

# Titanium powder production by preform reduction process (PRP)

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## Abstract

To develop an effective process for titanium powder production, a new preform reduction process (PRP), based on the calciothermic reduction of preform containing titanium oxide ( $\text{TiO}_2$ ), was investigated. The feed preform was fabricated from slurry, which was made by mixing  $\text{TiO}_2$  powder, flux (e.g.  $\text{CaCl}_2$ ) and binder. Various types of preforms in the form of plates, spheres, or tubes were prepared using a conventional technique, and the fabricated preform was sintered at 1073 K before reduction in order to remove the binder and water. The sintered solid preform containing  $\text{TiO}_2$  was then placed in a stainless steel container, and reacted with calcium vapor at a constant temperature ranging between 1073 and 1273 K for 6 h. Titanium powder was recovered from the reduced preform by leaching it with acid. As a result, pure titanium powder with 99 mass% purity was obtained. This process was found to be suitable for producing a homogeneous fine powder when the composition of flux and the size of the preform are controlled.

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## 1. Introduction

The application of titanium has been widening and the demand for titanium has also been increasing in recent years as it is a lightweight, extremely strong, and highly corrosion resistant metal. Titanium is currently produced by means of a metallothermic reduction process called the Kroll process [1]. In this process, titanium tetrachloride ( $\text{TiCl}_4$ ) is reduced by metal magnesium in an airtight metallic reactor at about 1100 K (Fig. 1(a)). The titanium deposit sticks to the reactor wall and grows in the form of massive sponge. As a result, it is rather difficult to recover product titanium. No continuous commercial process has been successfully established so far; and currently batch-type processes are employed for mass production. The chemical reaction during the Kroll process is a huge exothermic reduction, and producing titanium on a large scale takes several days [2].

To overcome the drawback of the current Kroll process, Fray et al. conducted extensive research on the direct electrochemical reduction of titanium dioxide ( $\text{TiO}_2$ ) to obtain high

purity titanium in molten calcium chloride ( $\text{CaCl}_2$ ) [3–6]. As schematically shown in Fig. 1(b), the FFC process, developed by Chen et al. [3], is being carried out on a larger scale. Their activity not only inspired academic materials research, but has also energized related industries all over the world. Currently, Ono and Suzuki are also developing a commercial process for titanium production by means of the calciothermic reduction of  $\text{TiO}_2$  in molten  $\text{CaCl}_2$  [7–9]. Their process, called the OS process, also involves the electrolysis of molten  $\text{CaCl}_2$ – $\text{CaO}$  [7]. The features of the FFC [3] and OS [8] processes and the differences between them are described elsewhere [10]. The application of an electronically mediated reaction (EMR) [11,12] to the titanium reduction process is also under investigation [12]. This method has the potential to prevent impurities from accumulating in metal deposits [13], and can increase energy efficiency when combined with conventional molten salt electrolysis (MSE) of  $\text{CaCl}_2$ – $\text{CaO}$  molten salt for the production of calcium alloy reductants.

The direct reduction processes of titanium oxide mentioned above have the potential to be the next generation titanium production processes. However, there are some drawbacks which have to be overcome before the process can be commercialized. Specifically, a large amount of molten salt is required, and the reduction process is slow.

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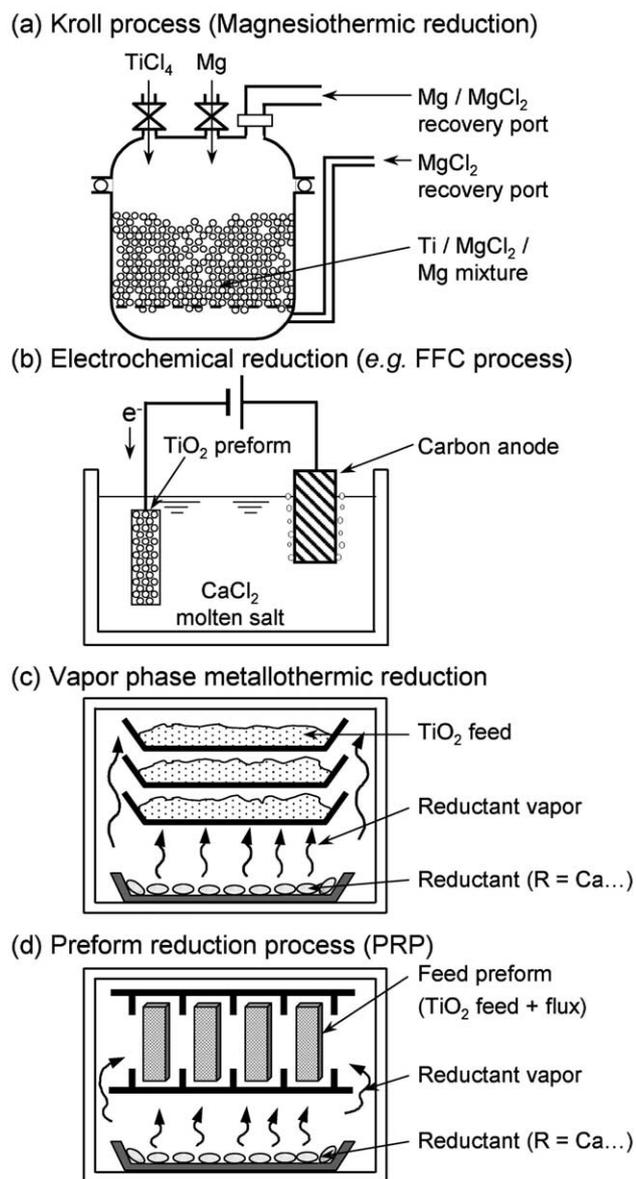


