POWDER METALLURGY- UNIT 3

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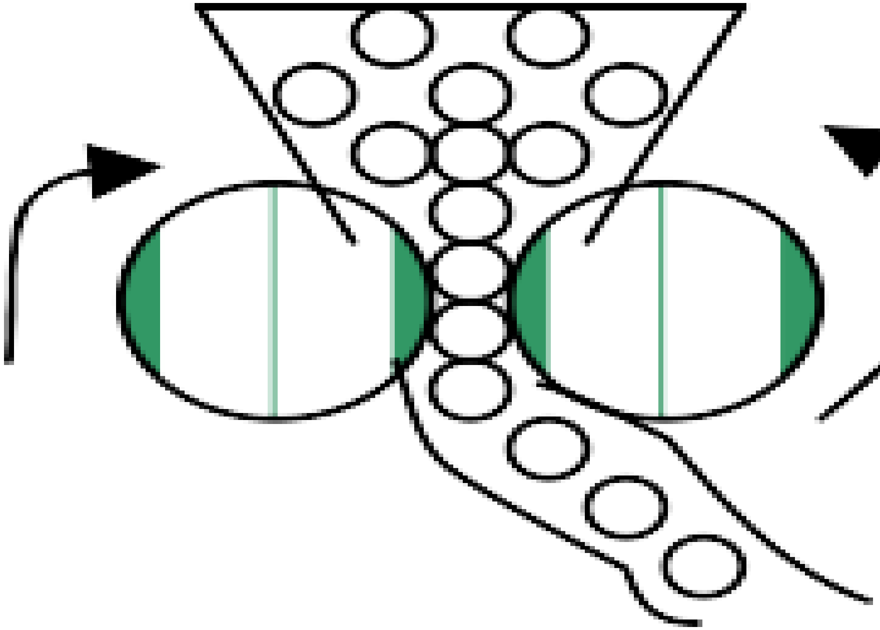
SYLLABUS

- Powder rolling, powder forging, powder extrusion and explosive forming technique
- Definition of sintering, stages of sintering, effect of variables on sintering, sintering atmospheres and sintering furnaces.

POWDER ROLLING

- This process involves feeding of powders between rolls to produce a coherent and brittle green strip. This green strip is then sintered & re-rolled to obtain a dense, finished product.



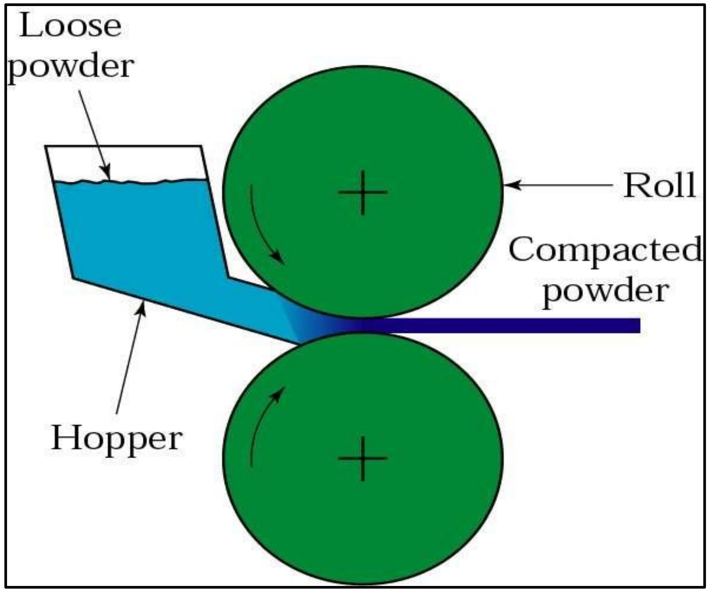
STEPS

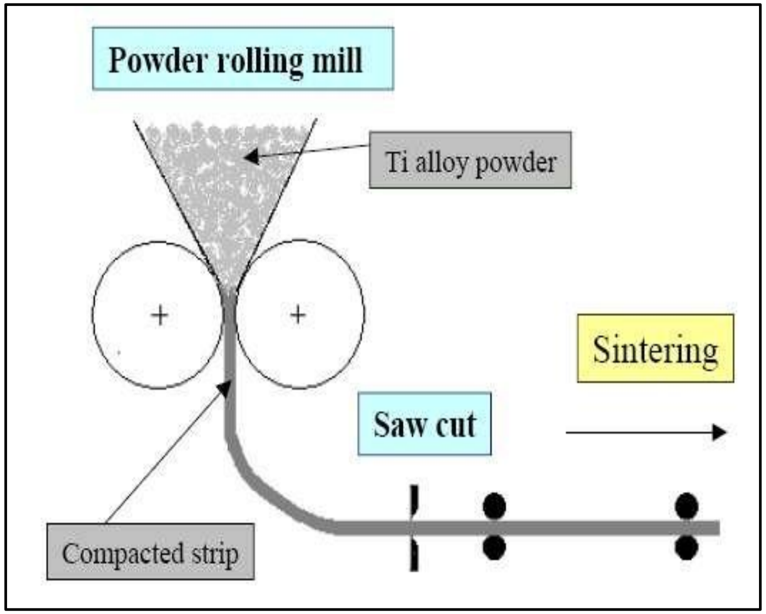
1) preparation of green strip,
2) sintering,
3) densification of sintered strip,
4) final cold rolling and annealing

PARAMETERS AFFECTING:

- roll gap,
- roll diameter,
- roll speed,
- powder characteristics;
  - Roll gap => large roll gap leads to decrease in green density; very small roll gap leads to edge cracking;
  - Roll diameter => increase in density and strength with increase in roll dia. for a given strip thickness;
  - Roll speed => Kept low, 0.3-0.5 m/s;
  - Powder => irregular powder with rough surfaces provide better strip density

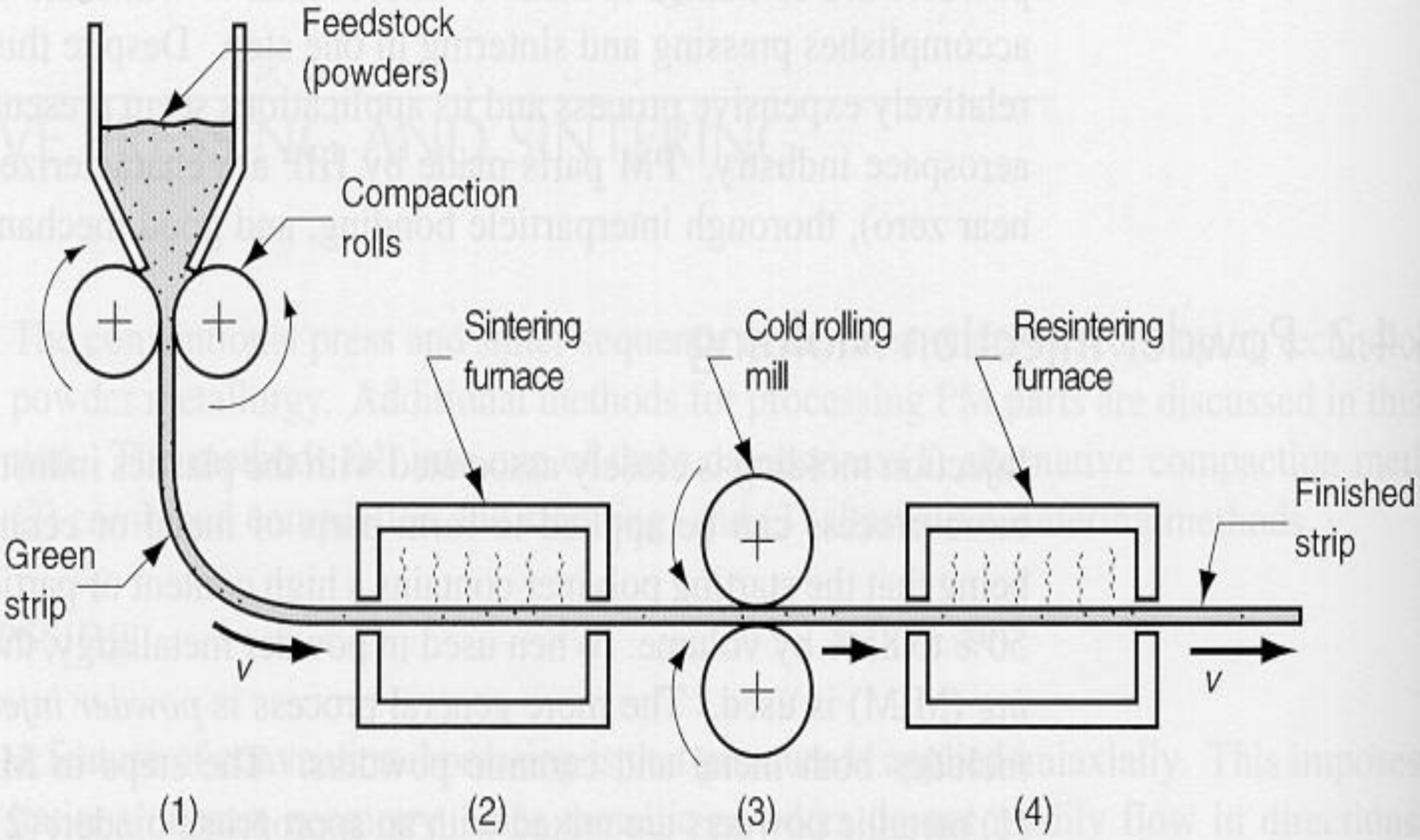
- In densification stage, either repeated cold rolling followed by annealing or hot rolling of strip can be followed
- Applications:
  - nickel strips for coinage,
  - nickel-iron strips for controlled expansion properties,
  - Cu-Ni-Sn alloys for electronic applications,
  - porous nickel strip for alkaline batteries and fuel cell applications





12” wide Ti-6Al-4V strip

FIGURE 18.15 Powder rolling: (1) powders are fed through compaction rolls to form a green strip; (2) sintering; (3) cold rolling; and (4) resintering.

