Preparation of Ultrafine Ceramic Powders by Vapor Phase Techniques

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Thank you Mr. Chairman, ladies and gentleman. It is my great pleasure to present a lecture at this conference.

As technology has advanced in recent years, needs for fine and homogeneous ceramic powders have increased. This situation has stimulated the study on the preparation of fine powders and many nonconventional powder preparation techniques have been developed. Vapor phase method is one of the nonconventional techniques and is receiving increasing attention as the production method of ultrafine ceramic powders.

In this presentation, after a brief introduction, I would like to talk about the principle of powder formation and particle size control in chemical vapor deposition method, and usefulness of CVD powders in study of particle size effects.

Uses of Ultrafine Powders

1. Uses in powder or dispersion state.
2. Uses in dispersion-strengthened M and C.
3. Raw powders of ceramics.
4. Uses as coating material.

As described in the text, the uses of ultrafine powders are divided into several types: (1) uses in powder or dispersion state such as pigments and fillers; (2) uses in dispersion - strengthened metals and ceramics ; (3) raw materials in fabrication of dense or porous ceramics; (4) uses as coating materials.

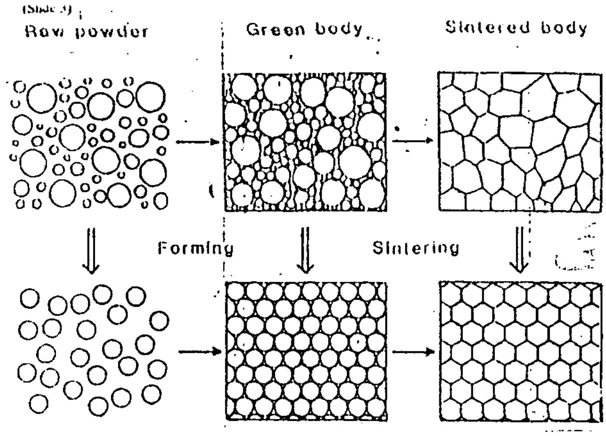
TiO_2 as white pigment:

Crystal structure : Rutile form.
Particle size : 0.2~0.4μm

γ-Fe_2O_3 for magnetic tape

Particle shape : needle-like.
Particle size : (0.05~0.07) × (0.3~0.5)μm

In the preparation of powders, it is importat to control the powder characteristics to meet the requirements of each application. For example, the light scattering power of TiO_2 powders as white pigment becomes the largest when TiO_2 takes rutile form which has the highest refractive index among the polymorphic forms and the particle size is in the this range. In the use of γ-Fe_2O_3 powders for magnetic tape, it shows the best performance when the powders consist of the needle-like particles with these sizes.

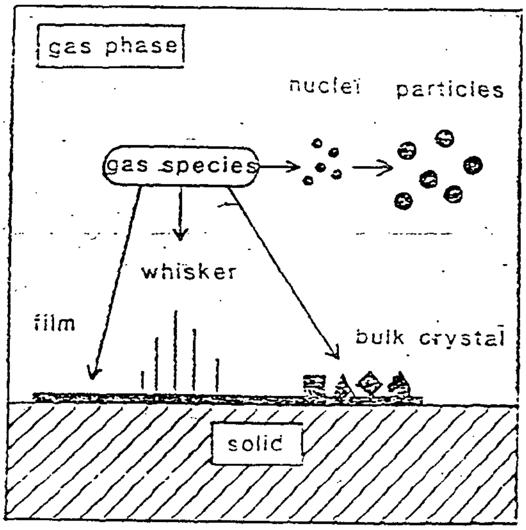


In the sintering, the shape and size of particles in the starting powders change remarkably during sintering. However, the characteristics of the raw powders give significant effects on the sintering process and microstructure of sintered body. To obtain a sintered body with a uniform and fine grained microstructure, the green body of the precursor should have a uniform and fine structure. To obtain such a green body, the raw powder of the precursor should be fine and uniform.

Requirements for Raw Powders in Sintering

1. Small and spherical particles with narrow size distribution.
2. Unaggregated powders.
3. Controlled chemical composition.

The generally accepted requirements for the starting powders are to be consisted of small and spherical particles with narrow size distribution, to be unaggregated powders, and to have high purity and uniform chemical composition. Smaller particles give a higher rate of densification both due to an increase in the driving force for sintering and due to a decrease in the distance of matter transport needed to fill pores, and permit the lowering of sintering temperature and the achievement of fine-grained ceramics. A few examples of particle size effect on the densification will be shown later.



Type of deposits from gas phase.

This slide shows the type of solid deposit from gas phase. There are four types of deposit: film, whisker, bulk crystal and powders.

② Physical Vapor Deposition (PVD)

○ $S(s) \longrightarrow S(g) \longrightarrow S(s)$

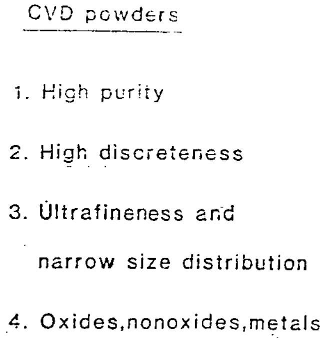
○ Film, Coating, Powder

③ Chemical Vapor Deposition (CVD)

○ $A(g) + B(g) \longrightarrow C(s) + D(g)$

○ Film, Coating, Whisker, Powder,
Bulk crystal

There are two techniques to deposit solid from gas phase: Physical vapor deposition (PVD) and chemical vapor deposition (CVD). Although both techniques give powders, the application of PVD technique which uses the evaporation-condensation is limited because of less volatile nature of refractory materials. On the other hand, CVD technique which uses the vapor phase chemical reaction is less limited and applicable for the preparation of powders of many compounds.

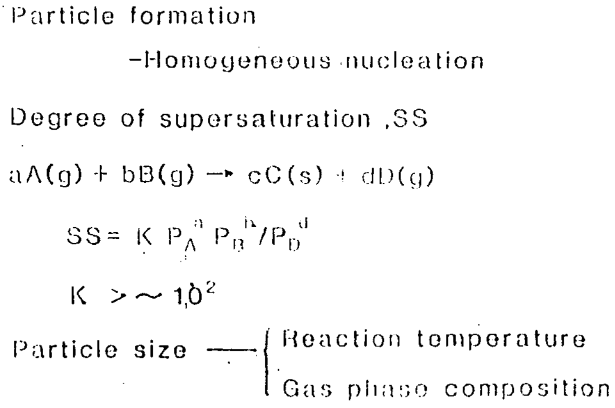


CVD powders

1. High purity
2. High discreteness
3. Ultrafineness and narrow size distribution
4. Oxides,nonoxides,metals

Chemical vapor deposition method as powder preparation technique can be characterized by: (1) high purity of powder products; (2) highly discrete particles; (3) ultrafine powders with narrow particle size distribution; and (4) versatility for the direct preparation of powders of metals and nonoxides in addition to oxides by the control of reacting atmosphere. The first three caracteristics are just the generally accepted requirements for the starting powders of sintering.