Fig. 1. Various titanium reduction processes.

Furthermore, the process lacks the flexibility to be scaled up, as it is based on electrochemical reduction. With this background, a new reduction process for producing fine metal powder by means of the metallothermic reduction of preform containing feed material was investigated in this study.

## 2. Preform reduction process (PRP)

When producing titanium by means of the metallothermic reduction of titanium oxide, the feed ( $\text{TiO}_2$ ) and reductant (Ca) are often mixed and heated to elevated temperatures. The overall reaction for this process is



This calciothermic reduction of  $\text{TiO}_2$  is a huge exothermic reduction process, and in general it is difficult to achieve a homogeneous reaction. When the reductant calcium comes into physical contact with  $\text{TiO}_2$  and reacts with it, the impurities in the reductant, such as nitrogen and carbon, are transferred to the metal phase, and contaminate the titanium metal. To avoid this problem, calcium vapor is often used to reduce  $\text{TiO}_2$  [14,15]. A vapor phase metallothermic reduction, as schematically shown in Fig. 1(c), is effective in preventing contamination from reductant, and it is possible to produce titanium powder with purity greater than 99%. However, this process is still very sensitive to contamination from reactor material, as the  $\text{TiO}_2$  feed is placed on the substrate material. Furthermore, it is difficult to scale up the process because it is difficult to facilitate a homogeneous reaction when reducing large amounts of feed material in a single reactor.

To develop an effective process for the titanium powder production, a new reduction process using feed preform was investigated in this study. As schematically shown in Fig. 1(d), this process is based on the metallothermic reduction of preform, which contains feed material [16]. Advantages of this process are its effective control of purity and morphology, and its flexible scalability. Contamination from the reaction container or reductant can easily be avoided because the feed material in the self-supporting preform does not come into physical contact with them. The process is highly flexible in terms of scalability as it is possible to simultaneously treat multiple pieces of preform in a single reduction chamber. Furthermore, the amount of chemicals such as  $\text{CaCl}_2$  can be reduced, compared to the direct electrochemical reduction processes mentioned before.

## 3. Experimental

Fig. 2 shows the material flowchart for the preform reduction process using calcium vapor as a reductant that was investigated in this study. The experiment consists of three major steps: preform fabrication, reduction by calcium vapor, and titanium powder recovery by leaching.

The feed preform was fabricated from slurry, which was made by mixing  $\text{TiO}_2$  powder, flux (e.g.  $\text{CaCl}_2$ ) and a binder. The purity and form of the starting materials used in this study are listed in Table 1. About 3–15 g of  $\text{TiO}_2$  powder (99.97% purity, Al: < 0.001%, Fe: < 0.001%, Cl: < 0.011% by mass, average particle size: 0.2  $\mu\text{m}$ ) was mixed with the flux and binder using a stirrer, and a homogeneous slurry was synthesized. About 0.5–6 g of  $\text{CaCl}_2$ , or CaO powder was employed as flux. A collodion solution, which is a mixture of 5 mass% nitrocellulose in ethanol and ether, was used as a binder solution. The viscosity of the slurry was controlled by varying the amount of flux and binder. The obtained slurry was then cast into a stainless steel mold and a preform plate of 3–10 mm thickness was produced. In some experiments,

