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A new production method for high-purity ZrO<sub>2</sub> powder has been developed. Non-stabilized ZrO<sub>2</sub> powder high purity can be produced by heating a mixture of zircon (ZrO<sub>2</sub>·SiO<sub>2</sub>) and carbon powders under reduced pressure. Stabilized ZrO<sub>2</sub> powders with high purity can also be obtained by adding a stabilizer such as CaO or Y<sub>2</sub>O<sub>3</sub> to the zircon/carbon mixture. The generation of SiO(g) from a zircon/carbon mixture is greatly accelerated by heating under reduced pressure compared with under atmospheric pressure. The purity of ZrO<sub>2</sub> powder produced by this method is about 99.8%, and the average grain size is smaller than 5μm. Fine Y<sub>2</sub>O<sub>3</sub>-partially-stabilized ZrO<sub>2</sub> powder (<1μm) can also be obtained by wet grinding for use as the raw material for a ZrO<sub>2</sub> sintered body with high strength and high toughness. In addition, ultra-fine SiO powder (<50nm) as a by-product of this process can be obtained by vapor-phase condensation of SiO(g).

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# High-Purity Zirconia Powder Produced from Zircon by Carbothermic Reduction under Reduced Pressure\*



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## 1 Introduction

Zirconia ( $ZrO_2$ ) is an oxide of high melting point (2 988 K), and is used as the material for various refractories. In recent years, zirconia has become more widely used as a principal material for solid electrolytes, abrasives, pigments, electronic ceramics, glass, etc. Sintered zirconia partially stabilized with  $Y_2O_3$  is finding increasing application as high-strength and high-toughness engineering ceramics. An expansion of the market for zirconia is expected especially in the field of various machine parts in which the high strength, high tough-

## Synopsis:

A new production method for high-purity  $ZrO_2$  powder has been developed. Non-stabilized  $ZrO_2$  powder with high purity can be produced by heating a mixture of zircon ( $ZrO_2 \cdot SiO_2$ ) and carbon powders under reduced pressure. Stabilized  $ZrO_2$  powders with high purity can also be obtained by adding a stabilizer such as  $CaO$  or  $Y_2O_3$  to the zircon/carbon mixture. The generation of  $SiO(g)$  from a zircon/carbon mixture is greatly accelerated by heating under reduced pressure compared with under atmospheric pressure. The purity of  $ZrO_2$  powder produced by this method is about 99.8%, and the average grain size is smaller than  $5 \mu m$ . Fine  $Y_2O_3$ -partially-stabilized  $ZrO_2$  powder ( $<1 \mu m$ ) can also be obtained by wet grinding for use as the raw material for a  $ZrO_2$  sintered body with high strength and high toughness. In addition, ultra-fine  $SiO$  powder ( $<50 nm$ ) as a by-product of this process can be obtained by vapor-phase condensation of  $SiO(g)$ .

ness and high wear resistance of zirconia are required.

To expand the lines of its business, Kawasaki Steel has started the research and development of various engineering ceramic. The company is now pushing forward the development ranging from the synthesizing of raw-material powders of boron nitride (BN), zirconia, silicon nitride ( $Si_3N_4$ ), etc. to the manufacture of sintered products. Concerning the production of zirconia powder, it was found that high-purity zirconia powder can be produced by a new production method involving mixing zircon powder with carbon powder and heat-treating the mixture under reduced pressure.<sup>1,2)</sup>

This report describes manufacturing techniques of zirconia powder by a newly developed carbothermic reduction method under reduced pressure and the thermodynamical background of this method. It also describes effects on the making of zirconia of raw materials mixing ratio, heat treatment conditions under reduced pressure, addition of a stabilizer, together with the characteristics of zirconia powder obtained. In addition, it outlines ultrafine  $SiO$  powder obtained as a by-product of this manufacturing process.

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## 2 Conventional Production Techniques of Zirconia Powder

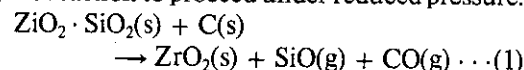
The production methods of zirconia powder are broadly classified into the dry method, involving electro-melting desiliconization (the electric arc fusion method), and the wet method.<sup>3,4)</sup> Table 1 gives an outline of the conventional production methods of zirconia powder and the characteristics and main applications of zirconia powder obtained by these methods. In the dry methods, zirconia powder is usually produced at low cost although its purity is relatively low; therefore, the zirconia powder produced by the dry methods is used as the materials mainly for refractories and pigments. On the other hand, high-purity and fine zirconia powder is obtained by the wet method and is used as the materials mainly for high-strength engineering ceramics, electronic ceramics, etc. However, the wet method is essentially expensive because the manufacturing process is long and complex.