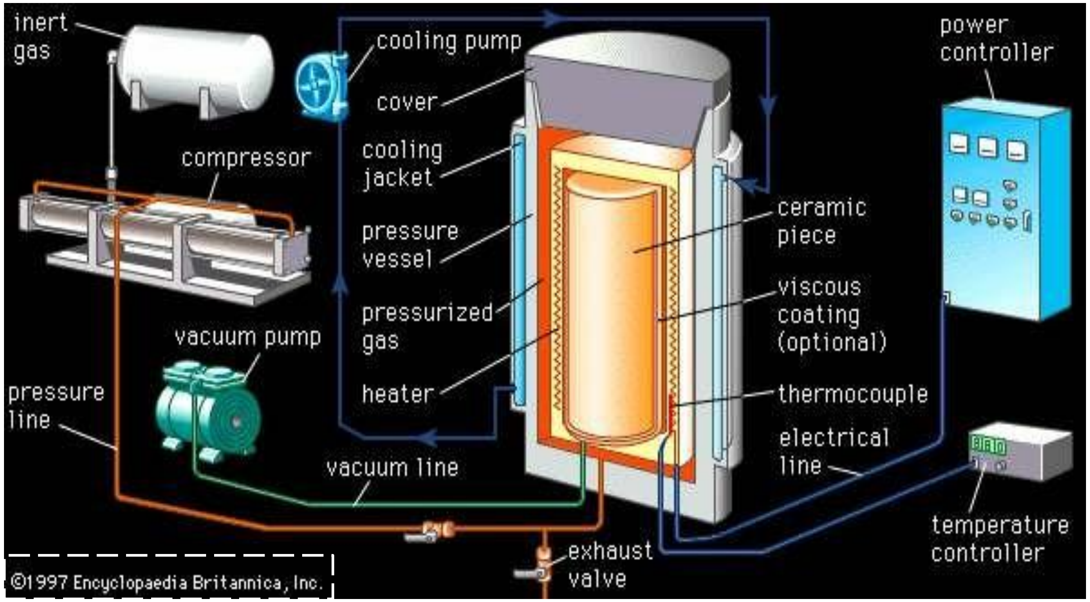


HOT ISOSTATIC PRESSING

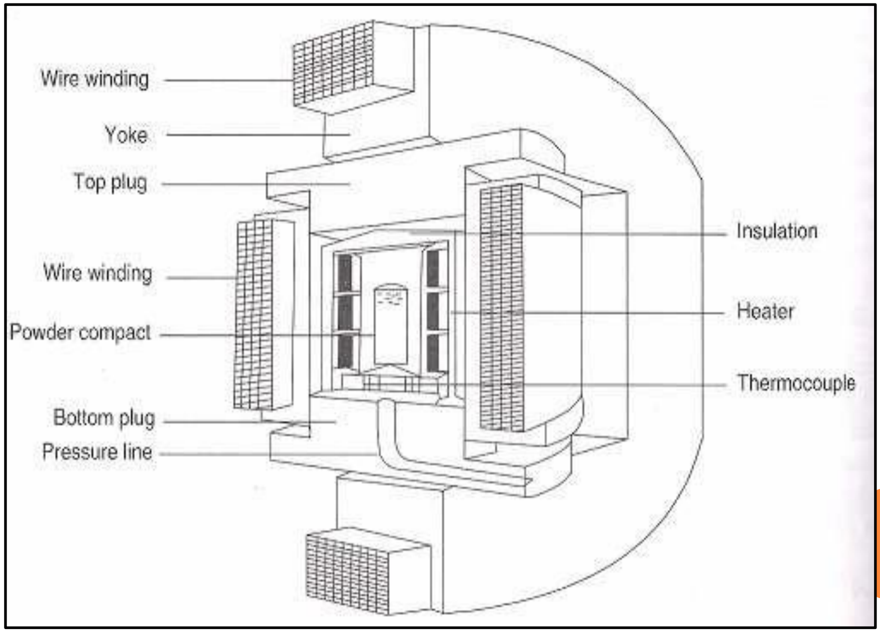
- Ideal method for consolidation of powders of nickel and cobalt base super alloys, tool steels, maraging steels, titanium alloys, refractory metal powders, cermets.
- It has got variety of applications including bonding of dissimilar materials, consolidation of plasma coatings, processing hard and soft magnetic materials etc.
- HIP is the application of pressure at elevated temperatures to obtain net or near net shape parts from metal, ceramic, cermet powders.

- HIP unit consists of a pressure vessel, high temperature furnace, pressurizing system, controls and auxiliary systems (material handling, vacuum pumps, metering pumps).
- The pressure vessel is made of low alloy steel. Its function is to heat the powders while applying uniform gas pressure on all the sides. Furnaces are of radiation or convection type heating furnaces with graphite or molybdenum heating elements.

- Nichrome is also used. The furnace heats the powder part, while pressurizing medium (a gas) is used to apply a high pressure during the process. Generally, argon, nitrogen, helium or even air is used as pressurizing medium.
- The pressurizing gas, usually argon, is let into the vessel and then a compressor is used to increase the pressure to the desired level. The furnace is then started and both temperature and pressure are increased to a required value.

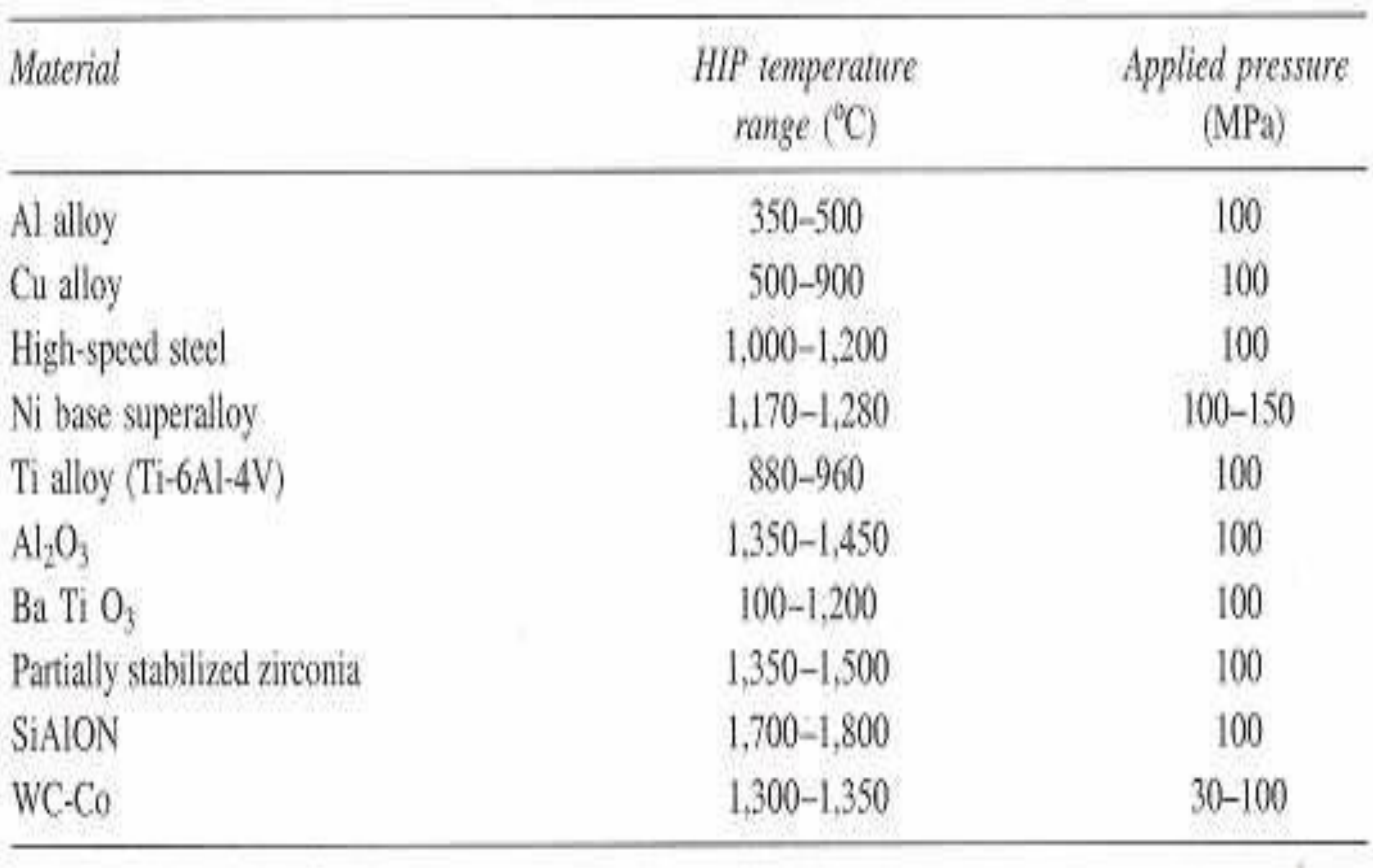


HIP UNIT– used for ceramic material (photo from public domain)



