

446.305A MANUFACTURING PROCESSES

Chapter 11. Properties and Processing of Metal Powders, Ceramics, Glasses, Composites, and Superconductors

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Introduction

- **Powder Metallurgy (P/M, 분말야금)**
 - Compacting metal powders in dies and sintering them.
- **Typical products**
 - Gears, cams, bushings, cutting tools, automotive components, and etc.
- **Advantages**
 - Material density in P/M is a controllable variable.
 - Low density : porous filters.
 - Full density : structural parts.
 - Competitive with processes such as casting, forging, and machining for relatively complex parts made of high-strength and hard alloys.

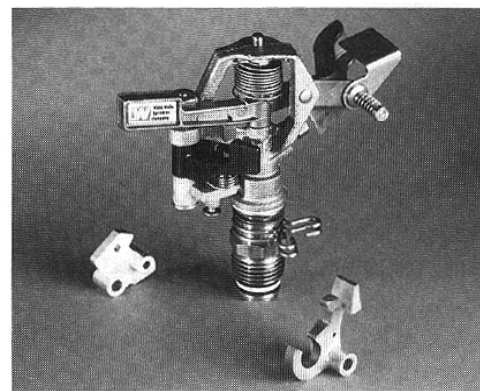
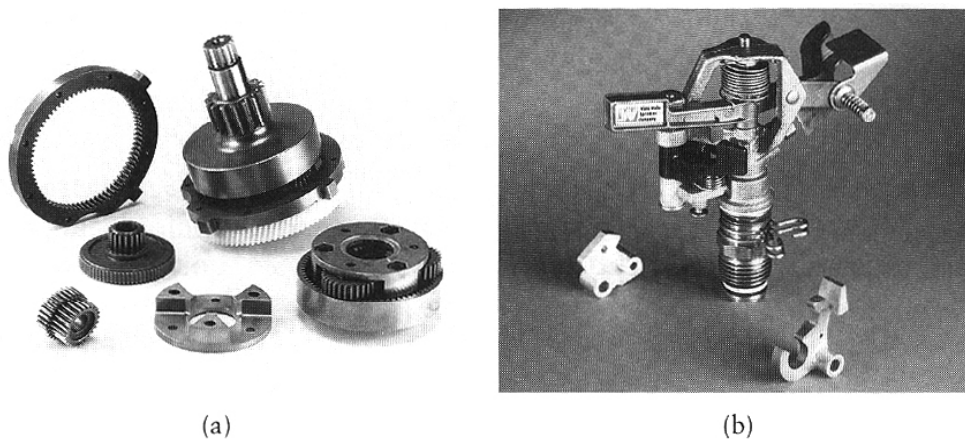
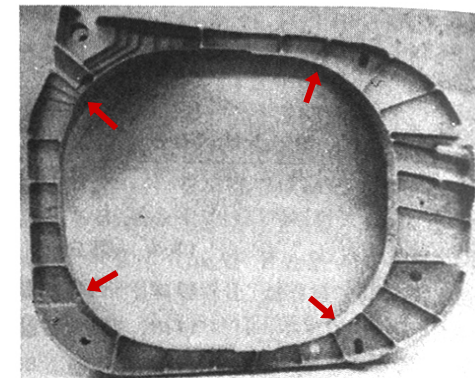


FIGURE 11.1
(a) Examples of typical parts made by powder-metallurgy processes. (b) Upper trip lever for a commercial irrigation sprinkler, made by P/M. This part is made of unleaded brass alloy; it replaces a die-cast part, with a 60% cost savings. *Source:* Reproduced with permission from *Success Stories on P/M Parts*, Princeton, NJ: Metal Powder Industries Federation, 1998.



Ref.
S. Kalpakjian, "Manufacturing Processes for Engineering Materials",
3rd/4th ed. Addison Wesley

Metal powders



- Powder production → Blending → Compaction → Sintering → Finishing operations

TABLE 11.2

CHARACTERISTICS OF POWDER-METALLURGY PROCESSING

ADVANTAGES

- Availability of a wide range of compositions to obtain special mechanical and physical properties, such as stiffness, damping characteristics, hardness, density, toughness, and electrical and magnetic properties. Some of the high alloyed new superalloys can be manufactured into parts only by P/M processing.
- A net- or near-net-shape technique for making parts from high-melting-point refractory metals, which would be difficult or uneconomical to make by other methods.
- High production rates on relatively complex parts, with automated equipment requiring little labor.
- Good dimensional control and, in many instances, elimination of machining and finishing operations, thus eliminating scrap and waste and saving energy.
- Capability for impregnation and infiltration for special applications.

LIMITATIONS

- Size of parts, complexity of part shapes, and press capacity.
 - High cost of powder metals compared to other raw materials.
 - High cost of tooling and equipment for small production runs.
 - Mechanical properties, such as strength and ductility, that are generally lower than those obtained by forging. However, the properties of full-density P/M parts made by HIP or additional forging can be better than those made by other processes.
-

Powder fabrication

- **Atomization (입자화)**
- **Reduction (환원) : reduction of metal oxides using gases such as hydrogen and carbon monoxide.**

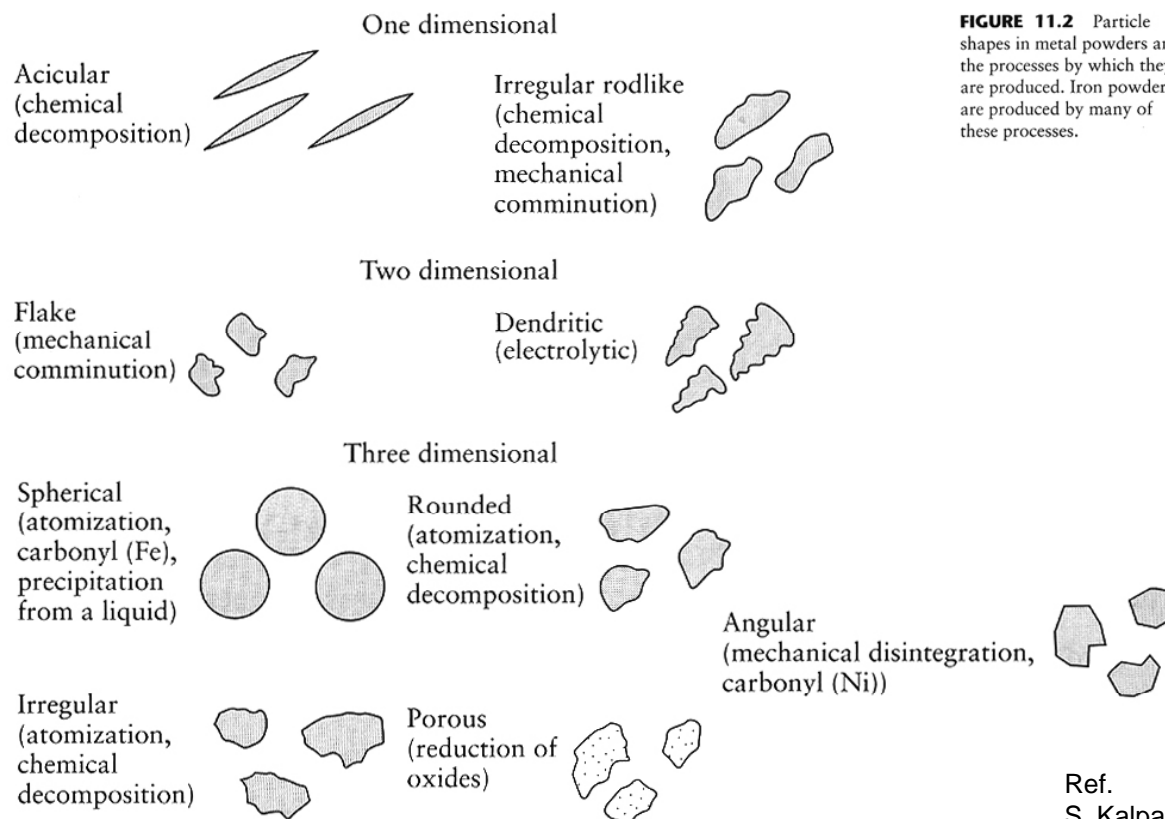


FIGURE 11.2 Particle shapes in metal powders and the processes by which they are produced. Iron powders are produced by many of these processes.

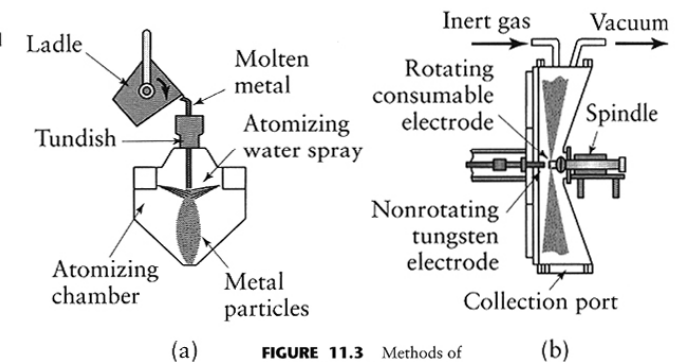


FIGURE 11.3 Methods of metal-powder production by atomization: (a) melt atomization and (b) atomization with a rotating consumable electrode.

Ref.
S. Kalpakjian, "Manufacturing Processes for Engineering Materials",
3rd/4th ed. Addison Wesley

Compaction (압축) (1)

- The step in which the blended powders are pressed into shapes in dies, using presses that are either hydraulically or mechanically actuated.

