

# Ceramics Materials Manufacturing

Dr. Alaa Aladdin  
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# SINTERING

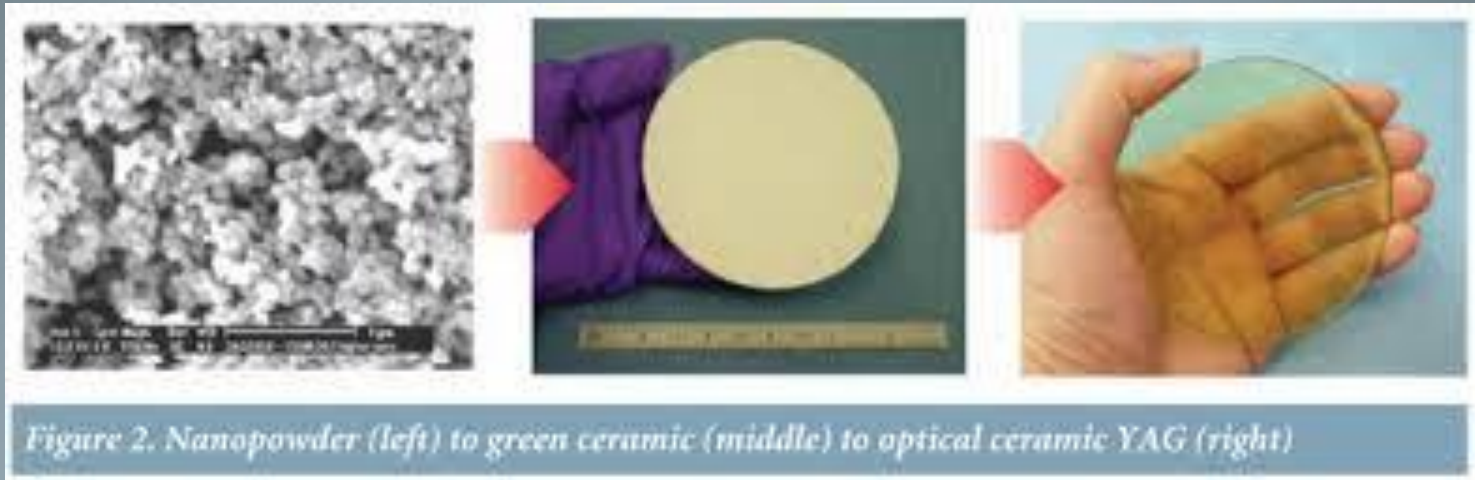
# Introduction

After binder burnout, we have a ceramic compact that consists of an aggregation of ceramic particles giving a porous ceramic green body.

At this stage the ceramic green body is at its most fragile state and must be handled with care or, better yet, simply not handled at all.

The next step, sintering, are done either:

- (1) In the same furnace but at different temperatures and different atmospheres.
- (2) In the same kiln in different sections.
- (3) Other furnace



How to change the optical properties of nano-powders after sintering



## PROPERTY CHANGES

Figure 1 show the sintered density obtained after 4 hr at each temperature for two log-normal sized distribution  $Al_2O_3$  powders.

In this figure, the finer powder, which has the lower density initially, sinters to a higher density at a lower temperature because it has a higher specific surface area.