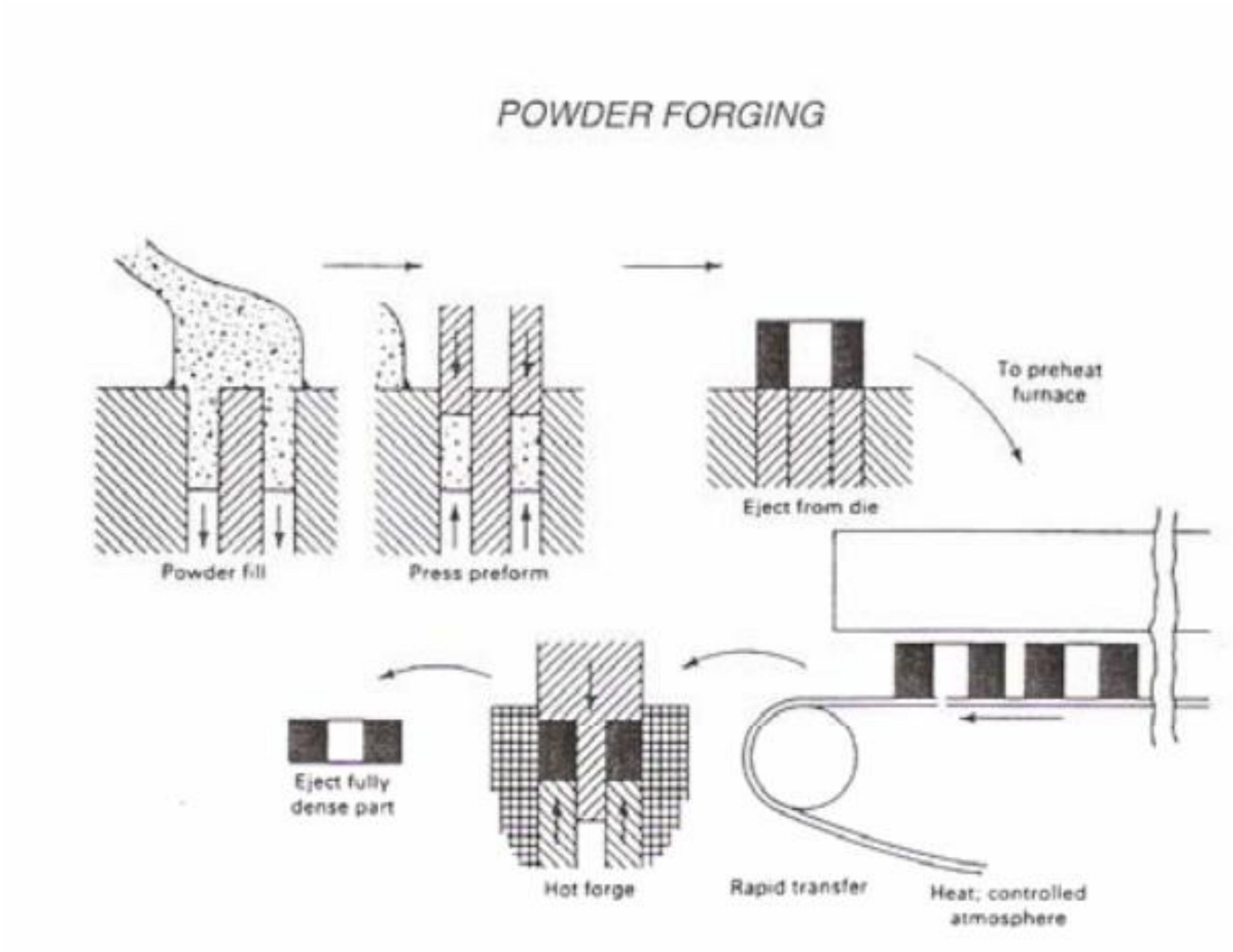
Schematic of HIP UNIT (cross-section)

- HIP presses are available in diameters up to 2m with pressures ranges from 40 to 300 MPa with temperature range from 500 to 2200 C. The processing time can last up to 4 hours depending on the material and size of the part.
- during HIP, the pores are closed by flow of matter by diffusion and creep, but also bonded across the interface to form a continuous material.
- Commonly used heating elements: Kanthal heating element – up to 1200 C; Molybdenum heating element – 1200 to 1700 C; Graphite heating element – 2000 to 2600 C
- Typical range of temperature & pressures used in HIP is given in table



Material	*HIP temperature range* (°C)	*Applied pressure* (MPa)
Al alloy	350–500	100
Cu alloy	500–900	100
High-speed steel	1,000–1,200	100
Ni base superalloy	1,170–1,280	100–150
Ti alloy (Ti-6Al-4V)	880–960	100
Al_2O_3	1,350–1,450	100
Ba Ti O_3	100–1,200	100
Partially stabilized zirconia	1,350–1,500	100
SiAlON	1,700–1,800	100
WC-Co	1,300–1,350	30–100

POWDER FORGING



- This is a rapidly developing technology in which pressed powder preform, usually produced by conventional die compaction, isostatic compaction or slip casting, is heated to hot working temperature in a protective atmosphere and immediately transferred to closed die and forging operation is carried out.
- After forging, the component is withdrawn from the die and its either quenched or cooled in protective atmosphere to minimize oxidation.

- This produces densification upto 100% and closed dimensional tolerances and involves lower sintering temperature to obtain metallurgical sound structure.
- The green compacts (referred as preforms) are given the combination of two thermal treatments i.e. sintering and forging pre-heating instead of normal sintering.
- The main problem involved in this process is die life particularly in complicated parts. Originally this technique has been developed for production of fuel elements used in nuclear reactors.

POWDER EXTRUSION

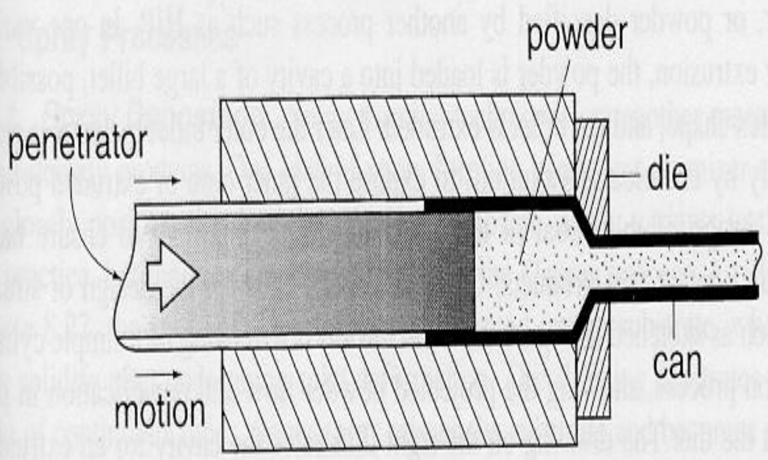


Figure 8.25. Hot powder extrusion involves forcing a can filled with powder though a die at the end of the extrusion barrel. Pressure is applied by a plunger or penetrator. With a large reduction in cross-sectional area, the powder is fully densified.

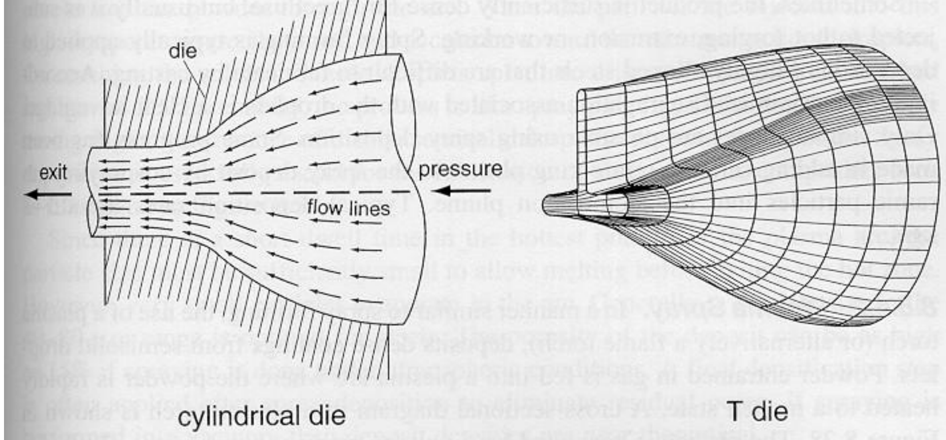


Figure 8.26. Streamlined die designs are used for smooth powder flow and densification. The contour changes in the die are calculated to ensure that the steel powder densifies into the final object shape without cracking. The left sketch shows an internal slice of a simple flow analysis, and the right sketch is an example die cavity for forming an extruded "T" section. (Based on work of H. Gegel, S. M. Malas, and S. M. Doraivelu.)

- Binding materials used has two fold function:
    1. Binds the powder particles
    2. Minimises the friction against the die wall.
- Paraffin wax is generally used (6-12 wt %).
- It has been recommended to use preforms rather than the mixture to get the desired density.
- The extruded components then goes for sintering.
- The mixture of powder and binding material is kept in the container and the plunger is pushed towards the die by a hydraulic press.

- Extrusion of canned powders may also be used for obtaining extremely high densities.
- This process is employed for consolidation of carbide, dispersion strengthened materials etc. in form of rods, bars etc.

HERF: EXPLOSIVE FORMING

- In these forming processes large amount of energy is applied for a very short interval of time.
- Many metals tend to deform more readily under extra – fast application of load which make these processes useful to form large size parts out of most metals including those which are otherwise difficult – to – form.
- The parts are formed at a rapid rate, and thus these processes are also called high – velocity forming processes.

- There are several advantages of using these forming processes, like die costs are low, easy maintenance of tolerances, possibility of forming most metals, and material does not show spring-back effect.
- The production cost of components by such processes is low.
- The limitation of these processes is the need for skilled personnel.