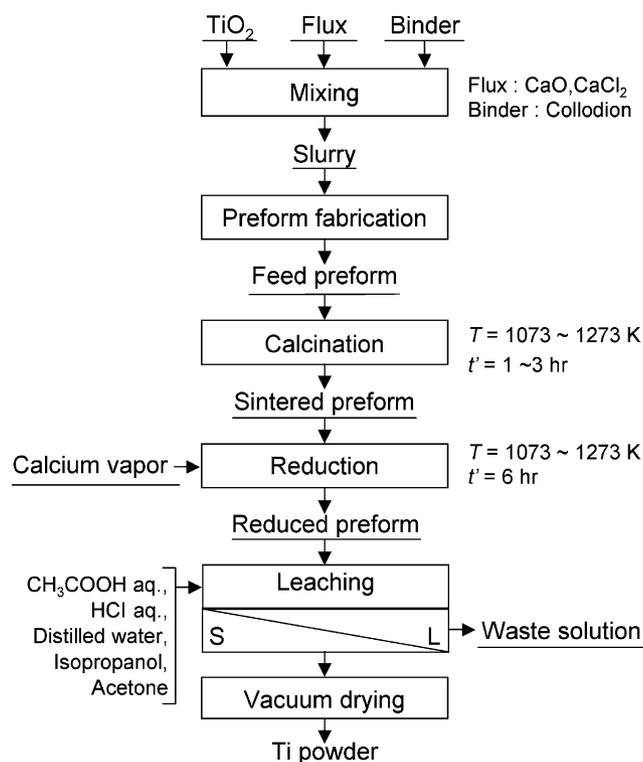


Fig. 2. Experimental procedures of titanium powder production by means of calciothermic reduction of oxides using a preform reduction process (PRP).

preform in a tubular shape or a spherical shape, 5–20 mm in diameter, was fabricated using slurry with high viscosity. Various types of preforms in the form of plates, balls, or tubes were prepared, and the fabricated preform was heated at 1073 K before reduction in order to remove the binder and water.

Fig. 3 is a schematic illustration of the experimental apparatus for producing titanium powder by means of the PRP. The sintered solid preform containing  $\text{TiO}_2$  was placed in a stainless steel vessel, and about 20 g of calcium shots were placed at the bottom. The calcium was physically isolated from the feed preform, and its vapor was

Table 1  
Starting materials used in this study

| Materials                | Form     | Purity or conc. (%) | Supplier                      |
|--------------------------|----------|---------------------|-------------------------------|
| $\text{TiO}_2$           | Powder   | 99.97               | Toho Titanium                 |
| $\text{CaO}$             | Powder   | 99.5 up             | Kanto Chemicals               |
| $\text{CaCl}_2$          | Powder   | 99.5 up             | Kanto Chemicals               |
| Collodion <sup>a</sup>   | Solution | 5.0 <sup>b</sup>    | Wako Pure Chemical Industries |
| Ca                       | Chip     | 98.0 up             | Mintech Japan                 |
| Ti                       | Sponge   | 98.0 up             | Toho Titanium                 |
| $\text{CH}_3\text{COOH}$ | Aqueous  | 99.7                | Kanto Chemicals               |
| HCl                      | Aqueous  | 35.0 <sup>b</sup>   | Kanto Chemicals               |

<sup>a</sup> 5 mass% nitrocellulose, 23.75 mass% ethanol, 71.25 mass% diethylether solution.

<sup>b</sup> Concentration of the solution.

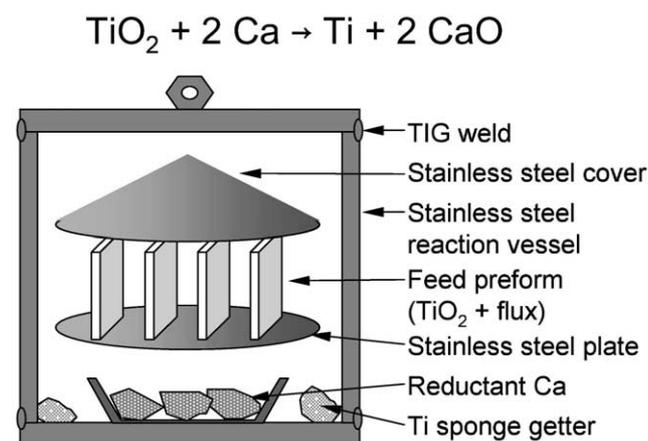


Fig. 3. Schematic illustration of the experimental apparatus for producing titanium powder by means of the preform reduction process (PRP).

supplied to the feed preform at unit activity through the gas phase while heating. As shown in Fig. 3, four to ten pieces of preform were installed in a thick-walled stainless steel reaction vessel, and were sealed by tungsten inert gas (TIG) welding. A sponge titanium was also placed at the bottom of the vessel for gettering nitrogen gas in the system. The sealed reaction vessel was then heated in an electric furnace maintained at a constant temperature ranging between 1073 and 1273 K, and the preform was reacted with calcium vapor. Some representative experimental conditions of the reduction experiments are summarized and listed in Table 2. After 6 h of reaction, the reaction vessel

