DIRECTIONAL SOLIDIFICATION USING MICROWAVE CASTING

A DISSERTATION

Submitted in partial fulfilment of the requirements for the award of the degree

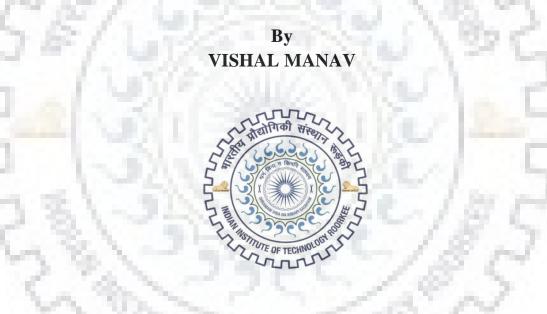
of

MASTER OF TECHNOLOGY

in

MECHANICAL ENGINEERING

(With Specialization in Production and Industrial Systems Engineering)



DEPARTMENT OF MECHANICAL AND INDUSTRIAL ENGINEERING INDIAN INSTITUTE OF TECHNOLOGY, ROORKEE ROORKEE-247 667 (INDIA) JUNE 2019

CANDIDATE'S DECLARATION

I hereby declare that the work carried out in this thesis titled "DIRECTIONAL SOLIDIFICATION USING MICROWAVE CASTING" is presented in the partial fulfillment of the requirement for the award of the degree of Master of Technology with specialization in Production and Industrial Systems Engineering submitted to the department of Mechanical & Industrial Engineering, Indian Institute of Technology Roorkee, India, under the supervision and guidance of Dr. APURBBA KUMAR SHARMA, Professor, MIED, IIT Roorkee, India.

I have not submitted the matter embodied in this report for the award of any other degree or diploma.

Date: June 2019 Place: Roorkee

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CERTIFICATE

This is to certify that the above statement made by the candidate is correct to the best of my knowledge and belief.

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ABSTRACT

Energy, time saving and reducing emissions are primary goals of all countries around the world. It is the need of time to use green technology for sustainable growth and development of the nation. According to worldwide census of casting production of 2017 over 109 million metric tons of castings are produced annually. India is the second largest producer of castings. Therefore, time and energy saying in foundries by increasing efficiency in production line can help to save resources for manufacturing sector and reduce emission is the main focused aim which is the mother for developing new processing and manufacturing technologies to reduce production or manufacturing cost, processing time, and enhancing manufactured product properties and thus reliability. It is very important that the developed processing techniques should be widely acceptable for all types of materials. Microwave materials processing is emerging as a novel processing technology which is applicable to a wide variety of materials system including processing of MMC, FRP, alloys, ceramics, metals, powder metallurgy, material joining, coating cladding, sintering, drilling and casting. In comparison to the conventional processes, microwave processing of materials offers better mechanical properties with reduced defects and economic advantages in terms of power and time savings. In the growing technology and the cut-throat competitive environment for engineering industries are demanding new and improved processing methods which can be implemented for a wider variety of materials and can yield better physical and mechanical properties of materials than conventional routes, so the present work focuses mainly how to get a product with tailored properties for the particular application by controlling solidification of molten metal in microwave casting process which depends on number of factors in which rate of heat transfer from the molten metal is the most crucial one which in turn depends on number of factors.

Keywords: Susceptor, Mold, In-situ Microwave Casting, Directional Solidification, Nucleation, Grain Growth.

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Microwaves are electromagnetic waves which is a combination of both the electric field and the magnetic field propagating perpendicular to each other. Frequency range is 0.3-300 GHz [1] and wavelength between 3 mm to 0.3 m as illustrated in Fig. 1.1. The domestic microwave applicators work on 2.45 GHz frequency. There are some frequencies reserved by the Federal Communications Commission (FCC) for heating and material processing purpose in industrial, medical and scientific applications i.e., 915 MHz (popular in United Kingdom), 2.45 GHz [2] (used in India and other Asian countries) and few more frequency [3].

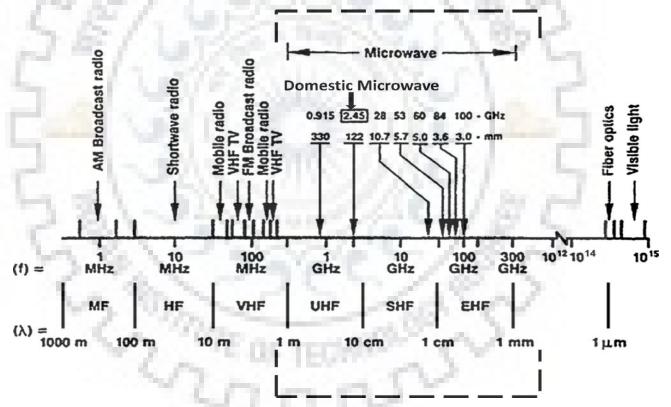


Fig. 1.1. Spectrum of electromagnetic radiations with wavelength (λ) and frequency (f) [4].

1.1. Microwave- material interaction

The electrical and magnetic properties of material play crucial role in effective heating of material through microwaves radiation. These properties (electrical and magnetic) of material

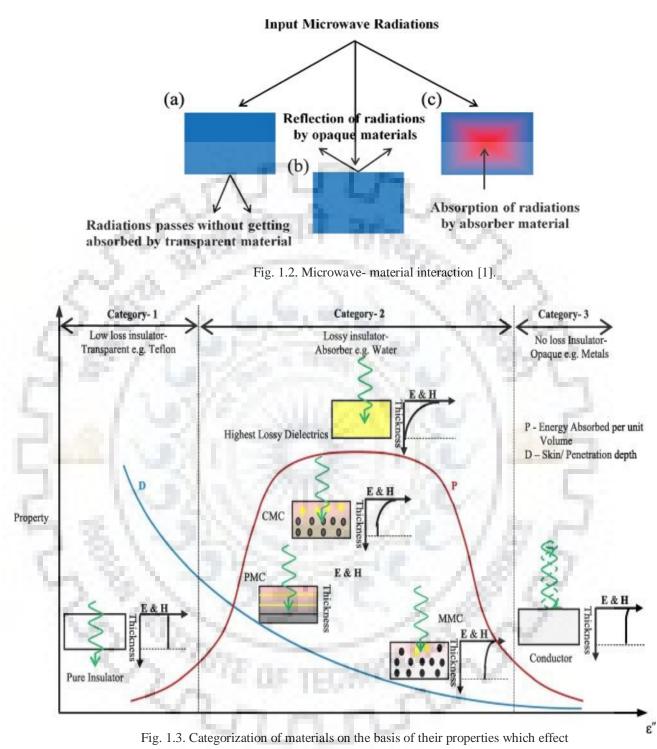
governs the way microwaves radiation couples with material. Generally materials are categorized into three major groups as presented in Fig. 1.2 and 1.3, moreover Fig. 1.3 showed the clear presentation of effect of penetration/skin depth of microwaves inside the material and dielectric loss factor over volumetric power absorption inside material when interacted with microwaves.

- a) Transparent: These materials act transparent to the microwaves thus microwaves when irradiated pass without getting absorbed as shown in Fig. 1.2(a), thus also called low loss materials, represented by category-1 in Fig. 1.3, for instance: Teflon, quartz etc. This is mainly because of very large penetration depth (skin depth) of microwaves inside the material and very low magnitude of loss factor (dielectric) of the material.
- b) Absorber: These materials readily absorbed the microwaves when irradiated and generate heat energy as shown in Fig. 1.2(c), also called high loss insulators it includes dielectric materials. Reason behind is the high magnitude of loss factor and moderate magnitude of skin depth, illustrated by category-2 in Fig. 1.3, for instance: water, SiC, charcoal etc.
- c) **Opaque**: These materials behave as opaque medium to the microwaves and get reflect thus shows surficial heating behavior as shown in Fig. 1.2(b), therefore also called no loss (conductor) materials. These materials possesses negligible skin depth but high loss factor as illustrated by category-3 in Fig. 1.2(b), for instance: all bulk metals.

One more category of material, or simply it is a multi-material system i.e., mixed absorbers or mix of any two or more above defined categories material thus also called as multi-phase material like composites, alloys etc. These advanced materials couple with microwaves and generate heat energy at localized points and with conventional modes of heat transfer all together gives the required heating result, for instance: PMC, CMC, and MMC [5].

1.2. Microwave Heat Transfer Modes

Modes of heat transfer in microwave material processing are of three different type but it consider only small volume of material for processing and combination of these modes may occur simultaneously, i.e., direct heating, selective heating and hybrid heating.



Microwave-material interaction [6].

1.2.1. Direct Heating

This mode of heating is feasible to the materials which are absorbers or belongs to category - 2 shown in Fig. 1.3. These materials when irradiated by microwaves radiation easily and

rapidly couple with microwaves and generate heat energy directly from microwaves radiation. For instance: food products, ceramics, metallic powder etc., as illustrated in Fig. 1.4(a) [6].

1.2.2. Selective Heating

This mode of heating is a subset of direct heating mode, thus similar to direct heating with some constraints, shown in Fig. 1.4(b). This mode of heating is used for particular applications like microwave welding or joining [7] etc., where only some portion of material is to be heated and rest material portion remain unaltered, it require masking material to cover the surface where heating is not required. Its advantageous when localized heating is required and some portion need to be remain undisturbed. A simple example of naturally selective heating through microwaves at low temperature applications, is heating food items in domestic microwave oven where the food is heated only and the container is not heated, this is because of the inherent properties of the materials which effect microwave-material interaction (section 1.1), like heating milk inside (borosilicate) glass [6].

1.2.3. Hybrid Heating

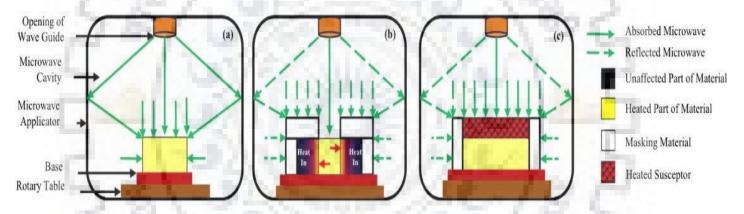
This mode of heating is used for processing of opaque materials (Fig. 1.3), as it is nearly impossible to process these materials directly by microwaves at room temperature [8] only because the opaque materials transformed into absorbers at higher temperature, it is a material specific temperature called critical temperature at and above which opaque material easily and rapidly start absorbing microwaves and generate heat energy thus the problem of processing opaque material rectified [9]. This rectification accomplished by combination of direct heating of intentionally added material to the system which is an absorber material at room temperature itself called susceptor. This require specialized tooling or setup consists of susceptor material, masking material and the material which is to be microwave processed called charge. The heating accomplished in combination of three stages:

- a) Direct heating of susceptor material thus called susceptor heating.
- b) Heat form hot susceptor transferred to the charge through conventional modes of heat transfer, includes conduction, radiation and little by convection also thus called conventional heating.

c) After reaching the critical temperature by conventional heating, charge transformed into an absorber material and then direct microwave heating initiated and rapidly increases the charge temperature.

Masking material may also be used to cover the opaque material initially. This mode of heating is a combination of conventional and microwave heating thus named as microwave hybrid heating (MHH), shown in Fig. 1.4(c). Microwave processing of bulk metals are done by microwave hybrid heating technique.

During microwave processing of materials, the heat flows from inside or core of the material to outside surface of material [1], this is called inside-out heating, which is just reversed to what happens in conventional mode of heating i.e., outside-in heating. And combination of these two results in uniform and volumetric heating of material as shown in Fig. 1.5.





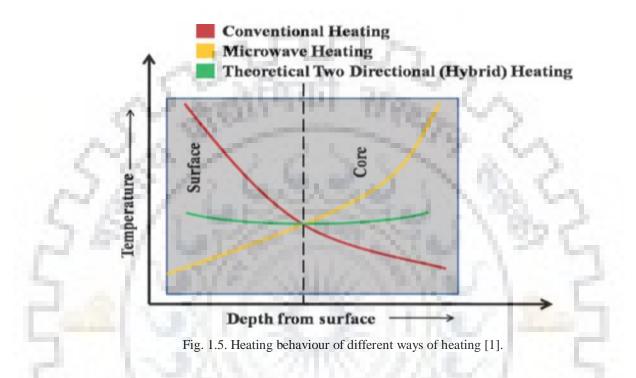
1.3. Physics Behind Heating Mechanism

Heat generation inside the material when irradiated by microwaves is completely depends upon the material own electric and magnetic properties. Mechanism of heating involves simple physics phenomenon but it is really difficult and complex to tell at what time which phenomenon is dominating because of dynamic nature of material properties. This involved the followings major phenomenon:

- (a) Dipolar loss
- (b) Conduction loss
- (c) Hysteresis loss

- (d) Eddy current loss
- (e) Domain wall resonance

Combination of these phenomenon working simultaneously inside the material which results in rapid, uniform and volumetric heat generation.



