Università degli Studi di Trieste
Dipartimento di Ingegneria ed Architettura

Scienza e tecnologia dei materiali ceramici

Prof. Valter Sergo

Contributi di:
Federico Antonelli
Elisa Favero
Silvia Dalla marta

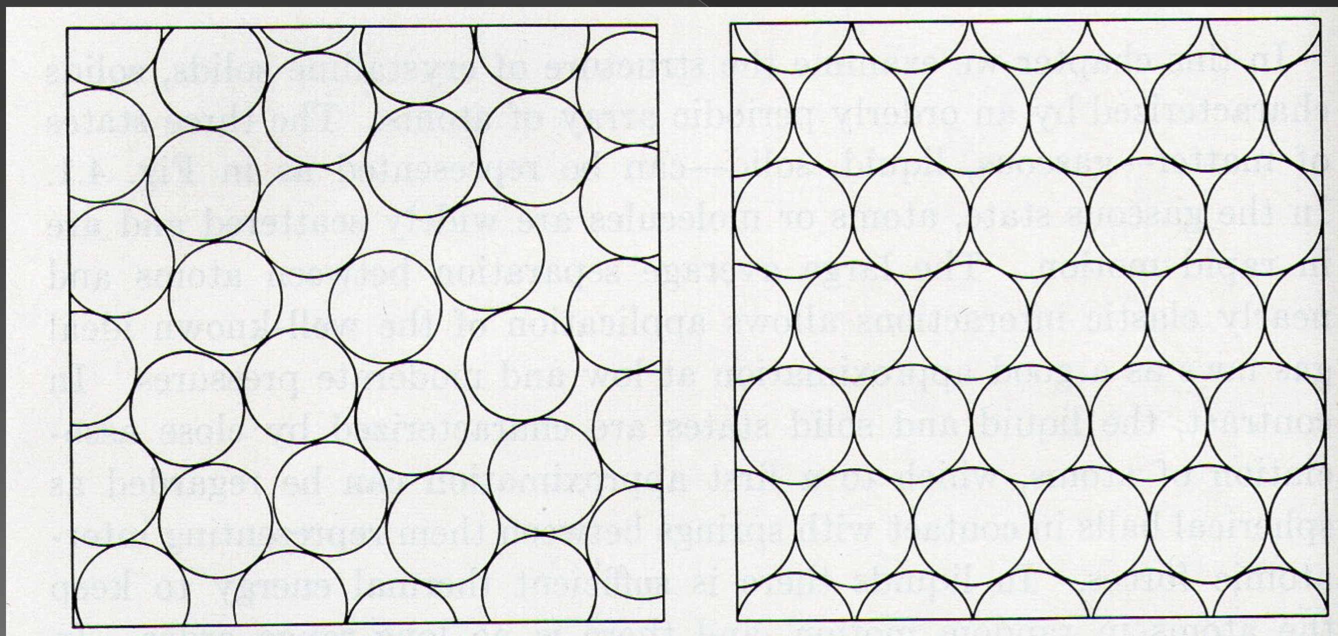
SINTERING PROCESS

Silvia Dalla Marta
(dal corso di Scienza e tecnologia dei materiali ceramici
prof. V. Sergo)

It is a thermal process of microstructural rearrangement in which the particles of powder are compacted and the porosity decreases to form a dense piece of ceramic.

monodisperse powder fcc or hcp: PF=74.5%

ceramic matherial with porosity:
before sintering



FORMATURE?

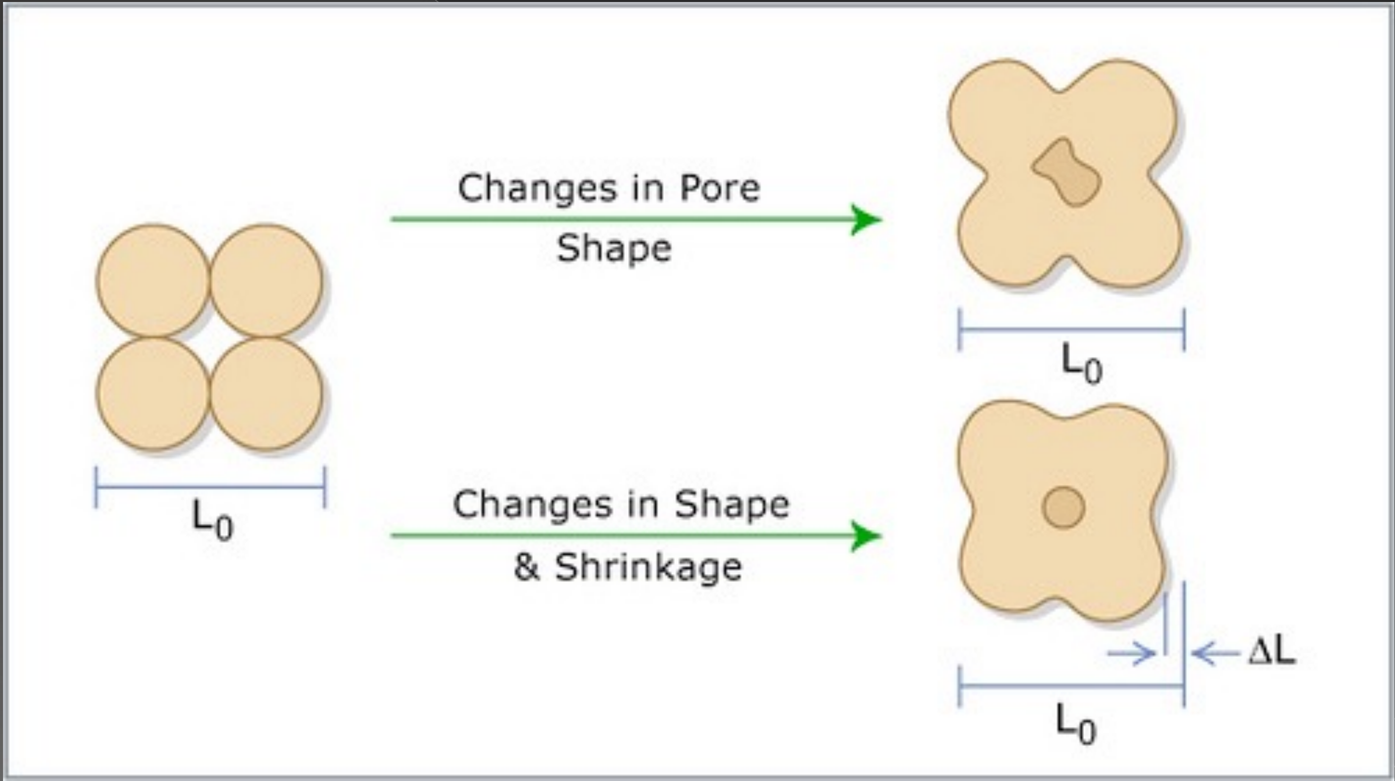
The absence of defects and porosity is very important for the *mechanical properties*:

$$K_{IC} = y\sigma\sqrt{c}$$

K: the parameter for the determination of the stress at the tip of the crack.
y: dimensionless constant that depends on defect's geometry and load
c: length of defect (m)

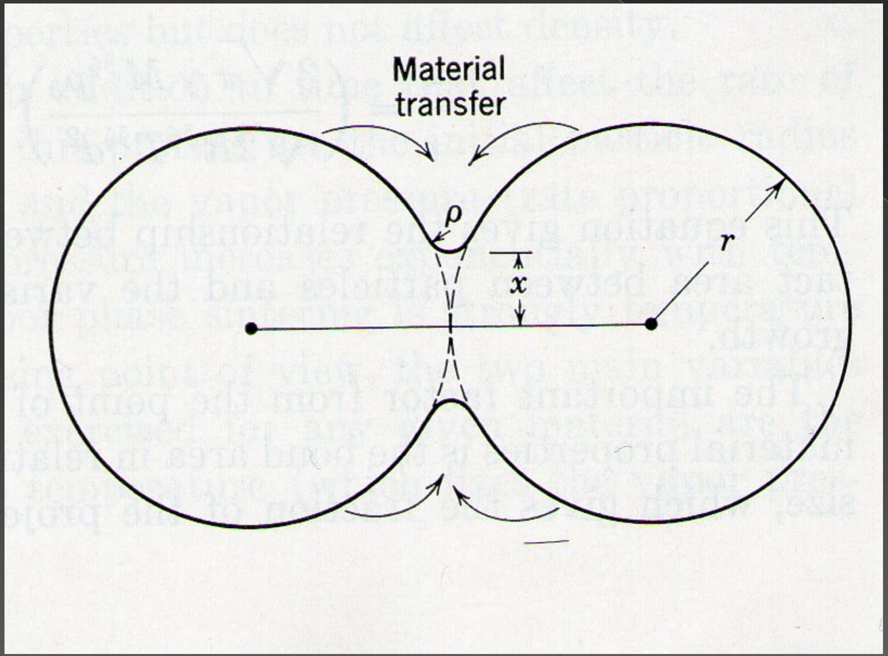
For the polycrystalline alumina: $K_{IC} = 3MPa\sqrt{m}$

In ceramics materials these values are very low compared to metals.
A very small defect or porosity lead to failure during an application of stress.


Changes in Pore
Shape
Changes in Shape
& Shrinkage
L_0
L_0
L_0
ΔL

BEFORE SINTERING:

- powder compact united by weaks Van der Waals forces
- individual grains separated by 25-60% of volume porosity



Considering two particles of ceramic material in contact with each other:

- concave zone
- convex zone

The atoms in a convex zone tend to *migrate* in a concave zone in according to a diffusion process actived by temperature.

DIFFUSION PROCESS

- Thermodinamically favored
- kinetically slow

FICK'S LAW (1D):

$$\frac{dC}{dt} = D\frac{d^2C}{dx^2}$$

Diffusion coefficient:

$$D = D_0 e^{-\frac{Ea}{RT}}$$

In order of kinetics to be fast enough for microstructural rearrangment to occur in *short time*, the sintering temperature must be:

$$T = \frac{2}{3}T_m$$

SINTERING MECHANISMS

- *SURFACE DIFFUSION*
- *VAPOR TRANSPORT*

↓

NO ***densification***

thinning of the particles

- *BULK DIFFUSION*
- *GRAIN BOUNDARY DIFFUSION*

↓

densification

decrease of the distance between particle centres

Thinning due to vapor phase matherial transfer:

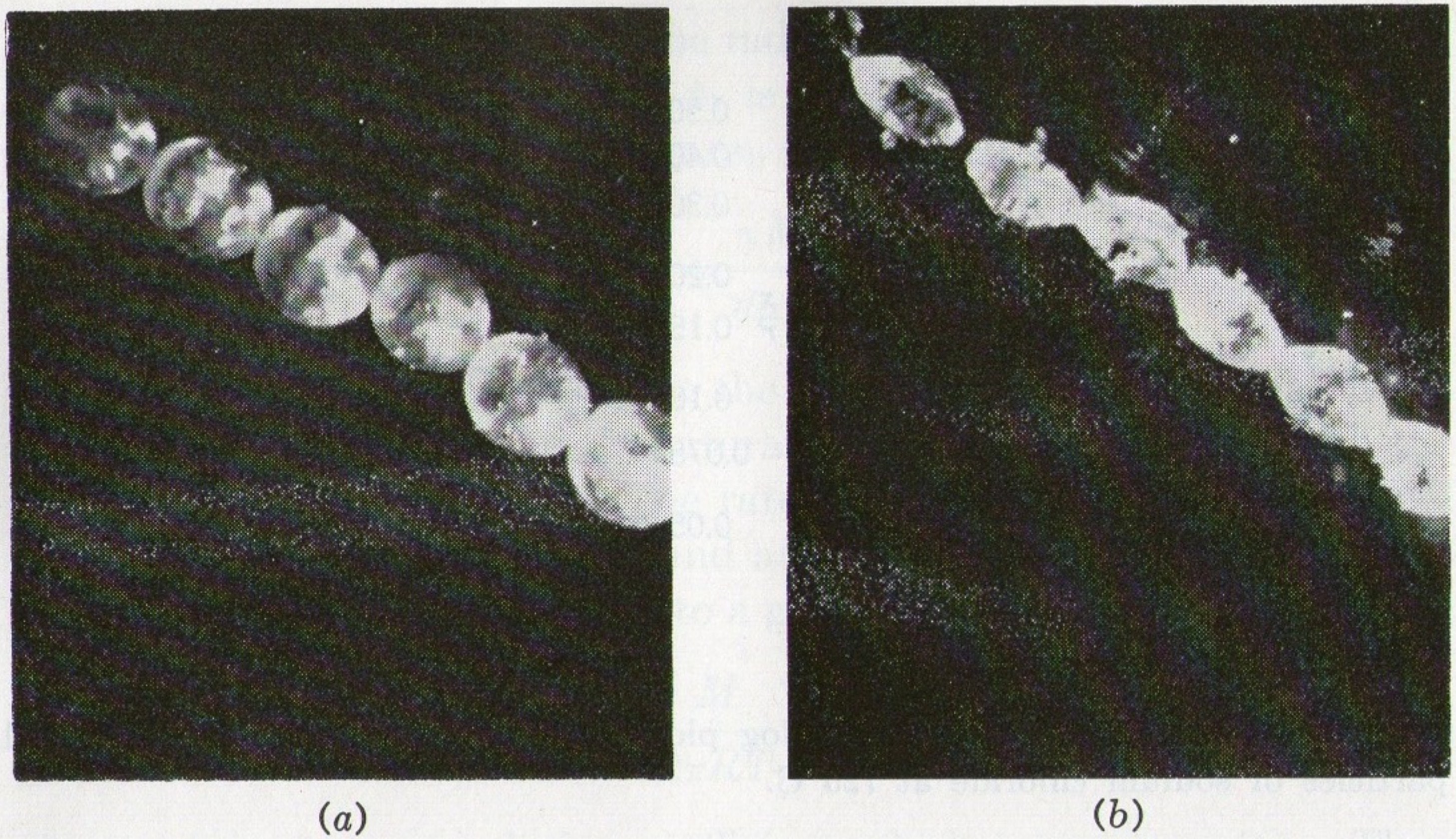
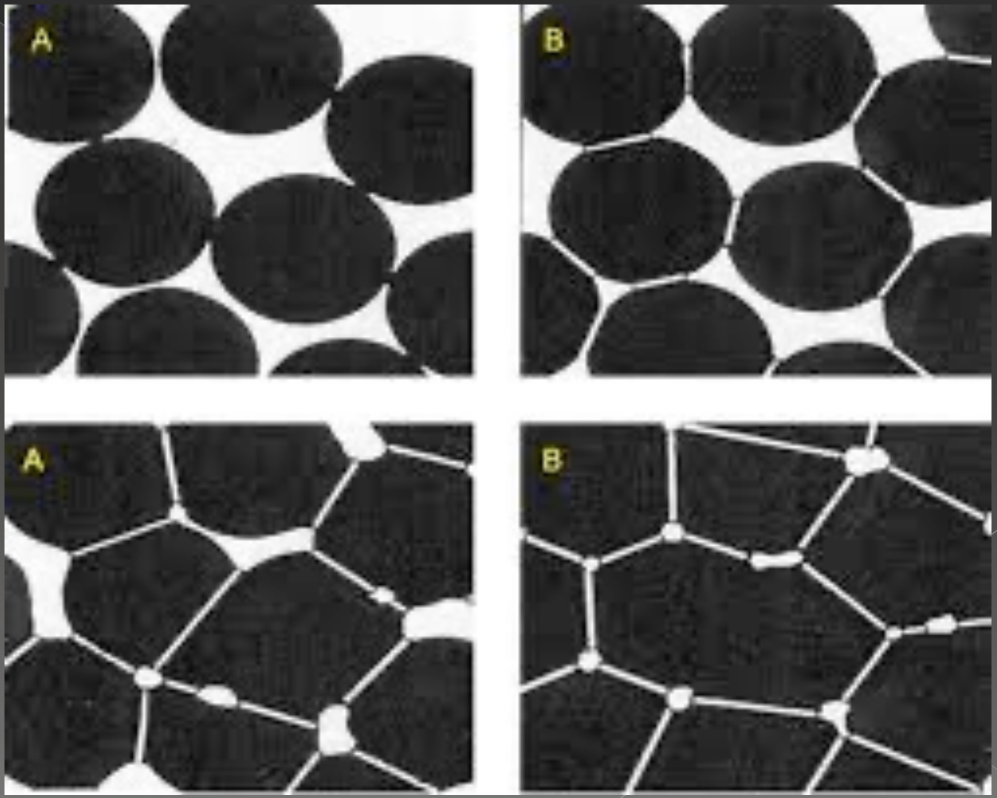


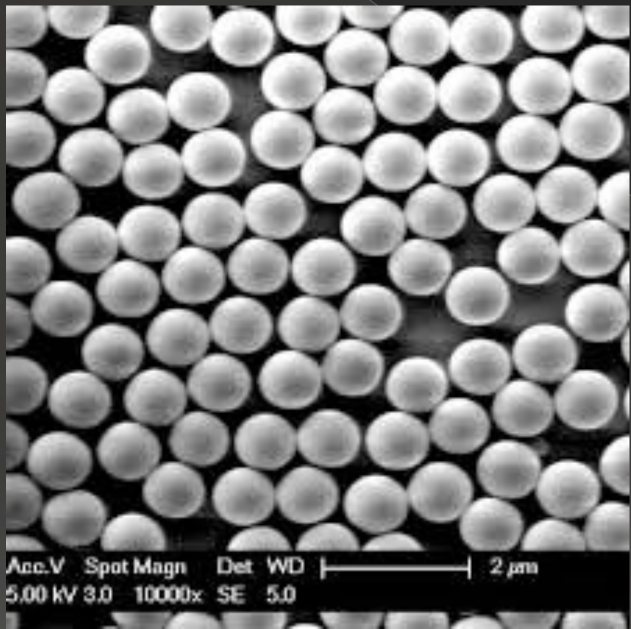
Fig. 12.17. Photomicrographs of sintering sodium chloride at 750°C: (*a*) 1 min, (*b*) 90 min.

DENSIFICATION:

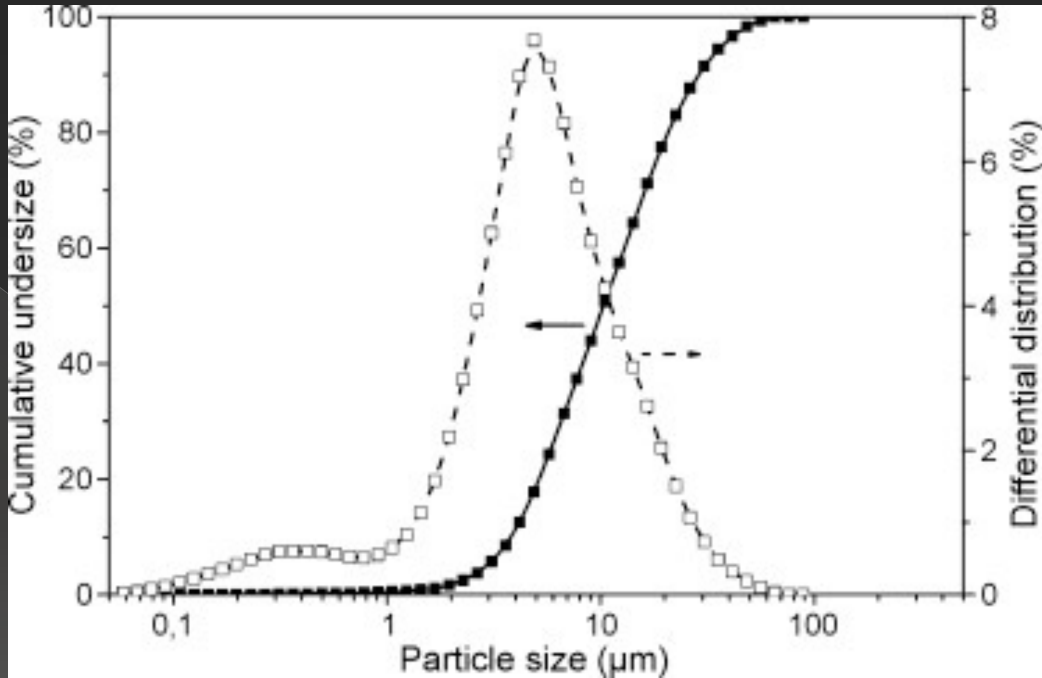
- atoms migration in the neck zone
- pores disappearence
- obtaining straight grain boundaries
- same chemical potential
- thermodynamically stable



Monodispersed powder:
rare and expensive!

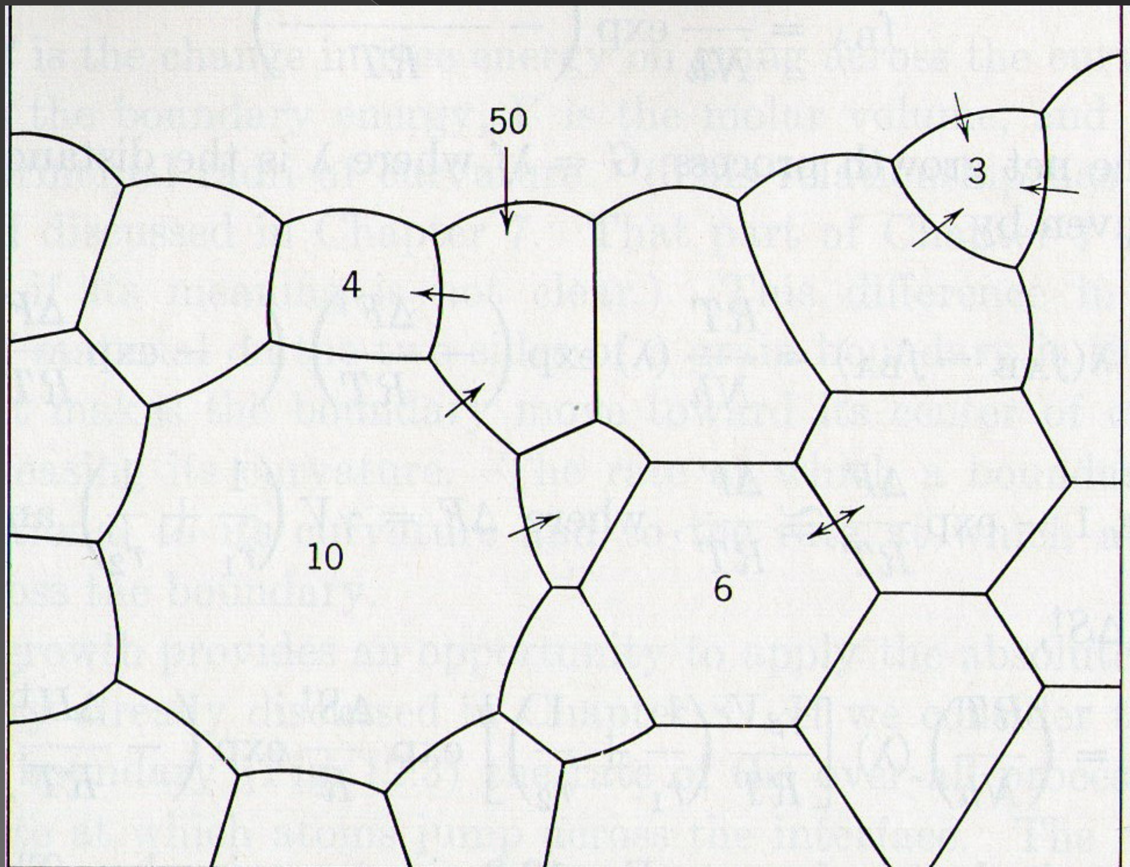


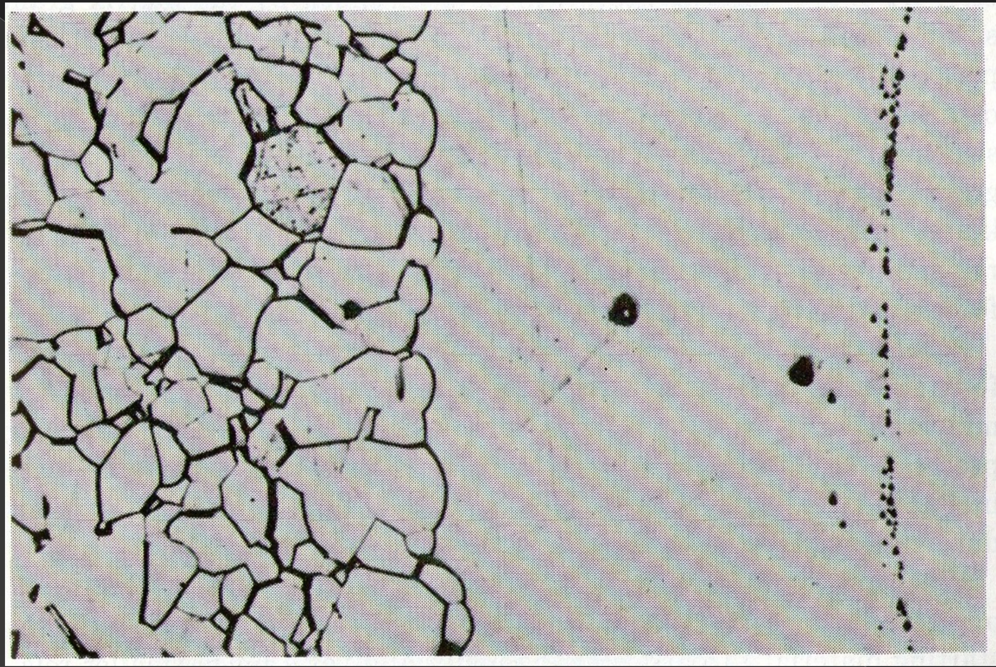
More frequent:
Grain size distribution!



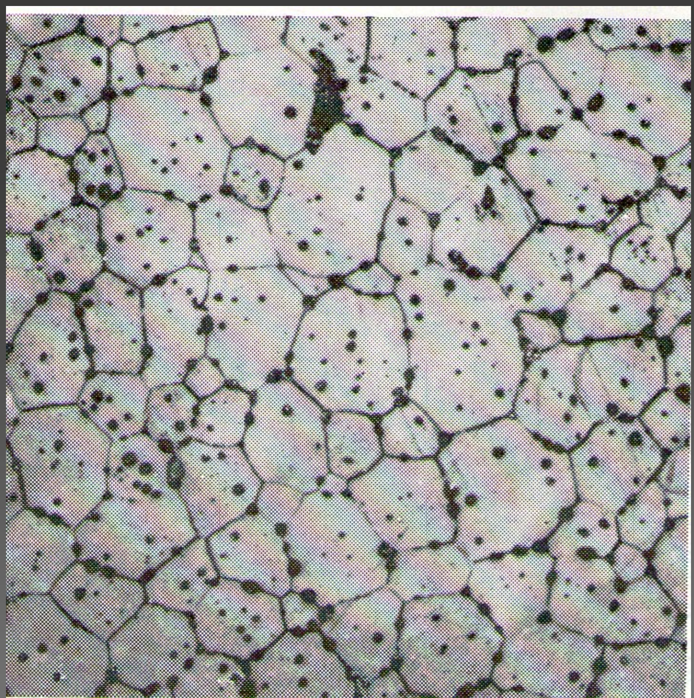
SECONDARY ABNORMAL OF BOUNDARY

Since grain boundaries migrate toward their centre of curvature,
grains with more than 6 sides tend to incorporate grains with less than 6 sides.





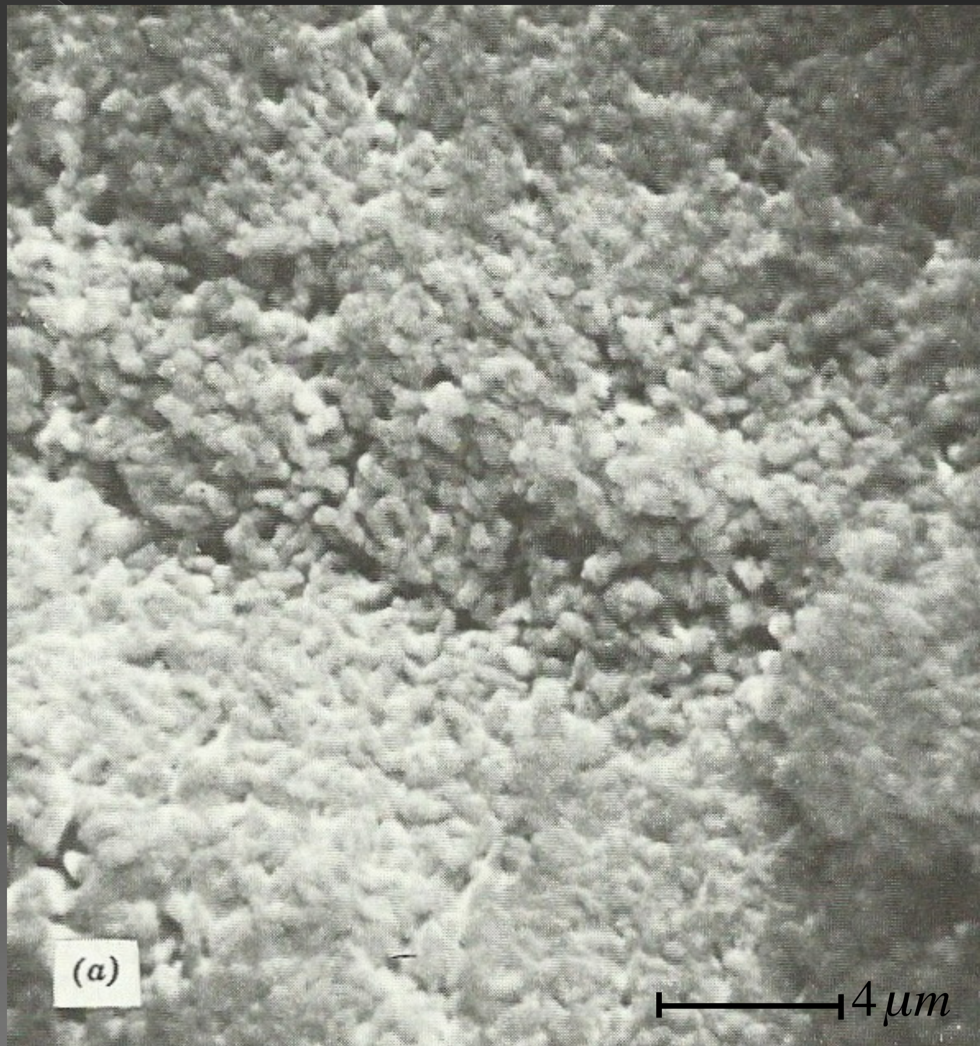
Growth of a large Al_2O_3 crystal into a matrix of uniformly sized grain.



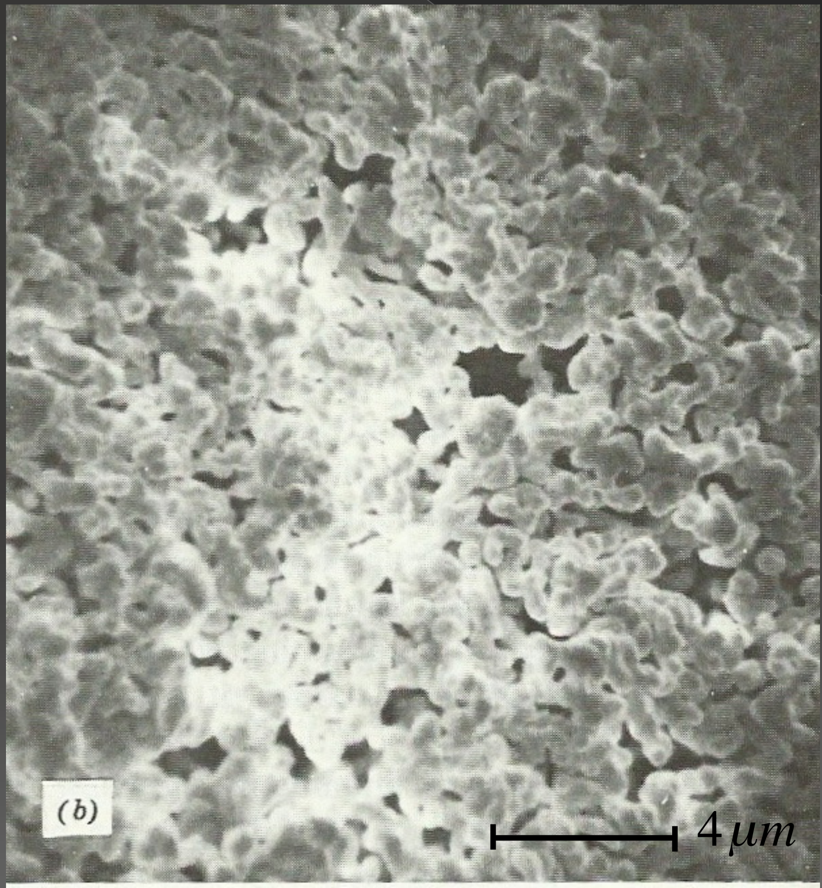
Polycrystalline flurite CaF_2 illustrating normal grain growth

PROGRESSIVE DEVELOPMENT OF MICROSTRUCTURE IN *LUCALOX ALUMINA*

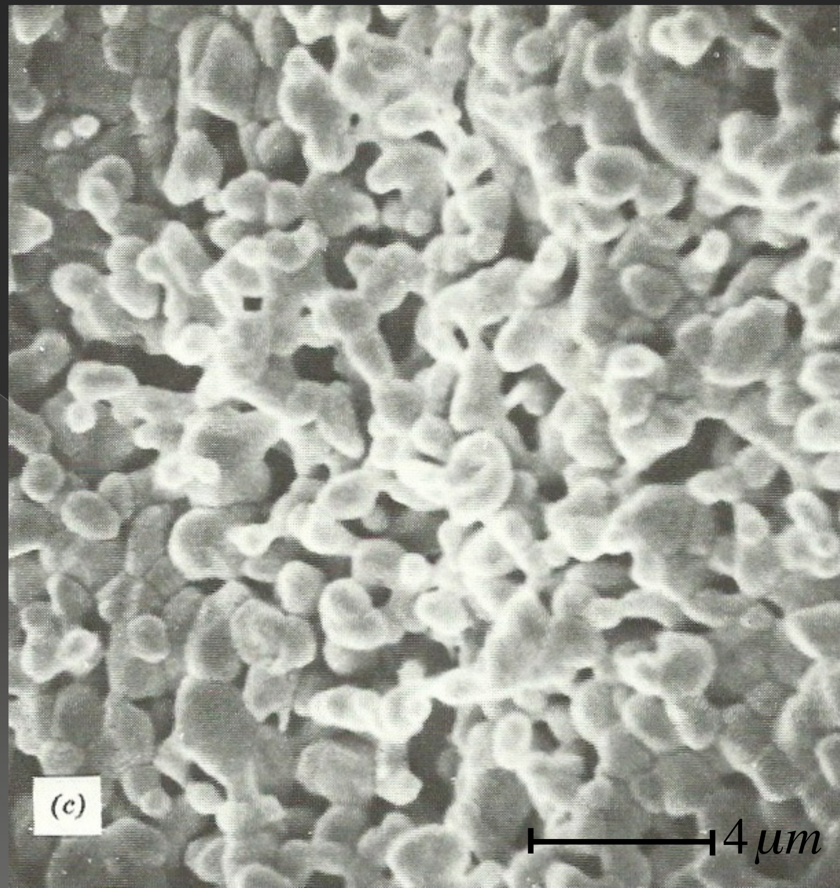
a) SEM of initial particles befor sintering (5000x)



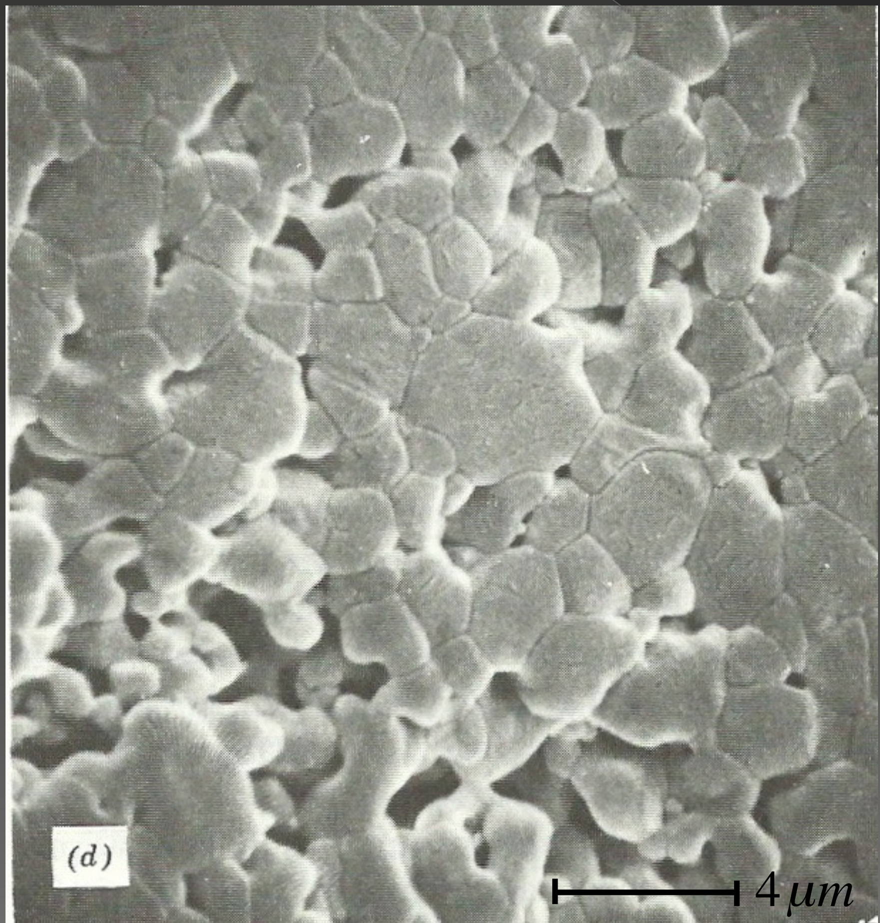
b) SEM of particles after 1 minute at 1700°C (5000x)



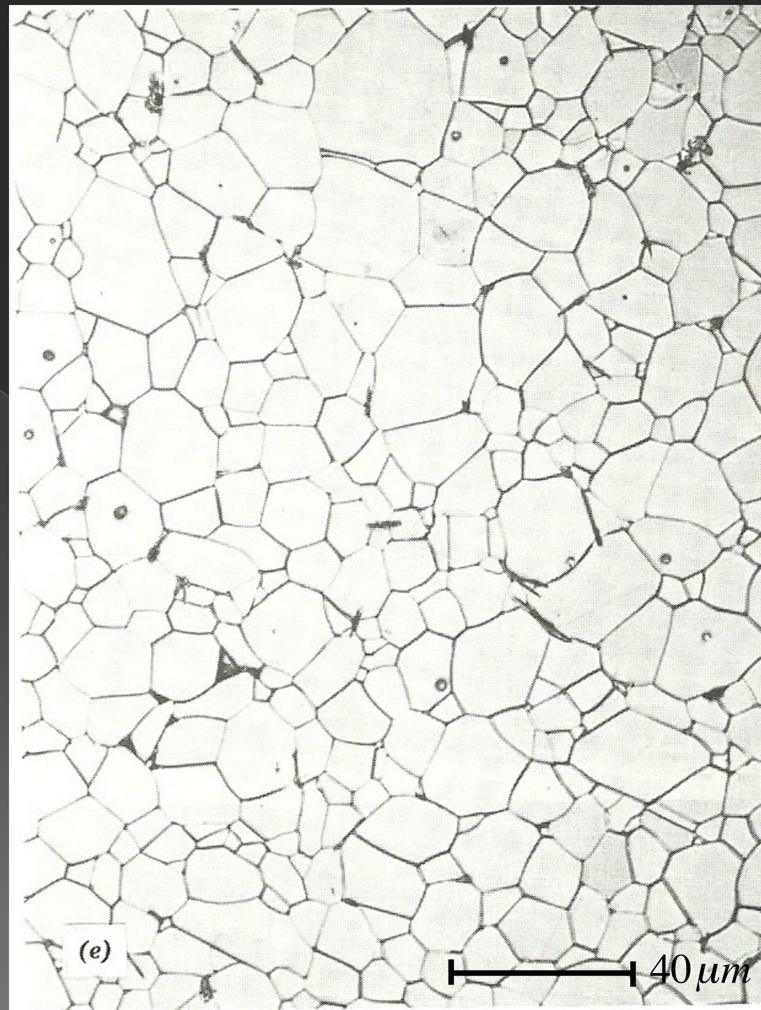
c) SEM of particles after 2 minutes at 1700°C (5000x)



d) SEM of particles after 6 minutes at 1700°C (5000x)

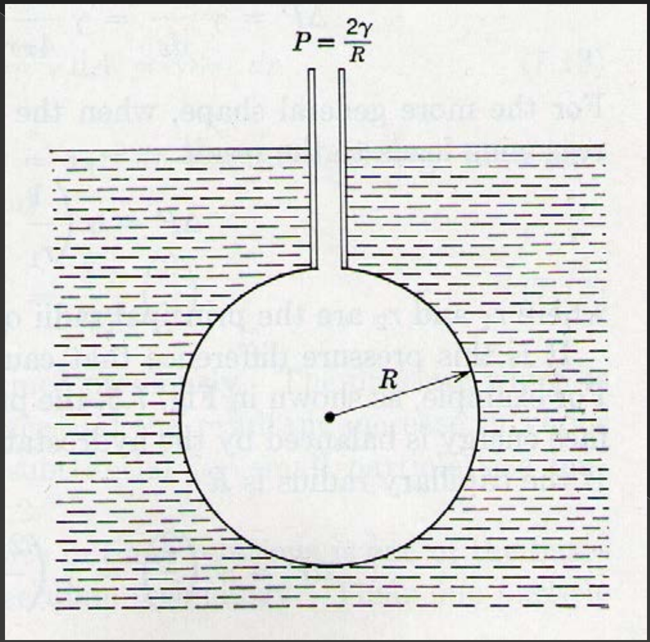


e) SEM of the final microstructure that is nearly porefree, with only a few pores located within grains (500x)



PRESSURE DIFFERENCE ACROSS A CURVED SURFACE

- The differences in the curvature of surface, causes a *pressure difference* in the various part of system, that leads to atoms transport.
- At the surface of the particle there is a positive radius of curvature, so that the vapour pressure is larger than would be observed in a flat surface.
- At the junction between particles there is a NECK whith a small negative radius of curvature and a vapour pressure lower than that for the particle itself.



