

**The Future of Manufacturing Business:
Role of Additive Manufacturing
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**Lecture-27
AM Materials and Metallurgy in LPBF**

Hello everyone, my name is Amit Powar and I am working as a materials application engineer in Wipro 3D. Today I am going to take a session on AM materials and metallurgy in LPBF.

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After this session user will learn about the available materials in additive manufacturing and specifically for the LPBF. The powder manufacturing processes and important powder properties which we check before certifying any powder and selection criteria of AM materials. We will go through some basic principles of metallurgy for the additive manufacturing.

We will see what type of different defects we can observe in the additive manufacturing and what we can do to avoid these defects or to mitigate this defect. So, we will see the strategies used for developing the end you have come 100% dense end use component.

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- What Influence Material Properties ?
- Metal Powders - Manufacturing
- Metal Alloys in LPBF & Wipro3D
- Metal powder characterization
- Consideration for selection of AM Materials
- Common defects in LPBF
- Mitigation Strategy
- Process for adoption of new alloy

This is the content for the today's presentation, we will check what parameters influence the material properties. Then metal powder manufacturing processes, metal alloys available in LPBF and specifically into Wipro 3D will check what are the materials available. Then metal powder characterization considerations used for selection of AM materials, common defects in LPBF and then mitigation strategies.

As I said, process for the adoption of new materials. If we are going to develop new materials for the additive manufacturing which is currently not available in the additive manufacturing domain, what processes we can take and how we can develop the material new alloy for the additive manufacturing will go through into one process development cycle.

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What Influence Material Properties ?

The diagram illustrates a six-stage process flow:

- Metal Powder** (Orange box): Chemistry, PSD, Morphology, Flow Rate, Number of Passes.
- Machine & Parameter** (Grey box): Layer Thickness, Laser Power, Laser Speed, Hatch Distance, Scan Strategy, Contour.
- Part Build** (Yellow box): Build Layout, Process Monitoring, Build Strategy.
- Thermal Processing** (Blue box): Stress Relief, HIP, Solution Treatment, Aging.
- Material Characterization** (Green box): Microstructure, Internal Defects (Porosity, lack of fusion zones etc), Location of defects, Surface characteristics.
- Material Properties** (Orange box): Physical, Tensile, Creep, Fatigue, Residual, Hardness.

So, whenever we are thinking about a new technology, we think that many people ask what are the parameters which influence the final mechanical properties or material properties. So, there in additive manufacturing there are 100 plus parameters which can affect the final mechanical properties, but in each stage we can while thinking about the any new technology, many people ask what are the important parameters which influence the final mechanical properties or material properties?

In additive manufacturing there are 100 plus parameters which can affect the influence the material properties. But we have identified few important parameters in each step of manufacturing. So, we going through into the details of this first stage is the metal powder.

In metal powder chemistry of the there are 5 important parameters of metal powder which can influence the properties. One first thing is a chemistry, as we know the chemical composition of steel is of a great importance and since it determines the potential mechanical properties of finished product and controls the degree of corrosion resistance and weldability of the material.

It should be in the specified limit of specification for that particular matter, for particular material. Second one is the particle size distribution. There are different particle sizes used for the different additive manufacturing processes, you may have seen the different additive manufacturers in the first two sessions. {article size for the MIM and binder jetting is a different particle size for the LPBF is different and particle size for the EBM is different. So, we will go into the size details into the next few slides.

But for consistent properties in each build and to ensure the repeatability of the properties it is very important that chemistry and particle size distribution results should be in the similar range. But however, if two chemistry and particle size distribution are same the third parameter which is morphology or flowability which comes into the picture that I have got picture.

Even though if the chemistry and particle size are close match it may possible property variation may come and that is because of the difference in the morphology of the powder. There are different atomization techniques for the powder manufacturing which results into different morphologies. Plasma atomization can give highly spherical particles and results in

a good flowability and packing density as compared to the gas atomizer powders. Review and then next is reuse.

Number of reuses also affects the material properties. So, number of users should be tracked after every build powder quality can degrade due to contamination. But only using one horizon powder is not economically viable. So, a well-structured characterization procedure combining with many existing techniques is proposed for determining the change in the morphology in each stage.

