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Additive Manufacturing

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8th Sem 'A' sec

Assignment – ①.

① Define AM process and its classification.

Ans:- Additive Manufacturing is a process by which digital 3D design data is used to build up a component in layers by depositing material. It is also called as 3D printing, rapid prototyping layer fabrication etc..

AM is suitable to produce complex metal shape parts and functional prototypes. It is used in various sectors such as aerospace, energy, automotive, medical etc...

* Classification of AM processes:-

Classification based on :-

a) Baseline technology.
b) Type of Raw material used.
c) 2-dimensional classification method.

ⓐ Liquid polymer systems – SLA (stereo litho graphy) – photo polymerization.

ⓑ Discrete particles systems – SLS – powder bed fusion.

ⓒ Molten material systems – FDM – material extrusion.

ⓓ Solid sheet systems – LOM – Laminated object manufacturing – sheet lamination.

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→ Liquid polymer systems – Building material is in liquid state.

→ Solid systems – Building material is in solid state, which includes the shape in the form of wire, rolls, laminates and pellets.

→ Powder / discrete particles system – Building material is powder.

All powder based AM processes employ the joining / building method.

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(2) Distinguish between AM processes and CNC machining.

Ans:-

AM processes	CNC machining
• Geometrical complexity can be high.	• Geometrical complexity can't be high.
• Internal features and undercuts can be ~~easily~~ easily built.	• Internal features and under cuts are difficult to design.
• Can be built directly.	• Complex components built seperately and then assembled.
• polymer materials, wax paper laminations, composites metals and ceramics.	soft materials – medium density fibre board, some polymers etc..
• May have voids, anisotropy	• Normally homogenous.
• Multiple parts can be produced simultaneously.	• High speed CNC machines can produce parts in a few ~~fe~~ weeks. It is a multistage process involving steps such as positioning relocation of parts etc..

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• Once the design data is available, the time to produce any parts will be a matter of hours.	
• In terms of tolerance, AM processes come below CNC machining process. They are more accurate.	• In terms of tolerance, CNC machining is superior to all 3D printing process.
• They are more accurate	• They are comparitevely less accurate.
• For low volumes, additive manufacturing is more appropriate cost wise.	• For large quantities, CNC machining is more appropriate
• Complex operation	• Easy operation
• Higher	• Lower
• Additive process	• Subtractive process.

39) Briefly Explain the AM process chain.

→ Additive manufacturing involves a number of steps that move from the virtual CAD model to 3D model. The steps involved in AM are :-

1. Conceptualization :- Initial stage of design for creating a product. This gives an idea as to how the product will look and function.
2. 3D modelling :- Advanced 3D CAD modelling is a general perequisite in the additive manufacturing process and is usually the most time consuming part of the entire process chain. All AM parts need to start

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from a software model which describes the geometry

3. Conversion to STL :- To establish consistency, STL (standard triangulation language), stereolithographic file format. STL format captures surfaces of the 3D model by stitching triangles of various sizes on its surface

4. Transfer to additive Manufacturing :- Once the correct STL file is available, a series of steps are required to transfer the information to start the build. These steps starts by repairing errors within the STL file, after this, proper orientation of the 3D model with respect to the build platform is decided, orientation, geometry, densities of structures are decided and generated in 3D model.

5. STL file manipulation :- After transfer to AM, CAD model preparation starts with importing an STL file into the pre process software program. The dimensions are then modified according to a number of steps are carried out to correct errors in the file.

6. Machine setup :-

a) Machine hardware setup :- includes cleaning of build chamber, loading of powder material and process controls → such as gas pressure, flow rate, oxygen sensors etc

b) Process control :- These tasks include,
→ accept and process build files
→ Start the build.

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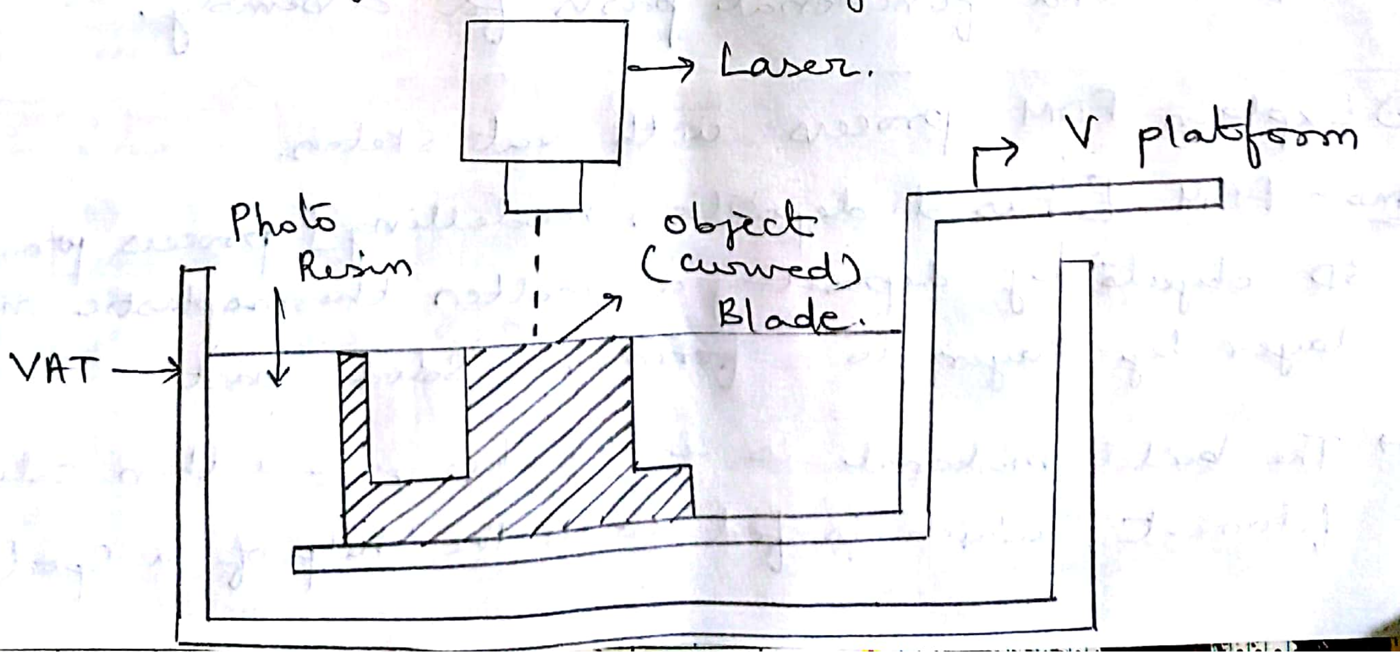
→ Interrupt and build if required.

(7) Building :- Mostly this region is fully automated. The building process may take up several hours to build depending on the size and number of parts required.

(8) Post processing :- The product once built may require an amount of additional cleaning up before they can be used. It may either contain no post operation or may contain several additional steps to change the surface, dimensions or material properties of the built part.

(4) Explain VAT photopolymerization with neat sketch :-

Ans :- VAT photo polymerization involves use of Vat of a liquid photopolymer resin, out of which the model is constructed layer by layer. An ultraviolet (UV) light is used to curve or harden the resin where required whilst a ~~platt~~ platform moves the object being made downwards after each new layer is curved.



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1. The build platform is lowered from the top of the resin VAT downwards by the layer thickness.
2. A UV light cures the resin layer by layer. The platform continues to move downwards and additional layers are built on the top of the previous.
3. Some machines use a blade which moves between the layers to provide a smooth resin base to build the next layer on.
4. After completion, the VAT is drained of resin and the object is removed.

* Advantages:-
1. Complex geometry parts can be built.
2. High quality surface finish.
3. Clear model with good resolution can be obtained.

* Applications:-
1. Manufacturing of medical models
2. Manufacturing parts for tooling and vacuum casting
3. Fit and functional parts for assembly.

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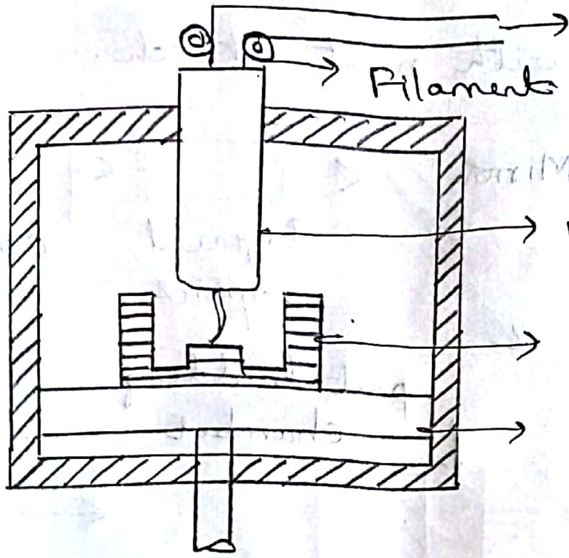
5) Explain FDM process with neat sketch.

Ans:- FDM [Fused deposition modelling] process produces 3D objects by depositing a molten thermoplastic material layer by layer to form a solid part.

→ The build material is in the form of a thin solid filament, which is fed with the help of a spool.



FDM.

→ As it reaches the head, the material is melted at 180°F by a resistance heater. This molten material is extruded through the nozzle, which gets deposited as fine layers.

→ On solidification, the desired object is achieved.