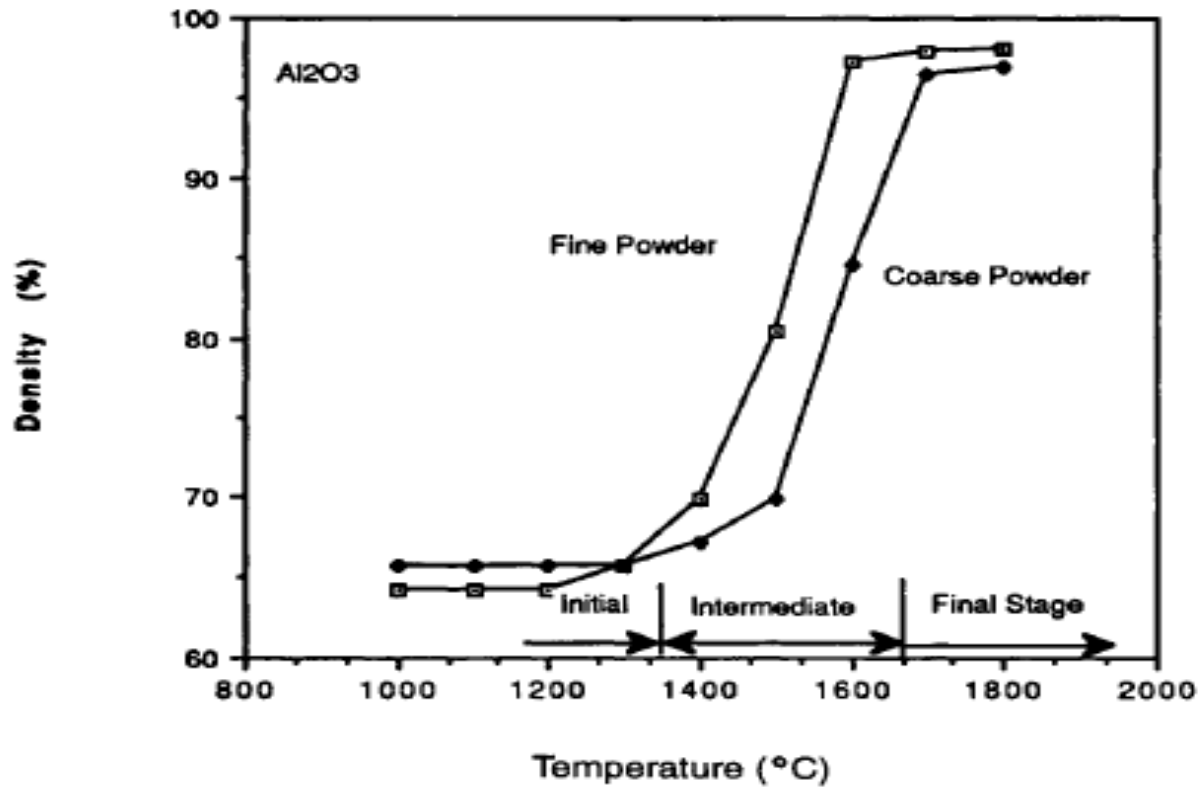
Initial



Intermediate



Final



{FIGURE 1 shows two log-normal alumina powders ( $d = 1.3$  and  $0.8 \mu\text{m}$ ) showing the initial, intermediate, and final stages of sintering. }

Sometimes the density does not increase even though the pores change shape.

This is called coarsening. The difference between sintering and coarsening is schematically shown in Figure 2.

If the inter-particle separation distance remains the same , coarsening takes place.

If the inter-particle separation distance decreases, sintering takes place. Only sintering leads to an increase in density, which is referred to as densification.