So, composition, flowability and number of uses can be decided. ONH analysis which is a part of chemistry that is ONH means oxygen nitrogen and hydrogen content. So, these are the non-metallic impurities extremely reactive with the metals like Titanium, Aluminum and Magnesium. Uptake of these impurities can happen at each process step beginning with the powder production, its storage the condition of the powder bed in the machine and the additive manufacturing process itself.

So, tracking of this ONH content is very important. We will go to the next manufacturing step and that is machine and parameter that is additive manufacturing. Machine and parameters. In this the layer thickness that is the depth of the each newly powder layer to be melted, then laser power the total energy emitted by the laser per unit time.

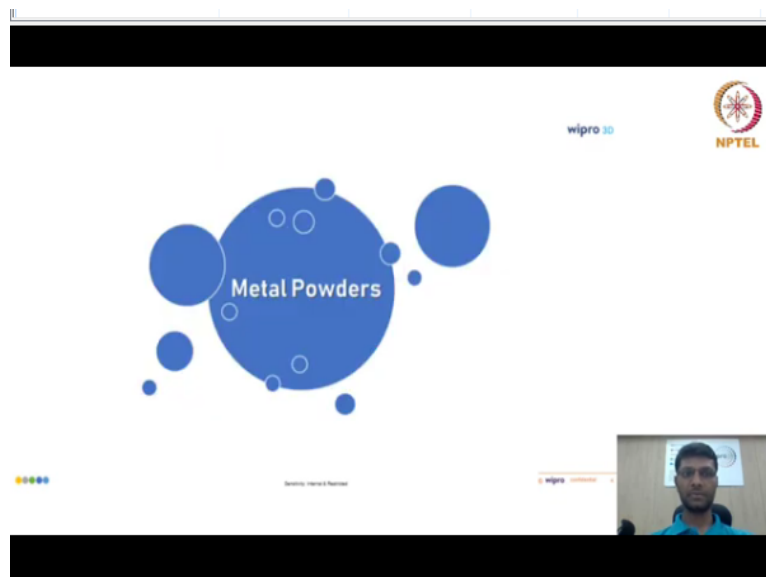
Then the scanning velocity laser speed that is the speed at which the spot is moving across the powder when along a scan vector. This is a defined by a point distance and exposure time on a modular laser system. Then hatch distance, the spacing between the neighboring scan vectors which is designed to allow a certain degree of re-melting of the previous well track to ensure the coverage of the region to be melted.

Then the scan strategy, there are different scan strategies that also affect your material properties and the contour which can affect the surface finish of the final product. Going into the next manufacturing step that is part build and where the build layout process monitoring tools used and the build strategy itself can affect the properties. Then comes the thermal processing where we are doing different thermal processing on the components manufactured through AM root.

That is stress relief hot isostatic pressing, HIP and then solution treatment and aging. So, these are also affect the final material properties. Then material characterization, microstructure study internal defects and the size and location both of that internal defect can affect. Surface characteristics which can affect your fatigue properties.

Next is the material property, what are material properties will check that is physical properties of the material, tensile properties, creep, fatigue and fracture toughness. So, all these parameters can affect the material properties and needs to be given extra attention during the manufacturing processes.

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So, we will go into the details of the metal powders. The major focus in this session will be on the metal powders, their manufacturing and the testing techniques.

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Powder is manufactured through the atomization technique. So, what is this atomization process? It is the dispersion of molten metal into particles by rapidly moving a gas or liquid stream. Here you can see the simplistic schematic view of the optimization process. Left-side schematic shows the gas atomization process while right-side schematic shows the plasma atomization process.

First stage of this manufacturing process is the segregation of the raw material as per the customer requirement. Then this raw material is melted in a vacuum melting furnace and next stage is the atomization process. In this case molten metal is passed through a small nozzle to create a molten metal stream. Then this molten metal stream is broken by a jet of inert gas or water.

If air is used to break the stream it is called as a gas atomization process and if the water is used to break the stream it is called as a water atomization process. The size and shape of particle form depend on the temperature of the molten metal rate of flow, nozzle size and jet characteristics. Gas and plasma atomization usually result in a more spherical particles as compared to water atomization.

