UNIVERSITY OF TRENTO
Department of Industrial Engineering

Thirteenth World Round Table Conference on Sintering

Tutorial for Young Researchers
Science and Technology of Sintering
Herceg Novi, Montenegro, 2024

Synthesis and Processing of Particulate Materials

Prof. Vincenzo M. Sglavo

vincenzo.sglavo@unitn.it - vincenzo@sglavo.it

University of Trento - Italy

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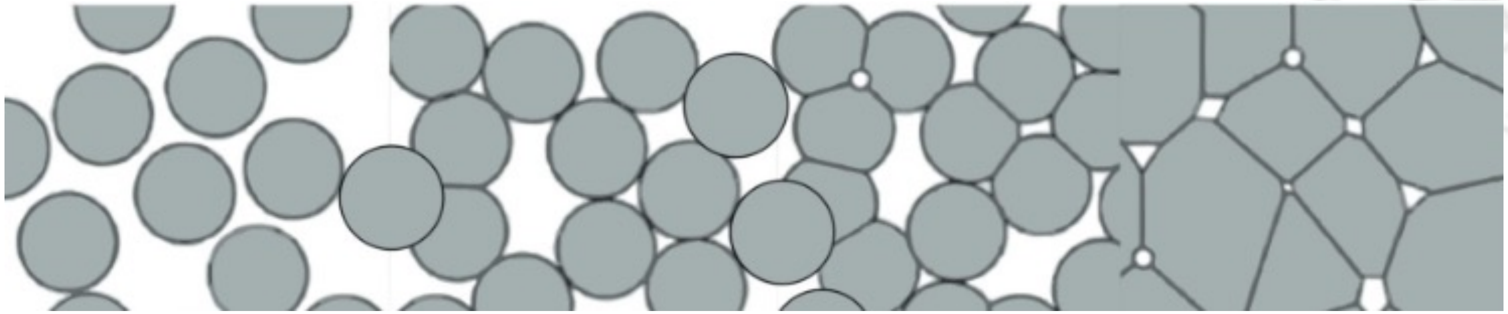
Founded in 1962

- **16.000 students (900 PhD students)**
- **750 professors**
- **750 technicians & administrative staff members**
- **11 departments**
- **4 research centers**

powder ⟷ **sintering**



Processing technologies for ceramic materials

- **from powders** →
  - **sintering**
  - reaction bonding ($Si_{(s)} + NH_{3(g)} \rightarrow Si_3N_{4(s)} + Si_{(s)}$)
  - thermal spray (→ thick coatings)

  Advantage:
  mass production
  Problems:

- from melt →
  - high temperatures (Al_2O_3: 2050°C; ZrO_2: 2700°C)
  - high volumetric shrinkage upon solidification (≈20-25%)
  - large crystalline grains (→ limited mechanical resistance)

- from vapour/gas (*CVD, sputtering*) →
- from solution (*sol-gel*) →
- from polymers →

  Advantage:
  high purity
  Problems:
  limited productivity (→ thin coatings, powders, microcomponents, fibers)

Powder metallurgy

Near net-shape production of metallic components

Advantages:
- *Uniform microstructure and distribution of alloying elements*
- *No machining*
- *Flexibility in component design*

Disadvantages
- *Porosity*
- *Fatigue and fracture resistance*

Outline

Starting from powders: ceramic and metallic powders
- *synthesis processes, challenges and issues*
- *powders from natural sources and wastes*

Colloidal suspensions and pastes for ceramic production
- *deflocculated systems: additives and stability, micro- and nano-powders*
- *viscous and plastic behaviour*

Conventional shaping methods for powder metallurgy and ceramics; features, shapes and technological issues
- *pressing or die-compaction; granulation*
- *extrusion*
- *slip and tape casting*
- *injection moulding*

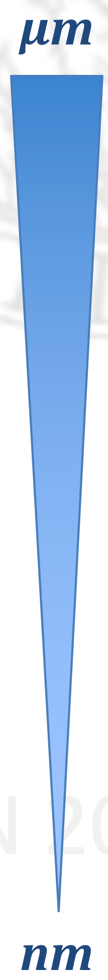
Innovative forming methods for ceramics and metals: additive manufacturing
- *powder bed fusion, directed energy deposition, binder jetting, direct inkjet printing, fused filament deposition, stereolithography and two-photon polymerization*

Drying and presintering processes

Innovative sintering processes

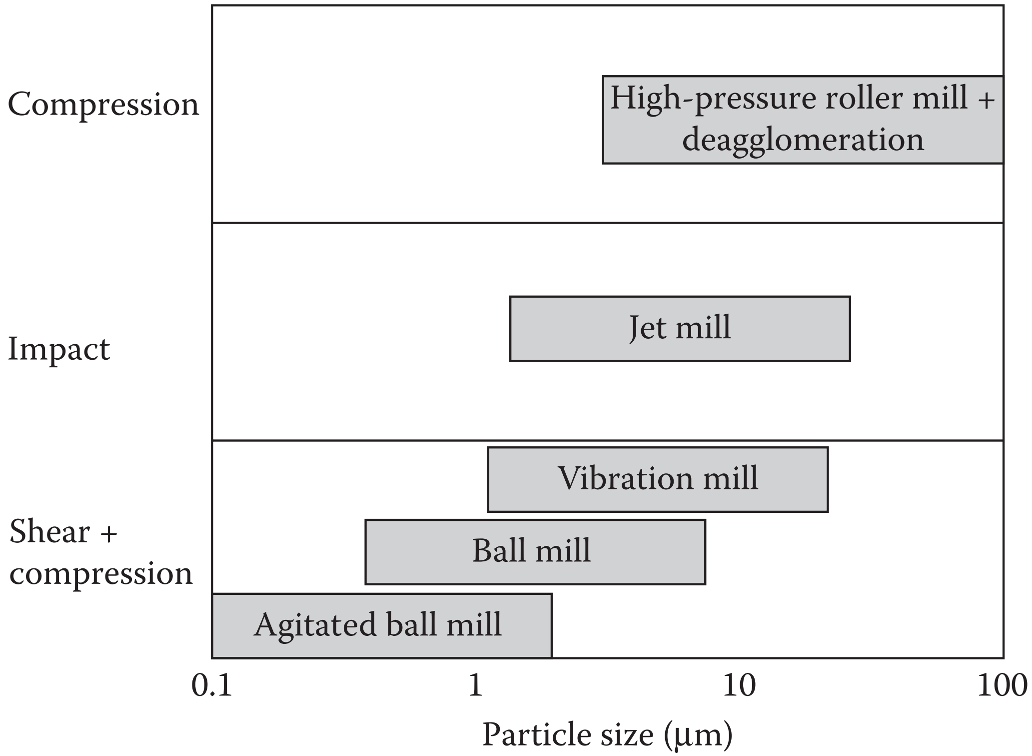
Powder preparation methods for ceramics

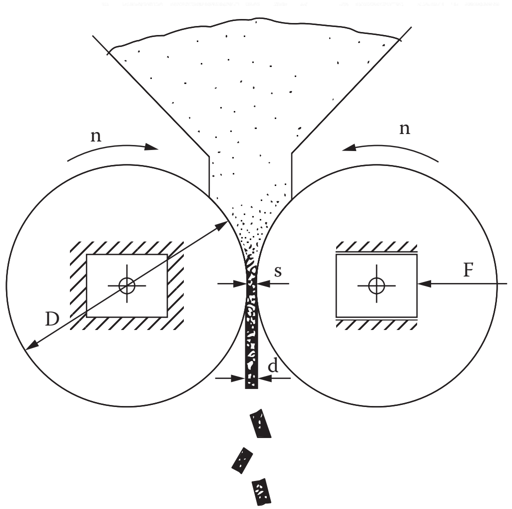
Powder Preparation Method	Advantages	Disadvantages
	Mechanical	
Comminution	Inexpensive; wide applicability	Limited purity; limited homogeneity; large particle size
High-energy ball milling	Fine particle size; good for nonoxides; low temperature route	Limited purity; limited homogeneity
	Chemical	
Solid-state reaction; decomposition; reaction between solids	Simple equipment; inexpensive	Agglomerated powder; limited homogeneity for multicomponent powders
	Liquid solutions	
Precipitation; coprecipitation; solvent vaporization (spray drying; spray pyrolysis; freeze drying)	High purity; small particle size; composition and particle size control	Expensive; limited applicability to nonoxides
Gel routes (sol–gel; Pechini; citrate gel; glycine nitrate)	High purity; good composition control; chemical homogeneity	Agglomerated powder; limited control of particle size
Nonaqueous liquid reaction	High purity; small particle size	Limited to nonoxides
	Vapor-phase reaction	
Gas–solid reaction	Inexpensive for large particle size	Low purity; expensive for fine powders
Gas–liquid reaction	High purity; small particle size	Expensive; limited applicability
Reaction between gases	High purity; small particle size; inexpensive for oxides	Expensive for nonoxides; agglomeration often a problem



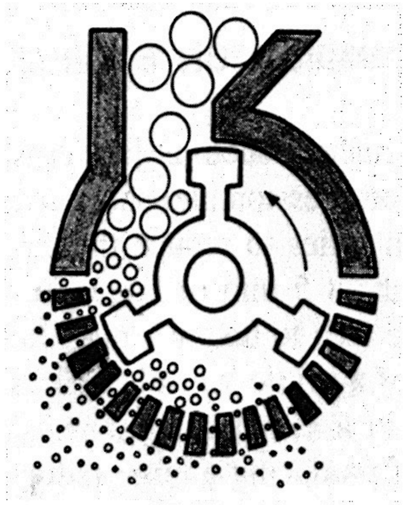
Milling or Comminution

Mechanical stresses among particles and sizes

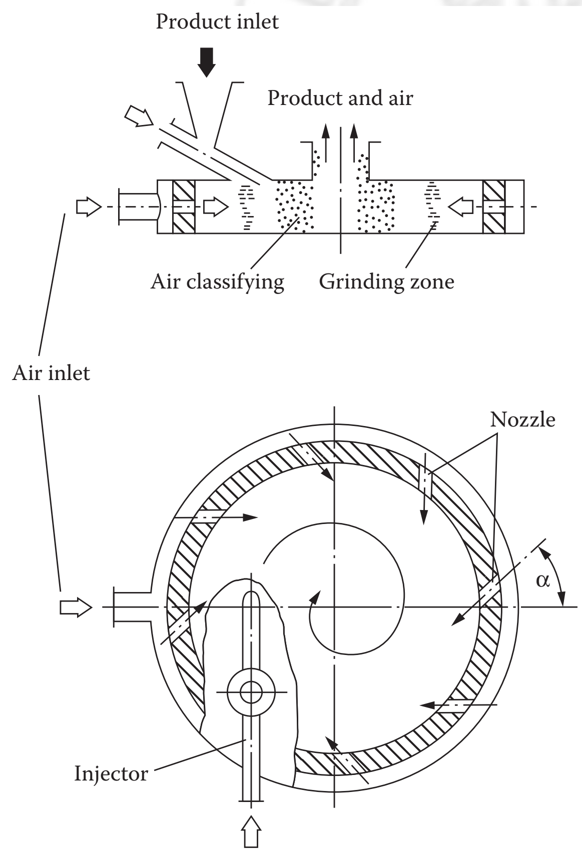




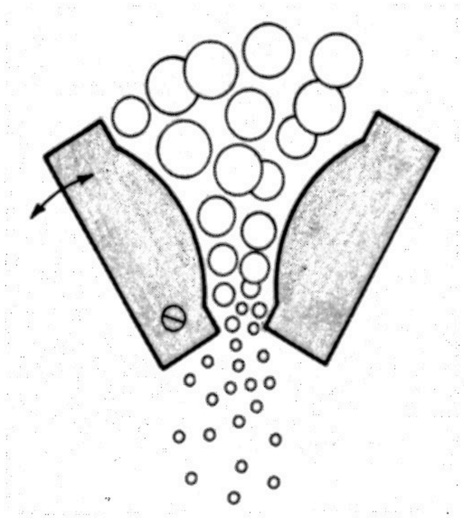
Crushing Rollers



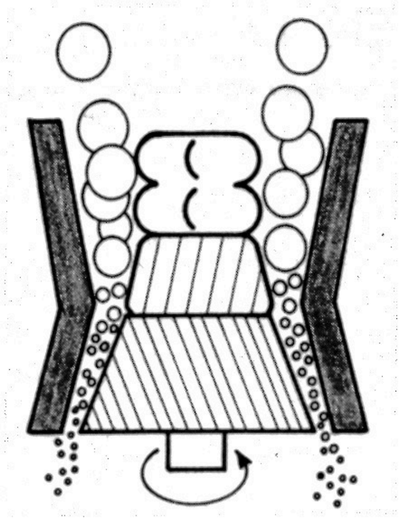
Hammermill



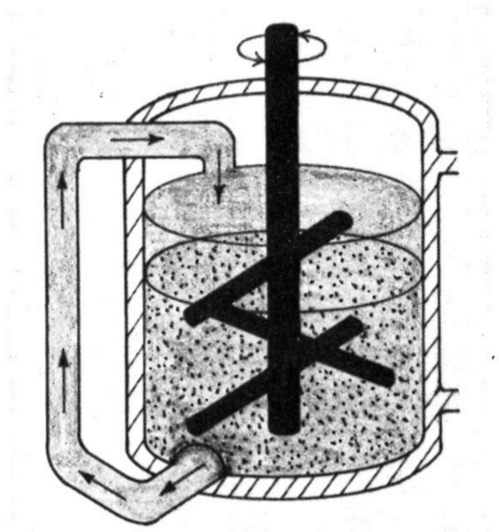
Jet Mill



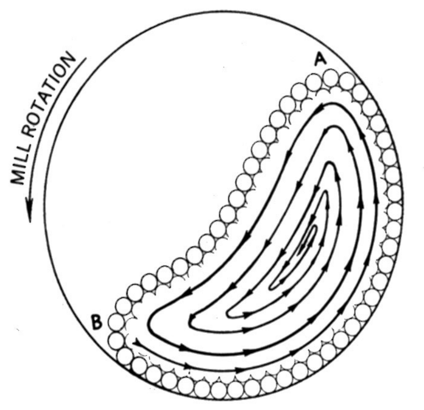
Jaw Crusher



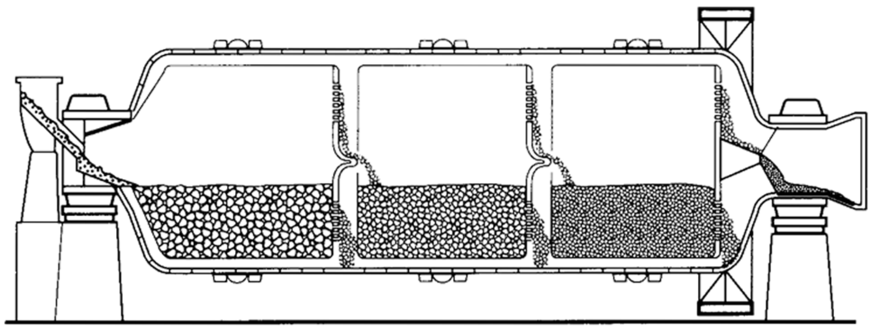
Rotary Crusher



Attrition Mill

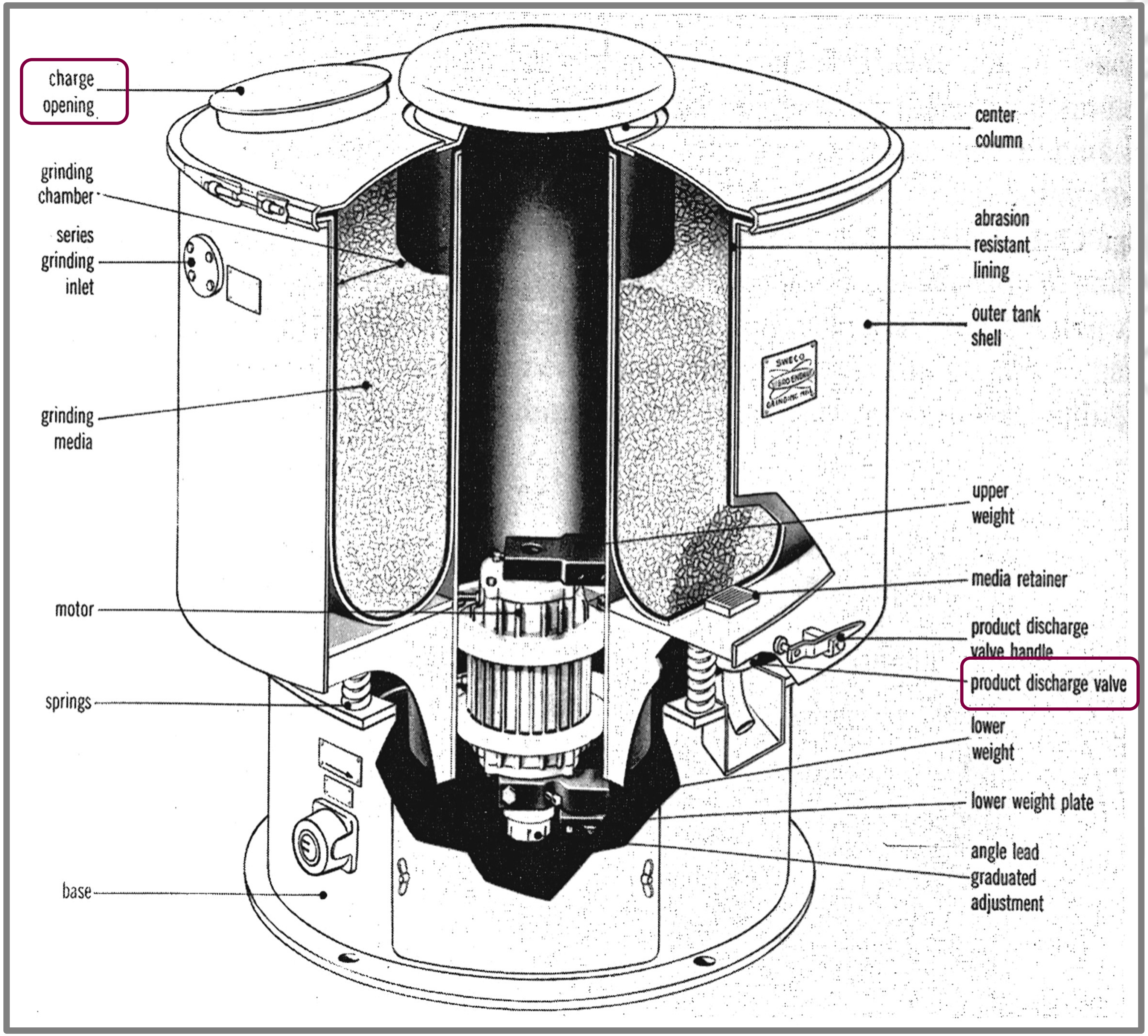


Ball Mill



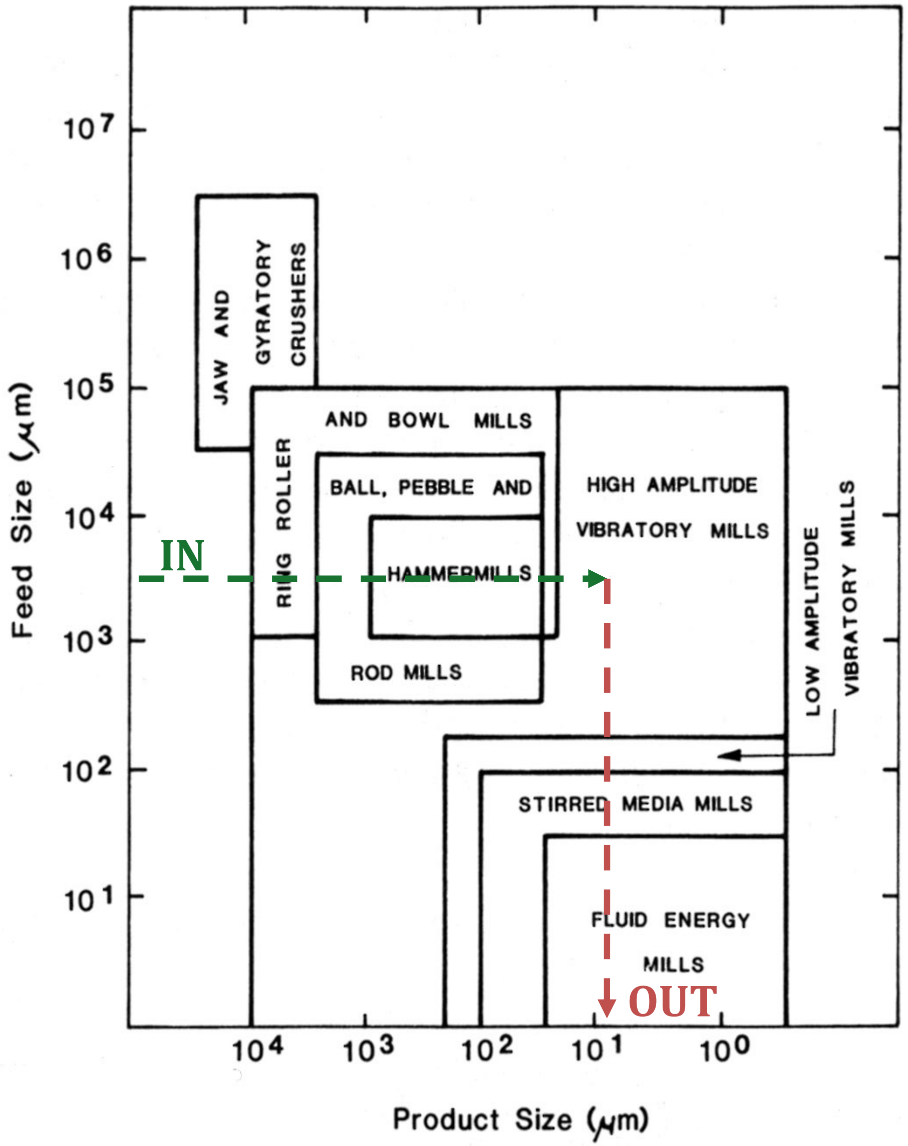
Commercially Available Grinding Media for Ball Milling

Grinding Media	Density (g/cm³)
Porcelain	2.3
Silicon nitride	3.1
Silicon carbide	3.1
Alumina	
Lower than 95% purity	3.4–3.6
Higher than 99% purity	3.9
Zirconia	
MgO stabilized	5.5
High-purity Y_2O_3 stabilized	6.0
Steel	7.7
Tungsten carbide	14.5



Vibratory Mill

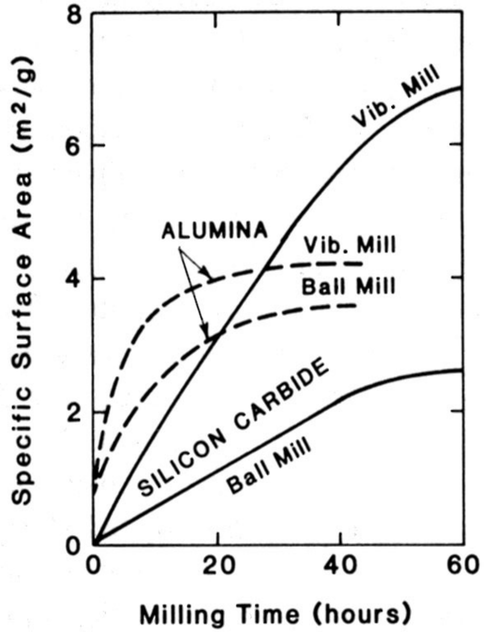
Comparison among systems

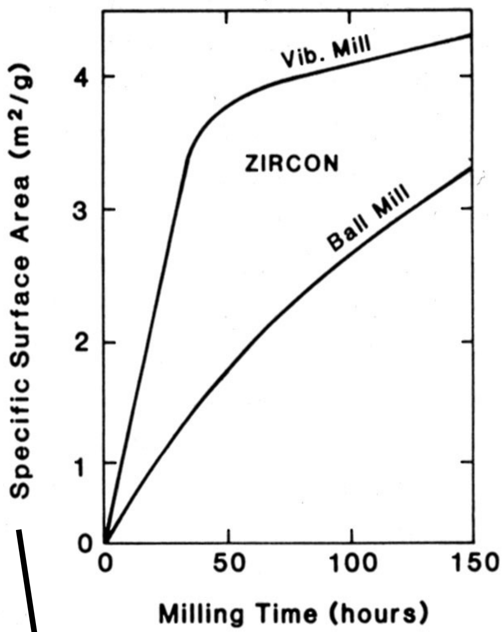


Jaw crushers	to 5 mm
Cone crushers	to 5 mm
Crushing rolls	to ~1 mm
Hammer mill	to ~0.1 mm
Jet mill	1 to ~50 μm
Vibratory mill	1 to ~50 μm
Ball mill	0.5–10 μm
Attrition mill	0.1–5 μm
Roller mill	0.1–5 μm

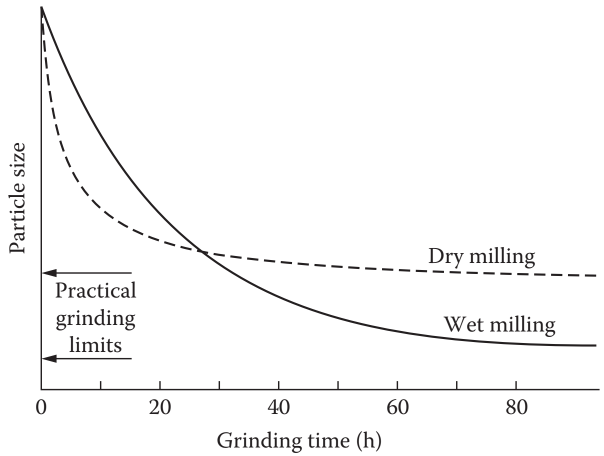
"Optimal milling time"

for discontinous processes





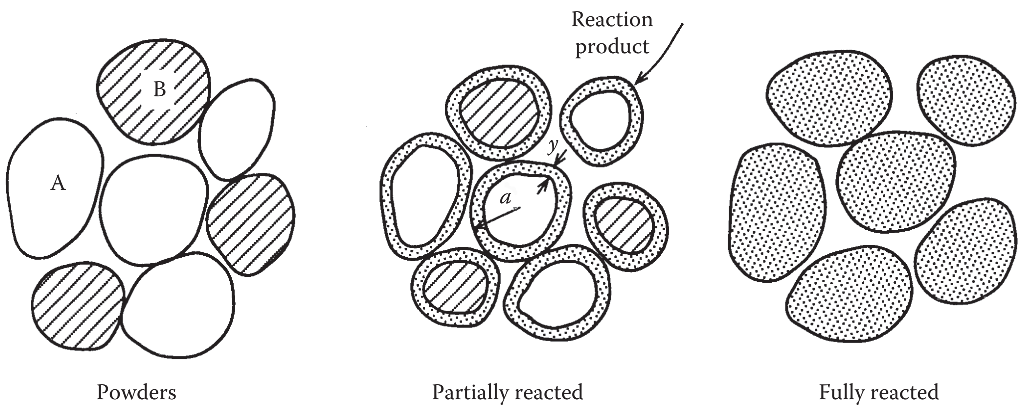
1/grain size



Chemical methods

- **Solid state reactions**
  coarse powders (above 10 µm)

$$SiO_2 + 3C \rightarrow SiC + 2CO$$ *Acheson process (2200-2500°C)*

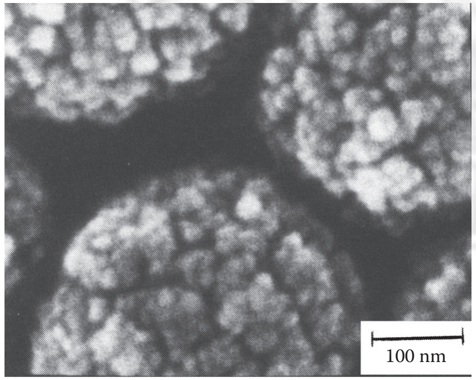


Issues:
- Homogeneity and size of the starting mix
- Presence of unreacted reagents
- Temperature
- Agglomeration (grinding)

- **Precipitation from solutions** *(by nucleation and growth)*

hydrolysis of solutions of metal-organic compounds (see sol-gel) or metal salts

$$Ti(OC_2H_5)_4 + (2+x)H_2O \rightleftarrows TiO_2.xH_2O + 4C_2H_5OH$$ *Stober process*



coprecipitation of complex oxides

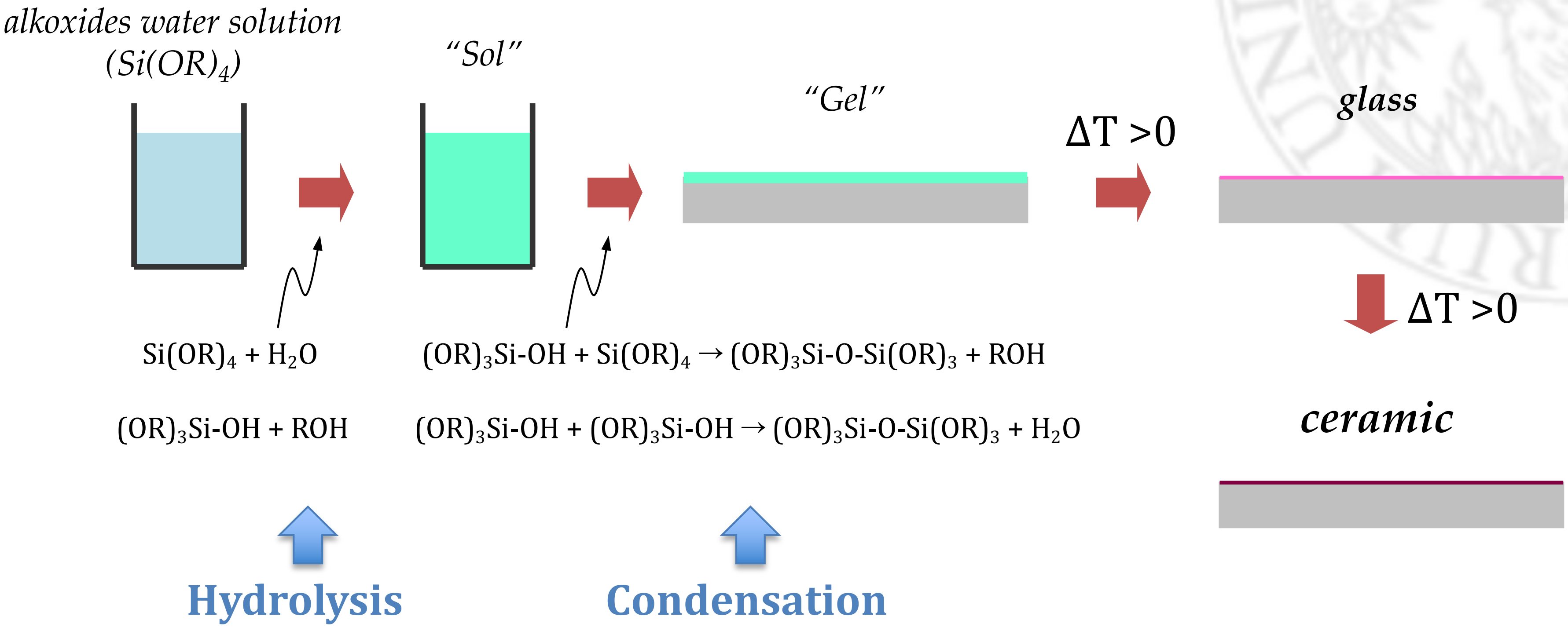
$$Ba(OC_3H_7)_2 + Ti(OC_5H_{11})_4 + 3H_2O \rightarrow BaTiO_3 + 4C_5H_{11}OH + 2C_3H_7OH$$

precipitation under hydrothermal conditions (100-374°C, 220 bar)

$$TiO_2 + Ba(OH)_2 \rightarrow BaTiO_3 + H_2O$$

Issues:
- Aggregation or agglomeration
- Particles growth by Ostwald ripening

Sol-gel



alkoxides water solution
($Si(OR)_4$)
"Sol"
"Gel"
$\Delta T > 0$
glass
$Si(OR)_4 + H_2O$
$(OR)_3Si\text{-}OH + ROH$
$(OR)_3Si\text{-}OH + Si(OR)_4 \rightarrow (OR)_3Si\text{-}O\text{-}Si(OR)_3 + ROH$
$(OR)_3Si\text{-}OH + (OR)_3Si\text{-}OH \rightarrow (OR)_3Si\text{-}O\text{-}Si(OR)_3 + H_2O$
$\Delta T > 0$
ceramic
Hydrolysis
Condensation

Typical alkoxides

Name	Chemical formula	Physical state
Aluminum *s*-butoxide	$Al(O^sC_4H_9)_3$	Colorless liq., T_B ~203°C
Aluminum ethoxide	$Al(OC_2H_5)_3$	White powder, T_M 130°C
Aluminum isopropoxide	$Al[O^iC_3H_7]_3$	White powder, T_M 118.5°C
Antimony ethoxide	$Sb(OC_2H_5)_3$	Colorless liq., T_B 95°C
Barium isopropoxide	$Ba(O^iC_3H_7)_2$	Off-white powder
Boron ethoxide	$B(OC_2H_5)_3$	Colorless liq., T_B 117.4°C
Calcium methoxide	$Ca(OCH_3)_2$	Off-white powder
Iron ethoxide	$Fe(OC_2H_5)_3$	T_M 120°C
Iron isopropoxide	$Fe(O^iC_3H_7)_3$	Brown powder
Silicon tetraethoxide	$Si(OC_2H_5)_4$	Colorless liq., T_B 165.8°C
Silicon tetraheptoxide	$Si(OC_7H_{15})_4$	Yellow liq.
Silicon tetrahexoxide	$Si(OC_6H_{13})_4$	Colorless liq.
Silicon tetramethoxide	$Si(OCH_3)_4$	Colorless liq., T_B 121–122°C
Titanium ethoxide	$Ti(OC_2H_5)_4$	Colorless liq., T_B 122°C
Titanium isopropoxide	$Ti[O^iC_3H_7]_4$	Colorless liq., T_B 58°C
Yttrium isopropoxide	$Y[O^iC_3H_7]_3$	Yellowish-brown liq.

CH_3CH_2OH ethanol

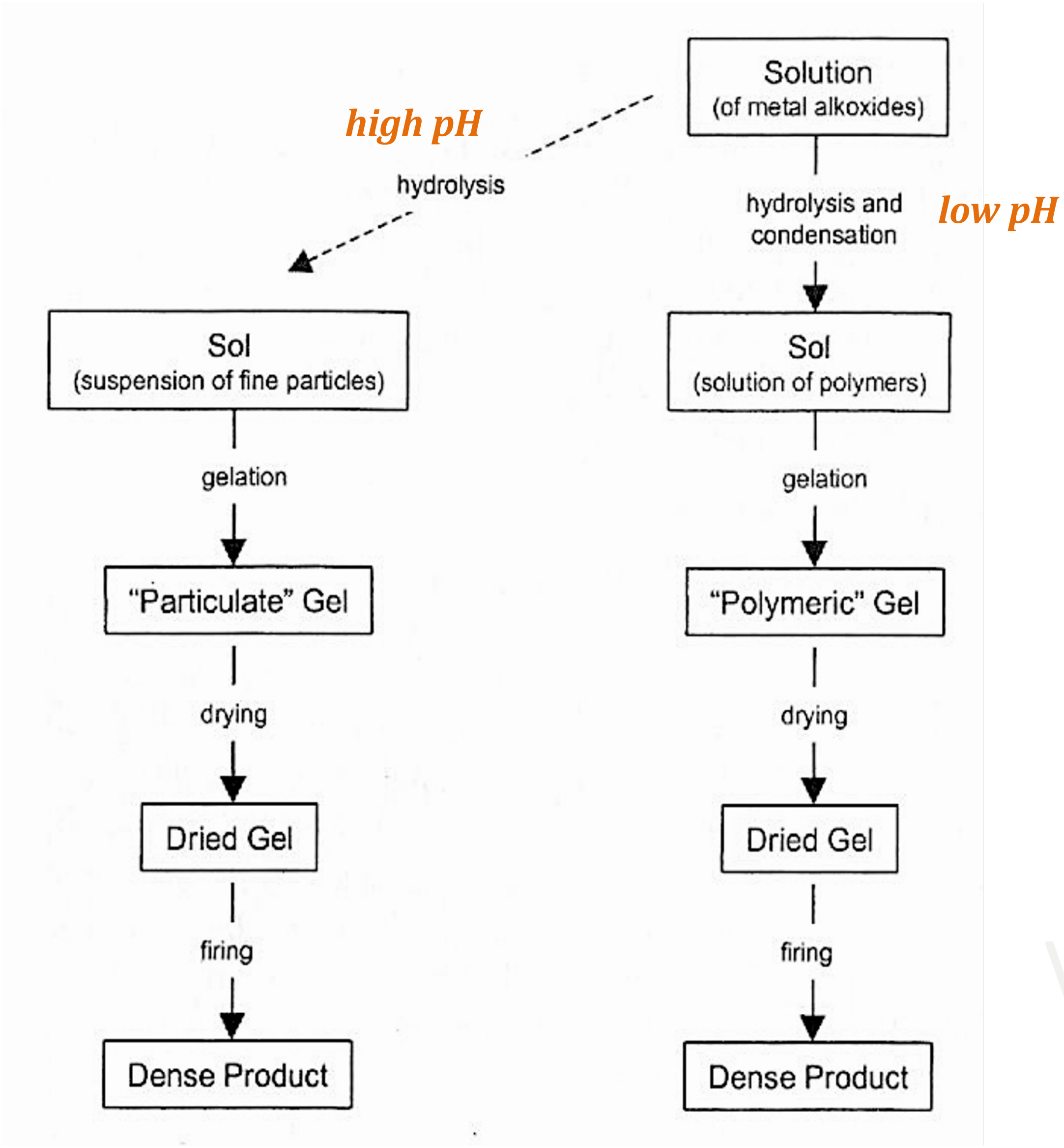
$CH_3CH_2O - Si - (OCH_2CH_3)_3$
silicon tetraethoxide

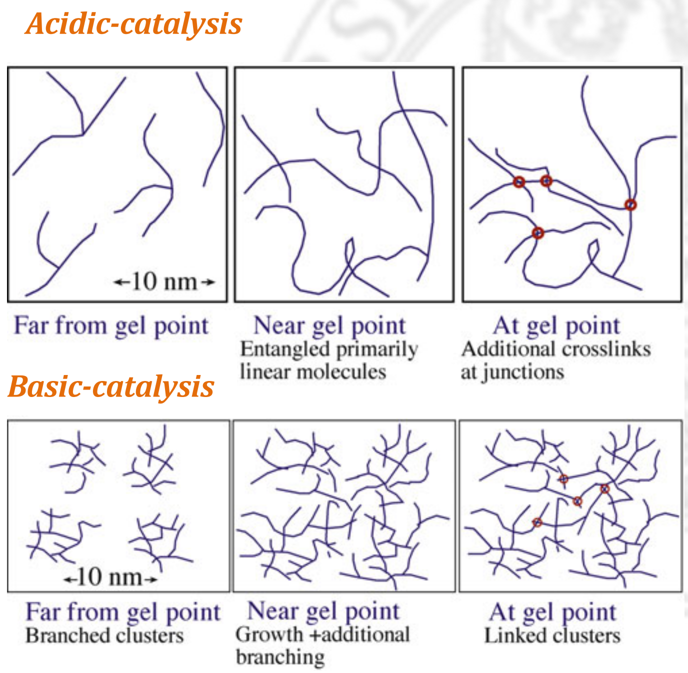
Examples of solution formulations

	One component: 100 mol% SiO_2				Two components: 94 mol% SiO_2 + 6 mol % TiO_2				Three components: 15 mol% Li_2O + 3 mol% Al_2O_3 + 82 mol% SiO_2			
	Soln. conc.	Oxide content			Soln. conc.	Oxide content			Soln. conc.	Oxide		
Precursor	wt%/100 g	vol%/ 100 mL	mol	g/mol	wt%/100 g	vol%/ 100 mL	mol	g/mol	wt%/100 g	vol%/ 100 mL	mol	g/mol
$Si(OC_2H_5)_4$	45	43			11	11			35	34		
$Ti(OC_3H_7)_4$					1	1						
$Al(OC_4H_9)_3$									1	1		
$LiNO_3$									3	1		
C_2H_5OH	40	43	4		36	41	14		29	34	4	
H_2O	16	14	4		52	47	50		33	30	8	
Oxide (Si + Ti + Al + Li)			1	11.3			1	3.15			1	10.6

Ceramic materials: science and engineering., C.B. Carter & M.G. Norton, Springer, 2007

Process scheme





Alumina produced by the Bayer process

digestion:

enriched and milled bauxite (≈2 mm) (Al_2O_3, Fe, Si, Ti oxides, etc.) + $NaOH_{aq}$ *(150-160°C, 5 bar)*

$$\Downarrow$$

$Al(OH)_4^-$ (aq) + Na^+ (aq) + solid (impurities, SiO_2, TiO_2, Fe_2O_3) – "*red mud*"

filtration: solution of $Al(OH)_4^-$ (aq) + Na^+ (aq)

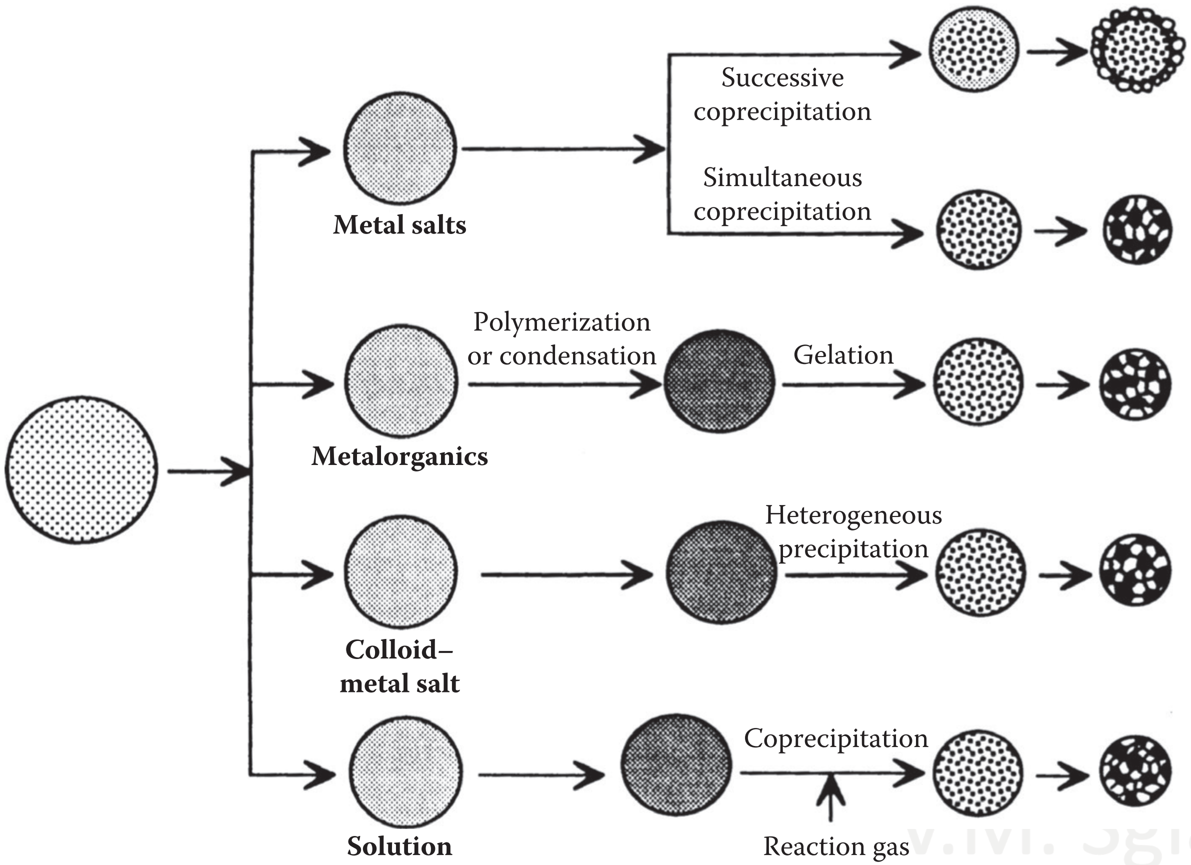
washing (resolubilization)
removal of Na

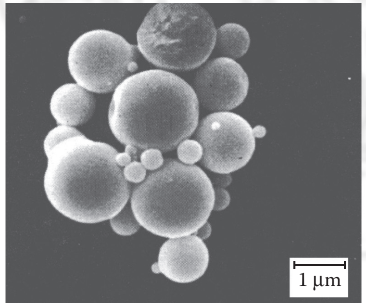
precipitation: *solution cooling* → α-$Al(OH)_3$ solid *(0.1 – 25 μm)*

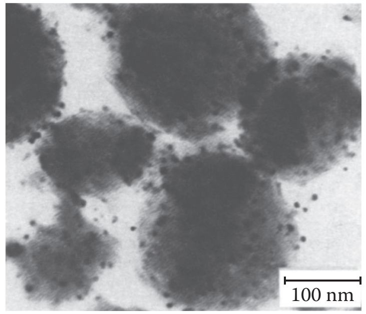
calcination: *treatment at 1100-1200°C* → Al_2O_3 – calcined alumina - *(0.5% Na_2O)*

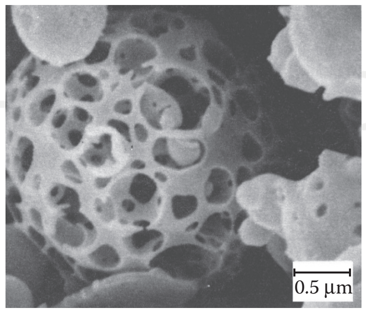
milling

- **Spray pyrolysis**
  solution (see also sol-gel) sprayed in hot and oxidizing atmosphere









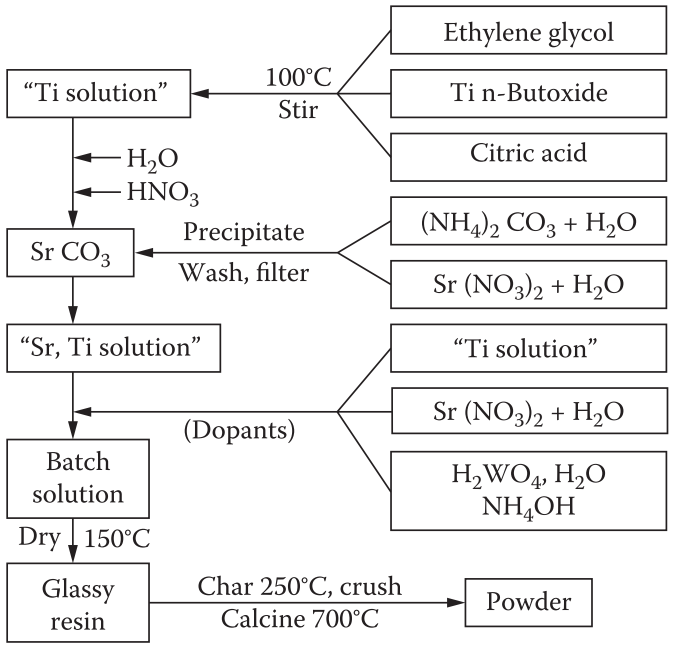
- **Gel routes**
  starting from a gel or viscous resin