P : Supplementary pressure to create the bubble.

γ: surface tension

SPHERICAL MODEL:

$$A = 4\pi R^2 \qquad V = \frac{4}{3}\pi R^3$$

$$p dV = \gamma_{LV} dA$$

$$\Delta p 4\pi R^2 dR = \gamma_{LV} 8\pi R dR$$

$$\Delta p = \frac{2\gamma_{LV}}{R}$$

GENERICALLY:

$$\Delta p = \gamma_{LV}\left(\frac{1}{R_1} + \frac{1}{R_2}\right)$$

$$H_2O(l) \rightleftharpoons H_2O(g)$$

in equilibrium condition

$$p° = K_e = e^{-\frac{\Delta G^0}{RT}}$$

vapour pressure of water
in a flat liquid-vapour interface

If the liquid-vapour interface is not flat, as in a small drops, the water has a vapour pressure that is larger than that in a flat surface:

$$e^{-\frac{\Delta G}{RT}} = e^{-\frac{\Delta G^0}{RT}} e^{-\frac{\bar{V}\Delta P}{RT}}$$

$$P_{H_2O} = P^0_{H_2O} e^{-\frac{2\gamma \bar{V}}{rRT}}$$

$P^0_{H_2O}$: standard vapour pression

ENERGY SURFACE

in a densification process in which the only energy is given by radius of curvature:

$$\overline{V} = \frac{MW}{\rho}$$

$$N = \frac{3MW}{4\pi a^3 \rho} = \frac{3\overline{V}}{4\pi a^3}$$

$$S_A = 4\pi a^2 N = \frac{4\pi a^2\, 3MW}{4\pi a^3 \rho} = 3\frac{\overline{V}}{a}$$

$$E_S = S_A \gamma = \frac{3\overline{V}\gamma}{a}$$

$\overline{V}$	: molecular volume	$\frac{cm^3}{mol}$
ρ	: density	$\frac{g}{cm^3}$
a	: particle radius	$\approx \mu m$
N	: number of particles in a mole of powder	
S_A	: surface area	m^2
E_S	:surface energy	$\frac{J}{mol}$
γ	: surface tension	$\approx 1\frac{J}{m^2}$
MW	: molecular weight	$\frac{g}{mol}$

- Energy available without added pressure in a sintering pocess of alumina:

$$E_S = \frac{3\overline{V}\gamma}{a} = 75\frac{J}{mol}$$

- Energy available whith added pressure in the same sintering:

$$w = P_A\overline{V} = 750\frac{J}{mol}$$

$$P = 30\,Mpa$$

$$\overline{V}_{Al_2O_3} = 25\cdot 10^{-6}\frac{m^3}{mol}$$

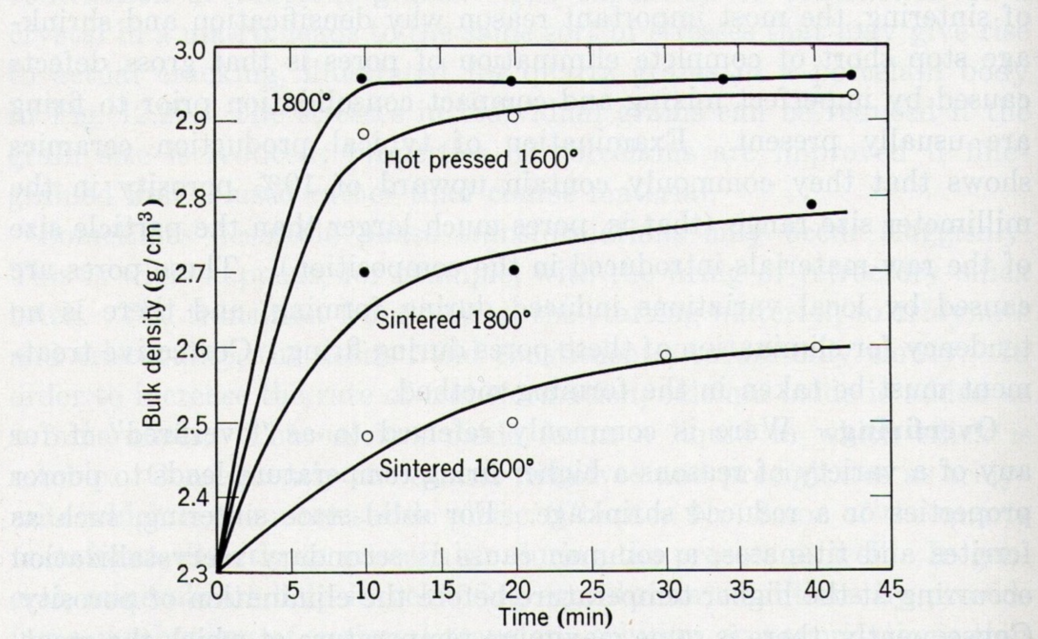


Fig. 12.30. Densification of beryllia by sintering and by hot pressing at 2000 psi.

Image from Kingery

DIFFUSION AND MOBILITY:

true in the absence of friction:

$$F = ma$$

otherwise:

$$F = m\frac{dv}{dt} + \frac{v}{M}$$

Einstein's generalized equation of mobility:

$$D = MRT$$

$\frac{v}{M}$: friction coefficient

M : mobility

D : diffusion coefficient

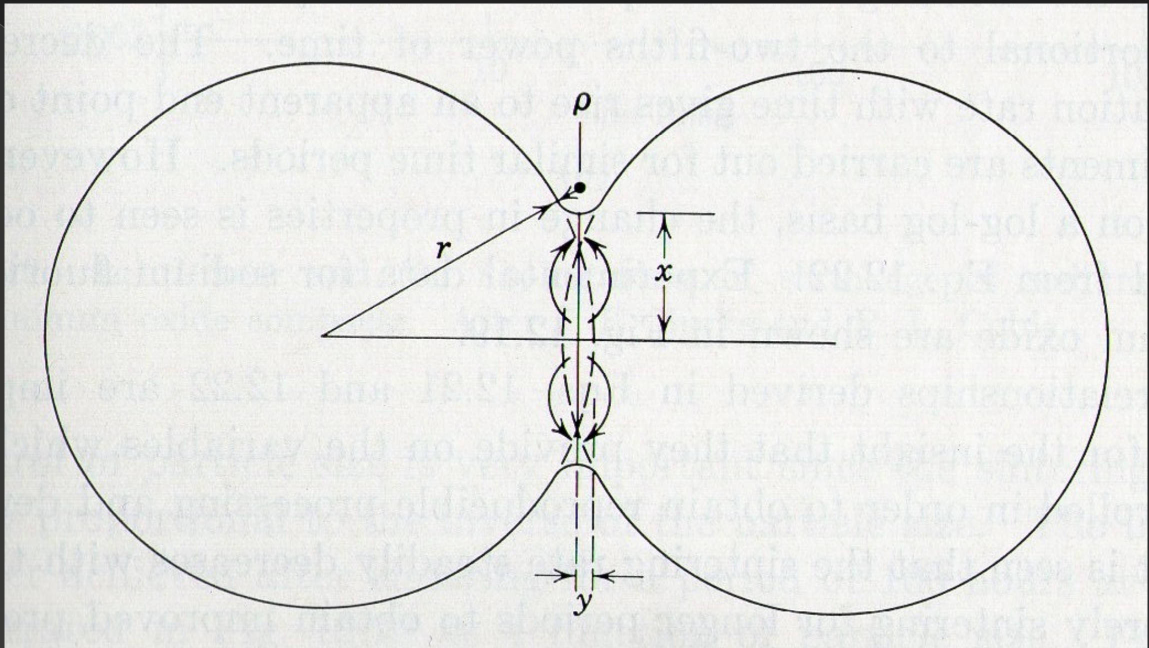
KINETIC MODELING OF SINTERING PROCESS

PARAMETERS TO DEFINE THE MODEL:

- *define a DRIVING FORCE*
- *define the GEOMETRY*
- *define the MECHANISM OF TRANSPORT*

STAGES OF THE SINTERING:

- *INITIAL STAGE :* from 50-55% to 75% of TD → *MODELING*
- *INTERMEDIATE STAGE :* from 75% to 92% of TD
- *FINAL STAGE :* from 92% to 100% of TD



ρ : radius of the neck's curvature

r : radius of particle

x : parameter indicated the progress of the sintering

GEOMETRY

$$(r+\rho)^2 = (r-\rho)^2 + (x+\rho)^2$$

$$\rho = \frac{x^2}{4r}$$

$$A_{Neck} = 2\pi x \cdot \pi\rho = \frac{\pi^2 x^3}{2r}$$

$$V_{Neck} = \frac{\pi x^4}{8r}$$

Approximations :

$\rho^2 = 0$

$x\rho = 0$

FLUX

The material transfer is linked to the flux.
Considering the area through which the transport takes (the neck area):

$$J = \frac{1}{A_{Neck}} \frac{d}{MW} \frac{dV_{Neck}}{dt}$$

d : density
MW : molecoular weight
J : flux

$$\frac{dV_{Neck}}{dt} = \frac{4\pi x^3}{8r}\frac{dx}{dt} = \frac{\pi x^3}{2r}\frac{dx}{dt}$$

Variation of the neck volume based on the increase of the 'x' parameter:

$$J = \frac{2r}{\pi^2 x^3}\frac{d}{MW}\frac{\pi x^3}{2r}\frac{dx}{dt} = \frac{1}{\pi \bar{V}}\frac{dx}{dt}$$

FLUX expressed as a DRIVING RORCE

$$J = cMF$$

c : concentration

M : mobility of bulk and grain boundary atoms

F : force

$$M = \frac{D}{RT}$$

$J = cMF$

$$F = -\nabla G = -\frac{dG}{dx} \approx \frac{dG}{\rho}$$

Variation of the free energy during the diffusion on the neck area:

$$\Delta G = \Delta p \overline{V} = \overline{V}\gamma\left(\frac{1}{r_1} + \frac{1}{r_2}\right) = \overline{V}\gamma\left(\frac{1}{x} - \frac{1}{\rho}\right) = \frac{\overline{V}\gamma}{\rho}$$

$$F = \frac{\Delta G}{\rho} = \frac{\overline{V}\gamma}{\rho^2}$$

$$J = cMF = c\frac{D}{RT}\frac{\overline{V}\gamma}{\rho^2}$$

$$\frac{1}{\overline{V}\pi}\frac{dx}{dt} = \frac{cD}{RT}\frac{\overline{V}\gamma}{\frac{x^4}{16r^2}}$$

integration between o and x

t=o, x=o

$$\frac{1}{5}x^5 = \frac{5\pi\overline{V}^2 cD\gamma r^2}{RT}t$$

$$x = \left(\frac{5\pi\overline{V}^2 cD\gamma r^2}{RT}\right)^{\frac{1}{5}} t^{\frac{1}{5}}$$

t: sintering time

Variation of the volume of the particles in the sintering process during the time:

Variation of the relative density variatung time and temperature:

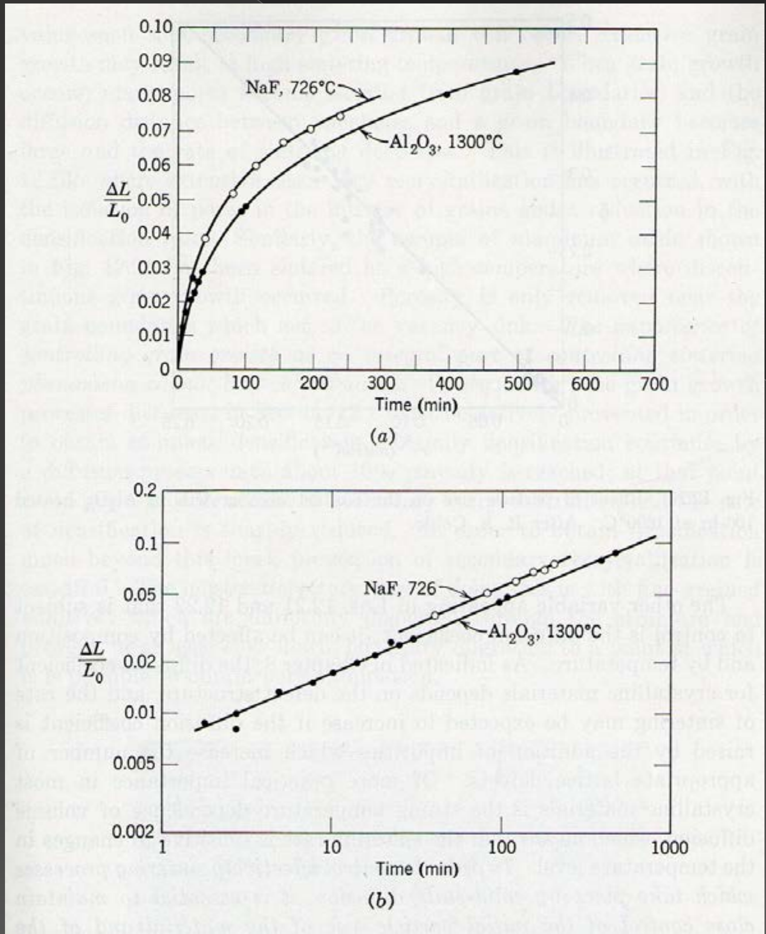


Fig. 12.19. (a) Linear and (b) log-log plots of shrinkage of sodium fluoride and aluminum oxide compacts. After J. E. Burke and R. L. Coble.

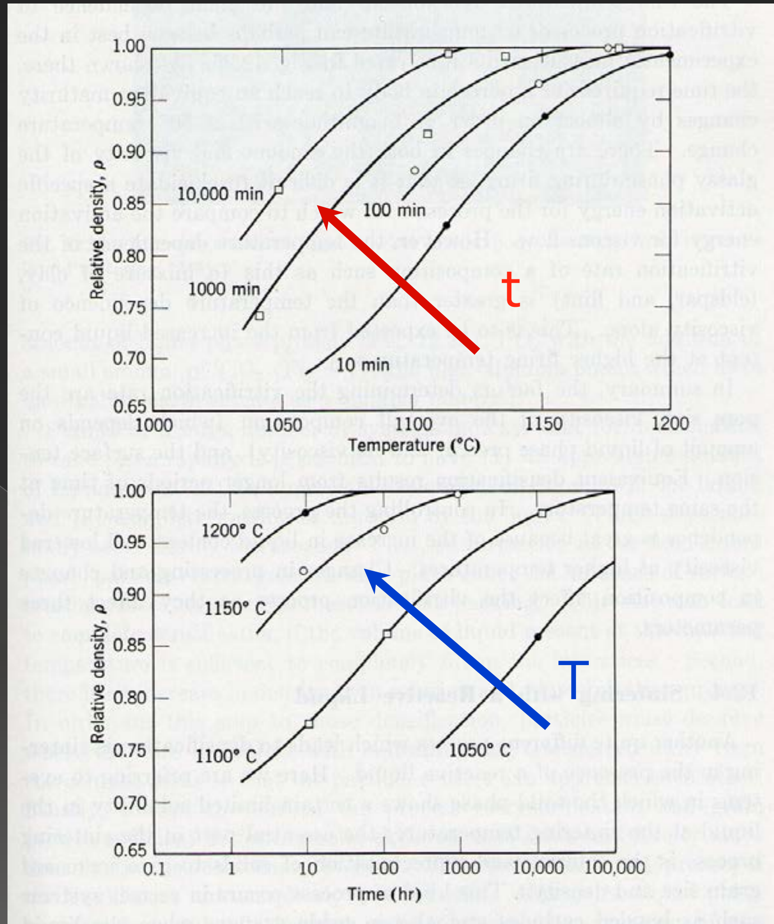


Fig. 12.25. Effect of time and temperature on the vitrification of a porcelain body. Data from F. H. Norton and F. B. Hodgdon, *J. Am. Ceram. Soc.*, **14**, 177 (1931).

The increase of a few degrees in temperature has much more influence on the grain size than the increase of a one order of magnitude of the time

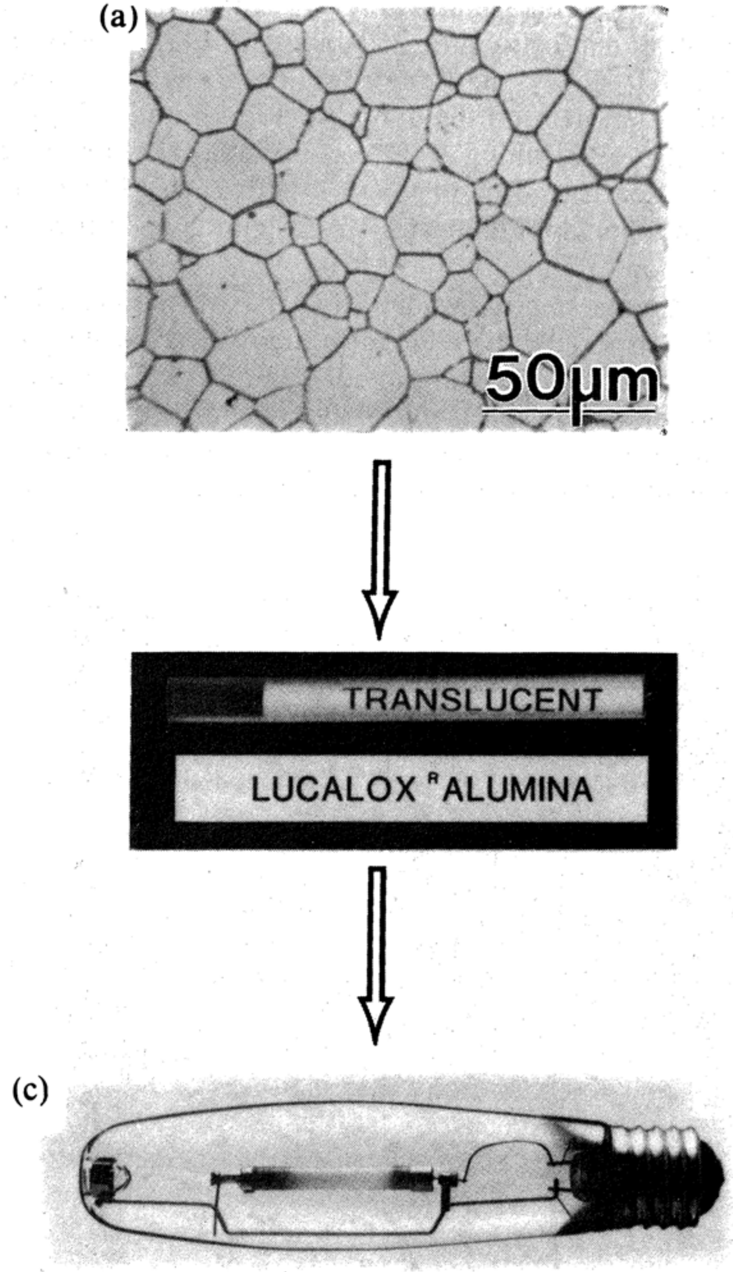


Figure 11.9 Comparison of the microstructure and translucency of relatively pore-free Al_2O_3 (a) with that of opaque Al_2O_3 containing pores trapped in grains (b). Translucent Al_2O_3 tubes are used in sodium vapor lamps that provide energy efficient street lights. (Courtesy of General Electric.)

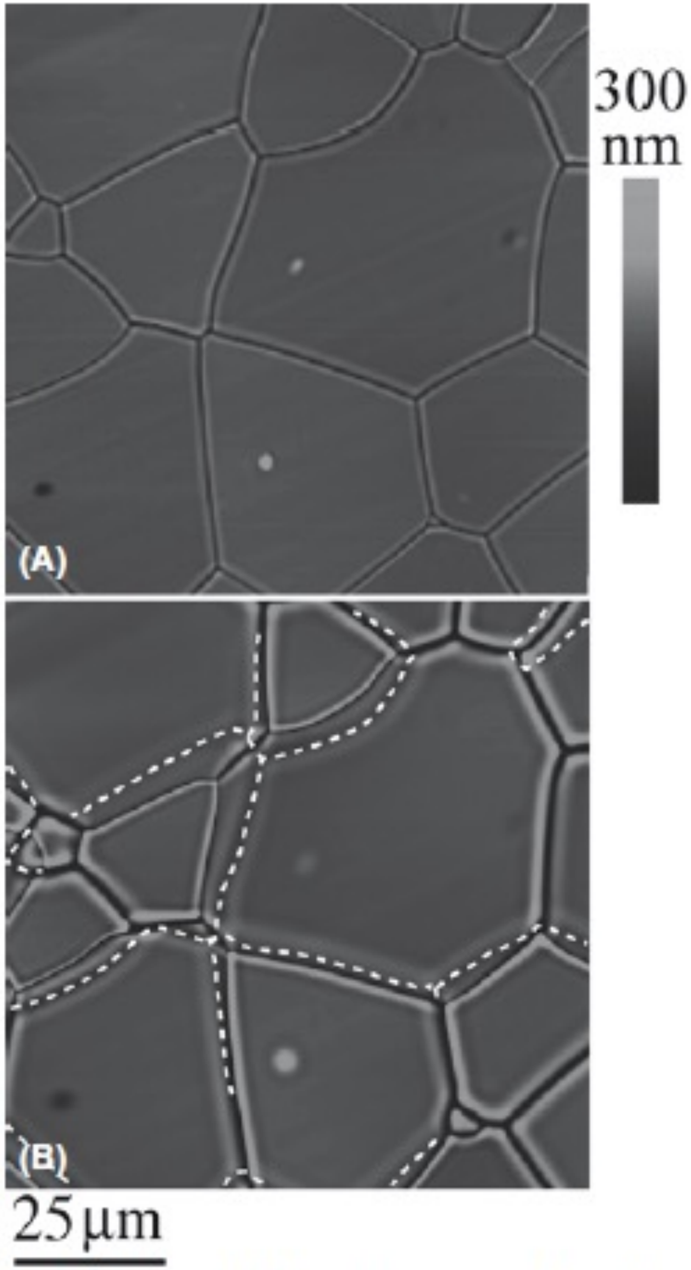


FIGURE 24.17 (a, b) AFM of grooves at migrating GBs

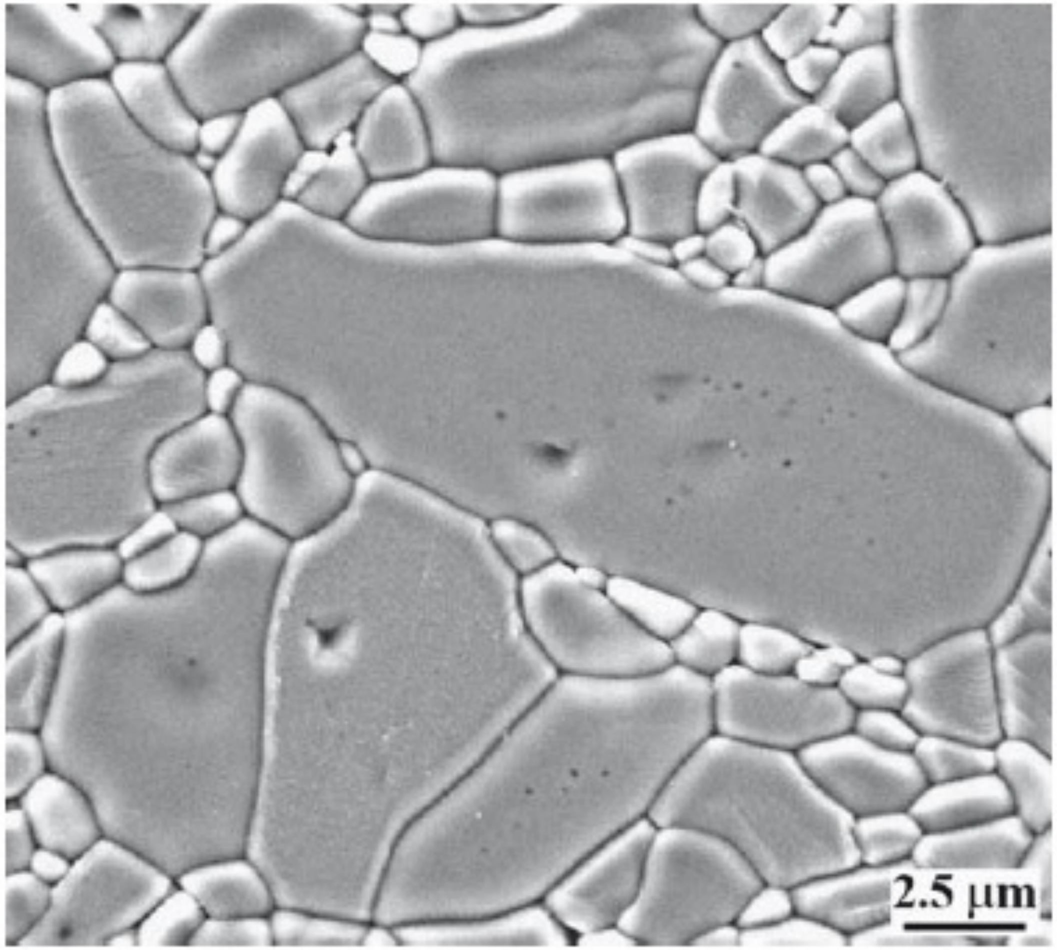
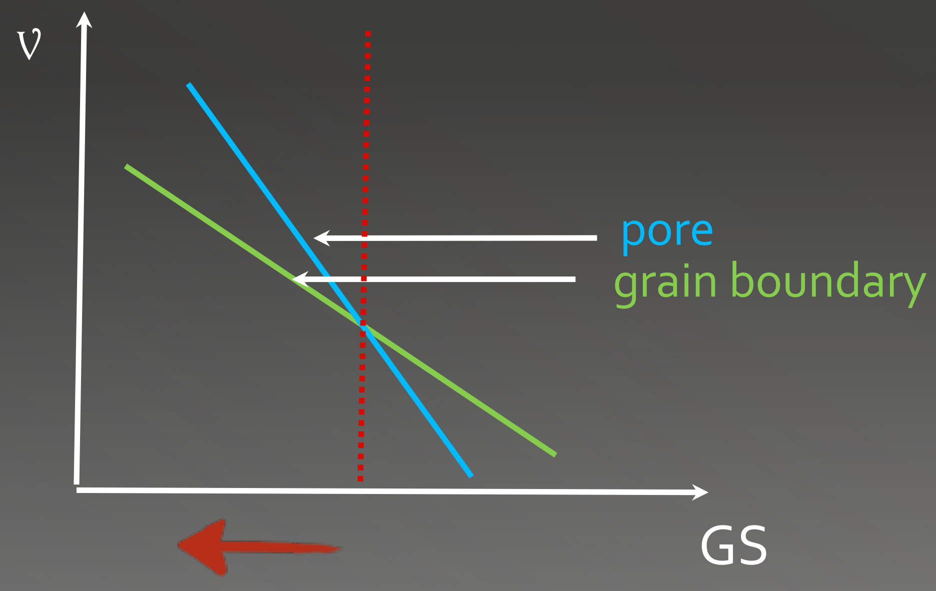


FIGURE 24.21 Elongated exaggerated grain in Al_2O_3.

- During the growth, the larger grain leaves behind a lot of pores and the piece can not achive the 100% of theoretical density (DT).
- To avoid the pore incorporation inside the grain, the speed of grain boudaries must be lower than that of the pores.
- Some impurities can segregate on grain boundary (*GRAIN BOUNDARY PINNING)* slowing the growth and so it’s possible to achive the 100% of DT.

v pore > v grain boundary



E.G. :

ALUMINA ‘LUCALOX ‘:
polycrystalline Al_2O_3 - 1% MgO

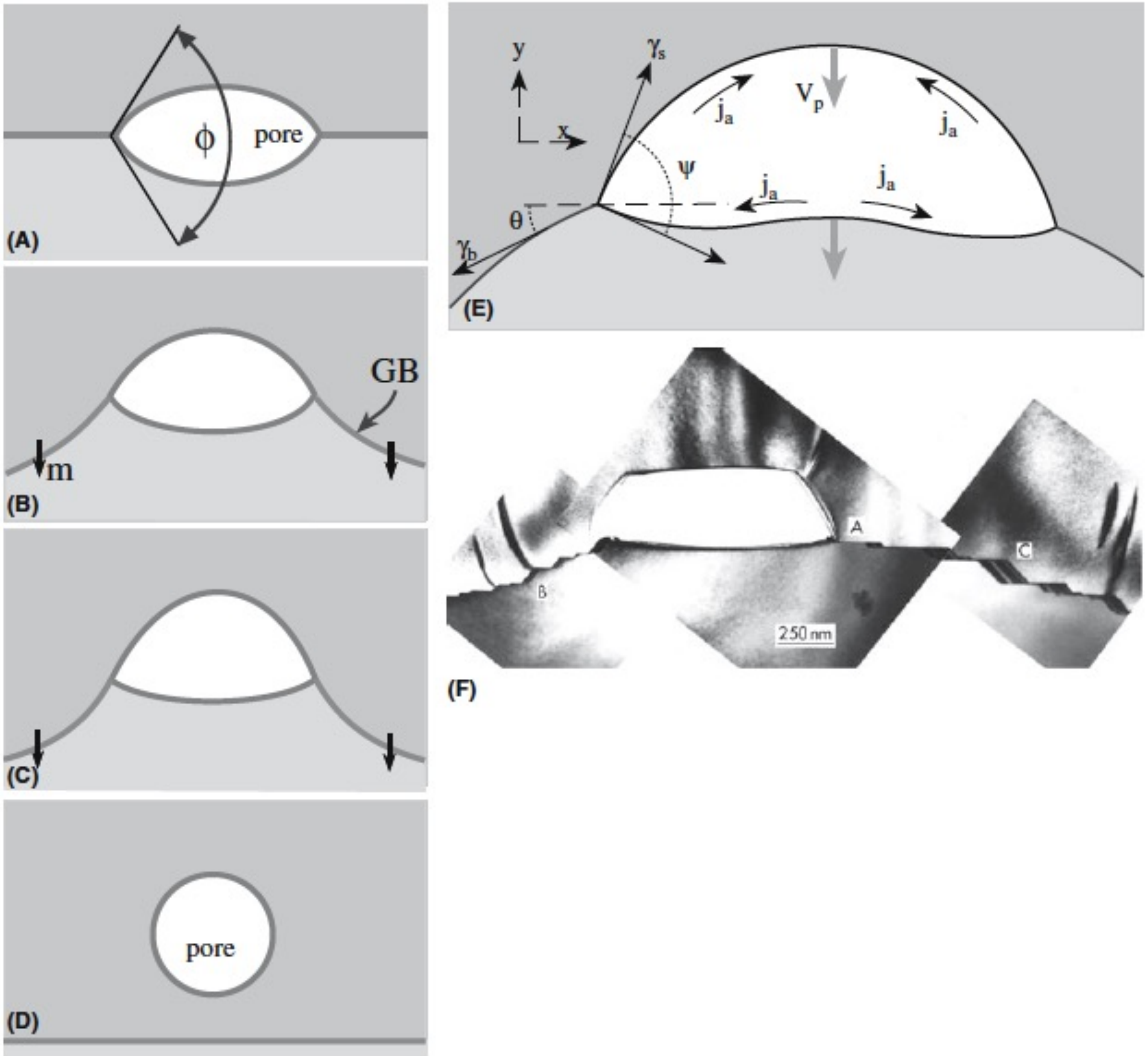
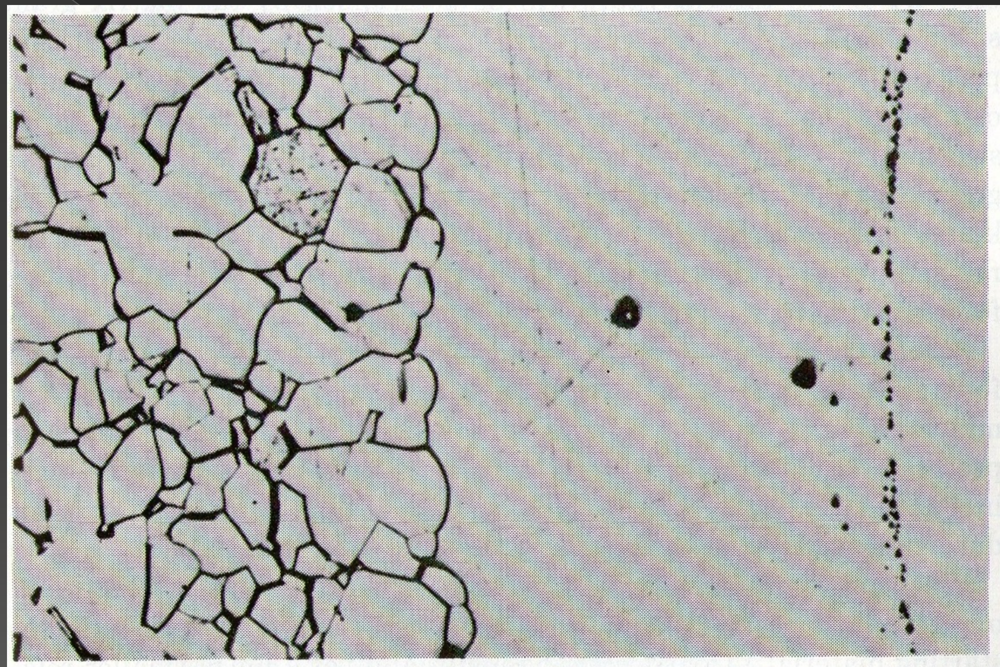


FIGURE 24.14 (a–e) GB/pore interaction: the break-away process.



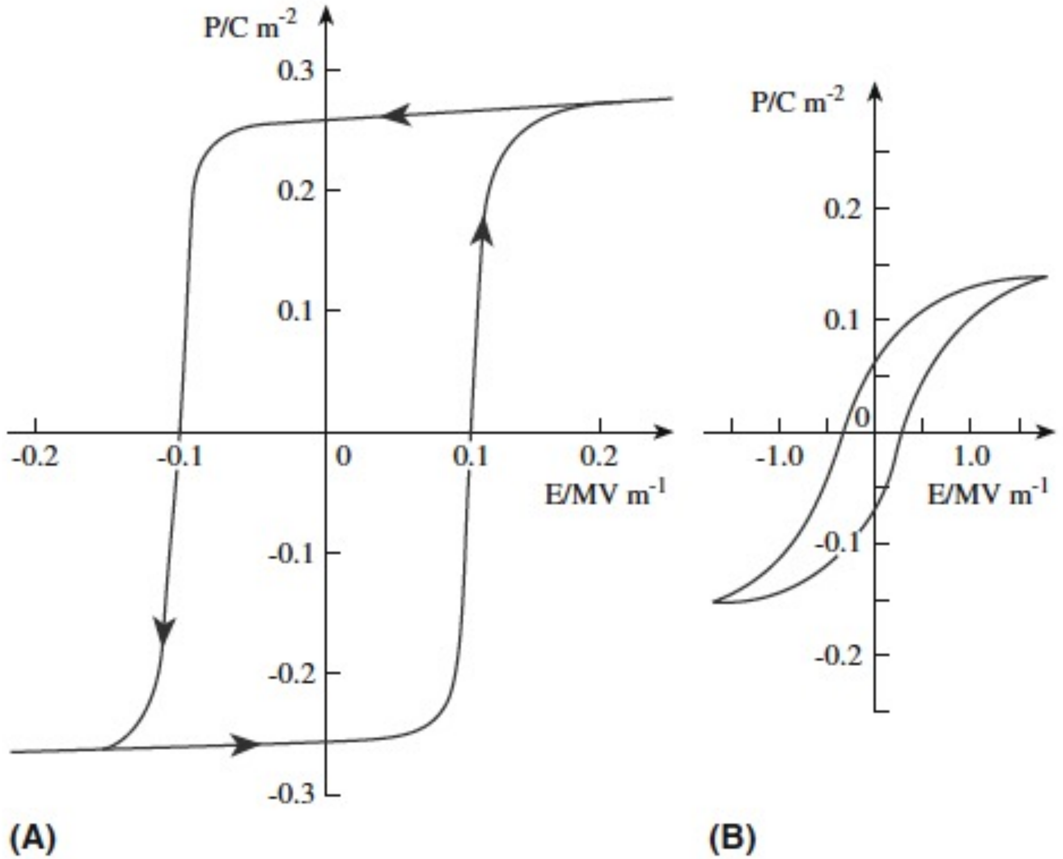


FIGURE 31.10 Hysteresis loops for $BaTiO_3$. (a) Single-domain single crystal. (b) Polycrystalline ceramic.

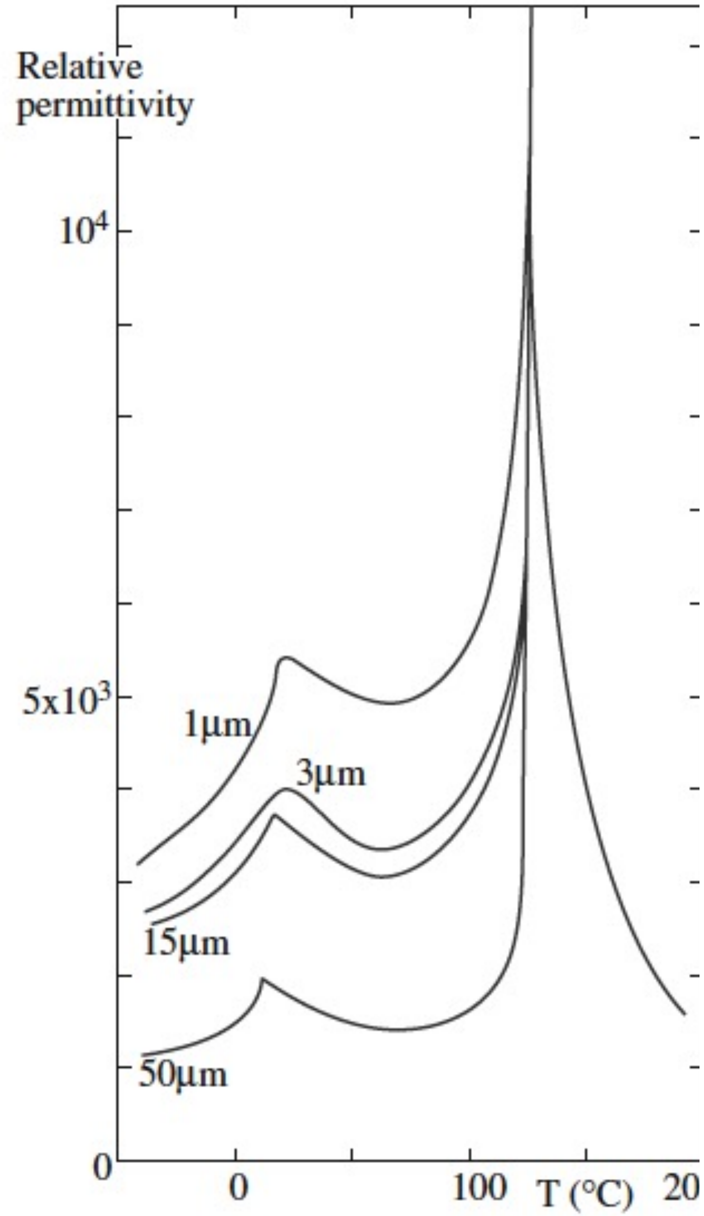


FIGURE 31.15 Effect of grain size on the dielectric con
$BaTiO_3$.

