


Powder Metallurgy
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Lecture – 09
Comparison of Atomization techniques


So, lecture series on Powder Metallurgy. So, far we have been talking about powder fabrication and we have discussed about different methods of powder fabrication.

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Powder Fabrication


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- The fabrication method decides the characteristics of the powder.
- All materials can be made into powder and the method selected for fabricating depends on the specific material properties.
- Primarily four fabrication methods are used
 - Mechanical ✓
 - Electrolytic ✓
 - Chemical ✓
 - Atomization ✓



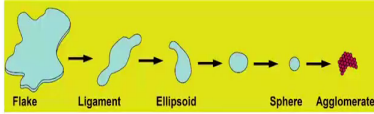
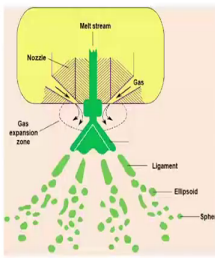


And, we have talked about them in great details about all of these techniques which are listed here as you could see. And, in the recent classes, we have been covering this particular topic atomization for making metal powders in large scale. And in that, we have already discussed about the basic principle of this process.

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Physics of atomization

- The expanding gas around the molten metal stream causes huge depressurization and disruption of the melt stream.
- This causes the melt stream to spread into a hollow cone after exiting from the nozzle.
- The thin cone is unstable due to high surface area to volume ratio.
- The liquid continues to shear this way due to acceleration forces from the gas. Sufficient superheat should be present to prevent premature solidification.
- The disintegrated melt gradually reduces to spherical particles going through some intermediate shapes.



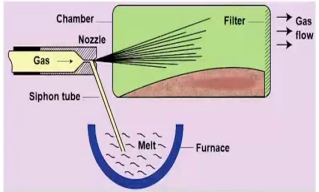


And also, the physics behind it as to how it happens you know, what are the different aspects of this technique? And, so on.

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Gas Atomization

- Air, N, He or Ar gas is used to breakup the molten metal stream.
- The liquid metal stream is disintegrated by rapid gas expansion out of a nozzle.
- Low temperature atomizers have horizontal design.
- The high velocity gas emerging from a nozzle creates a siphon and pulls the molten metal into the gas expansion zone.
- High velocity of the gas breaks up the molten stream producing a fine spray of droplets.
- The droplets lose heat during flight and solidify as powder in the collection chamber.

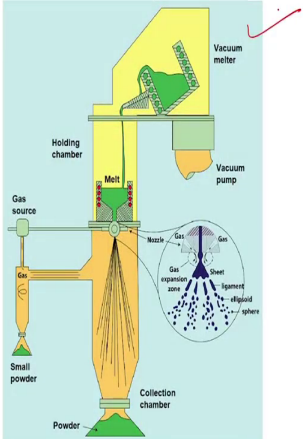




And then, we started talking about the different methods or different kind of equipment, which are used for atomization, for the process of atomization; like, the gas atomization process, we have discussed and in that, we have seen two kinds of atomization

equipment in case of gas atomization and these were the horizontal one as you can see over here.

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Vertical atomization contd....

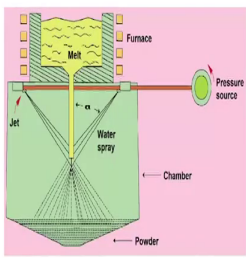






And, the vertical one that you can see in this particular image.

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Water Atomization

- Basic design of water atomizer - identical to gas atomizer.
- Spray or Jet - high-pressure water stream through nozzles far away from melt nozzle.
- The impingement zone - much lower than that in gas atomizers (due to higher cooling)
- Production rate is very high about 400 kg/min.
- Commonly used to produce powders of noble metals and of low carbon and stainless steels.
- Synthetic oils are other non-reactive liquids can be used for better control on particle size and oxidation.

And then, we also talked about the other atomization techniques like water atomization.