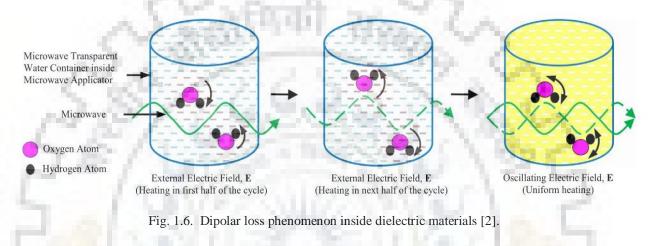
1.3.1. Heating Mechanism for Non-Magnetic Materials

It include conducting and non-conducting/dielectric materials, such as Copper, Aluminium, polymers, water, ceramics etc. This class of materials get majorly disturbed by electric field component of microwaves radiation and a little by magnetic field component as none material is completely non- magnetic. Heat generation mechanism involved mainly two phenomenon i.e., dipolar loss and conduction loss.

1.3.1.1. Dipolar Loss

This phenomenon is dominating in non-conducting or insulator or dielectric materials, for instance: ceramics, ceramics-matrix composites, polymer-matrix composites, water and food items etc. In this the vibrating or oscillating electric component of microwaves radiation disturbed the molecular dipoles inside the dielectric material. In the Fig. 1.6, water is taken as an example, where water molecules i.e., dipole formed by oxygen and hydrogen atoms.

Negative polarity with oxygen atom and positive polarity with hydrogen atoms, these dipoles reorient themselves according to the oscillating electric field waves so that they can be in phase with waves due to alternative positive and negative cycle of electric field waves. This happens 0.49 billion times in one second hence, due to Inertial, frictional, elastic, and molecular-interaction forces there exists strong resistance forces against the reorientation of dipole molecules, thus increase of molecular kinetic energy takes place which results in rapid, uniform and volumetric heating of material [10].



1.3.1.2. Conduction Loss

This phenomenon is dominating in conductor and semi-conductor materials, for instance: Copper, Nickel, Aluminium, Iron, Silicon, and metal- matrix composites etc. This mechanism is shown in Fig. 1.7. Conductors and semi-conductor have free electrons as illustrated in Fig. 1.7(a), when electric field component of microwave irradiated on these materials motion of free electrons starts with velocity 'v' in the same direction that of external electric field 'E' as presented in Fig. 1.7(b) and this motion of free electrons inside the generated/induced large current 'I_i' as illustrated in Fig. 1.7(c). This induced current inside the material in turn induced magnetic field 'H_i' in the direction opposite to that of external magnetic field and due to inertial, frictional, elastic and molecular-interaction forces which restricts the motion of free electrons. This happens in to and fro fashion according to oscillating electric field which results in generation of heat rapidly, volumetrically and uniformly inside the material as illustrated in Fig. 1.7(d) [11].

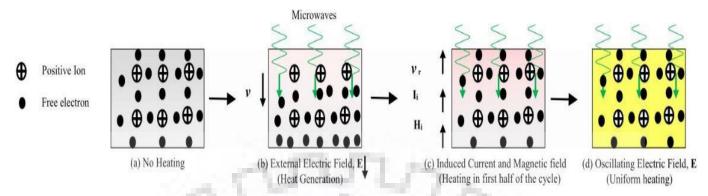


Fig. 1.7. Conduction loss phenomenon inside conductors and semi-conductors [2].

1.3.2. Heating Mechanisms for Magnetic Materials

Heating mechanism for magnetic materials also includes mechanisms of non-magnetic materials for example, free electron motion. Materials like Fe, Ni, Co etc., are disturbed by magnetic field component of microwaves because of being magnetic in nature but also affected by electric field component. The magnetic field component majorly disturb the spin of electron, domain walls and their orientations [12]. The following major phenomenon takes place inside magnetic materials:

- (a) Hysteresis loss
- (b) Eddy current loss
- (c) Domain wall resonance

1.3.2.1. Hysteresis Loss

Hysteresis loss referred as the disturbance created in terms of orientation of magnetic domains inside the material by external magnetic field component of electromagnetic waves as illustrated in Fig. 1.8. Magnetic domain treated as a tiny magnet which is responsible for magnetic moments inside the material due to electrons spinning inside the domains which generates the net magnetic moment or magnetic effect of the material. Fig. 1.8(a) picturized random arrangement (orientation) of domains thus net magnetic moment of material is null and called un-magnetized. When un-magnetized material irradiated by external magnetic field then magnetic domains realigned or get oriented in the same direction that of external magnetic field as illustrated in Fig. 1.8(b) and when oscillating external magnetic field changes its direction then again magnetic domains realigned in the same direction that of external magnetic

field as presented by Fig. 1.8(c). When external magnetic field becomes zero then gain randomization of magnetic domains occurred thus material became un-magnetized again as shown in Fig. 1.8(d). This rapid and repeated alignment and mis-alignment is restricted by elastic, inertial and frictional forces which transformed magnetic energy into heat energy. Therefore, the material gets quickly heated up uniformly as illustrated in Fig. 1.8(e). This phenomenon of magnetization and de-magnetization traces the B-H loop called hysteresis loop

[13].

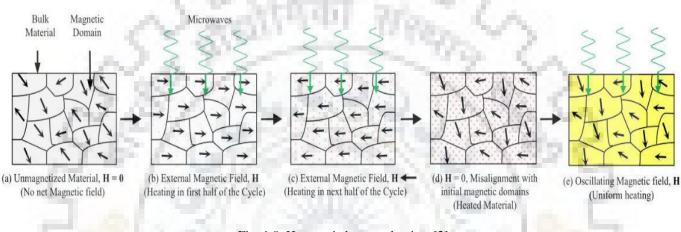


Fig. 1.8. Hysteresis loss mechanism [2].

1.3.2.2. Eddy Current Loss

This phenomenon takes place in any kind of conductor placed in varying magnetic field as illustrated by Fig. 1.9(a). Each domain on the material surface acts as a closed loop in which current flows called eddy current as shown in Fig. 1.9(b) when irradiated by external magnetic field which in-turn generates induced magnetic as illustrated in Fig. 1.9(c) that opposes the external magnetic field. When the direction of external magnetic field changes then same way eddy current direction also changes and when the magnitude of external magnetic field increases or decreases likewise eddy current magnitude also increases and decreases respectively as shown by Fig. 1.9(d). This changes in direction of eddy current and opposition of external magnetic field by induced magnetic field transform magnetic energy into heat energy. As the cycles external magnetic field repeats very quickly thus, it generates heat inside material rapidly and uniformly as illustrated in Fig. 1.9(e) [12].

1.3.2.3. Domain Wall Resonance Loss

This phenomenon is demonstrated in Fig. 1.10. Randomized domains results in zero net magnetic moment of magnetic effect as illustrated in Fig. 1.10(a) and called as un-magnetized. When the un-magnetized material irradiated by external magnetic field components of electromagnetic wave during microwave material processing, then the domains which are aligned in the same direction that of external magnetic field increases their size and rest of domains decreases their size as illustrated in Fig. 1.10 (b). When the direction of external magnetic field changes then again the size of domains increases which aligned in the same direction to that of external magnetic field, and rest domains size decreases as shown in Fig. 1.10 (c) [12].

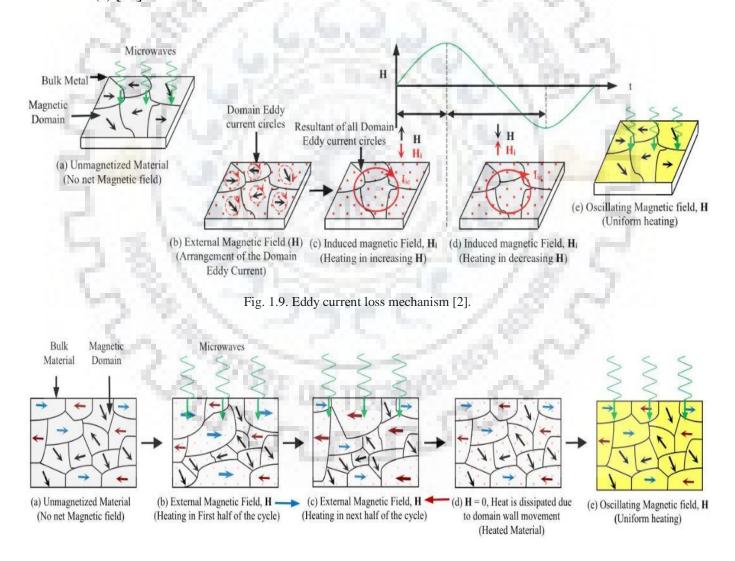


Fig. 1.10. Domain wall resonance loss mechanism [2].

When external magnetic field is absence then again the size of all domains comes to their size as illustrated by Fig. 1.10 (d). This cyclic size changing phenomenon creates vibration effect which transforms fraction of magnetic energy into heat energy inside the material because of frictional, elastic and inertial forces between the domains. This phenomenon repeats very quickly and thus it rapidly and uniformly generates volumetric heat inside the material as shown in the Fig. 1.10(e).

1.4. Numerical Modelling of Microwave –material interaction

The properties of material that causes the coupling of microwaves are described by Eq. (1.1) and (1.2) [14, 15]:

$$\varepsilon = \varepsilon_{o} \left(\varepsilon' - j \varepsilon'' \right) = \varepsilon_{o} \varepsilon' (1 - j \tan \delta)$$
(1.1)

$$\tan \delta = \varepsilon''/\varepsilon' \tag{1.2}$$

where, ε stand for medium electric constant which tell how much polarization can occur in the material upon exposure of microwave radiations. ε_0 is free space electrical permittivity, ε'' stand for medium electrical permittivity, ε''' is (dielectric) loss factor which responsible for transforming microwave energy into heat energy, j is polarizability (electrical), δ stand for loss tangent. Eq. (1.3) tells about how the temperature of material rises if microwaves couples with the material [14, 16]:

$$\Delta T / \Delta t = 2\pi f \varepsilon_{o} \varepsilon'' | E |^{2} / \rho c_{p}$$
(1.3)

Heat generated by polarization process and lost by conduction, convection and radiation from the materials results in higher heating rates in microwave processing of materials. Heat generation and uniformity of heat generation these are the key events happen with material upon microwaves exposure and these events majorly depends upon two parameters, these power dissipation (P) and skin depth (D) which is also known as depth of penetration of microwaves that means at this depth power of microwaves lost to 1/e of initial power at the surface. The power dissipation is given by Eq. (1.4) [16]:

$$P = \frac{1}{2} \omega \varepsilon_{o} \varepsilon'' E^2 V e^{-2\alpha Z}$$
(1.4)

where, E stands for electric field intensity at the surface of material, V stand for volume of material exposed to microwaves, ω stand for angular frequency, z is the distance travelled

inside the material and α represents the constant of attenuation. Eq. (1.5) governs the skin depth into the material which is the most crucial factor of uniform heat generation into the material, which is one of the unique properties of microwave material processing [14, 17]:

$$D = \frac{3\pi_{\circ}}{8.686\pi tan\delta(\epsilon'/\epsilon_{\circ})^{1/2}}$$
(1.5)

Eq. (1.6) gives the skin depth empirical relation in terms of microwaves frequency [18]:

$$D = \frac{C}{2\pi f \sqrt{2\epsilon'} (\sqrt{1 + tan^2 \delta} - 1)^{1/2}}$$
(1.6)

The value of dielectric loss tangent and ε' (electrical permittivity) are inversely proportional to the skin depth at a specific frequency of microwave radiation. Reason of surficial heating is low value of skin depth because of high frequency of microwave radiation and large value of dielectric properties of the material. Uniform – volumetric heating is due to higher skin depth because of low frequency value and moderate dielectric properties of material under exposure. Electrical as well as magnetic properties are responsible for uniform or surficial heating of material at a particular frequency of microwave radiation. Skin depth helps in categorization of materials on the basis of interaction behavior with microwaves at room temperature and also gives an understanding about the thickness of material which can be processed efficiently. Skin depth value not constant, it changes when the properties of material changes upon temperature change during microwave processing of material. Equation (1.7) gives the relation between the temperature dependent properties and skin depth [16].

$$\delta = \sqrt{\frac{\rho}{\pi f \mu_r \mu_0}} \tag{1.7}$$

where, δ stand for skin depth in μ m, ρ represents the resistivity in $\mu\Omega$ -cm, f stand for the frequency of microwaves irradiated in GHz, μ covers the magnetic permeability of material in H/m, μ_0 gives the absolute permeability in H/m and μ_r stand for relative permeability. That's how magnetic and electric properties of material effects the heat generation inside the exposed material.

1.5. Development in Microwave Processing Technology for Metals-Based Materials

Development in the microwave material processing technology categorizes the microwave processing applications into the three broader groups i.e., low temperature application during 1950 to 1970, at temperature less than 500°C which includes curing, drying and synthesis, moderate temperature applications works at temperature 500°C to 1000°C, popular between 1970 to 1999 used for sintering applications and high temperature applications from 1999 onwards, works at more than 1000 °C for advanced material processing applications. It took 65 years of development to arrive at metal casting using microwave energy as illustrated in Fig. 1.11 [19].

1.6. Processing with Microwave Energy

There are numbers of advantages of using microwaves than conventional resources for material processing like heating, sintering, melting etc., proven by researchers. Some are as follows:

- a) Uniform and volumetric heating
- b) Selective heating
- c) Rapid heating
- d) Less power consumption
- e) Kind of green technology (alternative)
- f) Economical than conventional modes of processing
- g) Microwave processed products show better mechanical and metallurgical results.

