



الجامعة التكنولوجية
قسم هندسة المواد
Department of Materials Engineering



Powder Metallurgy

3rd Level

Team Work

By

Dr. Arwa Faraj Tawfeeq

Dr. Ali AbdAlKareem

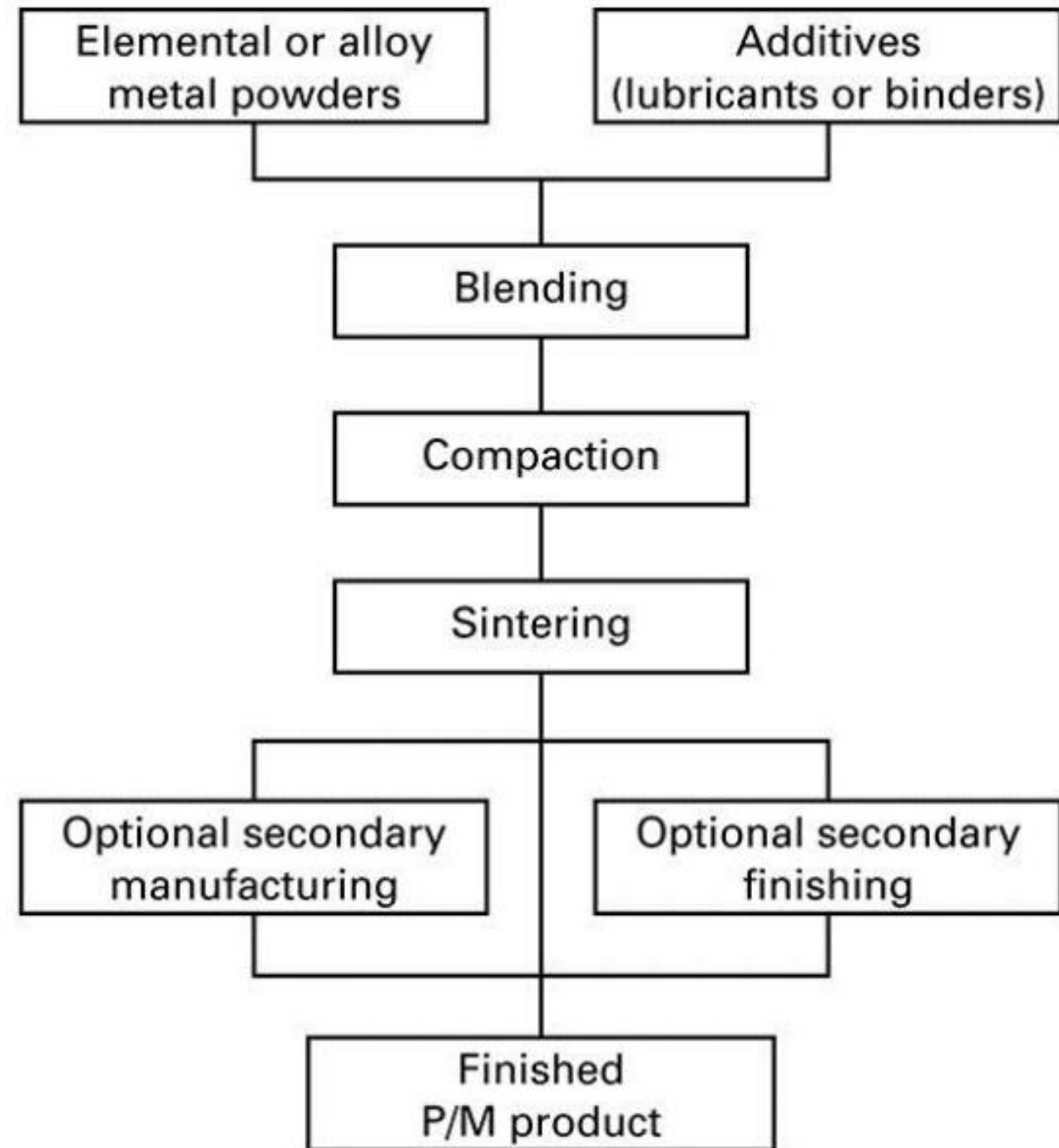
Content

1. Powder metallurgy,
2. Steps In powder Metallurgy,
3. Metal powder production,
4. Advantages,
5. Limitations,
6. Metal Powder Production Methods,
7. Mechanical Methods,
8. Milling (Objective),

Powder Metallurgy: is defined as the art and science of producing fine metal powder and making finished/semi-finished objects from mixed or alloyed metal powders with or without the addition of non-metallic constituents.

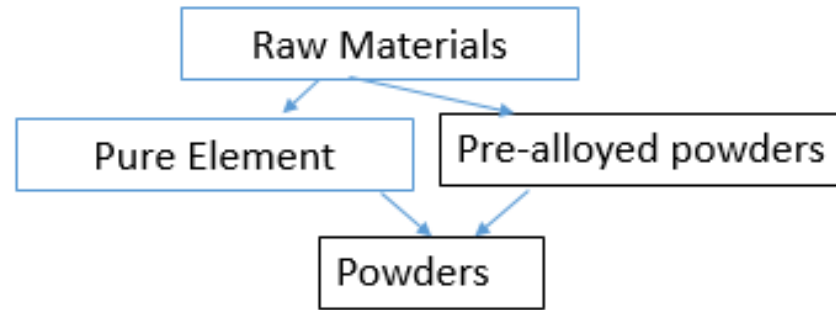
Steps In powder Metallurgy

- 1- powder production
- 2- Blending and mixing
- 3- compaction
- 4- Sintering
- 5- Secondary finishing processes.



1.1 Metal powder production.

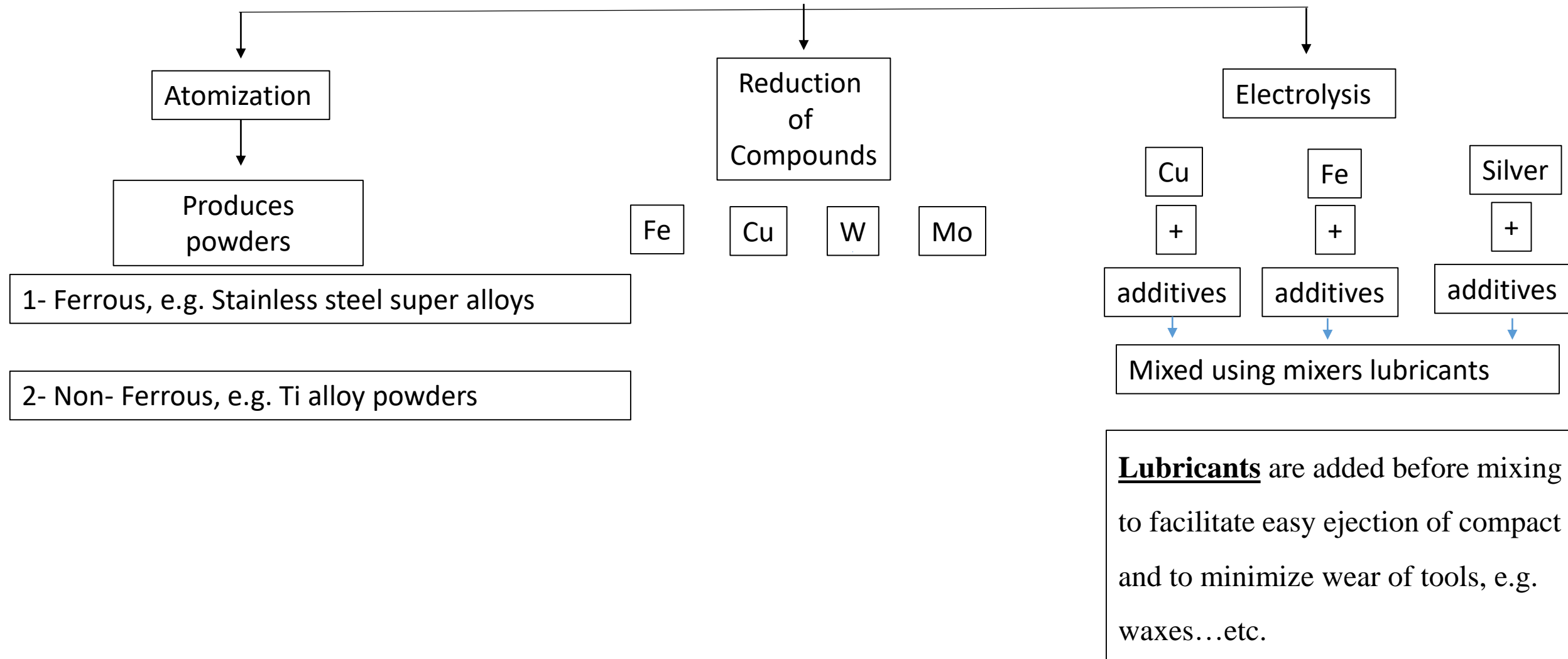
- It includes disintegration (تفتيت) of solids (without phase change), and **liquids, or gas, or oil, or mechanical dispersion** Incase of changing phase, to produce metal powder.
- The solids to disintegrate are possible to be taken from pure element or pre-alloyed powders.



- The produced metal powder is considered to be the main constituent of (PM) product, so, it is responsible on the final properties of the finished product (PM), e.g. size, shape, and surface area of the powder particles.
- However, the single powder production method is not sufficient for all applications.

1.1 Metal powder production.

➤ **Methods:** there are mechanical, physical, chemical methods to produce metal powder. See Example below.



Powder Metallurgy Advantages

Efficient material utilization: considered an economic process because involve about 97% metal powder, while, a very little amount of material's wastes, when converted to a some product, compared with the casting process. In addition, it is automated process.

Considered a dimensional control method of the product compared with the casting.

Enables close dimensional tolerances – near net shape possible, eliminating or reducing the need for subsequent processing.

Could be made Parts with controlled porosity: the possibility to produce a porous metal parts, e.g. filters. This is due to the nature of the raw materials used in PM process, which they already have a specified level of porosity.

Manufacture of complex shapes: the possibility to produce very complex shapes of parts, that, are difficult to produce by other methods, e.g. Tungsten, (Tungsten wire used in incandescent (متوهجه) lamp are made using PM technology). Also, combinations of certain metal alloy with cermet (سببکه معدنيه خزفیه) could be produced by PM process that could not be produced by other methods.

Provide good surface finish

Environment friendly, energy efficient

Powder Metallurgy Limitations

- (1) High cost of powder material & tooling.
- (2) there are difficulties with storing and handling metal powders (such as degradation of the metal over time, and fire hazards with particular metals). Also,
- (3) there are limitations on part geometry because metal powders do not readily flow in the die during pressing, and allowances must be provided for ejection of the part from the die after pressing.
- (4) variations in material density throughout the part may be a problem in PM, especially for complex part geometries.
- (5) Suited for moderate to high volume component production only.
- (6) Powders of uniform chemical composition => reflected in the finished part wide variety of materials => miscible (قابل له للامتزاج بآية نسبة), immiscible systems; refractory metals (المواد العاكسه).
- (7) Less strong parts than wrought alloys.

1.1 Metal powder production Methods.

1- Mechanical method.

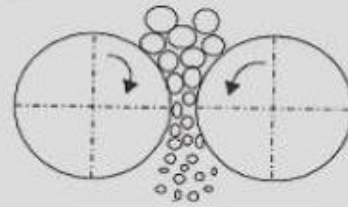
(1) Jaw crusher, (2) Gyratory crusher, (3) Roll crusher, (4) Ball mill, (5) Vibratory ball mill, (6) Attritor, (7) Rod mill, (8) Hammer mill, (9) Planetary mill.



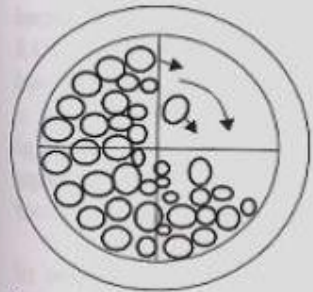
Jaw crusher



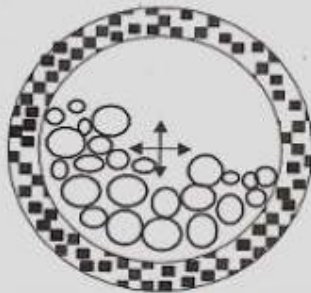
Gyratory crusher



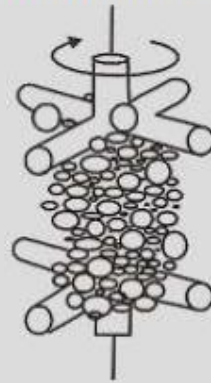
Roll crusher



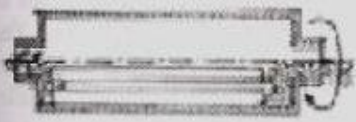
Ball Mill



Vibratory Ball Mill



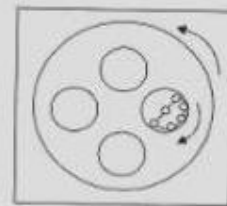
Attritor



Rod Mill

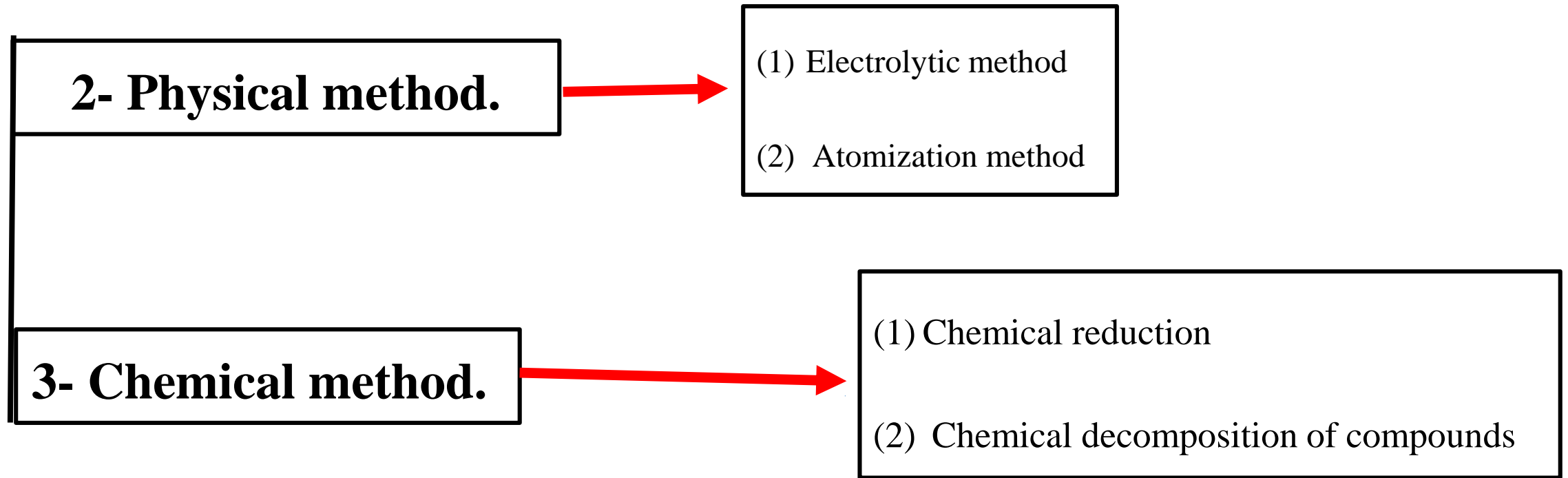


Hammer Mill



Planetary Mill

1.1 Metal powder production Methods.



Selection best method to produce powder metals depends on; (1) application type; (2) desired properties; (3) structure of the final product.

(Mechanical Method).

- The cheapest method of powder production;
- These methods involve using mechanical forces such as compressive forces, shear or impact to facilitate reducing the particle size of bulk materials; Eg.:Milling.
- Disintegration (تفتتیت) of solids (without phase change) and liquids (with phase change) can be applied to produce metal powder.
- These processes are not much used as primary methods for the production of metal powders. On the other hand, used as primary methods in the case of materials, easy to fracture e.g. (pure antimony and bismuth), hard and brittle metal alloys and ceramics, Reactive materials such as beryllium and metal hydrides, and, common metals such as (AL, Fe) which are required sometimes in the form of flake powder.

(Mechanical Method).

Milling

- During milling, impact, attrition, shear and compression forces are acted upon particles of the material, disintegrating particles into a smaller.
- During impact, striking of one powder particle against another occurs.
- Attrition refers to the production of wear debris (مخلفات على شكل شظايا) due to the rubbing action (تأثير الصقل) between two particles.
- Shear refers to cutting of particles resulting in fracture.
- The particles are broken into finer particles by squeezing action in compression force type.
- The milling process leads to an increase of the dislocation density and, therefore, reduces the compactibility.
- In most cases, subsequent heat treatment is necessary to soft-anneal the powders before use.

(Mechanical Method).

Milling Objective

- ❑ **Main purpose of milling** is to reduce particle's size, growth and (تكتلات و تجمعات) agglomeration (joining of particles together), also, changing their shape, solid state mixing, modification and hence, changing material properties.

(Mechanical Method).

Milling Mechanism

Changes in the morphology of powder particles during milling results the following situations:

Micro-
forging طرق دقيق

Fracture or fragmentation

تكسير ام تفتيت الى شظايا

Agglomeration

تكتلات

De- agglomeration

تفكيك التكتلات

Individual particles or group of particles are impacted repeatedly so they flatten with very less change in mass

Individual particles are deformed by initiation cracks, then propagate resulting in Fracture.

Mechanical interlocking (متشابكات) due to atomic bonding.

Breaking of agglomerates

- Powder characteristics such as shape, size, texture, particle size distribution, crystalline size, chemical composition, hardness, density, flow-ability, compressibility, sinter-ability, sintered density are influenced by milling.

(Mechanical Method).

Milling Equipment

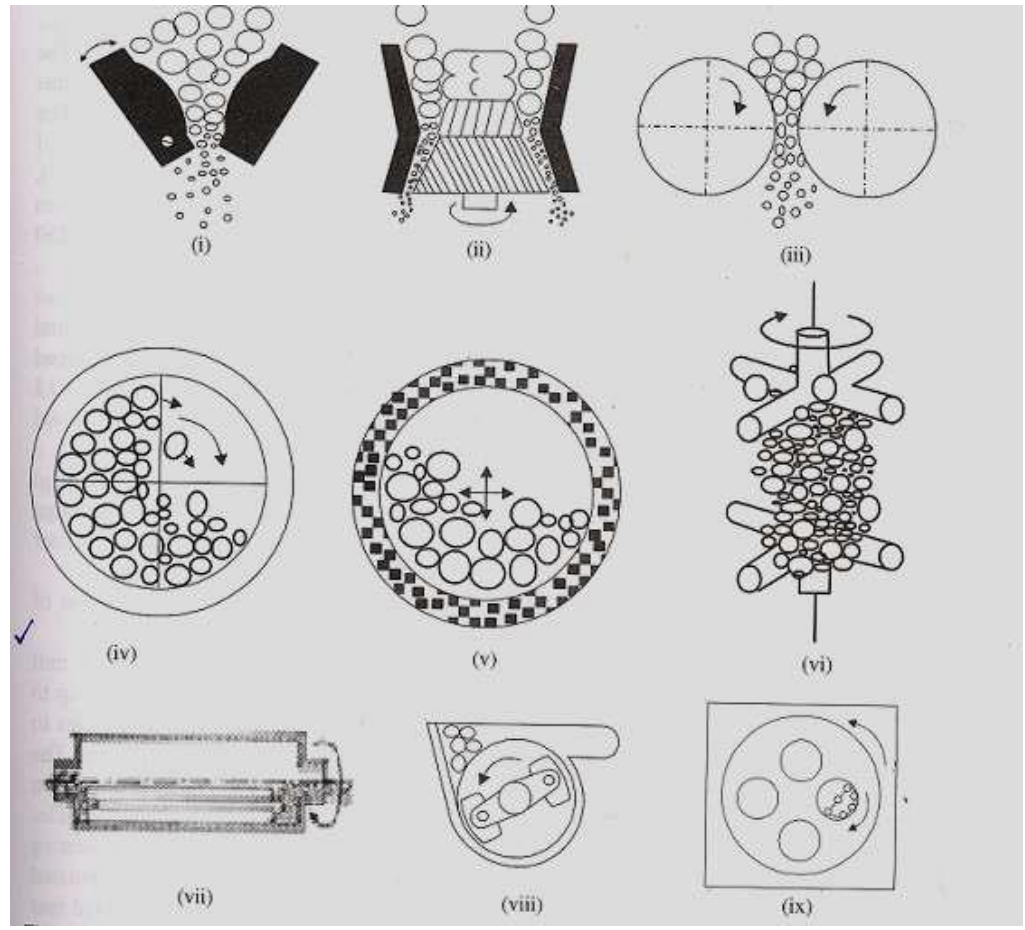
Are classified into:

Crushers & Mills Crushing

For making ceramic materials such as oxides of metals.

Grinding

For reactive metals such as titanium, zirconium, niobium, tantalum



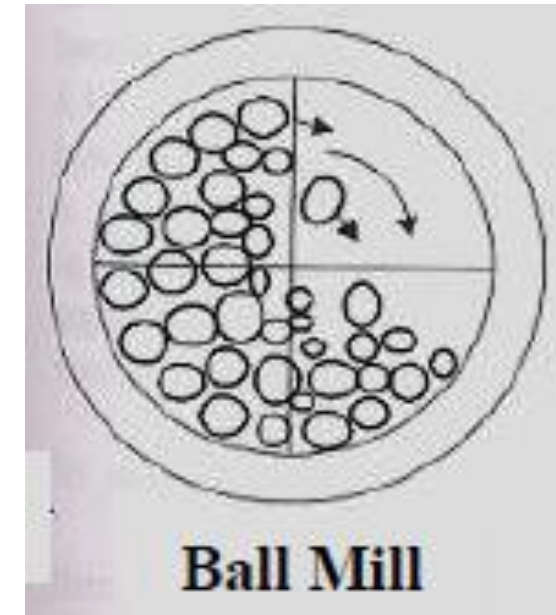
Different types of grinding equipments/methods are shown in the Figure.

(Mechanical Method).

Milling Equipment

Ball Mills

- Contain cylindrical vessel rotating horizontally along the axis.
- Length of the cylinder is more or less equal to diameter.
- Optimum diameter of the mill for grinding powders is about 250 mm.
- The vessel is charged with the grinding media (الوعاء يحمل بعدد للتنعيم كالكرات).
- The grinding media may be made of hardened steel, or tungsten carbide, ceramics like agate (عقيق), porcelain, alumina, zirconia.
- During rolling of vessel, the grinding media & powder particles roll from some height.
- This process grinds the powder materials by impact/collision & attrition (احتكاك).



Milling

Dry Milling

25 vol% of powder + 1 wt% of a lubricant such as stearic or oleic acid

Wet Milling

30-40 vol% of powder + 1 wt% of dispersing agent such as water, alcohol is employed

(Mechanical Method).

Milling Equipment

Vibratory Ball Mills

- This type of milling is more suitable for producing the finer powder particles as they need longer period for grinding.
- This is because vibratory ball mills have a higher amount of energy which is imparted to the particles by accelerating the vibration movement in the container.
- This mill contains an electric motor connected to the eccentric shaft of the drum (أسطوانه جوفاء) by an elastic coupling (ذراع مزدوج مرن).
- The drum is usually lined with wear resistant material.
- During operation, the rotation of the eccentric shaft causes the drum to oscillate (تتأرجح).
- In general, vibration frequency is equal to 1500 to 3000 oscillations/min. The amplitude of oscillations is 2 to 3 mm.
- The grinding bodies (العدد المستخدمه للتنعيم) is made of steel or carbide balls, that are 10-20 mm in diameter.
- The mass of the balls is 8-10 times the charged particles.
- Final particle size is of the order of 5-100 microns.

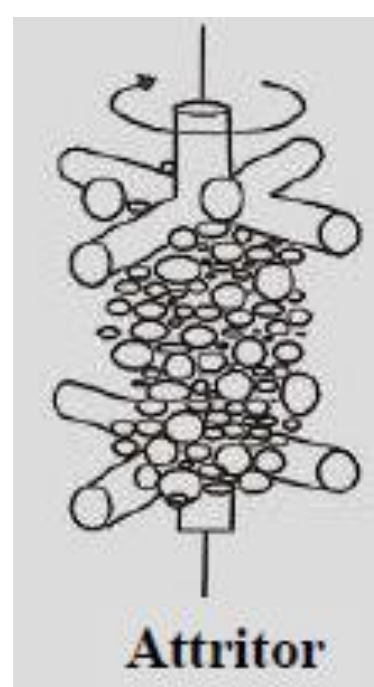


(Mechanical Method).

Milling Equipment

Attrition Mills

- IN this case, the charge is ground to fine size by the action of a vertical shaft with side arms attached to it. The ball to charge ratio may be 5:1, 10:1, 15:1. This method is more efficient in achieving fine particle size.



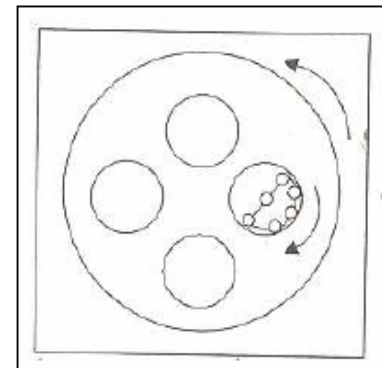
Rod Mills

- Horizontal rods are used instead of balls to grind the raw material's charge. Milling speed here varies from (12-30)rpm, to granulate (يجعل شكله حبيبي) discharged material from about (40 to 10)mm.



Planetary Mills

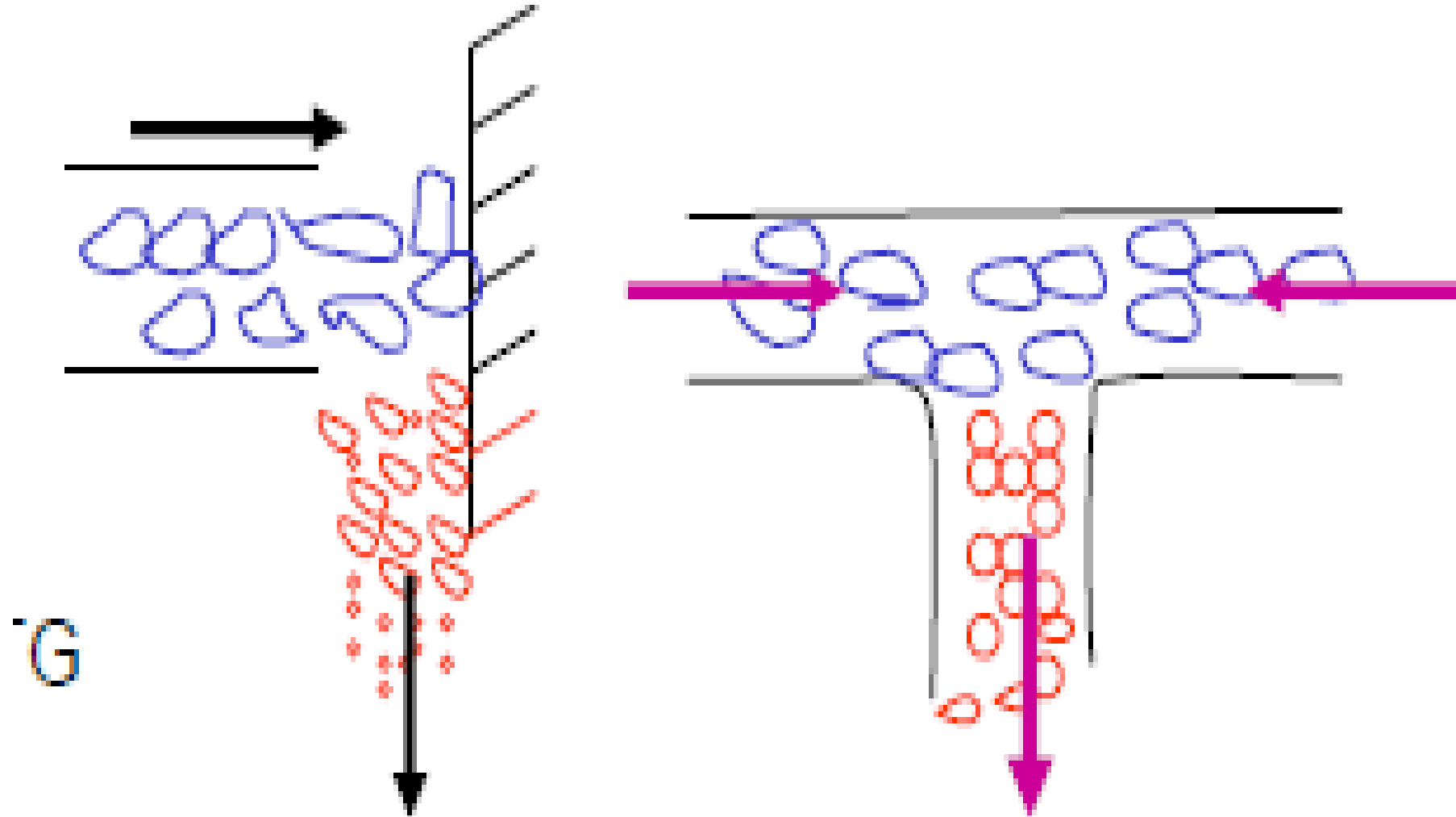
- High energy mill widely used for producing metal, alloy, and composite powders.



(Mechanical Method).

Milling Equipment

The mechanism of Fluid Energy Grinding or Jet Milling machine.



(Mechanical Method).

Milling Equipment

The mechanism of Fluid Energy Grinding or Jet Milling machine.

- ✓ Includes inducing (حث) particles by colliding (تصادم) against each other, at high velocity making them to fragment (تتجزأ) to finer particles .
- ✓ The continuous collision for the big particles with each other with incorporation for the multiple jet arrangements in the mill design enhances reduction the particle's size of the material.
- ✓ The fluid used is about to be either air or steam.
- ✓ Jet mills arrangements are based on the rotating motion of a particle-loaded gas stream at high speed in a round chamber. The high-velocity collision of the particles leads to fragmentation, and the small particles are removed in the center of the chamber whereas large particles are retained in the stream until the desired particle size is reached.
- ✓ تنسيقات التفريز او التنعيم النفائث: وهي قائمه على اساس الحركة الدورانيه للاجزاء المحمله مع بخار الغاز وبسرعه عاليه جدا في حجره او غرفه دائريه. سرعة التصادم العاليه للأجزاء تؤدي الى التجزئه وبالتالي فإن الاجزاء التي تم تنعيمها تزال من وسط الحجره بينما الأجزاء الكبيره تظل تحت تأثير البخار حتى تصل للحجم المطلوب.

(Mechanical Method).

Milling Equipment

Fluid Energy Grinding or Jet Milling

- In the case of **volatile** (المواد المتبخرة) materials, the protective atmosphere of Nitrogen and carbon di-oxide is used.
- The pressurized fluid is introduced into the grinding zone through special designed nozzles which convert the applied pressure to kinetic energy.
- Also, materials to be powdered are introduced simultaneously بالتزامن into the turbulent zone (المنطقة المضطربة).
- Fluid's velocity which comes out the nozzles is proportional $\alpha \sqrt{(Absolute Temp)}$ for the intering Fluid into the nozzle (سرعة المائع التي تخرج من الفتحة متناسبه طرديا مع درجة حرارة المطلقه للمائع الداخل الى الفتحة)

Hence it is preferable to raise the temperature of fluid to the maximum possible level without affecting the feed material.

ولهذا يفضل رفع درجة الحرارة داءما للمائع قدر الامكان الى اعلى مستوياتها وبالشكل الذي لايؤثر على نسيجها التركيبي.

(Mechanical Method).

Milling Equipment

Machining (خراطه Turning)

- For example, dental alloy (سبائك المتعلقة بالاسنان), e.g. Mg, Be, solder, are specifically made by machining. During machining (turning), chips are formed, these chips are crushed or ground into powders later on.

Shooting

- The molten metal is poured through a vibratory screen (مصفي هزاز) into air or into a medium (وسط) of a protective gas.
- During the process, the molten metals disintegrate (يتفكك) and solidify as spherical particles.
- The size of the obtained particles depends on pore size of screen, temperature, gas used, vibration frequency,
- However, the particles get oxidized.
- Metal produced by this method are Cu, Brass, Al, Zn, Sn, Ni.

