

MECH 3700

Part VI – Powder Metallurgy

Powder Metallurgy



Gears

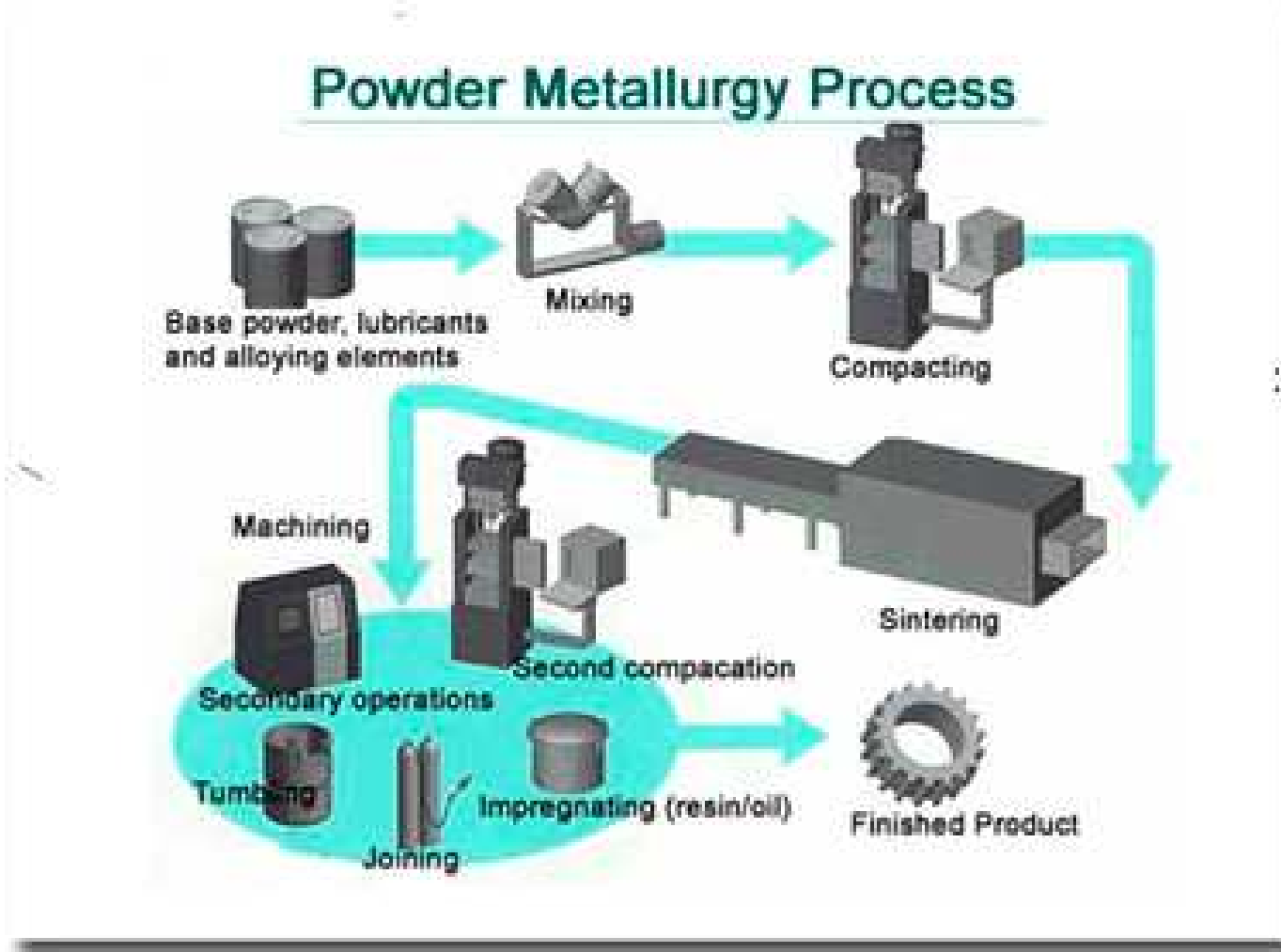


Machining tools
esp. tungsten carbide



Bushings

Powder Metallurgy



[Video](#)

Part 6: Goals / key points

1. Main reasons for powder metallurgy
2. Powder production methods
 - Main advantages and disadvantages of each method
 - Comparison of different methods
 - Methods that produce fine powder
 - Methods that produce coarse powder
 - Methods with good powder size control
 - Methods that produce spherical powder
 - Methods that produce irregularly-shaped powder

Part 6: Goals / key points

3. Powder characteristics
4. Powder conditioning
5. Consolidation mechanisms and methods
 - Cold compaction and sintering
 - Hot compaction
 - Advantages and disadvantages

Powder Metallurgy

Powder metallurgy is a group of manufacturing processes in which solid components are made by consolidating powders

- compaction and sintering

Powder metallurgy first used in ancient Egypt ~3000 B.C. to make iron tools

Modern use started in early 1900s: tungsten filament in light bulbs

Tungsten has high melting temperature (3400°C) and very high hardness

- other manufacturing processes (casting, machining and deformation processes) are thus difficult



6.1 Advantages of Powder Metallurgy

Advantages of powder metallurgy:

- Produces *net* or *near-net* shape with minimal material wasted and requires minimal finishing machining
- Produces parts with a composition that cannot be obtained by processing the material through a liquid state (e.g., for hardening with disperse oxide particles)
- Avoids solidification defects
- Direct and precise control over the grain structure or microstructure
- Controls the size of voids

6.1 Advantages of Powder Metallurgy

Advantages of powder metallurgy:

- Eliminates composition segregation effects
- Can produce components with controlled levels of porosity (filters, oil impregnated bearings); can *impregnate* porous components with lubricants e.g. bushings
- Can process metals that cannot be processed in a liquid state e.g. due to melting temperature (tungsten)

6.1 Advantages of Powder Metallurgy

Example with tungsten

Symbol: W (*Wolfram*)

$\rho=19.25 \text{ g/cm}^3$ (1.7x lead)

$T_m=3387\text{-}3422^\circ\text{C}$, brittle at low T, oxidizes at high T

Ores:

Wolframite $(\text{Fe,Mn})\text{WO}_4$

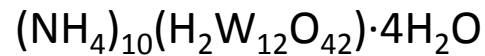
Scheelite - CaWO_4

WO_3 (minor quantities)

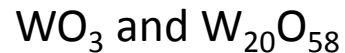


6.1 Advantages of Powder Metallurgy

Ores are processed into ammonium paratungstate



Thermally decomposed at 600°C into



Oxide powder reduced to metal powder by reaction with hydrogen

Tungsten powder consolidated into solid by *sintering*; filaments are produced by drawing

- flow stress ~ 1.5 GPa for 5% tensile strain

6.1 Advantages of Powder Metallurgy

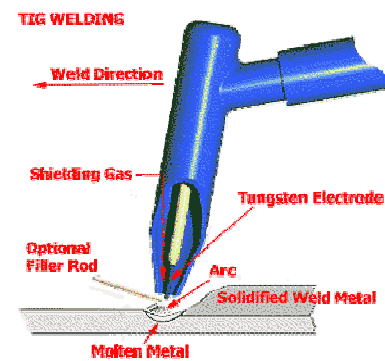
Used where high density is required

- Balancing weights, counterbalances



Used in high temperature applications

- Rocket engines, welding electrodes, spark plug electrodes



6.2 Powder Production

6.2 Powder Production

Several process are used to produce powdered metal

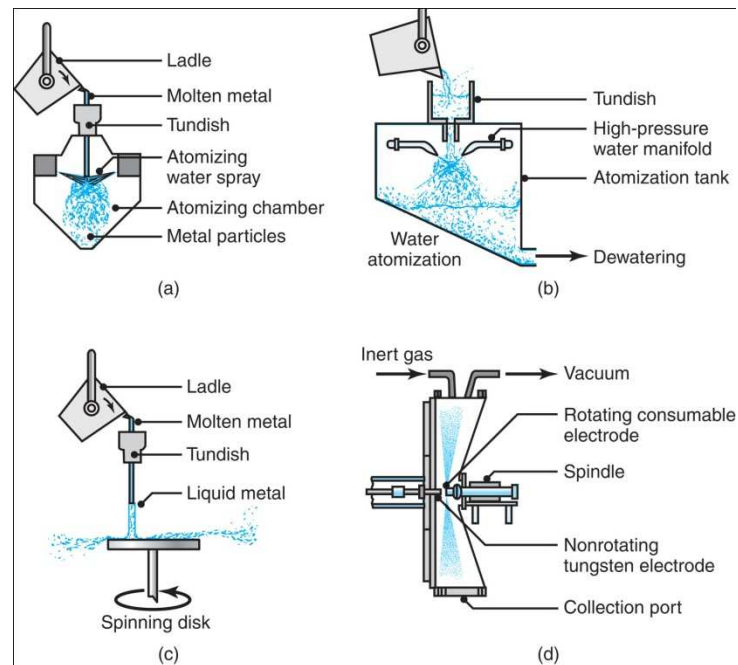
- Depends on metal produced
- Affects size/shape of particles

6.2 Powder Production

Atomization

A liquid metal is dispersed into fine droplets by impingement of water or gas jets, or by centrifugal force.

Most common method of powder production



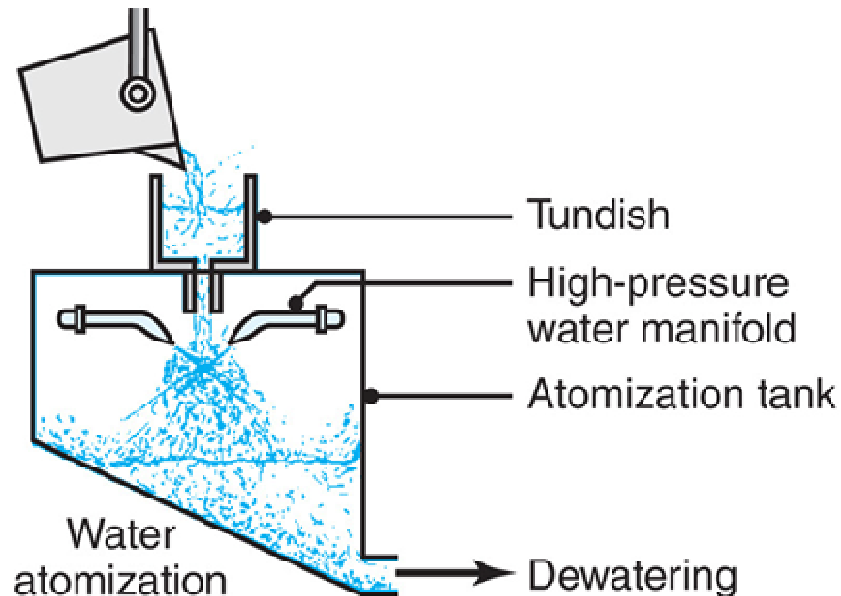
6.2 Powder Production

Water Atomization

Molten metal flows by gravity

Water jets 5 – 20 MPa hit the molten metal and disperse into fine droplets

Very rapid cooling into irregularly-shaped particles



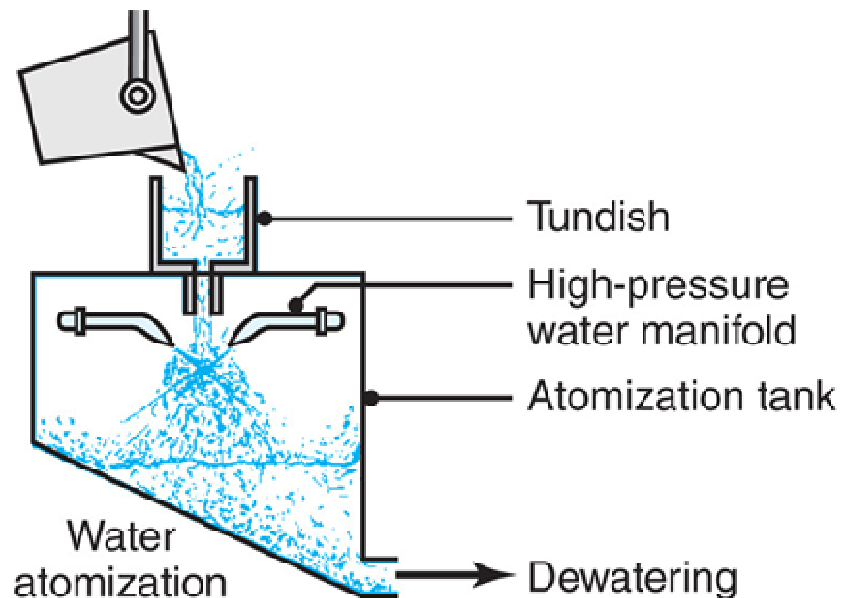
6.2 Powder Production

Water Atomization

Particle size typically 1.5 to 800 μm ; 95% of particles < 150 μm

Very high production rates

Main problem is contamination by surface oxides (can be removed by reduction process)



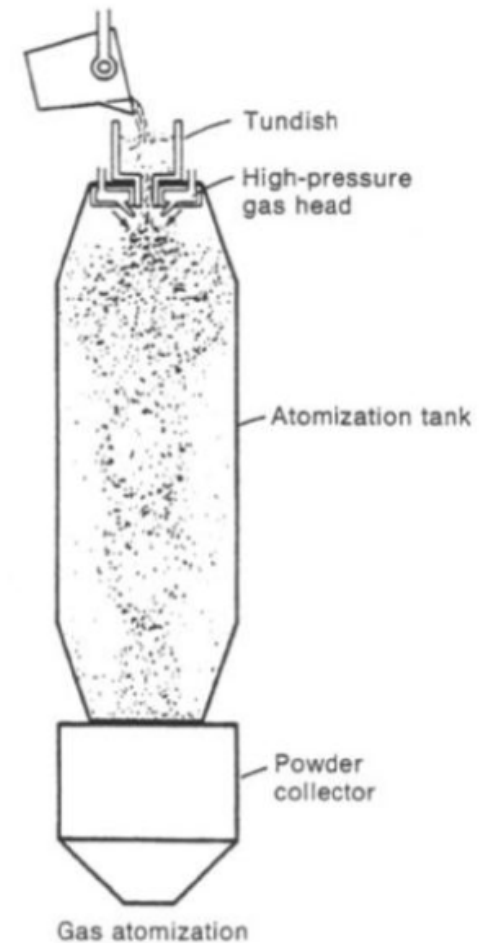
6.2 Powder Production

Gas Atomization

Cooling produced by radiation and convection

Cooling rate lower than in water atomization; produces approximately spherical particles

