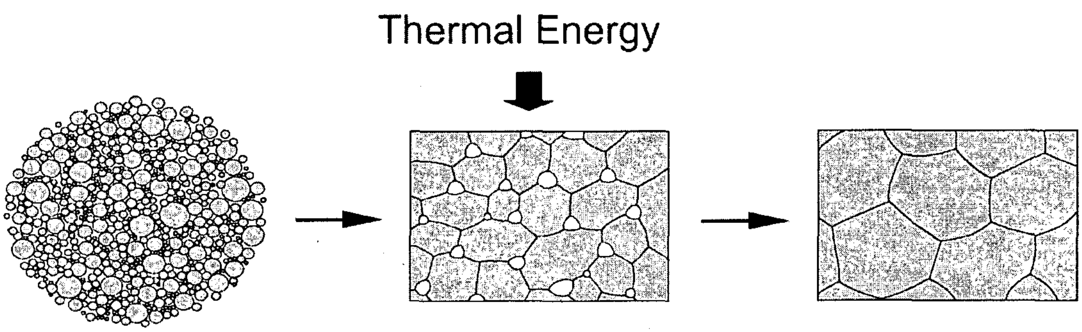
# UNDERSTANDING OF SINTERING PHENOMENA

## - Densification and Grain Growth -

Suk-Joong L. Kang

Materials Interface Laboratory
Korea Advanced Institute of Science and Technology

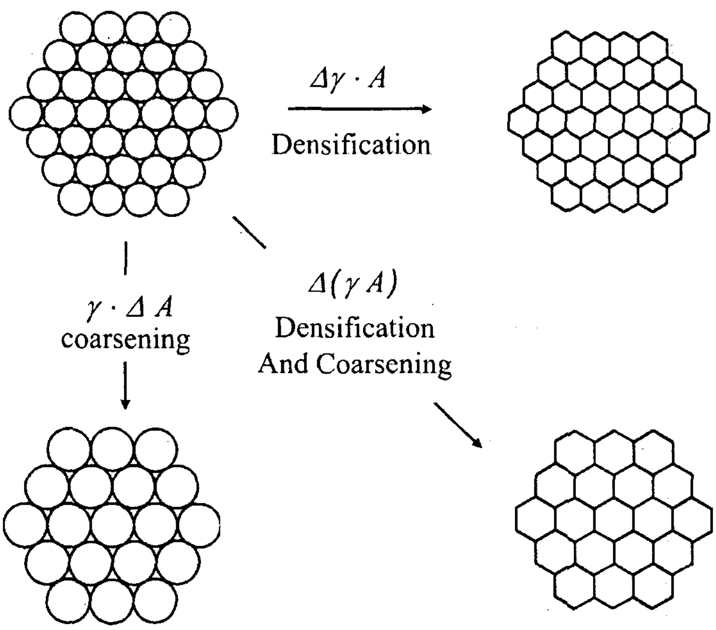
## What is Sintering?