* **Advantages of FDM:-**

1. FDM parts are tougher and more durable than other techniques
2. Low cost
3. Accurate prototypes can be produced.

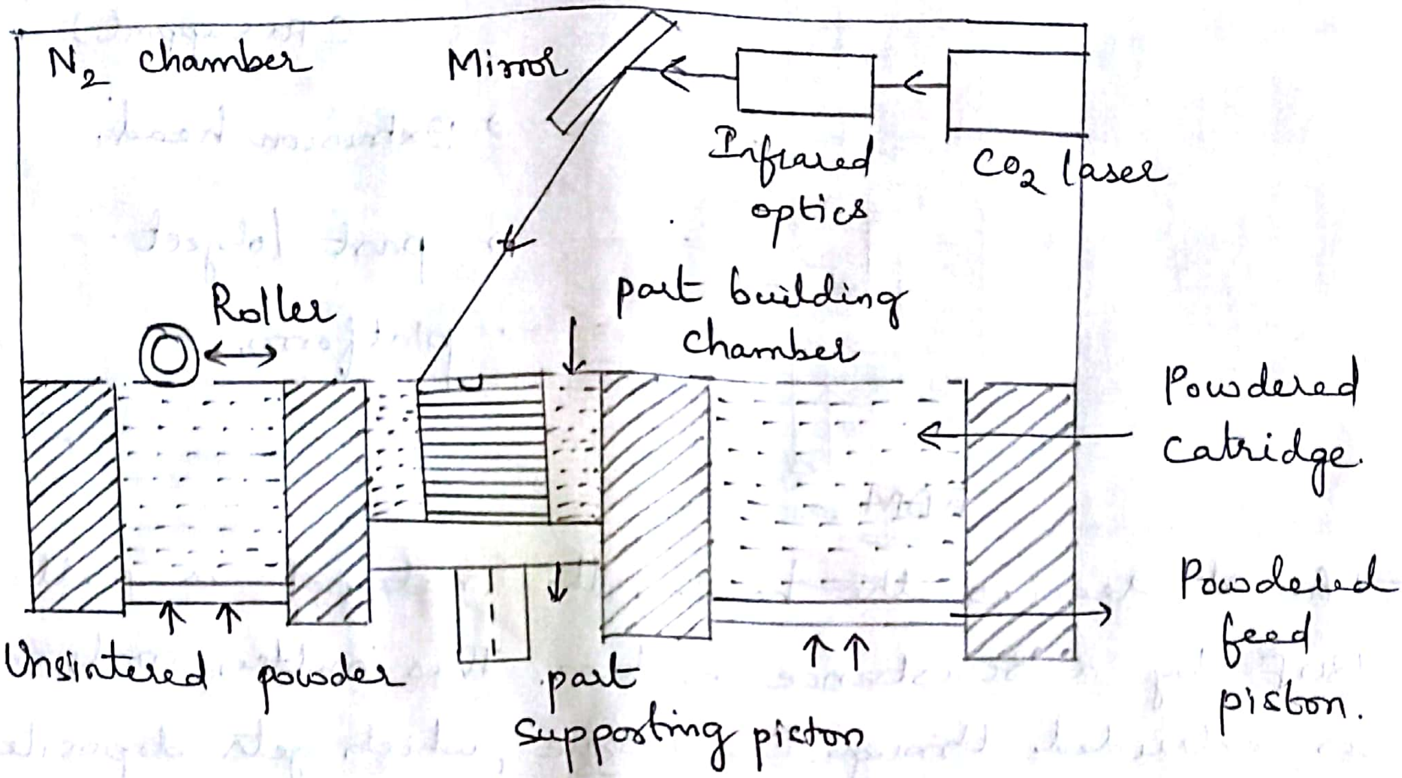
* **Disadvantages of FDM :-**

1. Difficult to produce very small features
2. Slow process for parts with wide cross-section.

* **Applications :-**

1. Medical feild
2. Quality parts with high stability.

6. Explain SLS process with neat sketch. 1CR17ME056.



→ In this, solid objects are produced layer by layer by fusing powdered thermoplastic material with the help of Infrared light beam

→ Selective laser sintering setup consists of a chamber with movable pistons.

→ When the laser beam is incident on the powdered particles, it becomes molten

→ After each cross-section is scanned, a new layer of powdered is applied on the top with the help of a roller

→ The layers are fused and the process repeats until the entire model is created.

* Advantages of SLS :-

1. wide range of materials can be used.
2. post curing is not necessary.
3. Support material can be removed easily.

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* Application of SLS :-

1. Rapid tooling applications
2. Soft tooling and short run production.

(10) Explain different challenges faced by AM processes :-

(a) Technological challenges :-

(i) Slow production speeds
(ii) Material development and inconsistencies in material preparation.
(iii) Manual post processing

(b) Software challenges

(i) Limited capabilities in data preparation and design
(ii) Quality assurance challenges
(iii) Part to part variation
(iv) Lack of Industry wide standards.

(c) Workforce challenges.

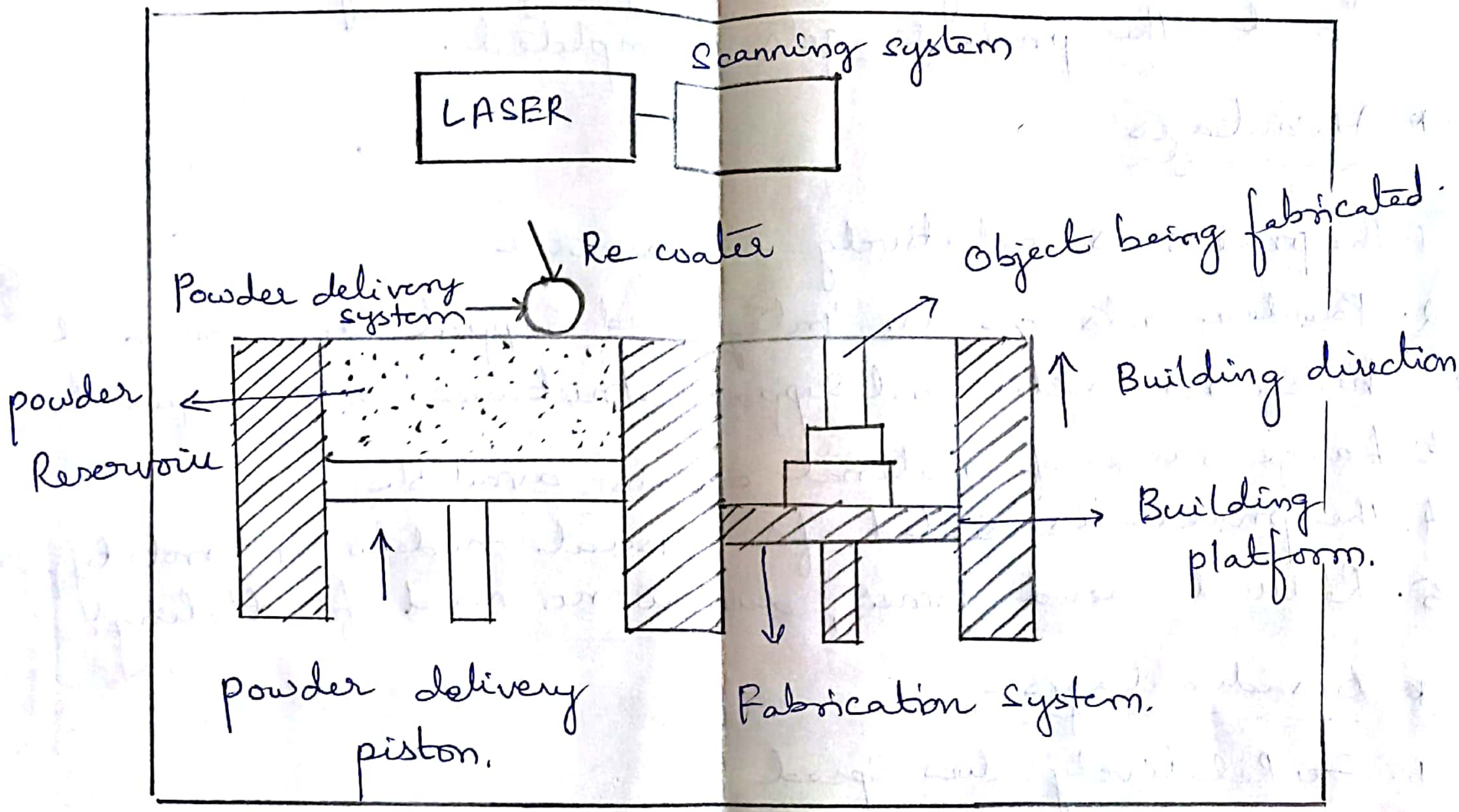
(i) Lack of understanding and expertise in additive manufacturing.

(d) Financial challenges

(i) initial investment
(ii) maintanance and repair charges
(iii) operation charges and electricity charges
(iv) Use of expensive materials and gases for manufacture.

(7) Selective Laser Melting process.

* Schematic diagram:-



Enclosed chamber filled with Argon gas.

→ Selective Laser melting (SLM) machine has a a chamber filled with metal powder. This metal powder is then spread across the substrate or build plate in very thin layers by a coater blade.

→ A high power laser fuses the 2D slice of the part by selectively melting the powdered material. The building platform then drops down by the height of one layer, the coater spreads another layer of fresh powder finely across the surface.

→ This process is repeated until the part is completly finished

→ Once the object is built, it is removed from the building platform and the supports are removed

→ further machining and Surface finishing is done and the product gets completed.

* Advantages:-

1. The process is relatively inexpensive.
2. Powder acts as an integrated support structure and hence no additional support structures are required.
3. Large range of material options available.
4. The process is suited for visual models & prototypes
5. Reduced lead times, due to no need for tooling.

* Disadvantages:-

1. Ex Relatively slow speed.
2. Lack of structural properties in materials.
3. process has high size limitations.
4. High power usage.

(Q9.) Applications of Additive Manufacturing:

Industry is taking advantage of aditive manufacturing to produce plastic metal, or composite parts and custom products without cost, time, tooling and overhead required in traditional machining or manufacturing processes.

→ Aerospace Industry is taking advantage of AM to simultaneously reduce material requirements and easily create engine parts.

→ Many military operations also require miniaturized, custom designed components in relatively small numbers.

→ For automotive industry, additive manufacturing holds great promise. Vehicle bodies and engines could be made using fewer parts and rapidly redesigned to minimize failures.

→ Health care industry is investing in tailored prosthetics dental implants, hearing aids, and other medical devices and tools.

→ They give industry new design flexibility, reduce energy use and shorten time to market.

→ Manufacturers of many consumer products may soon be using additive techniques to improve electrical performance and reduce weight and volume.

→ Additive manufacturing is used for aesthetic purposes such as for design, show case items, gifts and also statues etc...

→ It is also used in food industry and other various industries for producing vessels and cookeries.

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(8) Post processing techniques.

Most AM processes require post processing after building to prepare the part for its intended use.

1. Support material removal :-

Support material can be classified as (a) material which surrounds the part as a naturally occuring by product.

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(b) rigid structures which are designed and built to support, restrain or attach the part being built. This has to be removed.

(ii) Surface Texture improvements:-

AM parts have common surface texture features that may need to be modified for aesthetic or performance reasons. Common surface textures are:- Stair steps, powder adhesion, fill patterns from extrusion or beam based systems, witness marks from support material removal.

The type of post processing utilized for surface texture improvements is dependent upon the desired surface finish outcome.

If a smooth or polished surface desired, then wet or dry sanding and hand polishing performed.

(iii) Accuracy Improvements:-

There is a wide range of accuracy capabilities in AM. Process dependent errors affect the accuracy of the part. These errors come from positioning and indexing limitations.

Repeatable shrinking and distortion can be compensated for by scaling the CAD model.