Larger size atomization tank is necessary



6.2 Powder Production

Gas Atomization

Typical particle size 100 to 300 μm

Maximum particle size can be estimated:

$$d_{\max} = 2.7 \cdot 10^4 \cdot \gamma^{0.85} \cdot \eta^{0.15} \cdot \rho^{-1} \cdot v^{-1.85}$$

where

γ is surface tension of liquid metal

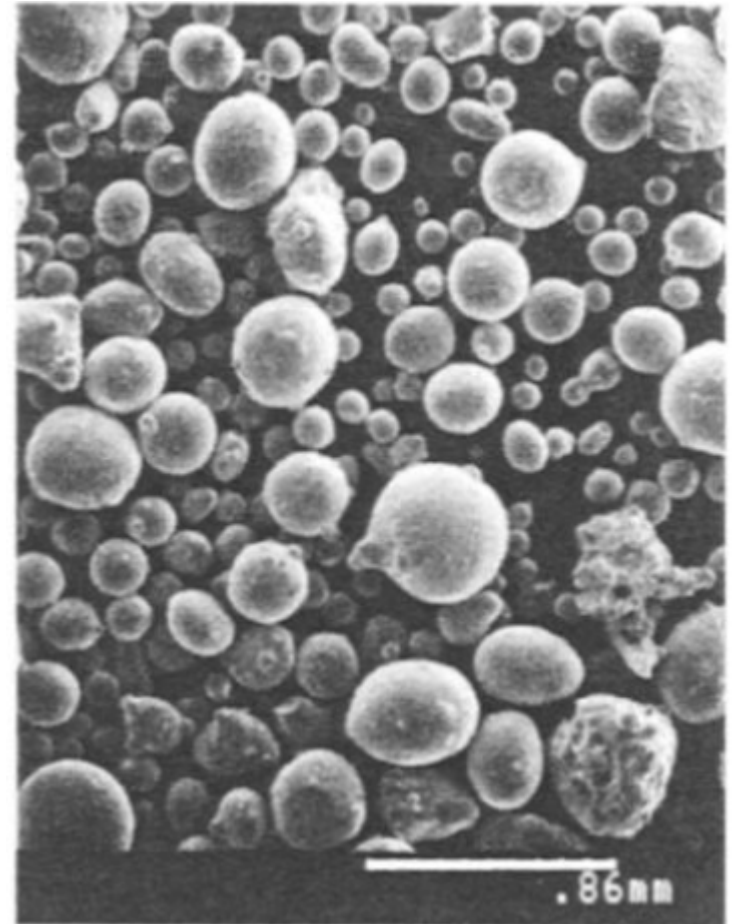
η is dynamic viscosity of liquid metal

ρ is density of liquid metal

v is velocity of the gas

(all parameters in base SI units)

Gas velocity is usually $> 100 \text{ m/s}$



6.2 Powder Production

Gas Atomization

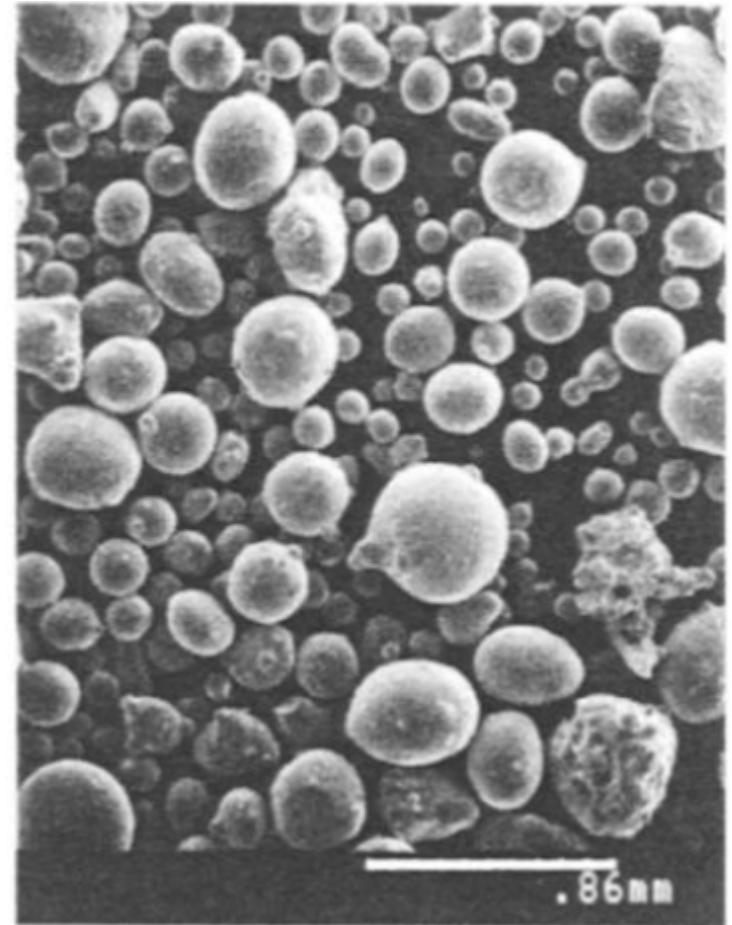
Contamination and oxidation is minimized:

- Tundish within vacuum chamber
- Inert gas used in atomization

Powder may be contaminated by refractory particles from nozzle, broken by molten metal flow (also applies to water atomization)

Argon gas may be entrapped in particles, increasing porosity

Better quality particles compared to water atomization but lower productivity and higher cost

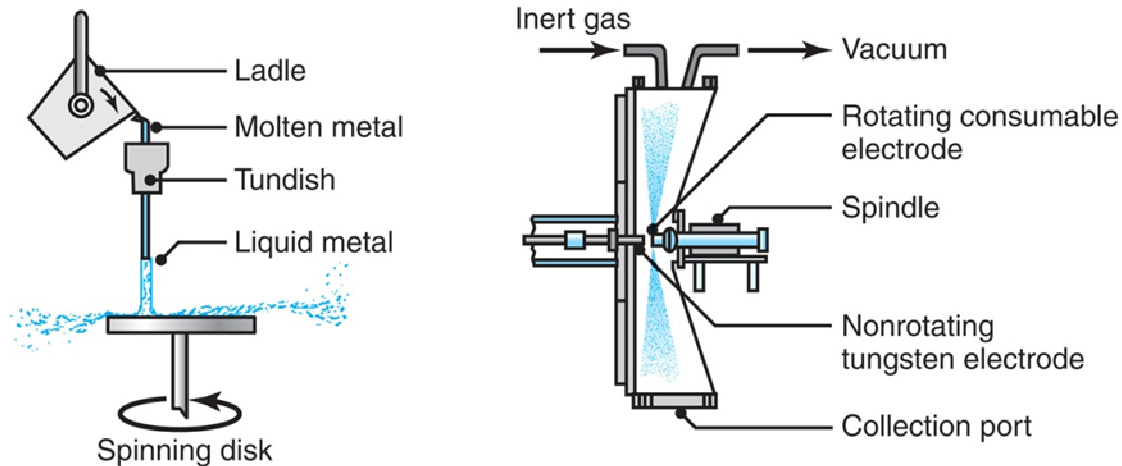


6.2 Powder Production

Centrifugal Atomization

Molten metal is dispersed into fine particles by centrifugal force

- A rotating disk or cup breaks up a molten stream (left)
- A molten (consumable) electrode breaks up into small particles (right). The electrode spins at $\sim 15,000$ rpm; inert gas (helium) limits oxidation



6.2 Powder Production

Centrifugal Atomization

Average powder size can be estimated:

$$d = \frac{0.4}{n} \cdot \left(\frac{\gamma}{R \cdot \rho} \right)^{\frac{1}{2}}$$

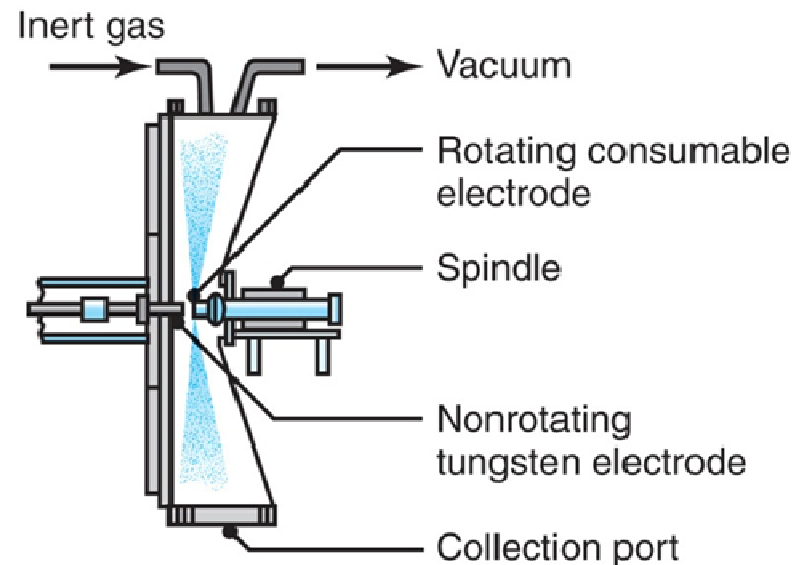
where

n is rotational speed (rpm)

γ is alloy surface tension

R is electrode radius

ρ is density of alloy



Rotational speed is usually 10,000 to 20,000 rpm

6.2 Powder Production

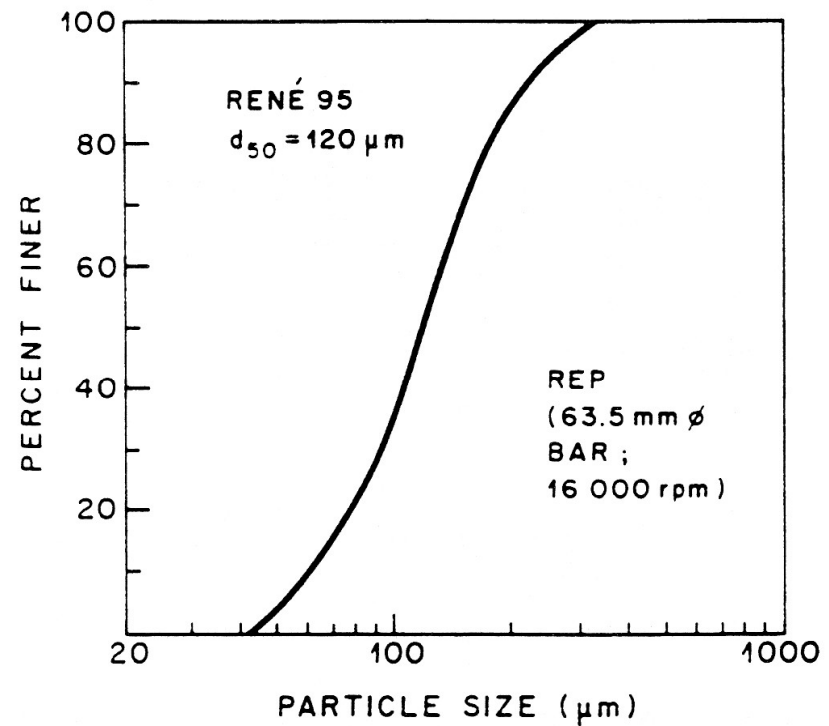
Centrifugal Atomization

No contamination effects, narrow distribution of particle sizes (50 – 250 μm)

Better quality than in gas atomization

Lower productivity

Higher cost



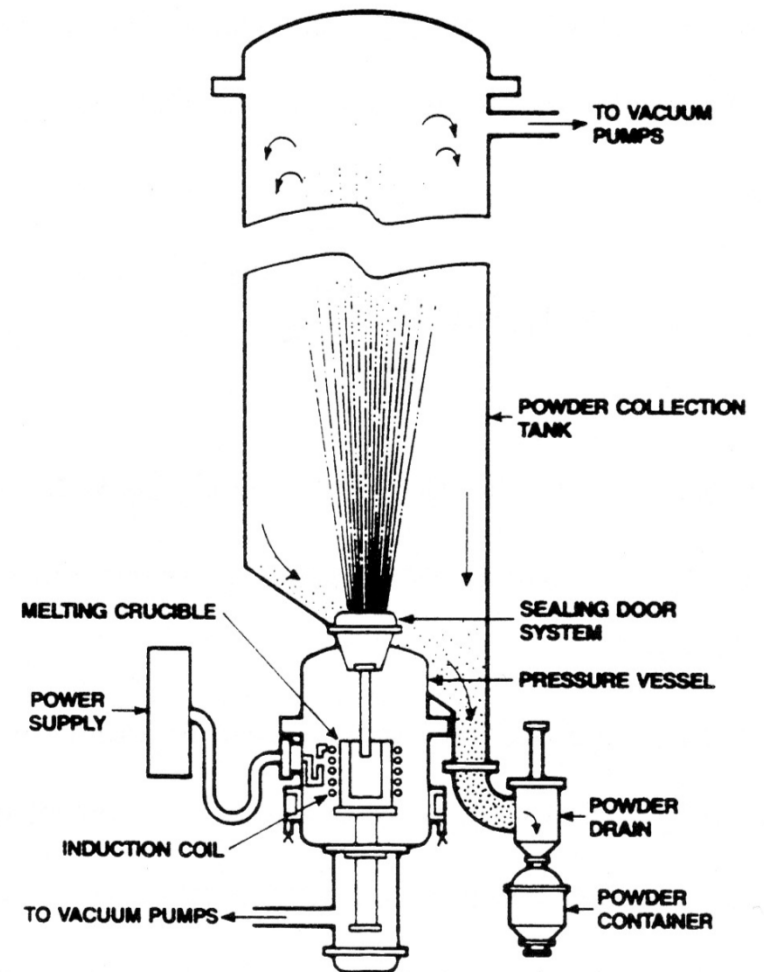
6.2 Powder Production

Soluble Gas Atomization

Alloy is saturated by hydrogen melted in melting crucible (recall gas solubility in molten alloys)

