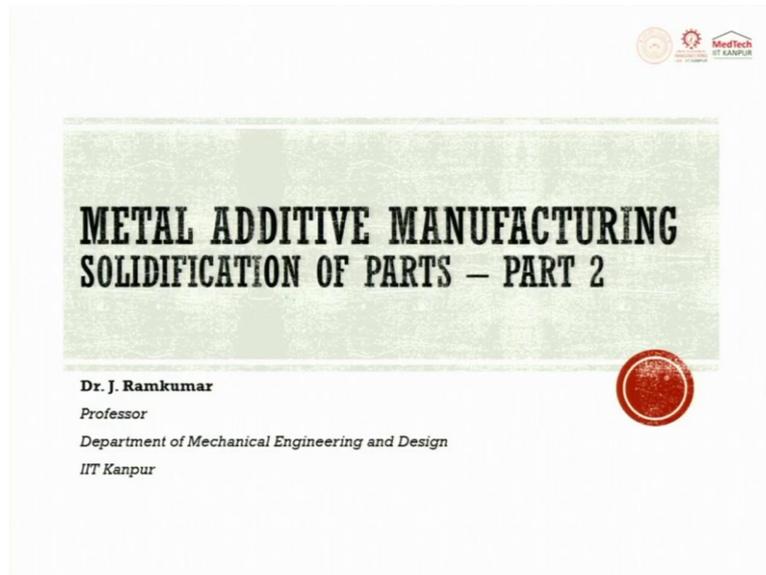


Metal Additive Manufacturing
Prof. Janakranjan Ramkumar
Prof. Amandeep Singh Oberoi
Department of Mechanical Engineering and Design
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Lecture 14
Solidification of Parts (Part 2 of 3)

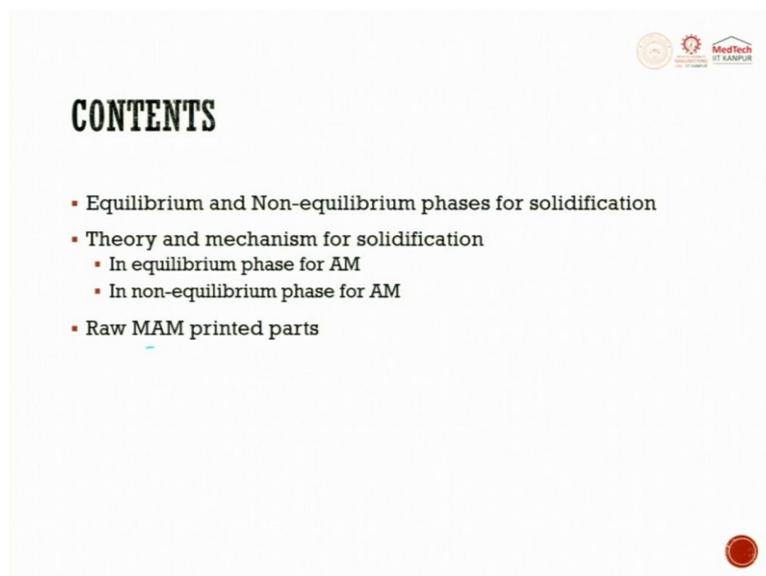
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METAL ADDITIVE MANUFACTURING
SOLIDIFICATION OF PARTS – PART 2

Dr. J. Ramkumar
Professor
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IIT Kanpur



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CONTENTS

- Equilibrium and Non-equilibrium phases for solidification
- Theory and mechanism for solidification
 - In equilibrium phase for AM
 - In non-equilibrium phase for AM
- Raw MAM printed parts



SOLIDIFICATION OF METALS: EQUILIBRIUM

- After melting in conventional casting or laser/electron-beam AM, the liquid metal solidifies through cooling.
- Thermodynamically, solid and liquid have the same energy at their melting point, so both are steady.
- As the liquid metal cools, it loses latent heat. Low liquid temperature reduces the average interatomic space between movable and disordered atoms.
- As atoms cool, their interaction forces stop them from moving apart, and the liquid turns solid.
- A large ingot of metal alloy is cast into a single crystal using a controlled solidification protocol.
- In practice, it's difficult to transform a homogeneous liquid into a perfect homogeneous solid. This requires knowledge at the angstrom, micron, meso, and macro levels.

Welcome to the second part of Solidification of Parts in Metal Additive Manufacturing series. So, in this lecture, we will try to understand more on equilibrium and non-equilibrium phases for solidification, then theory and mechanism for solidification. Last time we saw very trivially the solidification zone, freezing zone and the melting zone.

So, we saw melting first, freezing next, and solidification. So, at each phase what happens? So, now, we will go a little bit in understanding the theory and mechanism for solidification. So, in equilibrium phase and in non-equilibrium phase, the last part of the lecture we will try to see what raw metal additive manufacturing printed parts look like.

So, when we talk about solidification of metal and we talk about equilibrium here after melting in conventional casting or laser/electron beam additive manufacturing, the liquid metal solidifies through cooling. So, cooling is one of the process through which the solidification starts happening and there is a heat transfer.

Thermodynamically, solid and liquid have the same energy at their melting point. So, both are steady. So, this is very important, because, when you try to do modeling of additive manufacturing part in a metal melting pool or in a molten pool then this point is very important. Thermodynamically, solid and liquid have the same energy at the melting point.

Now, after the melting is done and cooling starts, there is a thermodynamical instability, as the liquid metal cools, it loses latent heat, low liquid temperature reduces the average inter atomic space between movable and disordered atoms. Very important low liquid temperature

reduces the average interatomic spacing. So, now, slowly the atoms are going closer between the movable and disordered atoms.

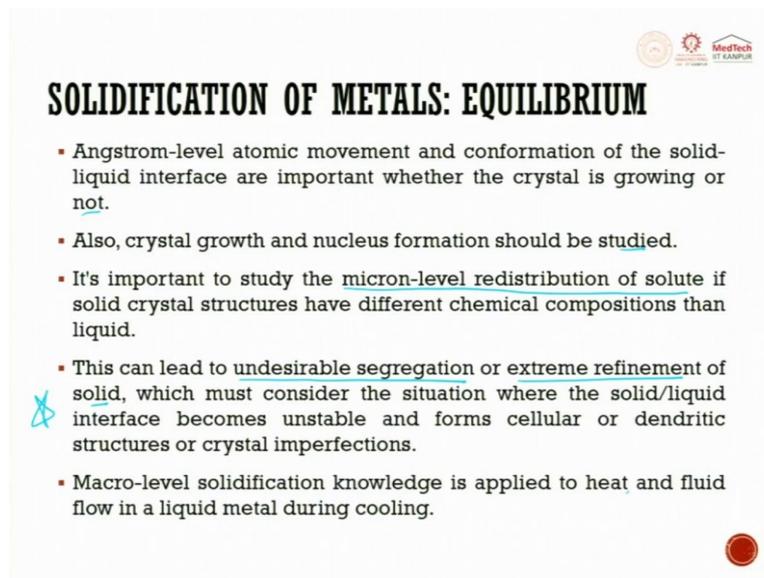
As atom cools, their interaction forces stops them from further moving apart and the liquid turns solid. So, atoms goes here and there randomly and then slowly, now it is put in a constrained fashion like a small kid left in a ground. It goes havoc here and there with lot of energy, but then the moment it hears the ringing of a bell and a school teacher coming in front slowly he gets confined and then he gets into his classroom.

So, it is almost like that, somuch of free energy. So, then as the atom cools, their interaction forces stop them from moving apart and then liquid turns solid. A large ingot of metal alloy is cast into a single crystal while controlled solidification protocol is done. So, when you do that, you can also have this grain orientation happening. In this grain orientation happening, you can try to have single crystal all throughout the ingot, which is used to be made for titanium.

So, titanium ingots are made for turbine blade applications to have better mechanical and service condition response. In practice it is difficult to transform a homogeneous liquid into a perfect homogeneous solid. This requires knowledge at the angstrom, micron, meso, and macro level.

That is why today additive manufacturing solidification part is done using molecular dynamic simulation tools. These tools will try to give more understanding at micro scale, meso scale and macro level. Macro and meso you can do it through any of the existing softwares like comsol. When it goes to micro, we always try to go towards molecular dynamics simulation and for angstrom, exactly only molecular dynamics simulation software works.

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SOLIDIFICATION OF METALS: EQUILIBRIUM

- Angstrom-level atomic movement and conformation of the solid-liquid interface are important whether the crystal is growing or not.
- Also, crystal growth and nucleus formation should be studied.
- It's important to study the micron-level redistribution of solute if solid crystal structures have different chemical compositions than liquid.
- This can lead to undesirable segregation or extreme refinement of solid, which must consider the situation where the solid/liquid interface becomes unstable and forms cellular or dendritic structures or crystal imperfections.
- Macro-level solidification knowledge is applied to heat and fluid flow in a liquid metal during cooling.

The angstrom-level atomic movement and confirmation of the solid liquid interface are important, whether the crystal is growing or not. If it is not growing, then you can use that time period for diffusing or doing something else, if it has started solidifying, then start properly using it. So, the heat is extracted when the grains are formed and the solid formed is something like your quenching cycle, you heat the liquid, you heat the solid to a particular temperature, try to deform it whatever you want and instantly quench it in water.

So, then when you quench it in water, you freeze in the same way you start playing with this angstrom-level atomic movement, you start playing it to get the output here. Friends, since metal is in a powder form, the surface area is large, volume is too low, the laser energies are very high, the melt-controlled solidification can be done very easily.

Whether the crystal is growing or not also, crystal growth and nucleus formation should be studied. This depends on the thermodynamic free energy available and the stability of the nucleus, it is important to study the micro level redistribution of solvent if solid crystal structure have different chemical composition than the liquid.

So, once the different phases are getting formed, then there is a solidification difference which is happening. So, the material gets redistributed and then it is trying to get formed. This can lead to undesirable segregation or extreme refinement of the solid.