1.6.1. Advantages of Microwave Energy in Metal Casting

Besides the above mentioned advantages, microwave gives better control over the following:

- a) Material saving (in-situ microwave casting)
- b) Cost saving
- c) Cast quality is better in terms of dense structure, strength and hardness.
- d) Reduced thermal gradient and casting defects
- e) Lower environmental hazards
- f) Types of grains formation and grain size
- g) Eliminate segregation or non-equilibrium cooling effect

h) Homogenization or equilibrium cooling

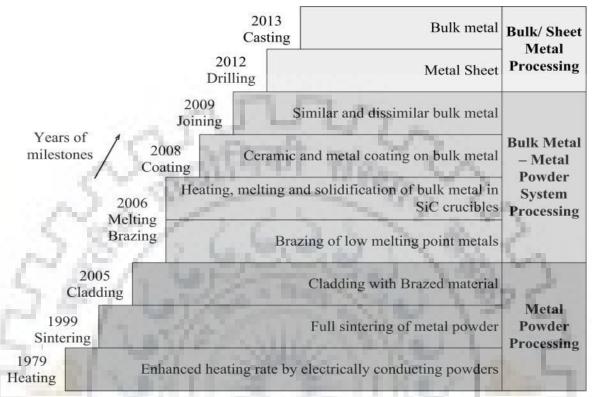


Fig. 1.11. Development in microwave processing applications for metal-based materials [19].

1.7. In-situ Microwave Casting

In-situ microwave casting itself a way in which events of casting like, heating, melting, pouring and solidification take place inside the microwave applicator with no human assistance once the setup is prepared as illustrated in Fig. 1.12. It is works on the principles of microwave hybrid heating (MHH). Hybrid heating means the heating of material takes place by combination of modes i.e., conventional heating and microwave heating. As we are aware of fact that bulk metals at room temperature don't couple with microwave rather reflect the irradiation as shown in Fig. 1.3, so to process the bulk materials which reflects or show insufficient coupling with microwaves MHH is used, as explained under section 1.2.3. The dynamics of the process and the cast quality are significantly influenced by the materials used for the microwave material processing.

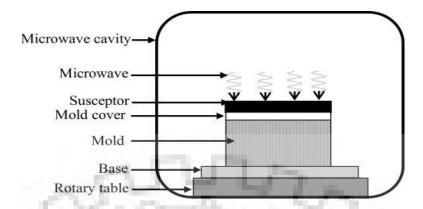


Fig. 1.12. Schematic diagrams of experimental setup of in-situ microwave casting [19].

1.8. Solidification of Casting

Casting is a versatile process known for production of intricate net shapes. But the end product i.e., cast whose properties depend upon the parameters and processes involved in casting. Solidification is that crucial process that decides majority of physical and mechanical properties of the product. Present work focuses on know-how of solidification with its mechanism and further takes towards the goal achieving i.e., Directional Solidification. Solidification means crystallization that is the conversion of liquid state material step by step into solid state.

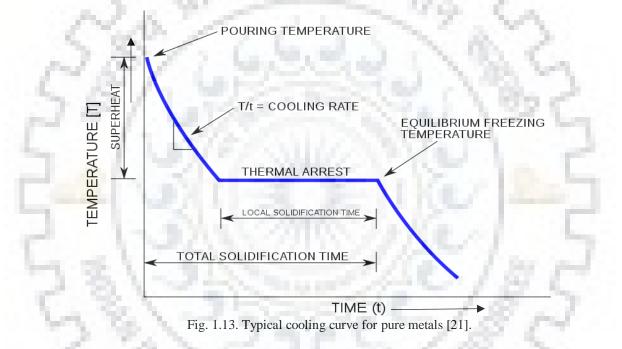
1.8.1. Cooling Curves

Cooling curve basically is a line graph which represents the change of phase of the matter. It may be from gas to liquid and it may be from liquid to solid .These are also knowns as time v/s temperature curves. There are two types of cooling curves represented by two different categories of metals, i.e., (a) pure metals and eutectic alloys (Al-Si), Fe-C with carbon composition 4.3%, (b) alloys.

1.8.1.1. Cooling Curve for Pure Metals and Eutectic Alloys

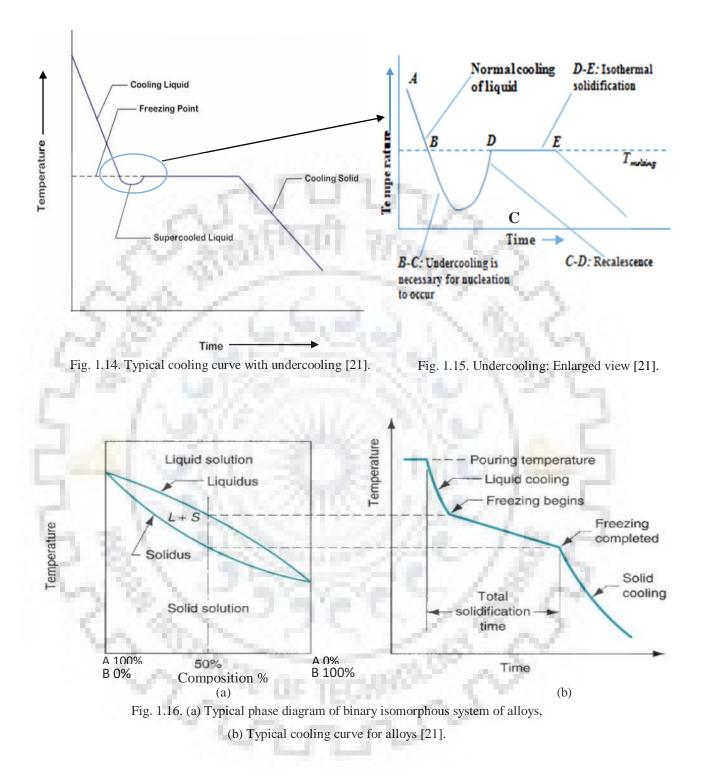
In pure metals and alloys with eutectic composition solidification occurs at a constant temperature as represented by Fig. 1.13. The temperature of the metal will come down steadily at one point of time, this temperature does not decrease for some time. So, this is a reason of thermal arrest. Once it comes here the liquid tries to lose the latent heat and the liquid is converting to the solid crystals slowly and this process is basically represented by this straight

line (horizontal). This is the idealized cooling curves, once the solid crystals are coming out of the liquid state, it does not occur at this temperature. So, for that, slight amount of under cooling is required. This is essential for starting the solidification initially, there has to do some decrease in the temperature and this decrease in temperature below its melting temperature is known as under cooling [20] and the amount by which temperature decreases down the freezing temperature that is known as degree of under cooling as illustrative in Fig. 1.14 and 1.15 (enlarged view). Therefore, degree of under cooling is a must for the solidification to start with and this is typically for a pure metal or even in case of eutectic composition like aluminum silicon alloys or iron carbon with carbon composition of 4.3 %.



1.8.1.2. Cooling Curve for Alloys

In alloys crystallization happens in different way. Alloys are having certain phase diagram as shown in Fig. 1.16(a). In this case the alloys are characterized by the property like it does not solidify at a constant temperature [22] because it is a mixture of two or more components as shown in Fig. 1.16(b). In this case, even during the solidification process also there is decrease of temperature, it does not occur at a constant temperature. The slope of these lines depends upon the freezing range of the alloys and accordingly it will vary for different materials different alloys and also the slope of these lines will be depending upon the how fast you are cooling or how slow you are cooling (same for pure metals cooling curve), but the characteristic of the lines or the nature of the lines will be similar.



1.9. Fundamentals of Solidification

One most important thing not only for materials but for everything in the universe is none other than stability. Everything which is existing in the universe is in its stable state that is the state of minimum energy. In my scenario this energy is known as free energy or Gibbs free energy. The principle behind the transformation from the liquid to solid state is the free energy, generalized free energy curve shown in Fig. 1.17.

Any material as a solid state or a liquid state at a particular temperature is governed by its free energy value, in the particular condition at the particular temperature, whichever state having the lower free energy the material will try to have that phase itself. Basically any material would like to be in a state which has the lower value of free energy to be stable. Above the melting temperature you have the liquid state which as a lower free energy that is why when you heat the material above a certain temperature, it is converted into a liquid state. Similarly once you are cooling it and going at a temperature lower than the melting temperature, here the solid state has lower free energy. That's why when temperature decreases the material has to be converted to a solid state which has lower free energy. At the temperature which is known as the melting temperature or even equilibrium temperature. At this temperature it is observed that the solid state and the liquid state both have equal amount of free energy, it means at this point solid and liquid states co-exist. Therefore, both solid as well as liquid state is there once the temperature go below this (equilibrium) temperature then only the conversion of liquid to solid starts [23].

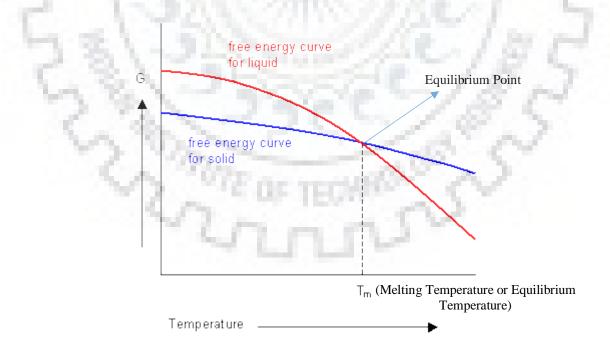


Fig. 1.17. Gibbs free energy v/s temperature (generalized) curve for liquid and solid states [24].

1.10. Mechanism of Solidification

During the process of phase transformation, free energy change determines the rate of transformation and for transformation to be possible and to proceed simultaneously free energy change must be negative, this is one criteria which must be satisfied so that the transformation in this case from liquid to solid state to be possible then free energy change has to be negative and this is one of the most crucial driving force, and a little degree of undercooling is must to start solidification or transformation process (Fig. 1.14). Once condition of a spontaneous of occurrence of phase transformation is satisfied transformation of crystalline configuration among few atoms of liquid starts. Fundamentally, it is a step by step process, from the liquid few atoms will come and among them there will be a solid crystal (surface) formed. This solid crystal formation will be based upon the "theory of existence". Therefore, there are principles behind how crystals will exist, they will be able to have their existence in the liquid phase as a solid particle [23] Further once they are meeting the condition of maintaining their existence in the liquid then at one point of time they are in position to further invite to more of the liquid atoms to come and join and then have the further opportunity to grow, that's how it goes in a step by step manner. Therefore, in this step by step transformation from liquid to solid state there are two steps majorly. First is formation of tiny stable particles of solid phase and then increase in size of these particles. Broadly these steps are:

1.10.1. Nucleation

Formation of the tiny stable particles of solid phase, this process is known as nucleation. Formation of embryos of the new phase which attained critical size (*theory of existence*) and therefore is stable to grow. There exists two type of nucleation:

1.10.1.1. Homogeneous Nucleation

Probability of having nucleation at any point in the whole domain is constant throughout the volume of solidifying material [20]. As temperature decreases, at every point in the liquid there will some nuclei which is forming this type of nucleation is called homogeneous nucleation.

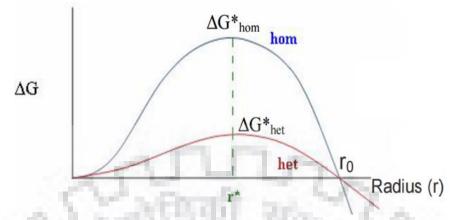


Fig. 1.18. Change in Gibbs free energy (energy barrier) v/s radius of nucleus [24].

1.10.1.2. Heterogeneous Nucleation

Probability of birth of embryo is more at certain sites because of favorable conditions, for instance: presence of foreign particles (nucleating agents), fast under-cooling sites (mold walls) etc. In general heterogeneous nucleation is more dominating because of lower free energy barrier than homogeneous nucleation, as shown in Fig. 1.18. For the same radius of tiny nucleating solid surface, Gibbs free energy barrier (change in Gibbs free energy) is more in homogeneous nucleation and less in heterogeneous nucleation [25]. That's why heterogeneous nucleation is dominating over homogeneous nucleation but it is nearly impossible in general condition to have only one kind of nucleation only, there exists both types always. For making only one kind of nucleation there required change in conditions of solidifications for example, Nucleating agents for promoting heterogeneous nucleation for making the whole volume of casting containing fine equi-axed or similar kinds of grains structures for uniform mechanical properties to achieve. Nucleating tiny embryo has to cross free energy barrier to get stable and after that only growth of stable nucleus starts.

1.10.2. Theory of Existence

This theory explains the formation of embryo to nucleus for further growth. Maintaining the position in liquid by a tiny crystal formed by arrangements of atoms explained by theory of existence. From the start of nucleation to the formation of complete grains shown in Fig. 1.19. Initially once few tiny particles take birth from liquid phase as shown in Fig. 1.19(a), among them which crosses the energy barrier or critical radius will be promoted from embryo to

nucleus and get ability to further grow as illustrated by Fig. 1.19(b) and Fig. 1.19(c), and thus formed gain boundaries when completely crystallized or solidified as shown in Fig. 1.19 (d). Considering the case of homogeneous nucleation during solidification (formation of surface): Let the tiny nucleating spherical particle of radius "r".