Next, I am going to talk about the principle of powder formation by CVD method. (See slide 5) The formation of films or coatings, the production of pure bulk crystals and the growth of whiskers occur via heterogeneous nucleation on a solid surface. On the other hand, powders are formed by homogeneous nucleation in the gas phase.



Particle formation

-Homogeneous nucleation

Degree of supersaturation ,SS

$aA(g) + bB(g) \rightarrow cC(s) + dD(g)$

$SS = K\,P_A^{a}\,P_B^{b}/P_D^{d}$

$K > \sim 10^2$

Particle size — { Reaction temperature / Gas phase composition

Homogeneous nucleation requires a higher degree of supresaturation. And the nucleation rate is an exceedingly steep function of supersaturation. The requirement for the production of a fine powder is initially for the achievement of high degree of supersaturation to produce a large number of nuclei by homogeneous nucleation. The degree of supersaturation for a deposition reaction is given by this equation and is proportional to the equilibrium constant for deposition reaction. On many reaction systems which deposit oxides and nonoxides, it has actually observed that powders are obtained only when the equilibrium constants have large values, something like this, in accordance with the thermodynamic consideration. This is shown in Table 1 in the text. However, a large value of the equilibrium constant is a necessary but not sufficient condition to produce powders by CVD method because the chemical reaction is involved. Both high reaction rate and large equilibrium constant are necessary to produce powders by CVD method.

Reactivity of MX_n with O_2

	E_a (kcal/mol)	log Kp (1000°C)	E_d (kcal/mol)
$SiCl_4$	90	10.6	100
$AlCl_3$	25	10.2	94
$ZrCl_4$	24	8.1	92
$TiCl_4$	17	5.6	82
$AlBr_3$	11	19.8	79
$FeCl_3$	<11	3.3	54

$$MX_{n}(g) \longrightarrow MX_{n-1}(g) + X(g)$$

In the production of oxide powders by CVD method, metal halide vapors are most commonly used as the metal sources. This slide shows the reactivity of several metal halide vapors with oxygen in the formation of oxide powders. The apparent activation energy is almost independent of the equilibrium constant or free energy change of CVD reaction. The reactivity of metal halide vapor with oxygen shows a good correlation with the dissociation energy of the first halogen as seen here. This reactivity order gives important effects for the formation process and structure of particles when polycomponent oxide powder is produced by the vapor-phase oxygenolysis of mixed halide vapors.

Control of Particle Size

Mass balance

$$d = \left(\frac{6}{\pi}\frac{M}{\rho}\frac{Co}{N}\right)^{1/3} = A\left(\frac{Co}{N}\right)^{1/3}$$

N: Temp.,Gas phase composition

The control of particle size is of prime importance in the production of powders. As the system which produces powders by CVD has a large equilibrium constant, the metal-carrying reactant can in practice be completely converted into the products by adjusting the other reactant until it is in excess. In this case, the material balance gives this relation for the particle size.

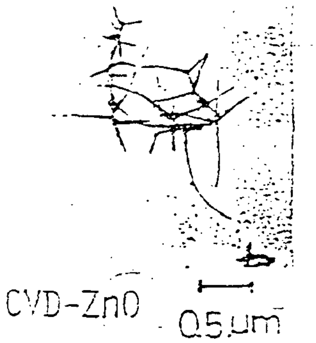
Where d is the diameter of particle produced, C_0 and N are the number of moles of metal source and the number of particle formed per unit volume of reacting gases, respectively. m is the molar weight of product and ρ is the density of product. Since M and ρ are constant for a given compound, the particle size is determined by the ratio of concentration of metal-carrying reactant to the number of nuclei formed.

Nucleation rate is a function of reaction temperatre and gas phase composition. Thus, one can control the particle size by manipulating the reaction temperature and gas phase composition as will be shown later.

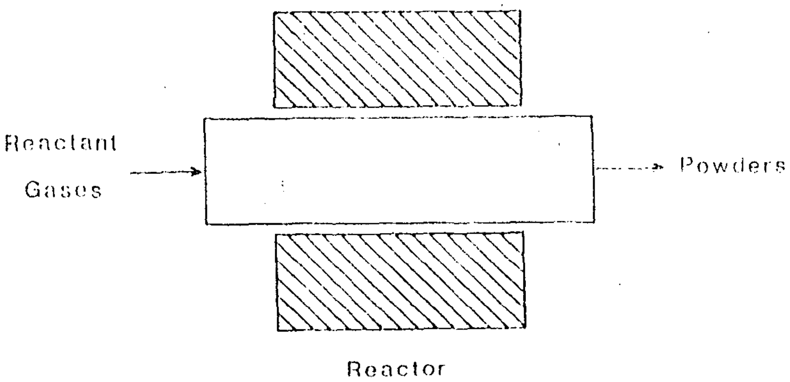
A particle is formed by the nucleation and growth. In a reacting system, there occur simultaneously the homogeneous nucleations and the growth of particles with various sizes. Under this condition, the particle size distribution of powders is determined by the relative magnitudes of the rates of the homogeneous nucleation and growth.

The effect of the consumption of reactants may be exceedingly larger on nucleation rate than on growth rate, because the nucleation is very sensitive to the supersaturation ratio which decreases rapidly with the proceed of reaction. In addition, when particles are once formed in a vapor phase, the deposition on the surfaces is more favorable thermodynamically than the formation of new nuclei. The reason why a vapor-phase reaction method gives a powder with a uniform particle size may be that the nucleation takes place predominantly in the very early stage of the reaction and stops in practice in the later stage.

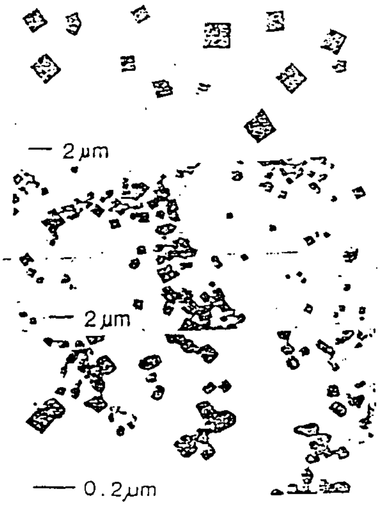
The control of particle shape is also interesting. Vapor phase reactions give single-crystalline or polycrystalline particles depending on reaction systems and reaction conditions used. The polycrystalline particles are spherical in shape. The single-crystalline particles have also spherical shapes as a whole, although they are often angular to some extent. In the reaction system which has so high degree of supersaturation as to cause a homogeneous nucleation, all kinds of growing crystal faces will have a large number of kinks or growth sites and then a similar growth rate. Thus, particles grow isotropically and result in spherical forms. Thus, the formation of anisotropically grown particles by vapor-phase reaction method is difficult in principle. An exception is the formation of needle-like particles of ZnO in the reaction of Zn vapor and oxygen.