Accordingly, the purpose of the present study is to establish a new production technique for heat-treating a mixture of zircon powder and carbon powder under reduced pressure by eliminating the respective disadvantages of the dry and wet methods, i.e., the low purity and high cost.

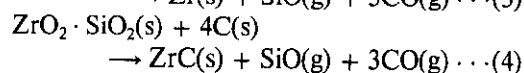
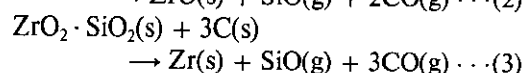
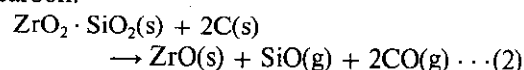
## 3 Thermodynamic Examination

Zircon is a compound oxide expressed by  $ZrSiO_4$  or  $ZrO_2 \cdot SiO_2$ . A thermodynamic examination was conducted beforehand to investigate whether it is possible to produce zirconia from zircon by the carbothermic reduction reaction under reduced pressure. It is thought that the formation of zirconia as reaction of zircon to carbon is given as a whole by Eq. (1). Therefore, it is expected that the acceleration of the reaction itself or a

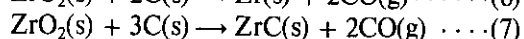
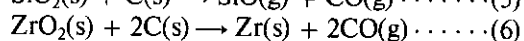
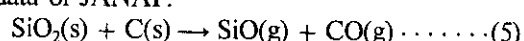
decrease in the reaction temperature is accomplished causing the reaction to proceed under reduced pressure.



However, it is considered that the following reactions expressed by Eqs. (2) to (4) also occur in the presence of excess carbon:



With respect to these reactions, the temperature- $P_{CO}$  equilibrium curve of a stable phase, formed by the reaction to the carbon of  $SiO_2$  or  $ZrO_2$  following to Eqs. (5) to (7), was determined with the aid of the thermodynamic data of JANAF.<sup>5)</sup>



Results of this examination are shown in Fig. 1. As is apparent from the equilibrium curve of  $SiO(g)$  determined according to the reaction formula (5) on the assumption that  $P_{SiO} = P_{CO}$ , the temperature at which  $SiO(g)$  is generated decreases by more than 100 K when the pressure is reduced by one order, and the accelerating effect of the heat treatment under reduced pressure on the reaction is considered noticeable. Furthermore, it is considered that  $SiO(g)$  is generated in a stable  $ZrC$  region and that the  $ZrC$  is inevitably formed at the same time with the formation of  $ZrO_2$ . In order to suppress the formation of  $ZrC$  to ensure single-phase  $ZrO_2$ , therefore, it is necessary that the composition of the raw material mix should have theoretical mixed-amounts corresponding to the reaction of Eq. (1), i.e., the molar ratio of the  $SiO_2$  in zircon to C,  $C/SiO = 1.0$ , and that a

Table 1 Main conventional production methods of zirconia powder and their characteristics

		Outline of production method	Characteristics of powder	Main uses
Dry	Electric arc fusion method	$\begin{array}{c} (CaO) \\ \downarrow \\ \text{Zircon + Carbon} \\ \text{Baddeleyite} \end{array} \xrightarrow{\text{Arc fusion}} \begin{array}{l} \text{Non-stabilized } ZrO_2 \\ \text{CaO-stabilized } ZrO_2 \end{array}$	Purity (~99%) Coarse Cheap	Refractories Pigments
	Plasma arc method	$\text{Zircon} \xrightarrow{\text{Plasma arc}} \text{Non-stabilized } ZrO_2$	Purity (~99%) Fine	Pigments
Wet	Alkali fusion method	$\begin{array}{c} \text{Zircon} \\ \text{Baddeleyite} \end{array} \xrightarrow{\text{NaOH}} \text{Alkali fusion} \xrightarrow{\text{H}_2\text{O, HCl}} \text{Na}_2\text{ZrO}_3 \xrightarrow{\text{YCl}_3} \begin{array}{l} \text{Non-stabilized } ZrO_2 \\ \text{Y}_2\text{O}_3 \text{ partially stabilized } ZrO_2 \end{array}$	High purity (>99.5%) Ultra fine Expensive	Advanced ceramics Electrical ceramics Glass composition

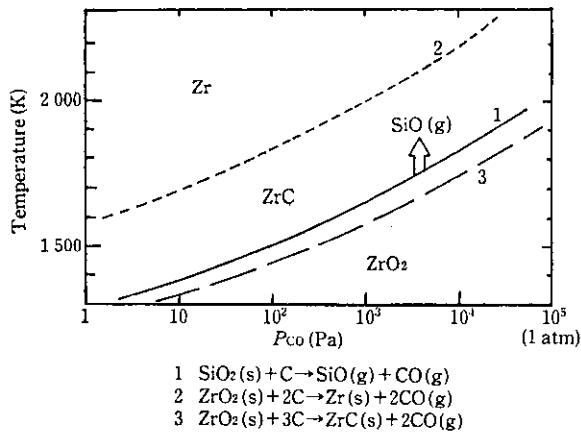


Fig. 1 Thermodynamic calculation

small amount of formed ZrC should be transformed into  $\text{ZrO}_2$  by oxidation.