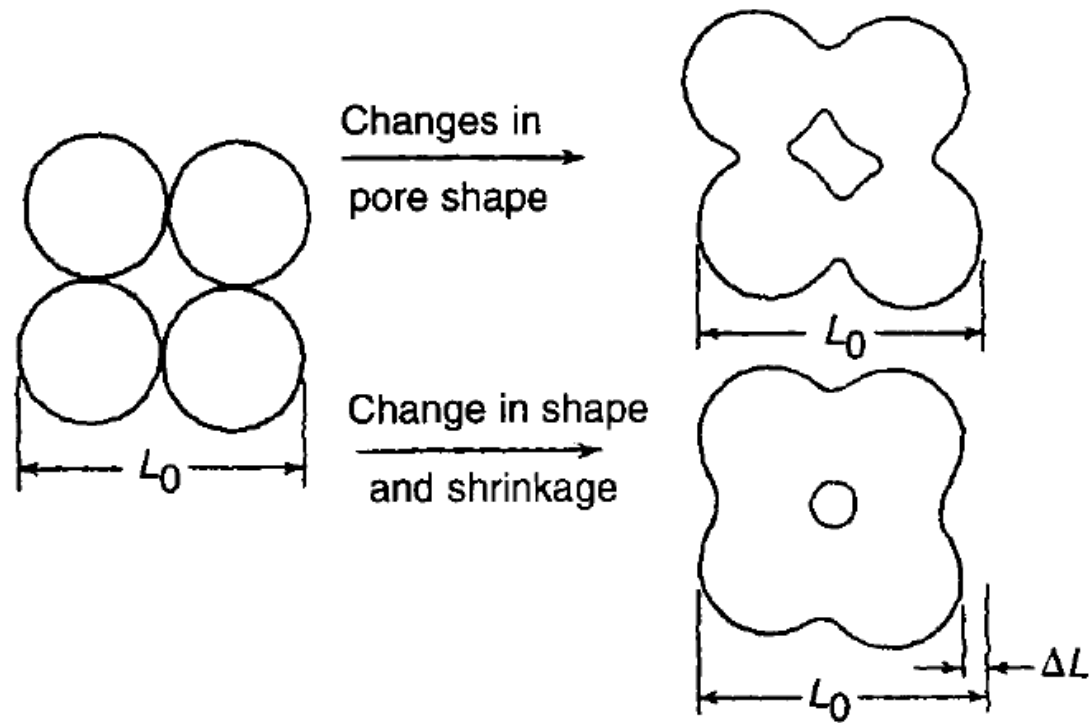
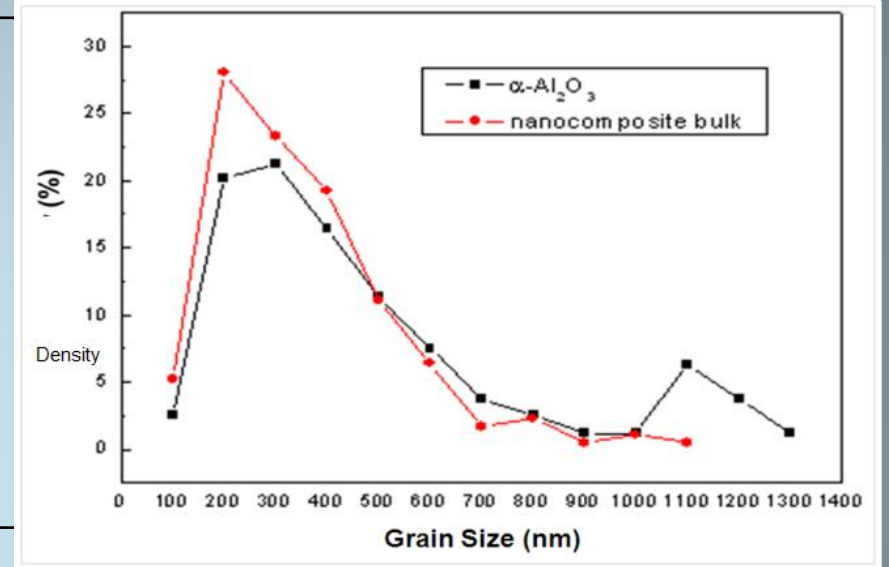
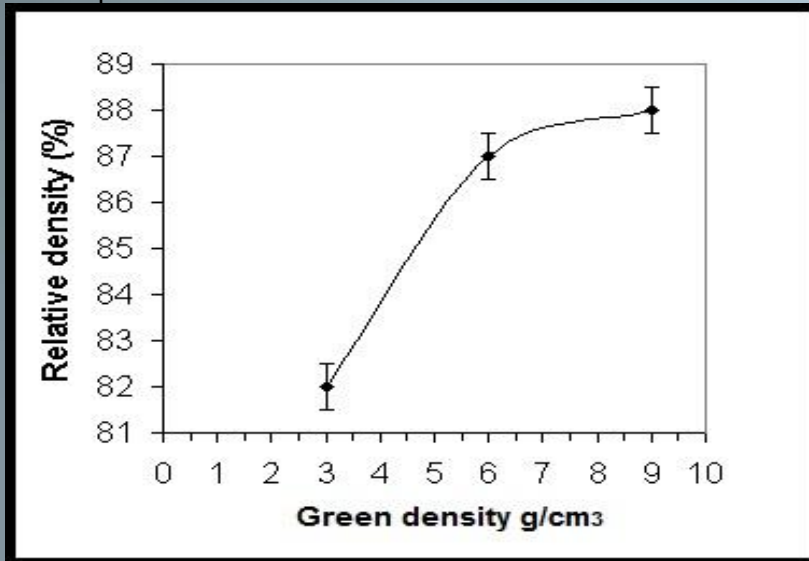