After this atomization stretch particles are collected into the collection chamber from where it is taken for the segregation according to their size classification, after that they are blended together to make in a particular size range with respect to the different additive manufacturing processes. Then the powder manufacturer will conduct a quality inspection of the manufactured powder. And finally, the powders are packed and send it to the customer.

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Manufacturing Process	Particle Size, μm	Advantages	Disadvantages	Common uses
Water Atomization	0 – 500	<ul style="list-style-type: none">High throughputRange of particle sizesOnly requires feedback in ingot form	<ul style="list-style-type: none">Post processing required to remove waterIrregular particle morphologySatellites present	Non reactive
Gas Atomization	0 – 500	<ul style="list-style-type: none">Wide range of alloys availableSuitable for reactive alloysOnly requires feedback in ingot formHigh throughput	<ul style="list-style-type: none">Satellites presentWide PSD Low yield of powder between 20–150 μm	Ni, Co, Fe, Ti (EXGA), Al
Plasma Atomization	0 – 200	<ul style="list-style-type: none">Extremely spherical particles	<ul style="list-style-type: none">Requires feedback to either be in wire form or powder form High cost	Ti (T64 most common)

Now in the next stage we will compare these all 3 different atomization processes. So, here are some advantages and disadvantages of this atomization processes. Water atomization is having high throughput and can produce the powders in a very wide range, but limiting factor is the post processing required to remove the water, irregular particle shape morphology particles, and satellite formation during the manufacturing.

Gas atomization is the process which is used widely and has a wide range of alloys available for processing. This process is suitable for reactive alloys and also having high throughput. But in this process also satellite formation can be seen. Plasma atomization is the process where we can get extremely spherical particles but the limiting factor is its cost. Cost are seen to be very high for the plasma atomizer powders.

So, here is the trade-off, if you want the good quality powders and if it is for the aerospace and defense applications you can go with the plasma atomizer powders and if the requirement is not so stringent and application is in the automotive domain then you can choose the gas atomizer powder or even water atomized powder in some cases to reduce the cost of the manufacturing.

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Metal Alloys available for metal AM

Material Base	Alloy Grade
Nickel Alloy	Inconel 718
	Hastelloy X
	Inconel 625
Iron Alloy	SS316L Stainless Steel
	17-4PH Stainless Steel
	15-5PH Stainless Steel
	M300 - Maraging Steel
Titanium Alloy	Ti-6Al-4V Grades 5 and 23 (ELI)
Aluminum Alloy	AlSi10Mg

Customized Metal Alloys by Wipro3D

Material Base	Alloy Grade
Iron based	A286 - Heat Resistant Steel
	SAF 2507 Super Duplex Stainless Steel
For development based on customer requirement_ with joint development project	
In Development	
Material Base	Alloy Grade
Nickel based	CM247 LC

This is the material portfolio available at Wipro 3D for processing under the LPBF. We have Inconel 718, Hastelloy x and Inconel 625 in nickel base alloys. We have SS316L, 17-4PH, 15-5PH, and Maraging steel M300 in iron-based alloys. In Titanium we have Ti6L4V grade 5 and grade 23 both and we can ELI is the extra low international element grade which is used in the medical field.

In the aluminum we have ALSI 10 mg and so there are current developments going on the ALSI 7mg. Also we are working on the custom process parameter development for new alloys for which the parameters are not available from the machine manufacturer. Until now we have developed A286 grade also called as a HRS heat resistance steel and super duplex stainless steel.

Now we are working on the development of nickel-based alloy CM247. I know there might be a lot of questions on the customized parameter development, for better understanding of this development we will be going through the development cycle of the one alloy at the end of this presentation.

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Metal Powders Properties – Chemical Composition

Chemical Analysis Techniques for Metallic Powders

- Inductively coupled plasma (ICP)
- Inert gas fusion
- Wet Chemical Analysis

ONH Content: Oxygen, Nitrogen and Hydrogen are the interstitial, impurity elements present in chemical composition of various alloys. These impurities extremely reactive with metals like Ti, Al which can degrade the mechanical properties if present above their maximum prescribed limits. These concentrations should be monitored regularly to prevent usage of contaminated powder.