Preprocessing of STL file to compensate for inaccuracies followed by finish machining of final part.

(iv) Aesthetic Improvements:-

Aesthetic Improvements such as painting, surface finishing and polishing increases the quality and look of the material. 1CR17ME056

Another Aesthetic enhancement is chrome plating through electrolysis process to enhance the properties and look of the material.

(v) Preparation for use as a pattern.

often parts using AM are intended as patterns for investment casting, sand casting, room temperature vulcanizations, and other pattern replication process. The accuracy and surface finish of AM pattern will directly influence the final part accuracy and surface finish. As a result special care must be taken to ensure pattern as accuracy and surface finish as the final part.

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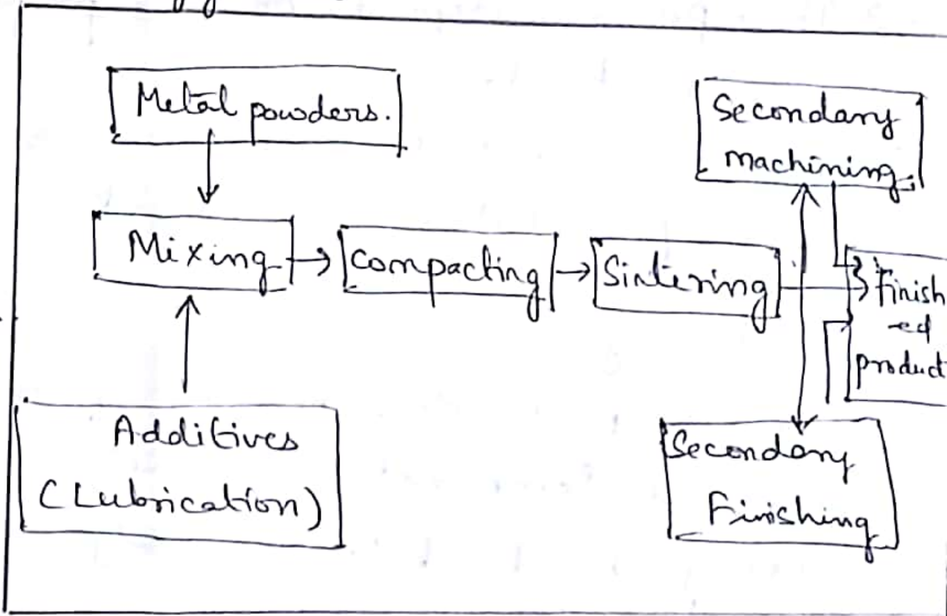
Additive Manufacturing

Assignment -02

① Powder metallurgy can be defined as the process of preparation and process the powdered iron or non-ferrous metals or materials.

The steps in powder metallurgy are:-

1. Powder preparation
2. Mixing and Blending
3. Compacting.
4. Sintering.
5. Secondary operations



Diagrammatic representation.

1. Powder preparation:-

→ First and Basic step in powder metallurgy. Any material can be converted into powder.

→ There are various process of producing powder such as atomization, grinding, chemical reaction, ball milling, Electrolysis etc...

2. Mixing and Blending:-

→ Blending imparts uniformity in the shapes of the powder particles

→ Process ensures even distribution of powder with additives binders etc

→ Lubricants are added to improve flow characteristic of powder.

3. **Compacting:-**

→ Compacting means compressing the prepared powder mixture into predefined dies

→ This step ensures to reduce the voids and increase the density of the product.

→ It involves pressure range from 80 to 1600 MPa

→ This pressure depends on the properties of metal powder and Binders.

→ Enhances contact area among the powder particles and facilitates subsequent sintering process.

4. **Sintering:-**

→ This step involves heating of green compact at an elevated temperature which ensures a permanent strong bond between adjacent particles.

→ This process provides strength to green compact & converts to final product

→ The sintering temperature is about 70-90% of melting temperature of metal powder

5. **Secondary operations.**

→ Sintered object is more porous compared to full dense material.

→ Some finishing operation such as repressing, sizing, hot forging, infiltration is done to further improve the quality of the product.

→ Most common secondary operation are sizing, coining, impregnation, hot forging etc.

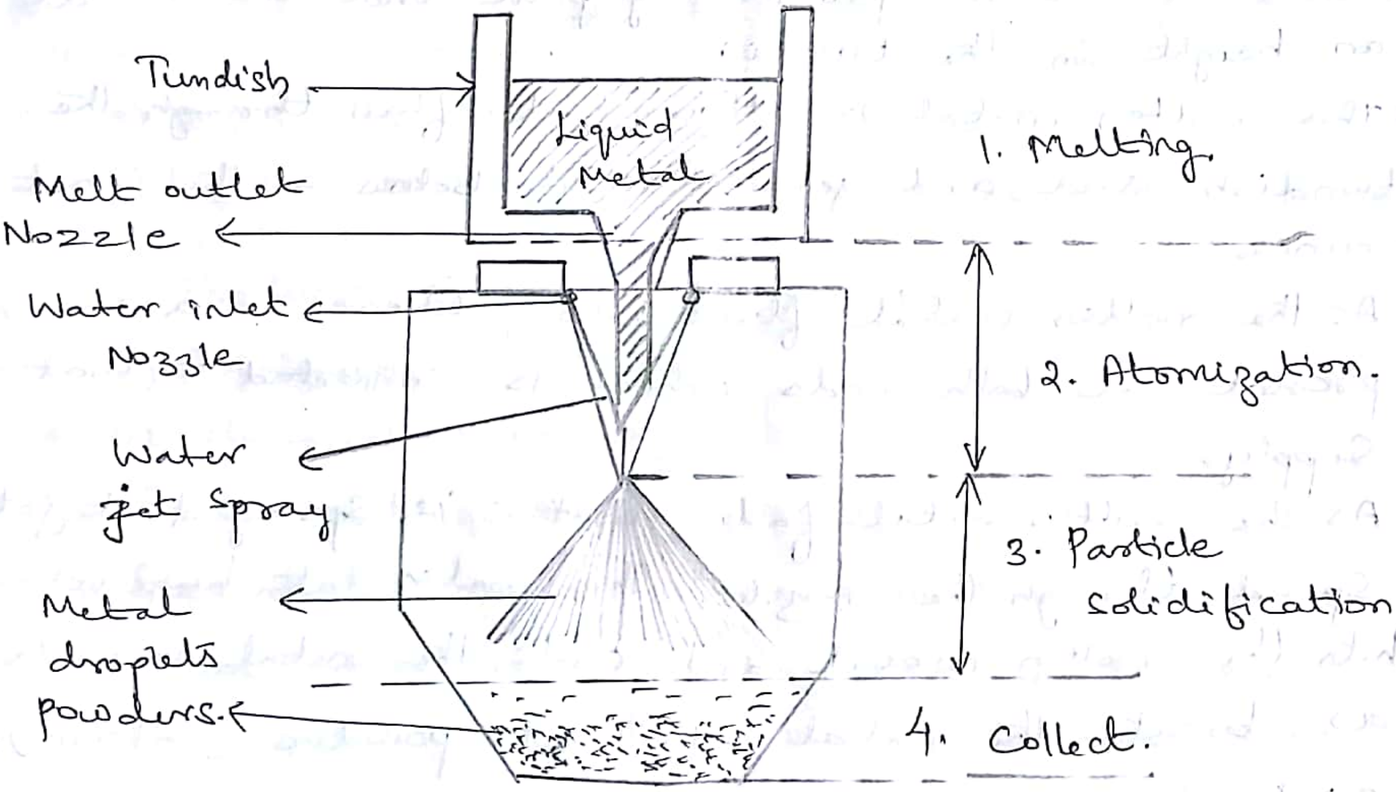
* **Advantages:-**

1. High production rate can be achieved
2. Machining operation is eliminated
3. Reduced production time

* **Disadvantages:-**

1. High initial cost of metal powder
2. High tooling cost
3. Equipments used are costly. and poor plastic properties

(5) Water Atomization process.



Schematic diagram.

* Water atomization of metals is process done inorder to achieve fine particle distribution for a range of materials.

→ <u>Construction</u> :-

a) Metal powder is heating upto its melting point and molten metal is placed inside the tundish.

b) There are water jet sprayers inorder to facilitate atomization

c) It consists of a collecting dish below inorder to collect the fine powder particles of the metal

→ <u>Working</u> :-

→ Metal is heated upto melting point and stored at an height in the tundish.

→ This molten metal is allowed to flow through the tundish and and get collected below in the container

→ As the molten metal flows below, there is a nozzle present at both ends which is connected to water supply.

→ As the molten metal falls, water is sprayed at jet speed through the nozzle. This water at huge velocity hits the molten metal and cools the metal as well as breaks the metal into small powders (<1mm). (>1mm).

→ As the metal becomes granules, it gets collected and cooled at the bottom of the setup and later is collected.

* Advantages:-

1. It is short process with high production efficiency
2. Suitable for large scale production.
3. Cheap raw materials can be used to produce high value and high quality powder.

* Disadvantages:-

1. Initial investment is very high
2. It is not conducive to the production of low density iron powder.

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(2) Explain advantages, disadvantages and applications of powder metallurgy

Ans:- The Major advantages of powder metallurgy are

→ Process advantages.

a) Eliminates or minimizes machining.

b) Efficient materials utilization - above 95% material utilization

c) Produces good surface finish.

d) Environment - friendly

→ Metallurgical Advantages.

a) Parts with controlled porosity.

b) Elemental and prealloyed powders.

c) Unique compositions including non-equillibrium compositions and micro structures. (crystalline, nano crystalline and amorphous)

d) Powders with uniform chemical composition with the desired characteristics, resulting from the absence of segregation during solidification.

e) Wide variety of materials - metals and alloys of miscible and immiscible systems, refractory metals like tungsten and ceramics, polymers, composite.

→ Commercial advantages :-

a) offers long term performance reliability in critical conditions.

b) Parts can be heated treated and plated if required

c) cost effective production of simple and complex parts.

* Applications of Powder Metallurgy.

a) Abrasive finishing - slot cleaning and abrasive wheels

b) Alloy production - steels, electroslag steel, free Nickel, lead, fe not Lings tin, ferrosilicon ferromolybdenum, ferromanganese, iron and aluminium, tungsten

c) Agriculture - Animal feed iron, animal medication cobalt, chelate fertilizers - Iron, seed cleaning - Iron, soil condition - Iron copper, Farm machinery - Iron, steel, copper, bronze.

d) Aerospace - Brake linings - copper, lead, tin, high nickel alloys, graphite, iron.

cores (electronic components) - Iron, permalloy

counter weights - Tungsten, copper, nickel, iron

Heat shields - Berilliyum, tungsten.