So, now, you see this undesirable segregation or extreme refinement of the solid that leads to better mechanical properties which must consider the situation where the solid-liquid

interface becomes unstable and form cellular or dendritic structure or crystal imperfections. If you understand this, then you try to play with the laser matter interaction.

What is the laser matter interaction? Laser power with respect to time, whether to stop for microsecond, millisecond, nanosecond or femtosecond at that spot and try to have multiple frequency hits to generate liquid and then you move in a feed rate such that this liquid is converted into a solid. During this conversion, it goes from liquid, solid liquid interface and then it goes to solid.

So, here when it goes into this transition, again time and temperature comes into existence. This time and temperature depending upon the composition tries to push people or the particles here and there forms precipitates, this is what happens. If you want to have equiaxed grains or if you want to have dendritic grains then you again play with the time.

So, all these things are getting made in a fraction of a second in a given spot. Macro level solidification knowledge is applied to heat and fluid flow in liquid metal during cooling. So, whatever was a knowledge which we get from solidification point of view in casting, the same thing can be brought in here, the only difference is going to be the interaction time and the interaction area.

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**SOLIDIFICATION IN ADDITIVE MANUFACTURING:
NON-EQUILIBRIUM**

- Since AM involves the rapid melting of metals, solidification theory is important for predicting and monitoring the process.
- Laser- or EB-based AM uses a moving heat source with a short interaction time.
- Laser powder bed fusion uses a 4 m/s laser with a 100 μm spot size.
- This means a 100 μm process zone is heated when its maximum temperature can be reached in less than 25 μs.

Handwritten diagrams: A blue sketch of a laser beam hitting a surface, and a blue sketch of a laser beam hitting a surface with a 'melt pool' and 'Area' labeled.

So, non-equilibrium let us see, since additive manufacturing involves rapid melting of metal, solidification theory is important for predicting and monitoring the process. See while doing the additive manufacturing we studied about closed loop system you can put sensors and find

out whether the fumes are created, where the temperature is going high or where there is an explosion of the powder. We saw that in the past lectures, what is the reason for exploding the powder while additive manufacturing is done through on a powder bed fashion?

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**SOLIDIFICATION IN ADDITIVE MANUFACTURING:
NON-EQUILIBRIUM**

- Since AM involves the rapid melting of metals, solidification theory is important for predicting and monitoring the process.
- Laser- or EB-based AM uses a moving heat source with a short interaction time.
- Laser powder bed fusion uses a 4 m/s laser with a 100 μm spot size.
- This means a 100 μm process zone is heated when its maximum temperature can be reached in less than 25 μs .

Hand-drawn diagrams: A blue circle labeled 'laser' with a horizontal line through it. To the right, a horizontal line with vertical tick marks is labeled 'Area'. Below it, a blue shape is labeled 'melt pool'. A blue arrow points from the 'Area' towards the 'melt pool'. A blue double-headed arrow above the 'Area' is labeled '15 - 100 μm '.

So, predicting and monitoring the process. So, for predicting and monitoring apart from all these things, you also use solidification theory. Laser or electron beam based additive manufacturing uses a moving heat source, this moving heat source is a small diameter feed rate given when it is moving, how is the heat getting conducted through the manufactured part with a short interaction time.

The laser powder bed fusion uses 4 m/s laser with a spot diameter of 100 μm size. So, that you can try to do all this rapid melting and solidification in a small spot and 100 μm , what we talk about is a spot size and our powder size varies from 15 μm to 100 μm . So, in a very crude fashion, you can see the spot diameter and the powder diameter is the same.

So, generally you will not melt 1 powder because these powders will be oriented in different directions. So, you will try to melt 3, 4 powders and these 3, 4 powders when you melt it can go, if you go along a straight line, it also can melt in this direction.

So, this makes an area and this area is a 2D. So, there is also the laser waste which is there in the focal plane. So, if you are trying to move along a line, so, the heat gets disseminated across laterally then it tries to have an area of melt, this area of melt is 2D.

So, when you try to put it in the powder bed fusion method, it will have a volume. So, a 3d instance of this is called as the melt pool. So, this means 100 μm process zone is heated with its maximum temperature can be reached in less than 25 μs .

Within a second you can try to melt and if the laser is going fast it starts solidifying. So, before that whatever we were thinking about rattling, moving, inter-atomic distance, freezing, everything happens in below 25 μs .

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SOLIDIFICATION IN ADDITIVE MANUFACTURING: NON-EQUILIBRIUM

Thermal Conductivity:

- Due to thermal conductivity, solidification and cooling can take twice as long as heating.
- Due to the substrate material, the surrounding heat conduction/convection regimes cause high solidification and cooling rates.
- The point heat source produces a highly localised heat flux in the melt pool zone and a massive temperature gradient in the deposited layers.
- The substrate bottom has a higher temperature gradient than the deposited top.

The slide includes a diagram showing a laser beam (Heat) hitting a substrate, with a melt pool forming. The diagram is annotated with 'Cool' and 'Heat' arrows, and 'Powder' is labeled. At the bottom, there are sketches of different powder bed fusion patterns: a cross-hatched area, a spiral, and a circular pattern. Logos for MedTech and ST KANPUR are visible in the top right corner.

So, thermal conductivity is a very important parameter which tries to dictate the solidification suppose, if the thermal solid its conductivity is very low, all the heat gets accumulated at that spot it does not allow the heat to go.

So, then the melt pool will be existing for a longer time. So, longer time means, then it will try to have different non-equilibrium conditions to do it. For example, in titanium what happens, when you start machining titanium, entire accumulation of the heat happens at the machine zone itself, it does not disseminate very fast this tries to distort the tool.

So, in the same way when we try to do copper powder, the thermal conductivity is extremely large and heat applied depth gets disseminated. So, you have a larger melt pool area. So, when you have a larger melt pool area, the grain, the grain boundary, the grain dynamics completely changes depending upon the thermal conductivity property of the powder, the equilibrium and non-equilibrium states gets achieved very fast.

Due to thermal conductivity, solidification and cooling can take twice as long as heating. So, this is a very important property when many of us do modeling for an additive manufacturing process. We try to take the thermal conductivity of a solid and then we try to fit into the program, but you should understand here you are trying to work with powder and this powder is not continuous, it is discrete.

So, when you try to take metal block, it is continuous and here the thermal conductivity is different. Now, here when we try to take powder, it is completely different and the response for a slab or a powder is different, the conductivity is different and here there is a discontinuity coming. So, here it is continuous. So, the modeling you will always try to have an error in the final prediction.

So, due to the substrate material, the surrounding heat conduction convection regimes causes high solidification and cooling rate. What are we talking about convection is something called as Marangoni convection. What is Marangoni convection? You have a melt zone in the melt zone, this is heat or hot zone and this is cold zone.

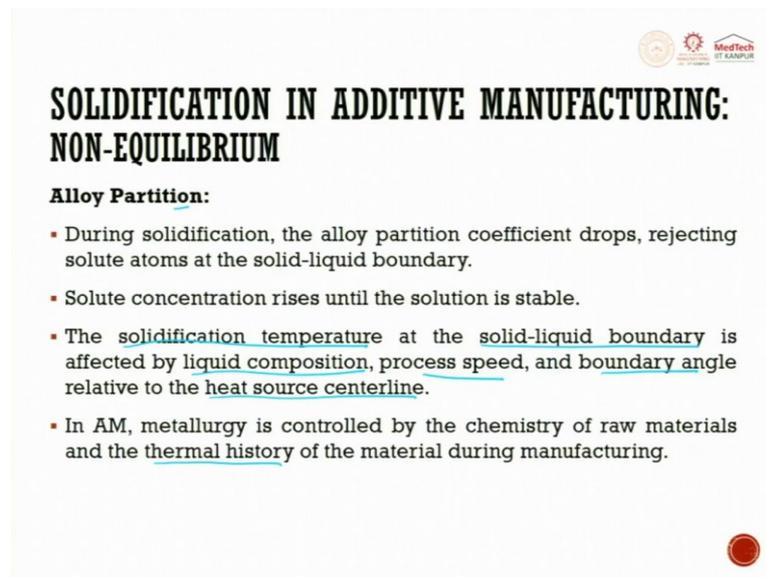
So, now, there is a difference in temperature this difference in temperature tries to bring in some small circulation of heat. So, this heat circulation creates a vortex. So, this vortex is called as Marangoni convection because of heat and cold zones you try to do this. So, that is what is the convection which is there, regime causes high solidification and cooling rates.

The point heat source produces a highly localized heat flux in the melt pool zone and a massive temperature gradient in the deposition area. So, now, it is very interesting. You are trying to move along a line. So, this is a straight line. So, now, if you try to have hatch patterns like this, then it is a different story. If you have something like out to in or in to out hatch patterns, then completely different phenomenas are happening. When the point heat source you try to move to a moving heat source, it is different.

So, the point heat source produces highly localized heat flux, point melting a powder or a set of powders in the melt pooled zone and a massive temperature gradient, because there are 4 powders which are melt, rest of the powders who are about to melt is perfectly powder at room temperature or at the bed temperature what ever we said in the deposition layer, the substrate bottom has a higher temperature gradient than the deposition top.

So, here at the bottom portion, the layer when it is recently formed, will be of a higher temperature as compared to that of a new layer which is brought in the powder bed fusion method for the laser to move on top of it. So, that is what the substrate bottom has a higher temperature gradient, from here it goes down than the deposited top.

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The slide features a title 'SOLIDIFICATION IN ADDITIVE MANUFACTURING: NON-EQUILIBRIUM' in bold black text. Below the title is a sub-heading 'Alloy Partition:' followed by a bulleted list of four points. The slide also includes logos for IIT Kanpur and MedTech at Kanpur in the top right corner, and a red circular logo in the bottom right corner.

**SOLIDIFICATION IN ADDITIVE MANUFACTURING:
NON-EQUILIBRIUM**

Alloy Partition:

- During solidification, the alloy partition coefficient drops, rejecting solute atoms at the solid-liquid boundary.
- Solute concentration rises until the solution is stable.
- The solidification temperature at the solid-liquid boundary is affected by liquid composition, process speed, and boundary angle relative to the heat source centerline.
- In AM, metallurgy is controlled by the chemistry of raw materials and the thermal history of the material during manufacturing.