- Oxygen in steel causes brittleness, Hydrogen in steels causes embrittlement.
- Nitrogen decreases ductility in Steels, brittleness in Titanium
- Uptake of these impurities can happen at each process step: beginning with the powder production, its storage, the conditioning of the powder bed in the machine, the additive manufacturing process itself, followed by powder recycling steps.

Test	ONH Analyzer
Model	Ultra ONH
Specification	Respective to material
Range of Detection (in μgms sample)	
	✓ Oxygen: 0.1 ppm – 2 %
	✓ Nitrogen: 0.1 ppm – 2 %
	✓ Hydrogen: 0.01 ppm – 1,000 ppm


Going into the details of the metal powder properties as I said earlier chemistry is very important. Mechanical properties and other properties are also dependent on this. We can check chemical composition of the powder by inductively couple plasma method or weight chemical analysis. ONH that is oxygen nitrogen and hydrogen content is very important in the chemistry and needs to be tracked with every bed.

These are the international impurity elements present in the chemical composition of various alloys. These impurities extremely reactivate the metals like Titanium and Aluminum which can degrade the mechanical properties if present above their maximum prescribed limit. These concentrations should be monitored regularly to prevent uses of contaminated powder. So, what happens if they go above the prescribed limit?

Oxygen in steel causes brittleness, hydrogen in steel causes embrittlement, nitrogen decreases ductility in steel and embrittlement in the Titanium and uptake of these impurities can happen at any stage during handling, beginning with the powder production its storage, the conditioning of the powder bed then yeah the additive manufacturing process itself followed by a powder recycling steps.

So, this ONH content should be monitored after every build and this will be the limiting factor for your powder reuse. This is ONH analyzer we are using at Wipro 3D which is from the ultra ONH and we are monitoring the ONH content after every build.

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Metal Powders Properties – Particle Size Distribution

The percentage by weight, or by number, of each fraction into which a powder sample has been classified with respect to sieve number or micron.

Particle Size in Different AM Processes

- Laser Powder Bed Fusion → 15 to 63µm
- Electron Beam Melting → 45 to 106µm
- Metal Injection Molding → <38µm

Test methods for PSD in AM

- Laser Diffraction
- Sieve analysis

Effect in Process **Effect in Product**

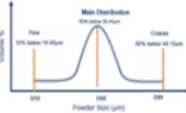
Coarser Particle Size

- More surface roughness
- low density
- more porosity

Influence: Minimum Thickness spread

Finer Particle Size:


- more cohesive
- less flow filling
- issues with laser interaction



Max Distribution
Minimum Inflow

Free 90% below 10 µm Coarse 90% above 40 µm

Particle Size (µm)




	Limit (µm)	Result 1 (µm)	Result 2 (µm)
D10%	20x5	19.00	19.35
D50%	30x5	30.40	30.50
D90%	50x5	48.15	47.50

Test PSD: Sieve Analysis

Model Sinc Sifter

Specification ASTM B214

Range <40µm



Moving to the next slide of the particle size distribution PSD is the percentage by weight or by number of each fraction into which powder sample has been classified with respect to the sieve number or micron. Different AM processes requires different powder size, laser powder bed fusion requires the powder size in the range of 15 to 63 micron. EBM requires the powder size in the range of 45 to 106 micron and metal injection molding requires the powder in a smaller size and below 38 micron. DD has its own requirements.

So, you need to be careful while choosing or ordering the powder. For testing there are two different methods acceptable as per the ASTM standard. One is the laser diffraction and other is a sieve analysis. The equipment shown here on the right side is for the sieve analysis.

Nowadays methods based on the image analysis are also available. The results of the laser diffraction are very useful. Here is the example if you look at the graph, the shape of the graph is always a bell shaped. You will get the micron values of D10, D50 and D90. What does this D10, D50 and D90 shows? If D10 is mentioned as a 20 micron, it means only 10% of powder is below 20 microns and if D50 is mentioned as a 30 micron, it means 50% of powder is below 30 micron and 50% of powder is above 30 micron. In the same way if D90 is mentioned as a 50 micron then only 10% of powder is above the 50 microns.

