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Project Title

Mold Construction for Visualization of Solidification of Transparent Alloys

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1

INTRODUCTION

Solidification processing is one of the oldest manufacturing processes as it is the principal component of metal casting processing. The manufacturing of all metallic products starting with raw materials it is coming from casting process and the materials can be ingots. To improve casing properties and reduce process defects, the phenomena related to solidification, e.g. melt undercooling, grain nucleation, microstructure and segregation formation, etc. have to be well understood. Solidification also exist in welding process. Solidification is, strictly speaking, the transformation of liquid matter into solid matter.

The microstructure that results from solidification may be the final one, in which case it directly affects the mechanical properties of the product. In other cases, heat treatment or other processes may be used after solidification to further modify the solidification microstructure. However, the outcome of this additional processing will be greatly affected by the solidification microstructure. The term microstructure is the classic term used in metallography to describe features observed under the microscope. The effect of solidification on the morphology can be deconstructed at four different length scales. The macroscale (macrostructure) is of the order of 10^{-3} to 1 m. Elements of the macroscale include shrinkage cavity, macro-segregation, cracks, surface roughness (finish), and casting dimensions. The mesoscale is of the order of 10^{-4} m. It allows description of the microstructure features at the grain level, without resolving the intricacies of the grain structure, mechanical properties are affected by the solidification structure at the mesoscale level. The microscale (microstructure) is of the order of 10^{-6} to 10^{-5} m. The microscale describes the complex morphology of the solidification grain in a sound casting, mechanical properties depend on the solidification structure at this scale level. To evaluate the influence of solidification on the properties of the materials, it is necessary to know the as-cast grain morphology (size and type, columnar or equiaxed) and the length scale of the microstructure (interphase spacing, dendrite arm spacing and eutectic lamellar spacing). The nanoscale (atomic scale) is of the order of 10^{-9} m (nanometers). At this scale, nucleation and growth kinetics of solidification are discussed in terms of the transfer of individual atoms from the liquid to the solid state.

Nucleation and growth are discussed in some detail in the following sections, two basic phenomena must take place in the liquid for solidification to occur: undercooling and nucleation. If these conditions are met, the nuclei can grow into the new solid grains. The monitoring of this stage of formation of solid structure is impossible in case of metallic alloy. As they are opaque, and the solidification temperature is relatively higher ($600\text{-}1000℃$) for non-ferrous alloy or very high ($> 1500 ℃$) for ferrous alloys. The current project aims at enabling observation of such very early stages of solidification. For this purpose, use of transparent alloys and mold walls allows witnessing and recording the event microstructure formation and melt convection. A copper-wall mold provided with transparent glass windows is to be constructed and provided with cooling/heating circuits to control the cooling rate inside the mold. Thus, the development of solidification structure and the associated phenomena can easily be observed by naked eyes and recorded for further analysis and studies.

CHAPTER 2

THEORETICAL REVIEW

2.1 SOLIDIFICATION PHENOMENA

2.1.1 Nucleation

Nucleation it can be define as a birth of the solid phase. It is genesis of a new phase in a distinct region, separated from the surroundings by a definite boundary. When a liquid cools a change of state from liquid to solid which may occur corresponding to a discontinuity in free energy. This results in the formation of small particles surrounded by liquid. The consequences of this process can be seen in the final microstructure and mechanical properties of the solidified metals and alloys. understanding the essence of this process will help to control the solidification morphology and reduce the defects which in turn improves the mechanical properties of the final components. It is convenient to classify the types of nuclei available in the melt as resulting from homogeneous nucleation, heterogeneous.

Solidification requires an outflow of heat which changes the free energy and the thermodynamic stability of the present phases. The transformation from one equilibrium state to another intermediate state of energy as shown in Fig.2.1.

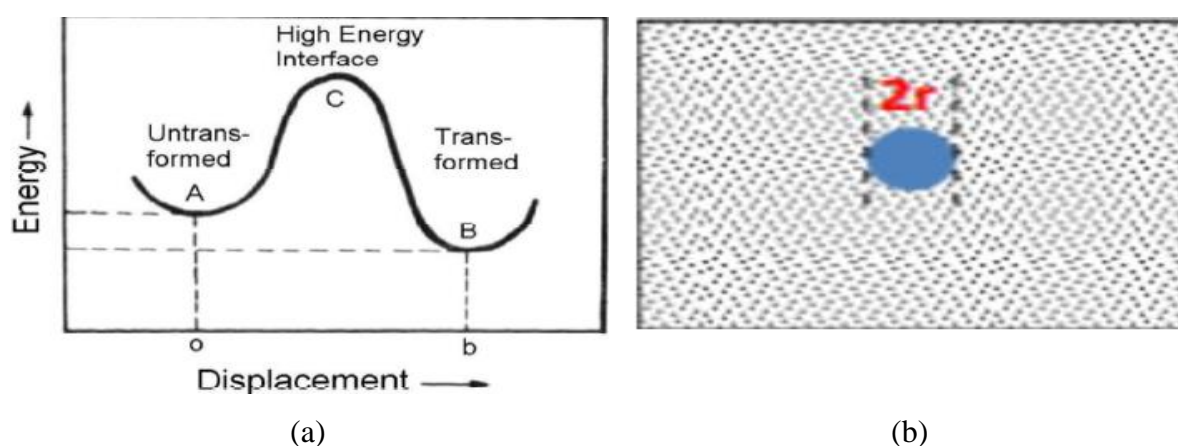(a)   (b)

Fig.2.1 (a) Energy relationship during a phase transformation and **(b)** A sketch showing a spherical nucleus of solid of radius r formed in a pool of liquid has volume $\frac{4\pi}{3}r^3$ and surface area $4\pi r^2$.

Movement of the atoms during nucleation to form clusters requires irreversible activation energy. From this it can conclude why solidification can never be an equilibrium process. It is necessary for the nucleation that clusters or embryos of solid atoms are formed where the liquid atoms can jump and stick. first clusters or embryos can exist randomly due to the thermal fluctuations, even over the melting point, but those will be unstable.

Below the melting point very small clusters continue to be unstable and remelted again due to the. relatively high energy required to create the new surface (where surface-to-volume ratio is too large), however the others with radius greater than a certain size ($r \geq r_0$) will survive to form the stable nuclei that grow steadily.

The phenomenon of nucleation of crystals from its melt depends on two processes: thermal fluctuations which lead to the creation of previously sized crystal embryos; and creation of an interface between the liquid and the solid. ΔG it is the total Gibbs free energy resulting from the local formation of a unit volume of solid arises from two sources as given in equation (2.1) and schematically represented in Fig.2.2 The first term, ΔG_i [J/mol], is the interface energy depends on free surface energy of the solid/liquid interface and is proportional to r^2; and the second term, ΔG_v [J/mol] Gibbs free energy per mol, depends on the difference in free energy between solid and liquid. ΔG_i always positive, while the ΔG_v is zero at the equilibrium temperature, negative below, and positive above.

$$\Delta G = \Delta G_i + \Delta G_v$$

$$\Delta G = \alpha \cdot \sigma + v \cdot \Delta_g$$

$$\Delta G = 4\pi r^2 \sigma + \frac{4}{3} \pi r^3 \Delta g_v \qquad \text{Eq(2.1)}$$
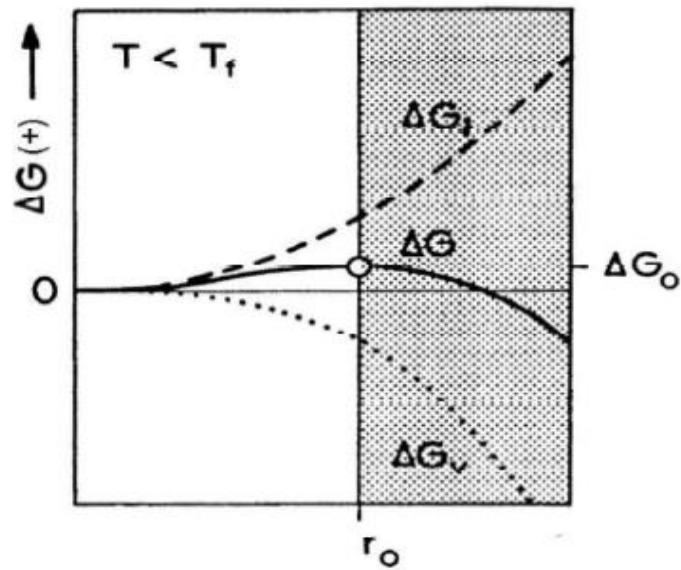
Fig.2.2 Free energy of a crystal cluster as a function of its radius at temperature below the melting point. A stable nucleus is likely to form in the grey zone where ΔG is starting to decrease.