#### 4 Development of Carbothermic Reduction Process under Reduced Pressure

##### 4.1 Experiment Method

##### 4.1.1 Production flow and vacuum furnace for heat treatment under reduced pressure

The production flow in this experiment is shown in Fig. 2. Specified amounts of zircon powder with a mean particle size of  $1 \mu\text{m}$  and carbon powder were measured out and were thoroughly mixed in a dry condition. After the addition of a small amount of organic binder (PVA), the mixture was formed into many cylindrical briquettes of 15 mm in diameter and 30 mm in height by a molding machine using a metallic mold. For producing stabilized zirconia powder, a specified amount of  $\text{CaO}$  and  $\text{Y}_2\text{O}_3$  was blended with zircon powder and carbon

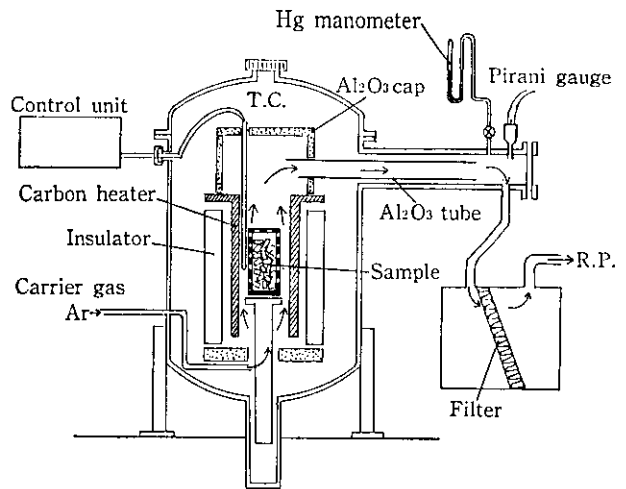


Fig. 3 Schematic representation of vacuum furnace for heating under reduced pressure

powder, thereby preparing similar briquettes. These briquettes were subjected to heat treatment under reduced pressure in the vacuum furnace illustrated in Fig. 3.

The vacuum furnace shown in Fig. 3 was obtained by modifying a resistance heating type vacuum furnace using a carbon heater for the present experiment. The space above the top of the heater is covered with an alumina crucible, and an alumina tube passes sideways through the wall of the crucible. A filter is installed in the exhaust tube midway between alumina tube and rotary pump (R.P.) to recover ultrafine  $\text{SiO}$  powder formed by the gaseous-phase condensation of  $\text{SiO}(\text{g})$ . Briquettes composed of zircon powder and carbon powder were heat-treated at temperature of 1 500 to 2 000 K under a 10 to  $10^5$  Pa (1 atm) reduced pressure in an argon atmosphere. The heating rate was 300 K/h and the holding time was 2 h. Furnace cooling was conduct-

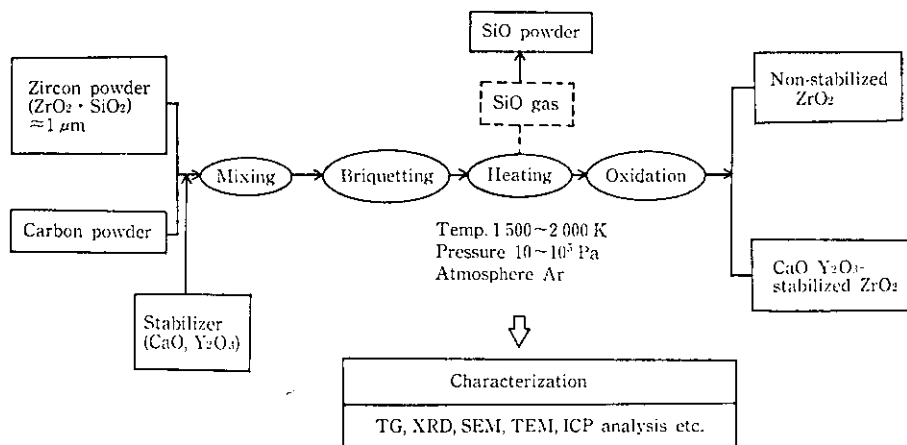


Fig. 2 Experimental procedure

ed after holding. As shown in Fig. 3, argon gas was injected into the furnace to control the pressure of the atmosphere and to carry away generated SiO(g) and the ultrafine SiO powder formed from it.