• Densification
• Grain Growth

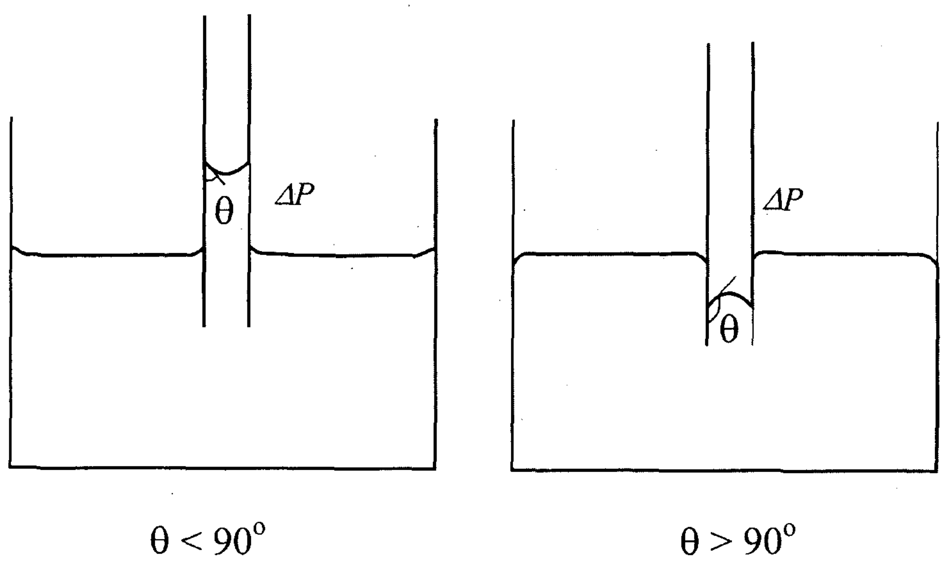
MILab KAIST

## Driving Force for Sintering, $\Delta(\gamma A)$

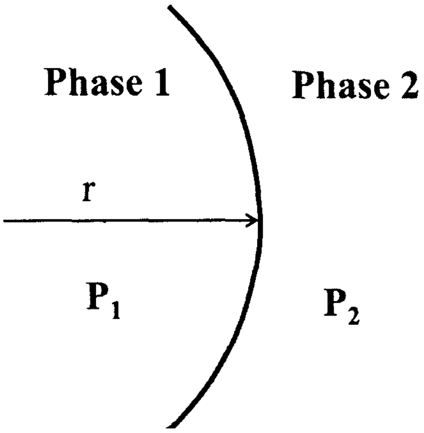



Basic phenomena occurring during sintering under the driving force for sintering, $\Delta(\gamma A)$.

## Capillarity

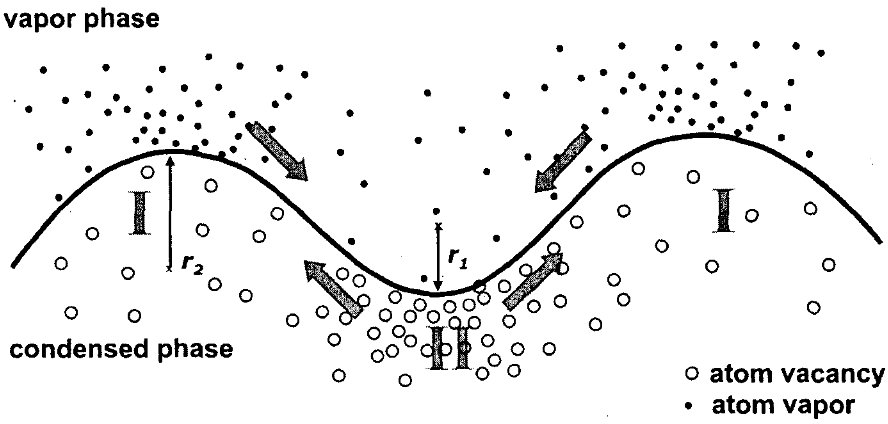


## Young-Laplace Equation




$$P_1 - P_2 = \gamma_{12} K$$
$$= \gamma_{12}\left(\frac{1}{r_1} + \frac{1}{r_2}\right)$$
$$\left(= \gamma_{12}\,\frac{2}{r}\right)$$

## Curved Interface and Material Transport




- Differences in i) Pressure, ii) Vacancy concentration, and iii) Vapor pressure(solubility).

<u>Sintering</u>

Curved interface : Particle geometry

(Interface energy)

⬇

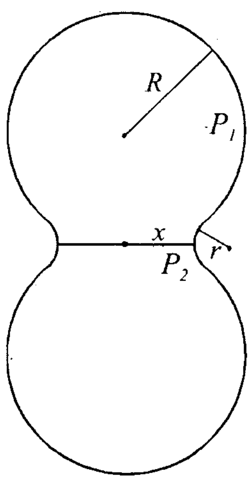
i) Pressure difference, ii) solubility difference, iii) Vapor pressure difference

Parallel Process (Kinetics)

| | | |
|---|---|---|
| Dominant mechanism<br>(Sintering Map) | ⟵ | System,<br>Particle size and geometry<br>temperature |

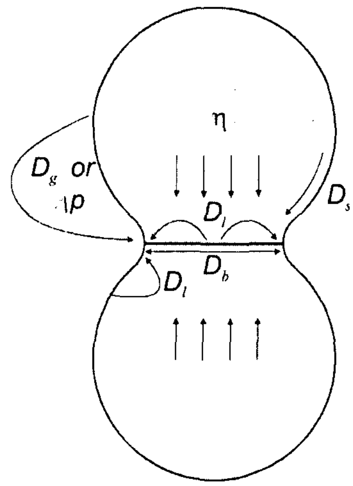
# Solid State Sintering

## 1. Densification



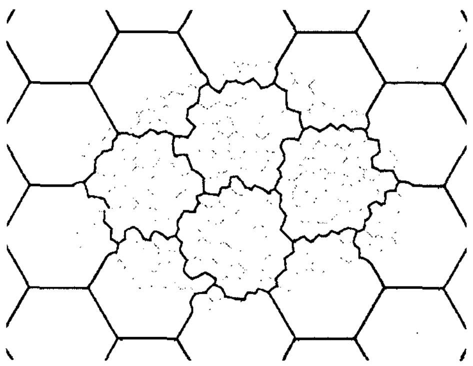

$$P_1 - P_2 = \left[\frac{2}{R} - \left(\frac{1}{x} - \frac{1}{r}\right)\right]\gamma$$

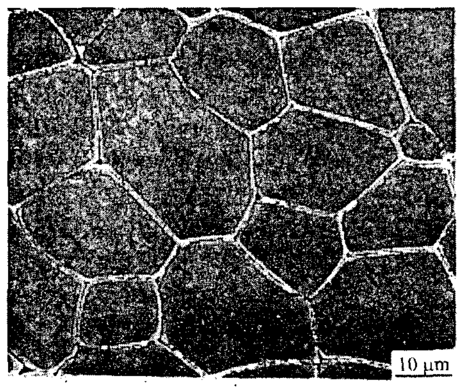
# Sintering Mechanisms



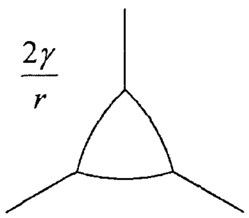

Material transport paths during sintering.