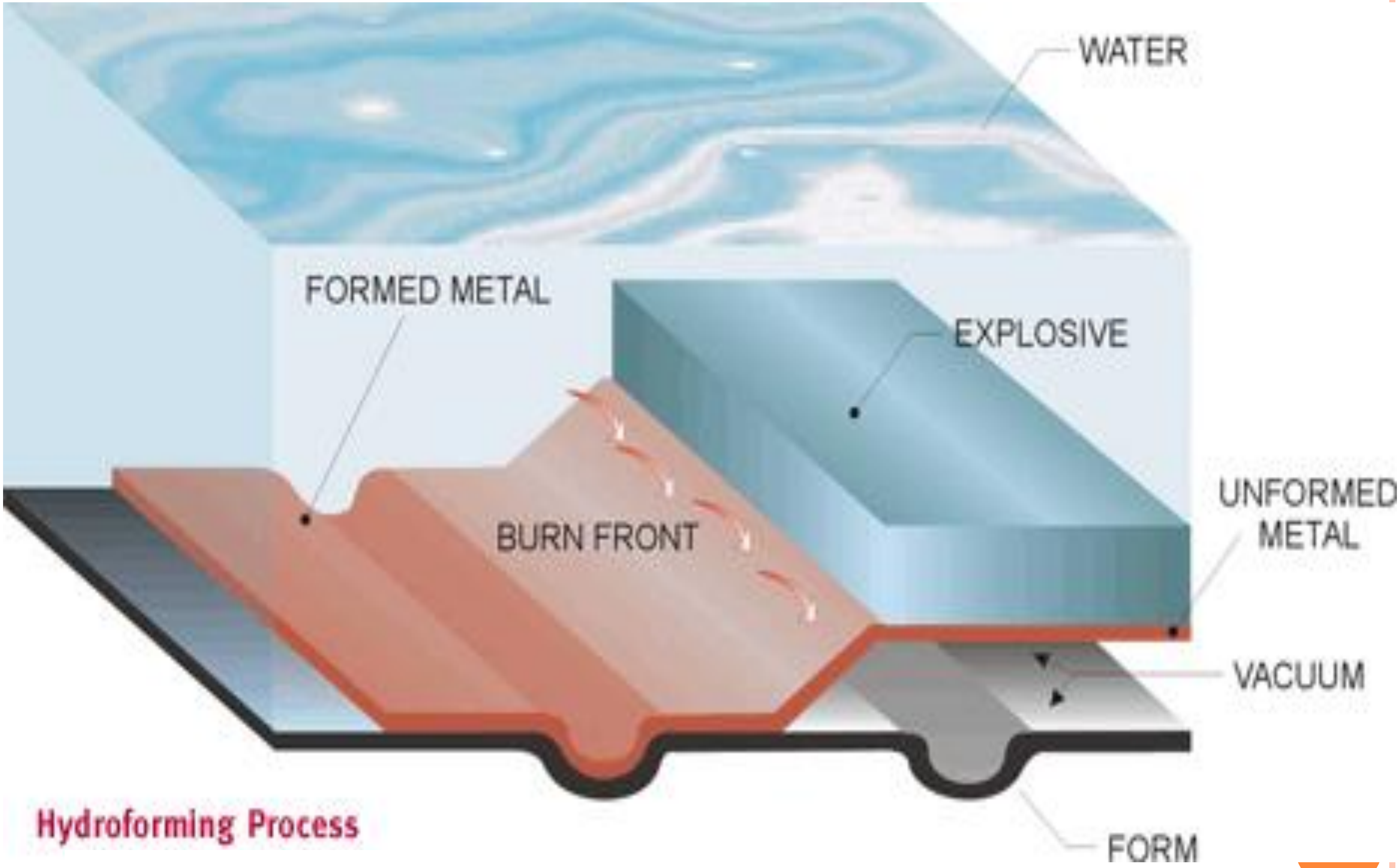
ADVANTAGES

- Parts can be formed that cannot be formed by conventional methods.
- Exotic metals, which do not readily lend themselves to conventional **forming** processes, may be formed over a wide range **of** sizes and configurations.
- The method is excellent for restrike operations.
- Springback after **forming** is reduced to a minimum.
- Dimensional tolerances are generally excellent.
- Variations from part to part are held to a minimum.
- Scrap **rate** is low.
- Less equipment and fewer dies cut down on production lead time.

- Explosive forming has evolved as one of the most dramatic of the new metalworking techniques.
- Explosive forming is employed in aerospace and aircraft industries and has been successfully employed in the production of automotive-related components.
- Explosive Forming can be utilized to form a wide variety of metals, from aluminum to high strength alloys.

- In this process the punch is replaced by an explosive charge.
- The process derives its name from the fact that the energy liberated due to the detonation of an explosive is used to form the desired configuration.
- The charge used is very small, but is capable of exerting tremendous forces on the workpiece.
- In Explosive Forming chemical energy from the explosives is used to generate shock waves through a medium (mostly water), which are directed to deform the workpiece at very high velocities.

- The process has been successfully used to form steel plates 25 mm thick x 4 m diameter and to bulge steel tubes as thick as 25 mm.
- Explosive forming is mainly used in the aerospace industries but has also found successful applications in the production of automotive related components.
- The process has the greatest potential in limited – production prototype forming and for forming large size components for which conventional tooling costs are prohibitively high.



Hydroforming Process

- The die assembly is put together on the bottom of a tank.
- Workpiece is placed on the die and blankholder placed above.
- A vacuum is then created in the die cavity.
- The explosive charge is placed in position over the centre of the workpiece.

- The explosive charge is suspended over the blank at a predetermined distance.
- The complete assembly is immersed in a tank of water.
- After the detonation of explosive, a pressure pulse of high intensity is produced.
- A gas bubble is also produced which expands spherically and then collapses until it vents at the surface of the water.
- When the pressure pulse impinges against the workpiece, the metal is displaced into the die cavity.

METHODS OF EXPLOSIVE FORMING

Explosive Forming Operations can be divided into two groups, depending on the position of the explosive charge relative to the workpiece.

Standoff Method

In this method, the explosive charge is located at some predetermined distance from the workpiece and the energy is transmitted through an intervening medium like air, oil, or water. Peak pressure at the workpiece may range from a few thousand psi to several hundred thousand psi depending on the parameters of the operation.

Contact Method

In this method, the explosive charge is held in direct contact with the workpiece while the detonation is initiated. The detonation produces interface pressures on the surface of the metal up to several million psi (35000 MPa).

SINTERING

- It is the process of consolidating either loose aggregate of powder or a green compact of the desired composition under controlled conditions of temperature and time.
- Types of sintering:
  - a) solid state sintering – This is the commonly occurring consolidation of metal and alloy powders. In this, densification occurs mainly because of atomic diffusion in solid state.

LIQUID PHASE SINTERING

- – The densification in improved by employing a small amount of liquid phase (1-10% vol). The liquid phase existing within the powders at the sintering temperature has some solubility for the solid.
- Sufficient amount of liquid is formed between the solid particles of the compact sample.
- During sintering, the liquid phase crystallizes at the grain boundaries binding the grains.
- During this stage, there is a rapid rearrangement of solid particles leading to density increase.

- In later stage, solid phase sintering occurs resulting in grain coarsening and densification rate slows down. Used for sintering of systems like tungsten-copper and copper-tin. Also covalent compounds like silicon nitride, silicon carbide can be made, that are difficult to sinter.

ACTIVATED SINTERING

- – IN this, an alloying element called ‘doping’ is added in small amount improves the densification by as much as 100 times than undoped compact samples. Example is the doping of nickel in tungsten compacts

REACTION SINTERING –

- IN this process, high temperature materials resulting from chemical reaction between the individual constituents, giving very good bonding.
- Reaction sintering occurs when two or more components reacts chemically during sintering to create final part.
- A typical example is the reaction between alumina and titania to form aluminium titanate at 1553 K which then sinters to form a densified product.
- Other than mentioned above, rate controlled sintering, microwave sintering, gas plasma sintering, spark plasma sintering are also developed and practiced

POWDER COMPACTION AND SINTERING

Definition

- Heat treatment to bond the metallic particles, thereby increasing strength and hardness
- Usually carried out at between 70% and 90% of the metal's melting point (absolute scale)
- Generally agreed among researchers that the primary driving force for sintering is reduction of surface energy
- Part shrinkage occurs during sintering due to pore size reduction

Stages

- Three stages of sintering
    - Burn-off (purge)- combusts any air and removes lubricants or binders that would interfere with good bonding
    - High-temperature- desired solid-state diffusion and bonding occurs
    - Cooling period- lowers the temperature of the products in a controlled atmosphere
- All three stages must be conducted in oxygen-free conditions

Sintering is carried out in three stages:

- First stage:

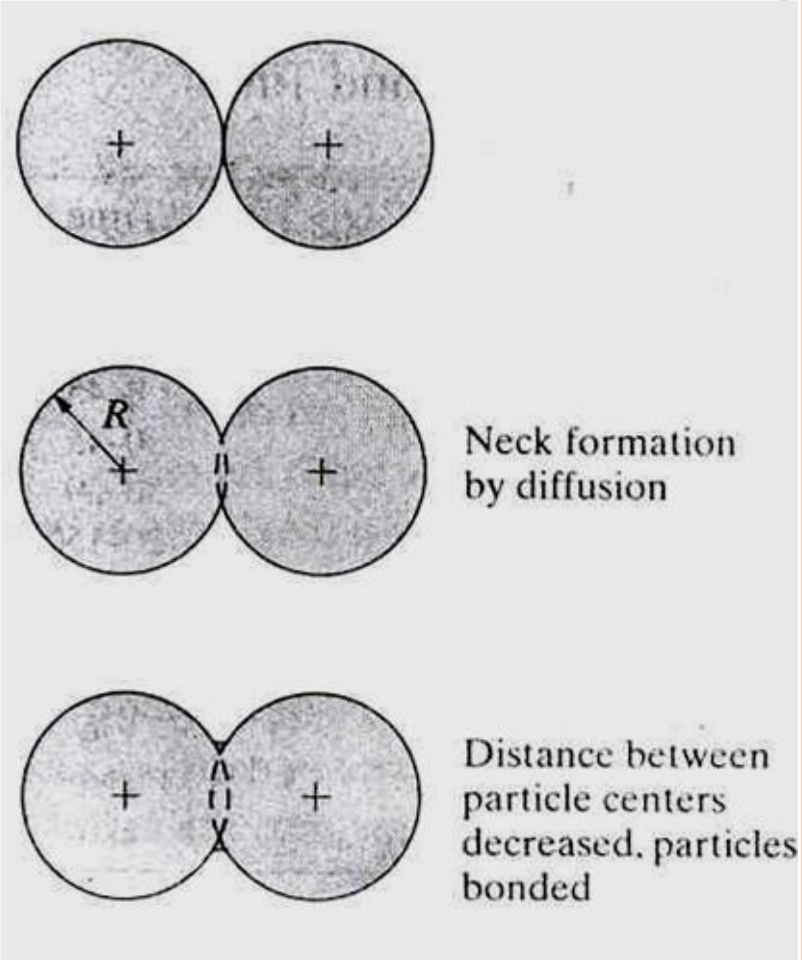
  Temperature is slowly increased so that all volatile materials in the green compact that would interfere with good bonding is removed

  - Rapid heating in this stage may entrap gases and produce high internal pressure which may fracture the compact

- Second stage:

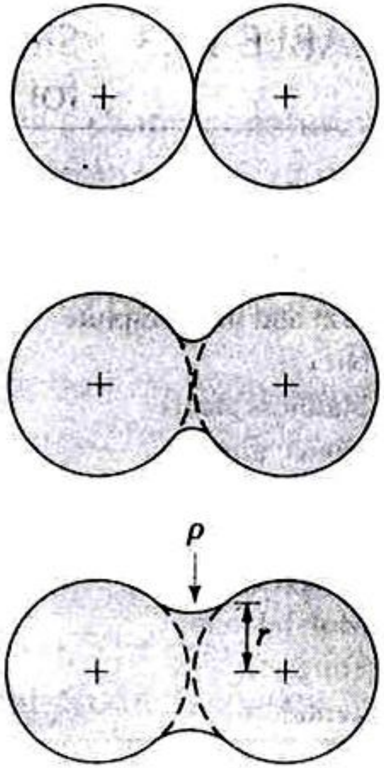
High temperature stage

- Promotes solid-state bonding by diffusion.
- Diffusion is time-temperature sensitive.
- Needs sufficient time



Second stage………

- Promotes vapour-phase transport
  - Because material is heated very close to MP, metal atoms will be released in the vapour phase from the particles
  - Vapour phase resolidifies at the interface

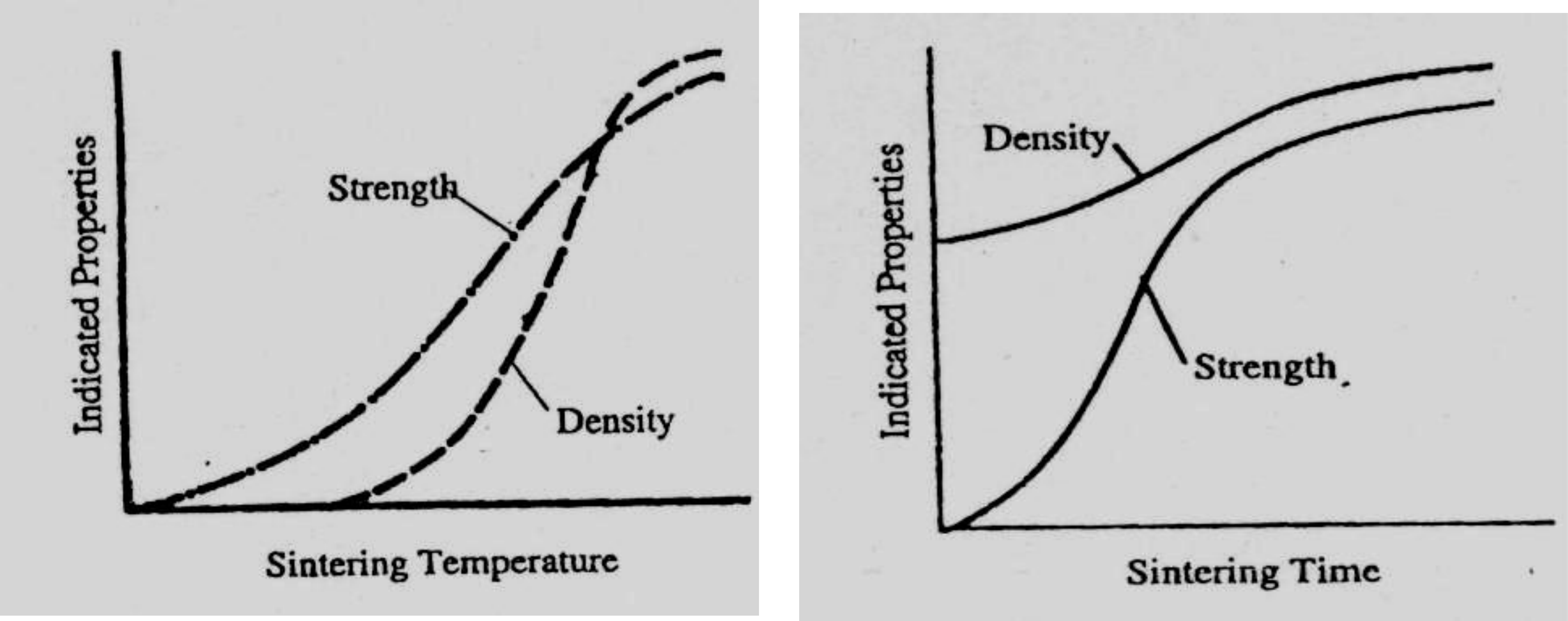


Neck formation by vapor phase material transport

Particles bonded, no shrinkage (center distances constant)

- Third stage:
- Sintered product is cooled in a controlled atmosphere
  - Prevents oxidation and thermal shock

Effect of variables



(a)

(b)

Effect of Sintering Temperature and Time on Strength and Density of Sintered product

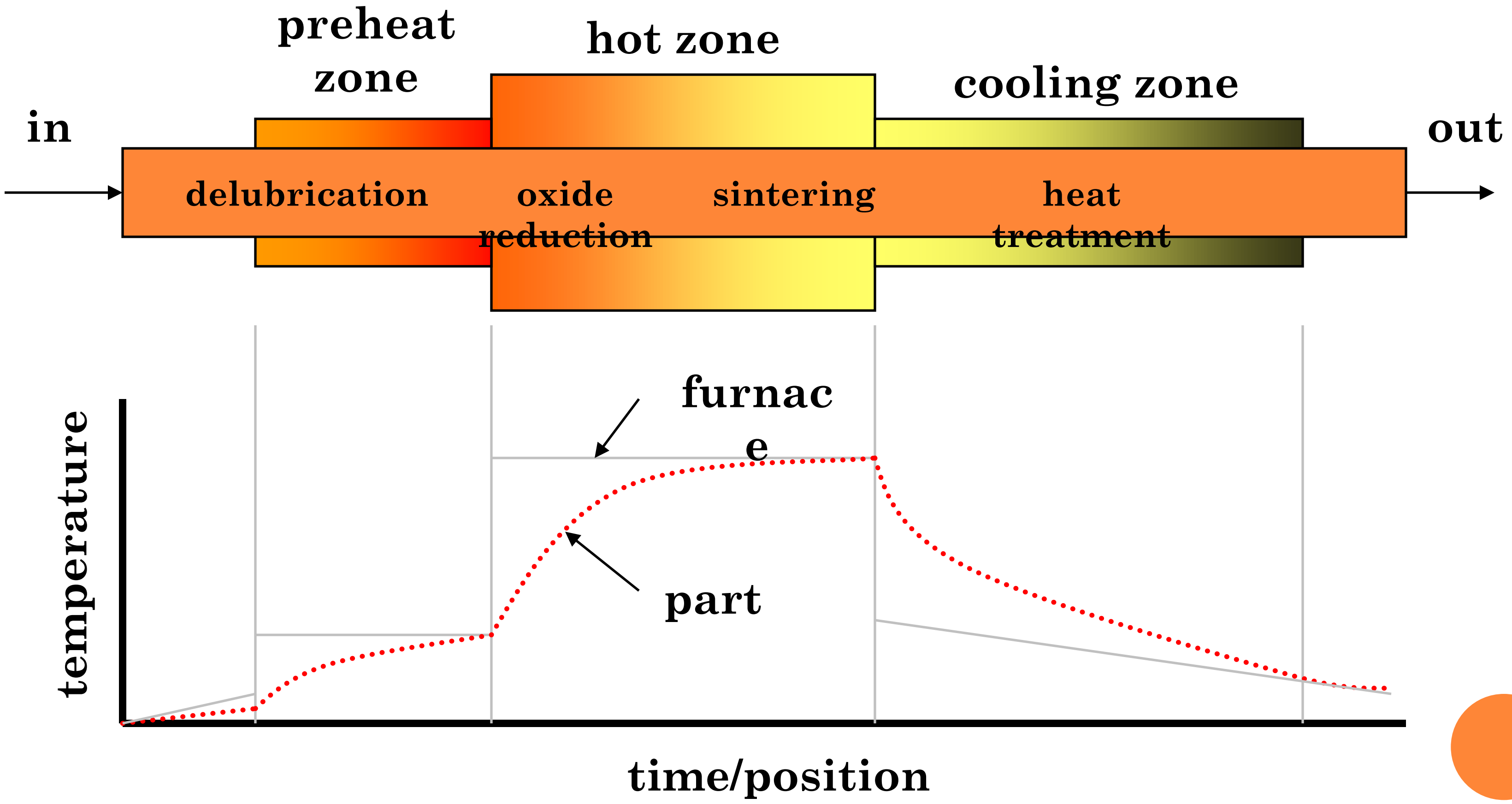
SINTERING TEMPERATURE AND TIME FOR VARIOUS METALS