The briquetted mixture of zircon powder and carbon powder becomes a very brittle coke-like lump containing small amounts of ZrO and ZrC after the heat treatment under reduced pressure. The briquette is then oxidized in the air at about 1 073 K. The purpose of this oxidation is to transform ZrO and ZrC into ZrO<sub>2</sub>, thereby finally obtaining nonstabilized or stabilized pure zirconia powder. The grayish-black coke-like lump is transformed into white fine powder by this oxidation.

#### 4.1.2 Method of evaluation

Effects of the mixing ratio of zircon and carbon, conditions for heat treatment, and addition of a stabilizer on the production of zirconia powder by carbothermic reduction under reduced pressure were investigated by thermogravimetric analysis (TGA) and X-ray diffraction (XRD). In addition, the characterization of the obtained zirconia powder and ultrafine SiO powder as the by-product was conducted by grain size analysis, observation under a scanning electron microscope (SEM) and a transmission electron microscope (TEM), and chemical analysis using high-frequency induction coupled plasma spectroscopic analysis (ICP).

### 4.2 Results of Experiment and Discussion

#### 4.2.1 Thermogravimetric analysis (TGA)

Results of the thermogravimetric analysis are shown in Fig. 4. Samples with a C/SiO<sub>2</sub> molar ratio of 1.0 were heat-treated in an argon atmosphere at two levels of pressure, i.e., reduced pressure (10<sup>2</sup> Pa) and 1 atm (10<sup>5</sup> Pa). The heating rate was 10 K/min. During heating and after reaching a temperature of 1 923 K, changes in weight were continuously measured. Weight

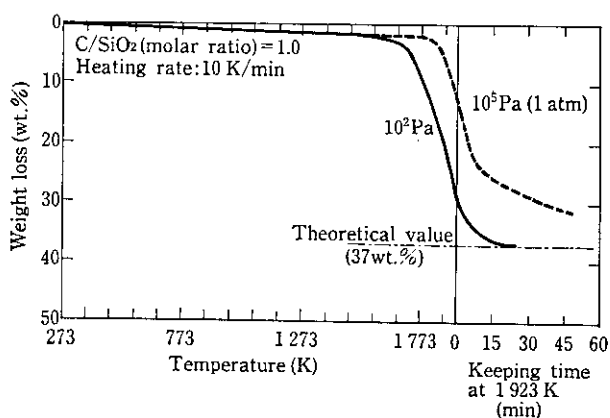


Fig. 4 Weight change of zircon/carbon mixture during heating

losses occur with the generation of SiO(g) and CO(g) according to the reaction formula (1). As is apparent from Fig. 4, the weight loss begins at a temperature of about 1 773 K under reduced pressure of 10<sup>2</sup> Pa, whereas it starts at about 1 623 K in the case of 10<sup>2</sup> Pa. Thus, it is understood that the reaction starting temperature is considerably lower under reduced pressure. The weight loss ratio at a holding temperature of 1 923 K is 37% at 10<sup>2</sup> Pa. This value is finally equal to the theoretical weight loss ratio resulting from the reaction given by Eq. (1). In contrast, the weight loss is about 31% at 10<sup>5</sup> Pa even after an elapse of 45 min at 1 923 K, suggesting that the reaction is not completed. From the above-mentioned results, it is apparent that the heat treatment under reduced pressure is effective in accelerating the reaction.

The effects of the pressure of the atmosphere on the weight loss caused by the heat treatment under reduced pressure were investigated. The results are shown in Fig. 5. The heat treatment was conducted at two levels of 1 723 K and 1 873 K, and the effect of the addition of stabilizers (CaO, Y<sub>2</sub>O<sub>3</sub>) was also investigated. As is apparent from Fig. 5, it is necessary to ensure the degrees of vacuum higher than 10<sup>3</sup> Pa at 1 723 K and higher than 10<sup>4</sup> Pa at 1 873 K in order to obtain weight loss ratios higher than 37%, which is the theoretical weight loss ratio when SiO<sub>2</sub> is completely dissociated from zircon. Large amounts of residual zircon were observed in all cases where samples were heat-treated at 10<sup>5</sup> Pa (1 atm) at low weight loss ratios. When the heat treatment is conducted at degrees of vacuum higher than 10<sup>3</sup> Pa, the weight loss ratio exceeds 37% and reaches as high as 40%. This is because ZrO or ZrC is formed according to the reaction (2) or (4), respectively. In addition, Fig. 5 reveals an important fact that the presence of stabilizers (CaO, Y<sub>2</sub>O<sub>3</sub>) has very little effect on