Vapor phase reaction of zinc vapor and oxygen at 850 to 1100°C gives granular and needle-like particles of zinc oxide. The amount of needle-like particles increased with increasing reaction temperature or at high concentration of zinc vapor. Particles smaller than 0.06μm are granular. On the other hand, particles larger than 0.1μm have needle-like shapes. These facts suggest that anisotropic growth become remarkable when particle grew above 0.1μm. The growth direction of needle particles is c-axis.

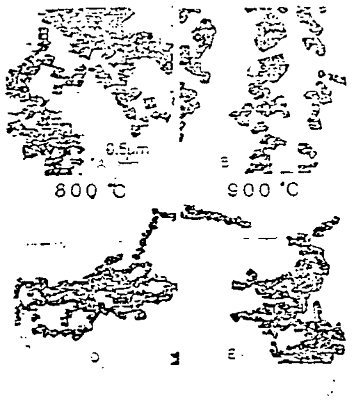


Next, I would like to illustrate a few oxide powders produced by CVD method and control of their particle sizes by reaction conditions. The following results were obtained by using such a horizontal flow reactor under atmospheric pressure.



$TiCl_4+O_2=TiO_2+2Cl_2$

This slide shows anatase particle produced by this CVD reaction. The particles are discrete and single crystalline regardless of their size. Large particles are square planar grown in parallel to c-plane.



A, B.

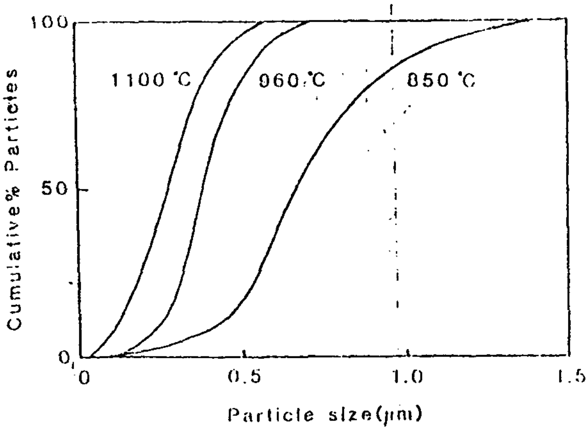
$FeCl_2-NiCl_2-O_2 \rightarrow NiFe_2O_4$

D, E.

$FeCl_2-CoCl_2-O_2 \rightarrow CoFe_2O_4$

This slide shows ferrite particles produced by oxygenolysis of mixed chloride vapors at 800° or 900°C. Particles are also discrete and single crystalline.

From such TEM photographs, one can obtain he particle size distribution. In the calculation, the size of particles is measured over several hundred particles.

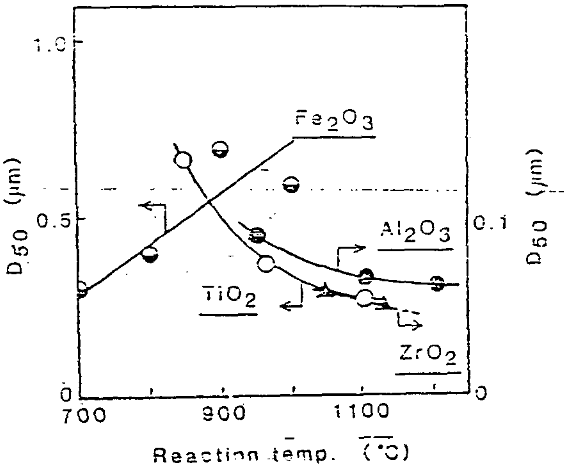


Reaction temperature and particle size

$TiCl_4 + O_2 = TiO_2 + 2Cl_2$

As mentioned above, in CVD method, the particles size can be controlled by reaction temperature and composition of reacting gases. This will be illustrated in the following slides.

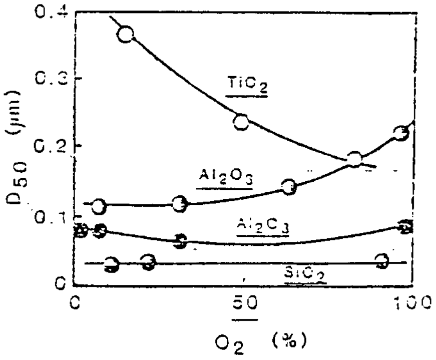
This slide illustrates the effect of reacton temperature on the particle size of titania produced by this reaction. Particle size decreases with increasing reaction temperatures.



Reaction temperature

and particle size

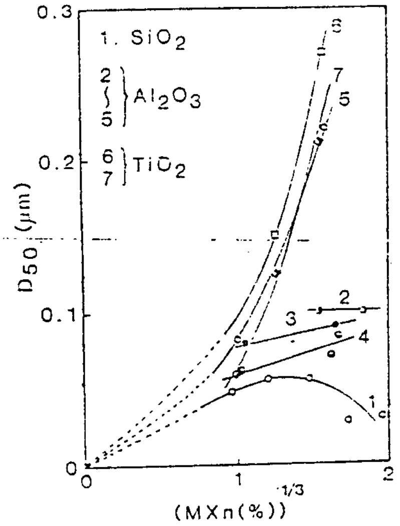
This slide shows the effect of reaction temperatures on particle size in several oxygenolyses of metal halides. In the formation of titania, alumina and zirconia powders, the particle size decreases with increasing reaction temperature. On the other hand, the particle size of ferric oxide increases with increasing reaction temperature. This reaction system is characterized by the small equilibrium constant of deposition in comparison with the other three reaction systems.



O_2 concentration and particle size

This slide shows the effect of oxygen concentration on particle sizes.

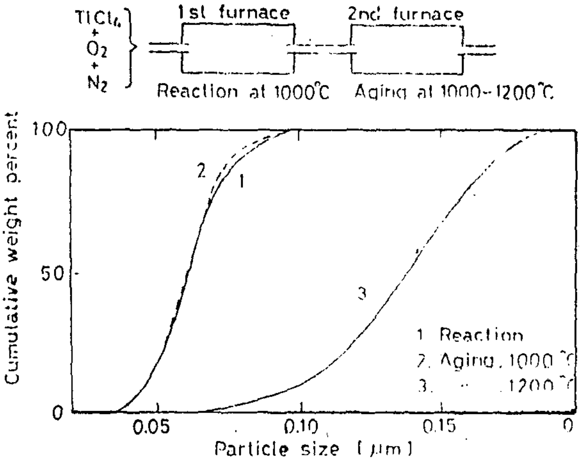
The particle size of titania decreases with increasing oxygen concentration. On the other hand, the effect of oxygen concentration on the particle size of alumina powders in reverse in the tendency. The particle size varies little with O_2 concentration in the formation of silica powders.



Halide concentration and particle size.

The effect of the metal halide concentration on particle size is more complicated as shown in this slide. Where particle size is plotted vs this value $(MX_n)1/3$ because particle size should be proportional to the cube root of the halide concentration if the nucleation frequency in vapor phase is the same at different halide concentration.

