Application of **forced vibration** and **agitation** to the molten metal during solidification are very effective for grain refining of cast structures

- it is believed earlier that grain refining by vibration is mostly due to the breakdown of dendritic crystals
- presently, vibration is believed to be the main cause of crystal separation
- common vibration methods are: mechanical vibrations, ultrasonics, bubbling-agitations, and magnetic and electric field interactions

☐ **Convection** is associated with temperature fluctuation is also important

- most violent at the solid/liquid interface when the top surface is cooled and is the smallest when the bottom surface is cooled

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☐ Presently, two important effects of forced vibration on solidified structure are considered :

1. promotes wetting of molten metal to the mould surface by breaking down the oxide film
    - helps heat extraction through the mould wall and increases the cooling rate and promotes nucleation at the mould wall
    - produces finer crystals at the surface
2. promotes the separation of narrow necked-shape crystals from the mould wall
    - vibrations in the early stage of solidification are the most effective for separation of crystals
    - if the pouring temperature is too high, the vibration action must be continued until the molten metal temperature is low enough for the separating crystals to survive

For metals that grow with planer solid-liquid interface (e.g. pure metals), separation of crystals, however, can not be expected

3. Effect of pouring methods

- If the pouring method produces stationary molten surface, grain refinement would not occur, even if the temperature is low
- Grain refinement would occur only if a pouring method creates a surface wave-motion near the mould wall
- The distance from pouring nozzle to mould top and number and size of nozzle greatly influence the solidified structure

  in other words, a pouring nozzle producing turbulence at the mould wall is the most effective in producing crystal separation and finer structures

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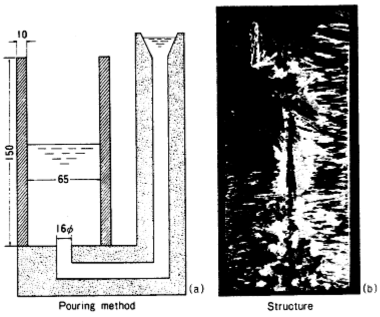
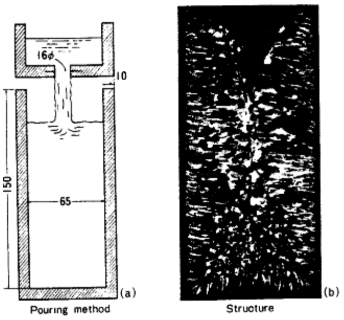
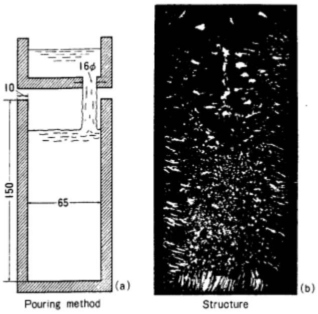


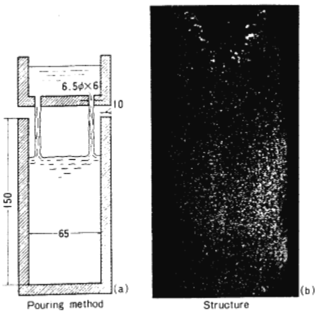
bottom pouring



central pouring by one nozzle



top pouring



side pouring by six nozzles

4. Effect of mould materials

- Number of nuclei at the mould surface increases with increased undercooling

- For a mould with **reduced cooling power** (e.g., a sand mould), a fewer nuclei at a far distance from each other are produced
  - will take a longer time to come in contact with each other to form a solid shell
  - a greater opportunity for crystal separation by convection and vibration

- Moulds with a **greater chilling power** (e.g. metal mould, graphite mould) produce more nuclei closely to each other on the mould wall
  - a solid shell is rapidly formed and starts to grow as columnar crystal even if violent convection exists in the liquid near the solid skin