## 2. Grain Growth





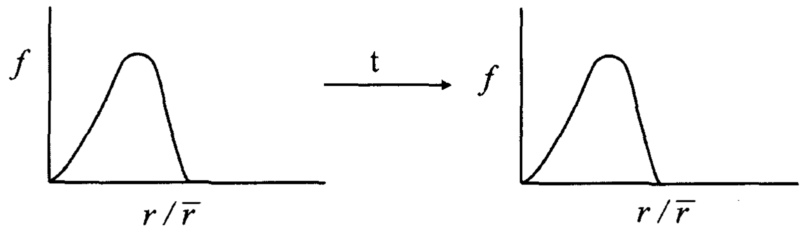

Etched and polished section of $Al_2O_3$ .



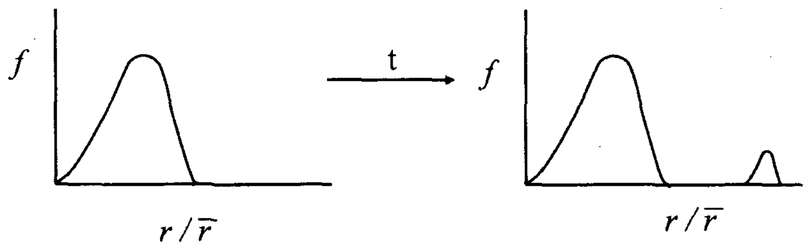

Kinetics : $G^2 \propto kt$

## Normal and Abnormal Grain Growth

Normal




Abnormal

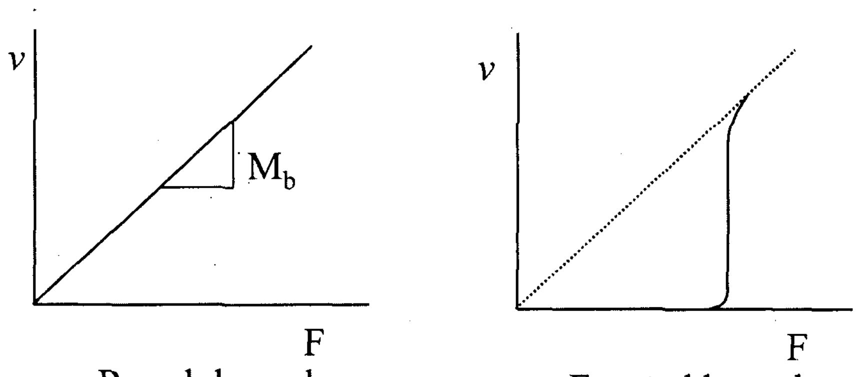



## Grain Boundary Migration

$$v_b = M_b F \propto M_b \left( \frac{\gamma_b}{r} \right)$$

i) $M_b$ = constant

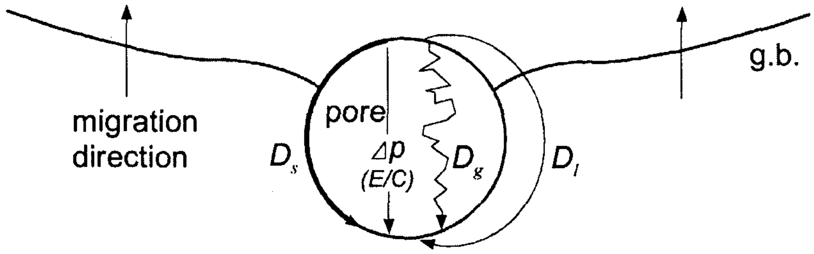
ii) $M_b$ = variable




Rough boundary

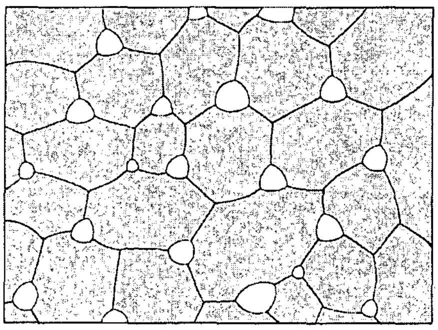
Faceted boundary

## Pore Migration




Zener effect

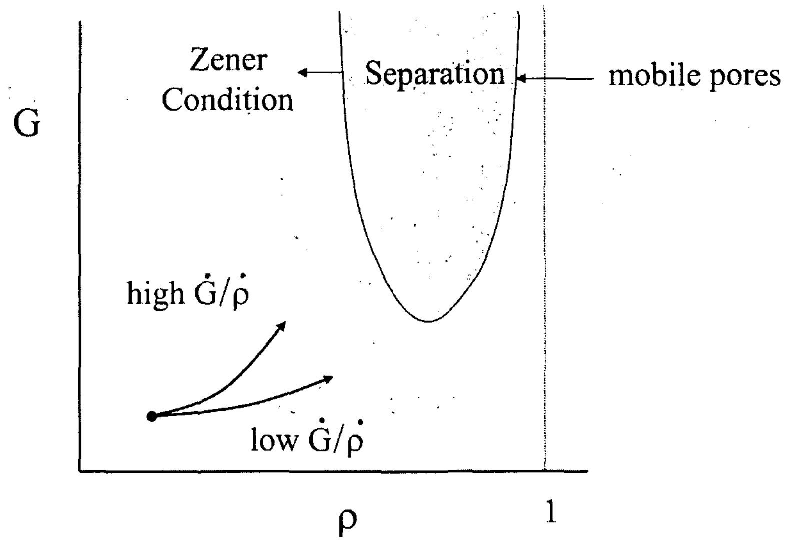
# 3. Densification and Grain Growth



Densification rate : $\frac{d\rho}{dt}$ $\left(\frac{d\rho}{dt}\frac{1}{\rho}\right)$

Grain Growth rate : $\frac{dG}{dt}$ $\left(\frac{dG}{dt}\frac{1}{G}\right)$

# Microstructure Development




## Sintering Parameters : *G, T(dT/dt), P*

### Sintering Equations

$$\frac{1}{\rho}\frac{d\rho}{dt} \propto \frac{V_m \gamma_s \delta_b^m D}{RTG^n}$$