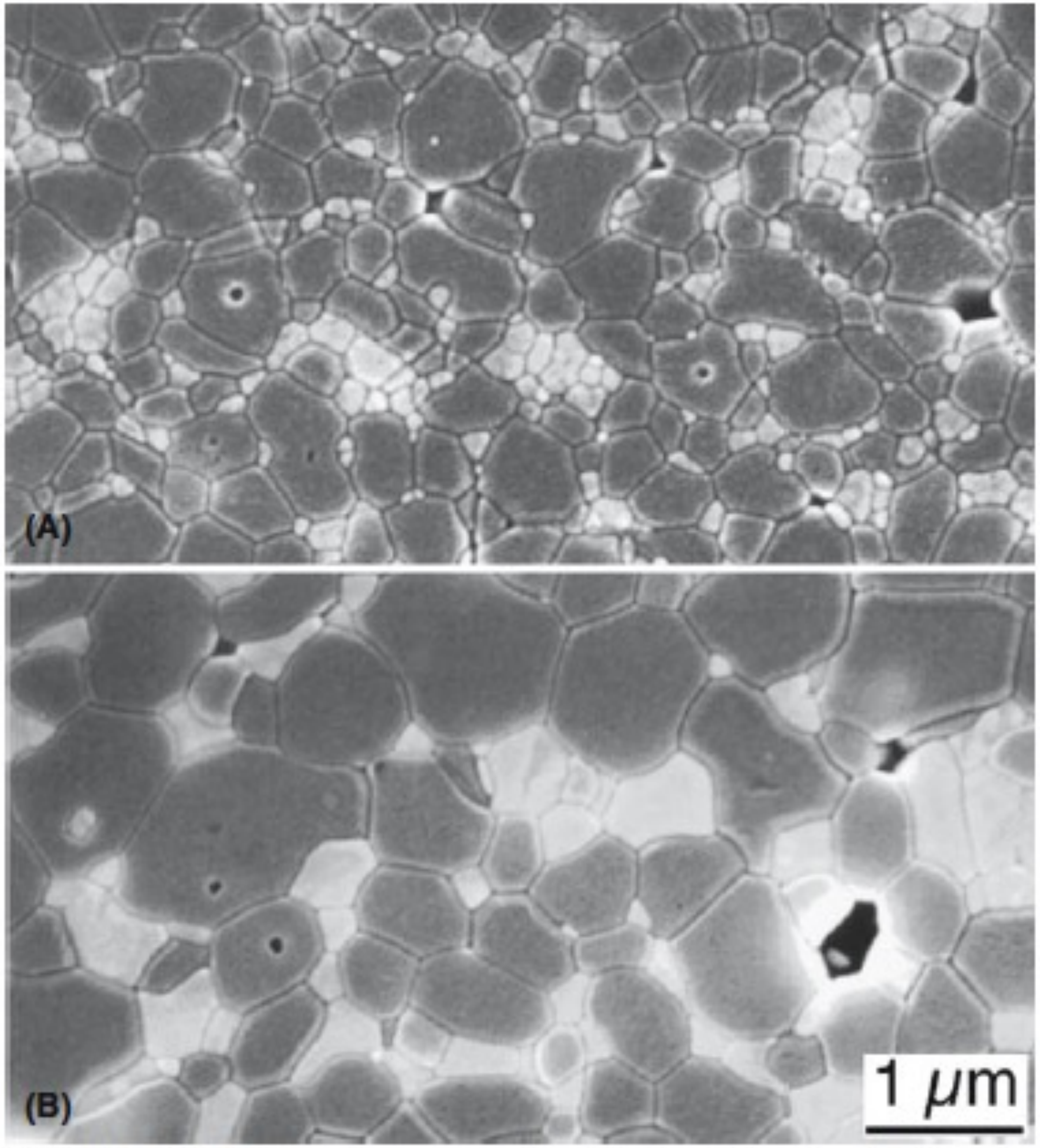
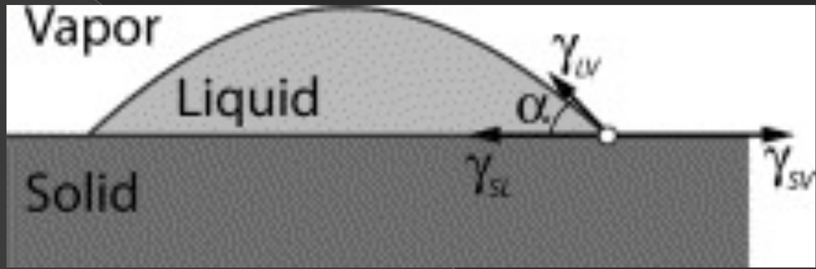


FIGURE 24.27 Two-phase ceramics. (a) As sintered and (b) heat treated at 1600°C for 30 hours. ZTA 30% (zirconia-toughened alumina with 30 vol% YSZ containing 10 molar% yttria).

WETTABILITY

Is the ability of a drop of liquid to recline on a solid surface.

Varying the pressur is changed the wettability.



α : contact angle

γ_{LV} : liquid-vapour interfacial energy

γ_{LS} : liquid-solid interfacial energy

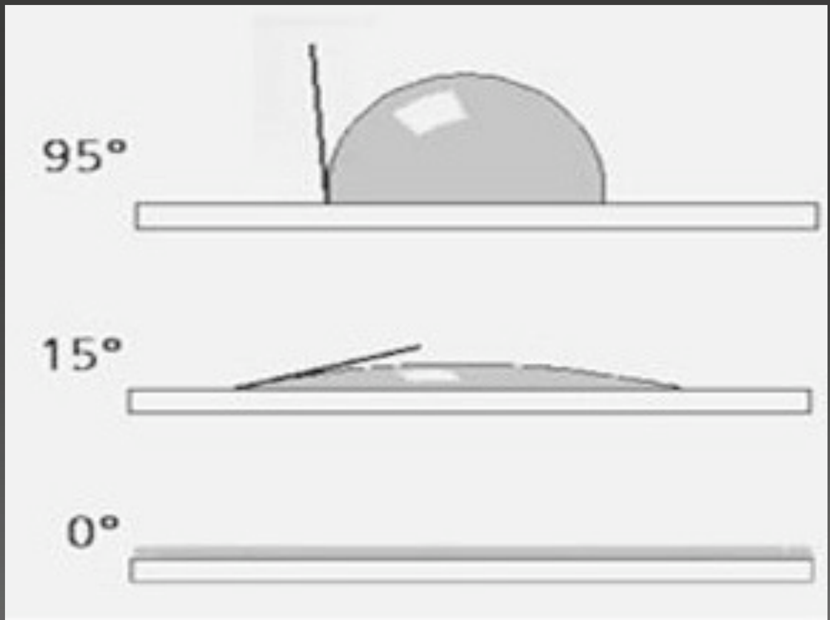
γ_{SV} : solid-vapour interfacial energy

The contact angle specifies the condition for minimum energy, according to the relation:

$$\gamma_{SL} + \gamma_{LV} \cos\alpha = \gamma_{SV}$$

$$\cos\alpha = \frac{\gamma_{SV} - \gamma_{SL}}{\gamma_{LV}}$$

possible cases:



$\alpha > 90°$ ⟶ non-wettability

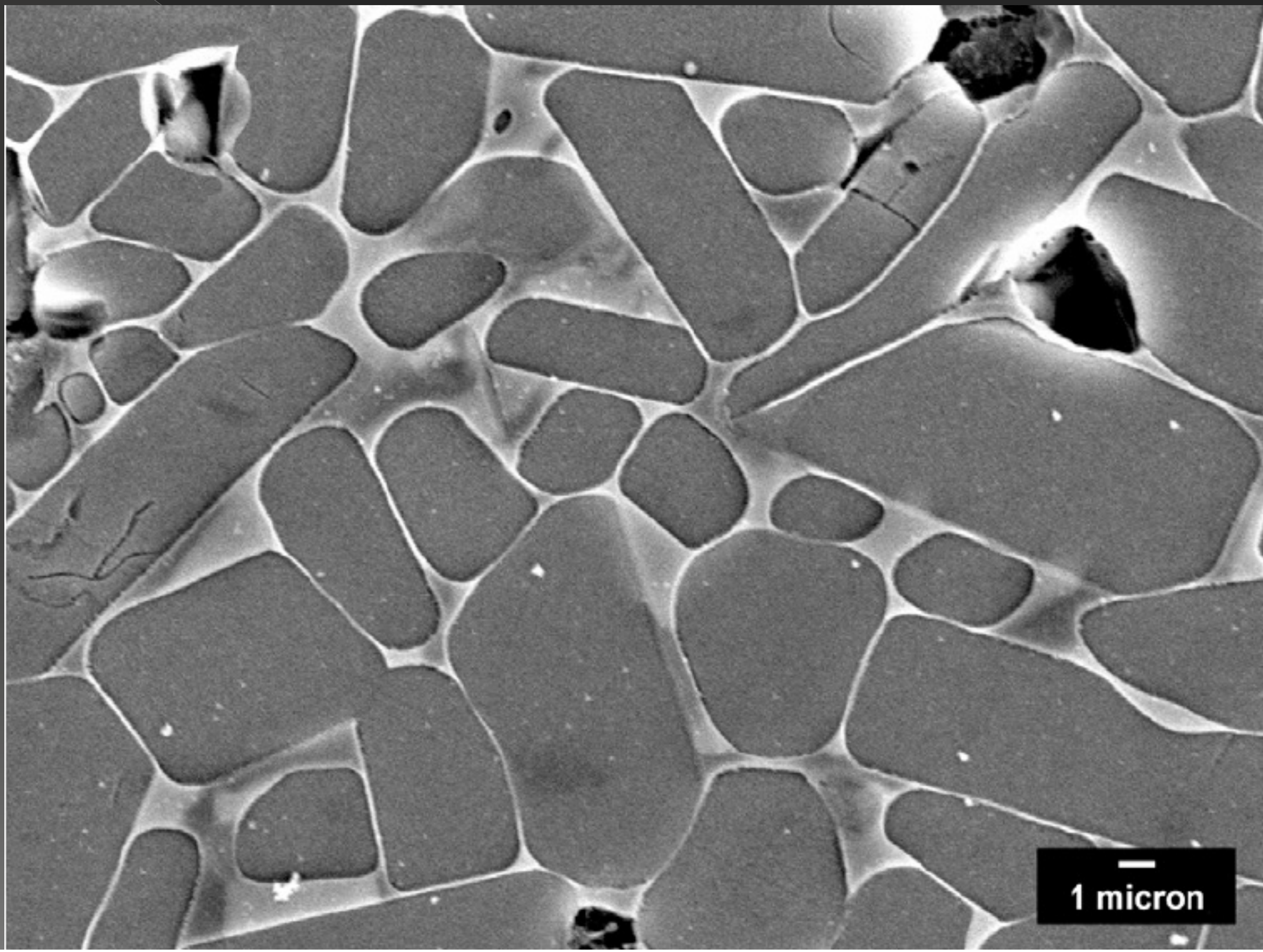
$\alpha < 90°$ ⟶ wettability

$\alpha = 0$ ⟶ spreading

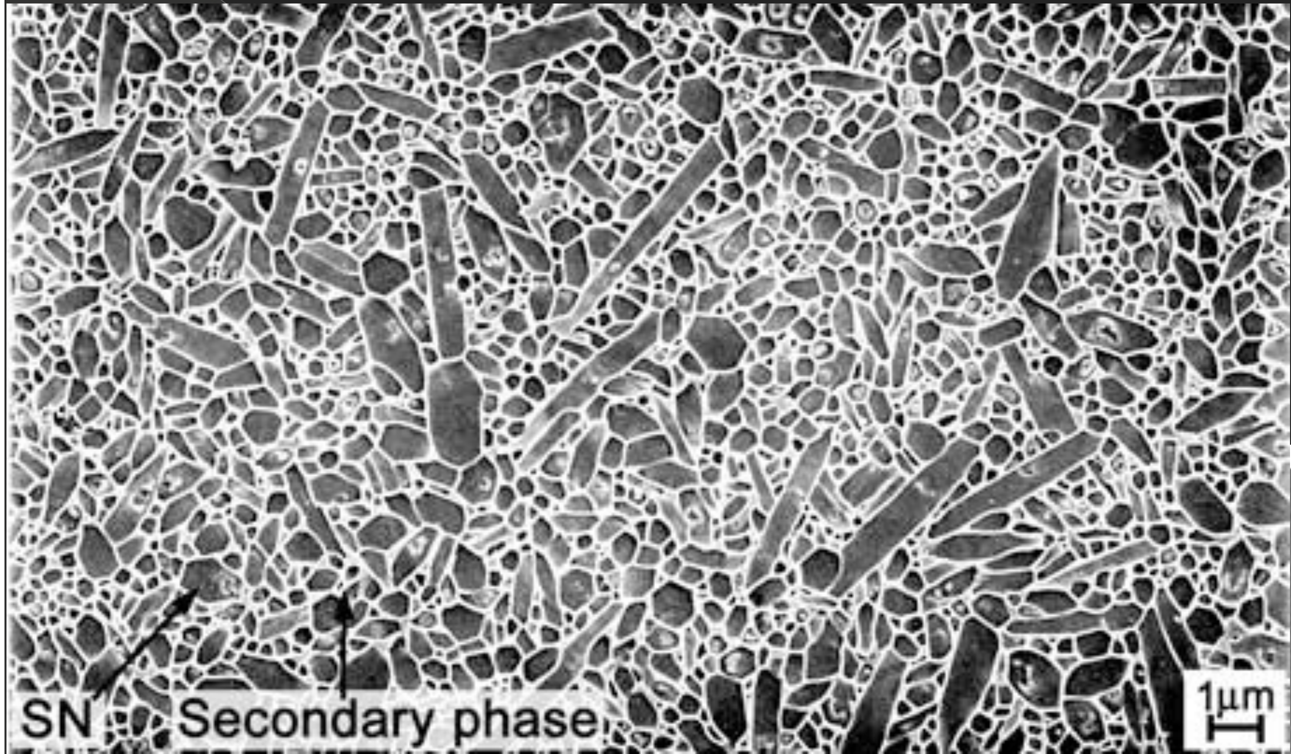
LIQUID PHASE SINTERING

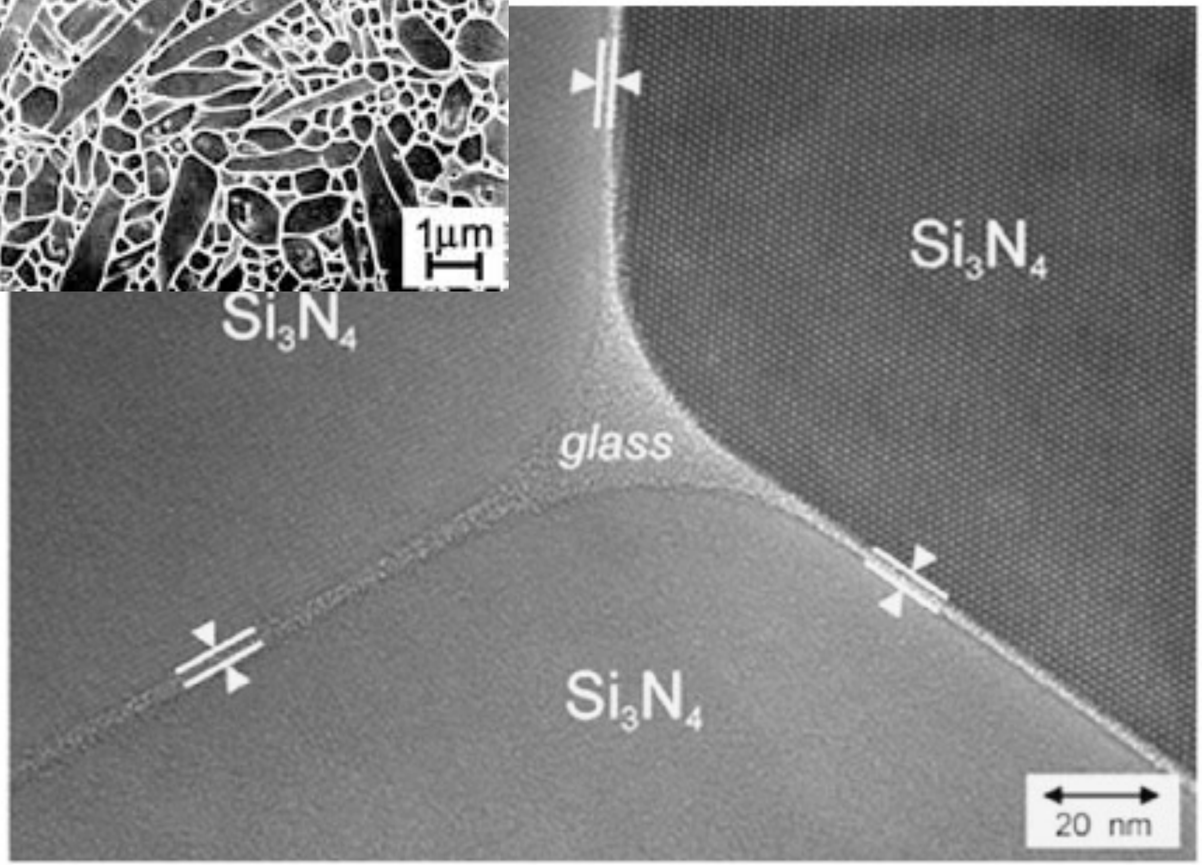
- It is the process of adding an additive to the powder which will melt before the ceramic grains.
- The metal added, at high temperatures, melt and WET the grains. The intergranulary spaces are such as to have a capillary forces which attract the grain one another.
- (By lowering the temperature, the amorphous phase does not wet the grains anymore and ritires in triple junctions.)
- (This gives good mechanical proprieties.)
- E.G. : WIDIA (93% WC in a Co matrix).

Liquid phase sintered SiC

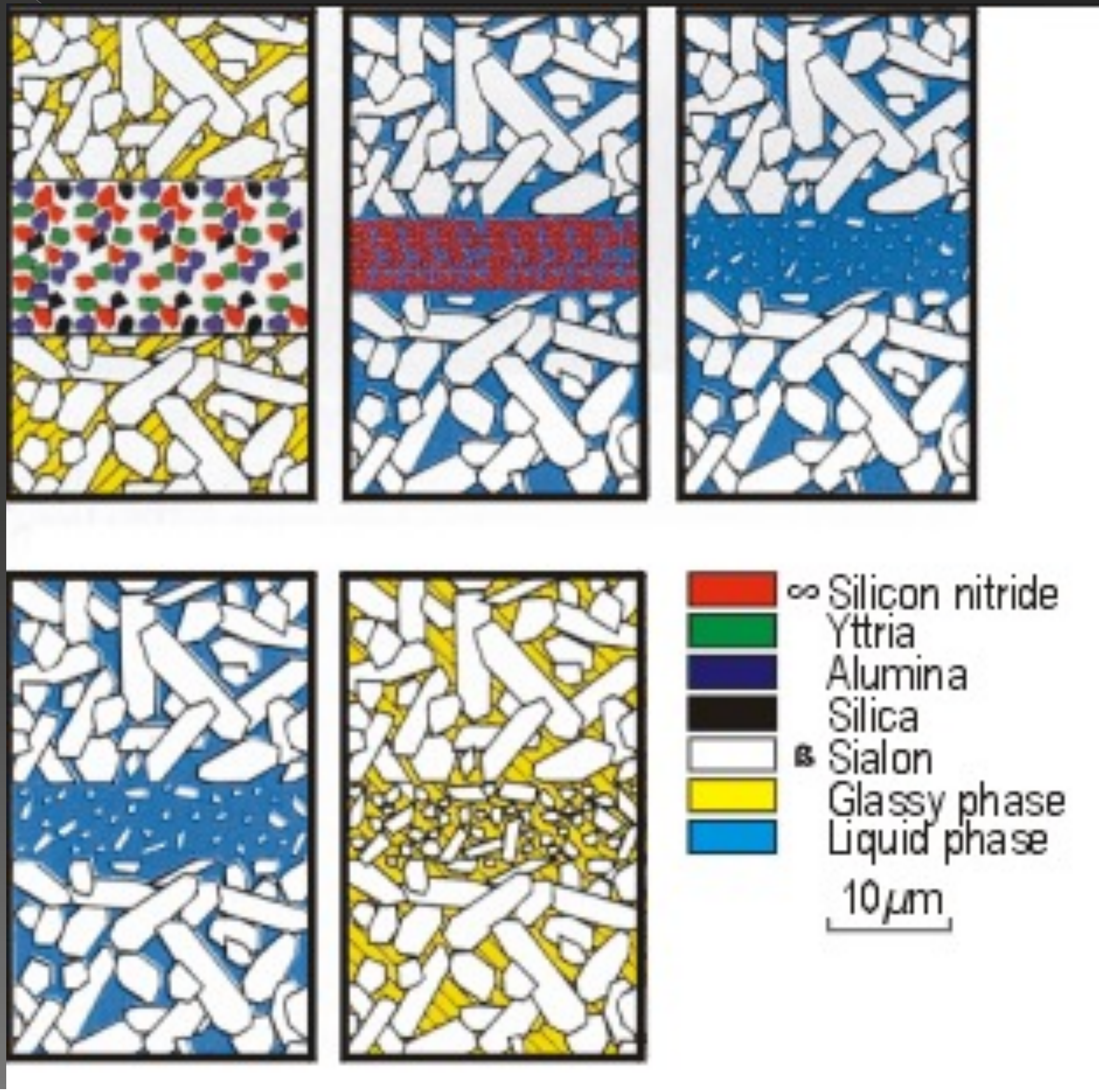


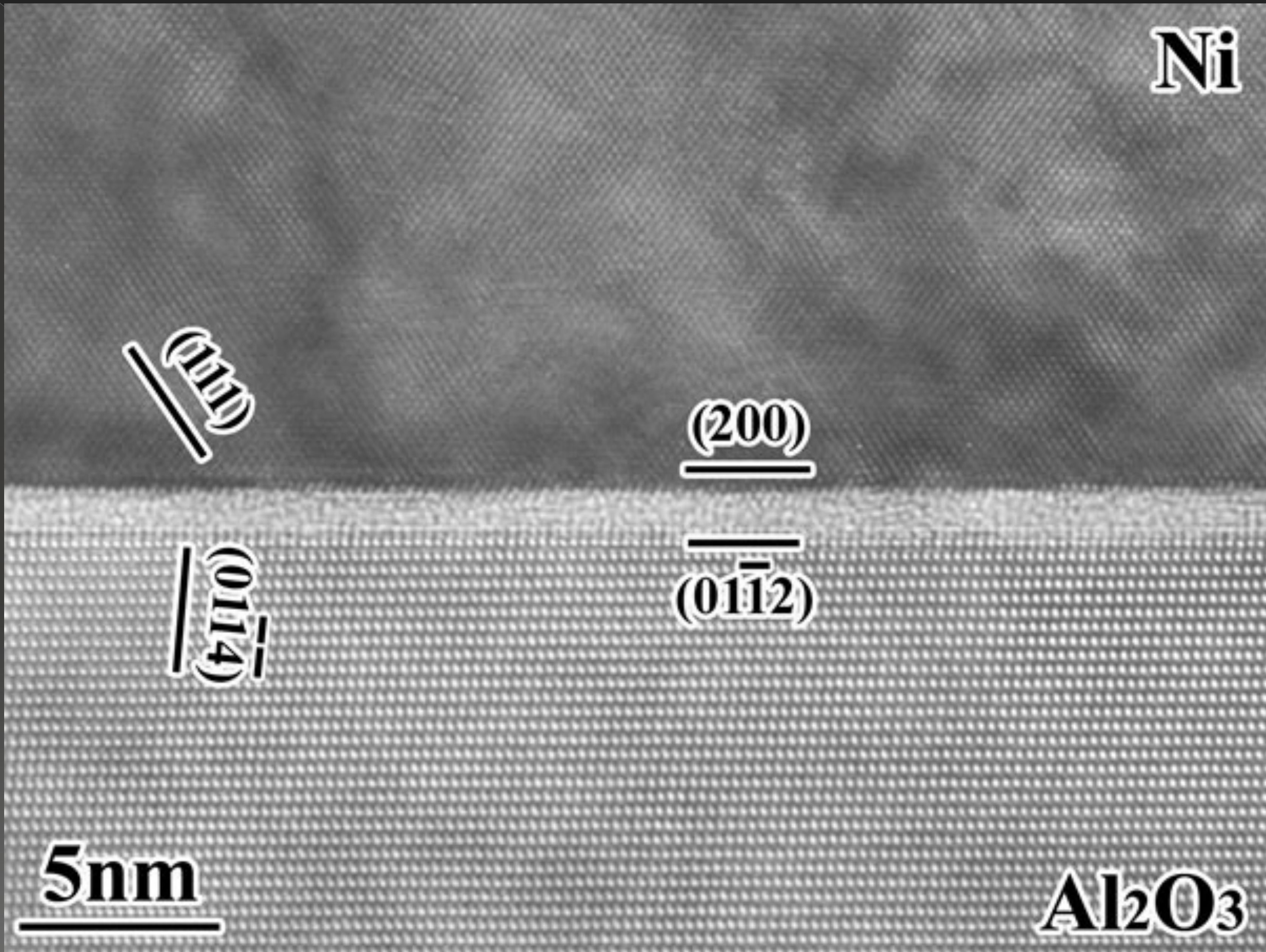
Liquid phase sintered Si_3N_4



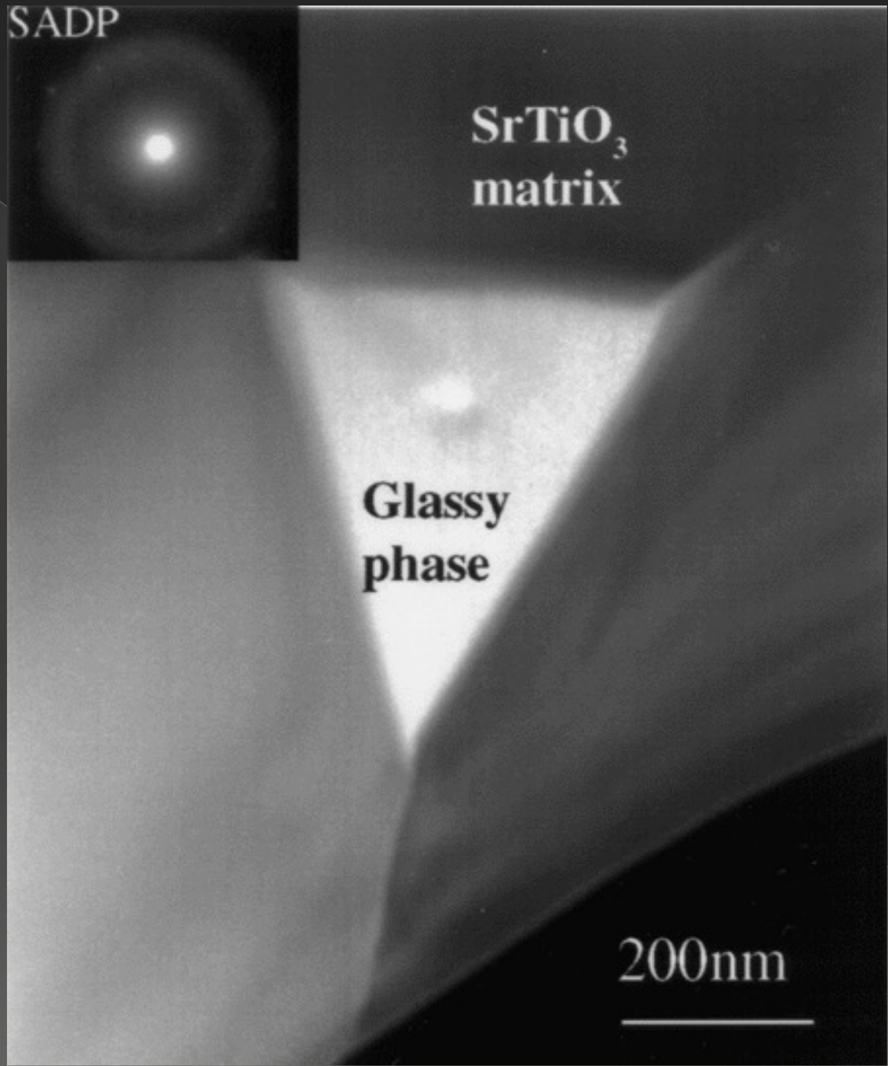


Lquid phase sintered SiAlON

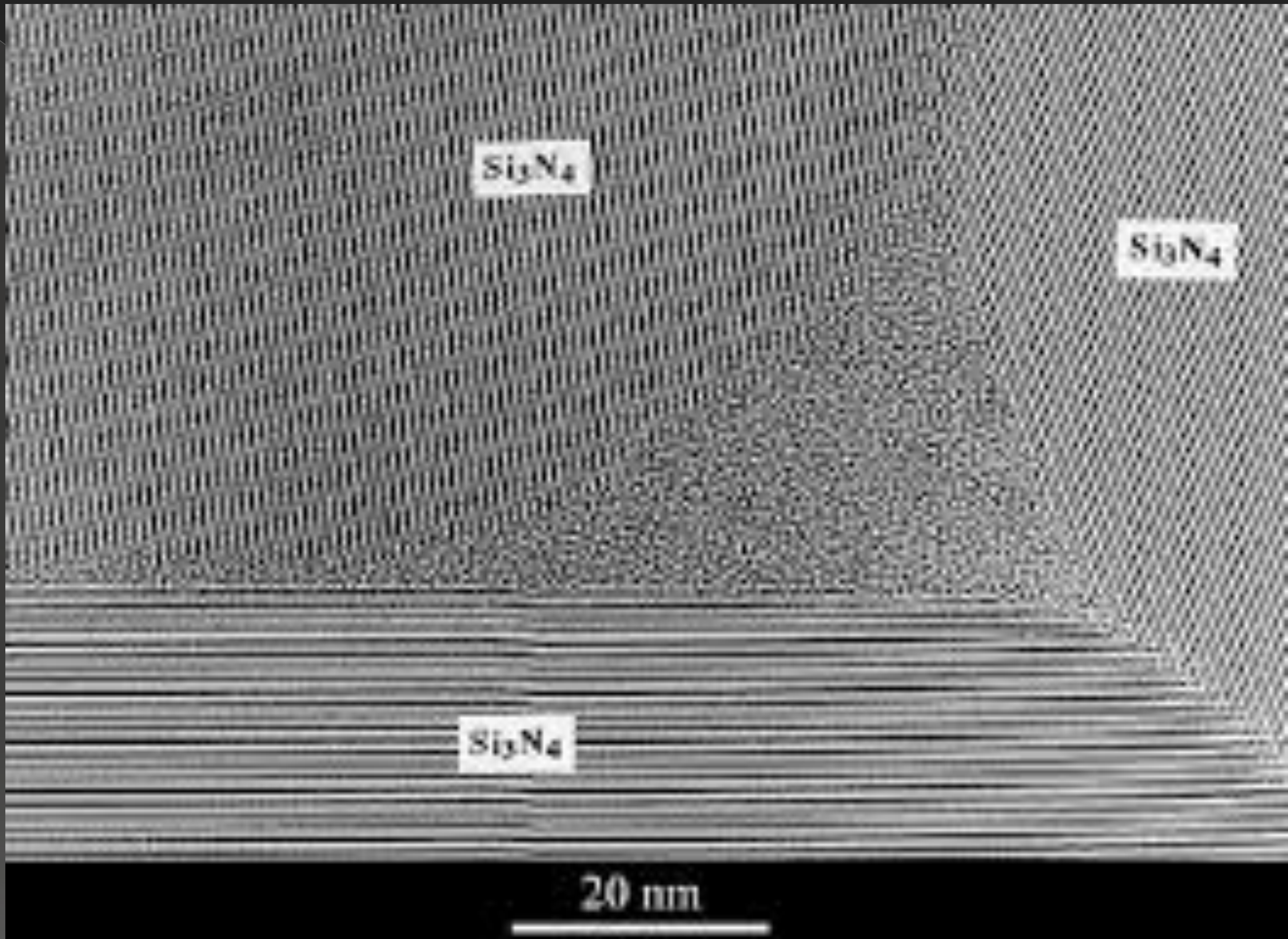




Ni
(200)
(01$\bar{1}$2)
5nm
Al_2O_3



SADP
SrTiO3
matrix
Glassy
phase
200nm


Si3N4
Si3N4
Si3N4
20 nm

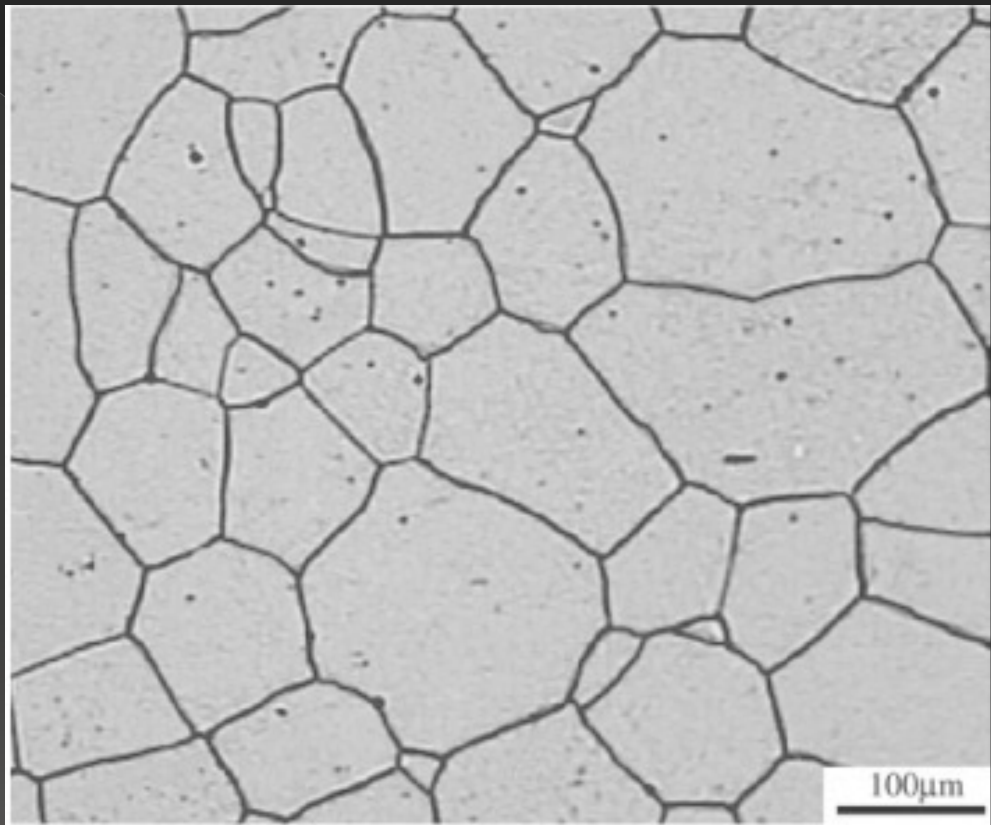
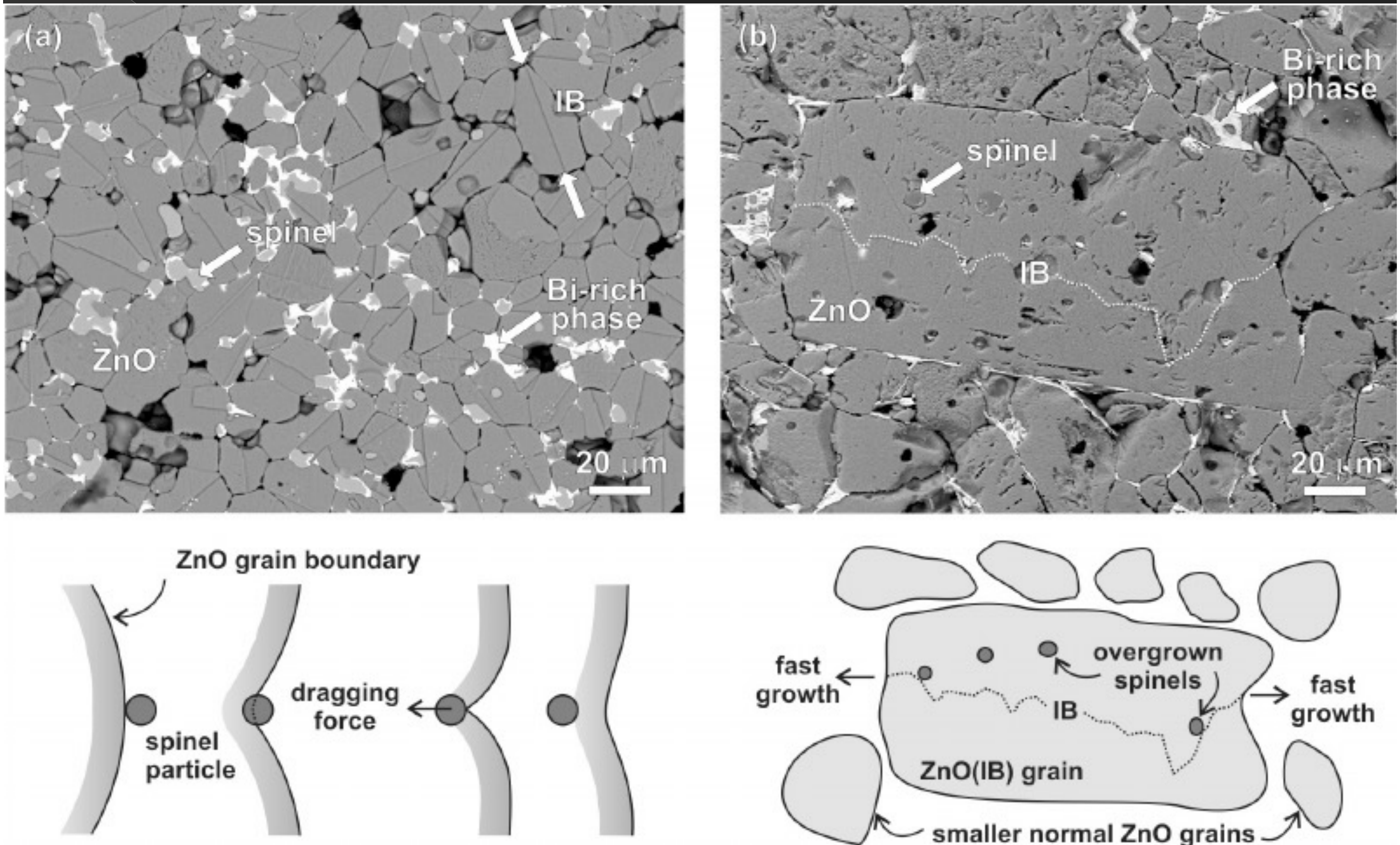


Figure 1. Typical microstructure of polycrystalline ZnO used in this work, after thermal etching at 1150 °C, for 1 h, in air.