Filters for fuel, air, hydraulic fluids - Bronze, nickel stainless steel.

Heat shield coating - Aluminium.

* Disadvantages.
  a) High initial cost of metal powder.
  b) High tooling cost.
  c) Equipments used are costly.
  d) Poor plastic properties.

③ Explain with neat sketch different mechanical way of powder making methods.

Ans:- Mechanical methods of powder production are:-
  a) Chopping or cutting.
  b) Abrasion methods.
  c) Machining methods.
  d) Milling.

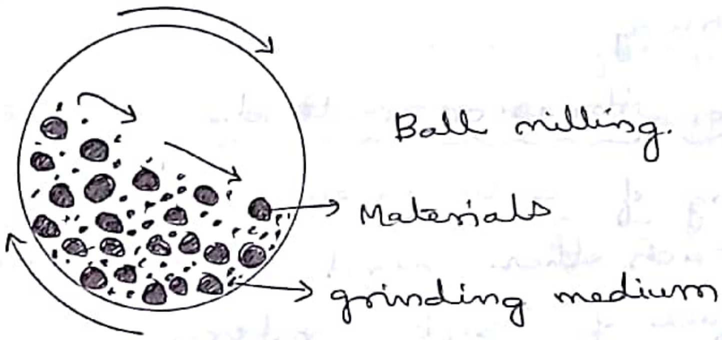
a) Chopping or cutting:- In this process, strands of hard steel wire, in diameter as small as 0.0313 inches are cut into small pieces by means of a milling cutter. This technique is actually employed in the manufacturing of cut wire shots which are used for peening or shot cleaning.

b) Rubbing or Abrasion methods.
  (i) Rubbing of 2 surfaces:- When we rub 2 surfaces against each other, hard surface removes material from the surface of soft material.
  (ii) Filing:- Filing as a production method has been frequently employed, especially to alloy powders, when supplies from conventional sources have been unobtainable.
  (iii) Scratching:- If a hard pin is rubbed on some soft metal the powder flakes are produced. Scratching is a technique actually used on a large scale for the preparation of coarse magnesium powders.

c) Machining – A machining process, using for example a lathe or a milling cutter in which something more than just scratching is involved, since the attacking tool actually digs under the surface of metal and it tears off. on lathe machine by applying small force we get fine chips. A large amount of machining scrap is produced in the machining operations.

d) Milling :- Most popular milling technique is Ball milling

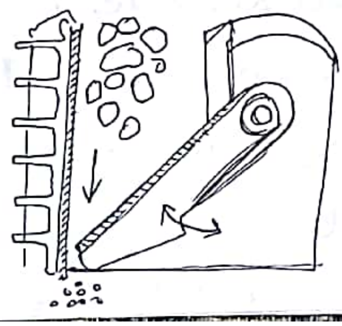
Ball mills :- This contains cylindrical vessel rotating horizontally along the axis. length of the cylinder is more or less equal to diameter. The vessel is charged with the grinding media. During rolling of vessel, the grinding media and powder particles roll from some height. This process grinds the powder materials by impact / collision.



Ball milling.

e) Grinding :- To grind a sintered sponge metal from reduction and electrolytic processes, jaw and hammer crushers are used above all, Grinding in a jaw crusher is performed between a fixed jaw and moving one.

Jaw Crusher.



(4.) Explain Reduction Method of powder making.

Ans:- Reduction of compounds, particularly oxides, by the use of reducing agents (solid or gaseous) is one of the most widely used and the oldest method of producing metal powders. other compounds such as hydrides, oxalates, and formates can also be used as starting method for reduction. This is a convenient, economical and extremely flexible method to control the properties of the product such as shape and porosity over a wide range. It is widely employed for producing powders of iron, copper, nickel, cobalt, tungsten, molybdenum, tantalum, thorium and titanium. Powders produced by this route exhibit pores within each powder particle and hence are called sponge powders. This sponginess, controlled by the amount and size of the pores, aids in improved compaction and sintering characteristics. Gases such as hydrogen, carbon monoxide, dissociated ammonia, coal gas, enriched furnace gas, natural gas, carbons etc.

The general equations for reduction of metallic oxides by hydrogen or carbon monoxide are:

$$MO + H_2 \uparrow \rightarrow M + H_2O \uparrow$$

$$MO + CO \uparrow \rightarrow M + CO_2 \uparrow$$

(6.) Explain electrolytic methods of powder making?

Ans:- Electrolytic deposition :-

→ used for producing copper, iron, tin, nickel, cadmium, antimony, silver, lead, beryllium powders.

→ Metals of high purity are precipitated from aqeous solution on the cathode of an electrolytic cell.

→ Copper powder ⇒ Solution containing Copper sulphate and Sulphuric acid and crude copper as anode
at anode : $Cu \rightarrow Cu^+ + e^-$; at cathode $Cu^+ + e^- \rightarrow Cu$

→ Iron powder ⇒ anode is low carbon steel, cathode is stainless steel.

→ Mg powder ⇒ electrode position from a purified magnesium sulphate electrolyte using insoluble lead anodes and stainless steel cathodes.

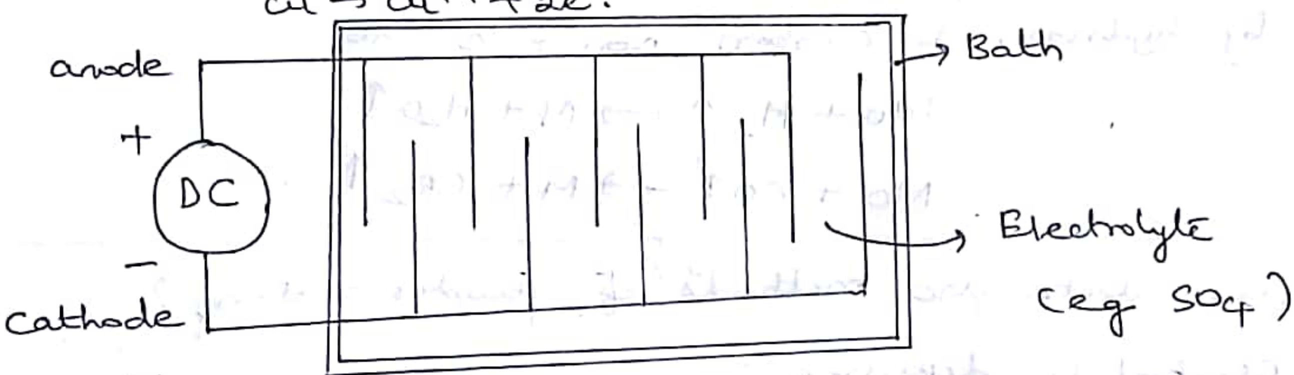
→ Final deposition occurs in 3 ways:-

a) A Hard brittle layer of pure metal which is subsequently milled to obtain powder (eg: Iron powder)

b) A soft, spongy substance which is loosely adherent and easily removed by Scrubbing.

c) A direct powder deposit from the electrolyte that collects at the bottom of the cell.

$Fe \rightarrow Fe^{++} + 2e^-$.
$Cu \rightarrow Cu^{++} + 2e^-$.



$Fe^{++} + 2e^- \rightarrow Fe$

$Cu^{++} + 2e^- \rightarrow Cu$

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4. Briefly explain physical methods of powder characterization

Answer:- Physical characteristics of metal powder characterization are size, shape and density.

* particle size:-

→ Particle size has a good importance in powder metallurgy because it affects some of the important properties such as green strength, density of compact etc.

→ Screening is the most widely recognised particle size technique, but is being displaced by automated size analyzes.

* Particle shape :-

→ The particle shape depends on the method of powder manufacture.

→ The various shapes are spherical, rounded, angular, dendrite etc..

→ The particle shape influences the flow characteristics of powders.

→ The particle shape has an effect on packing of a powder and has an influence on the compacting and sintering properties and the mechanical strength of the sintered product

* Microscopic method :-

→ microscopy is the most definitive method of particle size analysis, as it involves actual counting of individual particles on a slide containing a sample of product.

→ optical microscope is used for particles ranging from 100 to 0.1 μm.

→ Scanning electron microscope (SEM) is used for particles ranging from 10 µm to 0.01 µm while transmission electro-n microscope (TEM) is used for particles in the range 10 µm to 0.0001 µm.

→ SEM and TEM are very useful since these allow examination of individual particles which can reveal features not given by other methods.

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⑧ Briefly explain chemical methods of powder characterization.

Ans:- Metal powders are characterized for their chemical characteristics such as composition and purity.

* Chemical composition Analysis.

→ Chemical composition influences further processing such as compaction, ease of ejection as well as the degree of densification

→ chemical analysis must give the amount of metallic and non-metallic impurities.

→ The impurities can be in elemental form, or chemical composition of powders can be analysed by chemical methods.

→ Composition analysis involves the estimation of gaseous, impurities like oxygen, hydrogen, sulphur etc..

Some important techniques for analysis include.

a) Wet Chemical Analysis.

- principle :- Dissolving the sample and using various techniques like titration, spectroscopy.

- Remarks : Inertness problem, detection upto ppm levels possible.

b) X-ray flourescence.

- principle : Records characteristic x-ray lines.
- Remarks : non destructive, Calibration necessary, Surface Sensitive

c) Emission and spectroscopy.

- Principle : Vapourisation of the sample and recording the optical spectra.
- Remarks : Detection upto the ppm level possible.

d) Neutron activation analysis.

- Principle : Irradiating the sample and recording the radio-activity.
- Remarks : Expensive method.

e) Energy dispersive X-ray analysis (EDXA)

- principle : Measuring the energy of x-rays losses.
- Remarks : Qualitative, flat surface required, light element, non-destructive.

f) Electron probe micro analysis (EPMA).

- principle : Measurement of wavelength of X ray lines
- Remarks : Quantitative, flat surface required, light element detection is a problem.

g) X ray diffraction (XRD)

- principle : Measurement of diffraction angles and comparison with JCPDS file.
- Remarks : Rapid, qualitative, non-destructive.

9. Briefly explain processing methods of powder characterization.

Ans:- a) Particle size:-

It is the total space occupied by a powder particles. It is of great importance as it affects the some important properties like green strength, density of compact, porosity and dimensional stability

b) Particle shape:-

This depends on the method of powder manufacture. It influences the flow characteristics of powders. The irregularly shaped particles have reduced apparent density and flow rate, but good pressing and sintering properties.

c) Microscopic Method:-

This is the most definitive method of particle size analysis, as it involves actual counting of individual particles on a slide containing a sample of powder. optical microscope is used for particles ranging from 100 to 01 um. Scanning electron microscope (SEM) is used for particles ranging from 10 to 0.01 um while transmission electron microscope is used for particles in the range of 10 to 0.001 um.

d) Chemical Composition Analysis: Chemical composition Influences faster processing such as compaction, ease of ejection and degree of densification. chemical Analysis must give the amount of metallic and non-metallic impurities. Composition analysis involves estimation of gaseous / impurities like oxygen hydrogen, sulphur etc...