FIGURE .2 The differences between coarsening and sintering



## *Effect of Green Density*

## *Effect of Particle Size Distribution*



## MECHANISM OF SINTERING

The various stages of sintering are described next

Initial Stage:

The initial stage of sintering is frequently referred to as the neck formation stage, as is shown in Figure 1.

The sintering driving force for the initial powder compact is due to the curvature difference between particle surface and that of the neck, see Figure 5.

# MECHANISM OF SINTERING

Initial Stage:

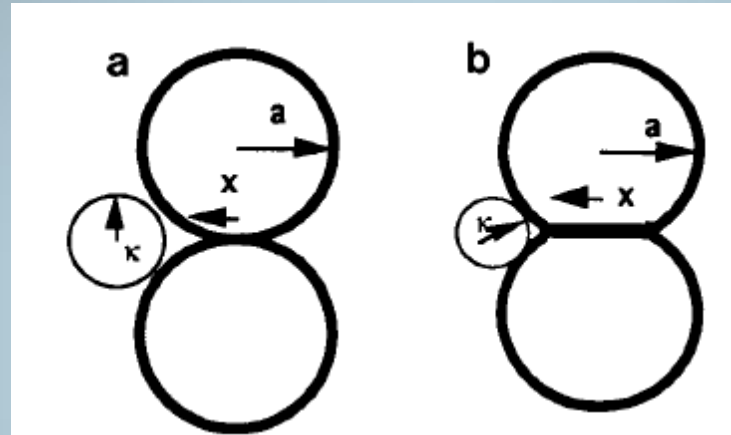


FIGURE 5 shows the contact area between two partially sintered spheres: (a) center-to-center distance is constant, (b) decreasing center-to-center distance.

## MECHANISM OF SINTERING

### Initial Stage:

The six distinguishable diffusion-controlled paths of material transport (**vacancies diffuse normally**), considered to be the most probable sintering mechanisms, are shown in Figure .3. Any one of these pathways increases the neck size, but not all time.

To clearly see this distinction, transport mechanisms :

- (1) vapor transport
- (2) bulk diffusion

# MECHANISM OF SINTERING

## Initial Stage:

- Particle surface smoothing and rounding of pores
- Grain boundaries form
- Neck formation and growth
- Homogenization of segregated material by diffusion
- Open pores
- Small porosity decreases  $<12\%$