So, when we are trying to work on alloy partition, during solidification the alloy participation coefficient drops, rejecting solute atoms at that solid liquid boundaries. The solute concentration rises until the solution is stable.

The solidification temperature at the solid liquid boundary is affected by liquid composition, process speed, boundary angle relative to the heat source centerline. The solidification temperature at the solid liquid boundary is affected by liquid composition because alloys have mixed processing speed and boundary angle.

In AM, metallurgy is controlled by the chemistry of raw material and the thermal history of the material during manufacturing, this is very important. This makes the life of simulation experts difficult to bring in reality, what happens during additive manufacturing. Still a long way to go in the research front also as far as powder bed fusion itself.

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SOLIDIFICATION IN ADDITIVE MANUFACTURING: NON-EQUILIBRIUM





Heat Transfer Mechanisms:

- LPBF and LDED may have different heat transfer mechanisms, but laser and EB AM have the same metallurgical principles for full melting.
- Cooling to ambient temperature after solidification controls the deposited layer's early phase distribution and grain morphology.
- Heat source speed, power, and size govern melt pool geometry, which controls solidification kinetics.
 - Thermal cycling and cooling control precipitation kinetics, phase growth, and grain growth.




Laser powder bed fusion and laser directed energy deposition may have different heat transfer mechanism. So, laser hitting at the powder bed, powder bed fusion and laser trying to have powder fed in either coaxially or laterally, this is laser, this is nozzle.

So, laterally or coaxially the heat transfer mechanisms are different for laser and electron beam. It is not the same heat transfer mechanisms, but laser and electron beam have the same metallurgical principles for full melting, selective laser sintering is different from selective laser melting.

Cooling to ambient temperature after solidification controls the deposited layers, early phase distribution and grain morphology. So, here when it cools from the freezing to the room temperature, here it tries to the deposited layers. Early phase distribution and grain morphology is very important.

The heat source speed, power and size govern melt pool geometry, which controls solidification kinematics. Heat source speed, power size governs the melt pool geometry, whether the melt pool also we assume it like a cylinder, but in reality it will have a shape something like this. So, melt pool geometry is very important.

I am not getting into details of solidification kinematics because that will be covered in few of the casting courses offered by books or Nptel. The thermal cycling and cooling control precipitation kind of metrics phase growth and grain boundaries are different. The thermal cycling and cooling controls.

The thermal cycling is a single spot what happens when you try to have a laser powder. So, that thermal cycling and cooling controls precipitation kinematics phase growth and grain growth.

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The slide features a title in bold black text: "SOLIDIFICATION IN ADDITIVE MANUFACTURING: NON-EQUILIBRIUM". Below the title is a sub-heading "AM vs casting solidification:" followed by a bulleted list of four points. The slide also includes logos for IIT Kanpur and MedTech at Kanpur in the top right corner, and a small red circular logo in the bottom right corner.

**SOLIDIFICATION IN ADDITIVE MANUFACTURING:
NON-EQUILIBRIUM**

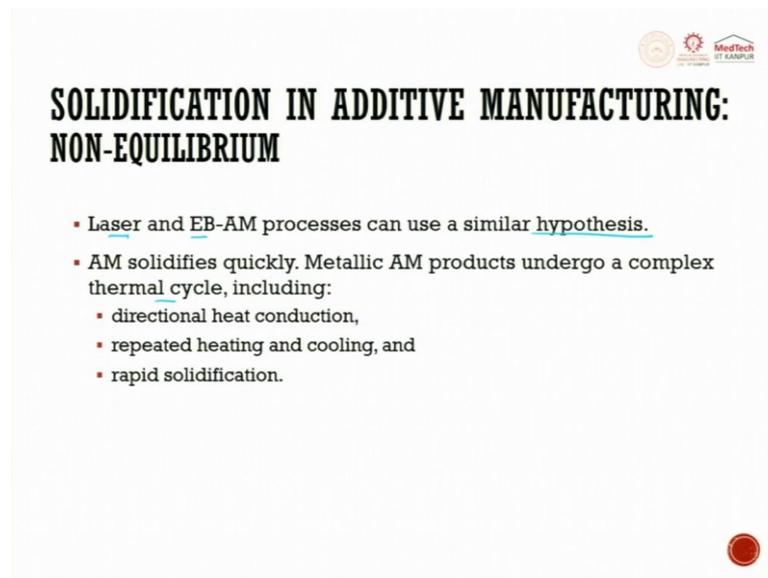
AM vs casting solidification:

- In some cases, AM solidification differs from casting.
- For AM, the melt pool size ranges from hundreds of micrometres to millimetres, regardless of the final component size, and is therefore different from castings with larger melt pool areas.
- Small melt pools have less macrosegregation than larger castings.
- In LPBF, the powder bed around the melt area has lower thermal conductivity (due to voids between particles) than bulk alloy material.

When we try to compare the solidification of additive manufacturing with casting solidification, in some cases, additive manufacturing solidification differs from casting. For additive manufacturing, melt pool size ranges from hundreds of micrometer to millimeter, regardless of the final components size and shape and is therefore, different from casting with large melt pool area, very different.

So, here it is hundreds of micro meter to a millimeter small melt pool have less macro segregation than larger castings. This is what I said you can dictate the product whatever you want and the quality and the mechanical properties of it. In laser powder bed fusion the powder bed around the melt area has lower thermal conductivity than bulk alloy material due to voids between the particles.

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The slide features a title in bold, uppercase letters: "SOLIDIFICATION IN ADDITIVE MANUFACTURING: NON-EQUILIBRIUM". In the top right corner, there are three logos: a circular emblem, a gear icon, and the text "MedTech UNIVERSITY OF KANPUR". The main content is a bulleted list with three primary points. The first point is "Laser and EB-AM processes can use a similar hypothesis." The second point is "AM solidifies quickly. Metallic AM products undergo a complex thermal cycle, including:" followed by three sub-points: "directional heat conduction," "repeated heating and cooling, and" and "rapid solidification." A small red circular logo is located in the bottom right corner of the slide.

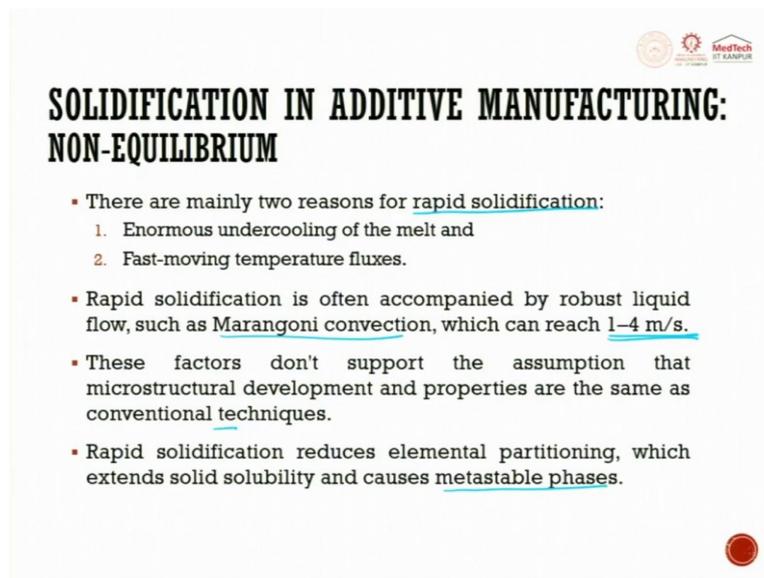
- Laser and EB-AM processes can use a similar hypothesis.
- AM solidifies quickly. Metallic AM products undergo a complex thermal cycle, including:
 - directional heat conduction,
 - repeated heating and cooling, and
 - rapid solidification.

Laser and electron beam additive manufacturing processes can use a similar hypothesis. AM solidifies quickly metallic AM products and undergo a complex thermal cycle including directional heat conduction, repeated heating and cooling and rapid solidification.

So, in order to make our life simple, we are trying to have this laser and electron beam because in electron beam it happens inside a vacuum you can still dictate the 3 of these processes but whereas, in laser you have an ambience which is created, but that is not as controllable as that of electron beam.

But in order to have a simplicity in life, we try to take laser and electron beam having the same hypothesis. These are the major complex thermal cycle phenomena which includes directional heat conduction, repeated heating and cooling and rapid solidification.

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The slide features a title 'SOLIDIFICATION IN ADDITIVE MANUFACTURING: NON-EQUILIBRIUM' in bold black text. Below the title is a bulleted list of four points. The first point is 'There are mainly two reasons for rapid solidification:' followed by a numbered list: '1. Enormous undercooling of the melt and' and '2. Fast-moving temperature fluxes.' The second point is 'Rapid solidification is often accompanied by robust liquid flow, such as Marangoni convection, which can reach 1-4 m/s.' The third point is 'These factors don't support the assumption that microstructural development and properties are the same as conventional techniques.' The fourth point is 'Rapid solidification reduces elemental partitioning, which extends solid solubility and causes metastable phases.' The slide includes logos for 'MedTech' and 'ST. KRUPUR' in the top right corner and a red circular logo in the bottom right corner.

**SOLIDIFICATION IN ADDITIVE MANUFACTURING:
NON-EQUILIBRIUM**

- There are mainly two reasons for rapid solidification:
 1. Enormous undercooling of the melt and
 2. Fast-moving temperature fluxes.
- Rapid solidification is often accompanied by robust liquid flow, such as Marangoni convection, which can reach 1-4 m/s.
- These factors don't support the assumption that microstructural development and properties are the same as conventional techniques.
- Rapid solidification reduces elemental partitioning, which extends solid solubility and causes metastable phases.

There are mainly two reasons for rapid solidification. Enormous under cooling of the metal and fast- moving temperature flux, these two tries to give a rapid solidification. What is enormous under cooling? Enormous under cooling is basically trying to go down rapidly cooling and then you come up, we will see that in detail in the forthcoming slides.