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Additive Manufacturing

Assignment - 03

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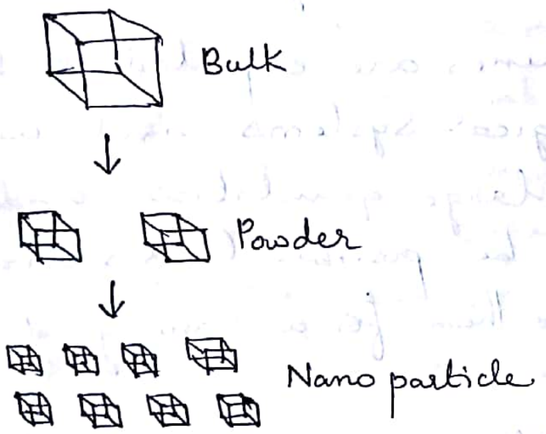
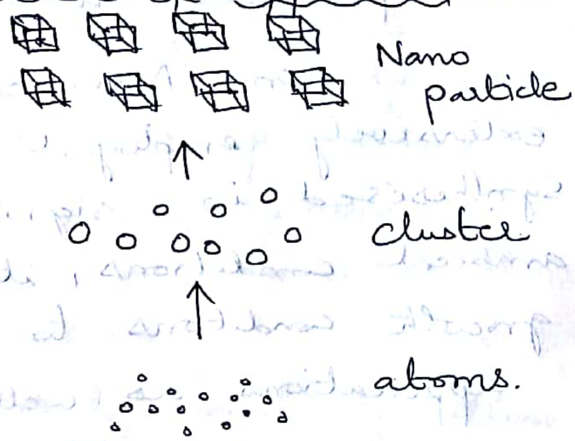
Write the difference between bottom up and Top down approaches in Nano technology.

Ans:- Top down approach.



* Top down approach starts from a large peice and subsequently uses finer and finer tools for correspondingly creating smaller structures.
* Various Nano manufacture techniques followed are :-
  a) Mechanical Alloying.
  b) Equal channel angular pressing. (ECAP)
  c) High pressure torsion (HPT)
  d) Accumulative roll bonding (ARB)
  e) Nanolithography.

Bottom up approach



* In bottom up approach, smaller components of atomic or molecular dimensions self assemble together, according to a natural physical principal or an externally applied driving force, to give rise to a larger and more organised systems.
* Various Nano manufacture techniques
  a) Physical vapour deposition (PVD)
  b) Chemical vapour deposition
  c) Spray conversion processing
  d) Sol-gel process
  e) Wet chemical synthesis
  f) Self - assembly.

2. Briefly describe challenges in nanotechnology.

Ans:- The Current applications of nanotechnology are much more lacking interest: strain resistant trousers, better sun screens, tennis rackets reinforced with nano tubes. There is a huge gap between what nanotechnology is believed to have promised and what is actually delivered so far.

Since Nanoscale machines are expected to be extensively employed in biological systems and would be synthesised in significantly large quantities under ambient conditions, it should be possible to discover the growth conditions to synthesize them for a variety of other applications as well.

The Beauty of nano technology is that it is truly multidisciplinary, re unifying the common threads between Science, engineering, and technology. It is so vivid, with possibilities left only to the constraint of perhaps human imagination. With little exaggeration, it seems possible that materials with any desired physical, chemical or electronic properties can be tailor made by playing with the nano dimensions.

The next generation is going to be directly or indirectly exposed to a variety of nano products ranging from cosmetics to sports, from medical to industrial, and also space applications. With the advent of any new revolutionary technology with enormous potential for applications. It is perhaps even more pertinent to assess the risks and challenges accompanying them.

5. Explain Wet chemical synthesis of Nano materials.

Ans :- a) This is a bottom up approach, solution based processing routes used for synthesis of nano particles include precipitation of solids from supersaturated solution, homogenous liquid phase chemical reduction and ultrasonic decomposition of chemical precursors.

b) This process are attractive due to their simplicity versatality and availability of low cost precursors.

c) Inorganic salt compounds used in wet chemical synthesis routes are more versatile and economical then alkoxides employed in sol gel process.

d) A typical example is the formation of nano crystalline titania powders via hydrolysis of $TiCl_4$ at lower temperatures

$$TiCl_4 + 2H_2O \longrightarrow TiO_2 + 4HCl.$$

e) Once the solution becomes saturated, crystallization of titania takes place either through homogenous or heterogenous nucleation, salt reduction is one of the most commonly adopted methods to generate the metal colloid particles

f) The process involves the dissolution of metal salts in aqeous or non aqeous environments followed by the reduction of metal cations to zero-valent state. The nature of the metal salts determines the kind of reducing agent to be applied.

g) Metal nano particles can also be generated via ultrasonic and thermal decomposition of metal salts or chemical precursors. Power ultrasonic waves can stimulate certain novel chemical processes due to formation of localised hot spots in liquid of extremely high temp

h) The main event in the process is nucleation, growth and collapse of cavitation bubbles formed in the liquid. The cooling achieved during the cavitation collapse is estimated to be greater than 2×10^9 K. It is called as sonochemical method.

i) Transition metal nanoparticles can be produced via sonication of their respective chemical precursors.
Eg:- $Ni(CO)_4$ has been sonicated under argon atmosphere to obtain amorphous liquid

j) one disadvantage of sonification process. is the difficulty in controlling the resulting particle size and distribution due to the agglomeration of particles into a porous coral like microstructure.

Explain with neat sketch the solgel synthesis of nano materials.

Ans:- The sol gel process involves the evolution of the inorganic networks through the formation of a colloidal suspension (sol) and gelation of the sol to form a network of a continous liquid phase (gel)

→ The starting material is processed to form a dispersible oxide and forms a sol in contact with water or dilute acid.

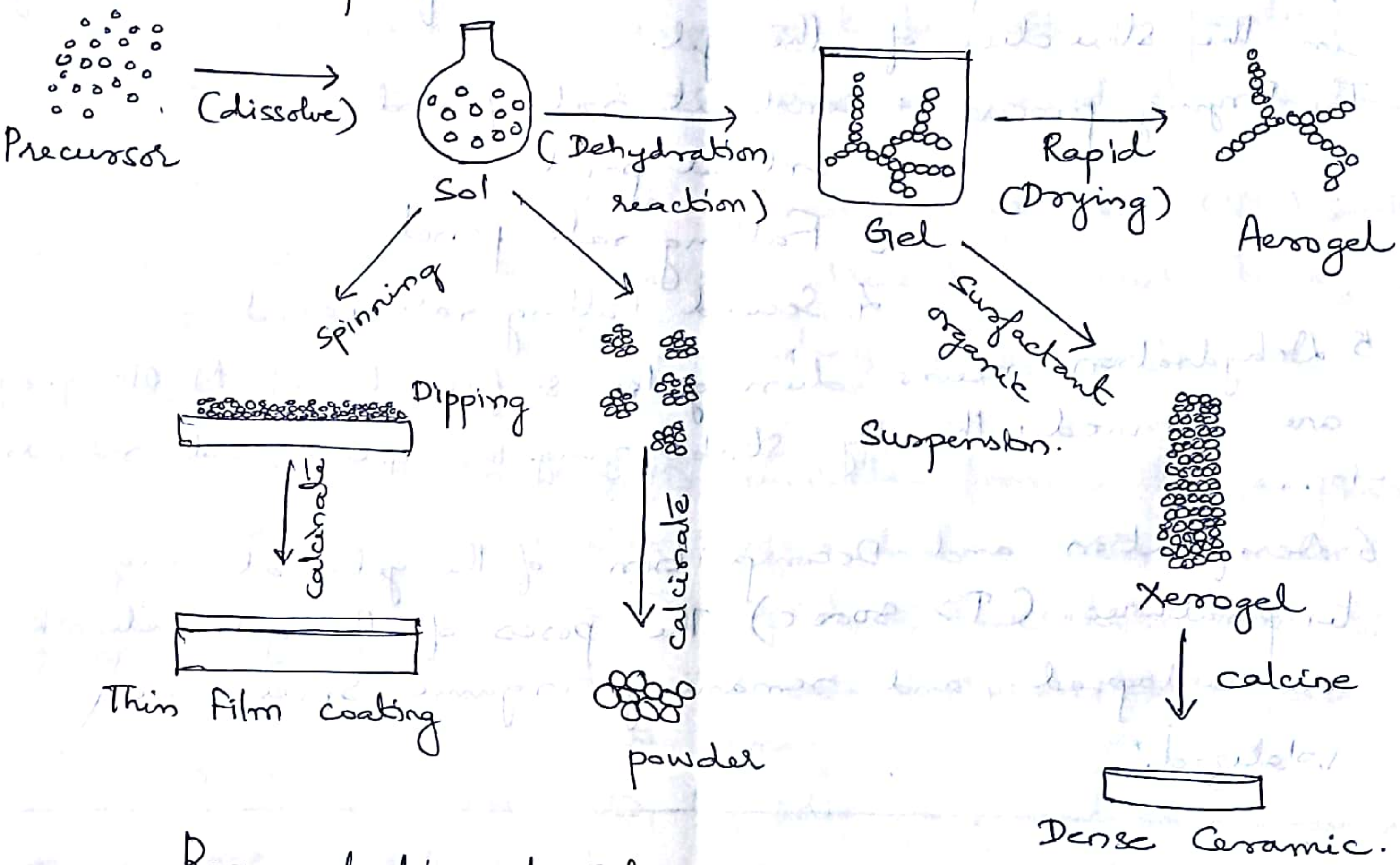
→ Removal of the liquid from the sol yeilds the gel, and the sol gel transition controls the particle size and shape

→ Calcination of the gel produces the oxide

→ sol gel processing refers to the hydrolysis of and condensation of the alkoxide based precursors.

→ Sol gel method of synthesising nano materials is very popular amongst chemists.

* The sol gel process can be characterised by series of distinct steps



Representation of Sol-gel process of synthesis of Nano materials.

1. Formation of different stable solutions of the alkoxide or solvated metal precursor.
2. Gelation Resulting from the formation of oxide or alcohol bridged network (the gel) by a polycondensation reaction results in a dramatic increase in the viscosity of the solution.