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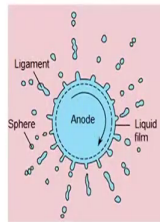
Other Atomization Techniques

Centrifugal Atomization

- Mechanical force from rotation forms the droplets from the melt. Melting can be achieved by electric arc, plasma or laser.
- The liquid spreads to form a thin film and the flow quickly becomes unstable due to high shear stresses forming ligaments. Centrifugal force ejects the perturbed ligaments at high speed resulting in the droplets.
- Low rpm, low melting rate – particles form directly at the anode lip. High melting rate – ligaments turn to droplets.
- Can be done in vacuum (for reactive metals), amorphous metal powder can be produced.

$$D = (A/\omega) \sqrt{\gamma/\rho_m R}$$

D – Particle size,
A – constant
 ω – angular velocity
R – electrode radius
 ρ_m – density of liquid



And, in the previous two classes, we spoke about the centrifugal atomization if you remember.

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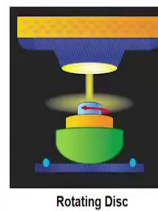
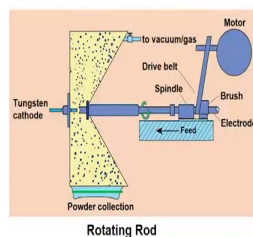
Centrifugal Atomizer



To control particle size and handle difficult to melt reactive metals.

Rotating Rod: Simultaneously melt and atomize a rotating feed stock bar. The bar is rotated at high speed up to 10,000 rpm.

Rotating Disc: Melt is poured on to a rotating water cooled disc. Disc rotation speed is in between 2000 to 3000 rpm. Finer powder compared to rod due to radial and azimuthal velocities. Disc also provides rapid cooling.



And here, we talked about two kinds of centrifugal atomizer; one is the rotating rod and the other one is the rotating disc.

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Centrifugal Atomization



- Rotational speed – 1000 to 50000 rpm. Melt rate – 10^{-7} m³/s. Anode dia – 2 to 5 cm.
- Advantages: Clean powder, spherical shape (better packing density and easy flow), uniform particle size, low contamination from crucible.
- Disadvantages: Low production rate, high equipment and processing cost, tungsten contamination (from cathode).



And then, we have also discussed about the different attributes of this centrifugal atomization process. So, now that we have covered different types of atomization processes, it is time for us to have a look as to what kind of process would give rise to what kind of powder particle and so on. So, let us have a look about all these atomization techniques back again and then see, what kind of powders do they produce?

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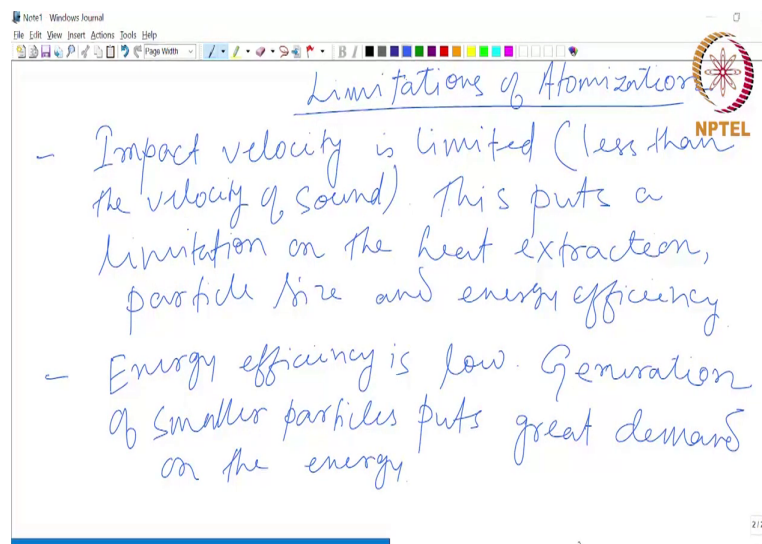
Process	Size range µm	particle shape	Cost-
Gas atomization	15-300	rounded, spherical	Moderate
Water atomization	5-800	irregular	low
Rotating electrode	200-600	nodular Spherical	high
Rotating disc	50-300	Spherical	high

So, let us have a look at the comparison between different atomization techniques. So, let us make a table like this, where we have this different processes, then the size of powder that they will generate, the particle shape and the cost. So, starting with gas atomization process; here, the size range that you can get is from 15 to 300 microns. As far as the particle shape is concerned, it is rounded and also spherical and cost is moderate.