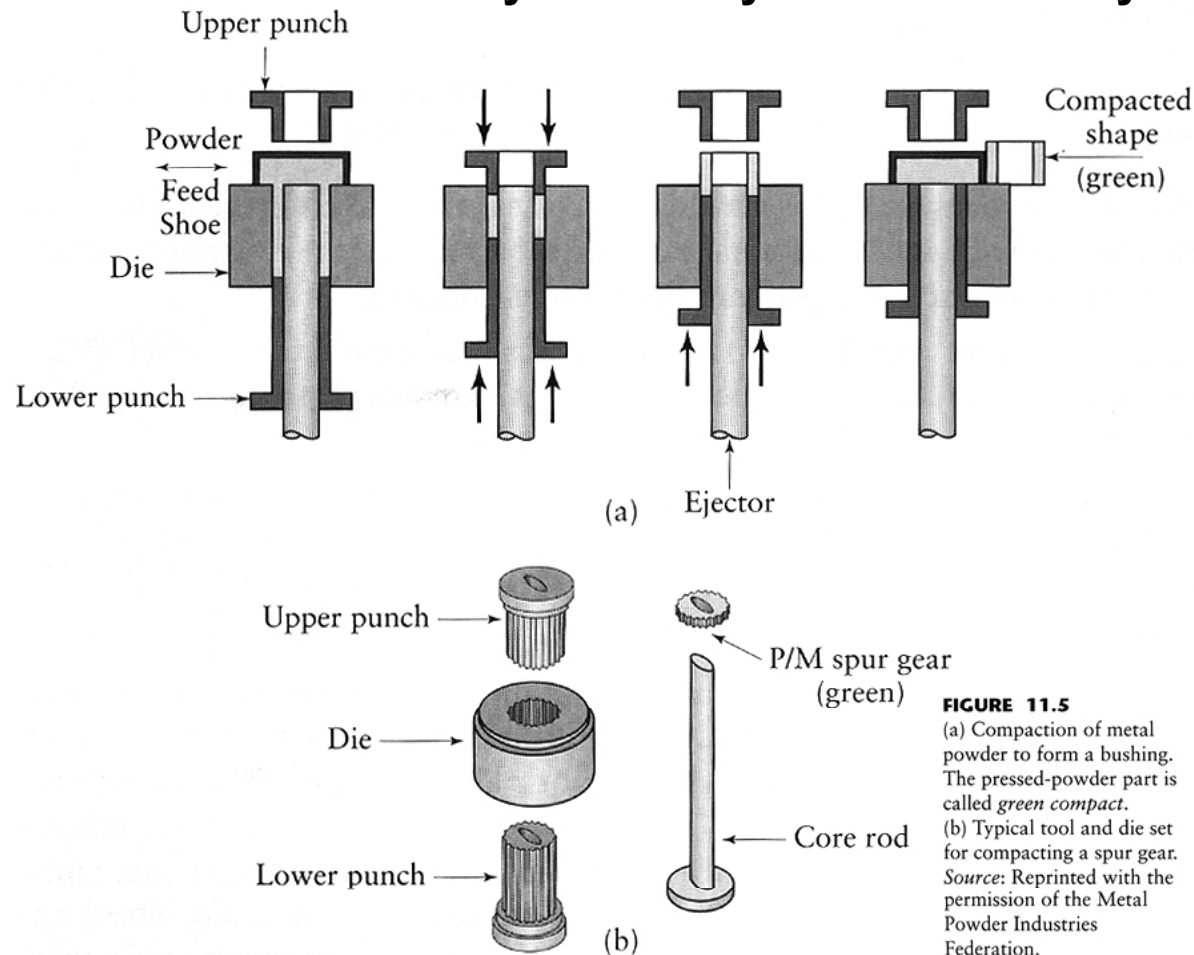


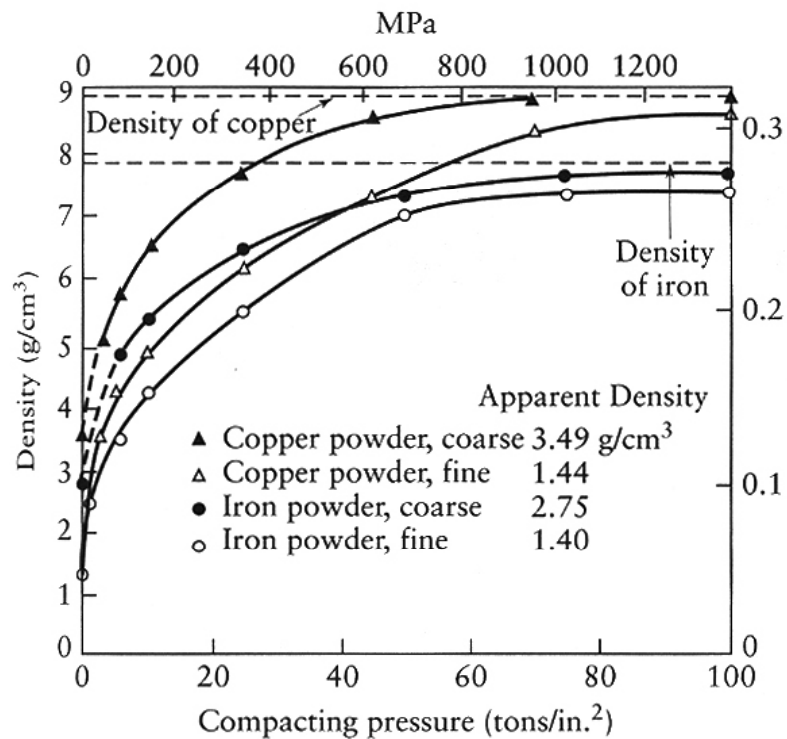
FIGURE 11.5

(a) Compaction of metal powder to form a bushing. The pressed-powder part is called *green compact*.
 (b) Typical tool and die set for compacting a spur gear.
 Source: Reprinted with the permission of the Metal Powder Industries Federation.

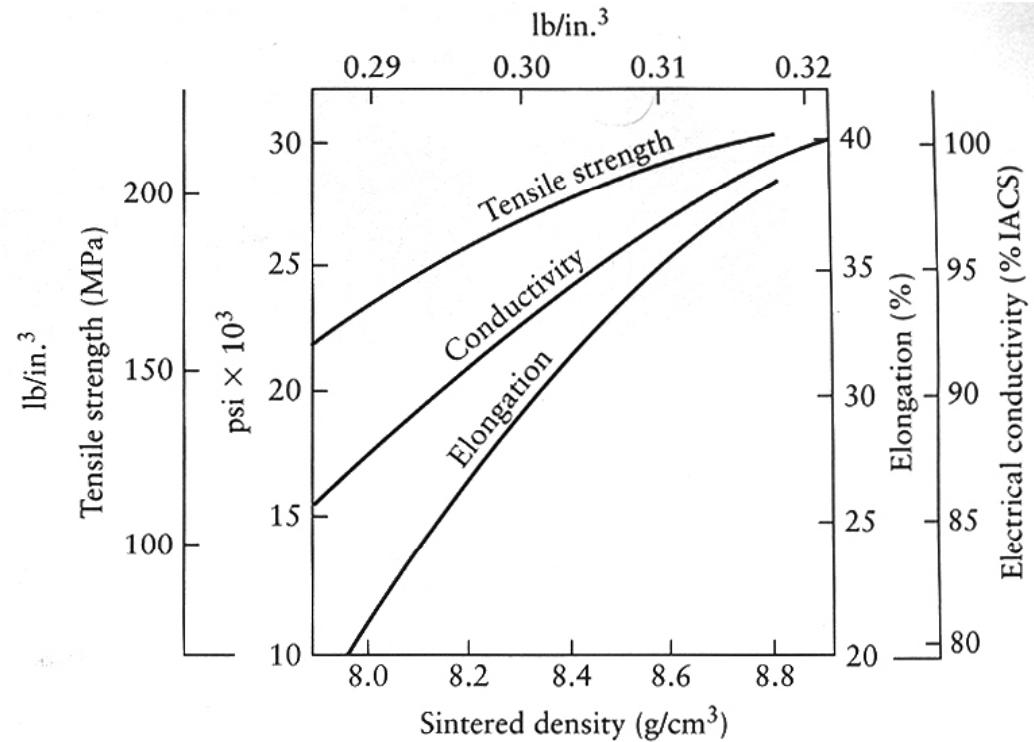
Ref.

S. Kalpakjian, "Manufacturing Processes for Engineering Materials", 3rd/4th ed. Addison Wesley

Compaction (압축) (2)



(a)



(b)

FIGURE 11.6 (a) Density of copper- and iron-powder compacts as a function of compacting pressure. Density greatly influences the mechanical and physical properties of P/M parts. Source: F. V. Lenel, *Powder Metallurgy: Principles and Applications*, Princeton, NJ: Metal Powder Industries Federation, 1980. Reprinted by permission of Metal Powder Industries Federation, Princeton, NJ. (b) Effect of density on tensile strength, elongation, and electrical conductivity of copper powder. IACS means International Annealed Copper Standard for electrical conductivity.

Ref.

S. Kalpakjian, "Manufacturing Processes for Engineering Materials", 3rd/4th ed. Addison Wesley

Isostatic pressing (균형가압)

- Cold isostatic pressing (CIP, 냉간균형가압)
- Hot isostatic pressing (HIP, 열간균형가압)

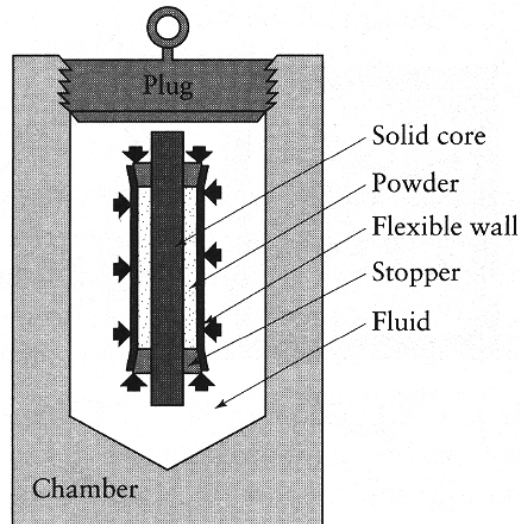


FIGURE 11.9 Schematic illustration of cold isostatic pressing as applied to formation of a tube. The powder is enclosed in a flexible container around a solid core rod. Pressure is applied isostatically to the assembly inside a high-pressure chamber. *Source:* Reprinted with permission from Randall M. German, *Powder Metallurgy Science*, Princeton, NJ: Metal Powder Industries Federation, 1984.

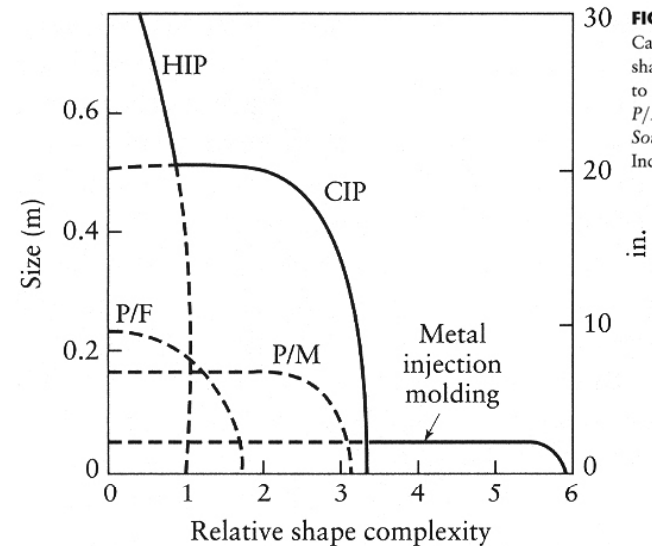


FIGURE 11.10 Capabilities of part size and shape complexity according to various P/M operations. P/F means powder forging. *Source:* Metal Powder Industries Federation.

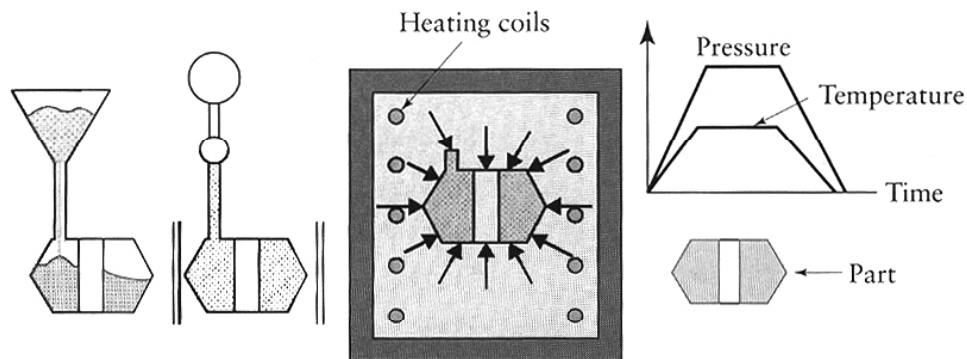


FIGURE 11.11 Schematic illustration of hot isostatic pressing. The pressure and temperature variation versus time are shown in the diagram. *Source:* Reprinted with permission from Randall M. German, *Powder Metallurgy Science*, Princeton, NJ: Metal Powder Industries Federation, 1984.