3. Aging the gel, during the poly condensation reaction continues until the gel transforms into a solid mass, accompanied by contraction of gel network and expulsion

of solvent from gel pores.

4. Drying of the gel is carried out when water and other voltatile liquids are removed from the gel network. This process is complicated due to the fundamental changes in the structure of the gel.

The Drying process :- 1. constant rate period.

2. critical point

3. Falling rate period

4. Second falling rate period.

5. Dehydration occurs during the surface bound M-OH groups are removed, thereby stabilizing the gel against rehydration.

6. Desification and Decomposition of the gels at high temperatures. (T> 8000°c). The pores of the gel network are collapsed, and remaining organic species are volatized.

Explain Gas phase of Nano materials.

Ans:- The Gas phase synthesis methods are of increasing interest because they allow elegant way to control process parameters in order to be able to produce size, shape and chemical composition controlled nanostructure.

In conventional chemical vapour deposition (CVD) synthesis, gaseous products either are allowed to react homogeneously or heterogeneously depending on the particular application.

(i) In Homogenous CVD, particles form in the gas phase and diffuse towards a cold surface due to thermophoretic forces, and can either be scrapped of the cold surface to give nano powders, or deposited onto a substrate to yeild what is called particulate films.

(ii) In Heterogenous CVD, the solid is formed on the substrate surface, which catalysis the reaction and a dense film is formed.

Inorder to form nano materials several modified CVD methods have been developed and gas phase processes have inherent advantages., Some of which are:-

a) a control of size, shape, crystallanity and chemical composition

b) Highly pure materials can be obtained

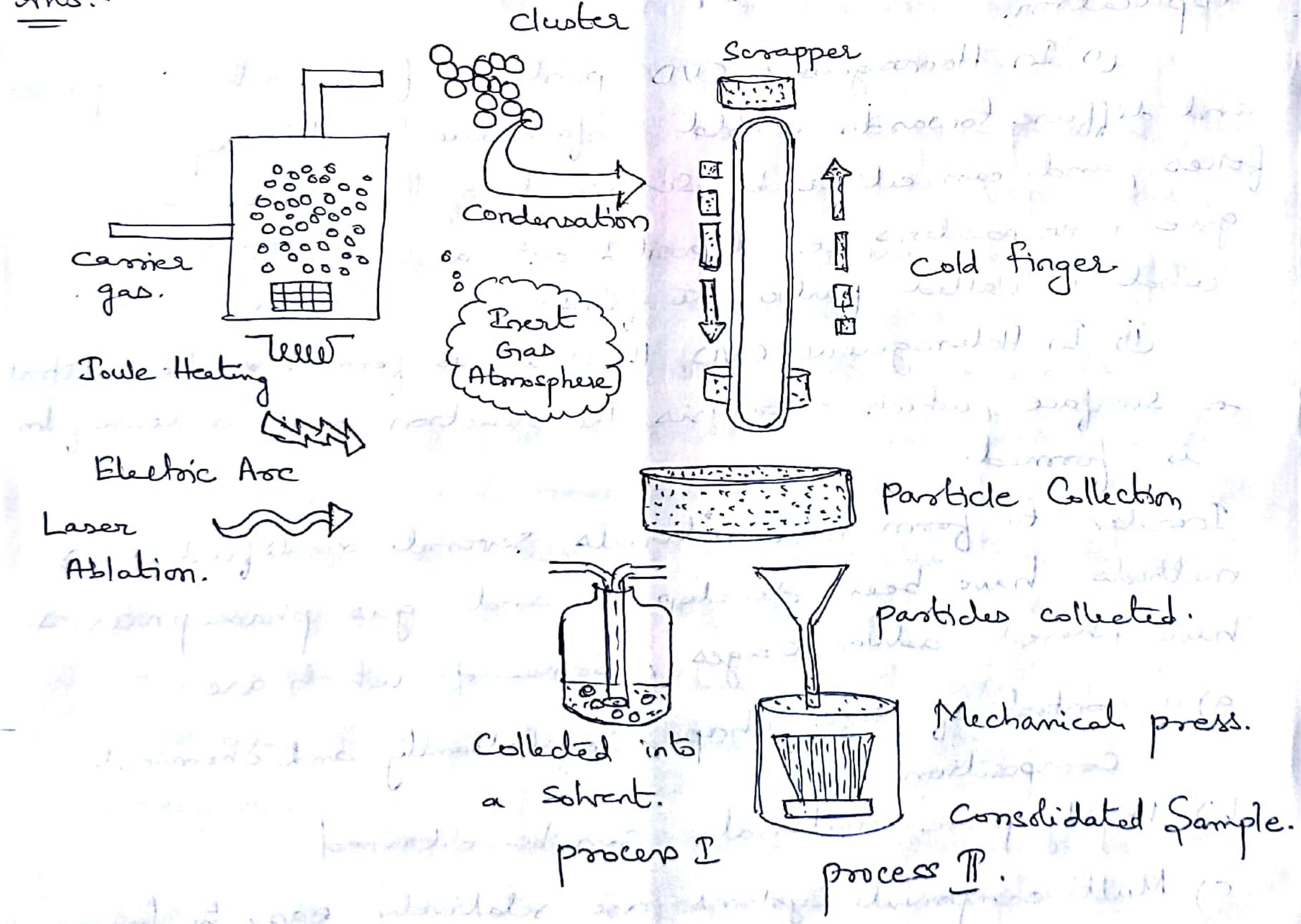
c) Multi component systems are relatively easy to form

d) Easy control of the reaction mechanisms.

Most of the synthesis routes are based on the production of small clusters that can aggregate to form nano particles. Condensation occurs only when the vapour is supersaturated and these processes homogenous nucleation in the gas phase is utilized to form particles. This Can be achieved by both physical and chemical methods.

Explain with neat sketch Gas Condensation processing (GPC) synthesis of nano materials.

Ans:-



* Schematic Representation.

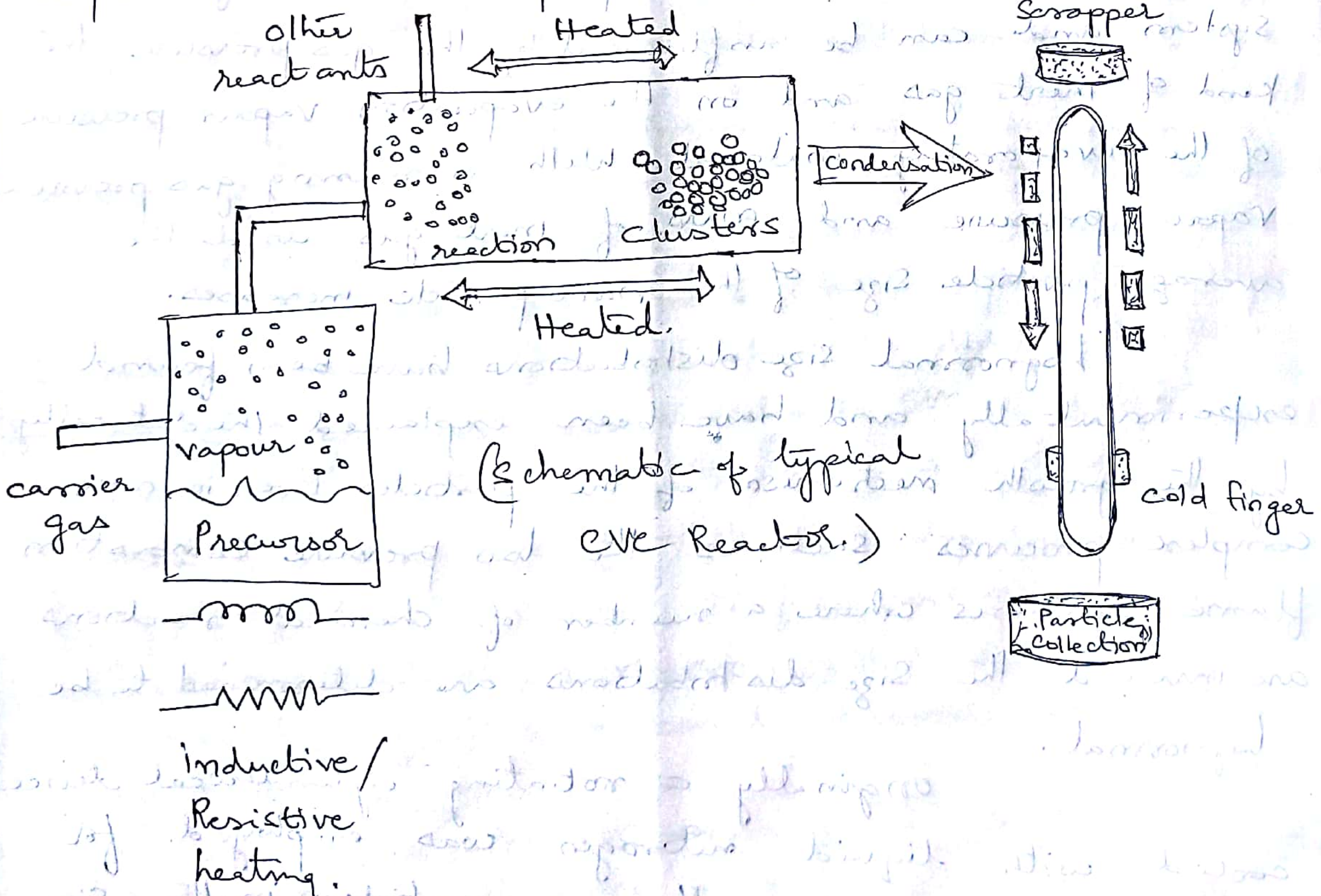
In this technique, a metallic or inorganic material eg:- Suboxide, is vapourised using thermal evaporation sources such as crucibles, electron beam evaporation devices or sputtering sources in an atmosphere of 1-50 m bar He (or another inert gas like Ar, Ne, Kr). Cluster form in the vicinity of the source by homogenous Nucleation in the gas phase and grow by Coalescence and Incorporation of atoms from the gas phase.

The cluster or particle size depends critically on the residence time of the particles in the growth system and can be influenced by the gas pressure, the kind of inert gas and on the evaporation vapour pressure of the evaporating material. With increasing gas pressure vapour pressure and mass of inert gas used the average particle size of the nano particle increases.