Rapid solidification is often accompanied by robust liquid flow such as Marangoni convection, which can read 1 to 4 m/s very high, these factors does not support the assumption that micro structural development and properties are the same as conventional technique. We try to understand casting very well, we have modelled casting process over a period of time to a large extent.

Now, from that understanding, we are trying to move to additive manufacturing where in which we see all these challenges. Enormous undercooling of the metal and the fast-moving temperature flux rapid solidification reduces elemental partitioning, which extends solid solubility and causes metastable phases. New phases, which was not earlier possible in casting could be achieved by fast moving temperature flux.

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The slide features a title in bold black text: "SOLIDIFICATION IN ADDITIVE MANUFACTURING: NON-EQUILIBRIUM". Below the title, the section "Heat conduction:" is followed by a bulleted list of four points. The slide also includes logos for IIT Kanpur and MedTech in the top right corner and a red circular logo in the bottom right corner.

**SOLIDIFICATION IN ADDITIVE MANUFACTURING:
NON-EQUILIBRIUM**

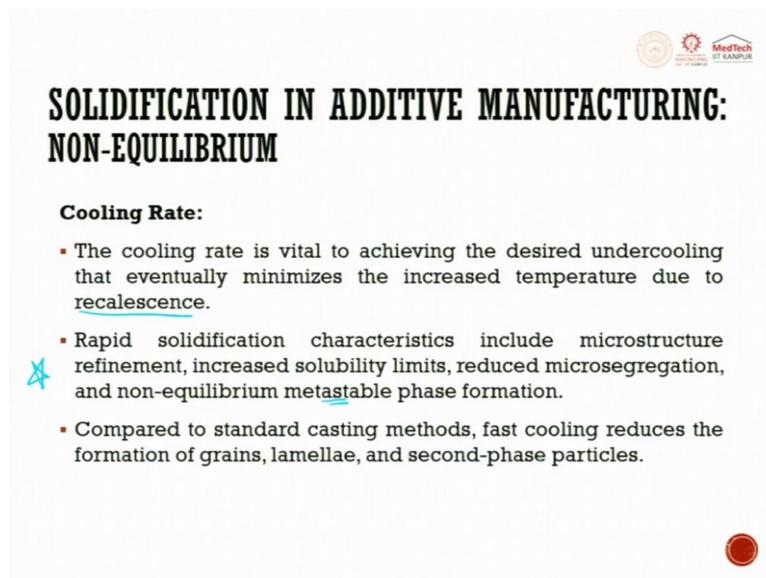
Heat conduction:

- Due to heat conduction, grain growth may be directional.
- Rapid and directional solidification and thermally-induced phase transformation affect the microstructure of deposited parts.
- Rapid solidification in the melt pool improves mechanical properties by allowing more structural latitude than in conventional manufacturing.
- Undercooling liquid before nucleation and solidification growth rate cause these unique structures.

Due to heat conduction, grain growth may be directional. Rapid and directional solidification and thermal induced phase transformation affects the microstructure of deposited parts. Rapid and solidification in the melt pool improves mechanical properties by allowing more structural latitude than in conventional manufacturing.

So, rapid solidification is nothing but something like quenching. It is not equivalent, but something like under cooling before nucleation and solidification growth causes unique structures. So, rapid solidification happens, directional solidification happens, you get this by doing thermal cycling.

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The slide features a title 'SOLIDIFICATION IN ADDITIVE MANUFACTURING: NON-EQUILIBRIUM' in bold black text. Below the title is a section header 'Cooling Rate:' followed by three bullet points. The first bullet point discusses the cooling rate's role in achieving undercooling and minimizing temperature increase due to recalescence. The second bullet point, marked with a blue asterisk, lists characteristics of rapid solidification: microstructure refinement, increased solubility limits, reduced microsegregation, and non-equilibrium metastable phase formation. The third bullet point compares fast cooling to standard casting methods, noting it reduces the formation of grains, lamellae, and second-phase particles. Logos for IIT Kanpur and MedTech are visible in the top right corner, and a red circular logo is in the bottom right corner.

SOLIDIFICATION IN ADDITIVE MANUFACTURING: NON-EQUILIBRIUM

Cooling Rate:

- The cooling rate is vital to achieving the desired undercooling that eventually minimizes the increased temperature due to recalescence.
- ✱ Rapid solidification characteristics include microstructure refinement, increased solubility limits, reduced microsegregation, and non-equilibrium metastable phase formation.
- Compared to standard casting methods, fast cooling reduces the formation of grains, lamellae, and second-phase particles.

So, here the other important thing is the cooling rate. We are only talking about heating, but also the cooling rate, it follows a fatigue cycle. So, the cooling rate is vital to achieve the desired under cooling, that eventually minimizes the increased temperature due to the recalescence.

Rapid solidification characteristics include micro structural refinement, increased solubility limits, reduced micro segregation and non-equilibrium metastable phase formation. Look at it, you have something like a khichadi everything mixed. Rapid solidification characteristics include micro structural refinement, increased solubility limits, reduced micro segregation and non-nuclear metastable phase formation.

This metastable phase sometimes can lead you to invisible alloys also. So, this metastable phase can also lead you to something like intermetallic phases forming. Compared to standard casting methods, fast cooling reduces the formation of grains, lamellae, and secondary phase particles.

(Refer Slide Time: 27:54)

**SOLIDIFICATION IN ADDITIVE MANUFACTURING:
NON-EQUILIBRIUM**

- Dendritic segregation can be reduced to achieve compositional homogeneity.
- * AM solidification affects grain size, shape, distribution, growth kinetics, elemental segregation and precipitation, phase transformation, and material properties.
- * The key factors in this process are multiscale multiphysics phenomena (i.e., fluid flow, heat and mass transfer, beam interaction with the material) caused by AM's localized heating and cooling.

Handwritten notes:
laser - cutting - polishing
- melting - sintering
- heat treatment

Dendritic segregation can be reduced to achieve compositional homogeneity in additive manufactured parts. In additive manufacturing, solidification affects grain size, shape, distribution, growth, kinematics, elemental segregation, precipitation, phase transformation and material properties. All are dictators.

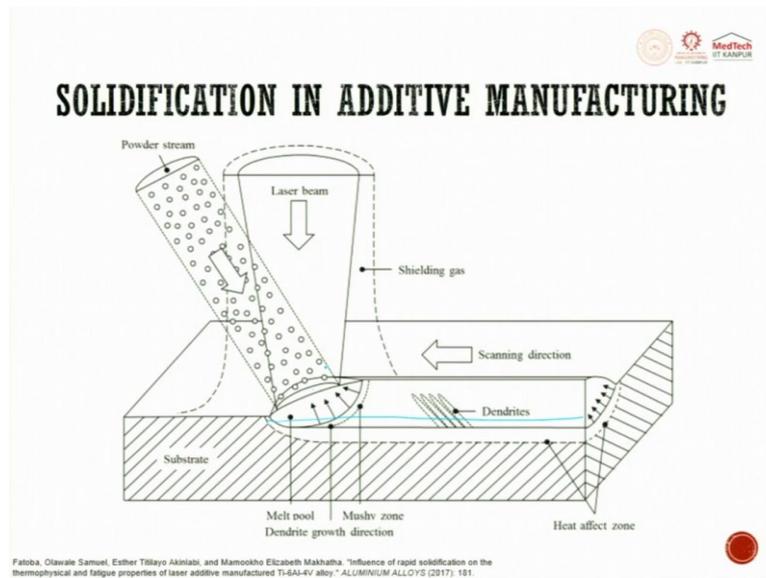
The key factor in the process are multiscale, multiphysics phenomena, what is that? Fluid flow heat and mass transfer beam interaction with material causes additive manufacturing localized heating and cooling.

It has multiscale multiphysics phenomena, because solid gets converted into a liquid, liquid will try to flow, in the flow you have viscosity, you have surface tension and when these 2 are there, there is a temperature gradient coming, then you have the heat and mass transfer happening, then the beam keeps moving very fast, beam interaction time.

So, all these things make additive manufacturing process a robust and a good process to make sound products selectively in a flexible manner. If you want to have on surface wear resistance property, you just play with laser interaction time with material. The laser can be used for cutting, melting, heat treatment.

It can also be used for polishing, the laser interaction material with time and power. You can do any of these things. You can also do so, sintering pole, same with electron beam also, you play with the energy densities you can get that.

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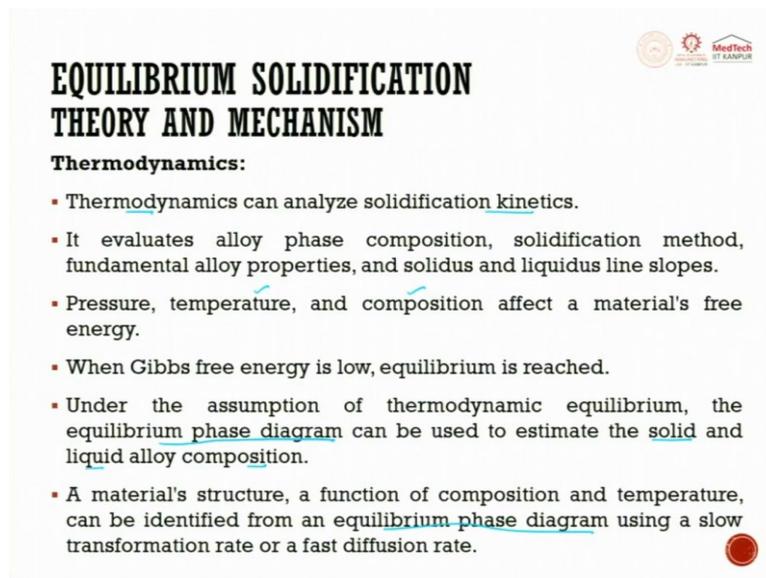
So, when we tried to talk about laser, so, this is the laser coming, this is directed energy deposition method where there is a powder stream coming. This is hitting the surface, you have a substrate, then you see the substrate as and when it is getting developed, it is getting cooled. So, you have dendrites getting formed.