Graining: Same as shooting except that the falling material through sieve is collected in water; Powders of cadmium, Bismuth, antimony are produced.

1.1.b Metal powder production (Physical method).

2.1 Electrolytic method. (التحليل الكهربائي),

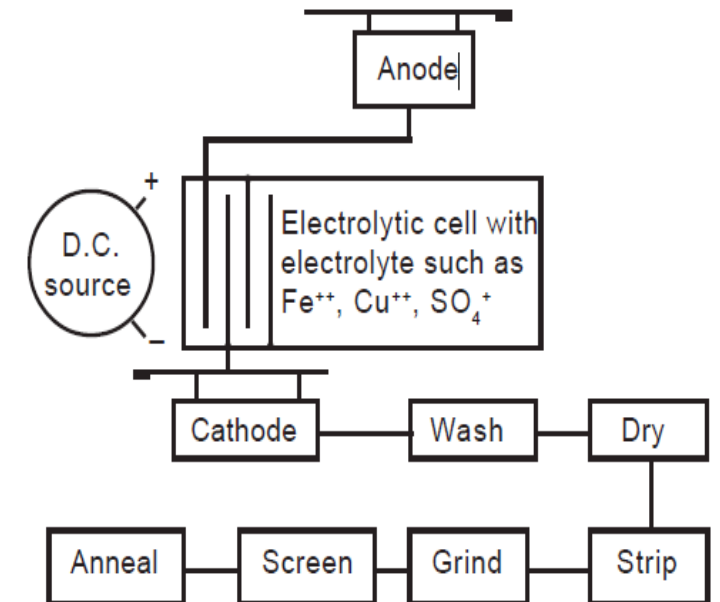
2.2 Atomization method(التحليل الذري),

1.1.b.1 Electrolytic Deposition Method.

- Condition method are based on precipitation (ترسيب) the metals of high purity from aqueous solution on the cathode of an electrolytic cell.
- This method is mainly used for producing Cu, Fe powder, Zn, Tantalum, Ni, Cadmium, antimony, silver, lead, beryllium powders.
- During electrodeposition, it possible to cause the metal to deposit loosely (بصوره طليقه حر أو متخلخل غير مضغوط) on the cathode of the cell either as a light cake or in flake form.

عملية التوافق في الظروف الكيمياوية والفيزياوية خلال عملية الترسيب الكهربائي تمهد لأمكانية أن يترسب المعدن على قطب الكاثود بصوره طليقه (لانه غير مضغوط), على شكل أما شرائح رقيقه أو رقائق مقطعه.

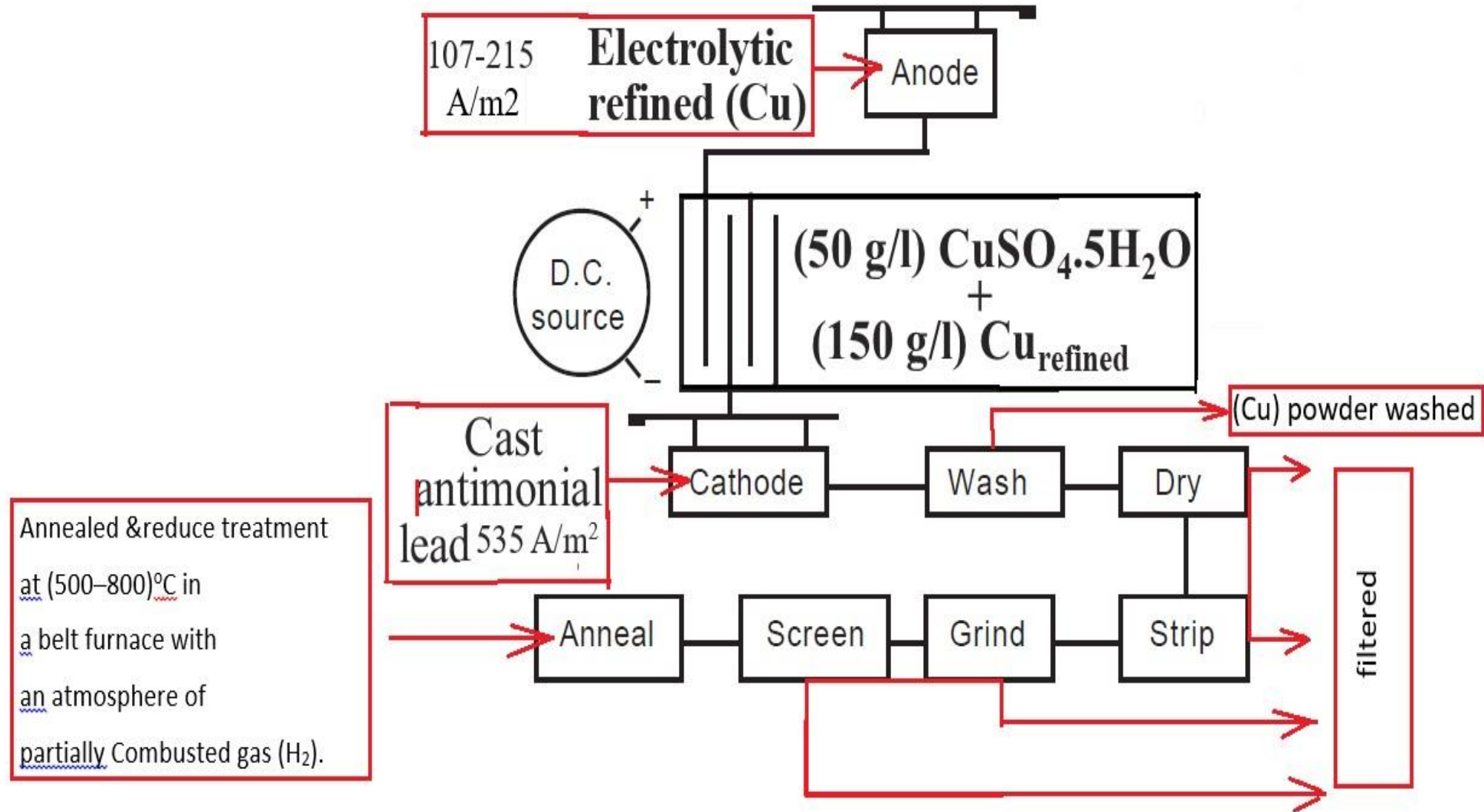
- Both (light cake or flake form) are readily crushed to a powder.
- The method yields a high purity metal with excellent properties for conventional powder metallurgy processing.



Schematic of the electrolytic process for making metal powders.

1.1.b.1 Electrolytic Deposition Method.

➤ Example 1: Copper powder, $\text{CuSO}_4 + \text{H}_2\text{SO}_4 \rightarrow$ at Anode: $\text{Cu}^- (\text{crude}) \rightarrow \text{Cu}^+ + e^-$; at Cathode: $\text{Cu}^+ + e^- \rightarrow \text{Cu}$



1.1.b.1 Electrolytic Deposition Method.

- Example 2, {first}Iron powder, **Anode**: low carbon steel [LCS]+ **Cathode**: stainless steel [SS] →
{second}the deposited Iron powder pulverized يسحق او يطحن using hammer mill. →
{third}annealing milled powder in Hydrogen (atmosphere) → Annealed (soft) Iron powder.
- **Example3**: Thorium, Tantalum, Vanadium powder →
fused salt electrolysis is carried out (محلول ملحي منصهر) at a temperature < melting point of the metal →
Deposition Results : occur in the form of small crystals with dendritic shape.



1.1.b.1.4 Factors promoting powdery deposit by electrolytic method

- (a) High current densities;
- (b) Weak metal concentrations (المعادن ذات التركيز الواطئ);
- (c) Additions of colloids and acids (بعض الإضافات الغرويه والحامضيه);
- (d) Low temperature;
- (e) High viscosities;
- (f) Avoidance of agitation (تجنب استخدام المواد الأنفعاليه);
- (g) Suppression of convection (حبس الحمل الحراري).

Advantage of electrolytic Method.

- Commonly, used in production powders of (Cu), beryllium, iron and (Ni) powders.
- The method produces metals with a high purity, excellent properties worthy for further conventional powder metallurgy processing.

Disadvantage of electrolytic Method

- More cost than other techniques. E.g., production iron powder electrolytic-ally costs higher than another atomization methods with the same characteristics.
- production electrolytic copper is competitive to that of atomized.
- Some electrolytic deposits powders are still solid even after finishing the deposition stage, as they are very reactive and brittle, so this type of material need a special annealing treatment , e.g. pulverization (تسحق), to reproduce a high purity powdery deposit, which is costly.
- A subsequent processing is applied to change the unwanted structure as the powders formed during electrolysis have a characteristic dendritic shape which is unwanted structure.

1.1.b.2 Atomization method

1.1.b.2.1 Atomization categories

Atomization Categories

(1)
Gas atomization

(2)
Water atomization

(3)
Centrifugal atomization
التذرية بطريقة الطرد المركزي

a-Liquid gas atomization

b-Vacuum atomization

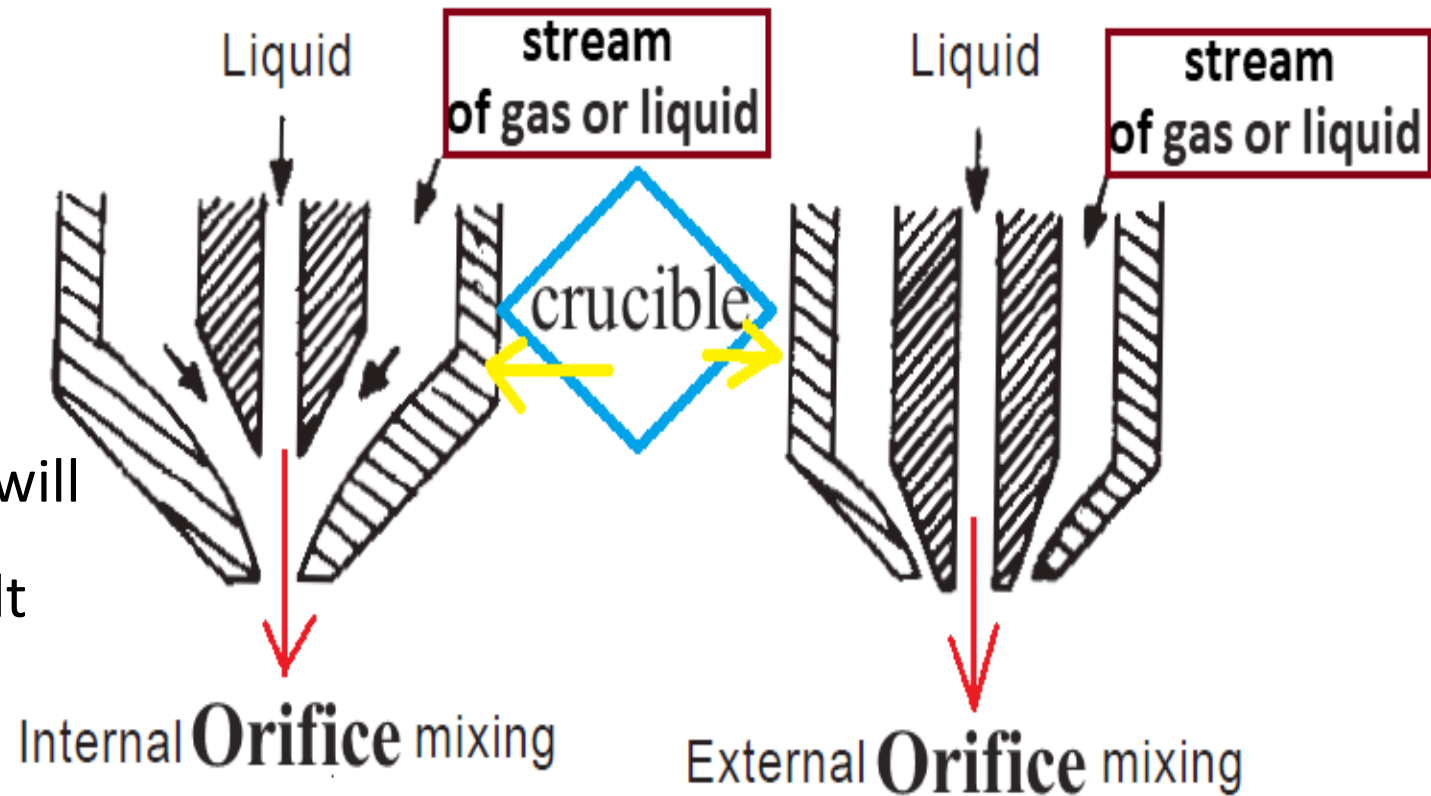
- 1- a high pressure water stream is forced through nozzles to form a disperse (مشتت) phase of droplets which then impact the metal stream.
- 2- Requires high energy to supply water. significant for low and high alloy steels,
- 3- water atomized powders are irregular in shape, with rough oxidized surfaces.

1.1.b.2.2 Atomization mechanism.

1- Pouring the melted metal into the crucible.

2- impinging اصطدام a stream of gas or liquid with the particles came out the orifice.

3- Mixing a stream of gas or liquid with the particles came out down the orifice will pulverize the disintegrated emerged melt particles .
الجزيئات المذابة المتكونه المتفتته



Two fluid atomization design

1.1.b.2.3 General steps to atomize (تذرية) a material.

(1)

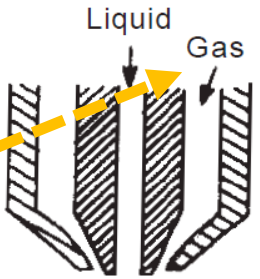
(Melting stage)

The selected material could a melt-able, = lead to (LIQUID).

(2)

[Liquid disintegration stage (تفكك السائل)]

By



Firstly - Forcing the melt-able liquid to pass through an escaped opening point (orifice). Then,

Secondly - allow Impinging (تصادم) a stream of gas or liquid on the emerging or formed (الناشئه) melted particles, lead to reduce particle's size in a very fast way directly, in the induced turbulence's manner (بطريقة الأضطراب المستحث).

Advantage of atomization Method.

1- Widely used because produces metals with a high purity, and pre-alloyed powders directly from the melt (المنصهر).

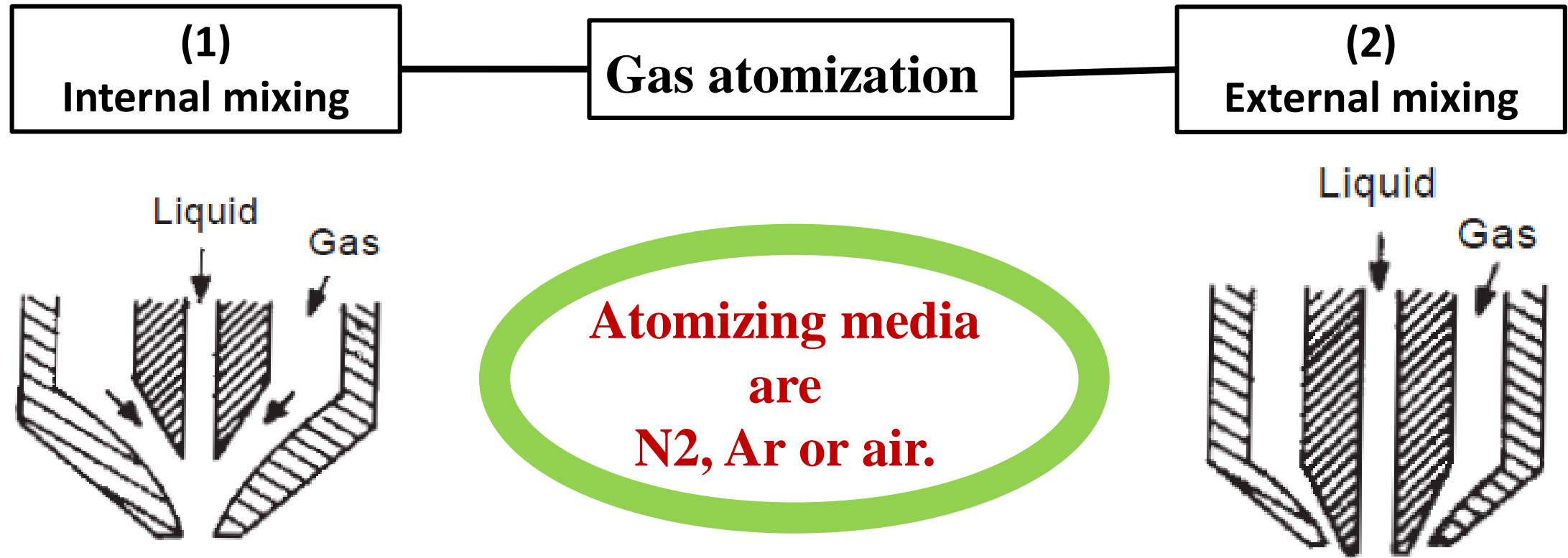
2- **An independent method**, does not affected by the normal physical and mechanical properties associated with the base solid material, regardless of chemical reactivity, which may necessitate specific atmosphere or materials.

طريقه مستقله: لاتتأثر بالخواص الفيزيائويه والكيميائويه الطبيعيه للماده الأساس, بغض النظر عن تأثير التفاعلات الكيميائويه التي تستلزم وسط أو ماده معينه.

Disadvantage of atomization Method

➤ **Orifice's design (تصميم الفوهه)**: it described as an **escaped opening point (فتحه تسريب)** that is necessary to locate in the crucible, in the way help forcing the melt particles come out it and Impinge the stream of gas or liquid during atomization process. So, selection the exact design for its location is of great deal to get successful achievement. See Figure (). Best selected location at the bottom of the crucible.

1.1.b.2.5 Gas atomization.



a- contact between the atomizing medium and melt takes place inside the respective nozzles.

b- used for atomization of materials which are liquid at room temperature.

a- contact between the atomizing medium and melt takes place outside the respective nozzles.

b- used for atomization of metals.

1.1.b.2.5.1 Operating parameters govern gas atomization process .

- 1) jet distance,
- 2) jet pressure,
- 3) Nozzle geometry,
- 4) Velocity of gas and metal, and
- 5) Melt superheat.

Advantage of gas atomization

- Used for preparing powders of the super-alloys, titanium, high speed steel and other reactive metals.
- Gas atomized powders are typically spherical(which is needed for applications where high bulk densities and flow rates are required), with relatively smooth surfaces, with higher pressure and/or a smaller jet distance produce finer powder.

Disadvantage of gas atomization

- The method suffers from a very low overall energy efficiency.
- An expensive type if inert gases other than nitrogen have to be used.
- Atomization by inert gases is used to produce powders from oxygen-sensitive elements in case of water atomization is not applicable due to the reactivity of the metals.

1.1.b.2.6 Water Atomization

1- a high pressure water stream is forced through nozzles to form a disperse phase of droplets which then impact the metal stream.

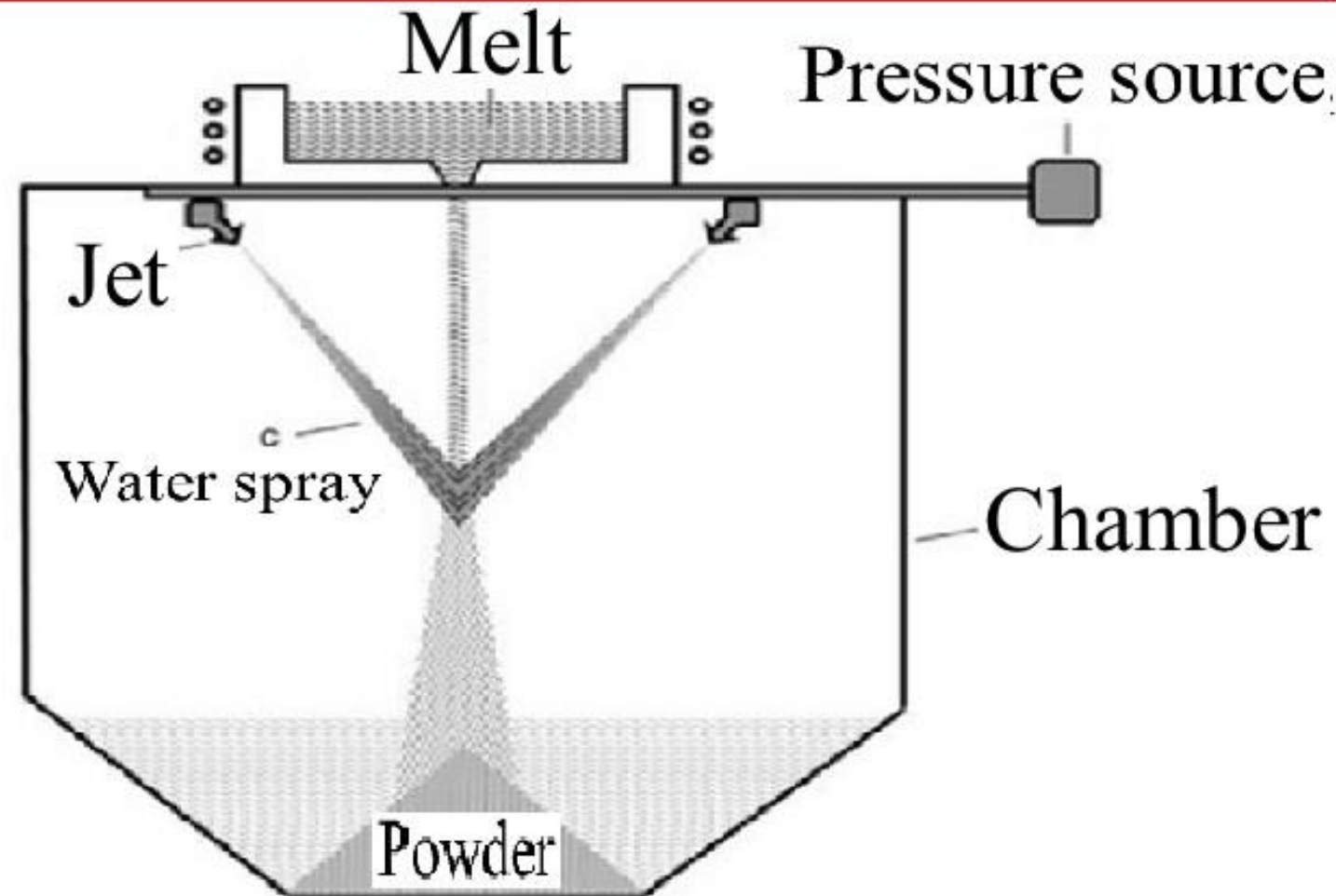
2- Requires high energy to supply water. significant for low and high alloy steels,

3- water atomized powders are irregular in shape, with rough oxidized surfaces.

4- The method can be used to produce iron and steel powders (alloyed with Mo, Ni, or Cr), stainless steel powders, copper, and copper alloys.

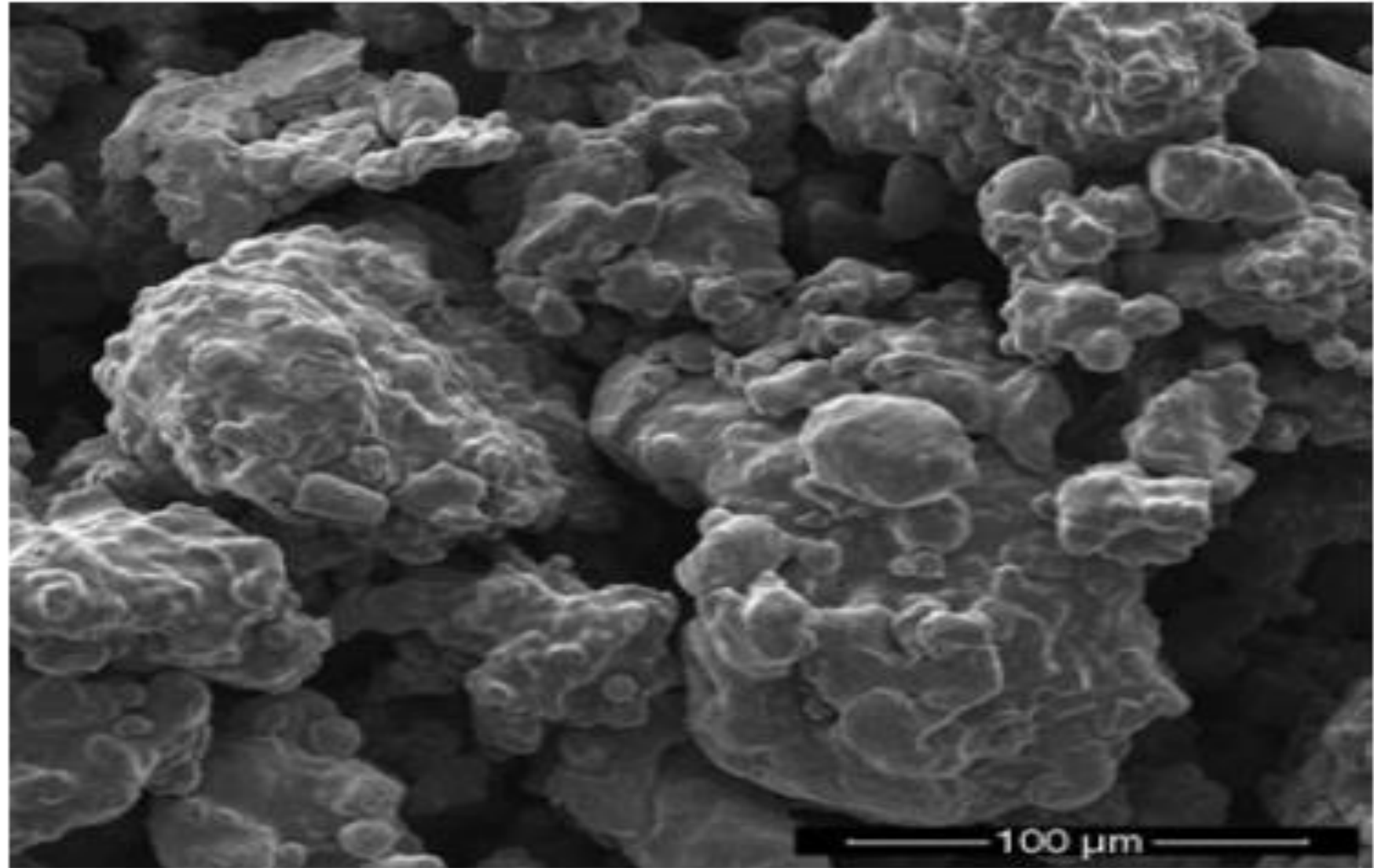
5- The final powders have an irregular shape that provides good green strength by interlocking تشابك of the particles.

Water atomization Principle arrangement of a water atomizer



1.1.b.2.6 Water Atomization

5- The final powders have an irregular shape that provides good green strength by interlocking of the particles.



Typical water-atomized powder.

1.1.b.2.6 Water Atomization

Water atomization Principle arrangement of a water atomizer

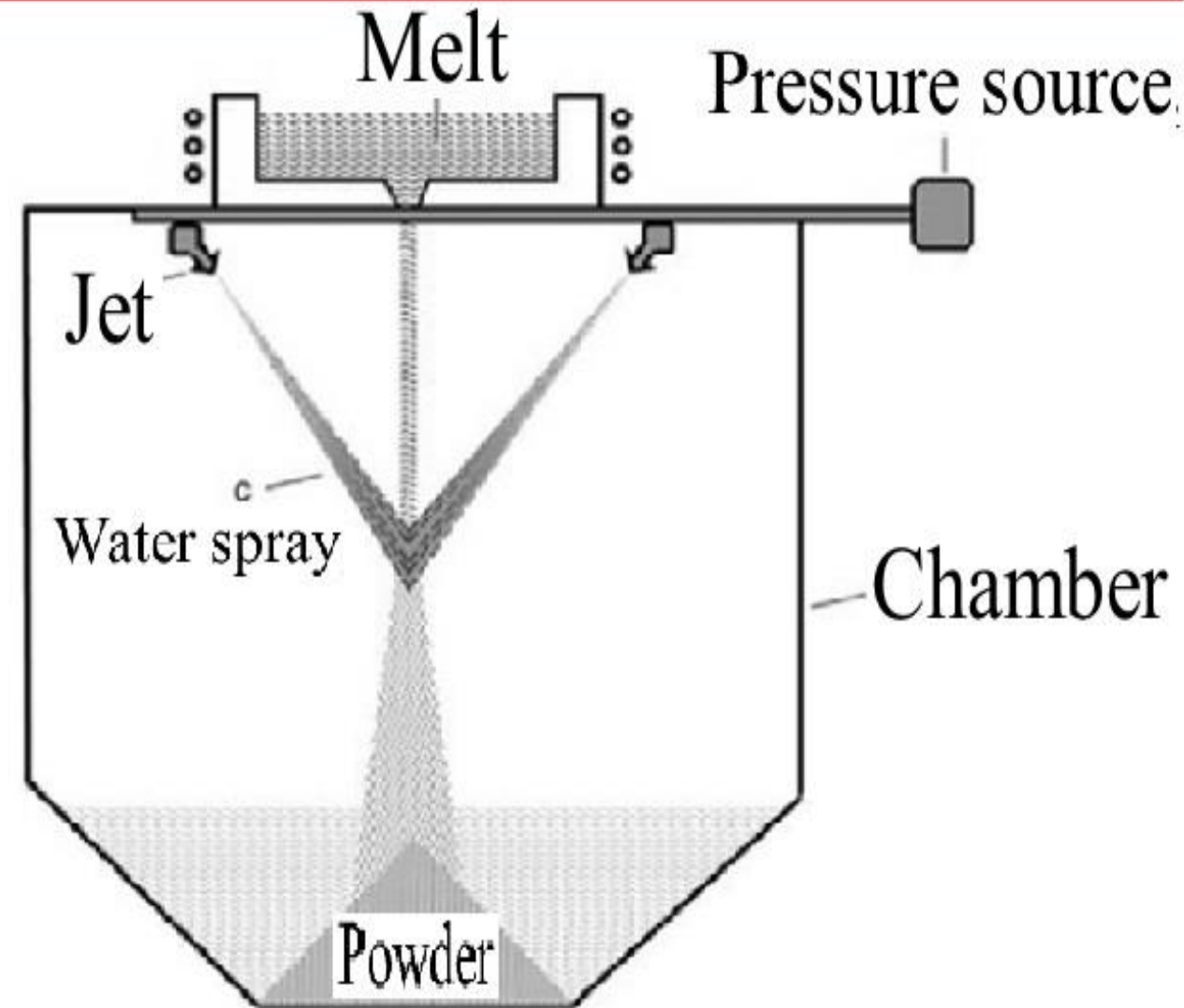
6- The process involves many variables:

a- Temperature and amount of superheated molten material (related to the composition),

b- $\frac{\text{water}}{\text{metal}} = \frac{10}{15}L/(1Kg \text{ of Produced water}),$

c- Diameter of the molten-metal stream,

d- Geometry of the nozzle (water jets amount, Incidence 'angle between water jet and molten metal stream, and water pressure.



Chemical method

Metal powder production

- 1- Chemical reduction method (طريقة الأختزال الكيميائي)
- 2- Chemical de-composition of compounds method
(طريقة التفكيك أو التحليل الكيميائي للمركبات)

1- Chemical reduction method.

- It is one of the methods to make powder metals, involves melting the chemical compound during melting process using a solid or gaseous reducing agent (chemical compounds).

هي إحدى الطرق المستخدمة لتصنيع مسحوق معدني، تتضمن ذوبان المركب الكيميائي باستخدام عامل مساعد للأختزال غازي أو صلب (مركب كيميائي).

- The solid or gaseous reducing agent (chemical compounds) are represented by using:

An oxide;

Carbonates;

Nitrates;

A halide [halogenides with gases (e.g. H₂) or solids (e.g. {C} or highly reactive

metals) (هاليدات: مركب كيميائي من الهالوجين مع عنصر أو مجموعه ذات شحنة كهربائية موجبه اكثر من غيرها)], or

Other salt of the metal.

Chemical reduction method *Cont.*

- During the process, the solid phase is deposited while all the metal contaminations with slag is removed. However, the obtained metal powder needs further refinement by mechanical, magnetic, or chemical procedures.

خلال عملية الذوبان, الطور الصلب المراد استخلاصه يترسب, بنفس الوقت الذي يتم إزالة الخبث والشوائب. على أية حال, يحتاج مسحوق المعدن المستحصل فيما بعد الى عمليات تنقيته أما بطرق ميكانيكية أو مغناطيسية, أو كيميائية.

- In most cases the metal compounds to be reduced are in the solid state provided as a feedstock (ماده خام تستخدم في عمليات التصنيع).