Lognormal size distributions have been found experimentally and have been explained theoretically by the growth mechanism of the particles. Even in more complex processes such as the low pressure combustion flame synthesis where a number of chemical reactions are involved the size distributions are determined to be lognormal.

originally a rotating cylindrical device cooled with liquid nitrogen was employed for the particle collection: the nanoparticles in the size range from 2-50 nm are extracted from the gas flow by thermophoretic forces and deposited loosely on the surface of the collection device as a powder of low density and no agglomeration. Subsequently the nano particles are removed from the surface of the cylinder by means of scraper in the form of a metallic plate. In addition to this cold finger device several techniques known from aerosol science has been implemented for the use in gas condensation systems such as corona discharge etc...

Explain chemical Vapour Condensation.



(Schematic of typical CVC Reactor.)

As shown schematically in the figure, the evaporative source used in GPC is replaced by a hot wall reactor in the CVC process. Depending on the processing parameters nuclea-tion of nanoparticles is observed during chemical vapour deposition of thin films and poses a major problem in obtaining good film qualities. The original idea of the novel CVC process which is schematically shown, it was intended to adjust the parameter feild during the synthesis inorder to suppress the film formation and enhance homogenous nucleation of the particles in the gas flow.

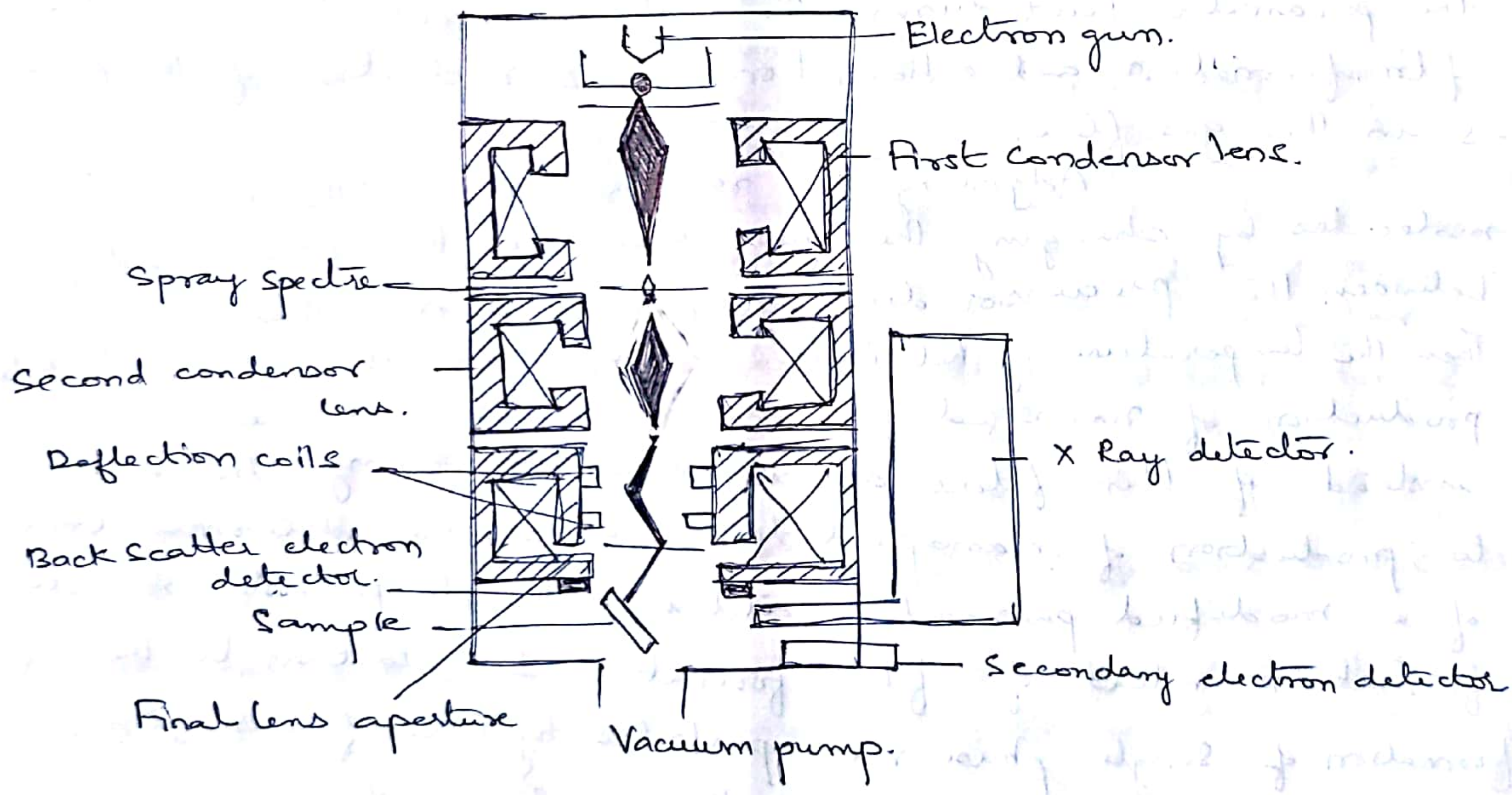
Adjusting the residence time of the precursor molecules by changing the gas flow rate, the pressure diff between the precursor delivery system and main chamber occurs. Then the temperature of hot wall reactor results in the fertile production of nanosized particles of metals and ceramics instead of thin films as in CVD processing. The extension to production of nanoparticles requires the determination of a modified parameter feild in order to promote particle formation instead of film formation. In addition to the formation of single phase nanoparticles by CVC of a single precursor the reactor allows the synthesis of

1. Mixture of nanoparticles of 2 phases or doped nanoparticles by supplying 2 precursors at the front end of the reactor.
2. Coated nanoparticles, i.e $n-ZrO_2$ coated with $n-Al_2O_3$ or vice versa by supplying a second precursor at second stage of the reactor.

Because CVC processing is continous, the production capabilities are much larger.

8) Explain scanning electron microscope (SEM) with neat sketch.

Ans:- SEM is a powerful magnification tool that utilizes focused beams of electrons to obtain information. The High resolution, 3 dimensional images produced by SEMS provide topographical, morphological and compositional information makes them invaluable in a variety of science & industry application.



SEM working principle and Imaging:

→ SEM provides surface information by tracing a sample in a raster pattern with an electron beam

→ The process begins with an electron gun generating a beam of energetic electrons down the column and onto a series of electromagnetic lenses. These lenses are tubes wrapped in coils and referred to as solenoids.

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→ The coils are adjusted to focus the incident electron beam on to the sample.

→ Controlled via computer, the sem operator can adjust the beam onto the sample to control magnification as well as determine the surface area to be scanned.

→ The beam is focused onto the stage, where a solid sample is placed. Most samples require some preparation before being placed in the vacuum chamber.

→ In addition, all the samples need to be able to handle the low pressure inside the vacuum chamber.

→ The interaction b/w the incident electrons and surface of the sample is determined by acceleration rate of incident electrons.

→ when the incident electrons come in contact with the sample energetic electrons are released from the surface of the sample. The scatter patterns made by the interaction yeilds information on size, shape, texture, and composition of sample.

→ A variety of detectors are used to attract different types of scattered electrons, including secondary and back scattered electrons as well as x-rays.

→ Back scattered electrons are incidents electrons reflected backwards, images provide composition data related to element and compound detection.

→ Diffracted back scatter electrons determine crystalline structures as well as the orientation of minerals and microfabrics.

→ sem produces black and white, three dimensional images. Image magnification can be upto to nanometers.

* Applications :-

→ number of scientific and industry related feilds especially where characterization of solid materials is beneficial.

→ Can detect and analyze surface fractures, provide info

, on microstructures, examine surface contaminations, reveal Spatial variations in chemical compositions, provide qualitative chemical analysis and identify christalline structures.

→ Sem have practical and industrial and technological applications such as semi conductor inspection, production line of miscule particles and assembly of microchip in computers

Advantages :-

a) wide array of applications, detailed 3 dimensional and topographical imaging and the versatile information garnered from diffrent detectors.

b) Easy to operate with proper training and advances in computer technology.

c) user friendly intuitive interfaces. Many application requires minimal sample preparation

Disadvantages :-

→ High cost and Large size

→ Expensive, large and must housed in area of possible, electric, magnetic and vibration interferences

→ High maintanance cost.

→ Special training is required to operate SEM as well as prepare samples.

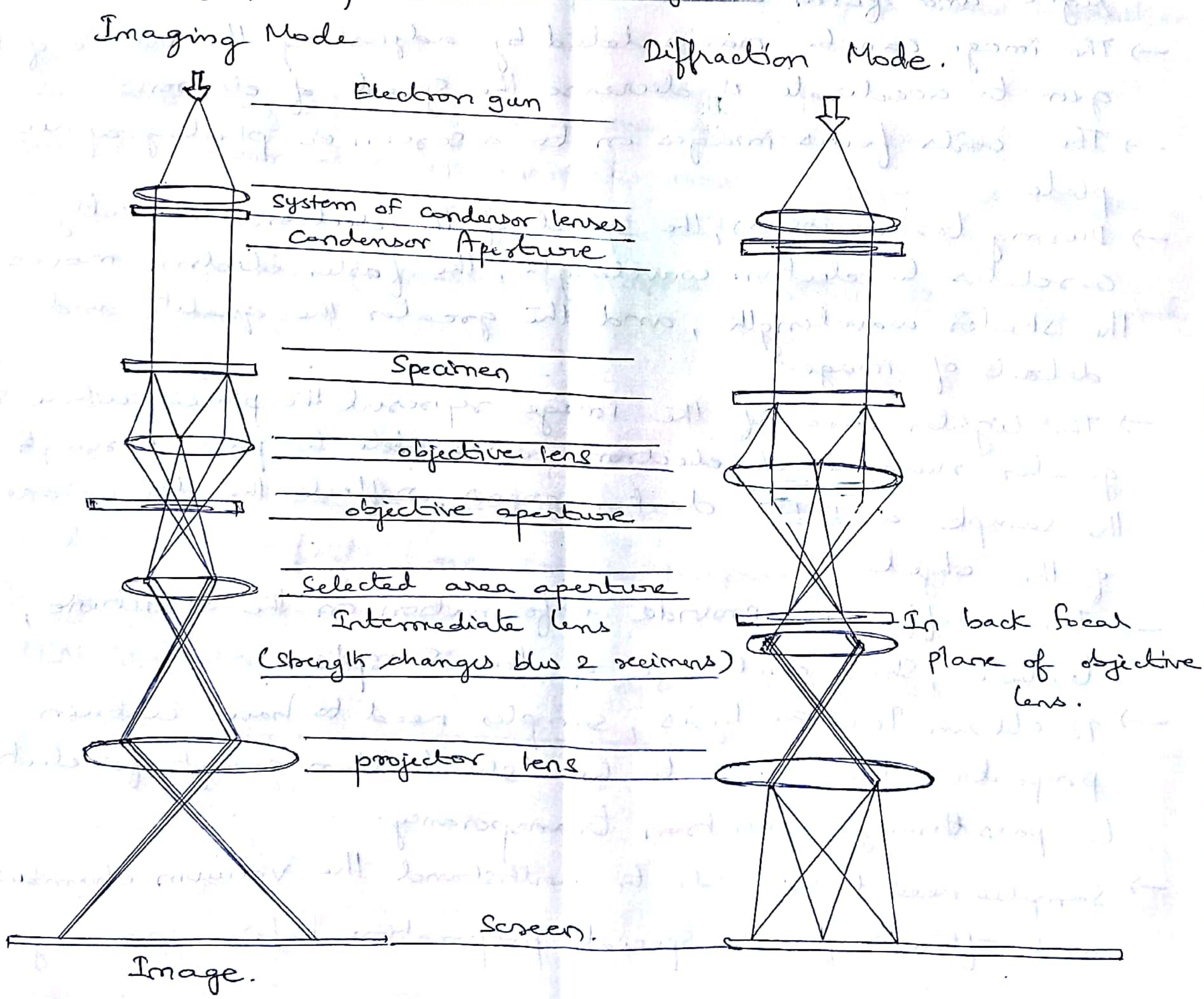
Scanned by CamScanner

9. Explain Transmission Electron Microscopy (TEM) - principles applications and limitations.

Ans:- A Transmission electron microscope utilizes energetic electrons to provide morphological, compositional and crystallographic information on samples.