The effects of the reaction conditions on particle sizes illustrated above are difficult to interpret in terms of the physicochemical propertieds of each reaction system at present. However, we have seen that one can control particle sizes by reaction temperature and gas phase composition.



Coalescence of TiO_2 particles in CVD

In recent years, many members of the Society of Chemical Engineering of Japan are studying on CVD method from the view point of chemical engineering. This is an unpublished result obtained recently by professor Morooka et al. of our department. They studied on the coalescence of titania particles in CVD method. Their method is shown here. The reactant mixture was introduced into the first furnace held at 1000°C. In the first furnace, titanium tetrachloride is converted completely into titania powder. The particle size distribution of powder left the first furnace is shown by curve 1. The suspension of titania powders in gas mixture was carried into the second furnace heated at 1000° to 1200°C. The particle size distribution of powder left the second furnace did not change when the temperature of the second furnace was 1000° or 1100°C, but shifted to curve 3 when the second furnace was heated at 1200°C. This result indicates that the coalescence of titania particles can occur in gas phase above 1200°C and that the coalescence of particles during CVD reaction should be considered when the materials are less refractory.

Formation Processes of Nitride and Carbide Particles

A. Formation of adduct particles between reactants and their thermal decomposition.

B. Nucleation and growth.

C. Formation of metal particles and their nitridation or carburization.

We can produce very fine powders of refractory nitride and carbide by CVD method as shown in Table 2 in the text. The particle formation processes can be classified into three types as described in the text:

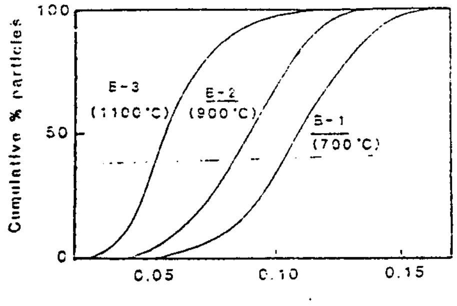
(A) Formation of adduct particles of reactants and their thermal decomposition into nitrides or carbides.

(B) Formation of nuclei of nitrides or carbides and their growth into particles.

(C) Formation of metal particles and their nitridation or carburization.

The formation process changes with reaction conditions, mainly with reaction temperature, even in the same reaction system. Furthermore, the method of introduction of the reactant gases into the reaction zone significantly influences the formation process and then the properties of resulting powders.

For example, in the formation of titanium nitride powders from titanium tetrachloride and ammonia system, the particle formation process changes form Process A to Process B with increasing the mixing temperature of titanium tetrachloride and ammonia. When the particles are formed by process B, the particles are single crystalline and the particle size can be controlled by reaction temperature as seen in the next slide.



TiN powders from $TiCl_4$-NH_3 System

The particle size decreases with increasing reaction temperature.

CVD Method for Powder Preparation

◎ Heating Method of Reactant

1. Electric furnace
2. Chemical flame
3. Plasma (hot)
4. Laser

◎ Reactant

1. Metal halide (Chloride)
2. Metal vapor
3. Organometallic compound (MOCVD)

   M(OR)n , MRn

   Si-N-C compound

In CVD method, we have to heat the reactant gas mixture to obtain a high reaction rate enough to produce a powder as explained above. The most common heating method is the use of electric furnace. I have just talked about the results obtained by this method. The other heating methods for powder production are chemical flame, high temperature plasma and laser method. In these new methods, one can keep the reactor wall at low tempeature. On concerning raw materials, the most common reactant for powder production is metal chloride vapors. Recently, organometallic compounds are receiving increasing attention as the reactants.

This time, I would like to talk about the formation of silicon carbide powders by plasma method.

Preparation of SiC powder by CVD method.

1) $Si(CH_3)_4 \xrightarrow[900\sim1400\,^{\circ}C]{H_2} SiC\ (0.01\sim0.2\,\mu m)$

2) $SiH_4 + CH_4 \xrightarrow[1200\sim1400\,^{\circ}C]{H_2} SiC\ (0.01\sim0.1\,\mu m)$

3) $CH_3SiCl_3 \xrightarrow[plasma]{H_2} SiC\ (0.01\sim0.03\,\mu m)$

4) $SiCl_4 + CH_4 \xrightarrow[plasma]{} SiC\ (0.01\sim0.1\,\mu m)$

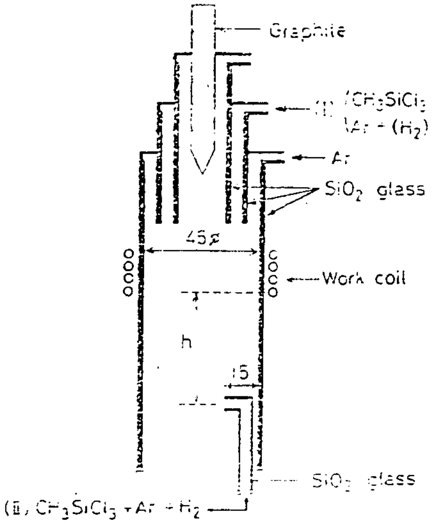
5) $SiO_2 + CH_4 \xrightarrow[plasma]{} SiC$

Si-C-O-H system

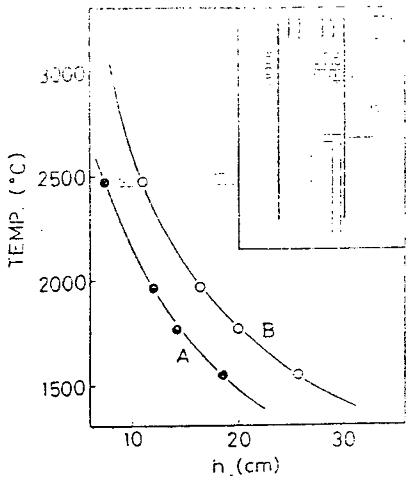
alkoxysilane $\longrightarrow$ SiC (~0.1μm)

Here, the reaction systems for the preparation of silicon carbide by CVD method are illustrated. When the reaction systems have large equilibrium constants and high reaction rates, carbide powders are obtained by CVD method at relatively low temperatures. However, these silicon compounds seem to be expensive as the raw materials for commercial porcess. If we use less expensive compounds such as methyl trichlorosilane or silicon tetrachloride, the carbide formation require a higher reaction temperature because of their lower reactivities.

From these reasons, the application of plasma to the powder production of refractory carbide is receiving increasing attention recent years. Today, the preparation of silicon carbide powders from CH_3SiCl_3 and $SiCl_4$-CH_4 systems by rf-plasma method will be illustrated.