في الأغلب, المركبات المعدنية المراد أختزالها تكون عادة بالحاله الصلبه.

However, hydrometallurgical processes have been developed also, especially for the reduction of nickel and cobalt solutions by pressurized hydrogen.

توجد معالجات هيدروميتالورجية مطوره لأختزال النيكل ومحلوا الكوبلت بطريقة الهيدروجين المضغوط.

Chemical reduction method.

Principle aspects.

- For kinetic reasons, the compound to be reduced has to have a suitably small particle size, which should not be exceeded otherwise the controlling diffusion processes may lead to very long reaction times which are not acceptable in practice.

لأسباب ديناميكية, المركب الكيميائي المراد أختراله لابد أن يمتلك ذرات صغيره مناسبه والتي **يجب** أن لايتجاوز حجمها, وألا سيؤدي الى أختلال في عمليات الأنتشار وبالتالي أطالة الوقت المطلوب للفاعل والذي هو غير مقبول بالتطبيق المطلوب.

Chemical reduction method.

Principle aspects (Cont.).

- The process is controlled by its free energy of reaction which has to be negative. The more stable the compound, the stronger the reduction media have to be.

عملية الأختزال تتم السيطرة عليها من خلال طاقتها الحرة للتفاعل والتي يجب أن تكون سالبة. فكلما كان المركب الكيميائي مستقرا كلما تحفز الوسط الكيميائي الذي يحدث فيه الأختزال ويصبح أكثر سيطرة.

- For example: alkali (القلويات), and especially alkaline earth metals (معادن الأرض القلوية) form a very stable oxides, so they are very strong reducing agents (معاملات أختزال).

Chemical reduction method.

Reducing agents*(Cont.)*

Chemical reduction method is carried out by involve chemical compounds (مركبات كيميائية) to make powder metals, through using the following types of reducing agents (Figure below):

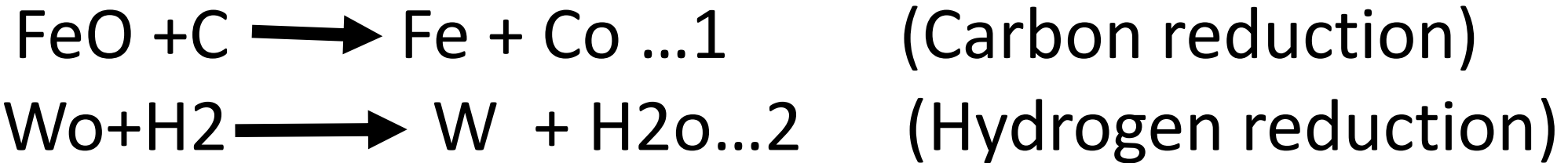
1. From the solid state (Oxide, Carbonate, and Hydrogen reduction).
2. From the aqueous solution (Precipitation).
3. From the gaseous state.

Reducing agents*(Cont.)*

1. From the solid state

a- Oxide reduction

As in the reduction of iron oxide with carbon (Eq. 1) or of tungsten oxide with hydrogen (Eq.2).



كما هو الحال في عملية أختزال أوكسيد الحديد مع الكربون للحصول على مسحوق راسب الحديد, أو أختزال أوكسيد التنكستن مع الهيدروجين للحصول على مسحوق راسب التنكستن.

This is a convenient, economical and extremely flexible method for controlling the properties of the product regarding size, shape and porosity over a wide range. The production of iron, copper, tungsten, and molybdenum powders from their respective oxides are well-established commercial processes.

From the solid state.

b- Carbon reduction

1) For example, the pure magnetic iron ore (Fe_3O_4 : خامات أكاسيد الحديد) is reduced using a carbonaceous material to produce sponge iron powder in **Höganäs process**.

2) The ore is ground to a particle size distribution determined by each of the desired iron.

3) The ore powder is placed in the center of cylindrical ceramic containers ('saggers' made of silicon carbide) surrounded on the outside by a concentric layer of a mixture of coke and limestone (CaCO_3).

مسحوق الخام يوضع في مركز حاويه سيراميكيه أسطوانيه (غطاء حام من الصلصال الحراري والمصنوع من كربيد السيليكون) محاطه من الخارج بطبقة كونكريت كخليط من الفحم وحجر الكلس (يستخدم في البناء وصناعة الجير).

4) The saggers (غطاء حام (من الصلصال الحراري) are placed in layers upon cars which are pushed through a fuel fired tunnel kiln (فرن نفق يعمل بالوقود).

6- The limestone (CaCO_3) serves to bind any sulphur in the coke and prevents its contaminating the iron

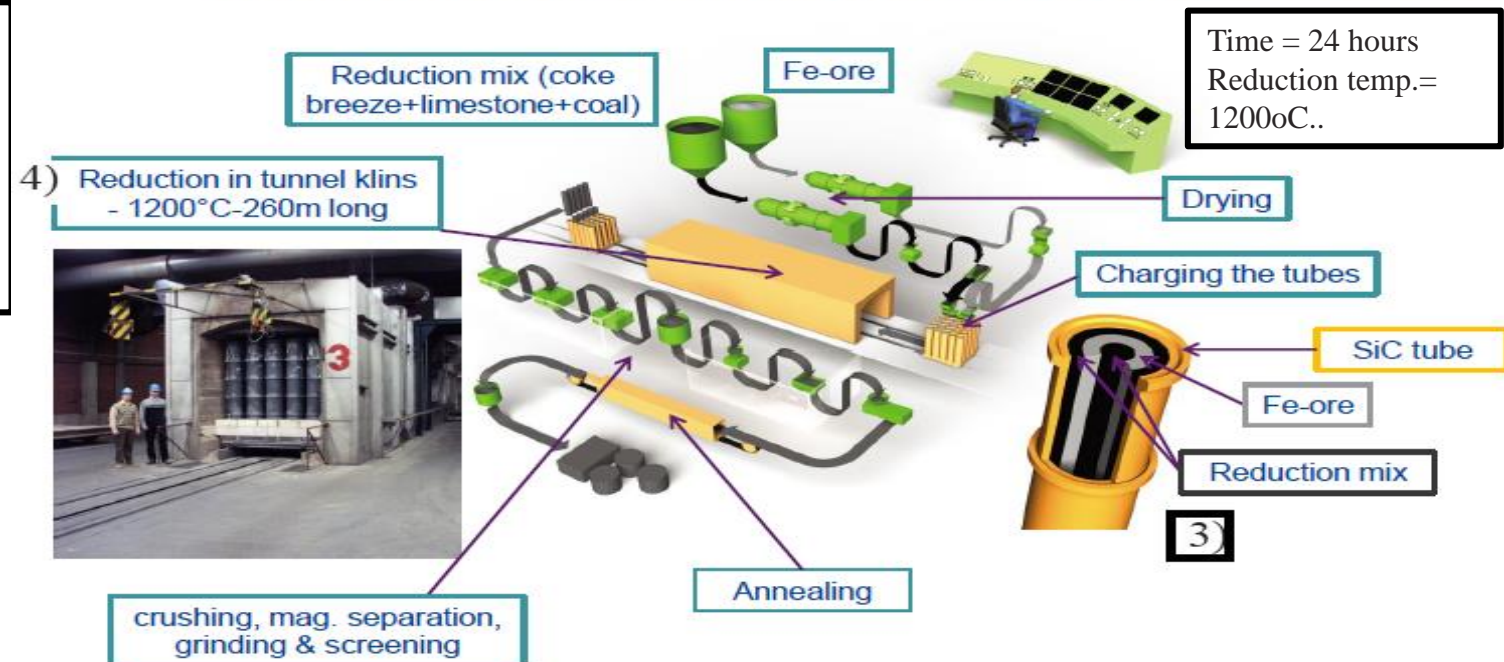
5) The carbon monoxide produced from the coke reduces the ore to iron.



7- The sponge iron is mechanically removed from the saggers, ground.

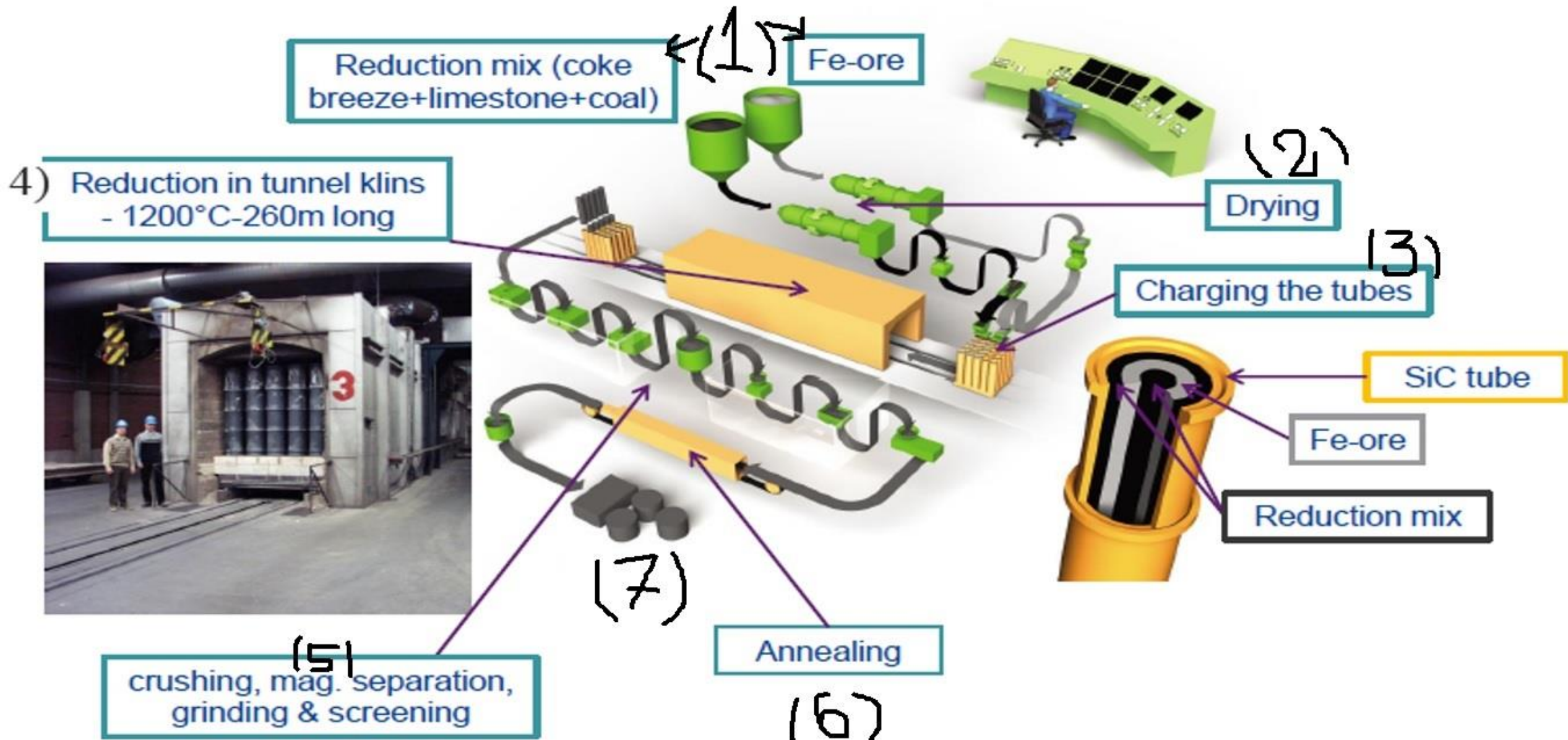
The Sponge Iron Process

Direct reduction of iron ore (magnetite) Fe_3O_4 (Höganäs process)



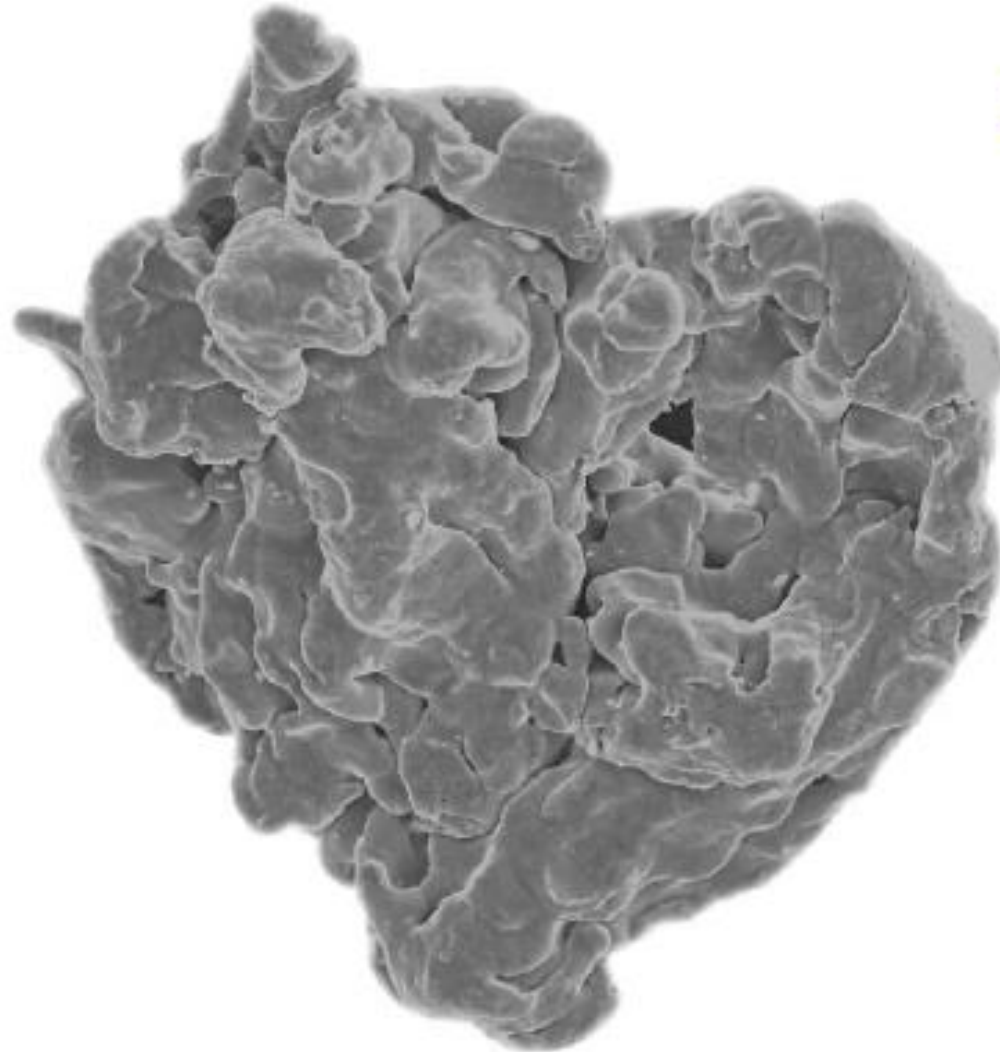
The Sponge Iron Process [From the solid state]

Direct reduction of iron ore (magnetite) Fe_3O_4 (Höganäs process)



From the solid state.

Carbon reduction



Sponge Fe-powder

Due to the reduction processes the powder has a «spongy» appearance – high internal porosity

From the solid state. $W_o + H_2 \longrightarrow W + H_2o \dots 2$ (Hydrogen reduction)

C- Hydrogen reduction

- 1- The (hydrogen reduction process) is undertaken in tube furnaces.
- 2- Generally, the (hydrogen reduction process) is undertaken well below the melting temperature (M) of the metal.
- 3- The (hydrogen reduction process), includes hydrogen + other compounds to reduction of oxides (Eq.2).
- 4- Very pure and fine powders can be obtained.
- 6- Examples are refractory metals (المعادن الحراريه) such as tungsten and molybdenum, ferrous metals and copper, which form compounds with only moderate stability.

Reducing agents_(Cont.)

2- From the aqueous solution (Precipitation).

- Precipitating principals of a metal from its aqueous solution is achieved by adding a less noble metal (معدن نبيل) which is higher in the electromotive series by applying it in numerous metallurgical processes.
- For example, silver (Ag) powder is produced in quantity from its nitride solution (AgN) by adding (Cu) or (Fe) depending on reaction. See the chemical reaction below:



Reducing agents_(Cont.).

From the aqueous solution (Precipitation).

- This method is used for producing metal powders of Ag, Sn, Pt and iron particles coated with copper.
- Precipitation is also synonymous with the term electrolytic precipitation to coat metals with a corrosion resistance film.
- The produced powder take the form of spongy mass which crashed into a hard and brittle powder.

Metal powder production (Chemical method)

2- Chemical de-composition of compounds method (Hydrides)

- There are two methods are very common in this category, (i) Decomposition of metal **hydrides** (ii) Decomposition of metal **carbonyls**.
- Decomposition of metal **hydrides** involves (a) hydriding; and (b) de-hydriding.
- Hydriding means heating the refractory metals e.g. (Ti, Zr, V, Th or U) in the form of sponge, chip or turnings (بقايا معدن ناتج من مخرطه - كشط) or even compact metal **in hydrogen**.
- For example, TiH_2 (Hydride titanium) is formed from titanium in the temperature range between $(300\text{--}500)^\circ\text{C}$. These hydrides are quite brittle and can be easily ball-milled into powder of the desired fineness.
- De- hydriding means heating the metallic hydrid, in a good vacuum at the same temperature at which the hydrid was formed. Example, heating (TiH_2) in the range between $(300\text{--}500)^\circ\text{C}$, in order to recover properties of (TiH_2) by remove brittleness.

Metal powder production (Chemical method)

Chemical de-composition of compounds method (Carbonyls)

- Decomposition of metal **carbonyls**: for example iron and nickel powder production.
- The carbonyls are liquids at normal temperature with a low boiling point.
- These are formed by reaction of the metal and carbon-monoxide gas under pressure.
- For example, iron carbonyl ($\text{Fe}(\text{CO})_5$) is formed at (70–200) atmosphere pressure and a temperature of (200–220) °C.
- The carbonyls can now be decomposed by heating the vapour at atmospheric pressure (70-200).

Powder Characterization.

Characterization powder properties properly achieved by applying the suitable methods for characterization, because the powder particles properties depend largely on the physical, chemical and mechanical properties of the initial metal powder. There are various methods to calculate average diameters of particles based on understanding the role of powder properties in the powder metallurgy process, as below:

- (1) Physically, by consideration (Particle size & Shape), using Particle size measurement techniques
- (2) Technological Properties (apparent density & tap density, Flaw ability, Segregation, compressibility).
- (3) Chemical properties, by consideration (Chemical composition, phase distribution).

Powder Characterization:

(1) Physically, by consideration (Particle size & Shape)

- Majority of metal powders employed in powder metallurgy industry vary in size from 4 to 200 microns.
- In practical P/M, metal powders are divided into three distinct classes: Sieve (غربال او مصفى), Sub-sieve (شبه غربال), Sub-micron (or ultrafine) (غربال شبه مايكروني).
- **Sieve size powders** are used for most ordinary mass production because of their good flow ability and lack of further processing requirement such as granulation (تحويل الى حبيبات).
- **Sub- sieve particles** are smaller than the aperture فتحة of such a screen but greater than 1μ . This class of powder is used for the production of refractory metals, hard carbides and magnetic cores.
- **The sub- micron powder** particle size is smaller than 1μ and is used for the manufacture of dispersion (خاصية الانتشار او التشتت) strengthened high temperature alloy (السبائك المصلده عالية الحرارة), bearing and micro porous components (المركبات ذات المساميه المايكرونيه), magnetic materials (المواد المغناطيسييه), nuclear reactor fuels (الوقود الفعال) (النووي).

1.1.d Powder Characterization

Particle size measurement techniques.

Q- Why sieving is the favored method for determination powder particles?

Sieving (غربله):

- widespread use of this method.
- Most satisfactory for plotting particle size distribution, in which the successive sizes form a geometrical series.
- Distinguishes between particles which are larger than 44 micrometers and smaller than 44 micrometers.

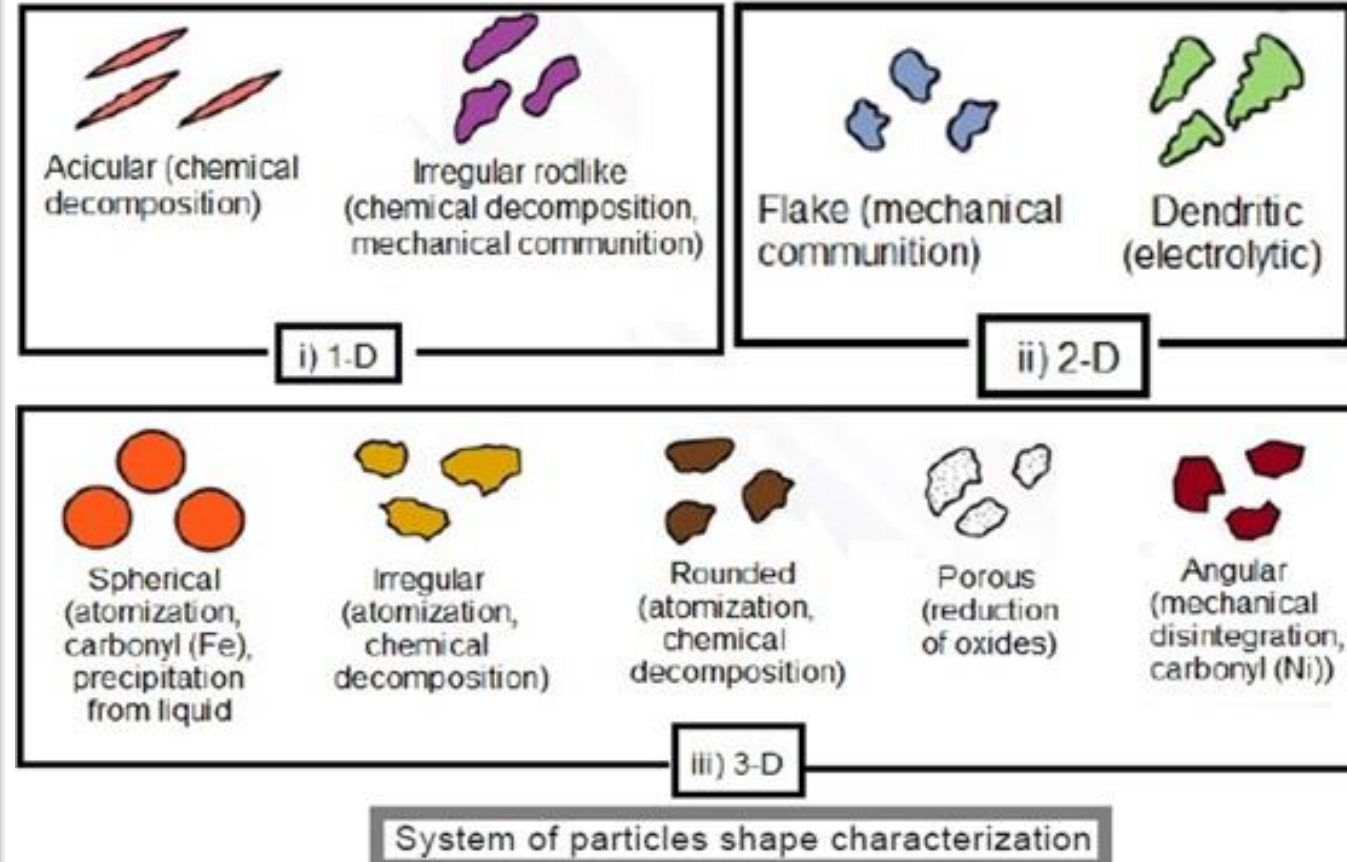
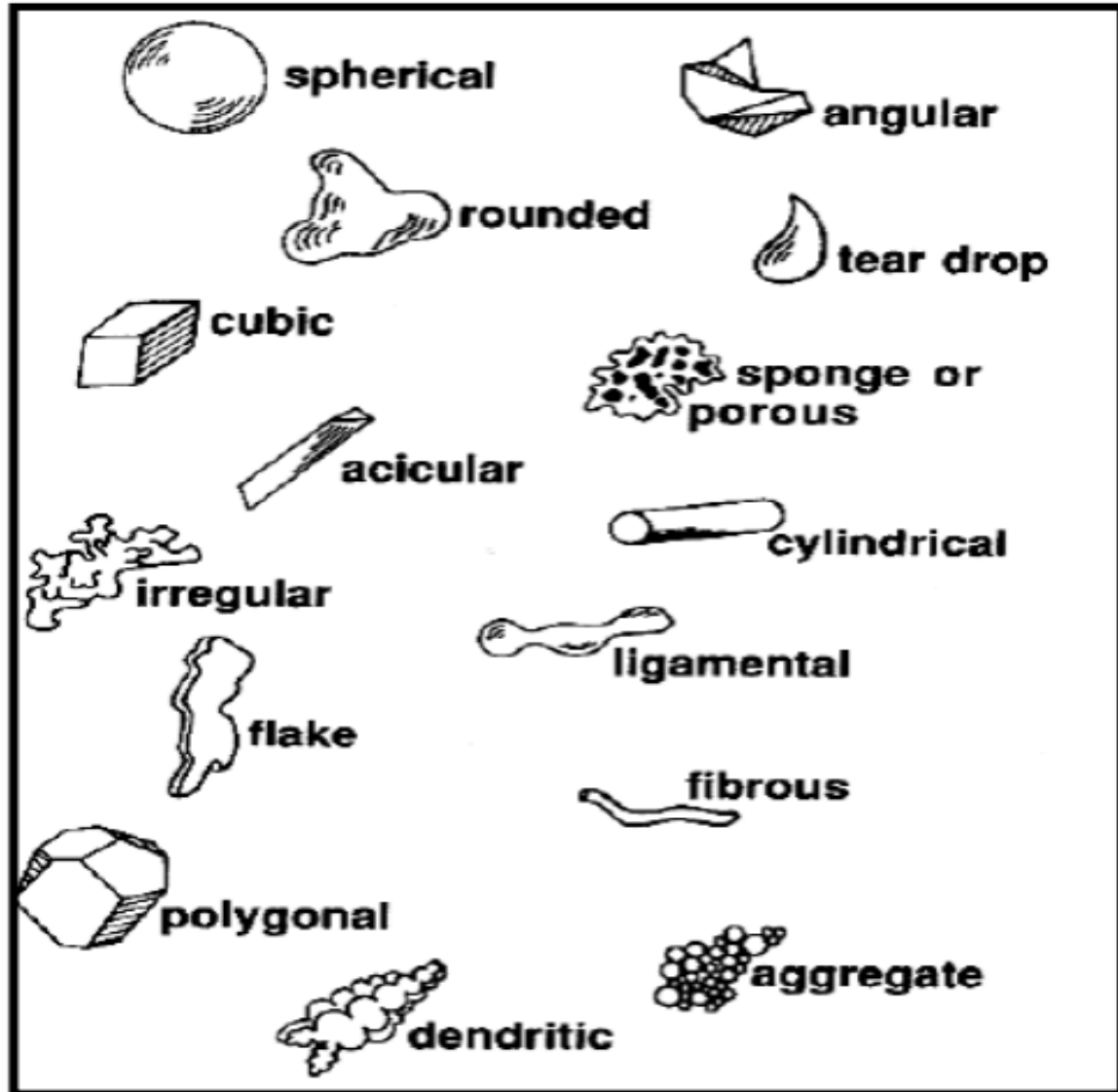
Table Common particle size determination methods and their limits of applicability²

Class	Method	Approximate useful size range (microns)
Sieving	Sieving using mechanical agitation or ultrasonic induced agitation and screens	44–800
Microscopy	Micromesh screens	5–50
	Visible light	0.2–100
	Electron microscopy	0.001–5
Sedimentation	Gravitational	1–250
	Centrifugal	0.05–60
Turbidimetry	Turbidimetry (light intensity attenuation measurements)	0.05–500
Elutriation	Elutriation	5–50
Electrolytic resistivity	Coulter counter	0.5–800
Permeability	Fisher sub-sieve sizer	0.2–50
Surface area	Adsorption from gas phase	0.01–20
	Adsorption from liquid phase	0.01–50

Table shows classifies some of the common methods of particle size determination and their limits of applicability.

Powder Characterization (Particle Size & Shape):

➤ Naturally, most powder particles are A (3-D) shape with different shapes and sizes. See the pictures.



Powder Characterization:

(1) Physically, by consideration (Particle size & Shape)

- **Particle sizes and shapes** are considered the basic characteristics of metal powder, such as the contour of the surfaces' particles (محيط سطح الجزيئه) also their distribution, purity, porosity and their chemical compositions.
- **Particle size definition** according to the **sedimentation** (ترسيب) method is defined as the diameter of the spherical particle (كرويّه) having the same specific gravity (ثقل او جاذبيه نوعيه) and settling velocity (سرعة الترسيب) as the non-spherical particle under test. (هو أن يمتلك قطر الحبيبه الكروية الشكل جاذبيه نوعيه وسرعة ترسيب مساوي لما في الحبيبه الغير الكرويّه) (الشكل)
- For example, the simplest and ideal particle's shape represents the (Equiaxed Spherical). (أبسط مثال هو شكل الحبيبه) (المثالي المتساوي المحاور) In this case, the calculation sizing method includes [Average. values of particle size], is a concise quantity for any given spherical particle, which may have several values with same shape, في هذه الحالة, طريقة الحساب بأخذ المعدل الحجمي للحبيبات, هي كميّه مناسبه لأي حبيبه كروية الشكل, والتي لها قيم متعدده لنفس الشكل.

Powder Characterization:

(1) Physically, by consideration (Particle size & Shape)

- When the method involves sizing, **the particle size** is measured as the opening of a standard screen which just retains or passes the particle. (هنا حجم الحبيبه يقاس بحجم الفتحة للمصفى التي تم اجتيازها من قبل الحبيبه)
- The average diameter in the case of **large particle size** can be determined by counting and weighing as the cube root of the volume. (معدل القطر للاحجام الكبيره يمكن حسابه بوزنه عن طريق الجذر التكعيبي للحجم)
- The average diameter in the case of **micro count method** can be determined by averaging several dimensions. (معدل القطر في طريقة العد المايكروبي يمكن حسابه عن طريق المعدل الحسابي لعدة ابعاد)
- Note that the average diameter is defined in different ways according to the method employed for size distribution. (معدل القطر يعرف بطرق مختلفه بناءا على الطريقه المطبقه لتوزيع الحجم)

Powder Characterization/ (1) Physically, by consideration (Particle size & Shape):

- Using particle size distribution method (التوزيع الحجمي للحبيبات)/the method employed for size distribution.

(a) It is the most accurate and a completed characterization method to describe powder materials. This is because in reality, all material's powder are prepared in same procedure, so all particles would have nearly a same looking shape, but not exactly same size

(الطريقة الأكثر دقة بسبب ان عملية تحضير المسحوق المادة تتم بنفس النسق فتؤدي الى ان يكون حبيبات المسحوق لها تقريبا نفس الشكل ولكنها مختلفة بالحجم).

(b) Particle size distribution curves relate the particle size to the corresponding fraction of the powder with that size.