## MECHANISM OF SINTERING

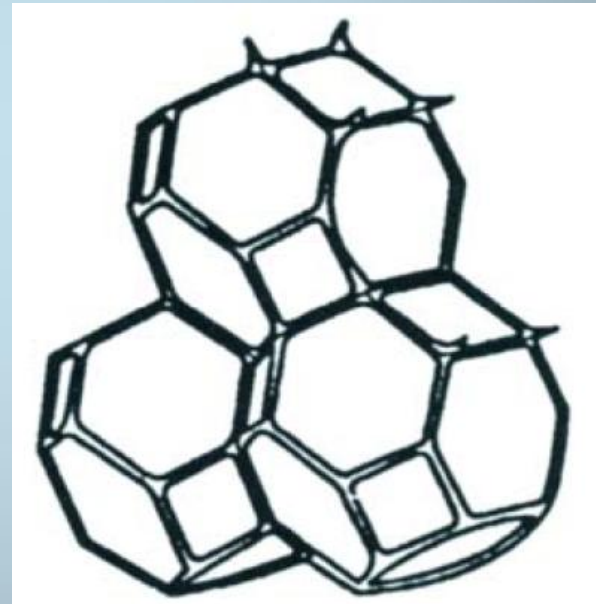
### **Intermediate Stage:**

The intermediate stage of sintering begins after grain and pore shape changes caused during the initial stage produce a pore and grain-boundary matrix consisting of equilibrium dihedral angles formed on the solid-vapor (pore) surface at the intersections with the solid-solid (grain-boundary) interfaces. At this stage, the pore shape approximates a continuous cylindrical channel coincident with three grain edges throughout the matrix as shown in Figure .9. During this intermediate stage of sintering, the cylindrical pore simply shrinks.

## MECHANISM OF SINTERING

### Intermediate Stage:

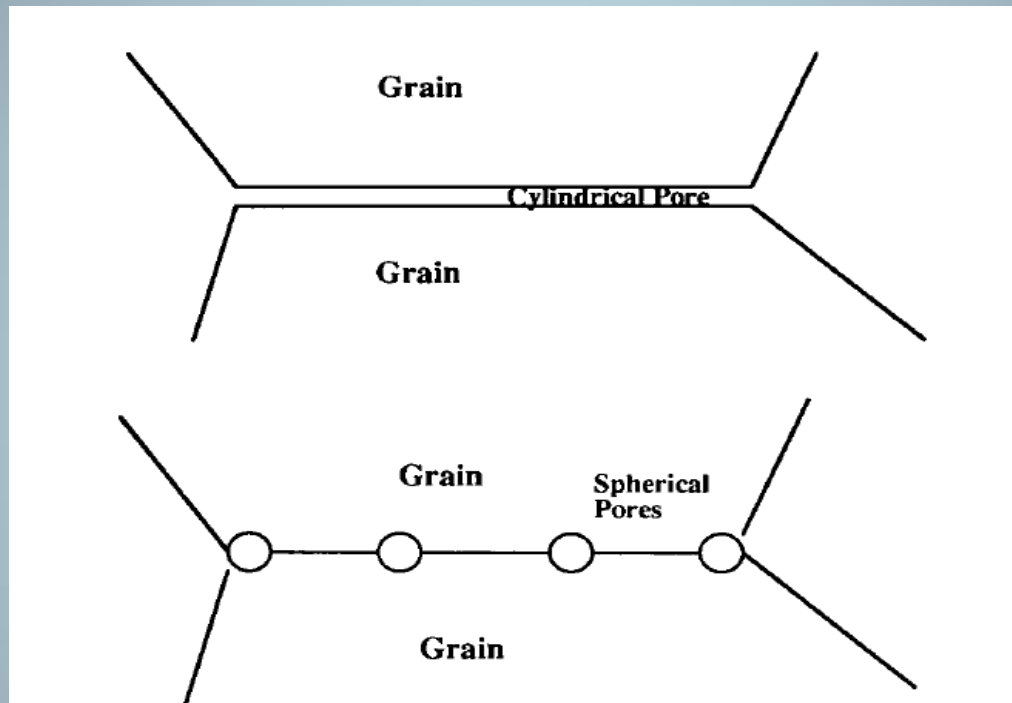
Assuming all grains have the same size and shape (cube, dodecahedron, or tetrakaidecahedron) and all pores are cylindrical and of the same size, the porosity function.



*FIGURE .9* The microstructure model used for intermediate stage sintering. This model consists of tetrakaidecahedra

# MECHANISM OF SINTERING

## Intermediate Stage:



*FIGURE 16.10* Breakup of a cylindrical pore into a string of spherical pores.