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Metal Powders Properties – Morphology

Plasma Atomization Gas Atomization

What we want ?

- High degree of sphericity
- controlled particle size
- minimal satellite content
- limited open porosity (or gas inclusion)

Results into:

- Excellent flowability
- high density (bulk and tapped)
- Perfect reproducibility

Powder defects - Scanning Electron Microscopy

Open Porosity Split Cap Elongated Satellites & Agglomeration Irregular Broken

Satellites are formed when the finer solidified particles stick to the molten or semi-molten surface of the coarser ones as a result of the in-flight collisions before the solidification of the coarser molten droplets.

Going to the next slide which is on a powder morphology it is very important that along with the chemistry and particle size distribution particles shall have good powder morphology. Here the powder morphology of two different manufacturing processes is shown. As mentioned earlier plasma atomization will produce powders with a good spherical morphology. In case of gas atomization, satellite formation and agglomeration can be seen.


This is images for the some of the defects is also shown here. So, this is open porosity some gas will get trapped during the powder manufacturing. This will prevent the part from getting full density. The next one is the split cap and some powder particles will be elongated and this can be seen majorly in the Aluminum powder. This next one is a satellite formation and agglomeration.


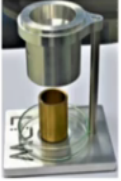

This is more common effect in powders. Satellites are formed when the finest solidified particles stick to the molten or semi-molten surface of the coarse one as a result of in-flight collision before the solidification of coarse molten droplets. Also, irregular shaped powder particles can be seen along with some broken powder particles.


Finally concluding this slide what we want is the high degree of sphericity, control particle size, minimal satellite count, and limited open porosity which results into excellent flowability, high density and perfect layer reproducibility. Next important test in the powdered test is the flowability test.

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Metal Powders Properties – Flow Rate, Apparent & Tap Density

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Time required for a powder sample of standard weight to flow through an orifice in a standard instrument	Mass of a unit volume of powder, usually expressed in grams, determined by a specified method	Density of the powder in a container that has been tapped under specified conditions
		
Test: Flow Rate Model: LPW Powder Flow Specification: ASTM B213/B214 Powder Type: Free & Non-Free Flowing	Test: Apparent Density Model: LPW Powder Flow Specification: ASTM B213/B214 Powder Type: Free & Non-Free Flowing	Test: Tap Density Model: LAB India Specification: ASTM B527



So, in this test time required for powder sample of standard weight to flow through an orifice in a standard instrument is recoater. This test is very simple and it gives good indication of the powder quality. Weigh 50 gram of powder, this quantity depends upon the metal order and pour into flow panel by static or dynamic method. Start the timing device when the powder discharges from the orifice.

And stop timer when the last powder exists the orifice. Report time for the powder to flow, shorter time reported for this test means that the powder flows more easily. Next test is the apparent density. It is a mass of unit volume of powder usually expressed in a gram per centimeter cube, determined by specified method. Here same equipment is used. This is also a quick cost effective and standardized method of characterizing the volume occupied by a given mass of metal powder when poured freely into calibrated container.

Apparent density is influenced by a powder shape, size and size distribution. If a powder sample results in a lower apparent density which is lower than the previous sample of same material it may suggest that the particle spacing has increased. Apparent densities calculated by weighing the mass of powder required to fill the volume calibrated apparent density cup.

Next test is the tap density. It is a density of powder in a container that has been tapped under specified conditions. Based on the apparent density choose the cylinder of for powder to fill, choose the number of tabs or minutes and then start the test. Note the reading at the initial of the test and the final values after the test and calculate the tap density.

Tap density values are higher for more regularly shaped particles. If the particle shape is sphere you will get a good tap density as compared to the irregular shaped particles, such as needles. A high relative tap density values should be helpful for powder spreadability and uniformity during the metal AM process. This is all about the powder manufacturing and testing. We will connect in the next session and we will go into the details of LPBF process from materials point of view. Thank you for attending the session.