Saturated alloy is sprayed into high vacuum chamber

Low hydrogen partial pressure causes 'explosive' evacuation of hydrogen, breaking alloy into small droplets

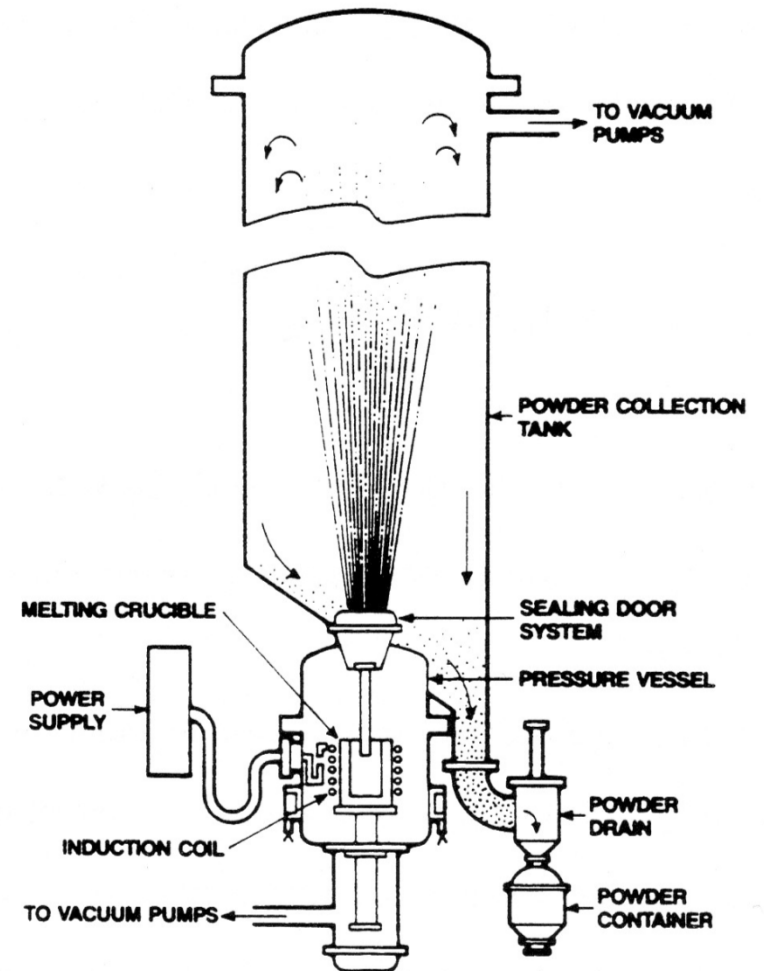


6.2 Powder Production

Soluble Gas Atomization

Fine powders with particle size $< 20 \mu\text{m}$ can be produced

Process parameters must be fine tuned to eliminate hydrogen (porosity) before solidification



6.2 Powder Production

Reduction of Metal Oxides

Metallic powders may be produced from reduction of ores



6.2 Powder Production

Reduction of Metal Oxides

Höganäs process: Magnetite (Fe_3O_4) is crushed, mixed with coke and limestone and heated to 1260°C

- Carbon reacts with oxygen, reducing magnetite to elemental iron
- Limestone reacts with impurities (e.g. sulphur) forming gangue, preventing contamination of metal



6.2 Powder Production

Reduction of Metal Oxides

Höganäs process: The powder produced after heating contains $\sim 0.3\%$ C and 1% O

- gangue and iron are magnetically separated
- iron powder is annealed at 870°C in atmosphere of dissociated ammonia ($2\text{NH}_3 \rightarrow \text{N}_2 + 3\text{H}_2$) to remove C and reduce oxygen to $\sim 0.3\%$
- typical powder size after subsequent grinding $< 180 \mu\text{m}$

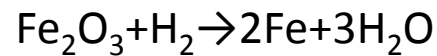


6.2 Powder Production

Reduction of Metal Oxides

Pyron process: Mill scale is crushed and oxidized to uniform Fe_2O_3 composition

Oxide reduced at 980°C with hydrogen



to produce coarse powder

Coarse powder is mechanically crushed to give particles $< 180 \mu\text{m}$

Oxide reduction is also used to produce tungsten, molybdenum and nickel powders

6.2 Powder Production

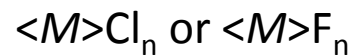
Precipitation from Gas

Some metals can form gas at low temperatures when combined with chlorine, fluorine or CO

Carbonyls:



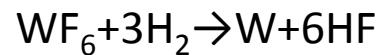
Halides (chlorides and fluorides of V, Nb, W, Hf, Ti, Ag, U):



6.2 Powder Production

Precipitation from Gas

Reaction in gas phase reduces metal to elemental form:



Solid metal particles precipitate from gas with size $< 1 \mu\text{m}$

Processing metals through gas compounds allows very precise composition control and isotope separation

Enrichment of uranium ($\text{U}^{235} + \text{U}^{238}$) with U^{235} isotope from natural composition is accomplished using centrifugal separation of UF_6 gas.



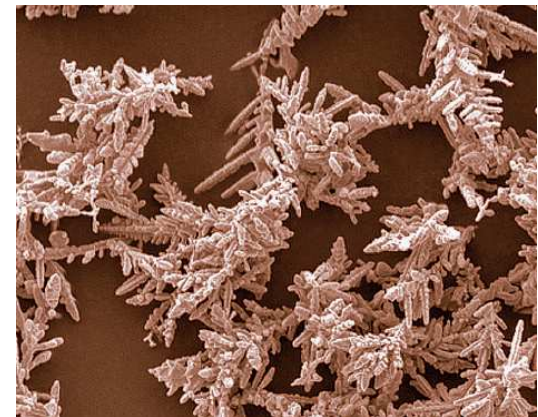
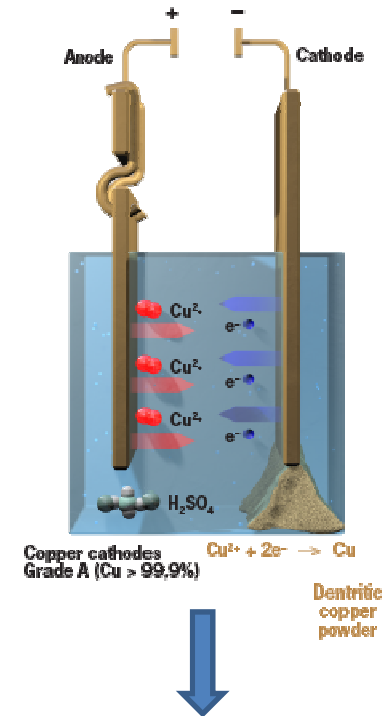
6.2 Powder Production

Electrolysis

Copper, manganese and chromium powders are produced by electrolysis

Copper powder produced by electrolysis of copper sulphate

Produces dendritic copper particles (99.9% Cu) on lead cathodes



6.2 Powder Production

Electrolysis

Produced powder is washed, filtered and dried in a furnace

95% of particles < 45 μm can be obtained

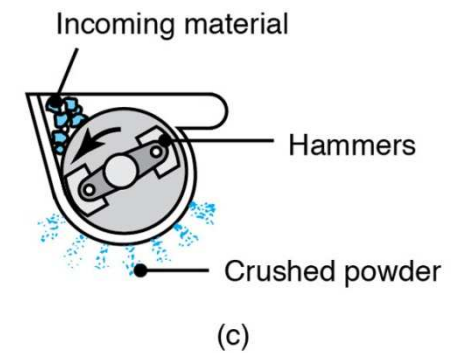
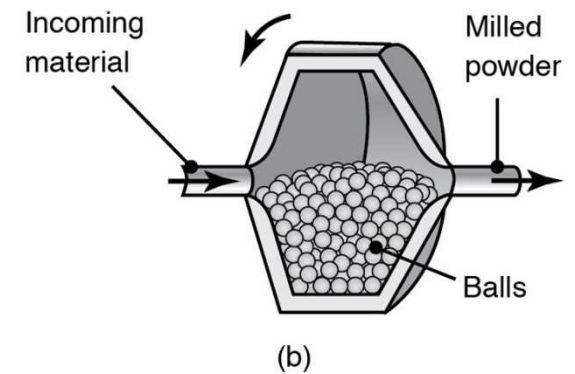
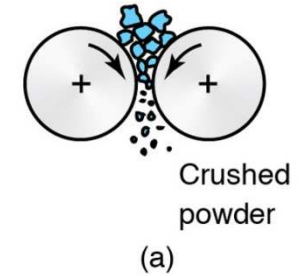
Electrolysis process produces some of the purest powders available



6.2 Powder Production

Pulverization

Mechanical process to produce powders of hard and brittle materials: *crushing, ball mill or hammer mill*



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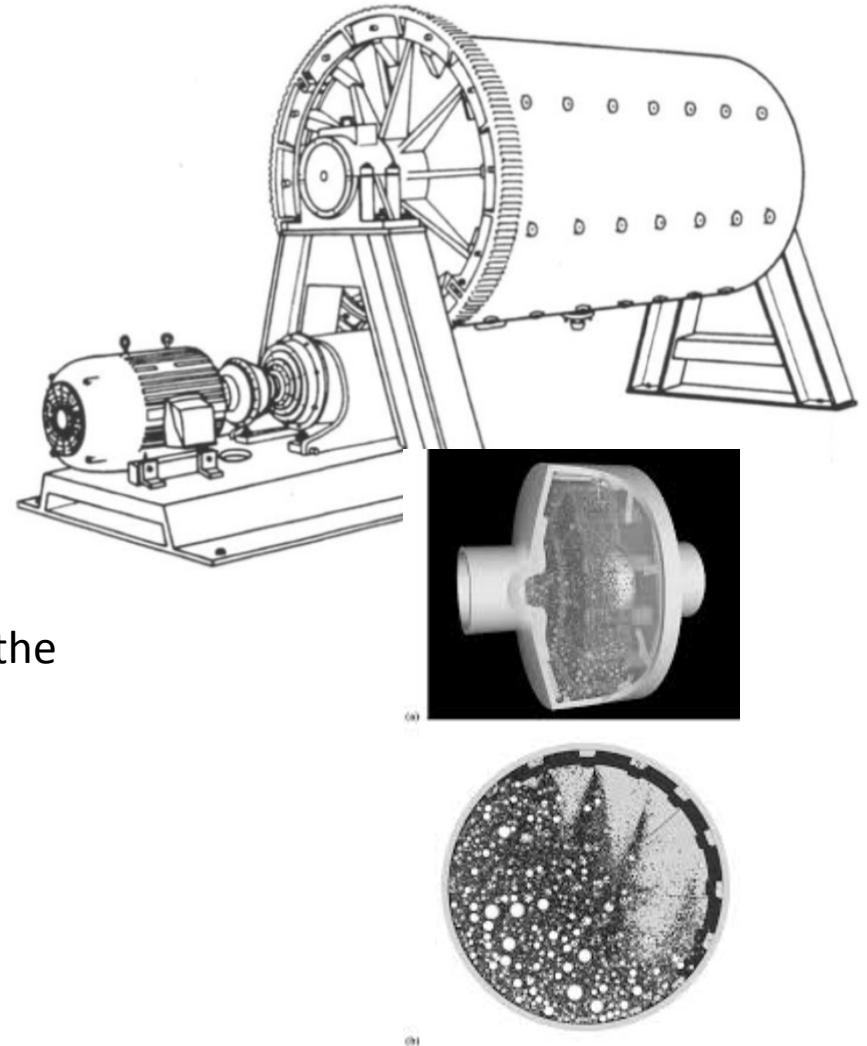
6.2 Powder Production

Pulverization

In a ball mill, hardened balls are placed in a rotating cylinder with the coarse powder (the charge)

Rotation of the tumbler produces pulverization by:

- the impact of hardened balls against charge
- the attrition due to rubbing action between the charge and hardened balls
- the shear or cleaving of charge particles