Then, we had talked about water atomization process. And in this case, the size range is 5 to 800 microns, particle shape is irregular and nodular and cost is low. Then, we covered the centrifugal atomization in which we had rotating electrode or rotating rod and then, we also had a rotating disc. So, let us look at the rotating electrode first.

This case, the size range is 200 to 600 microns; particles are spherical in shape and cost is high. And finally, for the rotating disc process, the size range is 50 to 300 microns; particles are spherical and here also, the cost is high. So, these are the different attributes of different kinds of atomization techniques that we talked about in terms of the characteristics of the particles and also, the cost of the process.

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And now finally, let us have a look at the limitations of this atomization process. As we have seen in this process, there is an impact from a gas jet or from a water jet which breaks down the molten metal into fine droplets to generate the powder particles. And, as

far as the impact velocity is concerned that is limited in this case. It is less than the velocity of sound and this limitation on the impact velocity also puts some kind of limitation upon the heat extraction, particle size and the energy efficiency of the process.

In fact, the energy efficiency is low for the atomization process. And, as the particle size decreases, it puts more demand on the energy. Smaller the particles that you need, higher will be the energy requirement for this process and if you remember, we talked about the parameter, which has an influence on the particle size and that was the weber number.

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Handwritten notes on a whiteboard:

Weber number, We

$$D = K d_m \left(1 + \frac{\rho_m}{\rho_g}\right) \frac{\eta_m}{\eta_g We}$$

$$We = \frac{\rho_g v_g^2 d_L}{2\gamma}$$

- We is in the range of $10^3 - 10^4$ for fine particles. High We will require high gas density and velocity. rocket nozzle technology.

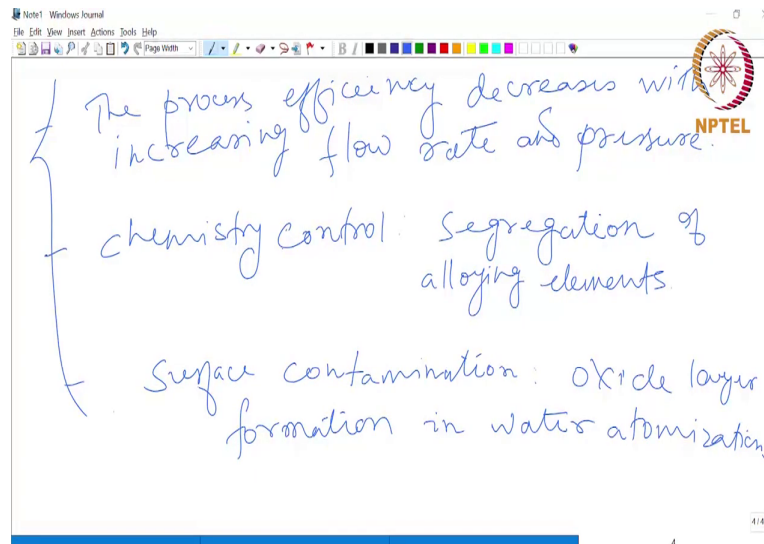
The particle size D is given by this relationship if you remember. The details of this different parameters have been already given before. Now, this weber number is given as, where ρ_g and v_g are the density and the velocity of the gas respectively; d_L , if you remember that is the ligament diameter and γ is the surface energy.

So, since the velocity is limited in this case as we discussed just now, the weber number also is low. And, it is in the range of 10^3 to 10^4 for fine particles and as you can again see from here, a high weber number, would require high gas velocity as well and high gas density also .

In fact, to generate high weber number for producing finer and finer particles, the normal nozzles which are used cannot be enough. And, this technology called rocket nozzle

technology may have to be used to generate high velocity and therefore, high weber number. So, the weber number also puts a limitation on the process.