It may happen if a surface appears or nucleation starts then due to unfavorable conditions it suppressed.

For a spherical particle of radius "r".

Surface area (S.A) to volume (V) ratio, (S.A/V) = 3/r

When r tends to zero then S.A/V ratio tends to infinity and this implies that the energy requirement for surface generation or nucleation is very high thus crystal is unable to sustain and get suppressed, because surface has positive energy which is needed for sustaining. Let, ΔG : Gibbs free energy change during transformation (liquid state to solid state)

 Δf : Gibbs free energy change /volume

 Γ : Surface energy/surface area

 Δf value can be positive and negative but for transformation to occur its value is negative and value of Γ is always positive.

So,
$$\Delta G = 4/3\pi r^3 \Delta f + \Gamma 4\pi r^2$$

(1.8)

Equation (1.8) [20], results in the formation of the curve shown in Fig. 1.20, which explains the concepts of energy barrier, critical radius and temperature dependence as well. According to equation (1.8), when tiny crystal takes birth form the molten material (metal), its size is very small (nano-scale) and Gibbs free energy per unit volume being negative in nature with higher power of radius than surface energy per unit area (always positive value), thus initially when embryo size increases the change in Gibbs free energy value increases with increase in size (radius) of embryo, after reaching a value (highest) called critical energy barrier (ΔG^*). After this point the factor of Gibbs free energy per unit volume (negative) becomes dominating over surface energy per unit area (positive) thus as radius increases, Gibbs free energy change decreases and from that highest point, the embryo reaches that point and now will be a stable nucleus and grow, and the radius of embryo corresponds to this highest point is called critical radius (r*), after attaining this critical radius the embryo changes to nucleus which is a stable form of crystal that grows.

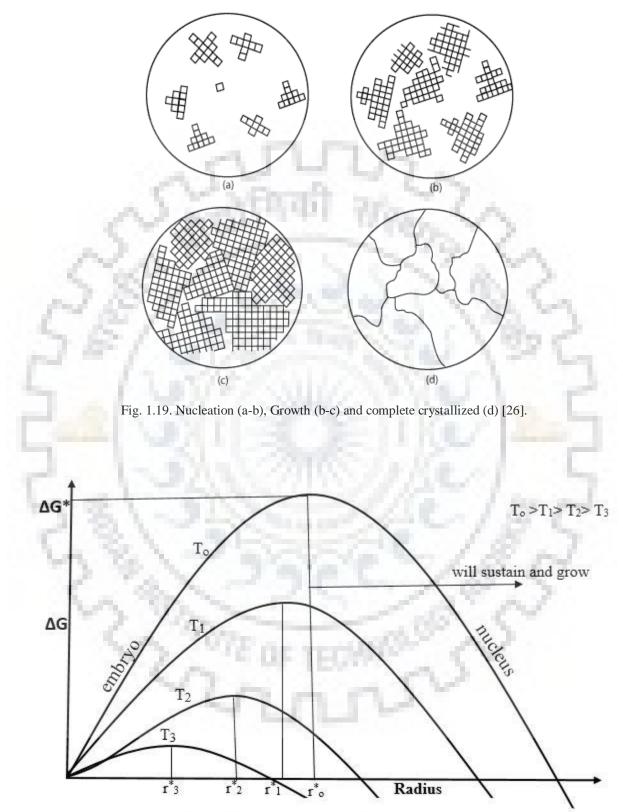


Fig. 1.20. Change in Gibbs free energy v/s radius curve.

This dome-like curve suppressed when the temperature is less, and requirement of critical energy barrier is also less and accordingly critical radius value is reduced, as shown the Fig. 1.20. T_o temperature is highest among all other temperatures (T_o>T₁> T₂> T₃), Thus it can be concluded from this analysis that at higher temperature it very difficult for embryo to reach the state of nucleus but as temperature decreases then it becomes easy conversion from embryo to nucleus and thus we can say at lower temperature number of nucleus is more. Therefore at lower temperature resultant grains structure in completely solidified volume is finer. The expression of critical energy barrier and radius can be calculated with the help of Equation (1.8), $\Delta G = 4/3\pi r^3 \Delta f + \Gamma 4\pi r^2$

Taking derivative of ΔG with respect to radius "r" and equating it to zero to get the expression of critical radius.

$$\frac{d\Delta G}{dr} = 0,$$

$$4\pi r^{2}\Delta f + \Gamma 8\pi r = 0$$

$$4\pi r^{2}\Delta f = -\Gamma 8\pi r$$

$$r^{*} (critical radius) = -\frac{2\Gamma}{\Delta f}$$
Putting equation (1.9) in equation (1.8), we get equation (1.10),

$$\Delta G^{*} = 16\pi\Gamma^{3}/3\Delta f^{2}$$
For liquid to crystallization takes place, can be written as equation (1.11)

$$\Delta f = \Delta h(T_{M} - T)/T_{M}, \text{ which implies that } \Delta f = \Delta h\Delta T/T_{M}$$
(1.11)
Putting equation (1.11) in (1.10), we get;

$$\Delta G^* = 16\pi\Gamma^3 T_m^2 / 3\Delta h^2 \Delta T^2$$

Where, T_m is the melting temperature of the metal, and T is the minimum temperature up to which under-cooling occurred.

If, ΔT = zero then ΔG^* will tends to infinity, so no nucleation will takes place, this proves that a small degree of under cooling is require for starting of nucleation.

If, T = 0 K (absolute zero temperature) then Δh will tends to zero and thus ΔG^* will again tends to infinity, results in nucleation will cease again [20]. This proves the point that undercooling is not the only driver of nucleation but also atomic mobility is an important factor of nucleation. The relation between the rate of nucleation and temperature is shown in Fig. 1.21.

1.10.2 Growth

Further, increase in size of the stable nuclei of the new phase. Growth is explained by Johnson Mehl Avrami Kolmogorov (JMAK) equation or simply Avrami equation. The characteristic of kinetics is that of the "S-curve", i.e., slow at first, the accelerating, then decelerating.

$$f(t) = 1 - e^{-kt}$$

Where, 'f (t)' is the fraction transformed, 'k' contains both nucleation and growth terms and exponent 'n' depends upon geometry of the transformation. Fig. 1.22, showed the fraction transformed v/s time (logarithmic) curve.

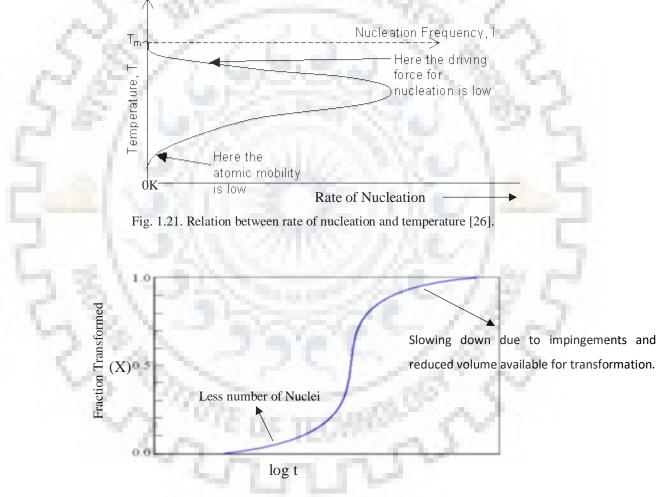


Fig. 1.22. Fraction transformed v/s logarithmic time curve [26].

1.11. Nucleation Rate, Growth Rate and Overall Transformation Rate

(a) Nucleation Rate, I (m⁻³s⁻¹): Number of nucleation events per unit volume per second.

- (b) Growth Rate, G (ms⁻¹): dr/dt ('r' is the radius or size of nucleus or tiny particle), rate of increase of size of growing particles.
- (c) Overall Transformation Rate, dX/dt (s⁻¹): Fraction transformed per second. Fig 1.23, showed the temperature dependence of Nucleation rate (I), Growth rate (G) and Overall Transformation rate (dx/dt), when liquid state transform into solid state.

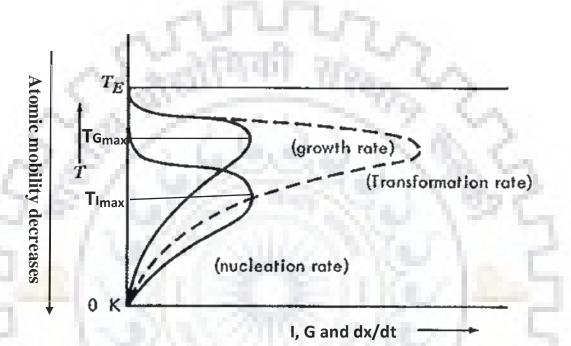


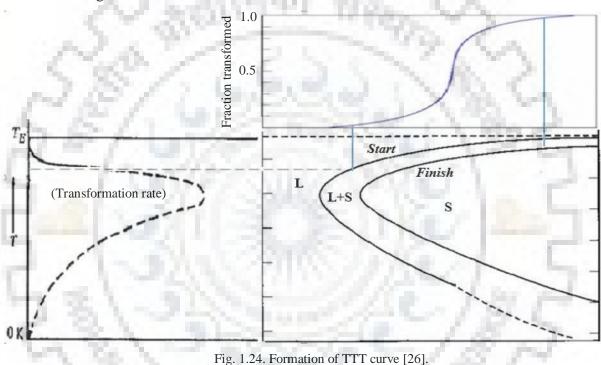
Fig. 1.23. Temperature dependence of growth rate, nucleation rate and overall transformation rate [26].

From the Fig. 1.23, these conclusions can be drawn:

- a) A little degree of under-cooling is essential for driving force to overcome the free energy barrier for starting the nucleation.
- b) For nucleation there are two major factors, under-cooling and atomic mobility.
- c) Temperature for maximum growth is higher than temperature for maximum nucleation.
- d) For overall transformation rate to be maximum the temperature falls in between the temperature at which growth is maximum and temperature at which nucleation is maximum because over-all transformation is dependent on both nucleation and growth.
- e) Nucleation rate is dominant at low temperature and growth rate is dominant at higher temperature. Hence, it can be concluded that if the cooling rate (of molten solidifying metal) is high, then number of nucleation is far more than growth of nucleus. Thus, results

in fine grain structures, and if cooling rate is slow than less number of nucleation with more growth of these nucleus which leads to the coarse grain structure [27].

Combination of Fig. 1.22 (fraction transformed v/s time curve) and Fig. 1.23 (temperature dependence of growth rate, nucleation rate and over-all transformation rate) give birth to very important curve called Time-Temperature-Transformation (TTT) curve as shown in the Fig. 1.24, which is also known as "C" curve. TTT curve is very important in phase transformation as shown in Fig. 1.25.

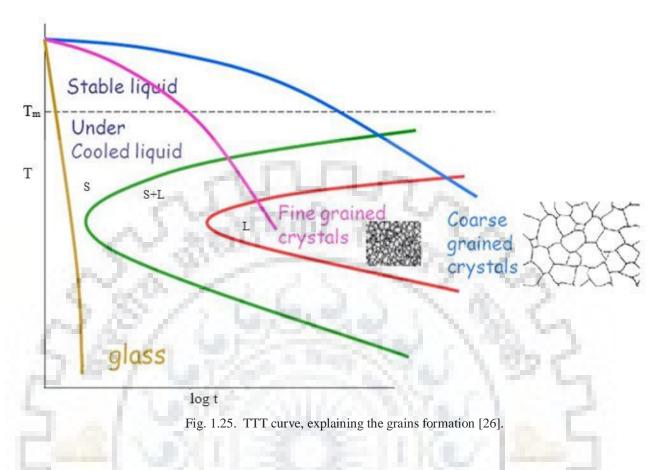


1.12. Types of Solidification

There are majorly two types of solidification:

1.12.1. Progressive Solidification

This solidification initiated at the walls of the casting and progresses perpendicularly from that surface as illustrated on Fig. 1.26. This type of solidification is unwanted and reason behind is number of defects like, shrinkage defects, hot tears or cracks etc. Also called parallel solidification because of its nature of occurrence of solidification.



1.12.2. Directional Solidification

Generally it is the solidification that occurs from farthest end of the casting and makes its way towards the sprue/riser as shown in Fig. 1.27, it is what the general requirement of the casting process but it further gives the meaning to solidification in a particular direction, with columnar like elongated grains grow in required direction as shown in Fig. 1.28 is the expected result of this experimental study and analysis. Directional solidification results in homogeneous or uniform mechanical properties which extend the service life of cast products by reducing specialized defects in alloys like segregation and making refining of grains thus improving the microstructure and most important is the growth of grains directionally, this work taken the direction form wall towards the center of cast, but directional orientation of grain growth can be in any particular direction according to the required application. In generally in a casting both types of solidification happens but when progressive solidification dominates then shrinkage defects, porosity and other defects occur in casting and thus undesirable.

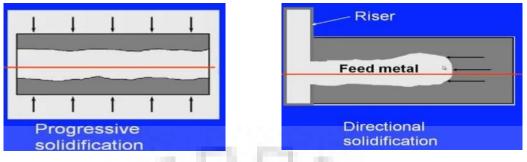


Fig. 1.26. Progressive Solidification [28].