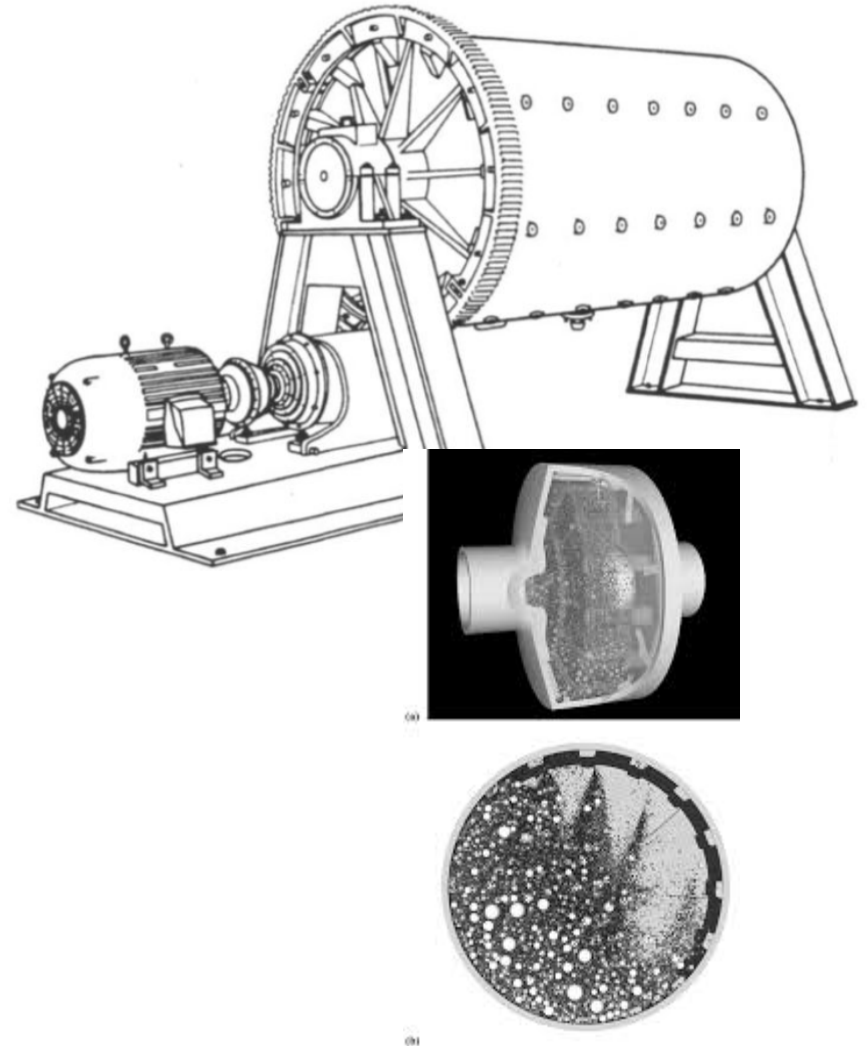
6.2 Powder Production

Pulverization

Fine powders with particles $< 10 \mu\text{m}$ can be produced

Main problem is contamination of the powder:

- metal transfer from hardened balls
- oxidation



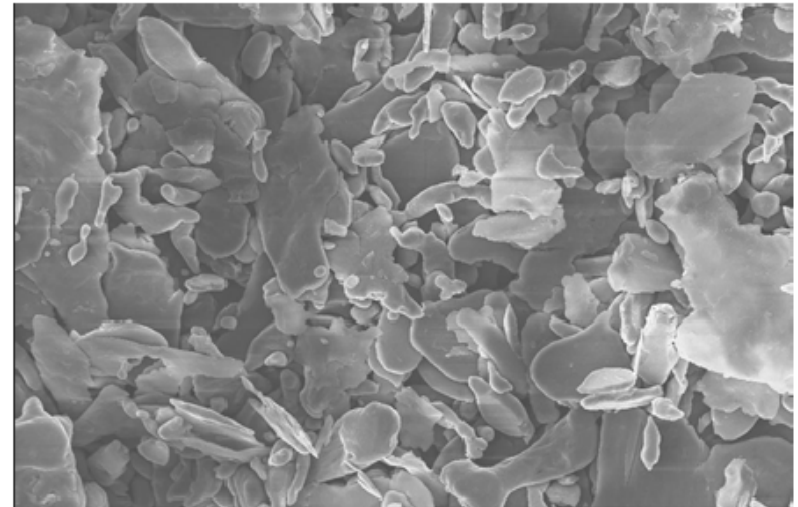
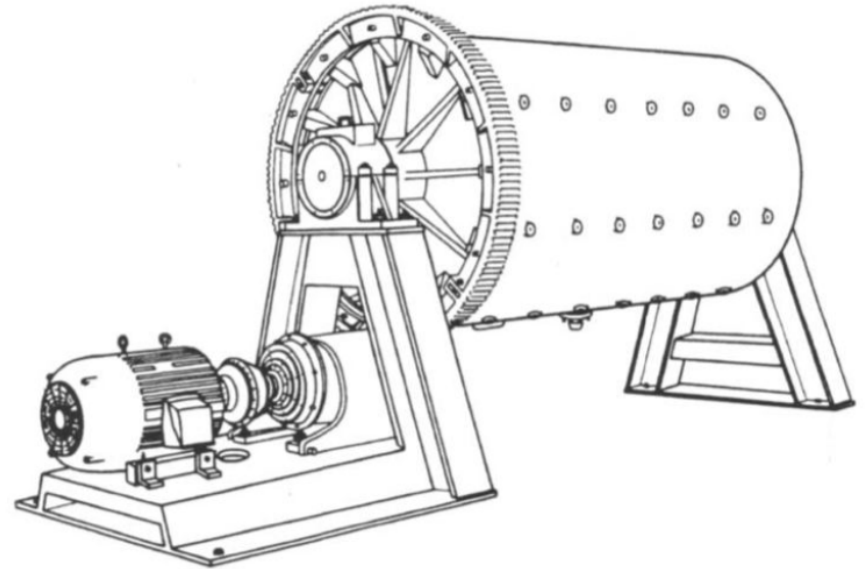
6.2 Powder Production

Pulverization

Process is suitable for *brittle* materials:

- Particles fracture producing angular shapes

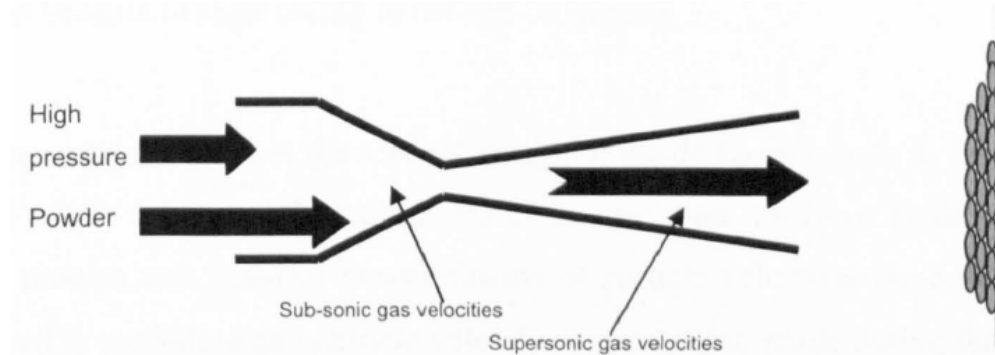
For ductile materials particles become flaky which is not particularly suitable for powder metallurgy



6.2 Powder Production

Pulverization

In the *coldstream* process granular metal is fed into supersonic stream at low temperature (similar to 'cold spray' coating technique)



Metal granules impinge on target causing fragmentation of particles

Target can be made of same metal as powder to prevent transfer contamination

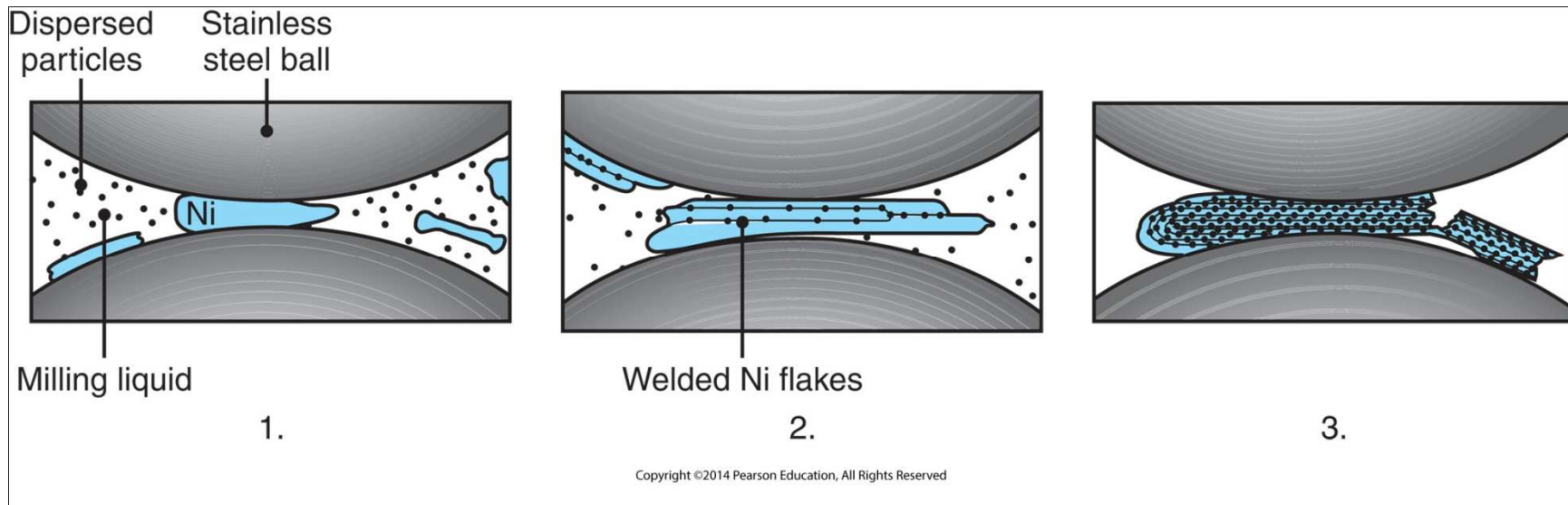
Fine powders with particles $< 10 \mu\text{m}$ can be produced

Used for tungsten, tungsten carbide, molybdenum and tool steels

6.2 Powder Production

Mechanical Alloying

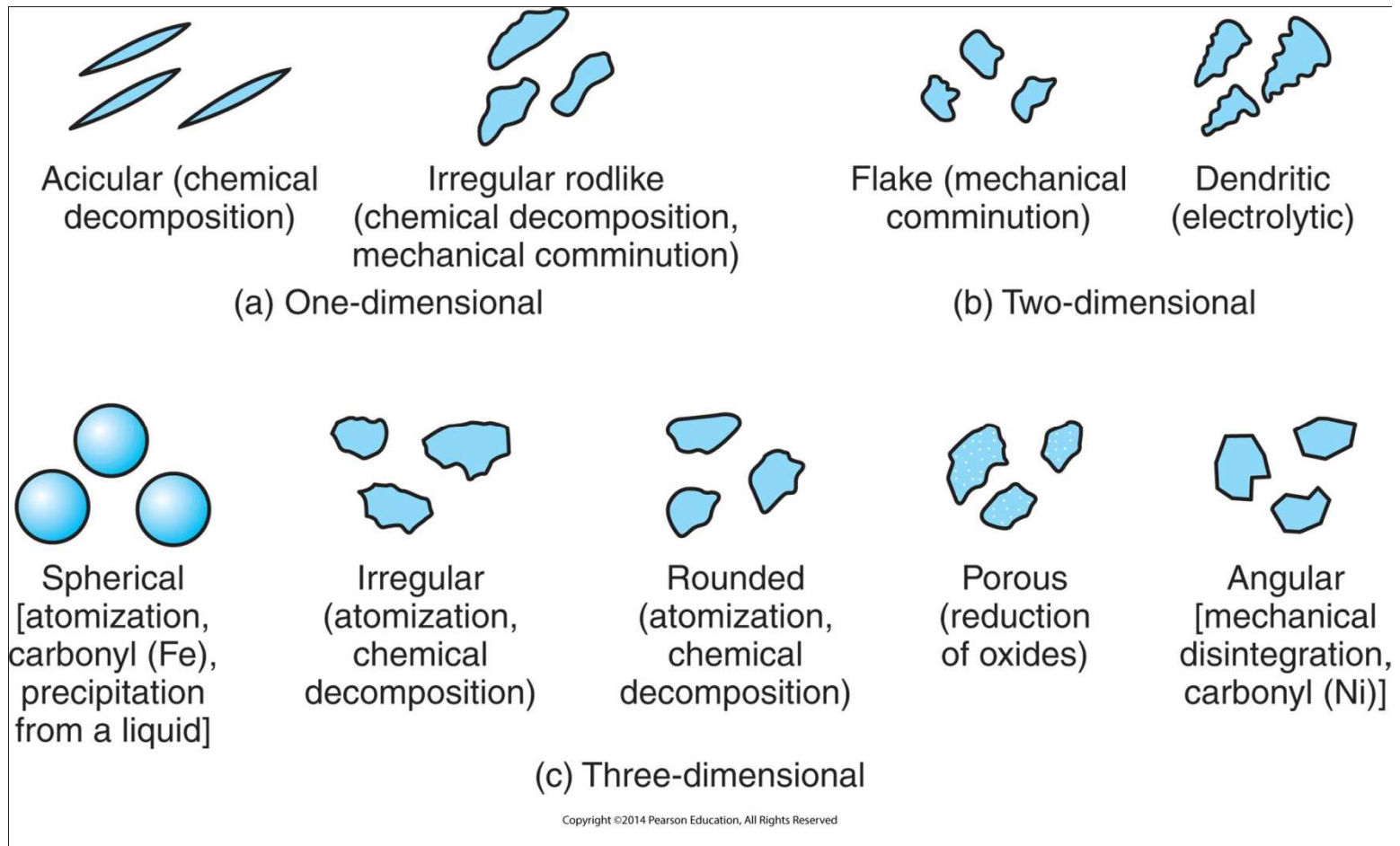
1. Powders of two or more pure metals are mixed in a ball mill
2. Under impact the powders fracture and bond together by diffusion
3. Smaller particles become dispersed phase which may increase strength or impart special electrical or magnetic properties