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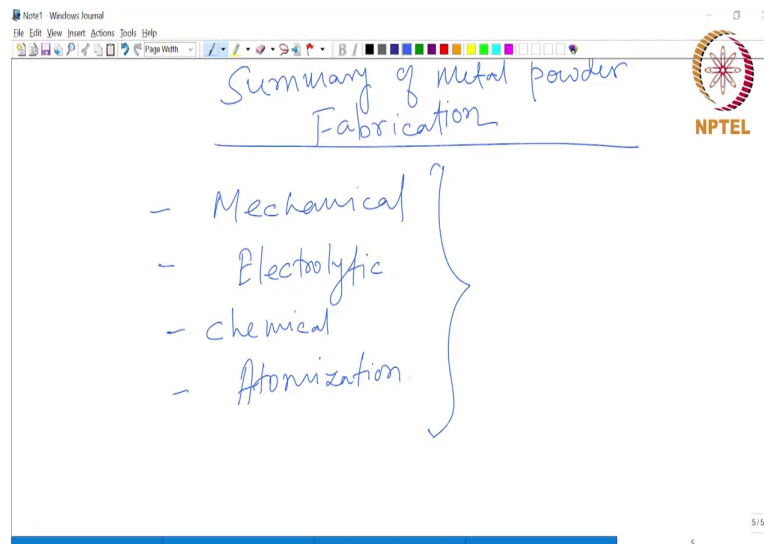
The process efficiency decreases with increasing flow rate and pressure. Chemistry control is also a challenge at times and segregation of alloying elements is an issue, when you are making alloyed powders by this process. And of course, we have seen in case of some of the atomization techniques like for example, the water atomization process, there is a problem of surface contamination that happens due to the oxidation of the powder during the process or formation of the oxide layer that happens in water atomization.

So, these are some of the limitations of the atomization process, but nevertheless this still remains one of the most commonly used methods for making metal powders in large quantities. So, this will kind of bring us to the different processes of atomization that we had lined up for this particular topic.

As far as the process is concerned, we can conclude it over here; but, there is one more thing, that we need to discuss before we actually close this topic and that has to be discussed in little more detail and we need to understand some concepts and some phenomena that happens during the process. So, we will take that up separately and

probably discuss that in a separate class. But for today, before we close this, let us try and summarize as to what we have learned so far about the different fabrication routes for making metal powders.

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The image shows a screenshot of a Windows Journal application window. The title bar reads "Note1 - Windows Journal". The menu bar includes "File", "Edit", "View", "Insert", "Actions", "Tools", and "Help". The toolbar contains various drawing and editing tools. The main content area has the handwritten title "Summary of metal powder Fabrication" underlined. Below the title is a list of four methods: "- Mechanical", "- Electrolytic", "- chemical", and "- Atomization". A large right-facing curly bracket groups these four items. In the top right corner of the journal window, there is a circular logo with a star-like pattern and the text "NPTEL" below it. At the bottom right of the journal window, the page number "5" and "5/5" are visible.


Essentially, we talked about four methods; which are mechanical, electrolytic; chemical and then finally, atomization, right. So, this is what we had and we have discussed all these processes in detail and learned about them in terms of their different attributes, process variables and so on.

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Mechanical fabrication

- Impaction
- Attritioning
- Shearing
- Compression

top down approach



Impaction - Rapid, instantaneous delivery of blows resulting in cracking and fragmentation of the material.


Attritioning - Size reduction by rubbing action

Shearing - Cleavage type of fracture by cutting. Powders formed by this method are coarse and not often used in P/M.

Compression - Breaking down a sufficiently brittle material into coarse powder by compressive forces.

The methods are often combined to make metal powders.

Powders produced by mechanical methods are typically irregular in shape.



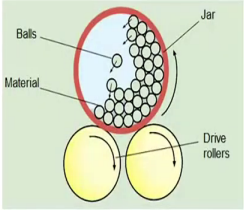


Starting from mechanical fabrication, we have seen how this technique is carried out and you know what kind of mechanical forces are used for making metal powders and we have also discussed that mechanical fabrication is a top down approach, wherein you start with the bulk material and break it down to smaller fragments to generate metal powders. And, in order to fragment it; in this case of mechanical fabrication, a mechanical force in terms of one of these processes is used to generate the powders.

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Milling

- Mechanical impaction using hard balls resulting in fragmentation.
- Jar mill or Ball mill - Container (Jar or vial) filled with balls and the material and rotated.
- Balls collide continuously with the material crushing it to powder.
- Rotation speed should be optimum - **Fast enough** to carry the balls to the top from where they fall on the bed of material. **Too slow speed** will result in balls rolling back. **Too fast** will result centrifugal force and balls will stick to the container wall.