  - sol-gel (the gel is dried and crashed)

  - Pechini method (polyesterification induced by ethylene glycol addition)

  - citrate method

  - glycine method

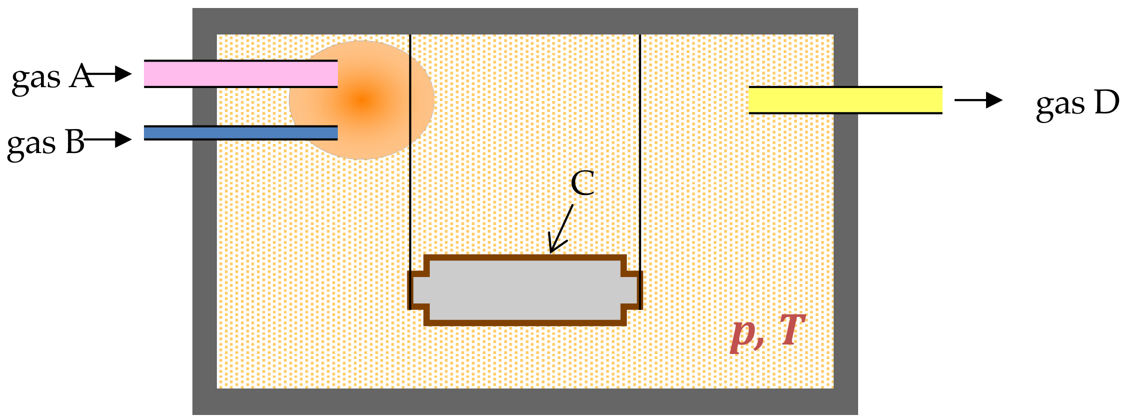


$SrTiO_3$ by Pechini method

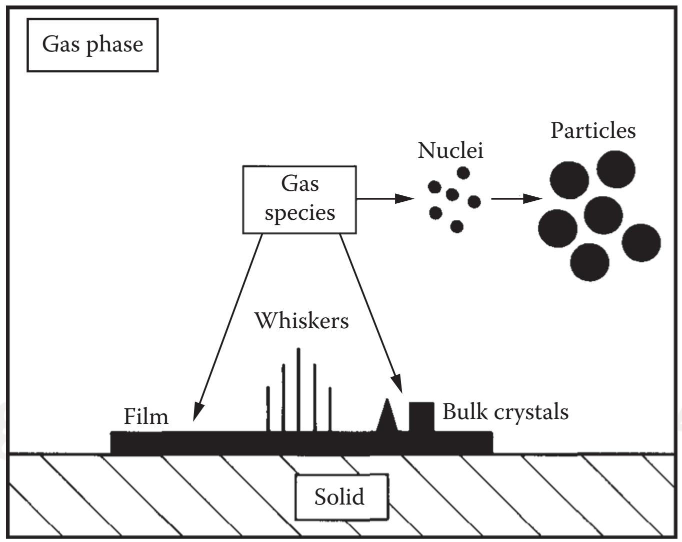
- **Vapour phase reactions**
  (see CVD – chemical vapour deposition)

$TiCl_4(g) + 2H_2O(g) \rightarrow TiO_2(s) + 4HCl(g)$

$3SiCl_4(g) + 4NH_3(g) \rightarrow Si_3N_4(s) + 12HCl(g)$



Steps:
1. reaction between A & B (gas) ➔ C (solid) & D (gas)
2. condensation of C (on the substrate) – *by nucleation & growth*



Fundamental reaction:
$aA(g) + bB(g) = cC(s) + dD(g)$

$K \approx (p_D)^d / (p_A)^a (p_B)^b$

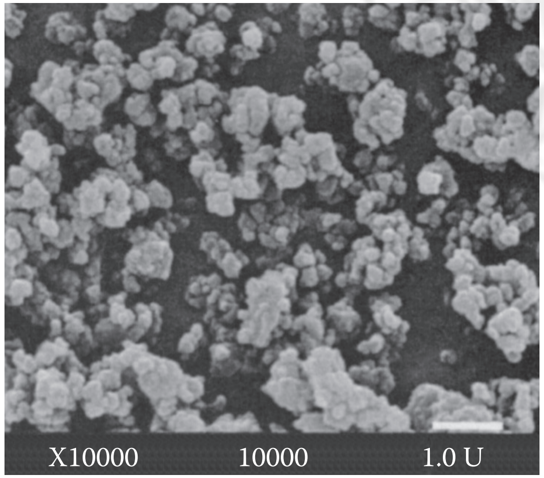
$\Delta G_0 = - R T \ln K$

- **Gas – solid reaction**

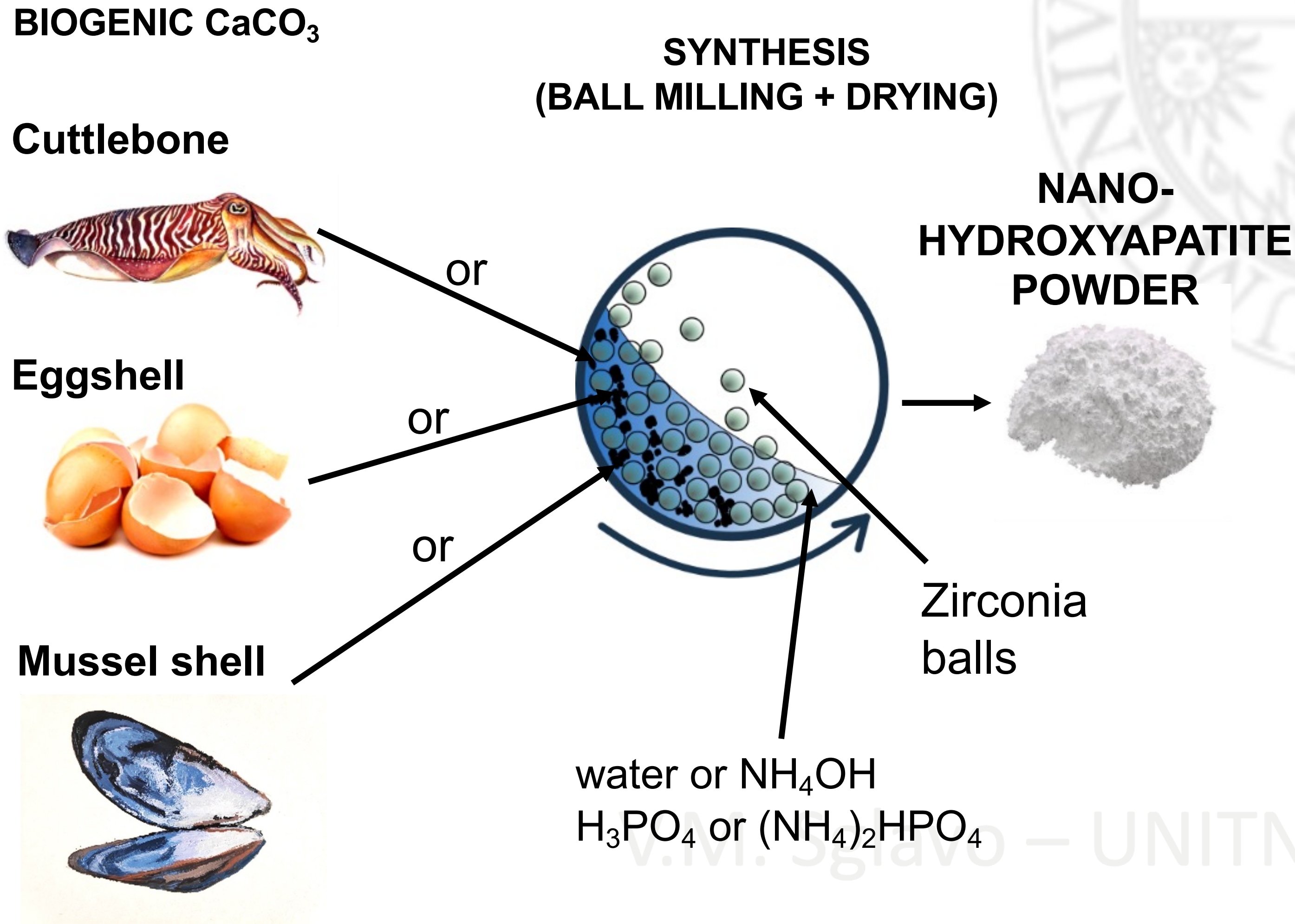
  - *nitrididation for Si_3N_4 (from Si powder)*

  - *carbothermal reduction*

$$3SiO_2 + 6C + 2N_2 \rightarrow Si_3N_4 + 6CO$$



Powder from natural sources or wastes



Powder preparation methods for metals

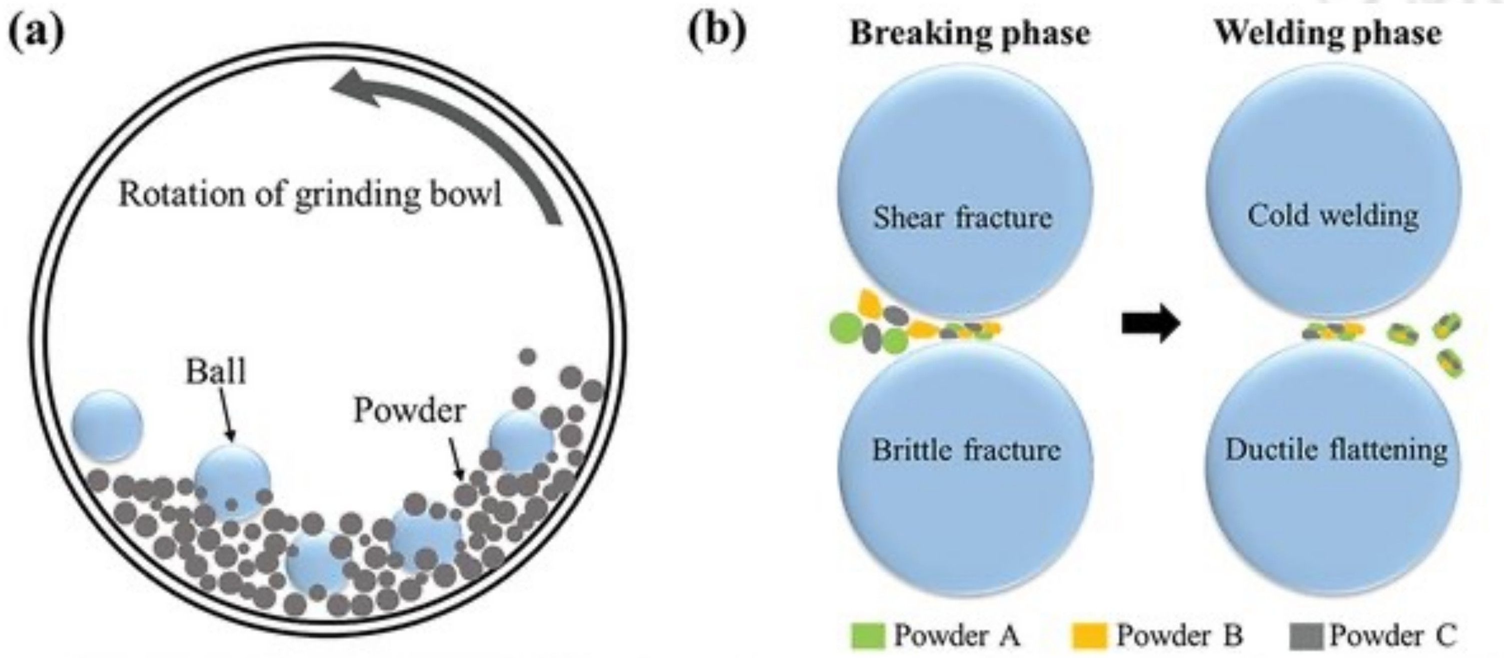
- **Mechanical**
  - Machining
  - Milling

  limited purity and homogeneity
  - Mechanical alloying
  - Cold stream process

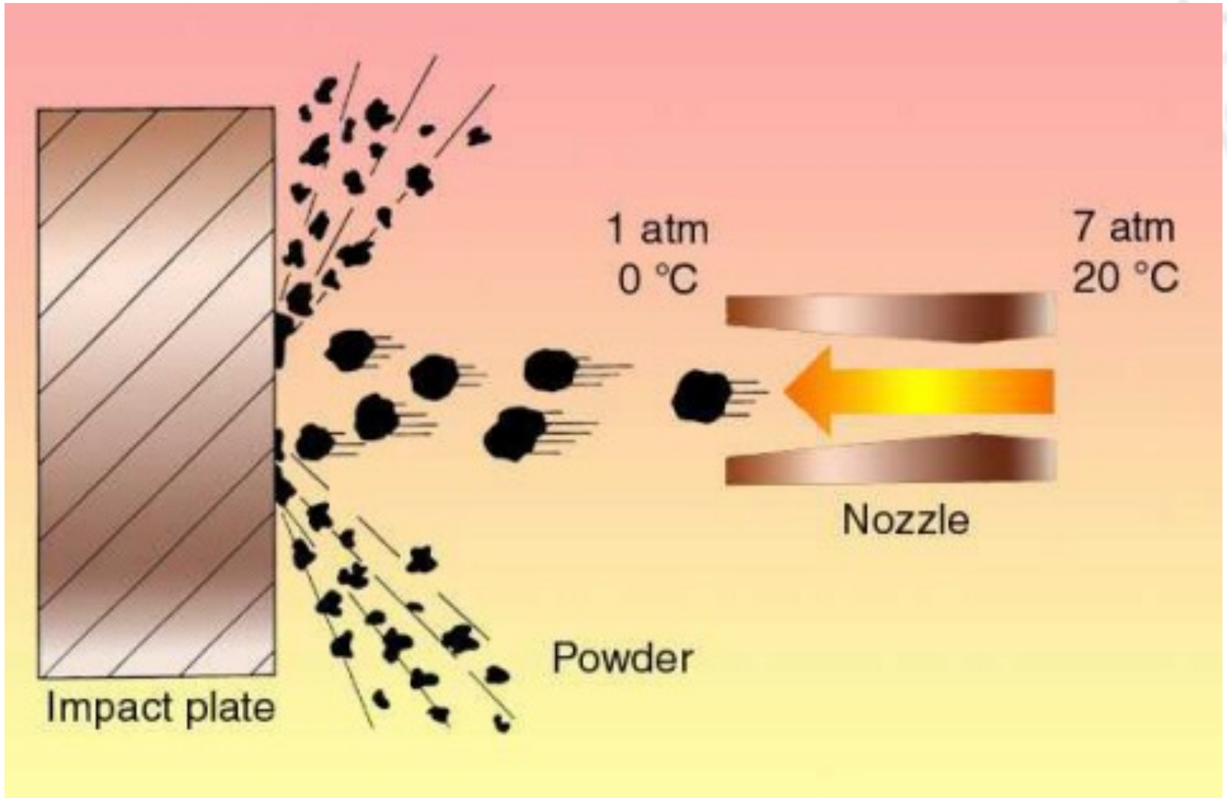
- **Chemical**
  - Electrolytic deposition
  - Decomposition of a solid by a gas
  - Thermal decomposition
  - Precipitation from a liquid
  - Precipitation from a gas
  - Solid-solid reactive synthesis

- **Physical**
  - Atomization techniques

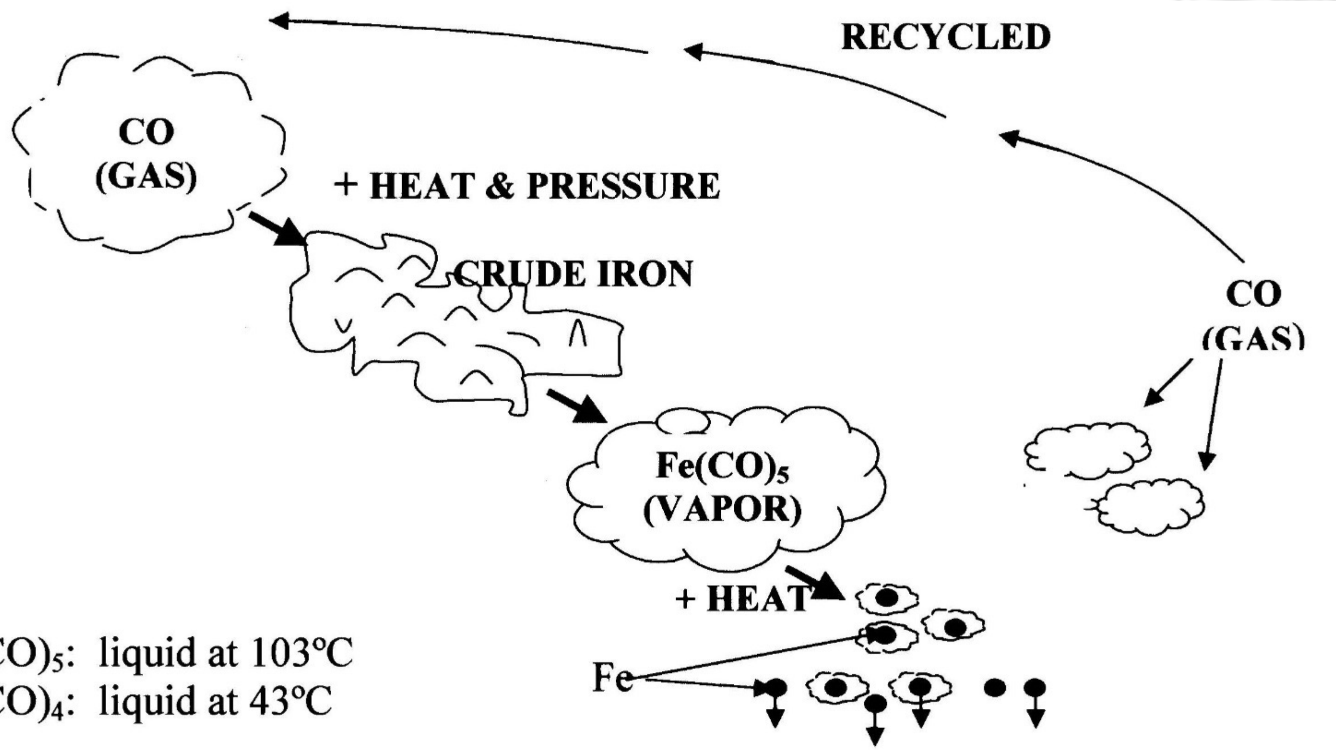
Mechanical alloying



Cold stream process



Carbonilic process

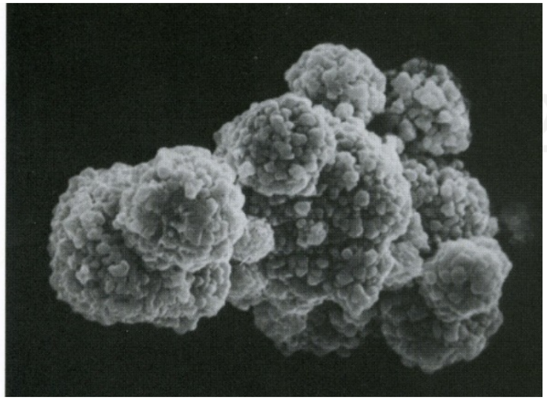


$Fe(CO)_5$: liquid at 103°C
$Ni(CO)_4$: liquid at 43°C

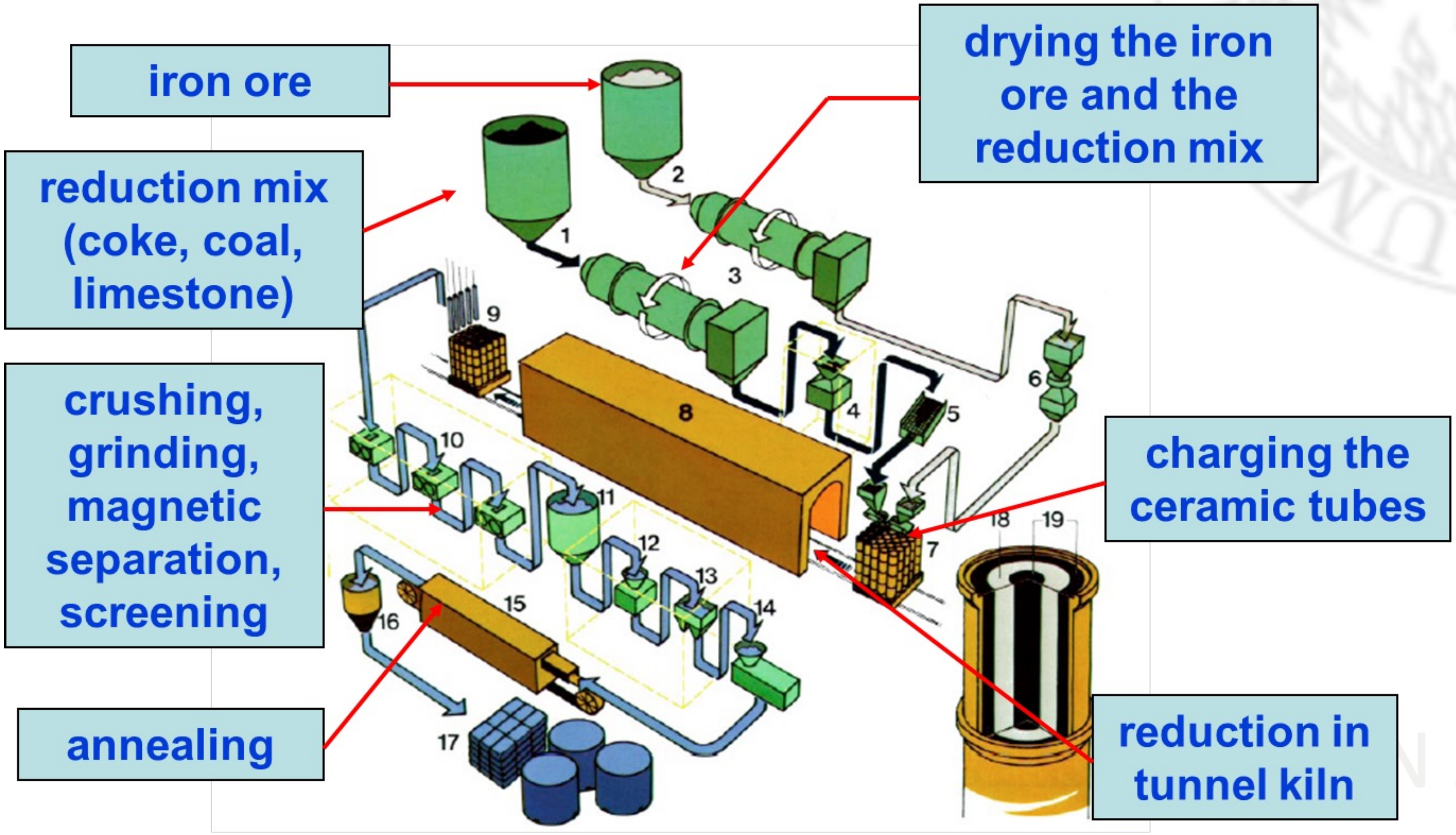
<u>Reactions</u>

$$\text{Fe (scrap or sponge)} + 5\,CO \xrightarrow[\text{200-400°F}]{\text{Pres.}} Fe(CO)_5$$

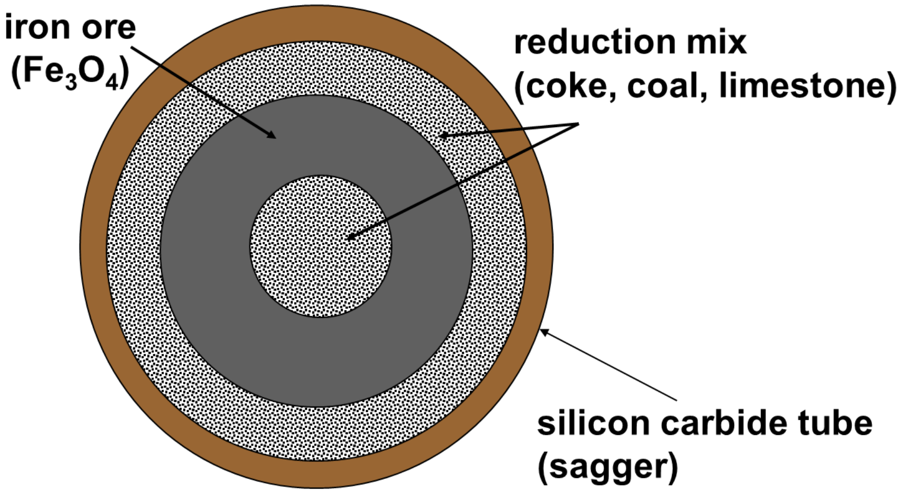
$$Fe(CO)_5 \xrightarrow[\text{300-750°F}]{\text{Atmosf.Pres}} 5\,CO + \text{Fe (powder)}$$



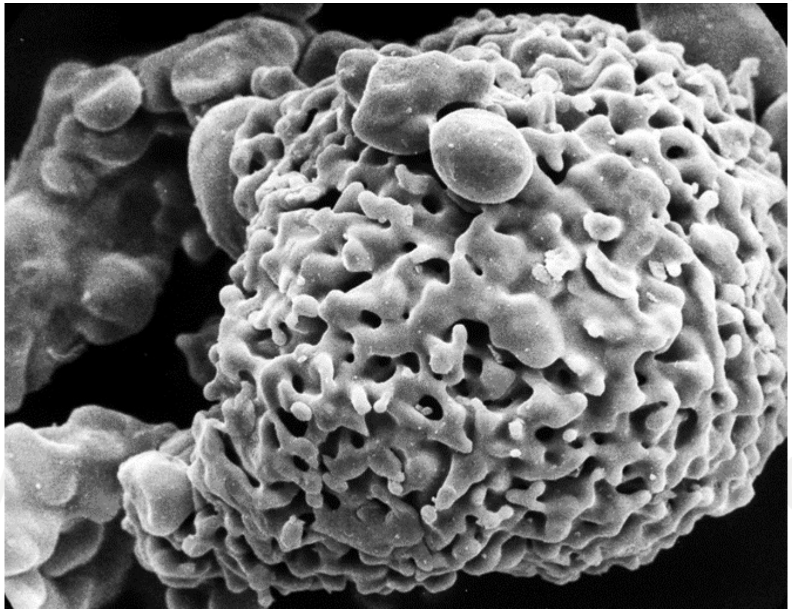
Sponge iron process



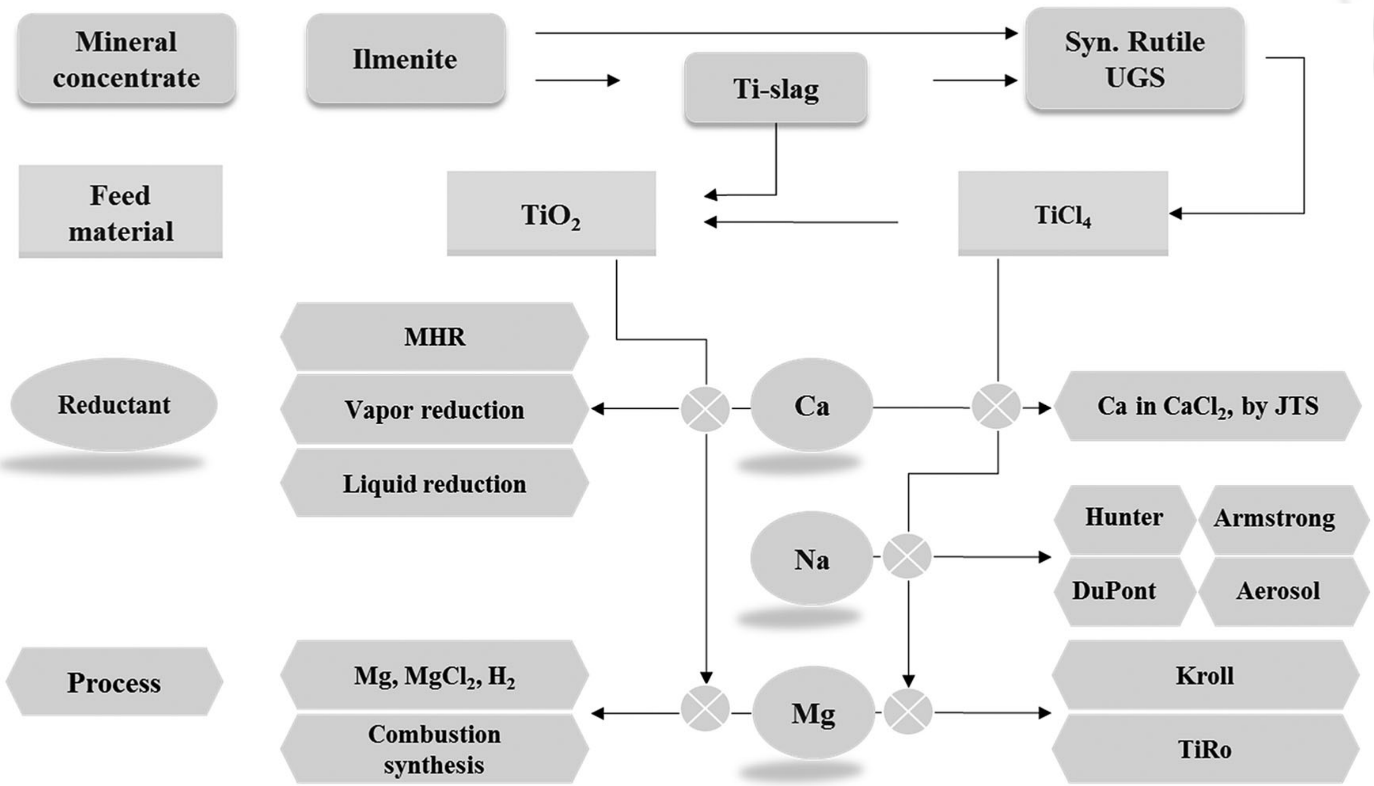
Ceramic tube cross section

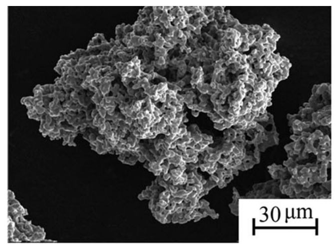


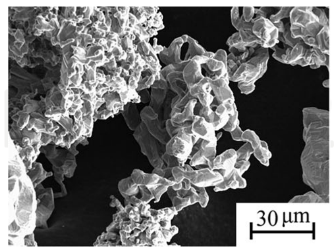
$$4\ CO + Fe_3O_4 = 3\ Fe + 4\ CO_2$$



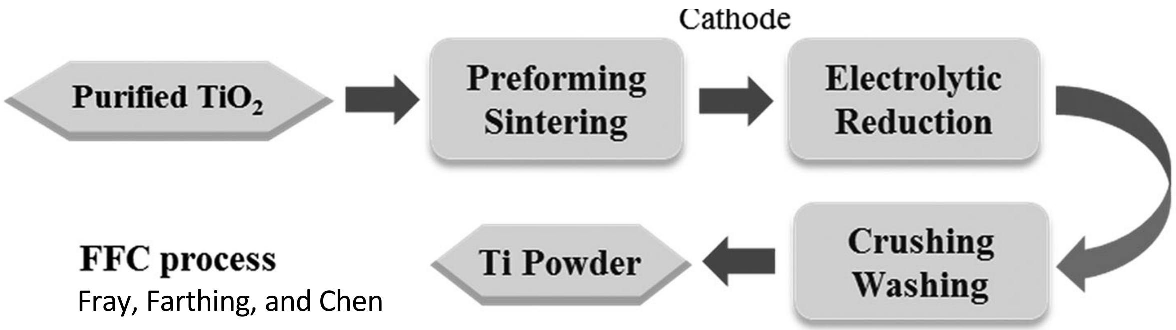
Thermochemical reduction routes to produce titanium







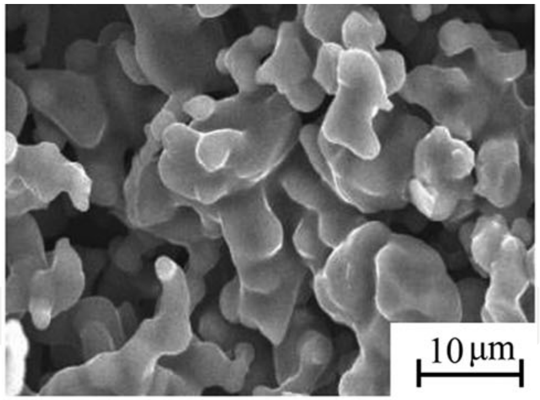
Electrochemical reduction to produce titanium



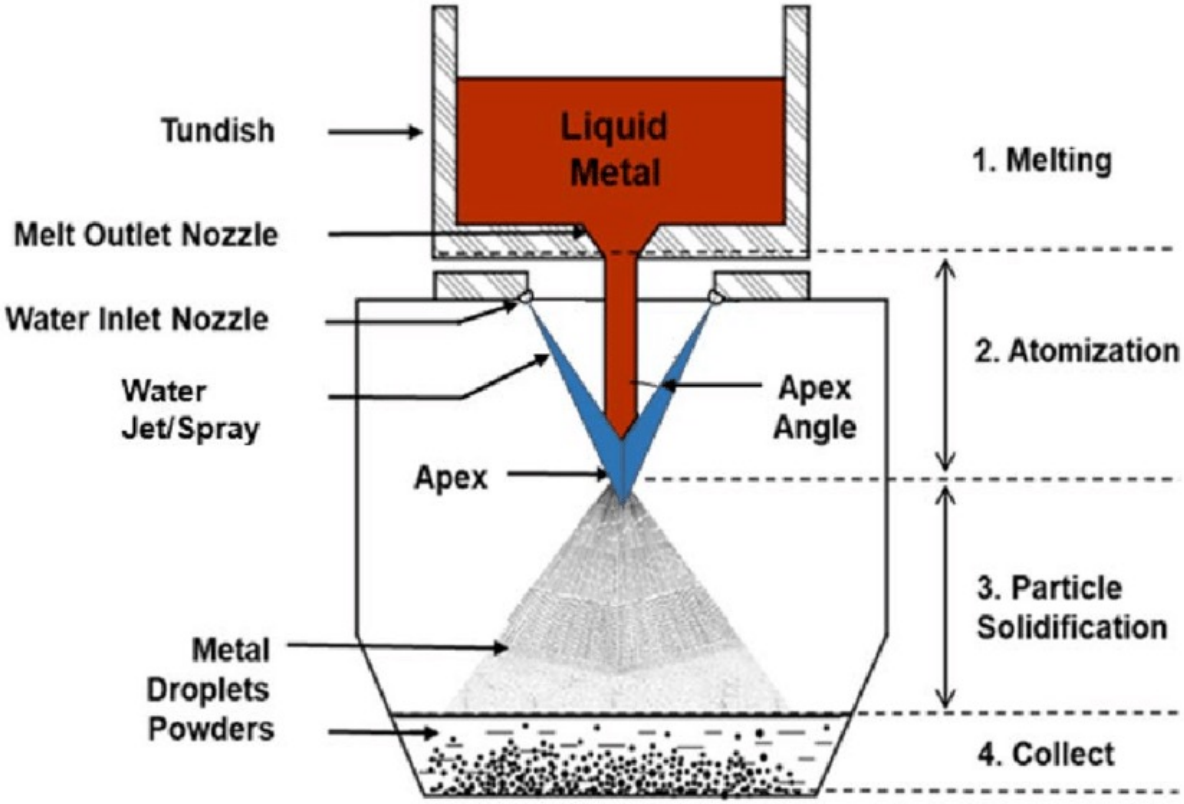
FFC process
Fray, Farthing, and Chen

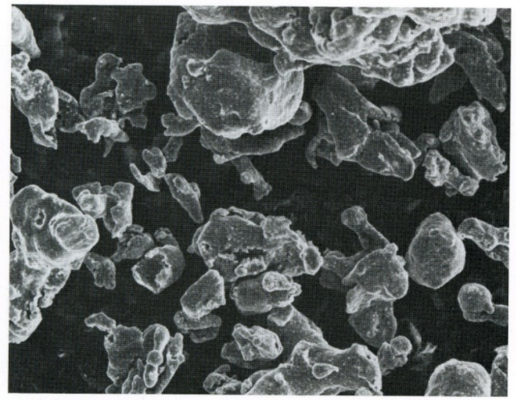
Cathod: $[O]_{TiO_2} + e^- \rightarrow O^{2-}$ Anode: $O^{2-} + C \rightarrow CO/CO_2 + e^-$

in molten $CaCl_2$ (800-1100°C)



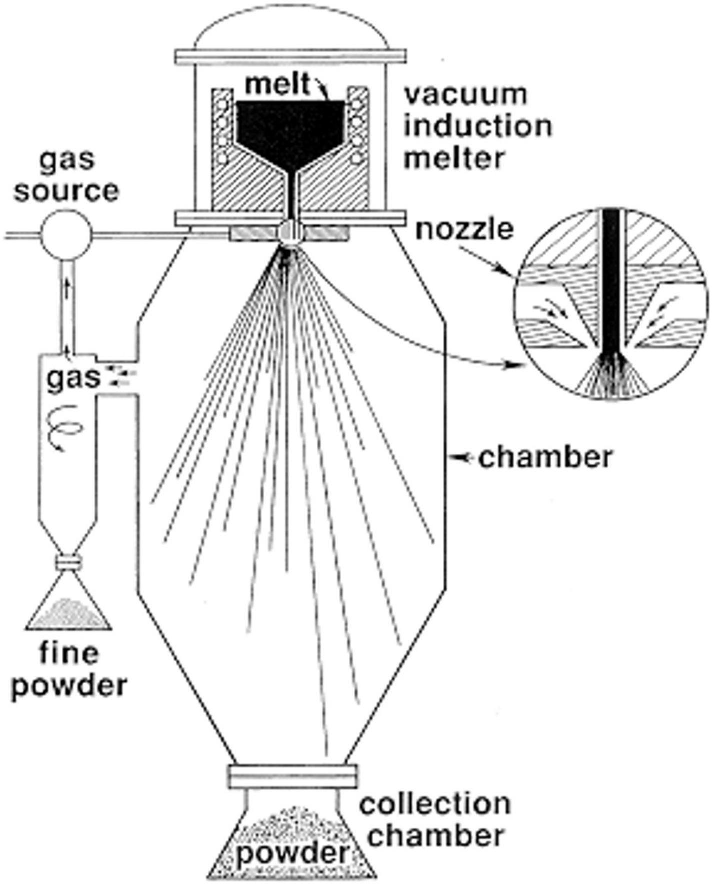
Water atomization

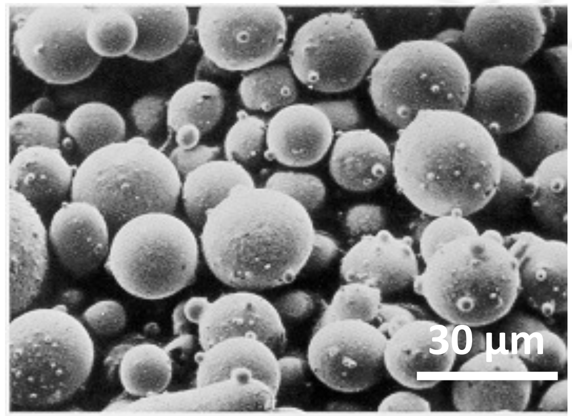




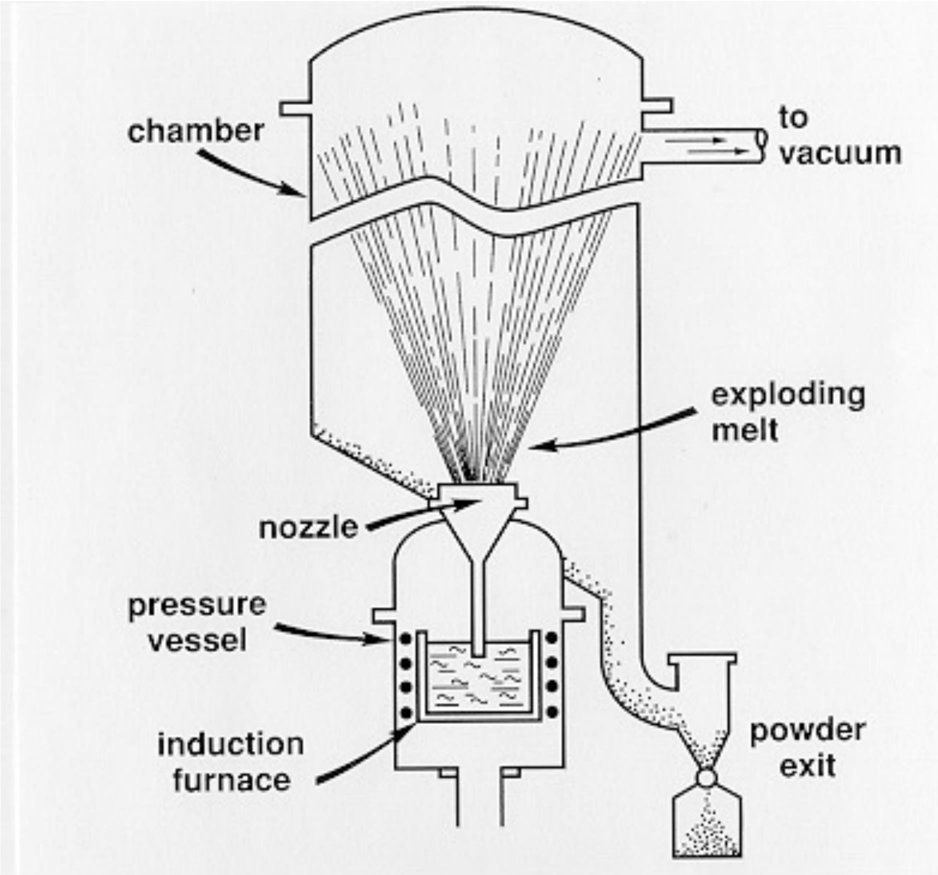
Surface oxidation!