## MECHANISM OF SINTERING

### **Intermediate Stage:**

- Intersection of grain boundaries
- Shrinkage of open pores
- Porosity decreases substantially
- Slow grain growth
- Differential pore shrinkage and grain growth in heterogeneous material

## MECHANISM OF SINTERING

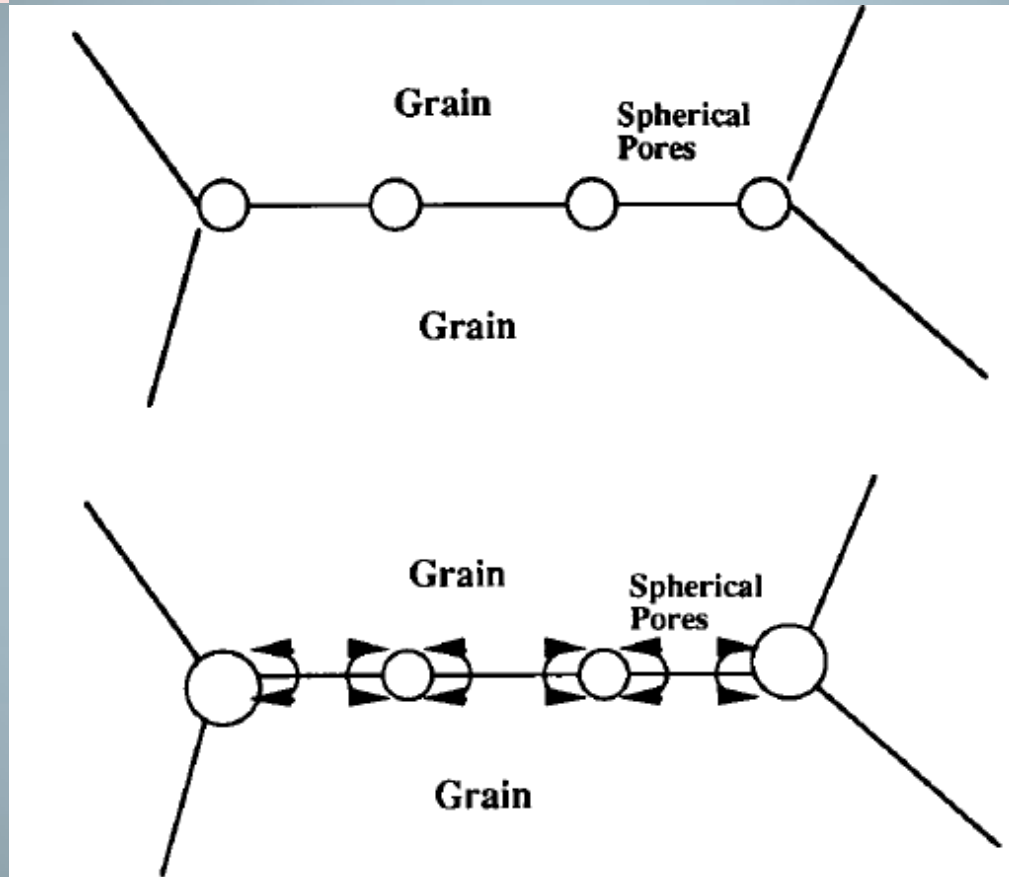
### **Final Stage:**

During the final stage the removal of these closed pores takes place. The final stage densification is dependent on the association of pores with grain boundaries and the rate of grain growth. The string of pores at the grain boundary will migrate to the point of lowest energy.

The energetically favored pore location is at the intersection of three grains in two dimensions, four grains in three dimensions. As shown in Figure 12, the movement of the string of pores to a single pore at the four grain intersection is a complicated process. In some cases it occurs but in others it is prevented from occurring by the movement of grain boundaries caused by discontinuous (or abnormal) grain growth.