| | n | m |
|---|---|---|
| $D_l$ | 3 | 0 |
| $D_b$ | 4 | 1 |

$$\frac{1}{G}\frac{dG}{dt} \propto \frac{C}{G^n(1-\rho)^m}$$

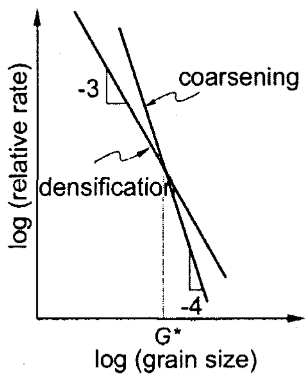
| | n | m |
|---|---|---|
| $D_s$ | 4 | 4/3 |
| $D_b$, $D_l$ | 3 | 1 |
| Evap./Cond. | 2 | 2/3 |
| Solute drag | 3 | 0 |

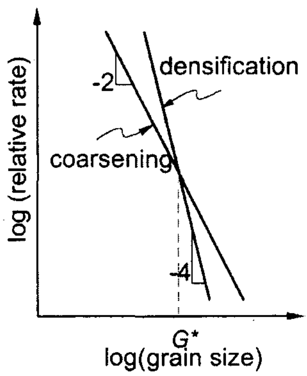
MILab KAIST

# Grain Size

## Examples

| Densification : lattice diffusion<br>Grain Growth : surface diffusion | Densification : grain boundary diffusion<br>Grain Growth : evaporation and condensation |
|---|---|

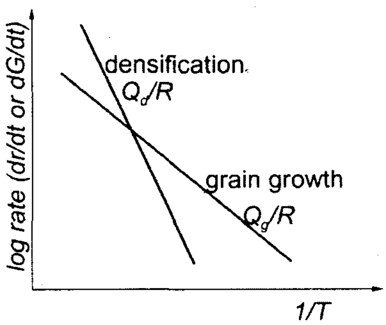


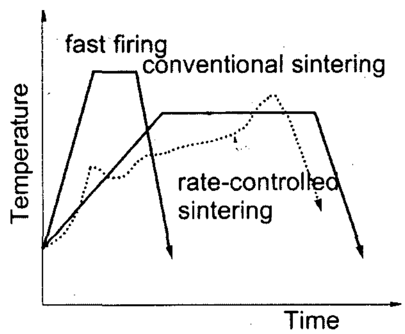

Relative densification and coarsening rates vs. grain size.

# Sintering Temperature ; Heating rate

$$\frac{d\rho}{dt} \propto exp\left(-\frac{Q_d}{RT}\right) \qquad \frac{dG}{dt} \propto \exp\left(-\frac{Q_g}{RT}\right)$$

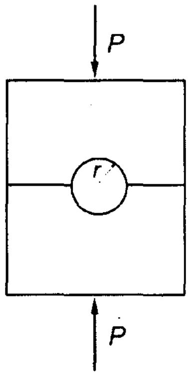



Temperature dependence of densification and grain growth for a material in which $Q_d > Q_g$.