And, we have discussed what kind of you know mechanical process or mechanical forces are used like milling, attrition and other impaction techniques like compressive crushing, cold streaming and so on.

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Other Impaction Techniques



- Compressive crushing down to 1 mm and below using WC blades.
- Cold stream method – Acceleration of coarse powders by a gas jet to collide with a cold target. ~10 μm , rounded but irregular shape powder.
- Applications: SS powders for filters, spray powders (for coating).
- Self-impact attritioning – variation of cold stream, two streams of same powder are directed at each other.



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Machining

- Huge amount of machining chips produced in the metalworking industry provide an abundant source of powder.
- Coarse powder is generated by grinding the chips.
- Easy and economical but lacks control on powder characteristics, contaminations, oxygen, oil and other metals.
- Powder is too coarse and irregular, requires further milling.
- Not preferred method, insufficient and slow
- Useful for small scale production
- Applications – High carbon steels, some dental amalgam powders.



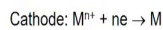
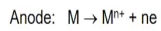
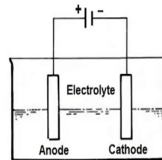
Also, we talked about making of metal powders by using machining chips.

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Electrolytic Techniques



- Precipitation of powders at the cathode in an electrolytic cell.
- Used for high purity metal powders – Fe, Cu, Zn, Mg, Ag, Pd, Ti, Ta, Nb
- Powder particles are spongy or dendritic in shape.
- Porous powders are favored by high current densities, low ion concentrations, acidic bath and colloidal additions.



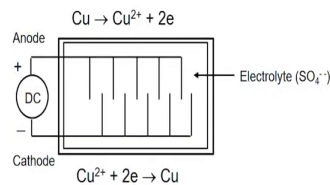
And then, we had discussed about the electrolytic technique in which, the metal powders are generated using an electrolytic cell, where the anode is the metal which is to be generated and then, through this electrolytic process, it is deposited on the cathode and from there, the powder is collected.

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Industrial production



- Dissolving impure metal in an electrolyte and depositing the metal ions on the cathode.
- Industrial cells are 3 – 4 m long, 1 m wide and 1 m deep and use a set of 20 – 40 anodes and cathodes connected in parallel to the power supply.
- The cell voltage is typically 1 – 2 V and current density is high, in the range of 300 – 4000 A/m².
- The container is sloped for easy collection of the deposited powder.
- Collected powder is washed, dried, ground into fine powder and annealed to remove any strain hardening.





And, on an industrial scale it is actually done in a big tank which is like 3 to 4 meter long and it carries a set of a series of anode and cathodes to generate the metal powders in large quantities from a particular batch, from a single batch.

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Industrial production

Conditions for Cu powder production

Cu concentration in electrolyte	30g/l
H ₂ SO ₄ concentration in electrolyte	150 to 250 g/l
Anodic current density	600 to 4000 A/m ²
Bath temperature	40 to 60 °C
Bath voltage	1 to 2 V
Electrode	88% Pb – 12% Sb





And then, we also talked about a particular metal production by this process taking an example of copper.

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Chemical Fabrication

- > Gas-Solid reduction reaction
- > Thermal decomposition
- > Precipitation from a liquid
- > Precipitation from a gas
- > Solid-Solid reactive synthesis




And then, we also discussed about chemical fabrication, where there are different routes again in this category; starting from gas solid reduction reaction.

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Thermal decomposition


➤ Another approach of thermal decomposition is to vaporize the raw material directly in a low Argon pressure atmosphere. 50-100 nm particles, faceted or cubic, high purity.



Flow chart of Carbonyl process

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graph TD
    A[Preparation of material for the process] --> B[Synthesis of carbonyl]
    B --> C[Residue for recycling]
    B --> D[Extraction, purification and separation of carbonyl]
    D --> E[By products]
    D --> F[Decomposition of carbonyl]
    F --> G[Carbon monoxide]
    F --> H[Metal powder]
    C --> A
    
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Then, we had thermal decomposition, where we used a process called carbonyl to generate metal powders by thermal decomposition of this volatile compound of a metal.