(a)
IB
spinel
Bi-rich phase
ZnO
20 μm
(b)
Bi-rich phase
spinel
IB
ZnO
20 μm
ZnO grain boundary
spinel particle
dragging force
overgrown spinels
fast growth
IB
fast growth
ZnO(IB) grain
smaller normal ZnO grains

PROCEDURE FOR THE SINTERING PROCESS

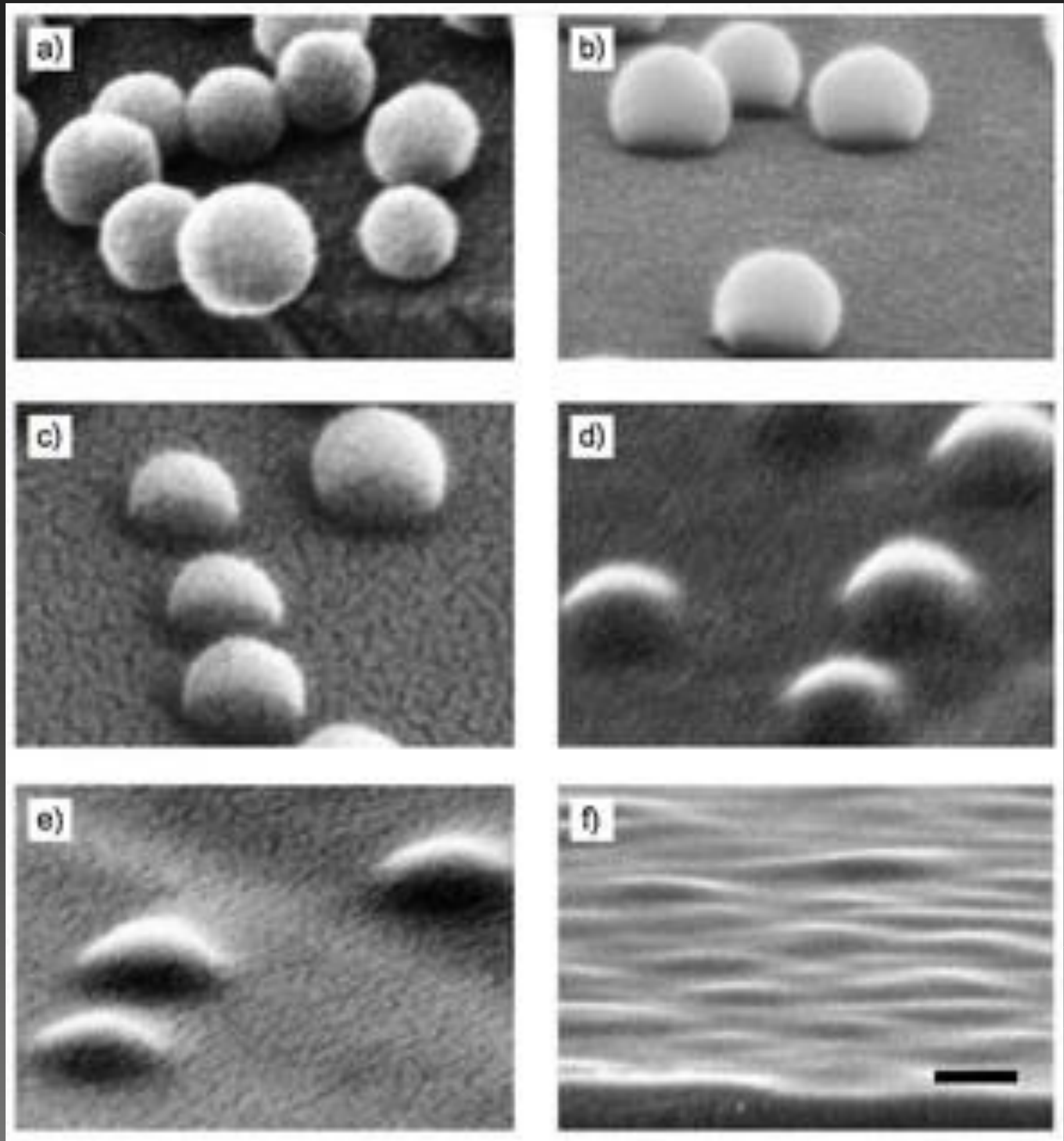
- Determination of the T_m $T_{sintering} = 2/3\, T_m$ E.G. : Al_2O_3
  $T_m = 2400°C$ $T_{sintering} = 1600°C$
- CALCINATION (200°C-300°C under the sintering temperature)
  E.G. : ZrO_2 stabilized by CaO, Y_2O_3, CeO_2
- FORMING the ceramic parts
- SINTERING

$$\frac{\sqrt{D \cdot t}}{a} \approx 1$$

DENSITY DETERMINATION
BY ARCHIMEDE'S PRINCIPLE

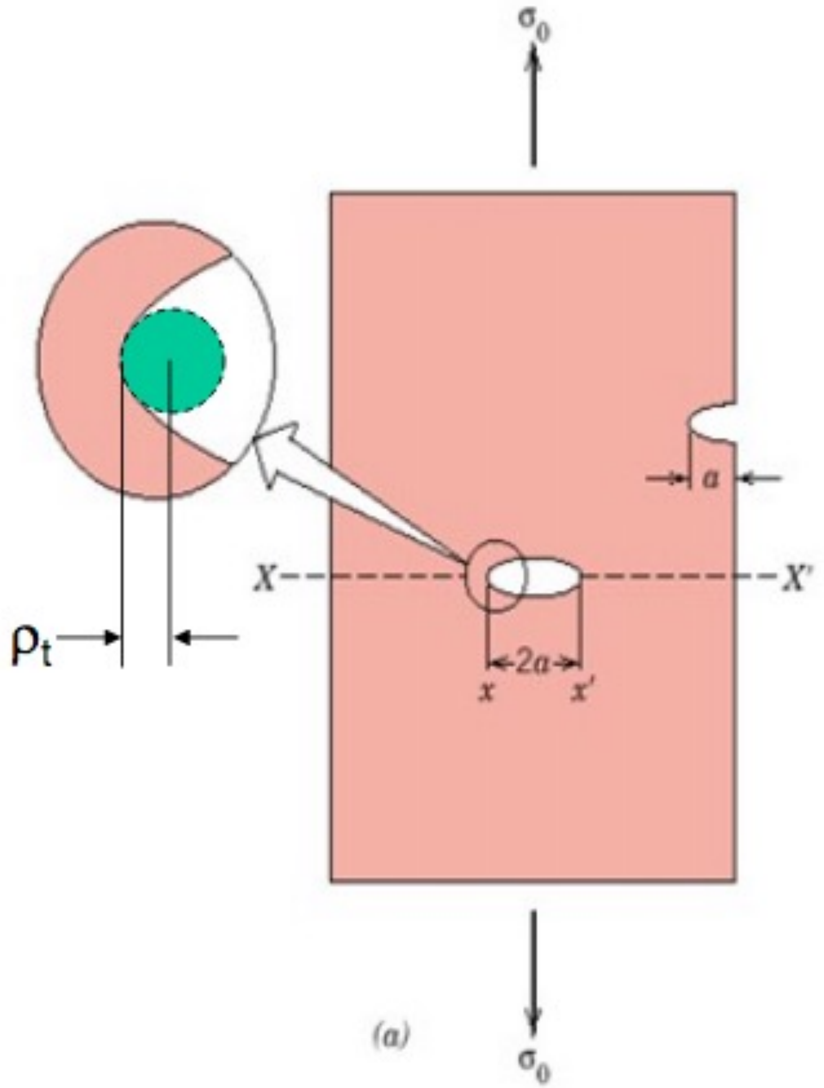
- D = dry weight
- boil the piece for 5 hours
- W = wet weight in air
- S = wet weight in water suspended
- V = external volume of the piece: $V = W - S$
- BULK DENSITY $B = D/V$
- P = apparent porosity $P = (W-D)/V$

TYPICAL SINTERING TIME-TEMPERATURE PROFILE



a)
b)
c)
d)
e)
f)

Flaws are Stress Concentrators



If the crack is similar to an elliptical hole through plate, and is oriented perpendicular to applied stress, the maximum stress σ_m=

$$\sigma_m = 2\sigma_o\left(\frac{a}{\rho_t}\right)^{1/2} = K_t\sigma_o$$

where

ρ_t = radius of curvature

σ_o = applied stress

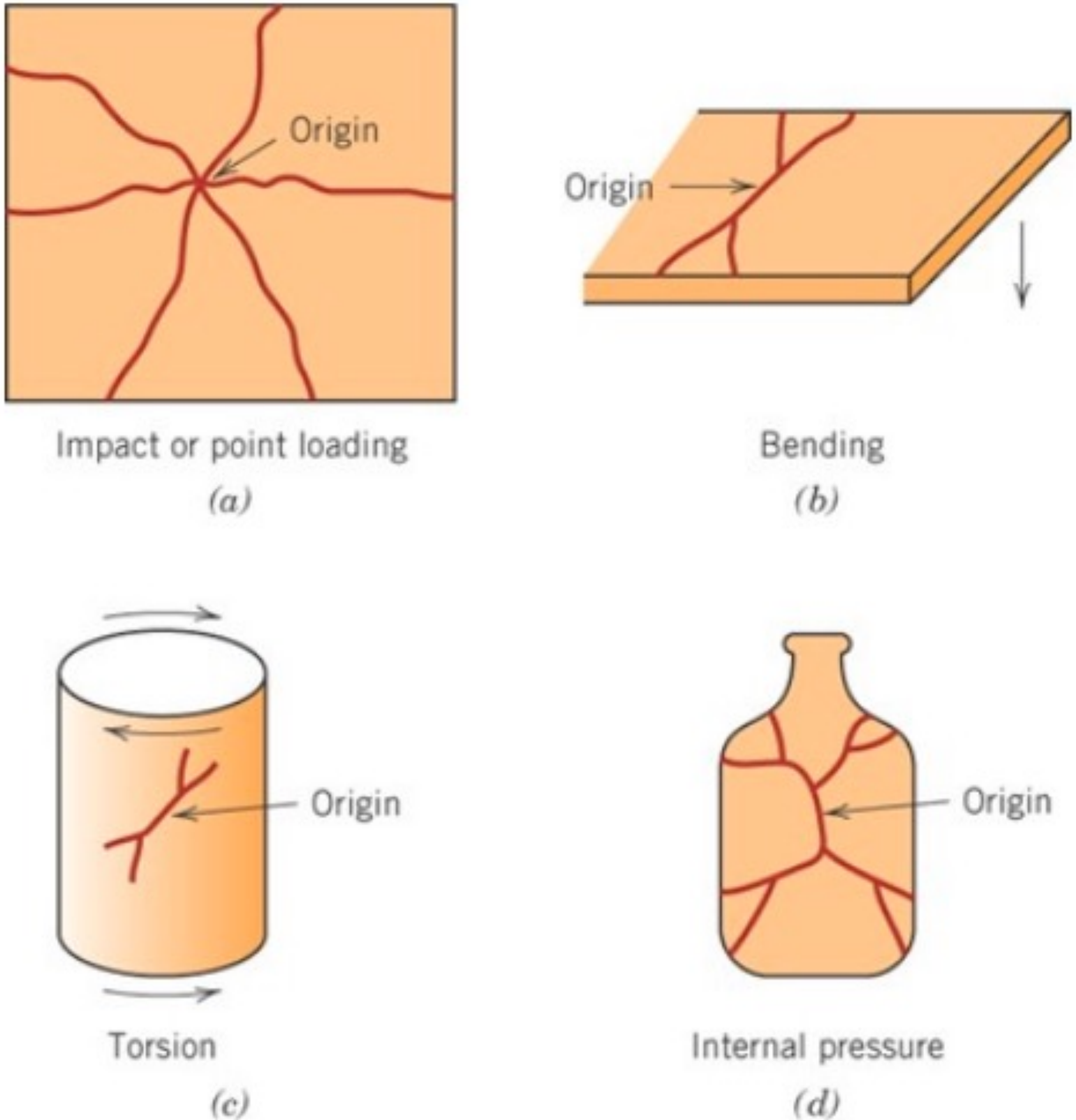
σ_m = stress at crack tip

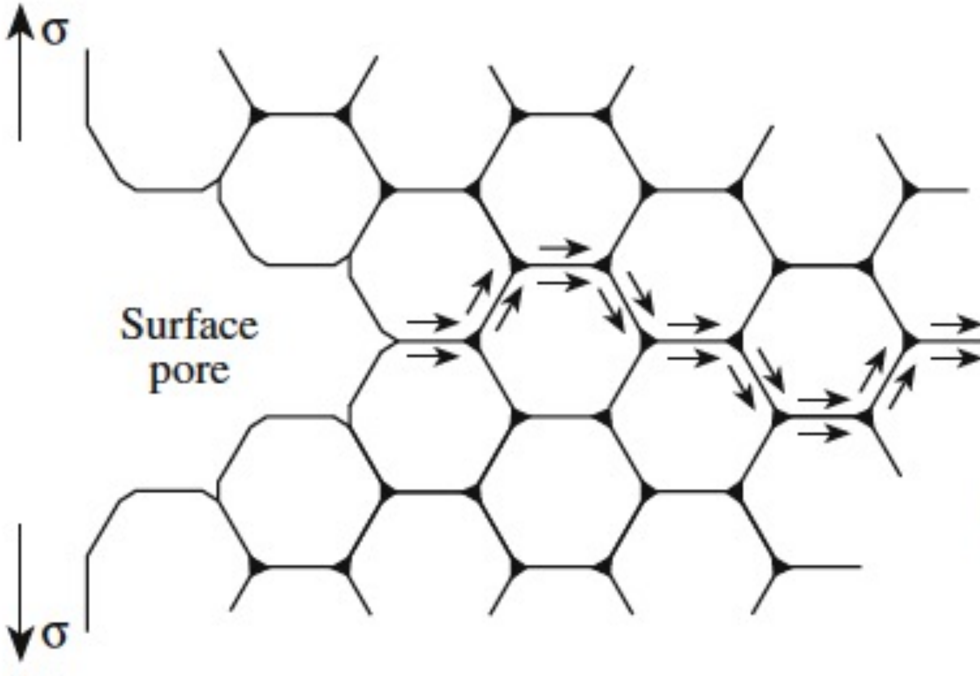
a = length of surface crack or ½ length of internal crack

$\sigma_m/\sigma_o = K_t$ the stress concentration factor

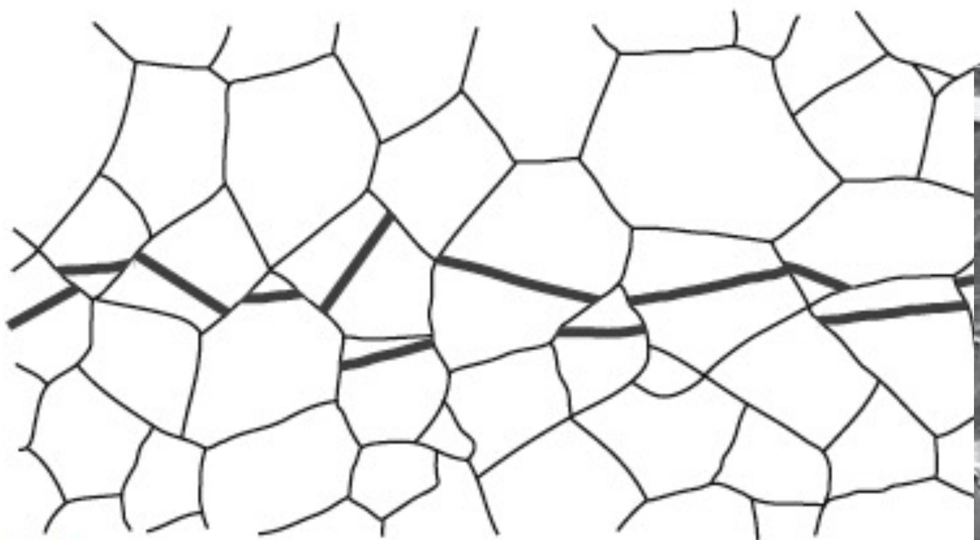
Brittle Fracture of Ceramics

- Most ceramics (at room temperature) fracture before any plastic deformation can occur.
- Typical crack configurations for 4 common loading methods.

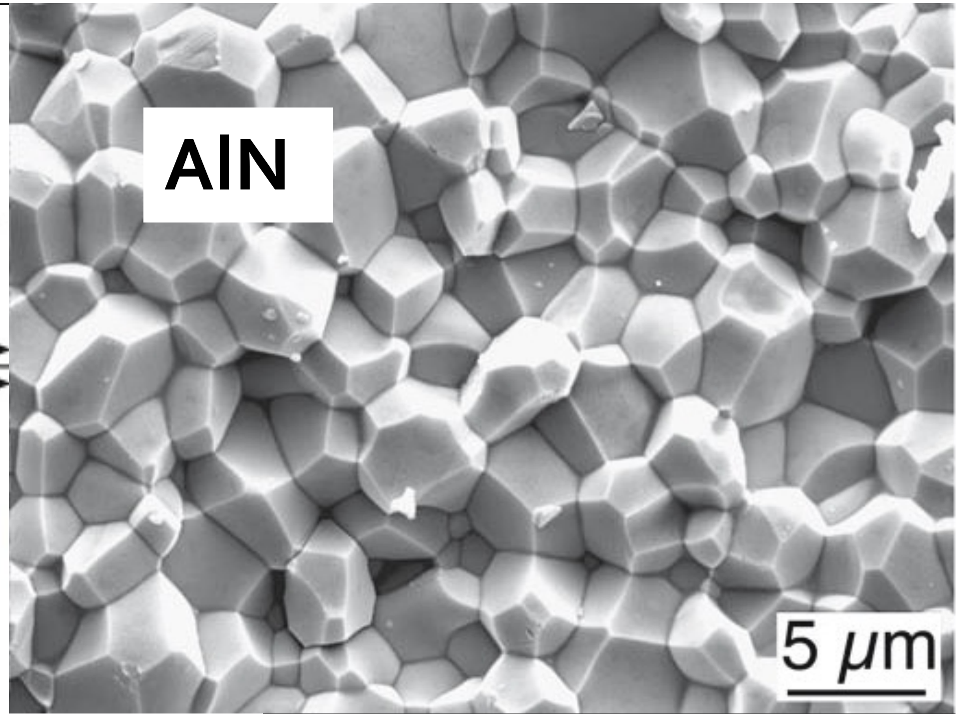




(A)



(B)



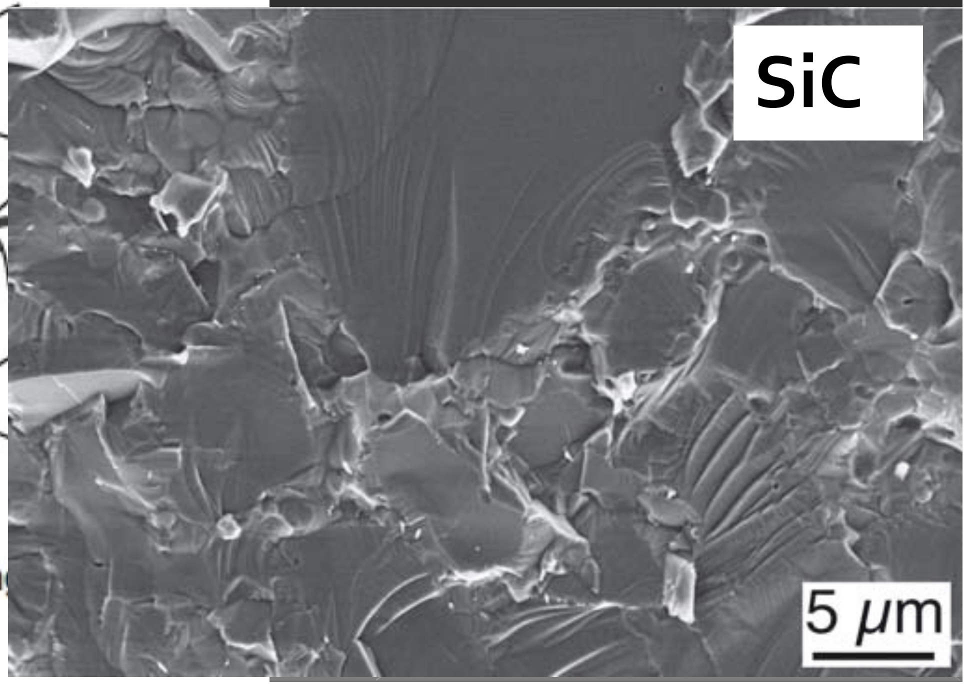
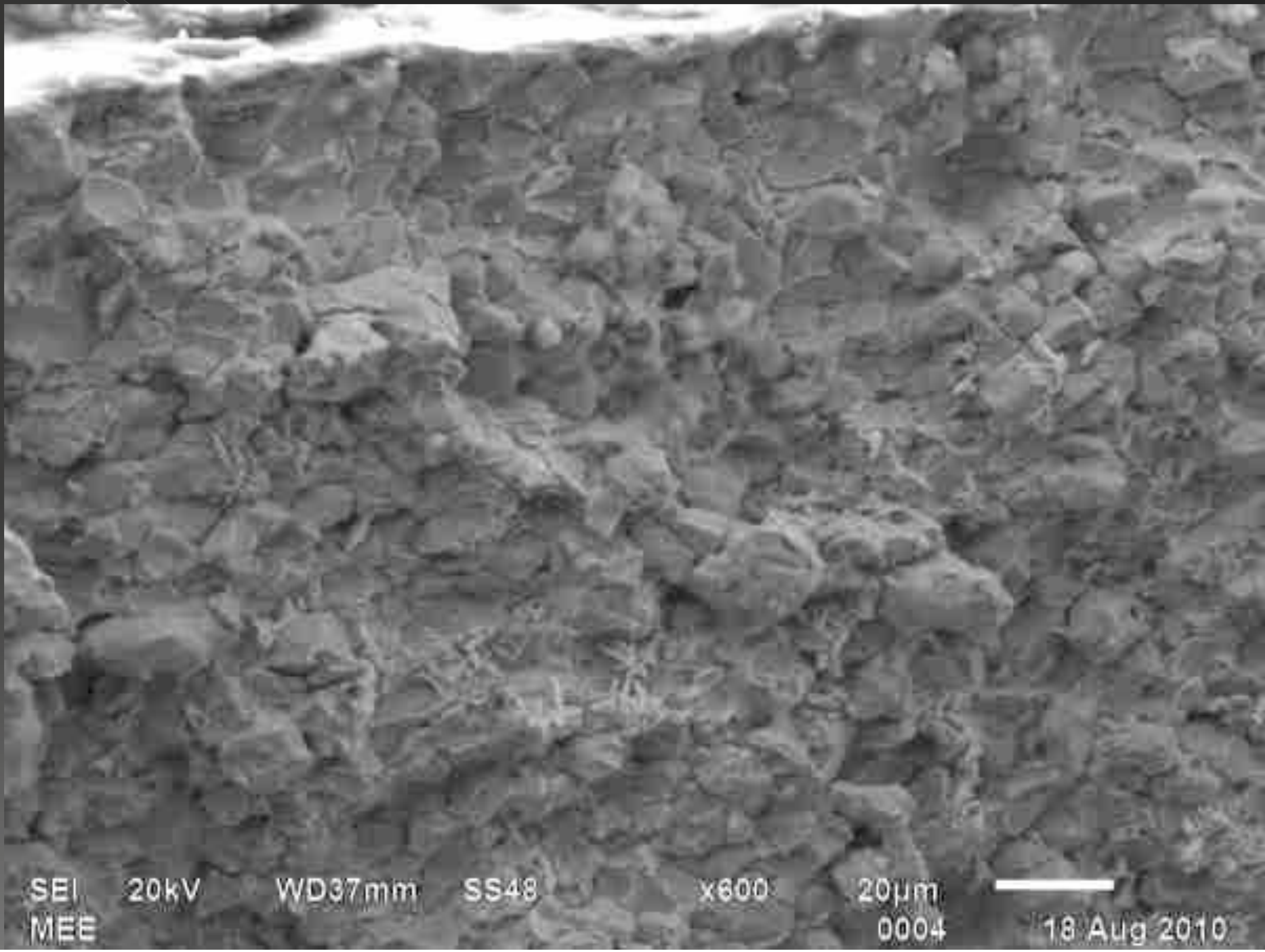


FIGURE 18.12 (a) Illustration of intergranular crackin
Illustration of transgranular cracking.

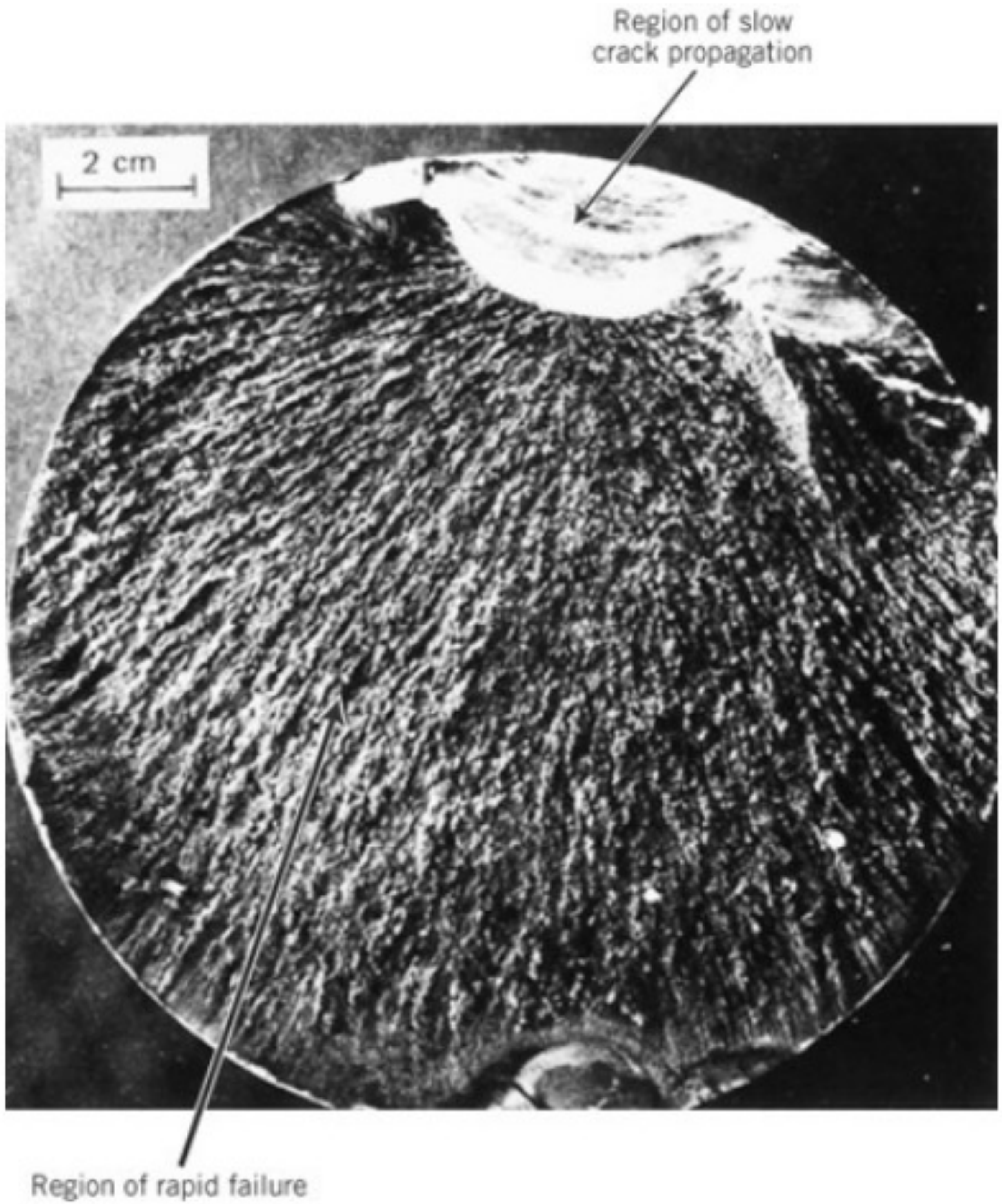
Undistinct features: brittle fracture (SiC)



Fatigue

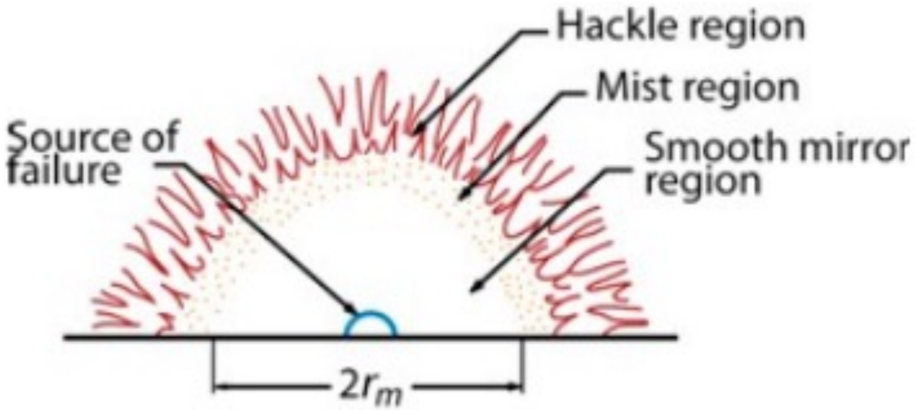
- Fracture surface with crack initiation at top. Surface shows predominantly dull fibrous texture where rapid failure occurred after crack achieved critical size.

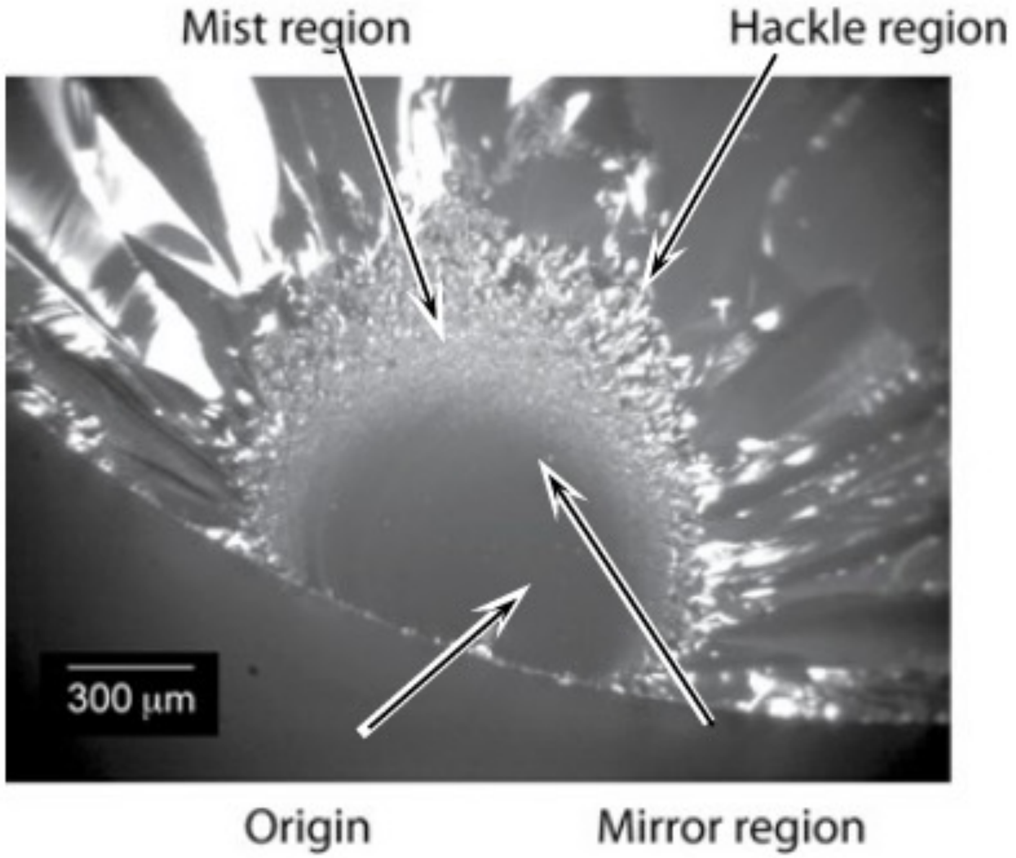
- Fatigue failure
  1. Crack initiation
  2. Crack propagation
  3. Final failure

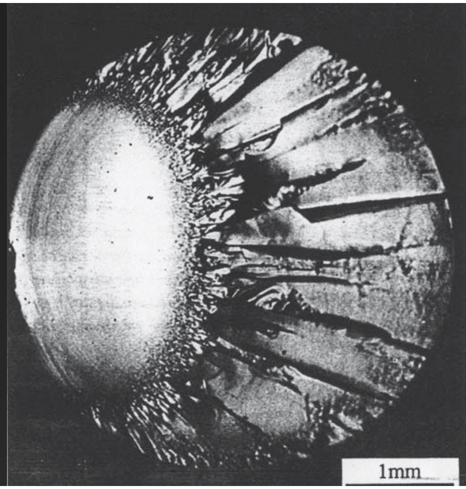


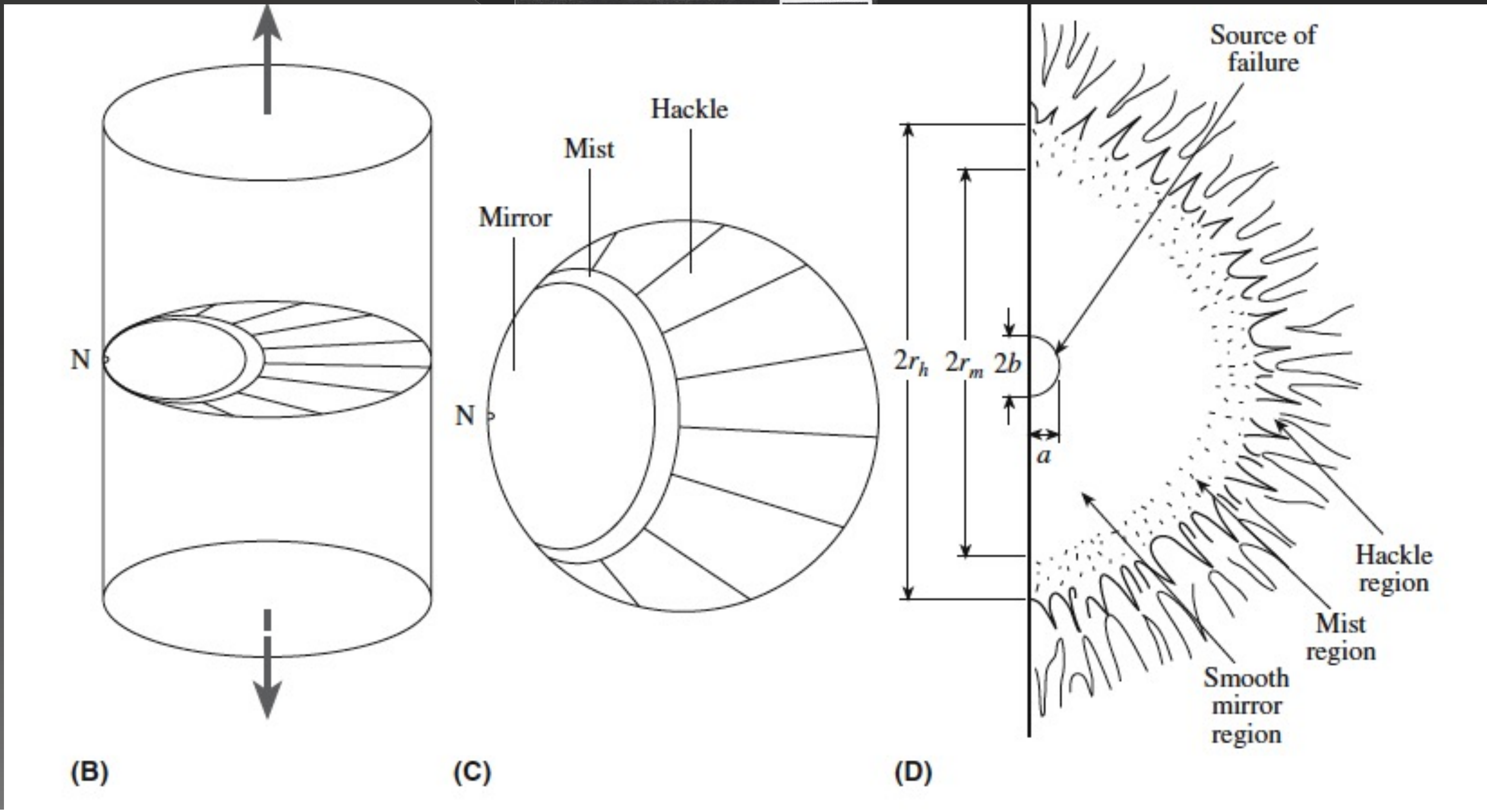
Brittle Fracture of Ceramics

- Surface of a 6-mm diameter fused silica rod.
- Characteristic fracture behavior in ceramics
  - Origin point
  - Initial region (mirror) is flat and smooth
  - After reaches critical velocity crack branches
    - mist
    - hackle

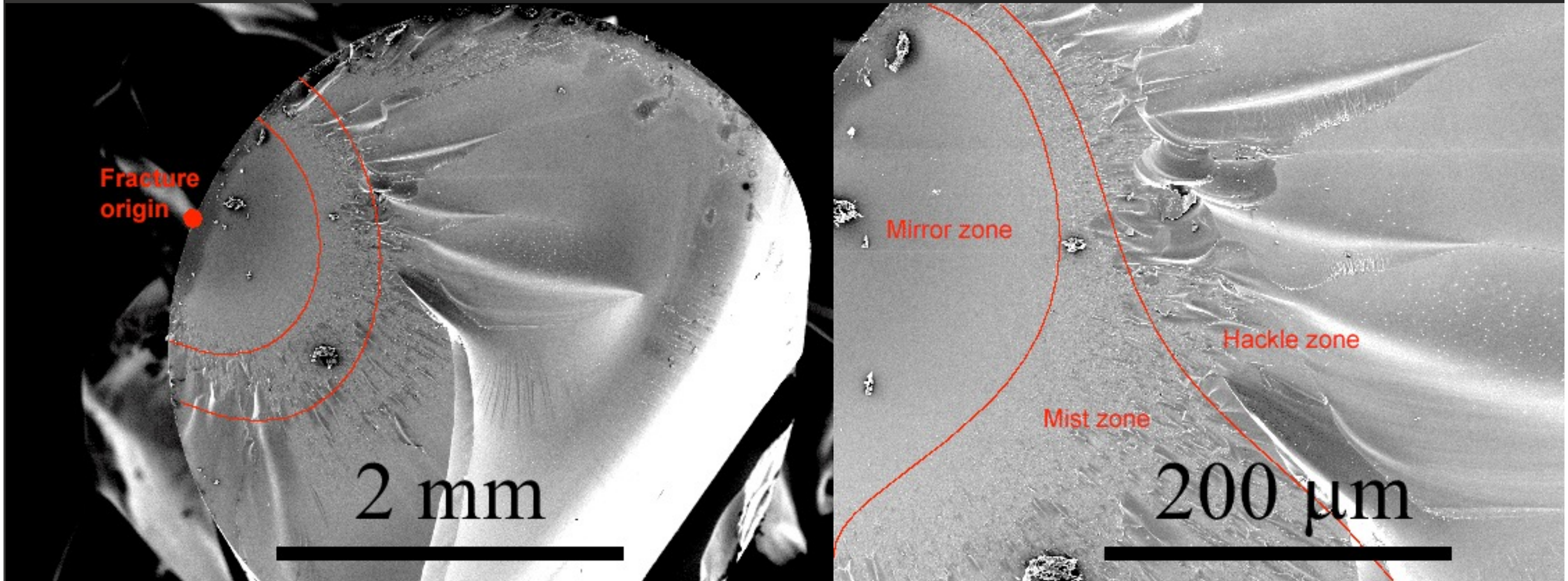




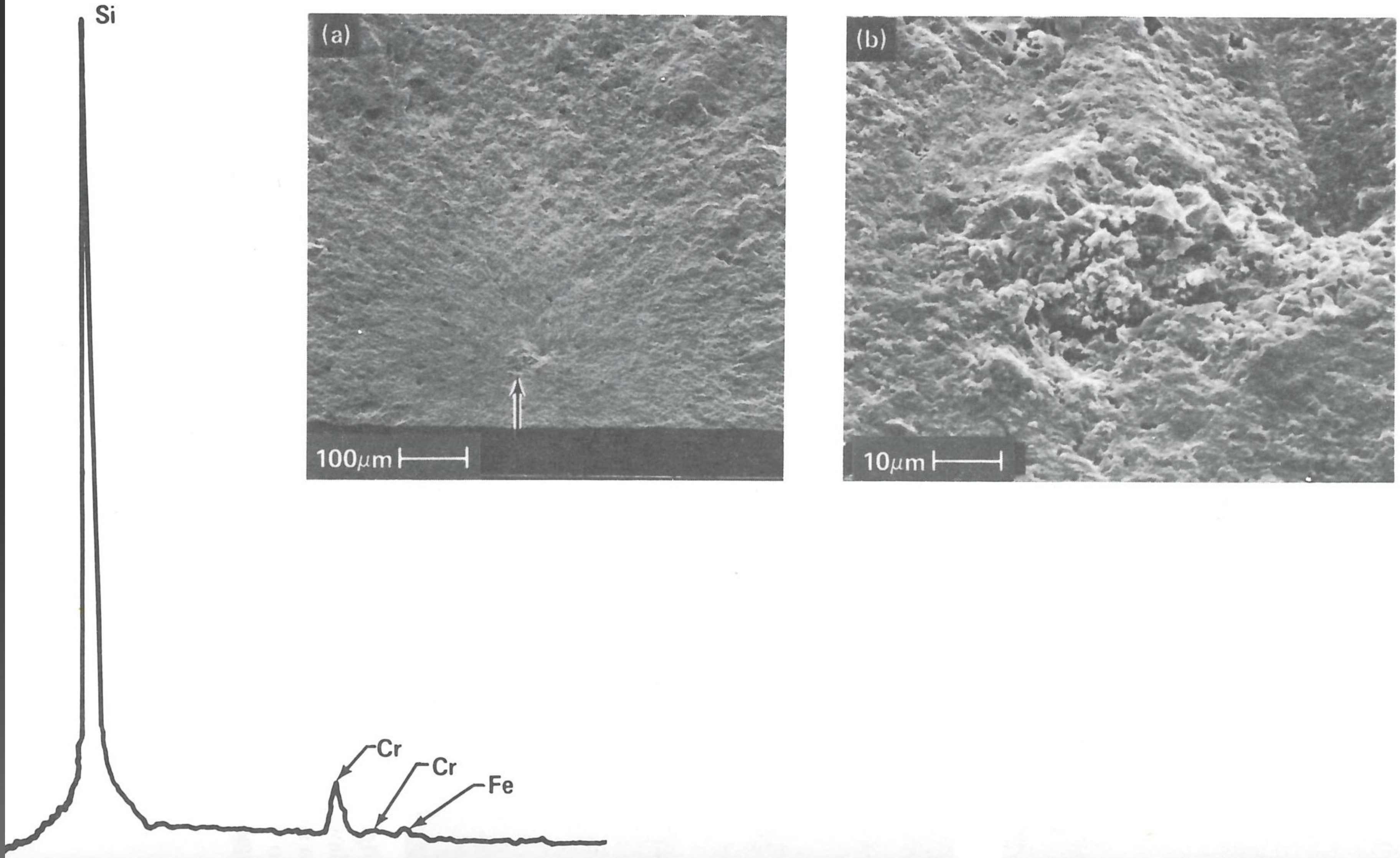

1mm


N
Hackle
Mist
Mirror
N
Source of failure
$2r_h$
$2r_m$
$2b$
a
Hackle region
Mist region
Smooth mirror region
(B)
(C)
(D)