Schematic showing the thermal cycles of conventional sintering, fast firing and rate-controlled sintering.

## Sintering Pressure

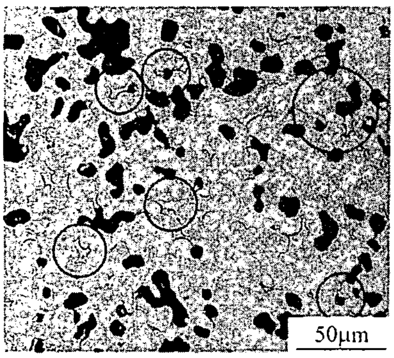



Driving pressure for sintering :

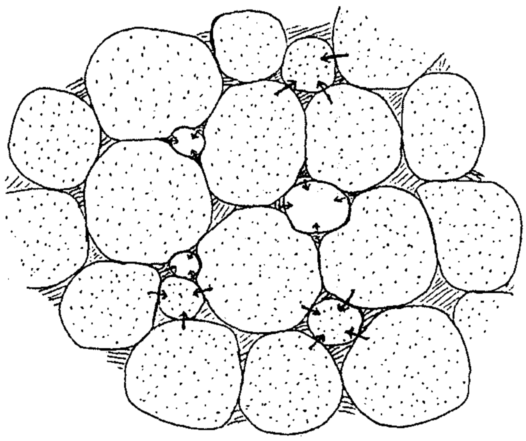
$$\sigma_t = \left(\frac{\gamma}{r}\right) + \sigma_{appl}$$

# Liquid Phase Sintering

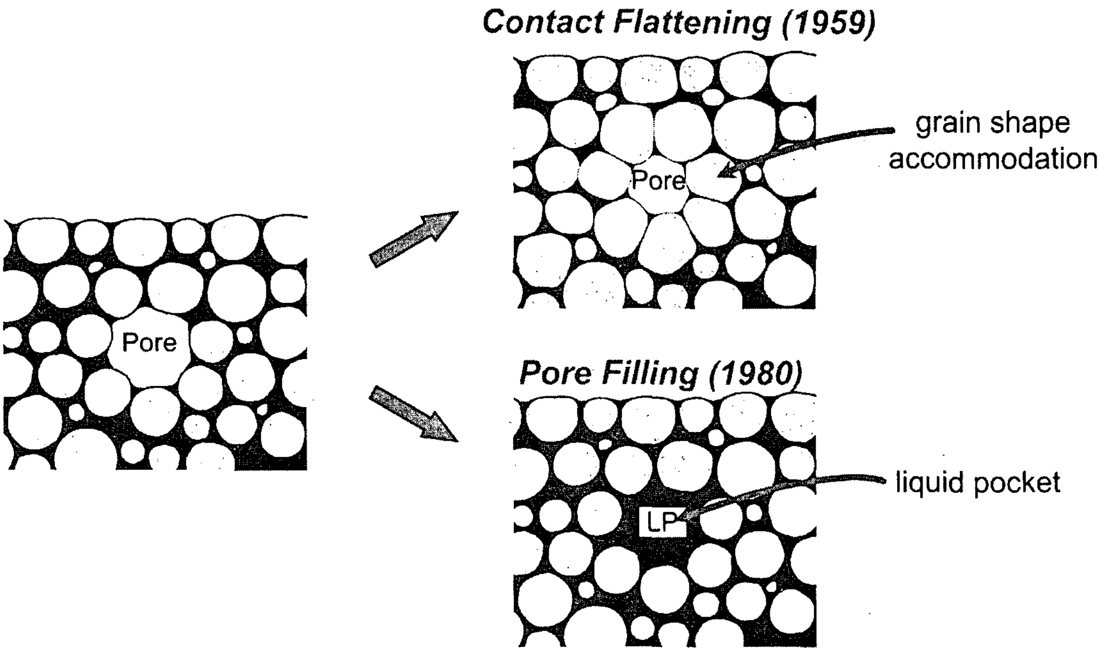
## Microstructure



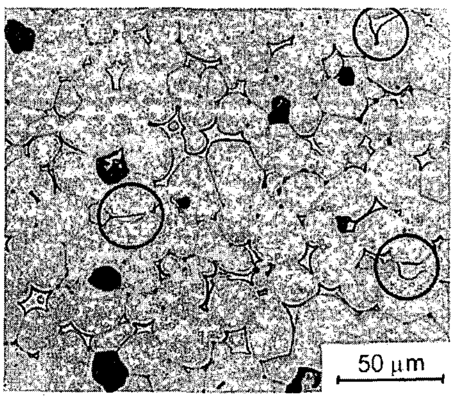

Densification of W(5 μm)-1Ni(4.6 μm)-1Fe(5 μm) by liquid filling of pores during liquid-phase sintering at 1460℃ for 10 min. The circles indicate liquid pockets.