When directional solidification dominated then, its gives less shrinkage cavities and other porosities, purifies the casing and increases the creep resistance to deformation (Fig. 1.28) beneficial mainly for the applications that is used in high temperature and under high stresses like blades of gas turbine.

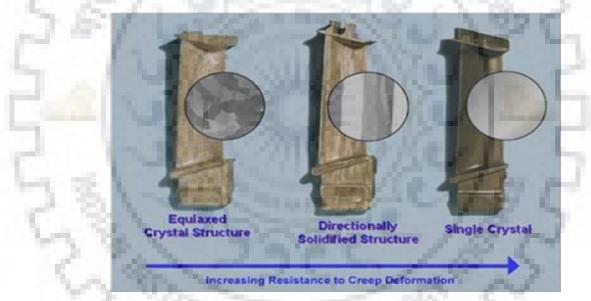


Fig. 1.28. Directionally solidified grains and comparison in terms of creep resistance [29].

In general casting process, solidification of pure metals leads to equi-axed fine grains at the mold wall and columnar grains towards the center of cast as shown in Fig. 1.29. When the solidification time is slow then less nucleation but more grain growth occurs that leads to non-homogeneous and inferior properties of the cast product which is undesirable. In alloys solidification, at center equi-axed coarsen gains, at wall same as pure metal and between wall and center dendritic columnar grains formed. These dendritic grains are formed because of constitutional solidification (in alloys) and one of the reasons for shrinkage cavities.

Fig. 1.27. Directional solidification [28].

1.13. Major Factors Effecting Directional Solidification

- a) Geometrical shapes of the mold cavity (inside and outside)
- b) Design of gating system
- c) Material of cast
- d) Pouring rate
- e) Initial temperature of gating system
- f) Pouring temperature of molten metal
- g) Heat transfer from the mold
- h) Nucleating agents
- i) Chvorinov's Rule
- j) Optimum no. of riser
- k) Chills
- 1) Insulating padding
- m) Heat generating material

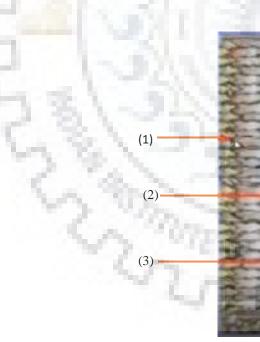


Fig. 1.29. Formation of different types of grains, showing (1) Equi-axed Fine Grains, (2) Columnar Grains and (3) Equi-axed Coarse Grains [30].

Today's era is all about less intensity and more energy which directly tells that the need of hour is to save the resources by optimally using them and taking sustainability hand in hand. Microwave material processing is that next revolutionary technology which will over-shadow all the conventional manufacturing techniques like, coating, cladding, sintering, joining, drilling, heat treatment, casting etc. The detailed literature review of microwave material processing fundamentals, casting and solidification.

2.1. Microwave Material Processing

Detailed review of microwave material processing fundamentals including mechanism of heat generation while microwave material interaction, challenges and opportunities. This helps in understanding the concept/physics behind the microwave coupling with material and generation of heat energy from microwave energy.

The detailed review of research trends in microwave processing of metal-based materials, also discussed the opportunities in metal casting via microwave processing route. They reported about the microwave processing of materials and its uses from the past to till date, and comparison of microwave material processing with other conventional and un-conventional methods. They studied all four categories of metal based materials, that is, the metal powders, bulk-metal powder system, bulk metals and sheet metals as well, in reference with sintering, cladding, joining , metal melting, metal casting, waste metal recycling or processing, heat treatment and microwave drilling. The outcome of study is that it is very advantageous to process materials with microwaves rather than conventional technologies in terms of taking less time in processing, saves energy and better quality of processed product [1]. The physics behind the heating mechanism when irradiated with microwaves. They studied the base of classifications of different materials on the basis of their interaction with microwaves, this all depends upon the material's own electric and magnetic properties which influence their coupling with microwaves radiation. Interaction of metals, non-metals, composites and their

different dominating heating mechanism are discussed [6, 31]. The processing of materials with microwaves and its applications in the manufacturing industries. They studied about the comparison between microwave and conventional heating with respect to time, energy and power required, uniform and volumetric heating of variety of metals. Results showed better productivity than conventional modes of processing of metals for the applications in sintering and cladding with better quality. Microwave hybrid heating have unlimited potential for processing of metals and their alloys, ceramics, and composites [32]. The microwave processing's fundamentals and its applications. The studied outcomes were that the volumetric heating characteristic of microwave processing is the key reason behind shorter processing time and energy saving. Better understanding of physics behind the heat generation inside the material while irradiated by microwaves and more knowledge of electric and magnetic properties of material helps in optimization of processing time and save energy [33, 34].

2.2. Microwave Melting and Casting

Review of literatures about melting of metal based materials and casting through microwave energy. This helps in understanding what are the materials used which help in fast coupling of microwaves with the material and minimizes the time of processing with getting better quality of cast.

The microwave melting of aluminium alloy A 1050, inside domestic microwave oven working with 2.45 GHz frequency at 900 W power. Result of study revealed that the ultimate tensile strength found to be 21% higher and micro-indentation hardness (viker's hardness) found to be 10% higher than the as received material [35]. The melting of bulk AA -7039 aluminium alloy of zinc and magnesium while microwave in-situ casting. In-situ microwave casting of Al-7039 bulk alloy using microwave energy at 2.45 GHz with 1.4 kW power. A 3-D modelling of the process was simulated in COMSOL multi-physics simulation software tool. The results showed the importance of placing location of casting setup inside the applicator and also the power level on the melting time, energy required to melt the metal, uniformity of heat generation inside the metal and rate of heating this is due to the fact that when setup which is a multi-material system placed inside the applicator then distribution of electric and magnetic field thus hotspots formed changes [36]. The novel microwave composite casting process using

hybrid heating mode. Nickel base powder with varying portions of silicon carbide were microwave casted. The outcomes of the study were that uniform and equi-axed grains structure were developed with uniform distribution of the reinforced phase of composite i.e., silicon carbide. Cast developed in less time the conventional casting mode [37]. Composites of nickelbased metallic powder as matrix and SiC powder as reinforcement were successfully casted by microwave energy with 900 W of power and at 2.45 GHz frequency. The result of study were lower casting defects like, shrinkage cavity, less than porosity 1.75% and thus obtained as dense cast of composite also microstructure of the cast obtained were uniform and equiaxed grains throughout the volume with uniform distribution of reinforcement in the matrix of nickel. These improvements are due the inherent characteristics of microwave heating or processing [38]. The processing of Ni-WC-8Co metal-matrix casting through microwave melting. They melted and casted the nickel-based metal matrix composites (MMCs) in powder form through microwave radiation. Nickel powder mixed with 5% and 10% volume fraction of the WC-8Co reinforcement powder and casted with 2.45 GHz frequency at 900 W of power. The development of casting took 25 mins exposure of microwaves radiation. Result of study revealed the formation of intermetallic such as NiSi and Cr₂₃C₆ which are hard phases, microstructure developed in equi-axed fashion, random distribution of tungsten carbide phase in the matrix of nickel. High micro-indentation hardness of 788 ± 52 HV was obtained because of hard particles of tungsten carbide and presence of intermetallic [39].

2.3. Solidification

Review of literatures about solidification and microstructure developed by in-situ microwave casting process. This helps in understanding the concept of solidification and accordingly microstructures developed.

Microstructure investigation of in-situ cast of AA 7039, aluminium alloy of Zn. Experimentation was performed using industrial microwave applicator working with 2.45 GHz frequency at 1.4 kW power by hybrid heating principle under atmospheric conditions. Outcome of the analysis is that the oxide formation during processing having supportive role in effective heating the metal and found denser cast and higher micro indentation hardness [40]. Relationship between the microstructure developed and responsible properties of in-situ

cast of AA 7039 aluminium alloy of Zinc and magnesium inside industrial applicator working with 2.45 GHz frequency at 1.4 kW power under atmospheric conditions. Relationship between mechanical properties like ultimate tensile strength, micro-indentation hardness, and others characteristics like porosity analysis, phase formation and their distribution resulting from microstructures developed by microwave casting discussed in detail [41]. The effect of susceptor materials and mold materials on microstructure of in-situ microwave casts of Al-Zn-Mg alloy. Susceptor materials and also mold materials significantly effects the need of exposure time of microwaves, time require for melting the charge and also the preheating of mold effects the cast microstructures developed and thus the properties. AA-7039 casts were developed under atmospheric condition inside the industrial microwave applicator working with 2.45 GHz frequency at 1.4 kW power using the hybrid heating technique. Susceptor materials used were SiC and ceramic crucible and mold materials were graphite and alumina. Outcomes of the study were that finer grains developed having more porosity when casted in alumina mold and susceptor used were SiC. The distribution and size of intermetallic phases formed depends upon the cooling rate of the molten metal inside the mold. Micro-indentation hardness found higher in case of alumina mold and silicon carbides a susceptor among other casts developed by other combinations of mold and susceptor materials [42]. The effect of solidification environment on the microstructure developed and micro- indentation hardness of AA 7039 aluminium alloy of zinc and magnesium in-situ casts developed using microwave hybrid heating done with 2.45 GHz frequency at 1.4 kW power of microwaves radiation. Three different cooling ambience were tested by casting number of times. The solidification conditions were, (a) closed cooling of applicator's cavity, (b) open cooling of applicator's cavity, (c) water-cooled cooling of applicators cavity and (d) casting with normal conditions for comparison. Result of study was that finer equi-axed grains structure, higher microindentation hardness among three different cooling conditions and denser casts were developed with water-cooled applicator cavity, but highest micro-indentation hardness were obtained with conventional casting condition due to the formation of intermetallic phases which is more effective in hardness improvement than the gains size refinement [43].

2.4. Directional Solidification

Review for literatures about directional solidification achieving by different techniques. This helped in understanding the clear picture of need of directionally solidifies grain structures, also the factors on which it depends most and different ways of achieving directional solidification in conventional techniques of castings.

The theoretical modelling of eutectic binary system of Ag-Cu alloy described columnar or directional growth of dendrites including growth rates. The changes in microstructures which may occur at high growth rates. The effect of the temperature dependent diffusion is also considered which is one of the mechanisms of creep deformation at high temperature and stress conditions [44]. The investigation of 'Bridgman' method which uses the Bridgman furnace allowed the determination of basic parameters of solidification process such as cooling rate, temperature gradient, and solidification rate during directionally solidification of Ni-based CMSX-4 super-alloy casting using vacuum furnace with graphite heaters [45]. The technique of cold crucible directional solidification (CCDS). Results described that the microstructure formation is significantly influenced by heat transfer and temperature distribution in CCDS also the heating process and temperature distribution in two square cold crucibles are so designed that the directional solidification can be achieved on Ti-46Al-6Nb ingots and finally the ingots are directionally solidified under the power of 45 kW [46]. The analysis of a transient process of directional solidification of TiAl alloys in the absence of forced convection. Centrifugal casting is used and the effect of gravity and the rotational speed is discussed. Formation of different types of columnar grains i.e., axial-columnar, radial-columnar and equi-axed grains are discussed with the factor on which their formation depends such as temperature distribution and applied cooling rates, as well as on properties such as the degree of inoculation of the melt and nucleation undercooling required [47]. The use of a low horizontal magnetic field during directional solidification. The external horizontal magnetic field was applied to the mushy zone of the directionally solidified Al-10 wt% Cu and Al-18 wt% Si samples, which was perpendicular to the growth direction. Experimental results demonstrate that the external horizontal magnetic field would be beneficial to fabricate product with directionally solidified grains structures [48].

2.5. Research Gap

Microwave material processing of metallic material is new and emerging field having greater potential of producing higher quality product than conventional manufacturing processes for sintering, coating, cladding, drilling, joining and casting. My work is confined to microwave metal casting. Processing with microwave consume much less energy and time, also have very high efficiency of processing. Solidification is that crucial event in the casting which decide the performance level of the product. Control over solidification is the key to excellent performance at higher temperature and stress conditions and other specialized applications also, so directional solidification is a step forward in quality improvement and increases the reliability of the product. There are lot of literatures available on directional solidification but applicable in conventional modes or processes of castings but not much directly applicator in microwave casting technology. This work is for achieving directional solidification using microwave casting technology by increasing the heat transfer rate from the mold.

2.6. Objectives of the Present Work

Based on the literature review and the gap areas identified, the objectives of the present work are formulated as follows:

- a) To develop a setup for in-situ microwave casting to promote directional solidification and to minimize the effect of parallel or progressive solidification
- b) To get directionally solidified grains structures by increasing the heat transfer rate from the mold and modifications of the heat sink on the external structure of mold.



A comparative study and analysis is being done to check the effectiveness of the process towards the goal achieving, that is to get directional solidification condition. Three different configuration as shown in Fig. 3.1, with three different cooling environments are taken for the analysis.



(a) 2 mm dia. holes (on four walls) (b) 4 mm dia. holes (on four walls) (c) Simple mold

Fig. 3.1. Configurations of mold used for experimental study (a) 2 mm diameter holes on side four walls, (b) 4 mm diameter holes on side four walls and (c) simple mold with no holes.