6.3 Powder Characteristics

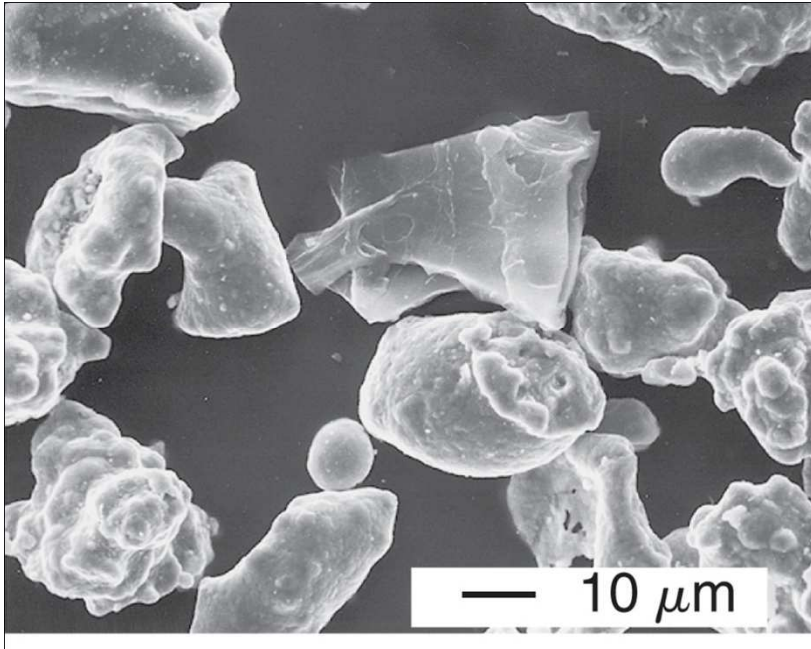
6.3 Powder Characteristics

Powders can be characterized by shape:

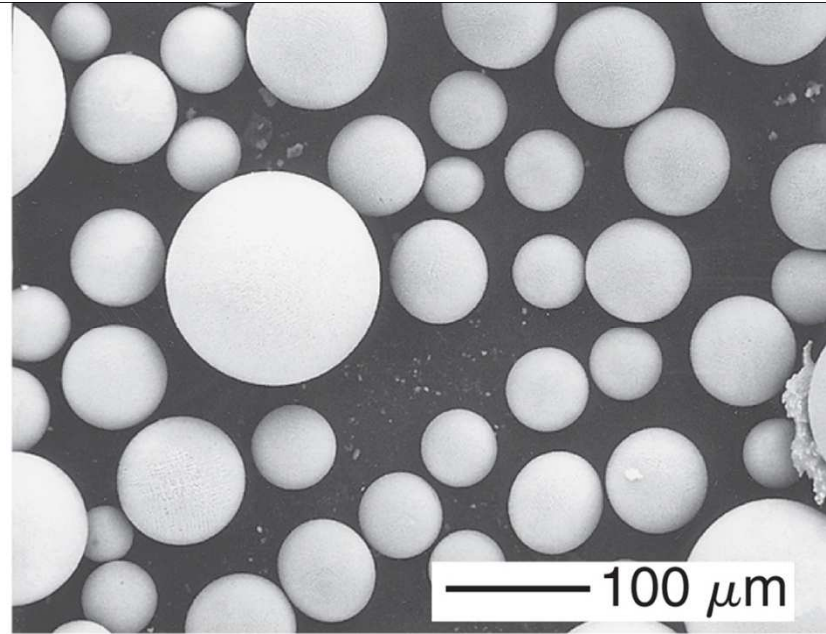


6.3 Powder Characteristics

Powders can be characterized by shape:



Fe particles made by atomization



Ni-based superalloy (Udimet 700)
particles made by spinning electrode

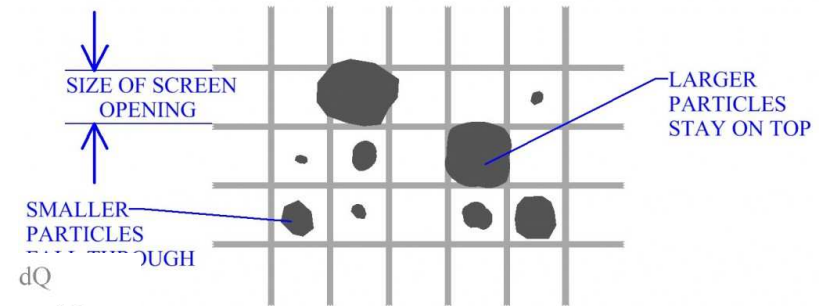
6.3 Powder Characteristics

Powders can be characterized by size:

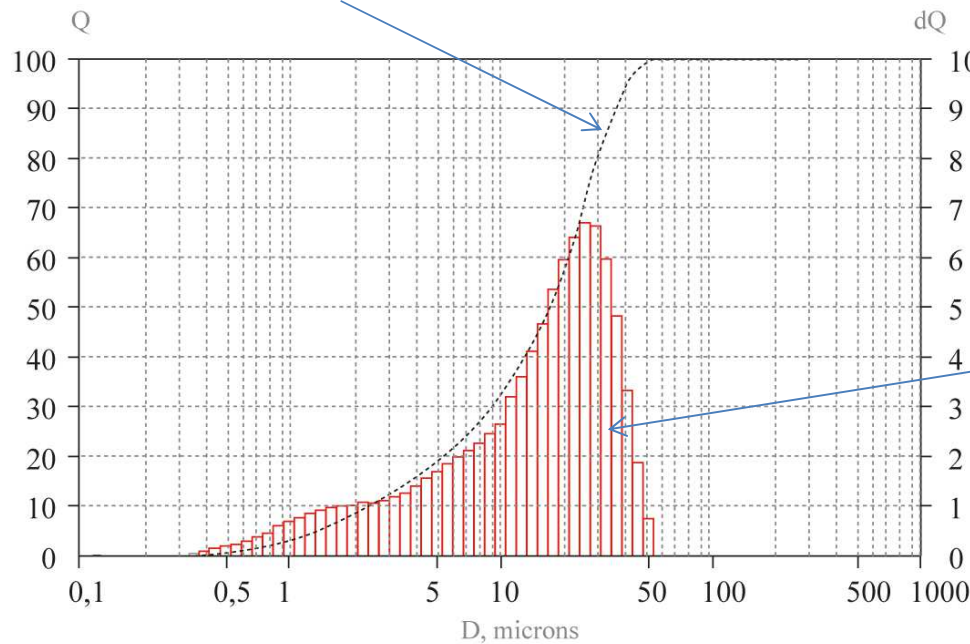
- Average size
- Maximum size
- Size distribution



MEASUREMENT OF POWDER PARTICLES



Cumulative distribution (Q: % finer than)



Histogram of particle sizes (dQ)

6.3 Powder Characteristics

Powders can be characterized by:

- Surface area to volume ratio
- Powder composition
- Contaminant particle content

6.3 Powder Characteristics

Powders can be characterized by:

- Density:
 - Apparent density (ρ_a): weight per unit volume of loose powder
 - Tap density (ρ_t): the receptacle is tapped or vibrated under specified conditions as it is filled with powder, then the density measured
 - Full (solid) density (ρ_s): density of bulk form of metal

- Degree of Densification

$$D_0 = \frac{\rho_a}{\rho_s} \cdot 100\%$$

6.3 Powder Characteristics

Density of common powders

Metal & Powder Type	ρ_a (g/cm ³)	ρ_t (g/cm ³)	ρ_s (g/cm ³)	D_o
Copper - Spherical	4.5	5.3	8.93	50 %
Copper - Irregular	2.3	3.14	8.93	26 %
Copper – Flake	0.4	0.7	8.93	4 %
Iron – Electrolytic	3.31	3.75	7.85	42 %
Iron – Atomized	2.66	3.26	7.85	33 %
Aluminum - Atomized	0.98	1.46	2.7	36 %

6.3 Powder Characteristics

Powders can be characterized by:

- Flash or explosion characteristics
 - Especially for nano-particles which are highly reactive
- Environmental and health characteristics

6.4 Powder Conditioning

6.4 Powder Conditioning

Metal powder is conditioned to improve handling characteristics, *green strength* and final part properties

- accomplished by blending powder(s)
- thorough mixing is important

Sieve separation and re-mixing: powder production batches may vary in size, shape and size distribution

- Powders mixed to obtain uniform distribution in order to improve (decrease) part-to-part variability

Blending of different metals: powders of different metals can be blended to obtain desired physical or mechanical properties of the final part; particles can also be produced of the equivalent alloy

6.4 Powder Conditioning

Lubricants: mixed with powder to improve powder flow characteristics (flow into die, flow/consolidation of particles) and improve die life

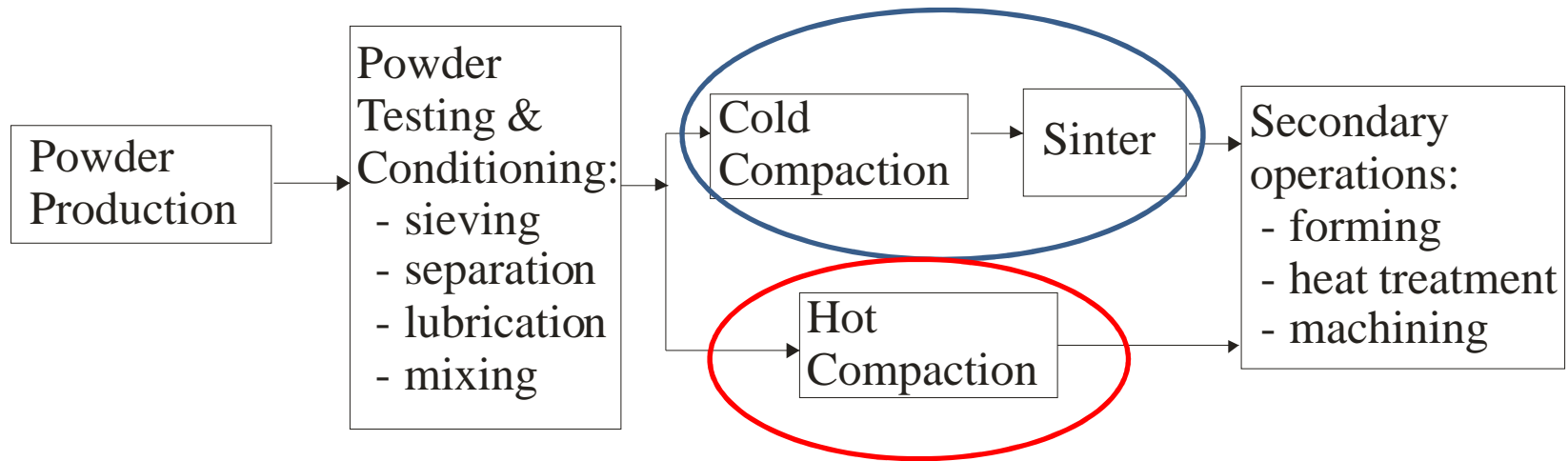
- stearic acid or zinc stearate are commonly used

Binders: similar concept to sand moulds i.e. improve *green strength* of compacted material prior to sintering

6.5 Consolidation Mechanisms

6.5 Consolidation Mechanisms

Conditioned powder can be consolidated into solid part using cold pressing with sintering or hot pressing

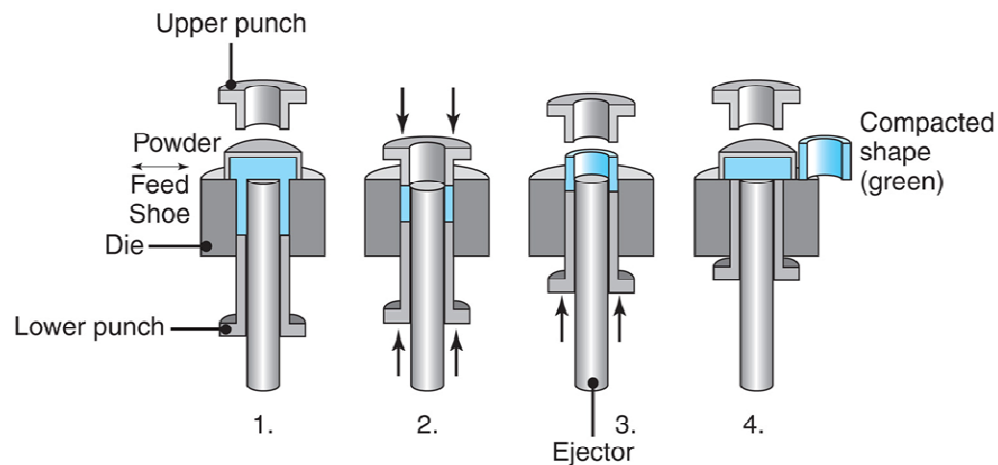


6.5 Consolidation Mechanisms

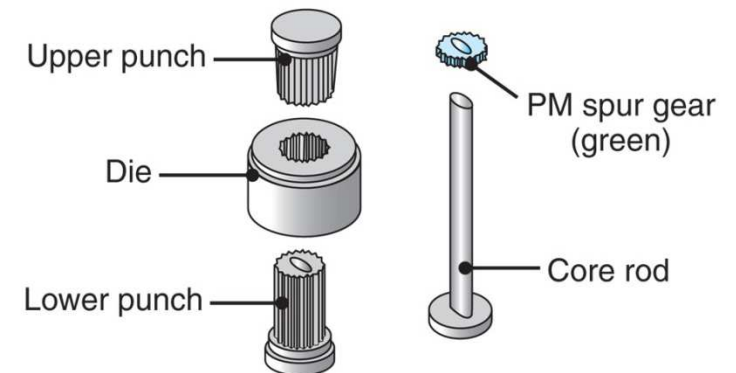
Cold Compaction

- press powder into shape close to that of final part
- impart sufficient green strength to maintain shape (under minimal forces)

Cold compaction often performed by pressing powder in rigid steel dies with pressures of 150 to 900 MPa



Bushing



Spur gear

6.5 Consolidation Mechanisms

Cold Compaction

- a pressed part is known as a *green compact* which has low strength as in green parts in slip casting of ceramics
- green parts are fragile (similar to chalk) and easily damaged
- green strength improved by proper powder feeding, compaction pressure and particle size distribution; binders can also be added in conditioning step