Gas atomization

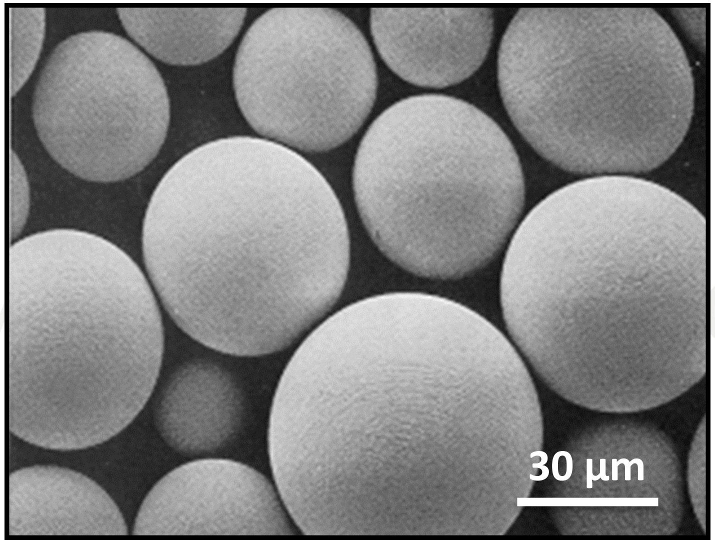




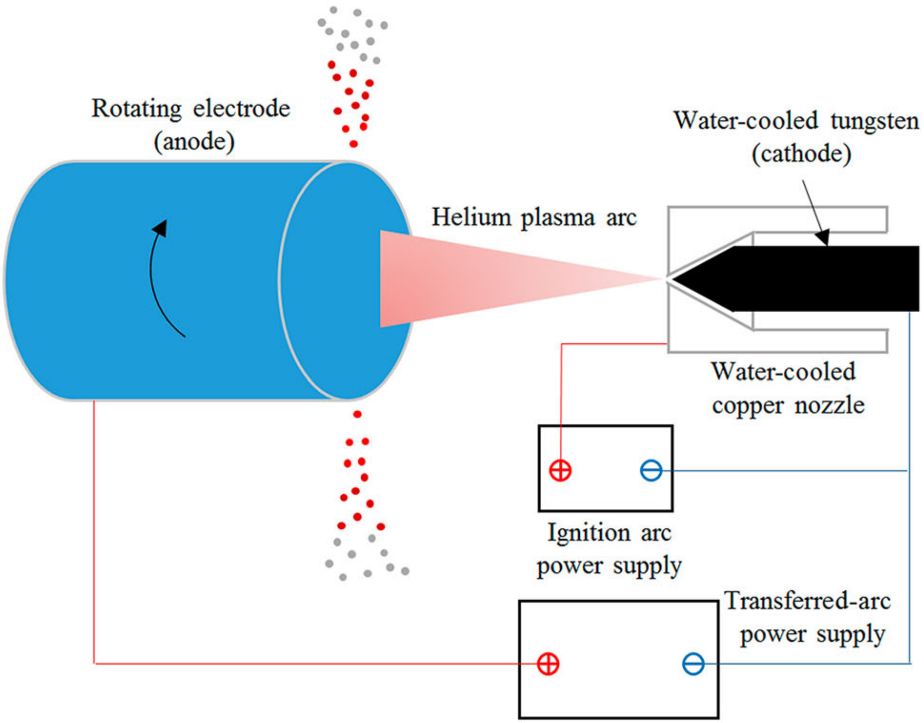
Vacuum atomization

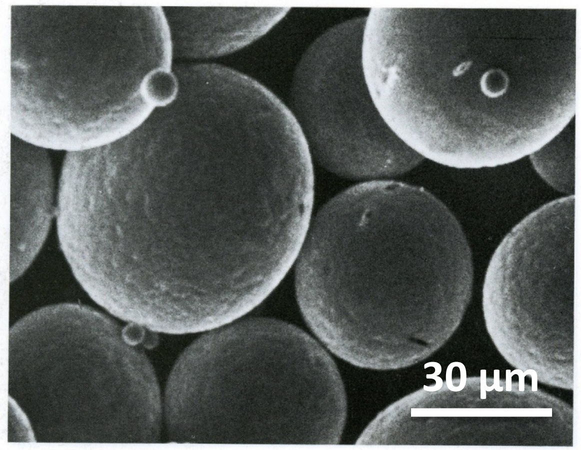


The melt is pressurized with hydrogen (1–3 MPa) and then released into vacuum



Plasma rotating electrode process





Ti-6Al-4V SEM, 100×

Powder characteristics for the green compact

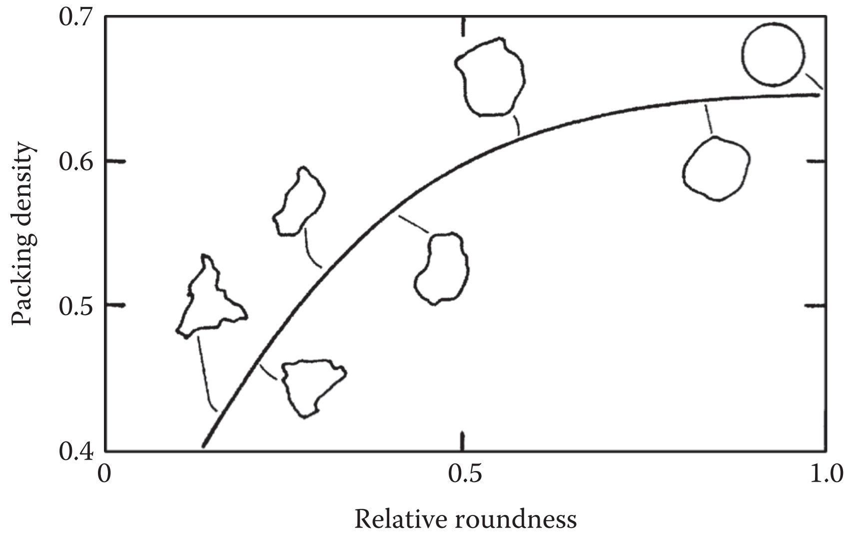
- **Aspect # 1: packing factor**

for particles of the same size

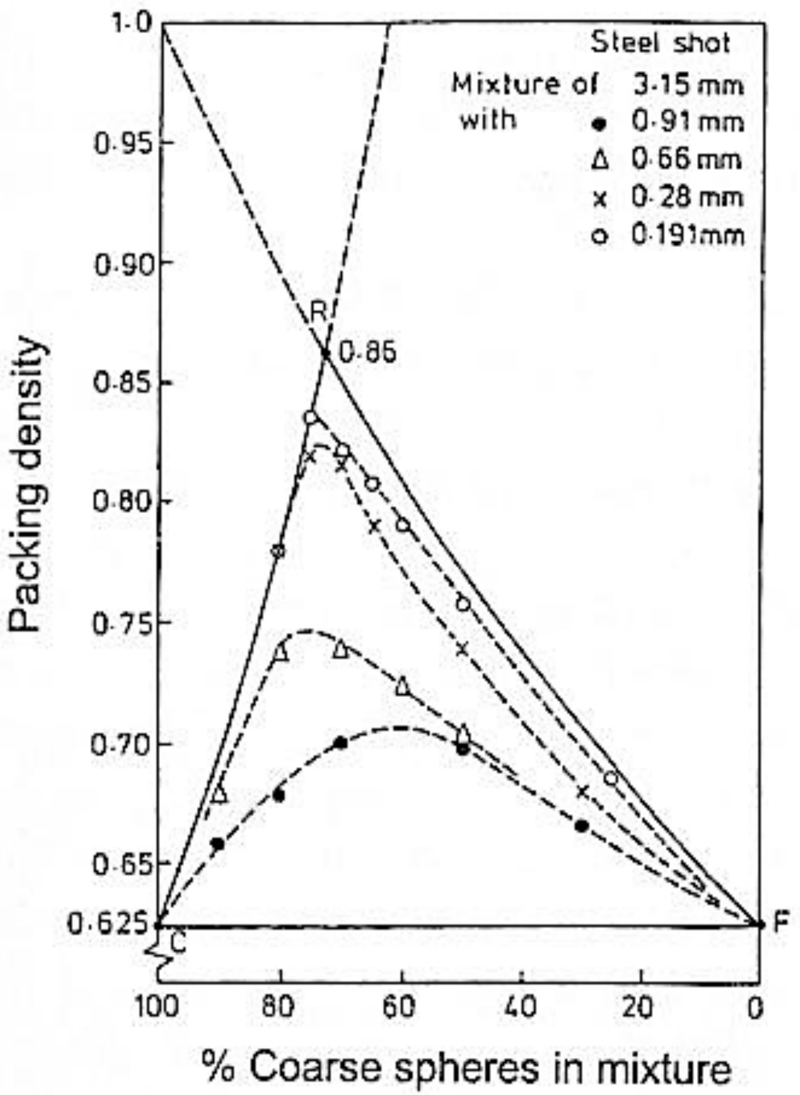
dense packing: ***PF = 0.635-0.64***
loose packing: ***PF = 0.57-0.61***

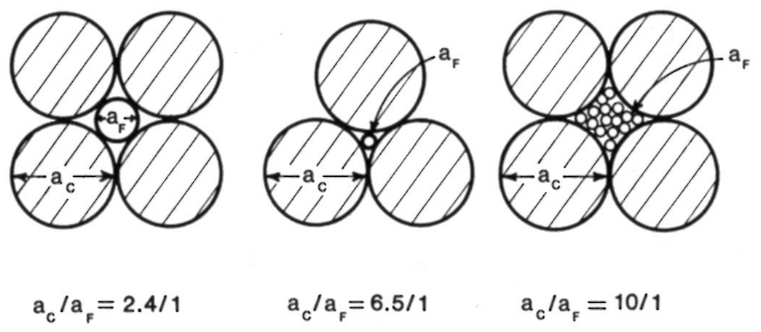
shape effect

Particle Shape	Aspect Ratio	Packing Density
Sphere	1	0.64
Cube	1	0.75
Rectangle	2:5:10	0.51
Plate	1:4:4	0.67
Plate	1:8:8	0.59
Cyclinder	5	0.52
Cyclinder	15	0.28
Cyclinder	60	0.09
Disk	0.5	0.63
Tetrahedron	1	0.5

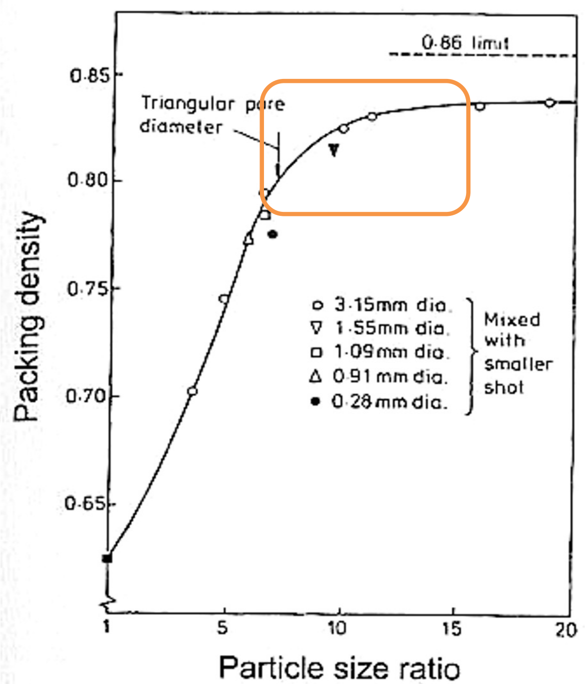


for particles with different size

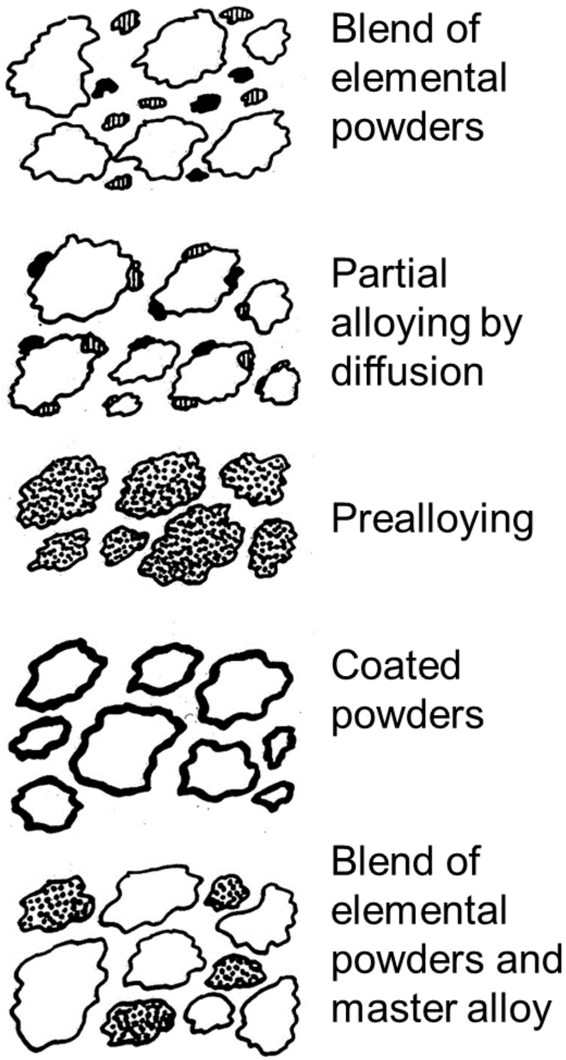




best condition: $15 > a_c/a_f > 7$



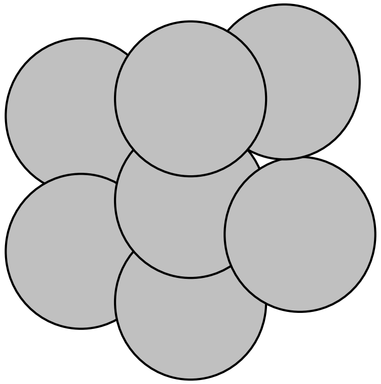
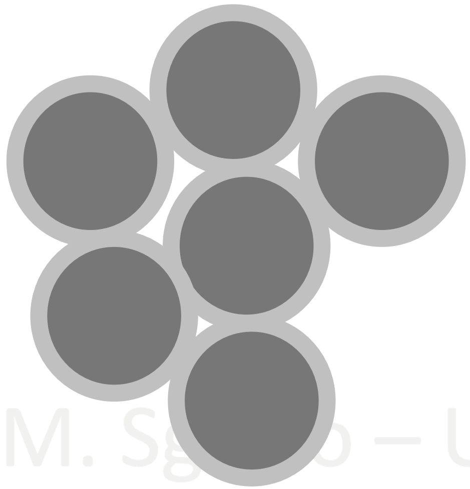
- **Aspect # 2: alloying in PM**



Suspensions

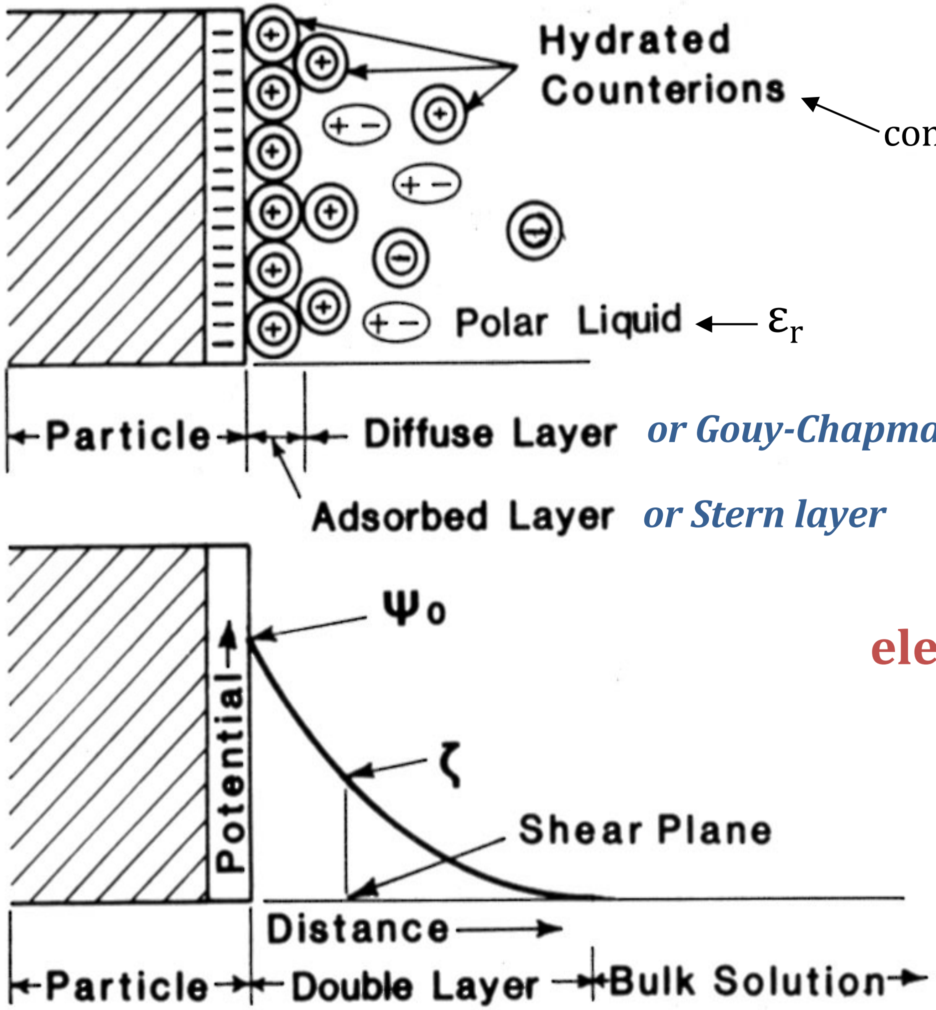
suspension = biphasic system constituted by a liquid and a solid

Possible configurations

Agglomeration	*Stabilization*
	
rigid system	*plastic (deformable) system*

Double electrical layer

the majority of the systems are negatively charged on the surface



concentration (molarity) = N_i

Z_i = ion charge

ε_r

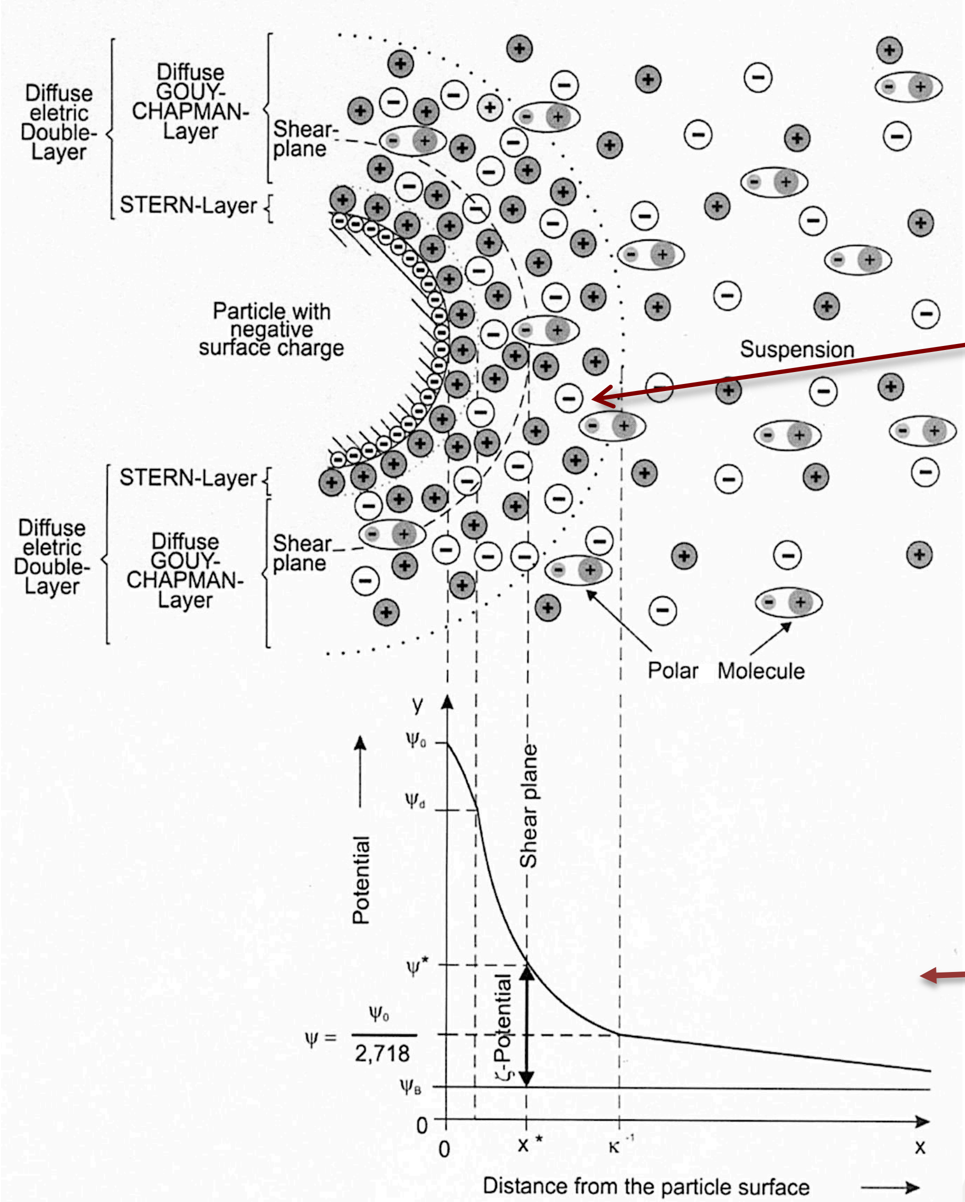
or Gouy-Chapman layer

or Stern layer

electrical potential (Gouy-Chapman)

$$\psi = \psi_0 \, e^{-\left(x/\kappa^{-1}\right)}$$

ψ_o = surface potential

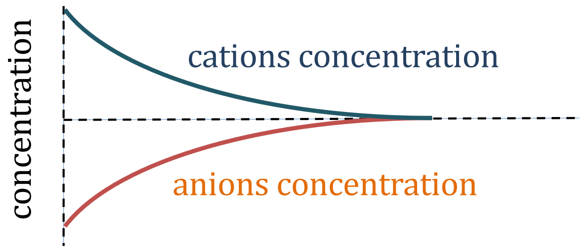


molar concentration of ion i

$$N_i = N_{i0} \exp\left(-\frac{U_i}{k_B T}\right)$$

potential energy of ion i (valence = Z_i)

$$U_i = Z_i e \psi$$



for a 1-1 electrolyte

$\boldsymbol{\kappa^{-1}}$ **=** distance at which $\psi = \psi_0/2.718$ **= Debye length** (κ = *Debye-Huckel parameter*)

$$\kappa^{-1} = \left(\frac{\varepsilon_r \varepsilon_0 N_A k_B T}{F^2 \underbrace{\sum N_i Z_i^2}_{\text{ionic strength}}} \right)^{0.5}$$

N_i, Z_i = molarity and valence of any ion type in the solution

Faraday constant
$F = N_A e^- = 9.649 \times 10^4$ C mol^{-1}

Permittivity of vacuum
$\varepsilon_0 = 8.854 \times 10^{-12}$ C^2 J^{-1} m^{-1}

Boltzman constant
$k_B = 1.381 \times 10^{-23}$ J K^{-1}

Zeta potential

ζ = electrical potential on the shear plane

viscosity

$$\zeta = \frac{f_H \, \eta \, v_e}{\varepsilon_r \, \varepsilon_0 \, E}$$

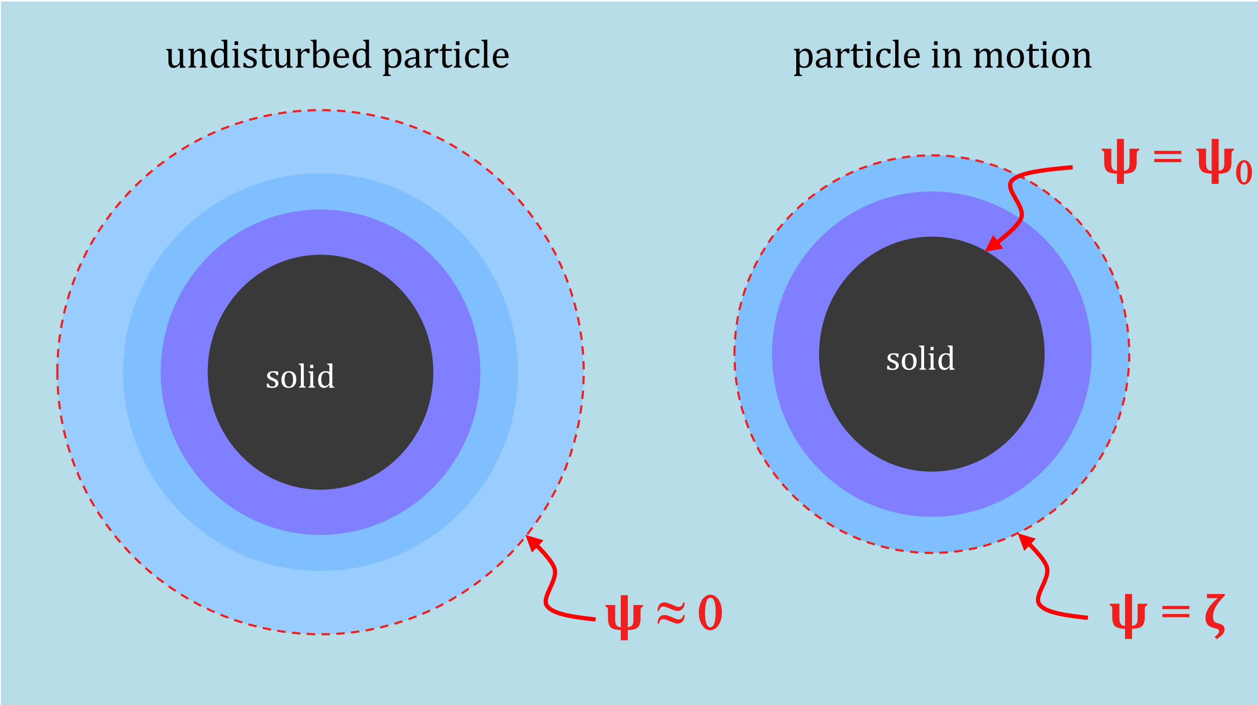
v_e/E = elettrophoretic mobility (measured independently)

$f_H = 1$ if $a/\kappa^{-1} = 0$

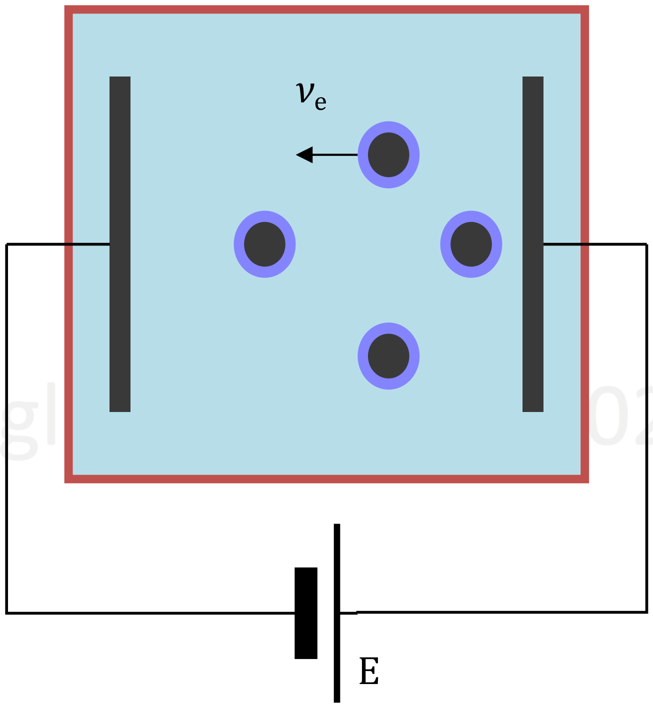
$= 1.5$ if $a/\kappa^{-1} = \infty$ (a = particle radius)

Compound		Dielectric Constant
Hydrogen-bonding		
methyl-formamide	$HCONHCH_3$	182.4
Formamide	$HCONH_2$	109.5
Hydrogen fluoride	HF	84[b]
Water	H_2O	78.5
Formic acid	HCOOH	58.5[c]
Ethylene glycol	$C_2H_4(OH)_2$	40.7
Methanol	CH_3OH	32.6
Ethanol	C_2H_5OH	24.3
n-Propanol	C_3H_7OH	20.2
Ammonia	NH_3	16.9
Acetic acid	CH_3COOH	6.2
Non-hydrogen bonding		
Acetone	$(CH_3)_2CO$	20.7
Chloroform	$CHCl_3$	5.0
Benzene	C_6H_6	2.3
Carbon tetrachloride	CCl_4	2.2
Cyclohexane	C_6H_{12}	2.0
Dodecane	$C_{12}H_{26}$	2.0
Hexane	C_6H_{14}	1.9
Crystals		
Sodium chloride	NaCl	6.0
Diamond	C	5.7
Quartz	SiO_2	4.5

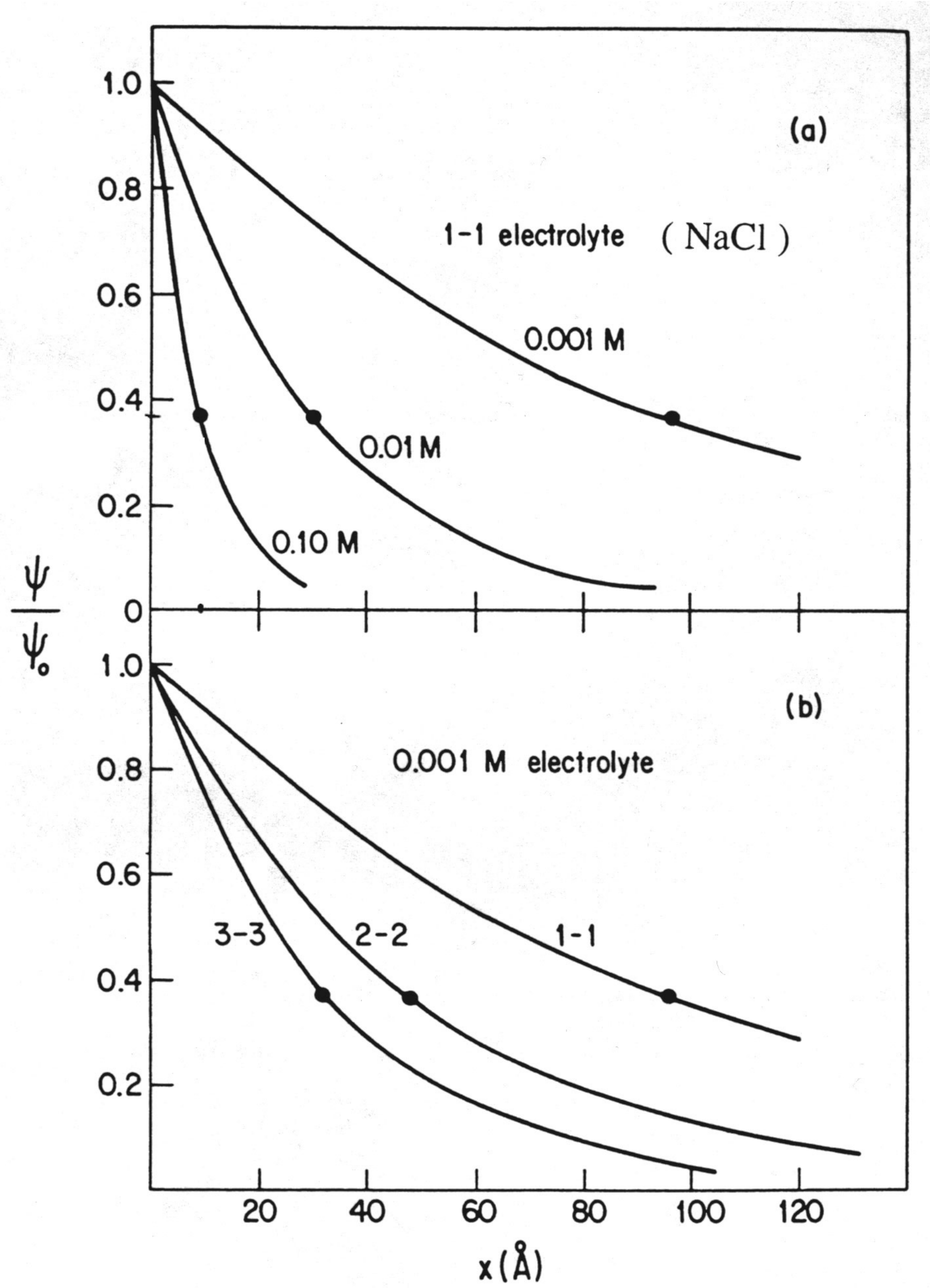
the zeta potenzial, ζ, is an index of the electrical potential (ψ) gradient



elettrophoresis



- *influence of ions in solution*



- if N increases κ^{-1} decreases

and ζ decreases, too

- if Z increases κ^{-1} decreases

Surface charge origin

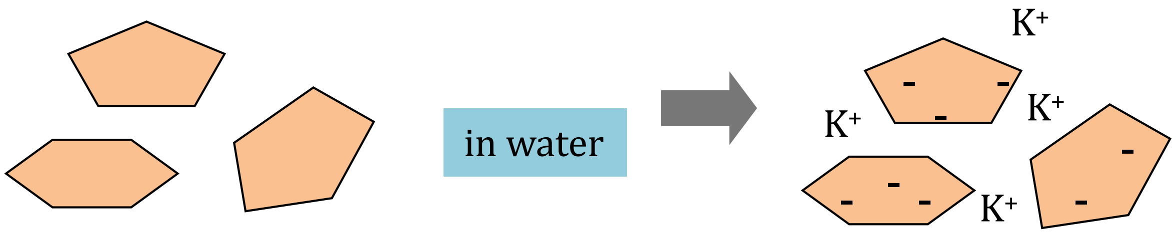
- **desorption & dissolution of ions** (clays – isomorphic substitution of Si & Al)

example: kaolinite

$$Si^{4+}_{(network)} \rightarrow Al^{3+}_{(network)} + K^{+}_{(surface)}$$

$$Si^{4+}_{(network)} \rightarrow Mg^{2+}_{(network)} + 2\ K^{+}_{(surface)}$$

weakly bonded

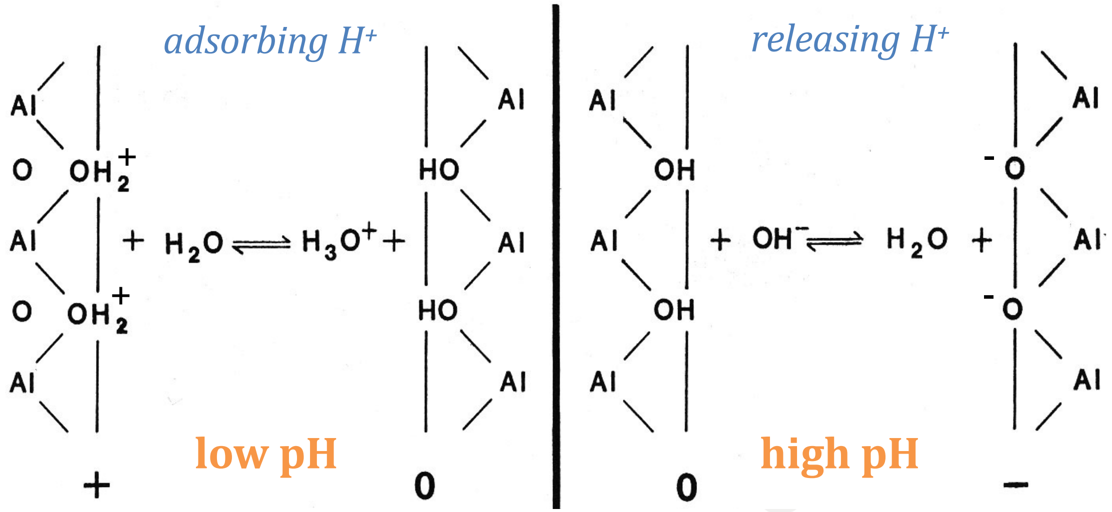


- **chemical reactions with aqueous medium**

$$\mathrm{MOH_{(surface)}} + \mathrm{H^{+}_{(solution)}} \overset{K_1}{\longleftrightarrow} \mathbf{MOH_2^{+}}_{\mathrm{(surface)}}$$

$$\mathrm{MOH_{(surface)}} \underset{K_2}{\longleftrightarrow} \mathbf{MO^{-}}_{\mathrm{(surface)}} + \mathrm{H^{+}_{(solution)}}$$

Example: alumina

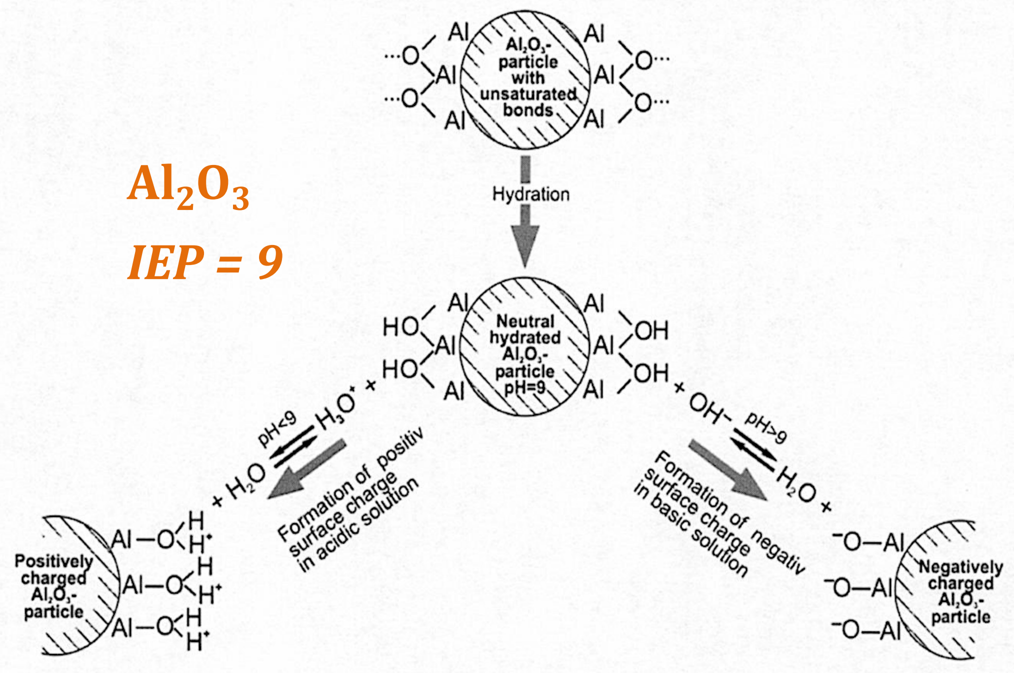


point of zero charge = PZC = $\dfrac{pK_1^{-1} + pK_2}{2}$ **= isoelectric point = IEP**

acid-base character of the surface
(K_1 and K_2 depend on the material)

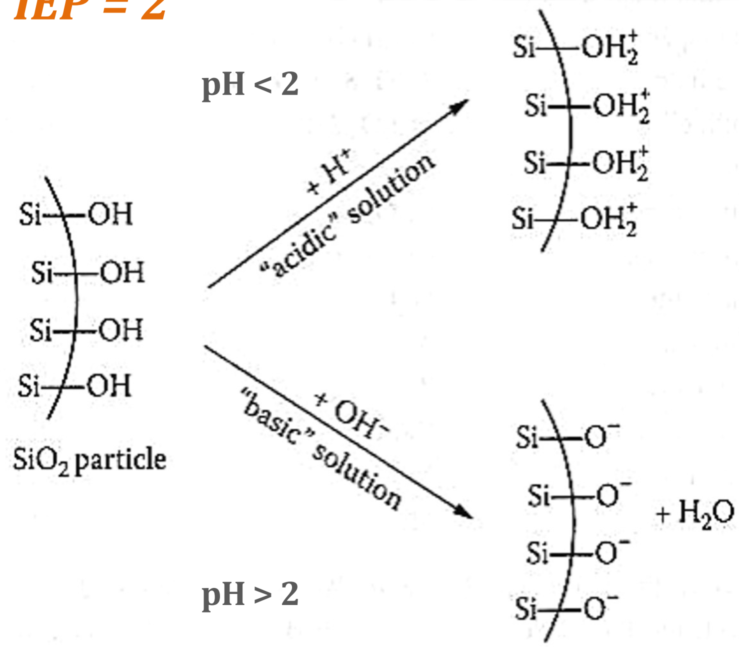
Al_2O_3

IEP = 9



SiO_2

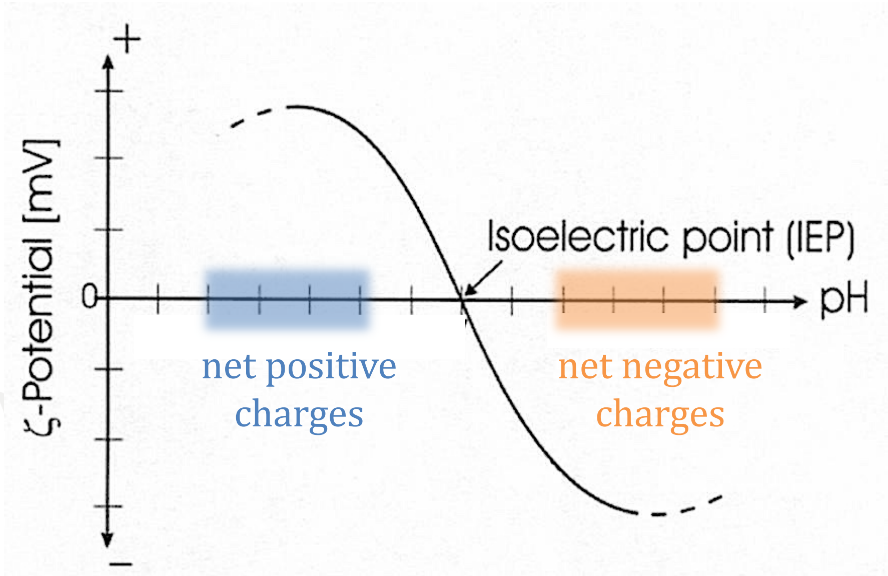
IEP = 2



Material	Nominal Composition	IEP
Muscovite	$KAl_3Si_3O_{11} \cdot H_2O$	1
Quartz	SiO_2	2
Delta manganese oxide	MnO_2	2
Soda lime silica glass	$1.00Na_2O \cdot 0.58CaO \cdot 3.70SiO_2$	2–3
Albite	$Na_2O \cdot Al_2O_3 \cdot 6SiO_2$	2
Orthoclase	$K_2O \cdot Al_2O_3 \cdot 6SiO_2$	3–5
Silica (amorphous)	SiO_2	3–4
Zirconia	ZrO_2	4–5
Rutile	TiO_2	4–5
Tin Oxide	SnO_2	4–7
Apatite	$10CaO \cdot 6PO_2 \cdot 2H_2O$	4–6
Zircon	$SiO_2 \cdot ZrO_2$	5–6
Anatase	TiO_2	6
Magnetite	Fe_3O_4	6–7
Hematite	αFe_2O_3	6–9
Goethite	$FeOOH$	6–7
Gamma iron oxide	γFe_2O_3	6–7
Kaolin (edges)	$Al_2O_3 \cdot SiO_2 \cdot 2H_2O$	6–7
Chromium oxide	αCr_2O_3	6–7
Mullite	$3Al_2O_3 \cdot 2SiO_2$	7–8
Gamma alumina	γAl_2O_3	7–9
Alpha alumina	αAl_2O_3	9–9.5
Alumina (Bayer process)	Al_2O_3	7–9.5
Zinc oxide	ZnO_2	9
Copper oxide	CuO	9
Barium carbonate	$BaCO_3$	10–11
Yttria	Y_2O_3	11
Lanthanum oxide	La_2O_3	10–12
Silver oxide	Ag_2O	11–12
Magnesium Oxide	MgO	12–13

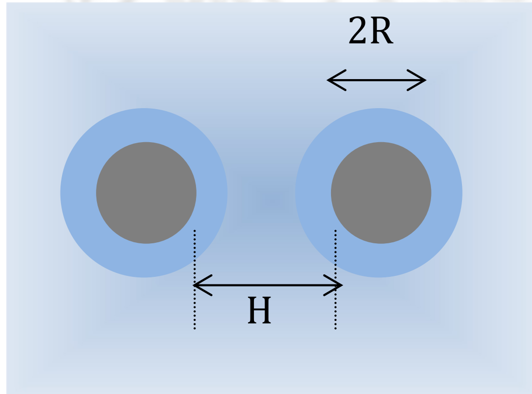
acidic-basic characteristics of surfaces

if pH > IEP → the surface charge is negative



Stability of suspensions

interaction between two colloidal particles in suspension



DLVO (Derjaguin e Landau / Verweij e Overbeek) theory

attraction: *Van del Waals force*

A_h = Hamaker constant ≈ 10^{-20} J

$$U_A = \frac{-A_h R}{12 H} \qquad F_A = \frac{-A_h R}{12 H^2}$$

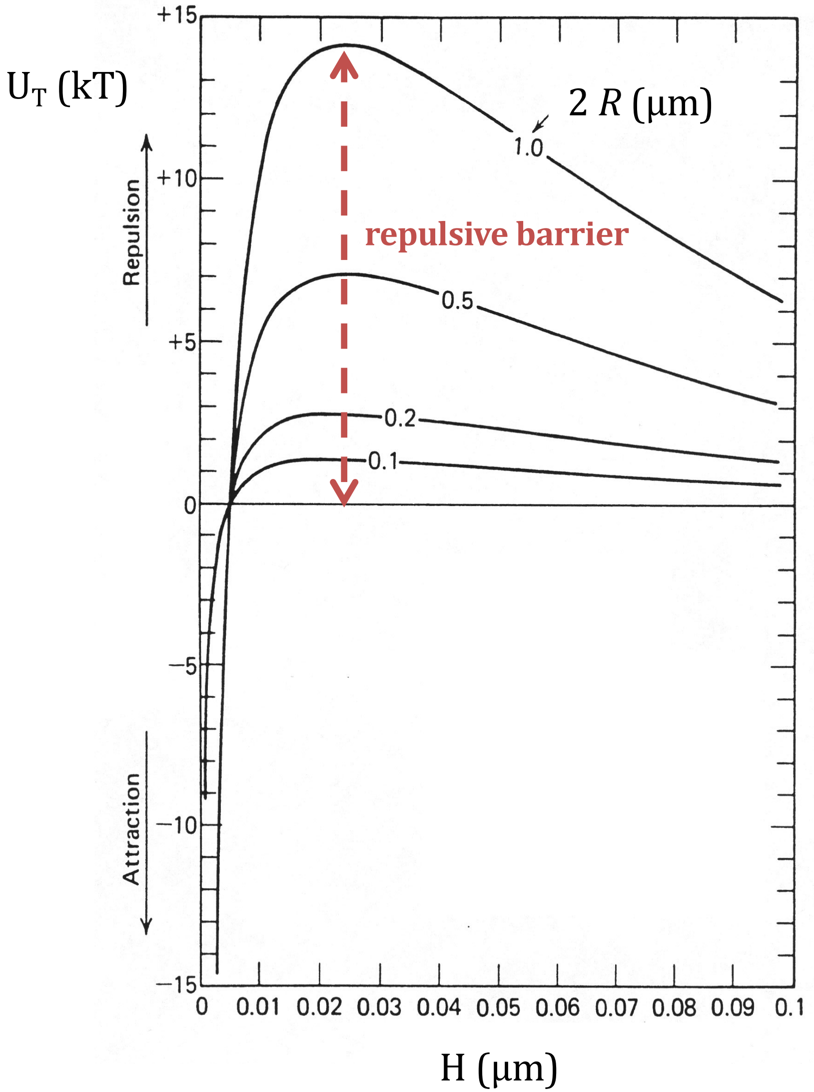
repulsion: *interaction between double electrical layers*

$$U_R = 2\pi\varepsilon\varepsilon_0 R\psi_0^2 e^{-\left(\frac{H}{\kappa^{-1}}\right)} \qquad R/\kappa^{-1} < 5$$

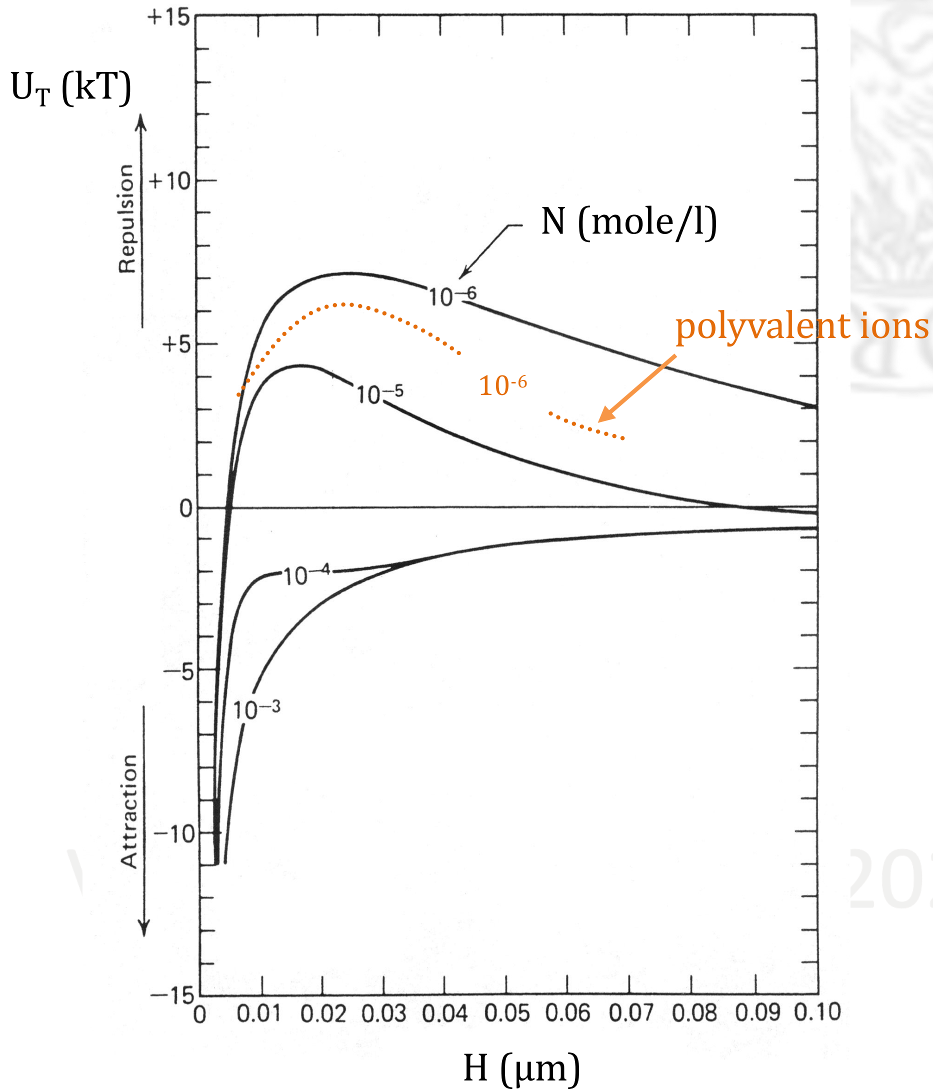
$$F_R = \frac{2\pi\varepsilon\varepsilon_0 R\psi_0^2}{\kappa^{-1}} e^{-\left(\frac{H}{\kappa^{-1}}\right)}$$