Different cooling ambiance taken for experimental study are:

1) Furnace cooling (inside microwave applicator cooling)

2) Ambient (outside microwave applicator cooling) and

3) Cooling with fan within natural convection range (75 W power ceiling fan)

Cooling rates in each above configuration with different cooling ambiance is recorded for theoretical analysis and justified with the experimental results as well. Comparative study is done on two different categories, i.e.

(a) Comparison between different mold configurations under same cooling conditions.

(b) Comparison between same mold configurations under different cooling conditions.

The temperature inside the microwave applicator is raised to 80 °C-110 °C during processing, which will affect the heat extraction rate form the mold and thus effect solidification/cooling rate of molten metal. Fig. 3.2, demonstrated the detailed schematic diagram of domestic

microwave applicator in which melting then pouring of molten metal inside the mold itself which is called in-situ casting took place.

3.1. Experimentations

(a) Mold: Single type mold, made up of graphite material from a rectangular shape with dimensions (L×B×H) 40×40×20 mm³, having cavity (mold cavity) at the center of cylindrical shape with 20 mm diameter and 10mm depth as shown in Fig. 3.1(c). Modification has been done in terms of making change in external shape of the mold by incorporating holes of 2 mm diameter (Fig. 3.1(a)) and 4 mm diameter (Fig. 3.1(b)) on four walls up to a depth of 9 mm, holes are close and equi-spaced to serve as heat sink (increasing effective surface area for fast heat extraction rate from the mold). Graphite is a microwave absorbing material because of sufficient skin depth and high dielectric loss factor (Table 3.1), it eliminates the preheating requirement of the mold during pouring and reduce the thermal gradient.

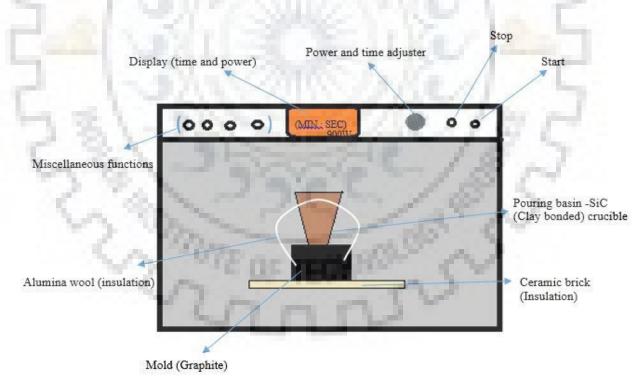


Fig. 3.2. Schematic of microwave applicator (LG SolarDom) with experimental setup.

(b) **Pouring Basin:** Silicon carbide clay bonded crucible itself used as a pouring basin and susceptor as well. Crucible is of conical shape with 12 mm thickness. Separate sprue is

not used for minimizing the effect of progressive solidification and to promote the directional solidification, by extracting the heat from the top at slow pace so that more solidification occurs from walls end towards the center of the cast not from upper and bottom walls (progressive solidification), this is why in-built sprue is made in the pouring basin by making a taper hole at bottom center of 8 mm to 5 mm diameter over 12 mm thickness. Taper sprue (passage) is made for eliminating the aspiration effect. Silicon carbide clay bonded crucible has higher value dielectric loss factor and rapidly interacted with microwave radiation. Initially it heats up the charge (aluminium) through conduction till the charge reaches its material specific critical temperature i.e., ~370 °C for aluminium [49] after which charge itself starts coupling with microwaves and thus heated up very rapidly by microwave hybrid heating.

- (c) Charge: Pure Aluminum is used as charge of 27-28 g mass.
- (d) Insulation: Alumina wool is for insulating the setup except the mold box. It covers pouring basin for making a hot zone inside it for fast melting of charge and reduce leakage of heat through radiation and convection mode of heat transfer. This alumina wool can withstand the temperature of 1200 °C.
- (e) Domestic Microwave Applicator: Domestic microwave applicator (oven) of LG (Solar Dom) of 900 W power working on 2.45 GHz frequency is used for experimentation Fig. 3.2 and Fig. 3.4, showed the schematic of the applicator with experimental setup and real time applicator with setup respectively.
- (f) Insulation Block : A ceramic brick is used on which complete setup is placed for avoiding thermal damage of the applicator surface and also to promote directional solidification, by extracting the heat from the bottom at slow pace so that more solidification occurs from walls end towards the center of the cast not from upper and bottom walls (progressive solidification).

3.1.1. Properties of Materials

Properties of material used in experimental work shown in table 3.1.

Material	Density	Heat capacity	Thermal	Dielectric	Penetration
	(kg/m ³)	(J/kgK)	conductivity	loss factor	depth
			(W/mK)	(F /m)	(mm)
SiC	3100	100	250	.02898	40
Graphite	1800	379	151	11.2	0.21
Aluminum	2700	903	237	negligible	0.0017
Insulation block	1 300		5-10	5.00	-

Table 3.1. Properties of materials (at room temperature) used in experimentation [3].

3.2. Experimental Analysis

Iterative experiments are being done for finding and optimizing the melting and casting time. Melting of charge and self- pouring together takes 10-11 min and around 1 min of extra microwave exposure is allowed so that mold and charge have less temperature gradient which led to more homogeneous nucleation dominating over heterogeneous nucleation and also setup is made in such a way that it promotes directional solidification over progressive solidification, Fig. 3.3-3.6, showed the experimental setup and processing.

Holes of 9 mm length and different diameters (2 mm and 4 mm) are being made on the four walls of the mold to increase the cooling rate because of increase in effective surface area. Effective surface area of simple mold is 3200 mm², effective surface area of 2 mm diameter holes mold is 3652.39 mm² and effective surface area of 4 mm diameter holes mold is 3954 mm². Mold cavity taken for experimentation is cylindrical with 20 mm diameter and 10 mm depth. Cylindrical shape, unlike rectangular cavity there is no diverging effect of heat transfer. Diverging effects comes with diverging section at the corners of the rectangular cavity which gives rise to high rate of heat transfer at the corners which results in non-uniform cooling/solidification effect.

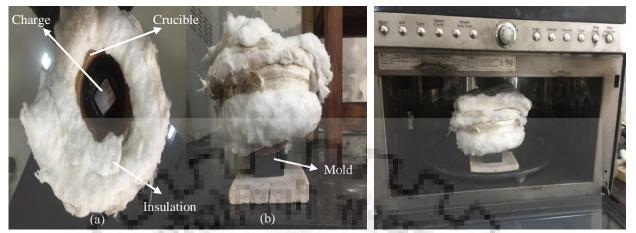


Fig. 3.3. Experimental setup used, (a) top view and Fig. 3.4. Setup inside microwave applicator cavity. (b) front view.



Fig. 3.5. Microwave exposure while processing.



Fig. 3.6. Cooling rate recording during solidification.

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4.1. Experimental Configurations

In this experimental work, three different cooling environmental conditions are explored together with three different mold configurations, combinations of these are used for comparison study to analysis their effectiveness towards achieving directional solidification.

4.1.1. Cooling Inside Microwave Applicator

- a) Simple mold
- b) Mold with 2 mm diameter holes
- c) Mold with 4 mm diameter holes

Samples of pure aluminum using different mold configurations while cooling inside the microwave applicator are shown in Fig. 4.1.

4.1.2. Cooling Outside the Microwave Applicator

- a) Simple mold
- b) Mold with 2 mm diameter holes
- c) Mold with 4 mm diameter holes

Samples of pure aluminum using different mold configurations with cooling outside the microwave applicator are shown in Fig. 4.2.

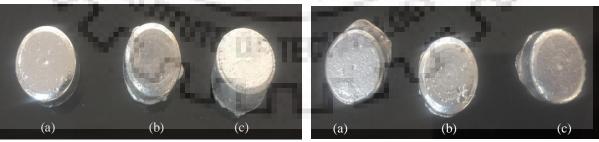


Fig. 4.1. Casts obtained by cooling inside microwave Fig. 4.2. Casts obtained by cooling outside microwave applicator.

4.1.3. Fan Cooling

Cooling of molten metal under fan running of 75 W power, at a height of ~6 ft., this condition give a little velocity to the still air around the hot mold containing solidifying molten metal, to check the effectiveness of hole drilled on the four walls of mold for heat sink purpose. This cooling comes under free convectional condition. Configuration of mold with 4 mm diameter holes only was allowed to cool under this condition.

Sample casted with 4 mm diameter holes under fan cooling is shown in Fig. 4.3.





Fig. 4.3. Cast developed under fan cooling. Fig. 4.4. Laser pyrometer (temperature measurement).

During solidification, the cooling rate was determined by recording temperature of mold during solidification of the cast by non-contact pyrometer (Fluke IR Thermometer) with range of temperature sensing from -40 °C to 800 °C as shown in Fig. 4.4. Cast samples were sectioned (perpendicular) as shown in Fig. 4.5, for further processing or characterization.

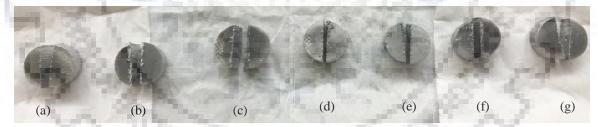


Fig. 4.5. Sectioned cast samples obtained by furnace cooling [(a), (b), (c)], ambient cooling [(d), (e), (f)] and fan cooling (g).

4.2. Cooling Curves and Microstructures obtained with Different Configurations

Cooling curves are generated using the data recorded by laser pyrometer, these curves will give the real time comparison of different cooling environments together with different mold configurations on the basis of cooling rate. Cooling of casting till 100 °C is considered and thus average cooling rate is calculated. Microstructures (grains and grain boundaries) are observed under optical microscope at 100X magnification from wall to the center of casting to see the directional orientation of gains developed as shown in the Fig. 4.6, which is considered as a reference figure for all the microstructure's figures.

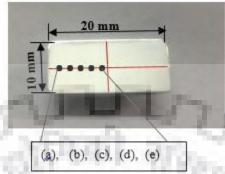
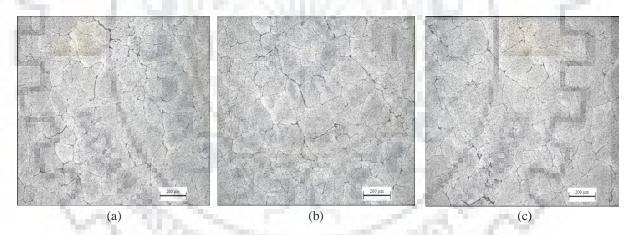


Fig. 4.6. Sectioned–polished–etched sample image for the reference of position.

4.2.1. As Received Sample Microstructure: Microstructures are observed and named according to the reference given in Fig. 4.7. Fig. 4.7 (a), (b), (c), (d), and (e) present the microstructures form wall towards center of casting, having porosity and coarser grains, this will be the reference sample for the samples casted under different configurations.



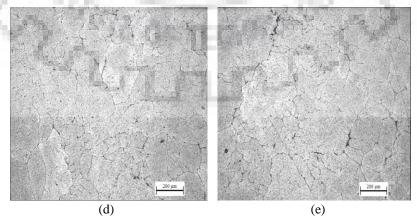


Fig. 4.7. Microstructures found in as received sample from wall (a), towards the center of casting (e) (100X).

4.2.2. Cooling Inside Microwave Applicator

Cooling inside microwave applicator with all three different configurations of mold are used to compare their heat removal rate capacity together with their potential to get directionally solidified grain structures.

4.2.2.1. Simple Mold

Simple mold getting cooled inside microwave applicator cooling curve shown in Fig. 4.8, which get cooled to 100 °C in about 70 mins.

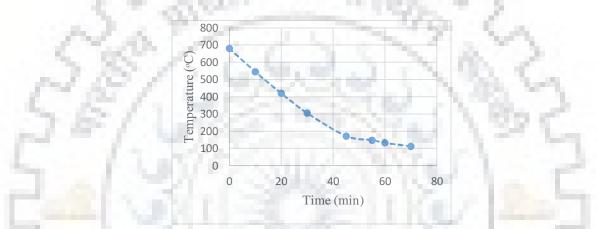
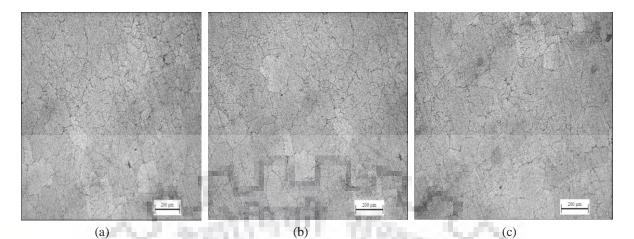
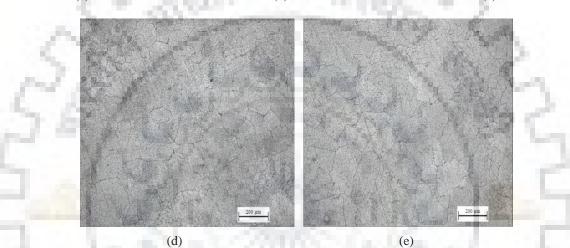
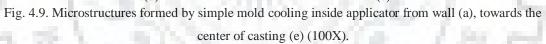


Fig. 4.8. Cooling curve of simple mold under inside applicator cooling.