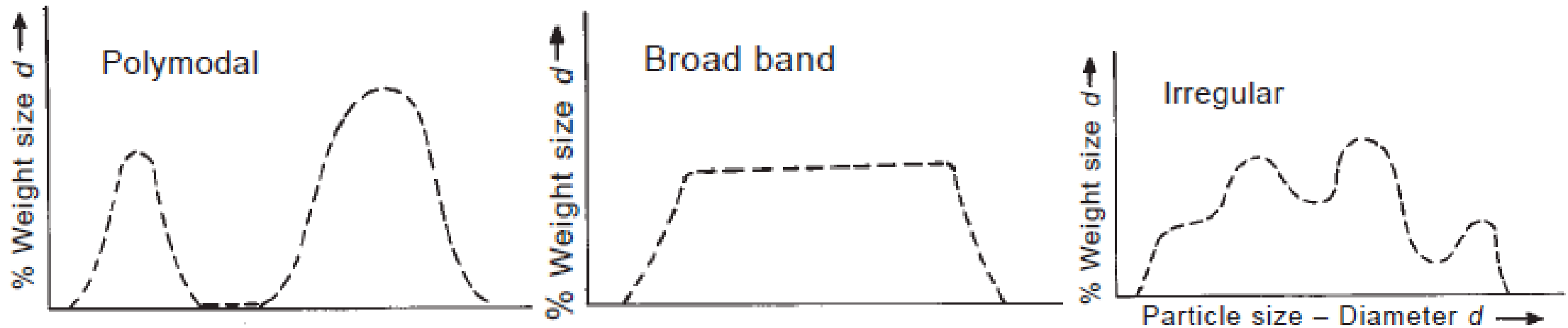


Figure below illustrates various size distributions

B- b- Using particle size distribution method: The curve has different sizes as below:

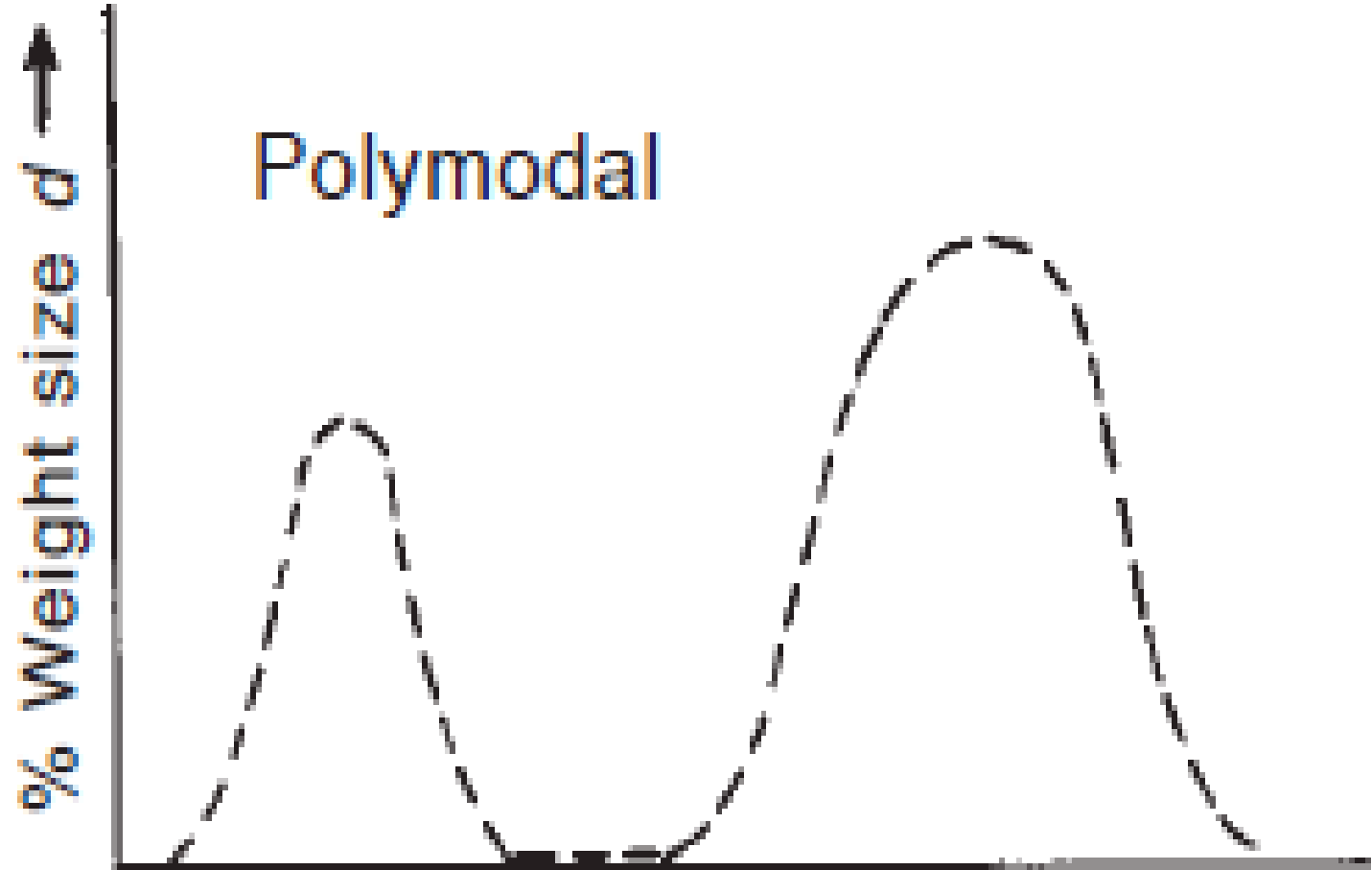
- **The poly-modal distribution** (التوزيع المتعدد الأشكال) consists of two or more narrow bands of particle sizes, each with a maximum, with virtually no particles between such band.

حبيبات منفردة أو منفصلة خلال الأشرطة الضيقة لأحجام الحبيبات, كل شريط له قمة عليا, ولاتوجد أي التوزيع المتعدد الأشكال يحتوي على اثنين أو أكثر من الأشرطة

Note:

In unimodal distribution (التوزيع الأحادي الشكل), there is one high point or maximum amount of a certain critical size.

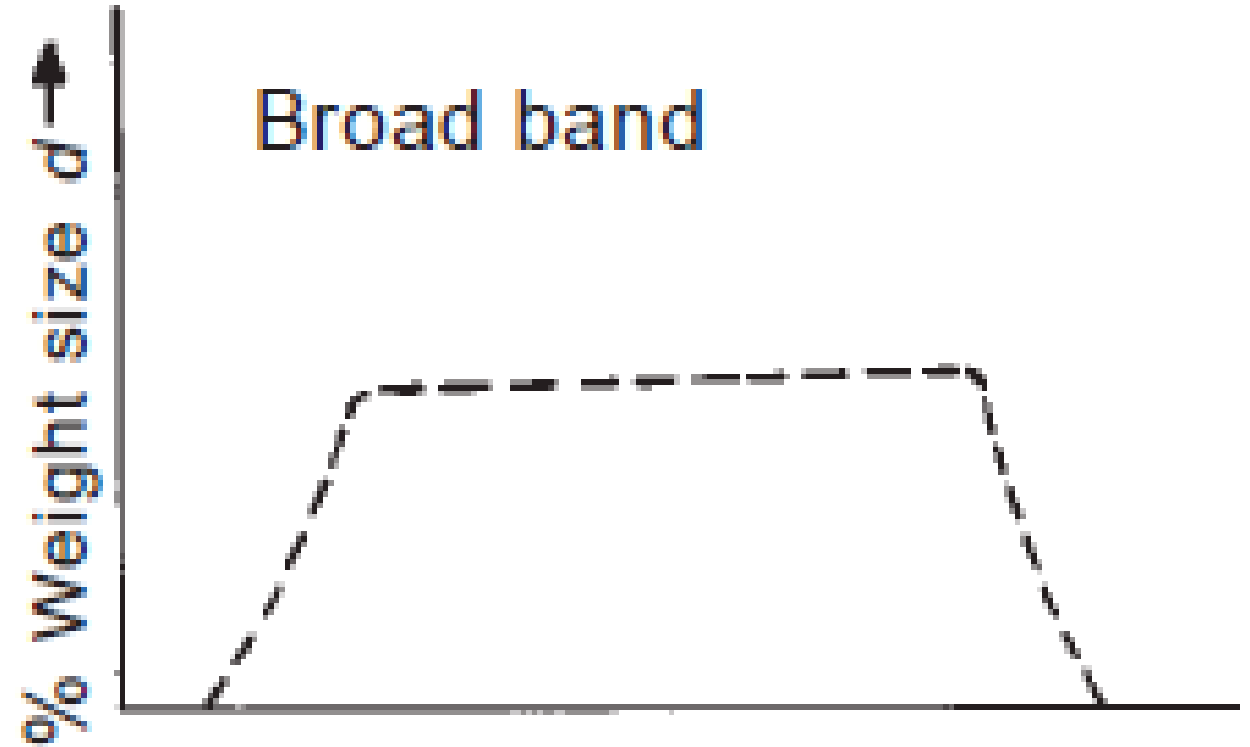
(توجد نقطه واحده عاليه أو أكبر قيمه لحجم معين حرج)



B- b- Using particle size distribution method: The curve has different sizes as below:

- **The broad band distribution** (التوزيع الواسع): represents a uniform concentration of particle sizes over a broad size of interval with practically no particles having sizes outside this range.

يمثل تركيز حجم متجانس ومتقارب من
حجم حبيبات معينه على طول مسافه واسعه
النطاق، وأي حبيبة لها حجم أصغر أو أكبر
تكون خارج هذا النطاق.



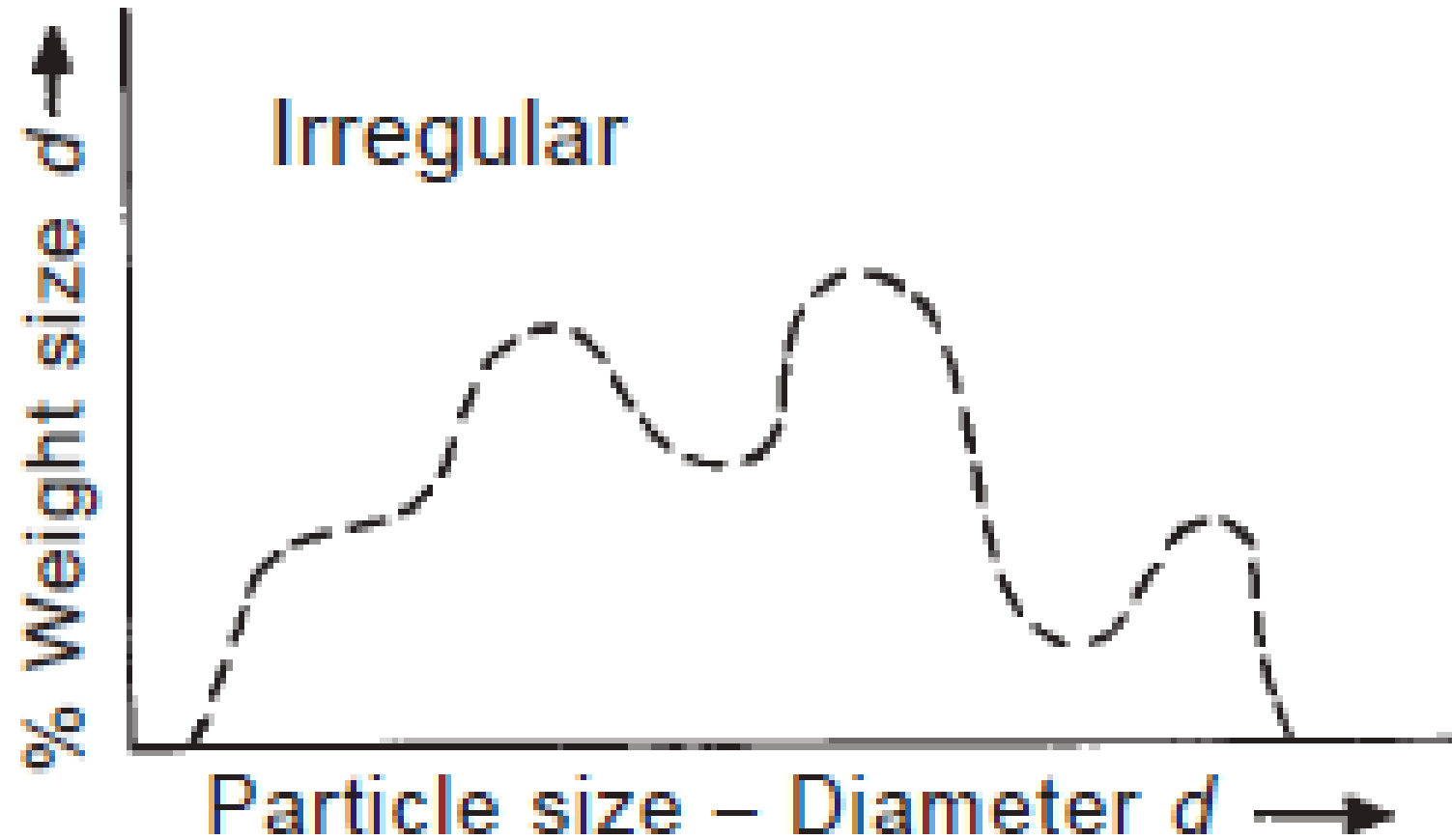
B- b- Using particle size distribution method: The curve has different sizes as below:

- **The irregular distribution** (التوزيع الغير منتظم): represents a continuous and finite variation of particle sizes within a relatively broad range.

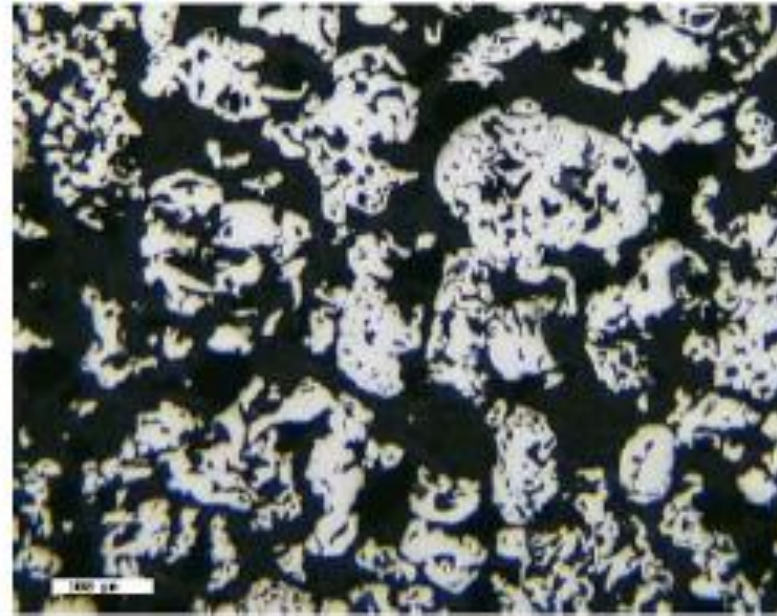
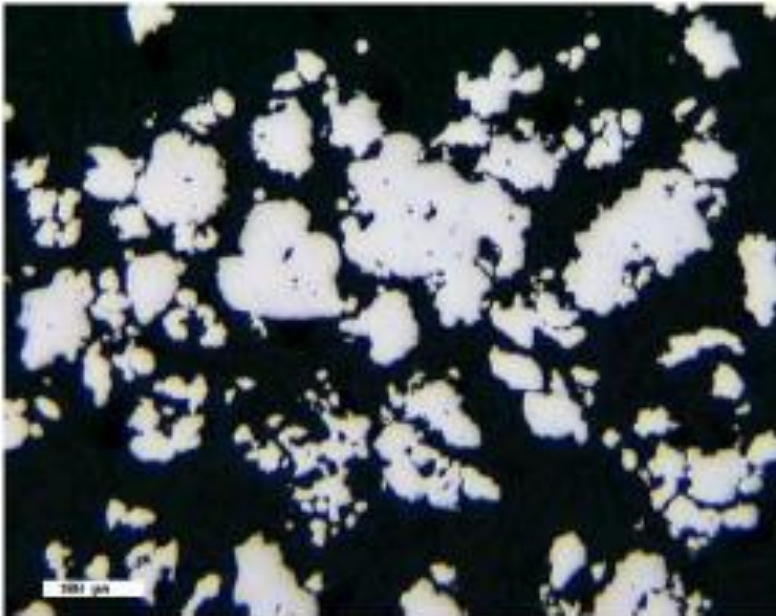
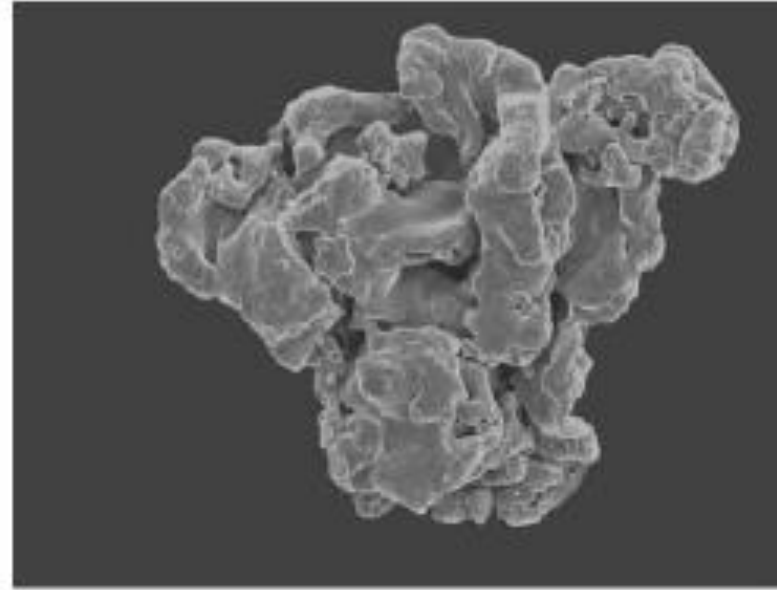
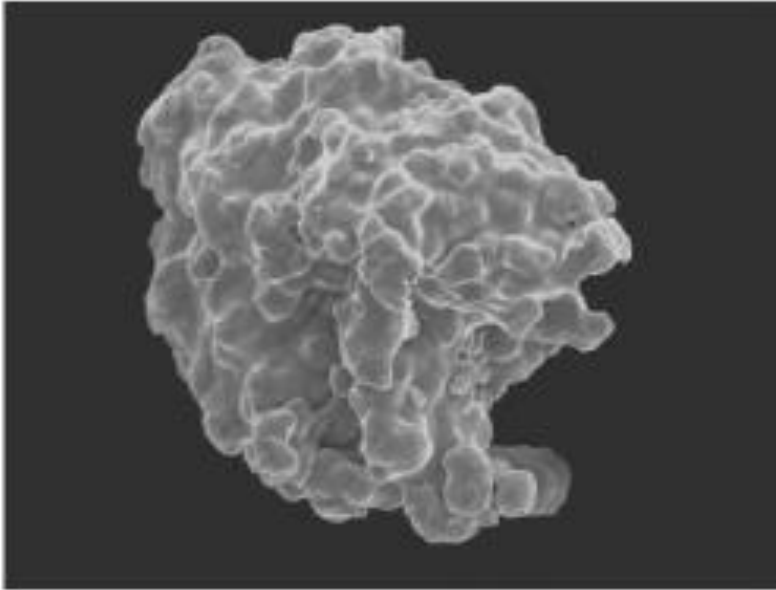
يمثل مدى عريض نسبيا من الأحجام الجزيئية
المستمره الغير متشابهه والتي ذات تنوع
محدود.

Note:

- Porous particles differ from irregular, due to porosity's presence, which itself may be very irregular in both size and shape.
- A large amount of porosity makes any shape characterization very difficult.



B- b- Using particle size distribution method:the curve could be applied at different sizes and shapes as below:



Powder Characterization.

(2) Technological Properties.

- The other characteristics which are dependent entirely on the above primary properties of metal powders: ,
Apparent density, Tap density, Specific surface , Flow rate, Compacting, Sintering
- **The apparent density of a powder** is defined as the mass of the particles divided by (per) unit volume of loose or unpacked (غير المضغوط) powder, usually expressed in g/cm^3 . Thus it includes internal pores but excludes (تستبعد) external pores. It is governed by chemical composition, particle shape, size, size distribution, method of manufacture of metal powders as well as surface conditions which can vary from 20-50% of the theoretical density.
- It is influenced by the lubricant/mix and is an indicator for filling the tool's depth (تتأثر بتماسك المزيج وهي مؤشر لمقدار (امتلاء عمق العده).
- It is a critical feature, because It specifies the type and size of tooling's necessary in compaction, and sintering processes later on, affecting either positively or negatively on the other characteristics of the powders, which have direct bearing on it (apparent density), e.g. density of the solid material, particle size and shape, surface area (m^2/gm), topography and its distribution.

Powder Characterization.

(2) Technological Properties.

Green density: the strength of the green compacted powder mainly depends on consolidation pressure (الضغط المحبوس), and increase from mechanical interlocking (التشابك الميكانيكي) neighboring particle and shearing particles. It could be improved by using soft irregular shaped particles with cleaned surfaces as the particle's shape and structure have a great effect on the properties of green strength, as well as, by employing regularly annealed smooth shaped particles with high densities (possessing no internal or interconnected porosity). In general, the green density increases with increasing particle's size, apparent density, or compaction pressure, and with decreasing particle's hardness, strength and compaction speed.

Powder Characterization, Technological Properties (Flaw ability).



Powder Characterization, Technological Properties (Flow ability).

- **Flow rate (Time /50g):** is the time required for **50g** of dry powder to pass the aperture (ثغره) of a standardized funnel (قمع) Influenced by the lubricant/mix and is an indicator for the tool filling rate (معدل أمتلاء العده) and consequently the productivity of the press (أنتاجية المكبس).
- The flow-ability is proportional with flow rate. The flow rate is a very important characteristic of powders which measures the ability of a powder to be transferred.
- It is defined as the rate at which a metal powder will flow under gravity from a container through an orifice both having the specific shape and finish.
- The powder filling of die must be rapid and uniform without bridge formation for obtaining a rapid rate of production, consistent compacts and economy.
- The standard apparatus (جهاز القياسي), known as Hall Flowmeter, is generally used for the determination of flow rate.
- The time required to flow the weighed sample of powder (usually 50 g) from the funnel into a cup held at a fixed distance below the orifice is a measure of flow rate

Powder Characterization.

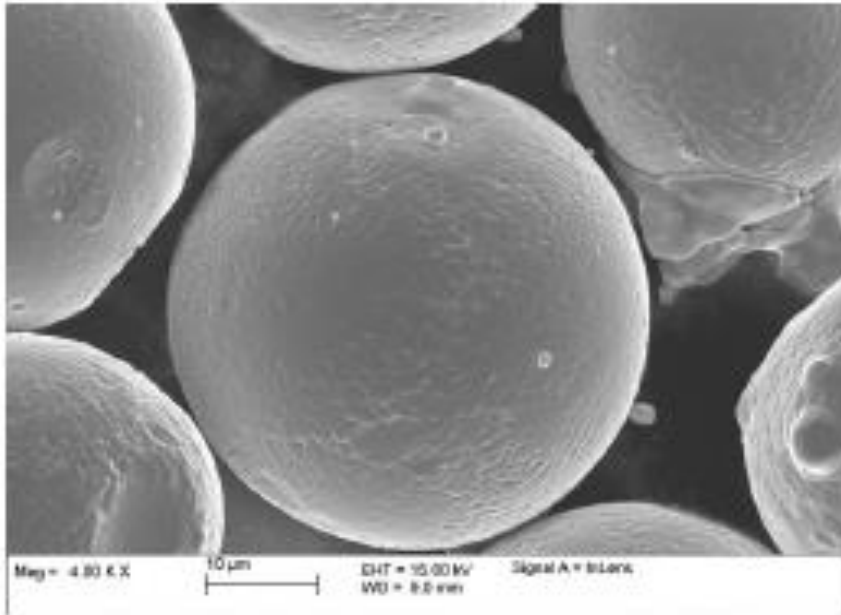
Example

With increasing non-uniform shape of particles, the flow rate increases.

كلما كان شكل الحبيبه غير منتظم كلما ازدادت معدل الانسيابيه.

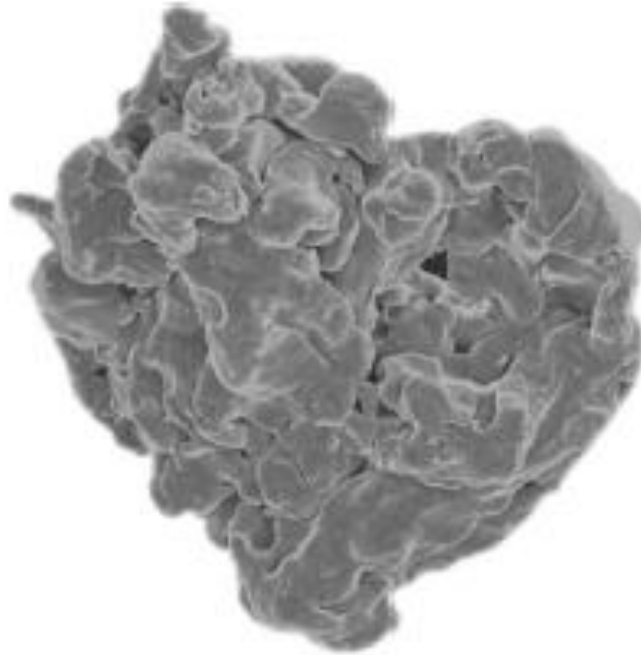
Bulk: density, flow

The relationship between flow rate and apparent density is reversed, with decreasing flow rate, the apparent density increases for sponge, irregular and spherical particles.



Spherical (stainless steel)

AD: 4-4,5 g/cm³
Flow: 13-15 s/50g



Sponge (irregular and porous)

AD: 2,3-2,65 g/cm³
Flow: 29-32 s/50g



Atomized (irregular)

AD: ~3 g/cm³
Flow: ~25 s/50g

Powder Characterization.

(2) Technological Properties (the effect of bulk/ segregation الانتشار).

In a particulate system (نظام الجزيئي) there are no motions equivalent to the molecular diffusion (الانتشار الجزيئي) of gases and liquids.

Segregation rate depends on

- 1) Particles' shape and size,
- 2) Induced movement, and
- 3) Density of additives.

Powder Characterization.

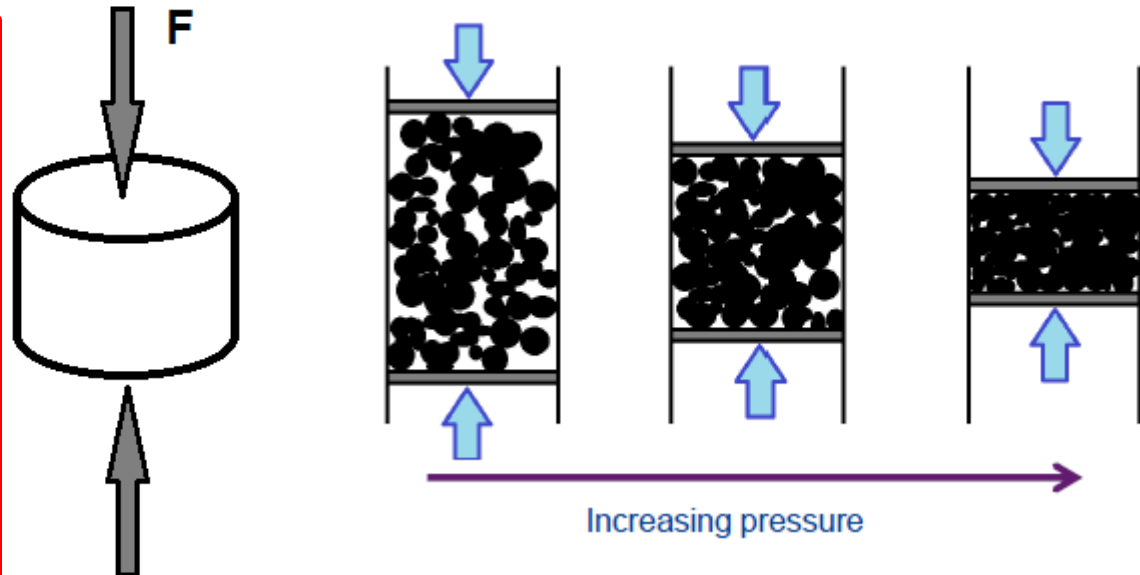
(2) Technological Properties (Compressibility - الأنضغاطية).

Compressibility is a measure of compression or densify the powder using external pressure.

Compressibility unit is the density in g/cm^3 , rounded to the nearest 0.01 g/cm^3 , at a specified compaction pressure.

Factors influencing compressibility:

- a) Compaction pressure
- b) Particle shape/size
- c) Particle porosity
- d) Lubricant/organics content / محتوى عضوي / type
- e) Powder composition



Powder Characterization.

(2) Technological Properties (Compressibility - الأنضغاطية)

- It is one of the most important characteristics of a metal powder since it affects the densification process.
- It is a measure of the powders ability to deform under applied pressure.
- It is represented by the pressure/ density (or pressure/ porosity) relationship.
- It is defined as the ratio (compression ratio) of: $\frac{\text{green density of the compact}}{\text{apparent density of the powder}}$.
- Maximum compression ratio = $\frac{\text{Ultimate or True density of a bulk material}}{\text{Apparent density}}$.
- The compression ratio can be varied from about (2 – 8), in most practical applications.
- Higher values of compression ratio require greater die depths and produce severe complications due to the introduction of friction between the powder and the die walls and internal friction of the powder.
- Thus a low compression ratio is preferred because of the reduction of: i. Die depths ii. Breakage and wear of tooling iii. Press movement and thereby it is possible to achieve higher production rate.

Powder Characterization.

(2) Technological Properties (Compressibility - الأنتضاغاطيه)

Compressibility, is defined in terms of the densification parameter, to:

$$\text{Densification parameter} = \frac{\text{Green density} - \text{Apparent density}}{\text{Theoretical density} - \text{Apparent density}}$$

- **Green spring:** another property of the green compact, associated with the difference between the size of the compact and the tools employed to prepare.
- It is usually termed green "spring" because the compacts expand both radially (باتجاه القطر) and longitudinally on ejection from the tools.
- During compacting, plastic deformation of the powder particles causes work hardening and an increase in the elastic limit.
- As the compact leaves the die, there is elastic recovery of the residual stresses and when it exceeds the green strength of the compact, cracking will occur on ejection.

Powder Characterization.

(2) Technological Properties (Compressibility - الأنضغاطيه)

- Green strength is the mechanical strength of a green – i.e. un-sintered powder compact.
- This characteristic is very important, as it determines the ability of a green compact to maintain its size and shape during handling prior to sintering.
- Green strength is promoted by increasing particle surface roughness and area, and decreasing the powder apparent density.

Compatibility is indicated by the pressure/green strength relationship. Compact ability of powder is defined as the minimum pressure required producing a compact of given "green strength". Both these terms are dependent on particle size, shape, porosity or density and hardness, surface properties, chemical composition and previous history (e.g. hardening, annealing treatment, etc) of the powder.

Powder Characterization.

(3) Chemical properties, by consideration (Chemical composition, phase distribution):

- The chemical composition of powders usually reveals the type and percentage of impurity.
- The term impurity refers to some elements or compounds which has an undesirable effect.
- Impurities influence not only the mechanical properties of the powder compacts, but also their chemical, electrical and magnetic properties.
- It may also exert (يبيذل جهدا يتحمل) a decisive (ثابت) effect on pressing, sintering, and other post- sintering operation which are essential for the production of finished product from powders.
- It determines the particle hardness and compressibility.
- The toxicity of powder is normally related to inhalation (أستنشاق) or ingestion (أبتلاع) of the material and the resulting toxic effect.
- The chemical reactivity of a material increases as the ratio of surface area-to-volume increases.