Table 2  
Experimental condition of the preform reduction process<sup>a</sup>

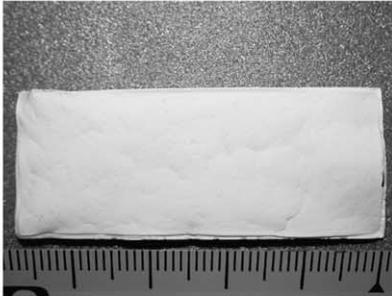
| Exp. no. | Mass of sample $w_i$ (g) |                   |                 |                  | Cationic molar ratio $R_{\text{Cat.}/\text{Ti}}$ <sup>b</sup> | Reduction temperature $T_{\text{red}}$ (K) |
|----------|--------------------------|-------------------|-----------------|------------------|---|--|
|          | Feed $\text{TiO}_2$      | Flux $\text{CaO}$ | $\text{CaCl}_2$ | Binder Collodion |   |  |
| A        | 4.99                     | 0.44              | –               | 6.02             | 0.1   | 1273                                       |
| B        | 3.93                     | 1.60              | –               | 6.32             | 0.5   | 1273                                       |
| C        | 3.11                     | 2.10              | –               | 5.50             | 1   | 1273                                       |
| D        | 5.40                     | –                 | 0.47            | 10.50            | 0.1   | 1273                                       |
| E        | 4.11                     | –                 | 1.97            | –                | 0.5   | 1273                                       |
| F        | 3.05                     | –                 | 2.83            | 11.15            | 1   | 1273                                       |
| G        | 5.00                     | –                 | –               | 5.00             | 0   | 1273                                       |
| H        | 3.98                     | 1.40              | –               | 4.63             | 0.5   | 1073                                       |
| I        | 3.98                     | 1.40              | –               | 4.63             | 0.5   | 1173                                       |
| J        | 3.98                     | 1.40              | –               | 4.63             | 0.5   | 1273                                       |
| K        | 3.97                     | –                 | 1.89            | 6.85             | 0.5   | 1073                                       |
| L        | 3.97                     | –                 | 1.89            | 6.85             | 0.5   | 1173                                       |
| M        | 3.97                     | –                 | 1.89            | 6.85             | 0.5   | 1273                                       |
| N        | 3.97                     | –                 | –               | 4.85             | 0   | 1073                                       |
| O        | 3.97                     | –                 | –               | 4.85             | 0   | 1173                                       |
| P        | 3.97                     | –                 | –               | 4.85             | 0   | 1273                                       |

<sup>a</sup> Preform was calcined at 1073 K for 1 h and reacted with Ca for 6 h.

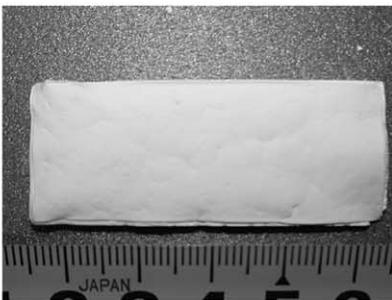
<sup>b</sup> Cationic molar ratio,  $R_{\text{Cat.}/\text{Ti}} = N_{\text{Cat.}}/N_{\text{Ti}}$ , where  $N_{\text{Cat.}}$  and  $N_{\text{Ti}}$  are the mole amounts of the cation in the flux and titanium, respectively.

was taken out of the furnace, and quenched in water. The preforms in the container were mechanically recovered at room temperature, and subjected to the following leaching process.

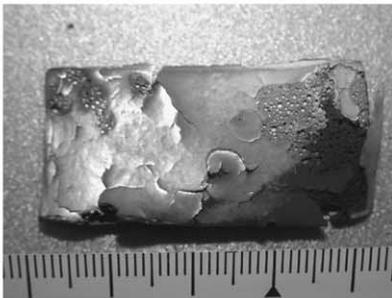
(a) Fabricated feed preform



(b) After calcination



(c) After reduction



(d) After leaching

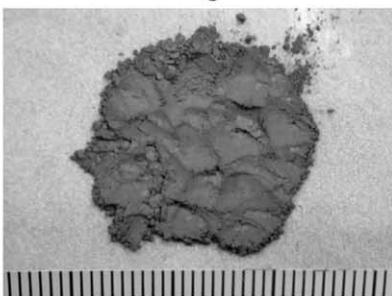


Fig. 4. (a) Fabricated platy feed preform (flux,  $\text{CaCl}_2$ ; binder, Collodion). (b) Preform after calcination (temperature: 1073 K, holding time: 1 h). (c) Preform after reduction (temperature, 1273 K; holding time, 6 h). (d) Obtained titanium powder after leaching (exp. no. M, Ti, 99.88%; Fe, 0.10%; Ni, 0.02%; analyzed by EDS).

The titanium powder in the preform obtained after the reduction experiment was recovered by leaching the preform with acid. The reaction product ( $\text{CaO}$ ), flux, and excess reductant in the sample were removed by dissolution in acetic acid solution. The obtained metal powder was rinsed in a hydrochloric acid at room temperature. It was then rinsed with distilled water, alcohol, and acetone, and then dried in a vacuum.