So, this is the direction in which you are moving and this in order to protect the melt pool getting oxidized or explosive reactions happening. So, we try to put a shield around it, so, this is almost like a neutral gas, which prevents it from getting interaction with the surface.

So, you see here the powder particles again, you will have alloys, when it melts, you will have different responses, the alloy solidification time are different, in alloys the grain boundaries happen against segregation.

So, you dictate the temperature whatever is getting formed. So, you not only melt the powder, you also try to melt one more layer down and these 2 fellows gets pitched because of the melt solidification process happening here.

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**EQUILIBRIUM SOLIDIFICATION
THEORY AND MECHANISM**

Thermodynamics:

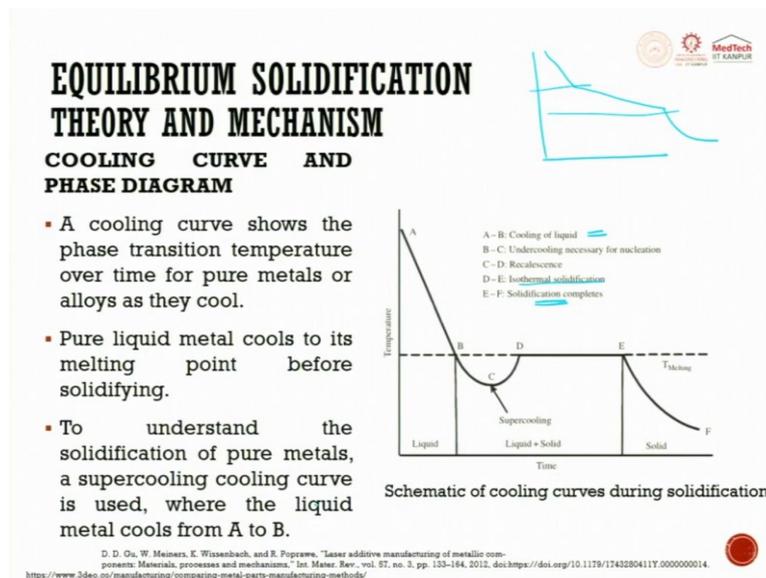
- Thermodynamics can analyze solidification kinetics.
- It evaluates alloy phase composition, solidification method, fundamental alloy properties, and solidus and liquidus line slopes.
- Pressure, temperature, and composition affect a material's free energy.
- When Gibbs free energy is low, equilibrium is reached.
- Under the assumption of thermodynamic equilibrium, the equilibrium phase diagram can be used to estimate the solid and liquid alloy composition.
- A material's structure, a function of composition and temperature, can be identified from an equilibrium phase diagram using a slow transformation rate or a fast diffusion rate.

So, a little bit on thermodynamics. So, thermodynamics can analyze solidification kinematics, it evaluates alloy phase composition, solidification method, fundamental alloy properties and solidus, liquidus line slope. So, all these things are part of thermodynamics and solidification kinematics. Pressure, temperature, composition affect materials free energy.

So, here we use these 2, we do not use pressure. When Gibbs free energy is low, equilibrium is reached. Under the assumption of thermodynamic equilibrium, the equilibrium phase diagram can be used to estimate the solid, liquid alloy composition by using the equilibrium phase diagram.

This equilibrium phase diagram is drawn for solid casting, solid material or bulk material powder interacting with laser short time, this equilibrium phase diagrams get tweaked a little bit for additive manufacturing parts and especially with respect to metal additive manufacturing parts. The materials structure a function of composition and temperature can be identified from an equilibrium phase diagram using your slow transformation rate or fast diffusion rate.

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This is the typical cooling curve, which we always discuss in casting process. So, you can see here A, B is the cooling from liquid to freezing B and E. So, BE is the liquid solid. So, you will have freezing going on and E to F will be solidification complete. So, you see here this is liquid, this is solid. So, in between B and E there are so many phenomena which happened and this so, many phenomena will try to dictate the output.

You take an example, AB is liquid, then BC what will happen, there is an undercooling which is necessary for initiating nucleation process, then from C to D there is recalescence happen, then DE is isothermal solidification, all these things happen in casting as well as in additive manufacturing in the melt pool, the time will be small. So, because of that rapid timing is going on.

So, you will have multiple new faces coming into action. So, the cooling curve shows the phase transition temperature over time for a pure metal or alloy as they cool. When you try to do it for a pure metal, it is like this, when you try to do for an alloy, it is something like this. So, you will have this freezing will go like this.

So, this is the freezing zone, this is the solidification zone in the liquid and in solidification zone there will be not much change except for the slope change, but in the freezing zone there will be a lot of change. So, it will not be isothermal solidification. The pure liquid metal cools to its melting point before solidifying to understand the solidification of a pure metal. Super cooling curve is used where the liquid metal cools from A to B.

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EQUILIBRIUM SOLIDIFICATION THEORY AND MECHANISM

COOLING CURVE AND PHASE DIAGRAM

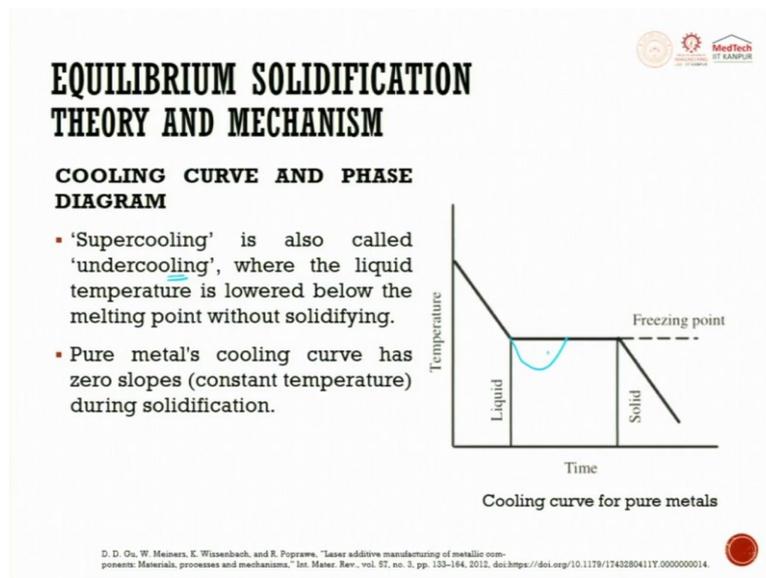
- B and C must be undercooled. Since nucleation begins at point C, the liquid's temperature rises due to the latent heat of fusion.
- This is recalescence (from point C to D). From here, solidification continues at (T_m) until point E.
- The cooling rate indicates when the solid starts to cool.
- Based on the atomic configuration, solid and liquid metals have different thermal conductivities.
- Different regions have different cooling curve slopes.
- Before solidification begins, the temperature falls below equilibrium cooling, known as "supercooling," to develop a thermodynamic driving force.



Then from B to C, it must be under cooled since nucleation begins at the point C, so, till that time there will be a rapid struggle of the fittest. So, nucleation will start and it will try to expand. The extra temperature will kill, so, this will go back and forth. So, since nucleation begins at point C, the liquid temperature rises due to latent heat of fusion, so it rises. So, this is recalescence from the point C to D. From here, solidification temperature at T_m , until the point E is the freezing zone.

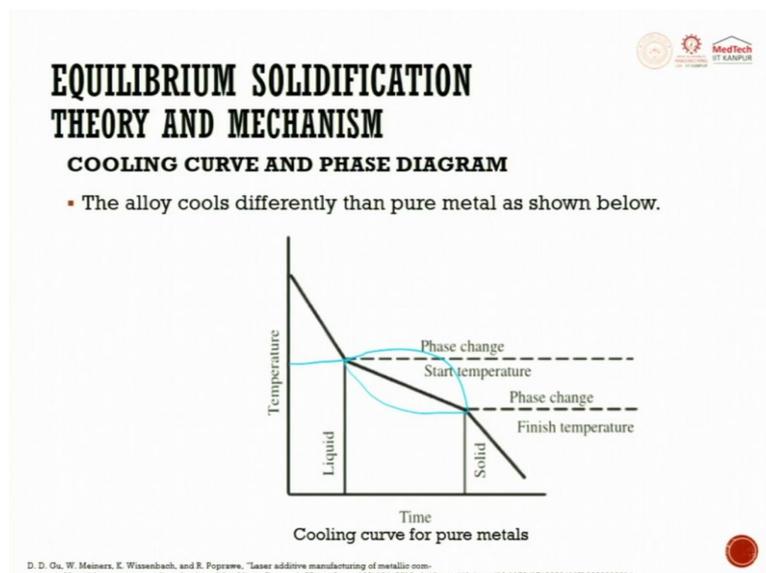
The cooling rate indicates that the solid starts to cool and based on the atomic configuration solid and liquid metal having different thermal conductivity. You have different atomic configurations, different direction, regions having different cooling curves. Slopes are seen before solidification begins, the temperature falls below the equilibrium cooling called as super cooling to develop a thermodynamic driving force.

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So, when we try to talk about this, what we have been talking in plenty, super cooling is also called under cooling. So, do not get confused. Super cooling is different under cooling, both are the same, this is what is under cooling, where the liquid temperature is lowered below the melting point without solidifying. Pure metal cooling curve has zero slope, constant temperature during solidification, this is an ideal case.