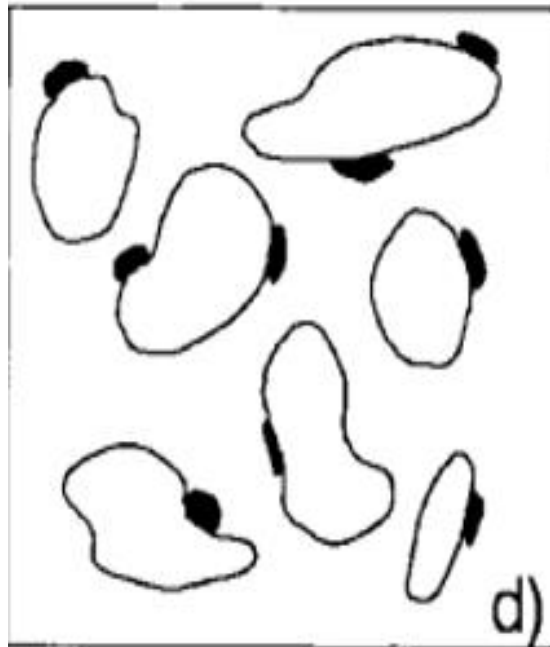
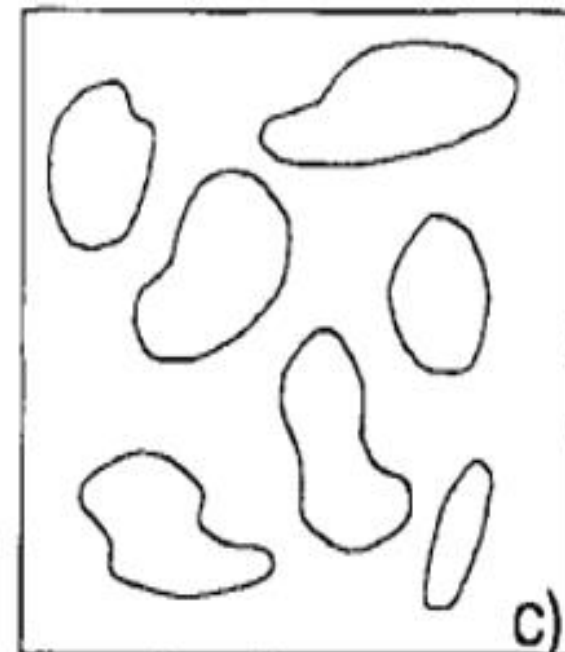
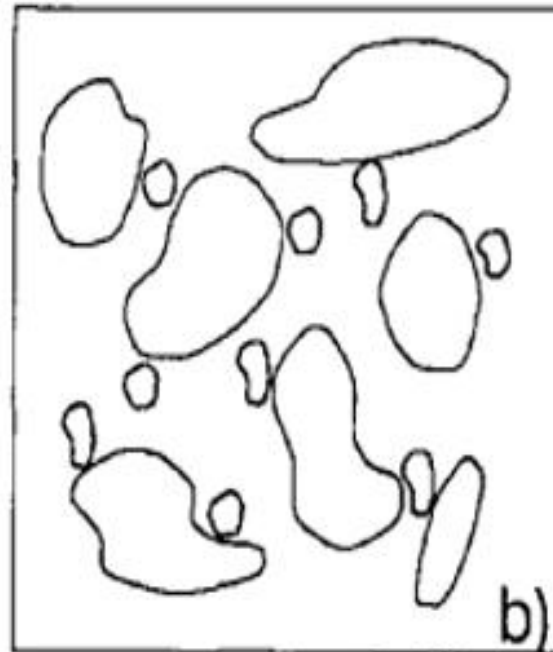
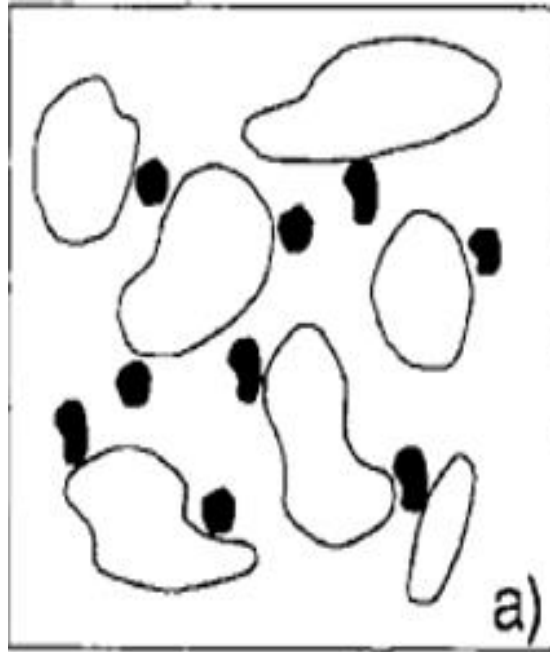
Powder Conditioning & Heat Treatment.

Powder conditioning: means additional treatments are required to make a powder suitable for further processing. They depend on the nature of the powder, the size and shape of the final part, and the subsequent operations to be carried out.

1) Powder mixing (additives): the temporary additives have the function of **lubrication, plasticizing and binding**. Lubricants and plasticizers facilitate movement of the particles under externally applied forces by reducing inter-particle and die wall friction forces. Binders enhance the strength properties of the green compacts, which have to be sufficient to withstand the stresses arising during subsequent handling. These stresses can be quite high during ejection of a compact from an axial die press.

خلط المسحوق (الأضافات): الأضافات المؤقتة تعمل على التزييت, تليين, ربط. المزيينات والملينات تسهل حركة الجزيئات والتي مسلط عليها قوى خارجيه عن طريق تقليل الجزيئات الداخلة وقوى الاحتكاكية لجدران القالب. الأربطه تعزز خصائص المقاومه للمسحوق المضغوط والذي من المفروض أن يكون كافي ليقاوم الأجهادات المرتفعه خلال المعاملات اللاحقه. هذه الأجهادات ممكن تكون عاليه جدا خلال عملية اخراج المسحوق المضغوط من قالب محوري الأتجاه.

Powder Conditioning & Heat Treatment.



a) elemental powder mixture

b) master alloy mixture

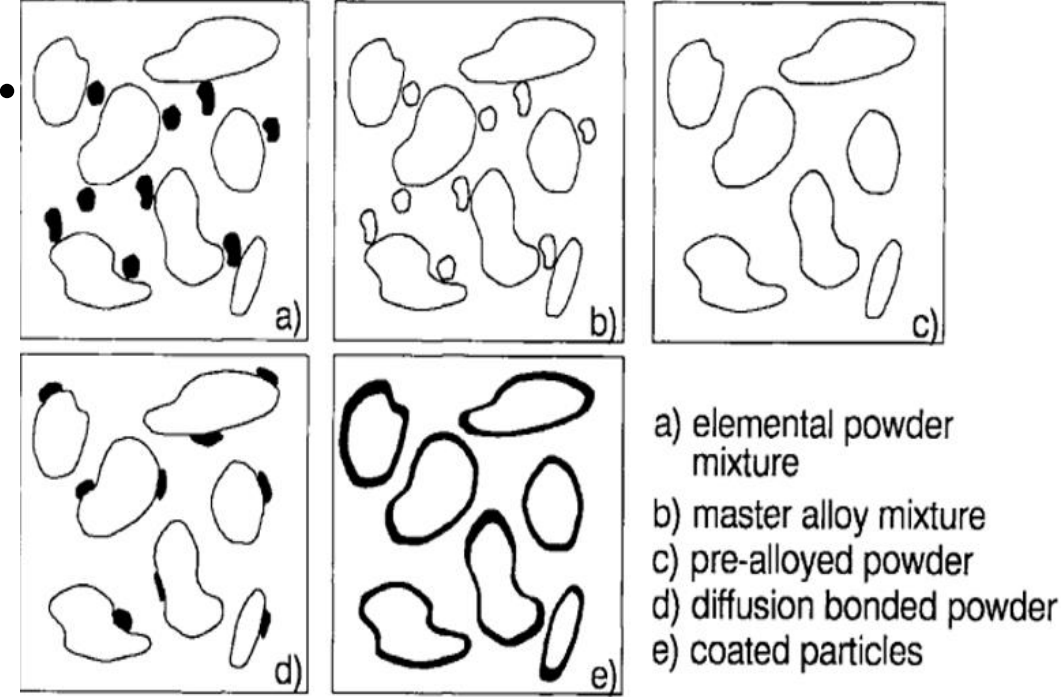
c) pre-alloyed powder

d) diffusion bonded powder

e) coated particles

Powder Conditioning & Heat Treatment.

2- Alloying powders: there are different types of alloying techniques are possible to be applied to the powder's systems, for example, the most important example of which are the coated particles shown in the figure. The purpose is to obtain the desired properties of the material in the final component. This could be occurred during sintering in order aid sintering through additional functions in powder metallurgy process, or during handling and formability of the powders.



تسبيك المسحوق: توجد عدة طرق لتسبيك مسحوق معين, على سبيل المثال, الجزيئات المطلية او المغلفة. الغرض هو الحصول على الخصائص المرغوب فيها بالنسبة للمنتج النهائي. هذه العملية ممكن حدوثها خلال عملية التلييد لتعزيز عملية السنتره من خلال عمليات اضافيه ضمن الية تكنولوجيا المساحيق, أو خلال المعاملات الحراريه وعمليات التشكيل للمسحوق.

Powder Conditioning & Heat Treatment.

3- **Particle size reduction/ Powder Mixtures of elemental (alloy constituents):** means mixing homogenous and heterogeneous powders, in order to provide the desired concentration of alloying elements. This process described as a versatile (متعدد الاستخدامات), relatively good compact ability (قابلية انضغاط نسبية). However, in many cases it is insufficient homogenization process during sintering, and the danger of segregation during powder handling.

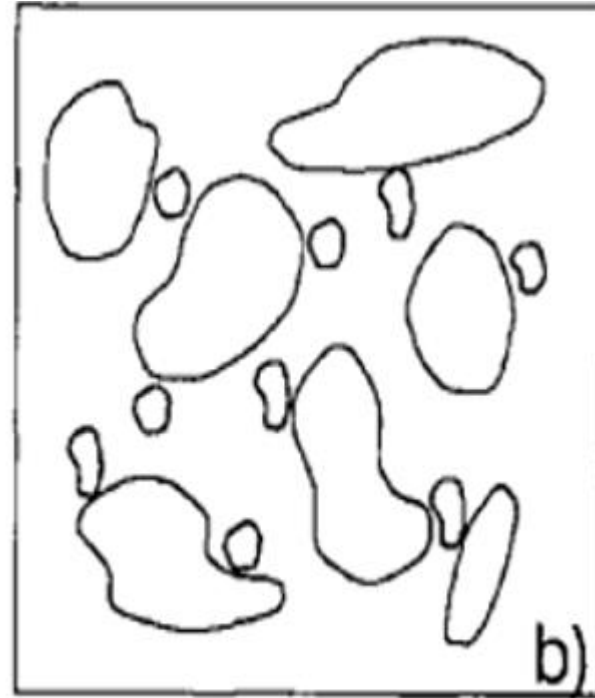


a) elemental powder mixture

Powder mixing(additives) + alloying powder= pre-alloyed powder

Powder Conditioning & Heat Treatment.

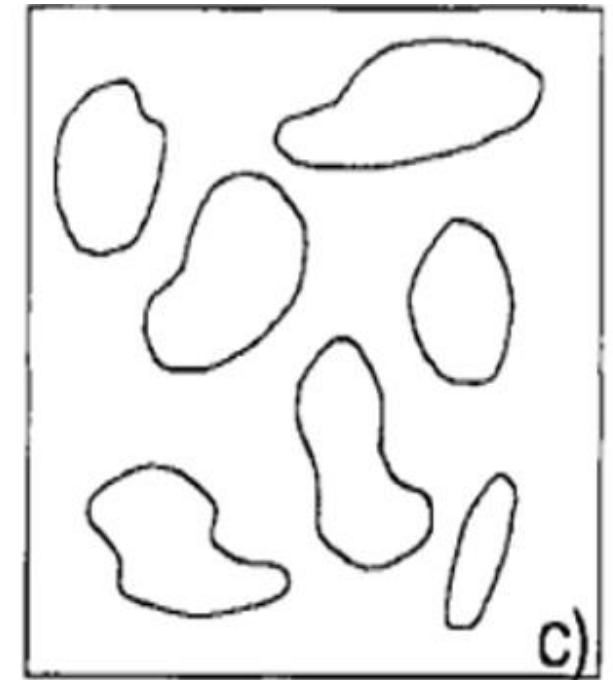
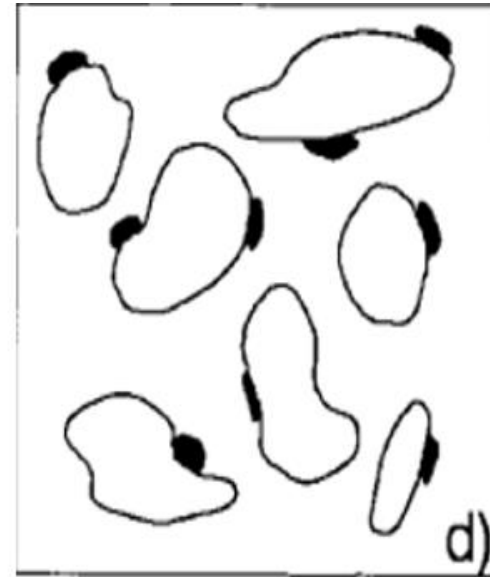
4- Evaluation powder mixtures quality in macro and micro volumes (Master alloy powders): means selection the suitable starting powder mixtures from various types, that enable overcome special problems in alloy system. For example, using alloying elements of high oxygen affinity (تقارب) such as Cr, Ti, Al, etc., which have stable oxide films inhibit (يمنع) sintering. They can be used as master alloy powders, in which their activities are reduced by forming compounds or solid solutions with the base powder, or which form transient or even stable liquid phases during sintering.



b) master alloy mixture

Powder Conditioning & Heat Treatment.

5- Diffusion bonded (diffusion stabilized): means combining the powder mixtures with the pre-alloyed powders. This is produced by special heat treatment of a mixture, within it, the particles of the alloying constituents are sintered (bond with base powder particles through diffusion). So, the danger of powder segregation could be completely eliminated.



d) diffusion bonded powder c) pre-alloyed powder

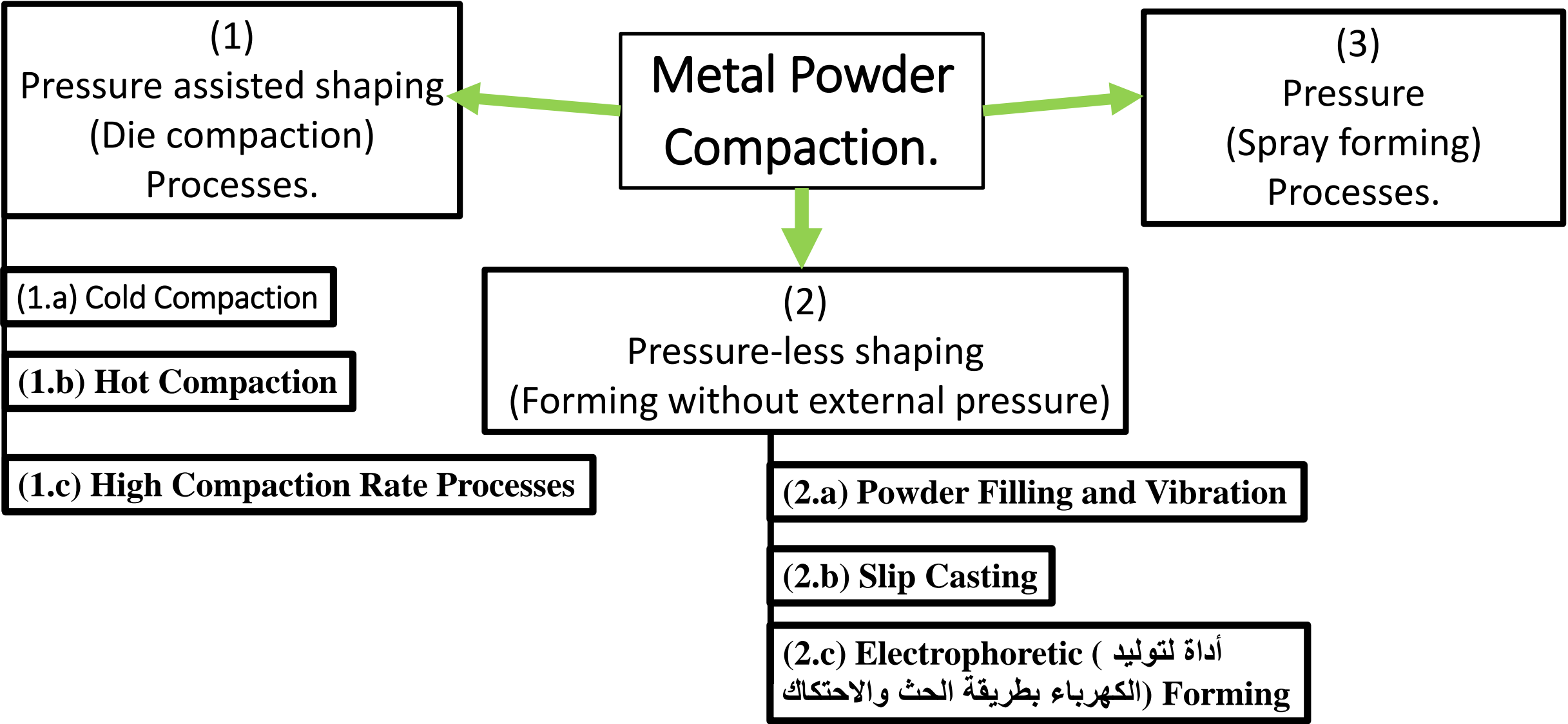
Powder Conditioning & Heat Treatment.

6- Particle coating: Means the alloying constituents is similar to diffusional alloying, but more effective for homogenization during sintering. It can be applied even in completely immiscible systems (انظمه غير قابله للامتزاج) like metal-ceramics, where it provides the basis for ideal microstructures with a continuous skeleton (هيكل مستمر) of the metal phase at metal contents of only a few percent. Coating of particles can be achieved via gas phase reactions or by precipitation from aqueous solutions.



e) coated particles

II. Metal Powder Compaction Processes.



III. Metal Powder Compaction Objective.

A. Consolidate (دمج) the powder into desired shape.

(دمج المسحوق الى الشكل المطلوب)

B. to Impart (منح او اعطاء او مشاركه), to as high a degree as possible, the desired final dimensions with due consideration to any dimensional changes resulting from sintering.

(للمشاركة في اعطاء الأبعاد النهائية أعلى درجة من الدقه مع الأخذ بنظر الاعتبار السماحات البعديه الحاصله بعد السنتره لتجنب التغيرات البعديه التي من المحتمل توقع حصولها بعد عملية السنتره)

C. To impart the desired level and type of porosity.

(لتحديد مستوى الضغط المطلوب وأيضا نوعية المسامات المتواجده)

D. To impart adequate strength for subsequent handling.

(لخزن طاقه مناسبه وكافيه للمسحوق تساعد في الحفاظ على خصائصه عند تعرضه للمعاملات اللاحقه أن استلزم الأمر)

IV. Metal Powder Compaction Categories

To achieve the goal of compaction process, should select one of the following categories:

(a) continuous vs discontinuous process,

(عمليات أنضغاط مستمره أو متقطعه)

(b) Pressures high vs low,

(باستخدام ضغط أما عالي أو منخفض)

(c) compaction velocity – high vs low,

(وبسرعه أما عاليه أو منخفضه)

(d) Temperature room to elevated temperature,

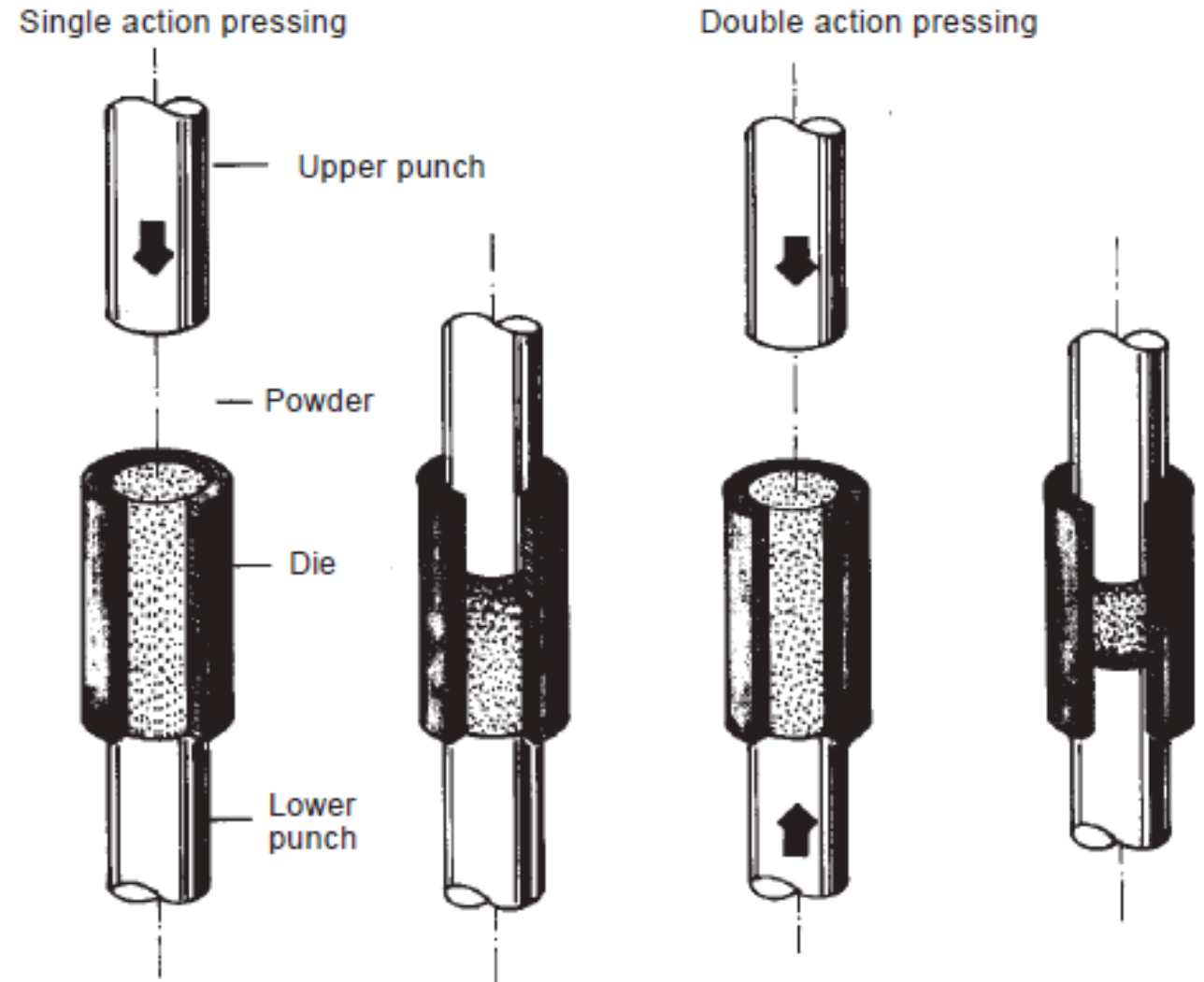
(وبدرجات حرارة الغرفه أو درجات حراره عاليه)

(e) uniaxial vs hydrostatic pressures.

(وباستخدام أجهزه ذات ضغط أحادي الأتجاه أو هيدروستاتيكي).

V. Die Compaction (Conventional) Technique.

- Represents the most widely used method.
- Involves rigid dies and special mechanical or hydraulic presses.
- Densities of up to 90 % of full density can be achieved following the compaction cycle, the duration of which may be of the order of just a few seconds for very small parts.
- Powders do not respond to pressing in the same way as fluids and do not assume the same density throughout the compact.
- The friction between the powder and die wall and between individual powder particles hinders the transmission of pressure.



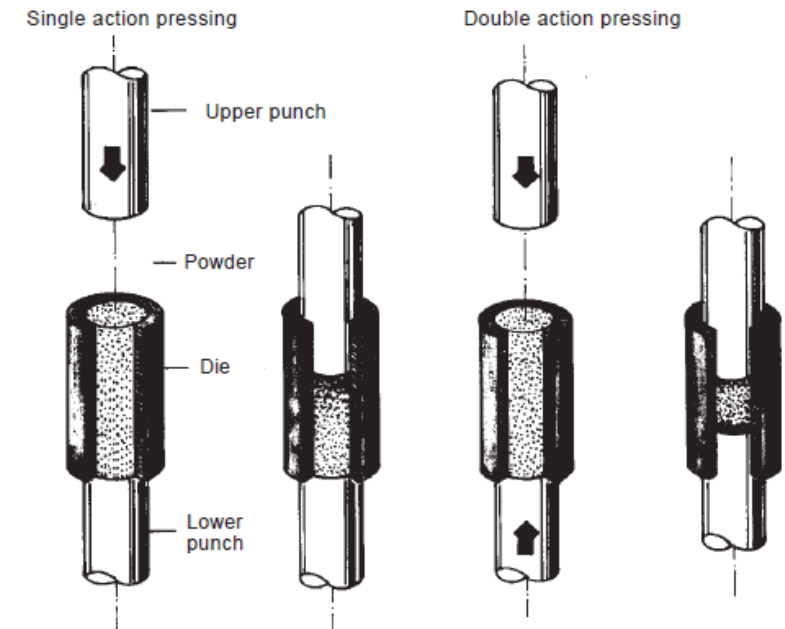
Single and double acting powder compaction.

V. Die Compaction (Conventional) Technique.

A high uniformity in green parts can be achieved depending on:

1- Kind of compacting technique: It refers to the movement of the individual tool elements (e.g. upper/lower punch, die relative to one another).

2- Type of tools: depends on pressing action, it refers to using two types of tools during compacting process within a fixed dies.



Single and double acting powder compaction.

VI. Pressing Action.

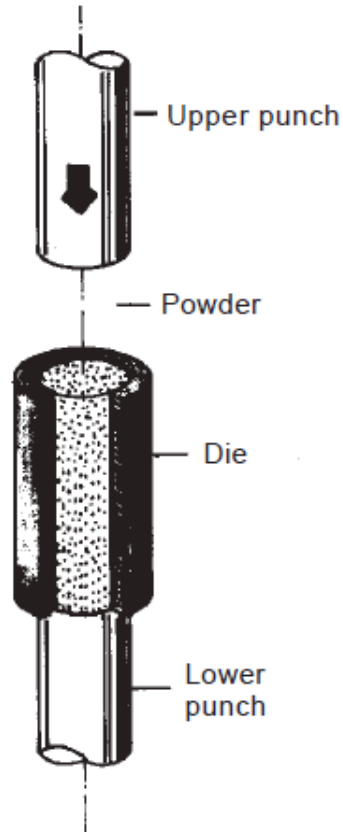
pressing action

Single

Double

- 1) Lower punch and die are both stationary (ثابت).
- 2) The pressing operation carried out solely by the upper punch as it moves into the fixed die.
- 3) The die wall friction prevents uniform pressure distribution.
- 4) The compact has a higher density on top than on the bottom.

- 1) Die is stationary in the press.
- 2) Upper and lower punches advance simultaneously (تتقدم بصوره تزامنيه) from above and below into the die.
- 3) The result powder is high density at the top and undersides of the compact.
- 4) In the center there remains a 'neutral zone' which is relatively weak.



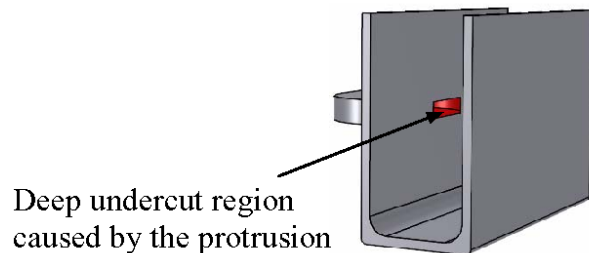
VII. Pressing Operation

can be sequenced as follows:

1. **Filling** of the die cavities with the required quantity of powder.
2. **Pressing** in order to achieve required green density (has porosity due to friction) and part thickness.
3. **Withdrawal of the upper punch from the compact**: a risk of cracking green parts is expected, as the upper punch withdraws the balance of forces in the interior of the die ends.

In the case of parts with two different thicknesses, the elastic spring back of the lower punch is the greatest danger.

Examples for parts with two different thicknesses



In the case of thin parts with large projected area, cracking is common due to **elastic spring back of the lower punch and the part itself**. The former (المشكل) pushes the part still lying in the die cavity upwards, while the latter tends to expand the part.

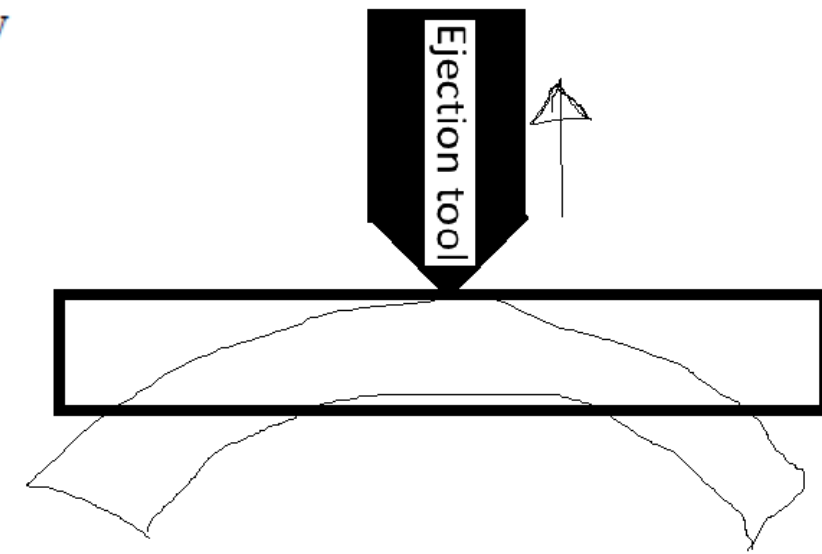
VII. Pressing Operation. *Cont.*

4. **Ejection**; the tooling must be done in such a manner to help ejection process to be possible as possible.

Ejection of a part with complex forms is a problem, as it involves friction between the green part and tool walls. The green strength must be high to resist the bending stresses introduced by the ejection force.

$$\text{Densification parameter} = \frac{\text{Green density} - \text{Apparent density}}{\text{Theoretical density} - \text{Apparent density}}$$

Green strength is the mechanical strength of a green – i.e. unsintered powder compact. This characteristic is very important, as it determines the ability of a green compact to maintain its size and shape during handling prior to sintering. Green strength is promoted by increasing particle surface roughness and area, and decreasing the powder apparent density.



1- Hydraulic Presses

Compaction Presses

2- Mechanical press

1-Produce working force through the application of fluid pressure on a piston (مكبس) by means of pumps (مضخه), valves (صمامات), intensifiers (مكثفات) and accumulators (المراكمات).

Compaction presses for powders are of two types



3- Mechanical hydraulic Presses

1-a flywheel stores energy, which is then released and transferred by one of a variety of mechanisms (eccentric اللامركزي, crank قرص تدوير, toggle وصله مفصليه) to the main slide.

المكبس الهيدروليكي: آلية العمل به ناتجه عن توليد قوة عامله (ويقصد قوه ضغطيه) من خلال ضغط السائل (المائع) على مكبس, وهو مايسمى بمضخات, صمامات, مكثفات, مراكمات.

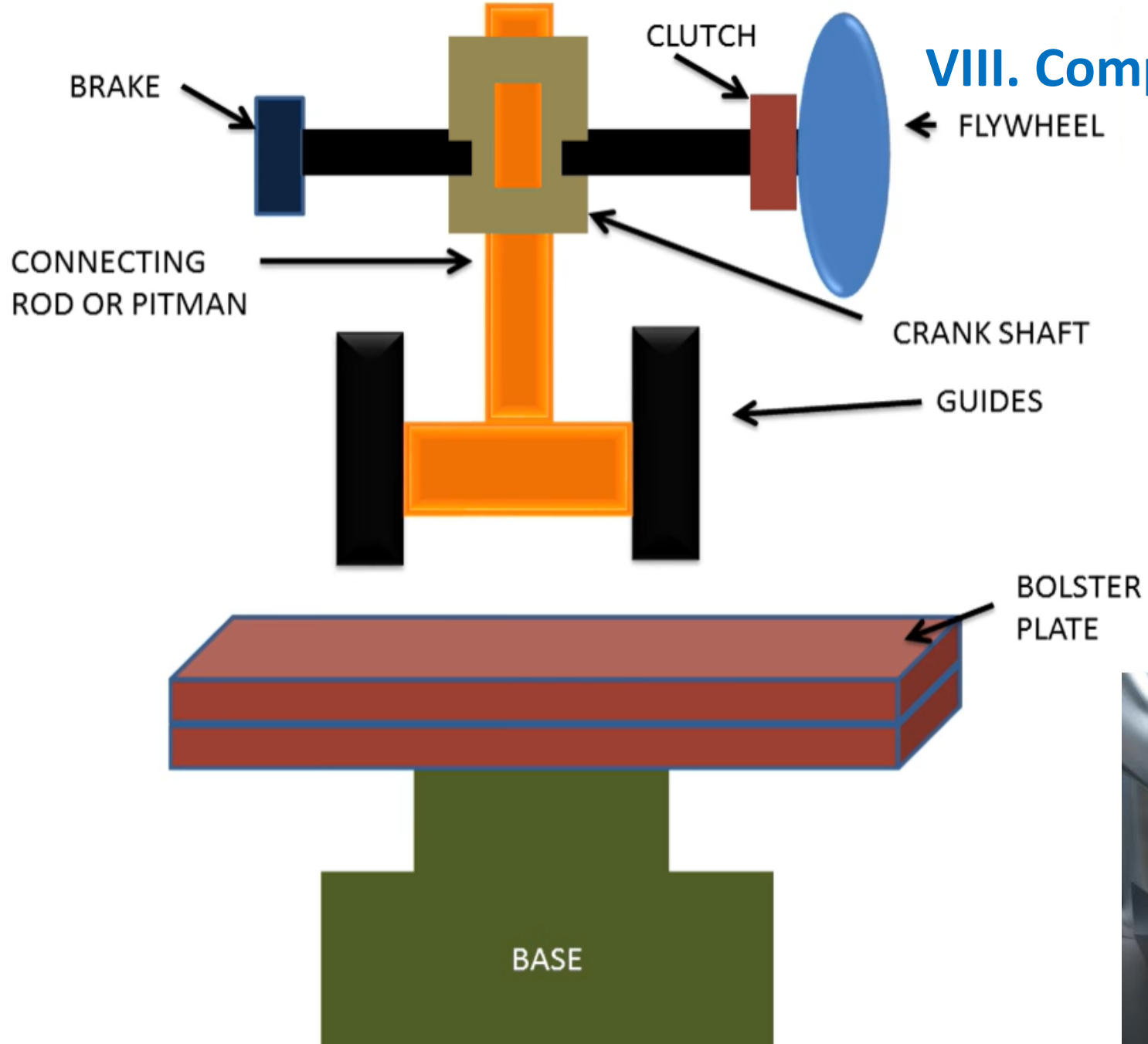
مستودع خزن الطاقه على شكل عجله أو دولاب دوار بصوره مستمره يوجه بنقل طاقه ميكانيكيه حركيه الى أحد أنواع المفاصل الميكانيكيه الناقله للضغط الميكانيكي الى المكبس الضاغط الذي يقوم بكبس المسحوق. على سبيل المثال, إحدى هذه الآليات الناقله هو ذراع التدوير اللامركزي, وذراع تدوير مركزي أو وصله مفصليه.