Where σ [J·m-2]: solid/liquid interface energy, Δh_f [J·m-3]: latent heat of fusion per unit volume, Gibbs free energy per unit volume $= \Delta g_v = \Delta h_f \cdot \Delta T / T_f$, a: interface surface area, and ΔT : undercooling. The critical radius, at which r_0, ΔG starts to change its sign towards the stability can be obtained by differentiating both sides of equation (2.1) with respect to r and equating them with zero. The resulting radius is given in equation (2.2). The maximum energy change that occurs at the critical nucleus, ΔG_O, is called the work of nucleation or the activation energy, A. The value of A is infinity at Tf although fluctuating clusters of atoms do exist. Below freezing temperature (T_f) values of both A and r_0 Decreases as ΔT increases.

$$r_0 = - \frac{2\sigma T_f}{\Delta h_f \cdot \Delta T} \qquad\qquad Eq(2.2)$$

9

Cooling curve

The best way to monitor the process of solidification is to measure its temperature by a thermocouple. Fig.2.3 gives a plot of temperature (T) as a function of time (t). It shows that temperature keeps dropping continuously till it reaches a temperature when cooling stops till the liquid gets transformed totally into solid. Thereafter temperature keeps dropping again.
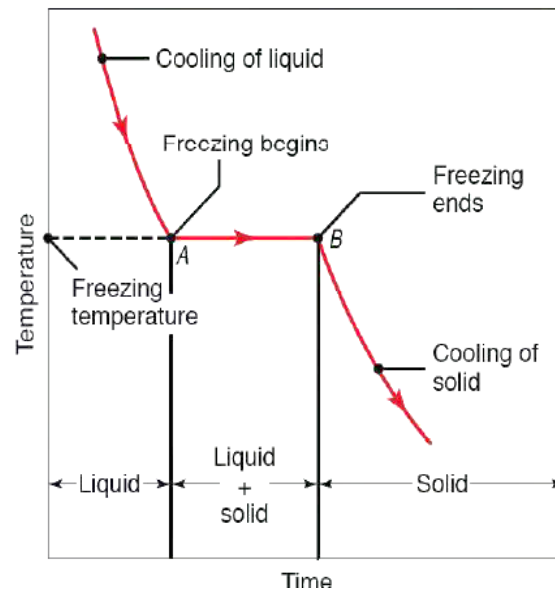
Fig.2.3 Temperature as a function of time for the solidification of pure metal.

Real cooling curve: super (under) cooling

The cooling curve of a pure metal as shown in Fig 2.3 is indeed an idealization of the solidification process. In reality the transformation from liquid to solid state begins only after it has cooled below its melting point. Fig.2.4 shows a sketch of real cooling curve. Once the process initiates the latent heat that is released by the metal raises the temperature back to its melting point. Thereafter the temperature remains constant till the solidification is complete.
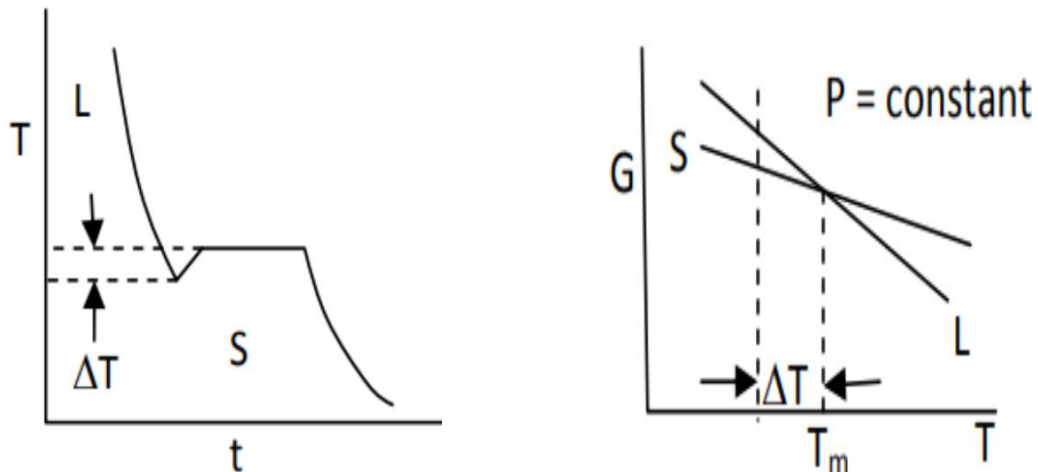
Fig.2.4 A sketch showing real cooling curve. Along with G versus T plot of the solid and liquid phases have been shown. At a temperature lower than its melting point $G_s < G_L$ signifying S is more stable than L.

Homogeneous nucleation versus Heterogeneous nucleation

The process of nucleation described above is totally random. Grains may nucleate anywhere in the melt. homogeneous nucleation it is not common in casting alloys. in the general case of freezing within the bulk of pure molten metal, minute crystalline nuclei form independently at random points continued removal of thermal energy from the system causes these small crystalline regions to grow independently at the expense of the surrounding melt. It is also homogeneous nucleation if the orientations of the grains are represented in a standard projection by their cube poles these will be located uniformly all over the entire standard projection. Such plots are known as pole as shown in Fig.2.5.

Homogeneous nucleation most likely to happen if the molten pool of metal has no free surface. This however is only a hypothetical case which may occur only at the center of the mold containing the molten metal. Mold surface provided an interface where solid may nucleate.

(a)   (b)
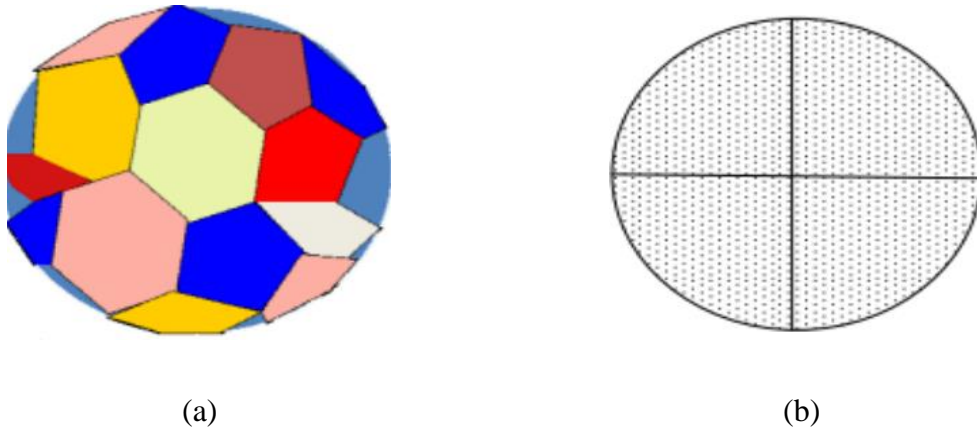
Fig.2.5 (a) Shows a typical microstructure of a pure metal each grain having different orientations. (b) This shows distribution of (001) poles of all the grains on a standard projection. This type of diagram is known as pole figure.

When it takes place on a pre-existing solid surface the process is known as heterogeneous nucleation. This is shown in Fig.2.6 Since the mold surface provides a part of the surface energy it would need less driving force (super cooling). Thus providing such nucleation sites it is possible to promote solidification.

More number of sites for nucleation finer are the grains. This is one of the ways of having fine grain structure on solidification. The process is known as inoculation where very small amount of easily dispersible fine solid particles are added to the metal as it poured into mold.

(a)   (b)

Fig.2.6 (a) A sketch showing homogeneous nucleation of a solid spherical ball in a molten pool of metal in a hypothetical mold without any boundary. (b) Sketch showing a case where apart from an embryo at the center there are several nuclei each formed on the interface between the mold wall and the liquid metal.

In Fig.2.7 Illustrates the role played by the respective surface tension (or surface energy; both represent the same physi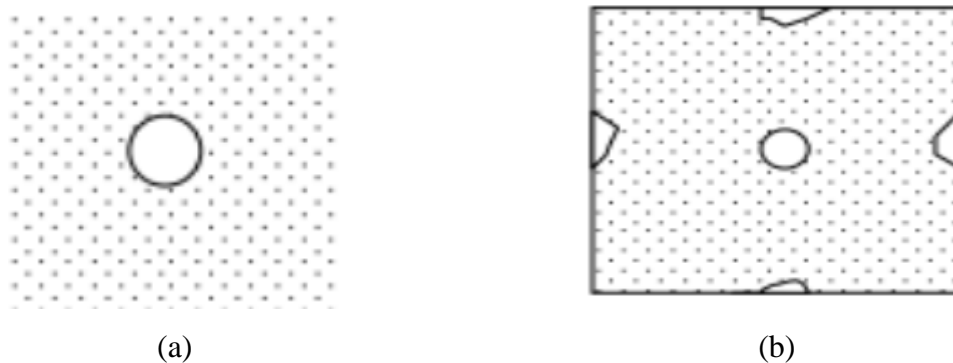cal parameter) and the contact angle (θ). In this case the mold surface acts as a substrate on which an embryo of the solid nucleates. At the point of contact there are three surface forces; one between the mold and the embryo (σ_{ME}), the second between the mold & the liquid (σ_{ML}) and the third between the embryo & the liquid (σ_{EL}). The angle between EL and ME is known as the contact angle. Invoking the condition of equilibrium between the three forces it is possible to find out the condition that would promote nucleation.

(a)   (b)
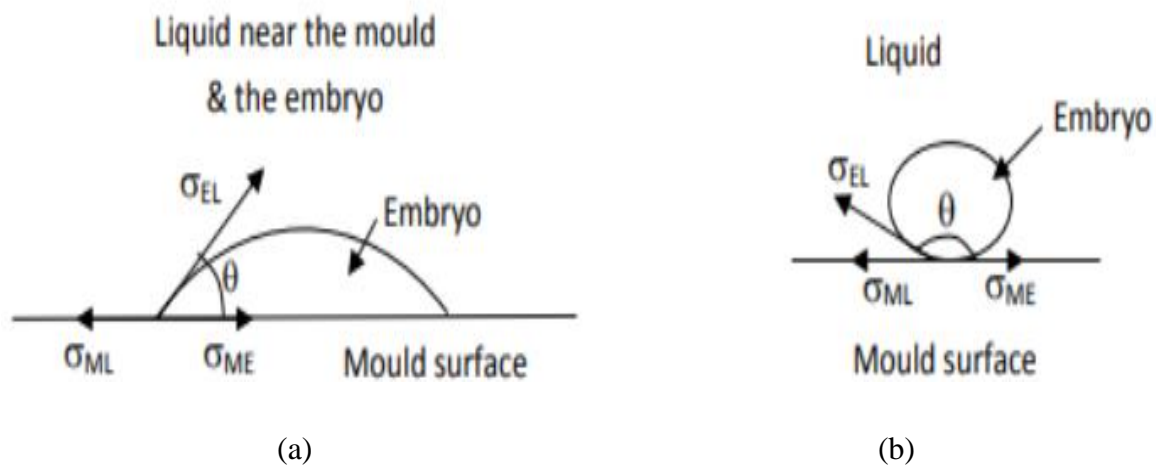
Fig.2.7 A sketch illustrating the role played by the surface tensions between the three surfaces that come in contact during the nucleation of an embryo. (a) Shows the case where θ < 90 or σ_{ML} > σ_{ME} and (b) shows the case where θ > 90 or σ_{ML} < σ_{ME}.