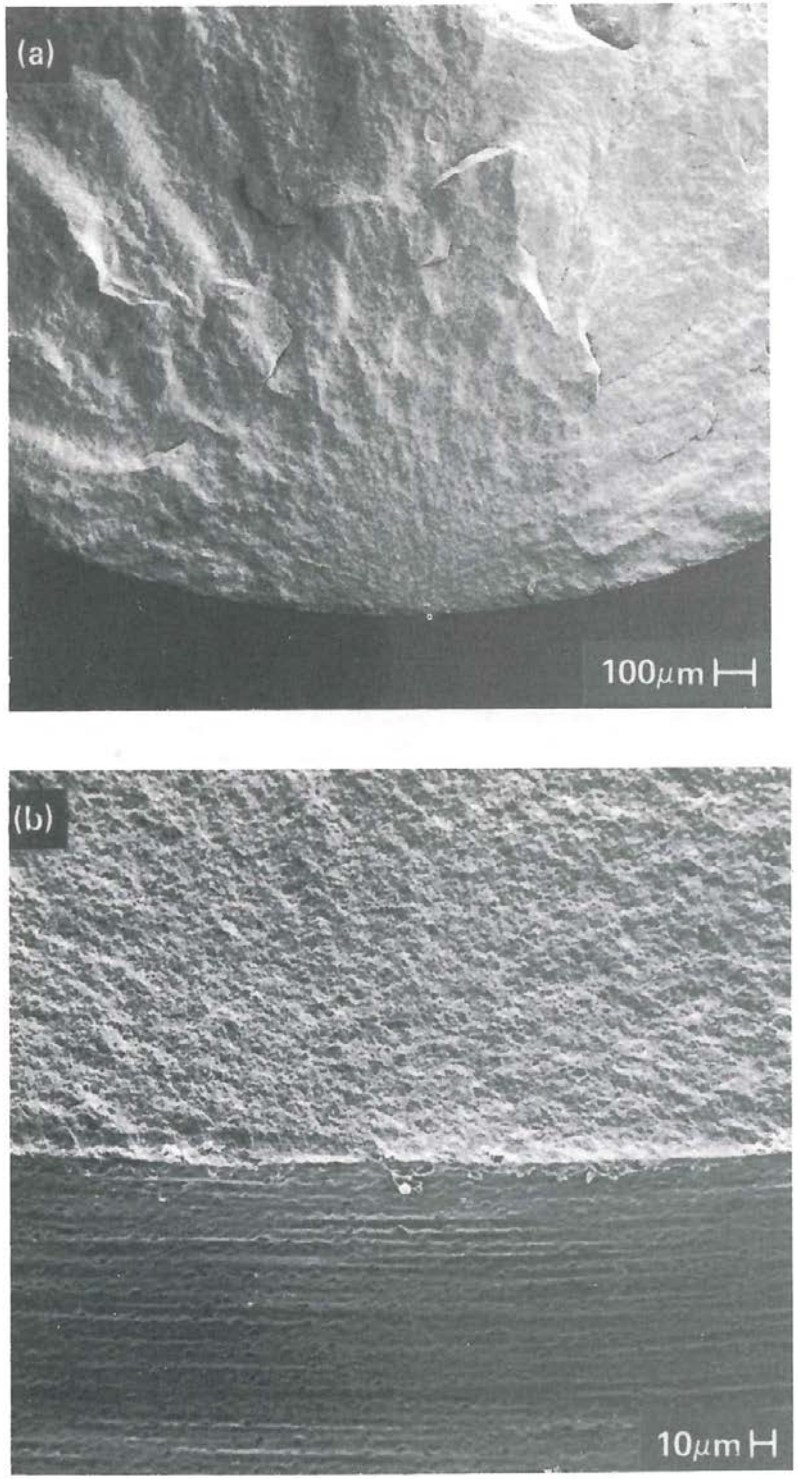
Fracture of glass



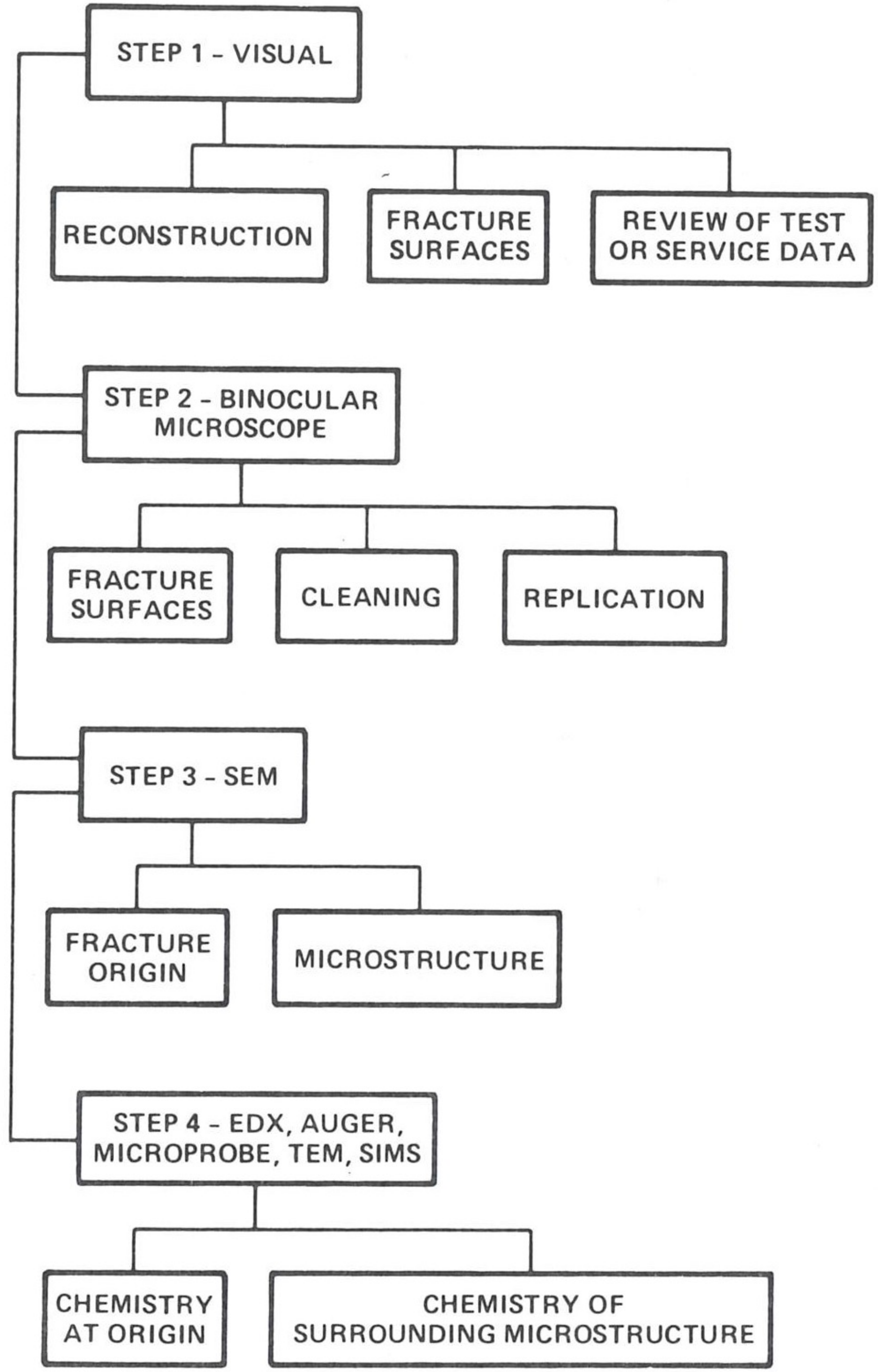
Fracture surface of silicon nitride with steel impurity



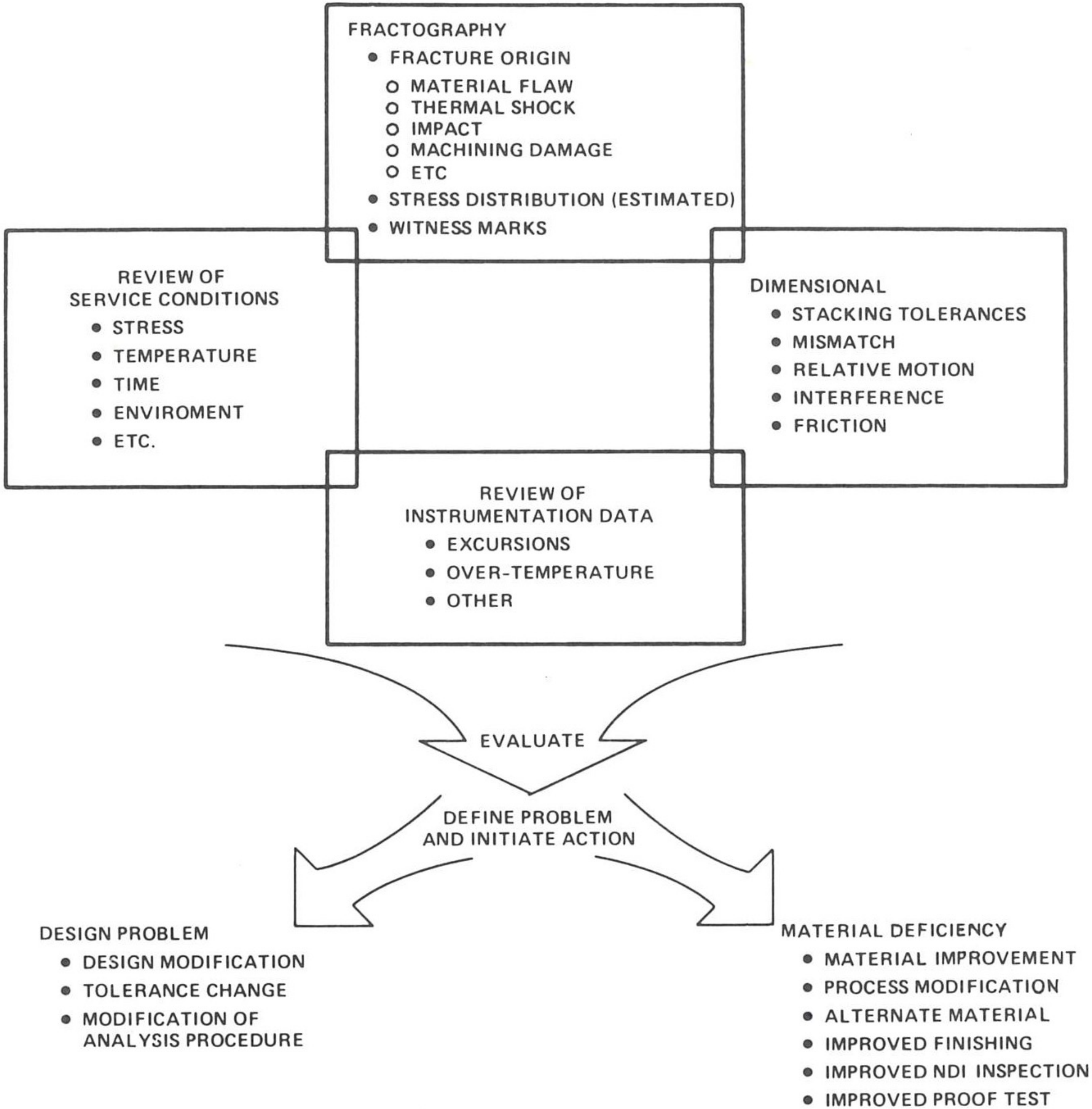
Fracture surface of lathe machined Silicon nitride



Roadmap for fractography



Roadmap for correcting failure



Toughening by whiskers and fibers

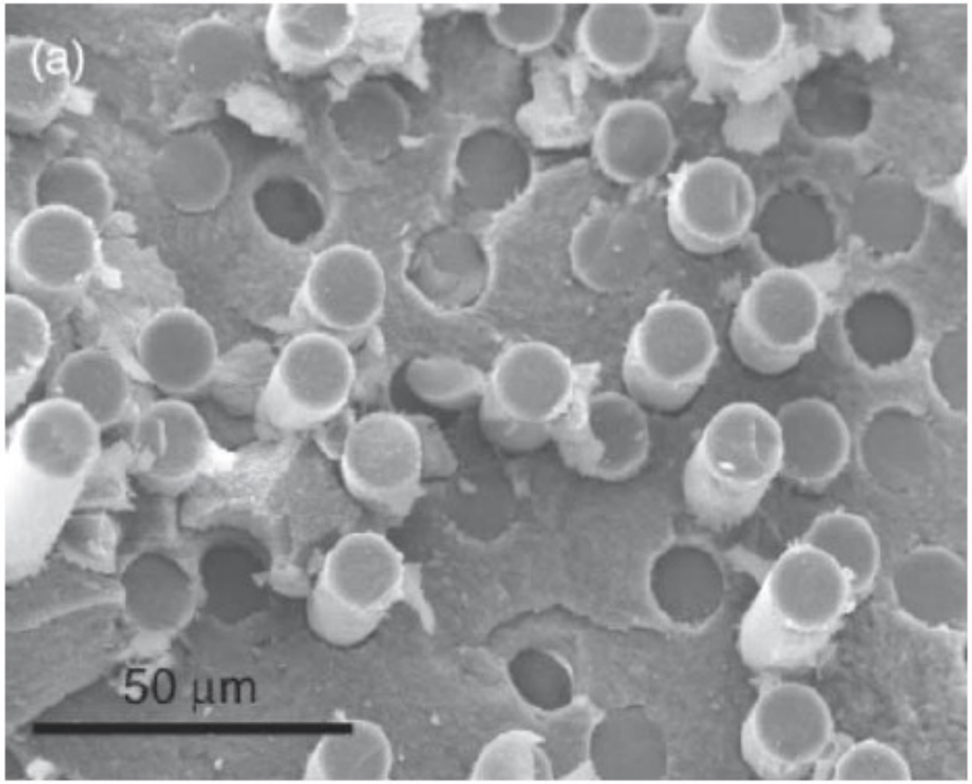
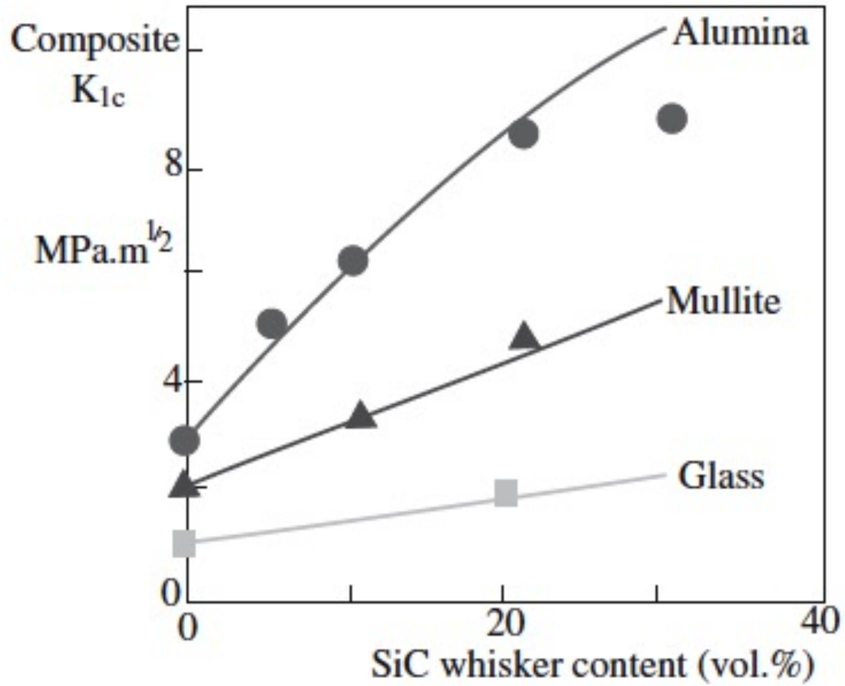
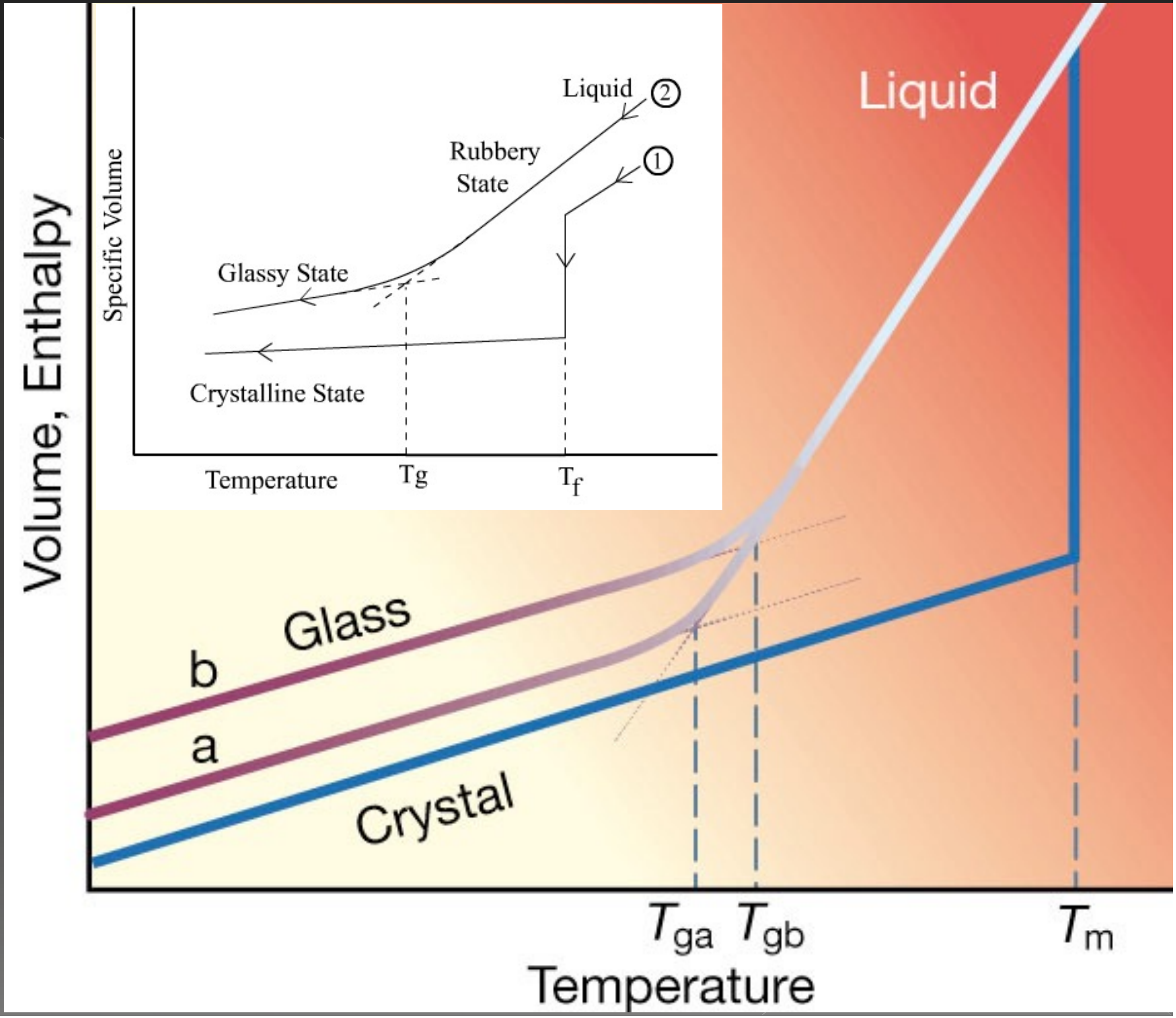


FIGURE 18.18 SEM image showing fiber pullout on the fracture surface of $AlPO_4$-coated alumina/mullite fiber/Al_2O_3 CMC, hot pressed at 1250°C for 1h.



Glass theory

- Glasses lack the periodic (long range) order of a crystal
- •
- Infinite unit cell (no repeating large scale structures)
- •
- 3D network lacking symmetry and periodicity
- •
- ISOTROPIC: same average packing and properties in all directions
- •
- Crystals in different directions(see above):
- •
- different atom packing and so different properties


Liquid
Specific Volume
Liquid
2
Rubbery
State
1
Glassy State
Crystalline State
Temperature
Tg
T_f
Volume, Enthalpy
Glass
b
a
Crystal
T_{ga}
T_{gb}
T_m
Temperature

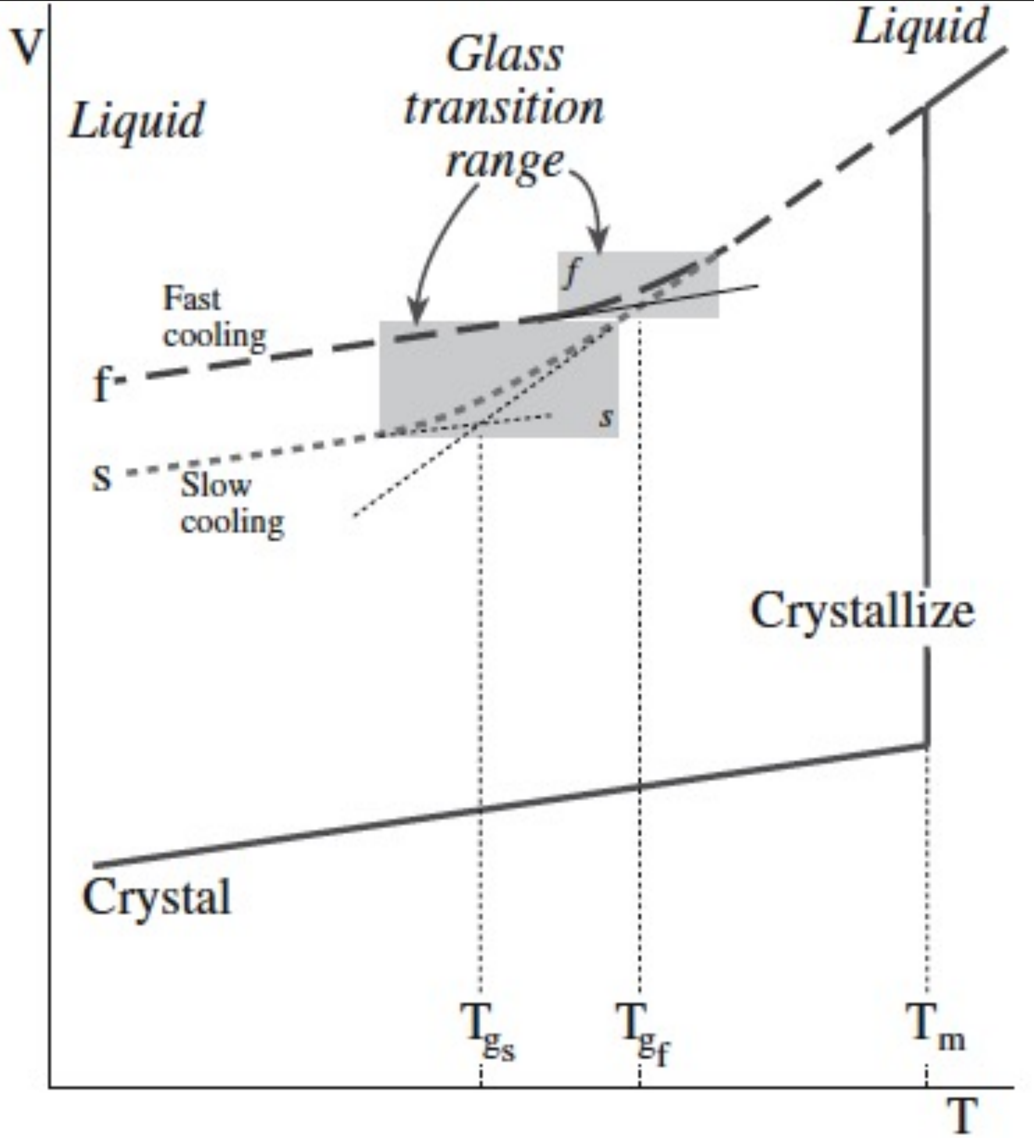
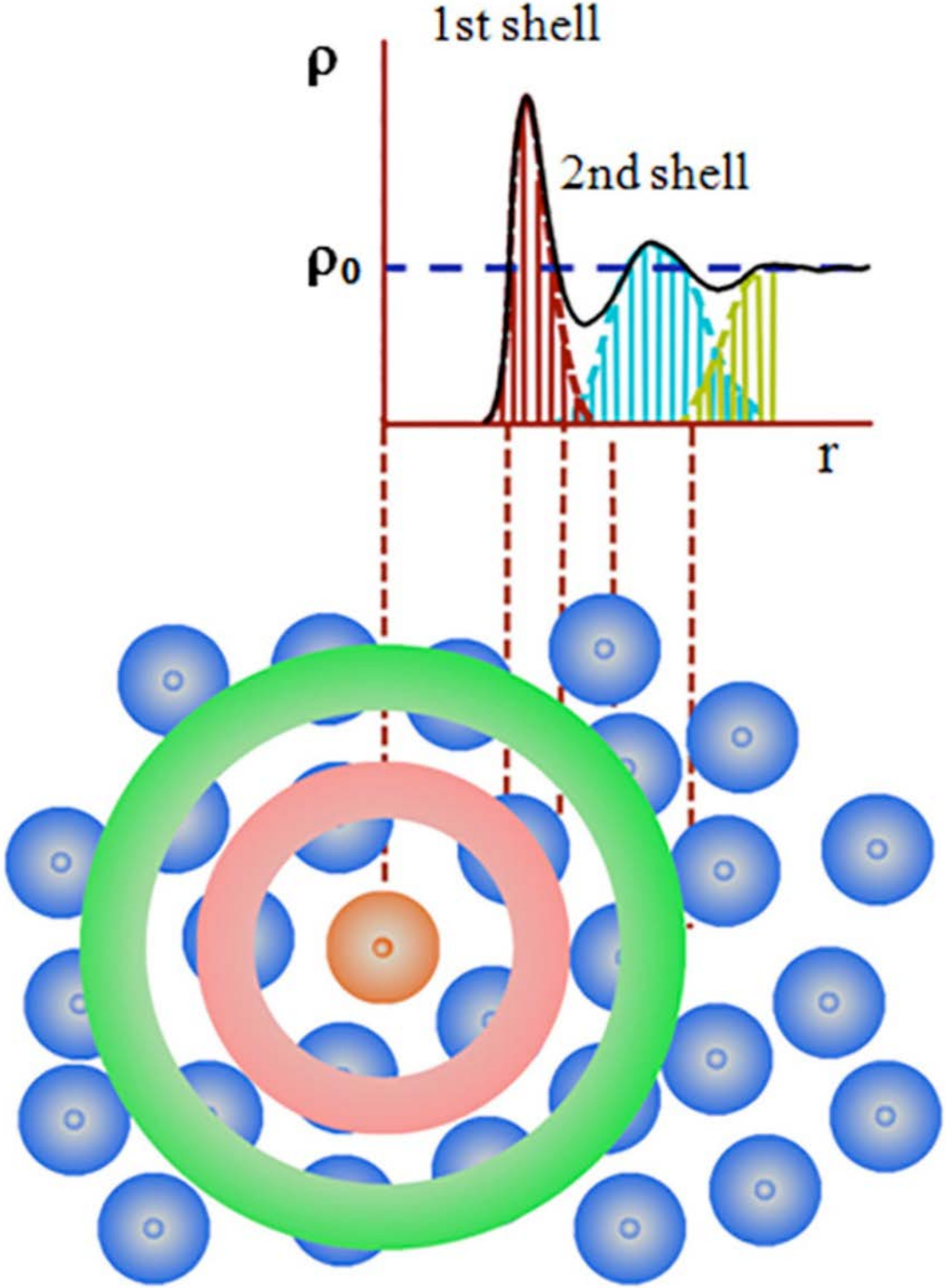
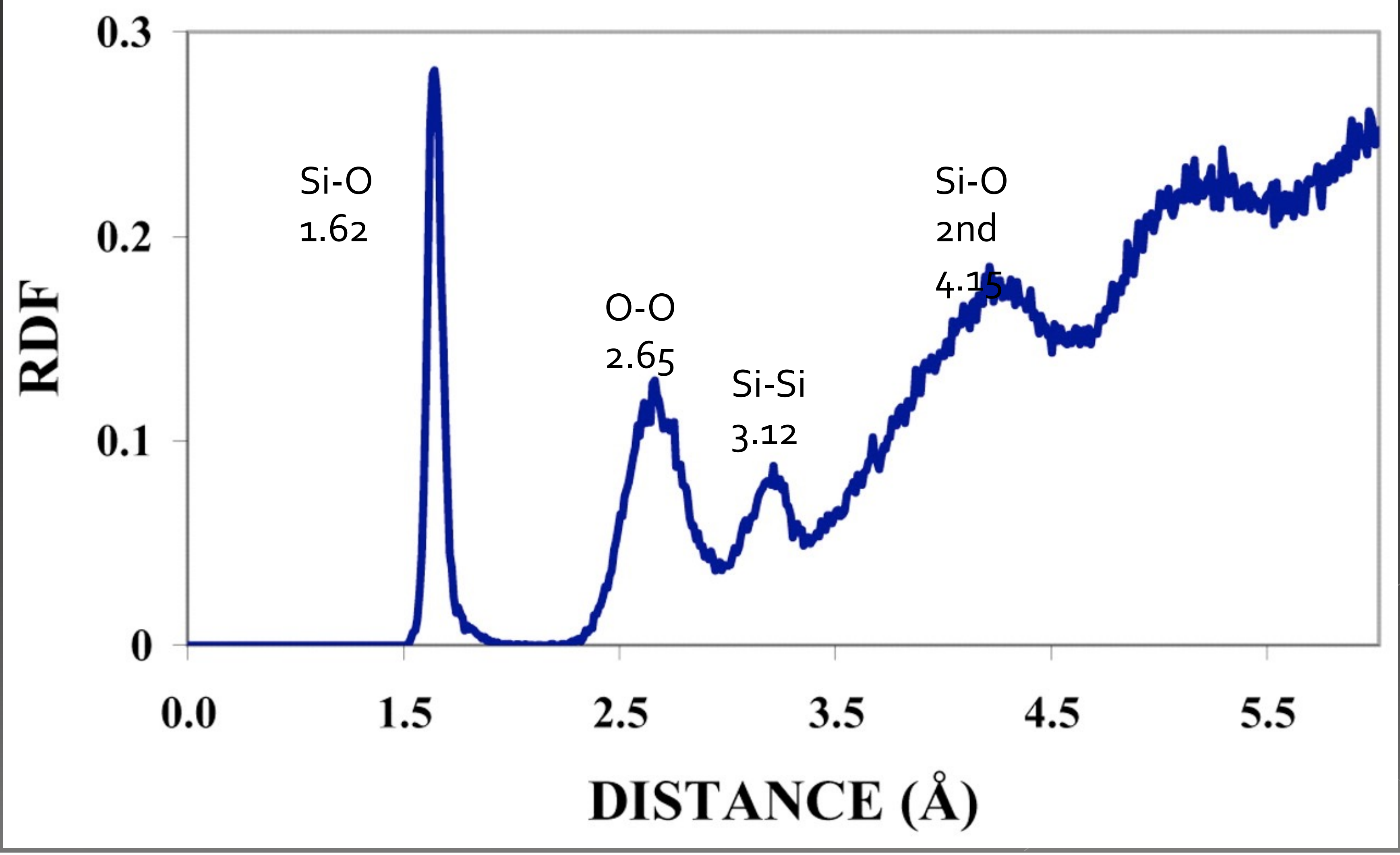


FIGURE 21.1 Plot of volume versus temperature for a liquid that forms a glass on cooling and one that forms a crystalline solid. The glass transition temperature, T_g, depends on the cooling rate and is not fixed like T_m.

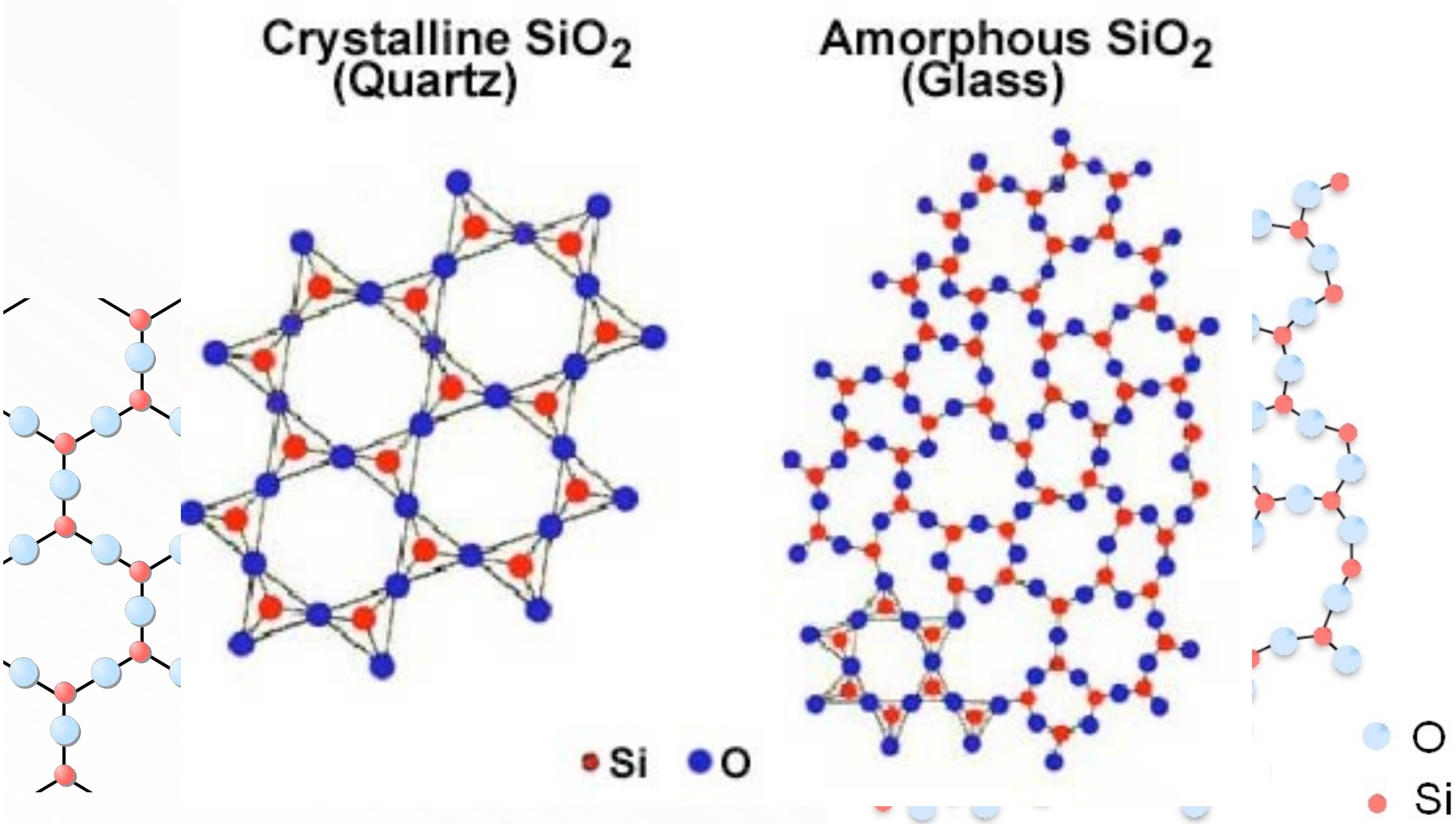
Pair distribution function of SiO2 glass

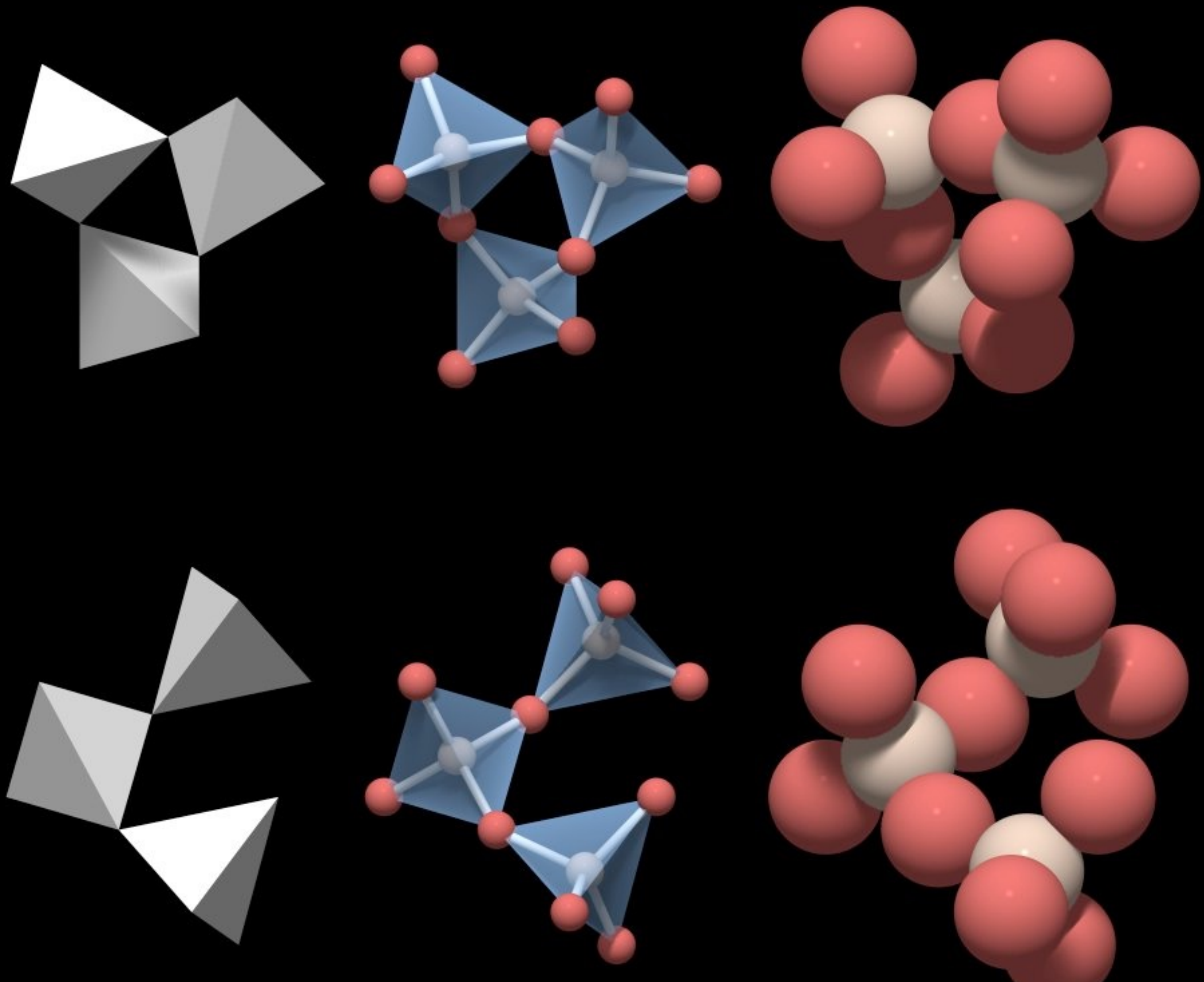


Radial distribution function for SiO_2



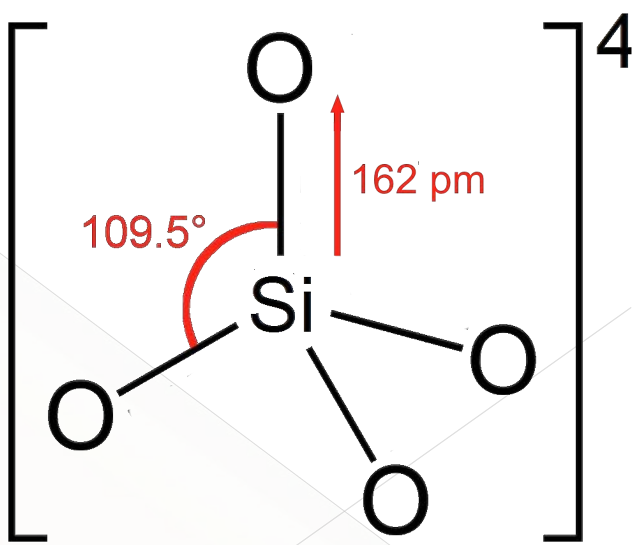
Glass structure:
SiO2 quartz compared to SiO2 glass





Zachariasen rules for glass A_mO_n

1) An oxygen atom is linked to no more than two glass-forming atoms A.
2) The number of oxygen atoms around each glass-forming atom A is small, perhaps 3 or 4.
3) Among the oxygen-containing polyhedra, a polyhedron cation A shares corners, but no sides or faces.
4) For three-dimensional networks of oxygen-containing polyhedra, at least three corners must be shared.