2- Inherent in the hydraulic method of drive transmission is the capability to provide infinite adjustment of stroke speed, length and pressure within the limits of press capacity.

2-For example, In the toggle type the eccentric or crank straightens a jointed arm, in the way, the upper end is fixed at the top, while the lower end is guided for controlled accurate punch guidance into the die.

أساس هذه الطريقه في نقل حركة المائع معتمد على قدرتها في تجهيز تعادل لامتناهي (موازنه مستمره) من سرعة للقوه الضغطيه الموجهه, طولها وضغطها ايضا ضمن السعه المحدده للمكبس الهيدروليكي.

على سبيل المثال, نقل الحركه باستخدام ذراع مفصلي يتم نقل الطاقه الميكانيكيه المتولده مركزيا من الكرنك أو لامركزيا من عجلة التدوير اللامركزيه الى المكبس الضاغط عن طريق جعل هذا الذراع (ذراع الوصله المفصليه) مستقيما بالشكل الذي يجعل النهايه العليا للذراع مثبتة باتجاه الأعلى بينما النهايه السفلى للذراع مصممه أن تكون حركتها للأسفل بالشكل الذي يدفع الكابس الضاغط نحو الأسفل وبحركه دقيقه مسيطر عليها بأن تكون باتجاه القالب.



VIII. Compaction presses,



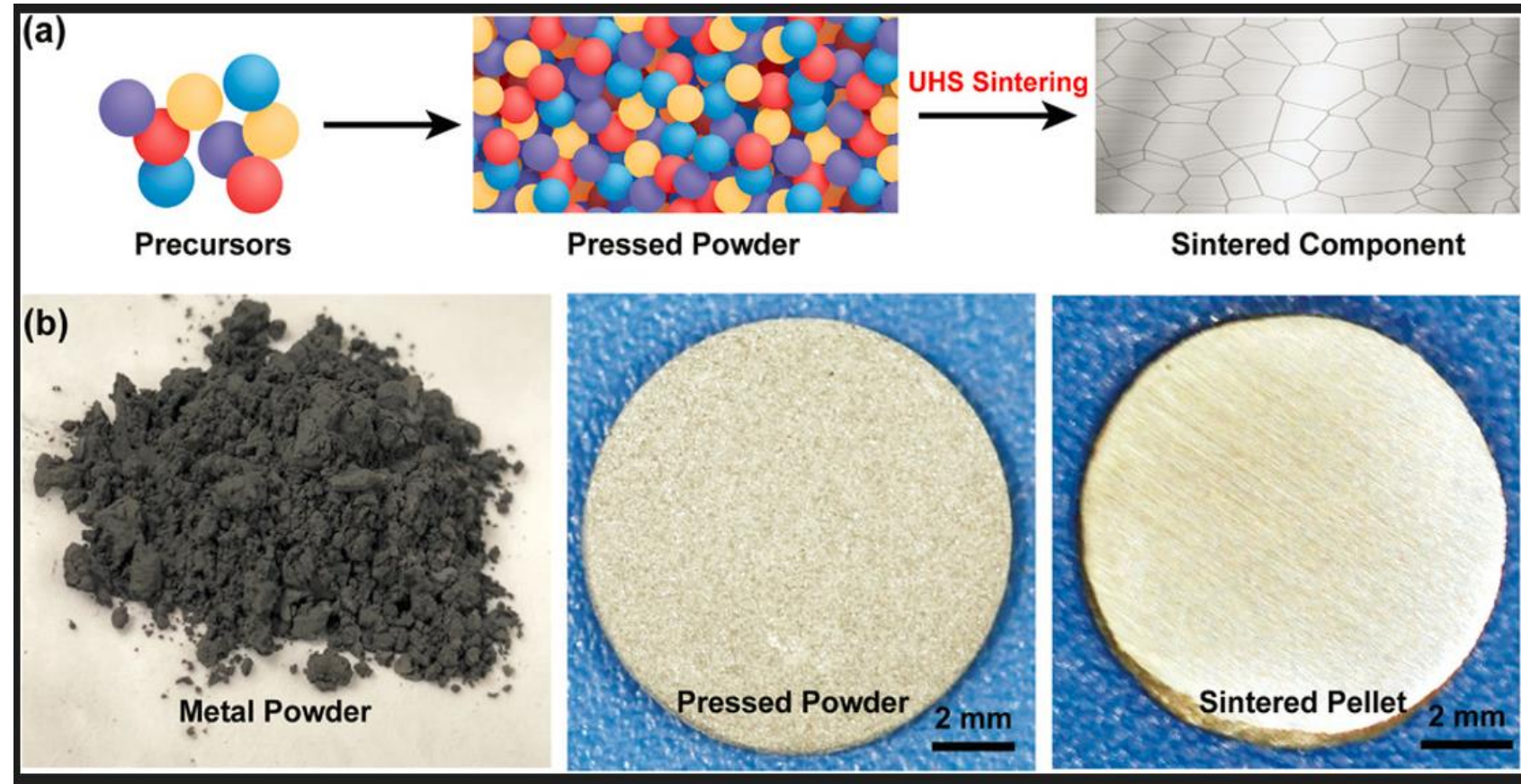
general view of the automatic mechanical press.

1.3 Sintering (تلييد أو ترسيب)

➤ Sintering: An assembly of the compacted particles under pressure or by confining (حصر) in a container (حاويه), chemically bond themselves by adhesive forces (قوة التلاصق) into a coherent body (جسم متماسك) under the influence of an elevated temperature.

➤ The temperature is usually below the melting point of the major constituent (القوام الرئيسي).

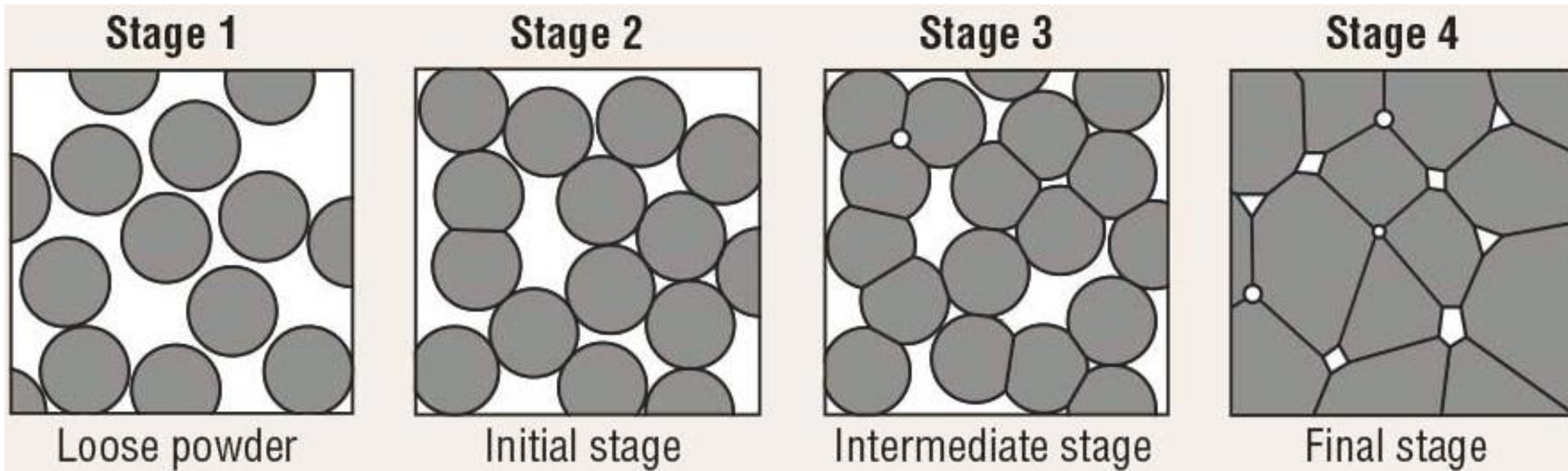
➤ During sintering, metallurgical changes within the material may take place simultaneously (على التزامن) or consecutively (على التعاقب).



Powder metallurgy steps to produce a part.

1.3 Sintering (تلييد أو ترسيب)

- It is achieved in stages, each stage has a set of condition, e.g. Driving force, & The mechanism of material transport.
- It is a complicated process as it is expected to face expansion or shrinkage in the sintered part leading to very large amount of dimensional changes.



Sintering steps to produce a part.

1.3.1 Sintering Stages.

(1) Initial bonding among particles,

(2) Neck growth,

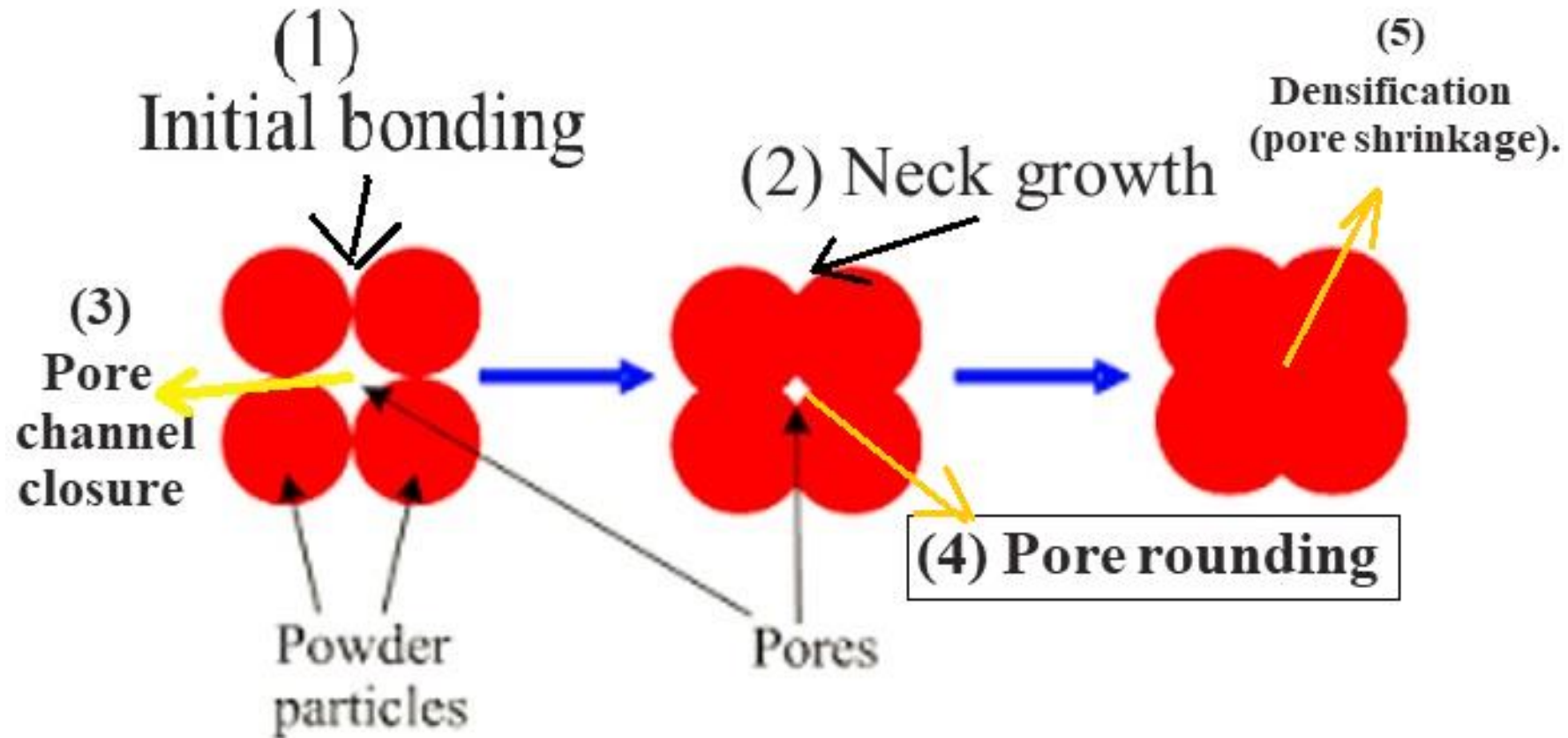
(3) Pore channel closure

(4) Pore rounding

(5) Densification

(pore shrinkage).

(6) Pore coarsening.



1.3.2 Liquid Phase and Activated Sintering Method:

Liquid Phase sintering Method:

- The most common method which include liquid phase during all or part of the cycle of the material's sintering in order to enhance densification.

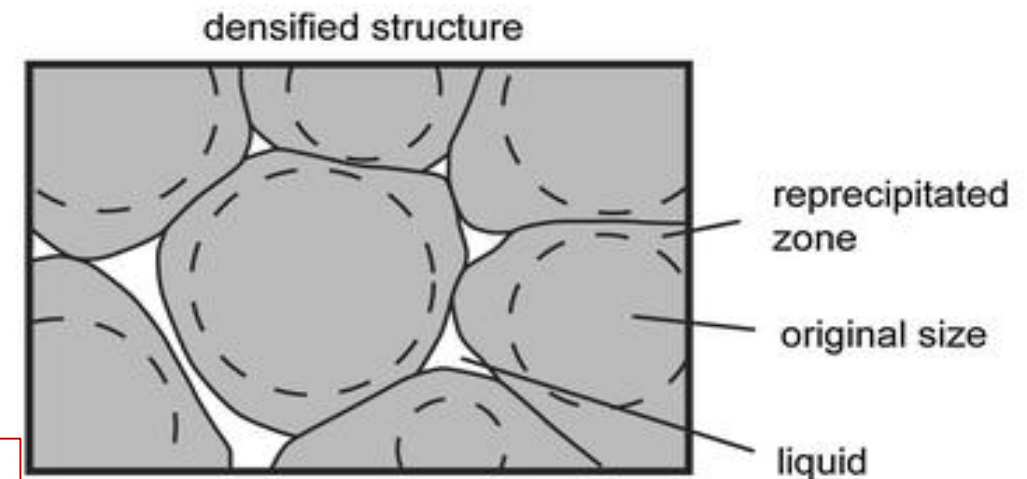
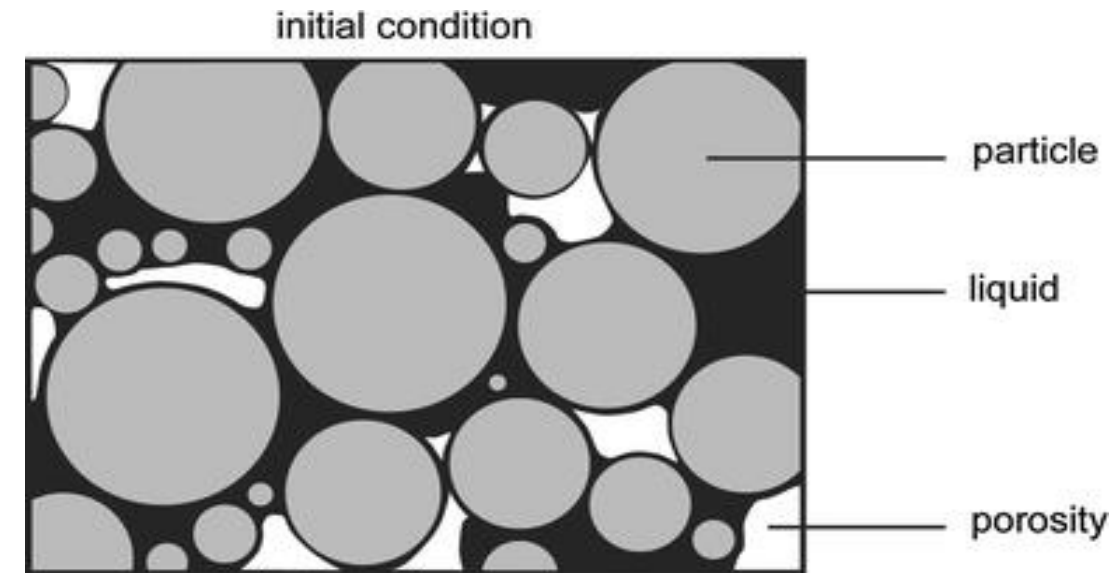
- There are two variations of the process:

- (a) normal liquid phase sintering for which the formation of the liquid phase is associated with one or more components contained in the original green compact.

التليبد بوجود طور السائل الطبيعي: فيه تشكيل الطور السائل متعلق بمركب أو أكثر متواجدين بصوره طبيعيه في الجزء الرطب الأصلي المضغوط .

- (b) Infiltration (تسريب \ ترشيح): occurs simultaneously during the very early period of sintering for the original green compact with the liquid formed outside the compact which is dominant, also, occurs for the previously sintered parts.

التسريب: يحدث بتزامن خلال المراحل المبكره جدا لعملية التليبد للجزء الرطب الأصلي المضغوط مع الماء المتشكل خارج عملية الانضغاط والتي هي العمليه المسيطره, وكذلك, تحدث للأجزاء الملبده التي قبلها.



Liquid Phase Sintering Process.

1.3.2.a Liquid Phase and Activated Sintering Stages:

Liquid Phase sintering steps (densification steps).

1) **Re-arrangement' / 'liquid flow'**

- Includes lower densification kinetics with progress in liquid phase sintering, cause increase liquid content up 35% volume.
- Increasing liquid 35% V, aids initial densification by start coarsening particles size,
- Coarsening particles size with high green density transfer the melt to an offset point.

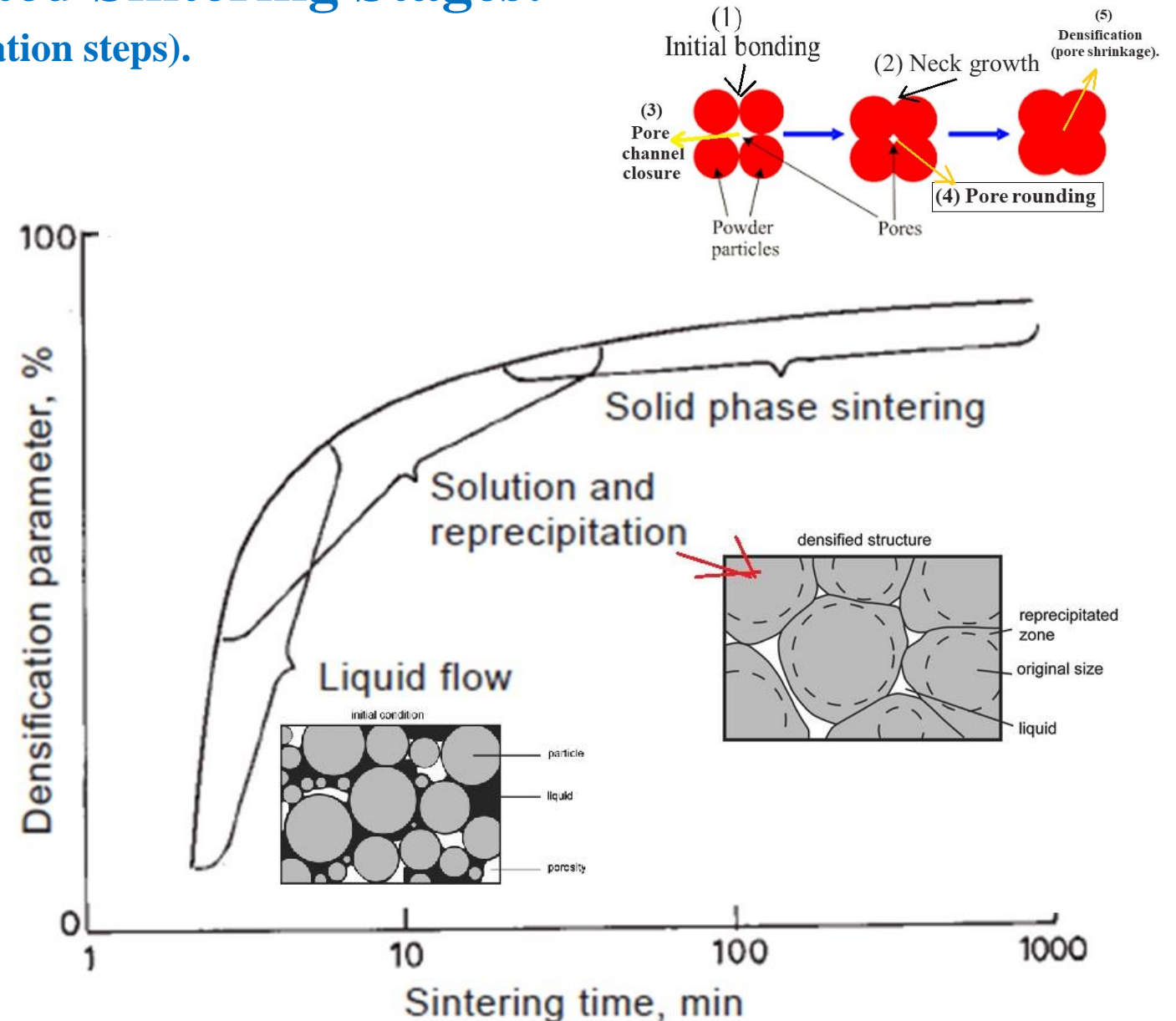


Figure below shows the densification stages during liquid phase sintering.

1.3.2.a Liquid Phase and Activated Sintering Stages:

Liquid Phase sintering steps (densification steps).

2) “Accommodation / ‘dissolution & re-precipitation”.

- The melt penetrates along the inter-particle interfaces causing separation, and contribute in swelling (انتفاخ او تضخم).
- A small dihedral angle (زاويه زوجيه) inhibits (يمنع) coalescence (التحام) of neighboring particles.
- This type of technology used in sintering steel, cemented carbides, heavy alloys, bronzes and silicon nitride systems.

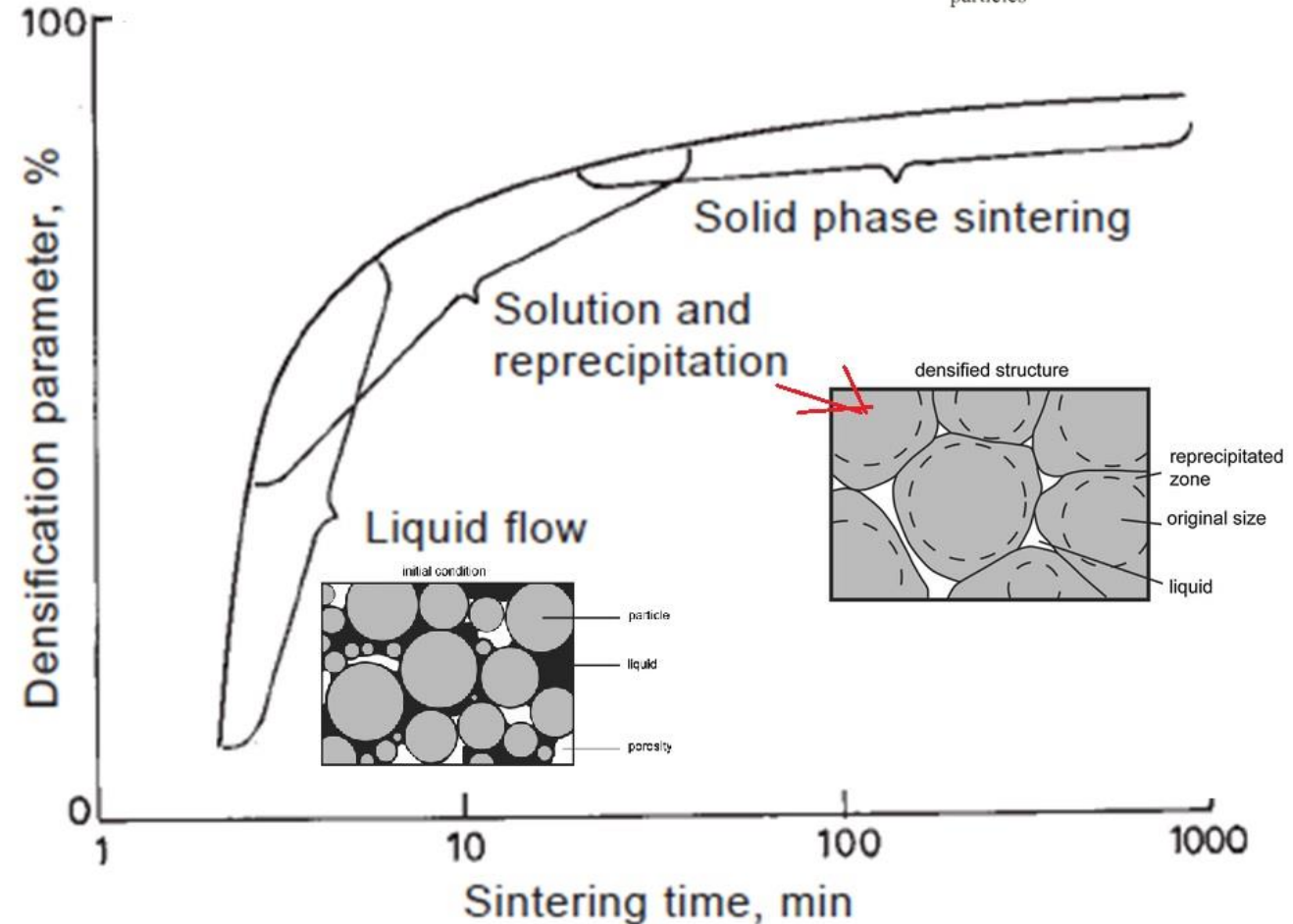
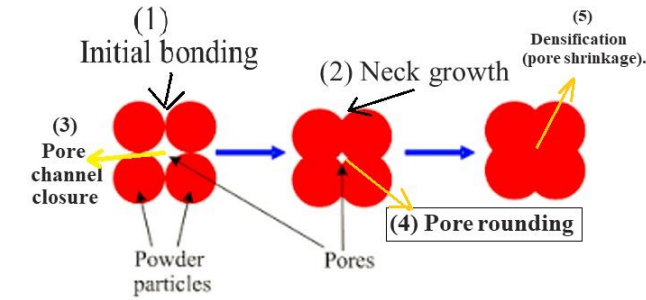


Figure below shows the densification stages during liquid phase sintering.

1.3.2.a Liquid Phase and Activated Sintering Stages:

Activated Sintered.

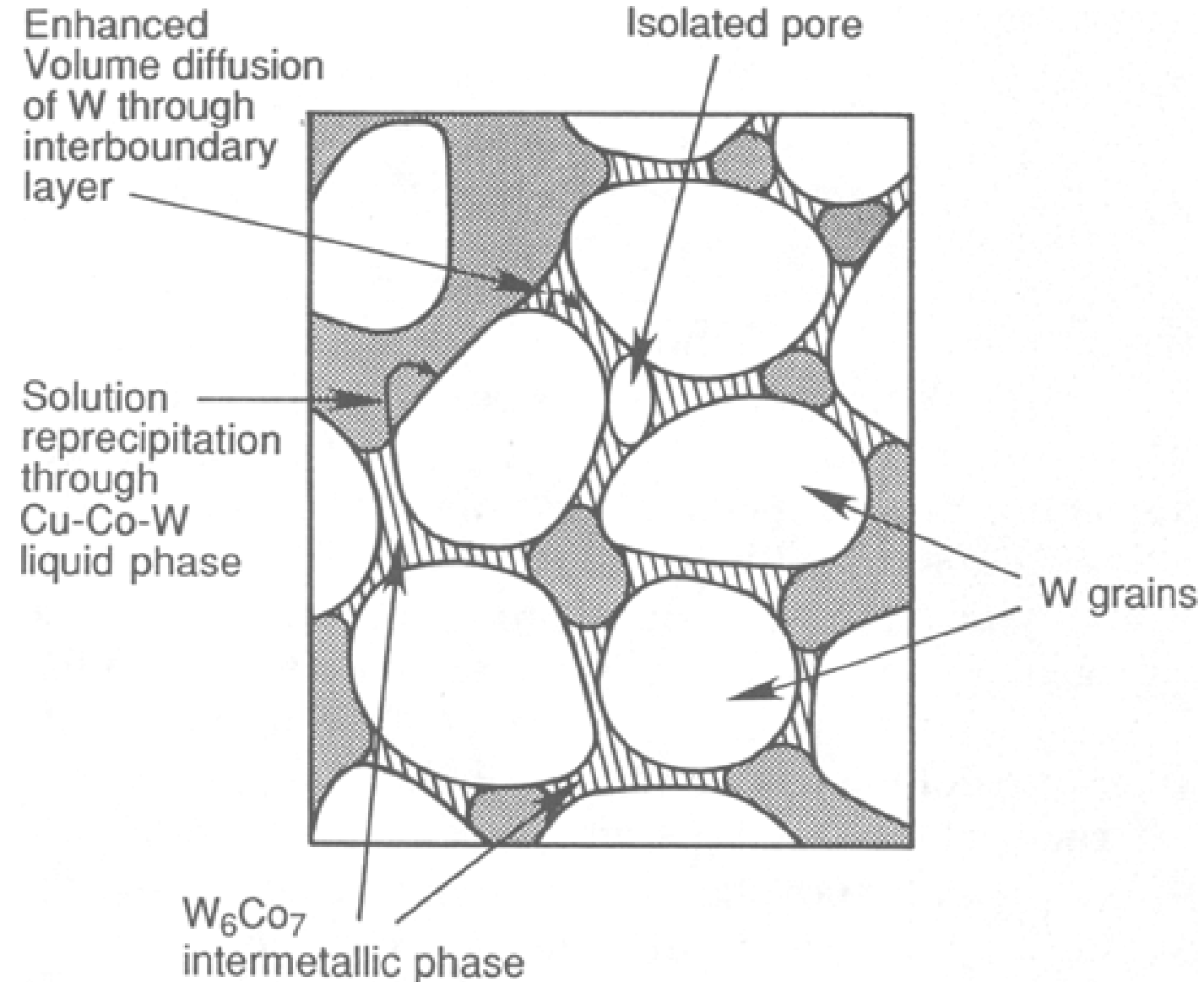
- Refers to lowering the activation energy for sintering by adding chemical addition to the powder.
- For example, the refractory metals (المعادن ذات درجة ذوبان عالية) (ومقاومه للصهر), the addition of dopant (منشطات) causes the densification kinetics to increase 100 times compared with unhoped compacts.
- The best activators are palladium and nickel, tungsten.

1.3.2.a Liquid Phase and Activated Sintering Stages:

Activated Sintered (Example).

- Example presents the proposed mechanism of activated liquid phase sintering through adding (W) activator.
- Solution re-precipitation through Cu-Co-W liquid phase is thought to be negligible in comparison to enhanced volume diffusion through the inter-boundary layer.

Negative side: Isolated porosity may result from the rapid solid-state sintering.