A chemical analysis of the sample was carried out using an inductively coupled plasma-atomic emission spectroscopy (ICP-AES; Seiko Instruments, SPS 4000). The metal powder was observed using scanning electron microscopy (SEM, Jeol, JSM-5600LV), and the composition of the sample was determined both by an energy dispersive X-ray spectrometer (EDS, Jeol, JED-2200) and ICP-AES. Phases in the sample were identified using an X-ray diffraction analysis

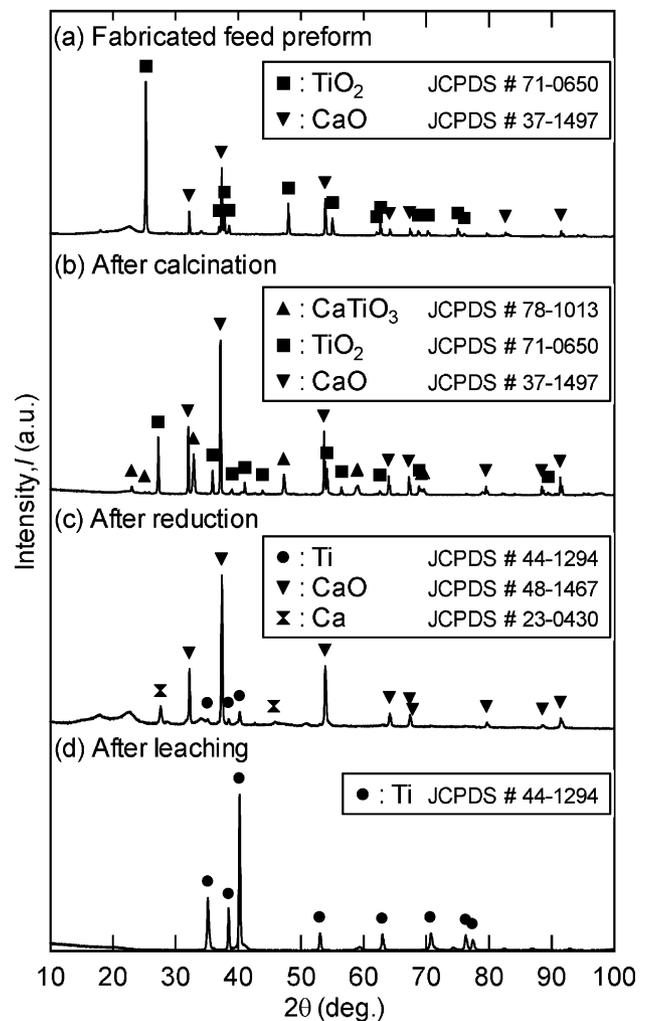


Fig. 5. X-ray diffraction patterns of the samples. (a) Fabricated platy feed preform (flux,  $\text{CaO}$ ; binder: Collodion). (b) Preform after calcination (temperature, 1073 K; holding time, 1 h). (c) Preform after reduction (temperature, 1273 K; holding time, 6 h). (d) Titanium powder obtained after leaching (exp. no. C, Ti, 99.88%; Fe, 0.10%; Ni, 0.02%; analyzed by EDS).

Table 3

Analytical results of the titanium powder obtained by means of the preform reduction process