Fill
can
(a)

Vacuum
bake-out
(b)

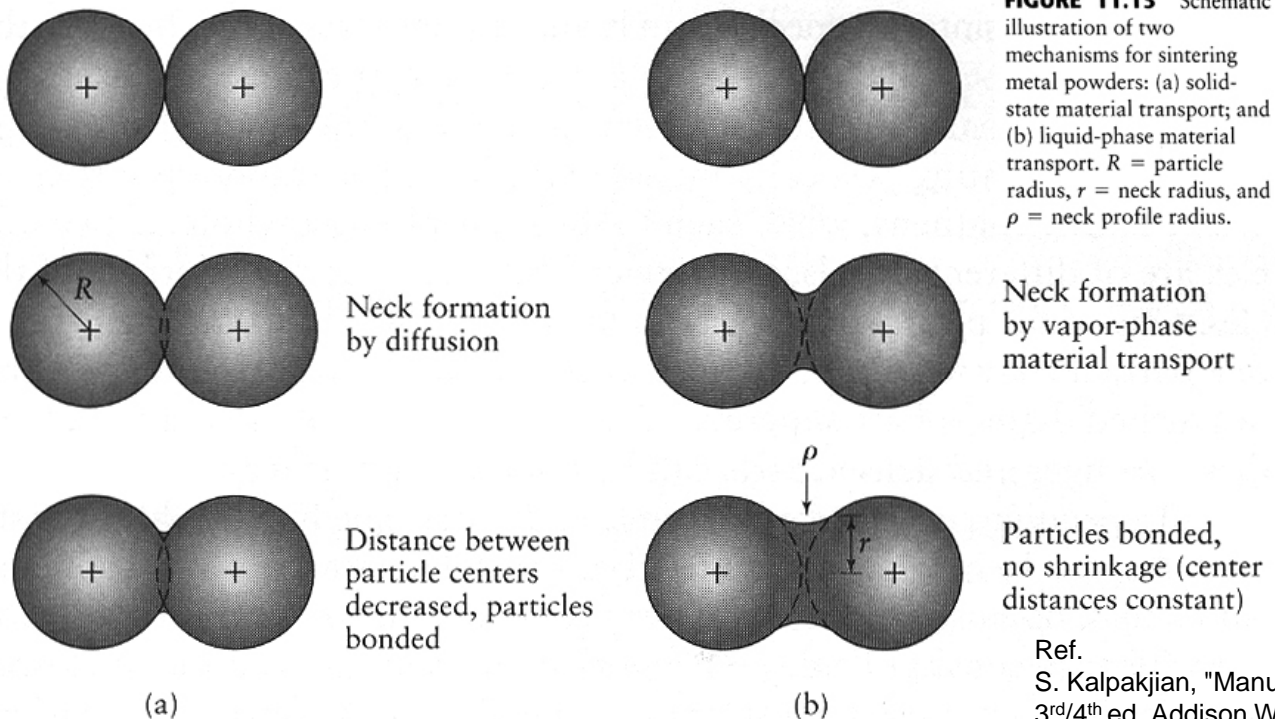
Hot isostatic
press
(c)

Remove
can
(d)

Ref.
S. Kalpakjian, "Manufacturing Processes for Engineering Materials",
3rd/4th ed. Addison Wesley

Sintering (소결) (1)

- The process whereby compressed metal powder is heated in a controlled-atmosphere (hydrogen, ammonia, nitrogen) furnace to a temperature below its melting point, but sufficiently high to allow bonding of the individual particles.
- Sintering temp. : 70% ~ 90% of the melting point of the metal or alloy.
- Continuous-sintering furnace : for most production today.



Ref.
S. Kalpakjian, "Manufacturing Processes for Engineering Materials",
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Sintering (소결) (2)

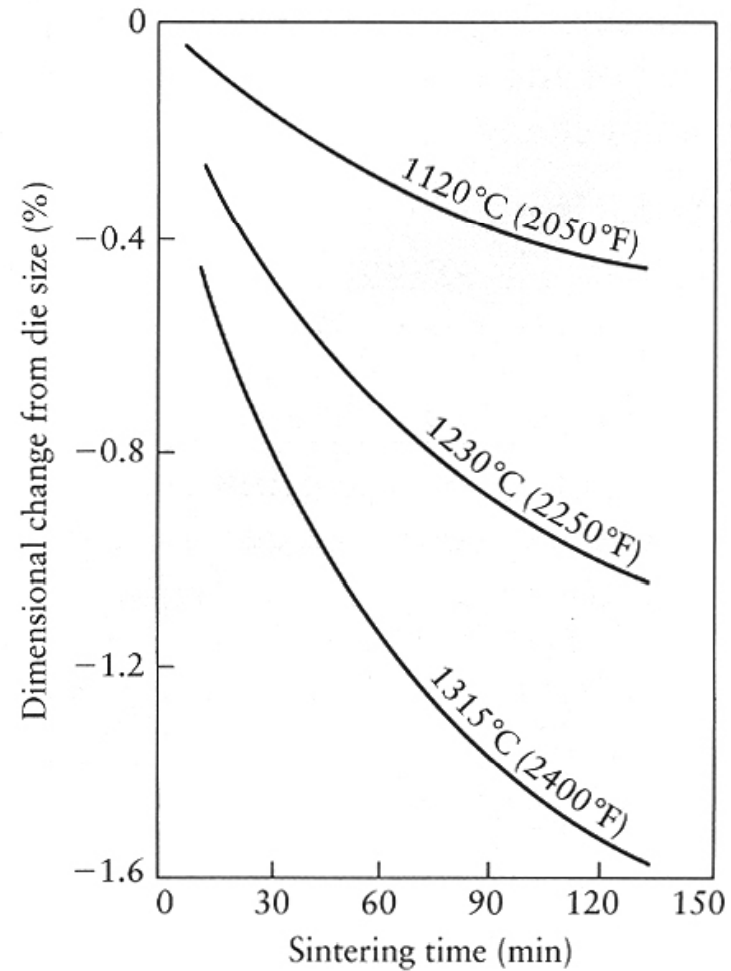
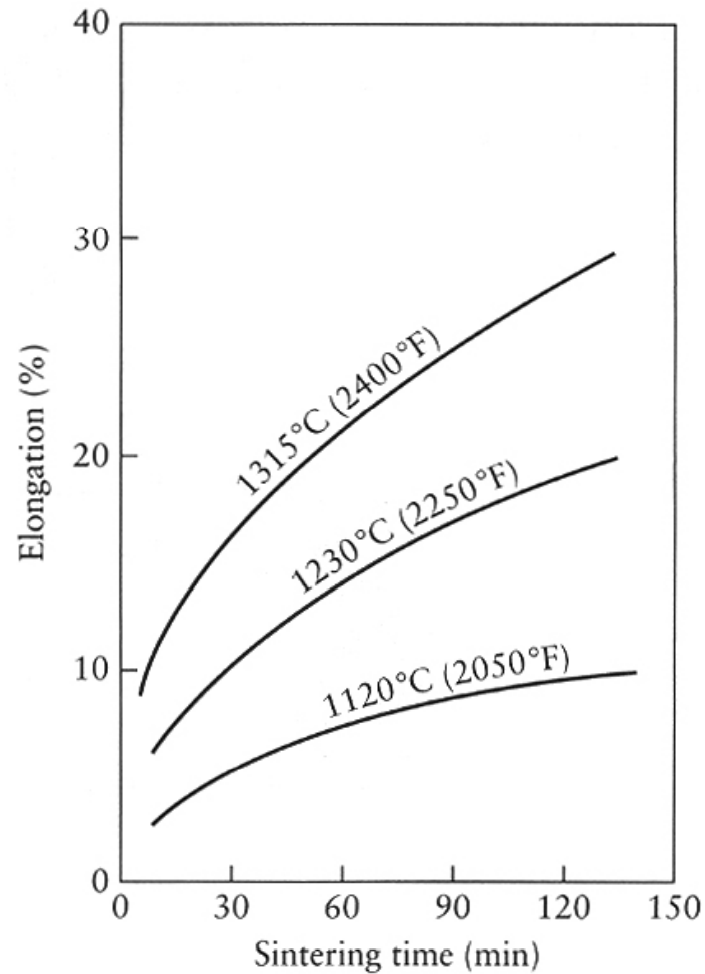


FIGURE 11.14 Effect of sintering temperature and time on elongation and dimensional change during sintering of type 316L stainless steel. Source: ASM International.

Ref.

S. Kalpakjian, "Manufacturing Processes for Engineering Materials", 3rd/4th ed. Addison Wesley

Sintering (소결) (4)

TABLE 11.4

Mechanical Property Comparisons for Ti-6Al-4V Titanium Alloy

Process	Density (%)	Yield Stress (MPa)	Ultimate Tensile Strength (MPa)	Elongation (%)	Reduction of Area (%)
Cast	100	840	930	7	15
Cast and forged	100	875	965	14	40
Powder metallurgy					
Blended elemental (P + S)*	98	786	875	8	14
Blended elemental (HIP)*	>99	805	875	9	17
Realloyed (HIP)*	100	880	975	14	26

(*) P + S = pressed and sintered; HIP = hot isostatically pressed.

Source: R. M. German.

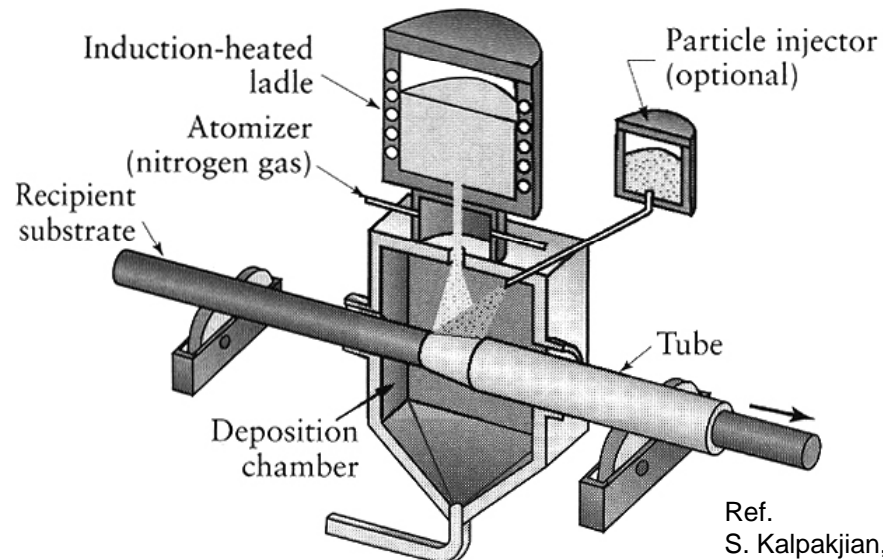


FIGURE 11.12 Spray casting (Osprey process) in which molten metal is sprayed over a rotating mandrel to produce seamless tubing and pipe. Source: After J. Szekeley, "Can Advanced Technology Save the U.S. Steel Industry," *Scientific American*, July 1987, © George V. Kelvin/Scientific American.