2.1.2 Growth

Growth process occurs in the solidification when molten metal changes from a liquid state to a solid state, the growth process is got when the heat is continuously extracted from the molten metal until the temperature is below the freezing point. As the heat extraction rate increases, the solid/liquid interface advance correspondingly rate increases.

For pure metals, as the solidification front is seen to go through a series of transitions. Initially it is planar; at higher rates of advance it develops deep protrusions, spaced regularly over the previously planer front. This type of growth is called cellular growth. At higher velocities still, the cells grow into rapidly advancing projections, sometimes of complex geometry. Their tree-like forms have given them the name dendrites.

In case of alloys, the three growth forms are similarly present as schematically illustrated in Fig.2.8 However, the driving force for instability is the constitutional undercooling of liquid which arises because of the segregation of alloying elements ahead of the front. The presence of this extra concentration of alloying elements reduces the liquids temperature of the newly developed alloy constitution in liquid. If this reduction is sufficient to reduce the melting point to below the actual temperature at that point, then the liquid is said to be locally constitutionally super cooled. The difference between the cellular grains with a planer front formed in pure metals and in alloys in case of alloys.
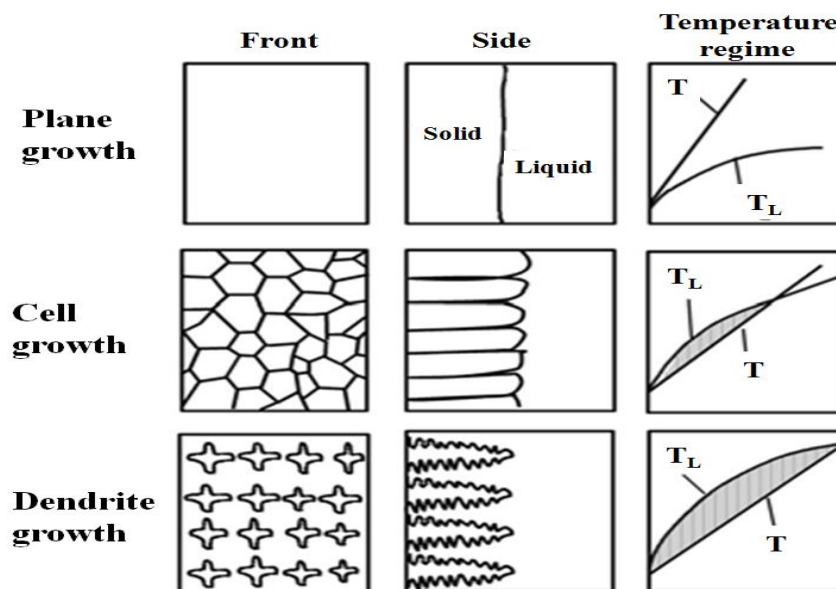
Fig.2.8 the transition of growth morphology from planar, to cellular, to dendritic, as compositionally (constitution) induced undercooling increases.

Cellular growth happens when the advancing planar solid-liquid interface becomes unstable shown Fig.2.9. and a small spike appears on the interface that then grows inside a cellular type structure. The planar surface becomes unstable because any section of the interface that grows ahead of the rest enters an area in the liquid that is at a lower temperature. The first spikes that form remain isolated at first, because as they grow by solidification, they release their latent heat of fusion into the next to something else liquid, causing a localized increase in the temperature. As a result, parallel spikes of almost equal spacing advance into the liquid. [1]

Dendritic growth is a further visible sign of cellular growth in which the spikes develop side protrusions. At still higher undercooling and higher growth speeds, the cells grow into quickly advancing projections, sometimes of complex geometry. Their treelike forms have given them the name dendrites, after the Greek word dendrons for tree shown Fig.2.9Fig.2.10 The secondary arms of dendrites develop perpendicular to the primary arms because, as the primary arm turns into a solid and gives off its latent heat of reaction, the temperature immediately next to the primary arm increases. [1]

Heat Flow
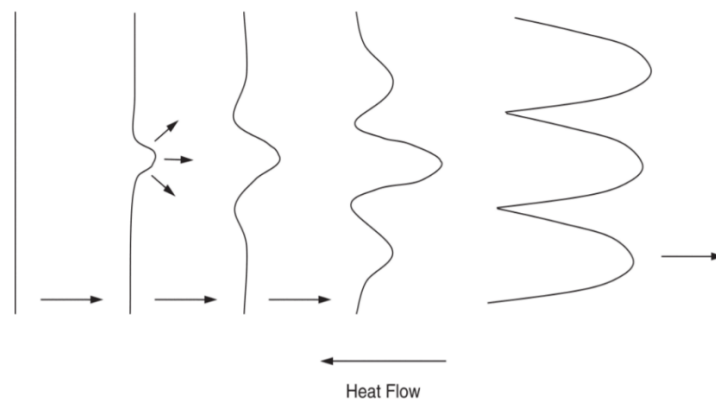
Fig.2.9 Transition from planar to cellular growth.
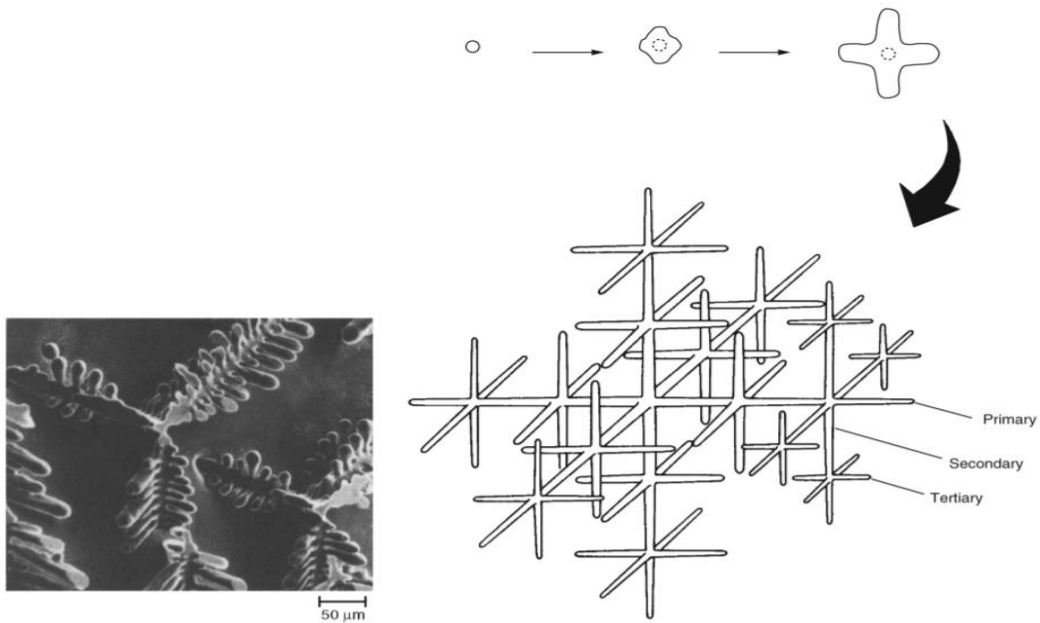
Fig.2.10 Dendrite formation.

2.1.3 Structure Formation

A metal cast into a mold can have up to two or three separate zones: a chill zone, a zone containing columnar grains, and a center-equiaxed grain zone shown Fig.2.11 .It should be noted that all three zones do not always happen. For example, pure metals can show a chill zone and a columnar zone but do not include a center-equiaxed zone.