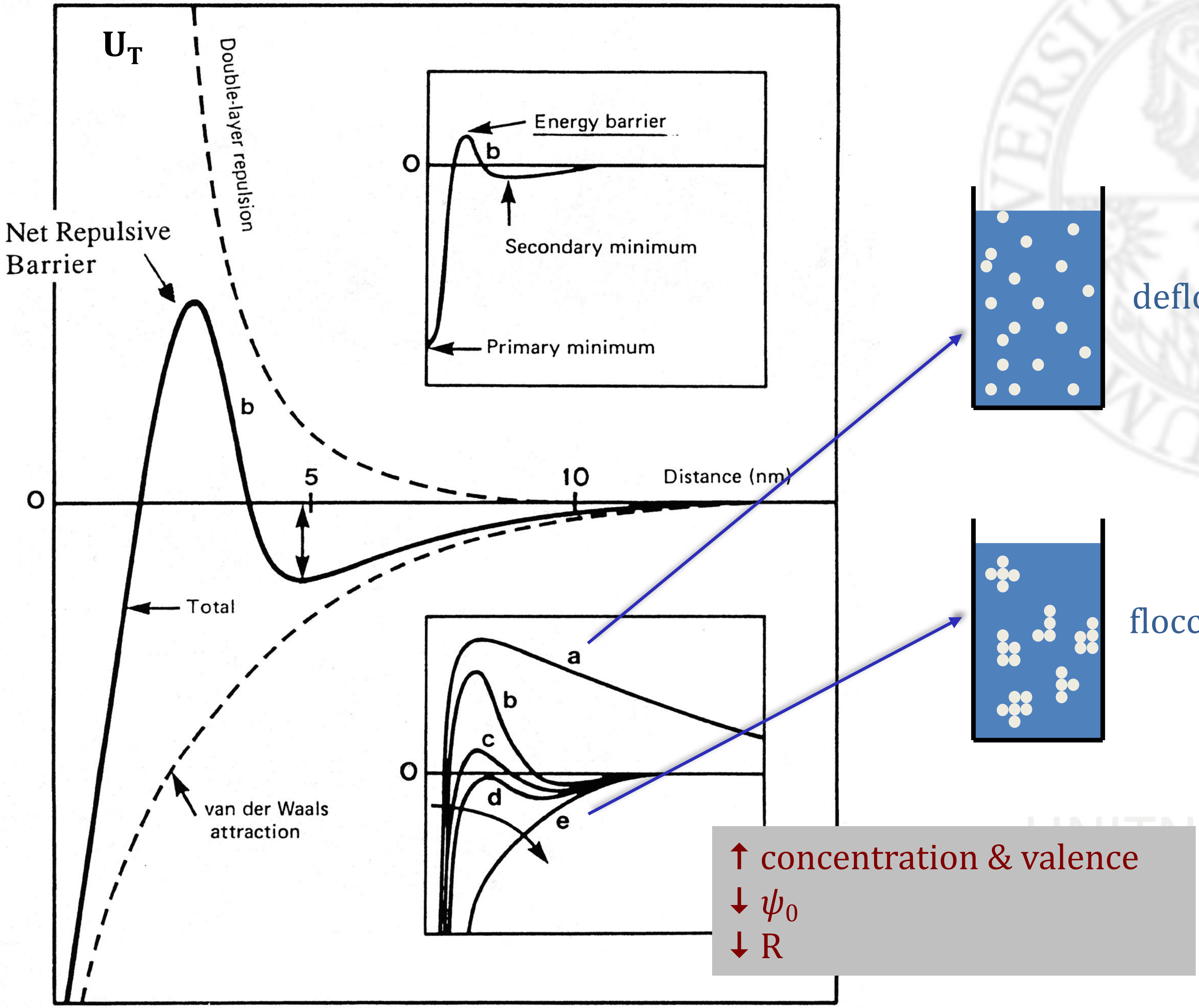
$U_T = U_A + U_R$

particles size



concentration of the solution



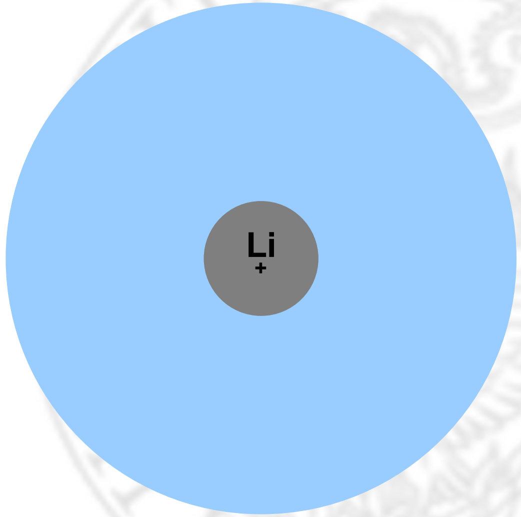


U_T
Double-layer repulsion
Net Repulsive Barrier
b
O
5
10
Distance (nm)
Total
van der Waals attraction
Energy barrier
b
O
Secondary minimum
Primary minimum
a
b
c
O
d
e
deflocculated
flocculated
↑ concentration & valence
↓ ψ_0
↓ R

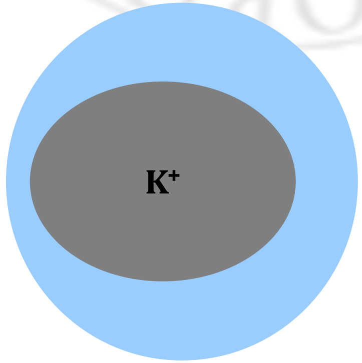
Coagulation *(not desired in ceramic systems!)*

- ζ < 20 mV (Brownian motions)
- presence of ions: $Li^+ > Na^+ > K^+ > NH_4^+$
  $Mg^{2+} > Ca^{2+} > Sr^{2+} > Ba^{2+}$
  $I^- > SO_4^{2-} > Cl^- > F^- > NO_3^-$

coagulation power



solvation cloud + polarizability



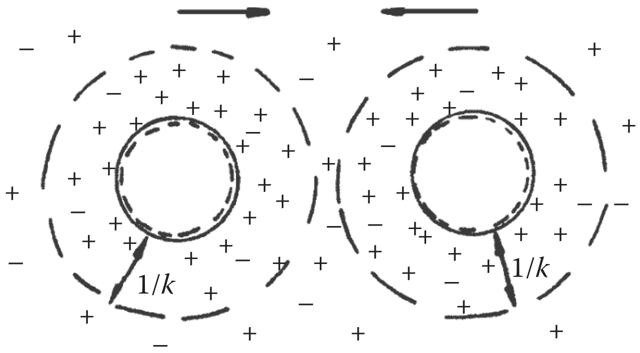
Ion	Radius (Å)	Hydration (mol H_2O)	Hydrated Radius (Å)
Li^+	0.78	14	7.3
Na^+	0.98	10	5.6
K^+	1.33	6	3.8
Rb^+	1.49	0.5	3.6
NH_4^+	1.43	3	—
Mg^{2+}	0.78	22	10.8
Ca^{2+}	1.06	20	9.6
Ba^{2+}	1.43	19	8.8
Al^{3+}	0.57	57	—

adsorbtion tendency

coagulant agents

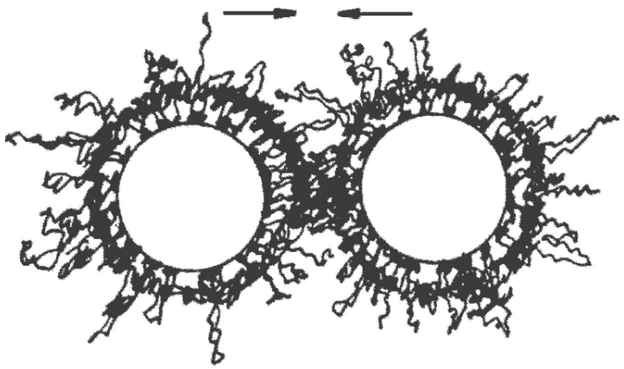
$CaCl_2$, $CaCO_3$, $MgCl_2$, $MgSO_4$, $FeCl_3$, $AlCl_3$

Deflocculation – stabilization of colloidal suspensions



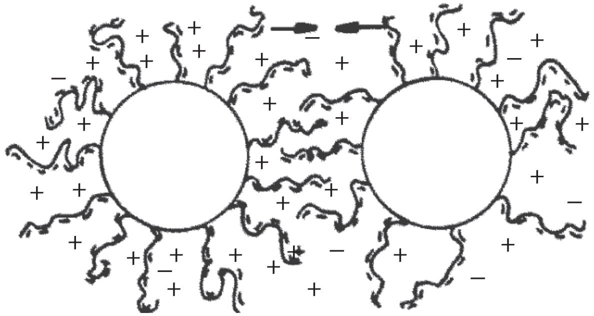
- ***electrostatic stabilization***

  when ζ > 20 mV



- ***steric stabilization***

  adsorbtion of polymers/surfactants



- ***electrosteric stabilization***

  adsorbtion of polyelectrolytes

electrostatic stabilization

$$U_A = \frac{-A_h R}{12H}$$

$$U_R = 2\pi\varepsilon\varepsilon_0 R\psi_0^2 e^{-\left(\frac{H}{\kappa^{-1}}\right)}$$

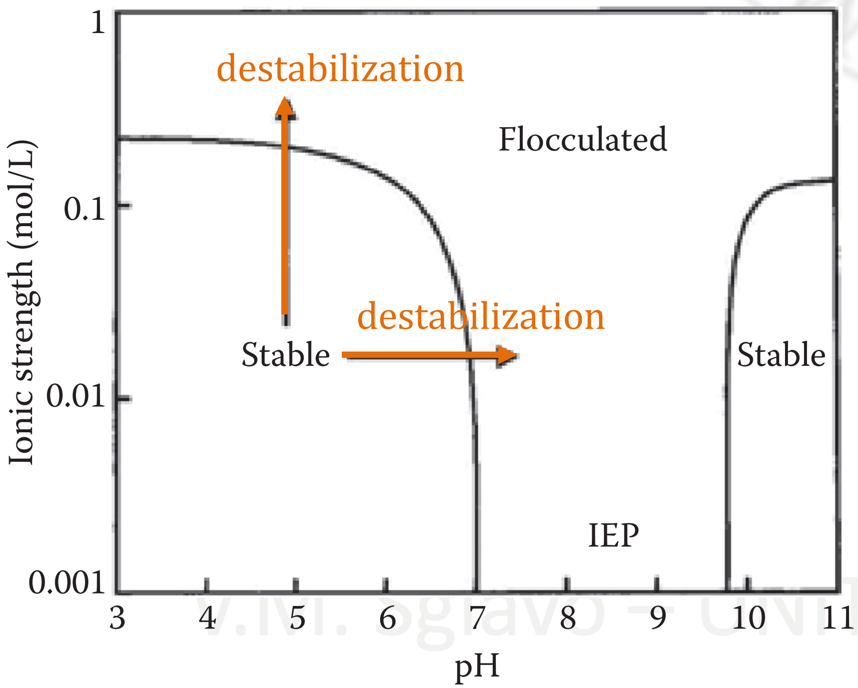
$$\kappa^{-1} = \left(\frac{\varepsilon_r \varepsilon_0 N_A k_B T}{F^2 \sum N_i Z_i^2}\right)^{0.5}$$

different effect of R, ψ_0, N_i, Z_i

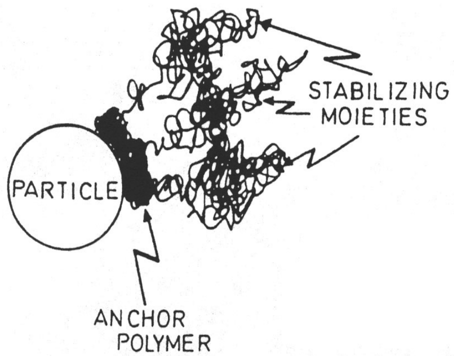
pH

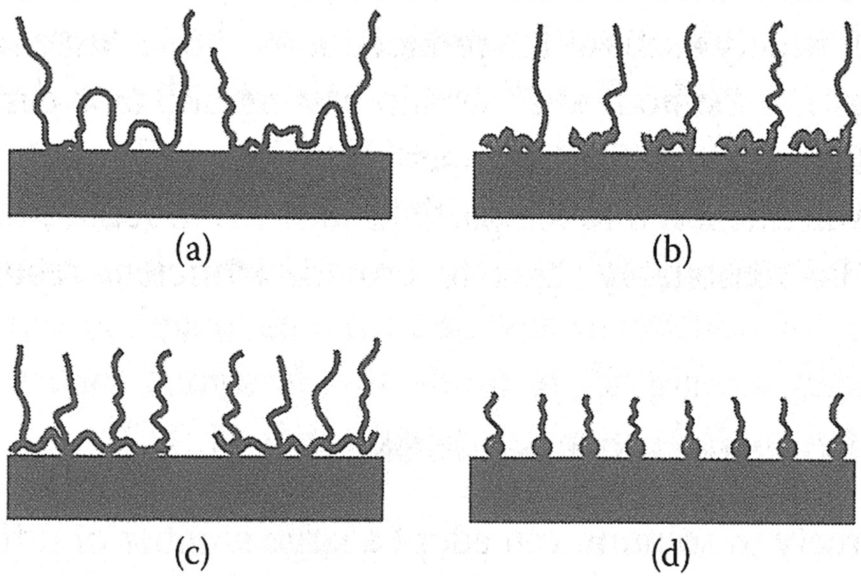
alumina

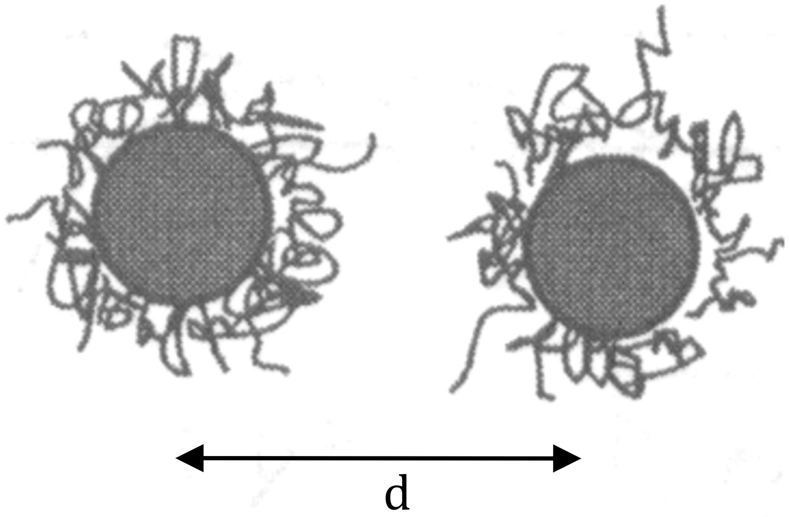
$N_i Z_i^2$



steric stabilization: adsorbtion of specific polymers /surfactants with high MW (>10^6)



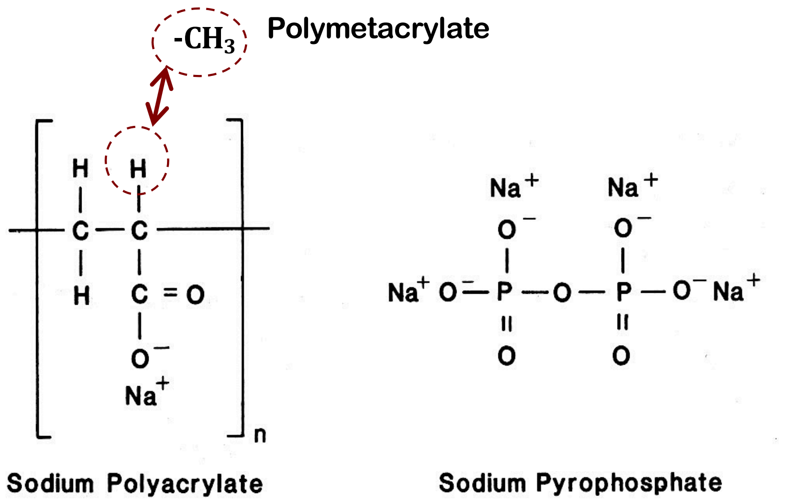


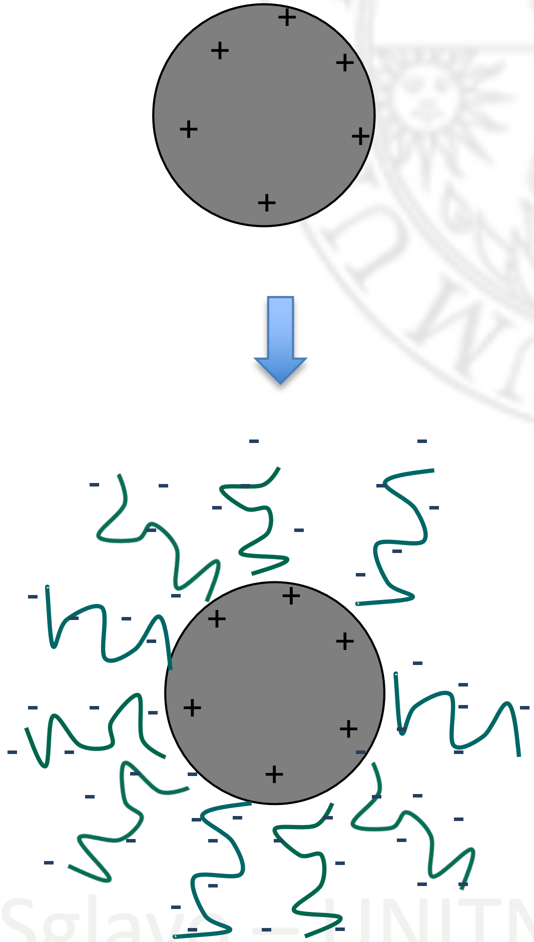


$$\Delta G_{steric}(\infty \rightarrow d) = \Delta G_{mix} + \Delta G_{entr}$$

Aqueous dispersions	
Anchor polymer	Stabilizing moieties
polystyrene	poly(oxyethylene)
poly(vinyl acetate)	poly(vinyl alcohol)
poly(methyl methacrylate)	poly(acrylic acid)
poly(acrylonitrile)	poly(methacrylic acid)
poly(dimethylsiloxane)	poly(acrylamide)
poly(vinyl chloride)	poly(vinyl pyrrolidone)
poly(ethylene)	poly(ethylene imine)
poly(propylene)	poly(vinyl methyl ether)
poly(lauryl methacrylate)	poly(4-vinylpyridine)
Nonaqueous dispersions	
Anchor polymer	Stabilizing moieties
poly(acrylonitrile)	polystyrene
poly(oxyethylene)	poly(lauryl methacrylate)
poly(ethylene)	poly(12-hydroxystearic acid)
poly(propylene)	poly(dimethylsiloxane)
poly(vinyl chloride)	poly(isobutylene)
poly(methyl methacrylate)	*cis*-1:4-poly(isoprene)
poly(acrylamide)	poly(vinyl acetate)
	poly(methyl methacrylate)
	poly(vinyl methyl ether)

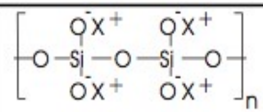
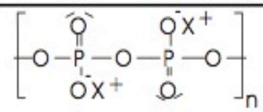
electrosteric stabilization: adsorbtion of ions / polyelectrolites

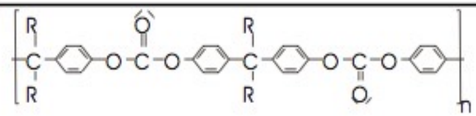
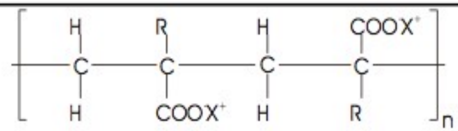
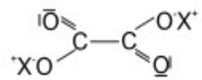
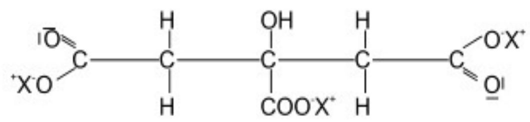
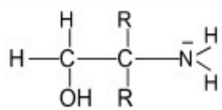




deflocculant agents

Inorganic	Organic
Sodium carbonate	Sodium polymethacrylate
Sodium silicate	Ammonium polyacrylate
Sodium borate	Sodium citrate
Tetrasodium pyrophosphate	Sodium succinate
	Sodium tartrate
	Sodium polysulfonate
	Ammonium citrate

Inorganic dispersing agents	X=Na, K, NH_4
Polysilicate	
Polyphosphate	

Organic dispersing agents	X=Na, K, NH_4; R=C_kH_{2k+1} with k=0,1,2,3,...
Polycarbonate	
Polyacrylate (*k=0*) and -methacrylate (*k=1,2,3,...*)	
Oxalate	
Citrate	
Alcanolamines (Aminoalcohols)	

Slurries and pastes

- *powder*
- *solvent*
- dispersant
- binder
- plasticizer
- lubricant
- others

Forming Method	Solvent	Dispersant	Binder	Plasticizer	Other Additives
			Dry Pressing		
Die compaction	—	—	<5	<2–3	<1[a]
Isostatic compaction	—	—	<5	—	—
			Slurry-Based Methods		
Slip casting	50–60	<0.5–2	<0.5–1	—	—
Tape casting	50–60	<0.5–2	10–15	5–10	<1–2[b]
			Plastic Forming Methods		
Extrusion	30–40	<0.5–2	5	1	<1–2[a]
Injection molding !!	—	—	25–30	5–10	<2–5[a]

!!

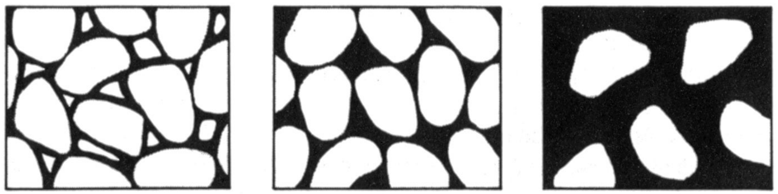
Note: The actual concentration in a given forming method can vary widely, depending on factors such as the particle size of the powder and the composition and molecular weight of the additive.

[a] Lubricant.
[b] Wetting agent; homogenizer; antifoaming agent.

Rheology of ceramic systems

system	reology	DPS
powder	elastic	0
granulated powder	elasto-plastic	<<1
paste or plastic mix	elasto-plastic	<1
slurry	viscous	>1

DPS = degree of pore saturation = volume of liquid / volume of pores



DPS < 1 DPS = 1 DPS > 1

Principles of ceramics processing, 2nd ed., J.S. Reed, J. Wiley & sons, 1995

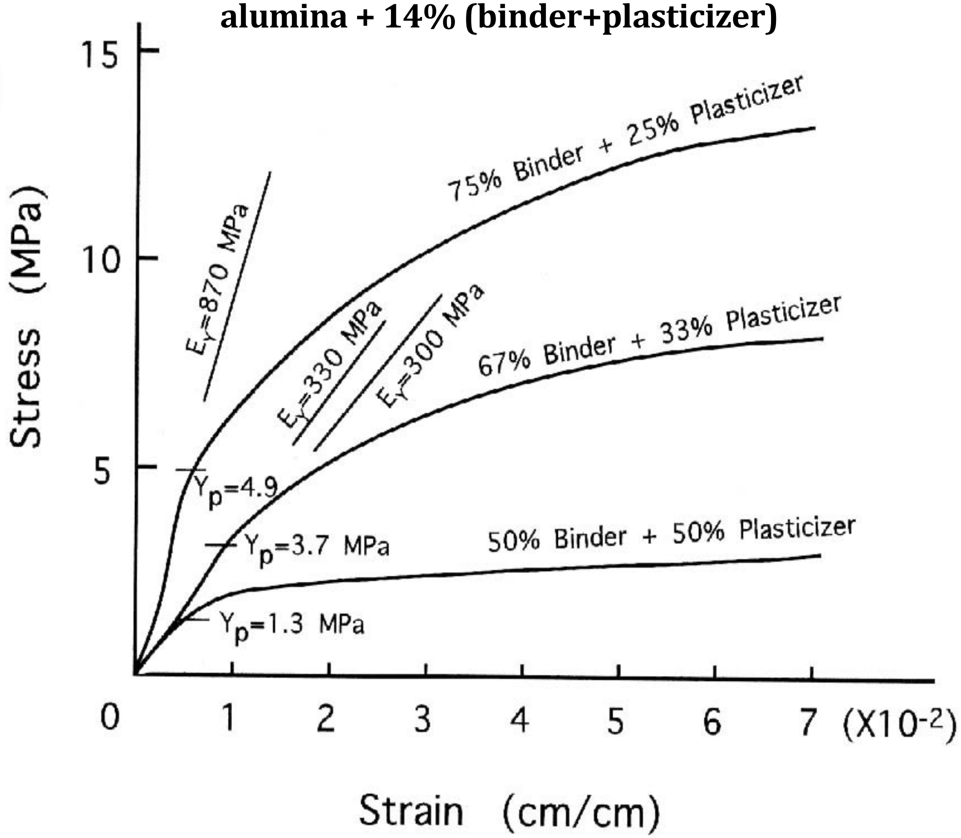
DPS < 1

elasto-plastic behaviour

- **interaction among particles** *(friction ↔ lubricant)*

energy & forces for the deformation (elastic & plastic)

tensile test
alumina + 14% (binder+plasticizer)



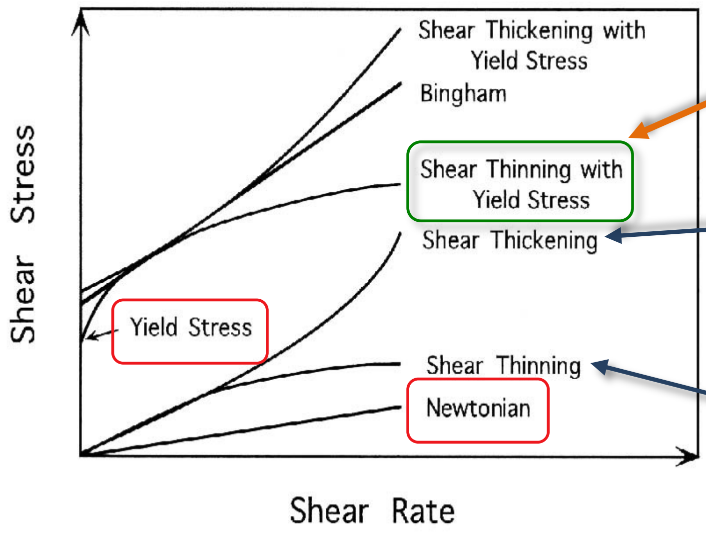
Fundamental parameters:

- *elastic modulus (elastic recovery)*
- *yield stress (deformability)*
- *maximum deformation (cracks)*

DPS ≥ 1

viscosity of the suspension

viscosity: $\eta = \dfrac{\tau}{d\gamma/dt}$



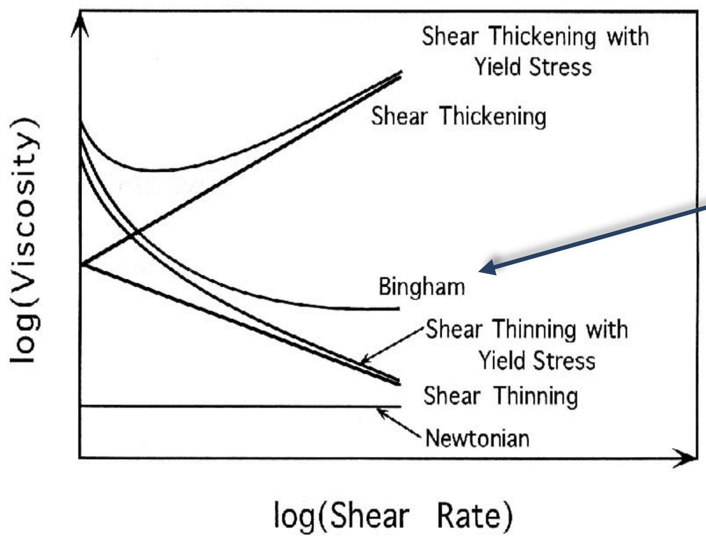
desired situation

$$\tau = K\left(\frac{d\gamma}{dt}\right)^n \qquad n>1$$

relatively concentrated suspensions

$$\tau = K\left(\frac{d\gamma}{dt}\right)^n \qquad n<1$$

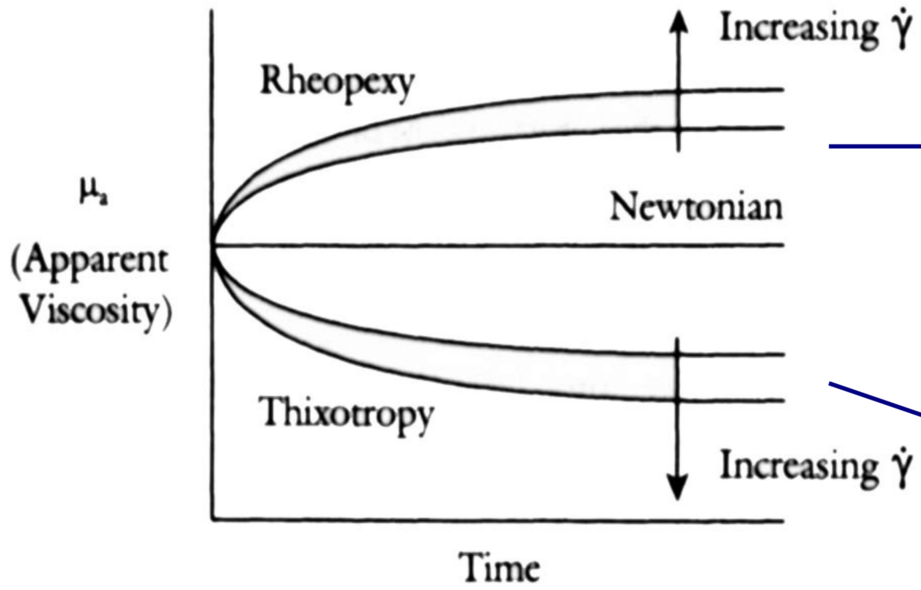
presence of large molecules or non-isometric particles with limited interaction/attraction



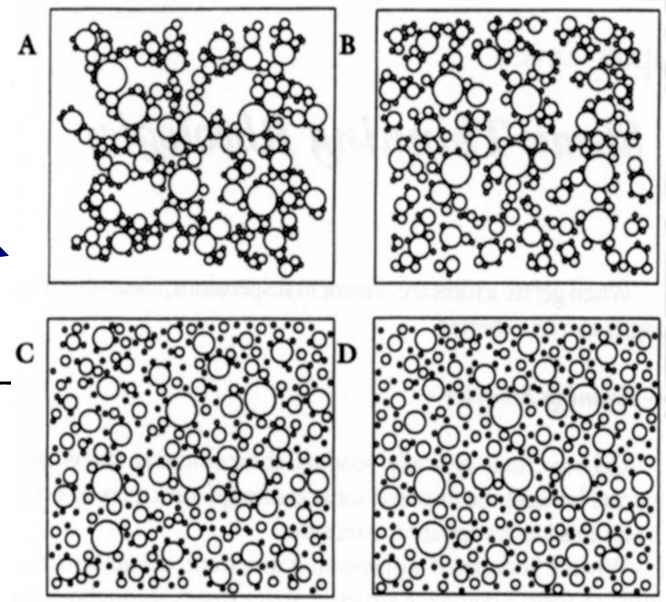
$$\tau = \tau_Y + \eta \frac{d\gamma}{dt}$$

$$\eta_{app} = \frac{\tau_Y}{d\gamma/dt} + \eta$$

particles bonded by large molecules (polyelectrolytes)

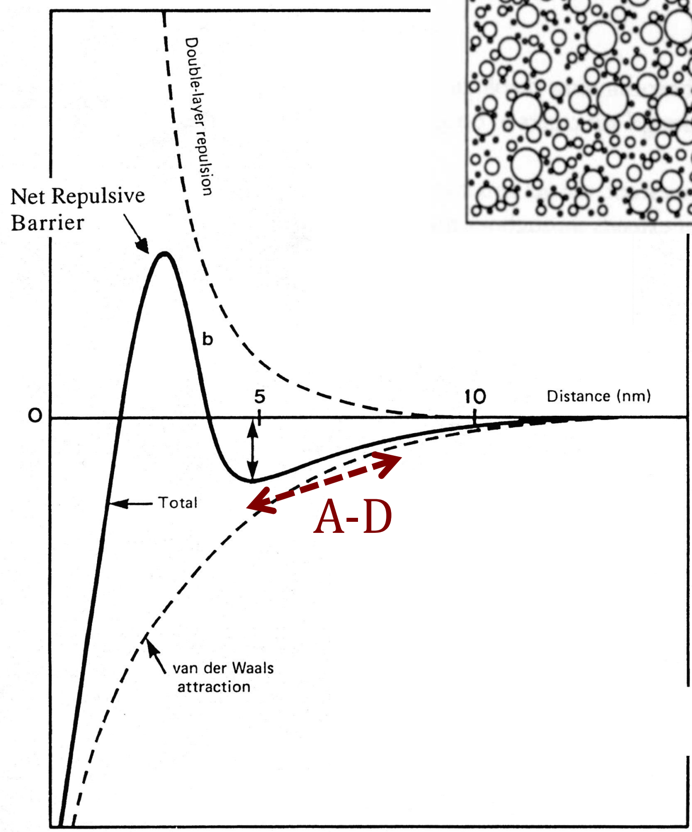


collisions & interaction among particles

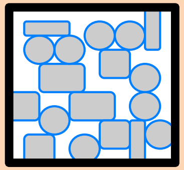
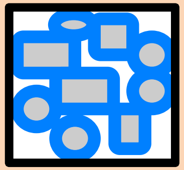
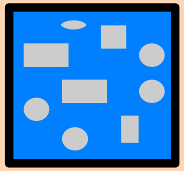


apparent viscosity:

$$\eta_a = \frac{\tau_0}{\left. d\gamma / dt \right|_0}$$



Forming technologies from powders

pressing	-	granulated powder	
extrusion, injection moulding	-	ceramic paste	
tape casting, slip casting	-	ceramic suspension	

solvent

water, organic solvent

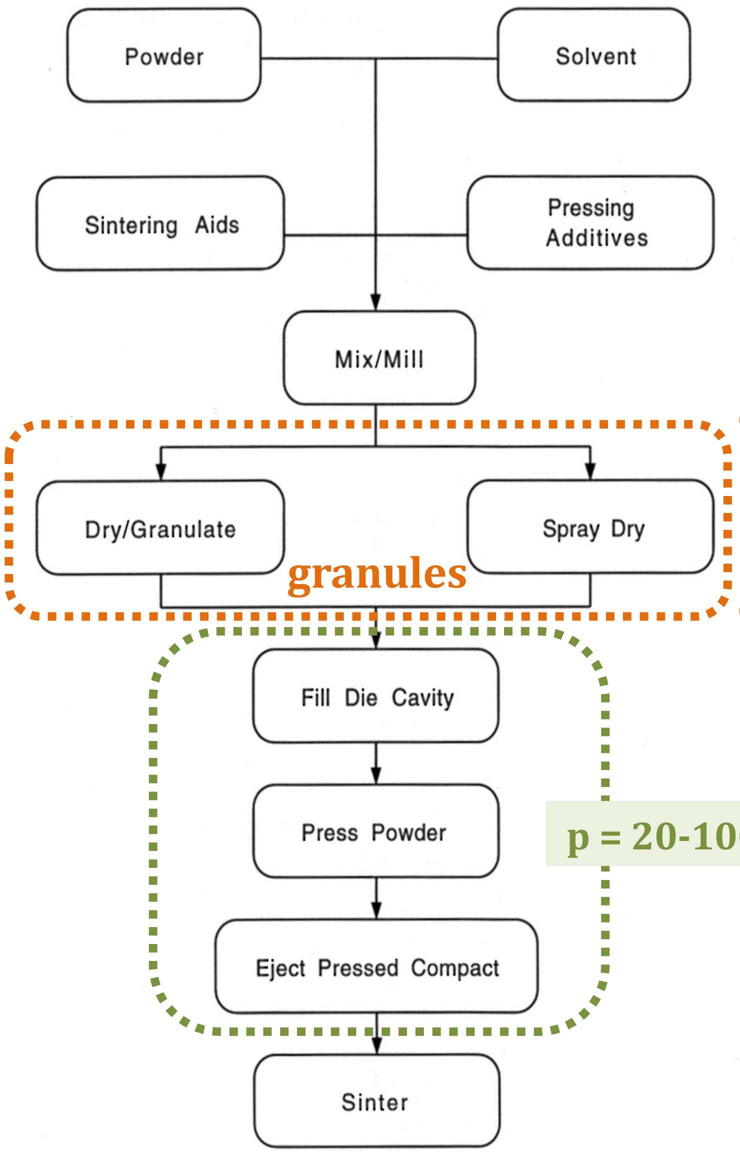
powder

additives

deflocculant, binder,
plasticizer, lubricant

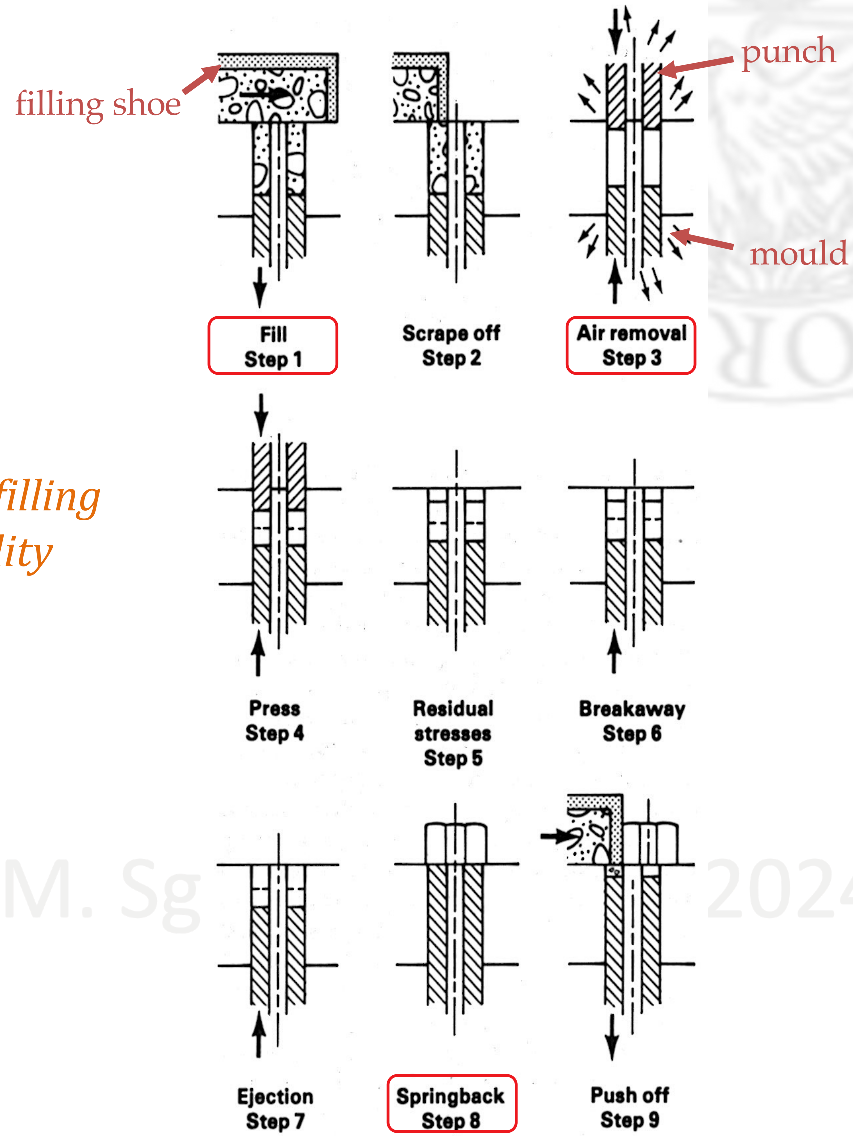
} *formability*

Pressing or die compaction



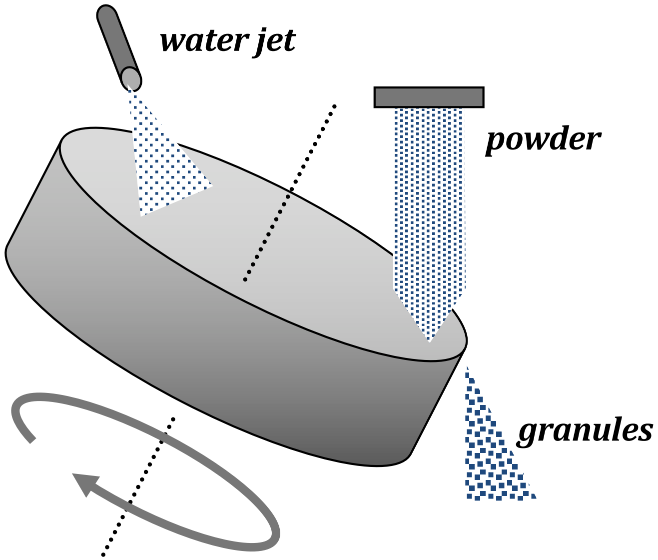
ease of flowing & filling
plastic deformability

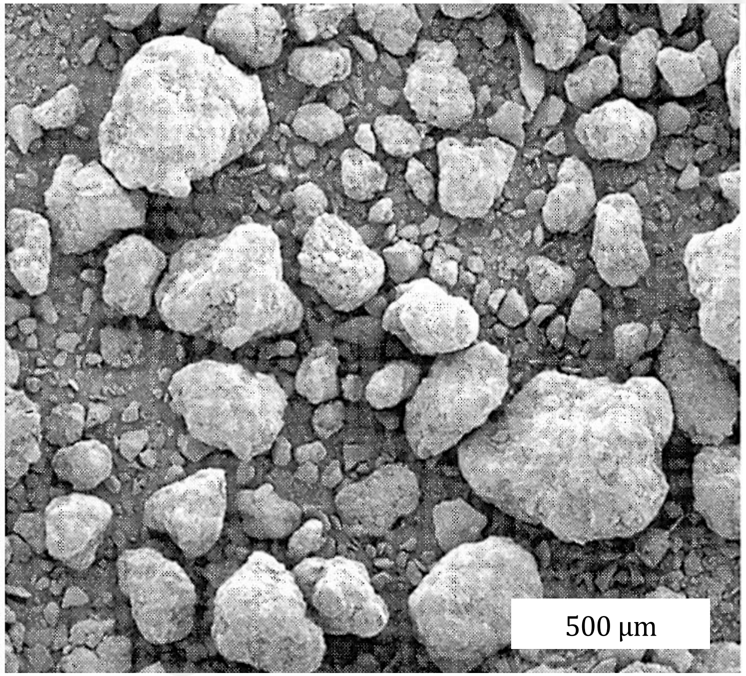
p = 20-100 MPa



Granulation

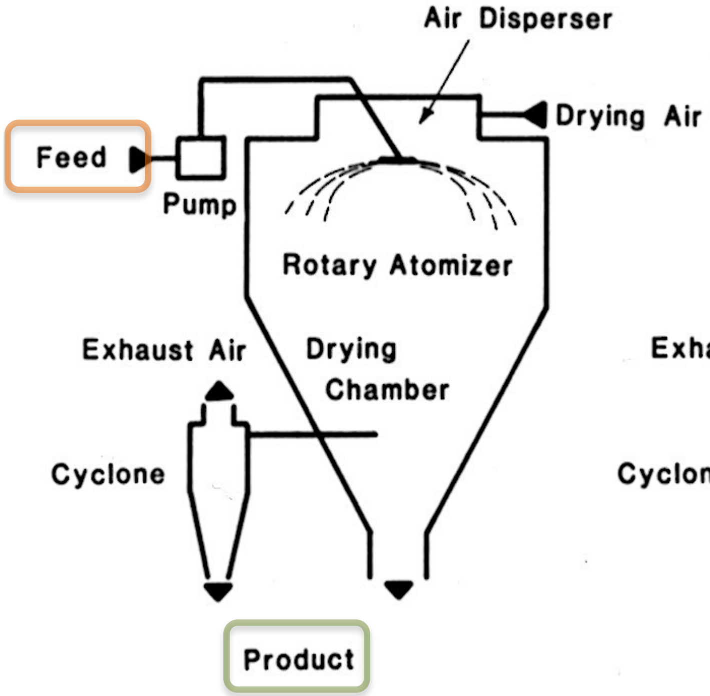
- **direct granulation (pellettization)**



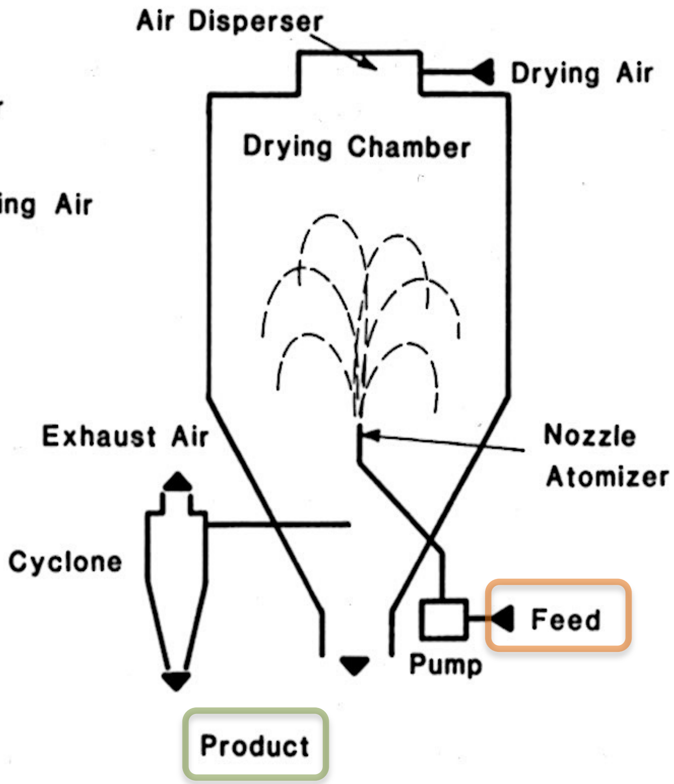


irregular shapes and distributed sizes

- **spray drying**

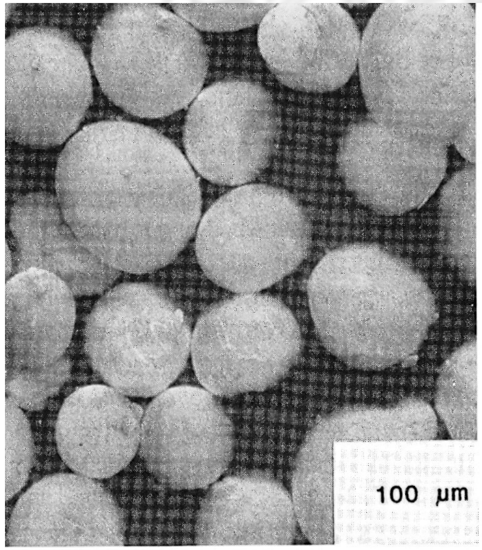


Co–current

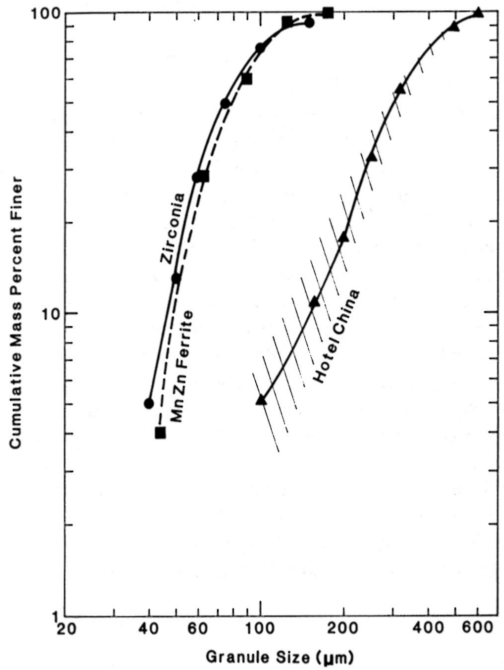


Mixed Flow

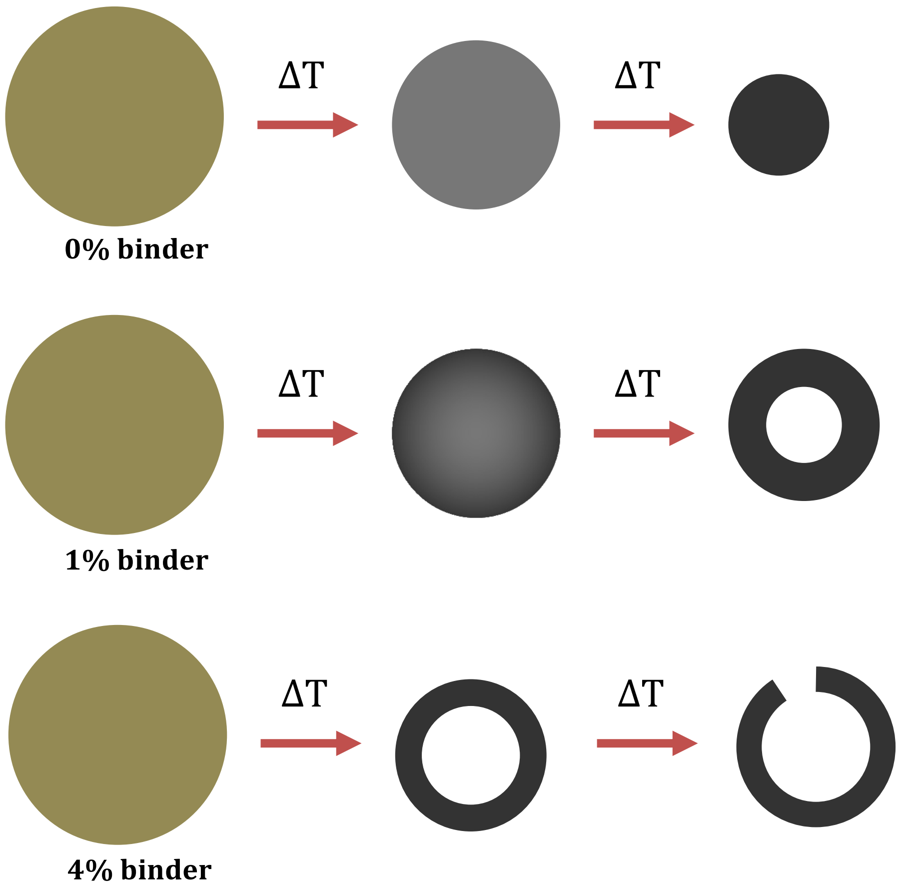
feed = suspension
product = granulated powder