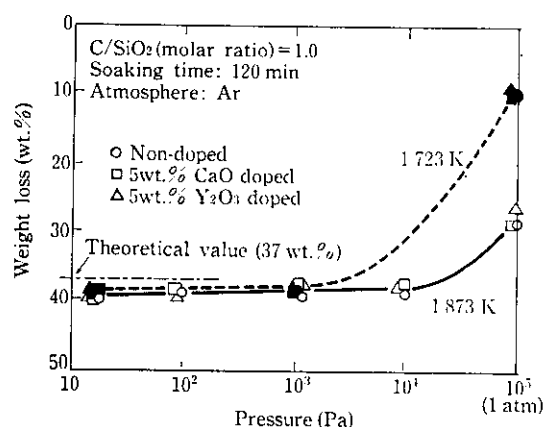


Fig. 5 Effect of pressure during heating and addition of stabilizer on weight loss of zircon/carbon mixture

the weight loss. This means that stabilized zirconia can be produced by quite the same process as with nonstabilized zirconia simply by changing the composition of the raw material mix.

#### 4.2.2 Amount of residual SiO<sub>2</sub>

The effects of the C/SiO<sub>2</sub> molar ratio on the SiO<sub>2</sub> content in zirconia powder oxidized after the heat treatment under reduced pressure were investigated. The results are shown in Fig. 6. At C/SiO<sub>2</sub> < 1.0, the SiO<sub>2</sub> content increases because zircon remains owing to an insufficient amount of reducing agent. At C/SiO<sub>2</sub> > 1.5, however, over-reduction occurs and a Zr-Si alloy is formed although zircon does not remain. In this case, the oxidation treatment results in an increase in the SiO<sub>2</sub> content. The optimum C/SiO<sub>2</sub> molar ratio for obtaining zirconia with a minimum SiO<sub>2</sub> content ranges from 1.0 to 1.3. The SiO<sub>2</sub> content of zirconia powder obtained from a composition with a C/SiO<sub>2</sub> molar ratio of about 1.0 is 0.05% or less, and this indicates that dissociation of SiO<sub>2</sub> from zircon is almost completed.

#### 4.2.3 Stabilization

Figure 7 shows X-ray diffraction patterns of stabilized zirconia powder of C/SiO<sub>2</sub> molar ratio of 1.0 and 1.3 and with an addition of 7 wt.% CaO after the heat treatment under reduced pressure and the oxidation treatment. When the C/SiO<sub>2</sub> molar ratio is 1.3, the cubic ZrO-ZrC solid solution formed after the heat treatment under reduced pressure becomes a monoclinic zirconia phase as a result of the oxidation treatment, and only cubic zirconia powder containing the monoclinic system is obtained. On the other hand, when the C/SiO<sub>2</sub> molar ratio is 1.0, the amount of formed ZrO-ZrC solid solution is very small, and single-phase cubic zirconia is formed after oxidation. To obtain fully stabilized cubic zirconia powder not containing monoclinic, therefore, it is necessary to control C/SiO<sub>2</sub> to about 1.0.

### 5 Characterization

#### 5.1 Evaluation of Characteristics of Zirconia Powder

Table 2 gives examples of chemical analysis of various

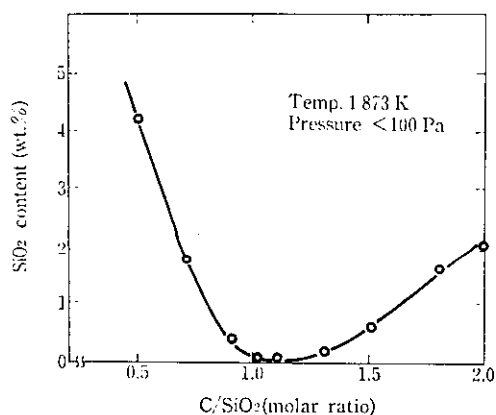


Fig. 6 Effect of C/SiO<sub>2</sub> (molar ratio) on SiO<sub>2</sub> content in nonstabilized ZrO<sub>2</sub> powder after heating under reduced pressure

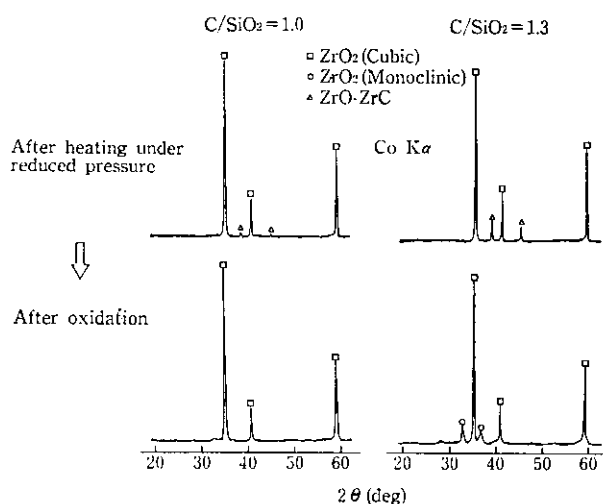


Fig. 7 XRD patterns of CaO fully stabilized ZrO<sub>2</sub> powder

types of zirconia powder obtained by the above-mentioned production method and of the raw material zircon powder. As is apparent from the table, the ZrO<sub>2</sub> (+CaO, Y<sub>2</sub>O<sub>3</sub>) contents of zirconia powders obtained by this production method are about 99.8% and the contents of impurities such as SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> are all lower than 0.1%. Thus, these purities of the zirconia