Fig. 4.9 (a), (b), (c), (d), and (e) presented the microstructures form wall towards center of casting. Microstructure at wall of the cast shown in Fig. 4.9 (a) are finer than the microstructures growing towards the center due to lower cooling rate at center than at wall of the cast. Denser microstructures than the as received sample with less porosity, microstructures are coarsen equi-axed at the center, finer and random at the wall, no directional orientation of grains are observed inside the cast. These microstructures will result in higher micro-indentation hardness at the wall and least at the center due the coarse grains at the center than at wall of the cast, this is verified by the 'Hall-Petch' relation of strengthening mechanism of grain boundary by gain refinement. The cooling rate of simple mold configuration cooling inside the microwave applicator is least among all other configurations under different cooling conditions, thus microstructures are so formed.







4.2.2.2. Mold with 2 mm Diameter Holes

Mold with 2 mm diameter holes cooled inside microwave applicator to 100 °C in around 78 mins as shown in Fig. 4.10.

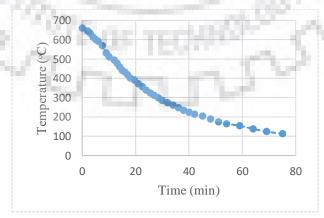


Fig. 4.10. Cooling curve of 2 mm diameter holes mold under inside applicator cooling condition.

Fig. 4.11 (a), (b), (c), (d), and (e) presented the microstructures form wall towards center of casting. Cooling rate of this configuration is higher than simple mold under both the cooling conditions i.e., cooling inside and outside the applicator. Therefore, microstructures so formed in this condition is more finer, uniform and equi-axed throughout the volume of cast also denser structure than as received sample is developed with less porosity. The trend of microstructure is obvious that finer at the wall and coarser at the center of the cast and the directional orientation of grains is missing.

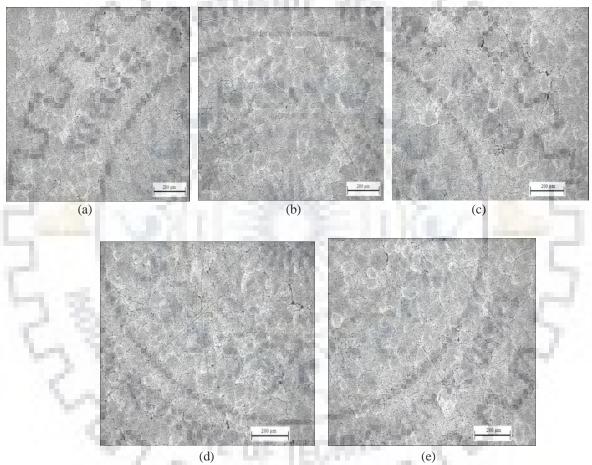


Fig. 4.11. Microstructures formed by 2 mm diameter holes mold cooling inside applicator from wall (a), towards the center of casting (e) (100X).

4.2.2.3. Mold with 4 mm Diameter Holes

Mold with 4 mm diameter holes got cooled inside microwave applicator to 100 °C in ~78 mins as shown in Fig. 4.12.

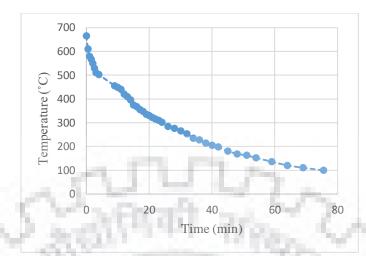


Fig. 4.12. Cooling curve of 4 mm diameter holes mold under inside applicator cooling condition.

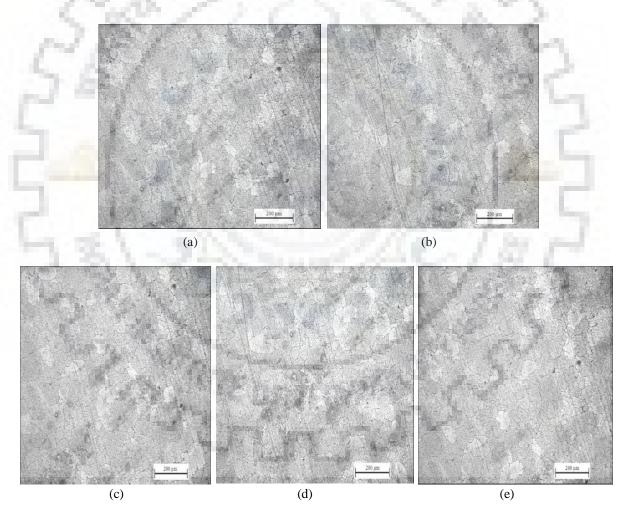


Fig. 4.13. Microstructures formed by 4 mm diameter holes mold cooling inside applicator from wall (a), towards the center of casting (e) (100X).

Fig. 4.13(a), (b), (c), (d), and (e) presented the microstructures form wall towards center of casting. The cooling rate of 4 mm diameter holes mold under furnace cooling condition is more than the cooling rate of other mold configuration under same cooling condition, thus the microstructure obtained are more uniform, finer at the wall and at the center and are of equi-axed nature. Denser cast structure is developed with less than 1% porosity and other defects. The directional orientation of growth of gains structure is not shown thus the microstructures formed are not directionally solidified and the configuration is not effective getting directional solidification.

It seem like mold having more effective surface area taking more time to cool which is reversed to the general theoretical concept of conventional modes of heat transfer i.e., conduction, convection and radiation, but more important thing is the rate of cooling at initial stage of solidification than latter stage and average time to get cooled, which will give clear idea about which configuration having more nucleation than growth and vice versa.

4.2.3. Cooling Outside the Microwave Applicator

Cooling outside microwave applicator with all three different configurations of mold to compare their heat removal rate capacity together with their potential to get directionally solidified grain structures.

4.2.3.1. Simple Mold

Simple mold cooling outside the microwave applicator shows cooling curve in Fig. 4.14, which gets cooled to 100 °C in about 77 mins, which is more than time taken by simple mold cooling inside the microwave applicator, this showed the ineffectiveness of the simple mold.

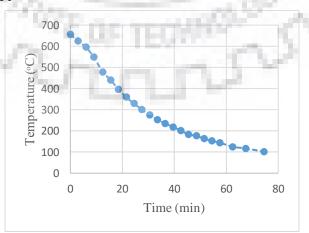


Fig. 4.14. Cooling curve of simple mold under outside applicator cooling condition.

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Fig. 4.15 (a), (b), (c), (d), and (e) presented the microstructures form wall towards center of casting. The cooling rate of this configuration is least among all the different mold configuration but, more than the same configuration under furnace cooling condition. Microstructure formed are not directional but are finer at the wall and coarse at the center and near center of the cast. At center gains are equi-axed but near the wall grains are finer and randomly developed. Denser structures developed with reference to the as received sample with less porosity and other defects. Thus this proves that this configuration is not effective not only for getting directionally solidified grains but also uniformed equi-axed grains.



Fig. 4.15. Microstructures formed by simple mold cooling outside the applicator from wall (a), towards the center of casting (e) (100X).

4.2.3.2. Mold with 2 mm Diameter Holes

Mold with 2 mm diameter holes cooled to 100 °C in around 68 mins as shown in Fig. 4.16, this

showed that mold of 2 mm diameter holes is more effective when cooling outside the applicator than inside.

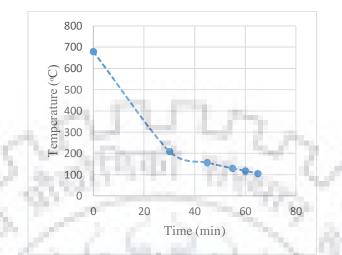
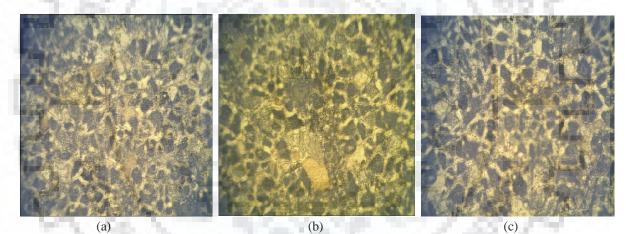


Fig. 4.16. Cooling curve of 2 mm diameter holes mold under outside applicator cooling condition.



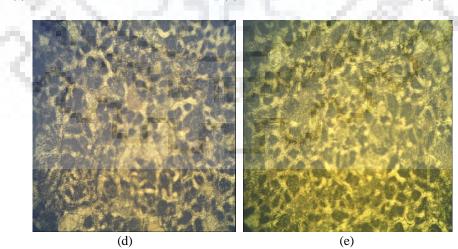


Fig. 4.17. Microstructures formed by 2 mm diameter holes mold cooling outside applicator from wall (a), towards the center of casting (e) (100X).

Fig. 4.17(a), (b), (c), (d), and (e) presented the microstructures form wall towards center of casting. The microstructures are uniform, equi-axed and finer than the all previous configurations. Denser cast structure is developed. Microstructure formation is finer at the wall and less fine at the center of the cast. Apart from high micro-indentation hardness and denser structure, directional orientation of grains from wall towards the center of cast is missing.

4.2.3.3. Mold with 4 mm Diameter Holes

Mold with 4 mm diameter holes cooled to 100 °C in ~64 mins outside microwave applicator as shown in Fig. 4.18, this proves that mold with 4 mm diameter holes is more effective when cooled outside the applicator cavity. The cooling curves obtained when cooling outside applicator cavity showed the heat transfer rate according to the surface area i.e., as surface area increases heat transfer rate also increases [50] but the more important thing is the cooling rate at initial state of solidification than latter stage and also than the average cooling rate.

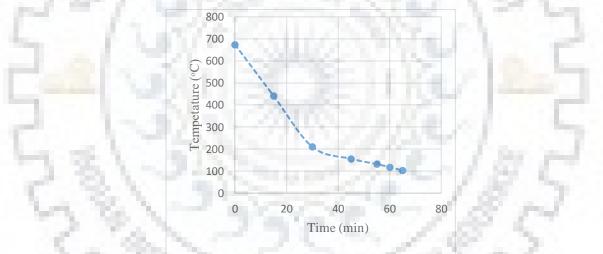


Fig. 4.18. Cooling curve of 4 mm diameter holes mold under outside applicator cooling condition.

Fig. 4.19(a), (b), (c), (d), and (e) presented the microstructures form wall towards center of casting. Microstructures observed show uniform and finer development. At wall microstructures are fine and uniform towards the center of cast microstructure are less fine, uniform and more equi-axed but directional orientation is not observed. Denser cast developed with $\sim 0.5\%$ porosity and defects free.

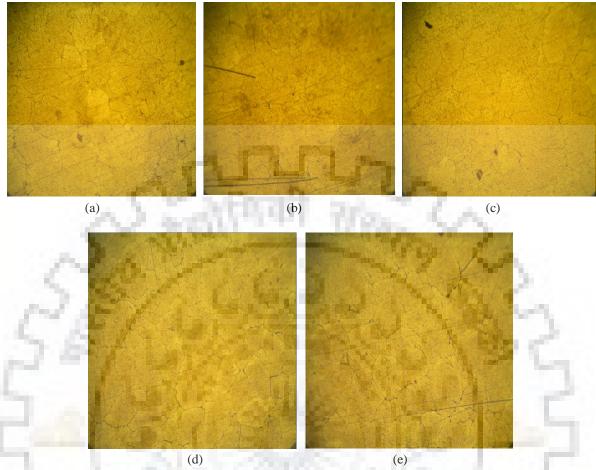


Fig. 4.19. Microstructures formed by 4 mm diameter holes mold cooling outside applicator from wall (a), towards the center of casting (e) (100X).

4.2.4. Fan Cooling

Mold with 4mm diameter holes cooled under fan of 75 W running at height of ~6 ft. for giving circulation to air around the mold but under natural convection range only, during solidification of molten metal inside the mold to check the effectiveness of 4 mm diameter holes in order to give higher cooling rate and to get the directionally solidified grains, for this case 4 mm diameter holes mold is taken only because, it has maximum effective surface area among all the configurations and it is the most suitable configuration that can authenticate the effectiveness of holes for achieving directional solidification. Fig. 4.20, showed the cooling curve under fan cooling condition, it took ~55 mins to reach the temperature of 100 °C. This clearly depicts that this configuration is most effective than others.

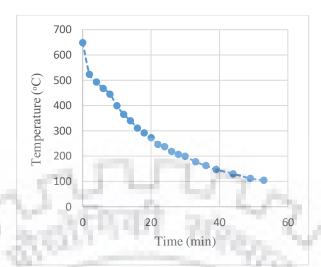


Fig. 4.20. Cooling curve of 4 mm diameter holes mold under fan cooling condition.

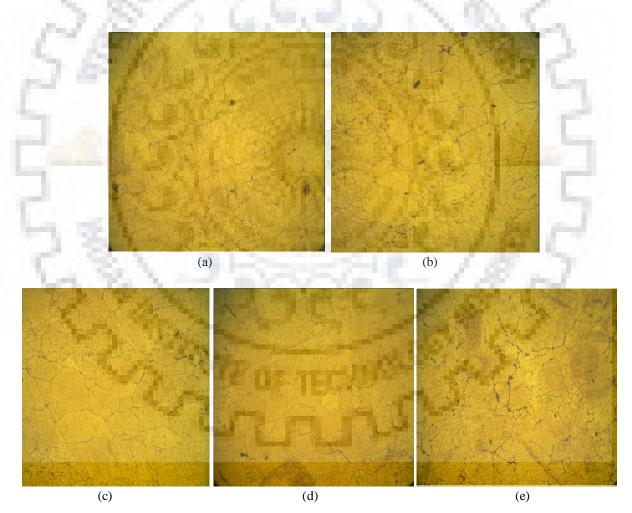


Fig. 4.21. Microstructures formed by 4 mm diameter holes mold under fan cooling condition from wall (a), towards the center of casting (e) (100X).