TABLE 11.4

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

- Three stages of sintering
  - Burn-off (purge)- combusts any air and removes lubricants or binders that would interfere with good bonding
  - High-temperature- desired solid-state diffusion and bonding occurs
  - Cooling period- lowers the temperature of the products in a controlled atmosphere
- All three stages must be conducted in oxygen-free conditions

SINTERING SEQUENCE



SINTERING FURNACE

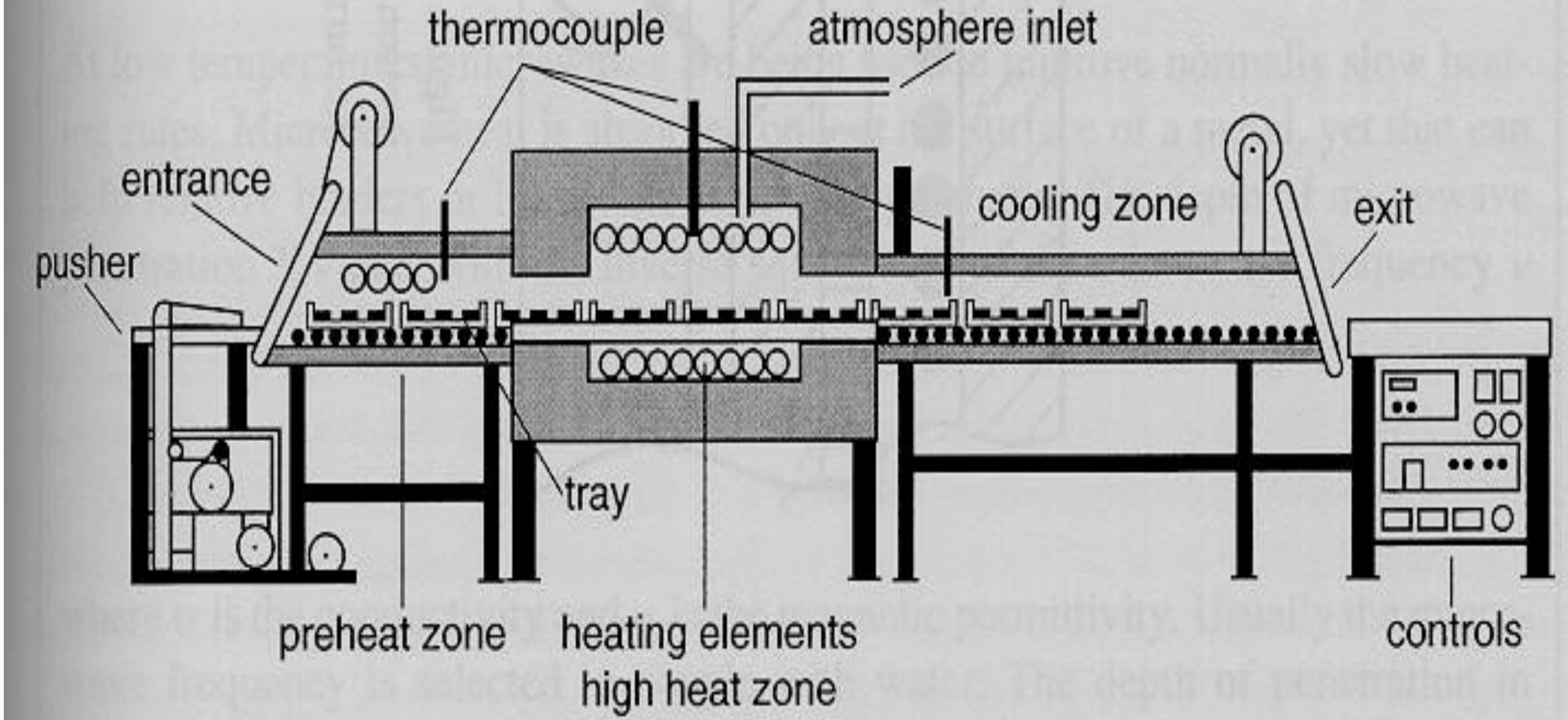


Figure 7.21. A sketch of a continuous pusher furnace, where trays are stoked one against another to create a constant flow of work through the preheat and high-heat zones.

SINTERING PRODUCTION LINES

Mechanism

- Sintering mechanisms are complex and depend on the composition of metal particles as well as processing parameters.
- As temperature increases two adjacent particles begin to form a bond by diffusion (solid-state bonding).

MECHANISM IN SOLID STATE SINTERING

- As discussed earlier, material or atom transport forms the basic mechanism for sintering process. A number of mechanisms have been proposed for sintering operation.
- These are:

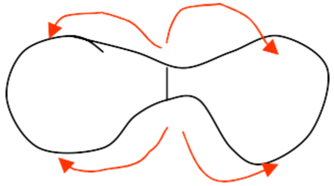
1. Evaporation condensation,

2. diffusion (can be volume diffusion, grain boundary diffusion, surface diffusion),

3. viscous flow,

4. plastic flow

EVAPORATION CONDENSATION,



- The basic principle of the mechanism is that the equilibrium vapor pressure over a concave surface (like neck) is lower compared to a convex surface (like particle surface).
- This creates the vapor pressure gradient between the neck region and particle surface.
- Hence mass transport occurs because of vapor pressure gradient from neck (concave surface) to particle surface (convex surface).

- The driving force of this is based on Gibbs-Thomson equation, $\mu - \mu_0 = RT \ln(p/p_0) = (-\gamma)(\Omega)/r$ where μ and μ_0 are chemical potentials of initial and final surfaces, R is universal gas constant, T is temperature in K, p and p_0 are partial pressures over the curved and flat surface respectively, γ is the surface free energy, Ω is the atomic volume

2. DIFFUSION MECHANISM

- Diffusion occurs because of vacancy concentration gradient. In the case of two spheres in contact with each other, a vacancy gradient is generated between the two surfaces. This condition can be given by,
  - $\mu - \mu_0 = RT\ \ln(C/C_0) = (-\gamma)(\Omega)/r$
  - Where C and C_0 are the vacancy concentration gradient around the curved and flat surface.
- Kuczynski has derived empirical relation for neck growth rate for the following two cases,
  - i) a sphere in contact with flat surface – the rate of neck growth is proportional to seventh root of time ($t^{1/7}$),
  - ii) when two spheres are in contact – the neck growth rate is, $x^5/a^2 = 40\ \gamma\ \Omega D.t/RT$ where a – radius of sphere, D – volume diffusivity of material, Ω – atomic or molecular volume.

- Neck growth due to surface diffusion, lattice diffusion, vapour transport, grain boundary transport from GB source, lattice diffusion from sources on GB, lattice diffusion from dislocation sources

3. VISCOUS FLOW MECHANISM

- According to this concept, sintering occurs due to the presence of lattice vacancies. This is important in sintering of glass. Frenkel developed the equation, $(x^2/a) = 3/2\ (\gamma/\eta).t$ where x is the neck radius & a is the radius of particle. Increasing the temperature results in increased plasticity of metal powders.
- Balshin proposed the following mechanisms to happen during sintering.

1.Particle rearrangement,

2. particle shape change,

3. grain growth.

- Frenkel suggested that surface tension lead to sintering and solids could behave like newtonian liquids at high temperatures i.e., sintering occurs by viscous flow.
- He derived an equation for the viscosity coefficient, $\eta = kT/D\Omega$ where k is the Boltszman's constant, T is the absolute temperature, D is the self-diffusion coefficient, Ω is the atomic volume.
- He derived an expression on neck growth during sintering of two spherical particles of radius *a* with time *t* as, $x^2 = 3a\gamma/2^{\eta}$ where *x* is the neck radius and γ is the surface energy

4. PLASTIC FLOW MECHANISM

- Bulk flow of material by movement of dislocations has been proposed as possible mechanism for densification during sintering.
- Importance was given to identify dislocation sources during the sintering process.
- Even if frank read sources are present in the neck region, the stress available for dislocation generation is very small, indicating that the generation of dislocation must come from free surfaces.

- Only if the surface is very small of the order of 40 nm, the stress required for dislocation generation will be sufficient.
- But experimental results have shown the absence of applied stress and plastic flow is expected to occur during early stages of sintering.
- Plastic flow mechanism is predominant during hot pressing.