6.5 Consolidation Mechanisms

Cold Compaction

TABLE 17.1

Compacting Pressures for Various Powders

Material	Pressure (MPa)
Metals	
Aluminum	70–275
Brass	400–700
Bronze	200–275
Iron	350–800
Tantalum	70–140
Tungsten	70–140
Other materials	
Aluminum oxide	110–140
Carbon	140–165
Cemented carbides	140–400
Ferrites	110–165

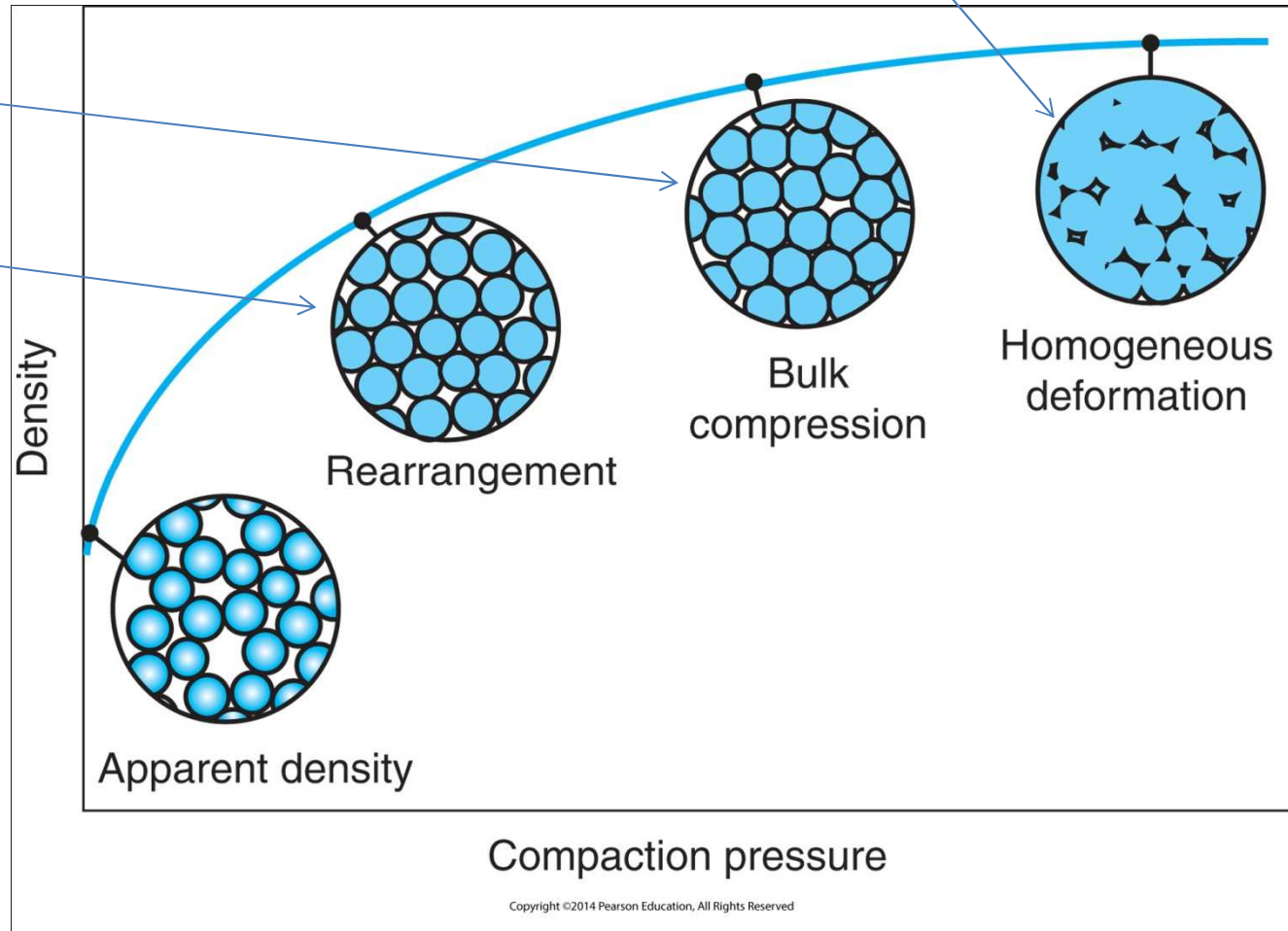
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6.5 Consolidation Mechanisms

Cold Compaction

Deformation at interfaces

No deformation of particles



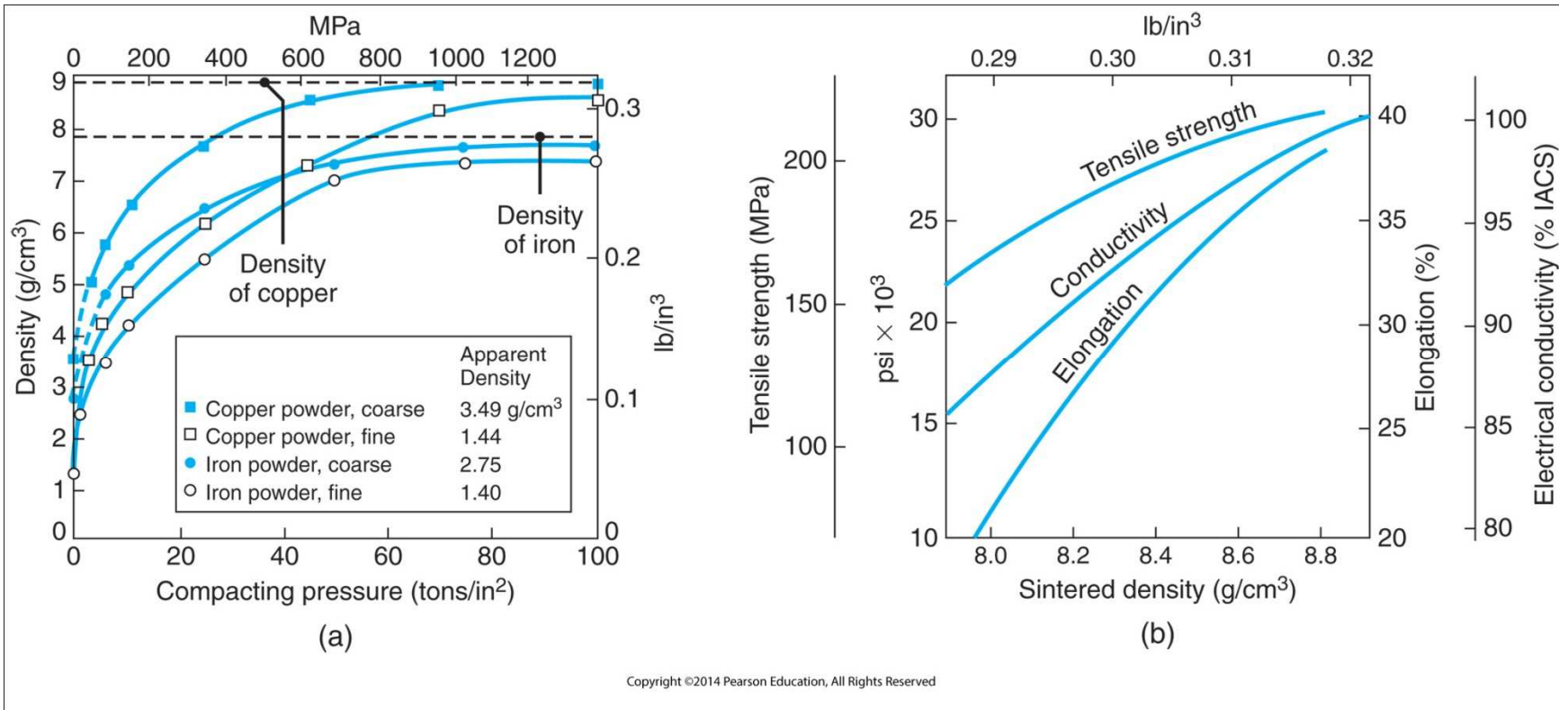
6.5 Consolidation Mechanisms

Cold Compaction

- rearrangement causes breaking of cold welds between particles and formation of new cold welds between new neighbours
- plastic deformation causes mechanical locking between particles and strain hardening
 - these limit the green density that can be obtained
- it is easier to compact irregularly-shaped particles

6.5 Consolidation Mechanisms

Cold Compaction



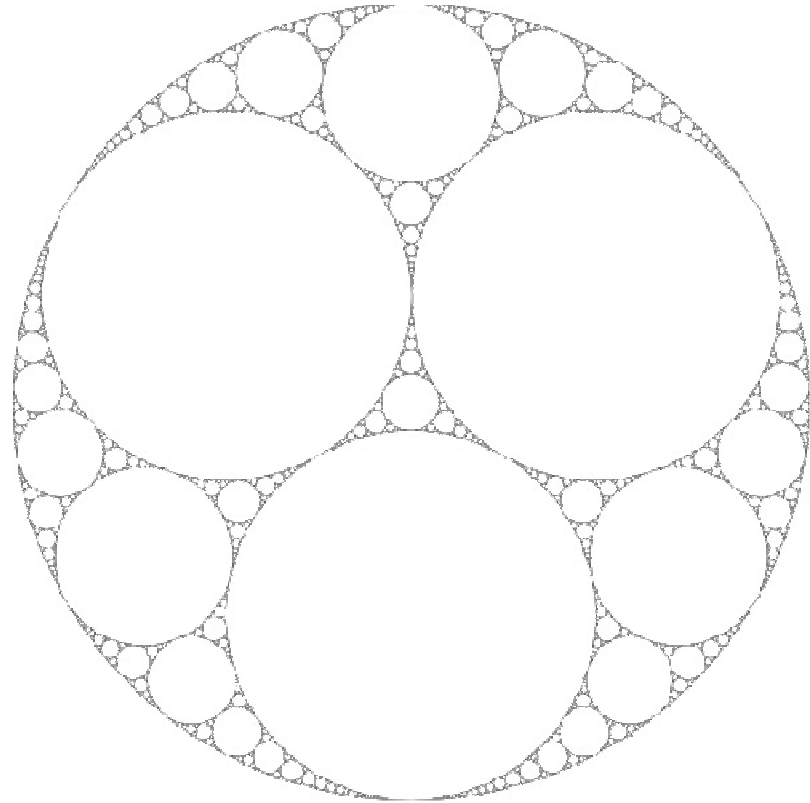
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6.5 Consolidation Mechanisms

Cold Compaction

- wide range of particle sizes increases density that can be achieved
- thus increases strength

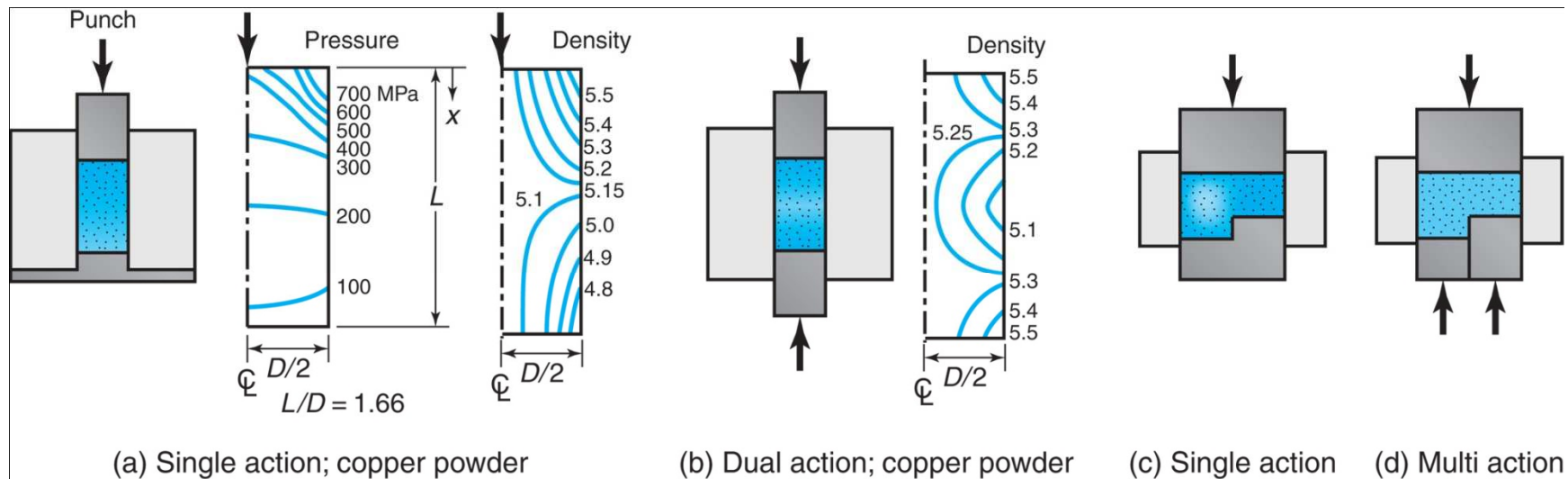
- coarse particles increase green strength due to interlocking of particles; however powder flow more difficult



6.5 Consolidation Mechanisms

Cold Compaction

- pressure, and thus density, distribution not even through compact; both are lowest at middle
- pressure and density more uniform if pressure applied by upper and lower punches



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6.5 Consolidation Mechanisms