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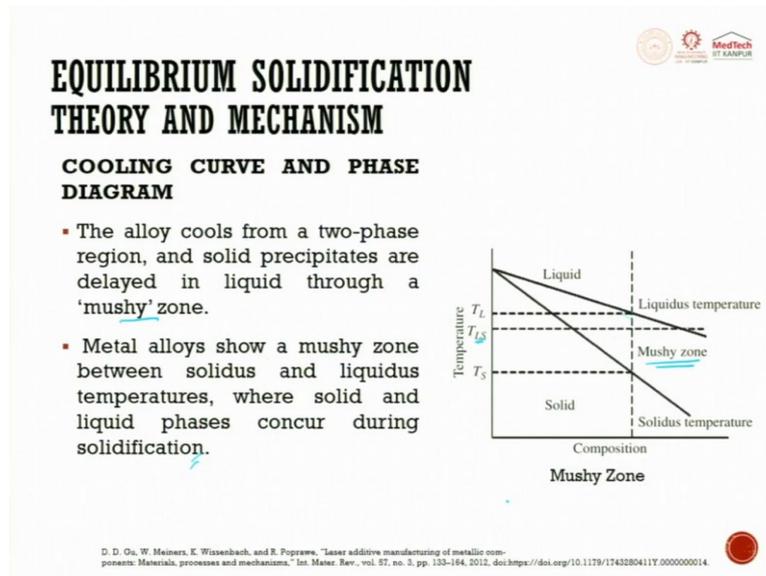


But whereas, when you do alloys this is what will happen for all engineering materials, let it be bulk or let it be powder you will have an alloy. So, if you see you will have melting then a phase change will happen. Start temperature, phase change, finished temperature, and here is

what a big paradigm shift happens from casting to additive manufacturing process and this henceforth, here will be almost the same.

The alloy cools differently than the pure metal as shown here. So, here there is lot of things in equilibrium like thermodynamic stability. Thermodynamic kinematics is there to dictate what you want.

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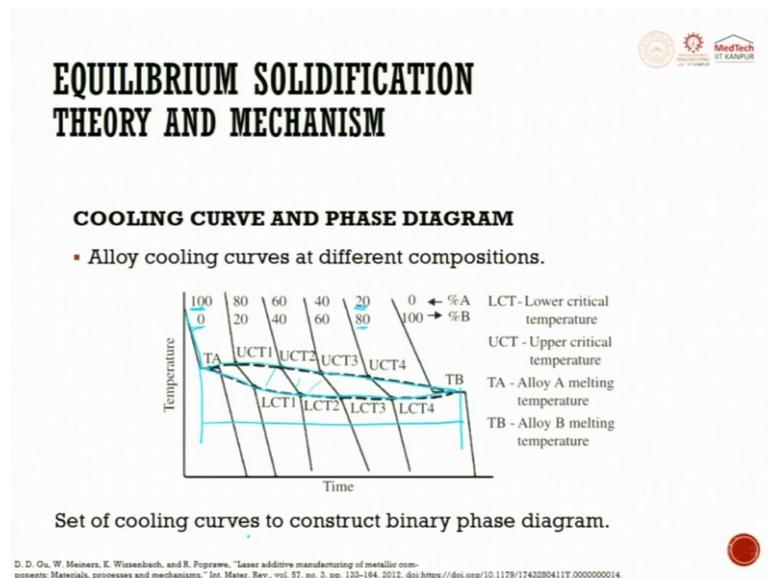


So, when we look at the cooling curve and phase diagram, you can see here this is liquid, there is a solid and in between the liquid and the solid, you have liquidus temperature line, you would have a solidus temperature line and in between you have a mushy zone, this mushy zone you try to dictate in additive manufacturing.

So, if you try to take the cooling curve and the phase diagram, for the cooling curve from a two-phase region the solid precipitates are delayed in liquid through a mushy zone. So, this is what is the liquid, this is what is the solid. So, start of liquid to get into a solid is called as liquidus temperature, the solid is called as solidus temperature.

So, you can see here this is T_L , temperature of the liquidus line, temperature of the solidus line, temperature of liquid solid line also you can see here. The metal alloy shows a mushy zone between solidus and liquidus temperature where solid and liquid phase occurs during solidification. So, here you can try to have various compositions and this various compositions with the heating cycle, can try to dictate your grain.

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So, if you see a cooling curve under phase diagram, alloy cooling curve at different compositions you can see this is, what is the temperature melting zone, this is called the freezing zone and then it is solidification zone. So, here if you see that in an alloy, you will try to have this as solidus, this is a liquidus line, this is a solidus line and now here is a mushy zone.

So, now, if you try to draw plots, you will try to see various compositions you can try to take for an alloy. This top one talks about here it is 100 % of A and 0 % of B and when it comes here, it is 20 and 80, you can try to have this what is called as upper critical temperature, lower critical temperature, T_A and T_B . T_B is a point when alloy B melting temperature is reached and T_A is a point where alloy A melting temperature is reached.

So, now with this composition, you are with this solidus line when you draw you can try to find out what are all the phases and what all segregations can happen.

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EQUILIBRIUM SOLIDIFICATION THEORY AND MECHANISM

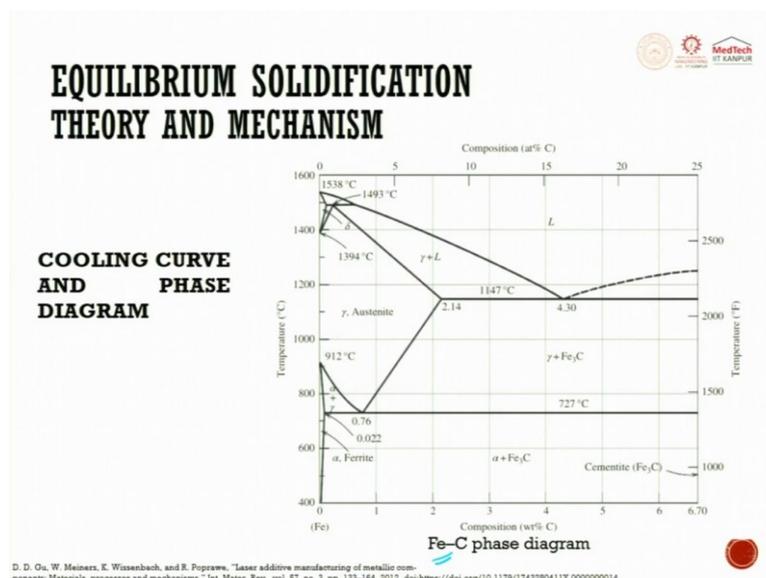
COOLING CURVE AND PHASE DIAGRAM

- This region of the curve is non-linear due to the evolution of latent heat caused by the solidification process.
- The temperature from which the slope first deviates corresponds to the formation of the first solid and is called the liquidus temperature.
- The alloy solidifies below the solidus temperature.

This curve tries to talk about cooling curve to construct binary phase diagram. But in reality, you will have a ternary phase diagram which makes the life a little more complex, the cooling curve and phase diagram. This region of cooling is non-equilibrium or non-linear.

So, the region of cooling is non-linear due to the evolution of latent heat caused by the solidification process. The temperature from which the slope first deviates, corresponds to the formation of the first solid it is called as liquidus temperature the alloy solidifies below the solidus temperature.

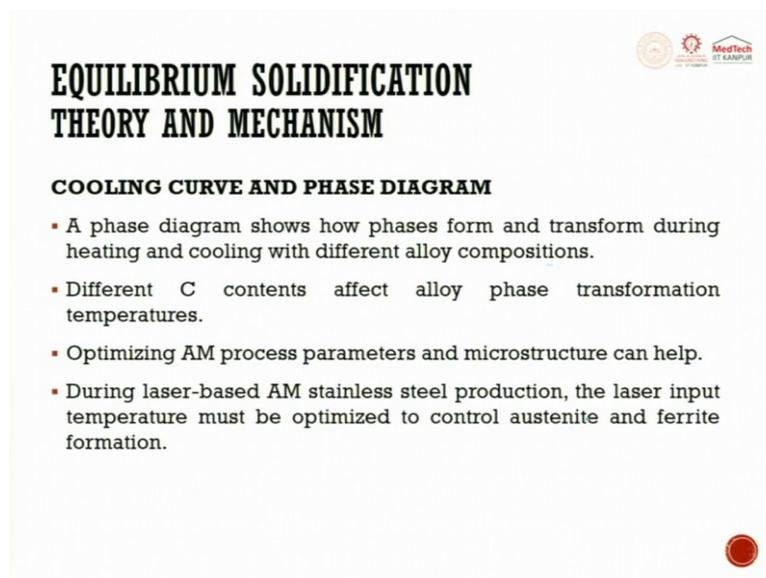
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This is a typical cooling curve and a phase diagram for steel. So, this is iron and carbide diagram, which you would have gone through in your initial days of your school or in your engineering materials. So, this is how we tried to get for iron carbide diagram at various temperatures, various compositions what are the different phases you get.

With this we can try to understand when iron and carbon are getting mixed, what will be the response, like this you will try to see for various alloys.

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**EQUILIBRIUM SOLIDIFICATION
THEORY AND MECHANISM**

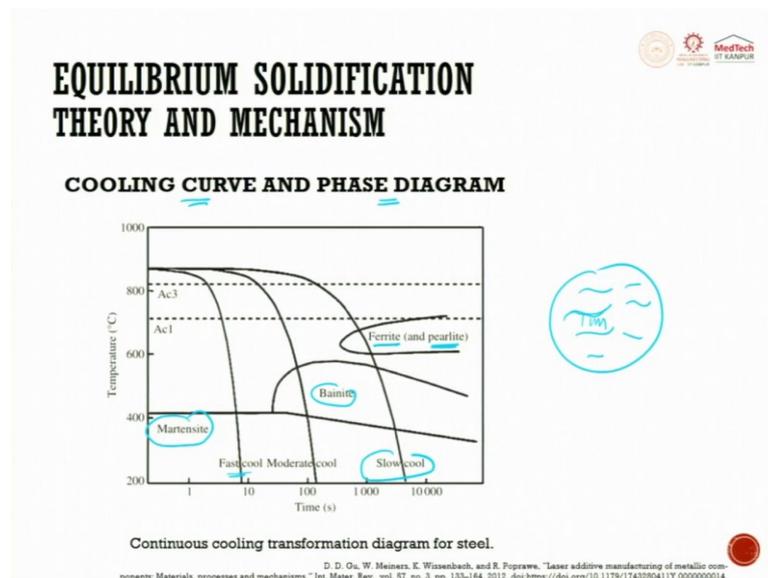
COOLING CURVE AND PHASE DIAGRAM

- A phase diagram shows how phases form and transform during heating and cooling with different alloy compositions.
- Different C contents affect alloy phase transformation temperatures.
- Optimizing AM process parameters and microstructure can help.
- During laser-based AM stainless steel production, the laser input temperature must be optimized to control austenite and ferrite formation.