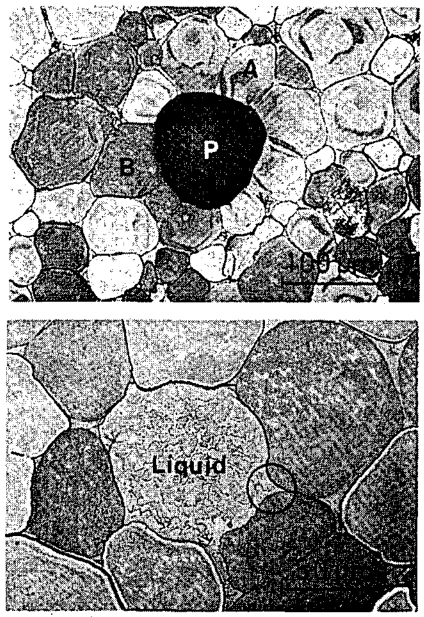
## Densification Mechanisms during LPS




## Pore Filling

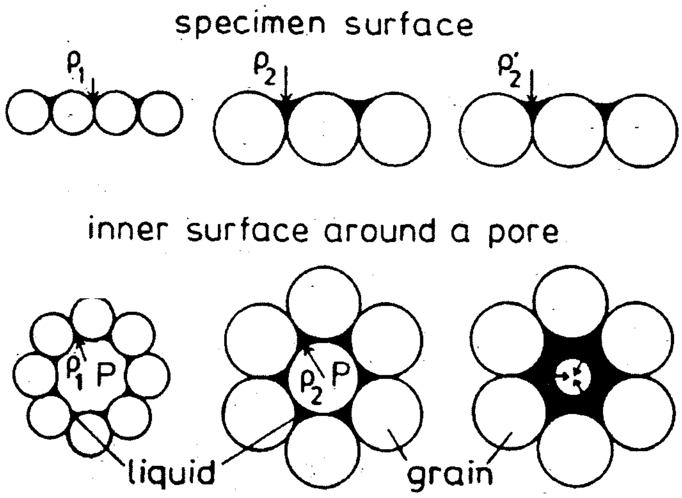



W - Ni - Fe

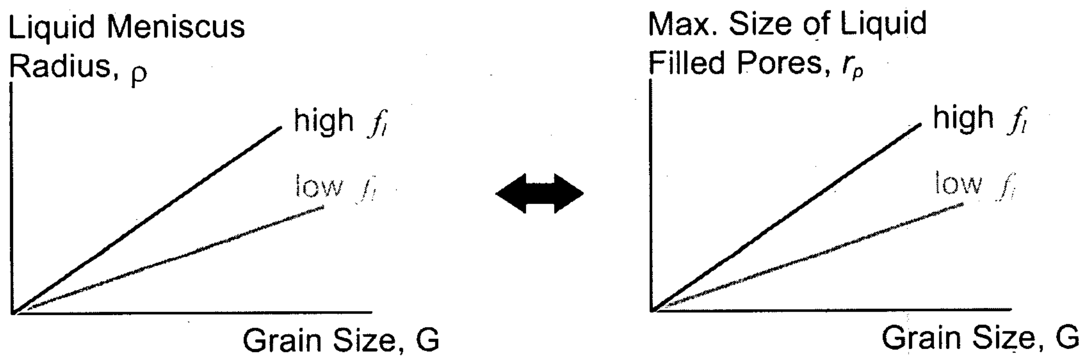



Mo - Ni

# Densification by Grain Growth



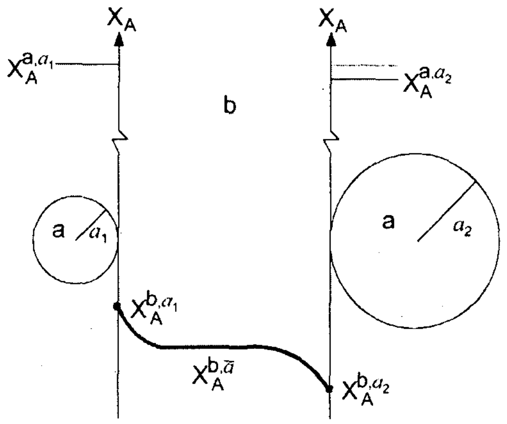

# Grain Growth and Pore Filling




$\rho \propto G$ $\rho \leftrightarrow r_p$ $r_p \propto G$

(H.-H. Park, et al. 1986)