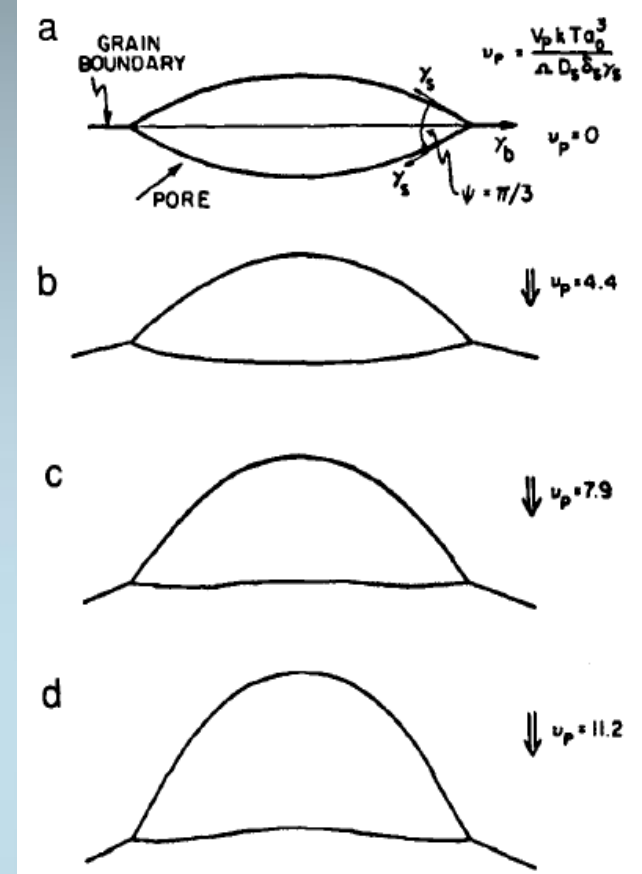
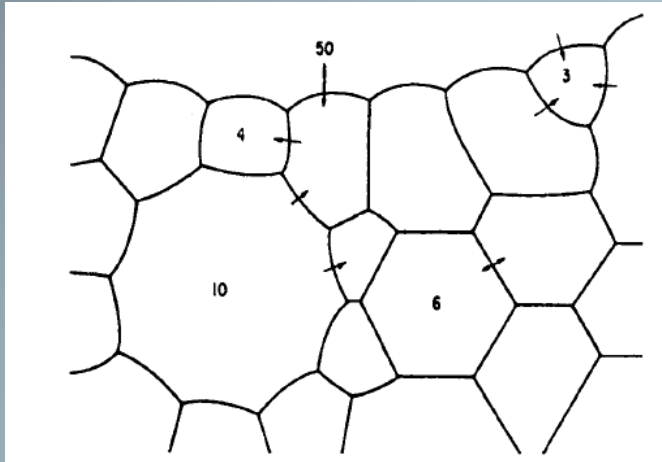
# MECHANISM OF SINTERING

Final Stage



*FIGURE .12* The mean diffusion distance for material transport is smaller when there are more of the same size of pores on the grain boundary

## Final Stage



- Closed pores--density >92%
- Closed pores intersect grain boundaries
- Pores shrink to a limiting size or disappear
- Pores larger than the grains shrink very slowly

## TYPES OF SINTERING PROCESSES

### **Solid state sintering,**

After composed of ceramic particles in contact as shown in Figure .2. As the temperature is increased, material flows come from various sources within the ceramic green body to the neck at the intersection between particles, as shown in Figure 3 .

This neck has a negative curvature, compared to the positive curvature of the spherical ceramic particle.