The slide includes logos for MedTech and IIT Kanpur in the top right corner and a red circular logo in the bottom right corner.

A phase diagram shows how phases form and transform during heating and cooling with different alloy composition, different C content affects alloy phase transformation temperature. Optimizing additive manufacturing process parameter and microstructure can help during laser based additive manufacturing. The stainless-steel production, the laser input temperature must be optimized to control austenite and ferrite formation.

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So, these are the cooling curve which we are again taking it for steel, because it is very much available, but when you do additive manufacturing, this is still a question. A lot of people are trying to understand. Will it follow the same or will there be a deviation, if there is a deviation. How is that? So, the cooling curve and the phase diagram says that this y-axis is the temperature, x-axis is with respect to time.

So, you will have fast cooling you say, you get Martensite when you do slow or when you do moderate cooling you try to get Bainite and when you do slow cooling you get Ferrite and Pearlite in the microstructure.

So, when we were looking into the microstructure, we were looking like a fingerprint impression, I was saying these are it can be Martensite or this can be Austenite in between whatever it is. So, this we will try to talk about the cooling curve and formation of different phases with respect to temperature for steel, like this, you have different cooling curves and phase diagrams for different materials.

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EQUILIBRIUM SOLIDIFICATION THEORY AND MECHANISM

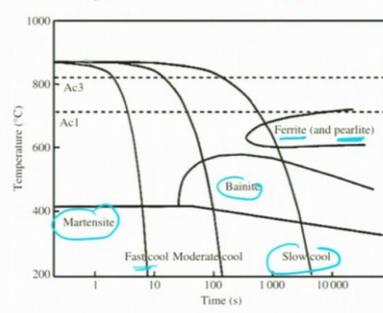
COOLING CURVE AND PHASE DIAGRAM

- The continuous cooling transformation (CCT) diagram shows the phases that form in alloys at different cooling rates.
- Each deposited layer undergoes repeated thermal cycles and has different cooling rates in AM.
- At fast cooling rates, a complete martensitic structure forms, while bainite, ferrite, and pearlite form at slower rates.
- In AM, the CCT diagram can be used to estimate phase formation when the thermal profile of each deposited layer is known.



EQUILIBRIUM SOLIDIFICATION THEORY AND MECHANISM

COOLING CURVE AND PHASE DIAGRAM



Continuous cooling transformation diagram for steel.

D. D. Gu, W. Meiners, K. Wissenbach, and R. Poprawe, "Laser additive manufacturing of metallic components: Materials, processes and mechanisms," Int. Mater. Rev., vol. 57, no. 3, pp. 133-164, 2012. doi:https://doi.org/10.1179/1742350411Y.0000000014.



The continuous cooling transformation diagram, CCT diagram tries to talk about the phase that forms in alloys at different cooling rate. So, the same if you want to do it for additive manufacturing, we might get the same response or we might not get the same response.

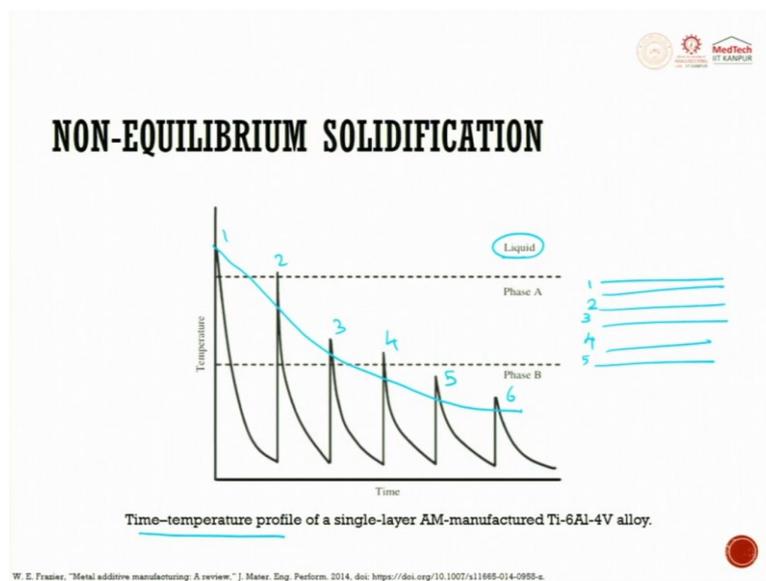
See, if we look at it going back, see the cooling rate in 10 s, you are trying to move from 800-830⁰, you are trying to come to 200⁰ within 10s, but whereas, in laser additive manufacturing, you get it within a second, tweak transformation. So, you might get completely Martensite or in Martensite also you might get some change in the microstructure, to do that.

Each deposition layer undergoes repeated thermal cycle and have different cooling rates in additive manufacturing. At fast cooling rates, a complete martensitic structure forms, while

bainite, ferrite and pearlite forms at slower cooling rates as compared to that of the faster one. In additive manufacturing, the CCT diagram can be used to estimate phase formation when the thermal profile of each deposition layer is known, which is a challenge.

This is a challenge we do not know today, because between one layer and the next layer, what will be the temperature dissemination happening? We are not able to measure even if you are using an infrared Pyrometer or thermocouple. The layer size is too small for a thermocouple to measure, when you use thermal imaging, it averages out. So, all these things make the life miserable very difficult to draw a CCT diagram for additive manufactured part.

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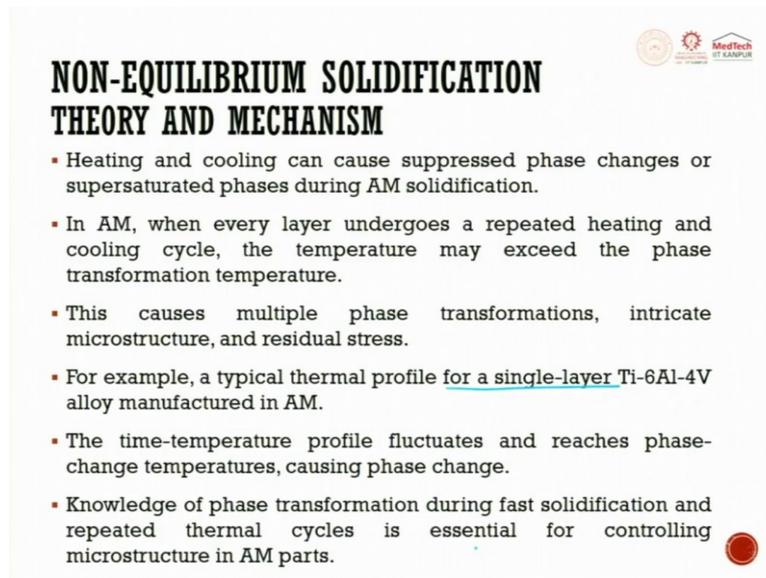


So, this is the cycle which it undergoes in temperature time profile, this is called as a thermal cycle. So, you can see in the first layer when it is moving, it will try to be in liquidus form. When the second layer is moving, this first layer is on the top, let us do it like this.

So, this is the first layer, this is the second layer. In the third layer, you see, the temperature will be like this, in the fourth layer it will be like this, in the fifth layer it will be like this and in the sixth. So, first, second, third, fourth, fifth and sixth layer.

So, you see, there is lot of thermal cycle happening. You aim that only the first layer, but second layer also gets the temperature diffused and the third layer also it is getting, and the fourth layer. Now, if you try to see this is a slope which we try to get, it is pretty interesting, and to get and validate the experiments to get with respect to timing cycle, it is a real challenge.

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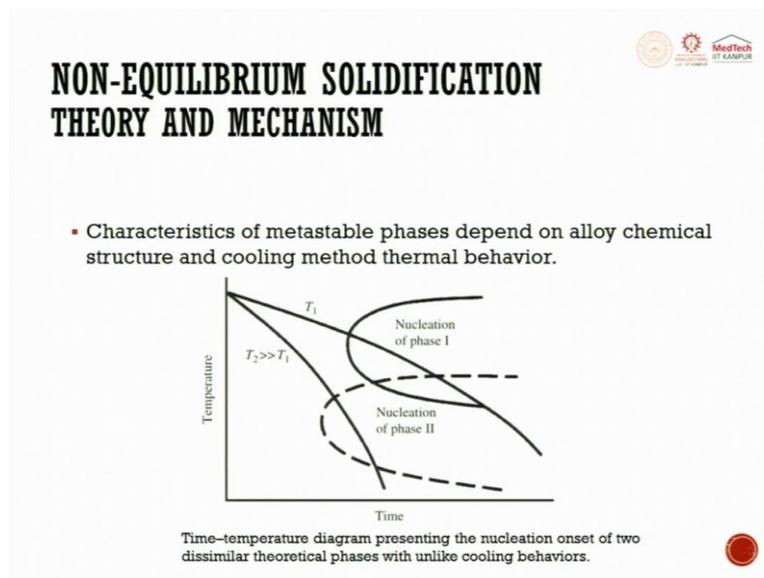
**NON-EQUILIBRIUM SOLIDIFICATION
THEORY AND MECHANISM**

- Heating and cooling can cause suppressed phase changes or supersaturated phases during AM solidification.
- In AM, when every layer undergoes a repeated heating and cooling cycle, the temperature may exceed the phase transformation temperature.
- This causes multiple phase transformations, intricate microstructure, and residual stress.
- For example, a typical thermal profile for a single-layer Ti-6Al-4V alloy manufactured in AM.
- The time-temperature profile fluctuates and reaches phase-change temperatures, causing phase change.
- Knowledge of phase transformation during fast solidification and repeated thermal cycles is essential for controlling microstructure in AM parts.

So, heating and cooling can cause suppressed phase changes or supersaturated phases during additive manufactured solidification in additive manufacturing. When every layer undergoes a repeated heating and cooling cycle, the temperature may exceed the phase transformation temperature. This causes multiple phase transformation and then intricate microstructure and residual stresses.