## Grain Growth




Ostwald ripening

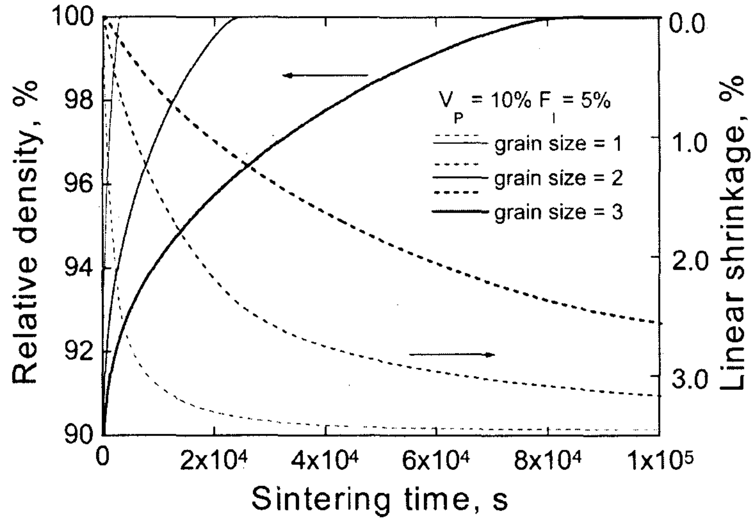
Kinetics : $G^3 \propto kt$

(diffusion control)

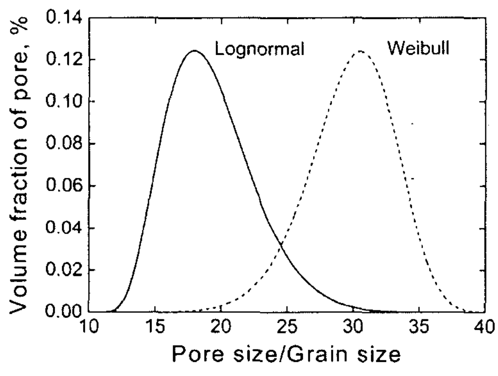
## Sintering Parameters

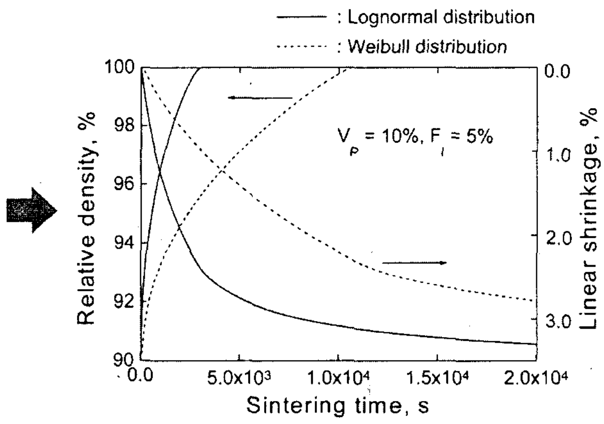
- **Particle Size of Raw Materials**
  - Solid particles ⟶ Initial grain size
  - Liquid forming particles ⟶ Pore size and size distribution, Porosity
- **Sintering Temperature**
  - Liquid volume fraction ⟶ Densification, Grain growth