Fig.2.11 The different zones in a cast ingot. [**Fehler! Verweisquelle konnte nicht gefunden werden.**]

Chill Zone, Because the mold is cool more than the metal, nucleation will happen over the interior surface of the mold. Because the mold wall is cool more than the liquid metal, the nucleation rate is very high and thus the average grain size is very small. Each nucleation event produces a single crystal or grain, that grows dendritically in a direction roughly perpendicular to the mold wall until it harms on 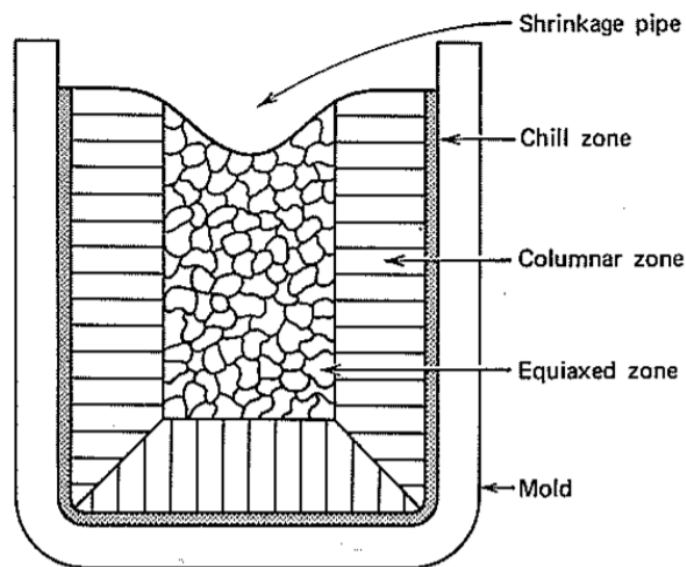other grains. As a large amount of latent heat of fusion is released from the indurate grains, and as the superheat of the liquid is wasted, the rate of solidification decreases. The chill zone grains are oriented at random with respect to the mold, that is the major axis of each grain is at random oriented. [1]

Columnar Zone, into of the chill zone, there are a series of columnar or column-formed, grains that are oriented parallel to the heat flow direction. Because each metal grows more positively in one principal crystallographic direction, only those grains positively oriented with their growth direction most perpendicular to the mold wall will grow into the middle of the casting. The axes direction of the columnar grains is parallel to the direction of heat flow and they grow along specific crystalline planes. As they grow, the more completely oriented grains grow ahead of the less completely oriented grains and crowd them out shown Fig.2.12.
The fact that the grains most positively oriented to the mold wall grow the fastest means that the final form of the grains in a pure metal casting is columnar, with the parallel columns growing progressively from the mold wall into the middle of the casting. The final columnar structure results from parallel growth of different colonies of dendrites and the progressive lateral ,growth or thickening between them.

Equiaxed Zone, a third area at the middle of some alloy casting consist of smaller grains that are randomly oriented and almost equiaxed. As freezing goes forward the thermal gradient decreases, and this causes the dendrites to become drawn-out. Breakdown of columnar growth may happen as a result of fracturing of the very long dendrite grains by convection currents in the melt. These broken arms Fig.2.13 can then avail as nuclei for new grains. Another chance is that new grains nucleate as a result of a low thermal gradient and segregation that is happening during freezing. At low temperature of casting the entire the casting may solidify with an equiaxed structure. [1]
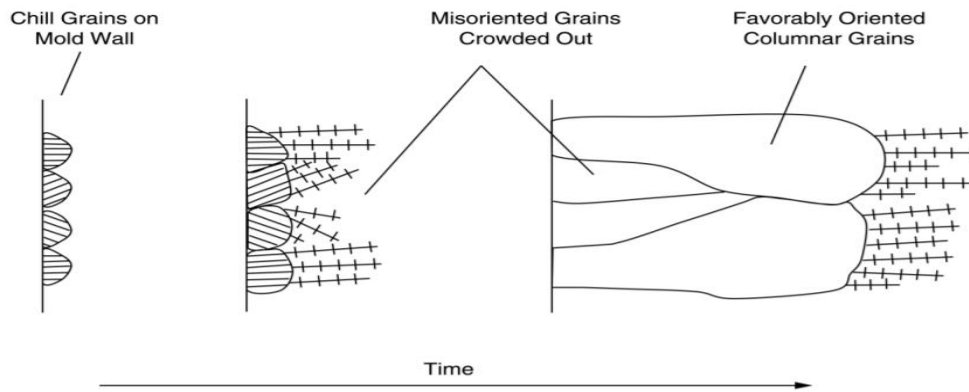
Fig.2.12 Dendritic growth from mold wall.
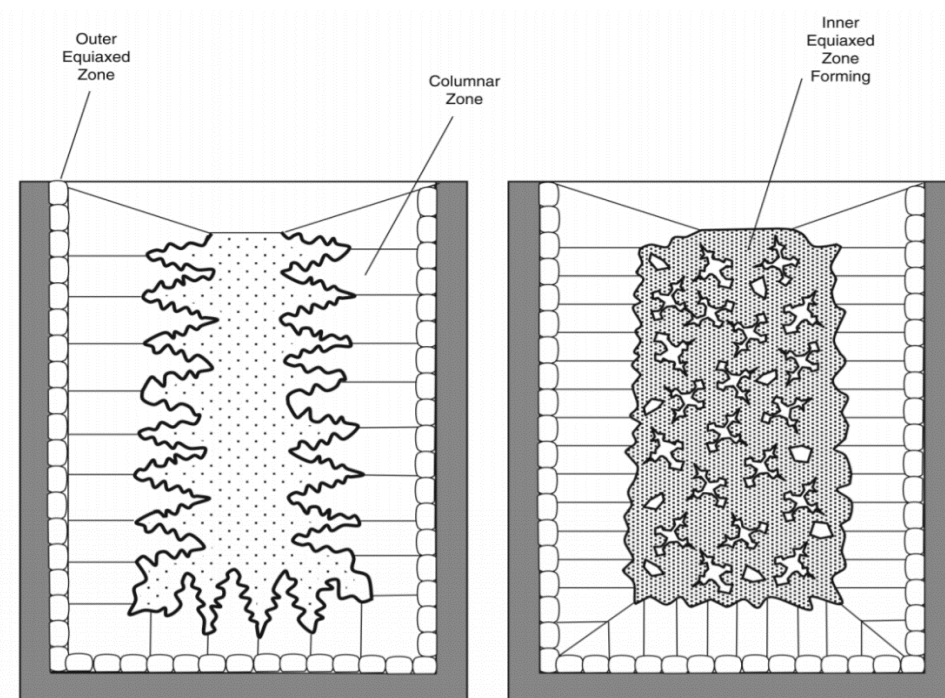
Fig.2.13 Formation of equiaxed zone in alloy casting.

The amount of the final form of cast structure that is columnar or equiaxed depends on the alloy complex and on the thermal gradient at the liquid-solid interface through solidification. A thermal gradient is most easy for controlled by controlling the heat extraction rate from the casting or the cooling rate. Alloys that have a very wide spread between the liquids and the solidus temperatures solidify with an especially equiaxed grain structure at normal cooling rates, while alloys with small differences in solidus and liquids temperatures solidify with an especially columnar structure. High cooling rates embolden columnar solidification because

they establish very high thermal gradients at the liquid-solid interface. Low thermal gradients embolden equiaxed solidification.

Temperature gradient G, and the solidification rate R, affects the solidification microstructure from the ratio (temperature gradient G, / solidification rate R,) can be determines the mode of solidification, the mode of solidification are planar, cellular, columnar dendritic or equiaxed dendritic structure shown in Fig.2.14.
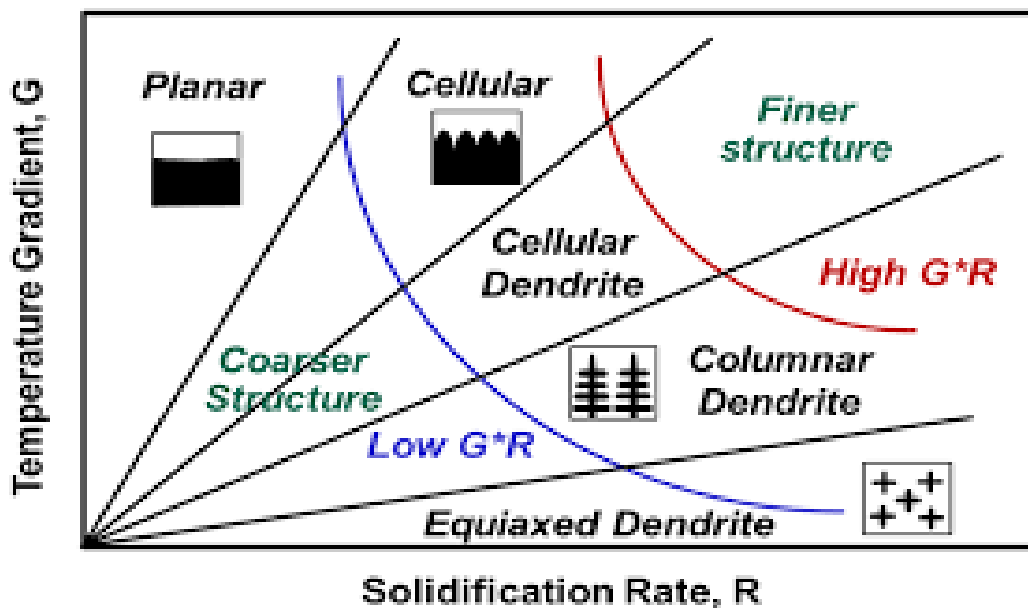

Fig.2.14 Effects of temperature gradient G, and solidification rate R, on the as-cast grain morphology.

Understanding the origin of the solidification structure is very helpful for controlling as-cast structure of the casting components and correspondingly, their mechanical properties. Three basic types of as-cast structures are known: (1) the columnar growth that is favored e g, in single crystal growth or in some turbine blades, (2) the equiaxed grain structure which exhibits more isotropic properties and characterizes with higher toughness, and (3) mixed columnar-equiaxed structure with a columnar-to-equiaxed transition zone. The latter as-cast structure exhibits undesirable anisotropic mechanical properties and have to be avoided either by modification of cooling conditions to get a fully columnar structure or by addition of grain refiners to the melt to change it to fully equiaxed.