In general, all four rules should be satisfied for glass formation to occur.
Low coordination numbers, corner-sharing rules imply that glass formation is more likely with open, low density polyhedral structures.

1. Consider Silica:
    - covalent Si-O bond: *sp^3* hybrid
        - tetrahedral bonding
- Pauling's packing rule:

$$\frac{r(Si^{4+})}{r(O^{2-})} = \frac{0.40}{1.40} \approx 0.29 \quad \textit{prefers tetrahedral bonding}$$

- satisfies Zachariasen's rule #2.

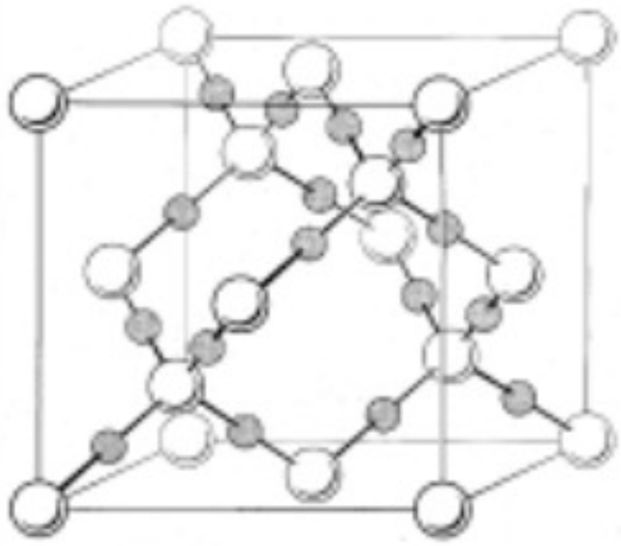
$$\frac{charge(Si^{4+})}{CN(Si^{4+})} = \frac{4}{4} = \frac{charge(O^{2-})}{CN(O^{2-})} = \frac{2}{2} \quad CN(O^{2-}) \text{ is } 2.$$

- satisfies Zachariasen's rule #1.

Crystal structure: sharing four corners:

All Rules are Satisfied: SiO_2 forms a glass.

β-cristobalite (SiO_2)



Cation CN:Anion CN = 4:2

2. Consider Magnesia (MgO):
- ionic Mg-O bond
  - Pauling's packing rule:

$$\frac{r(Mg^{2+})}{r(O^{2-})} = \frac{0.72}{1.40} \approx 0.51 \quad \textit{prefers octahedral bonding}$$

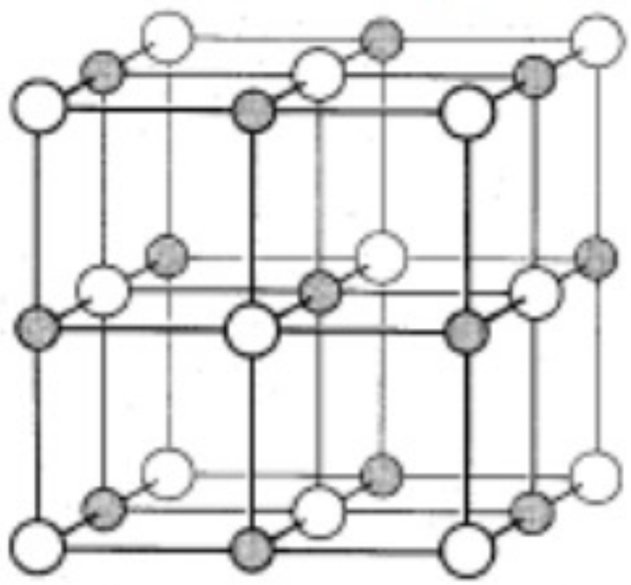
  - violates Zachariasen's rule #2.

$$\frac{charge(Mg^{2+})}{CN(Mg^{2+})} = \frac{2}{6} = \frac{charge(O^{2-})}{CN(O^{2-})} = \frac{2}{6} \quad CN(O^{2-}) \textit{ is } 6.$$

  - violates Zachariasen's rule #1.

Crystal structure: edge-sharing polyhedra;
Rules are Not Satisfied: MgO does not form a glass.

Rock salt (MgO)



Cation CN:Anion CN = 6:6

3. Consider Alumina (Al_2O_3):

- Pauling's packing rule:

$$\frac{r(Al^{3+})}{r(O^{2-})} = \frac{0.53}{1.40} \approx 0.38 \quad \textit{octahedral / tetrahedral boundary}$$

- octahedral CN preferred in Al_2O_3.

$$\frac{charge(Al^{3+})}{CN(Al^{3+})} = \frac{3}{6} = \frac{charge(O^{2-})}{CN(O^{2-})} = \frac{2}{4} \quad CN(O^{2-}) \text{ is } 4.$$

- violates Zachariasen's rule #1.

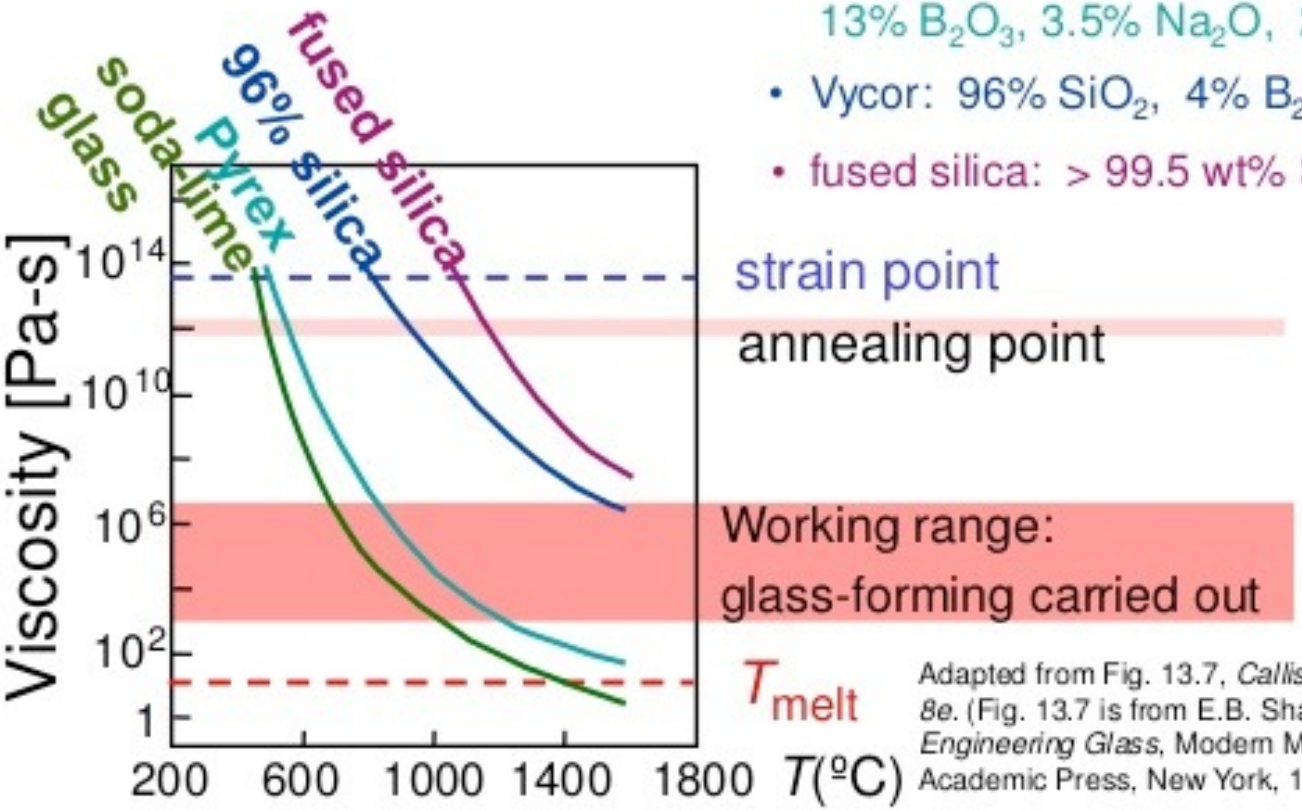
Al_2O_3 does not form a glass.

Elements for glass formation

Formers	Modifiers	Intermediate
B	Sc	Ti
Si	La	Zr
Ge	Na	Pb
Al	K	Al
V	Rb	Th
As	Cs	

Log Glass Viscosity vs. Temperature

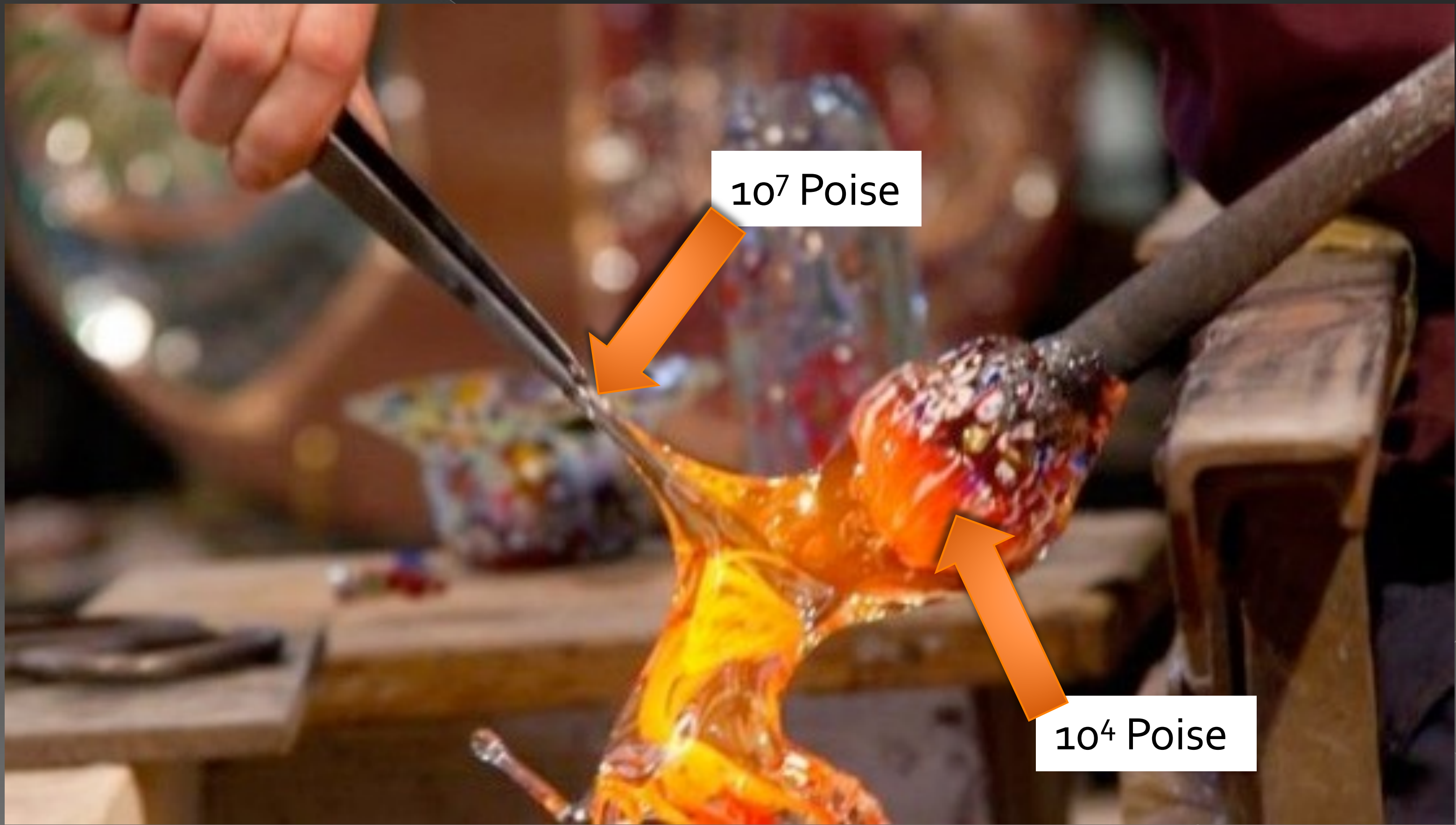
- Viscosity decreases with *T*



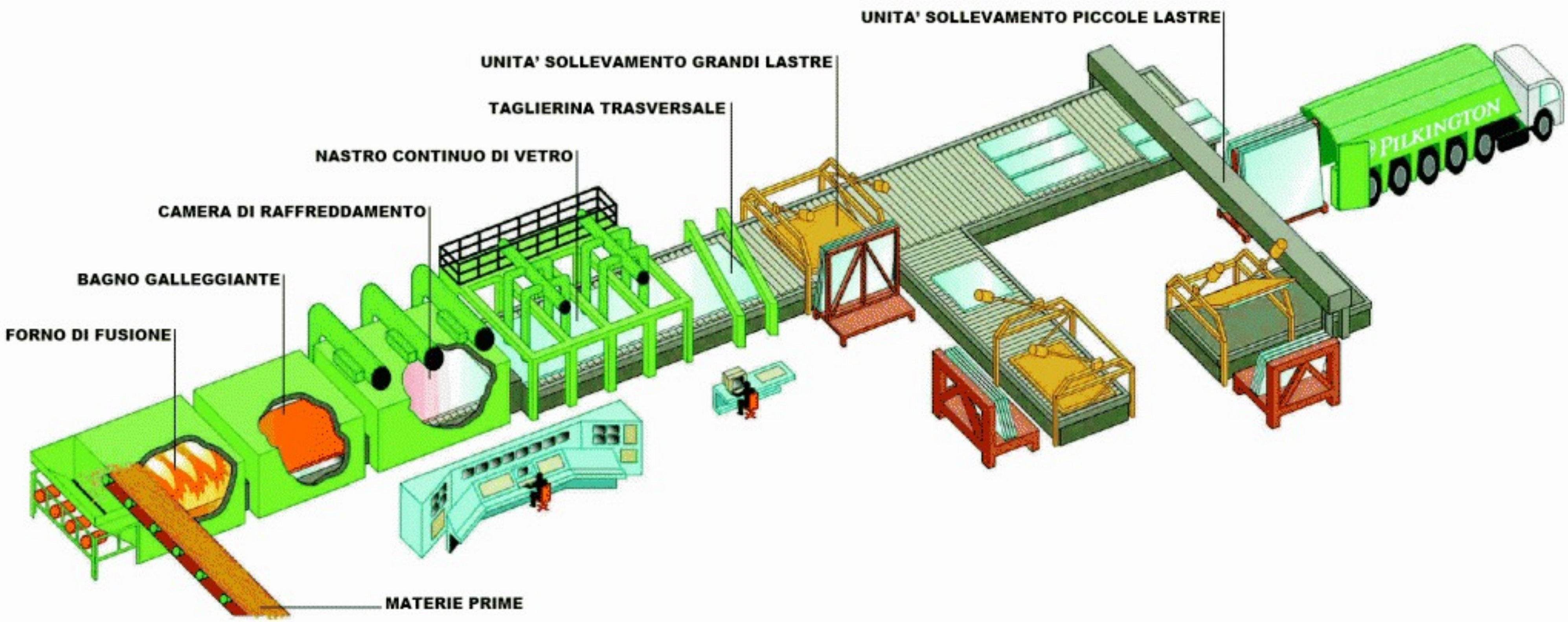
- soda-lime glass: 70% SiO_2 balance Na_2O (soda) & CaO (lime)
- borosilicate (Pyrex): 13% B_2O_3, 3.5% Na_2O, 2.5% Al_2O_3
- Vycor: 96% SiO_2, 4% B_2O_3
- fused silica: > 99.5 wt% SiO_2

Adapted from Fig. 13.7, *Callister & Rethwisch 8e*. (Fig. 13.7 is from E.B. Shand, *Engineering Glass*, Modern Materials, Vol. 6, Academic Press, New York, 1968, p. 262.)

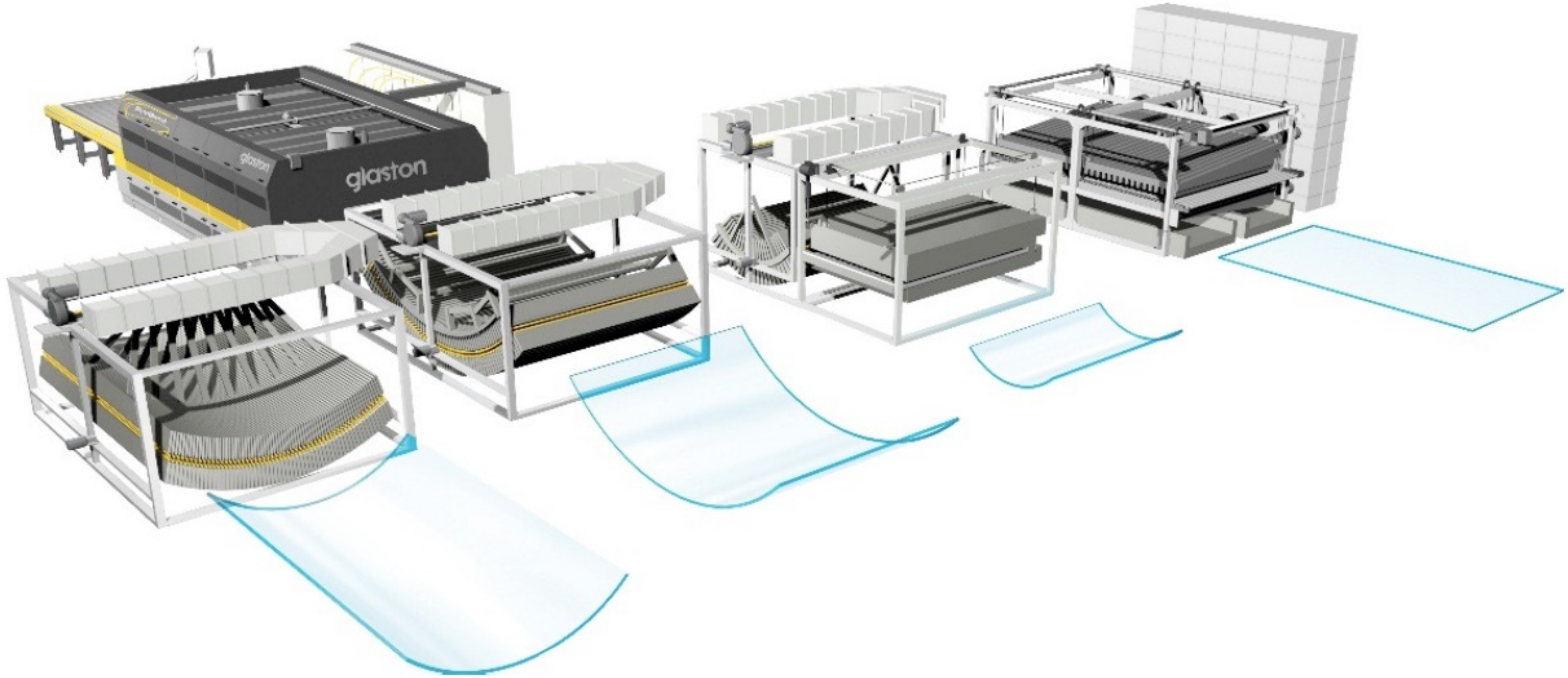
Glass Viscosity and Workability

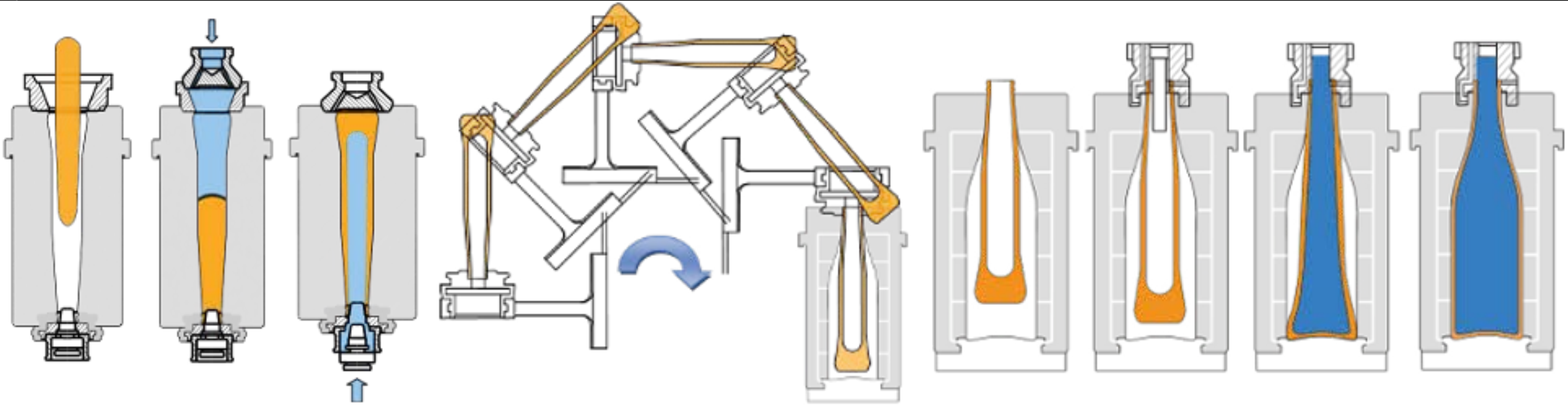


Pilkington process



Glass bending





Bottle production line

://www.youtube.com/watch?v=k8MmEuvugG4

Dielectrics

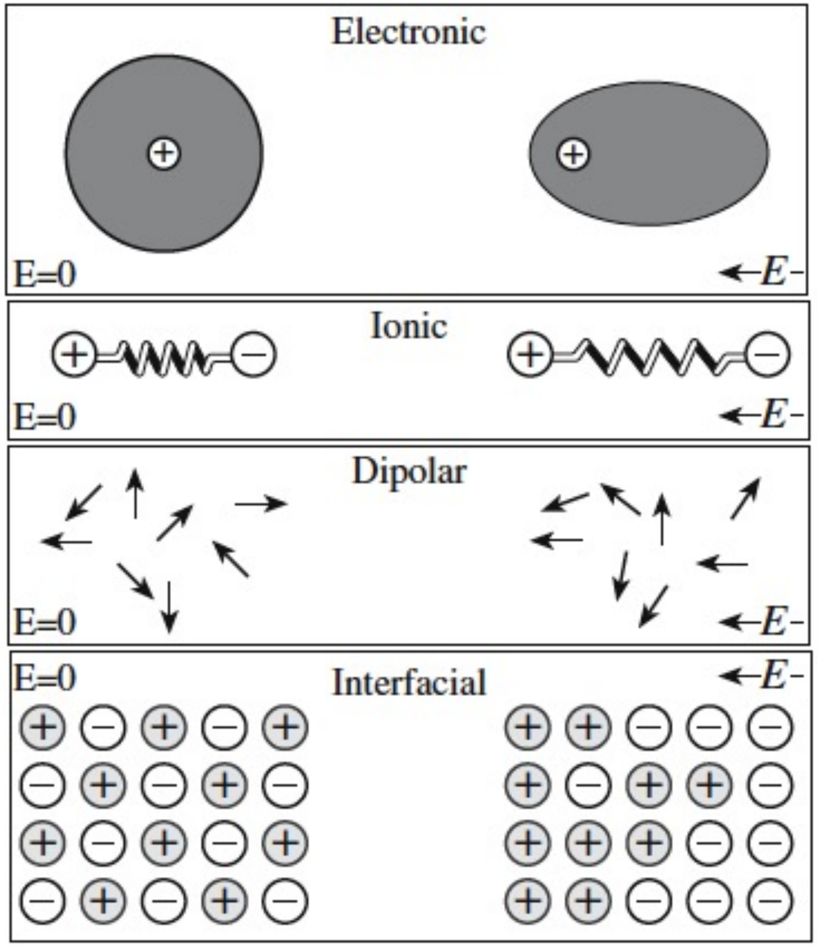


FIGURE 31.1 Illustration of the different polarization mechanisms n a solid.

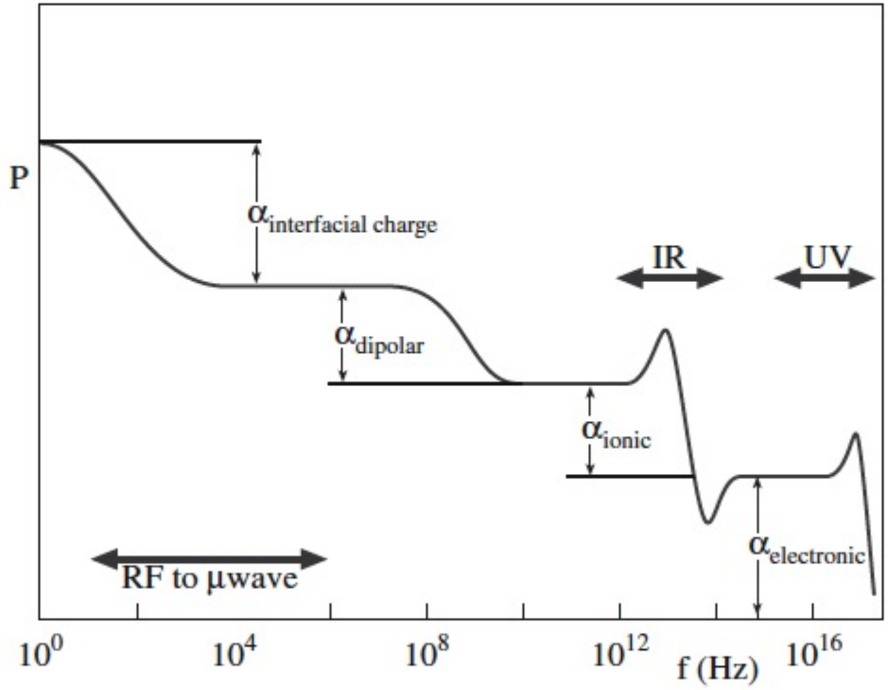


FIGURE 31.2 Frequency dependence of polarization.

TABLE 31.2 Dielectric Constants of Various Ceramics

Material	*κ at 1 MHz*	*Material*	*κ at 1 MHz*
Diamond	5.5–6.6	Al_2O_3	8.8
SiO_2	3.7–3.8	MgO	9.6
NaCl	5.9	$BaTiO_3$	3000
Mica	5.4–8.7	Pyrex glass	4.0–6.0
Soda-lime glass	7.0–7.6	TiO_2	14–110
Steatite (SiO_2 + MgO + Al_2O_3)	5.5–7.5	Forsterite ($2MgO \cdot SiO_2$)	6.2
Cordierite (SiO_2 + MgO + Al_2O_3)	4.5–5.4	Mullite	6.6
High-lead glass	19		

[redacted]

[redacted]

TABLE 31.4 Dielectric Strengths for Various Ceramics

Material	*Dielectric strength (MV/cm at 25°C)*
Al_2O_3 (99.5%)	0.18
Al_2O_3 (94.0%)	0.26
High-voltage porcelain	0.15
Steatite porcelain	0.10
Lead glass	0.25
Lime glass	2.5
Borosilicate glass	5.8
Fused quartz	6.6
Quartz crystal	6.0
NaCl [100], [111], [110]	2.5, 2.2, 2.0
Muscovite mica	10.1

Non destructive testing Techniques

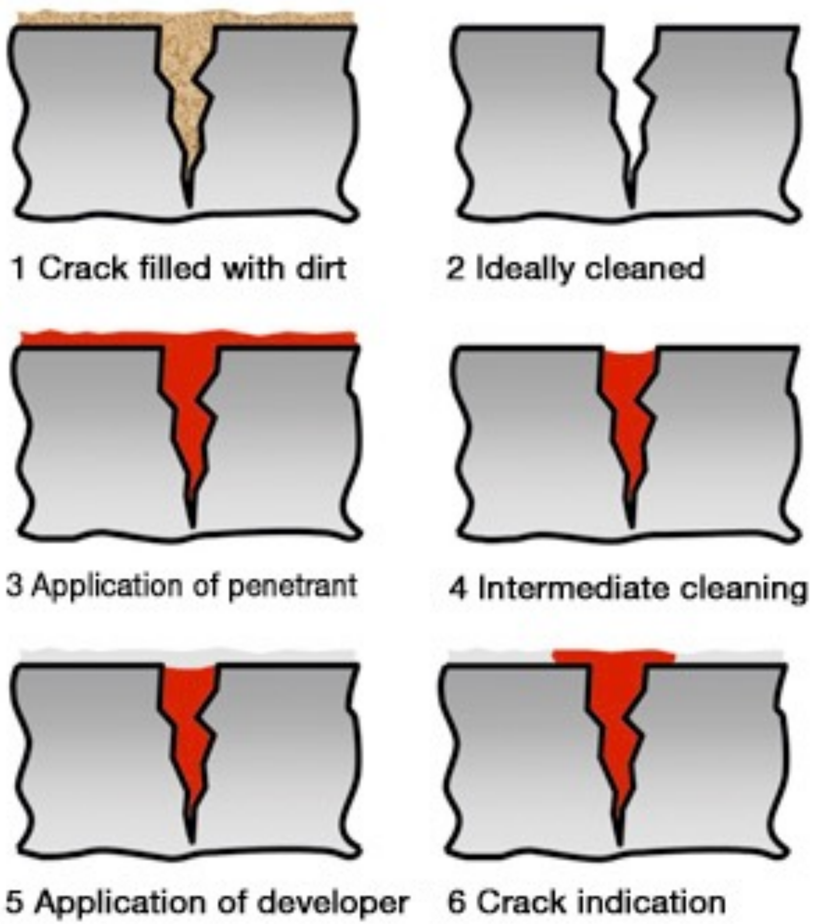
- Visual inspection
- Penetrant dyes
- Ultrasonic testing
- Radiographic testing
- Magnetoscopic testing
- Eddy currents

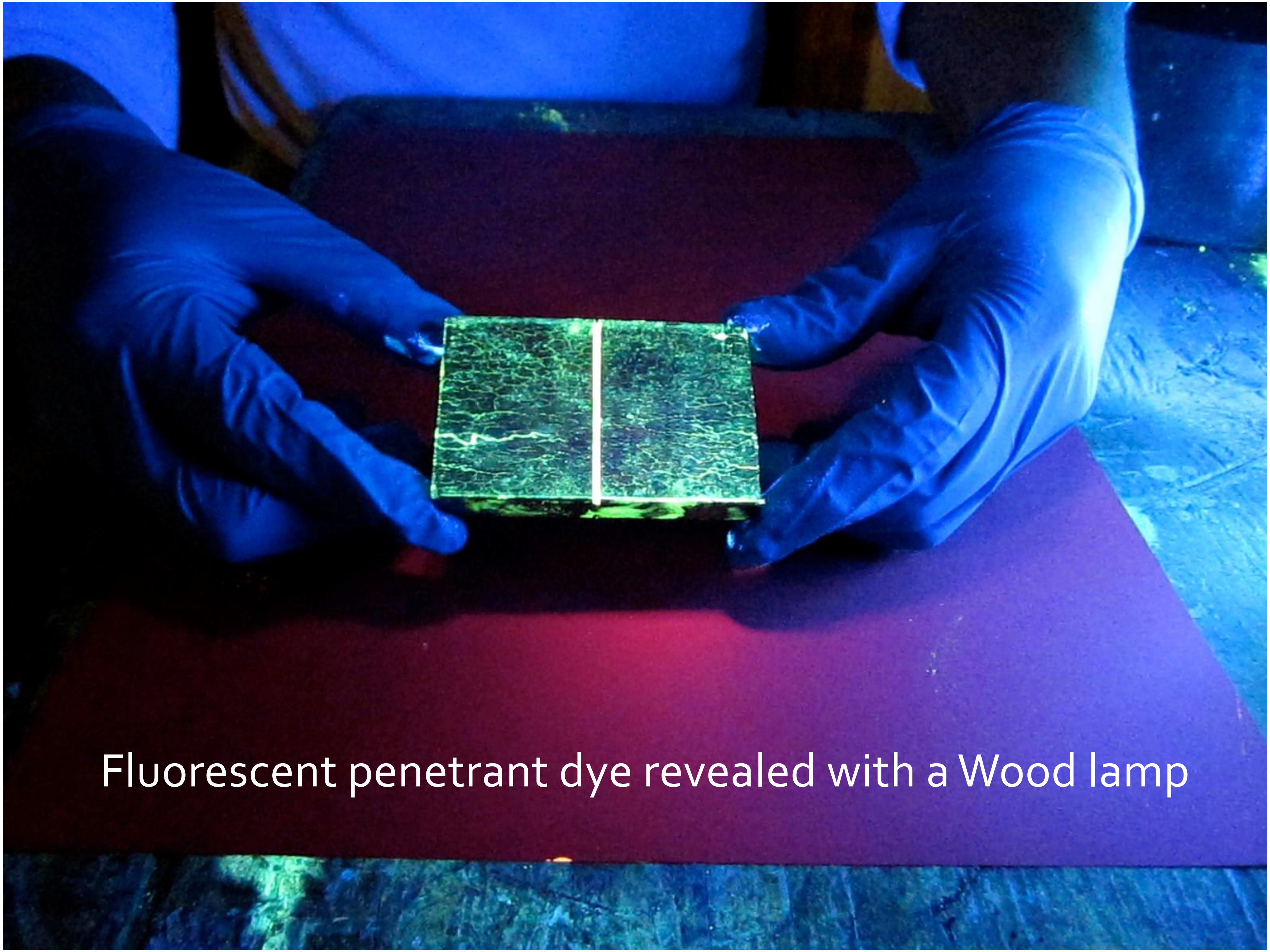
Proof testing:

1) load configuration as similar as possible to service condiction

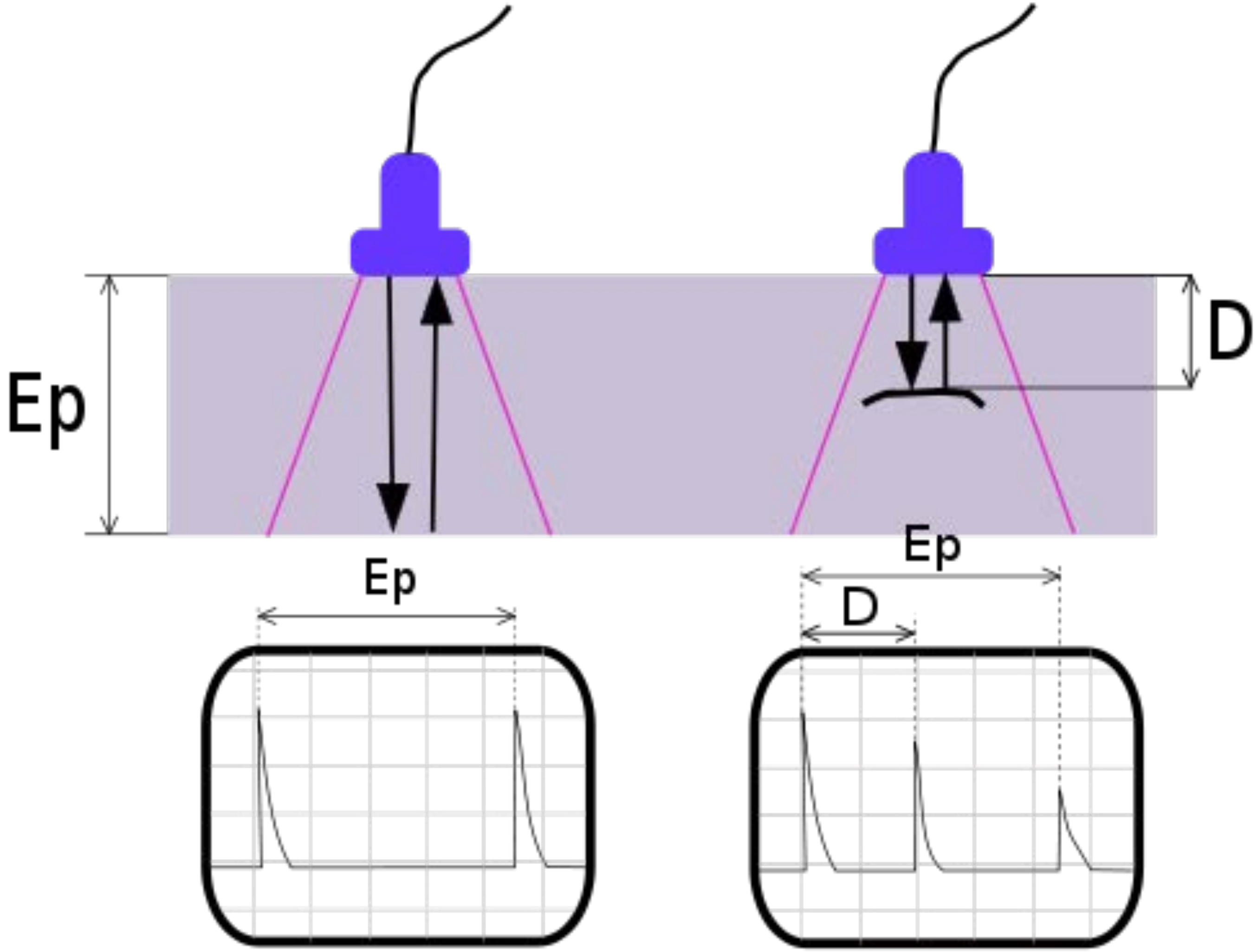
2) one single test slightly above load/stress values in service