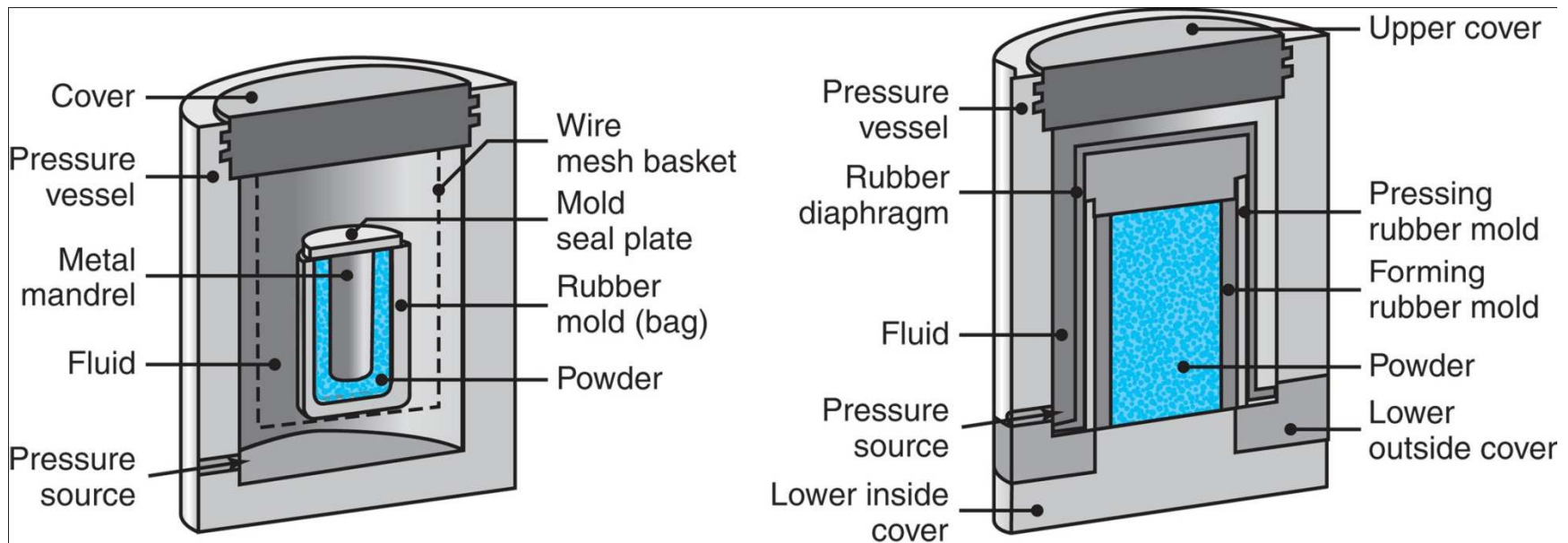
Cold Compaction

- Non-uniform density limits length:diameter ratio to about 3
- More uniform cold compacts can be produced by hydrostatic pressure (cold isostatic pressing)

6.5 Consolidation Mechanisms

Cold Compaction

- cold isostatic pressing involves application of pressure isostatically inside a high pressure chamber
- can use *wet bag* (left) or *dry bag* (right) process



6.5 Consolidation Mechanisms

Sintering

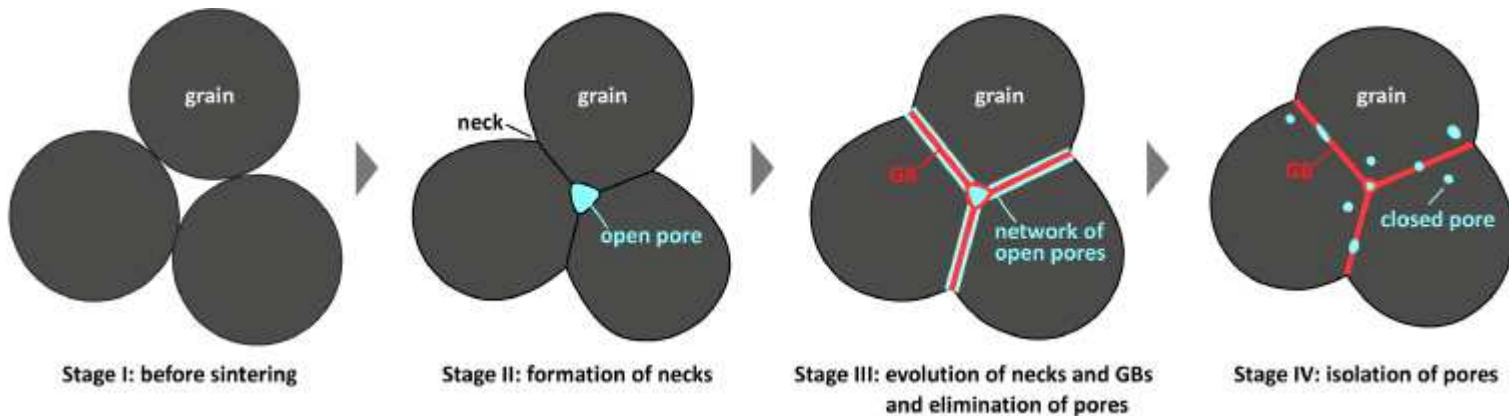
- after cold compaction sintering is required to fuse the particles together
- compact is heated to below the melting temperature
- bonding of particles occurs

6.5 Consolidation Mechanisms

Sintering

Bonding between particles depends on:

- diffusion
- plastic flow
- evaporation of volatile materials (e.g. binders)
- recrystallization
- grain growth
- pore shrinkage



6.5 Consolidation Mechanisms

Sintering

- main variables are temperature, time and furnace atmosphere
- temperature typically 70 to 90% of melting temperature

TABLE 17.2

Sintering Temperature and Time for Various Metals

Material	Temperature (°C)	Time (min)
Copper, brass, and bronze	760–900	10–45
Iron and iron graphite	1000–1150	8–45
Nickel	1000–1150	30–45
Stainless steels	1100–1290	30–60
Alnico alloys (for permanent magnets)	1200–1300	120–150
Ferrites	1200–1500	10–600
Tungsten carbide	1430–1500	20–30
Molybdenum	2050	120
Tungsten	2350	480
Tantalum	2400	480

6.5 Consolidation Mechanisms

Sintering

Sintering must be performed in a protective atmosphere

- Prevent oxide formation which impedes diffusion bonding of particles
- Vacuum; hydrogen with CO and CO₂; nitrogen

Types of sintering:

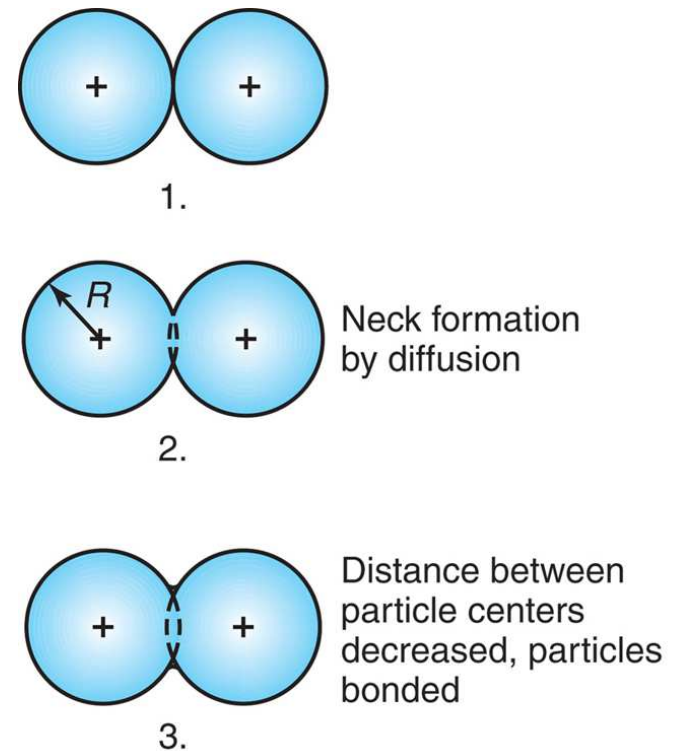
- Solid phase sintering
- Vapour-phase transport
- Liquid phase sintering

6.5 Consolidation Mechanisms

Sintering – Solid phase

All powder remains in the solid state

- Two adjacent particles form a bond through diffusion of atoms
- Strength, density, ductility, thermal and electrical conductivity increase
- Individual particles move closer together to supply material in bond, thus the compact shrinks and shrinkage allowances are required

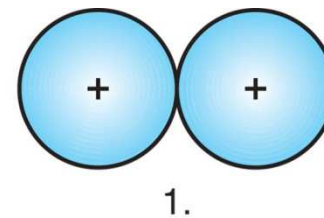


6.5 Consolidation Mechanisms

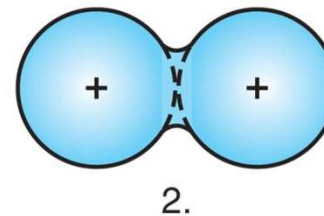
Sintering – Vapour phase transport

Almost all powder remains in the solid state

- Temperature is near T_{melt} a small number of atoms released from surface directly to vapour phase
- Atoms transported to convergent region i.e. between particles (due to lower vapour pressure) and precipitate forming neck
- Particles shrink but bulk volume unchanged

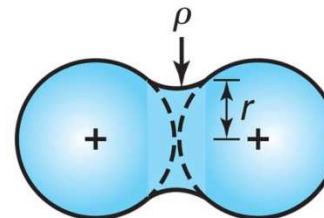


1.



2.

Neck formation
by vapor-phase
material transport



3.

Particles bonded,
no shrinkage (center
distances constant)

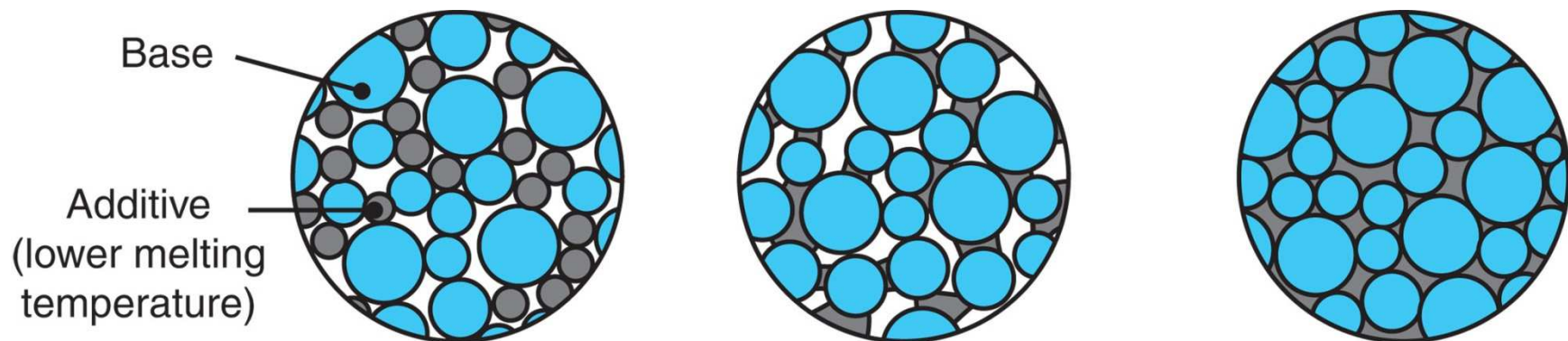
6.5 Consolidation Mechanisms

Sintering – Liquid phase

Most common when two or more powders mixed and one has a lower T_{melt}

As one powder melts it surrounds the solid particles due to surface tension

Example: Co in tungsten-carbide used for cutting tools



6.5 Consolidation Mechanisms

Sintering results in

- Formation of strong bonds between grains
- Reduction in size and number of voids; increased density
 - May result in part shrinkage
- Grain growth

6.5 Consolidation Mechanisms

Driving force for processes in sintering is provided by surface energy

- Free surfaces of particles
- Grain boundaries

Rate of these processes is governed by diffusion rate and the diffusion constant:

$$D = D_0 \exp\left(\frac{-Q}{RT}\right)$$

D_0 is the frequency factor (material property)

Q is the activation energy (material property)

T is the absolute temperature

R is the universal gas constant

6.5 Consolidation Mechanisms

$$D = D_0 \exp\left(\frac{-Q}{RT}\right)$$

Rate of diffusion (*flux*) is linearly related to the diffusion (rate) constant

Thus diffusion process in sintering will provide faster rate of densification at higher temperature

But, higher temperature also increases rate of grain growth

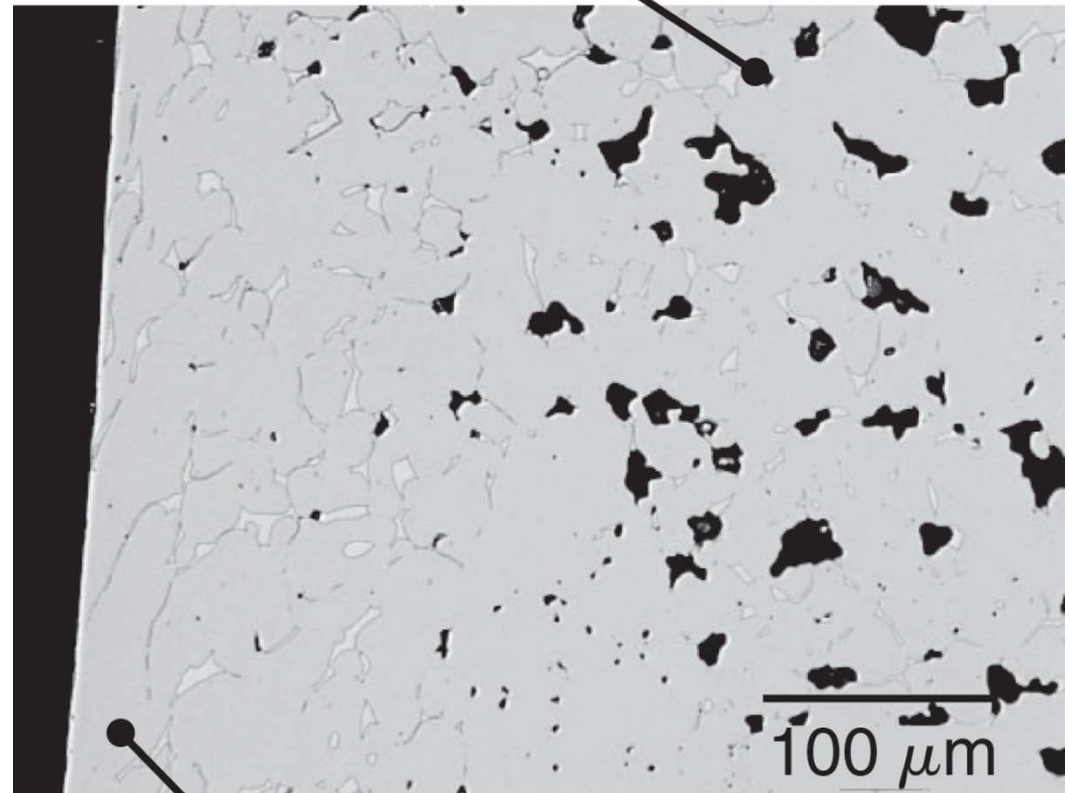
Final grain size can be modified by thermo-mechanical treatment