2.1.4 Segregation

Segregation can be defined as any deviation from a uniform distribution of the chemical elements in the alloy. It is that develops within the grain due to the interaction of solute rejection by solidification with solute diffusion in the liquid and solid phases. Some variation in composition occurs on a microscopic scale (range in 10-100 μm) that's called microsegregation. There are many forms in microsegregation that may exist in castings as a consequence of the solidification process. when diffusion is allowed, the atoms migrate from higher concentration areas to the lower concentration areas. Diffusion is occurring only at high temperatures, for which reason a rapidly cooled alloy will be appreciably cored and a slowly cooled alloy only slightly cored, shown in Fig.2.15.
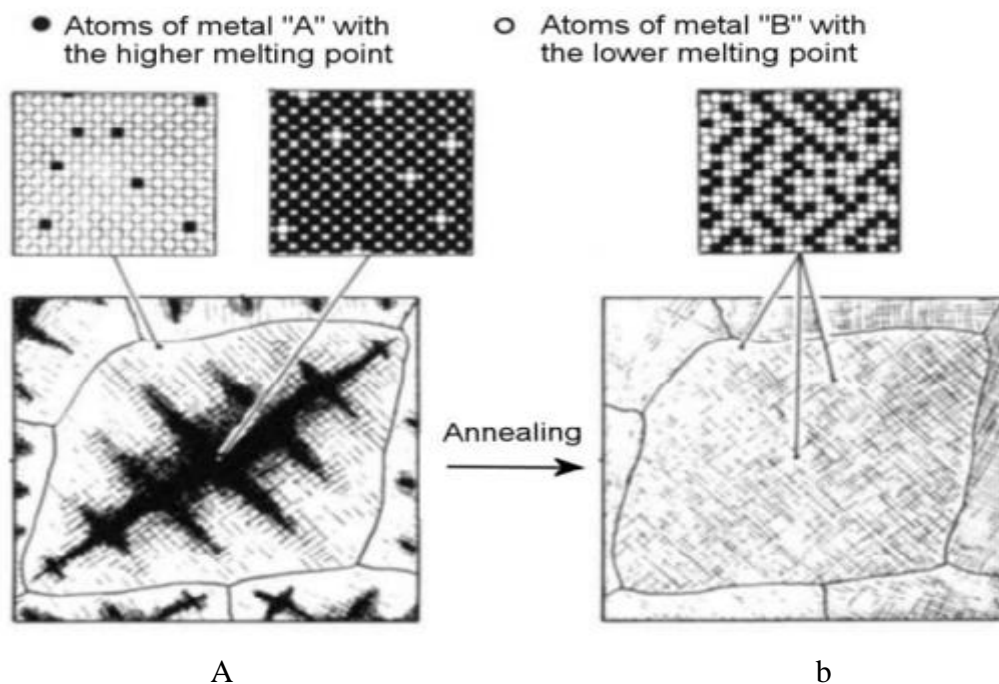

A   b

Fig.2.15 (a) Schematic of the formation of the cored structure,(b) the homogenization of the grains by annealing heat treatment.

It can occur microsegregation in other forms may arise as a result of impurities and oxide inclusions dissolved in the liquid metal. During solidification, impurities are pushed gradually out of the advancing solid front. The impurities in liquid increases gradually. At the end of solidification, the impurities either entrapped at the inter-dendrite spacing or precipitated at the grain boundaries as shown in Fig.2.16.
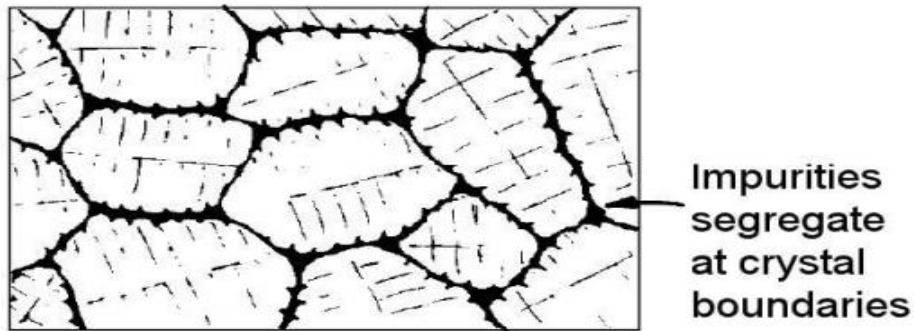
Fig.2.16 Segregation of impurities at grain boundaries

This type of segregation is the most damaging in its effect, since it will cause overall brittleness of the castings and, depending upon the nature of the impurity. It can be partially eliminated through subsequent forming processes. Also has another type of segregation which called macrosegregation this type is composition changes over distances comparable to the size of the specimen. It exists When casting size gets bigger, the melt and the inter-dendritic fluid flow becomes stronger. Thus become macro-scale-segregation forms. The natural melt convection is considered the essential driving force for the formation of the A-type and V-type defects in steel ingots and sand mold roller castings as shown in Fig.2.17 The interaction between the effects of gravity driven natural convection and the solute type and concentration leads to the formation the various macrosegregation defects in the cast ingots. Can be controlled the channel (V or A) segregates by decreasing the time available for their formation by increasing the rate of solidification.
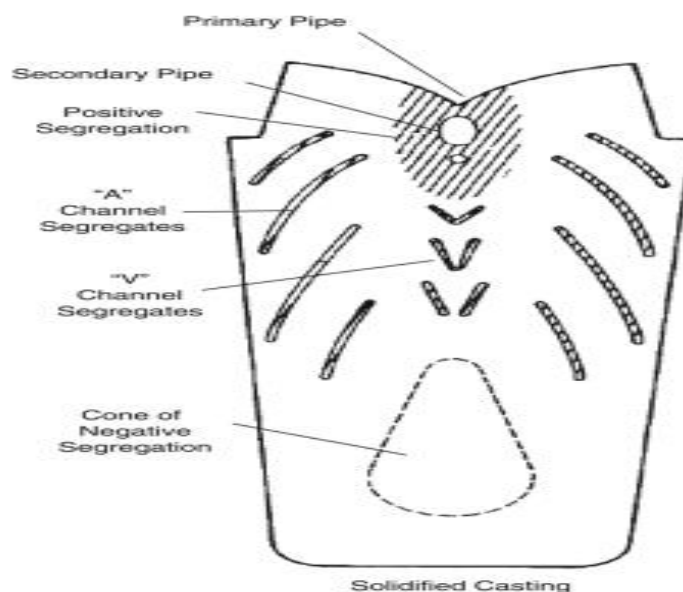

Fig.2.17 Macrosegregation defect in a conventional large steel ingot.

2.2 TRANSPARENT ALLOYS

2.2.1 - Why transparent alloys used?

Macrosegregation not only renders the composition inhomogeneous at the scale of the entire product, but it can also induce segregation at the mesoscopic scale through the formation of freckles (important defect in casting). The transparent NH_4Cl-water system used to visualize freckle formation as shown in Fig.2.17. [3]
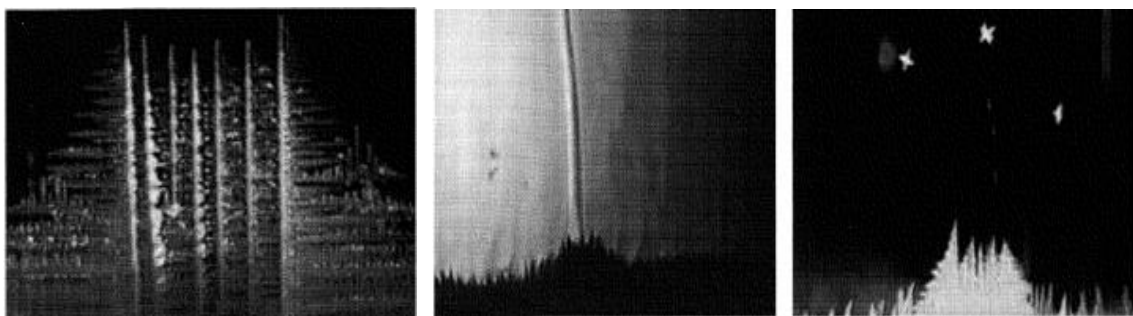

Fig.2.18 Freckle formation in the NH_4Cl-water system. The top left panel shows a plume of liquid, visible due to the variation of the refraction index with composition. In the top right panel, the entrainment of some fragments by the plume is displayed using dark filed illumination. The bottom panel shows the interior of chimney with the accumulation of fragments.

2.2.2 Types of transparent alloys

i - Succinonitrile (SCN)-Salol alloys

Fig.2.19 shows some examples of microstructures observed during the directional solidification of Succinonitrile (SCN)-Salol alloys. In this experiment, is confine a thin specimen of a dilute alloy between two microscope slides that are placed on a microscope stage between a hot block and cold block Fig.2.19. The specimen is then translated at constant pulling speed (VP) and microstructure is observed. By using a fixed temperature gradient, (G) and various pulling speeds (VP) obtained sequence of micrographs.