| Exp. no. | Flux              | Cationic molar ratio $R_{\text{Cat./Ti}}^{\text{a}}$ | Reduction temperature $T_{\text{red.}}$ (K) | Impurity content, $C_i^{\text{b}}$ (mass% ) |                         |                       |                      |                        |                          |
|----------|-------------------|--|---|---|-------------------------|-----------------------|----------------------|------------------------|--------------------------|
|          |                   |  |   | Titanium $C_{\text{Ti}}$                    | Calcium $C_{\text{Ca}}$ | Oxygen $C_{\text{O}}$ | Iron $C_{\text{Fe}}$ | Nickel $C_{\text{Ni}}$ | Chromium $C_{\text{Cr}}$ |
| A        | CaO               | 0.1  | 1273  | 99.66                                       | 0.33                    | (0.00)                | 0.00                 | 0.00                   | 0.01                     |
| B        | CaO               | 0.5  | 1273  | 99.62                                       | 0.11                    | (0.00)                | 0.12                 | 0.15                   | 0.00                     |
| C        | CaO               | 1  | 1273  | 99.80                                       | 0.03                    | (0.00)                | 0.23                 | 0.17                   | 0.00                     |
|          |                   |  |   |   | 0.03 <sup>c</sup>       | 0.28 <sup>d</sup>     | 0.10 <sup>c</sup>    | 0.09 <sup>c</sup>      | 0.66 <sup>c</sup>        |
| D        | CaCl <sub>2</sub> | 0.1  | 1273  | 97.75                                       | 1.07                    | (1.03)                | 0.00                 | 0.14                   | 0.00                     |
| E        | CaCl <sub>2</sub> | 0.5  | 1273  | 99.17                                       | 0.38                    | (0.00)                | 0.00                 | 0.33                   | 0.11                     |
| F        | CaCl <sub>2</sub> | 1  | 1273  | 99.84                                       | 0.07                    | (0.00)                | 0.09                 | 0.00                   | 0.01                     |
| G        | No flux           | 0  | 1273  | 97.11                                       | 1.71                    | (1.08)                | 0.11                 | 0.00                   | 0.00                     |
| H        | CaO               | 0.5  | 1073  | 87.15                                       | 2.94                    | (9.69)                | 0.06                 | 0.00                   | 0.17                     |
| I        | CaO               | 0.5  | 1173  | 88.73                                       | 7.61                    | (3.41)                | 0.00                 | 0.24                   | 0.00                     |
| J        | CaO               | 0.5  | 1273  | 99.22                                       | 0.51                    | 0.00                  | 0.23                 | 0.04                   | 0.03                     |
| K        | CaCl <sub>2</sub> | 0.5  | 1073  | 88.79                                       | 6.40                    | (4.62)                | 0.16                 | 0.00                   | 0.03                     |
| L        | CaCl <sub>2</sub> | 0.5  | 1173  | 99.92                                       | 0.02                    | (0.00)                | 0.00                 | 0.06                   | 0.00                     |
|          |                   |  |   |   | 0.04 <sup>c</sup>       | 0.66 <sup>d</sup>     | 0.01 <sup>c</sup>    | 0.01 <sup>c</sup>      | < 0.01 <sup>c</sup>      |
| M        | CaCl <sub>2</sub> | 0.5  | 1273  | 99.88                                       | 0.00                    | (0.00)                | 0.10                 | 0.02                   | 0.00                     |
|          |                   |  |   |   | < 0.01 <sup>c</sup>     | 0.57 <sup>d</sup>     | < 0.01 <sup>c</sup>  | 0.02 <sup>c</sup>      | < 0.01 <sup>c</sup>      |
| N        | No flux           | 0  | 1073  | 82.48                                       | 0.68                    | (16.32)               | 0.52                 | 0.00                   | 0.00                     |
| O        | No flux           | 0  | 1173  | 78.47                                       | 0.16                    | (21.20)               | 0.07                 | 0.00                   | 0.10                     |
| P        | No flux           | 0  | 1273  | 86.41                                       | 9.31                    | (3.94)                | 0.17                 | 0.00                   | 0.16                     |

<sup>a</sup> Cationic molar ratio,  $R_{\text{Cat./Ti}} = N_{\text{Cat.}}/N_{\text{Ti}}$ , where  $N_{\text{Cat.}}$  and  $N_{\text{Ti}}$  are the mole amounts of cation in the flux and titanium, respectively.

<sup>b</sup> Analyzed by EDS. Values in parenthesis are listed for reference.

<sup>c</sup> Determined by ICP analysis.

<sup>d</sup> Determined by inert gas fusion-infrared absorption spectroscopy (LECO).

(XRD, Rigaku, Rint 2000). Oxygen analysis was carried out by inert gas fusion–infrared absorption spectroscopy (LECO).

#### 4. Results and discussion

By regulating the amount of binder, it was possible to control the viscosity of the slurry and obtain preform in different shapes. A preform plate, 3–6 mm thick could easily be produced by casting the slurry into the mold. Fig. 4(a) shows fabricated platy preform, 50 × 20 × 4 mm in dimension. This was made by casting slurry that was produced by mixing TiO<sub>2</sub>, CaCl<sub>2</sub> and collodion solution (exp. no. M). As shown in Fig. 4(b), the shape of the preform after calcination at 1073 K for an hour remained unchanged. Fig. 4(c) shows the preform after reduction at 1273 K for 6 h. The preform became slightly deformed after reduction, and was covered with metallic calcium. It was easy to recover the preform from the reaction vessel because it is physically isolated from it. These results indicate that this preform reduction process is suitable for separating feed material from reactor material, and it is easy to control contamination from the reaction vessel. When the preform obtained after reduction was put into an acetic acid solution, its shape was lost and titanium powder was obtained.

It was not necessary to pulverize the reduced preform before the leaching treatment. Titanium powder obtained after rinsing and drying is shown in Fig. 4(d). The EDS analysis revealed the purity of the obtained titanium powder to be 99.9 mass% Ti, 0.10 mass% Fe, and 0.02 mass% Ni (exp. no. M). A summary of the analytical results related to the powder as well as to the other samples is listed in Table 3.

Fig. 5 shows representative XRD patterns of the samples obtained during the experiment (exp. no. C). The analysis revealed that the fabricated feed preform was a mixture of TiO<sub>2</sub> and CaO, and some portion of these phases reacted to form CaTiO<sub>3</sub> after calcination. Fig. 5(c) shows an XRD pattern of the preform after reduction at 1273 K for 6 h. Excess calcium reductant was found in the mixture of titanium and CaO after reduction. One can see from Fig. 5(d) that pure titanium was produced after leaching.

