Abridged version

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

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A new production method for high-purity ZrO2 powder has been developed. Non-stabilized ZrO2 powder high purity can be produced by heating a mixture of zircon (ZrO2·SiO2) and carbon powders under reduced pressure. Stabilized ZrO2 powders with high purity can also be obtained by adding a stabilizer such as CaO or Y2O3 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 ZrO2 powder produced by this method is about 99.8%, and the average grain size is smaller than 5μm. Fine Y2O3-partially-stabilized ZrO2 powder (<1μm) can also be obtained by wet grinding for use as the raw material for a ZrO2 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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Synopsis:

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1 Introduction

Zirconia (ZrO₂) is an oxide of high melting point (2988 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 Y₂O₃ 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 toughness 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 (Si₃N₄), 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.¹,²

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. 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 ZrSiO₄ or ZrO₂ · SiO₂. 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.

\[ \text{ZrO}_2 \cdot \text{SiO}_2(s) + \text{C}(s) \rightarrow \text{ZrO}_2(s) + \text{SiO}(g) + \text{CO}(g) \]  \hspace{1cm} (1)

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

\[ \text{ZrO}_2 \cdot \text{SiO}_2(s) + 2\text{C}(s) \rightarrow \text{ZrO}(s) + \text{SiO}(g) + 2\text{CO}(g) \]  \hspace{1cm} (2)

\[ \text{ZrO}_2 \cdot \text{SiO}_2(s) + 3\text{C}(s) \rightarrow \text{Zr}(s) + \text{SiO}(g) + 3\text{CO}(g) \]  \hspace{1cm} (3)

\[ \text{ZrO}_2 \cdot \text{SiO}_2(s) + 4\text{C}(s) \rightarrow \text{ZrC}(s) + \text{SiO}(g) + 3\text{CO}(g) \]  \hspace{1cm} (4)

With respect to these reactions, the temperature-\( P_{\text{CO}} \) equilibrium curve of a stable phase, formed by the reaction to the carbon of SiO₂ or ZrO₂ following to Eqs. (5) to (7), was determined with the aid of the thermodynamic data of JANAF.\(^3\)

\[ \text{SiO}_2(s) + \text{C}(s) \rightarrow \text{SiO}(g) + \text{CO}(g) \]  \hspace{1cm} (5)

\[ \text{ZrO}_2(s) + 2\text{C}(s) \rightarrow \text{Zr}(s) + 2\text{CO}(g) \]  \hspace{1cm} (6)

\[ \text{ZrO}_2(s) + 3\text{C}(s) \rightarrow \text{ZrC}(s) + 2\text{CO}(g) \]  \hspace{1cm} (7)

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 \( P_{\text{SO}} = P_{\text{CO}} \), 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 Zr:C region and that the Zr:C is inevitably formed at the same time with the formation of ZrO₂. In order to suppress the formation of ZrC to ensure single-phase ZrO₂, 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 SiO₂ in zircon to C, C/SiO = 1.0, and that a

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

<table>
<thead>
<tr>
<th>Method</th>
<th>Outline of production method</th>
<th>Characteristics of powder</th>
<th>Main uses</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry</td>
<td>Electric arc fusion method</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Zircon + Carbon → Arc fusion</td>
<td>Purity (~99%)</td>
<td>Refractories</td>
</tr>
<tr>
<td></td>
<td>Baddeleyite</td>
<td></td>
<td>Pigments</td>
</tr>
<tr>
<td></td>
<td>(CaO)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>NaOH + H₂O → Non-stabilized ZrO₂</td>
<td>Purity (~99%)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Zircon → Plasma arc</td>
<td></td>
<td>Pigments</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Plasma arc</td>
<td>method</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Zircon → Alkali fusion → Na₂ZrO₄</td>
<td>High purity (&gt;99.5%)</td>
<td>Advanced ceramics</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Electrical ceramics</td>
</tr>
<tr>
<td>Wet</td>
<td>Alkali fusion method</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Glass composition</td>
</tr>
</tbody>
</table>

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small amount of formed ZrC should be transformed into ZrO₂ 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 μm 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 CaO and Y₂O₃ was blended with zircon powder and carbon 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 SiO powder formed by the gaseous-phase condensation of SiO(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 10³ 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-
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 ZrO₂, 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/SiO₂ molar ratio of 1.0 were heat-treated in an argon atmosphere at two levels of pressure, i.e., reduced pressure (10⁶ Pa) and 1 atm (10¹² Pa). The heating rate was 10 K/min. During heating and after reaching a temperature of 1923 K, changes in weight were continuously measured. Weight 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 10⁶ Pa, whereas it starts at about 1 623 K in the case of 10¹² 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 10⁶ 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 10⁶ Pa even after an elapsed time 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, Y₂O₃) was also investigated. As is apparent from Fig. 5, it is necessary to ensure the degrees of vacuum higher than 10⁹ Pa at 1 723 K and higher than 10⁸ Pa at 1 873 K in order to obtain weight loss ratios higher than 37%, which is the theoretical weight loss ratio when SiO₂ is completely dissociated from zircon. Large amounts of residual zircon were observed in all cases where samples were heat-treated at 10⁶ Pa (1 atm) at low weight loss ratios. When the heat treatment is conducted at degrees of vacuum higher than 10⁸ 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, Y₂O₃) has very little effect on

![Fig. 4 Weight change of zircon/carbon mixture during heating](image)