## Initial Grain Size



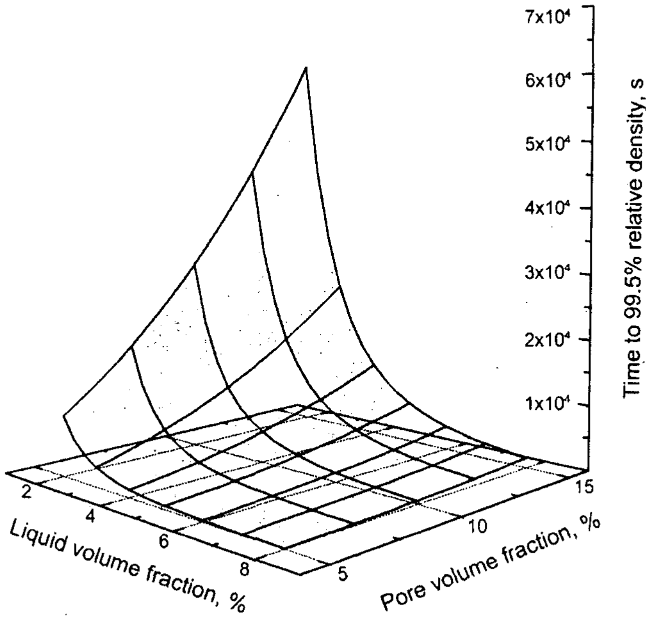

## Pore Size and Size Distribution




Particles with low m.p. ↑ ⟶ Densification ↓

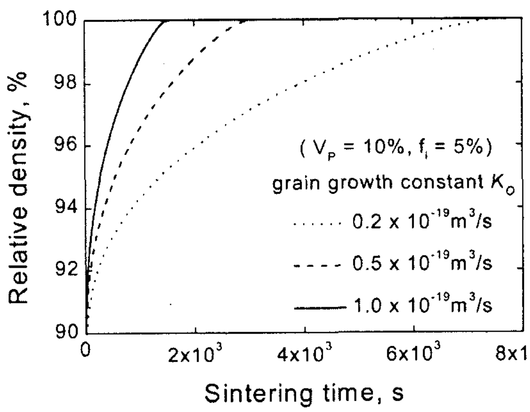
# Liquid Volume Fraction, Porosity

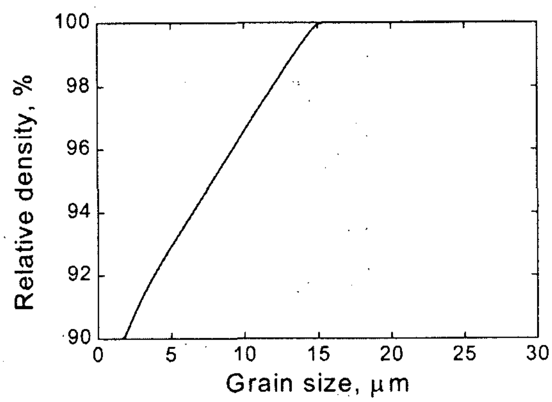



$$t \propto (f_l)^{-2.9}$$

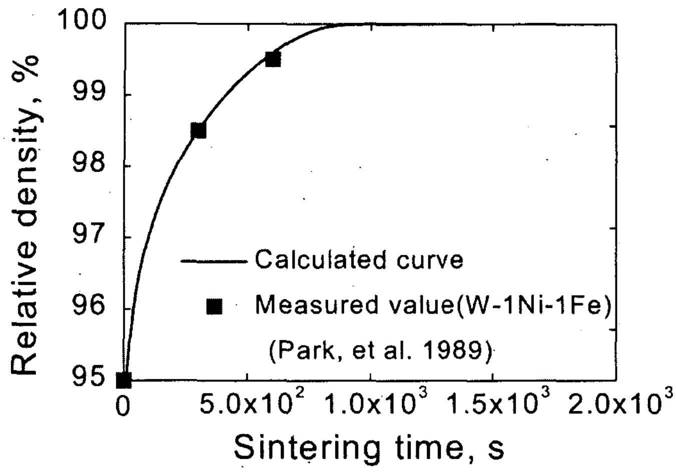
$$t \propto (V_p^i)^{2.2}$$

$$G^n - G_o^n = Kt\ , \qquad K = K_o\left(\frac{0.05}{f_l^{eff}}\right)^{0.8}$$



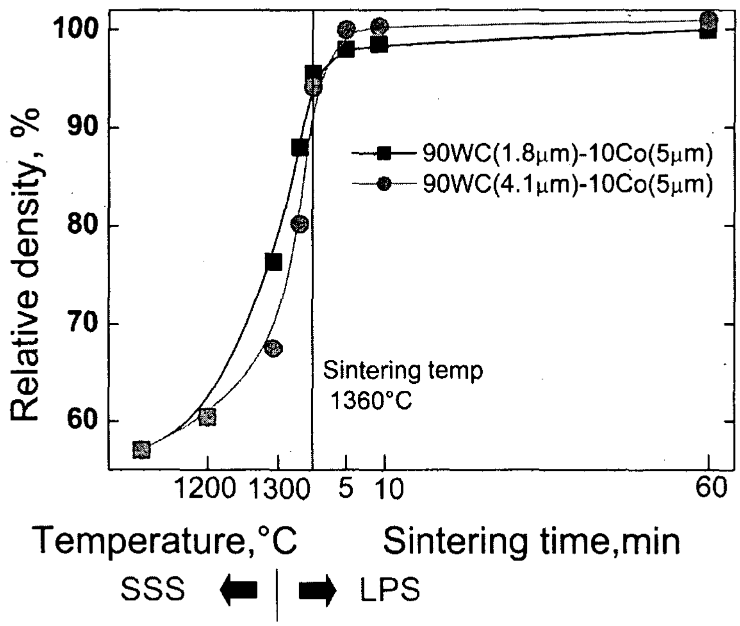




$$t \propto \frac{1}{K}$$




Comparison of measured densification with calculated one

# Densification of WC-Co during SSS and LPS



# Conclusions

- Solid-State Sintering

  - Densification $\xrightarrow{\text{pore}}$ Grain Growth
  - Grain Growth $\xrightarrow{\text{grain size}}$ Densification

- Liquid Phase Sintering

  - Densification $\xrightarrow{\text{liquid fraction}}$ Grain Growth
  - Grain Growth $\xrightarrow{\text{pore filling}}$ Densification

---

Understanding of Sintering : Densification, Grain Growth, And Their Interrelationship

MILab KAIST