Ref.
S. Kalpakjian, "Manufacturing Processes for Engineering Materials",
3rd/4th ed. Addison Wesley

Design considerations

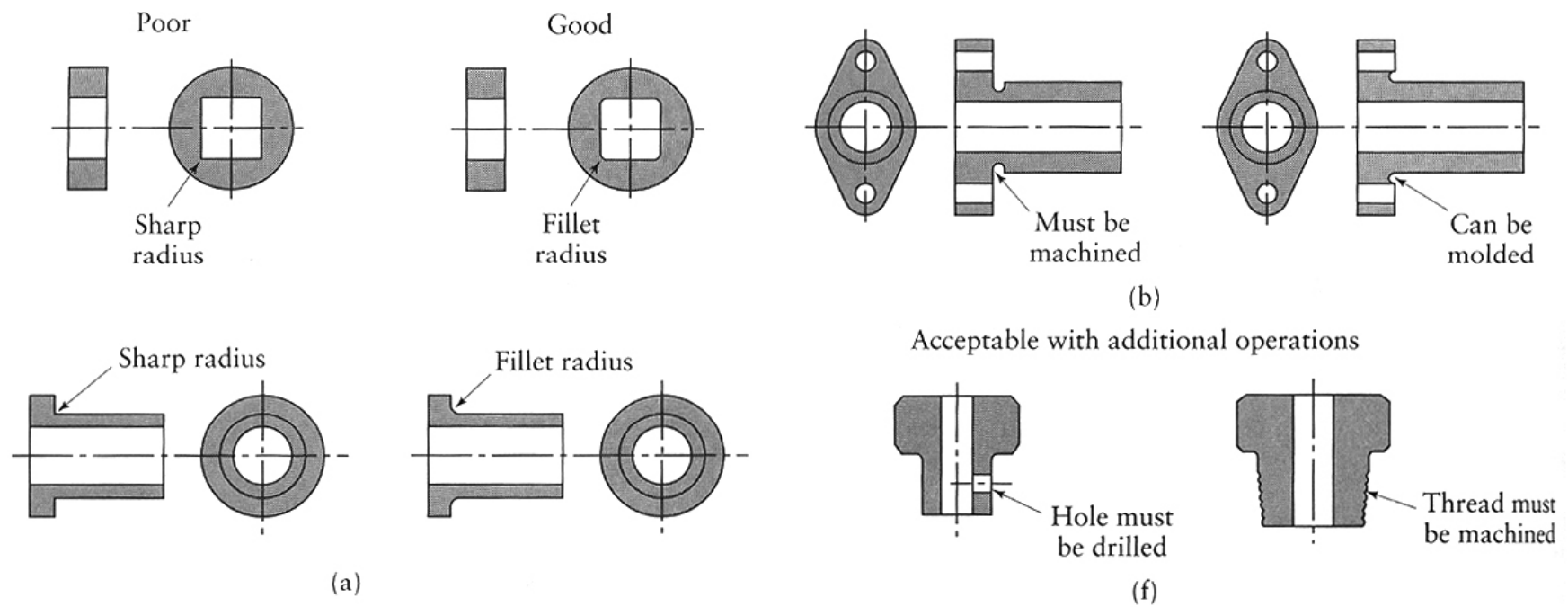


FIGURE 11.17 Examples of P/M parts, showing poor and good designs. Note that sharp radii and reentry corners should be avoided, and that threads and transverse holes have to be produced separately by additional machining operations. *Source:* Metal Powder Industries Federation.

Ceramics

TABLE 11.6

Types and General Characteristics of Ceramics and Glasses

Type	General Characteristics
Oxide Ceramics	
Alumina	High hot hardness and abrasion resistance, moderate strength and toughness; most widely used ceramic; used for cutting tools, abrasives, and electrical and thermal insulation.
Zirconia	High strength and toughness; resistance to thermal shock, wear, and corrosion; partially-stabilized zirconia and transformation-toughened zirconia have better properties; suitable for heat-engine components.
Carbides	
Tungsten carbide	High hardness, strength, toughness, and wear resistance, depending on cobalt binder content; commonly used for dies and cutting tools.
Titanium carbide	Not as tough as tungsten carbide, but has a higher wear resistance; has nickel and molybdenum as the binder; used as cutting tools.
Silicon carbide	High-temperature strength and wear resistance, used for heat engines and as abrasives.
Nitrides	
Cubic boron nitride	Second hardest substance known, after diamond; high resistance to oxidation; used as abrasives and cutting tools.
Titanium nitride	Used as coatings on tools, because of its low frictional characteristics.
Silicon nitride	High resistance to creep and thermal shock; high toughness and hot hardness; used in heat engines.
Sialon	Consists of silicon nitrides and other oxides and carbides; used as cutting tools.
Cermets	Consist of oxides, carbides, and nitrides; high chemical resistance but is somewhat brittle and costly; used in high-temperature applications.
Nanophase ceramics	Stronger and easier to fabricate and machine than conventional ceramics; used in automotive and jet-engine applications.
Silica	High temperature resistance; quartz exhibits piezoelectric effects; silicates containing various oxides are used in high-temperature, nonstructural applications.
Glasses	Contain at least 50% silica; amorphous structure; several types available, with a wide range of mechanical, physical, and optical properties.
Glass ceramics	High crystalline component to their structure; stronger than glass; good thermal-shock resistance; used for cookware, heat exchangers, and electronics.
Graphite	Crystalline form of carbon; high electrical and thermal conductivity; good thermal-shock resistance; also available as fibers, foam, and buckyballs for solid lubrication; used for molds and high-temperature components.
Diamond	Hardest substance known; available as single-crystal or polycrystalline form; used as cutting tools and abrasives and as die insert for fine wire drawing; also used as coatings.

Ref.

S. Kalpakjian, "Manufacturing Processes for Engineering Materials", 3rd/4th ed. Addison Wesley

Ceramic Bonding

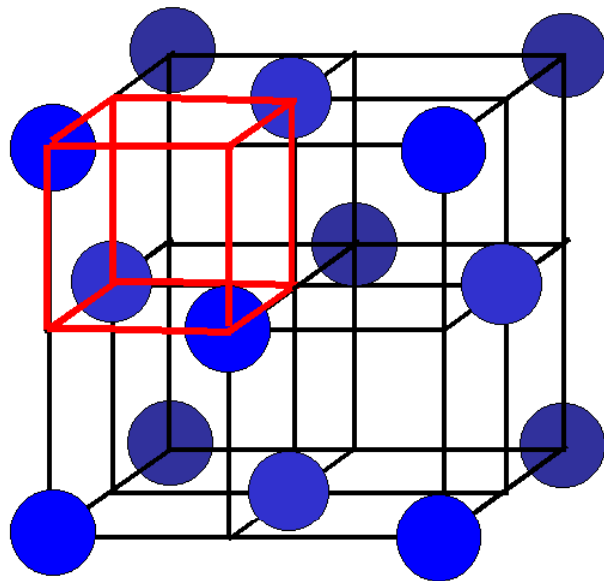
- **Bonding :**
 - Mostly ionic, some covalent.
 - % ionic character increases with difference in electronegativity.
- **Large vs. small ionic bond character :**

IA	IIA												IIIA	IVA	VA	VIA	VIIA	0
H 2.1	Li 1.0	Be 1.5											B 2.0	C 2.5	N 3.0	O 3.5	F 4.0	He -
Na 0.9	Mg 1.2	Ca 1.0	Sc 1.3	Ti 1.5	V 1.6	Cr 1.6	Mn 1.5	Fe 1.8	Co 1.8	Ni 1.8	Cu 1.9	Zn 1.6	Ga 1.6	Ge 1.8	As 2.0	Se 2.4	Br 2.8	Kr -
Rb 0.8	Sr 1.0	Y 1.2	Zr 1.4	Nb 1.6	Mo 1.8	Tc 1.9	Ru 2.2	Rh 2.2	Pd 2.2	Ag 1.9	Cd 1.7	In 1.7	Sn 1.8	Sb 1.9	Te 2.1	I 2.5	Xe -	
Cs 0.7	Ba 0.9	La-Lu 1.1-1.2	Hf 1.3	Ta 1.5	W 1.7	Re 1.9	Os 2.2	Ir 2.2	Pt 2.2	Au 2.4	Hg 1.9	Tl 1.8	Pb 1.8	Bi 1.9	Po 2.0	At 2.2	Rn -	
Fr 0.7	Ra 0.9	Ac-No 1.1-1.7																

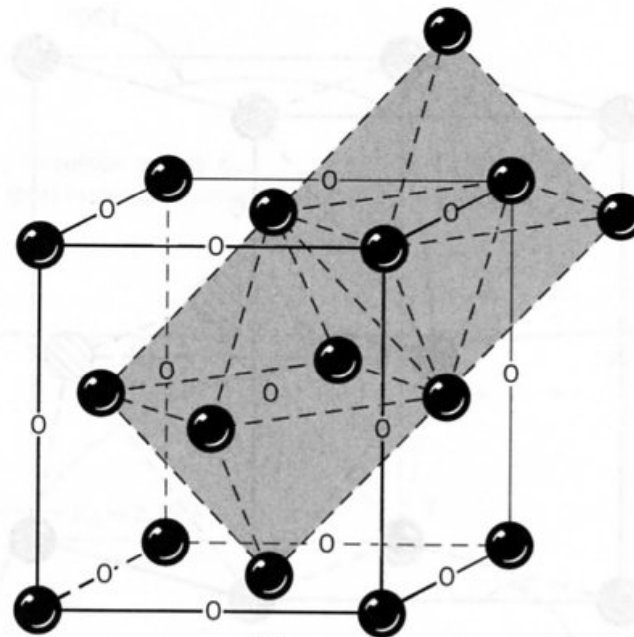
CaF₂: large
SiC: small

Ceramic Crystal Structures

- **Oxide structures**
 - Oxygen anions are much larger than metal cations.
 - Close packed oxygen in a lattice (usually FCC).
 - Cations in the holes of the oxygen lattice.



Tetrahedral site



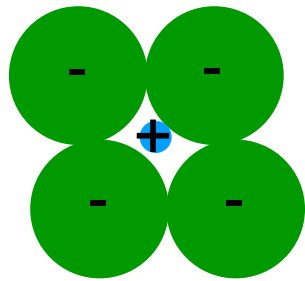
Octahedral site

What determines crystal structure?

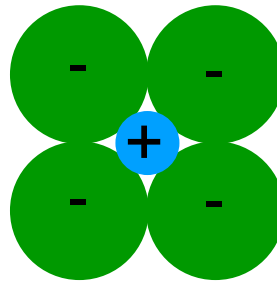
1. **Magnitude of electrical charge** on each of component ions.
 - Crystals must be electrically neutral.
2. **Relative sizes of cations and anions.**
 - Does the cation fit in the site.

Ionic Bonding & Structure

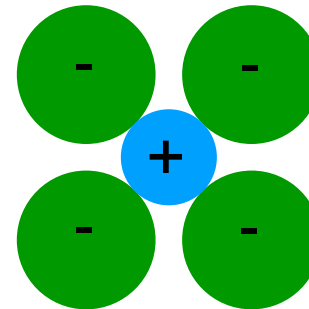
- Size - stable structures :
 - maximize the number of nearest oppositely charged neighbors.



unstable

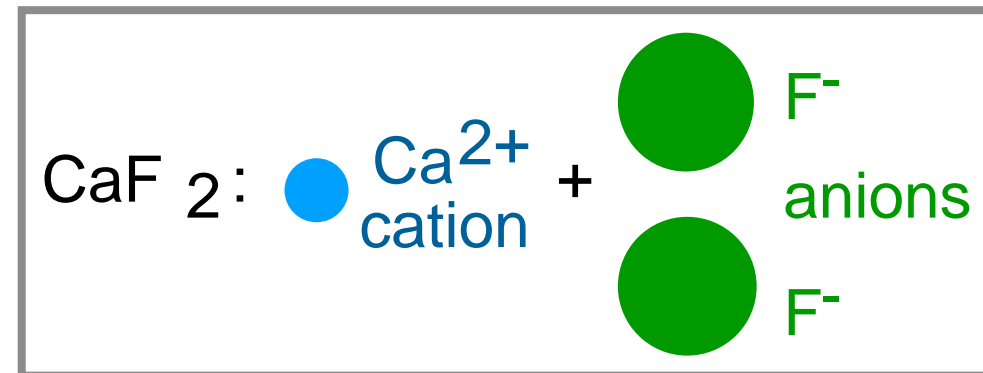


stable



stable

- Charge neutrality :
 - Net charge in the structure should be zero.