Maximum potential of 1 nanometer magnification. TEMs are the most powerful microscopes. TEMs produce high resolution, 2 dimensional images, allowing for a wide range of educational, science and Industry applications.

* TEM working principle and Imaging



A TEM produces a high resolution, black and white image from the interaction that takes place between prepared samples and energetic electrons in the vacuum chamber.

→ Air needs to be pumped out of vacuum chamber, creating a space where electrons are able to move.

→ The electrons then pass through multiple electromagnetic lenses. These solenoids are tubes with coil wrapped around them.

→ The beam passes through the solenoids, down to column, makes contact with the screen where electrons are converted to light and form an image.

→ The image can be manipulated by adjusting the volume of the gun to accelerate or decrease the speed of electrons.

→ The coils focus images on to a screen or photographic plate.

→ During transmission, the speed of the electrons directly correlates to electron wavelength, the faster electron moves the shorter wavelength, and the greater the quality and detail of image.

→ The lighter areas of the image represent the places where a greater number of electrons were able to pass through the sample and the darker areas reflect the dense areas of the object.

→ These differences provide information on the structure, texture, shape and size of the sample.

→ To obtain TEM analysis, samples need to have certain properties. They need to be sliced thin enough for electrons to pass through, electron transparency.

→ Samples need to be able to withstand the vacuum chamber and often require special preparation before viewing.

→ Types of preparation include dehydration, sputter coating of non-conductive materials and staining.

* TEM applications:-

→ ideal for feilds such as life sciences, nanotechnology, medical, biological, gemology etc.

→ provide topographical, morphological, compositional and crystalline information

→ The images allow researchers to view samples on a molecular level, making it possible to analyse structure and texture.

→ Tems can be used in semiconductor analysis and production

Advantages:-

→ powerful magnification, over one million times or more.

→ provide information on element and compound structure

→ Images are high quality and detailed.

→ able to yeild information of surface features, shape, size and structure.

Disadvantages:-

→ Large and very expensive

→ Laborious sample preparation.

→ potential artifacts from sample preparation.

→ Images are black and white.

→ TEM requires special housing and maintenance.

10. Explain XRD with neat sketch.

Ans:- X ray powder diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analysed material is finely ground, homogenised and average bulk composition is determined.

* Construction:-

a) X ray tube.

b) Incident beam optics.

c) The goniometer

d) The sample and sample holder.

e) Recieving side optics

f) Detector.

* Principle of Working.

→ X ray diffraction is based on constructive interference of monochromatic xrays and a crystalline sample. These X rays are generated by a cathode ray tube, filtered to produce monochromatic radiation, collimated to concentrate and direct towards the sample. The interaction of incident rays with sample produces constructive interference.

→ These diffracted X rays are then detected, processed and counted by scanning the sample through a range of 2θ angles, all possible diffraction directions of the lattice should be attained due to random orientation of powdered material.

→ Conversion of the diffraction peaks to d-spacing (distance between planes of atoms that give rise to diffraction peaks) allows identification of mineral because each mineral has a set of unique d-spacing.

→ All diffraction methods are based on generation of X rays in an X ray tube. These x rays are directed at the sample and the diffracted rays are collected. A key component of all diffraction is the angle between the incident and diffracted rays.

* Applications :-

→ Characterization of crystalline materials.
→ Determination of unit cell dimensions.
→ Measurement of sample purity.
→ Determine of modal amounts of minerals.

* Advantages :-

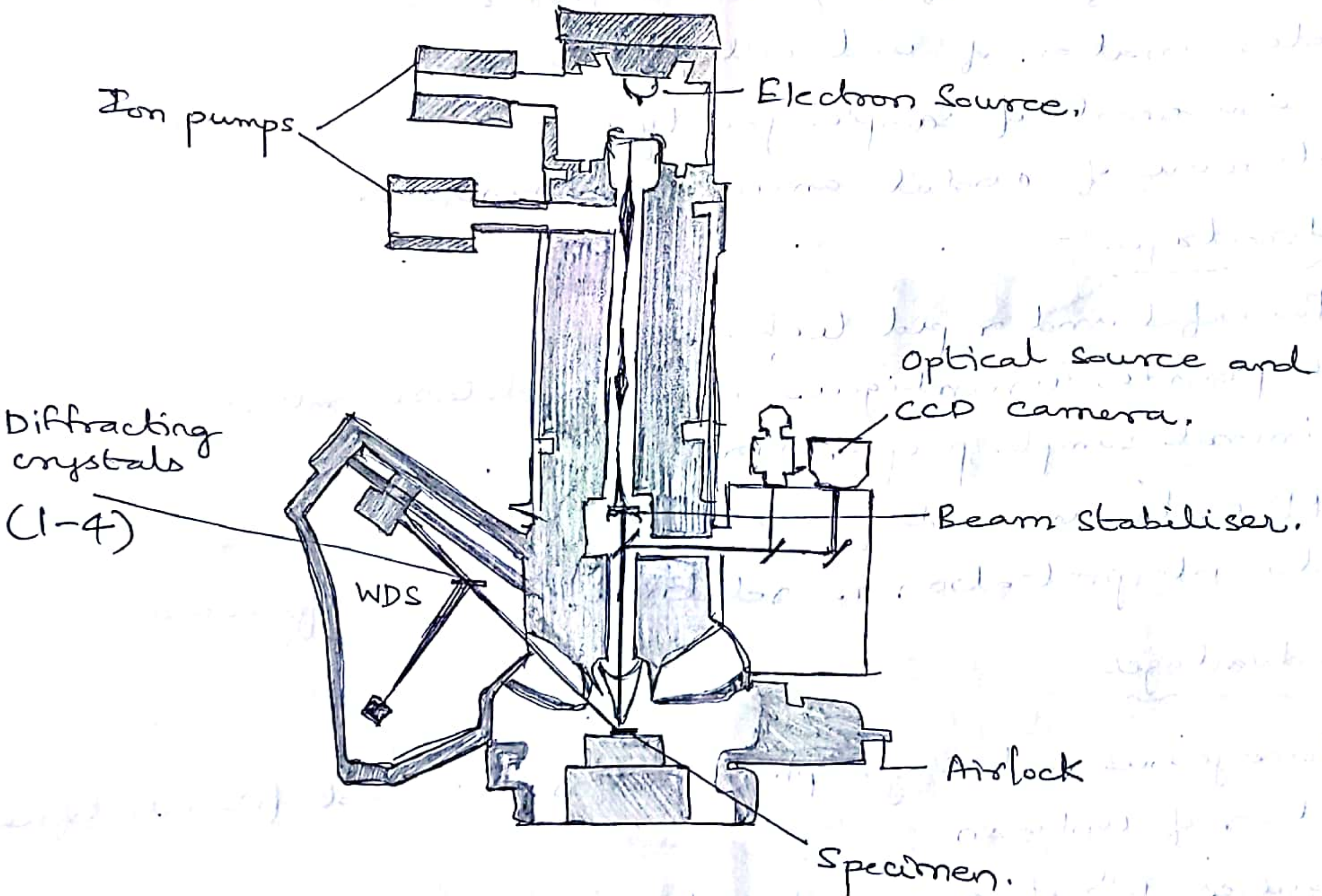
→ Powerful and rapid technique.
→ It provides unambigous mineral determination
→ Minimal sample preparation
→ XRD units are widely available.
→ Data interpretation is relatively straight forward.

* Disadvantages :-

→ Homogenous and single phase material is best for identification of unknown
→ Requires 10th of gram of material which must be ground into a powder.

(13) Electron probe micro analyser :-

An electron probe micro analyser is a micro beam instrument used primarily for the in-situ non-destructive chemical analysis of minute solid samples. It is also informally called an electron microprobe or just probe. It is fundamentally same as SEM with added capability of chemical Analysis.



Electron-probe-micro analyser.

* Working principle :-

Electron probe micro-analyzer works by bombarding a microvolume of a sample with a focused electron beam and collecting the X ray photons thereby emitted by the various elemental species. As the wavelengths of these X rays photons thereby emitted by the various elemental species. ~~= the wavelengths~~, the sample composition can be easily identified by recording wavelength Dispersive Spectroscope. Wavelength Dispersive Spectrometers operate based on Bragg's law and use various moveable, shaped mono crystals as mono chromators.

→ Electron probe micro analyzer is a fully qualitative and quantative method of non-destructive elemental analysis of micron sized volumes at the surface of materials, with sensitivity at the level of ppm.

→ It is fully compatible with routine analysis sessions, with easy and direct interpretation of the results.

→ Determination of thickness and elemental composition from nm to mm thick layers in stratified materials is possible.

* Applications :-

→ Quantitative Electron probe micro-analyzer analysis is the most commonly used method for chemical analysis of geological materials at small scales.

→ It chooses where individual phases need to be analysed, or where the material is of small size.

* Advantages :-

→ Equipped with wide range of crystal spectometers that enable quantative chemical analysis

→ Electron probes commonly have an array of imaging detectors.

* Limitations.

→ Unable to detect the lightest elements such as Hydrogen, helium and Lithium

→ Some elements generates X rays with overlapping positions.