At very low velocities (not clear), the interface is planar above a critical speed, which we compute below, shallow cells develop, separated by grooves parallel to G. The cells do not

follow any particular crystallographic orientation and are characterized by a large radius of curvature. When the solidification speed increases, the grooves will be deeper and the cell spacing decreases. At higher speed, there is a gradual transition to dendrites characterized by much sharper tips and by trunks whose orientation follows the preferred growth direction, and by side branches. [3]
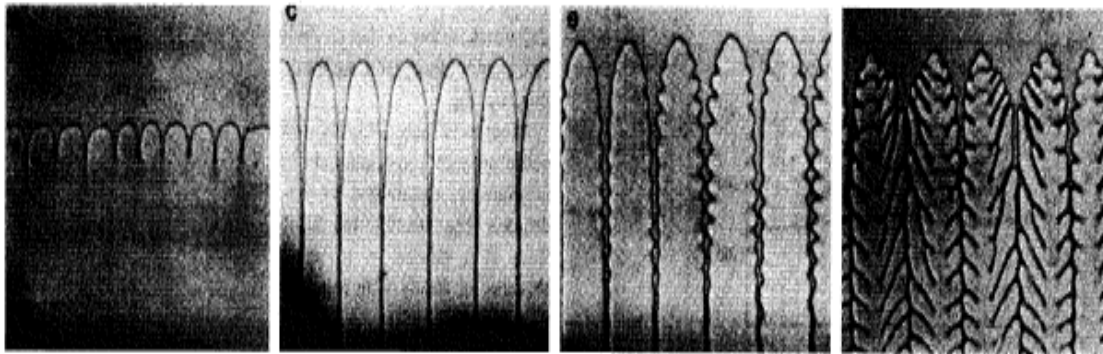

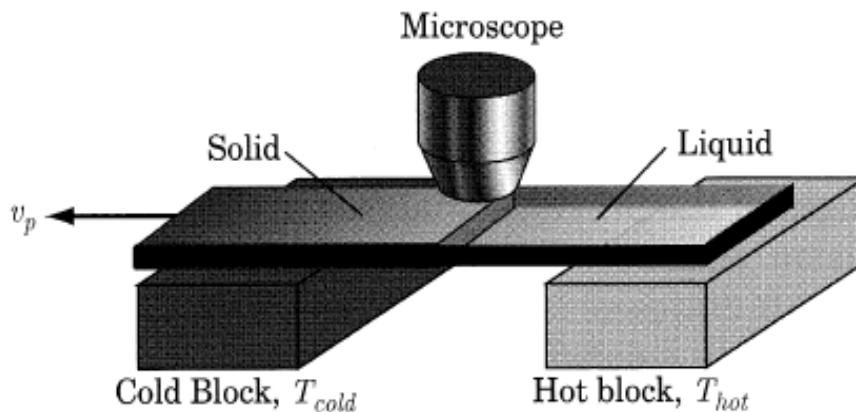**Fig.2.19** Solidification of Succinonitrile (SCN)-Salol alloys.

Fig.2.20 A schematic drawing of a laboratory scale directional solidification apparatus.

ii - Succinonitrile-Camphor alloy

Fig.2.21 shows partial remelting and detachment of side arms in columnar dendrites in a Succinonitrile-Camphor alloy after an abrupt change in growth rate and gradient temperature during Bridgman solidification. Fragmentation by thermal effects without convection is of course much less effective in promoting a transition from columnar to equiaxed formation, because the fragments remain more or less in place. This effect can be detrimental to the

production of single crystal turbine blades, leading to so called (zebra grains) that are slightly misoriented with respect to the main grain. [3]
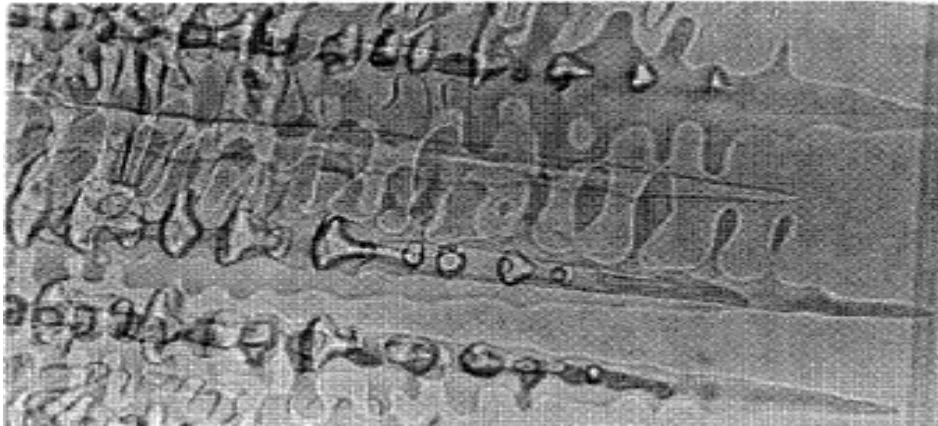

Fig.2.21 The partial remelting and detachment of dendrites side arms in the Succinonitrile-Camphor alloy during a change in growth rate and thermal gradient during Bridgman solidification.

iii - Borneol-Succinonitrile alloy

For irregular eutectics when one of the two phases, e.g., a, grows with a non-faceted morphology while the 3-phase is faceted, triple junctions can be maintained: the non-faceted phase follows the plates or needles of the faceted phase. The faceted phase grows along well-defined directions by the help of defects such as twins or screw dislocations. Therefore, the resulting eutectic structure is irregular and very complex, giving rise to an irregular eutectic morphology, as sketched schematically on the right-hand side of Fig.2.22. This type of eutectic is found in metalic-non metalic systems e.g. Fe-C (gray cast iron) and Al-Si(Fig.2.22 b), and in various organics such as the Borneol-Succinonitrile alloy (Fig.2.22 d). [3]
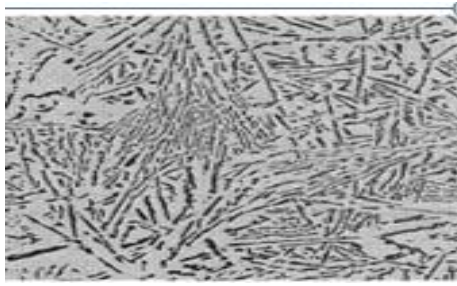
(a) Al-Au
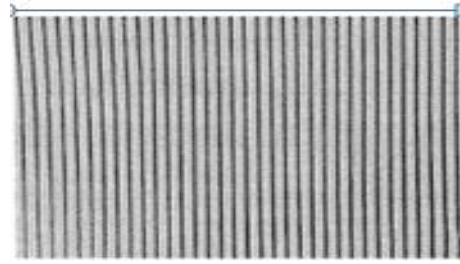
(b) Al-Si
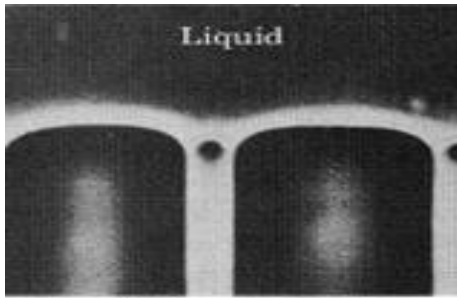
(c) CBr$_4$-C$_2$Cl$_6$

(d) Borneol-SCN
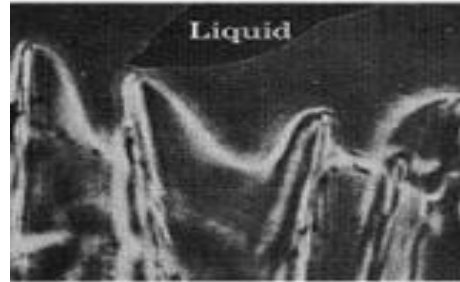
Fig.2.22 show regular and irregular eutectics: (a) regular Al-Au eutectic observed in a longitudinal section, (b) irregular Al-Si eutectic observed in a transverse section, (c) regular CBr$_5$-C2Cl$_6$ eutectic observed during growth, and (d) an irregular Borneol- Succinonitrile eutectic observed during growth. The faceted borneol phase leads the growth of Succinonitrile in between.

iv- Ammonium chloride (NH$_4$Cl)

Advection is the transport of heat and solute by fluid flow. It plays a very significant, or even dominant role in transport near solidifying interfaces. The fluid flow can be induced by pouring of the melt into a mold, buoyancy-driven flow due to thermal and solutal gradients, which affects the dendritic microstructures. Fig.2.23 shows the shape of a denser NH$_4$Cl crystal falling while growing in an undercooled NH$_4$Cl-water melt at uniform temperature. The falling crystal experiences an apparent incoming flow, caus- ing the downward pointing primary arm to grow faster than the upward pointing one. Primary arms growing perpendicular to the relative move- ment have their orientation biased downward slightly by the same mech- anism. This also occurs for dendrite trunks of columnar grains growing in a flow parallel to the isotherms. In order to model the structures that result from the movement of grains during solidification, one must also take into account the momen-tum transfer between the solid and liquid phases.Consider again the single grain shown in Fig.2.23 a, growing and settling in an

otherwise quiescent liquid.The drag force on the dendritic grain is complicated to express analytically, because the flow passes both through and around the grain.
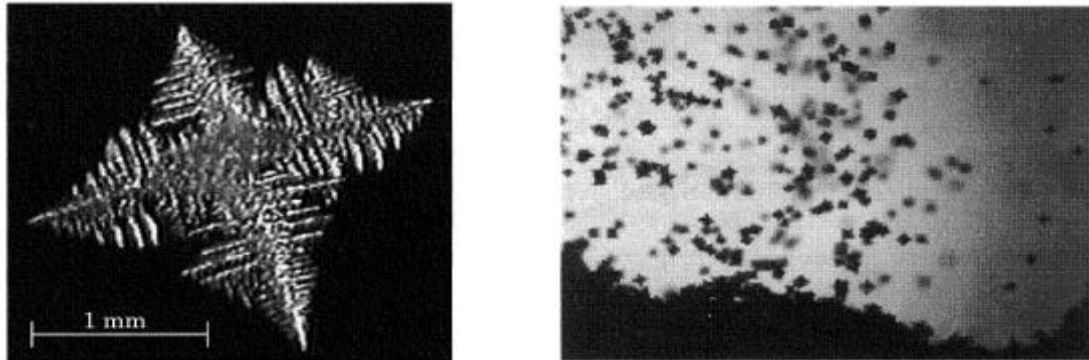
<center>(a)   (b)</center>

Fig.2.23 (a) The shape of an Ammonium Chloride dendritic crystal growing and settling in an undercooled NH4Cl-water melt. (b) The movement and settling of a family of Ammonium Chloride crystals in a rectangular cavity cooled from all sides.[3]

2.2.3- Properties of Ammonium Chloride

Table.2.1 Properties of Ammonium Chloride – NH4Cl

NH4Cl	Ammonium Chloride
Molecular Weight/ Molar Mass	53.491 g/mol
Density	1.53 g/cm³
Freezing Point	-0.322 °C
Melting Point	338 °C
Boiling Point	520 °C

2.3 PREVIOUS STUDIES FOR NH$_4$Cl

1- Experimental observation of convection during solidification of transparent alloys.