- General form : $A_m X_p$


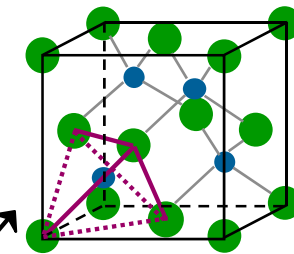

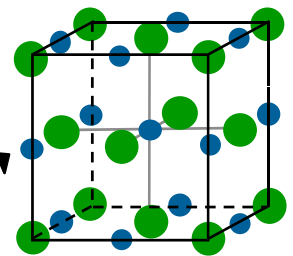

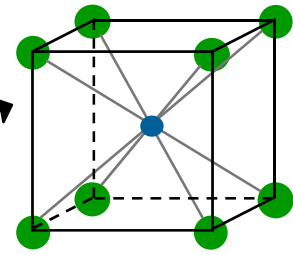
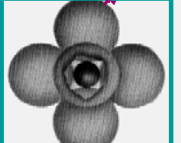
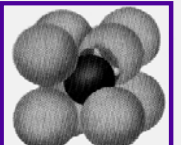


m, p determined by charge neutrality

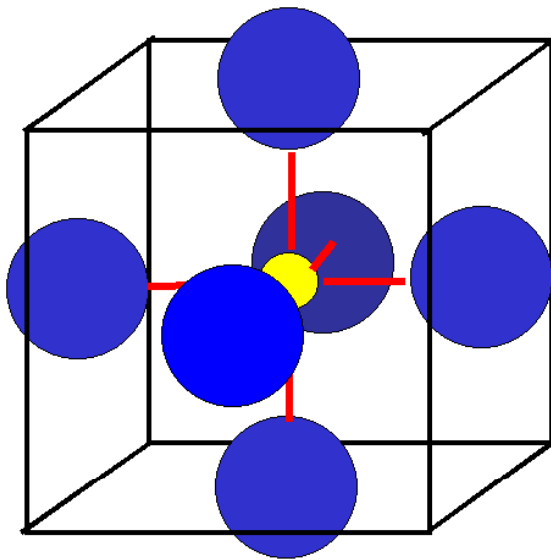
Coordination number and Ionic Radii

- Coordination number increases with $\frac{r_{\text{cation}}}{r_{\text{anion}}}$

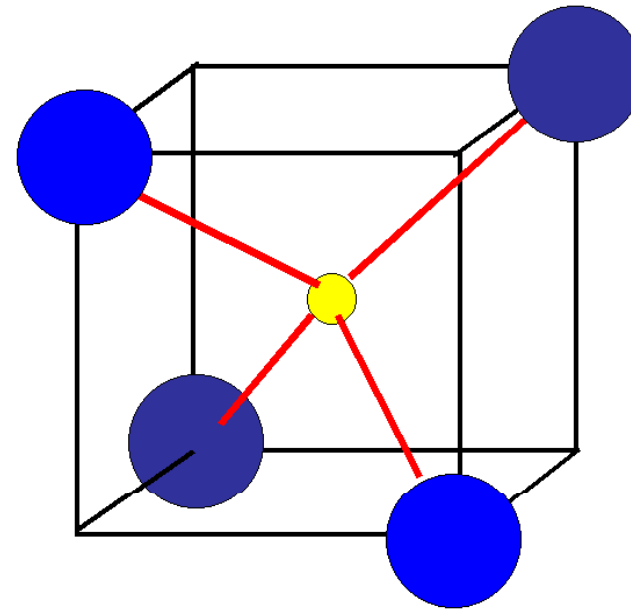
Issue: How many anions can you arrange around a cation?

$\frac{r_{\text{cation}}}{r_{\text{anion}}}$	Coord. #				
< 0.155	2	linear			ZnS (zincblende)
0.155 - 0.225	3	triangular			NaCl (sodium chloride)
0.225 - 0.414	4	T_D			CsCl (cesium chloride)
0.414 - 0.732	6	O_H			
0.732 - 1.0	8	cubic			

Interstitial Sites



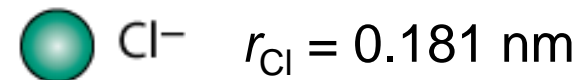
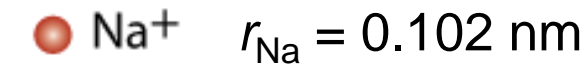
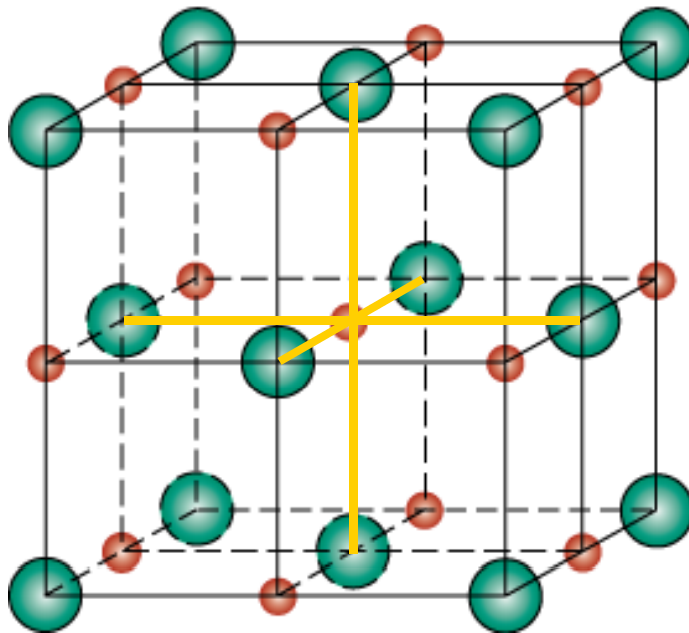
Octahedral sites



Tetrahedral sites

Rock Salt Structure

- Same concepts can be applied to ionic solids in general.
example : NaCl (rock salt) structure



$$r_{\text{Na}}/r_{\text{Cl}} = 0.564$$

∴ cations prefer O_H sites

Effect of Porosity

- Residual Porosity :
Elastic Modulus and Strength \Rightarrow Lower
Elastic Modulus, E

$$E = E_0 (1 - 1.9P + 0.9P^2)$$

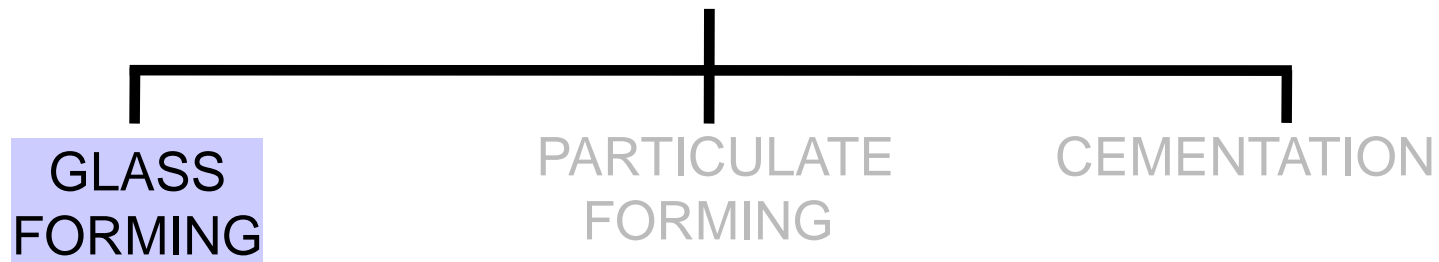
P ; Volume Fraction of Porosity

E_0 ; Elastic Modulus of Material

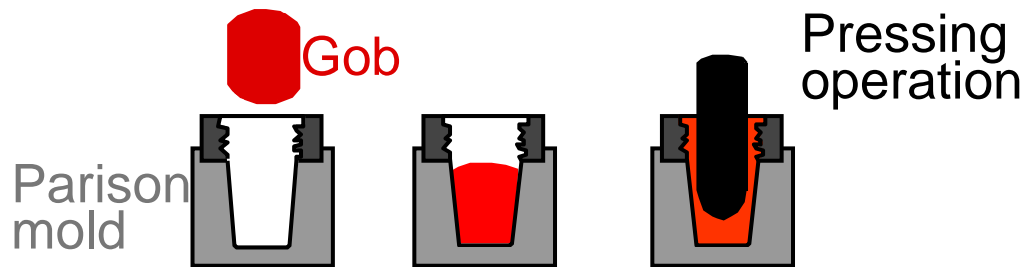
Reasons : strength lowered by porosity.

- (1) Cross-Sectional Area \Rightarrow Reduce
- (2) Stress Concentrators

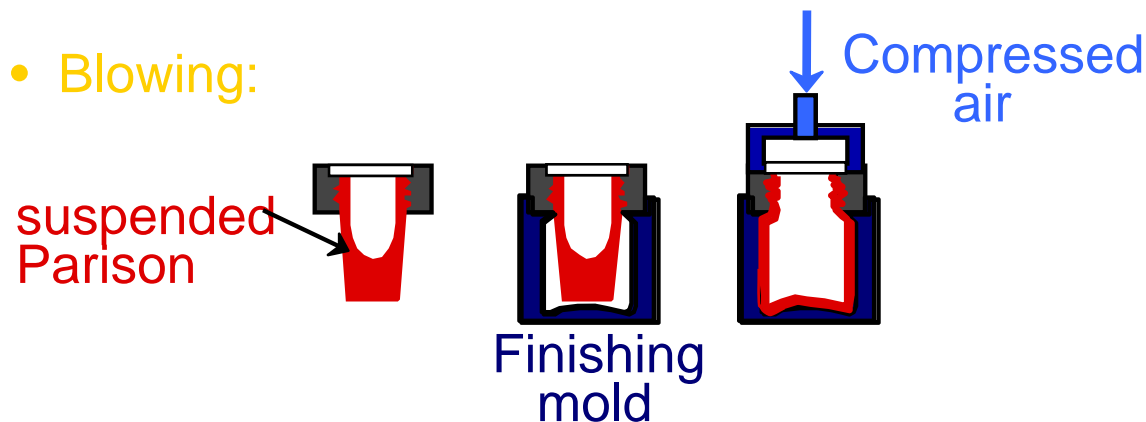
Ceramic Fabrication Methods (1)



- Pressing:

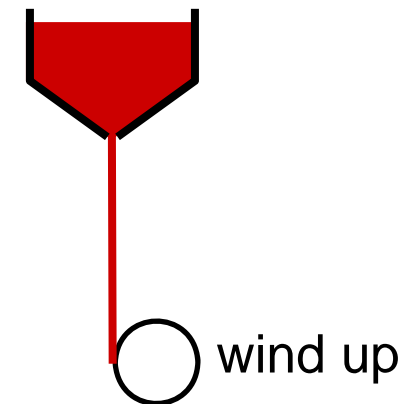


- Blowing:



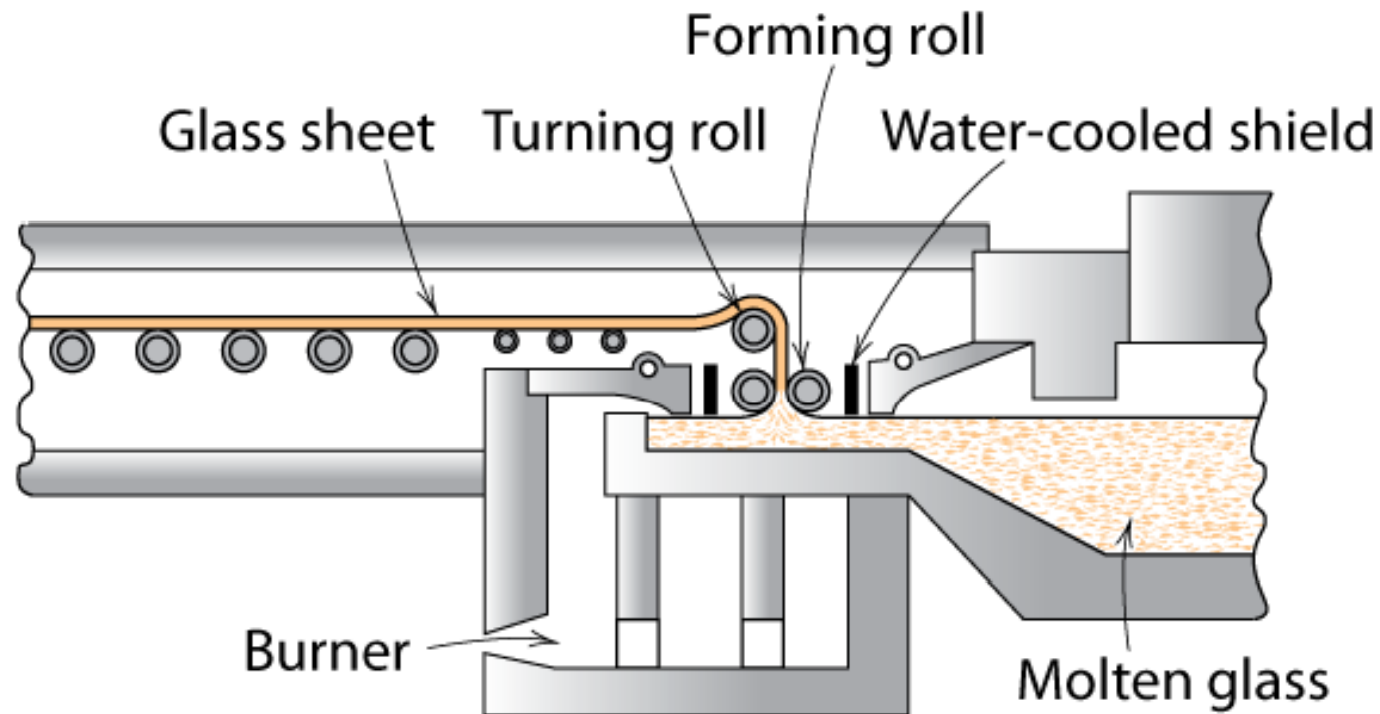
- Fiber drawing:

plates, dishes, cheap glasses
 --mold is steel with graphite lining



Sheet Glass Forming

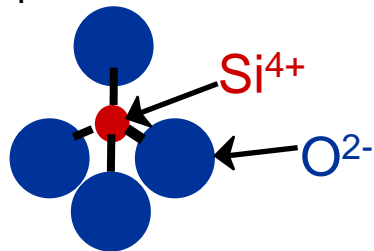
- **Sheet forming – continuous draw**
originally sheet glass was made by “floating” glass on a pool of tin.



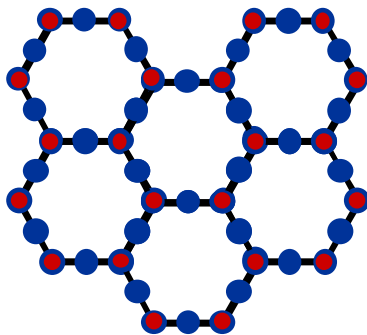
Glass Structure

- Basic Unit:

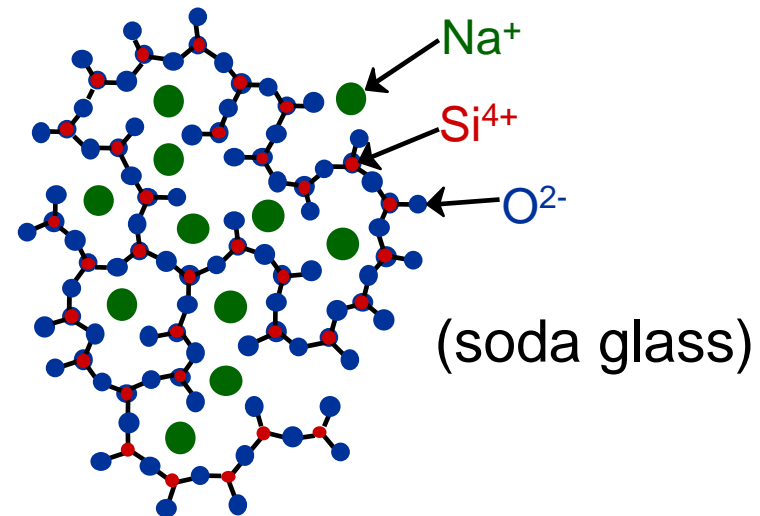
SiO_4^{4-} tetrahedron



- Quartz is **crystalline**.
 SiO_2 :

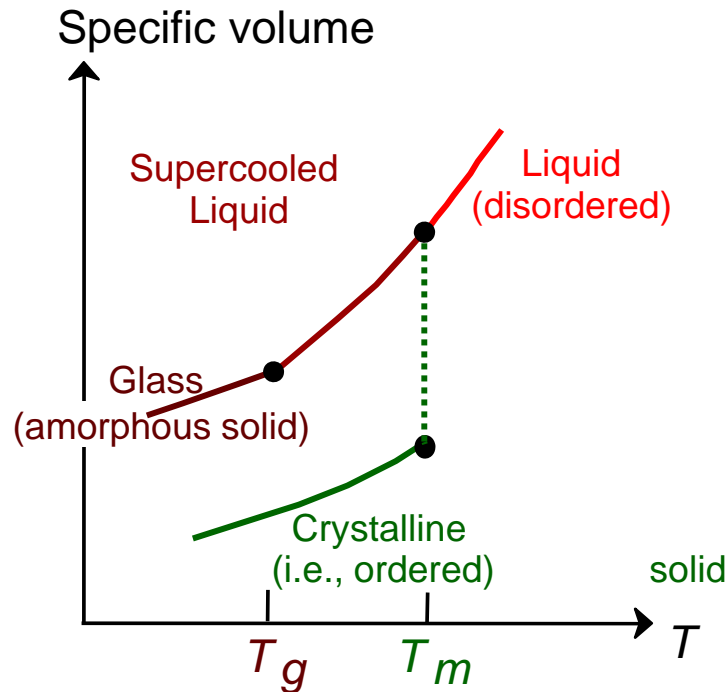


- Glass is **amorphous**.
- Amorphous structure occurs by adding impurities. (Na^+ , Mg^{2+} , Ca^{2+} , Al^{3+})
- Impurities: interfere with formation of crystalline structure.



Glass Properties

- **Specific volume** ($1/\rho$) vs. Temperature (T):



- **Crystalline materials:**
 - crystallize at melting temp, T_m .
 - have abrupt change in spec. vol. at T_m .
- **Glasses:**
 - do not crystallize.
 - change in slope in spec. vol. curve at **glass transition temperature**, T_g .
 - transparent
no crystals to scatter light.

Heat Treating Glass

- **Annealing:**
 - removes internal stress caused by uneven cooling.
- **Tempering:**
 - puts surface of glass part into compression.
 - suppresses growth of cracks from surface scratches.
 - sequence :

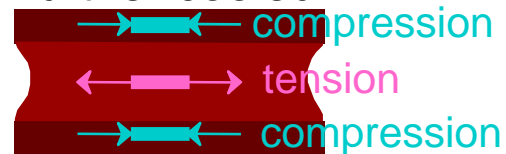
before cooling



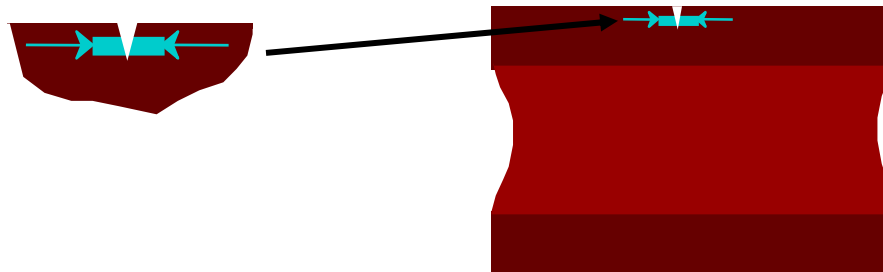
surface cooling



further cooled



--Result: surface crack growth is suppressed.



Ceramic Fabrication Methods (2)

GLASS
FORMING

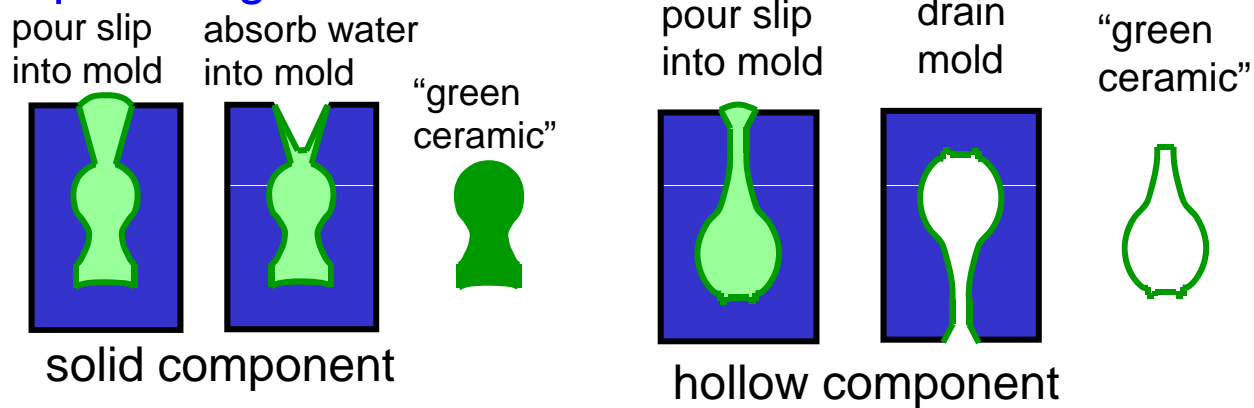
PARTICULATE
FORMING

CEMENTATION

- Milling and screening : desired particle size
- Mixing particles & water : produces a "slip"
- Form a "green" component

- **Hydroplastic forming:**
extrude the slip (e.g., into a pipe)