Sintering stages

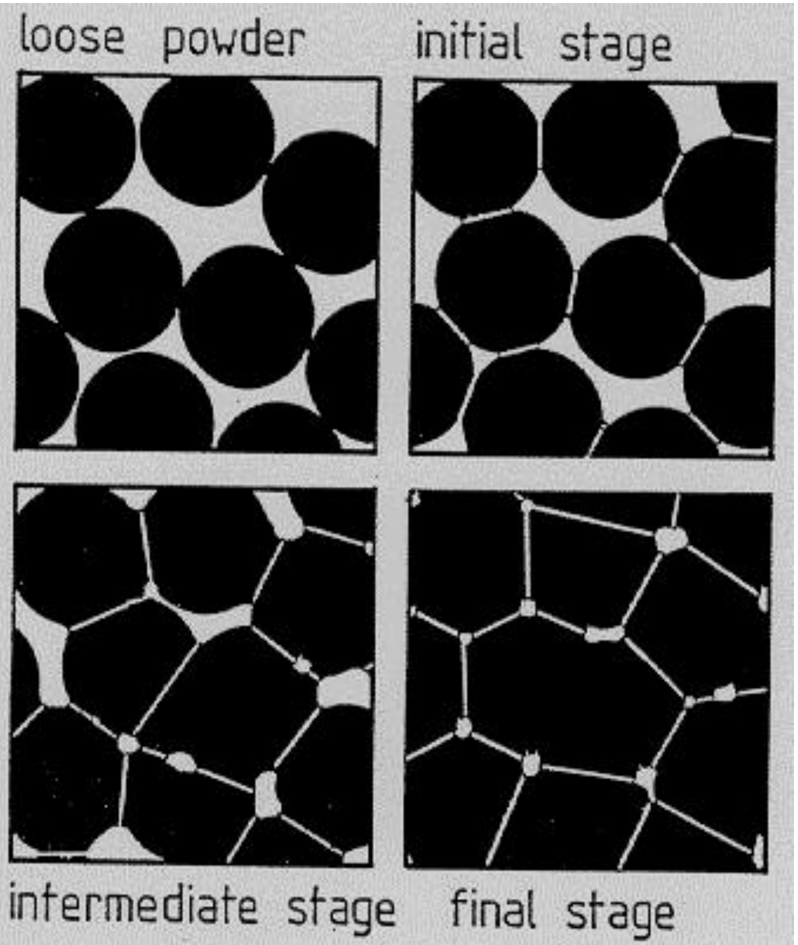
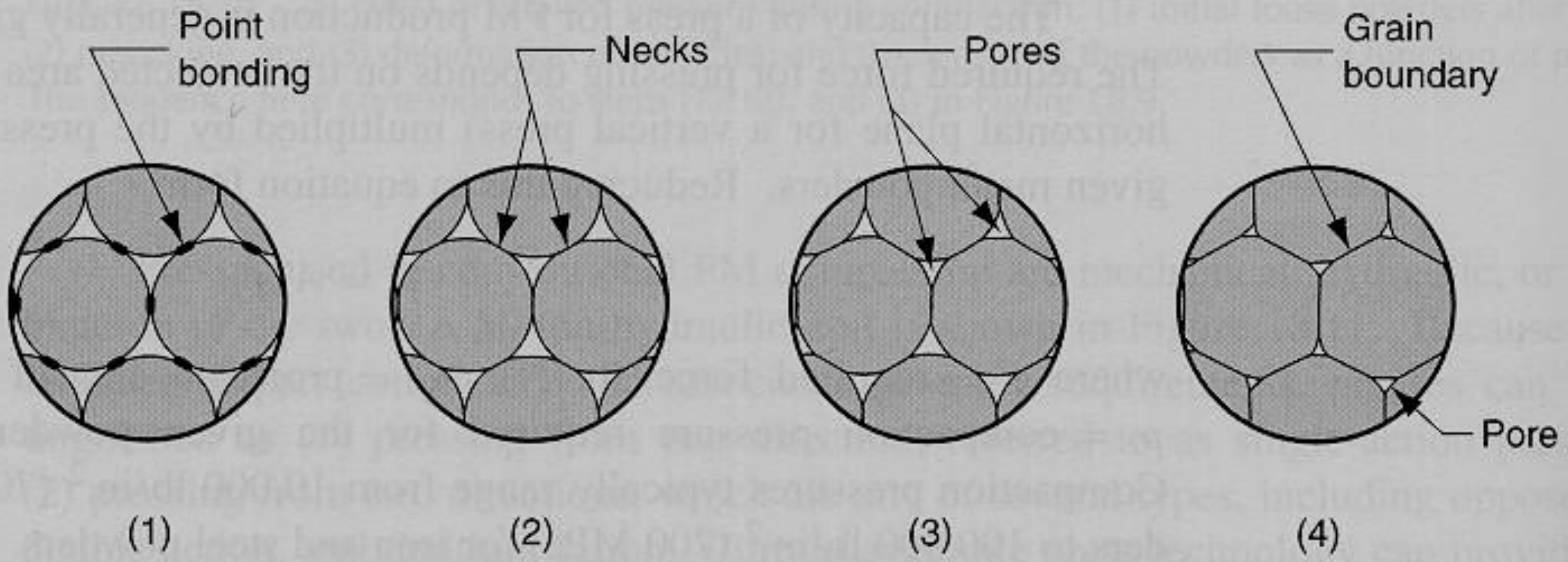


Figure 1: Schematic of loose powder sintering (20).

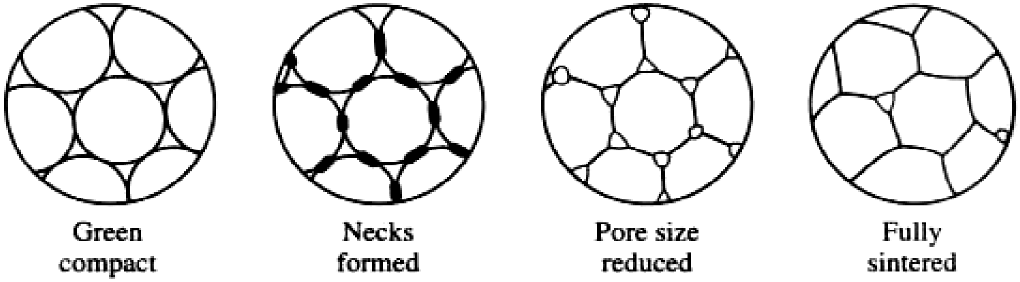
SINTERING STAGES

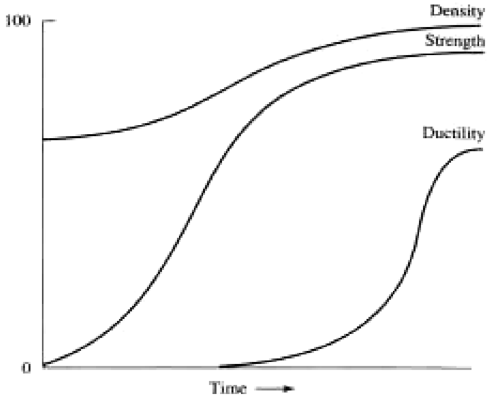
FIGURE 18.12 Sintering on a microscopic scale: (1) particle bonding is initiated at contact points; (2) contact points grow into "necks"; (3) the pores between particles are reduced in size; and (4) grain boundaries develop between particles in place of the necked regions.



SINTERING

- Parts are heated to 0.7~0.9 T_m.
- Transforms compacted mechanical bonds to much stronger metallic bonds.





- **Shrinkage always occurs:**

$$Vol\_shrinkage = \frac{V_{sintered}}{V_{green}} = \frac{\rho_{green}}{\rho_{sintered}}$$