Table 2 Examples of chemical analysis

Material	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	P <sub>2</sub> O <sub>5</sub>	Na <sub>2</sub> O	CaO	Y <sub>2</sub> O <sub>3</sub>	ZrO <sub>2</sub> +HfO <sub>2</sub>
Non-stabilized ZrO <sub>2</sub>	0.039	0.033	0.11	0.005	<0.001	<0.001	0.011	—	—
CaO-fully stabilized ZrO <sub>2</sub>	0.092	0.052	0.13	0.011	<0.001	<0.001	6.2	—	—
Y <sub>2</sub> O <sub>3</sub> -partially stabilized ZrO <sub>2</sub>	0.047	0.035	0.12	0.007	<0.001	<0.001	0.010	5.3	—
Zircon powder (ZrO <sub>2</sub> ·SiO <sub>2</sub> )* <sup>1</sup>	32.5	0.28	0.080	0.072	0.059	0.002	0.007	—	66.3

\*<sup>1</sup> Starting material

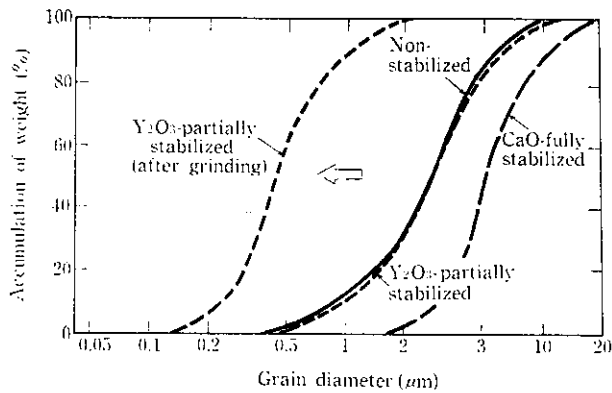


Fig. 8 Particle size distribution of various kinds of ZrO<sub>2</sub> powder

powders thus obtained are almost equal to those obtained by the wet method shown in Table 1, which is a method for obtaining high-purity zirconia powder. A comparison with the analytical values of the raw material zircon powder reveals an interesting fact that the contents of impurities in zirconia powders, such as Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, P<sub>2</sub>O<sub>5</sub> and Na<sub>2</sub>O, are very low compared with those of zircon powder. This means that these oxides are reduced during the heat treatment under reduced pressure to form metals of high vapor pressure and such suboxides as Al<sub>2</sub>O and FeO, which are vaporized and removed. The heat treatment under reduced

pressure contributes not only to the acceleration of the dissociation of SiO<sub>2</sub> from zircon, but also to greatly obtaining zirconia powder with high purity. This is one of the important features of this production method.

Figure 8 shows results of a measurement of particle size distribution of various types of zirconia powder by the laser scattering method (microtrack particle size analyzer). Fine zirconia powder with 50% mean particle sizes of 5 μm or less can be obtained easily by crushing after the oxidation treatment. It is also possible to produce ultrafine zirconia powder containing 3 mol% Y<sub>2</sub>O<sub>3</sub> as the raw material powder for high-strength and high-toughness sintered zirconia body by wet grinding. An SEM image of partially stabilized ultrafine zirconia powder containing 3 mol% Y<sub>2</sub>O<sub>3</sub>, thus obtained after wet grinding, is shown in Photo 1.

After compacting this ultrafine zirconia powder using a metallic mold press, the compacts were sintered in the air under various temperature to produce a sintered zirconia body, and the bending strength was measured at room temperature. The effect of the sintering temperature on the bending strength is shown in Fig. 9. A high-strength sintered body of 100 kgf/mm<sup>2</sup> or more is obtained by conducting sintering at temperatures higher than 1 450°C. Thus, it is found that the zirconia powder obtained by this production method is very promising as the raw material powder for high-strength sintered products.