Liquid penetrant dyes




Fluorescent penetrant dye revealed with a Wood lamp

Ultrasonic testing



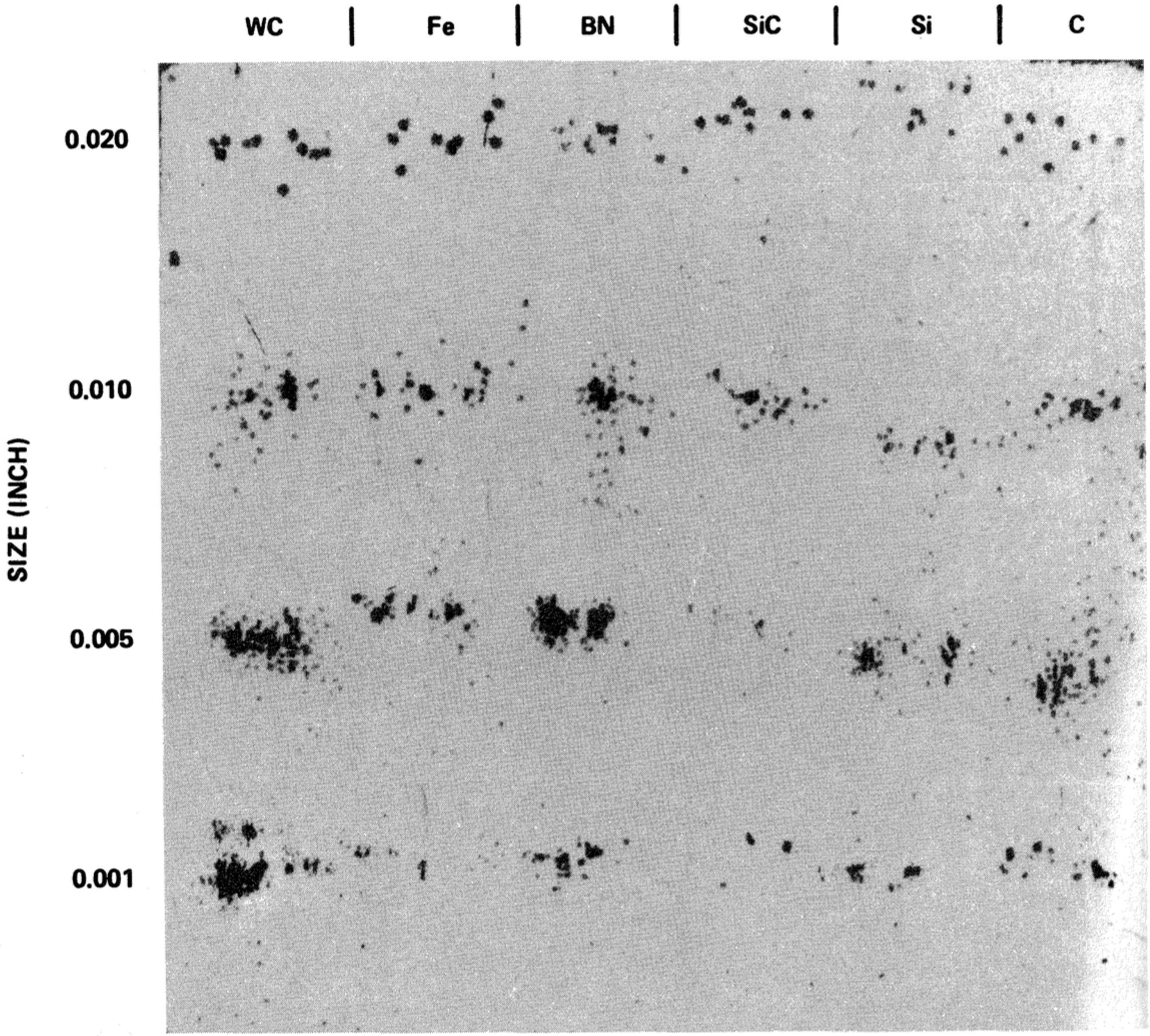
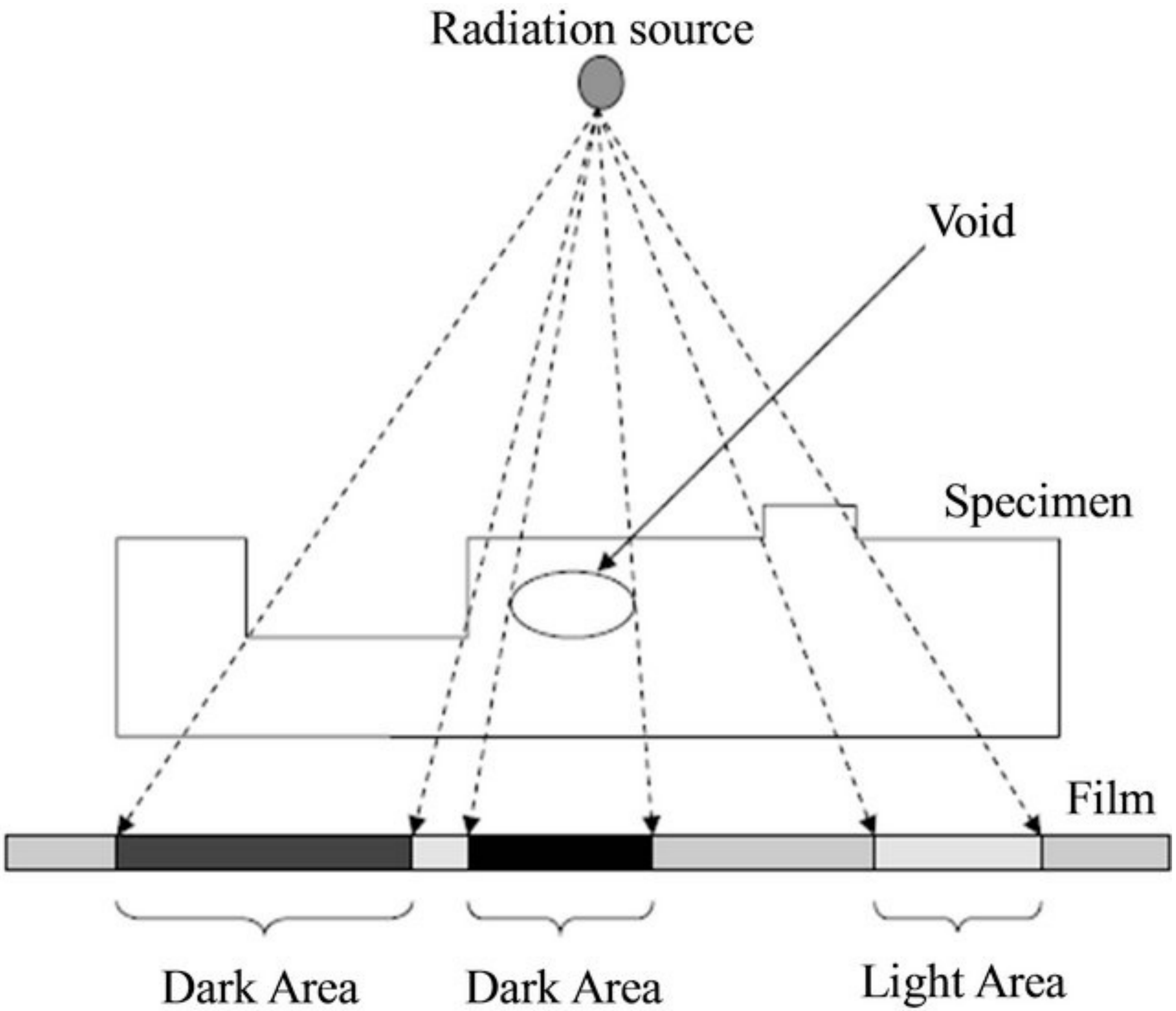
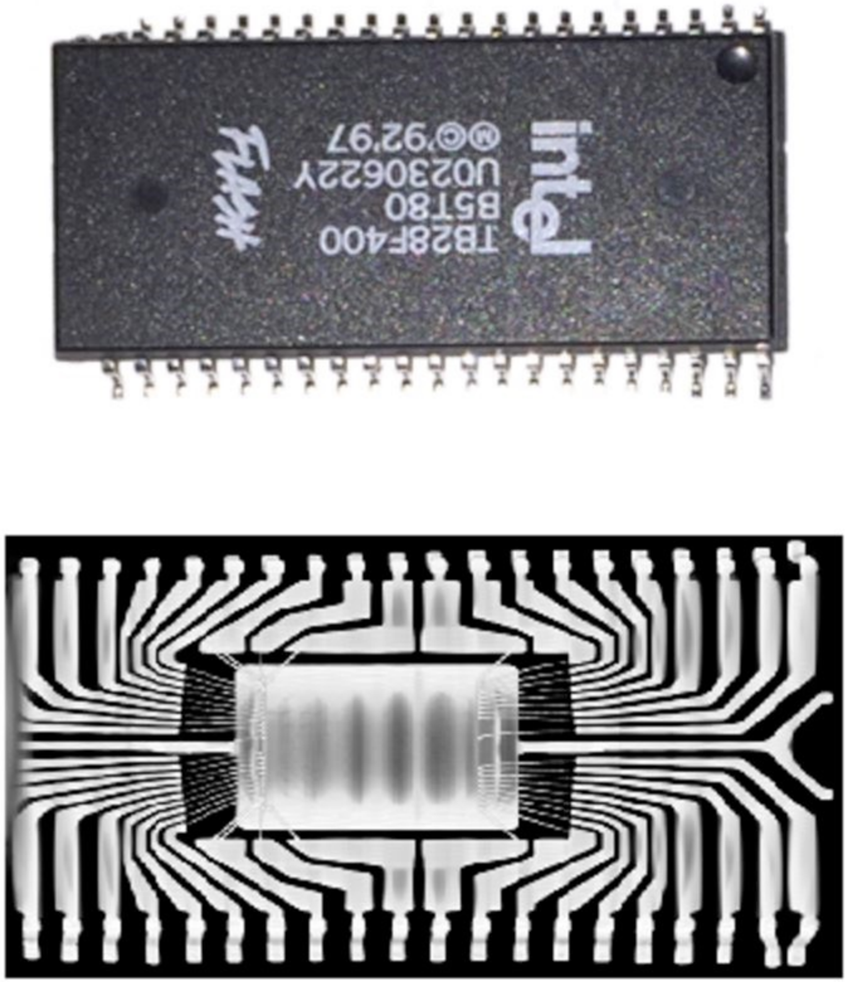


Figure 13.10 Ultrasonic C-scan with a 25-MHz transducer of a 0.64-cm (0.25-in.)-thick hot-pressed Si_3N_4 plate. (Courtesy Garrett Turbine Engine Company, Phoenix, Ariz., Division of Allied-Signal Aerospace.)

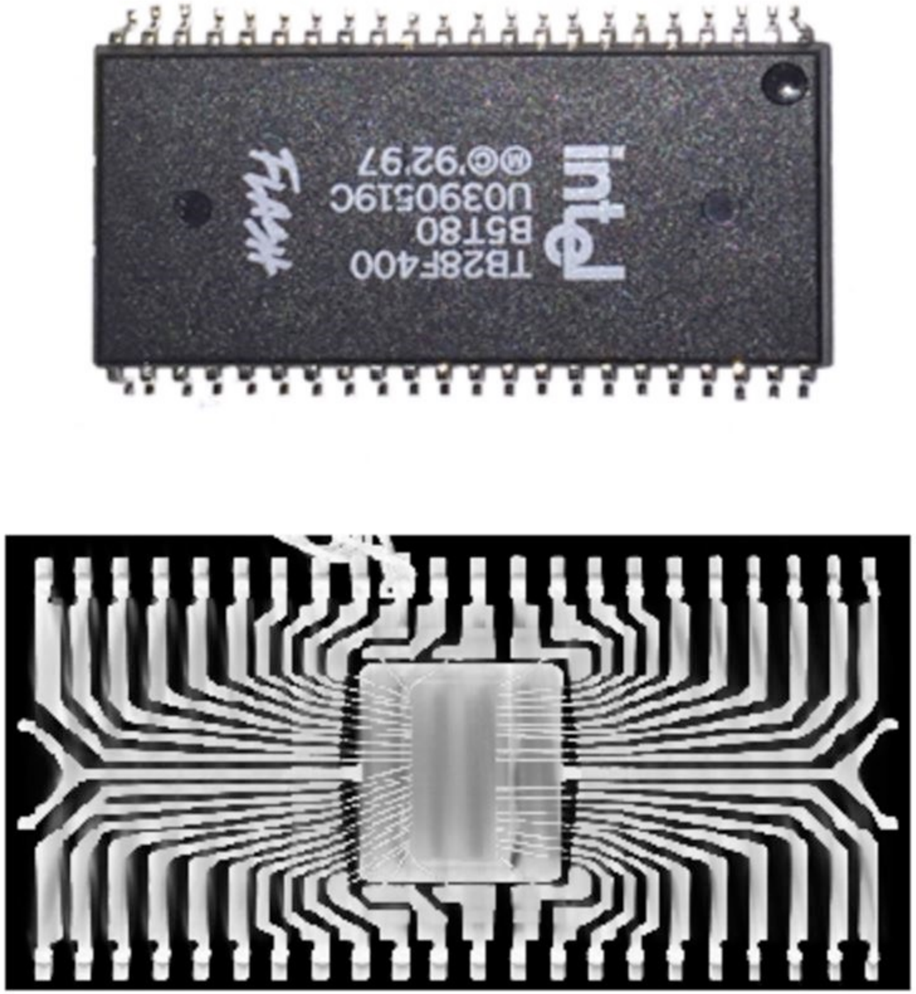
Radiographic testing



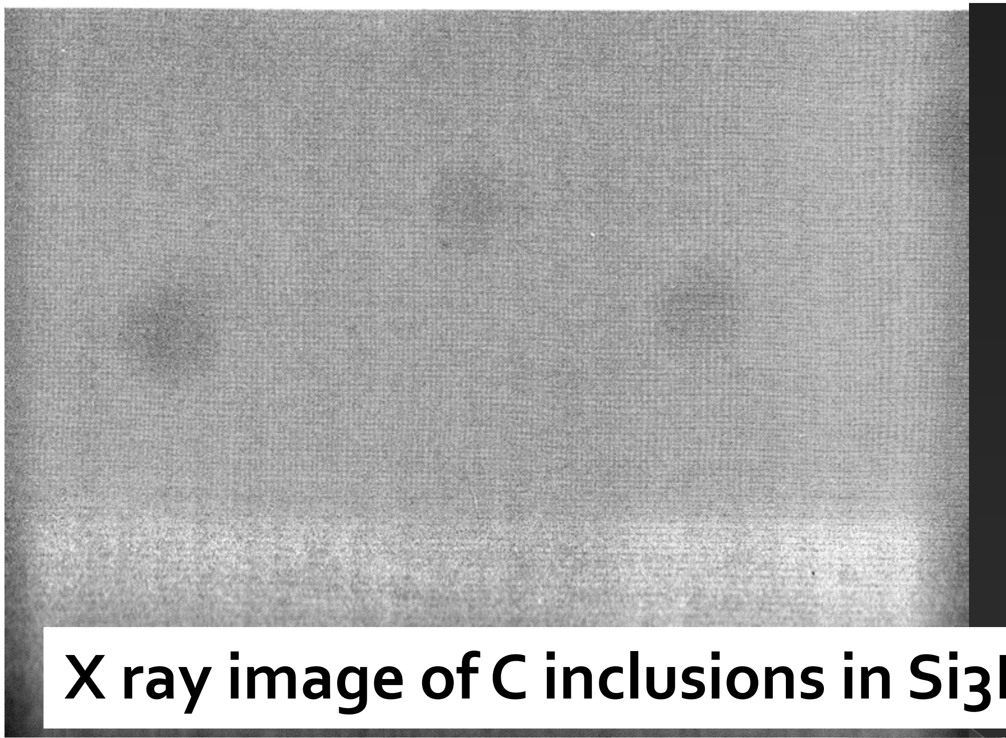
Radiographic testing of two chips

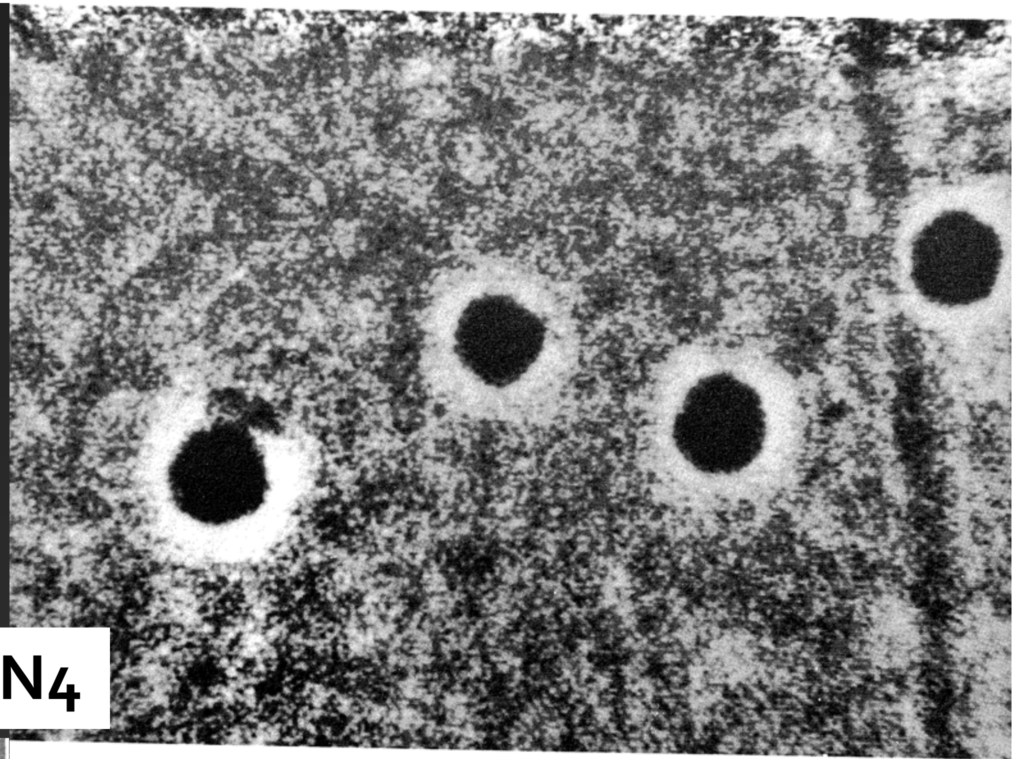


Counterfeit

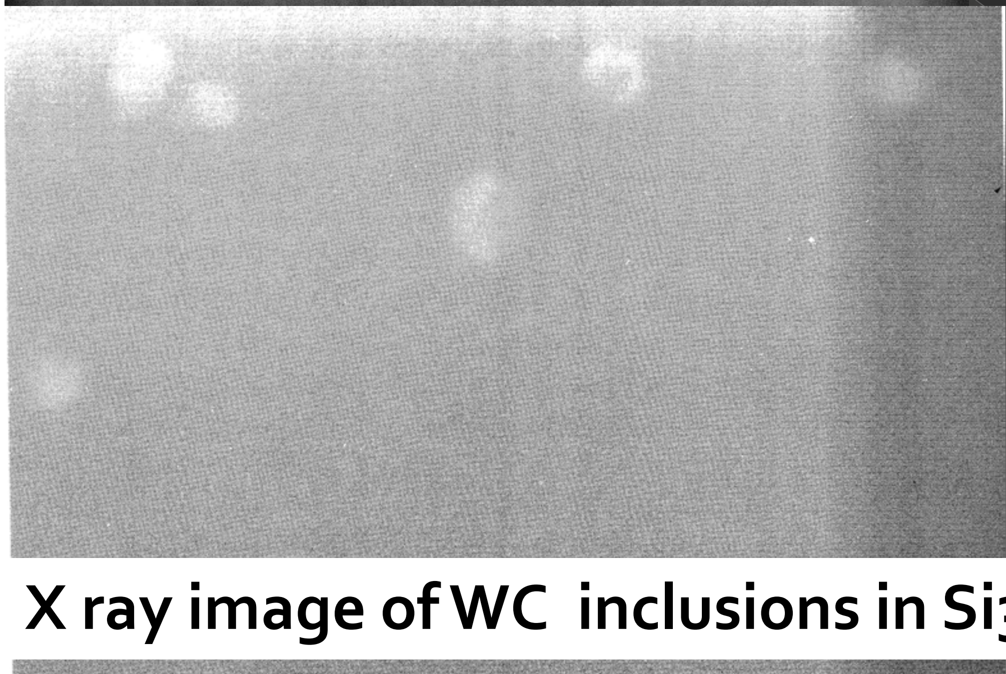


Authentic





X ray image of C inclusions in Si_3N_4



X ray image of WC inclusions in Si_3N_4

Magnetoscopic testing

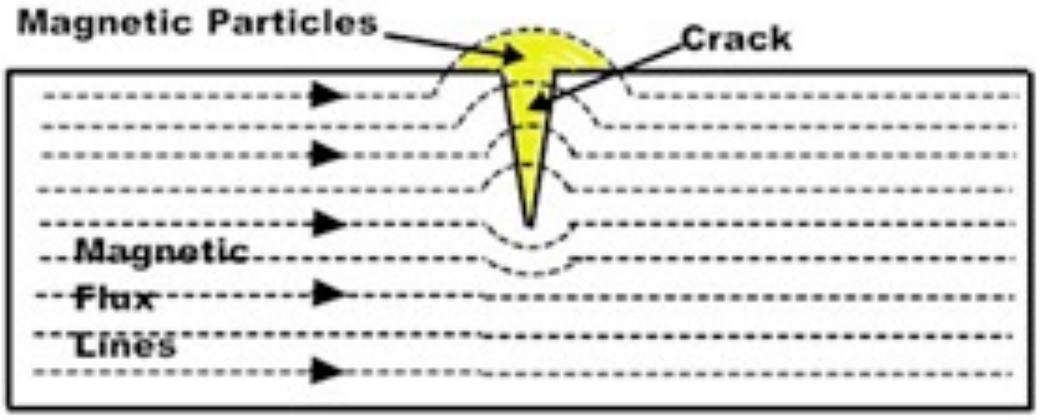


Figure 1

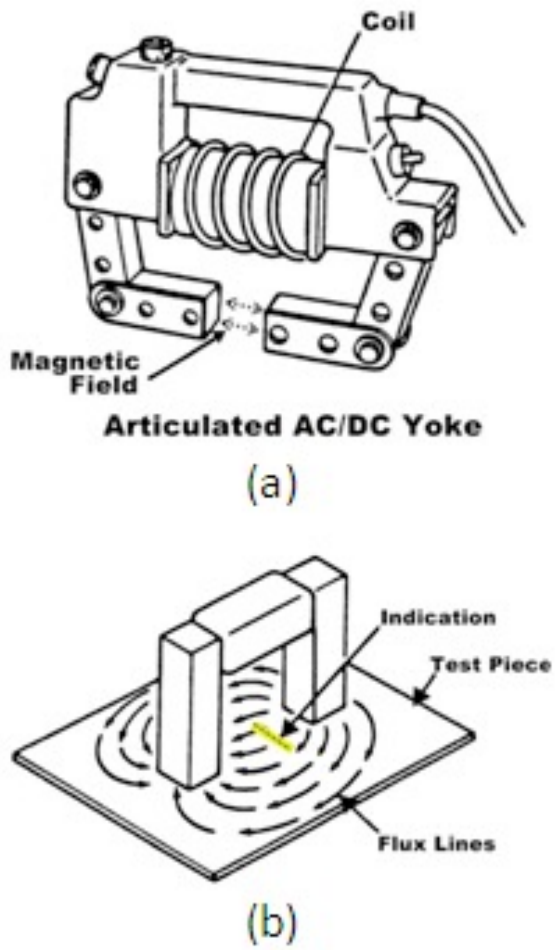


Figure 2

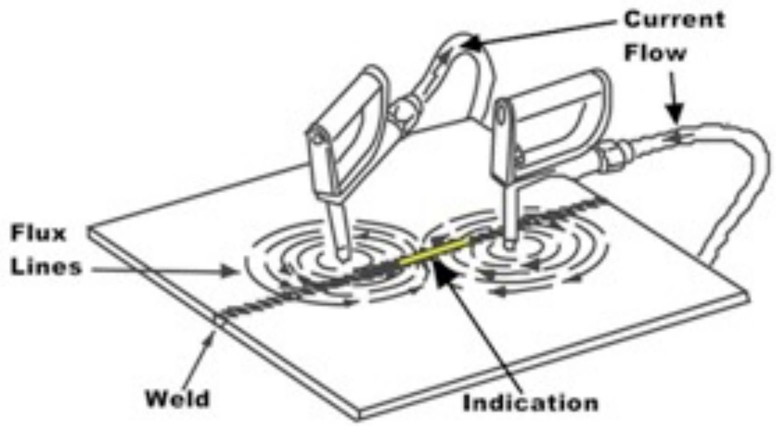


Figure 3

Eddy current testing

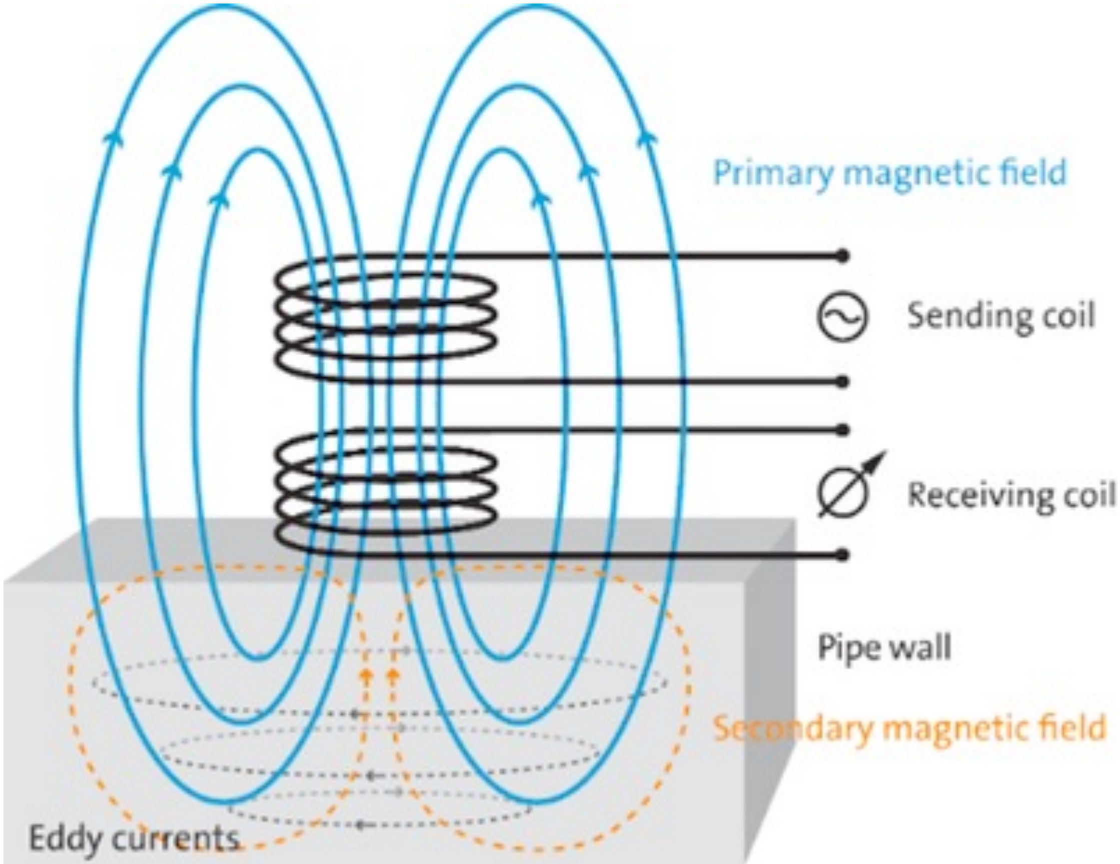


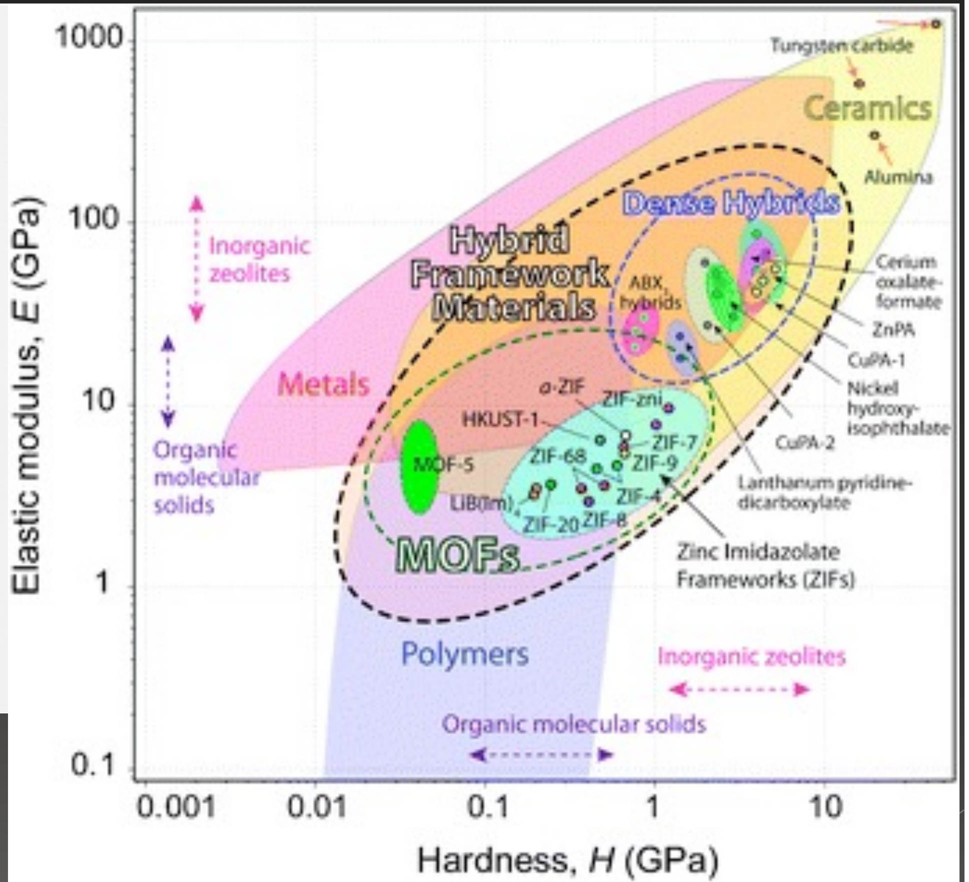
Table 14.2 Examples of Design Requirements of Various Applications and Ceramics with Properties Which Match the Requirements

Application	Requirements of the applications	Candidate ceramics	Key properties
Seal			
Turbine stator			
Heating element			
Rotary heat exchanger			
Heat sink for IC and transistor devices			
Furnace insulation			
Miniature capacitor			
High-speed, high-load bearing			
Segments of watch band			