Fig. 4.21(a), (b), (c), (d), and (e) presented the microstructures form wall towards center of casting. This configuration is having negligible but a little potential to get directionally solidified grain structures when air is circulated around the mold. Cooling rate is maximum in this configuration and the micro-indentation hardness is also maximum among all the configurations under different cooling conditions. Microstructures formed are fine, uniform and a little elongated towards the center of the casting from the wall, this shows the potential of the configuration to get directionally solidified grains structures but at the center of the cast the grains are equi-axed and denser cast is developed.

Microstructures developed using all the different mold configurations with different cooling conditions are uniform and equi-axed, no directional orientation is observed. Obvious pattern of grains refinement is observed from more fine at the wall and less fine towards the center of the cast. Development of these microstructures will results in the higher micro indentation hardness and denser volume of cast developed with minimum defects like porosity, shrinkage defects etc.

4.3. Comparing Different Mold Configurations with Different Cooling Conditions

In this section, comparison between different combinations of mold configurations under different cooling conditions is made to further clarify which combinations is better in terms of getting higher cooling rate at initial stage of solidification and at latter stage as well. Higher cooling at initial stage of solidification is of more importance than at latter stage because higher rate of cooling at initial stage will result in more nucleation than growth and latter will result in more growth than nucleation.

4.3.1. Comparison between Different Mold Configurations under same Cooling Condition

This compared and more clearly picturized the potential of each configuration. Here, overlapping of all the three different mold configuration's cooling curves obtained under different cooling conditions taken one at a time is being done. This clearly showed the rate heat removal potential at the initial and latter stages of solidification of each configuration and also define the effectiveness of each mold configuration under different cooling conditions.

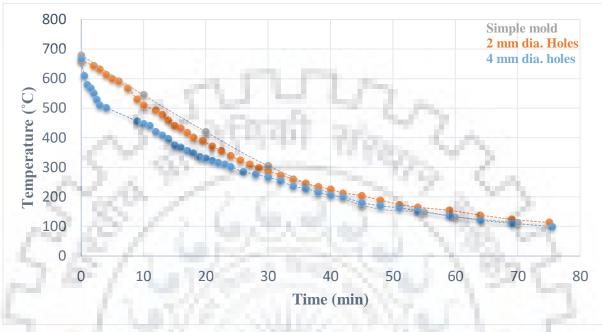


Fig. 4.22 showed the cooling curves of all three mold configuration cooling inside the microwave applicator.

Fig. 4.22. Furnace cooling (inside applicator) of different mold configurations.

Simple mold and 2 mm diameter holes mold shows almost similar cooling rate that means the 2 mm diameter holes mold is not effective under furnace cooling condition, but 4 mm diameter holes mold shows very high rate of cooling at initial stage of solidification than the other two configurations but after 30 mins all the mold configurations showed similar rate of cooling i.e., at later stage of solidification.

The comparison between the mold configurations cooling outside the microwave applicator shown in Fig. 4.23. All the mold configurations showed similar cooling rate at initial stage of solidification, but after ~20 mins 2 mm diameter holes mold shows higher rate of cooling than the other configurations. At the initial stages 4 mm diameter holes mold is more effective when cooling inside the microwave applicator than outside as depicted by the cooling curves, but at latter stages of solidification the situation get reversed and average cooling rate is more when cooling outside the applicator.

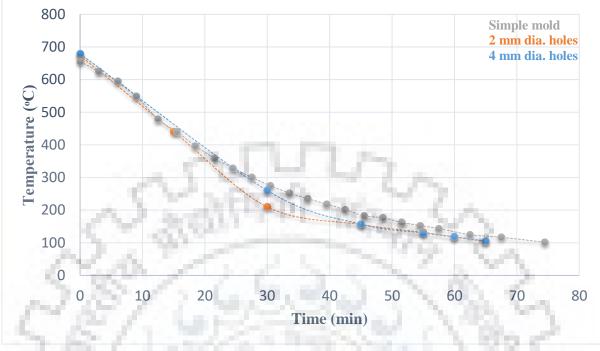


Fig. 4.23. Ambient cooling (outside applicator) of different mold configurations.

4.3.2. Comparison between same Mold Configuration under Different Cooling Conditions

This compared and more clearly picturized the potential of each configuration. Here, overlapping of same mold configurations cooling curves obtained under different cooling conditions is being done which will clearly show the potential of heat removal rate at the initial and latter stages of solidification of each configuration and also describe the effectiveness of each mold configuration under different cooling conditions.

Fig. 4.24, showed the comparison between different cooling conditions i.e., furnace and ambient cooling with simple mold configuration, cooling rate under both the different cooling conditions are almost similar thus the simple mold configuration has negligible effect of changing cooling condition from inside to outside of the applicator. The 2 mm diameter holes mold cooling curves with cooling under furnace and ambient cooling are shown in Fig. 4.25. This depicts that 2 mm diameter holes mold is more effective than the simple mold under furnace and ambient cooling rate at initial stage of solidification under both the cooling conditions.

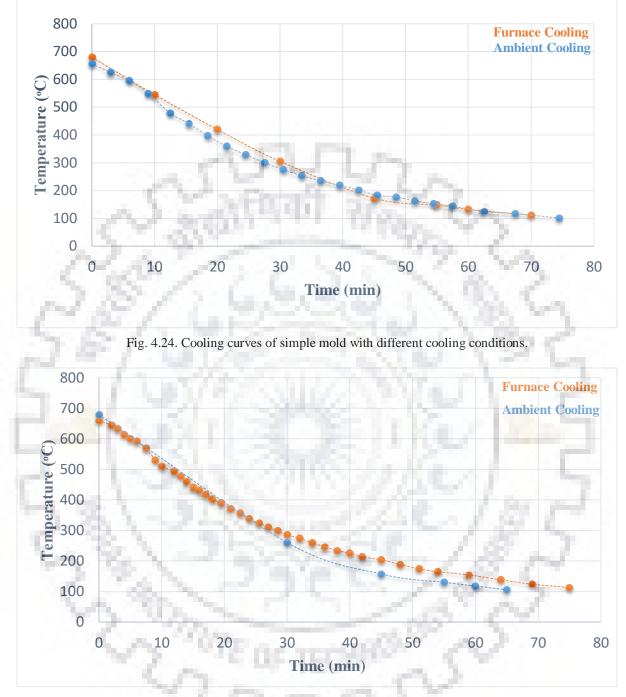


Fig. 4.25. Cooling curves of 2 mm diameter holes mold with different cooling conditions.

Cooling curves of 4 mm diameter holes mold under three different cooling conditions i.e., furnace, ambient and fan cooling are shown in Fig. 4.26. Comparison depicted that 4 mm diameter holes mold is more effective than other mold configuration and it is more effective under fan cooling condition than ambient cooling and least under furnace cooling condition.

Highest cooling rate is achieved by 4 mm diameter holes mold under fan cooling condition. But higher cooling rate is not sufficient alone, it should impart directional orientation to the solidified grains structures.

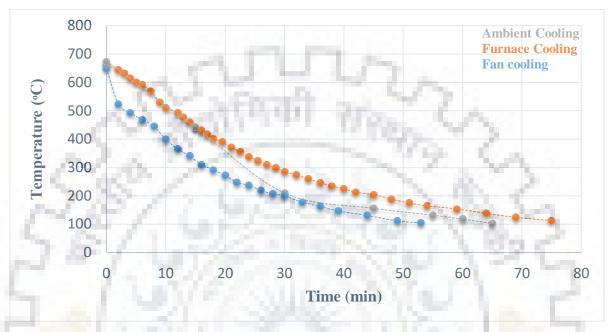


Fig. 4.26. Cooling curves of 4 mm diameter holes mold with different cooling conditions.

This can be concluded that 2 mm diameter holes mold is not effectiveness towards increasing the cooling rate under different cooling conditions and same for simple mold but the 4 mm diameter holes mold found effective and gives maximum cooling rate under fan cooling condition under natural convection range, minimum with the furnace cooling and ambient cooling is same as the furnace cooling initially but later its cooling rate increased.

4.4. Grain Size, Micro-indentation Hardness and Porosity

Average grain size found out by 'Line Intercept' method, micro-indentation harness was tested by Vicker's micro-hardness tester at 100 grams load for 30 seconds, micro-indentation hardness of simple mold under furnace cooling is shown in Fig. 4.27(a) which showed the minimum value of micro-hardness among all the mold configurations, Fig. 4.27(b) and 4.27(c) showed the second highest and highest value of micro-indentation hardness given by 4 mm diameter holes mold cooling outside applicator and under fan cooling conditions respectively.

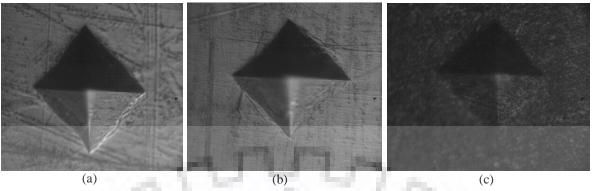


Fig. 4.27. Micro-indentation hardness (a) minimum value, (b) second highest value and (c) highest value.

Porosity was found out by Archimedes' principle, shown in Table 4.1. Distilled water at 37 $^{\circ}$ C-40 $^{\circ}$ C with density 995.65 kg/m³ is used to find porosity.

Serial No.	Mold configurations	Cooling rate (°C/min)	Average grain size (µm)	Average micro- hardness (HV)	Average bulk porosity (%)
1.	Simple mold with furnace	8.21	120±25	24.76	0.491
2.	Mold 2 mm diameter holes with furnace cooling	8.571	100±14	25.82	0.872
3.	Mold 4 mm diameter holes with furnace cooling	9	85±20	29.94	0.94
4.	Simple mold with outside furnace cooling	8.837	98±18	26.66	0.894
5.	Mold 2 mm diameter holes with outside furnace cooling	9.375	88±28	30.43	0.654
6.	Mold 4 mm diameter holes with outside furnace cooling	9.677	83±22	33.71	0.535
7.	Mold 4 mm diameter holes with fan cooling	12.23	70±25	35.56	0.743
8.	As received sample	-	180±40	21.34	3.23

Table 4.1. Grain size, micro-hardness and porosity values with respective configurations.

5.1. Conclusion

In the present study, effects of the different configurations and cooling conditions on properties of the pure aluminum in-situ cast were studied during microwave casting at power of 900 W with the radiation frequency of 2.45 GHz. Properties of the casts were characterized in terms of directional orientation of microstructures, micro-indentation hardness and average porosity.

- The study confirms that properties of the cast can be effectively controlled by controlling the cooling rate and thus by controlling the solidification of molten material inside the mold. Higher cooling rate favors grain refinement and thus increases the hardness and strength of the cast because cooling rate of the casts affects the nucleation rate, growth rate and overall growth rate of grains structure.
- 2. Microstructures are fine and uniform in every configuration but the directional orientation of grains formed could not be identified with reasonable clarity except in 4 mm diameter holes mold under fan cooling which showed a little directional orientation of grain growth.
- 3. Micro-hardness of the in-situ casts increased as grains became finer. Then, it was found to follow the "Hall-Petch" relation for grain boundary strengthening.
- 4. Denser volume of cast is obtained with porosity less than 1% in all the different configurations.
- 5. Mold with 4 mm diameter holes under fan cooling within natural convection range, showed the finest grains among the other configurations and highest average microindentation hardness value i.e., 35.56 HV. This showed that modification of mold with holes will become more effective under the presence of notable air velocity i.e. forced convection but fins configuration will be suitable for free convection condition and may give better results.
- 6. Experimental setup of in-situ microwave casting is ineffective in getting directionally solidified grains because of possible impingement of two dimensional directionally solidified grains which disturbed the one dimensional directionally solidified grains.

Directionally solidified grains have greater potential of working under higher temperature and stress conditions and are creep resistant. For instance: gas turbine blades.

5.2. Future Scope

In the present study, the effect of only mold external surface modification in terms of making blind holes was investigated which requires more studies in respect of 'Directional Solidification'. In future the following can be investigated to get directionally solidified grain structures.

- 1. Fins on single side or opposite sides of mold walls may be integrated or externally applied.
- 2. Use of through holes, where feasible, inside the mold with or without the use of fluid flow across the holes by external energy source like, pump for liquid and blower for gases.
- 3. Combination of fins, blind holes and through holes accordingly.
- 4. Different materials for the mold can be attempted.
- 5. Use of vacuum or inert gases inside the applicator cavity during processing can be investigated.
- Application of heat at one end and cooling at the other end during solidification should be explored to get directionally solidified grain structures.
- 7. Every configuration must be first simulated to compare the cooling potential and pattern for filtering the alternatives to save time and energy.

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