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Precipitation from a liquid

➤ Precipitation of the metal or a metal containing precipitate from the metal salt solution.

➤ The metal salt (metal nitrate, chloride or sulfates) is dissolved in water and the metal is precipitated by a second compound

$$2\text{AgNO}_3 (\text{aq}) + 2\text{K}_2\text{SO}_3 (\text{aq}) \rightarrow 2\text{Ag}^+ + 2\text{NO}_3^- + 4\text{K}^+ \rightarrow 2\text{Ag} (\text{s}) + \text{K}_2\text{SO}_4 (\text{aq}) + 2\text{KNO}_3 (\text{aq}) + \text{SO}_2 (\text{aq})$$

➤ The metallic ions can be also reacted with H_2 to form metallic precipitates.


➤ Powder characteristics can be controlled by controlling the process parameters.


➤ Powders are in the range of 1 μm and are of high purity. Flow and packing characteristics are poor.

➤ Composite or alloyed powders can be made. Co-precipitation technique, which uses the precursors of the constituents, is used for this purpose.

➤ One phase is used to nucleate the precipitation reaction in this method. Example, ThO_2 , TiO_2 and WC are coated with Co, Ni or Fe.

➤ The precipitation technique is also useful for making powders of reactive metals such as Zr and Ti.





And then, we talked about precipitation from a liquid wherein, you have a metal salt which is dissolved in a solution and with the help of a precipitating agent, that metal is


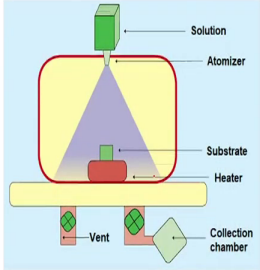
released and precipitated in the form of powders and you know with the help of this a variety of metal powders can be generated.

And, we have also seen that using the same process, it is also possible to coat a particular surface. For example, a ceramic material surface like thoria, titanium or tungsten carbide can be coated with the metal like cobalt and nickel or iron when we use the process of co precipitation, where both of these materials will be precipitated together.

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Spray pyrolysis

- > Solution containing metallic ions is sprayed into a heated chamber.
- > The solution goes through thermal decomposition (pyrolysis) in the chamber.
- > The particles generated can be deposited on a pre-heated substrate or collected in a collection chamber.
- > Inert atmosphere is needed to prevent oxidation of metal powders.
- > Rapid pyrolysis and heating can generate variety of particles shapes.



And then, we talked about this process called spray pyrolysis, wherein a spray of the metal salt solution is sent into a chamber, where it is thermally decomposed to generate the metal powders, which are collected at the end in this collection chamber. And, we have also seen that the powder which is generated inside this chamber by the thermal decomposition process can also be coated on a preheated substrate. So, this is also a process of making a coating apart from making metal powders.




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Hydrometallurgical precipitation

- The metal is dissolved to form ion complexes which are then precipitated.
- For example, CuSO_4 dissolved in aqueous solution is reacted with hydrogen at 130°C and 3 MPa to produce solid Cu. H_2SO_4 is the by product.

Refractory metals

- Ta can be released from K_2TaF_7 salt solution by sodium reduction.
- Molten sodium is added to the solution under inert atmosphere to start the reduction reaction.
- The reacted mass or cake that contains Ta is then processed through a series of aqueous leaching steps to obtain pure Ta powder.

Then, we talked about the process of hydrometallurgical precipitation, wherein the particular metal is in a compound which is dissolved in a salt solution and in this case, we took the example of tantalum which is dissolved in sodium and it is made into a cake and once, it is into the cake, the metal in this case, tantalum is released by washing and once it is obtained as powder, it is crushed followed by milling, ok.




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Precipitation from a gas

- Reactive metals can be precipitated as nano scale particles from gaseous phase.

$$\frac{1}{3} \text{Cu}_3\text{Cl}_3 (\text{g}) + \frac{1}{2} \text{H}_2 \xrightarrow{1000^\circ\text{C}} \text{Cu} (\text{s}) + \text{HCl} (\text{g})$$

- Powders form without melting or contact with a crucible.
- Spongy particles, spherical polycrystalline aggregates can also form.
- The method can be used for making composite powders or for refractory coating.