It has been solidified NH$_4$Cl-H$_2$O solutions under defined experimental condition. The happening melt convection was scanned by Particle Image Velocimetry (PIV). the observed of NH$_4$Cl crystals attempts were made to quantitatively measure its number density and sedimentation rate by (PIV) and Particle Tracking. In order to prove the of the results several experimental runs with equal and slightly modified conditions were analyzed. This study has demonstrated the use of Particle Image velocimetry and Particle Tracking in a solidification experiment. Have illustrated that the conventional flow field, particle numbers (PN) , and sizes can be quantified with this technique. In combination with the initial concentration and the measured temperature, these quantities will be taken as benchmark for numerical.[4]

2- Measurement and simulation of temperature and velocity fields during the cooling of water in a casting model.

Simultaneous measurements of temperature and velocity have been performed by applying combined laser induced fluorescence (LIF) and particle image velocimetry (PIV) techniques to physical convection in a cast model. A 10 cm×10 cm×3 cm U-shape rectangular casting model has been filled with pure water and have been cooled the cell walls continuously between 41 and 6 ℃ The resulting natural convection pattern and temperature field have been measured with (PIV) and LIF. Furthermore, the convection pattern has been simulated in (2D) and (3D) using the commercial (CFD) software FLUENT. The comparison experimental and numerical results given shows the good agreement both qualitatively and quantitatively. The achieved agreement between numerical prediction and experimental results confirms the applicability of the reported setups as starting points for both experimental and numerical investigations of the convection during hypereutectic NH$_4$Cl–H$_2$O solidification in casting model.[5]

CHAPTER 3

CONSTRUCTION OF SOLIDIFICATION MOLD

3.1 PHASE DIAGRAMS OF (NH₄Cl-H₂O)

The analysis of problems involving the casting of metal and alloys requires a complex combination of fluid mechanics, heat flow, chemical diffusion and solid mechanics. However, the phase diagram, especially when applied to the character of a material at a given position and time, provides the basic constitutive relation of the alloy regarding the physical state. The information from a phase diagram must be placed upon a framework of mass, momentum and energy balance equations that describe the kinetics of a given situation.

A major use of phase diagrams comes about in the prediction of the degree of microsegregation and second-phase formation. Additionally, the path solidification can be calculated, and the prediction made of the fraction solid when the liquid can start formation of second-phase particles, inclusions or eutectics. Such liquid compositions are directly read from phase diagrams.

It is intended in the current work to focus the study on the aqueous Ammonium Chloride solution with composition NH_4Cl-70%Water. As shown in Fig.3.1, this solution starts solidification at $\sim 30°C$, and ends its solifdification at the eutectic line at $-15.4°$ C. As heat is removed from the mold walls, grwoth of solidifed crystals continues starting from the walls towards the bulk liquid. Since the solidification of a binary solution occures over a range of temperatures; hence, a mushy zone containing dendrites appears during the solidification process. [6]
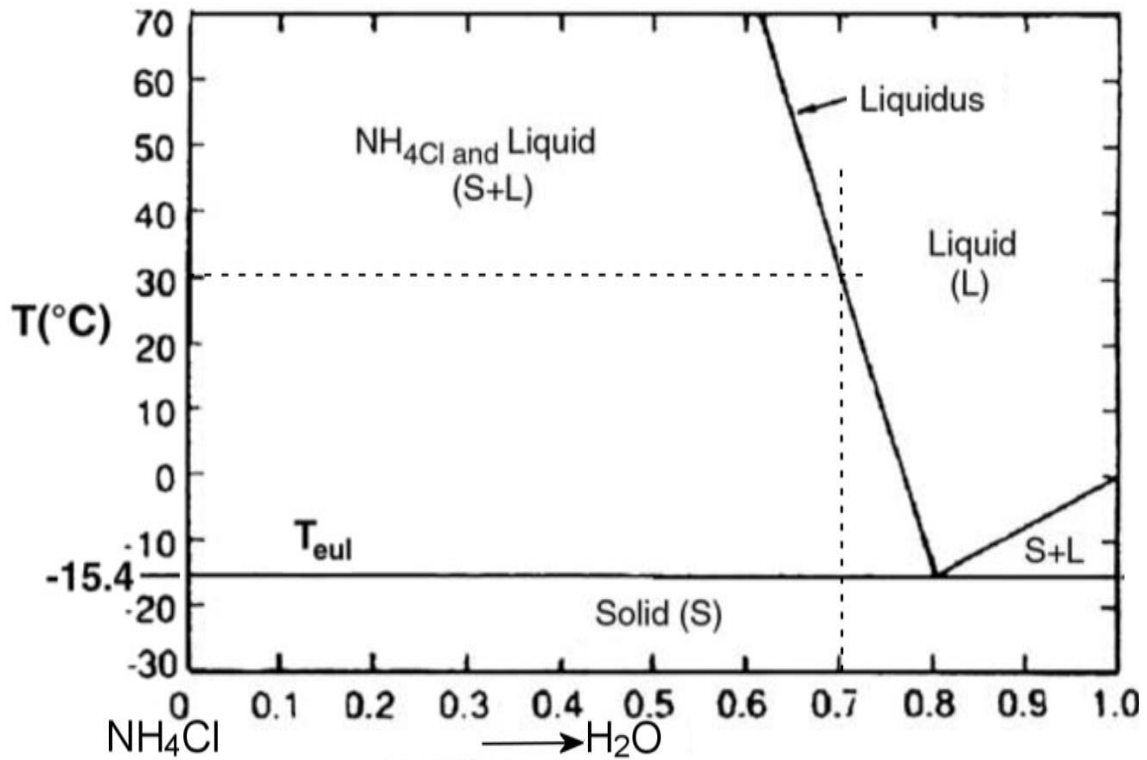
Fig.3.1 A typical phase diagram of NH$_4$Cl+H$_2$O ($\omega_e = 19.7\%$ and $T_e = -15.4°C$ [1]

Table3.1 show the thermo-physical properties of **NH$_4$Cl-70%H$_2$O.** [6]

Thermo-physical properties	Value
Specific heat(C_p)	3000 $J/(kg.k)$
Thermal conductivity (k)	0.4 W/mk
Density (p)	1000 kg/m^3
Specific diffusion coefficient (D$_1$)	4.8×10^{-9} m^2/s
Eutectic temperature (T$_e$)	-15.4 °C
Eutectic concentration (C$_e$)(wt%NH$_4$CL)	19.7
Latent heat of fusion (L$_a$)	3.0× 10^5 j/kg
Melting point of pure H$_2$O (T$_m$)	0 °C

3.2 DESIGN OF MOLD

The materials will used to construct this mold is double Plexiglass from two sides with small space between them show in Fig.3.2. Transparency and other outstanding properties including toughness, light weight, formability, the control of light ray paths, and good chemical resistance allow Plexiglass sheet to be used where other transparent materials may fail. In colorless form, Plexiglass is as transparent as the finest optical glass. Its total light transmission is 92%, and its measured haze averages only 1%.