By increasing the amount of flux and using CaCl<sub>2</sub> as a flux, it was possible to synthesize high-viscosity slurry, and fabricate it to form spherical or tubular-shaped preform. Fig. 6(a) shows fabricated spherical feed preform, and Fig. 6(b) shows preform that has been calcined at 1073 K for 1 h. Fig. 6(c) shows the preform that has undergone reduction at 1273 K for 6 h. One can see from the sectioned image [Fig. 6(c) right] that the reduced preform is homogeneous within the sensitivity of the optical observation. A

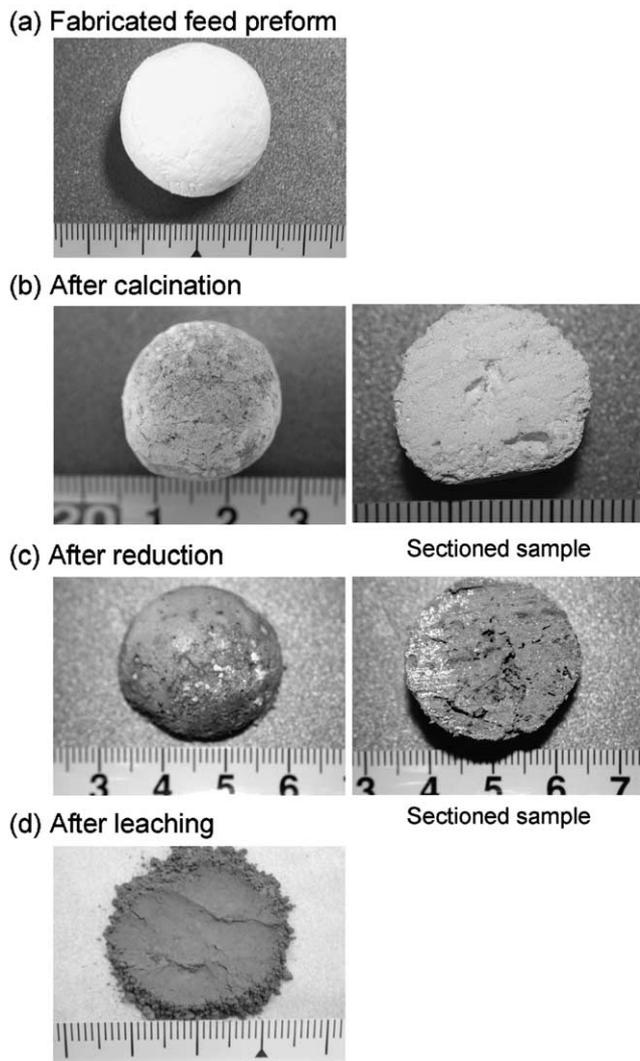


Fig. 6. (a) Fabricated spherical feed preform. (Flux,  $\text{CaCl}_2$ ; binder, Colloidion). (b) Preform after calcination (temperature, 1073 K; holding time, 1 h). (c) Preform after reduction (temperature, 1273 K; holding time, 6 h). (d) Titanium powder obtained after leaching (Ti, 99.70%; Ca, 0.06%; Fe, 0.10%; Ni, 0.10%; analyzed by EDS).

metallic phase was observed at the center of the preform, which confirms that the reduction was completed in this experimental condition. The EDS analysis revealed the purity of the obtained titanium powder after leaching to be 99.7%. Fig. 7 shows the results for the tubular preform. One can see from the sectioned preform after reduction [Fig. 7(c) right] that a uniform cake was obtained after reduction. The EDS analysis revealed the titanium powder that was obtained after leaching to be 99.9 mass%Ti, 0.04 mass%Ca, and 0.03 mass%Ni.

Fig. 8 shows SEM images of the titanium powder obtained after reduction at 1273 K for 6 h followed by leaching (exp. no. J, M, P). When flux was not used, the size of the titanium particles could be measured only in sub- $\mu\text{m}$  amounts. However, when flux was used, this size increased