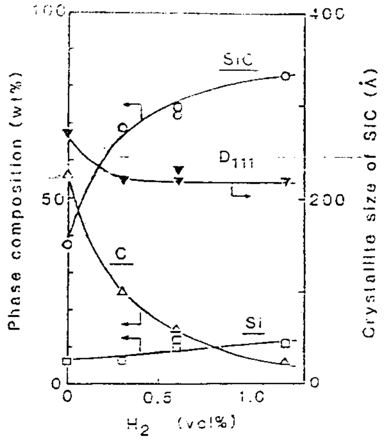


This slide shows rf-plasma reactor used. The frequency is 4MHz.



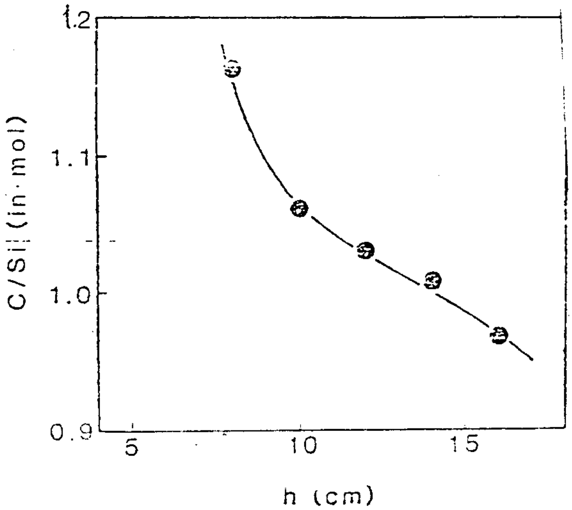
This slide shows the temperature distribution in plasma reactor. This h is the distance from the bottom of work coil. Curve B shows the temperature along the center line of reactor. Curve A shows the temperature at 15mm from the center line. In the carbide formation, the position of the inlet of reacting gases, h, is very important.

See slide 24. When the reactant gas mixture of CH_3SiCl_3-Ar-H_2 was introduced from the top of reactor, the reaction temperature was too high and a large amount of free carbn was by-produced as seen in the next slide.



$CH_3SiCl_3 \longrightarrow SiC$

Addition of H_2 into the reactant gas suppresses significantly the formation of free carbon and increases the amount of silicon carbide. However, one can not increase the concentration of H_2 beyond this value because of the quenching of plasma flame . Further, the addition of H_2 increases the content of Si in the product.

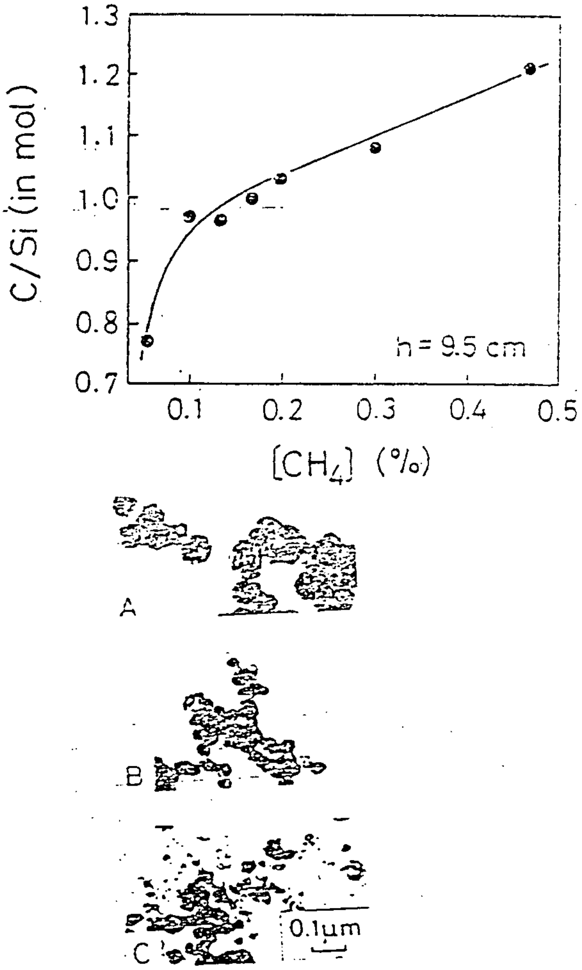


Composition of Product

See slide 24. On the other hand, when the reactant gases were introduced into the tail of plasma flame, the formations of free carbon and silicon could be suppressed below the detectable amount by X-ray diffraction. In this case, the atomic ratio C/Si of the product could be controlled by the position of the introduction of reactants, h, that is, by the reaction temperature as seen in the next slide.

This slide shows the change of the C/Si ratio by h.

The similar method gives B-SiC powders from the $SiCl_4$-CH_4 system. In this reaction system, the position of the introduction of reacting gases also gives a remarkable effect on the composition of product. When the position, h, is properly selected, the atomic ratio C/Si could easily be controlled by the concentration of methane as seen in the next slide.



SiC ($SiCl_4$-CH_4)

This slide shows the particles of β-silicon carbide produced from $SiCl_4$-CH_4 system. The particles are smaller than 0.1μm and are single crystalline.

Although a thermal plasma is characterized by its very high temperature, the above results show that the control of reaction temperature is important in carbide formation to suppress the excess formation of free carbon.

Next, I am going to talk about the use of CVD powders in study of particle size effect. Because of the high discreteness and ultrafineness, CVD powders are very suitable to elucidate the particle size effects in various fields.

$$\underline{TiO_2+BaCO_3 = BaTiO_3+CO_2}$$

Reactivity of TiO_2

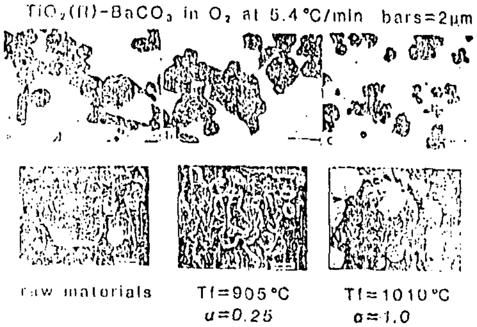
- independent of crystal form
- depend on particle size

Shape and size of $BaTiO_3$

- determined by these of TiO_2
- independent of these of $BaCO_3$

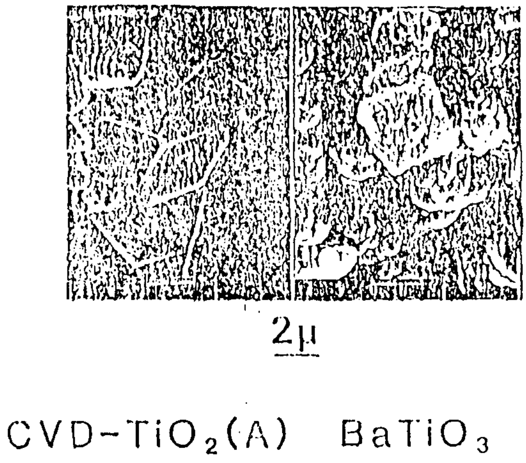
The first talk is about reactivity of titania powders with barium carbonate. There are many studies on this reaction. And it was generally accepted that anatase is more reactive than rutile. However, in the previous studies on the effects of the particle size and crystall type of titania powders on the reactivity in the solid-state reactions with barium carbonate, anatase powders were produced by wet methods and rutile powders were prepared by the calcination of anatase at high temperatures. In these titania powders, the primary particles are considered to be aggregated, especially in calcined rutile powders. Therefore, it is questionable whether or not the results truly represent the effect of the size or crystal type of the primary titania particles. On the other hand, as shown above, CVD titania powders (both anatase and rutile forms) are characterized by a very closely controlled particle-size distribution and high discreteness and consist of single-crystalline particles. Accordingly, CVD titania powders are expected to show exactly the effects of the size and crystal type of titania particles on the reactivity. Studies on the reactions between CVD titania powders and barium carbonate revealed clearly that the reactivity of the titania powders is independent of the crystal form of titania and depend only on the particle size.