$$Linear\_shrinkage = \left(\frac{\rho_{green}}{\rho_{sintered}}\right)^{1/3}$$

SINTERING

Diagram of particles showing the possible movements of atoms during sintering

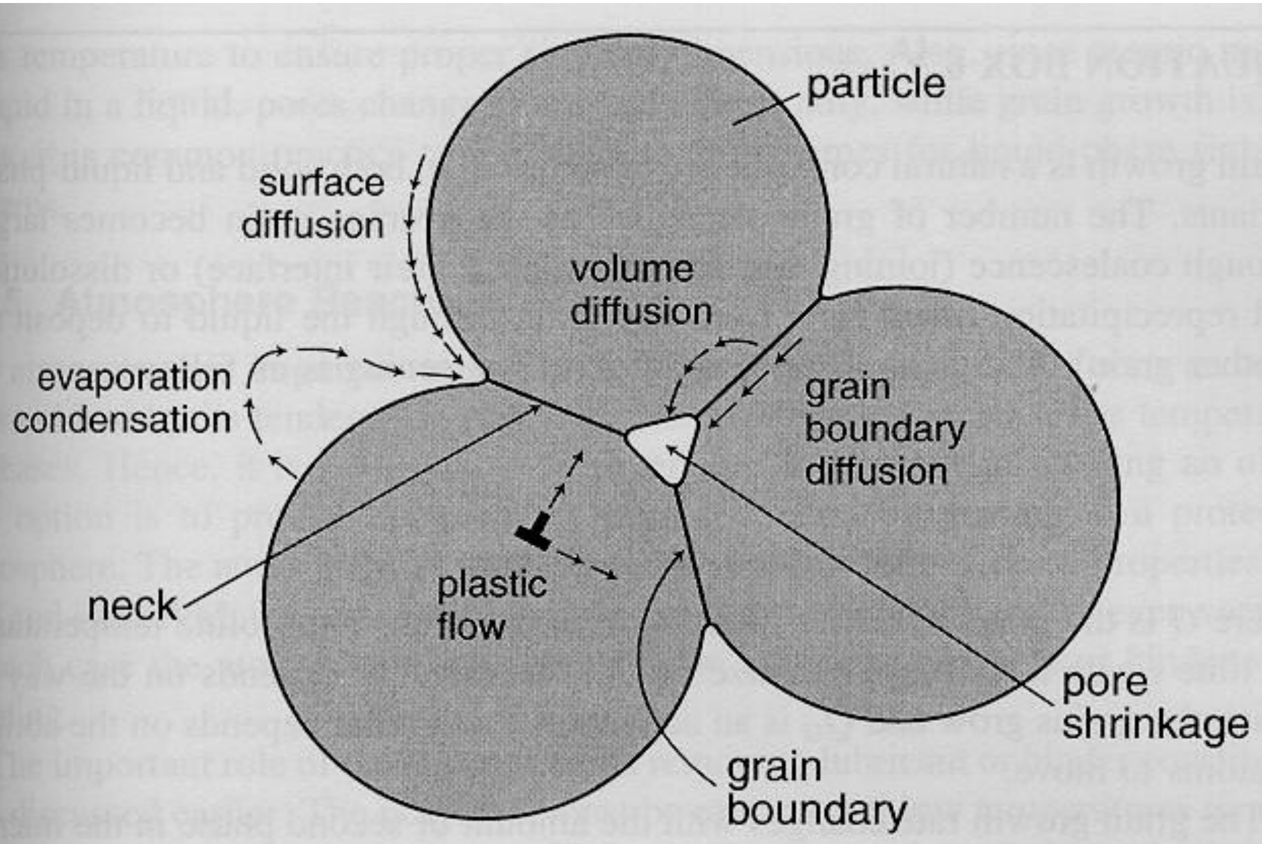
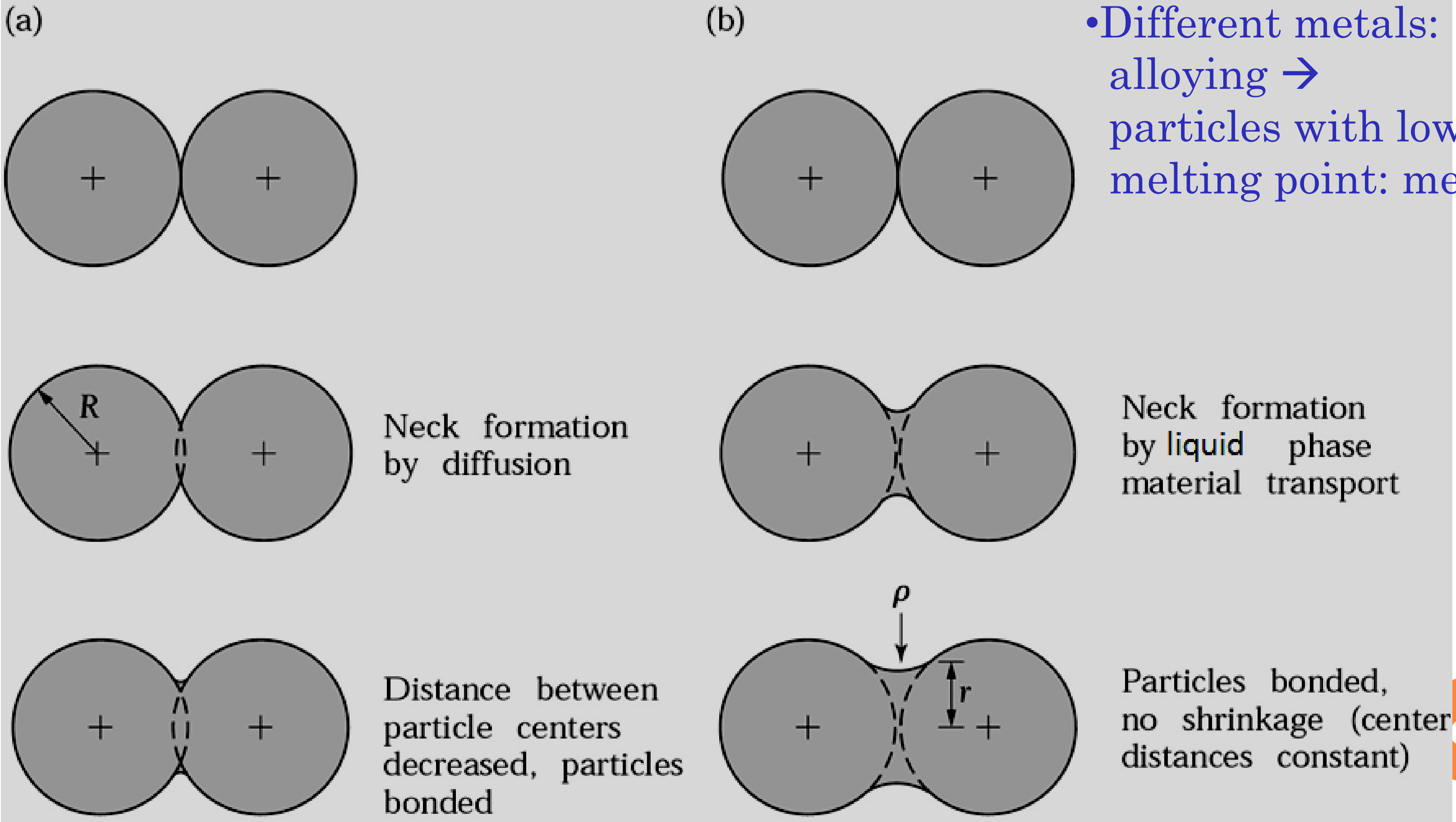


Figure 6.4. A three-particle sketch of sintering, showing the several possible paths of atomic motion involved with particle bonding (neck growth) and pore shrinkage (densification).

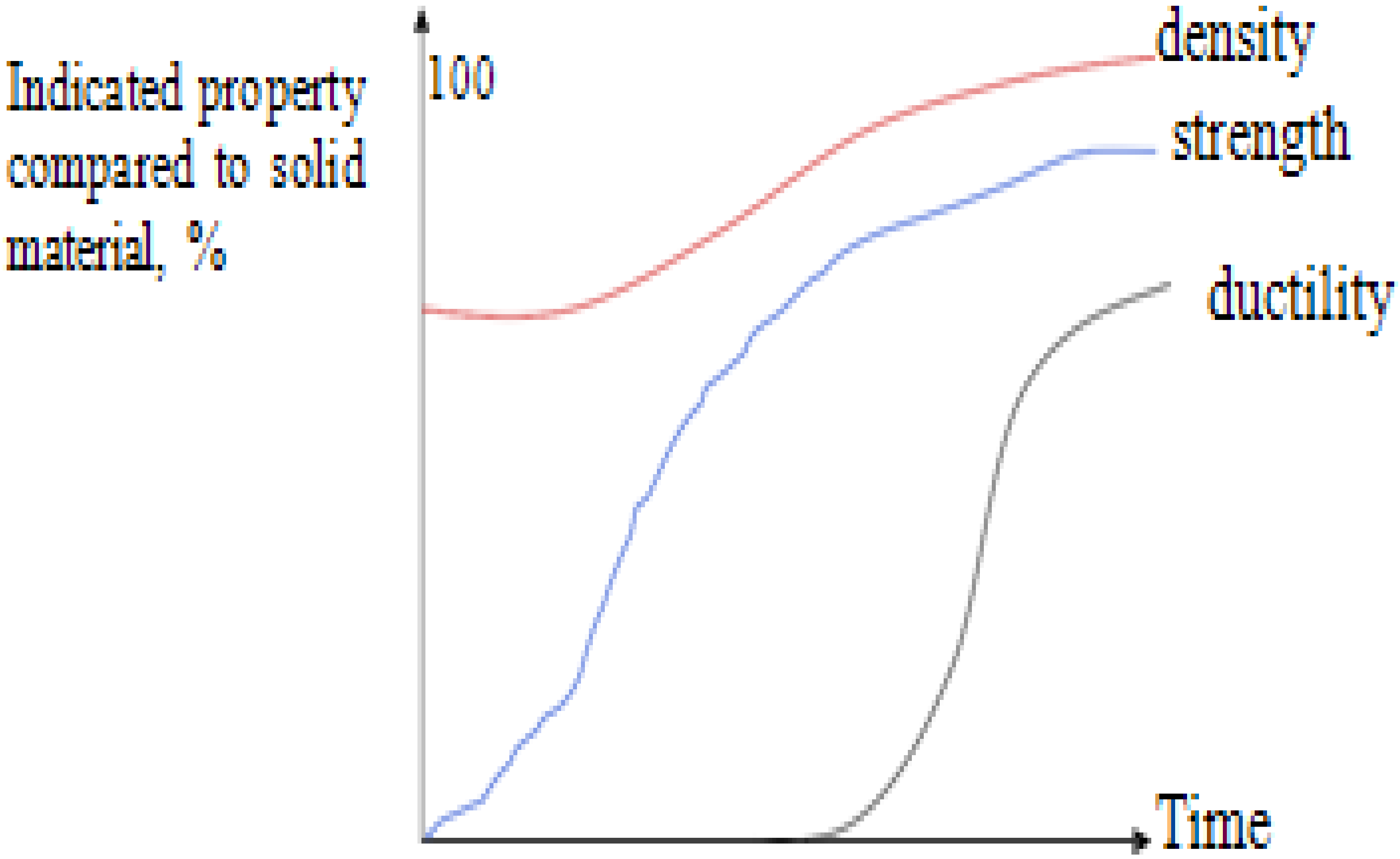
Schematic illustration of two mechanisms for sintering metal powders: (a) solid-state material transport; (b) liquid-phase material transport. *R* = particle radius, *r* = neck radius, and ρ = neck profile radius.



- Different metals: alloying → particles with low melting point: melt

PROPERTY CHANGES DURING SINTERING

- Densification is proportional to the shrinkage or the amount of pores removed in the case of single component system
- IN multicomponent system, expansion rather than shrinkage will result in densification and hence densification can not be treated as equal to the amount of porosity removed.
- densification results in mechanical property change like hardness, strength, toughness, physical properties like electrical, thermal conductivity, magnetic properties etc. Also change in composition is expected due to the formation of solid solution.


Indicated property compared to solid material, %
100
density
strength
ductility
Time