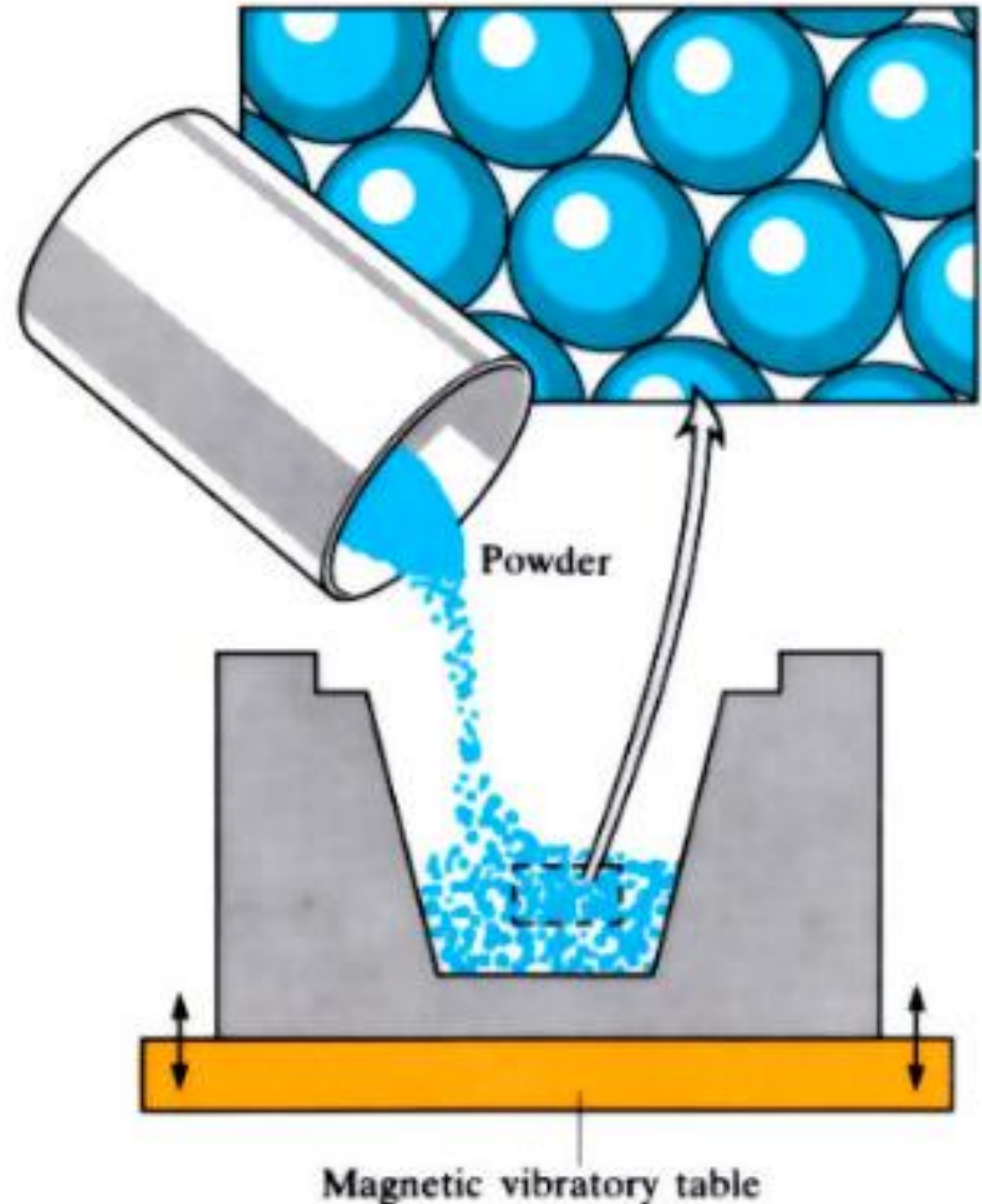
1.3.2.a.2 Loose Sintering Method.

- Widely used for manufacturing highly porous parts, e.g. filters.
- Metal powder is poured or vibrated into a mould.
- The poured metal powder in the mould is then heated to the sintering temperature in an appropriate atmosphere.
- Complexity resulted shape made by this method depend on powder's flow-ability.

“Mould material (مادة القالب) characteristics”

Should:

- ❖ Be easily machined or formed into the required shape,
- ❖ Withstand the sintering temperature without appreciable deformation
- ❖ Not weld to the powder during sintering.



1.3.3 Sintering Process Variables

The most important factors involved during sintering process are described below:

1. **Sintering Temperature:** Increasing the sintering temperature greatly increases the rate and magnitude of any changes occurring during sintering.
2. **Sintering Time:** the desired properties of the sintered parts possible to obtain by setting shorter sintering times and higher temperatures. The time has less affect than the temperature. This is due to loosing of driving force with increasing time at any temperature, the matter that makes remove all porosity by sintering is difficult. In turn, using furnace sintering at higher temperature is very costly due to maintenance costs and energy consumption, with elevating temperature.
1. **Sintering Atmosphere:** to control of sintering process, the proper sintered product depends on using the optimal gas atmosphere.

1.3.4 Sintering Compacted Material Powder Variables

- 1. Particle Size:** decreasing particle size leads to increased sintering. The smaller particle size has a greater pore/solid interfacial area producing a greater driving force for sintering. It promotes all types of diffusion transport, e.g. greater surface area leads to more surface diffusion, small grain size promotes grain boundary diffusion and a larger inter-particle contact area to volume diffusion.
- 2. Particle Shape:** with decrease sphericity shape in particles and increasing macro/or micro surface roughness, greater intimate contact between particles occur lead to increase internal surface area. This promote sintering.
- 3. Particle Structure:** A fine grain structure within the original particles can promote sintering because of its favour-able effect on several material transport mechanisms.
- 4. Particle Composition:** Alloying additions or impurities within a metal can affect the sintering kinetics. The effect can either be deleterious or beneficial depending upon the distribution and reaction of the impurity. Surface contamination, such as oxidation is usually undesirable. Dispersed phases within the matrix may promote sintering by inhibiting grain boundary motion. Reaction between impurities and either the base metal or alloying additions at the relatively high sintering temperature may be undesirable.
- 5. Green Density:** A decreasing green density signifies an increasing amount of internal surface area and consequently, a greater driving force for sintering. Although the percentage change in density, increases with decreasing green density, the absolute value of the sintered density remains highest for the higher green density material.

1.3.5 Dimensional Changes Resulted From Sintering

The fundamental process of sintering leads to a reduction in volume because of pore shrinkage and elimination.

Following factors should be considered:

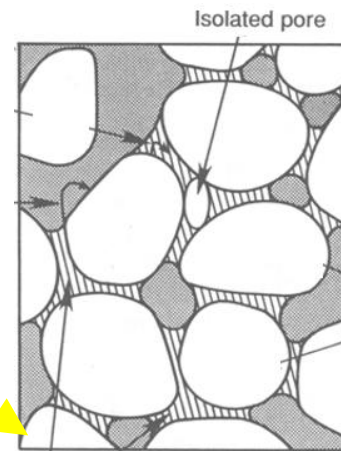
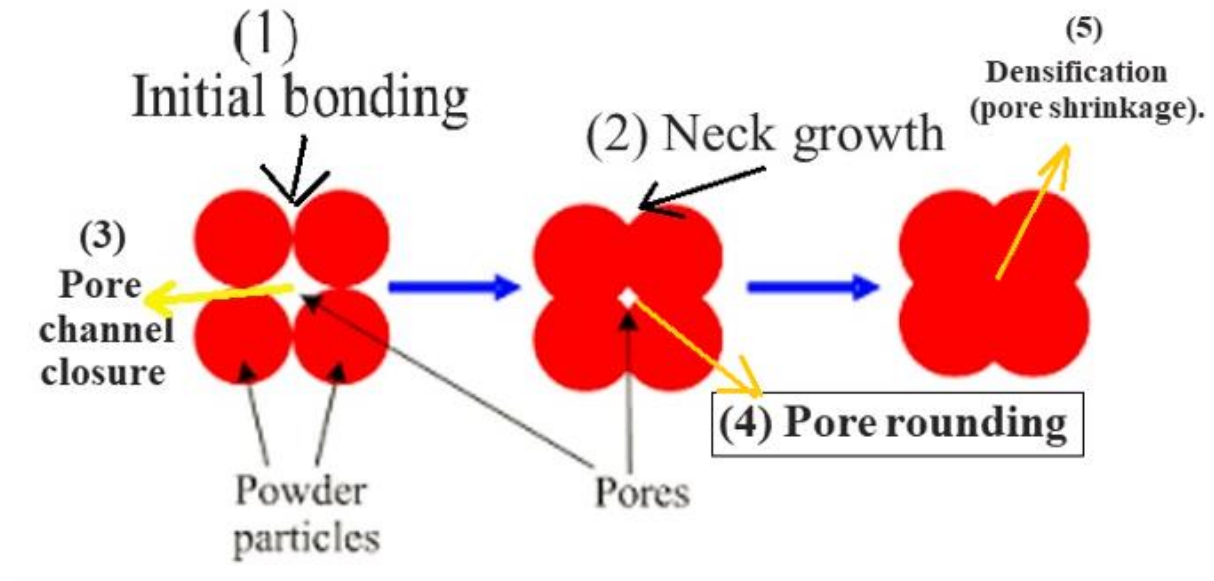
- ❖ **Entrapped Gases:** The expansion of gas in closed porosity produces compact expansion.
- ❖ **Chemical Reactions:** Hydrogen atmospheres cause diffuse through the metal to isolated portions of the compact, and reacts with oxygen to form water vapour. The pressure of the water vapour lead to expansion of the entire mass, or have reactions that lead to the loss of some element from the sinter mass to the atmosphere, such as volatilizing(التطاير), and result in a shrinkage of the material.
- ❖ **Alloying:** Alloying between two or more elemental powders due to formation of a solid solution leads to compact expansion. This effect is offset by shrinkage of the original porosity. Dimensional changes may also occur in a binary system where the rate of diffusion of each metal into the other is different.
- ❖ **Shape Changes:** Low green density regions will exhibit a greater amount of shrinkage during sintering.

1.3.6 Microstructural changes (Sintering Mechanism)

- Sintering the green compacted powder causes meshing the boundaries of the original particles with each other (unseen), leading to make the particle's structure looks like in an annealed wrought conditions, except incase of pores availability.

Pore's availability might have negative or positive affect on the diffused structure during sintering. This is because with progression of sintering, pores continue to shrink commencing formation of closed pores at about 5% of total porosity.

Basically, many parts produced by powder metallurgy are formed by porosity (closed), or by blending powders elements that constitutes many alloys, blending process possible produces non-homogenous structure, due to formation **opened pores**, which might affect negatively or not in the sintered structure.

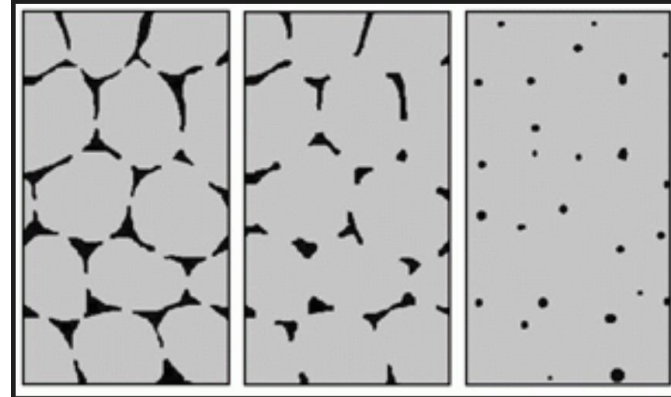


1.3.6 Microstructural changes(Sintering Mechanism).

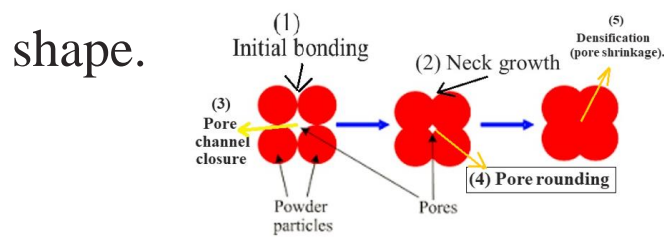
- During sintering the driving force for **grain growth** is very large,
- **Grain growth** in sintering formed from **two phases**.
- **Two phases**= (phase's material + **formed pores**).
- **Formed pores**: represent a hinder to grain growth in sinter mass,
- On the other hand, **the additions to the powder material** possible cause **hinder** to bonding adjacent particles (ربط الجزيئات المتجاوره) and **encourage** formation grain boundary.
- In addition, the grooves (الحزوز) in the grain boundary may also hinder grain growth by movement the grain boundaries away from its groove lead to increasing the energy and area of grain boundaries.
- Many types of phase transformations may occur in the solid state during sintering at a constant temperature or during the cooling of the metal from the sintering temperature. Porosity and fine grain structure influence on the transformation.

1.3.6 Microstructural changes (Sintering Mechanism).

- Precipitation from solid solution is also a very common type of transformation associated with sintering.
- During liquid phase sintering porosity level falls down, while grain size increases. Figure below.



- The shape of the pores varies rapidly during liquid phase sintering.
- In the first stage, the pores are irregular. Later they form a cylindrical network and finally attain a spherical shape.



- The interfacial energies can change during liquid phase sintering, since they depend on solubility, surface contamination and temperature.

So during liquid phase sintering, the microstructural parameters change with time, which affect significantly on mechanical properties of liquid phase sintered part, particularly ductility.

1.3.7 Sintering Atmosphere

- Generally, all materials' type react with their surroundings because they react with the natural atmosphere that is already available in the environment.
- Same situation happens during sintering process due to technical matters.
- For example, all metals could react with the gas of their surrounding atmosphere at room temperature, and elevated temperatures.
- Reaction metals with the surrounded gas atmosphere increases with increasing temperature.
- Also, the sintering process for the compacted metal powders to produce parts need special sintering atmosphere in order to provide protection against oxidation and re-oxidation of the sintered metal powders specifically. This is conducted by addition one type or more of gases, as an activators.

1.3.7.a Sintering Atmosphere Functions

- The addition of one type or more of gases into the atmosphere of sintering environment represents an activator for the following functions played by the added gas:
 - ✓ provide protection against oxidation and re-oxidation of the sintered metal powders specifically.
 - ✓ Provide protection against oxidation inside sintering atmospheres means reducing oxides.
 - ✓ Reducing oxides makes the atmosphere may create highly mobile metal atoms leading to direct reaction between the gas atoms inside the sintering equipment and the sintering compacted metals.
 - ✓ Gas atoms of the sintering atmosphere enter the sintering compact via interconnected pores.
 - ✓ Leading to either trapping by closed pores , so hinders formation shrinkage, or, diffuse into the metal itself and become alloyed with it.

1.3.7.a Gases used in atmosphere sintering.

- 1) Hydrogen,
- 2) Reformed hydrogen gases (Hydrocarbon) / (Exothermic & Endothermic),
- 3) Nitrogen and Nitrogen-Based Atmospheres,
- 4) Dissociated Ammonia,
- 5) Argon and Helium.
- 6) Vacuum Furnaces.

1.3.7.a.1 Sintering Characteristics of using Hydrogen Gas.

- 1) Pure hydrogen is an excellent reducing gas used for high valued products only.
- 2) Expensive , so not economical.
- 3) Very flammable, having an extremely high rate of flame propagation.
- 4) The lightest element; its specific gravity 0.069 compared to 1.0 for air.
- 5) Its thermal conductivity (7) times greater than air. Because of this, it accelerates both the heating and cooling rates of the work in sintering furnaces.
- 6) The thermal losses in furnaces are higher with hydrogen than when using heavier, less conductive gases.
- 7) Typical applications of hydrogen are in the reduction of oxides of iron during annealing.



(H₂N₂) Atmosphere Sintering Muffle Furnace



High Temperature (H₂) Atmosphere Sintering Box Muffle Inert Gas Furnace With Optional Sizes.



1.3.7.a.2 Sintering Characteristics of using Nitrogen and Nitrogen-Based Atmospheres.

- 1) Nonflammable due to it is inert gas.
- 2) Used as a safe cleaner for flammable atmospheres.
- 3) The main constituent of the nitrogen-based system is molecular (جزيئي)nitrogen.
- 4) Molecular nitrogen is obtained from air, which consists of ~ [78% N₂, 21% O₂, 0.93 % argon, 0.03 % CO₂ (dioxide carbon) and a small amount of such rare gases as neon and helium.
- 5) Very dry with a dew point (درجة تكثيف) (< 65°C).
- 6) Very pure, having < 10 ppm(parts per million) of oxygen.
- 7) It is essentially inert to materials most commonly sintered and to furnace components such as muffles, conveyer belt, heating element, radiant tube, fixtures, etc.



(H₂N₂) Atmosphere Sintering Muffle Furnace



High Temperature (H₂) Atmosphere Sintering Box Muffle Inert Gas Furnace With Optional Sizes.

1.3.7.a.3 Sintering Characteristics of using Argon and Helium Atmospheres.

- 1) Argon and helium are nonflammable.
- 2) Inert to all application.
- 3) Used for sintering refractory (مقاوم للصهر بسبب درجة الذوبان) (العاليه لها) and reactive metals and also as a back fill in vacuum furnaces.
- 4) Argon is cryogenically (درجات الحرارة المنخفضه) produced from air.
- 5) Its purity very high, less than 0.0005 % oxygen and a dew point <68oC.
- 6) Its specific gravity is 1.379 g/cm³, while thermal conductivity is 0.745.



1.3.7.a.4 Sintering Characteristics of Using Vacuum Sintering Furnace.

- 1) Vacuum retains the proper chemistry of the parts during sintering.
- 2) More economical than atmosphere gases, particularly bottled gas.
- 3) Only costly when using electrical energy and oil for the pumps of the vacuum furnace (افران محتويه هواء).
- 4) There are medium, high and low vacuum sintering furnace. But most sintering furnaces applied are of medium or high levels.
- 5) During sintering an alloy, the selective evaporation of some alloying elements must be taken into account, due to the different vapour pressures of the individual metals.
- 6) The efficiency of vacuum furnaces depends on the duration and temperature of vacuum sintering.

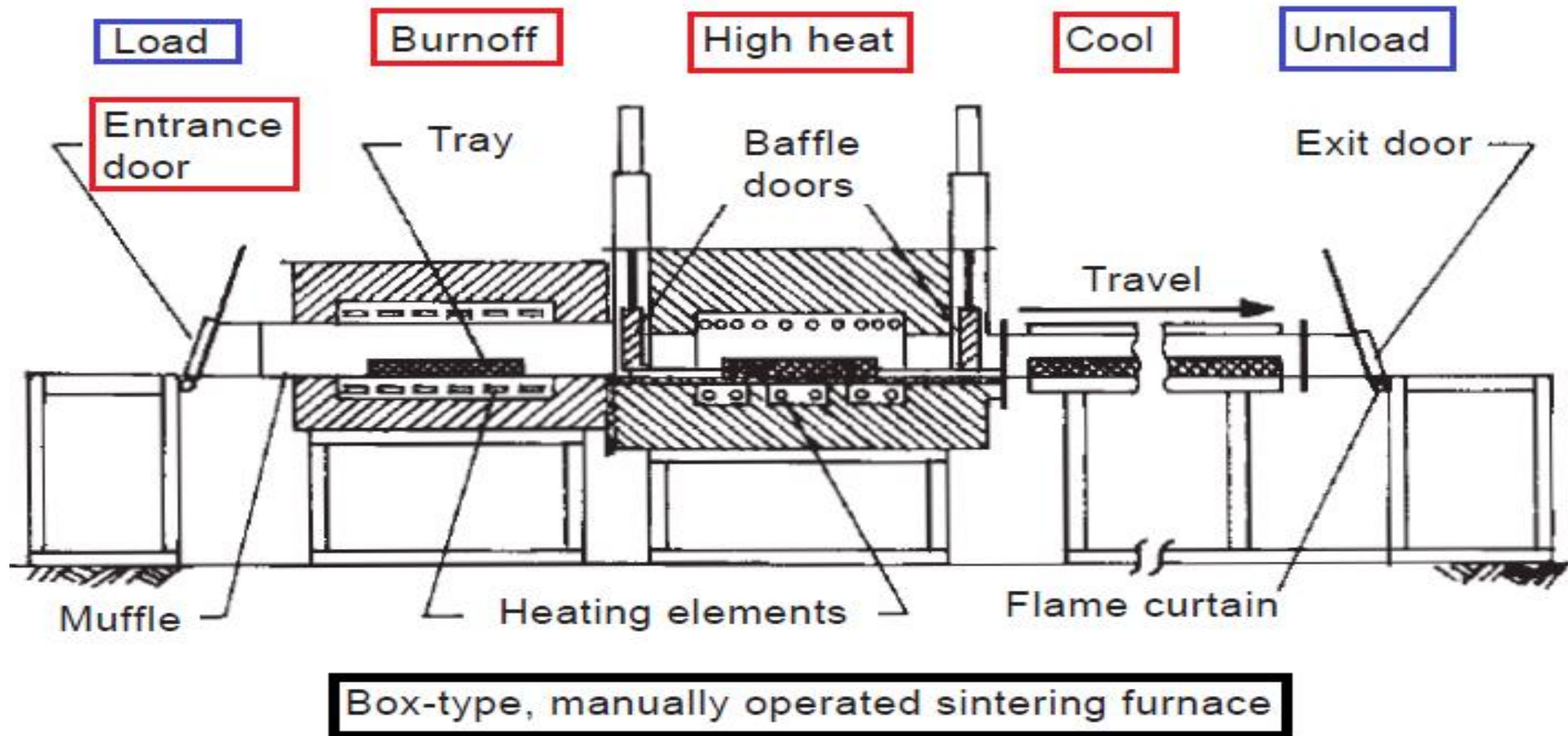


Vacuum furnace

1.3.8 Sintering Zones.

A conventional sintering furnace can be divided into three distinct zones. See Figure.

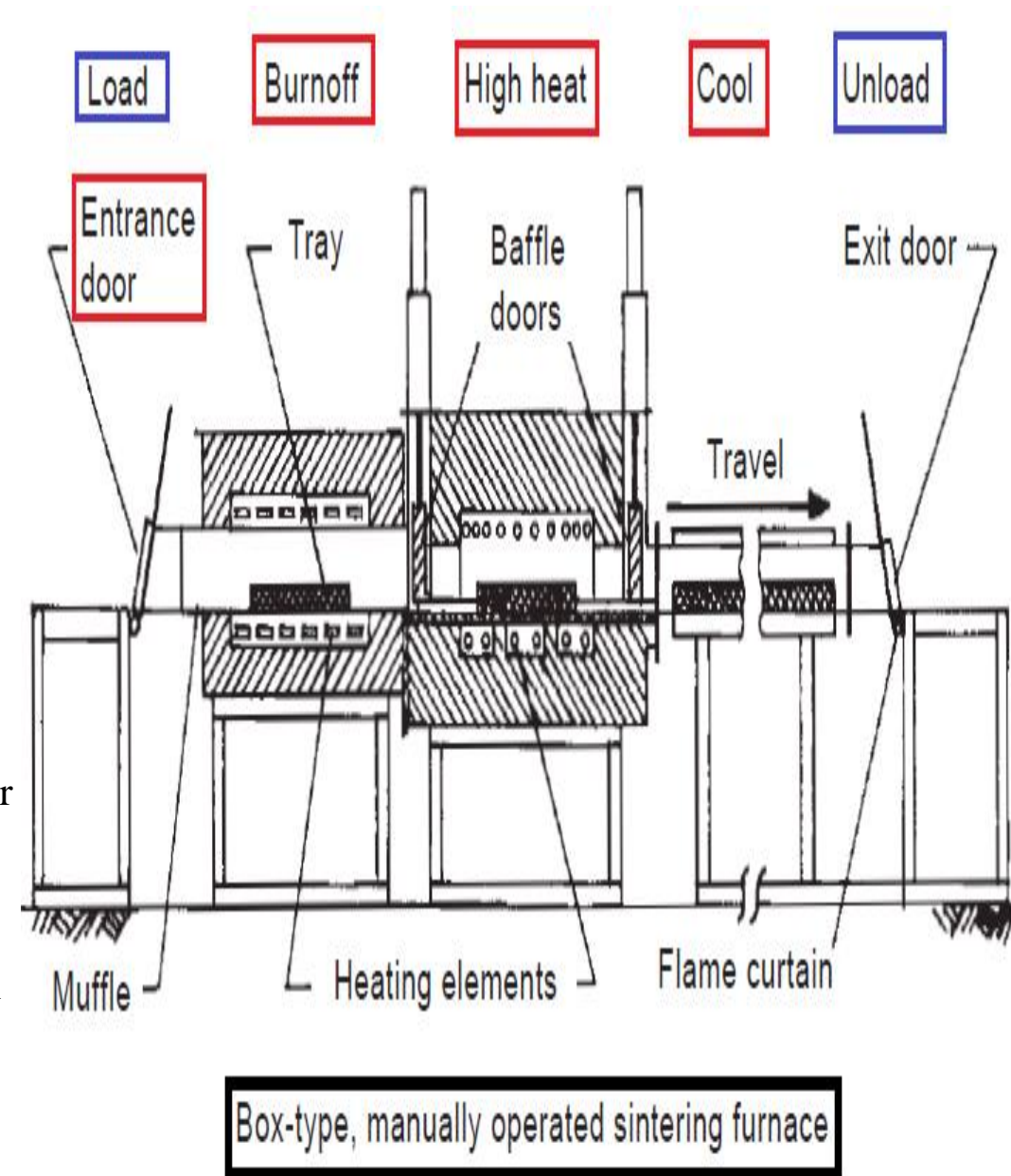
1. Burn-off and entrance zone.
2. High temperature sintering zone.
3. Cooling zone.



1.3.8 Sintering Zones.

First: Burn-Off and Entrance Zone.

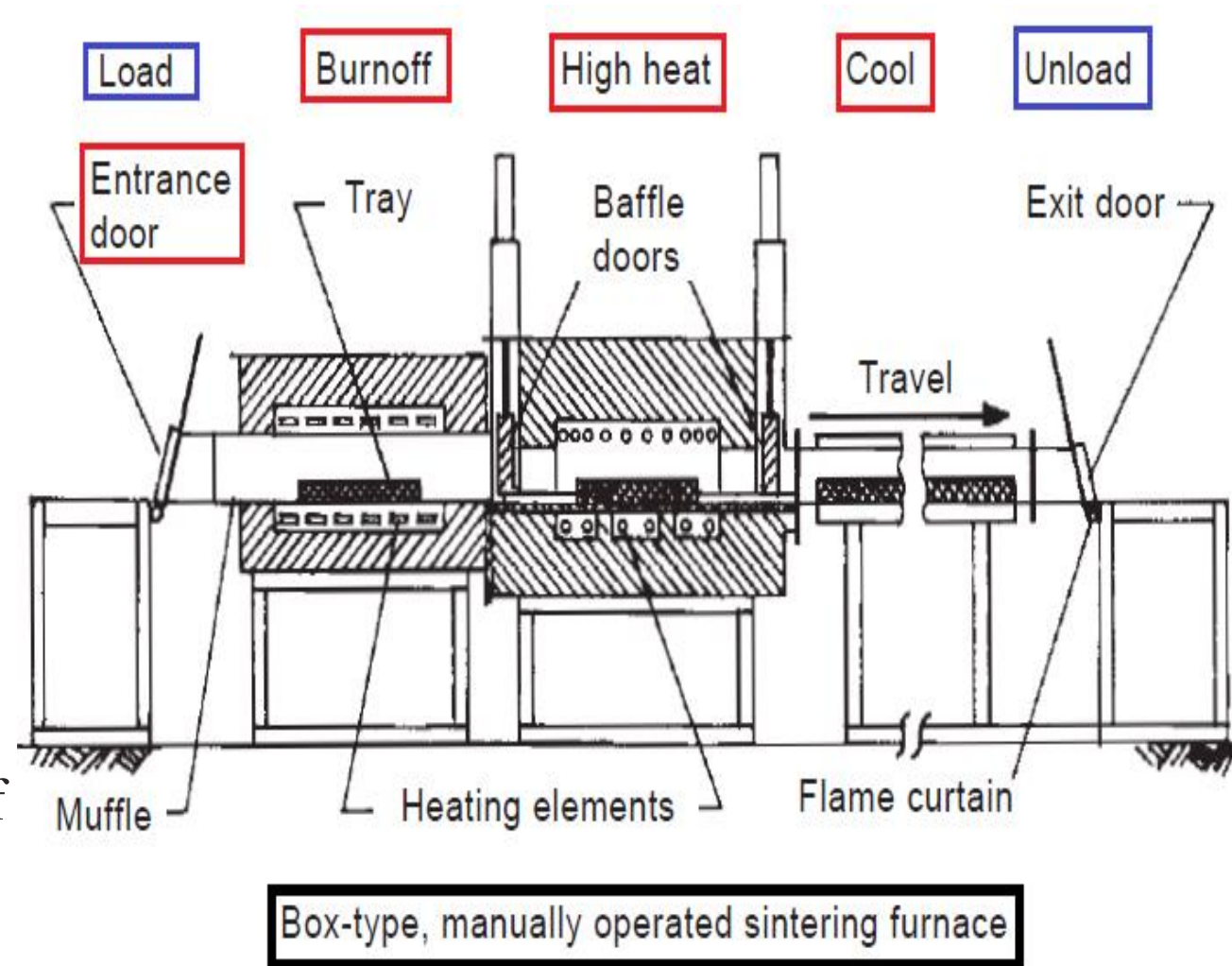
- ✓ Includes heating the green compacts slowly to a moderate temperature in order to avoid excessive pressures within the compact and possible expansion and fracture.
- ✓ The main function of this zone is the volatilizing (تطاير) and elimination of the admixed lubricant (الزيوت المخلوطة).
- ✓ The length of this zone must be sufficient to allow complete elimination of the lubricant before the compacts enter the high temperature zone. This is because the metal (ستيرات الزنك) resulted from volatilization or carbon resulted from elimination process could deposit on the furnace heating element and promote premature failure. Such deposits on refractory walls and cooling zones lead to poor heat transfer. On the other hand the compacts may be subjected to discoloration and possible undesirable chemical reactions.
- ✓ Atmosphere flow-ability achieved by provide sufficient atmosphere gas in order to discharge (expel - طرد) the lubricant vapours from entrance zone towards the furnace entrance, not into the high heat zone.
- ✓ The burn-off zone either separated with an air gap using a flame before the high heat zone, or using instead stearic acid or a wax compound during vacuum sintering metallic furnaces.
- ✓ Other wise the furnace and vacuum pumps get contaminated.



1.3.8 Sintering Zones.

Second: High Temperature Zone.

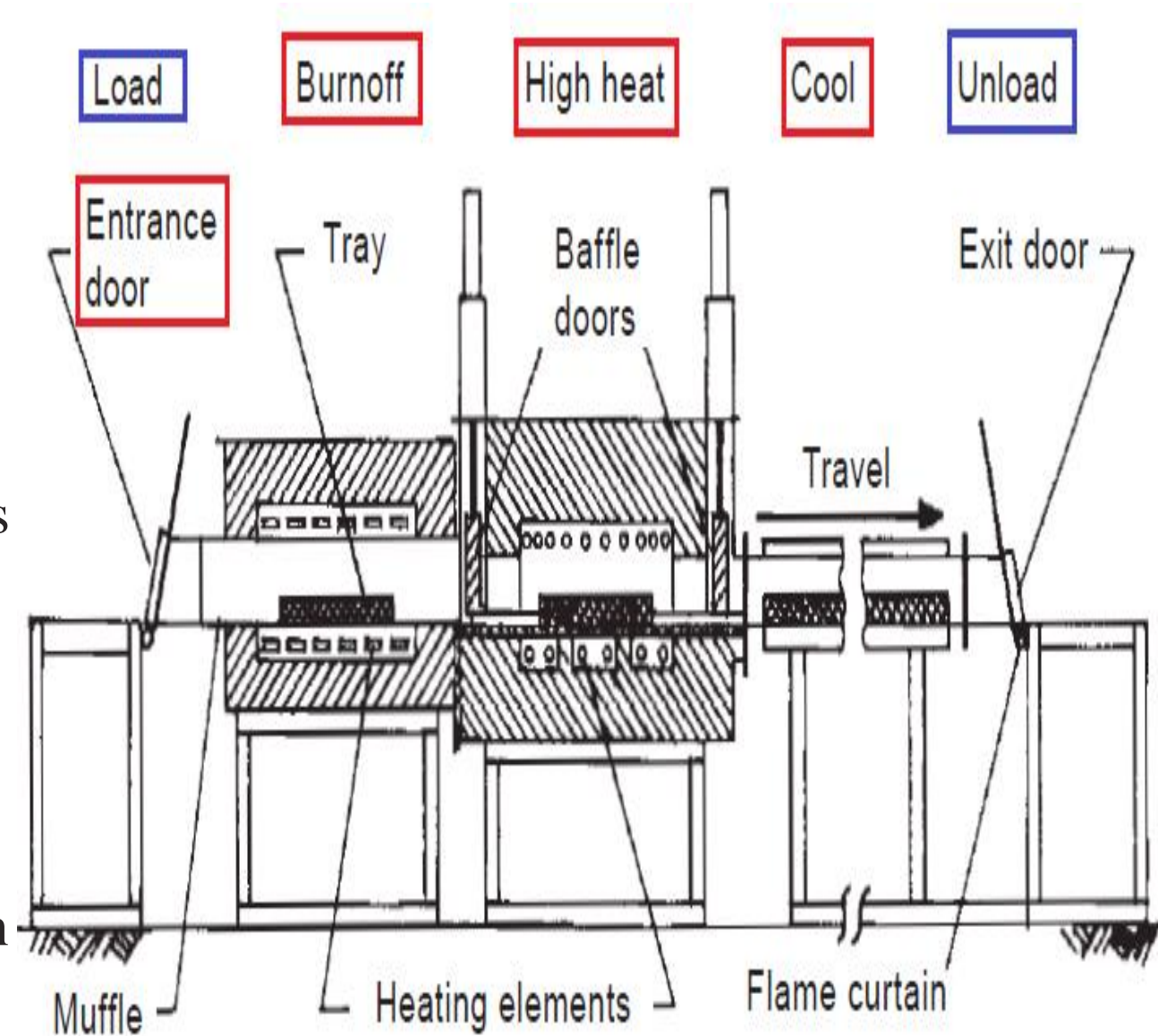
- ✓ In this zone, the actual sintering of the compacts takes place.
- ✓ Must properly heated to reach the desired temperature.
- ✓ So, sufficient length's zone with enough time at the desired temperature is attained to achieve the necessary end properties in the sintered parts.
- ✓ The lengths of this zone = length of burn-off zone.