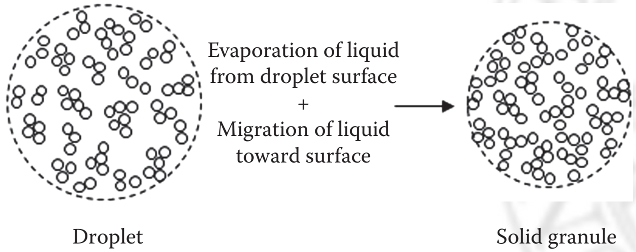


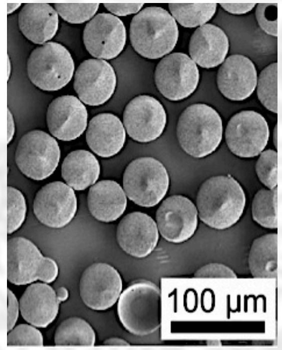
regular shapes and well distributed sizes

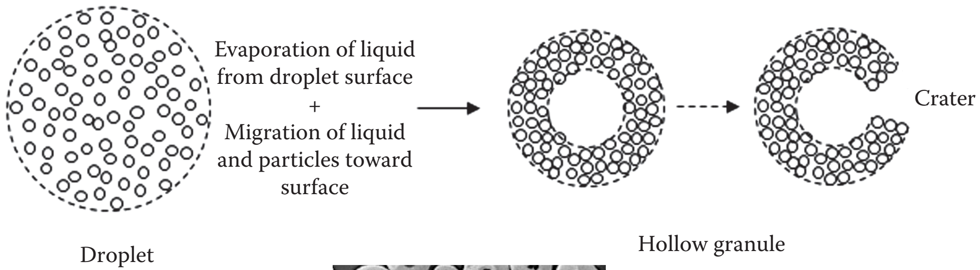


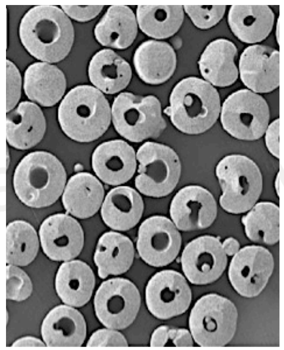
Granules shape



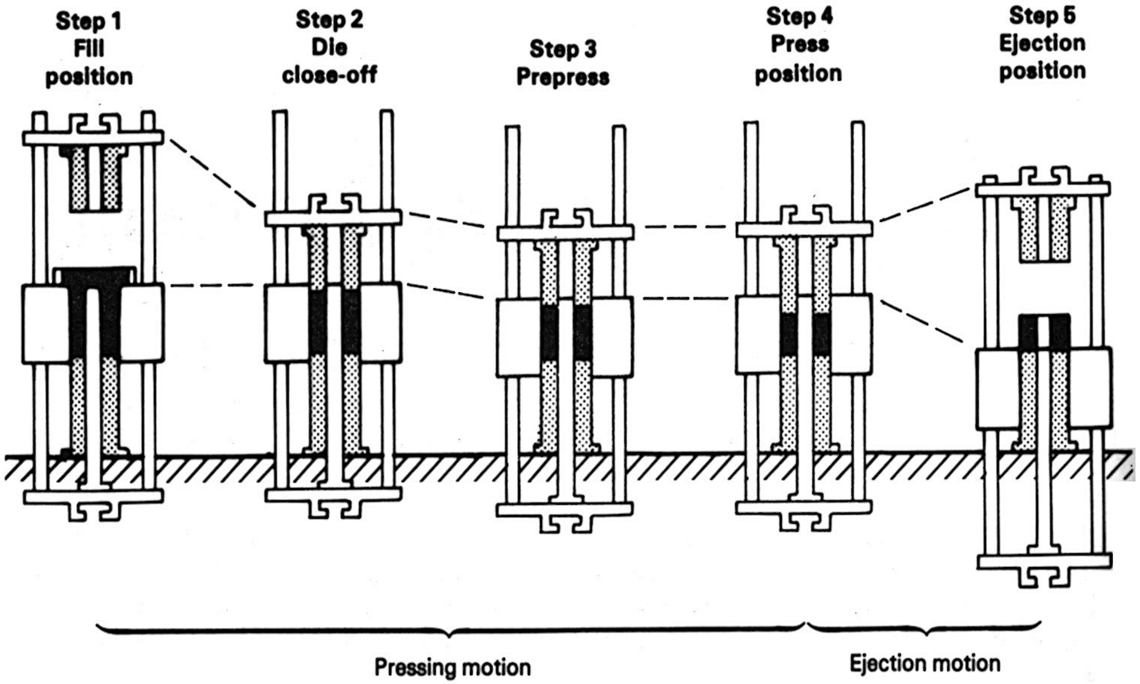


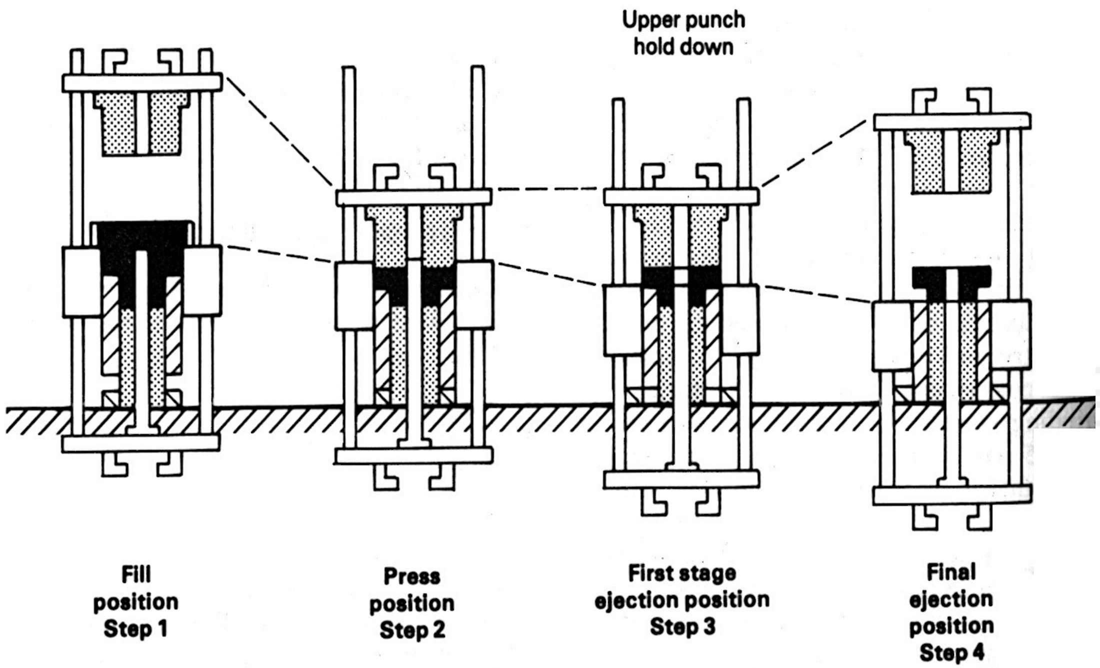






➡ ***plastic deformability during pressing***





segmented dies for complicated shapes

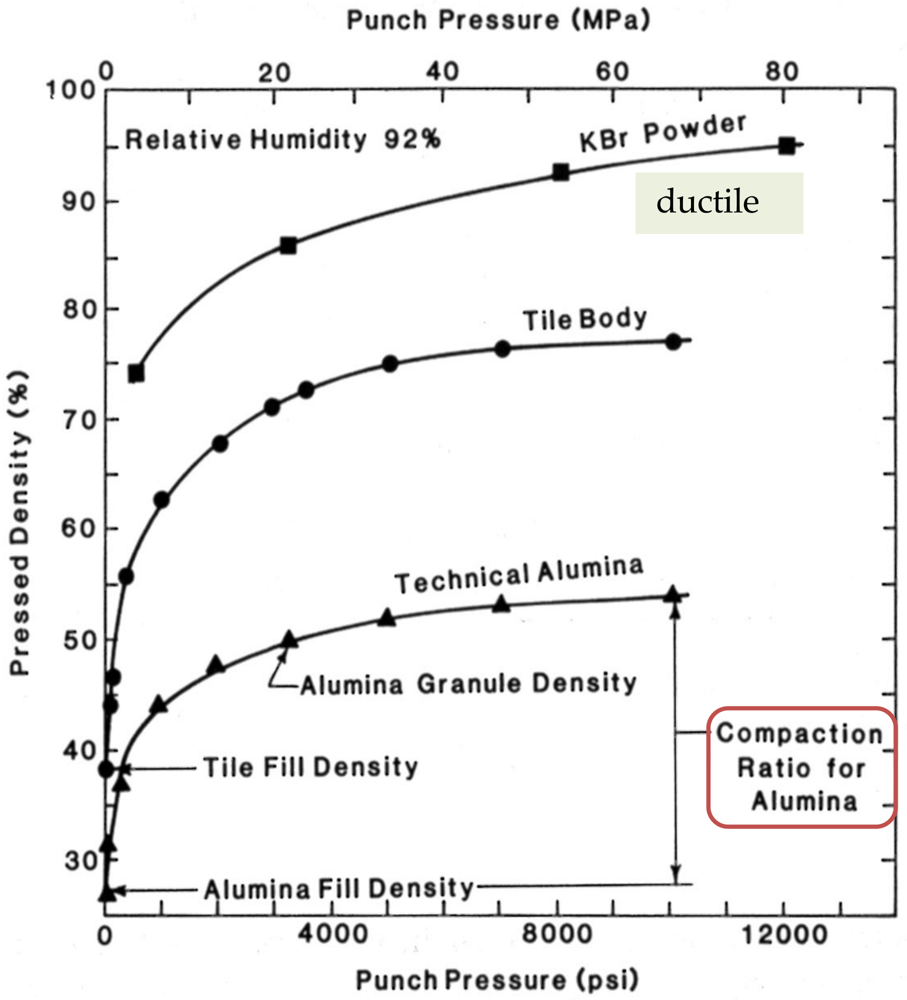
additives

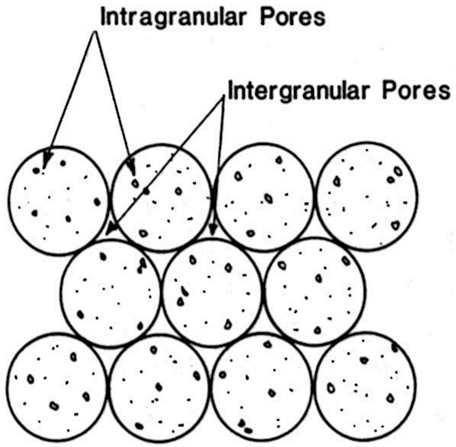
Product	Binder	Plasticizer	Lubricant
Alumina	Polyvinyl alcohol	Polyethylene glycol	Mg Stearate
96% alumina substrates	Polyethylene glycol	None	Talc, clay
Alumina spark plug insulation	Microcrystalline wax emulsion	KOH + tannic acid	Wax, talc clay
MnZn Ferrites	Polyvinyl alcohol	Polyethylene glycol	Zn Stearate
Ba Titanate	Polyvinyl alcohol	Polyethylene glycol	
Steatite	Microcrystalline wax, clay	Water	Wax, talc, clay
Ceramic tile	Clay	Water	Talc, clay
Hotel china	Clay, polysaccharide	Water	Clay
Refractories	Ca/Na lignosulfonate	Water	Stearate

granules

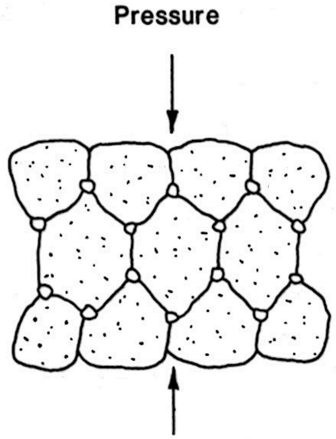
	Particle Size[a] (μm)	Organics (vol%)	Granule Size[b] (μm)	Granule Density (%)	Fill Density (%)
Alumina substrate	0.7	3.6	92	54	32
Spark plug alumina	2.0	13.3	186	55	34
Zirconia sensor	1.0	10.5	75	55	37
MnZn ferrite[c]	0.7	10.0	53	55[d]	32
MnZn ferrite[e]	0.2	8.5	56	31	18
Silicon carbide	0.3	20.4	174	45	29
		Water (vol%)			
Wall tile	9	13–21	134		38

Pressing & density

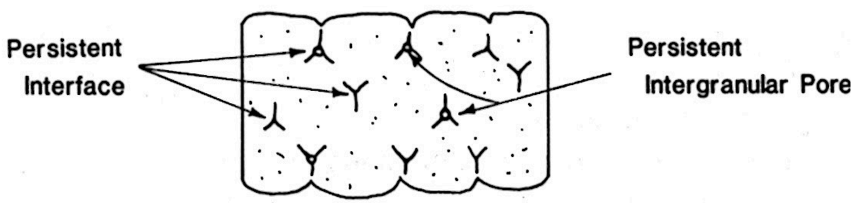




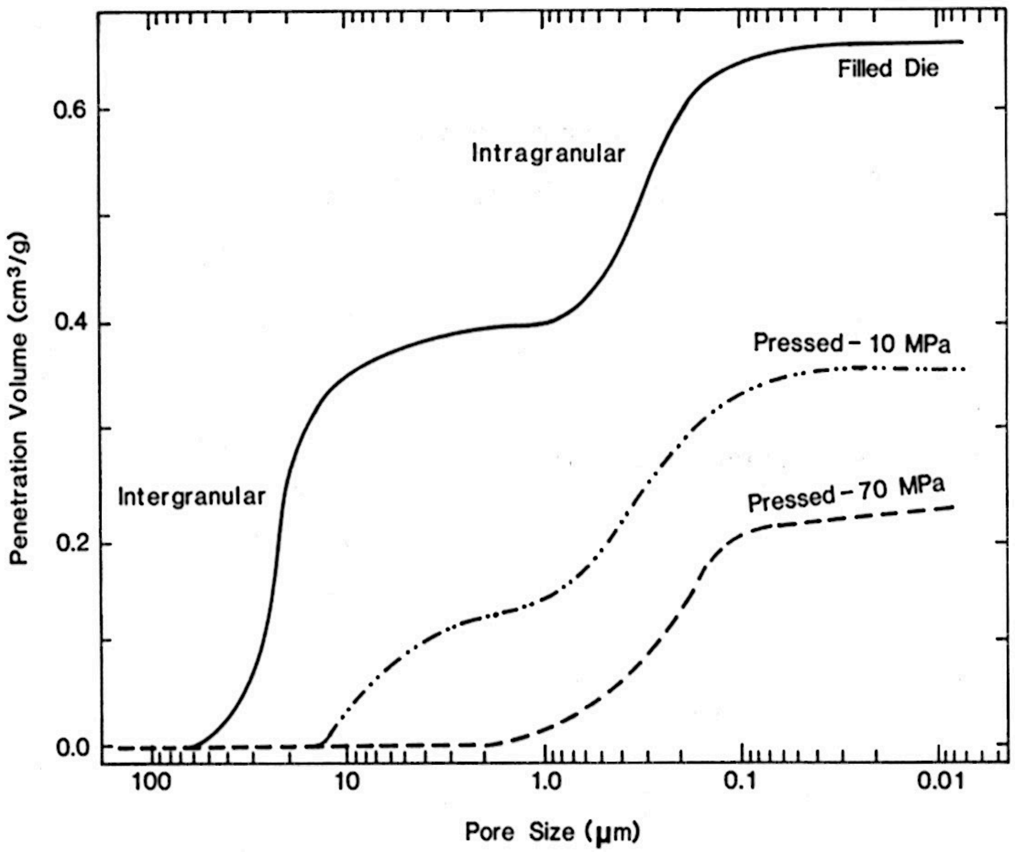
Packed Spherical Granules



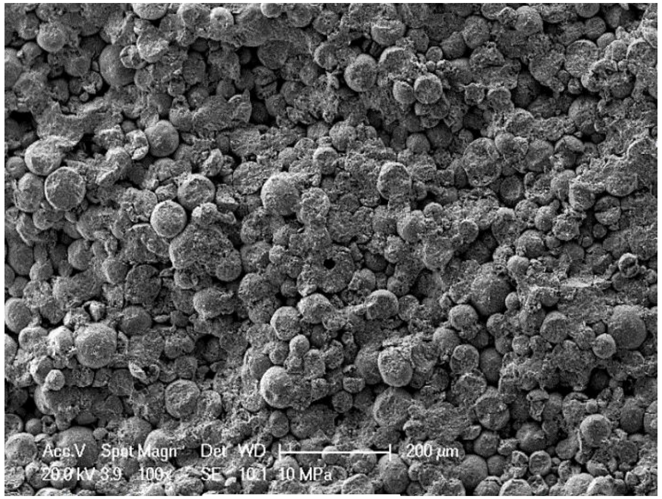
Deformed, Packed Granules



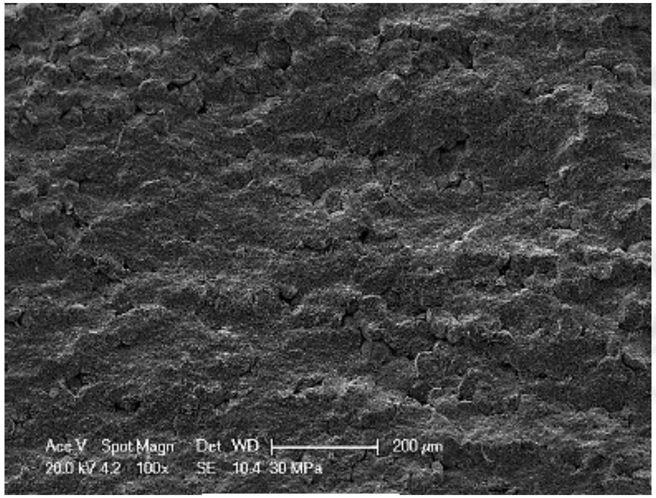
Pressed Piece



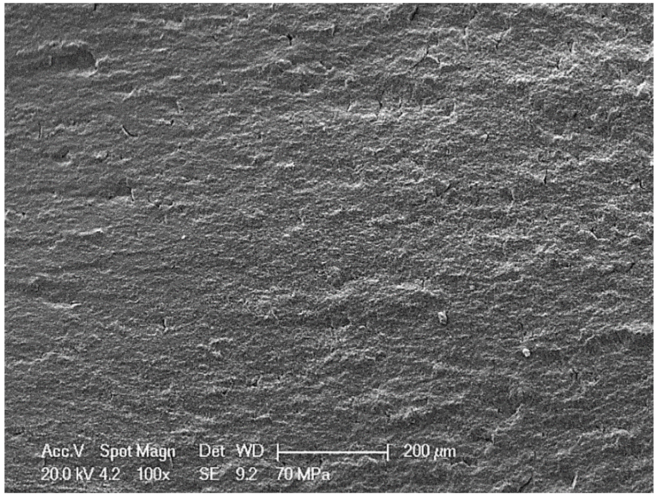
pressing of 3 mol% Y_2O_3 – 97 mol% ZrO_2 granules



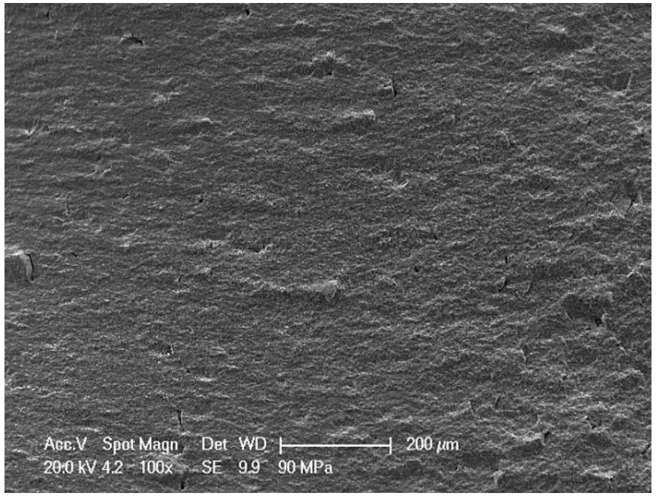
(a). 10MPa.



(b). 30MPa.



(c). 70MPa.

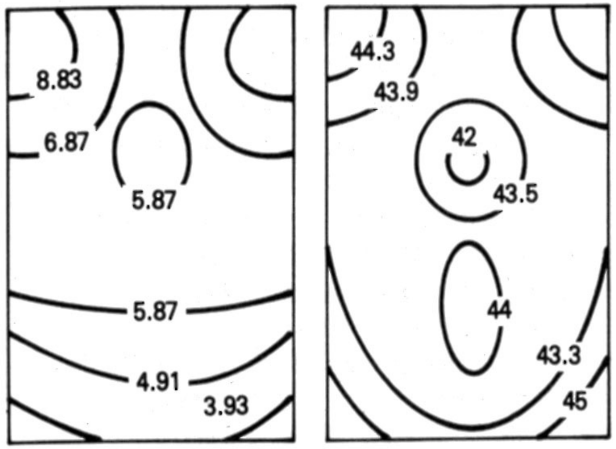


(a). 90MPa.

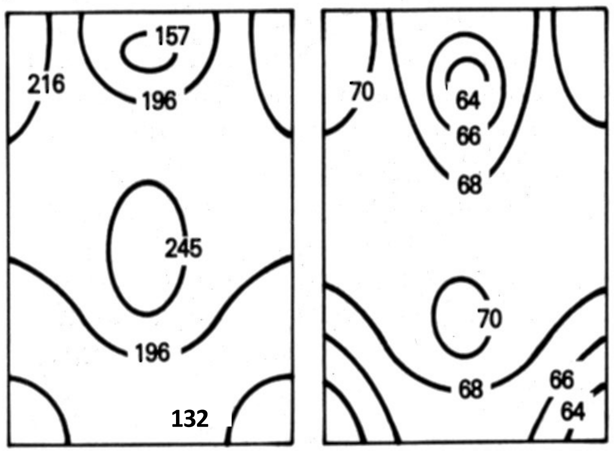
Pressure distribution

without lubricant

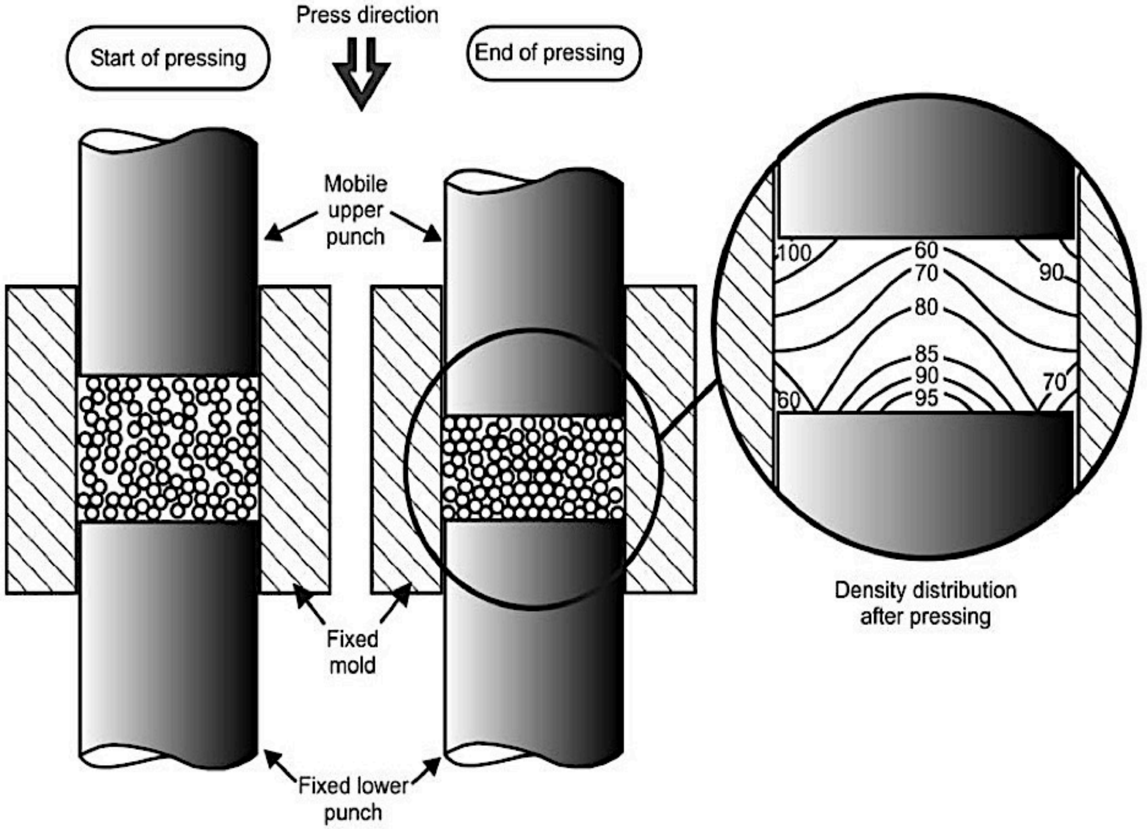
p = 8.83 MPa



p = 200 MPa



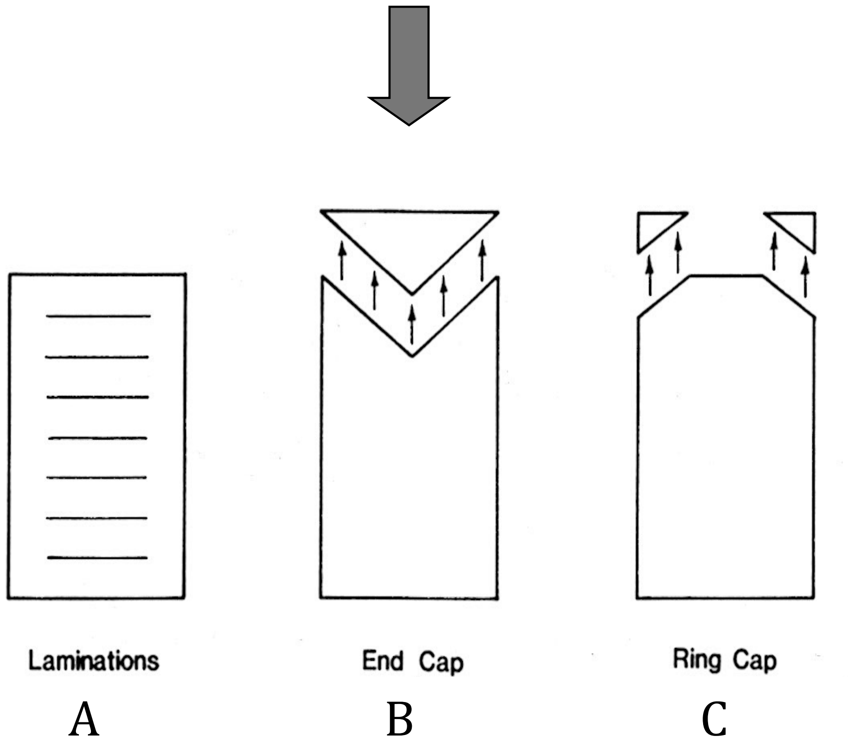
pressure (MPa) density (%)

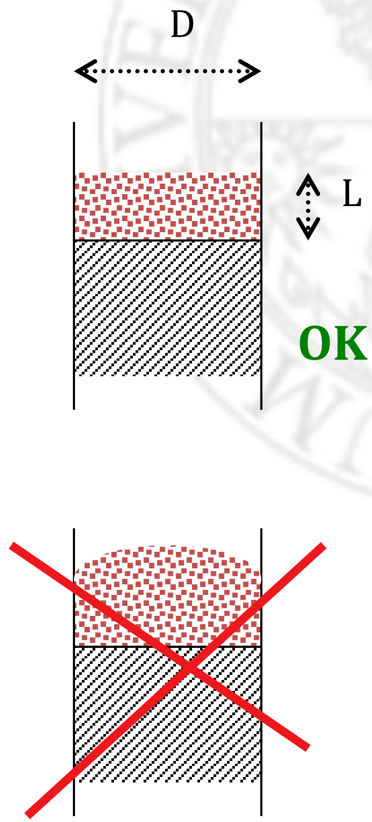


density gradients → differential springback → failures!!

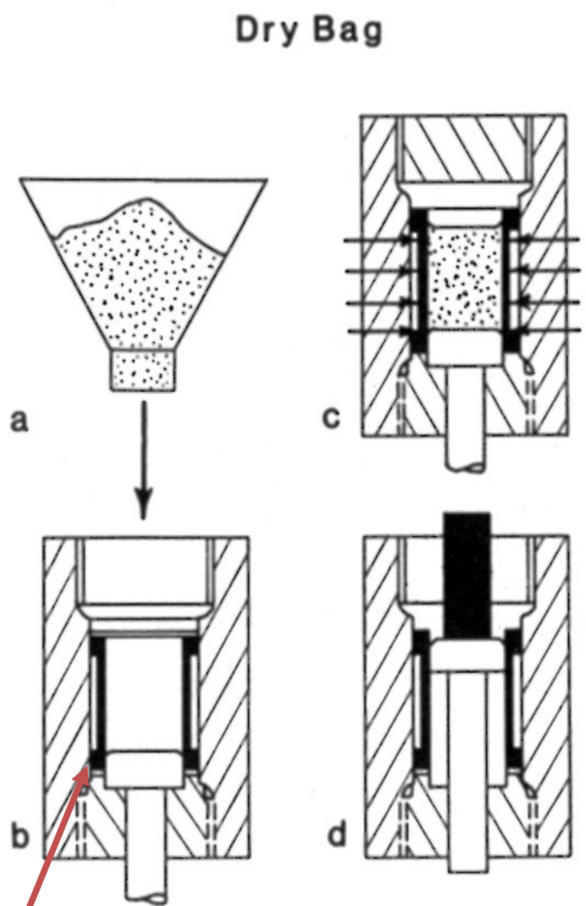
density gradients:

- absence of lubricant (B, C)
- uneven filling (B)
- L/D > 2 (A, B)
- too high compaction (*spingback*) (A, B, C)



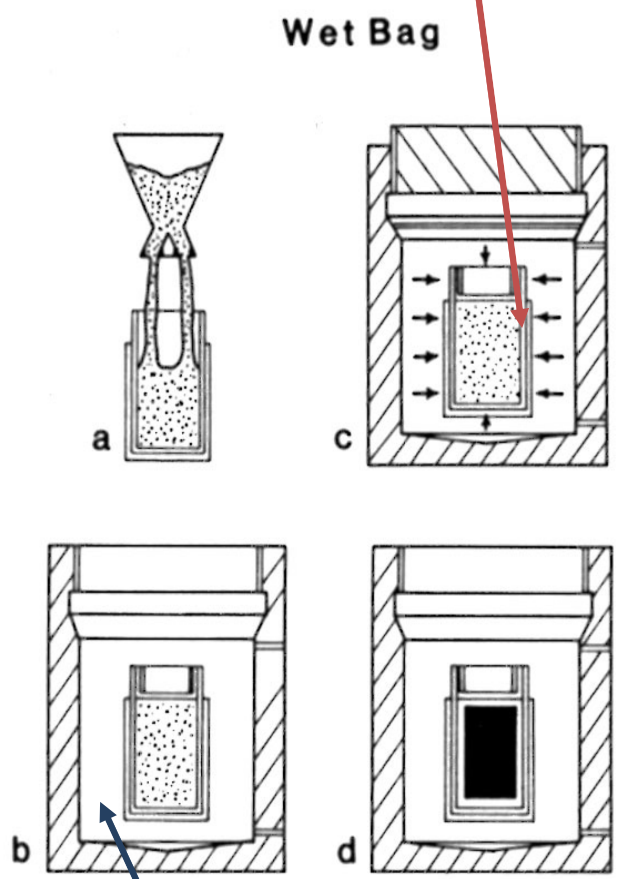


Cold isostatic pressing



p ≤ 100 MPa

flexible cylinder (siliconic rubber)



p ≤ 500 MPa

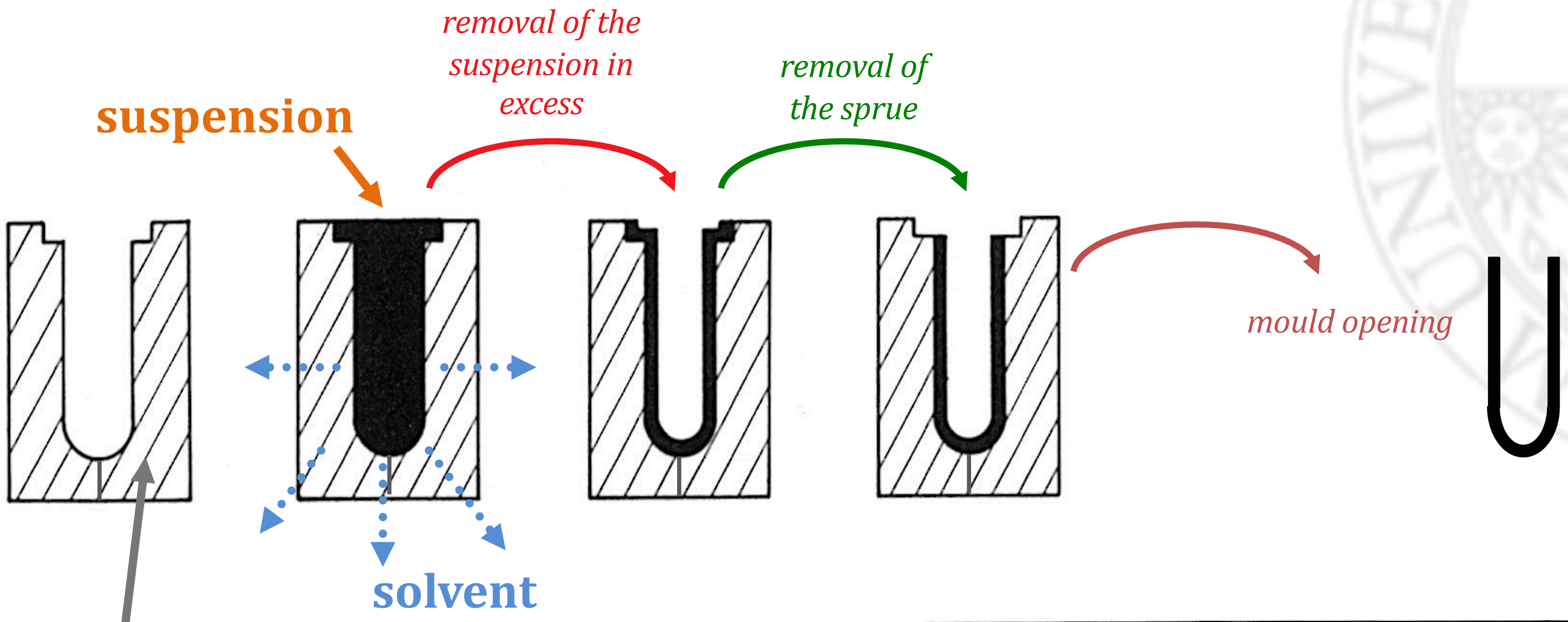
flexible mould (siliconic rubber)

liquid (oil)

↑ limited pressure gradients

↓ limited geometric shapes

Slip casting



porous mould

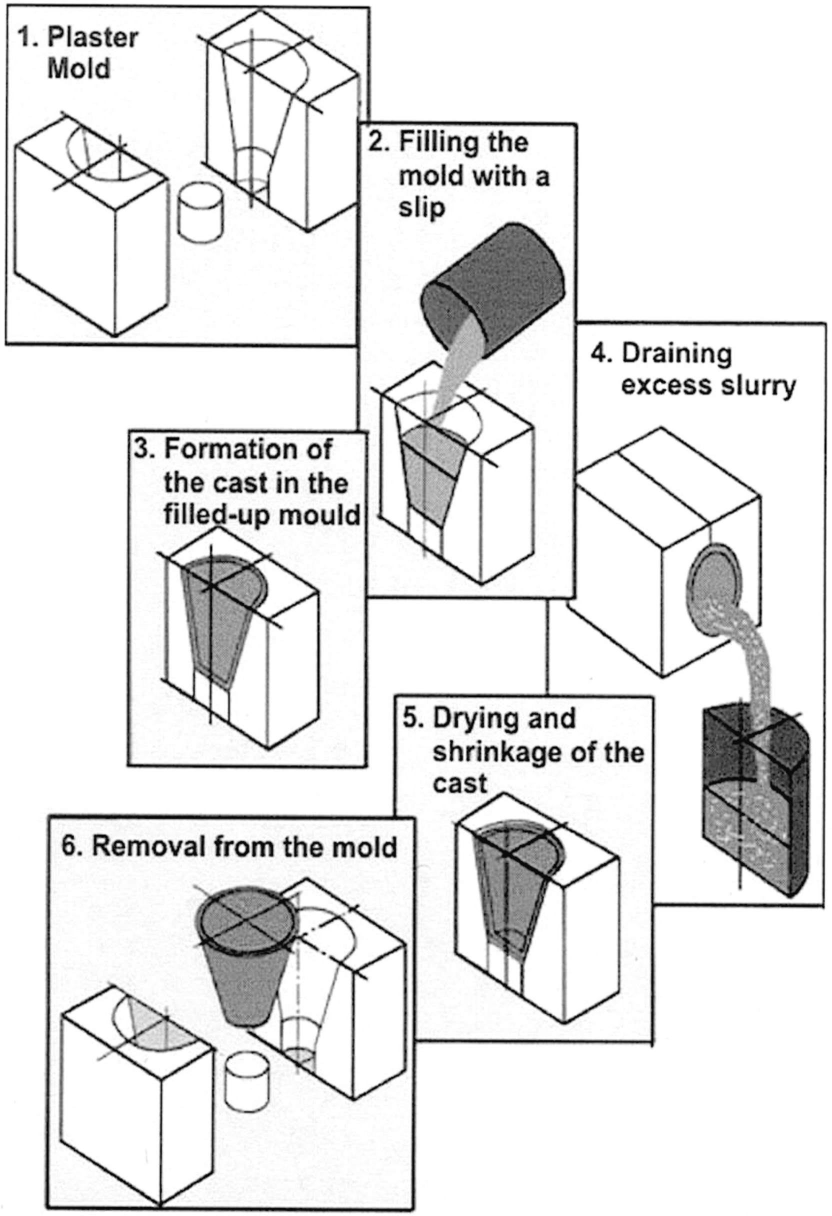
- *plaster of Paris - $CaSO_4 \cdot 2H_2O$ –*
- *polymeric resin*

porosity = 40-50%

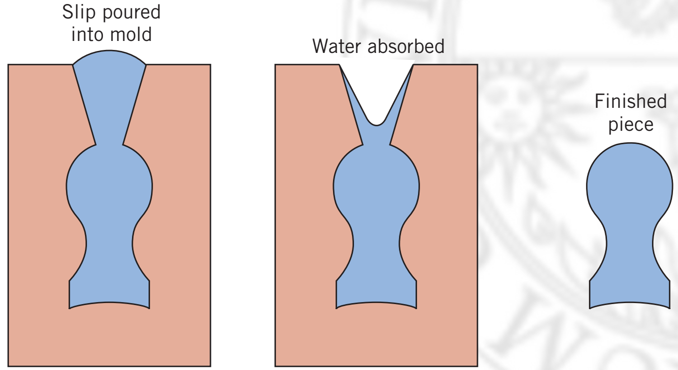
max pore size ≈ 5 µm

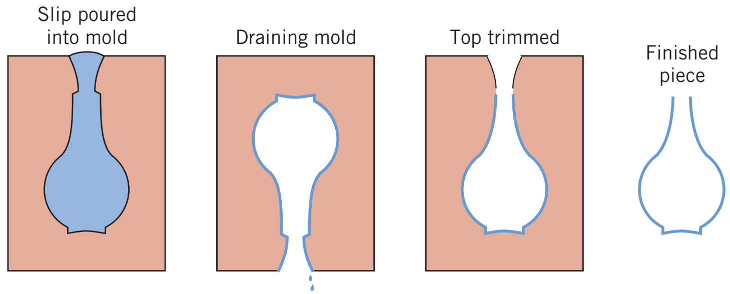
Material	Concentration (vol%) Porcelain	Alumina
Alumina		40–50
Silica	10–15	
Feldspar	10–15	
Clay	15–25	
Water	45–60	50–60
	100 vol%	100 vol%
Processing additives (wt%[a])		
Deflocculant		
Na silicate	<0.5	
NH_4 polyacrylate		0.5–2
Na citrate		0.0–0.5
Coagulant		
$CaCO_3$	<0.1	
$BaCO_3$	<0.1	
Binder		
Na carboxymethylcellulose		0.0–0.5

Principles of ceramics processing, 2nd ed., J.S. Reed, J. Wiley & sons, 1995



bulk bodies





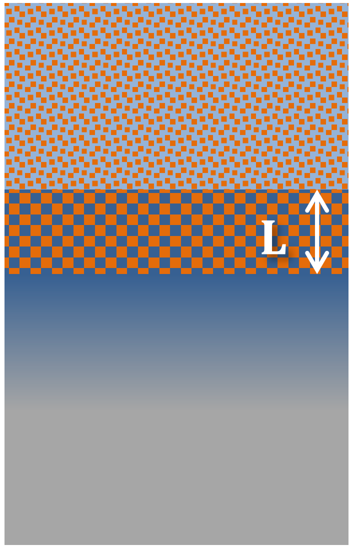
hollow bodies


t = 0


t ≈ 2 min


t ≈ 2 h

Formation of the “cake”