- **Slip casting:**

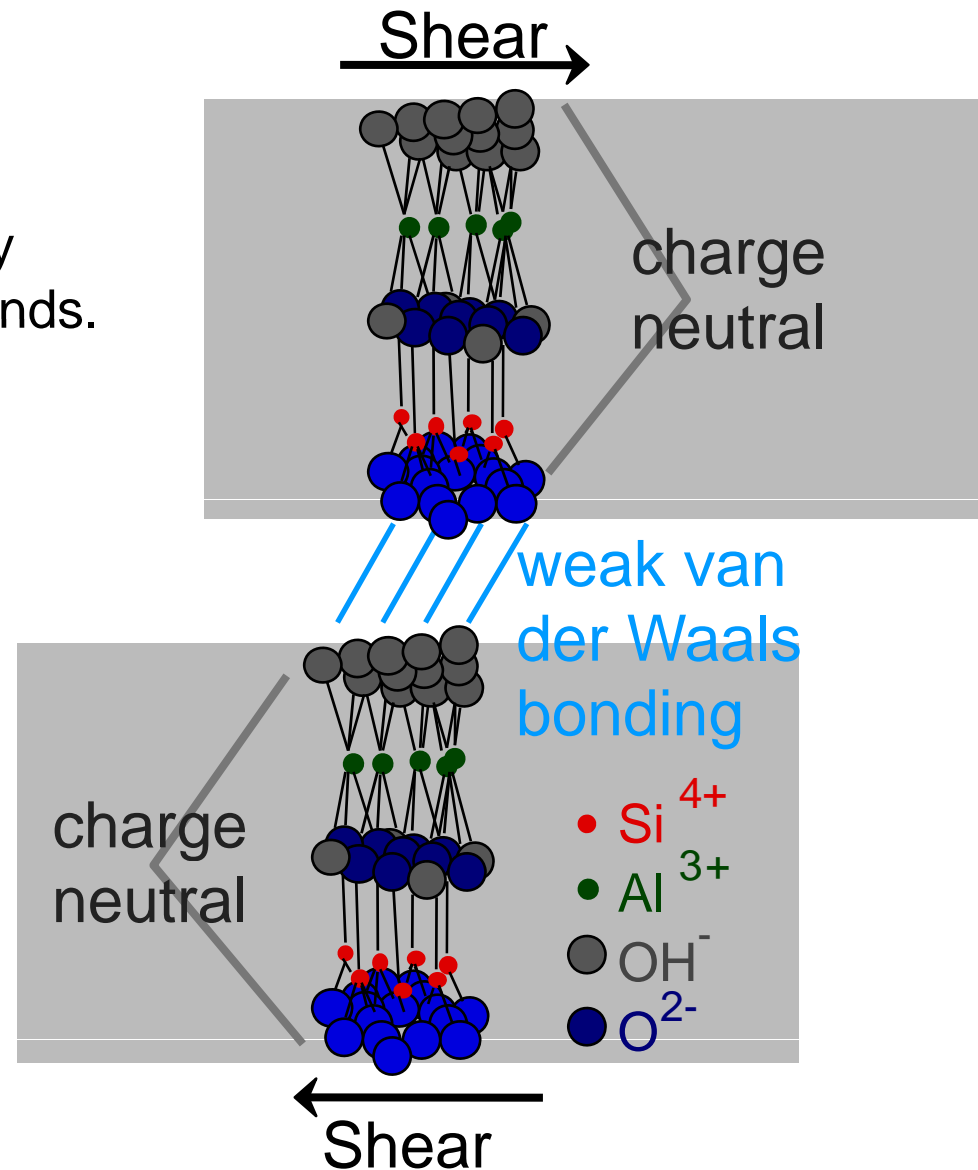


- **Dry** and **fire** the component.

Features of a Slip

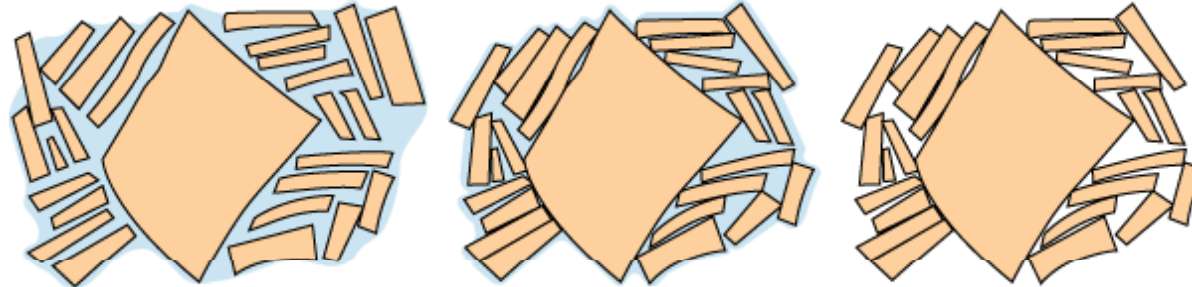
- Clay is inexpensive.
- Adding water to clay
 - allows material to shear easily along weak van der Waals bonds.
 - enables extrusion.
 - enables slip casting.

- Structure of Kaolinite Clay :
(고령토)



Drying and Firing

- **Drying:** layer size and spacing decrease.



wet slip

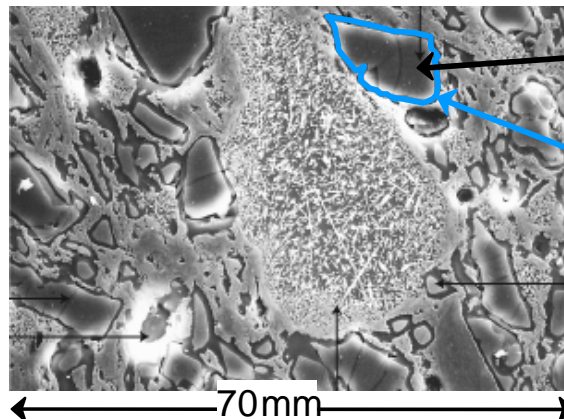
partially dry

“green” ceramic

Drying too fast causes sample to warp or crack due to non-uniform shrinkage.

- **Firing:**
 - T raised to $900\sim 1400^{\circ}\text{C}$
 - **vitriification** : liquid glass forms from clay and flows between SiO_2 particles. Flux melts at lower T .

micrograph of
porcelain



SiO_2 particle
(quartz)

glass formed
around
the particle

70mm

Ceramic Fabrication Methods (3)

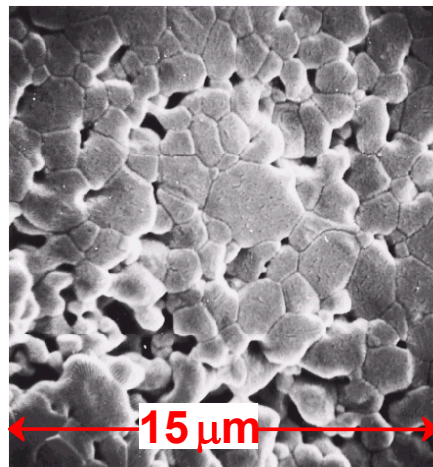
GLASS
FORMING

PARTICULATE
FORMING

CEMENTATION

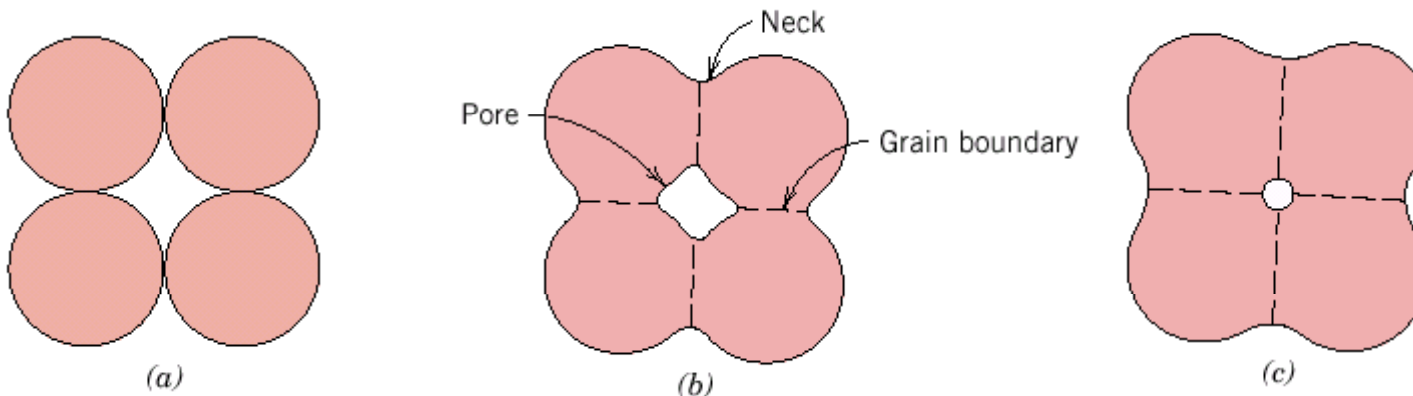
Sintering: useful for both clay and non-clay compositions.

- Procedure:
 - produce ceramic and/or glass particles by grinding.
 - place particles in mold.
 - press at elevated T to reduce pore size.
- Aluminum oxide powder:
 - sintered at 1700°C for 6 minutes.



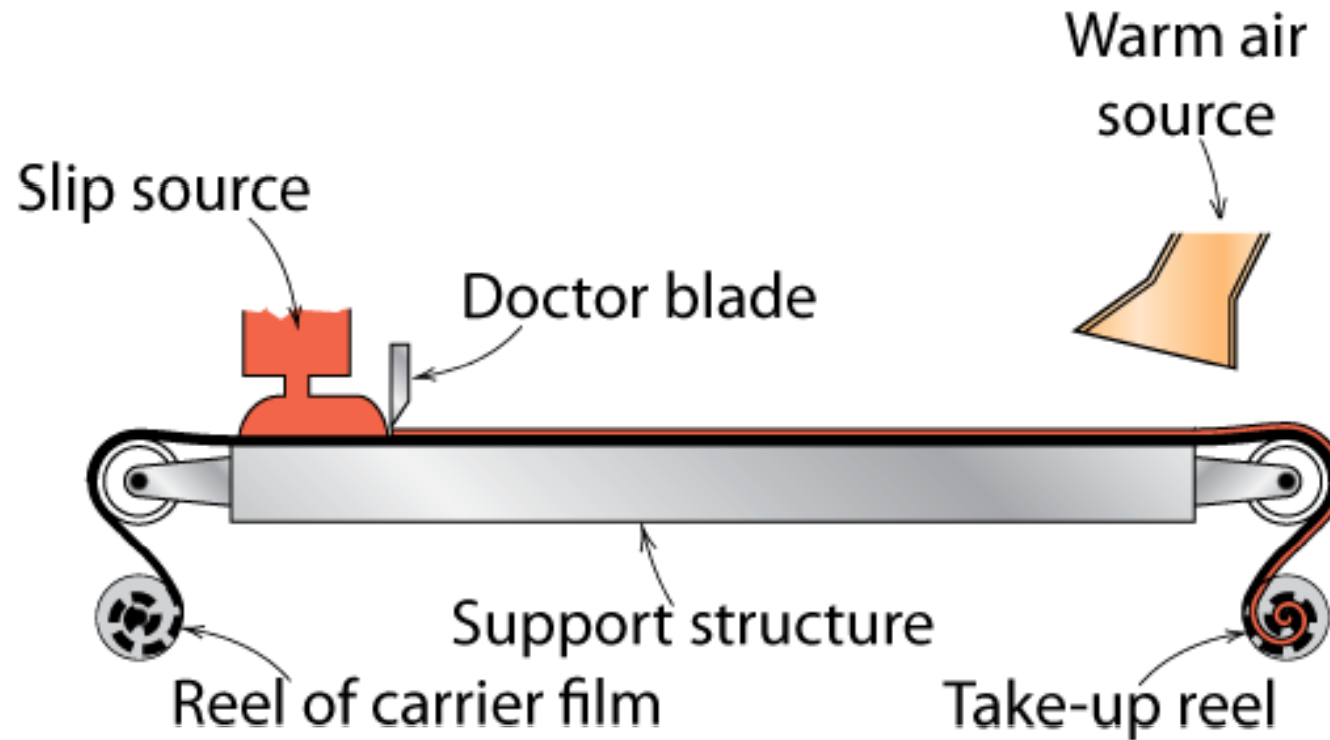
Powder Pressing

- **Sintering** - powder touches - forms neck & gradually neck thickens
 - add processing aids to help form neck
 - little or no plastic deformation
- **Uniaxial compression** - compacted in single direction.
- **Isostatic (hydrostatic) compression** - pressure applied by fluid, powder in rubber envelope.
- **Hot pressing** - pressure + heat



Tape Casting

- Thin sheets of green ceramic cast as flexible tape.
- Used for integrated circuits and capacitors.
- Cast from liquid slip (ceramic + organic solvent).