6.5 Consolidation Mechanisms

Roll densification after sintering decreases porosity near surface

- Better resistance to contact stresses

Subsurface porosity



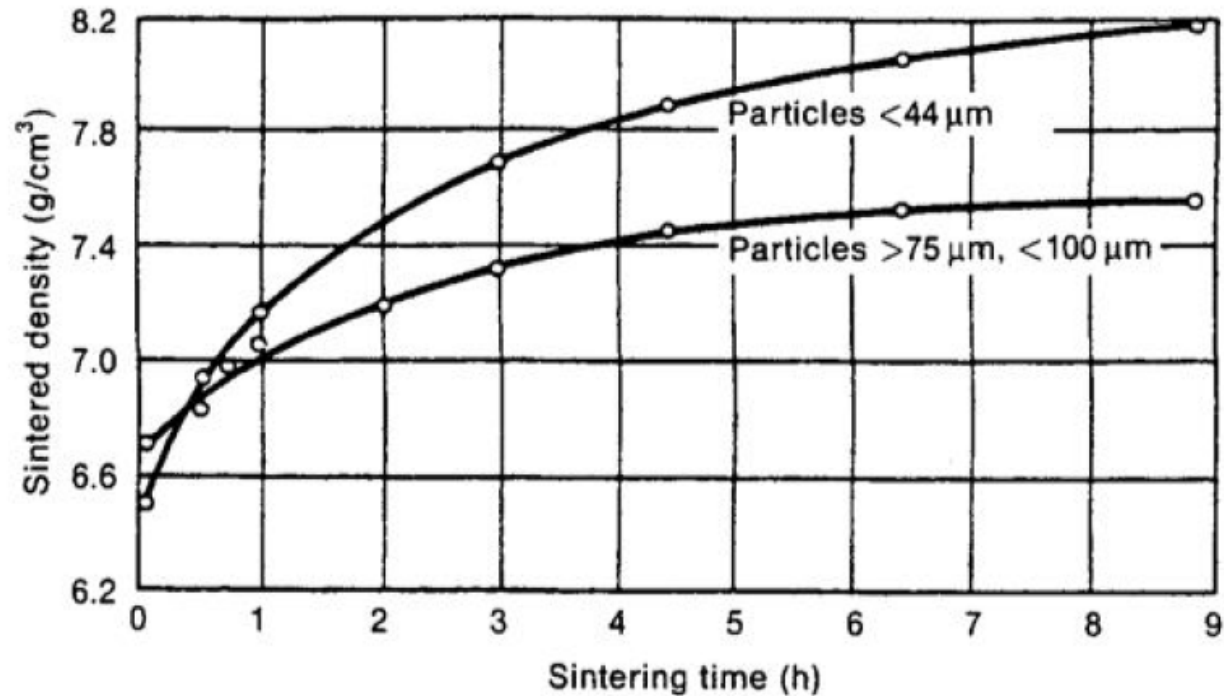
Densified surface

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6.5 Consolidation Mechanisms

Smaller powder size increases rate of densification and allows for higher final density

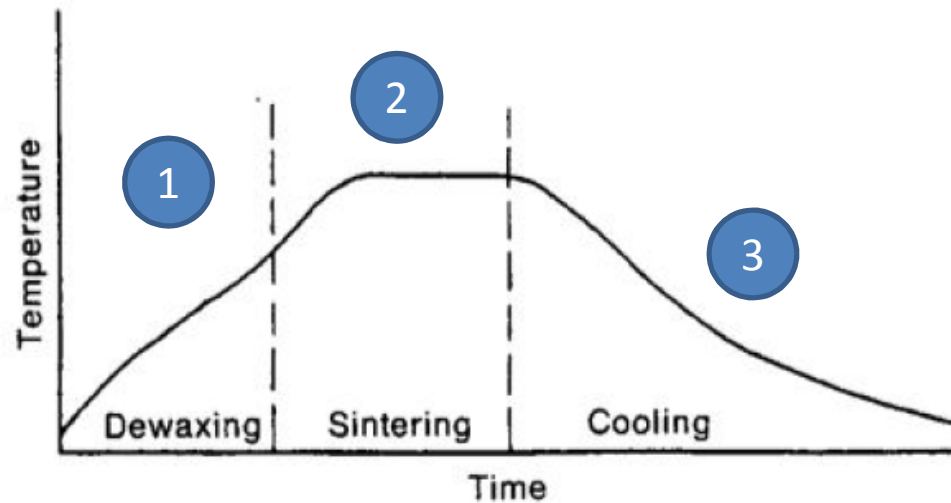
- Increased surface area thus surface energy



Sintering of copper with varying initial particle size; $\rho_s = 8.9 \text{ g/cm}^3$

6.5 Consolidation Mechanisms

Heating cycle in sintering has three stages



1. Initial heating phase; temperature rise slow enough:
 - to ensure all volatile compounds are driven off (binders, lubricants)
 - to allow heat transfer through part to obtain uniform sintering
2. Sintering time and temperature set to obtain desired strength, density (usually maximize)
3. Cooling slow to avoid thermal distortion; must be done in furnace atmosphere (protective) to avoid oxidation

6.5 Consolidation Mechanisms

Hot compaction

Powder may be compacted when hot

Softening of metal at elevated temperatures allows greater density of green compact to be achieved

Subsequent sintering may not be necessary

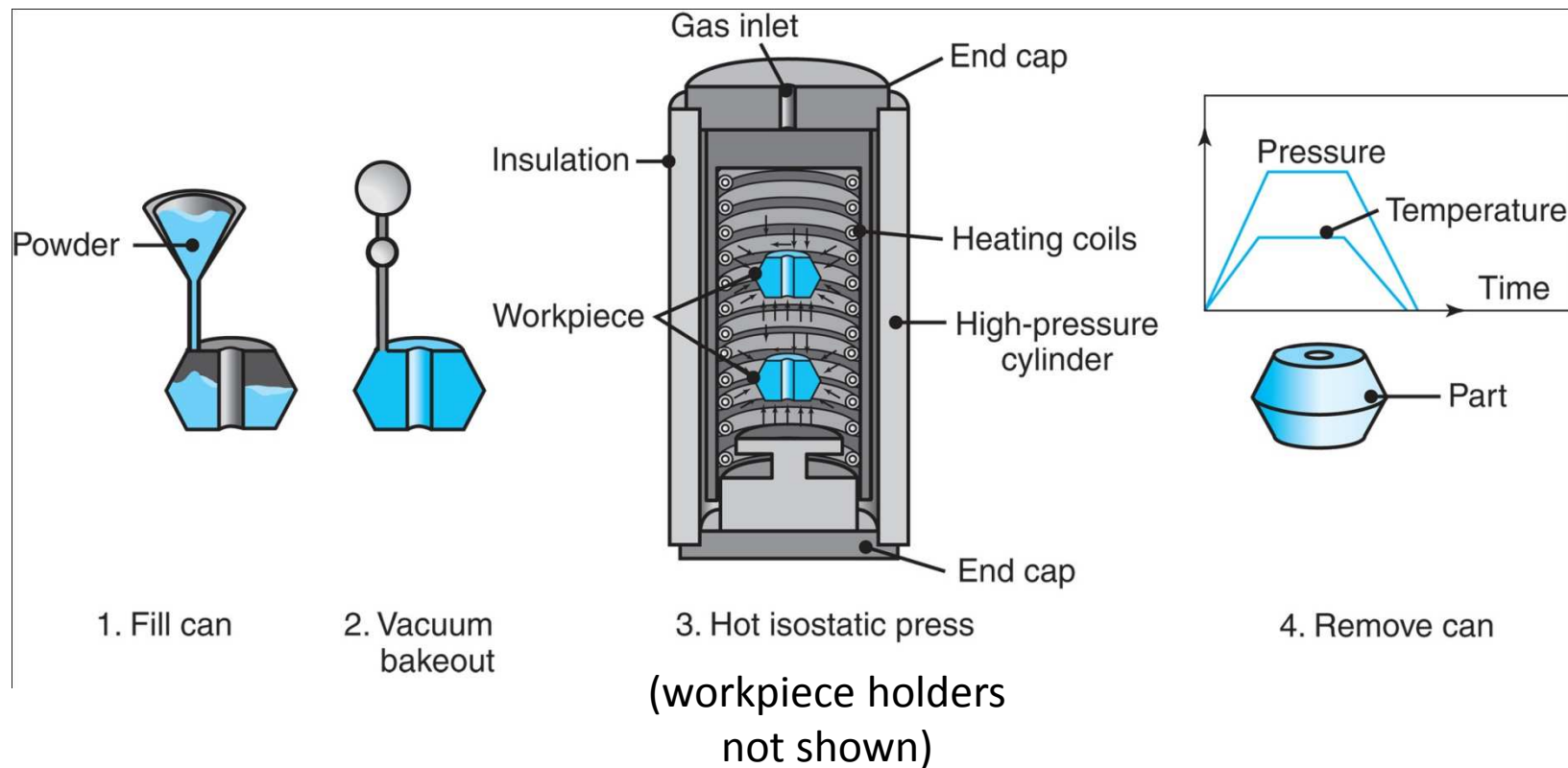
However hot compaction in powder metallurgy is usually achieved using *hot isostatic pressing (HIP)*

6.5 Consolidation Mechanisms

Hot isostatic pressing

Similar to cold isostatic pressing

- simultaneous application of heat eliminates separate sintering step



6.5 Consolidation Mechanisms

Hot isostatic pressing

Powder is placed in flexible container which is vacuumized

Powder/container is placed in HIP press which is then pressurized with inert gas (argon)

High pressure, typically 100 MPa, is transferred to powder through flexible container

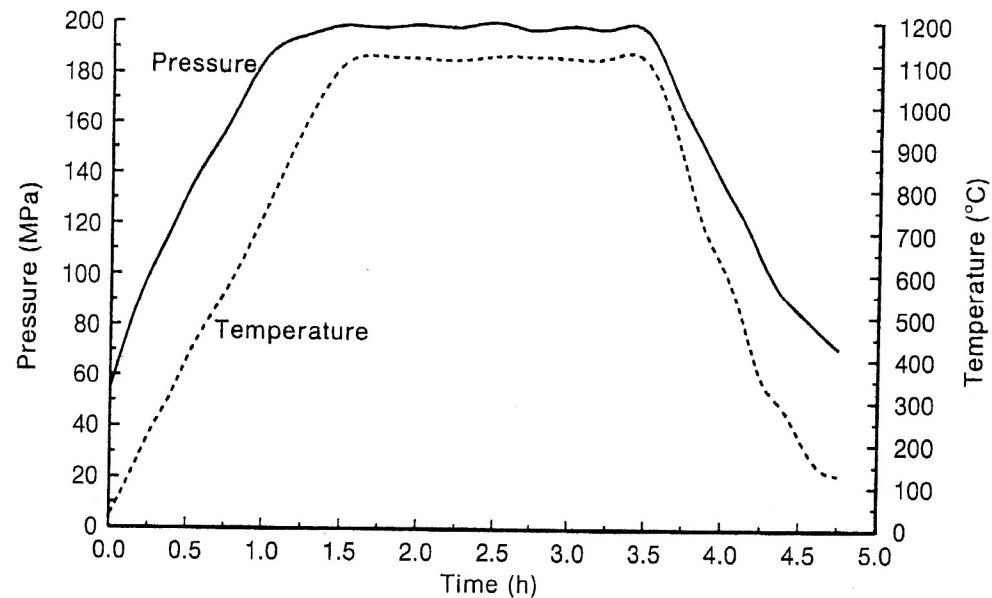
Commercial systems capable of 280 MPa and 1200°C available

6.5 Consolidation Mechanisms

Hot isostatic pressing

Typical pressure, temperature cycle

Note application of pressure at room temperature to obtain compaction before diffusion processes start



6.5 Consolidation Mechanisms

Hot isostatic pressing

Densification during HIP is obtained by plastic deformation and diffusion

HIP can also be performed on solidified castings to reduce porosity

Produces high quality parts of almost 100% density, good particle bonding and mechanical properties

Used mainly for production of superalloy components in aircraft and aerospace industries; also for tungsten carbide cutting tools

6.5 Consolidation Mechanisms

Hot isostatic pressing: Limitations

- Wider dimensional tolerances due to use of flexible containers
- High equipment costs and longer production time
- Lower productivity, typically limited to < 10,000 parts/year

Typical parts made by powder metallurgy

Application	Metals	Products
Automotive	Cu, Fe, W	Gears, bushings, valves, connecting rods, crankshaft bearing caps, belt pulleys
Gas Turbines	Ni base superalloys	Turbine discs
Heat treating	Mo, W	Furnace elements
Lubrication	Cu, Fe, Zn	Abradable seals, self-lubricating bearings
Tooling	W, Mo, Mn	Dies, punches, machining tools
Dental	Ag, Au	Amalgams