## TYPES OF SINTERING PROCESSES

### Solid state sintering,

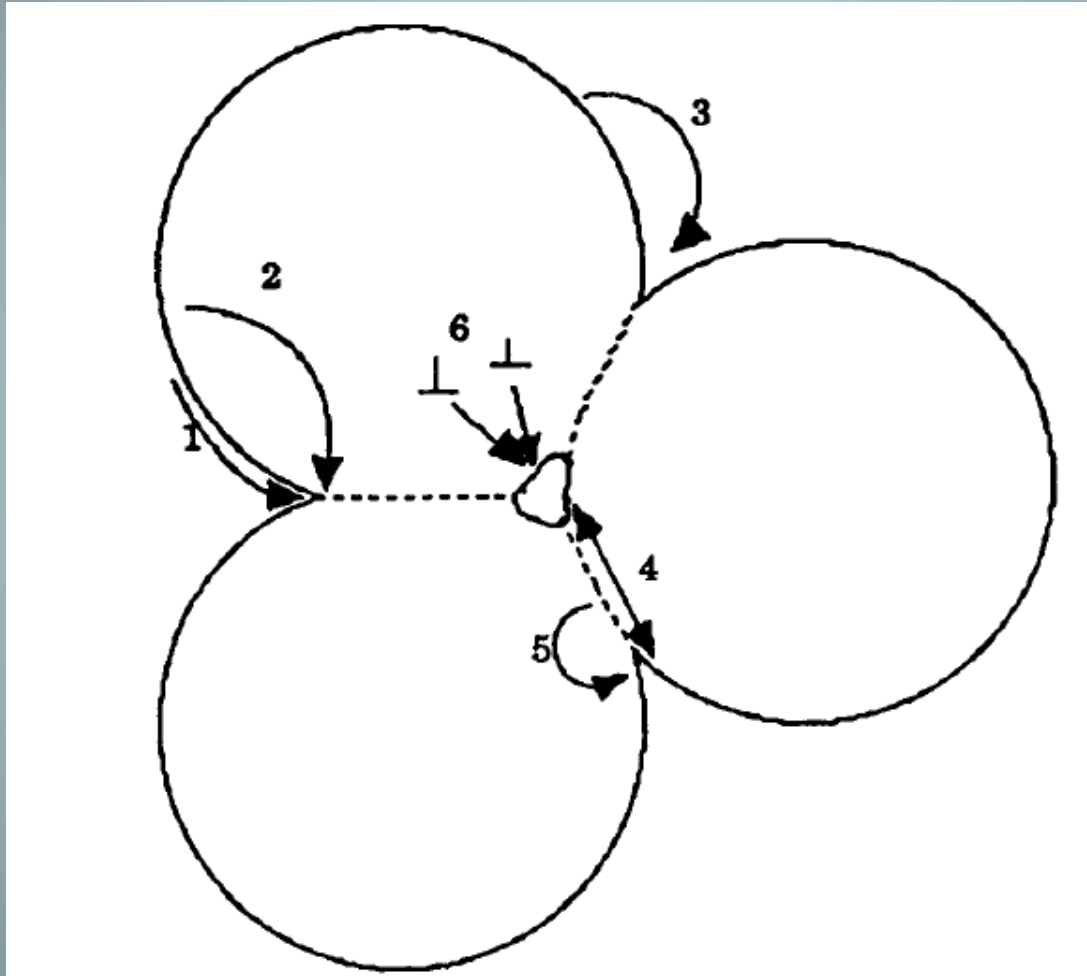
A tabulation of the possible sources of material is given in Table 1 with the transport pathway used to transport the material from the source to the neck.

These pathways are drawn on Figure 3 and consist of *surface, lattice, and grain boundary diffusion*, as well as vapor phase diffusion.

These pathways give rise to different mechanisms of sintering.

Each of these mechanisms is used to establish the kinetics of sintering.

## TYPES OF SINTERING PROCESSES



{FIGURE 3 Pathways for the transport of material during the initial stage of sintering}

## TYPES OF SINTERING PROCESSES

TABLE1 Pathways for the Transport of Material during the Initial Stage of Sintering

<i>Pathway (on Figure 16.3)</i>	<i>Transport path</i>	<i>Source</i>	<i>Sink</i>	<i>Process</i>
1	Surface diffusion	Surface	Neck	Coarsening
2	Lattice diffusion	Surface	Neck	Coarsening
3	Vapor transport	Surface	Neck	Coarsening
4	Boundary diffusion	Grain boundary	Neck	Sintering
5	Lattice diffusion	Grain boundary	Neck	Sintering
6	Lattice diffusion	Dislocations	Neck	Sintering