Ceramic Fabrication Methods (4)

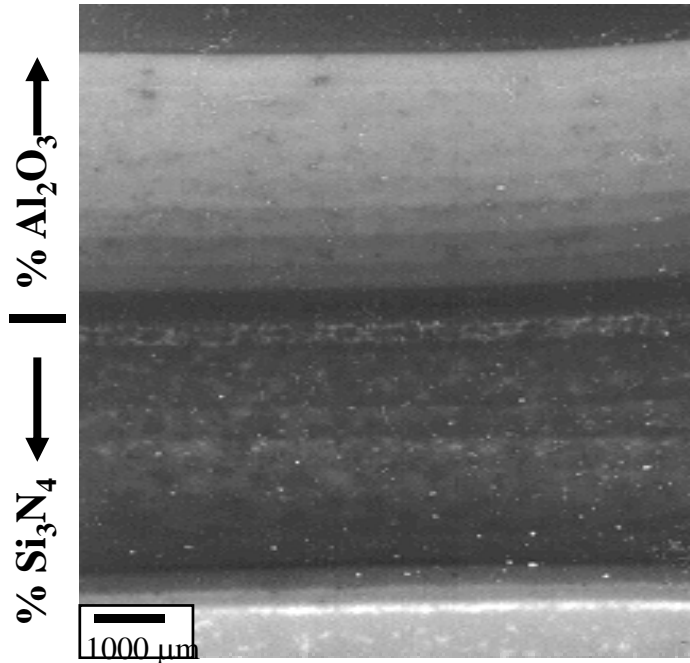
GLASS
FORMING

PARTICULATE
FORMING

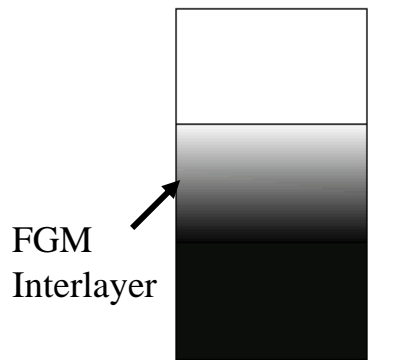
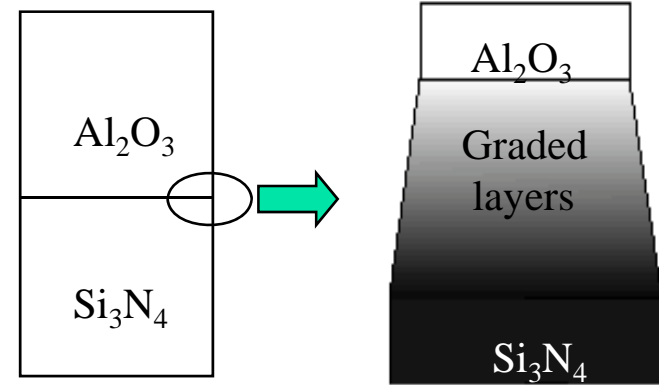
CEMENTATION

- Produced in extremely large quantities.
- Portland cement:
 - mix clay and lime bearing materials
 - calcination (heat to 1400°C)
 - primary constituents:
 - tri-calcium silicate
 - di-calcium silicate
- Adding water
 - produces a paste which hardens
 - hardening occurs due to hydration (chemical reactions with the water).
- Forming: done usually minutes after hydration begins.

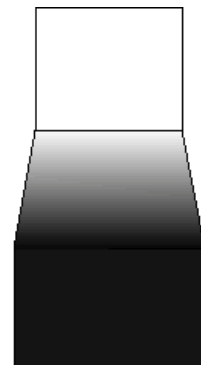
FGM (Functionally Graded Material)



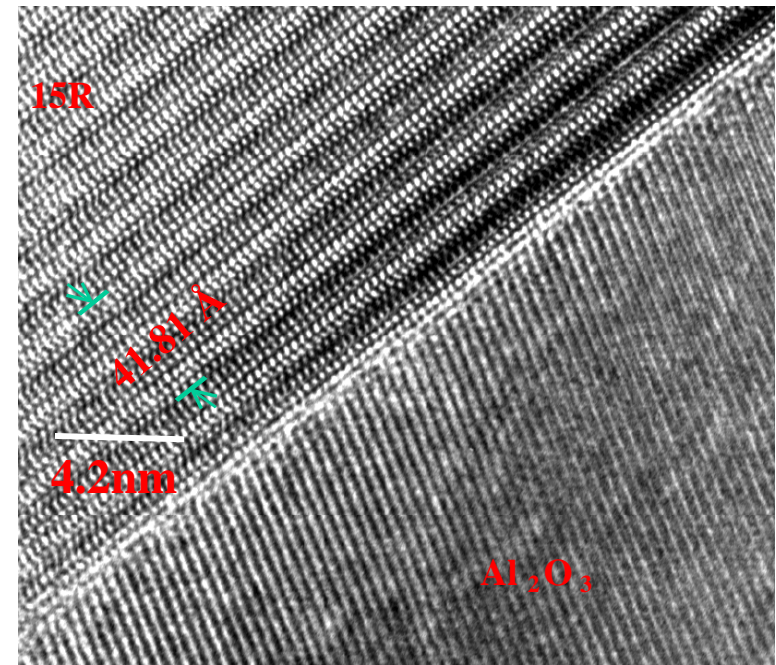
- ← 100% Al₂O₃
- ← 10wt% 12H/90wt% Al₂O₃
- ← 20wt% 12H/80wt% Al₂O₃
- ← 30wt% 12H/70wt% Al₂O₃
- ← 40wt% 12H/60wt% Al₂O₃
- ← 50wt% 12H/50wt% Al₂O₃
- ← 60wt% 12H/40wt% Al₂O₃
- ← 70wt% 12H/30wt% Al₂O₃
- ← 80wt% 12H/20wt% Al₂O₃
- ← 90wt% 12H/10wt% Al₂O₃
- ← 90wt% 12H/10wt% Si₃N₄
- ← 80wt% 12H/20wt% Si₃N₄
- ← 70wt% 12H/30wt% Si₃N₄
- ← 60wt% 12H/40wt% Si₃N₄
- ← 50wt% 12H/50wt% Si₃N₄
- ← 40wt% 12H/60wt% Si₃N₄
- ← 30wt% 12H/70wt% Si₃N₄
- ← 20wt% 12H/80wt% Si₃N₄
- ← 10wt% 12H/90wt% Si₃N₄
- ← 100% Si₃N₄



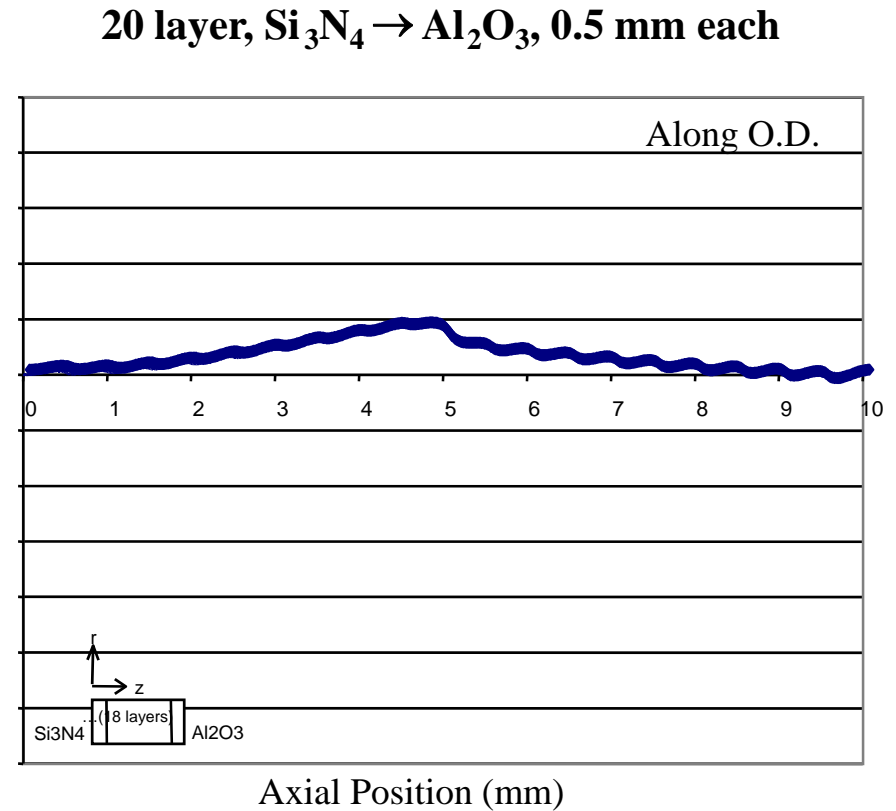
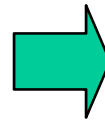
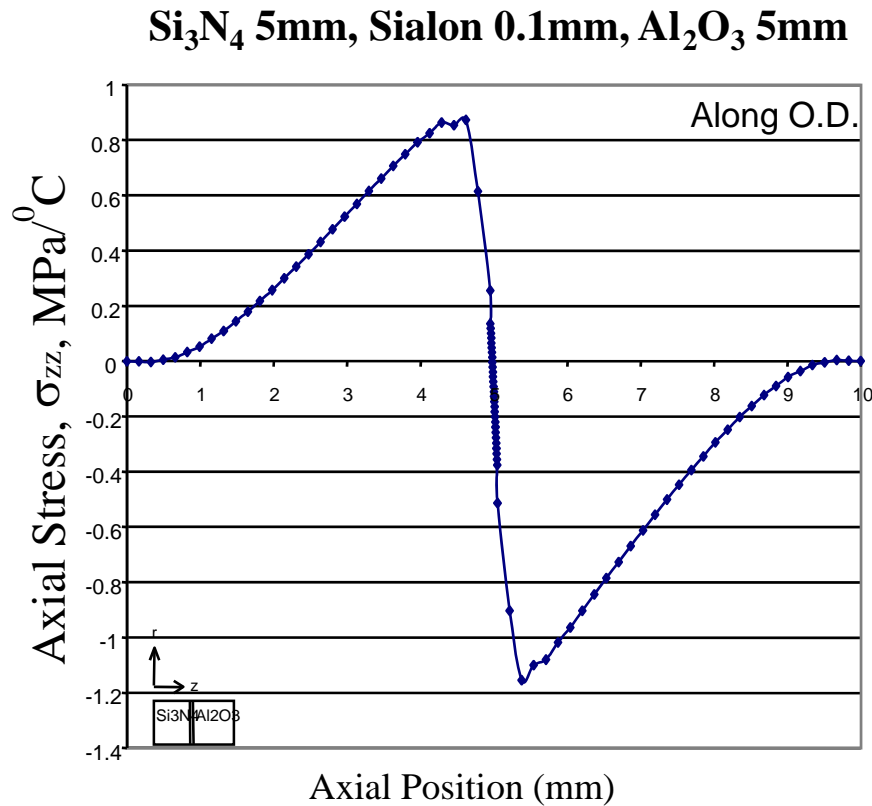
Joining temperature



Room temperature



Residual stresses calculation of FGM joint



- Significant reduction in residual stresses confirms experiment (Lee et al., Acta Mat. (2001))