Secondly, the study revealed thatthe shape and size of barium titanate particles produced are determined by these of starting titania particles and independent of these of barium carbonate. This is due to that the reaction proceeds topotaxially on titania particles. The conclusion will be shown by the next few slides.

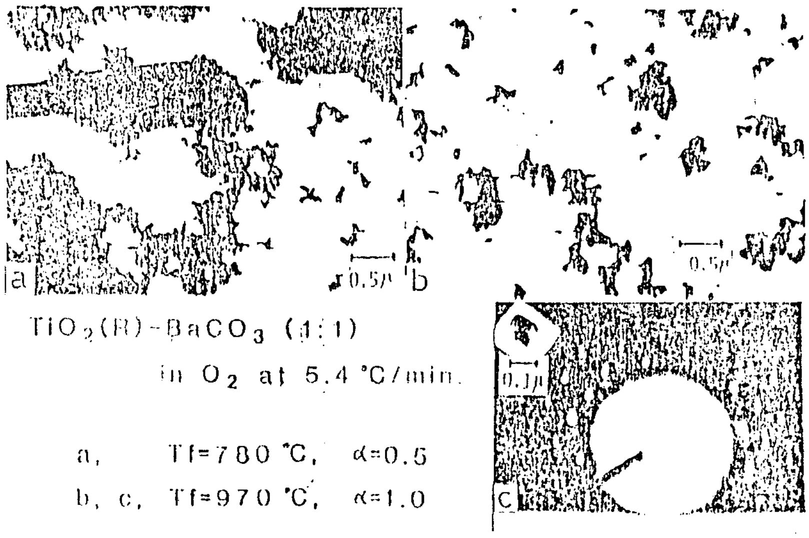


This slide shows the change of reactant mixture when equimolar mixture of CVD titania and barium carbonate mixture was heated at a constant rate in oxygen.

The left photograph is the starting mixture. The round particles are rutile and rod-like particles are barium carbonate. The right photograph shows barium titanate after the completion of reaction. Rod-like particles of barium carbonate disappeared.



This photograph shows that the shape and size of barium titanate are very similar to those of starting titania particles.



TiO_2(R)-$BaCO_3$ (1:1)
in O_2 at 5.4 °C/min.

a, Tf=780 °C, $\alpha=0.5$
b, c, Tf=970 °C, $\alpha=1.0$

This slide shows the drastic change of the shape and size of starting materials. This photograph shows the reactant mixture when the reaction proceeded upto 50%. Large rod-like particles are barium carbonate and small particles are titania or barium titanate. After the completion of reaction, large barium carbonate particles disappeared. Particles of barium titanate are single-crystalline and their size distribution is very close to that of starting titania powders and independent of the particle size of barium carbonate.

1. CVD-MgO

Mg vapor + O_2 ⟶ MgO

2. CVD-WC

$WCl_6 + CH_4 + H_2 \longrightarrow WC$

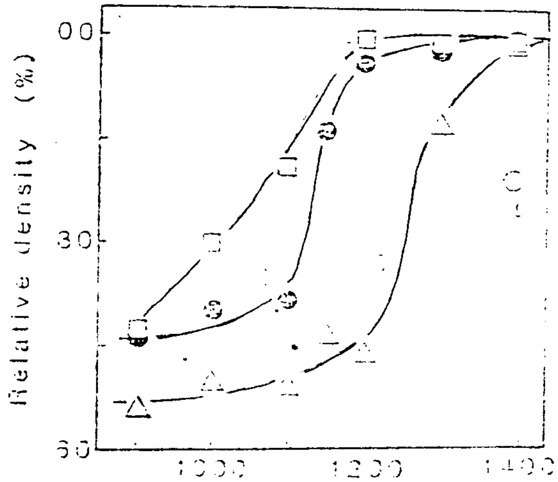
3. CVD-SiC

$SiH_4 + CH_4 \longrightarrow SiC$

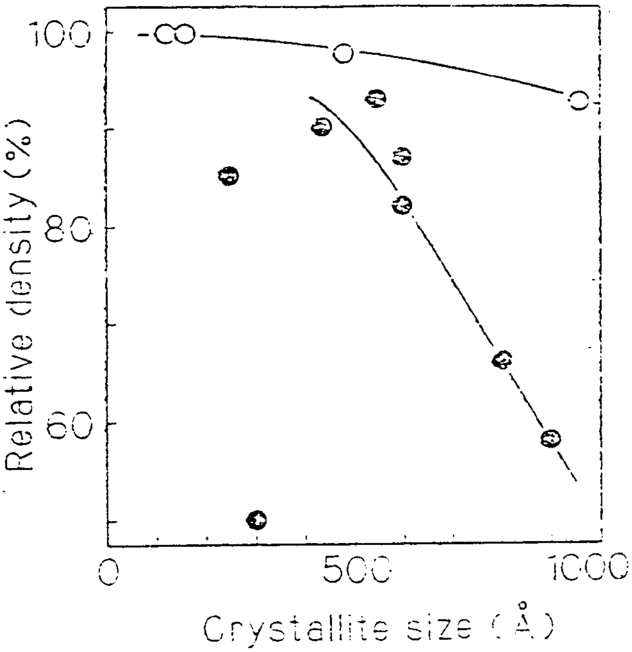
$Si(CH_3)_4 \longrightarrow SiC$

$Si(CH_3)Cl_3 \longrightarrow SiC$

Next, I am going to talk about the sinterability of ultrafine CVD powders. The powders were produced by CVD methods shown here.