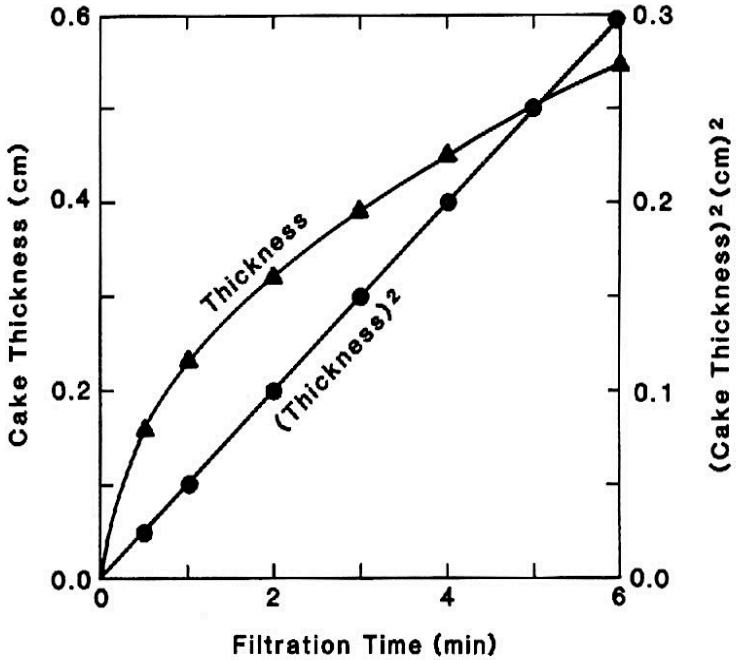
suspension (DPS >1)

consolidated layer (DPS =1)

wet

mould

dry



permeability of the consolidated layer

pressure (capillar + external)

$$L = \left[\frac{2 k_m P t}{\alpha \eta}\right]^{0.5}$$

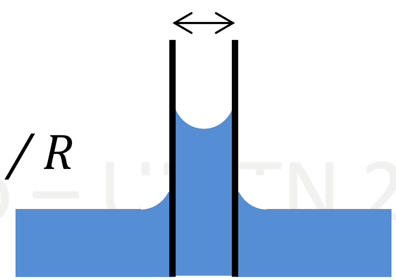
viscosity of the liquid

solid fraction in the consolidated layer

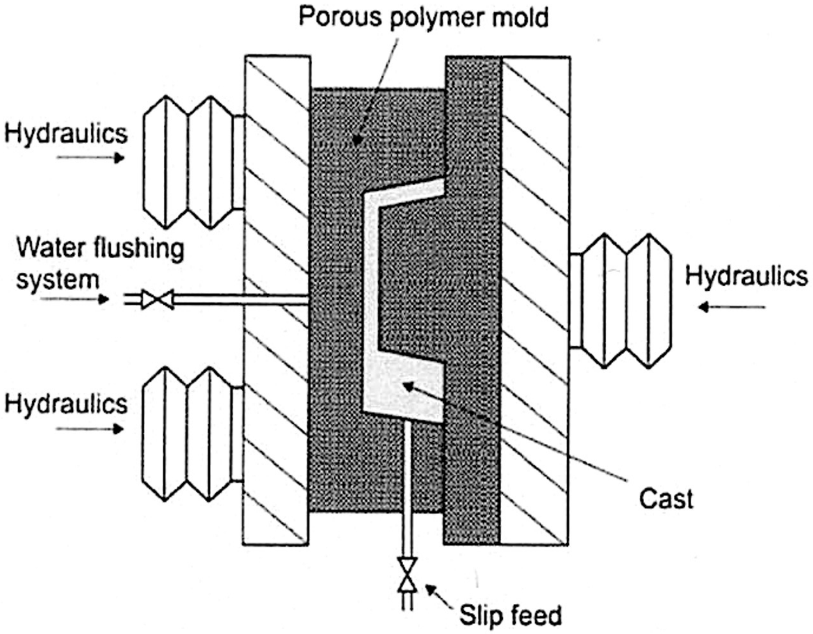
$$\alpha = \frac{V_c}{V_s} - 1$$

solid fraction in the suspension

$$P_{cap} = 2\, \gamma_{LV} \cos\theta \,/\, R$$

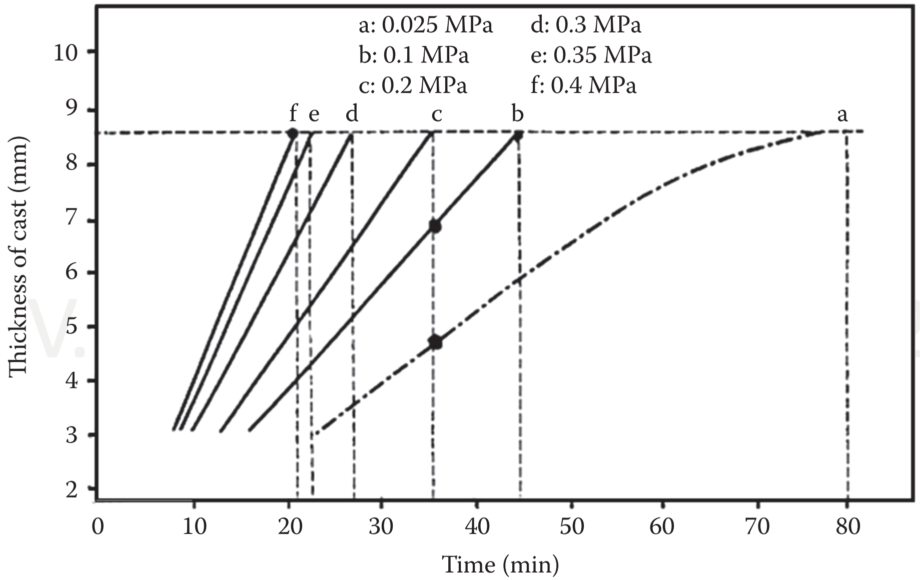


Pressure casting

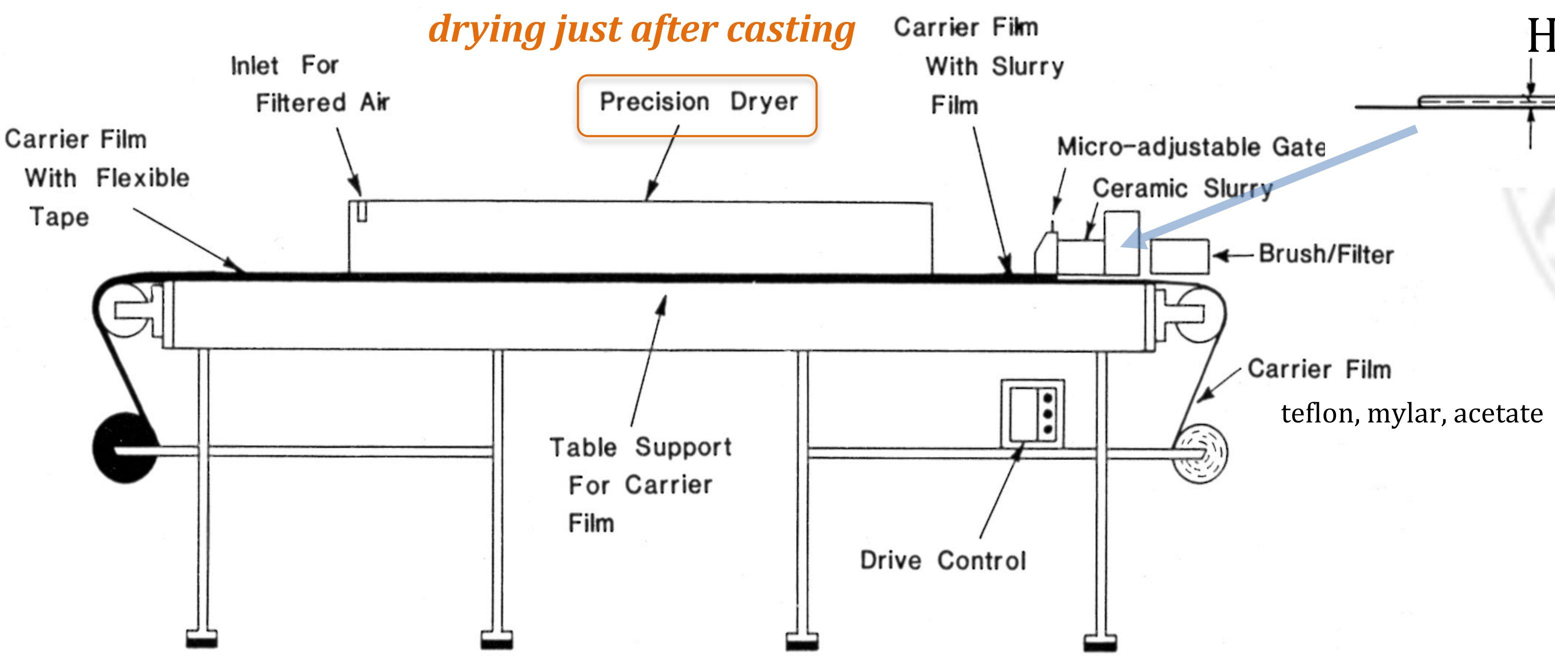


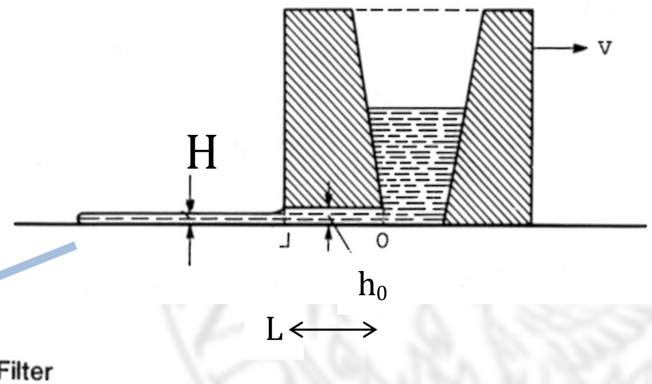
+0.5 wt% PVA binder

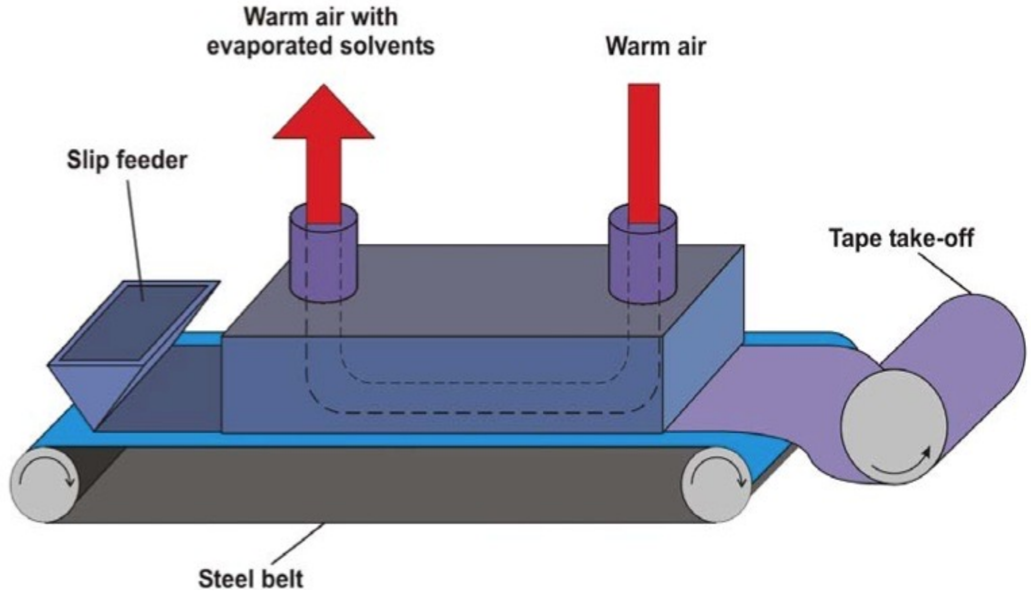
Pressure (kPa)	Casting Rate (mm^2/min) Porcelain	Alumina	Alumina	Zirconia
140	2.0	2.4	0.3	
280	3.1	4.9		
560	5.2	10		0.1
1400		25		0.3
2800		50		0.8



Tape casting





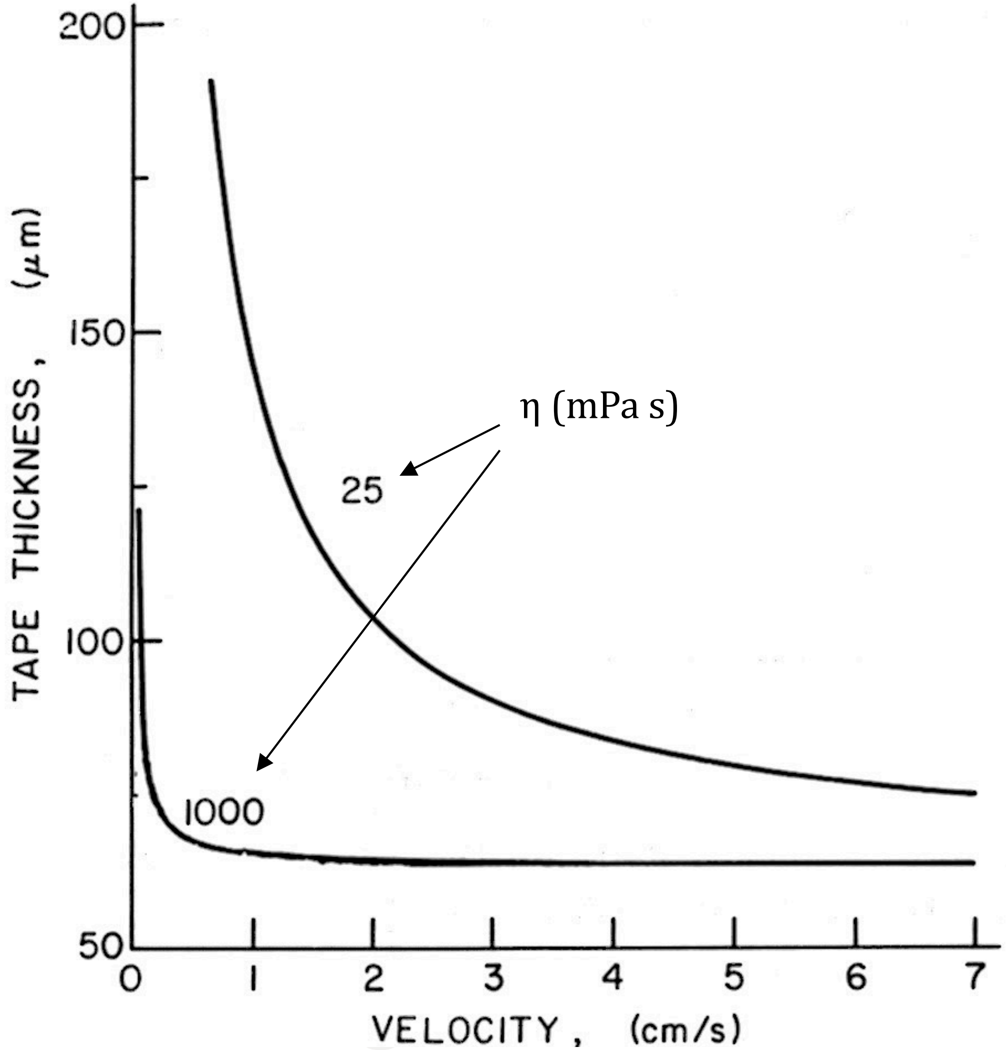


pressure (hydrostatic)

$$H = AD_r\, h_0 \left[1 + \frac{h_0^2 P}{6\eta\, v L} \right]$$

viscosity of the suspension

density of the suspension / density of the dry layer

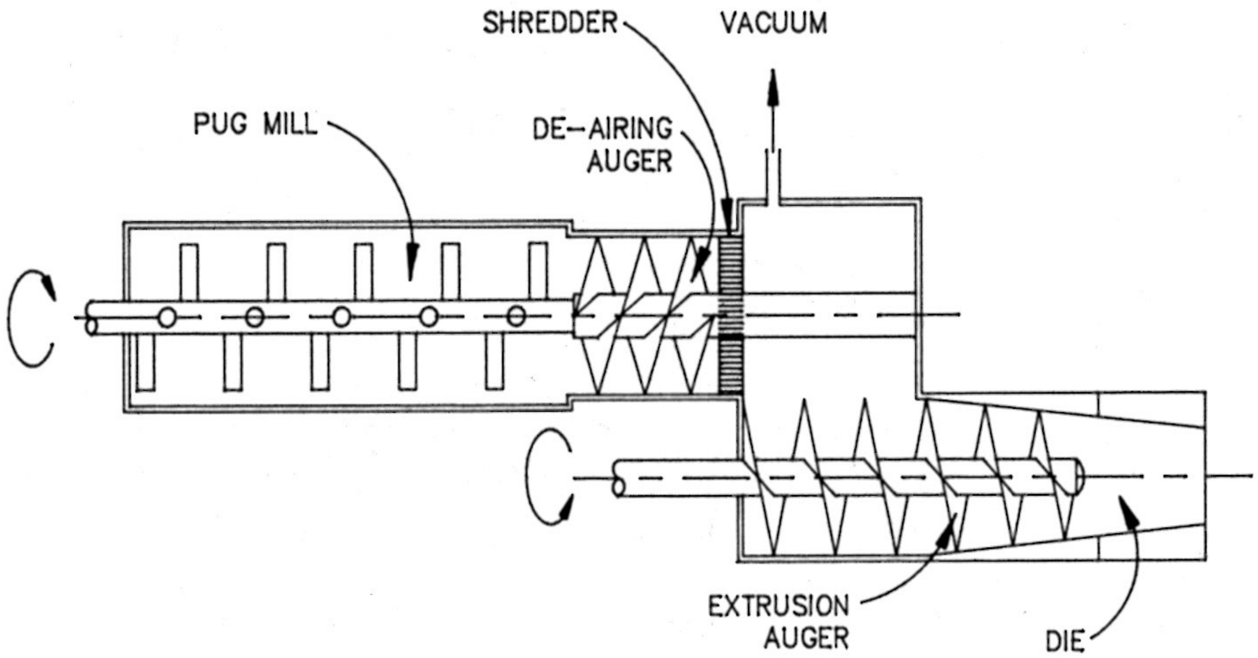


suspensions

Al_2O_3 powder	Matrix	40 -50
MgO powder	Grain growth inhibitor	0,5 - 3
Destilled water	Solvent	30 - 50
Polyvinylacetate	Binder	5 - 10
Polyethylenglykol	Plasticizer	0,5 - 3
Dibutylphthalate	Plasticizer	0,5 - 3
Arylic sulphuric acid	Deflocculant	0,1 - 0,3
Octylphenoxyethanol	Wetting agent	0,1 - 0,3

SiC	Matrix	40 – 50
Trichlorethylen	Solvent	15 - 25
Ethylalkohol	Solvent	15 - 25
Polyvinylbutyral	Binder	5 - 10
Polyethylenglykol	Plasticizer	0,5 - 3
Octylphthalat	Plasticizer	0,5 - 3
Menhaden fish oil	Deflocculant	0,5 – 3

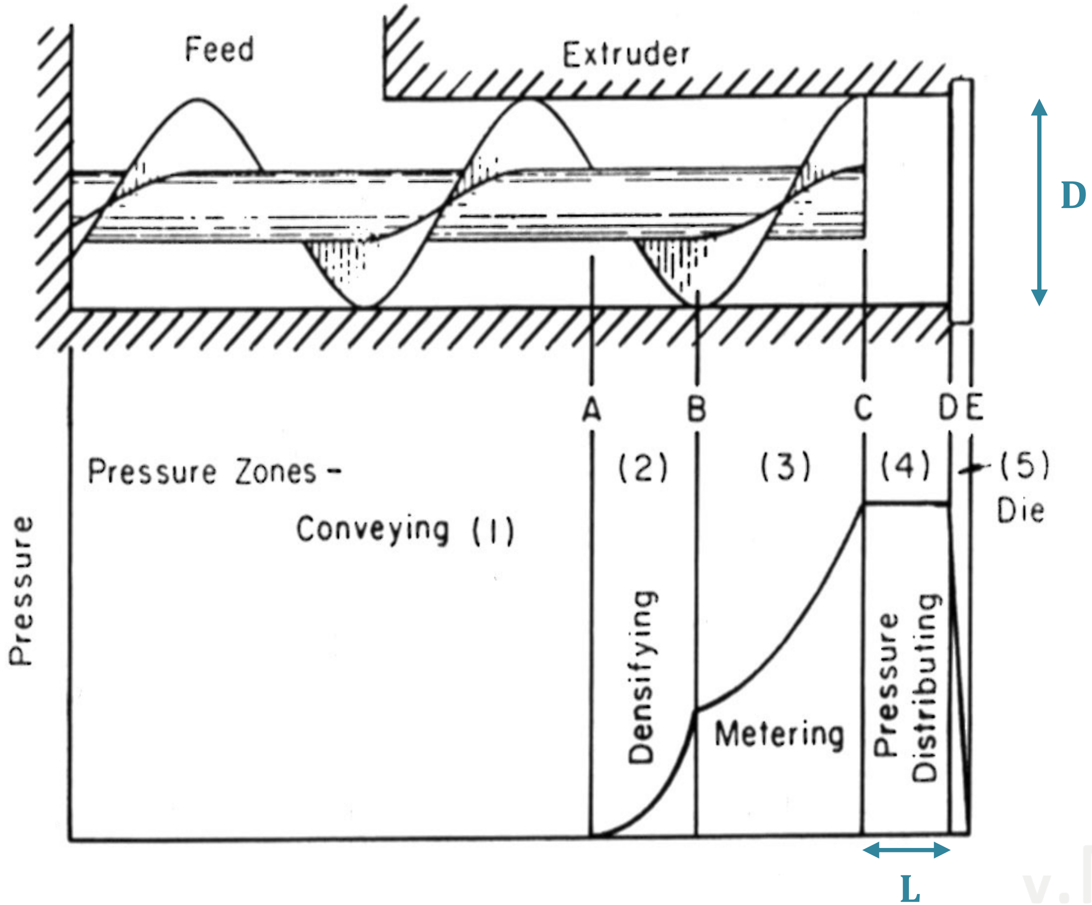
Extrusion



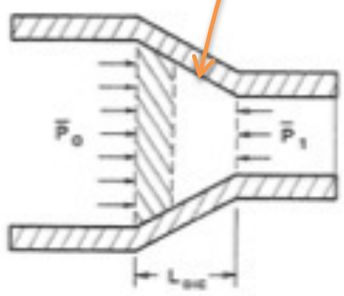
vol%

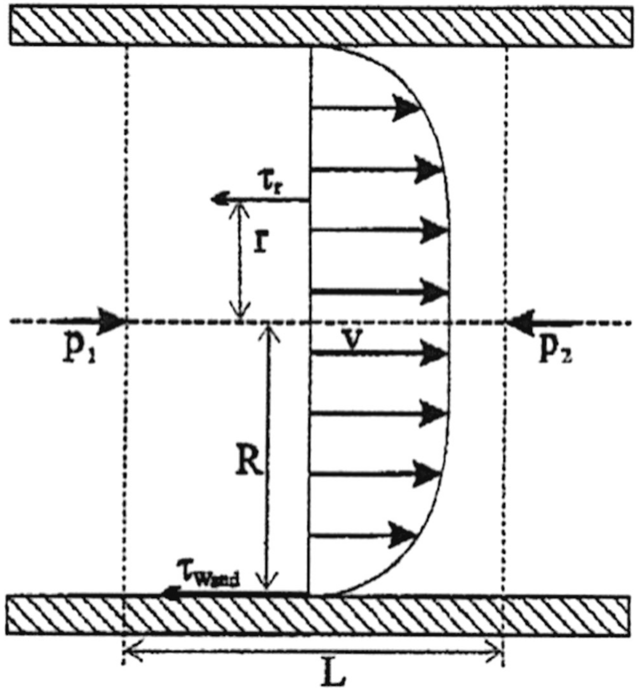
Silicon Carbide		High Alumina		Electrical Porcelain	
Silicon carbide	50	Alumina	46	Quartz powder	16
Hydroxyethyl cellulose	6	Ball clay	4	Feldspar	16
Water	42	Methyl cellulose	2	Kaolin	16
Polyethylene glycol	2	Water	48	Ball clay	16
				Water	36
				$CaCO_3$	<1

Pressure distribution and gradients



ΔP increases with
- absence of lubricant
- absence of plasticizer
- extrusion average velocity
- entrance angle

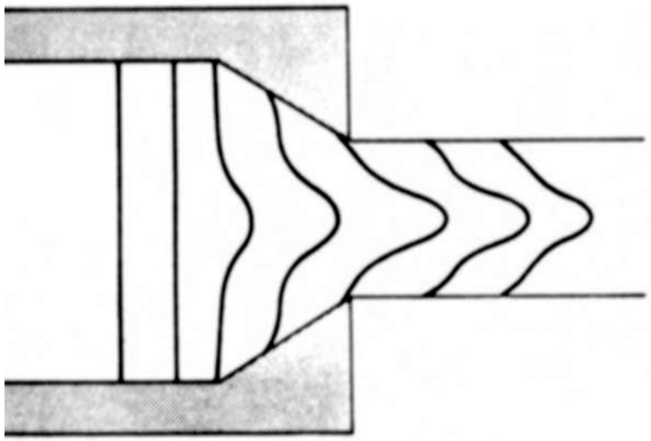


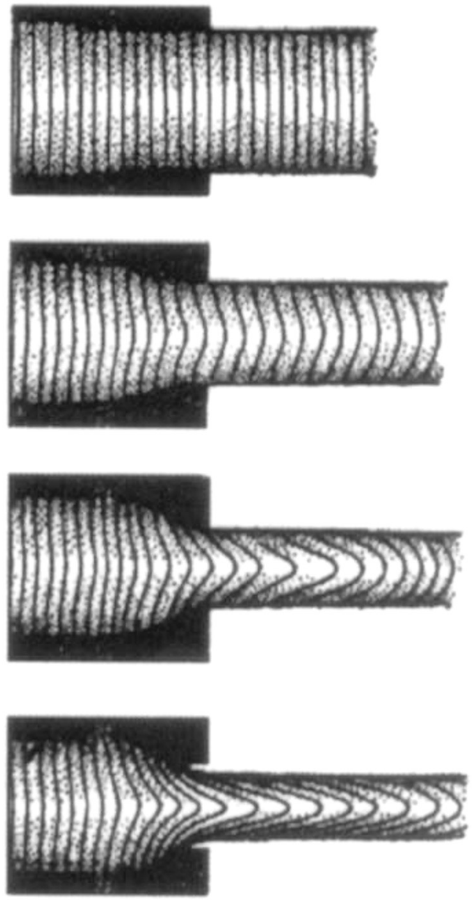


shear stresses

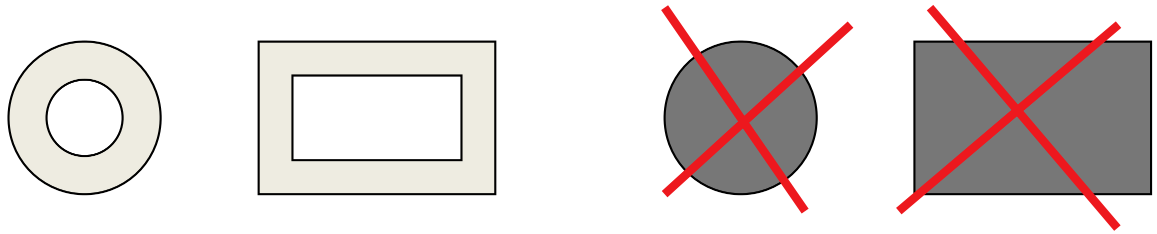
$$\tau = \frac{r\,(P_1 - P_2)}{2L}$$

flow velocity gradient

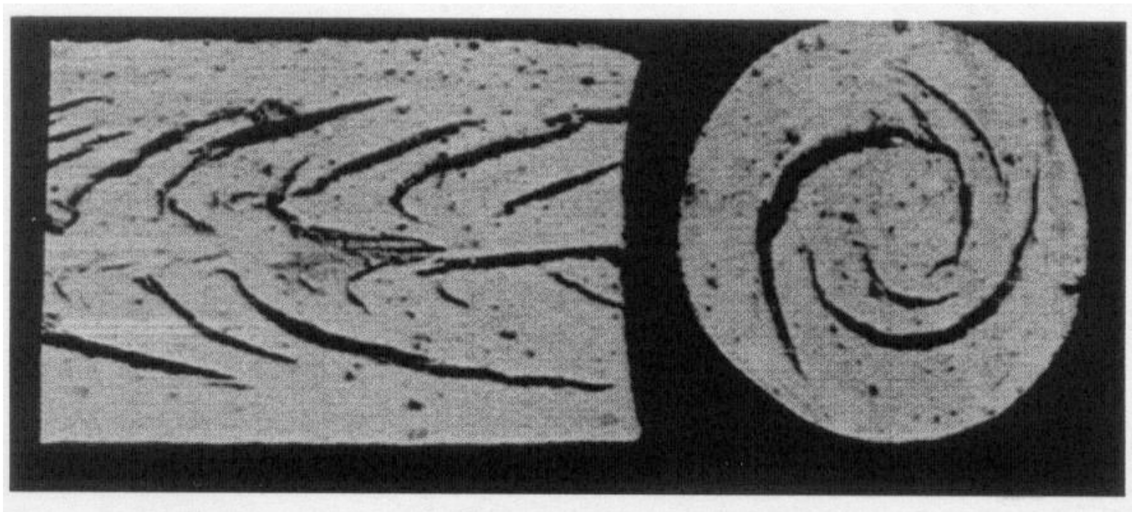




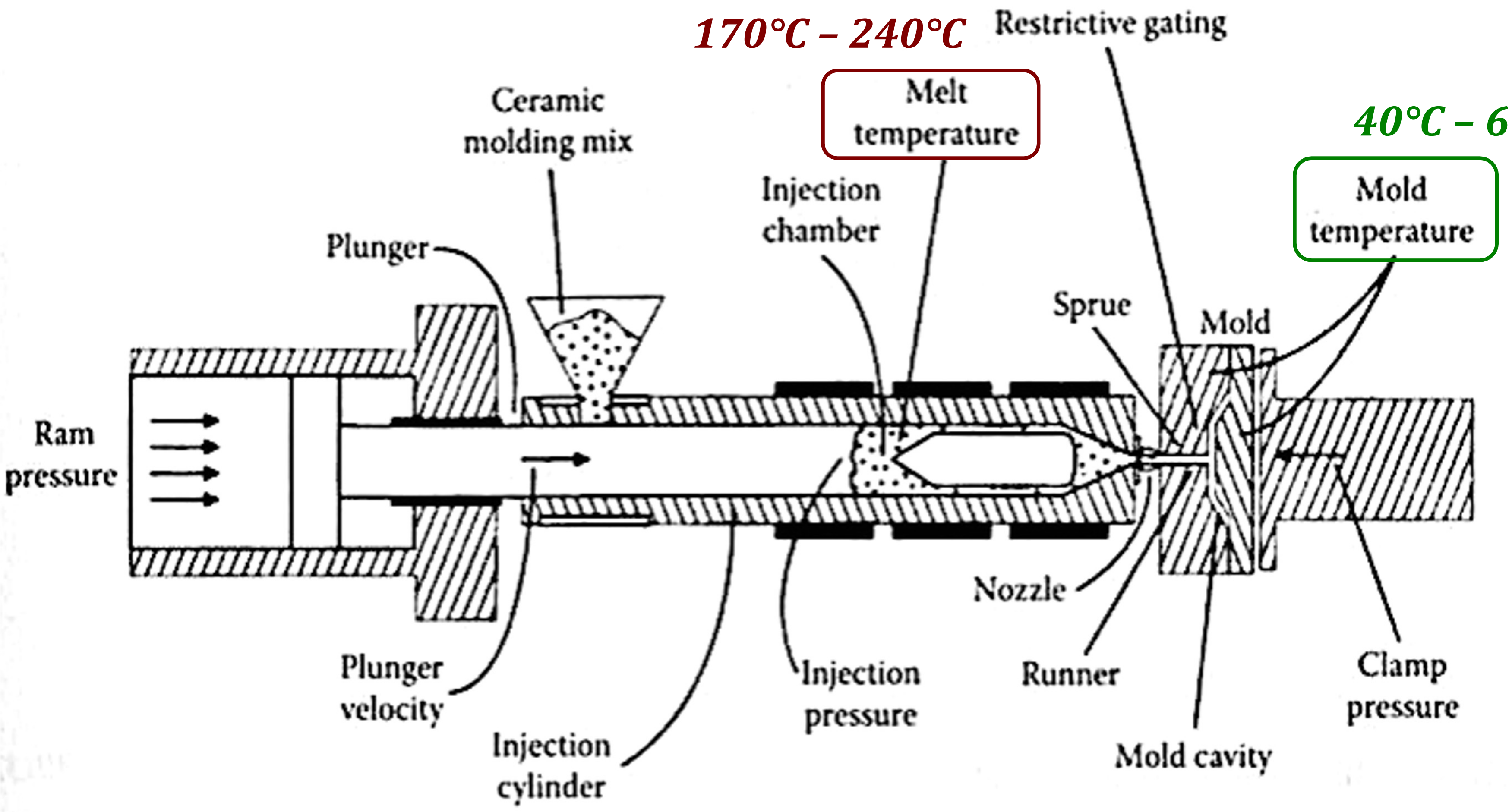
optimal sections for extrusion



gradients → defects



Injection molding



Major Binder	Minor Binder	Plasticizer	Other Additives
Polypropylene	Microcrystalline wax	Dimethyl phthalate	Stearic acid
Polyethylene	Paraffin wax	Diethyl phthalate	Oleic acid
Polystyrene	Carnauba wax	Dibutyl phthalate	Fish oil
Poly(vinyl acetate)		Dioctyl phthalate	Organo silane
Poly(methyl methacrylate)			Organo titanate

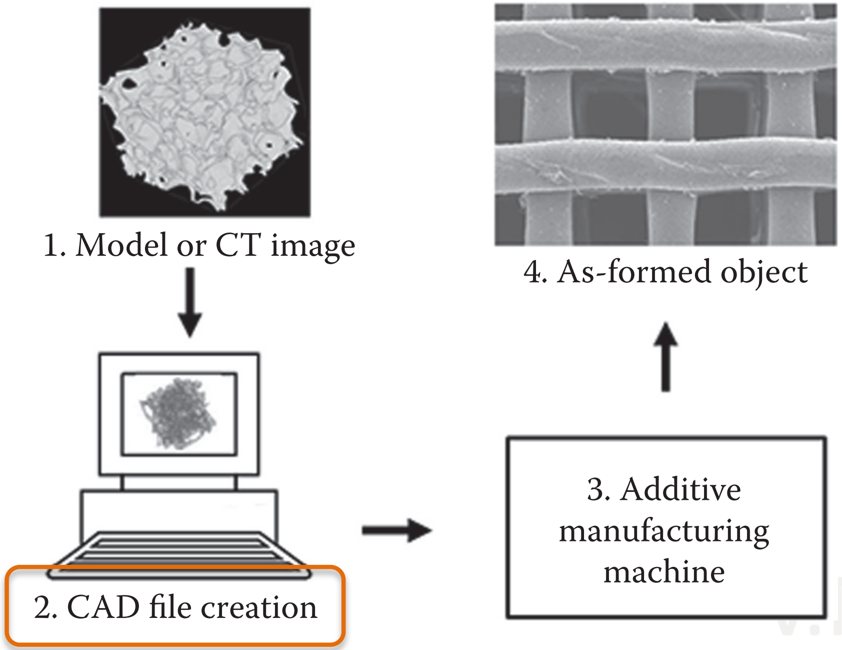
Examples

Component	Composition (wt%)	
Powder	1-µm Al_2O_3 (85)	20-µm Si (82)
Major binder	Paraffin wax (14)	Polypropylene (12)
Minor binder	—	Microcrystalline wax (4)
Other additives	Oleic acid (1)	Stearic acid (2)

Additive manufacturing

New technologies:

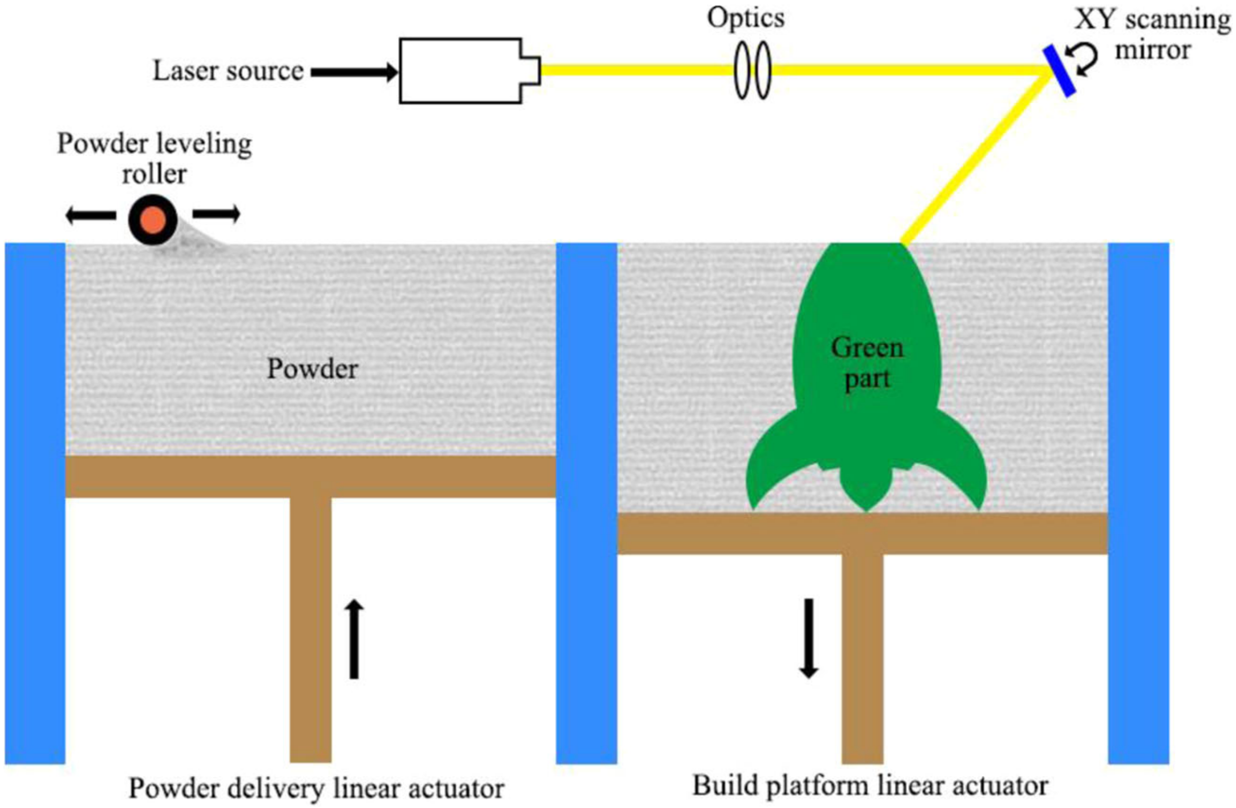
- *rapid prototyping*
- *solid freeform fabrication*
- *additive manufacturing* or *three-dimensional (3D) printing*



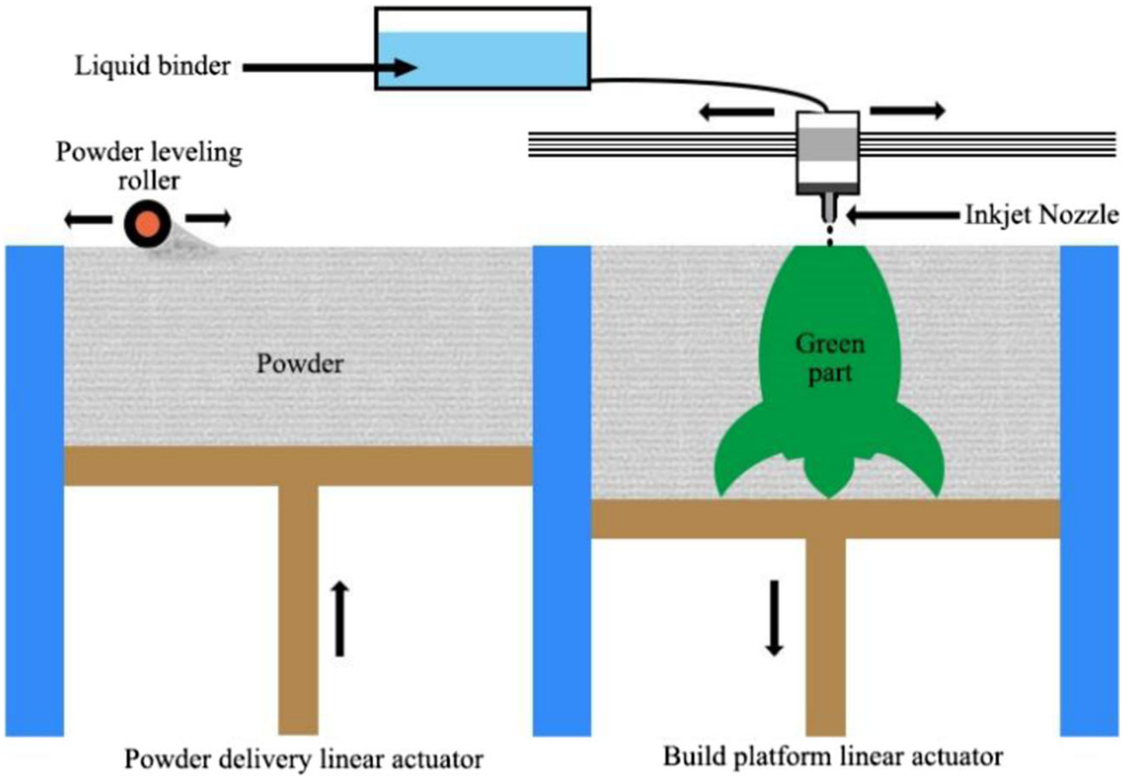
3D printing for ceramics

Feed Material	Method	Process
Powder		
Powder-binder mixture or binder-coated powder	Selective laser sintering (SLS)	Laser beam scanned on layer of material to soften binder and bind particles
Powder or dried powder from a slurry	Three-dimensional printing (3DP)	Binder solution sprayed on powder bed to bind particles
Particle-Filled Polymer		
Particle-filled polymer filaments	Fused deposition modeling (FDM)	Extrusion of softened filaments through a heated nozzle
Tape-cast sheets	Laminated object manufacturing (LOM)	Sheets cut by laser beam and stacked
Suspension, Slurry, or Paste		
Particles dispersed in monomer solution	Stereolithography	Laser beam scanned on suspension to polymerize monomer solution
Moderately dilute suspension stabilized with organic additives	Inkjet printing	Printing of droplets from printer nozzle followed by drying by evaporation
Concentrated slurry or paste stabilized with organic additives	Robocasting	Extrusion of slurry or paste through a nozzle followed by drying or gelation
Concentrated slurry or paste stabilized with organic additives	Freeze extrusion fabrication (FEF)	Extrusion of slurry or paste through a nozzle and freezing in a cold chamber

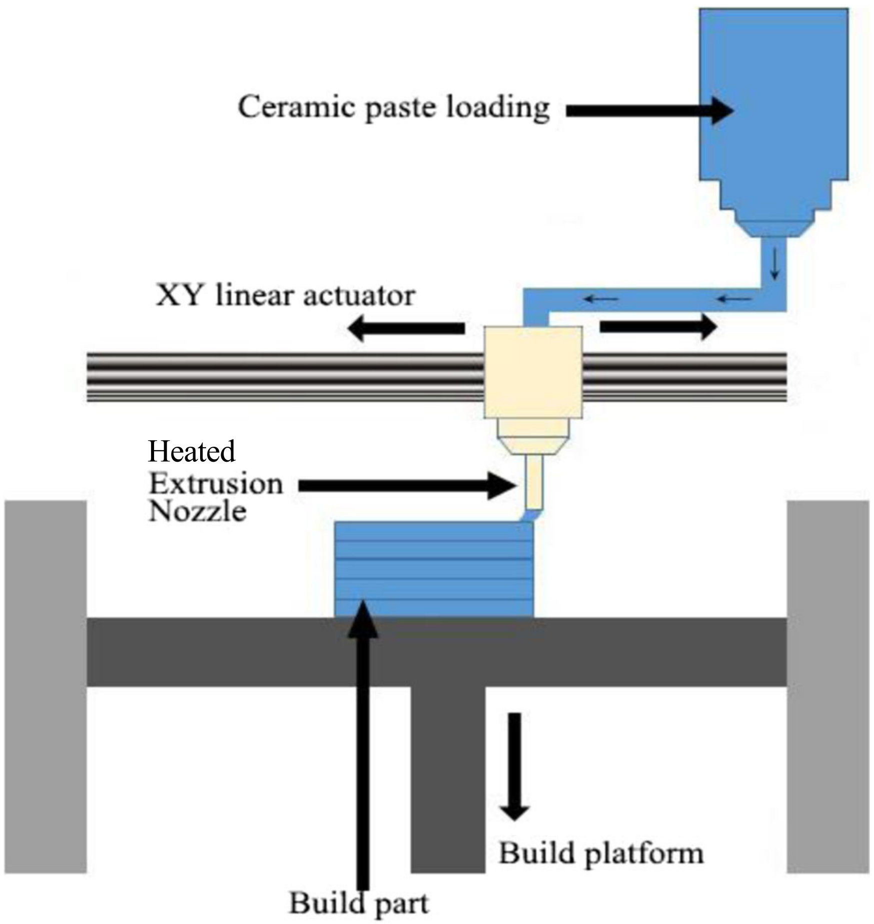
Selective laser sintering



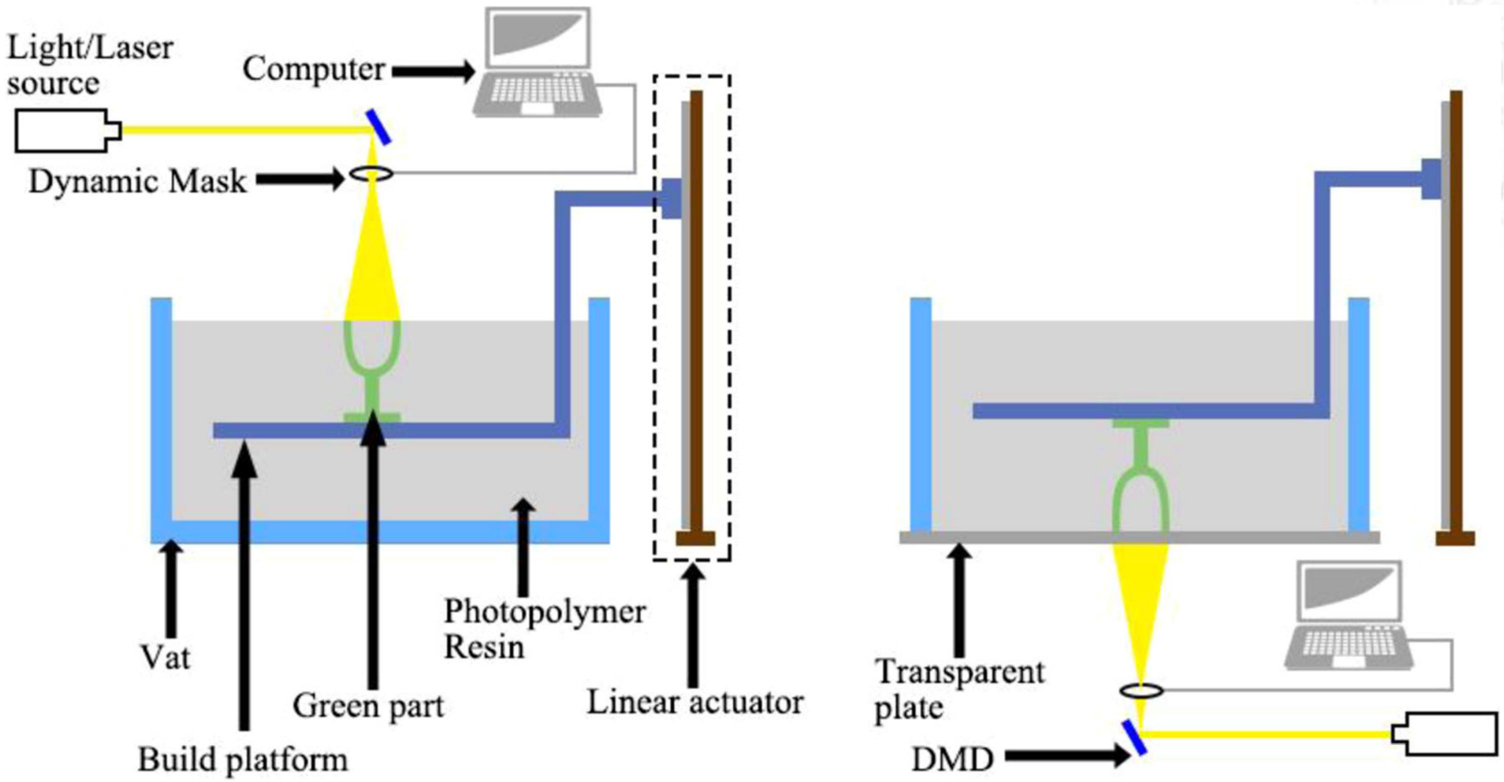
Binder jetting



Fused deposition modeling – fused filament modeling - robocasting



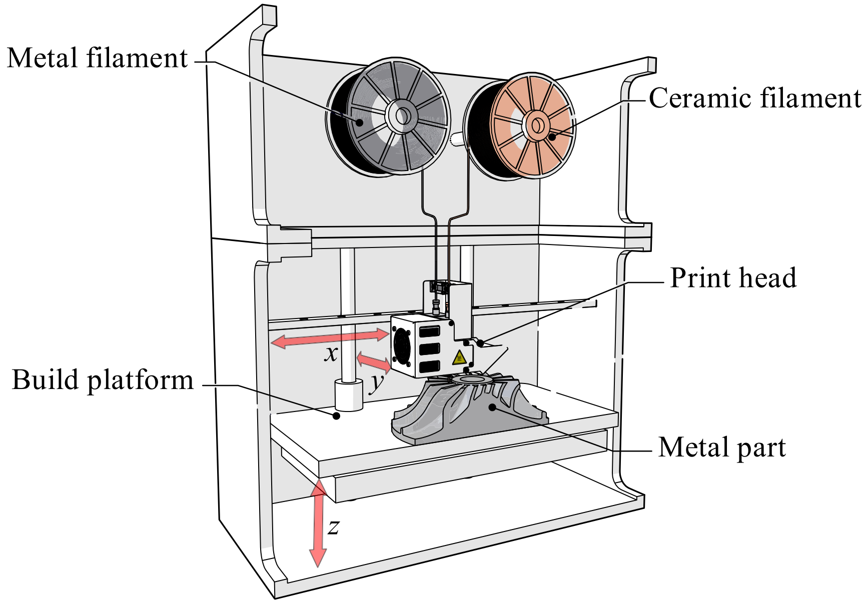
Stereoliothography – two photon polymerization