For example, typically thermal profiling of a single layer Ti-6Al-4V alloy is manufactured in AM for a single layer. The time-temperature profile fluctuates and reaches phase change temperature, causes phase changes to happen. Knowledge on phase transformation during faster solidification and repeated thermal cycle is essential to control the microstructure and additive manufacturing.

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Additive manufacturing process optimization involves equilibrium, non-equilibrium phase diagram thermodynamics, thermo physical quantities, laser electron beam material interaction, diffusion kinematics. So, this laser electron beam material interaction is completely different. When we are talking about laser, we talk about the surface reflection and thermal conductivity. When we talk about electron beam, we talk about the electronic structure which is there.

During rapid solidification, some phases that form under equilibrium condition may not form metastable phases. The figure on the next slide compares Ti-6Al-4V CCT diagram for standard casting and additive manufacturing, which is published in one of the renowned journals. In additive manufacturing, the alloy cooling is faster and forms a metastable martensitic structure.

So, you can see that the characteristics of metastable phase depend on alloy chemical structure and cooling method during thermal behavior. So, this is nucleation of phase 1, then it is nucleation of phase 2, this is T_2 this is T_1 , this is for titanium. So, the time-temperature diagram presents the nucleation onset of two dissimilar theoretical phase with unlike cooling behavior.

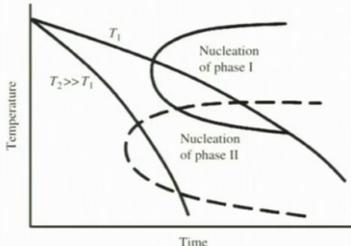
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NON-EQUILIBRIUM SOLIDIFICATION THEORY AND MECHANISM

- From the time-temperature diagram, at cooling rate T_1 , the primary phase is nucleated as phase I, whenever at faster cooling rate T_2 , ($T_2 \gg T_1$), another phase, phase II is nucleated by detouring phase I.
- Figure on the next slide shows a phase diagram with phase δ at equilibrium.
- Due to rapid cooling, a dashed metastable phase diagram may be created.

NON-EQUILIBRIUM SOLIDIFICATION THEORY AND MECHANISM

- Characteristics of metastable phases depend on alloy chemical structure and cooling method thermal behavior.

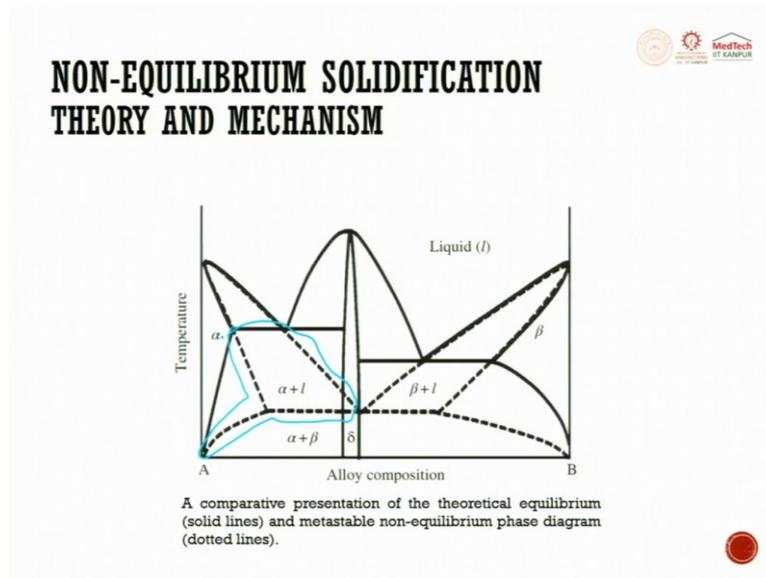


Time-temperature diagram presenting the nucleation onset of two dissimilar theoretical phases with unlike cooling behaviors.

So, from the time-temperature diagram at cooling rate T_1 , the primary phases are nucleated as phase 1, whenever, at faster cooling rate T_2 , another phase phase T_2 is nucleated by detouring phase 1. So, you do not even get into phase 1 you get into T_2 .

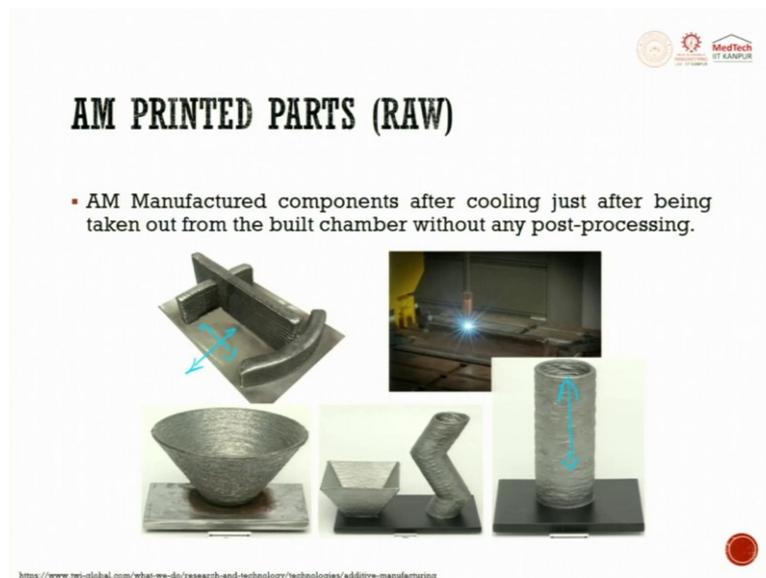
So, it is repeated thermal cycles. So, you do not even get phase 1, which is called as Taylor grain boundary engineering. Figure on the next slide shows the phase diagram with phase delta at equilibrium. Due to rapid cooling, your dashed metastable phase diagram may be created.

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So, you see here, this is a comparative presentation of theoretical equilibrium and metastable non-equilibrium phase diagram which is drawn. It makes life very complex. So, you see here these are all dotted lines.

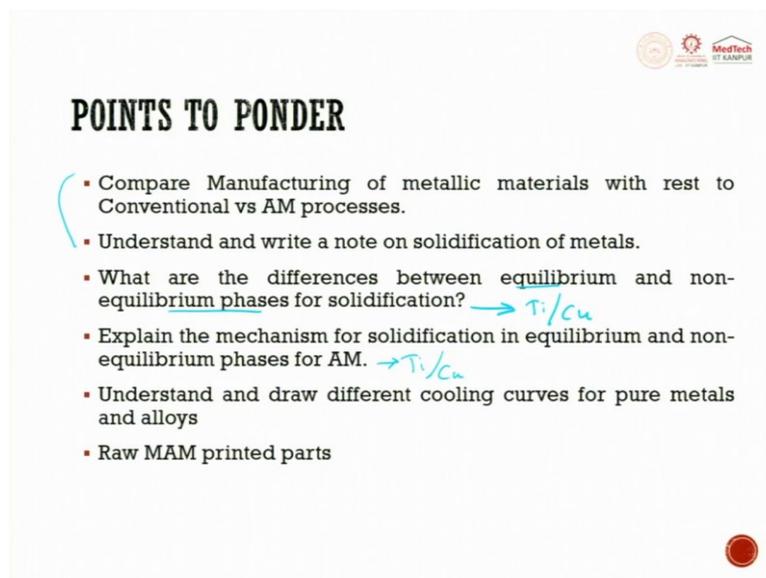
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So, it completely changes. So, some of the raw printed parts, which is made through additive manufacturing are shown. You can make a bowl, multiple parts can be made in one shot, high aspect ratio can be made in one direction, zero direction, high aspect ratio parts have been made in xy-plane.

Additive manufactured components after cooling just after being taken out from the built chamber without any post processing looks like this. You can see there is lot of undulations on the surface, which can be more because of staircase effects or it can be due to shrinkage or improper solidification. In order to avoid such things, we try to decide the orientation of the part before printing and keep in mind that it is not a single part getting printed, you can print multiple parts in one shot also.

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POINTS TO PONDER

- Compare Manufacturing of metallic materials with rest to Conventional vs AM processes.
- Understand and write a note on solidification of metals.
- What are the differences between equilibrium and non-equilibrium phases for solidification? → Ti/Cu
- Explain the mechanism for solidification in equilibrium and non-equilibrium phases for AM. → Ti/Cu
- Understand and draw different cooling curves for pure metals and alloys
- Raw MAM printed parts

So, in this lecture, what did we go through, we tried to compare manufacturing of metallic material with rest of the conventional additive manufacturing. We understood and we wrote notes on solidification, which we saw these two in the previous lecture. In this lecture, what we saw was, the difference between equilibrium and non-equilibrium phase for solidification.

Try to draw for titanium and copper, these are equilibrium and non-equilibrium phase diagrams for solidification. Try to do it by yourself as an assignment so, that you get more insight. Then, explain the mechanism of solidification in equilibrium and non-equilibrium phases. We saw for the same can you do it for titanium and copper, then understanding and draw different cooling curves for pure metal and alloy we saw and finally, we saw some of the raw printed parts, so, that you get an understanding how different it is from that of your casting.

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ASSIGNMENT

① Varying Thermal cycling graph for AM process both laser + EB separately

② Multi part printing process thro' AM what is their microstructural variation along the C-S

Cu/Ti

Bed

The assignment which is given here is try to look at varying thermal cycling graphs for AM process, both for laser and E beam separately. Multiple part printing process through AM, what is their micro structural variation along the cross section? For example, you had this is the bed, so you had a component something like this.

And you have this component. Look at some literature for it and then you try to take a cross section and see what is the difference in the microstructure if somebody has published? You can take it for again copper or titanium because copper is highly conducting. Titanium it is conducting but all the heat gets accumulated in one place.

So, because of that I am trying to always give you try to look at these two steels available in the open literature. So, let us look into these two things, varying thermal cycle whatever we said is this, this is the varying thermal cycle. So, you try to see what are the different phases you get again for copper. Thank you very much.