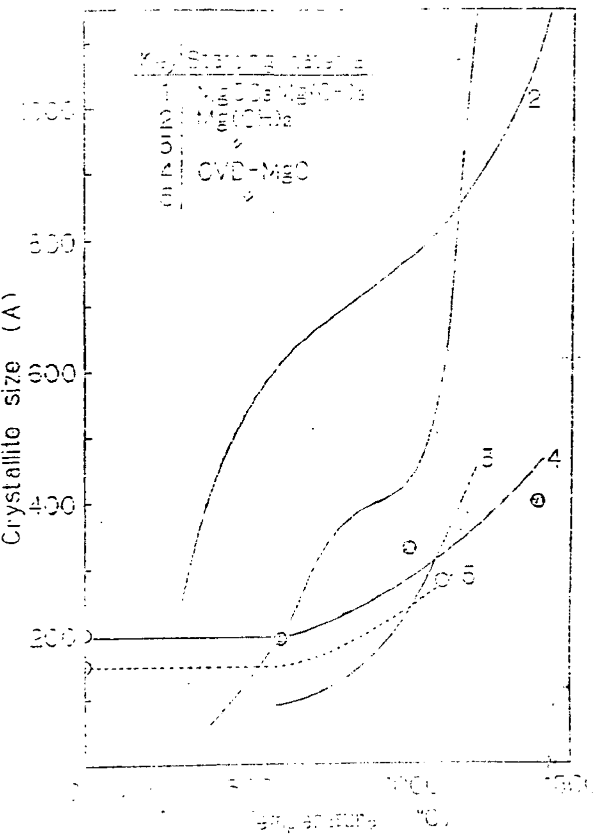


This slide shows the sintering behavior of CVD magnesia with different particle sizes. CVD magnesia consists of single crystalline particles. The average particle size calculated from surface area are CVD magnesia consists of single crystalline particles Firings were done for 6hr in air at temperature shown here. We can see a remarkable effect of particle size for densification. With decrease in particle size, the densification curves move from the high temperature region to the low temperature region, and powder

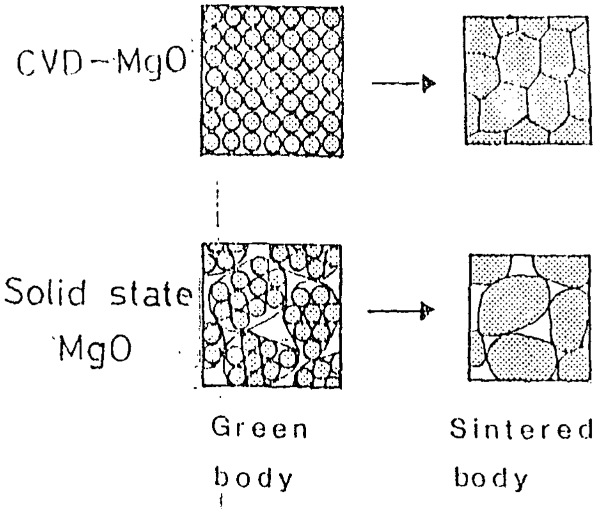


with particle size of 120Å densified nearly to the theoretical density at temperature as low as 1200°C. When the particle size is smaller than 200Å, one can obtain a sintered body close to theoretical density within an hour at 1300°C.

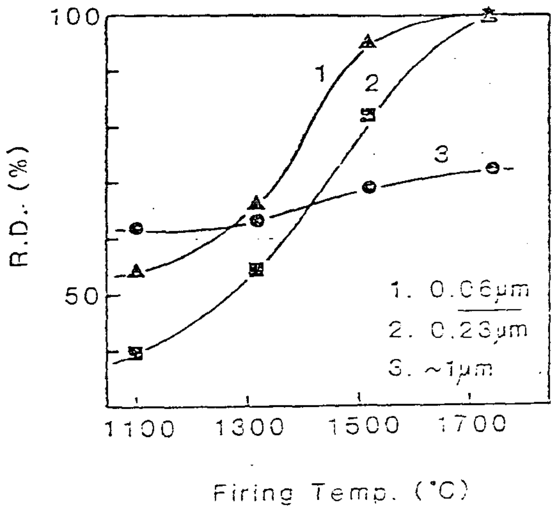
In this slide, the sinterabilities of CVD magnesia are compared with those of magnesia produced by the solid-state decomposition of various magnesium compounds. The sinterability at 1400°C is plotted against crystallite size of magnesia in the starting powders. As you can see, CVD magnesia shows a very high sinterability. The higher sinterability of CVD-magnesia is considered mainly due to the nonagglomeration. This is supported by the following fact.



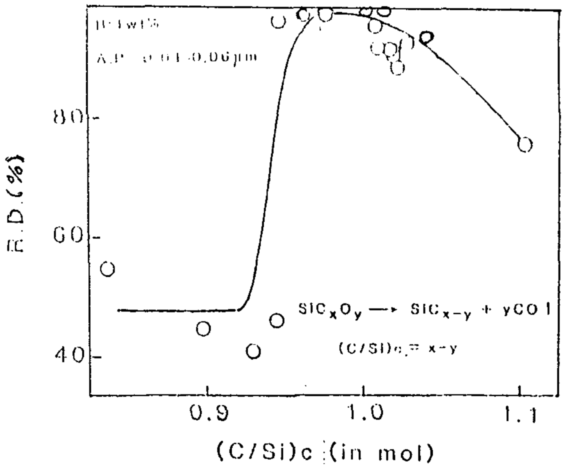
This slide shows the particle growth of magnesia produced by different method. In magnesia powders prepared from the decomposition of magnesium compounds, particle grows remarkably at temperatures above 1000°C. This may be due to the fact that the primary particles remain aggregated usually in the shape similar to that of the precurs or compound. On the other hand, the particle growth is very slow in CVD-magnesia because of the high discreteness of particles.



From these facts, in the sintering of magnesia produced by solid state reaction, the sintering or grain growth within aggregates occurs at the initial stage of sintering and results in a low final density. In such powders, the aggregation obscures the effect of the size of the primary particles on the sinterability.

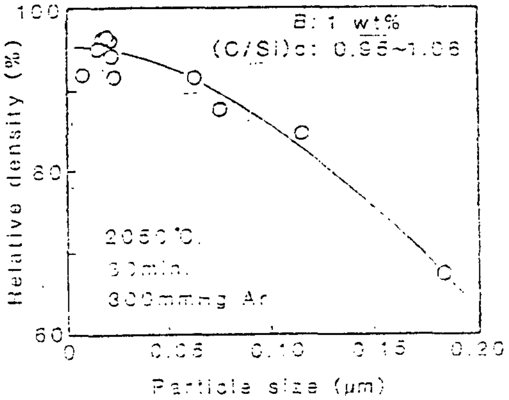


This slide shows the sintering behavior of tungsten carbide. Samples 1 and 2 are CVD-powders, and sample 3 is a commercial powders produced by solid state reaction. The average particle sizes are shown here. Fine powders, samples 1 and 2 densify to the theoretical density at around 1700°C. On the other hand, coarse powders, sample 3 densify little at such a low temperature, indicating the remarkable particle size effect on the sinterability.