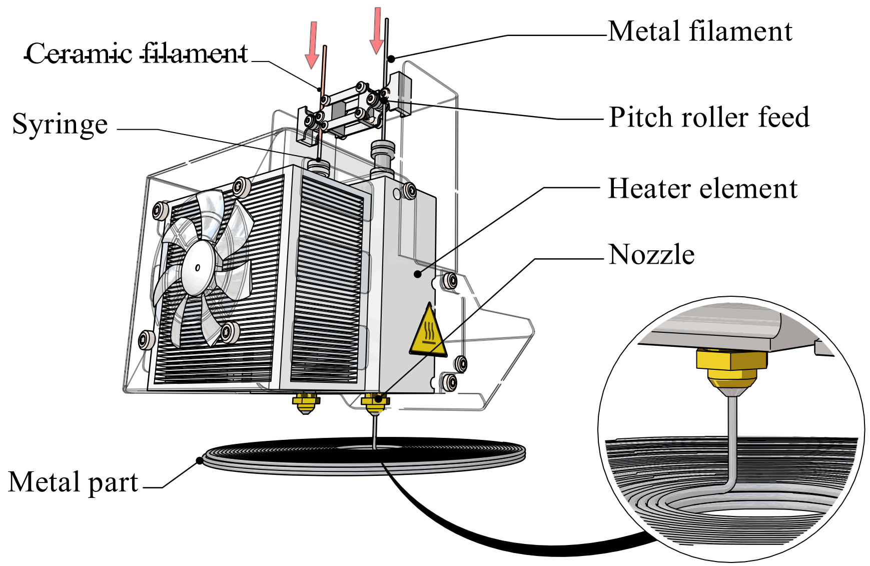


3D printing for metals

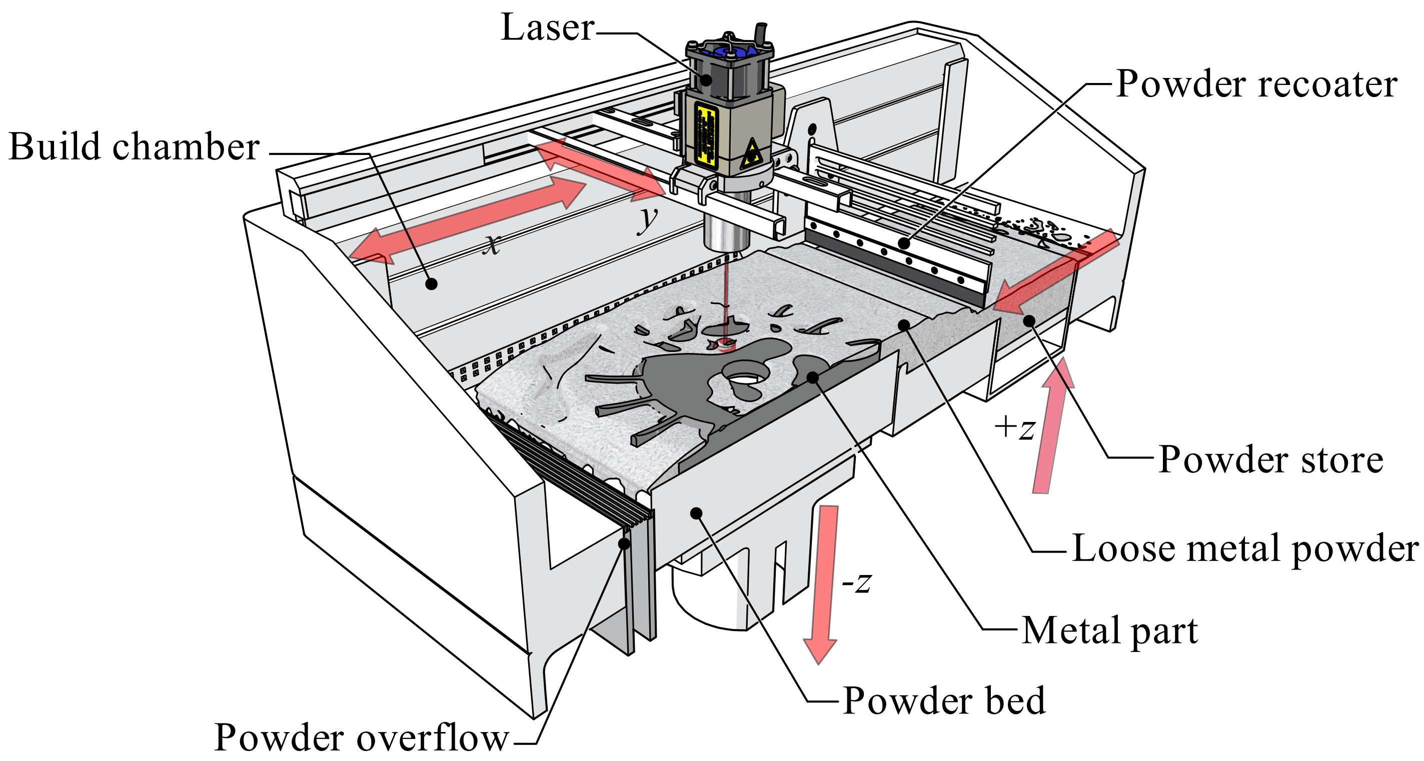
Process	Abbreviation	Mode	Typical Applications
Material extrusion	ME	Material is selectively dispensed through a nozzle or orifice	Functional metal parts, prototyping,
Binder jetting	BJ	A liquid bonding agent is selectively deposited to join powder materials	Functional metal parts, low-rate production runs of non-critical components
Directed energy deposition	DED	Focused thermal energy is used to fuse materials by melting as they are being deposited	Functional metal parts, repairs, adding material to existing parts
Powder bed fusion	PBF	Thermal energy selectively fuses regions of a powder bed	Functional metal parts, functional prototyping

Metal extrusion

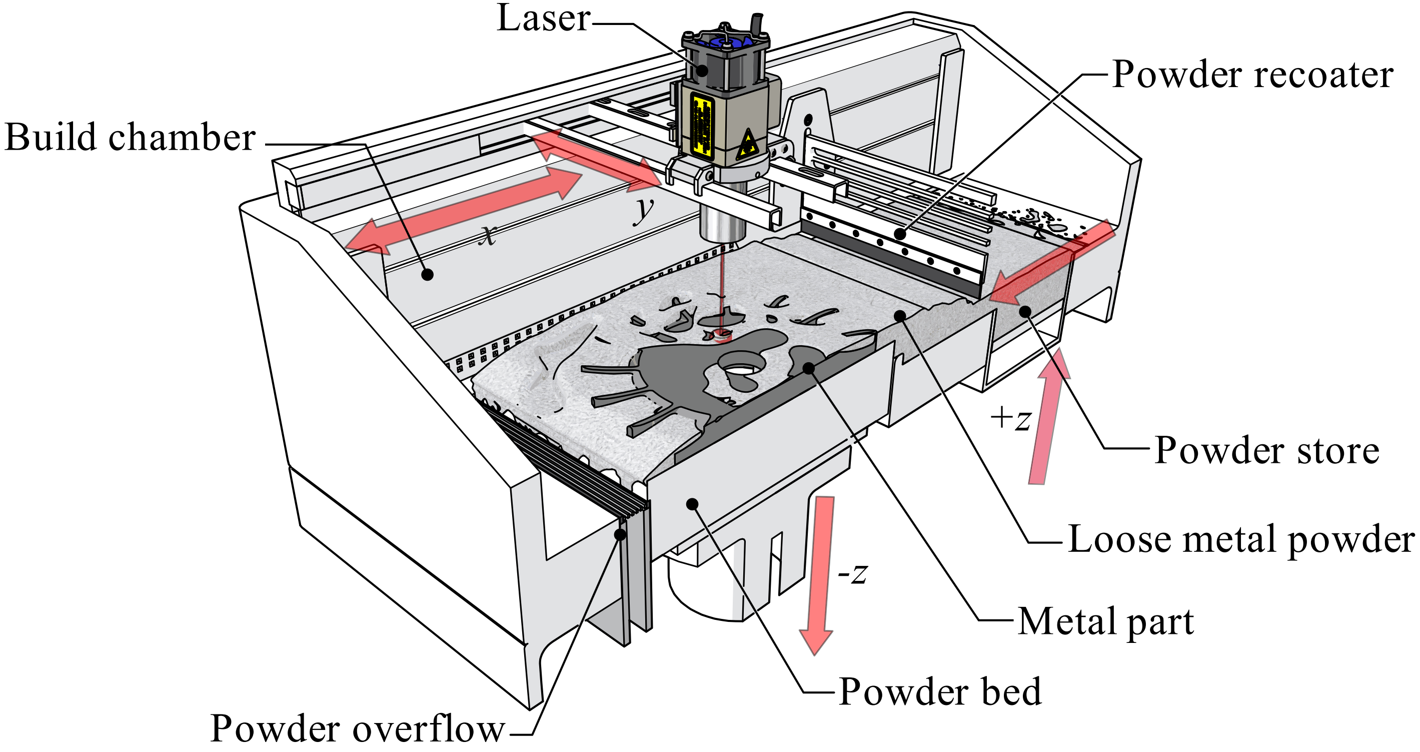




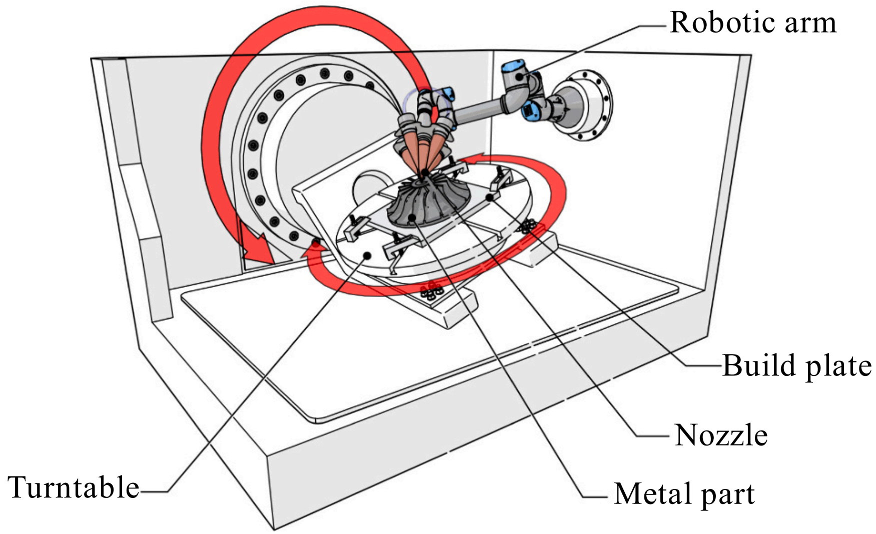
Powder bed fusion

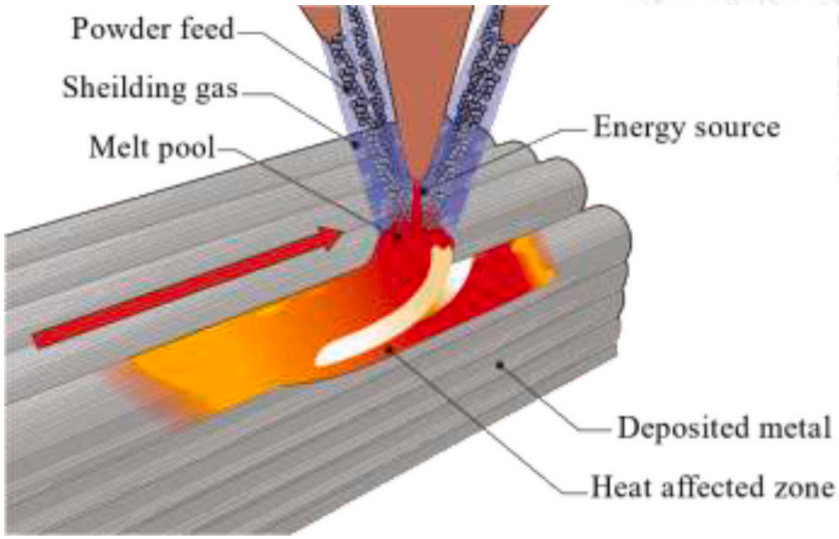


Powder bed fusion



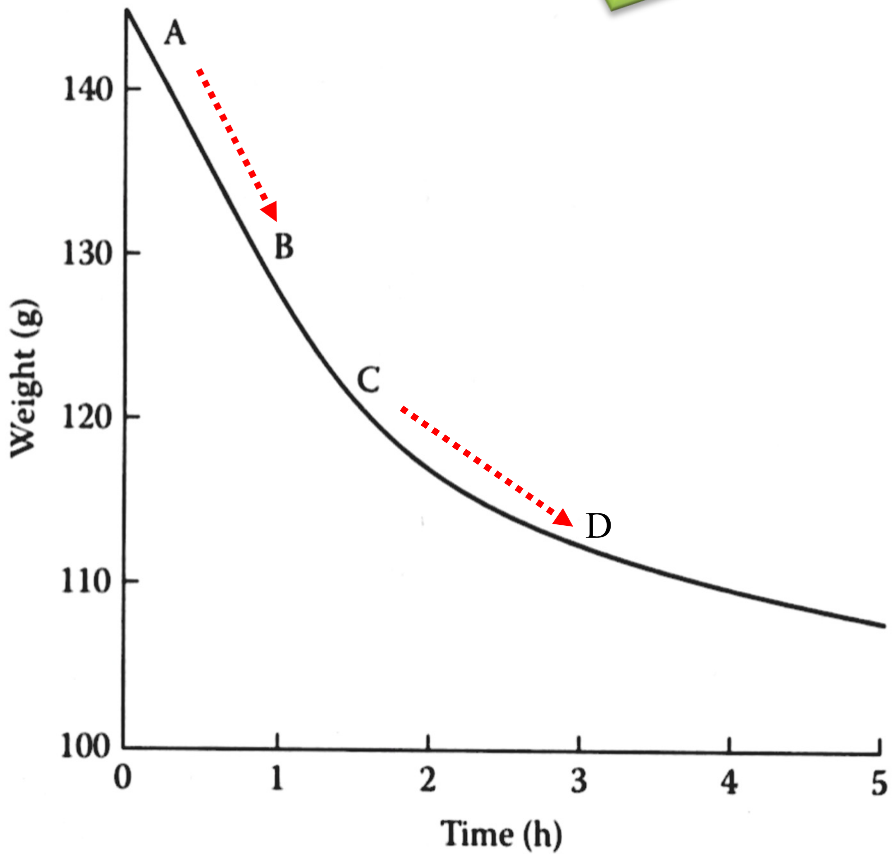
Directed energy deposition

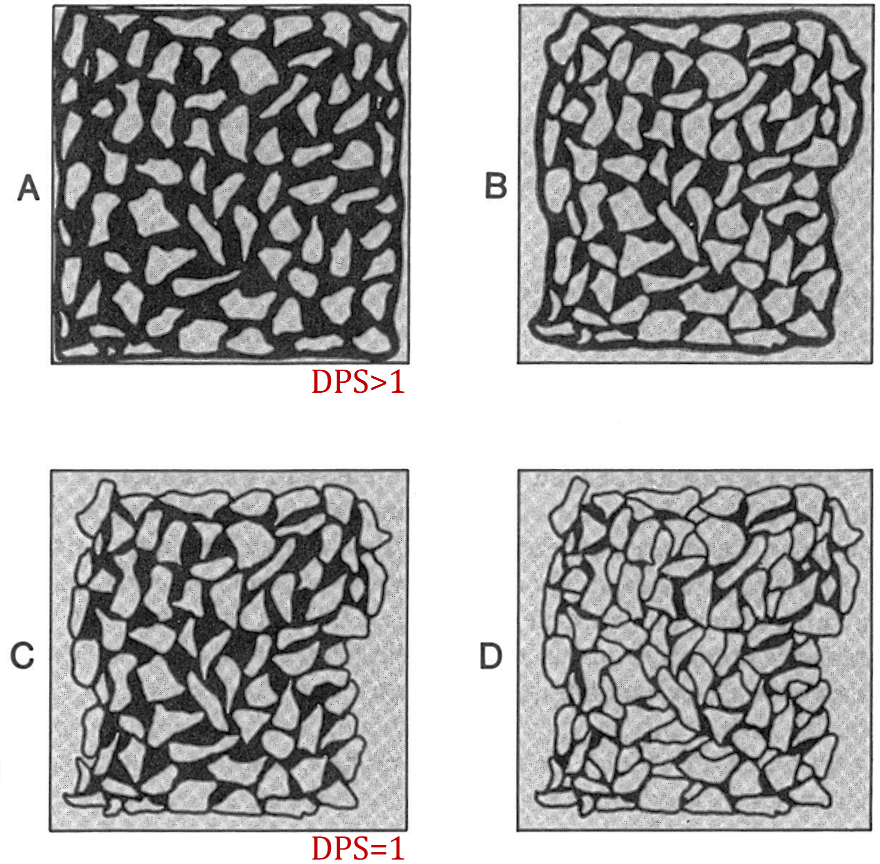


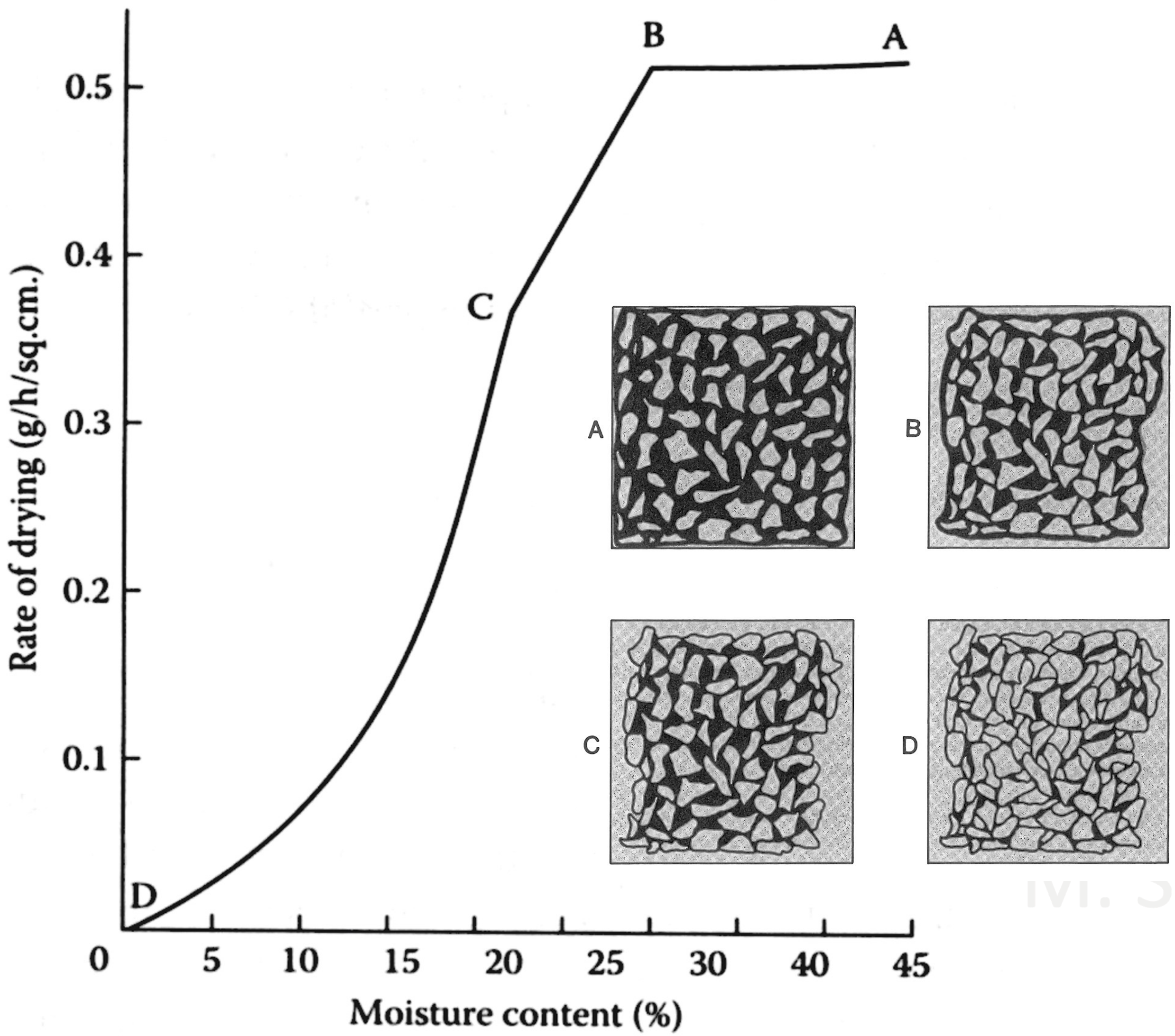


Drying

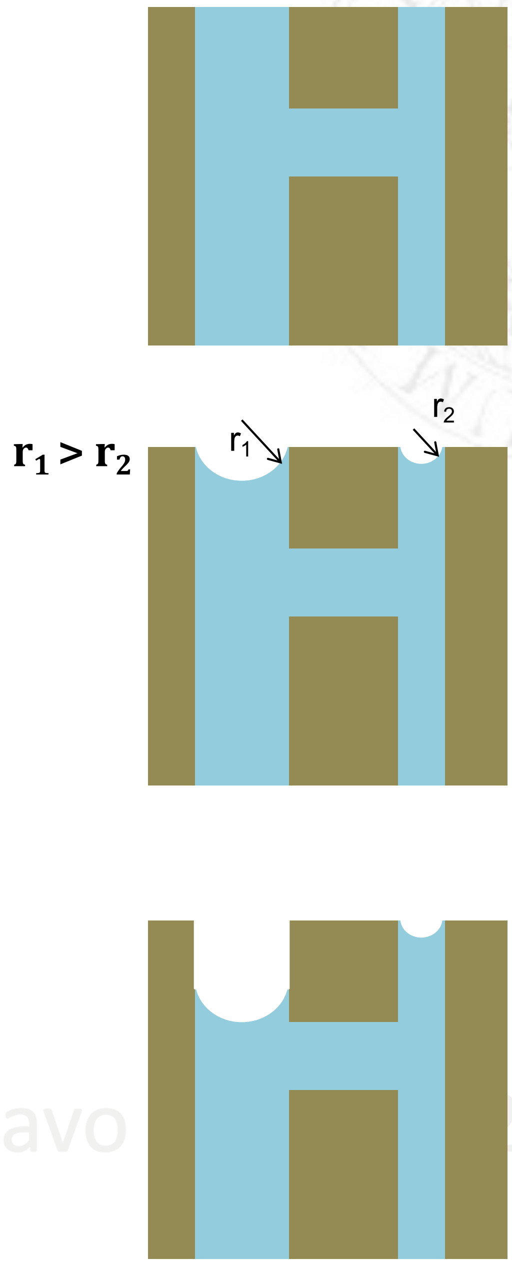
slow and uniform removal of the solvent







Rate of drying (g/h/sq.cm.)
0.5
0.4
0.3
0.2
0.1
0
5
10
15
20
25
30
40
45
Moisture content (%)
A
B
C
D
A
B
C
D



$r_1 > r_2$
r_1
r_2

evaporation speed

(mass evaporating per unit area x time)

$$R_E = K_E(p_W - p_0)$$

vapour pressure

partial pressure of the liquid in the air

air flow conditions

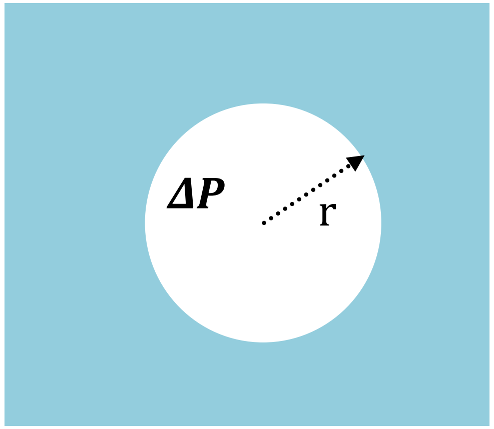
highly energetic process with liquid-gas transition ($\Delta V >> 0$)

evaporation heat

	Heat of Vaporization (MJ/kg)		
Temperature (°C)	H_2O	CH_3OH	C_2H_5OH
20	2.45	1.17	0.91
40	2.40	1.14	0.90
60	2.36	1.11	0.88
80	2.31	1.06	0.85
100	2.26	1.01	0.81

surface tension effects

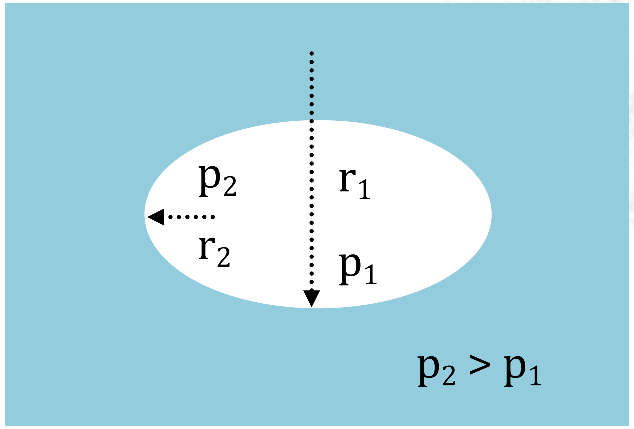
bubble in a liquid



Laplace law

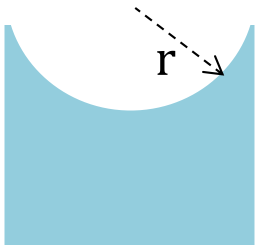
$$\Delta P = \frac{2\gamma}{r}$$

$$\Delta P = 2\gamma\left(\frac{1}{r_1} - \frac{1}{r_2}\right)$$



vapour pressure above the surface

P < P₀

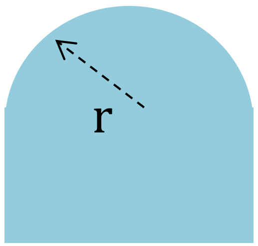


$\infty < r < 0$

P₀

$r = \infty$

P > P₀



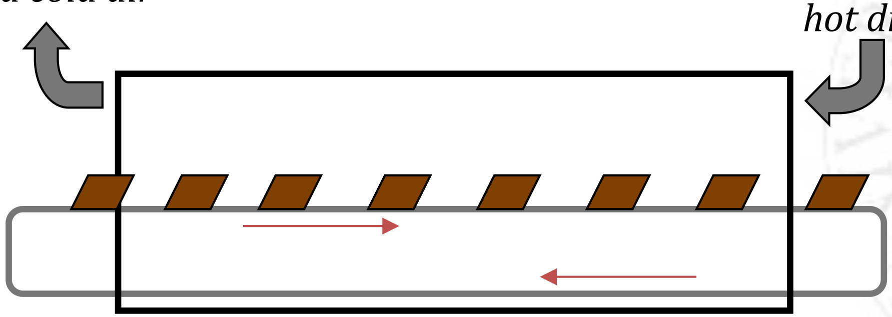
$0 < r < \infty$

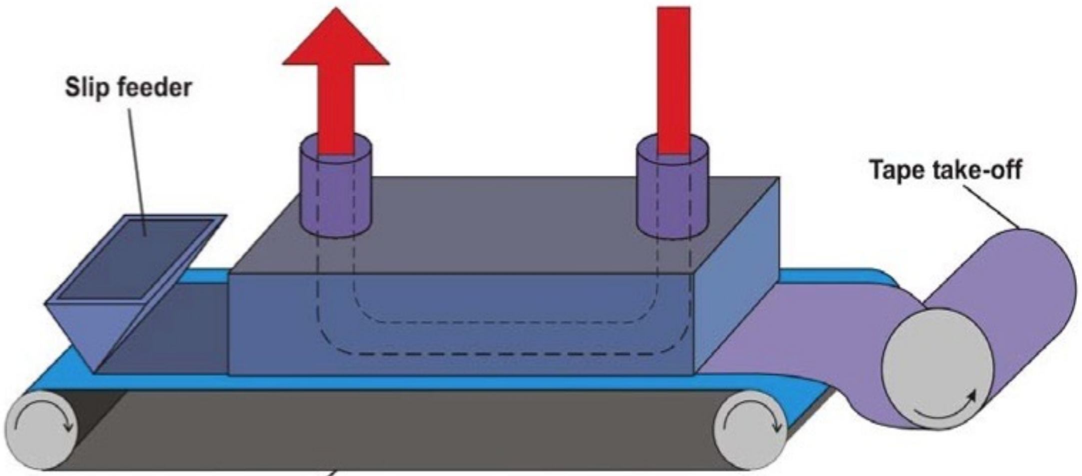
Kelvin law

$$\ln\frac{P}{P_0} = \frac{2\gamma\, V_{mol}}{r\, R\, T}$$

uniform drying:

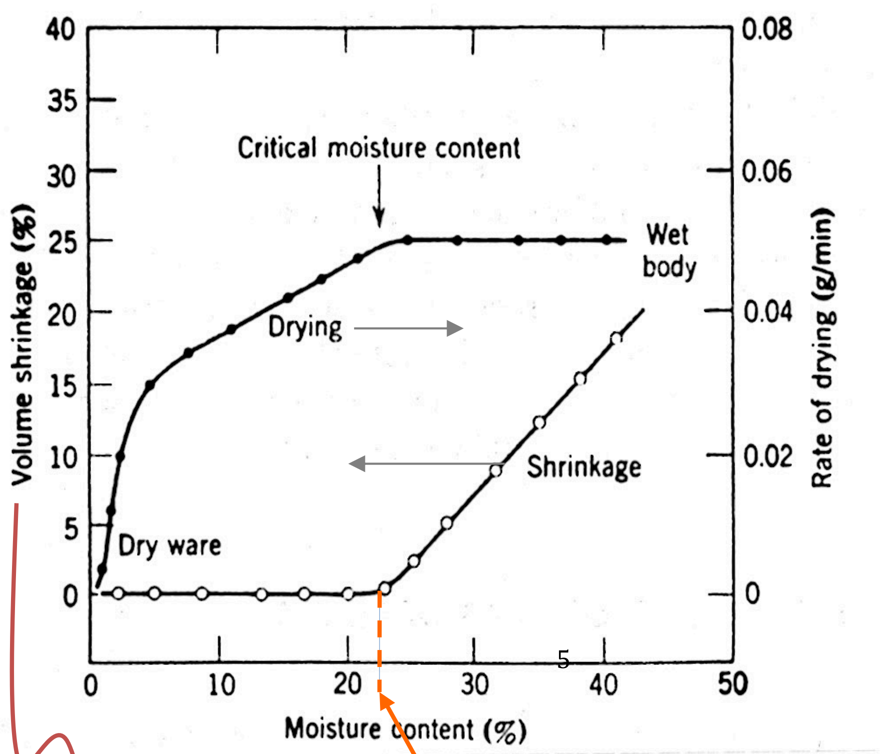
➡ counter-current process





Principles of ceramics processing, 2nd ed., J.S. Reed, J. Wiley & sons, 1995

shrinkage and stresses upon drying



influence of forming process (DPS value)

$$\frac{\Delta V}{V_0} = 1 - \left[1 - \frac{\Delta L}{L_0}\right]^3$$

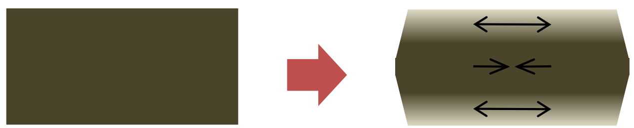
$$\frac{\Delta L}{L_0} = N_p \ \Delta l$$

"Bigot point" or critical moisture content

mean number of interparticle liquid films per unit length

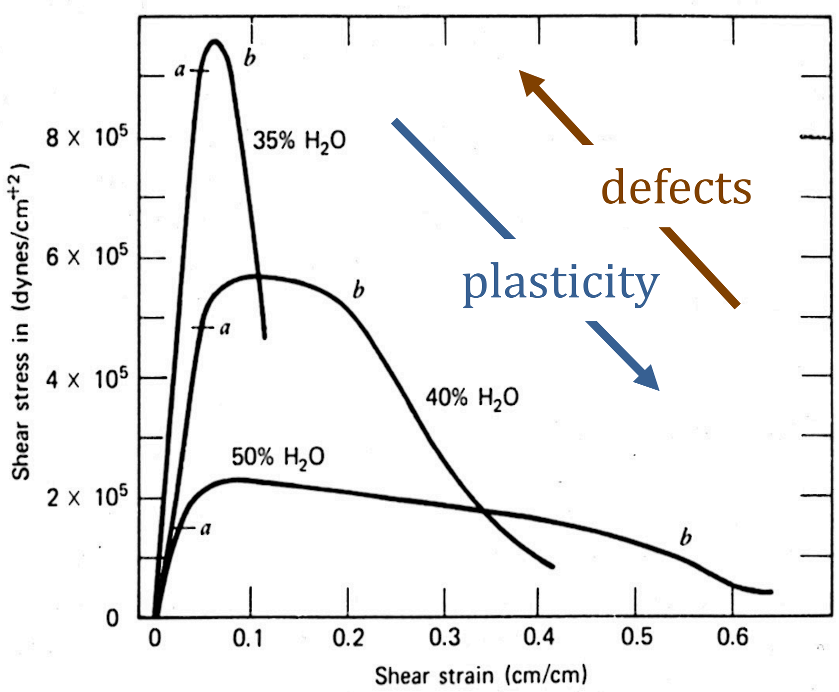
mean reduction of interparticle spacing

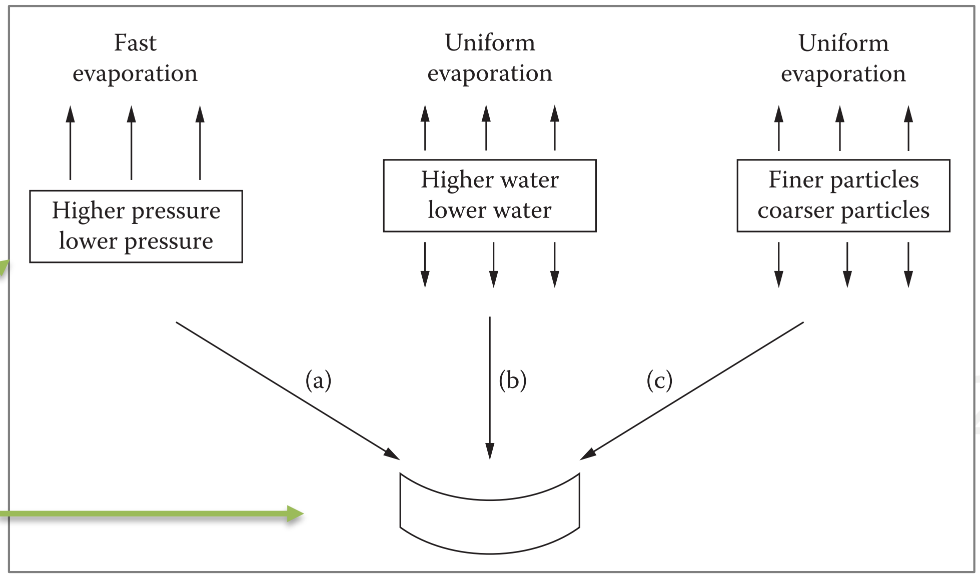
$\Delta V \rightarrow \Delta\varepsilon \rightarrow \sigma = \varepsilon E$



Principles of ceramics processing, 2nd ed., J.S. Reed, J. Wiley & sons, 1995

deformation and mechanical behaviour





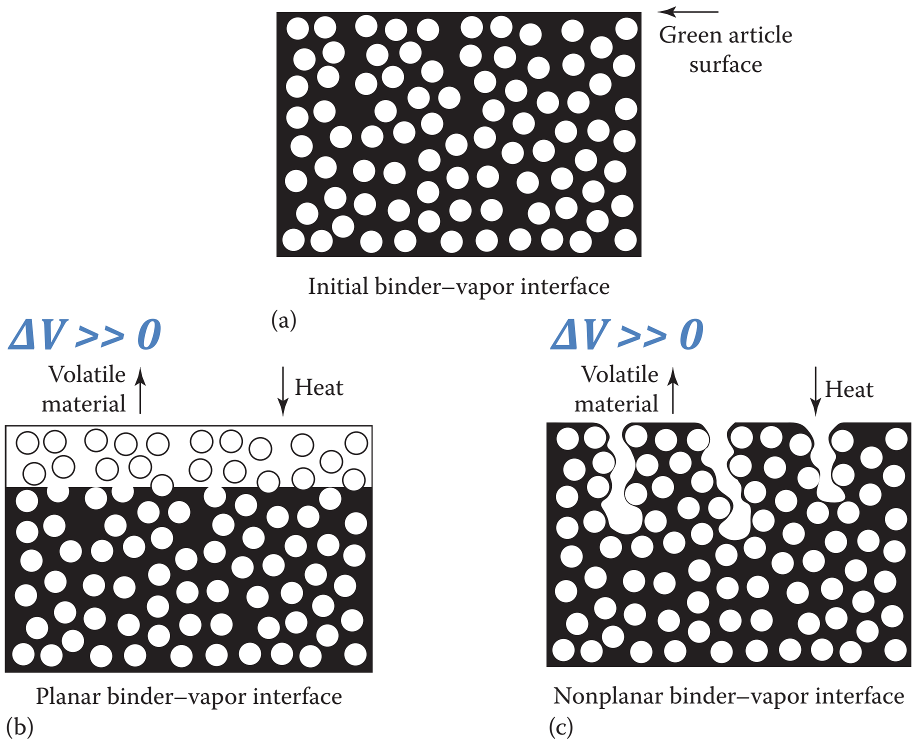
wet body

dried body

Principles of ceramics processing, 2nd ed., J.S. Reed, J. Wiley & sons, 1995

Pre-sintering processes

Thermolysis of organic compounds



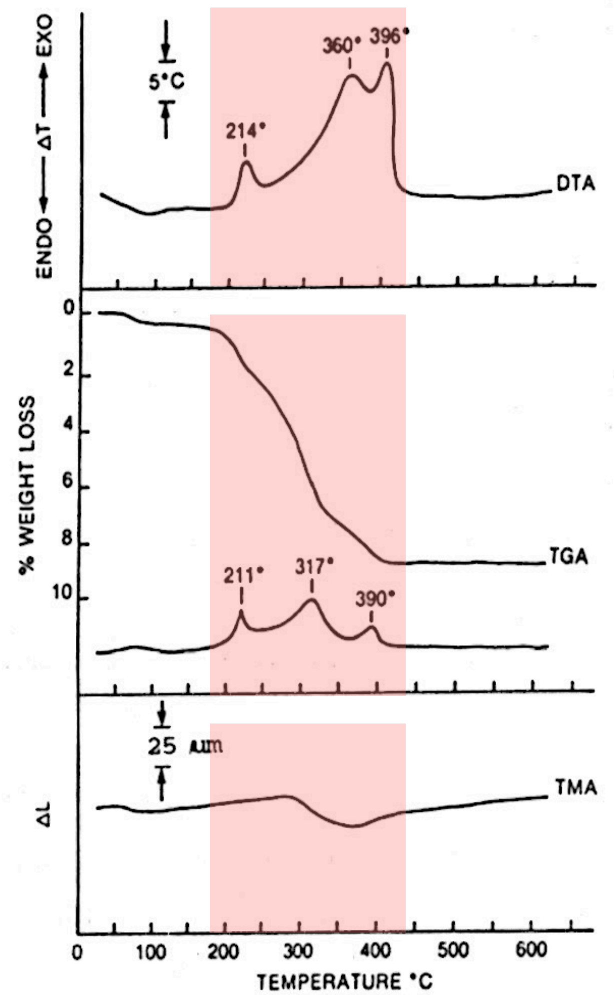
slow and uniform removal of the "binders", decomposition/reaction of inorganics

heating rate (T = 200 - 500°C) ≈ 0.1-1°C/min

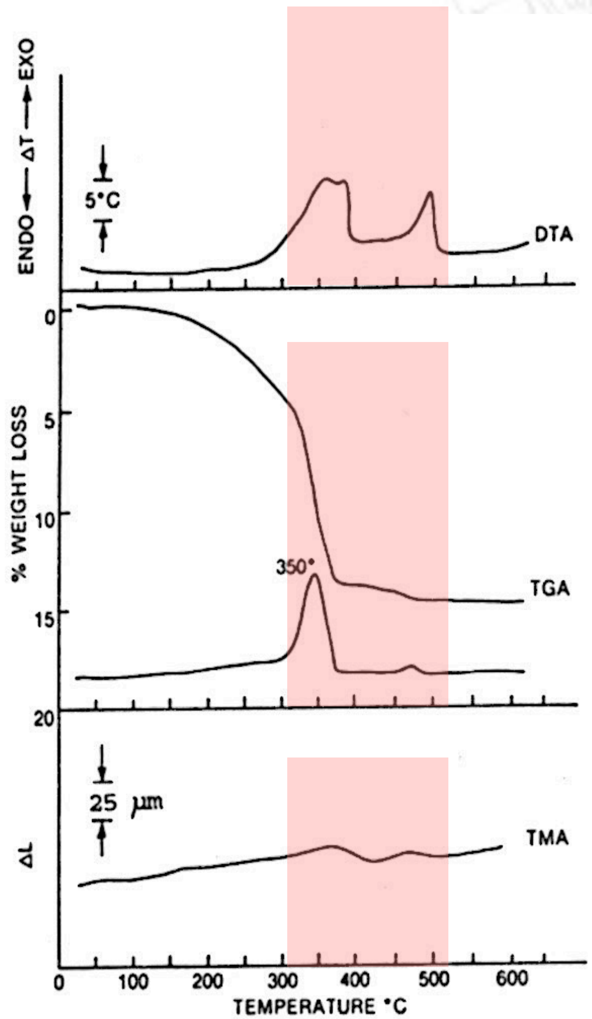
Principles of ceramics processing, 2nd ed., J.S. Reed, J. Wiley & sons, 1995