Metal of three walls used in this solidification mold it is Copper. Every wall mold contain plate of Copper and ceiling cover joint together by a bolts and there path between them to pass the cooling and heating water. The walls it can be cooled and heated separately or together in a controlled manner. The reason of used copper because it is a good conductor of heat. thermal conductivity of Copper is higher than most other metals.
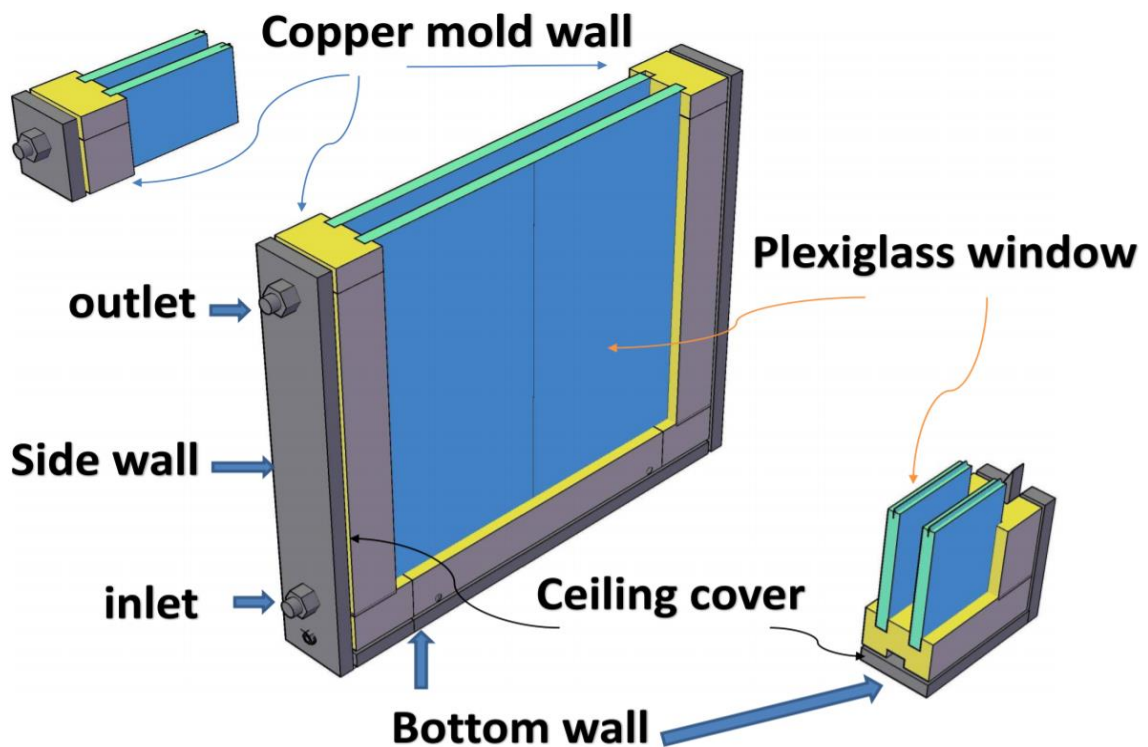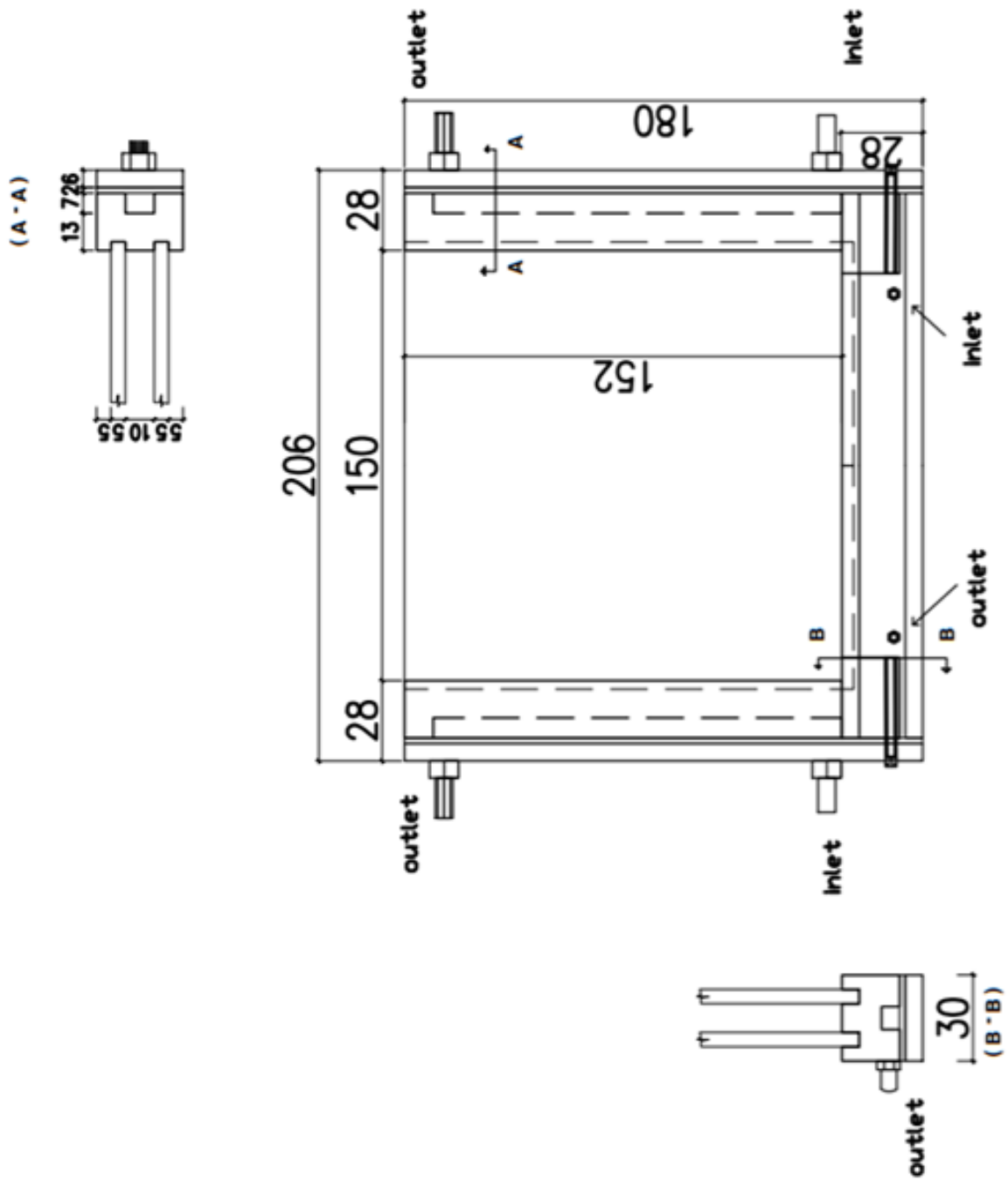

Fig.3.2 Design of mold (3D)

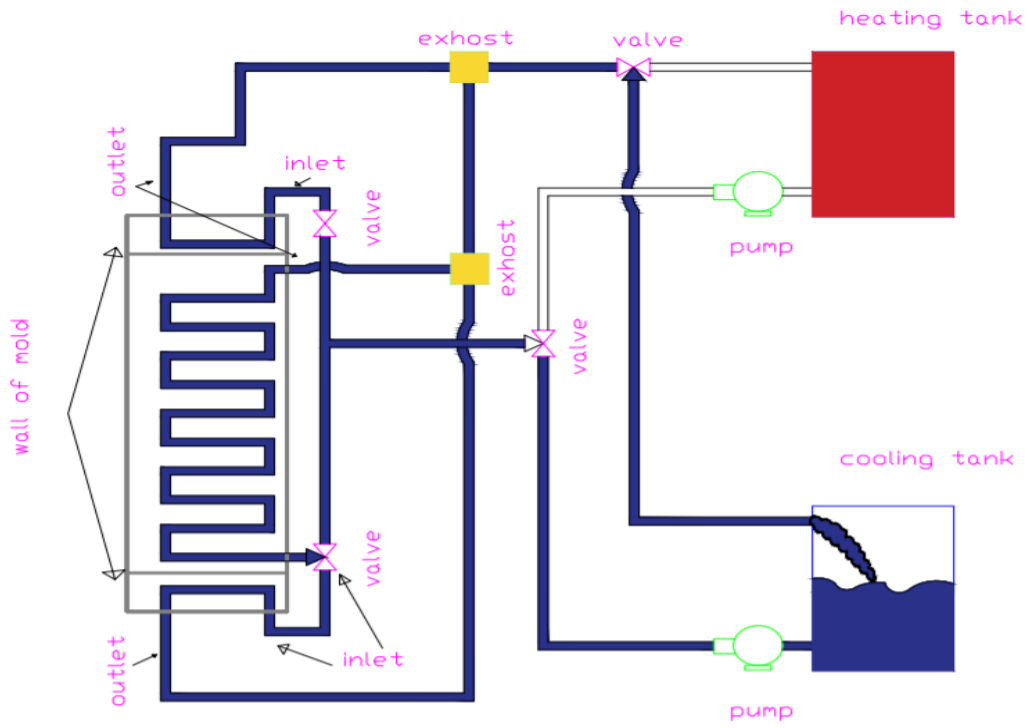**Fig.3.3** Design of mold with dimensions in (mm) (2D).
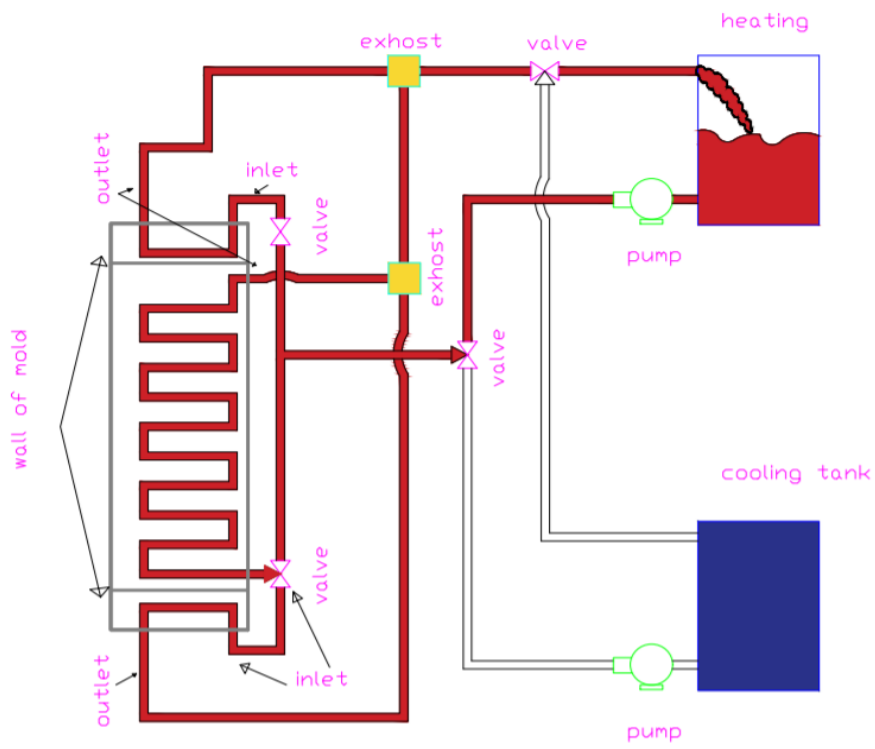
Fig.3.4 Mold-cooling cycle.

Fig.3.5 Mold-heating cycle.

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