We also talked about precipitation from a gas, wherein the metal compound is in the form of a volatile gas, which is reduced by reducing gas at high temperatures to generate the metal powder. We also talked about the different attributes of the powders generated by a particular process like in this case, the powders are spongy and spherical in shape.

Similarly, we talked about the other processes also as to what kind of powder morphology and powder characteristics, they generate. If you remember for example, in the mechanical case, the powder particles are generally irregular in shape and their packing and flow characteristics are also not that great, due to that particular shape or that irregular shape.



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Solid state reaction synthesis

- > Highly alloyed or intermetallic compounds with precise stoichiometric ratio such as Fe₃Cl, NiTi or Ti₃S₅ powders can be processed by solid state reaction.
- > The process involves mixing of constituent powders in a loosely packed bed, ignition and self-propagating reaction.
- > Heat is released when the compound is formed.
- > NiAl – Equiatomic ratio of Ni and Al. Melting point = 1649 °C

$$\text{Ni (s)} + \text{Al (s)} \rightarrow \text{NiAl (s)} + \text{Heat}$$

- > Ni and Al reacts spontaneously analogous to explosion reaction between hydrogen and oxygen to produce water.
- > Uncontrolled reaction can generate high amount of heat that may be sufficient to melt NiAl.
- > If the heat is properly extracted the end product is a porous mass that can be easily converted into powder by milling.
- > Many variant of the process are in use.
- > Mostly used for ceramics powders.






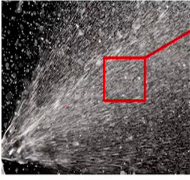
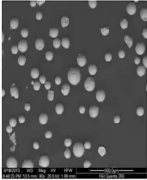
Then, we also talked about this process of reacting to solids you know to make another solid in the form of powder, but this is kind of more suitable for making intermetallic or ceramic powders rather than metal powders.


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Atomization

- Other methods – irregular shape, poor flow characteristics.
- How to improve the flow property – What shape/morphology, which process?







And finally, in last few classes, you know we have talked about this process called atomization, which is a large scale fabrication route for metal powders with production rate of as high as 400 kilogram per minute.


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Atomization

- Formation of powder from molten metal using a spray of droplets.
- Provides majority of the metal powders at rates as high as 400 kg/min.
- Attractive process because of applicability to several alloys and easy process control.
- It is a fusion based technology that provides control over melt purification and alloy chemistry.

Principle of atomization

- Disintegration of the melt into fine droplets due to surface instabilities induced either by hydrodynamic forces of the flowing stream, gas jets or mechanical, electrostatic or electromagnetic forces.
- The phenomena involves development of flow in the bulk liquid due to applied external forces, surface instabilities due to imbalance of forces at the free surface, formation of ligaments and detachments of the ligaments into droplets.







And in this case, you know what we have seen is that, the powder particles are spherical. And as a result, their flow and packing characteristics are very good. And, in this process

of atomization, we have also seen different types of atomization processes like gas atomization, then water atomization and finally, the centrifugal atomization process.

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Limitations of Atomization

- Impact velocity is limited (less than velocity of sound). This puts a limitation on heat extraction, particle size and energy efficiency.
- Energy efficiency is low. Generation of smaller powders puts great demand on the energy.
- Weber numbers in the range of $10^3 - 10^4$ for fine particles. High W_e required high gas density and velocity (rocket nozzle technology).
- The process efficiency decreases with increasing flow rate and pressure.
- Chemistry control: segregation of alloying elements.
- Surface contamination: Oxide layer formation on powder particles in water atomization.



And then, we also had a comparison between them to show you what kind of powder particles are generated by each of these atomization processes and also, what is the cost for this atomization processes. So, with this, I think we can conclude this topic of powder fabrication. And, as I said in the next class, we have one more very important aspect of powder fabrication process; particularly, with regard to these atomization process wherein, you start with a liquid stream and then, solidify that liquid into powders, right.

So, there is a solidification aspect which is involved here and we need to understand that. So, that is a very important aspect of this atomization process and that is something that we are going to take up separately, as I said before in a different class. But for today, this is all I have.

Thank you for your attention.