1.3.8 Sintering Zones.

Third: Cooling Zone.

- ✓ This zone Consists of a short insulated section and a relatively long water jacketed section.
- ✓ The former cools down (يبرد بالتدریج) the parts at slow rate to avoid thermal shock in the compacts and the furnace.
- ✓ The former also provides cooling to a temperature low enough to prevent oxidation of the material upon exposure to the air. This is designed either automatically or manually.



Box-type, manually operated sintering furnace

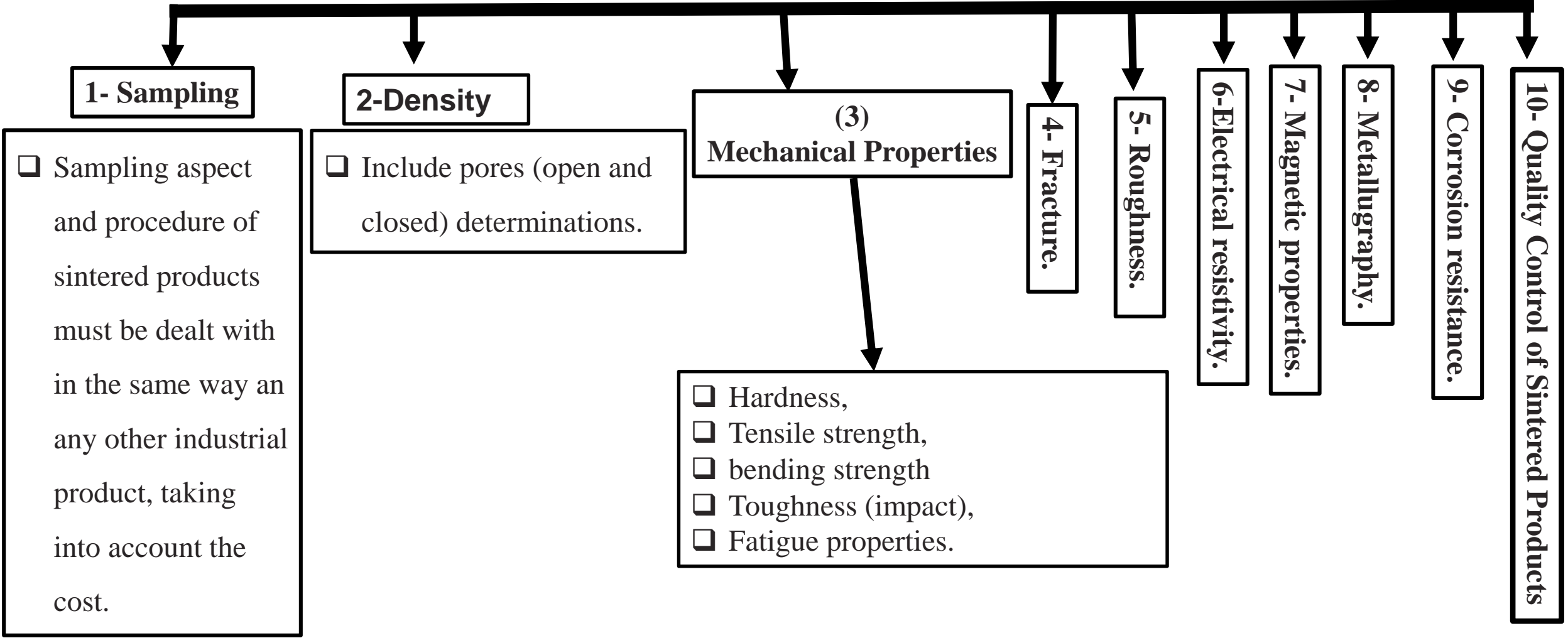
1.3.9 Joining Process For powder Metallurgy Parts.

- ✓ The ability to manufacture complex geometrical configuration of (PM) parts is represented by joining the (PM) parts to one another or to other cast/wrought products using welding technique.
- ✓ But welding of PM parts has concern with formation porosity, impurities, or a high carbon content in some PM parts.
- ✓ Porosity acts as a trap for impurities/inclusions which impact negatively on secondary operations such as welding when entrapped impurities in the pores could be deleterious to the weldability of the PM part.
- ✓ Therefore, widespread success in welding PM parts requires understanding of the influence of porosity, chemical composition, impurity level and overall cleanliness, upon weldment properties such as cracking, ductility and toughness, residual stresses and distortion in the weld zones of (PM) parts.
- ✓ Joining processes applicable for PM parts can be categorized as solid state and liquid (fusion) state.
- ✓ The solid state processes such as **diffusion bonding** and **brazing** are dominantly used for **lower density porous** parts.
- ✓ The liquid state (fusion weld) such as **arc welding** using gas tungsten arc (GTA), gas metal arc (GMA), **electron beam (EB)** and **laser welding** are dominantly used for **higher densities or minimal** porosity parts.
- ✓ Shrink fitting (press fitting), adhesive bonding, Joining metal injection moulded (MIM) parts, and Laser metal deposition are types of welding techniques used for bonding (PM) parts depending on the products conditions.

1.3.10 Sintered Metal Carbides

- Hard metals represent, together with sintered steels, the most important powder metallurgy products.
- They consist at of least one **basic metallic hard compound** + a **binder metal** + **alloy**.
- The **basic metallic hard compounds**, are carbides WC and TiC.
- The **binder metal** is mostly cobalt ranging between 3- 25 wt%.
- **WC-Co** represents the largest group in the entire hard metal field.

Quality Control OF Powder Metallurgy Materials.



1.3.11. a Relative density of a powder compact (Porosity).

- Defined as the total void part of the volume related to the entire volume of any porous material (ماده مسامیه). [total voids/ entire volume]
- It is subdivided into open and closed pores.
- **Open pores:** are the inter-connected pores, which reach the surface of the specimen at least on one side.
- **Closed porosity** represents the pores located inter-between the powder's particles and possible to melt and penetrate along the inter-particle interfaces of the particles during sintering causing separation (*lec.7*).
- **Porosity measurements** is achieved by determination its main parameters: Size , Distribution, Geometries, and its mechanical/physical/ or metallographic properties.

1.3.11.b Porosity Measurements.

➤ The total porosity (P_t) is measured by determining: mass (kg) and dimensions (mm).

➤ The total porosity (P_t) measurement in (%) is: $(P_t\%) = \frac{[\rho_{(fpm)} - \rho_s]}{\rho_{(fpm)}} * 100\%$.

Where:

$(P_t\%)$: total porosity percent; $\rho_{(fpm)}$: density of free porosity material.

➤ The open (inter-connected) porosity (P_o) is measured in (%) by the following equation:

$$(P_o\%) = \frac{(VP_o)}{V} * 100.$$

➤ The standard technique to count the porosity for hard metals is done by using the optical microscopy on ground and polished cross sections.

➤ The numbers of pores are counted or measured in % of the surface.

1.3.11.c The size distribution of inter-connected pores (open pores) measurements.

- The size distribution of interconnected pores is measured preferably by mercury porosimetry.
- Mercury is pressed into the porous solid and the intruded volume (الحجم المتداخل) is measured as a function of applied pressure.
- The method is used in powder metallurgy and ceramics for many other porous materials.
- Using the equation of Washburn: $r(p) = \frac{2\gamma \cdot \cos \theta}{P}$
- **where:**
 - **r**: radius of pore (assume circular);
 - **p**: the given pressure help penetrate the pore;
 - (**γ**): surface tension of the mercury ($\gamma = 480 \text{ MN m}^{-1}$);
 - (**θ**): contact angle between mercury and the material to be analyzed.

[Mercury does not wet most materials ($\theta \sim 90$ to 180°)].

Note: The method does not provide absolute data of pore sizes.

Mechanical Properties

1.3.11.d

1- Hardness.

- Hardness of a sintered material strongly affected by its density because voids in the structure of a material do not support of indenter.
- The indentation of a porous material should be considered in apparent hardness.
- The ISO standard on apparent hardness recommends Vickers as the reference method. However, allows Brinell and Rockwell methods as alternatives.

2-Tensile strength.

- Tensile strength (R_m) linearly with density range.
- Elongation at fracture (A) and impact strength (O_k) exhibit a stronger dependence on porosity.
- This holds for a number of sintered steels, whilst others show somewhat different behavior in strength with increasing slope at high densities.

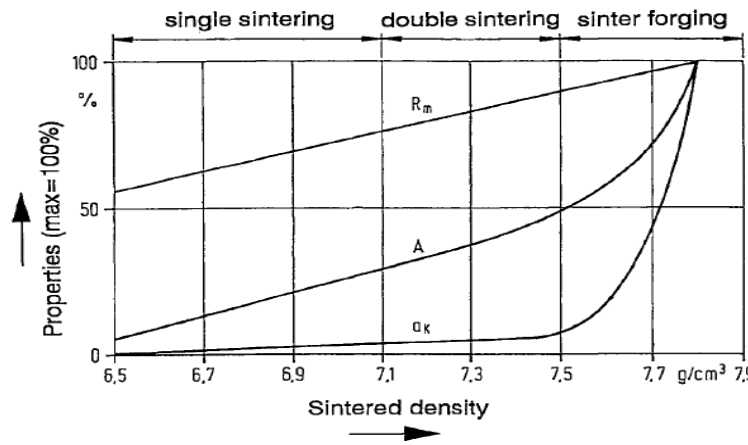
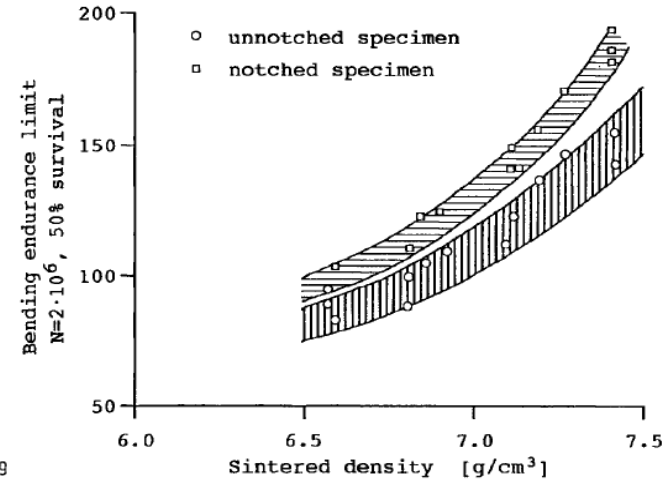


Figure shows materials with different porosities, in sintered iron and steel.

For the sintered ductile materials only

(3) Toughness (Impact tests).

The pores act as initiation sites for fatigue cracks more than the effect of notches sensitivity with conventional steels since the porosity diminishes (يقلل) the external notch effects.



Endurance limits on notched and un-notched sintered steel samples as a function of sintered density.

4- Bending Test.

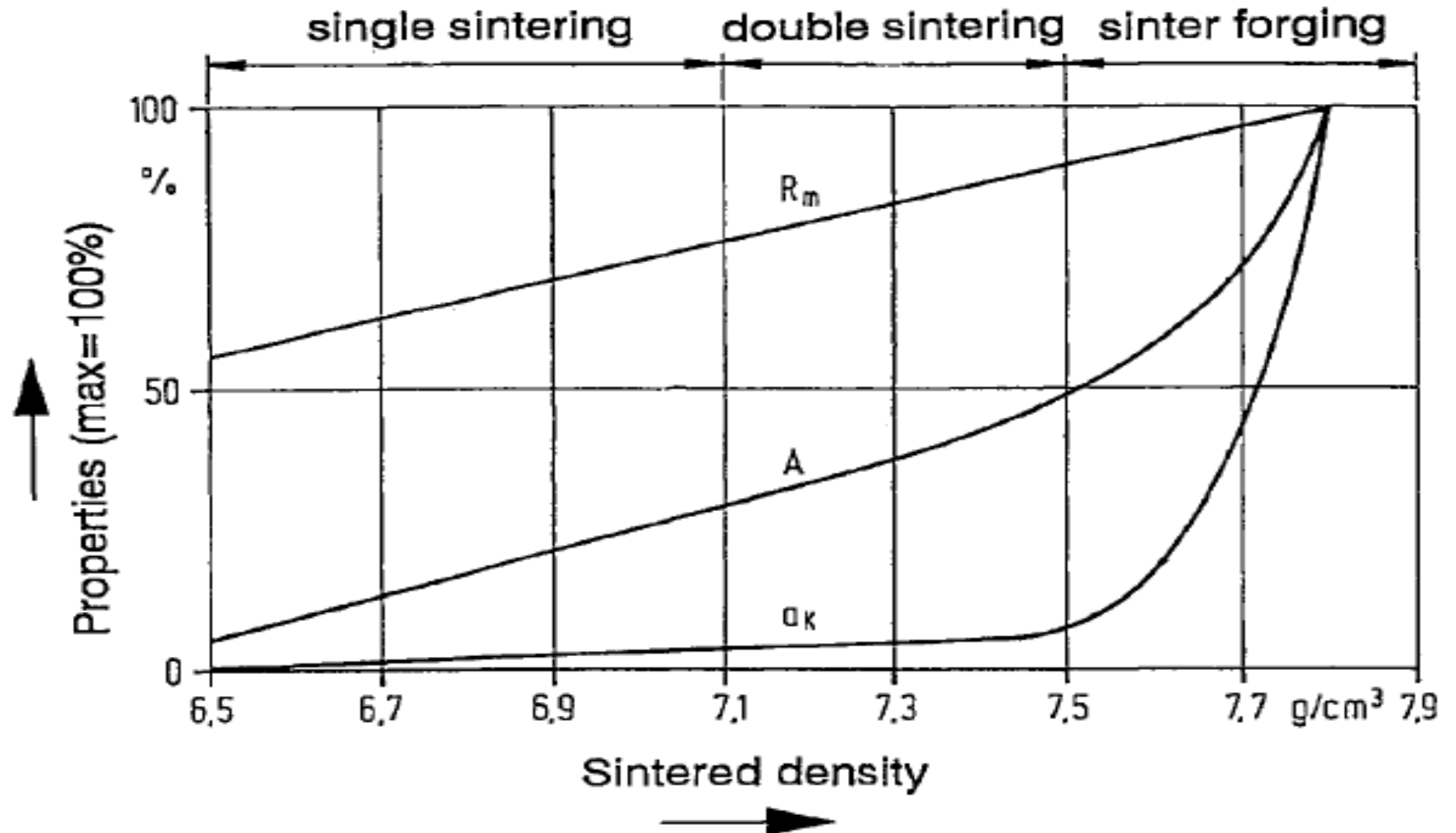
For Brittle hard metals & ceramics only.

5- Fatigue Test.

For the sintered ductile materials only

- Porosity affect is more important in fatigue tests than in other mechanical tests.
- Conducted after surface treatment, hardening and nitriding of sintered steels.
- All such treatments raise the fatigue limit, as in pore free materials.

Tensile strength



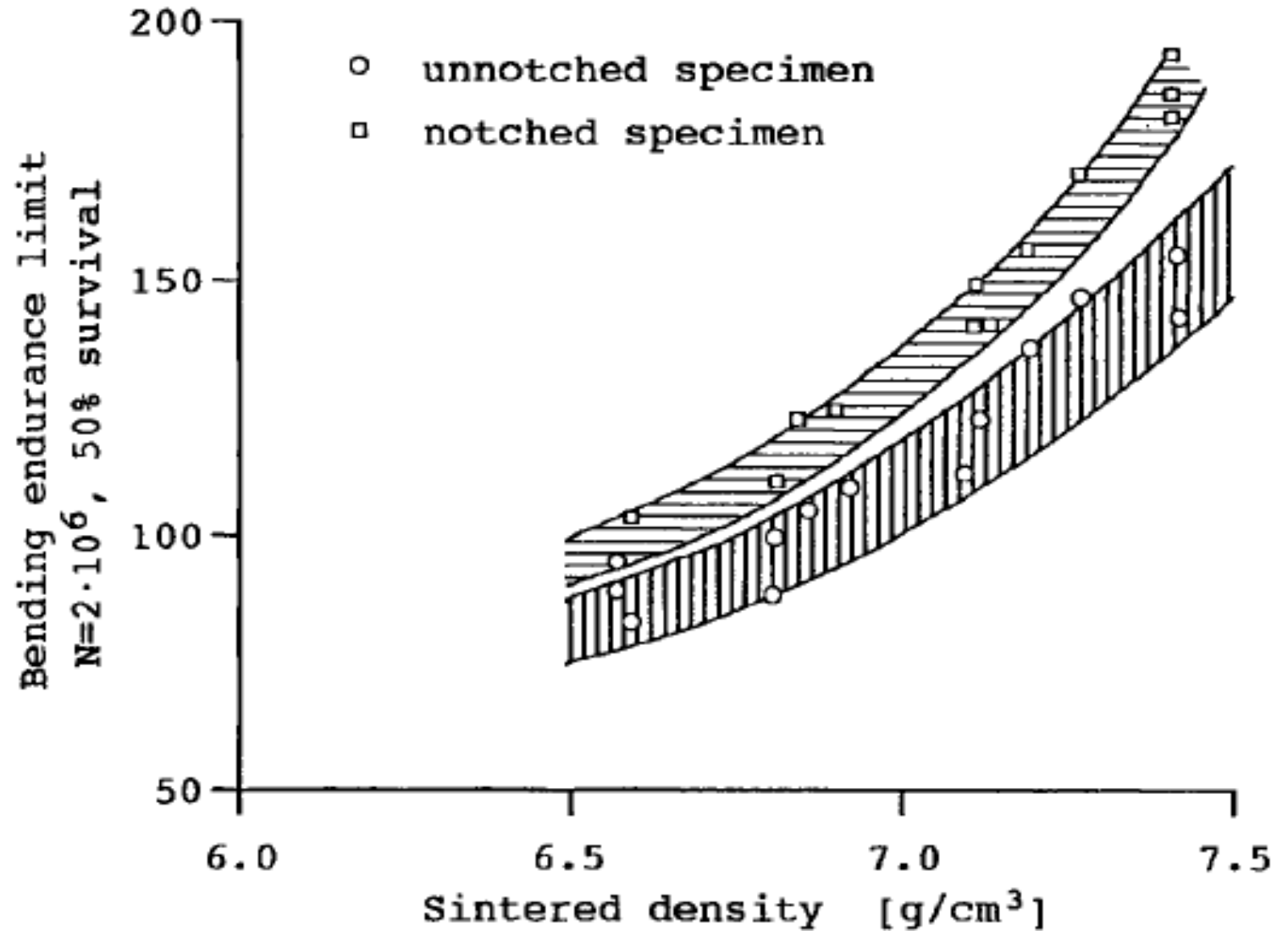
- ❑ Tensile strength (R_m) linearly with density range.
- ❑ Elongation at fracture (A) and impact strength (α_K) exhibit a stronger dependence on porosity.
- ❑ This holds for a number of sintered steels, whilst others show somewhat different behavior in strength with increasing slope at high densities.

(3)

Toughness (Impact tests).

□ For the sintered ductile materials only

The pores act as initiation sites for fatigue cracks more than the effect of notches sensitivity with conventional steels since the porosity diminishes (يقلل) the external notch effects.



Endurance limits on notched and un-notched sintered steel samples as a function of sintered density.

1.3.11 e Fracture properties for sintered products.

- Fracture in sintered materials has similar mechanisms known in wrought materials.
- Because inclusions and cavities are important in ductile fracture, the porosity in the sintered materials controls fracture.
- As sintered materials generally contain relatively large volume of coarse pores, localized internal necking is able to start at relatively low plastic strains.
- The large pores lead to high stress concentrations, thus accelerating the spread of fracture.
- So, the sintered materials are more complex in the stress strain response; their behavior depends on the work hardening characteristics of the matrix and on the pore size distribution and shape.
- Intergranular and cleavage (أنقسام) fractures are less common than ductile failures in sintered materials, although intergranular fracture may occur in sintered parts at inter-particle necks which may be imperfectly bonded, e.g. due to segregation of impurities at the interface. The pulling out of particles from a fracture surface, may be an indication of intergranular fracture.

1.3. 11. f Roughness properties for sintered product.

- The roughness of sintered parts is governed by the successive operations (عمليات متتاليه) the material is subjected to.
- In the powder pressing stage, it is influenced by the wear (نخر) of the dies.
- In the sintering stage the governing factors are the lubricant burn off and the reduction of any oxide on the powder surface.
- The roughness would be different if measured on the surfaces normal(vertical) to the pressing direction or on surfaces parallel to it.
- **A chisel stylus** is preferred for P/M parts because it bridges the negative gaps caused by the pores and will still measure any protrusions on the surface of the part.

1.3.11 g Physical Properties.

❖ **Electrical Resistivity for sintered product.**

- ❑ The evaluation of electrical resistivity for materials sintered is affected by several factors: e.g. oxide films, lubricant, compacting pressure, sintering parameters and powder characteristics.
- ❑ The measurement of electrical resistivity is done by a **Kelvin double bridge set up test.**

❖ **Magnetic properties for sintered product.**

- ❑ Affected by the presence of pores (size, shape, and their distribution in the mixture).
- ❑ Magnetic properties measurement devices for soft magnetic materials permit two types of test pieces: ring or bars.

❖ **Metallography for sintered product.**

- ❑ The purpose of this test is to study the type and morphology of pores which affect various properties.
- ❑ Metallographic preparation of sintered materials might lead to changes in the specimen surface which can cause erroneous (خاطيء) interpretation (تحليل) of the microstructure.

❖ **Corrosion Resistance for sintered product.**

1.3.12 Application of powder technology.

□ PM is widely used for a range of applications, such as dental restorations, automotive transmission parts, from biomedical to automotive industry sectors.

Material processes technology

- Casting
- Forging
- Stamping
- Powder metallurgy
- Metal injection molding
- Machining

- Ceramics
- Nuclear fuel
- Diamond tool
- Magnetic part*
- High melting point/High density material (W*, Mo)
- Cemented carbide
- Structural part* (iron, aluminum)
- Tribological part*
 - Oil impregnated bearing
 - Wear-resistant part
 - High heat-resistant wear-resistant part
- Electrical contact, electricity collecting part
- Friction material (break, clutch)

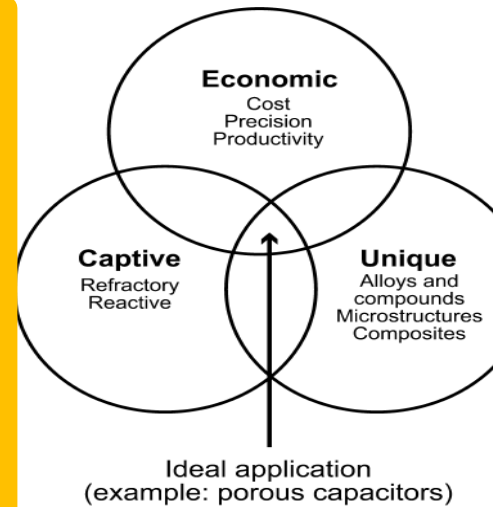
* Products produced by Powder Metallurgy Business Division

Position of powder metallurgy in material process technologies

1.3.12 Application of powder technology.

- Powder metallurgy (PM) is the production and utilization of metal powders.
- The three main reasons for using PM are **economic**, **uniqueness**, and **captive** (مقيد بتأثير البيئه) applications.
- For example, the dominant factor for the applications that require high volumes of parts with high precision, productivity, and cost is the **Economic Factor**. A good example of this segment is parts for the automotive industry (where approximately 70% of ferrous PM structural parts are used).

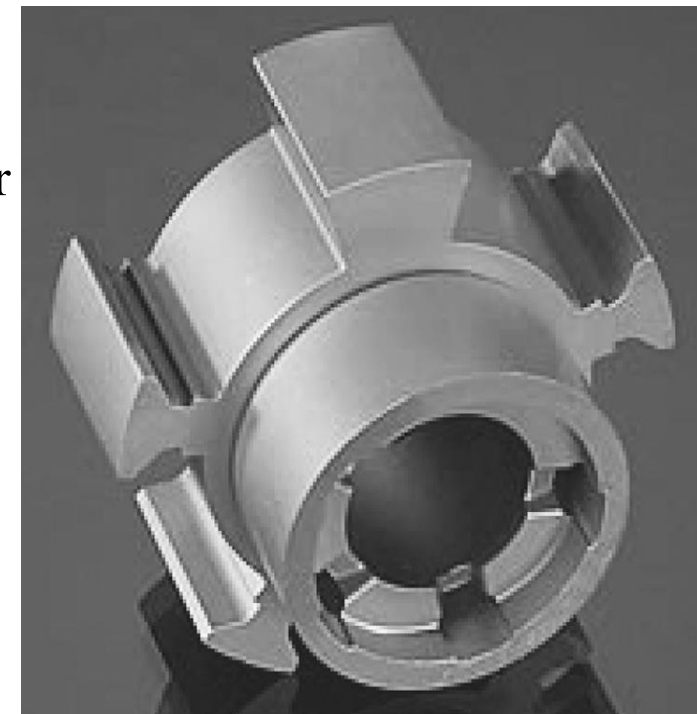
- On the other hand, some (PM) applications require using powder metals of materials that are difficult to process by other techniques, such materials are refractory and reactive metals, the dominant factor in this case is the **Captive Factor**.
- Other examples in this category are special compounds such as molybdenum disilicide and titanium aluminide, or amorphous (عديم الشكل) metals.



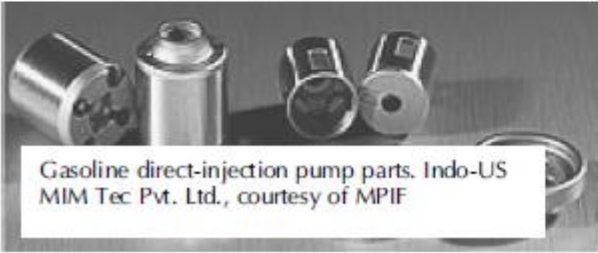
- While the dominant factor for the applications that require understanding the metallurgy field of the (alloys and compounds microstructures composites) that are used in engine, transmission, and chassis (هياكل) applications is the **Unique Factor**.
- This enables the engineers to use PM processing: for example, porous filters, self-lubricating bearings, dispersion strengthened alloys, functionally graded materials (e.g., titanium-hydroxyapatite), and cutting tools from tungsten carbide or diamond composites.

1.3.12 Application of powder technology/ Example.

- The variable valve timing (VVT) rotor consists of an assembly of a PM steel rotor and an adapter.
- The parts are joined by an adhesive, which joins them during the machining of cross-holes and other features on the inside diameter, and seals the joint between them.
- The assembly, used in a Chrysler V-6 engine, is mounted to the engine camshaft.
- The rotor is formed to a density of 6.8 g/cm^3 , with mechanical properties are ($\sigma_{\text{max}} = 415 \text{ Mpa}$), ($\sigma_y = 380 \text{ Mpa}$), and a (160 Mpa) fatigue limit.
- The adapter is formed to a density of 6.9 g/cm^3 , with ($\sigma_{\text{max}} = 400 \text{ Mpa}$), and ($\sigma_y = 365 \text{ MPa}$).
- After sizing and grinding, there is no other machining performed on the rotor.
- The adapter is not machined prior to assembly and is made to net shape with vertical slots for oil feeding.



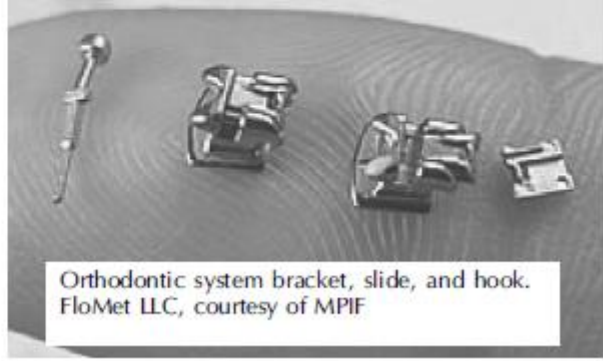
1.3.12 Application of powder technology.



Gasoline direct-injection pump parts. Indo-US MIM Tec Pvt. Ltd., courtesy of MPIF



Powder metallurgy aluminum camshaft-bearing cap. Metal Powder Products Co., courtesy of MPIF



Orthodontic system bracket, slide, and hook. FloMet LLC, courtesy of MPIF



Carrier and one-way rocker clutch assembly. GKN Sinter Metals LLC, courtesy of MPIF



Sector gear and fixed ring. Cloyes Gear & Products Inc., courtesy of MPIF



Helical gear and spur pinion. Capstan Atlantic, courtesy of MPIF

1.3.13 Advantage of powder technology.

- Powder technologies processing has the potential to permit the selective placement of phases or pores to tailor the component for the application.
- The capability to replicate parts in high volumes is very attractive to design engineers using different (PM) production methods.
- The ability to fabricate complex shapes to final size and shape is valuable.
- Powder metallurgy offers the potential to produce parts with high volumes and for applications where the volumes are large or small.
- The PM process is material and energy efficient compared with other metal forming technologies.
- Powder metallurgy is cost effective for making complex-shaped parts and minimizes the need for machining.
- A wide range of engineered materials is available, and through appropriate material and process selection the required microstructure may be developed in the material.
- Powder metallurgy parts have good surface finish and they may be heat treated to increase strength or wear resistance.
- Dimensional precision for PM is a good,

1.3.14 Disadvantage of powder technology.

- Powder technologies processing does not have the enough potential to increase the impact resistance properties in case of using the ferrous PM parts, as they have lower ductility which reduces the impact resistance compared with wrought steels.
- The majority concerns of PM parts are porous and consideration must be given to this when performing finishing operations.
- The strength and toughness of powder metallurgy products are poor, this is because the compaction formed by powder, the internal pores cannot be completely eliminated. Therefore, the strength and toughness of powder metallurgy products are worse than those of castings and forgings with corresponding components.
- Powder metallurgy cannot be made into large products. Since the fluidity of metal powder is worse than that of molten metal, its shape and size will be limited to a certain extent.

1.3.13 Advantages of the Powder Metallurgy Process *cont..*

- ✓ Special materials can be processed.
- ✓ Material powder metallurgy can manufacture refractory metals as well as compounds, and porous materials.
- ✓ Saves metal and reduces costs.
- ✓ Preparation of high-purity materials.
- ✓ Sintering is carried out in a vacuum and a chemically reducing atmosphere so oxidation will not contaminate the material.
Therefore, product purity is relatively high.
- ✓ Correctness of material distribution.
- ✓ The powder metallurgy method can ensure the correct composition and uniform proportioning of the material being used.
- ✓ Powder metallurgy is suitable for the production of products with a large number of uniform shapes, such as gears and other products, greatly reducing production costs.