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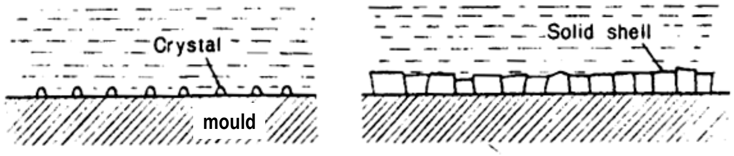
(a) Sand mould



(b) Chill mould

nucleation of crystal on mould surface during the initial stages of solidification

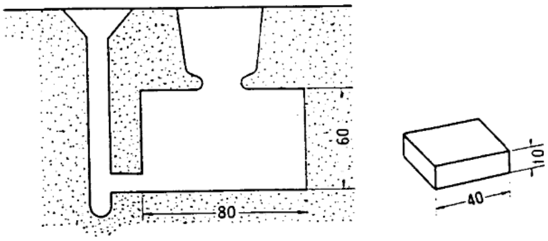
- However, if free crystals that are formed elsewhere are carried to the mould, the mould with higher cooling capacity produces finer grain structure on its wall

  because many crystals can precipitate without being re-melted in the liquid

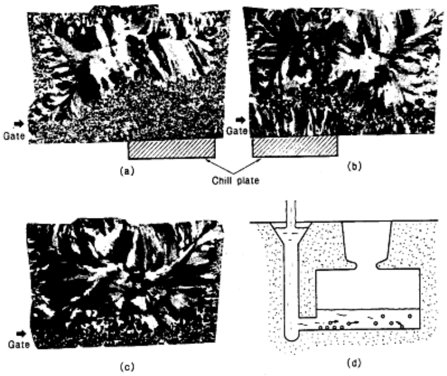
- Use of a chill has the similar effect

- But a chill plate will prevent crystal separation and produce columnar grains if it is placed at a location where crystal separation is most expected during solidification

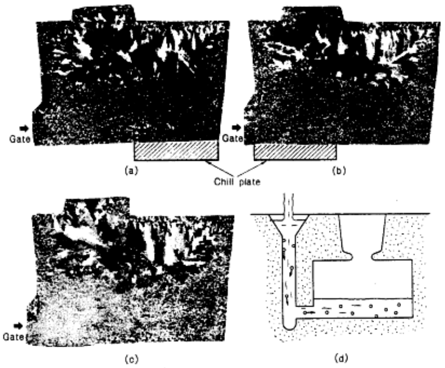
casting of aluminium plate with and without using a chill



(a) Sand mould **(b) Chill plate**



99% pure Al, pouring temperature 700 C



99% pure Al, pouring temperature 680 C

- At 700 C, stable crystals begin to form at the bottom of mould, right in front of the gate

  In (a), separated crystals carried on to the chill plate and form stable equiaxed crystals
  In (b), most of the crystals grew rapidly to form solid shell of columnar crystals
  In (c), most crystals were separated by the flow and were re-melted into the liquid metal

- All structures for metals poured at 680 C are similar, because crystal separation has already been taken place near sprue due to the low liquid temperature

5. Effect of pouring temperature

- ☐ It is generally well-known that the grain size in the solidified structure of ingots generally increases as the pouring temperature of molten metal increases
  - ❖ to produce finer crystals,
    the free crystals must survive the temperature to form finer grains
  - ❖ re-melting of separated crystals must be prevented
- ☐ High temperature **increases thermal convection** and higher crystal separation, but it also **increases re-melting** of separated crystals
- ☐ Lower temperatures generate lower thermal convection and fewer crystal separations with lower re-melting. But crystals that do not separated from the stable solid skin grow to form the columnar zone.

Thus, to obtain an ingot consisting entirely of equiaxed crystals, the pouring temperature should be as low as possible to cause enough crystal separation without re-melting of separated crystals

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Next Class
MME **345**, Lecture **B:07**

The Feeding Design

1. Necessity and requirements of feeding