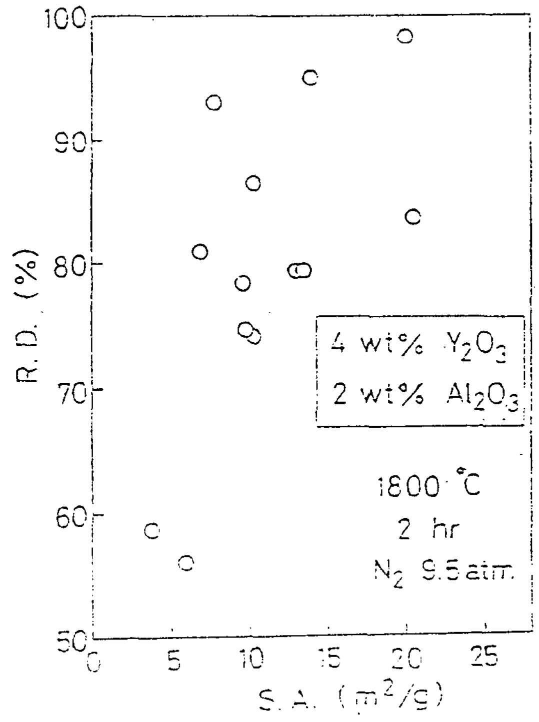


Sintering of CVD-SiC
(2050 °C, 30min, 300mmHg Ar)

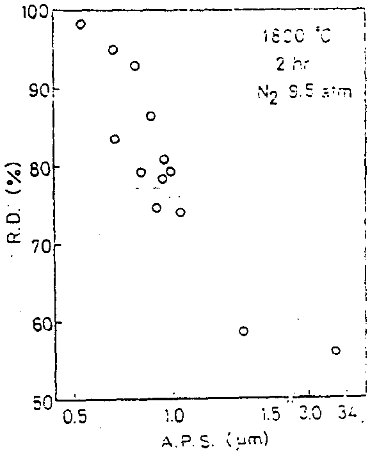
Next, I am going to talk about the sintering of ultrafine β-silicon carbide prepared by CVD method. Silicon carbide is a very good example to illustrate the importance of powder conditioning for the sinterability. Their mean diameters (median diameter) were in the range of 0.01 to 0.06μm. In the sintering, 1 wt% of boron powders was added as the sintering aid. The sintering was done under the condition shown here. This slide shows the effect of chemical composition of silicon carbide powders on the sinterability. Because of the fineness of silicon carbide used, large amounts of oxygen were contained in the original powders. This ratio C/Si shows the value when the surface oxygen was assumed to be remove as carbon monoxide during the sintering. As seen in this figure, silicon carbide powders having the ratio C/Si close to unity show the highest sinterability. When the ratio C/Si is below 0.95, the densification does not occur although the particle growth occurs to a remarkable extent. This result strongly supports the proposal that the carbon added as a sintering aid accelerates the sintering of silicon carbide by the removal of surface oxide of SiC particles. Accordingly, the control of chemical composition of original powders is very important in the sintering of fine silicon carbide. In addition, it has also been found that the carbon as the sintering aid should be added uniformly on the surface of silicon carbide particle to increase the sinterability.



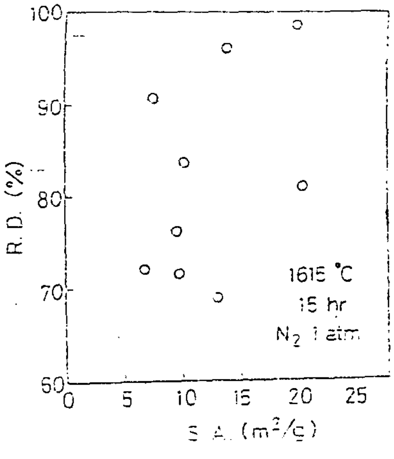
This slide shows the relation between the average particle size of silicon carbide and the relative density of sintered body. The sinterability decreases gradually with an increase in the particle size of silicon carbide.



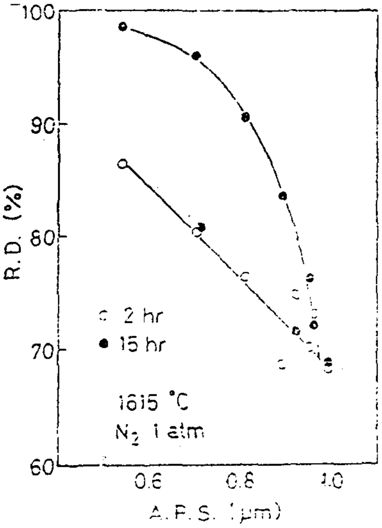
Next, I will show you the particle size effect in the sintering of silicon nitride. Silicon nitrides examined were commercial powders. In this slide, the relative density of sintered body is plotted against the surface area of the raw powders. The sintering aid used was the mixture of 4 wt % Y_2O_3 and 2 Wt % Al_2O_3. Firing was done at 1800°C for 2 hr under 9.5 atm of nitrogen. The samples were embedded in silicon nitride powders with sintering aids of the same composition. As you can see, the correlation between surface area and relative density is very poor.



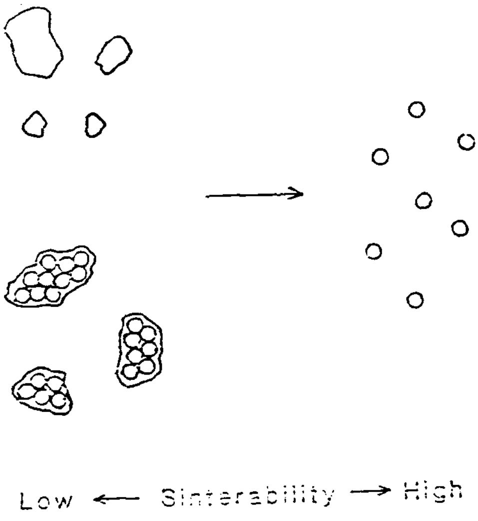
This slide shows the relation between the relative density of sintered body and the average particle sizes. This particle sizes were the weight mean diameters determined by a centrifugal sedimentation method*, that is Stokes diameter. We can observe a good correlation between the relative density and particle size. The relative density decreases rapidly with increase in the particle size.



This slide shows the relation between the relative density of sintered body and surface area of raw powder in the firing at 1615°C. The correlation is poor.



On the other hand, one can observe again a better correlation between relative density and Stokes diameter. The fact that the relative density has a better correlation with Stokes diameter than with surface area suggests that the agglomeration of raw powders has an important effect on the sinterability. The observation of microstructure of sintered bodies showed that sintered bodies with high density consisted of fine grains, whereas those with low density consisted of large grain with rod-like shape.



I have talked about the sintering of fine powders and illustrated that, in order to increase the interability, the decrease of particle size and decrease in the degree of aggregation or agglomeration are important. Powder produced by CVD method can satisfy these requirements (See slide 7)

Today one can produce the pure and fine powders of many oxides and nonoxides by CVD method. However, they seem to be expensive at present as the raw powder for ceramics. The high cost of fine powders may be an importnt problem for their wide applications. This problem may be solved when the excellent characteristics of fine and pure powders are recognized and the demand for them has increased.

Thank you for your attention.