SiC Heat exchanger

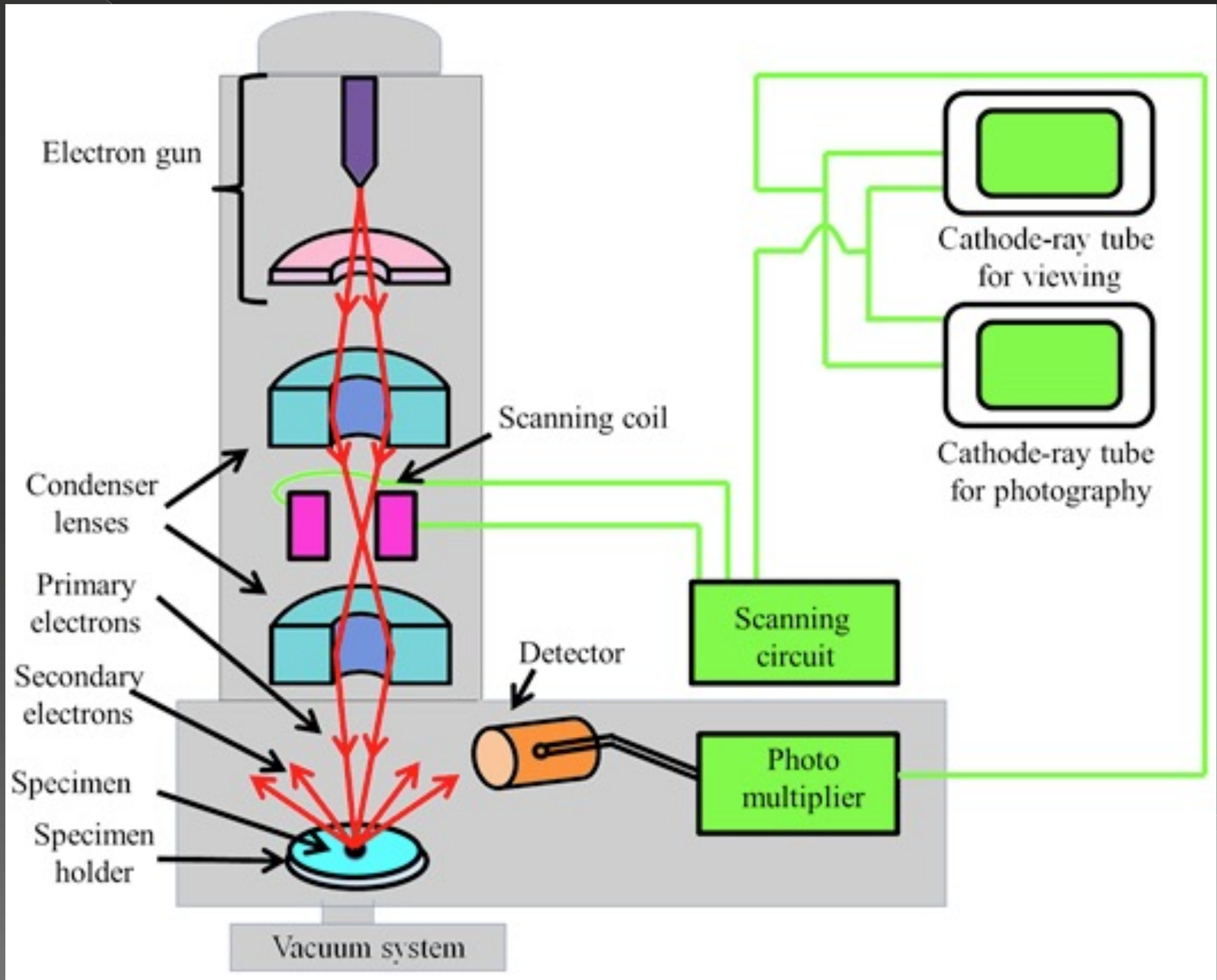
Ceramic seal for taps

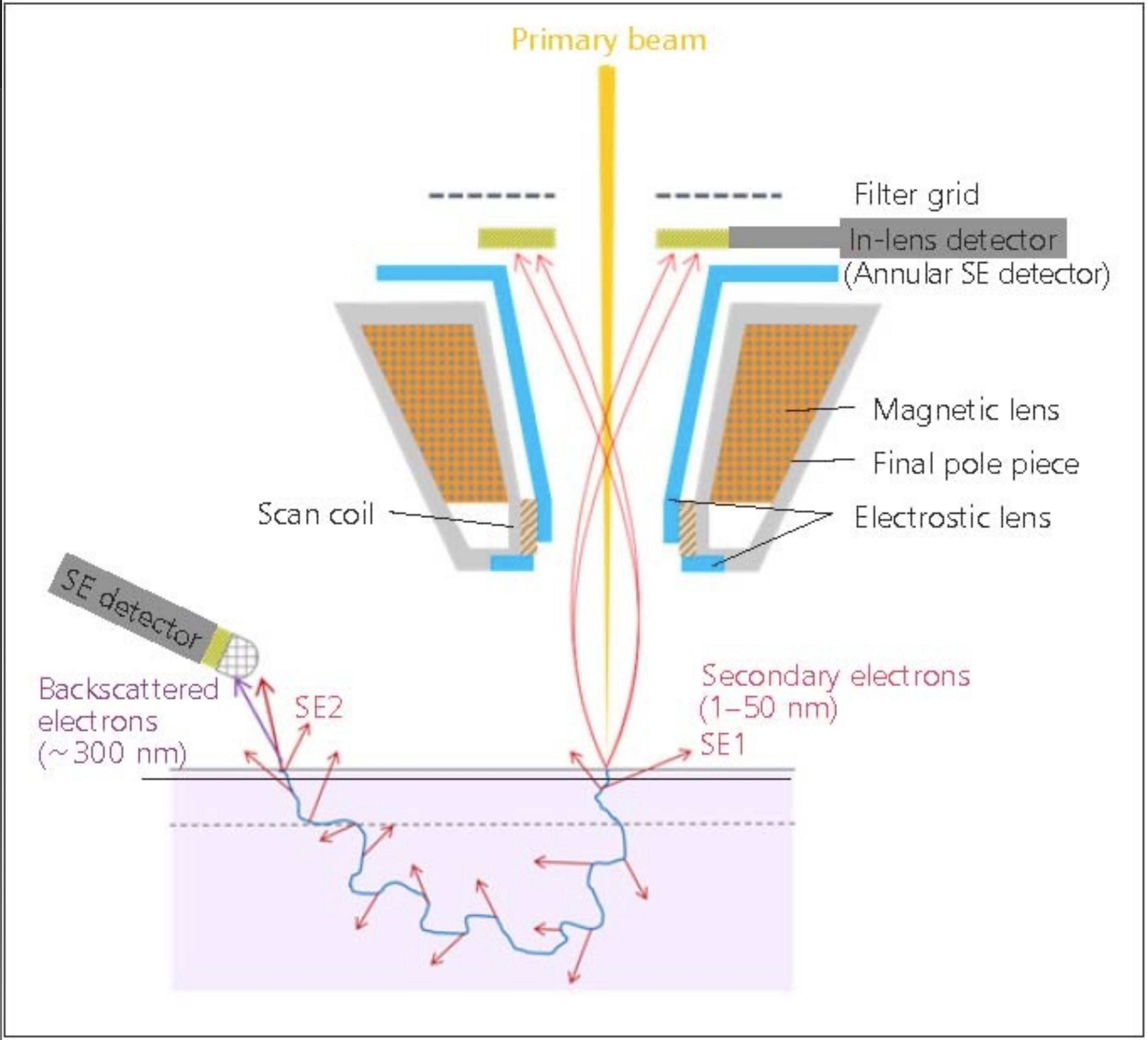
Sandblast nozzles



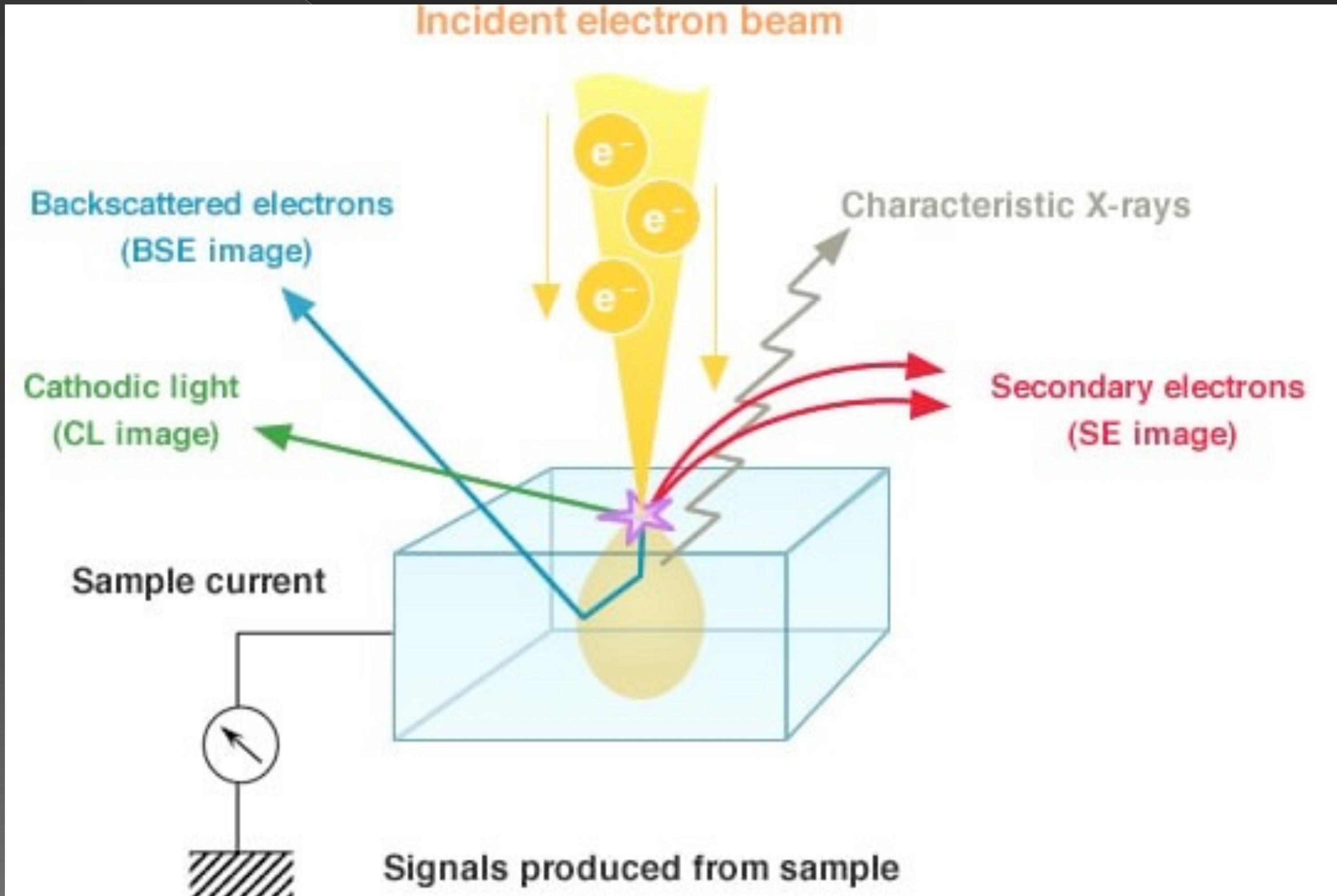
Rado watches

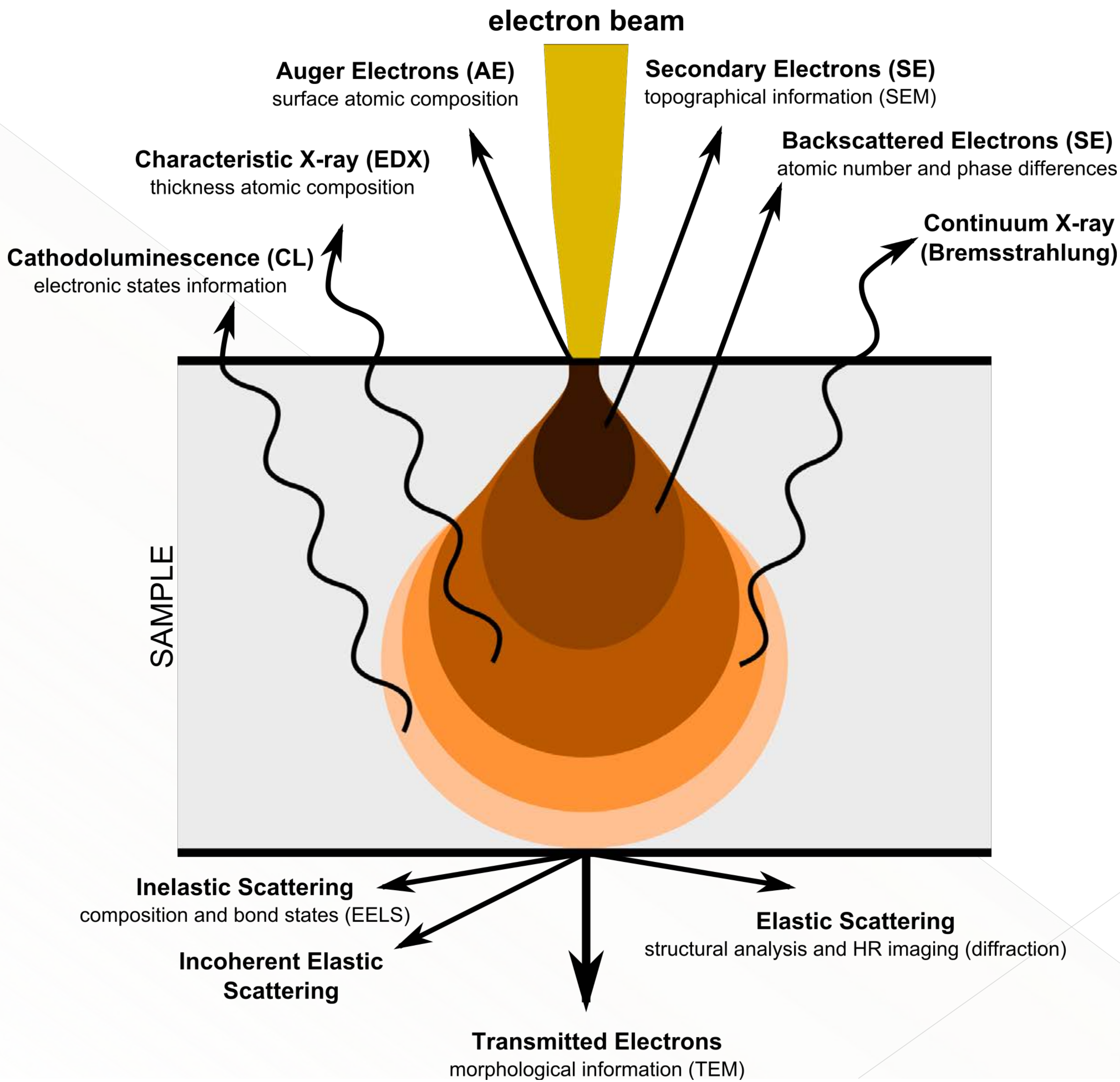
SEM fundamentals

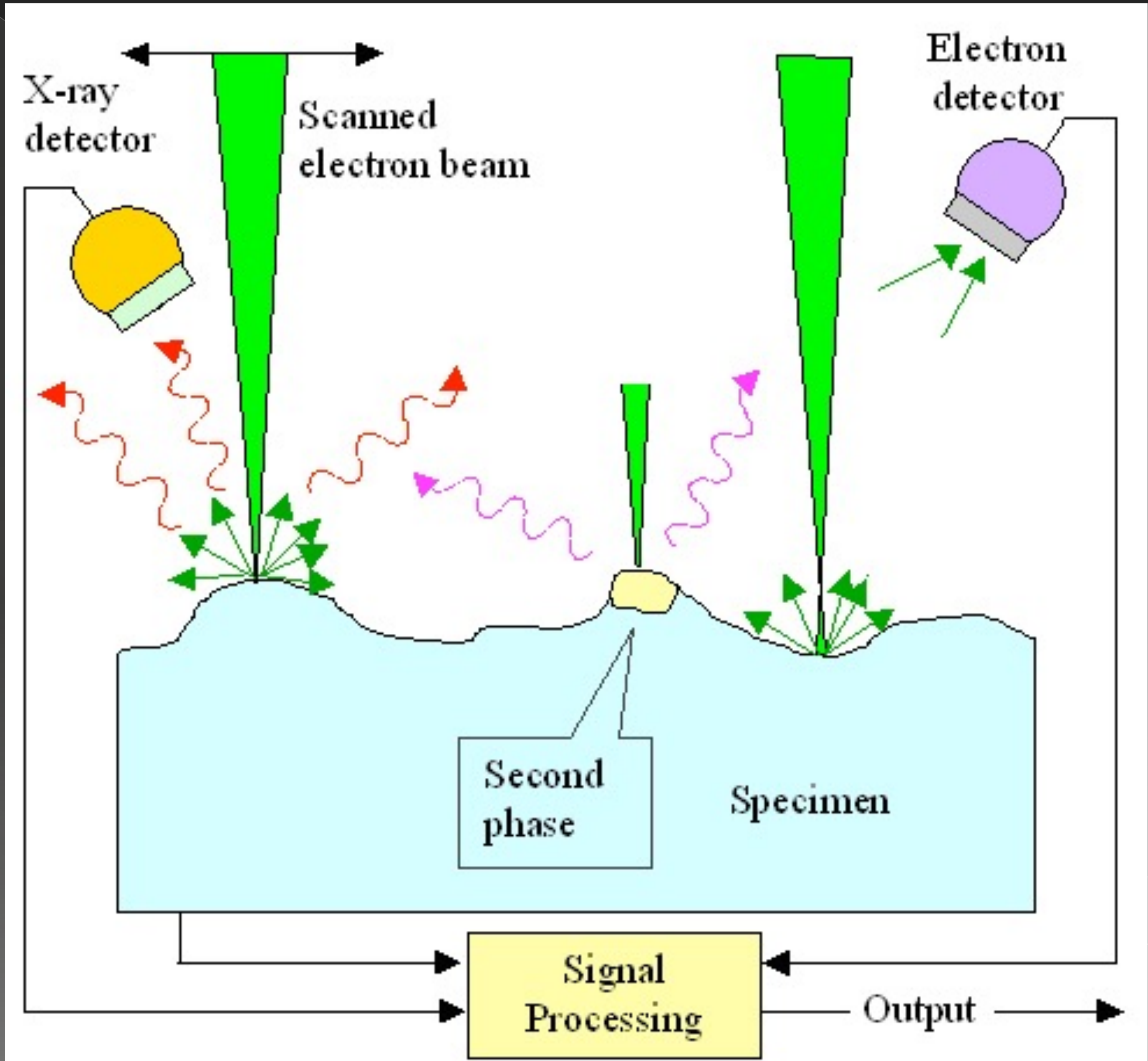



Primary beam
Filter grid
In-lens detector
(Annular SE detector)
Magnetic lens
Final pole piece
Electrostic lens
Scan coil
SE detector
Backscattered
electrons
(~300 nm)
SE2
Secondary electrons
(1–50 nm)
SE1

E-beam sample intercation




electron beam
Auger Electrons (AE)
surface atomic composition
Secondary Electrons (SE)
topographical information (SEM)
Characteristic X-ray (EDX)
thickness atomic composition
Backscattered Electrons (SE)
atomic number and phase differences
Cathodoluminescence (CL)
electronic states information
Continuum X-ray
(Bremsstrahlung)
SAMPLE
Inelastic Scattering
composition and bond states (EELS)
Incoherent Elastic
Scattering
Elastic Scattering
structural analysis and HR imaging (diffraction)
Transmitted Electrons
morphological information (TEM)


X-ray detector
Scanned electron beam
Electron detector
Second phase
Specimen
Signal Processing
Output

Compositional contrast

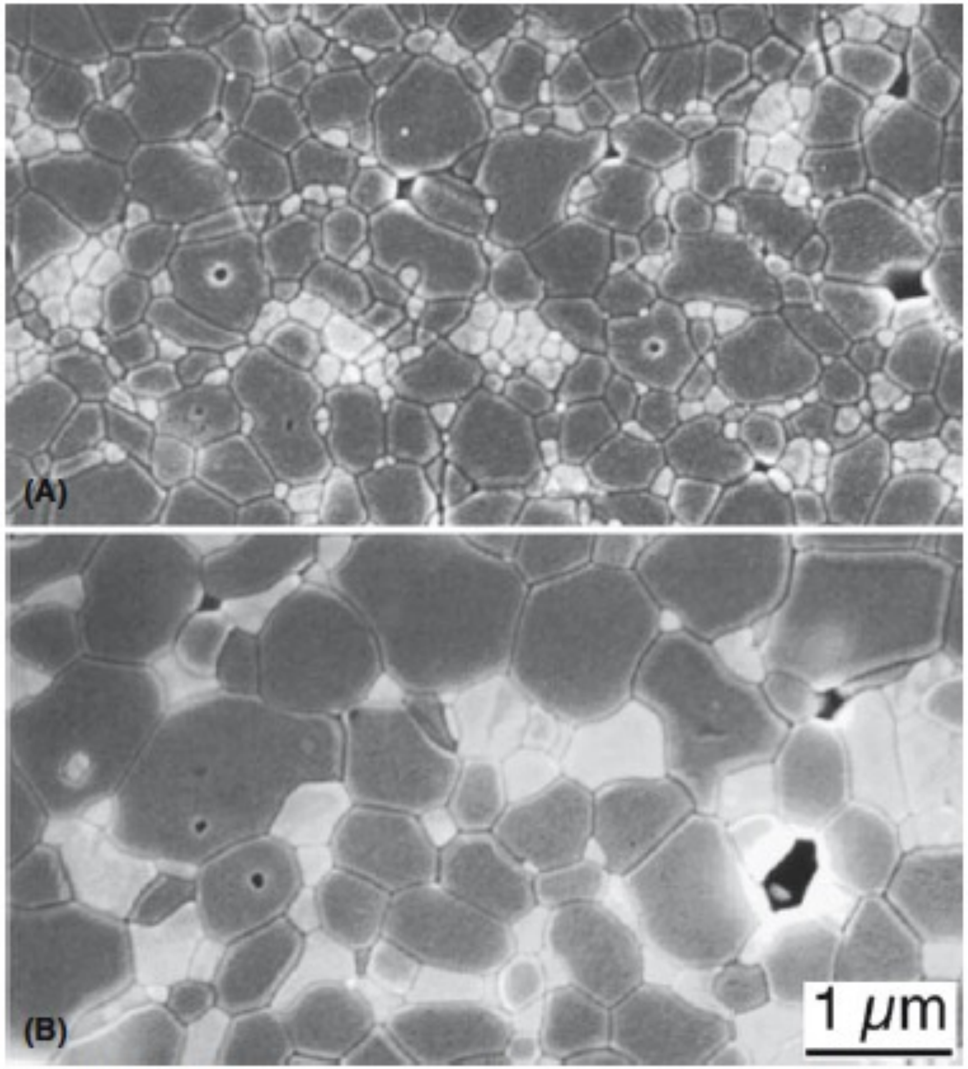
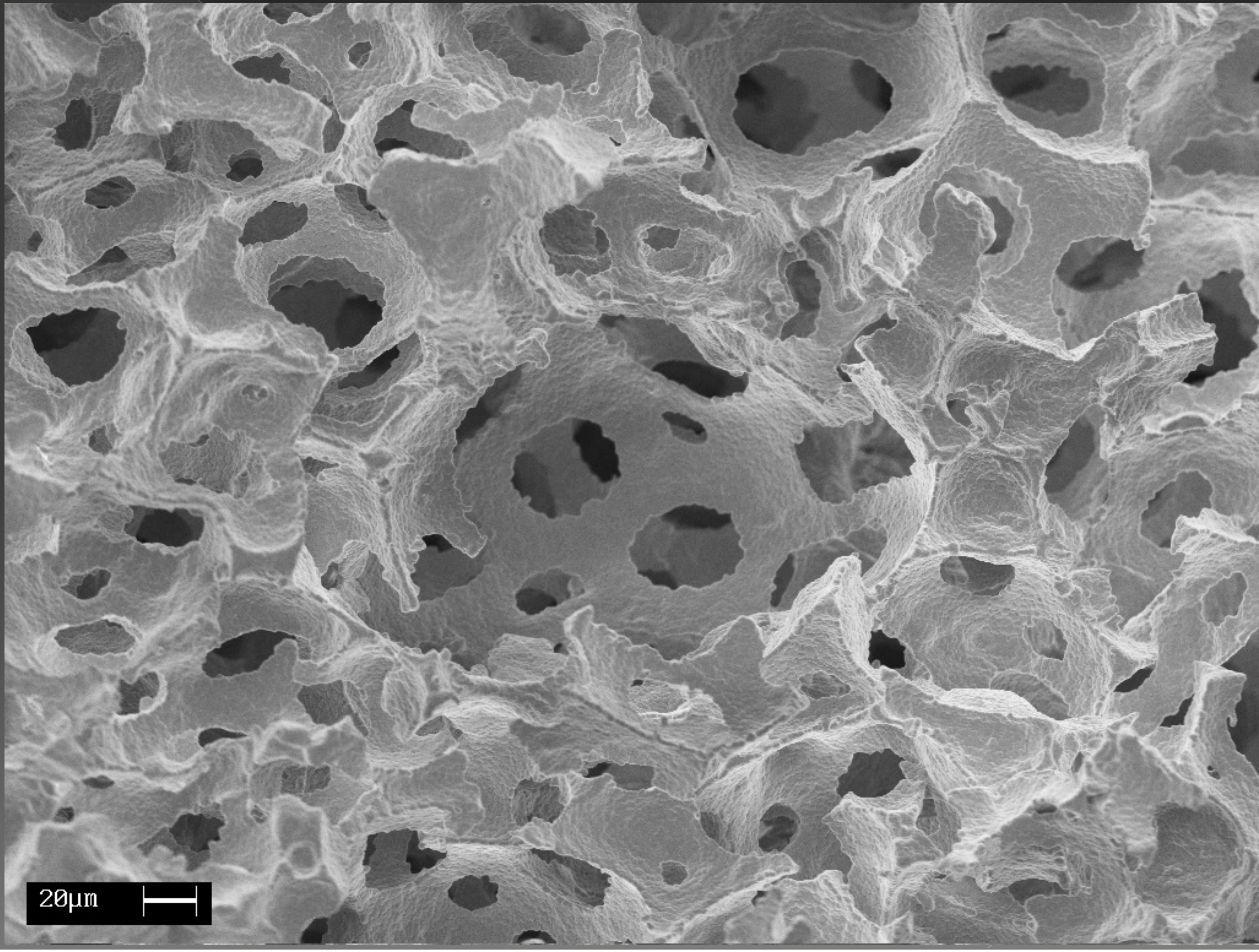
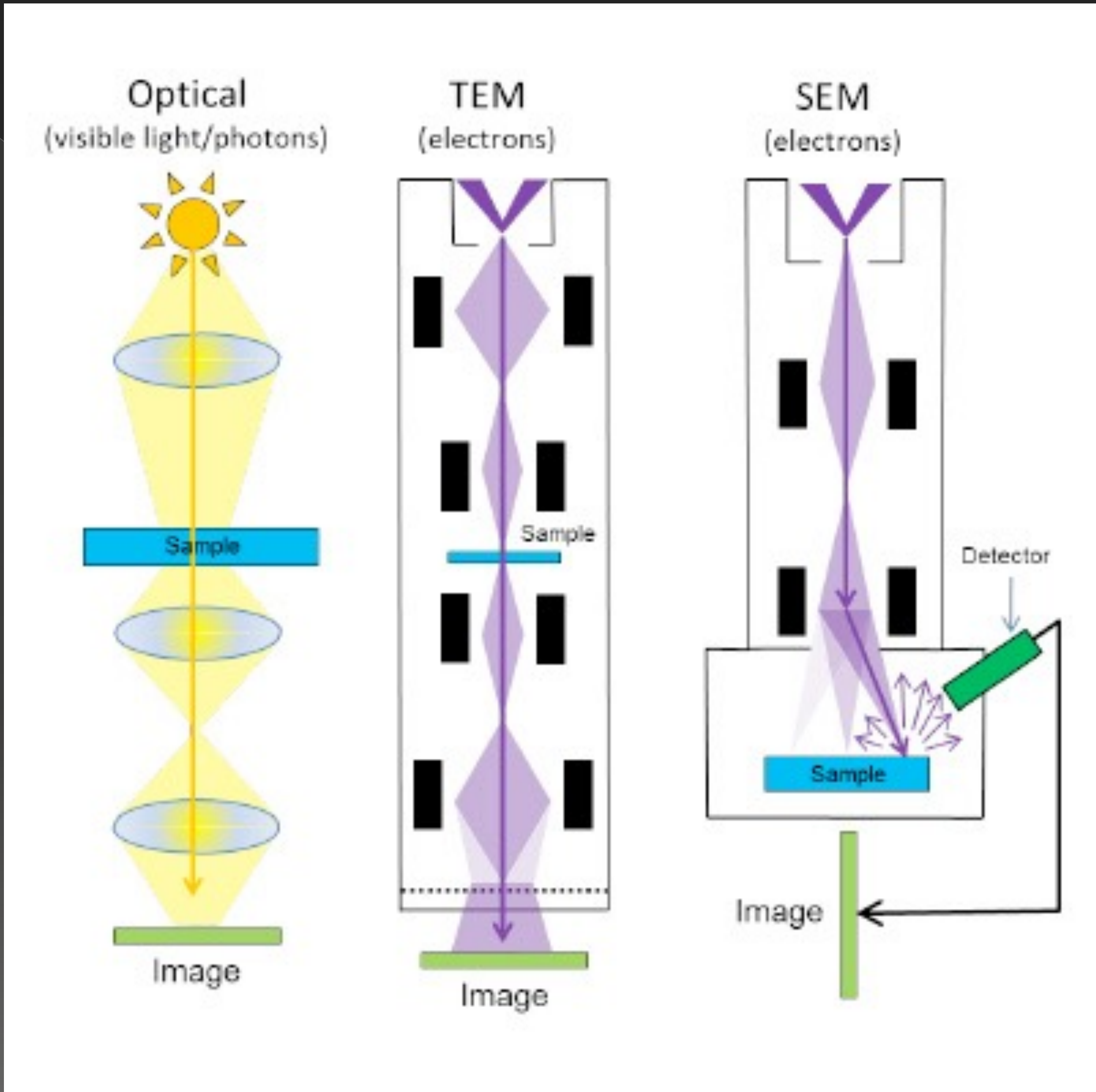
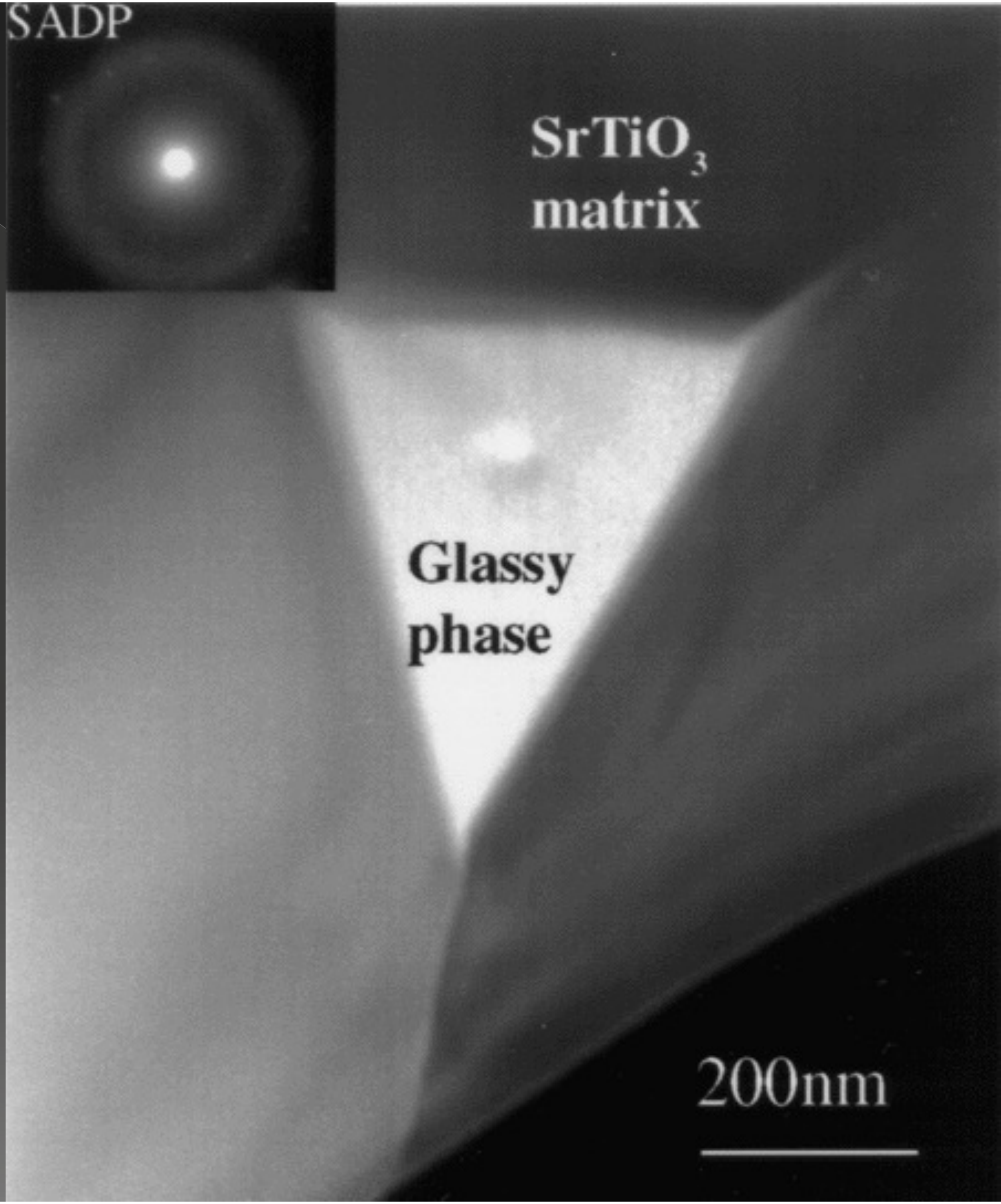


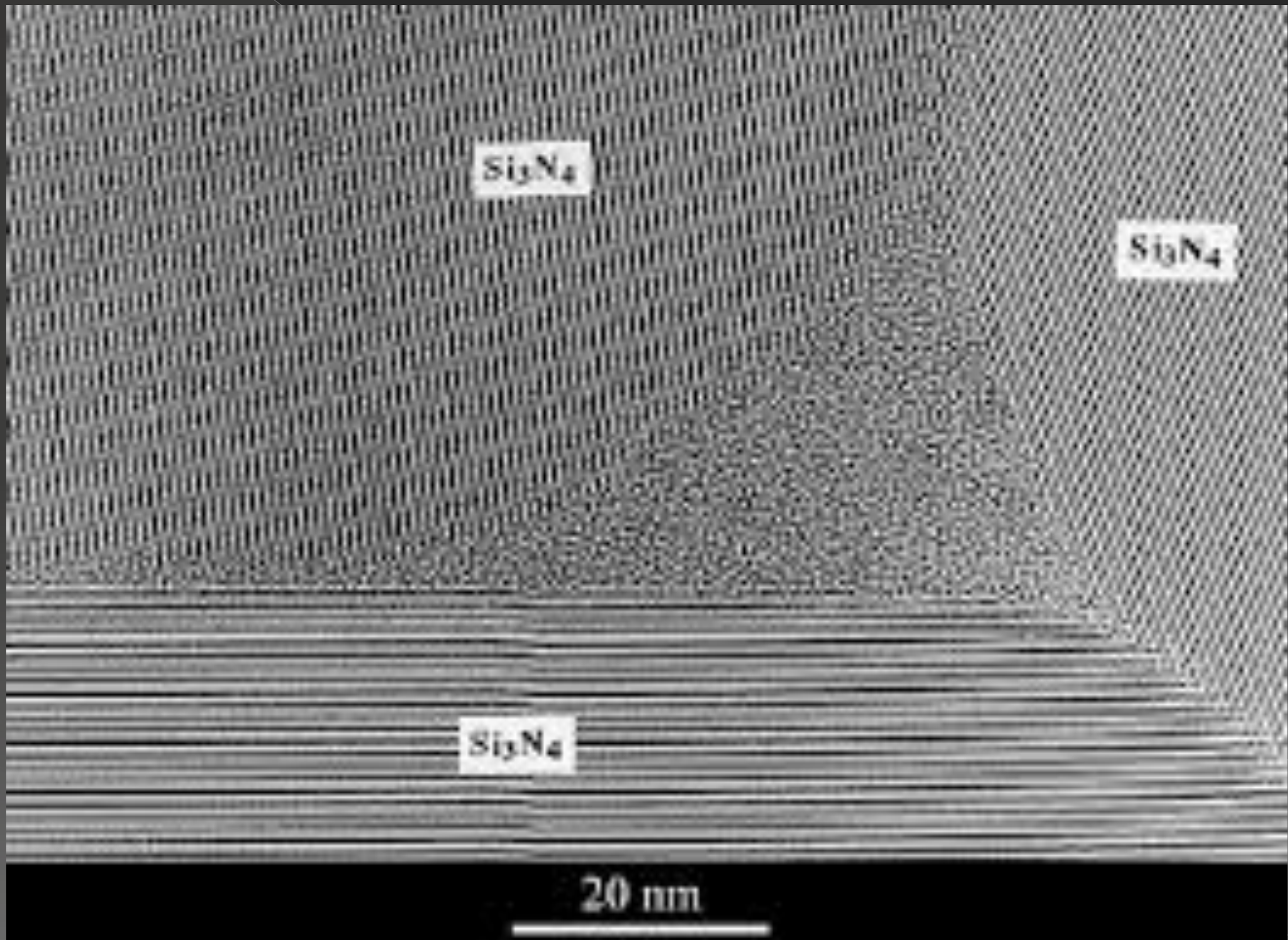
FIGURE 24.27 Two-phase ceramics. (a) As sintered and (b) heat treated at 1600°C for 30 hours. ZTA 30% (zirconia-toughened alumina with 30 vol% YSZ containing 10 molar% yttria).

Topographycal contrast

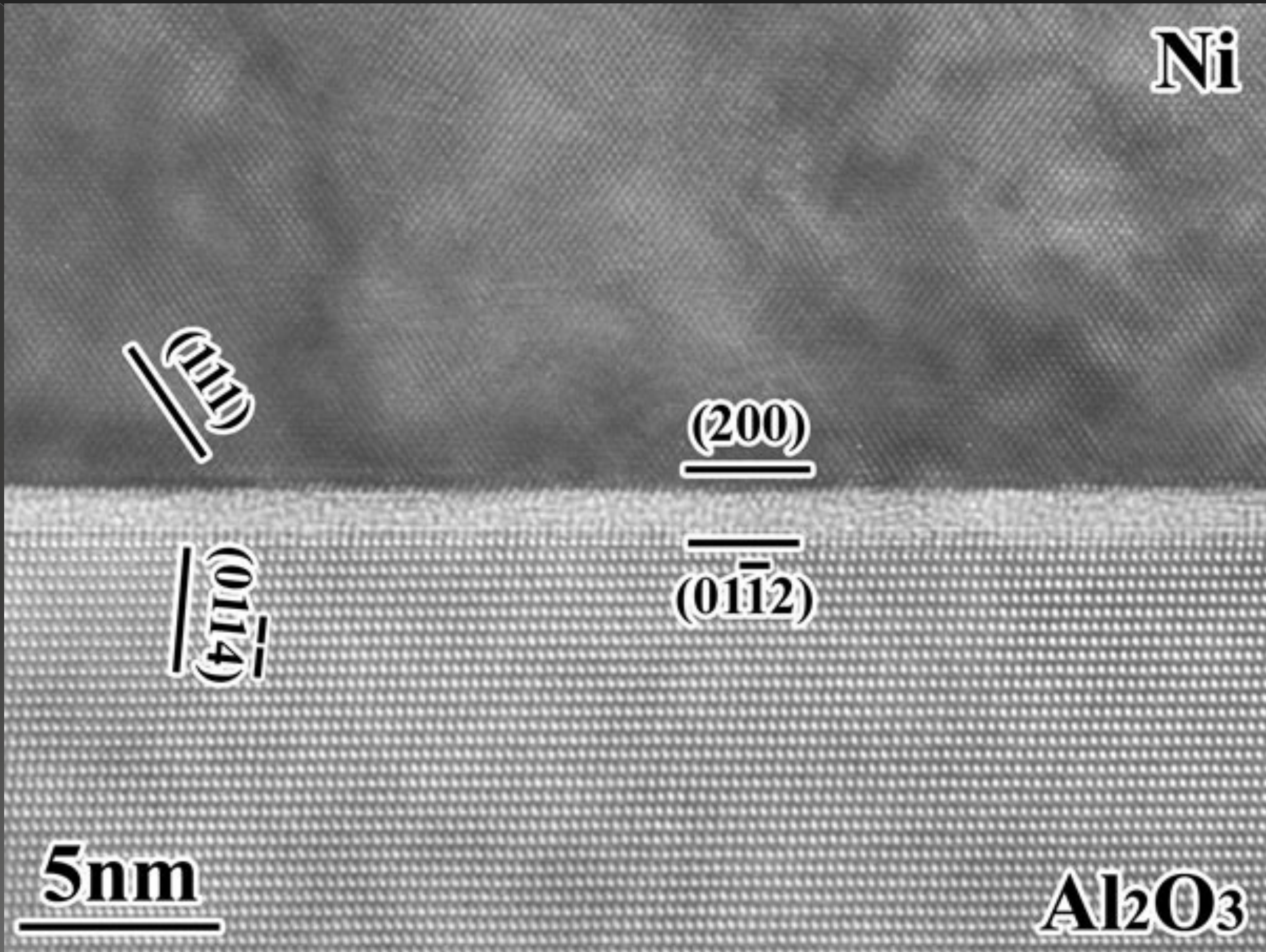


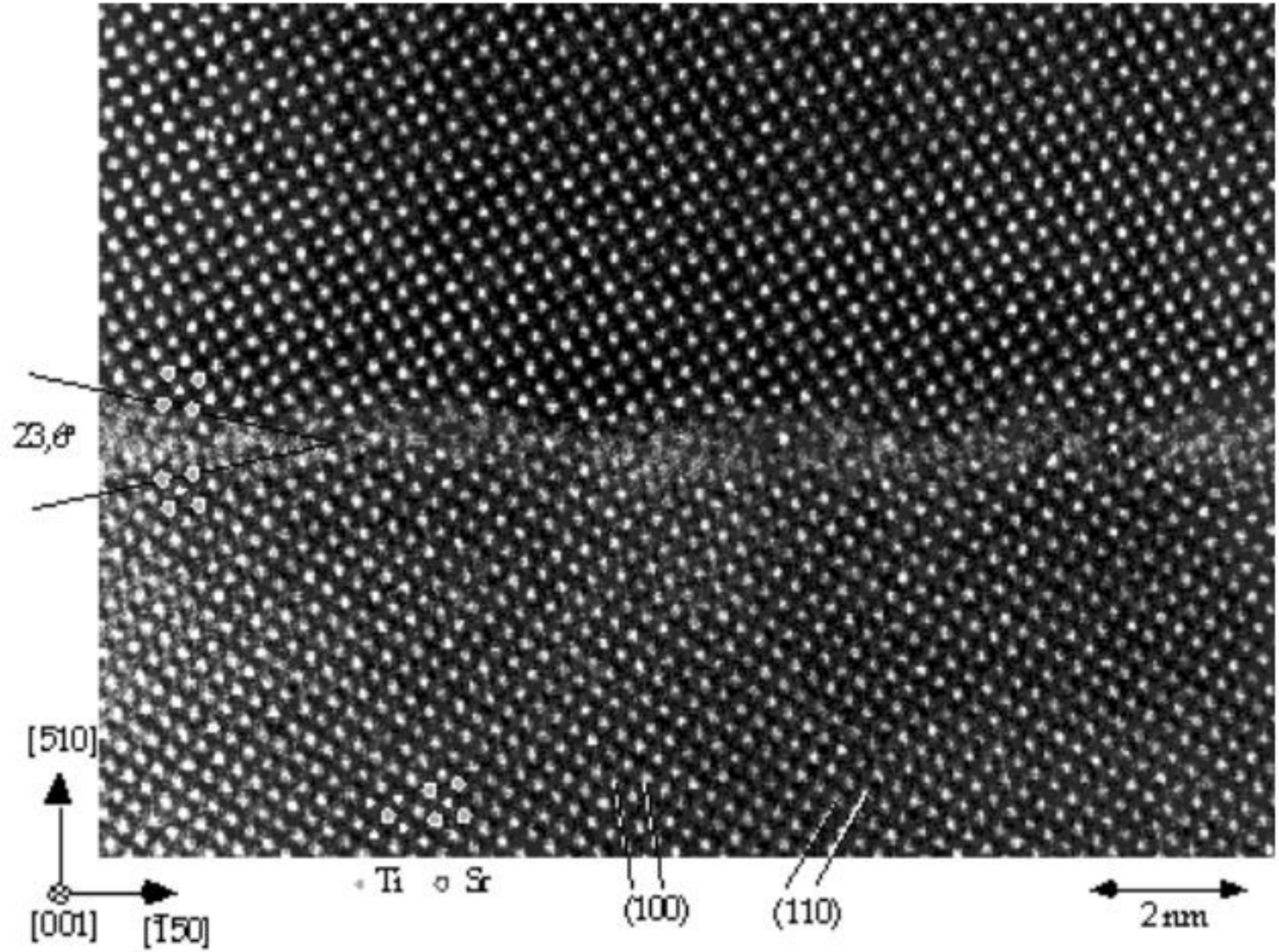

Optical
(visible light/photons)
TEM
(electrons)
SEM
(electrons)
Sample
Sample
Detector
Sample
Image
Image
Image

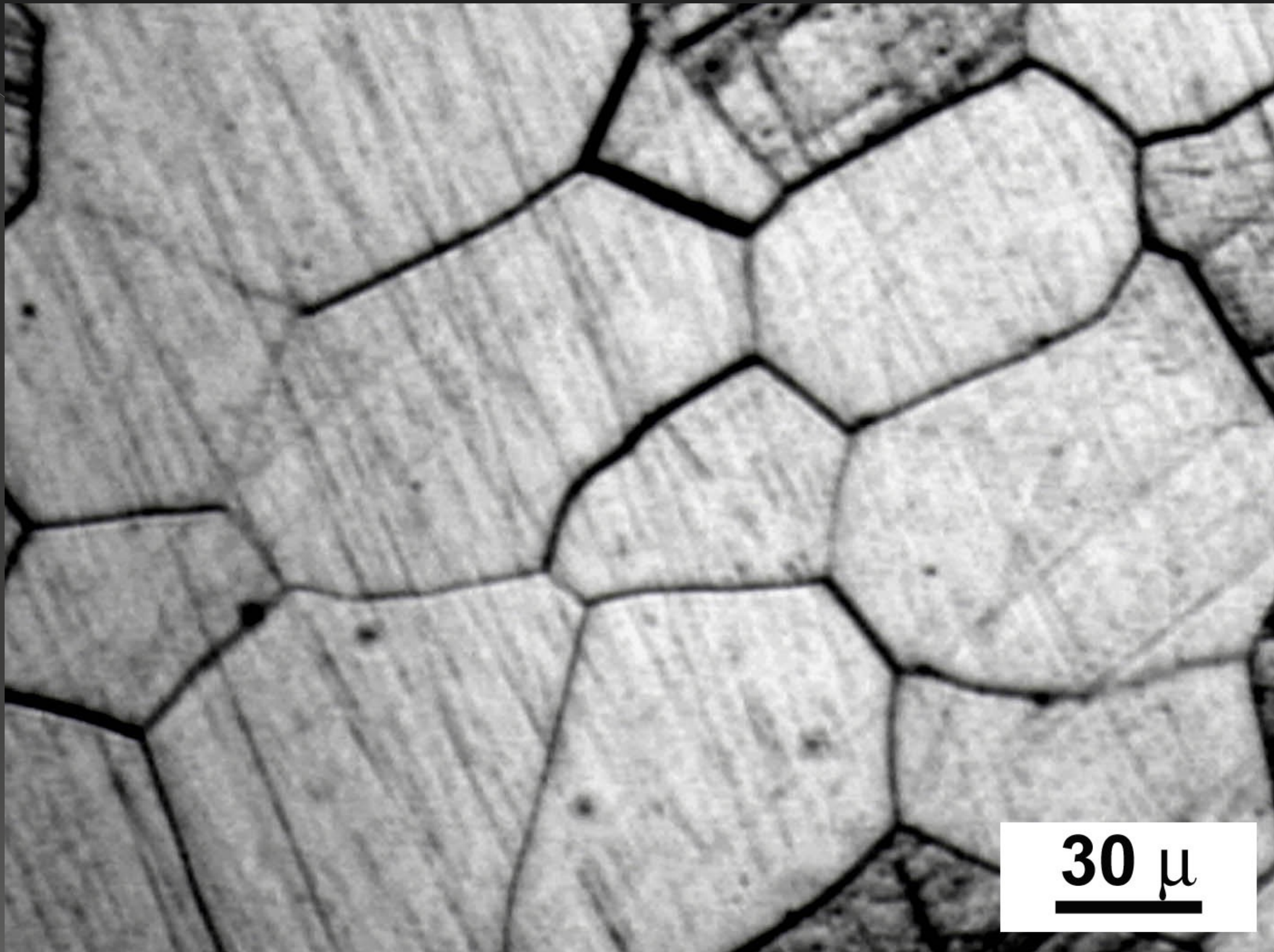

SADP
SrTiO3
matrix
Glassy
phase
200nm



Si_3N_4
Si_3N_4
Si_3N_4
20 nm


Ni
(200)
5nm
Al2O3


23,6°
[510]
[001]
[1̄50]
Ti
Sr
(100)
(110)
2 nm


30 μ