![Fig. 5 Effect of pressure during heating and addition of stabilizer on weight loss of zircon/carbon mixture](image)
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 SiO₂

The effects of the C/SiO₂ molar ratio on the SiO₂ content in zirconia powder oxidized after the heat treatment under reduced pressure were investigated. The results are shown in Fig. 6. At C/SiO₂ < 1.0, the SiO₂ content increases because zircon remains owing to an insufficient amount of reducing agent. At C/SiO₂ > 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 SiO₂ content. The optimum C/SiO₂ molar ratio for obtaining zirconia with a minimum SiO₂ content ranges from 1.0 to 1.3. The SiO₂ content of zirconia powder obtained from a composition with a C/SiO₂ molar ratio of about 1.0 is 0.05% or less, and this indicates that dissociation of SiO₂ from zircon is almost completed.

4.2.3 Stabilization

Figure 7 shows X-ray diffraction patterns of stabilized zirconia powder of C/SiO₂ 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/SiO₂ molar ratio is 1.3, the cubic ZrO₂-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/SiO₂ molar ratio is 1.0, the amount of formed ZrO₂-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/SiO₂ to about 1.0.

5 Characterization

5.1 Evaluation of Characteristics of Zirconia Powder

Table 2 gives examples of chemical analysis of various 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 ZrO₂ (+CaO, Y₂O₃) contents of zirconia powders obtained by this production method are about 99.8% and the contents of impurities such as SiO₂, Al₂O₃, and Fe₂O₃ are all lower than 0.1%. Thus, these purities of the zirconia

<table>
<thead>
<tr>
<th>Material</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>TiO₂</th>
<th>Fe₂O₃</th>
<th>P₂O₅</th>
<th>Na₂O</th>
<th>CaO</th>
<th>Y₂O₃</th>
<th>ZrO₂+HfO₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>Non-stabilized ZrO₂</td>
<td>0.039</td>
<td>0.053</td>
<td>0.11</td>
<td>0.005</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td>0.011</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>CaO-fully stabilized ZrO₂</td>
<td>0.092</td>
<td>0.052</td>
<td>0.13</td>
<td>0.011</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td>6.2</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Y₂O₃-partially stabilized ZrO₂</td>
<td>0.047</td>
<td>0.035</td>
<td>0.12</td>
<td>0.007</td>
<td>&lt;0.001</td>
<td>&lt;0.001</td>
<td>0.010</td>
<td>5.3</td>
<td>—</td>
</tr>
<tr>
<td>Zircon powder (ZrO₂-SiO₂)*1</td>
<td>32.5</td>
<td>0.28</td>
<td>0.080</td>
<td>0.072</td>
<td>0.059</td>
<td>0.002</td>
<td>0.007</td>
<td>—</td>
<td>66.3</td>
</tr>
</tbody>
</table>

*1 Starting material

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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 Al₂O₃, Fe₂O₃, P₂O₅ and Na₂O, 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 Al₂O and FeO, which are vaporized and removed. The heat treatment under reduced pressure contributes not only to the acceleration of the dissociation of SiO₂ 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% Y₂O₃ 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% Y₂O₃, 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/mm² or more is obtained by conducting sintering at temperatures higher than 1450°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.
Fig. 9  Bending strength of sintered body prepared from 3 mol% Y$_2$O$_3$-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 1073 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.6,7

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$_3$N$_4$ and SiC.

Table 3  Characteristics of SiO ultrafine powder

<table>
<thead>
<tr>
<th>Characteristic</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Color tone</td>
<td>Yellowish-brown</td>
</tr>
<tr>
<td>Particle size</td>
<td>&lt;80 nm</td>
</tr>
<tr>
<td>Specific area</td>
<td>100~300 nm$^2$/g</td>
</tr>
<tr>
<td>(→Calculated particle size)</td>
<td>(10~24 nm)</td>
</tr>
<tr>
<td>Bulk density</td>
<td>40~90 g/l</td>
</tr>
<tr>
<td>Purity</td>
<td>&gt;99.5% SiO$_2$</td>
</tr>
</tbody>
</table>

* After heat treatment for oxidation at 1073 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 \(\text{ZrO}_2 \cdot \text{SiO}_2(s) + C(s) \rightarrow \text{ZrO}_2(s) + \text{SiO}(g) + \text{CO}(g)\), and the generation of \(\text{SiO}(g)\) 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 \(\text{SiO}_2\) is within the range of 1.0 to 1.3 expressed by the molar ratio of the \(\text{SiO}_2\) in zircon to carbon \((\text{C}/\text{SiO}_2)\).

3. By adding a stabilizer, such as \(\text{CaO}\) and \(\text{Y}_2\text{O}_3\), 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 \(\text{Al}_2\text{O}_3\), \(\text{Fe}_2\text{O}_3\), and \(\text{P}_2\text{O}_5\) 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 \(\mu\text{m}\) or less. By wet grinding, it is also possible to produce ultrafine partially stabilized zirconia powder of 1 \(\mu\text{m}\) or less containing \(\text{Y}_2\text{O}_3\) as the raw material for high-strength and high-toughness sintered products. High-strength sintered products with bending strength of 100 kgf/mm\(^2\) can be obtained.

6. The \(\text{SiO}(g)\) 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 \(\text{Si}_{3}\text{N}_4\) and \(\text{SiC}\).

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