Thermal analyses on green ceramic compacts → critical temperature range

Tape cast binder system



Injection molding binder system



thermal effects

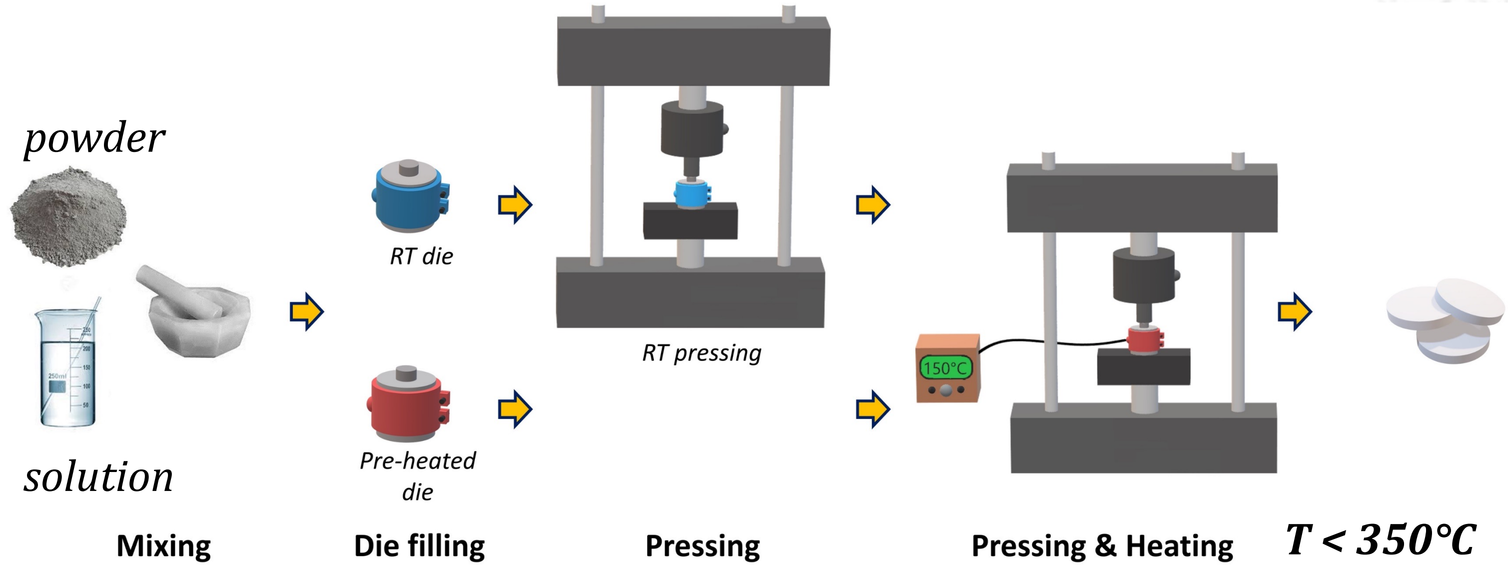
weight effects

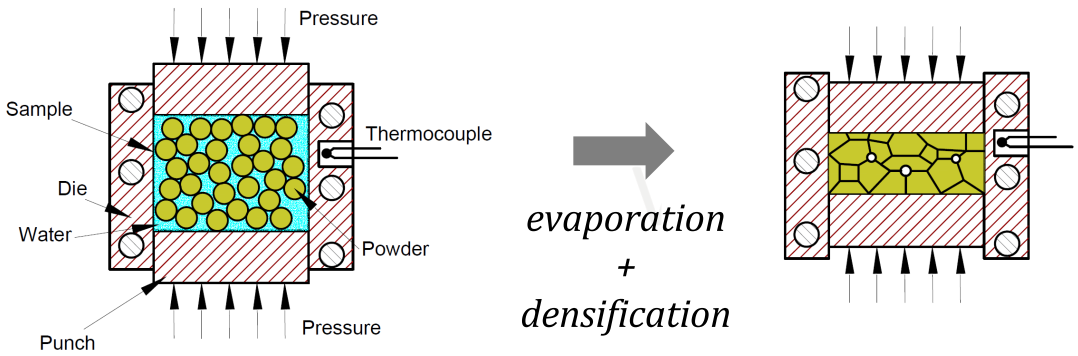
length effects

Principles of ceramics processing, 2nd ed., J.S. Reed, J. Wiley & sons, 1995

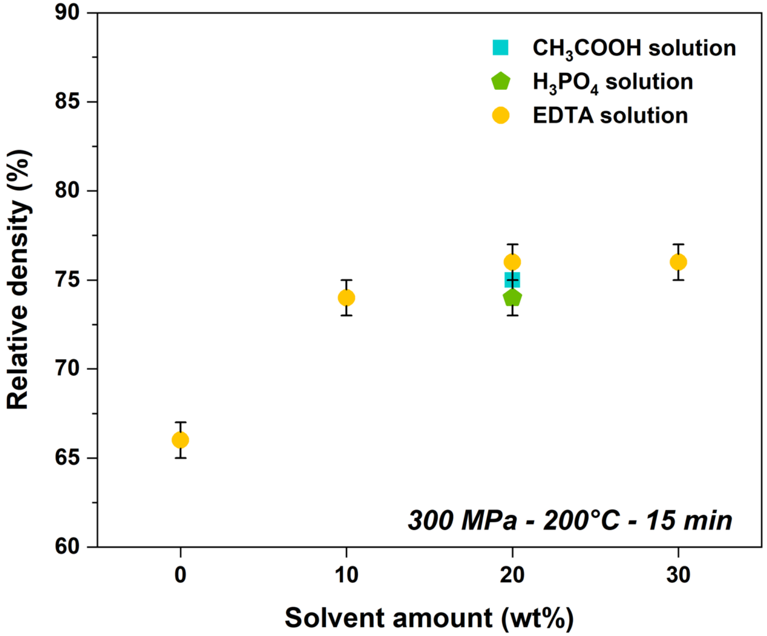
Cold Sintering

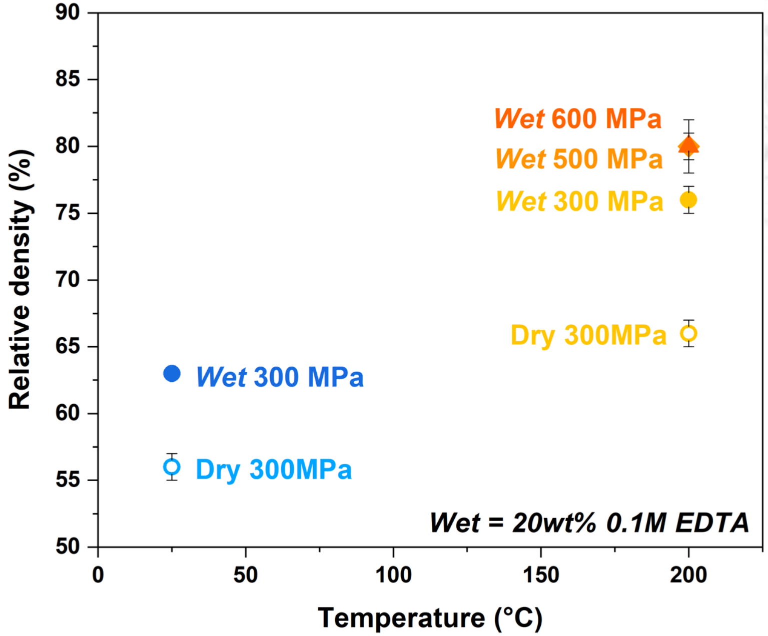
Introduced in 2016

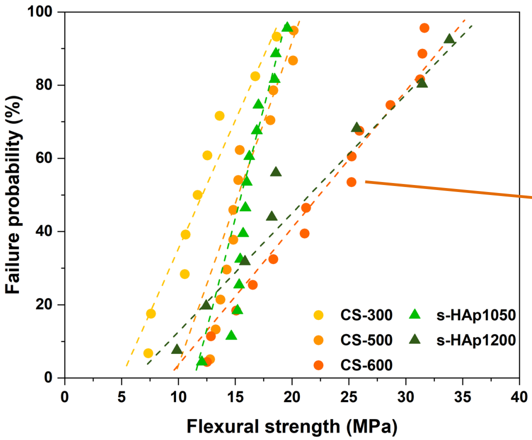


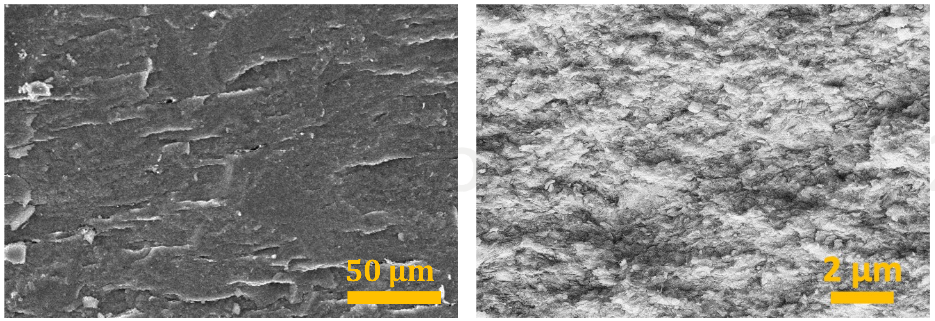


Cold sintering of HA

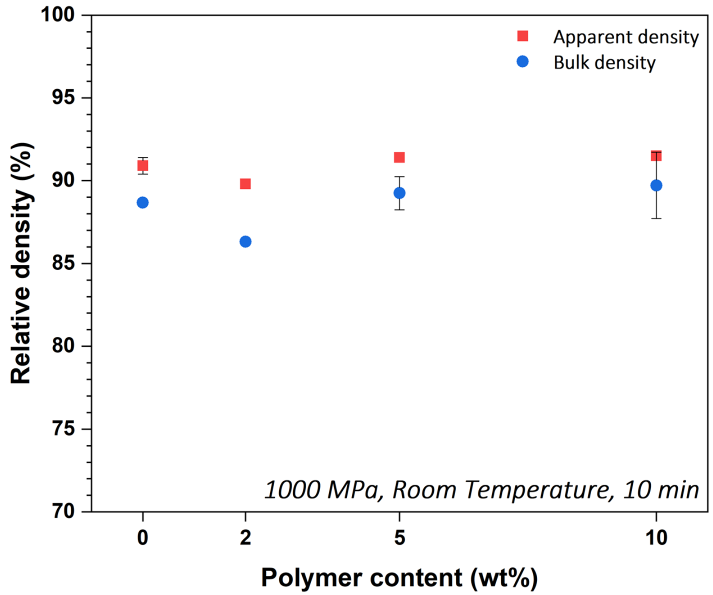


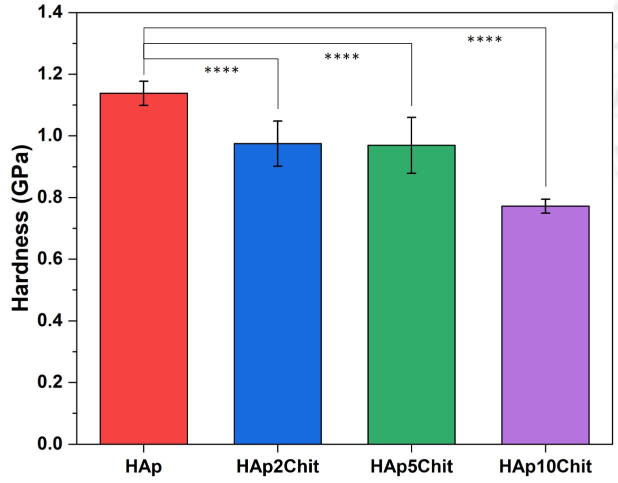


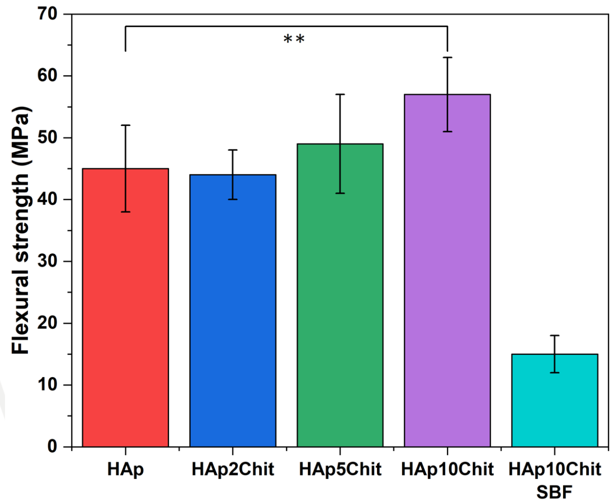




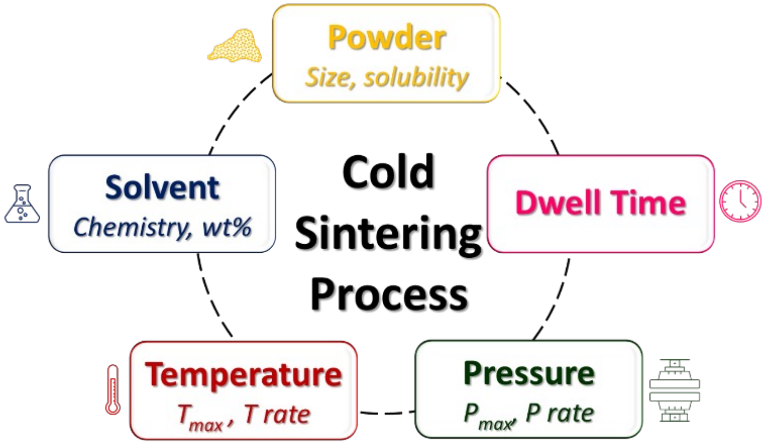
Cold sintering of HA/chitosan composites

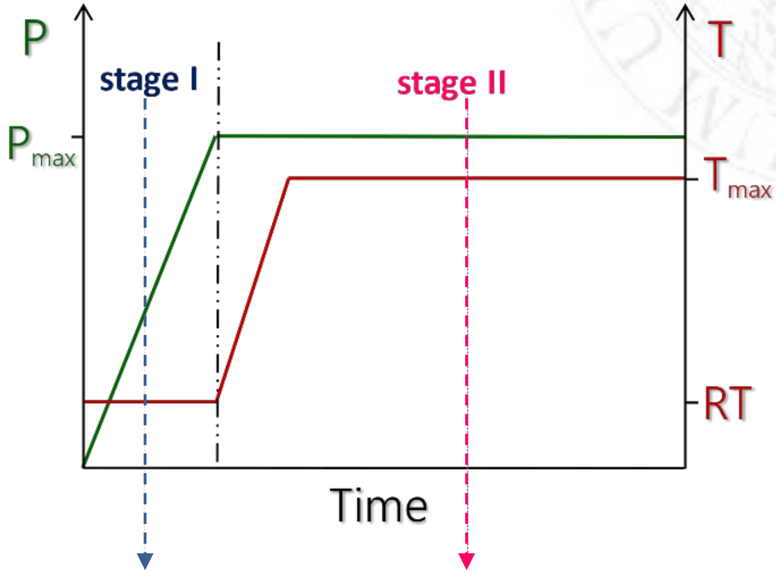






Proposed mechanisms



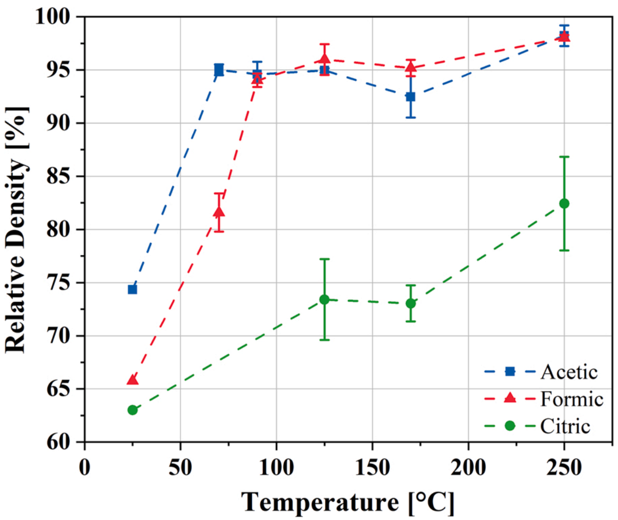


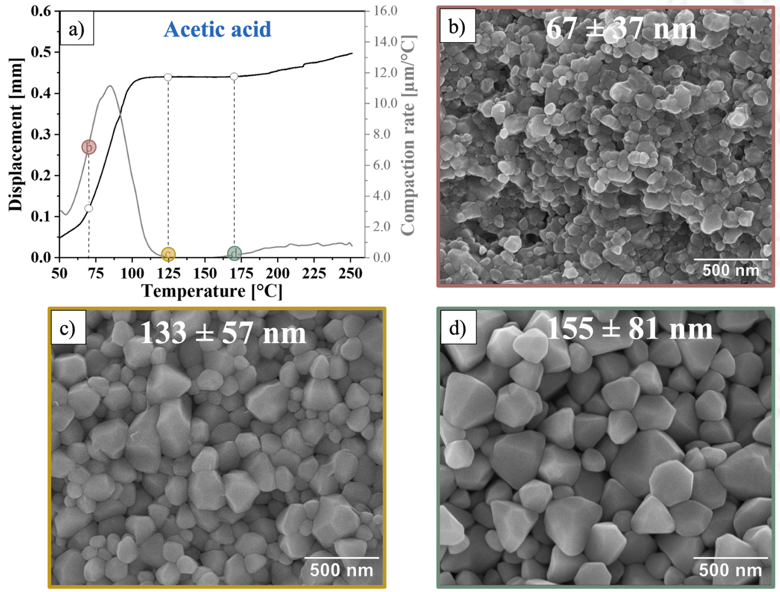
- ✓ particles rearrangement, sliding compaction
- ✓ grain boundary flattening and sliding

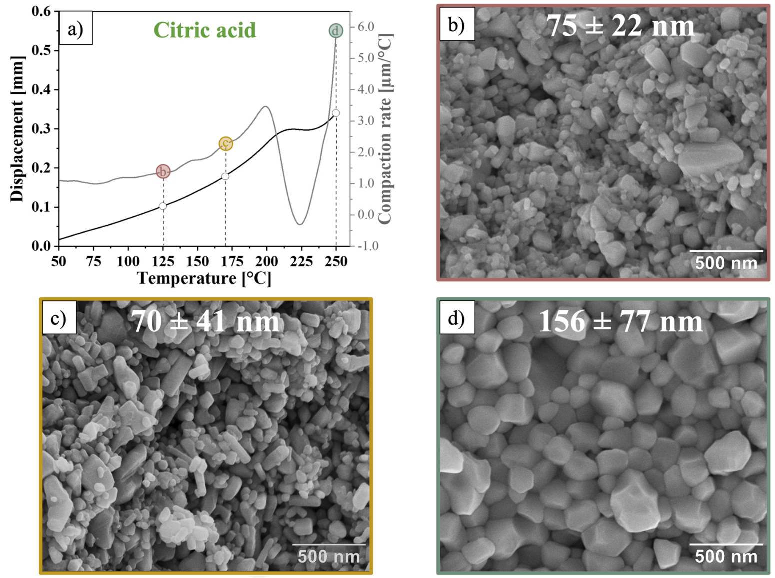
- ✓ pressure-solution creep
- ✓ dissolution and precipitation
- ✓ plastic deformation
- ✓ viscous flow

Effect of liquid phase chemistry

ZnO







Zinc Carboxylate	Formation constant, Log k_1	Acid pKa	Solubility [g/100 g]	Acid Decomposition Temperature [°C]	Complex Decomposition Temperature [°C]
Zinc Acetate	0.88 [30]	4.76 [31]	40 [35]	230 [36]	240–271 [37]
Zinc Citrate	4.93 [30]	3.13 [31]	40 [35]	165 [38]	300–400 [39]

Flash sintering

Discovered in 2010

(accidentally... during experiments on field effect on grain growth)

electric field

⬇

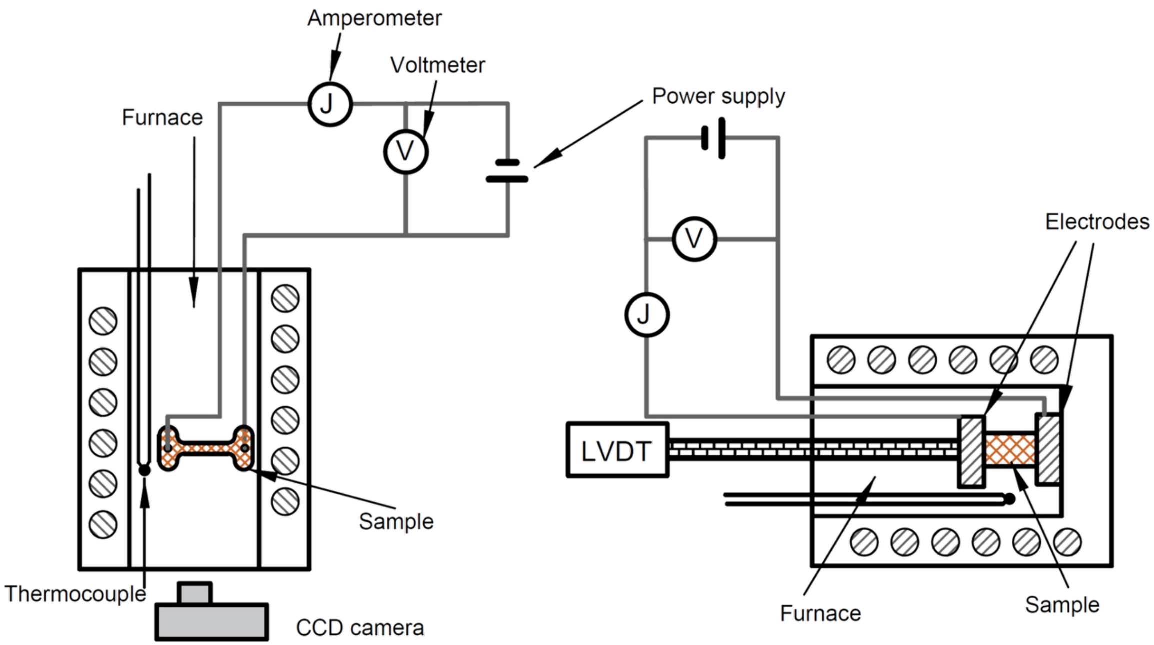
current flow through the green compact

⬇

sintering in just few seconds

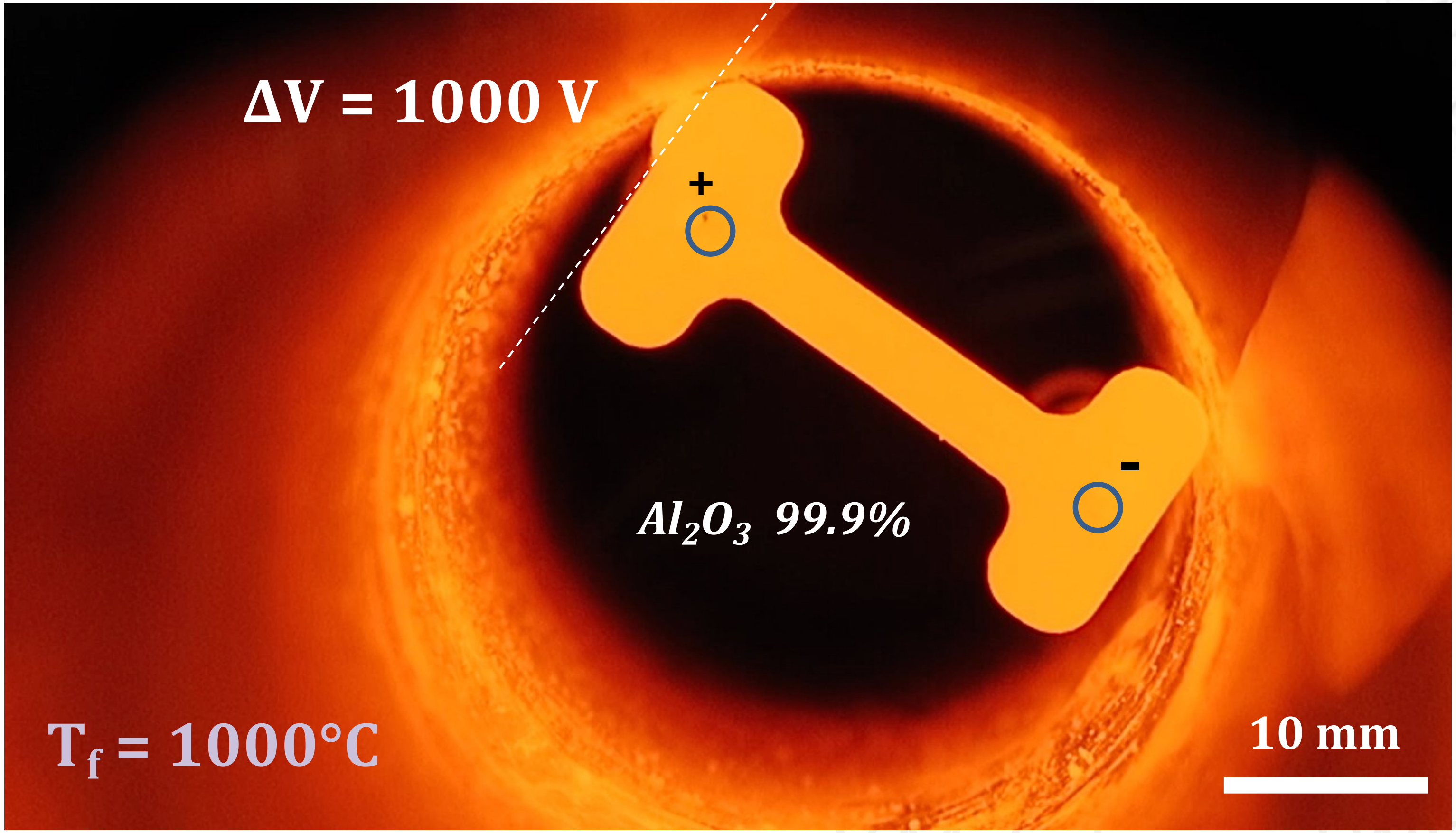
(combination of T and E)

thermal runaway of Joule heating

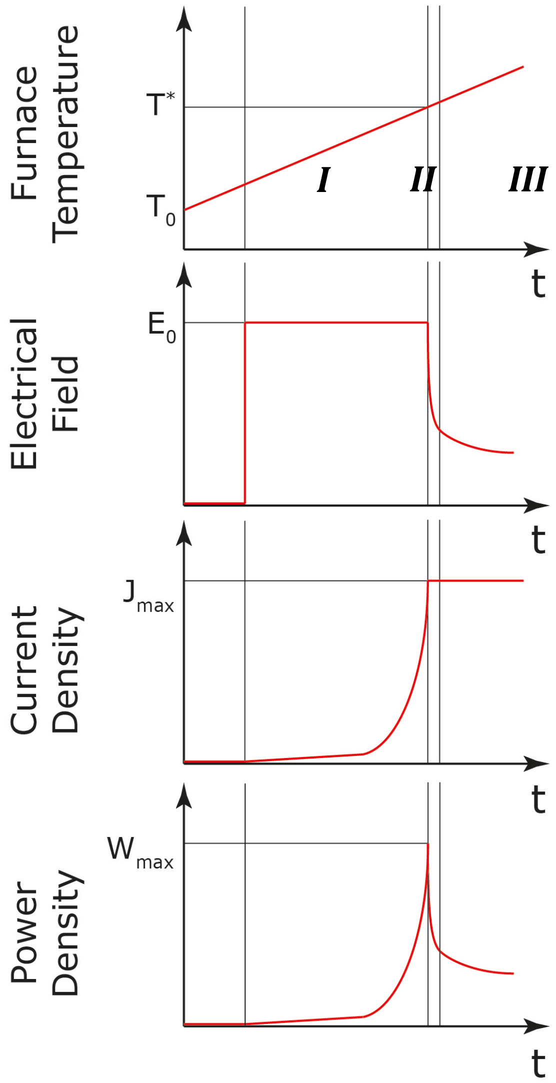


Phenomenology

speed = 4x



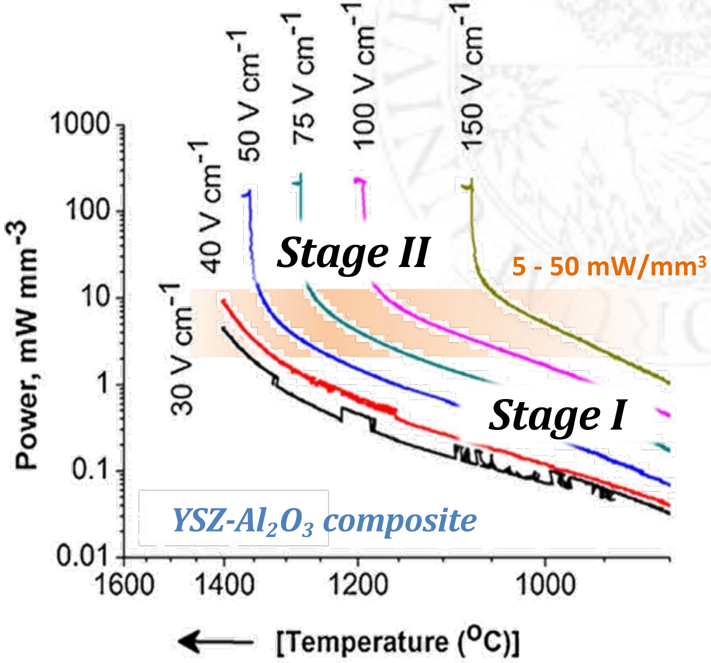
Stages and fingerprints



Stage I at E_0

Stage II "flash event"
thermal runaway

Stage III at J_{max}
stationary condition



- ***Sintering ≡ Resisitivity drop ≡ Very rapid heating (≈10^3-10^4 K/min)***
- ***Bright light emission («flash»)***
- ***A minimum conductivity is needed to produce the flash***
- ***The onset temperature descreases with the applied field***

Thermal runaway for Joule heating

Ceramics with negative temperature coefficient (NTC) for electrical resistivity

$$W_{in} = Volume\,\frac{E^2}{\rho} = Volume\,\frac{E^2}{\rho_0}\,e^{-Q/RT_{sample}}$$

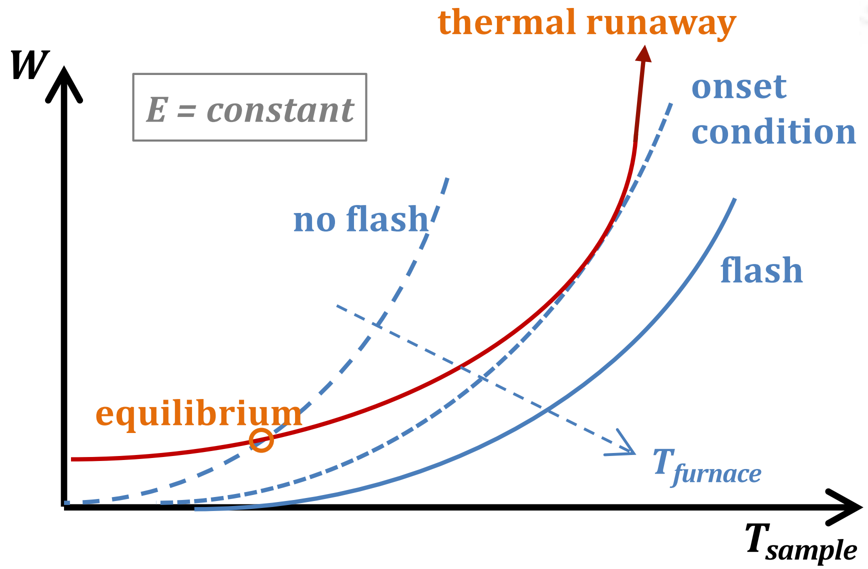
$$\Updownarrow$$

$$W_{out} = Surface\ \varepsilon\ \sigma_{SB}\left(T_{sample}^4 - T_{furnace}^4\right)$$

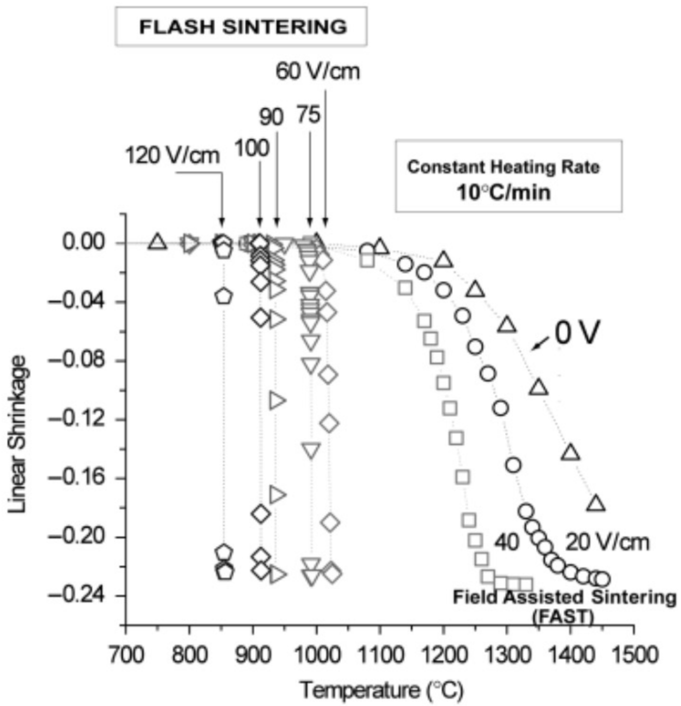
$$E^2 = \frac{4p\varepsilon\sigma\,\rho_0 R}{AQ}\left(T_f + \Delta T_c\right)^5 \exp\left(\frac{Q}{R\left(T_f + \Delta T_c\right)}\right)$$

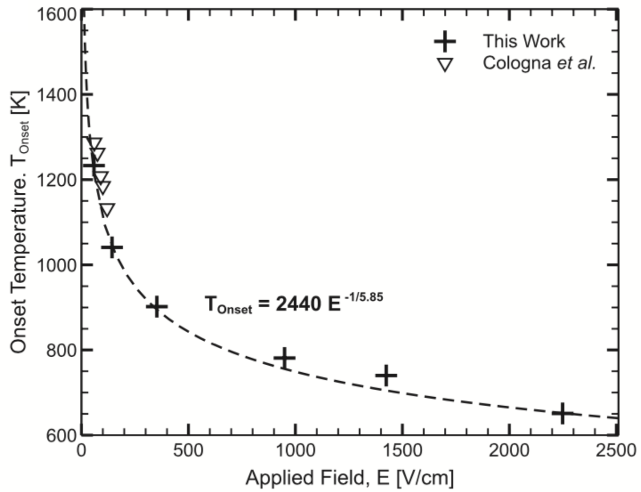
$$\Delta T_c \approx \frac{RT_f^2}{Q - 5RT_f}$$

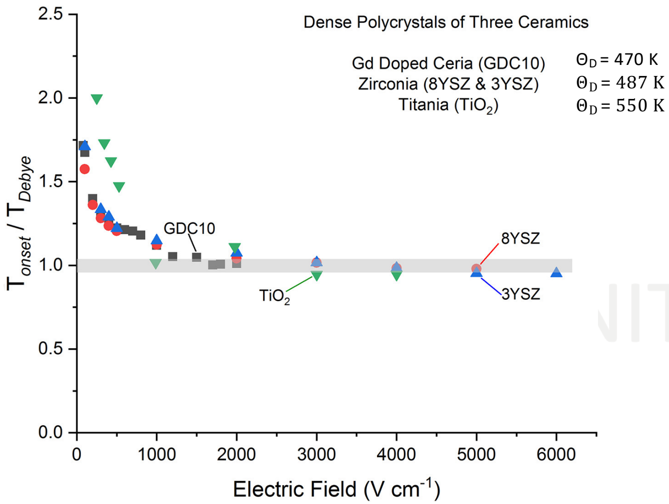
$$\Delta T_c = T_s - T_f \ll T_f$$



Electric field and onset temperature



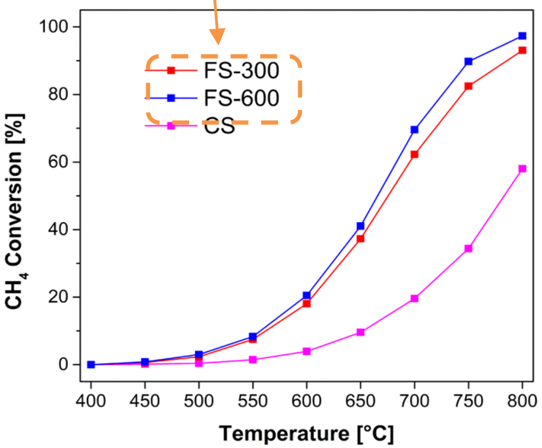


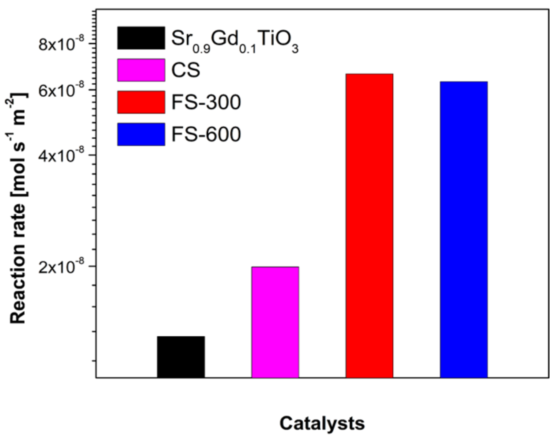


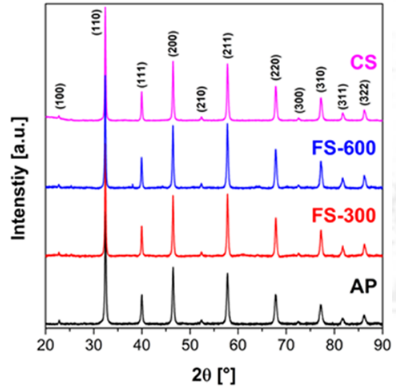
Useful defective structure from the high heating rate

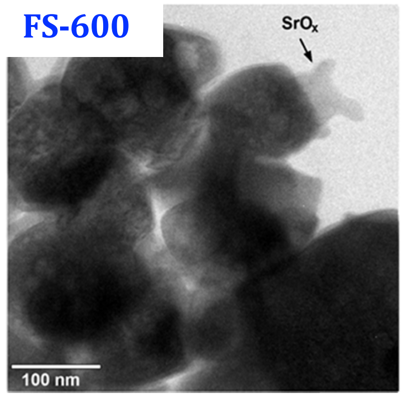
Chemical reactivity (CH_4 oxidation) of $La_{0.1}Sr_{0.9}TiO_3$

flash sintered $SrTiO_3$



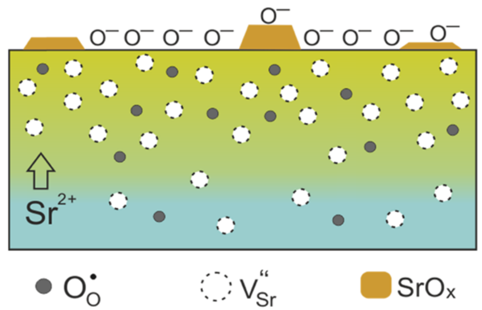






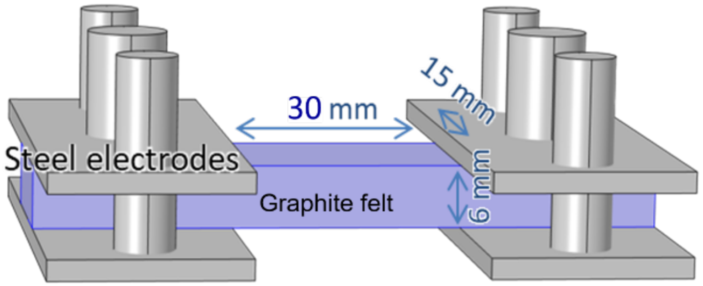
electric field ⟶

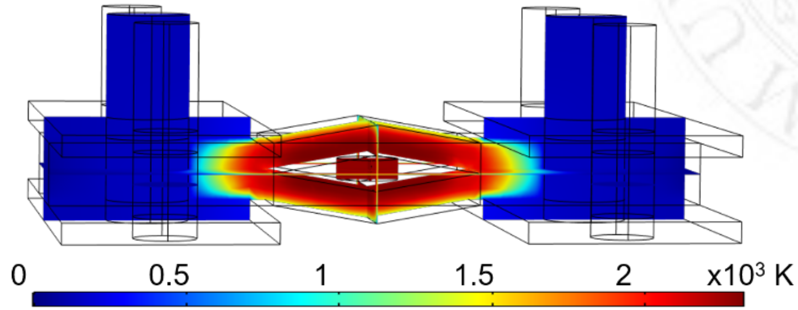
- ➢ *SrO_x segregation*
- ➢ *O^- defects*



Ultra-fast High-temperature Sintering (UHS)

Proposed in 2020





argon atmosphere

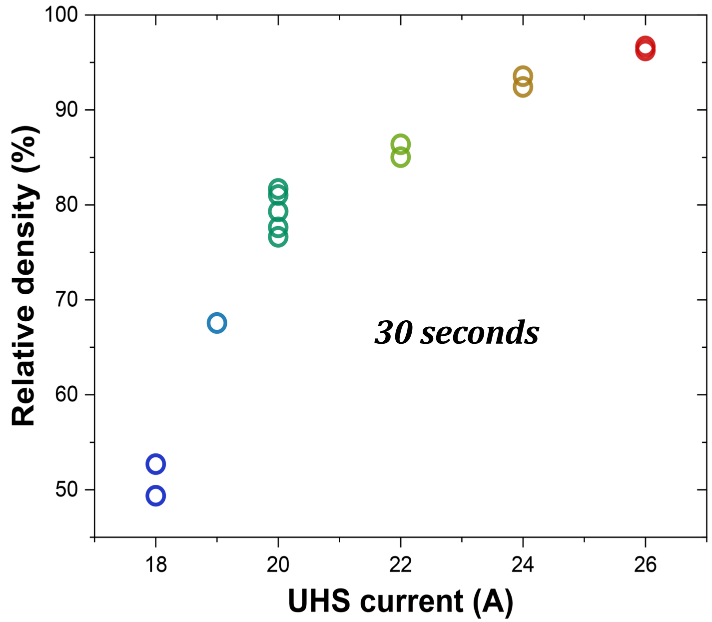
- Very high heating rates
- Possiblility to achieve 'very high' temperature
- Simple equipment (<10 k€)

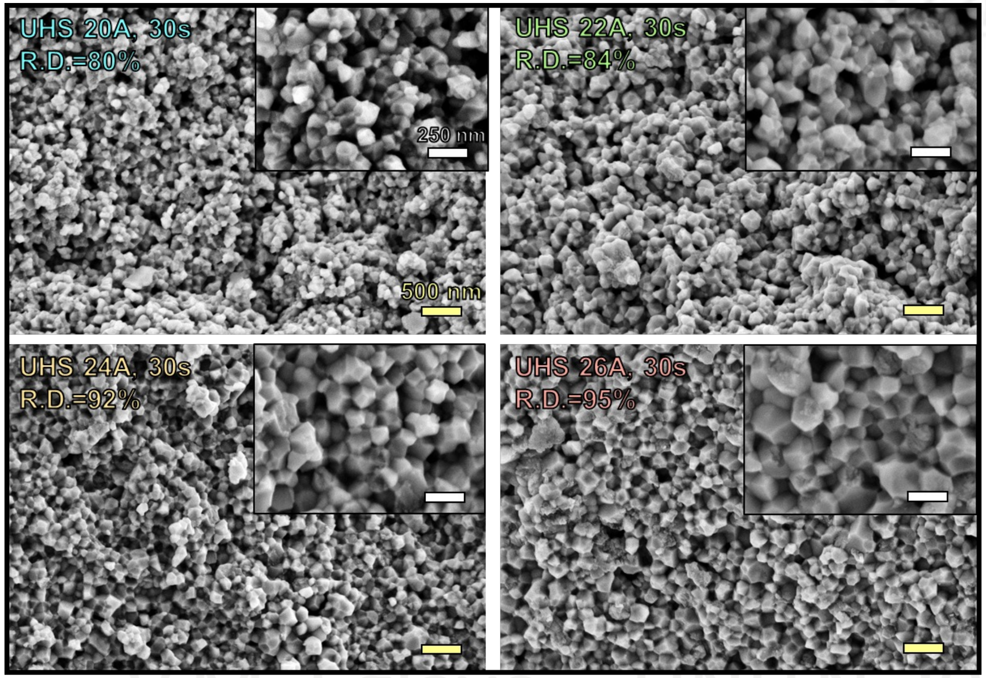
- *Small components*
- *Reducing conditions*

Typical UHS Process for LLZTO

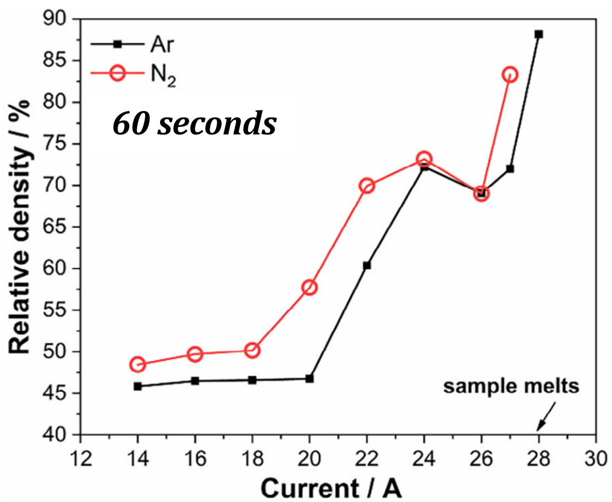
Heating rate: ~20,000 C/min
Sintering: ~6 s at ~1,500 C.

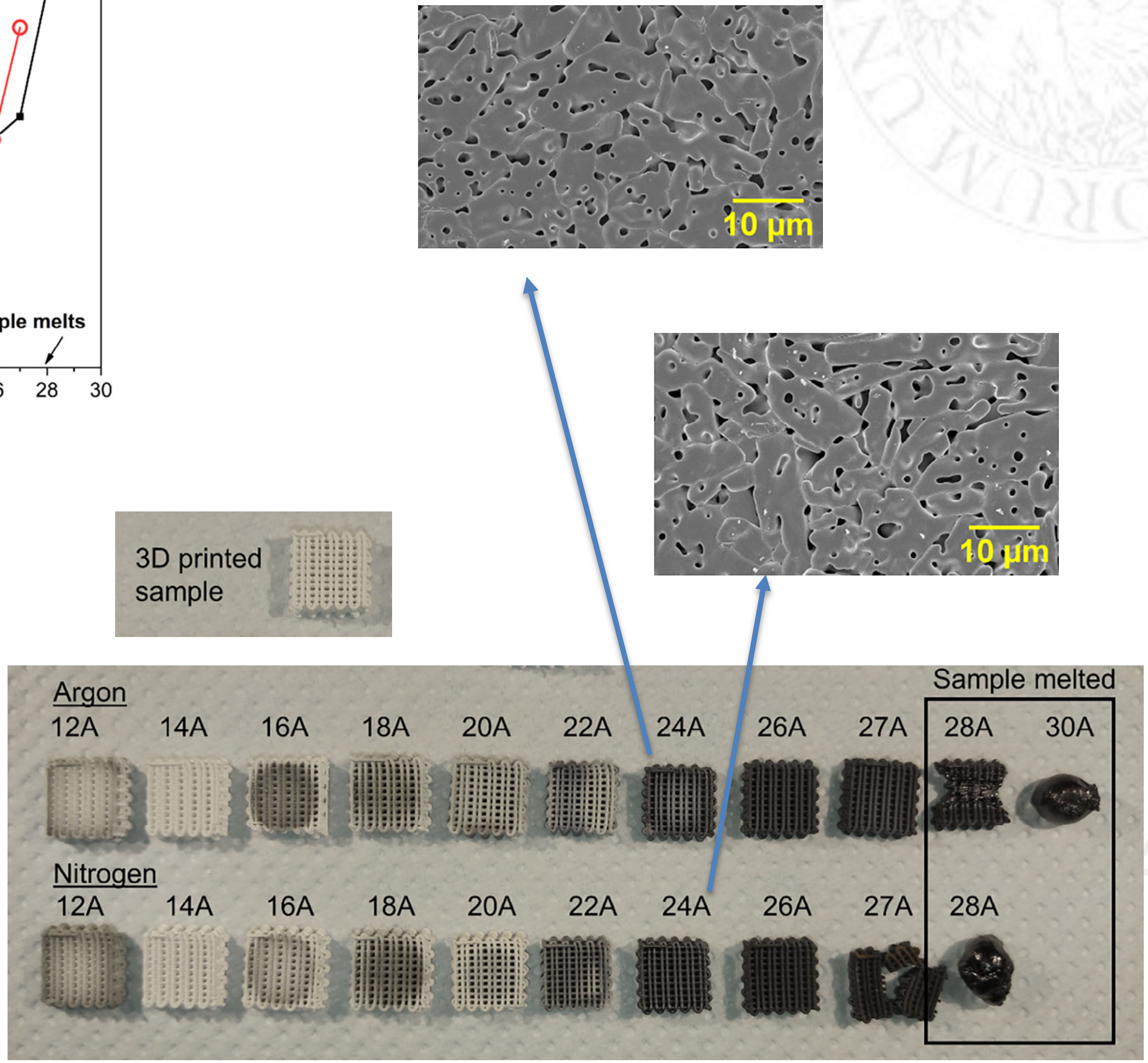
UHS of YSZ nanopowders





UHS of 3D printed $BaTiO_3$ bodies

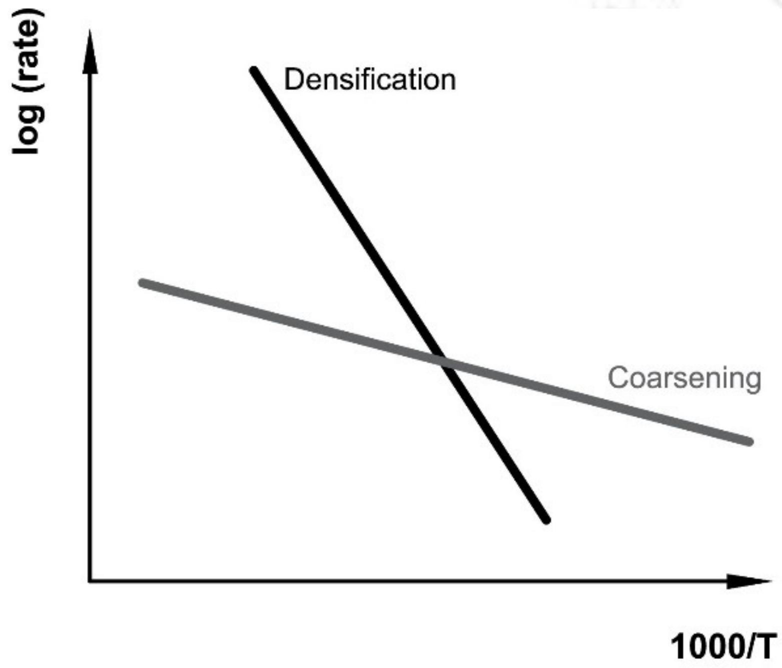




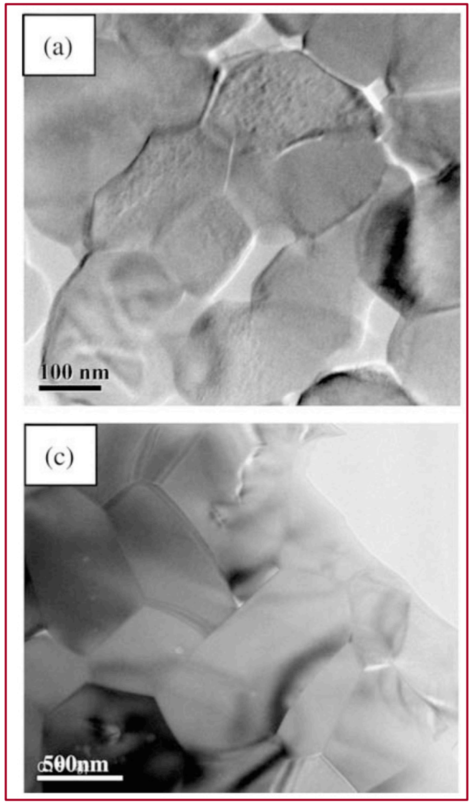
Very high heating rate effects

Enhanced densification

- Reduced coarsening / densification rate ratio



- Non-equilibrium grain boundaries?



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Thank you!
And enjoy the sun…