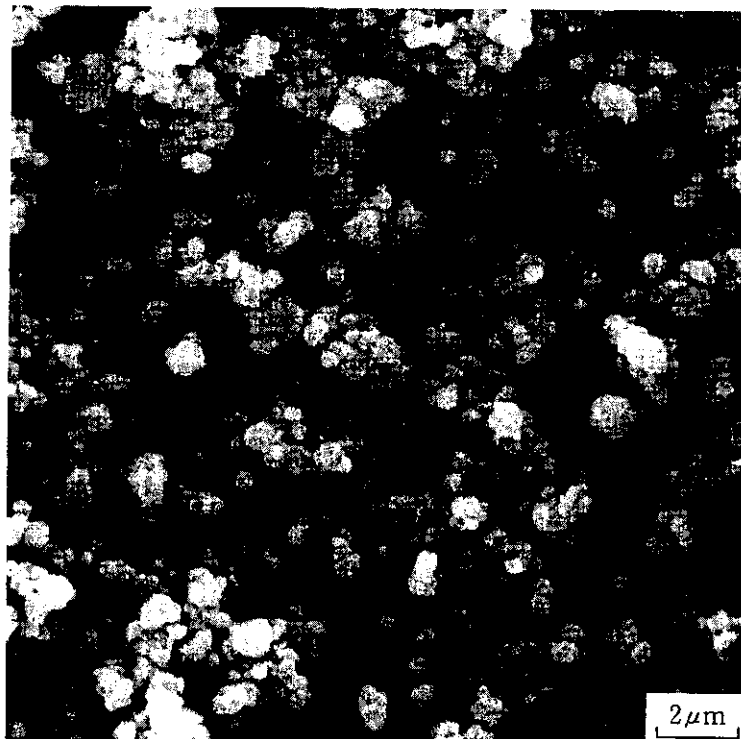


Photo 1 SEM photograph of Y<sub>2</sub>O<sub>3</sub>-partially stabilized powder after wet grinding

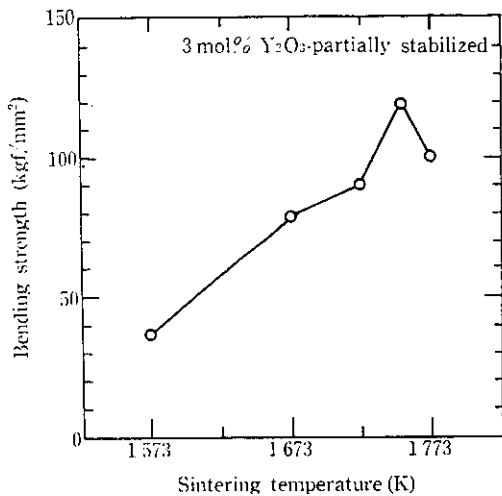


Fig. 9 Bending strength of sintered body prepared from 3 mol%  $Y_2O_3$ -partially stabilized  $ZrO_2$  powder

### 5.2 Ultrafine SiO Powder as By-Product

One of the features of this production method is that ultrafine SiO powder is obtained as a by-product. The SiO(g), generated during the heat treatment under reduced pressure using the vacuum furnace shown in Fig. 3, is carried up by the argon gas injected as the carrier gas to the upper part of the furnace and is rapidly cooled there to form ultrafine SiO powder. This ultrafine powder is carried by the argon gas into a filter box on the exhaust side of a rotary pump and is recovered there. The amount of formed ultrafine powder calculated by the reaction formula (1) is about 40 parts by weight relative to 100 parts by weight of  $ZrO_2$ . TEM images of the ultrafine SiO powder thus obtained are shown in Photo 2. It is found that the obtained powder has primary particles of 50 nm or less in diameter and is very fine. This powder is yellowish-brown and is found to be amorphous when examined by X-rays. It becomes a purely white amorphous powder when oxidized in the air at about 1 073 K. The ultrafine powder obtained by this production method is identified as SiO because of its increase in weight owing to the oxidation treatment, and because the purely white powder after the oxidation treatment shows exactly the same infrared absorption spectrum as that of amorphous  $SiO_2$  commercially available or shown in literature.<sup>6,7)</sup>

The characteristics of the ultrafine SiO powder obtained by this production method are shown in Table 3. This ultrafine SiO powder has high activity, with its purities as high as 99.5% or more. Therefore, this powder is promising as the raw material for other ceramic powders such as  $Si_3N_4$ <sup>8)</sup> and SiC.

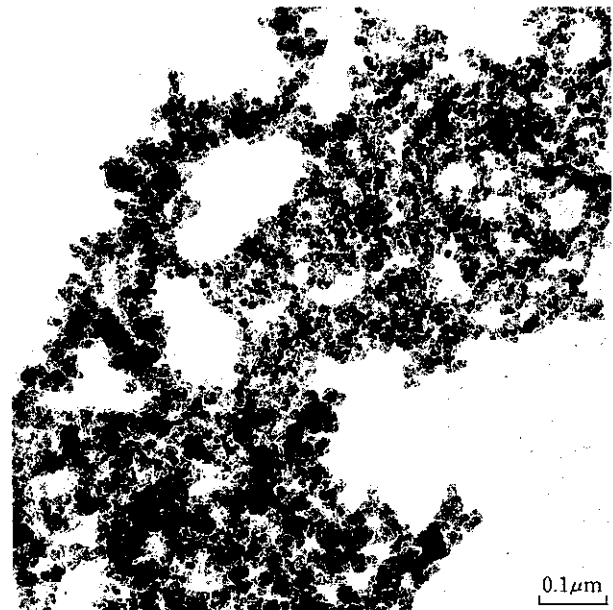
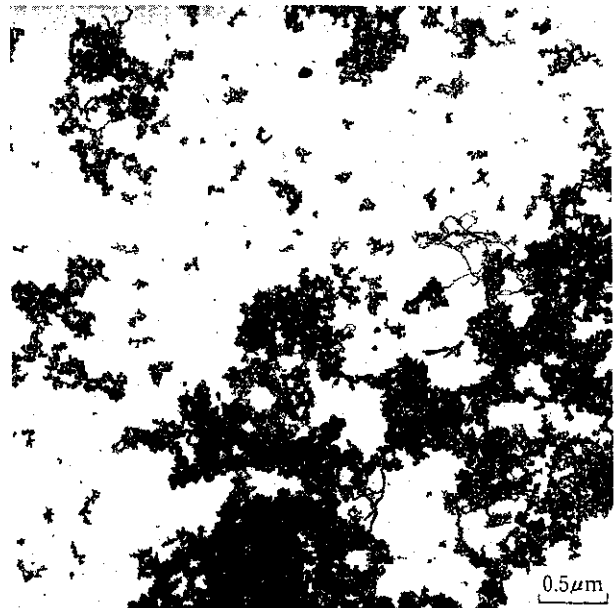


Photo 2 TEM photographs of SiO ultrafine powder

Table 3 Characteristics of SiO ultrafine powder

Color tone	Yellowish-brown
Particle size	< 50 nm
Specific area	100 ~ 300 m <sup>2</sup> /g
(→Calculated particle size)	(10 ~ 24 nm)
Bulk density	40 ~ 90 g/l
Purity	> 99.5% SiO <sub>2</sub> *

\* After heat treatment for oxidation at 1 073 K in air



## 6 Conclusions

It was found that high-purity zirconia powder can be produced by a new production method by which zircon ( $\text{ZrO}_2 \cdot \text{SiO}_2$ ) powder is heat-treated under reduced pressure in the presence of carbon powder. An investigation was made into the effects of the composition of the raw material mix, conditions for heat treatment under reduced pressure, etc. on the formation of zirconia by the carbothermic reduction reaction of zircon under reduced pressure. The characteristics of various types of zirconia powder and ultrafine SiO powder as a by-product of this production method were also evaluated. The following results were obtained:

- (1) The reaction proceeds as a whole according to the reaction (1),  $\text{ZrO}_2 \cdot \text{SiO}_2(\text{s}) + \text{C}(\text{s}) \rightarrow \text{ZrO}_2(\text{s}) + \text{SiO}(\text{g}) + \text{CO}(\text{g})$ , and the generation of SiO(g) resulting from this reaction is substantially accelerated by the heat treatment under reduced pressure compared with the treatment at 1 atm.
- (2) The optimum composition of the raw material mix for obtaining single-phase zirconia powder containing little SiO<sub>2</sub> is within the range of 1.0 to 1.3 expressed by the molar ratio of the SiO<sub>2</sub> in zircon to carbon (C/SiO<sub>2</sub>).
- (3) By adding a stabilizer, such as CaO and Y<sub>2</sub>O<sub>3</sub>, to zircon and carbon, it is also possible to produce stabilized zirconia powder by quite the same manufacturing process as with nonstabilized zirconia powder.
- (4) The purity of obtained zirconia powder is about 99.8% and the contents of impurities such as Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and P<sub>2</sub>O<sub>5</sub> are 0.1% or less. Thus, the purity of zirconia powder obtained is almost equal to that of zirconia powder produced by the wet method. The heat treatment under reduced pressure is also effective

in obtaining high purities by the vaporization of impurities.

- (5) The particle size of obtained zirconia powder is as fine as 5 μm or less. By wet grinding, it is also possible to produce ultrafine partially stabilized zirconia powder of 1 μm or less containing Y<sub>2</sub>O<sub>3</sub> as the raw material for high-strength and high-toughness sintered products. High-strength sintered products with bending strength of 100 kgf/mm<sup>2</sup> can be obtained.
- (6) The SiO(g) generated during the heat treatment under reduced pressure becomes a fine amorphous powder as a result of the condensation of the gaseous phase. The ultrafine powder of this by-product has primary particle sizes as small as 50 nm or less in diameter and provides high purity and activity. Therefore, this ultrafine by-product powder is promising as the raw material for other ceramic powders such as Si<sub>3</sub>N<sub>4</sub> and SiC.

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