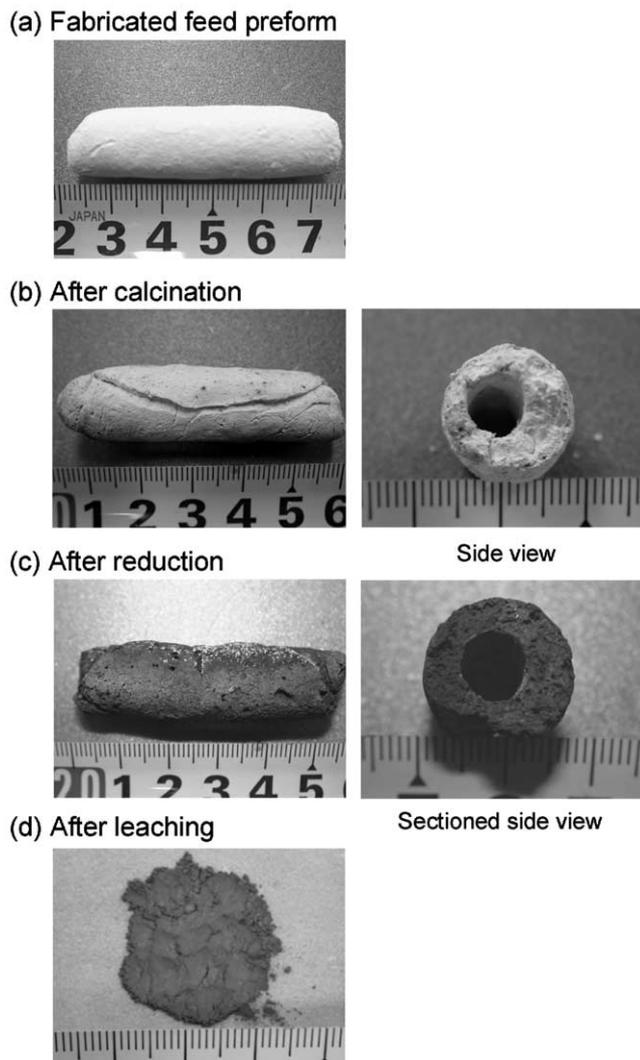


Fig. 7. (a) Fabricated tubular feed preform. (Flux,  $\text{CaCl}_2$ ; binder, Colloidion). (b) Preform after calcinations (temperature, 1073 K; holding time, 1 h). (c) Preform after reduction (temperature, 1273 K; holding time, 6 h). (d) Titanium powder obtained after leaching (Ti, 99.93%; Ca, 0.04%; Ni, 0.03%; analyzed by EDS).

to 1–2  $\mu\text{m}$ . Interestingly, it was found that homogeneous titanium powder was obtained when  $\text{CaCl}_2$  was used as flux. Furthermore, the purity of the titanium powder obtained was more than 99% when flux was used (see exp. no. A–F in Table 3), whereas it decreased when flux was not used (exp. no. G and P). The detailed mechanism of the reduction process and its influence on the morphology of the deposit are currently under investigation.

As shown in Fig. 9, the particle size of the obtained titanium powder increased as the reaction temperature increased (exp. no. K, L, and M). Analytical results of the titanium powder obtained are listed in Table 3. The purity of the sample was low when the reaction temperature was 1073 K (exp. no. H, K and N). It was found that the vapor pressure of calcium at this temperature ( $p_{\text{Ca}} = 8.5 \times 10^{-4}$  atm. at 1073

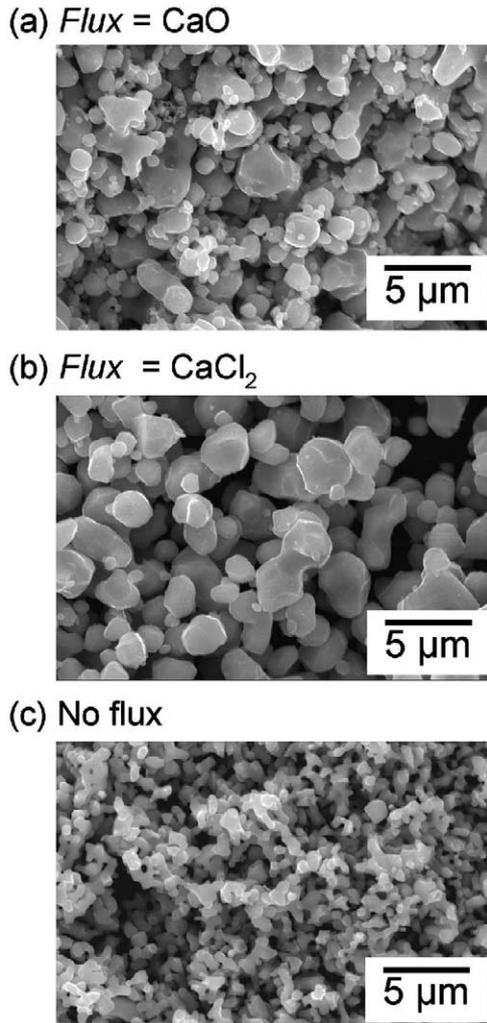


Fig. 8. SEM images of the titanium powder obtained after reduction using various fluxes. (Reduction conditions: flux = CaO, CaCl<sub>2</sub>, no flux, Cationic molar ratio,  $X_{C/Ti} = 0.5$ ; reduction temp.,  $T = 1273$  K; holding time,  $t' = 6$  h; exp. no. J, M, P).

K [17]) was too low for reduction by vapor. When the preform containing CaCl<sub>2</sub> as flux was exposed to calcium vapor at temperatures above 1173 K ( $p_{Ca} = 4.4 \times 10^{-3}$  atm. at 1173 K [17]), titanium powder with more than 99% purity by mass was successfully obtained (exp. L and M).

Fig. 10 shows an enlarged SEM image of the titanium powder obtained after reduction at 1273 K for 6 h using CaCl<sub>2</sub> as flux (exp. no. L). The LECO and ICP chemical analyses revealed that the oxygen content in the powder was 0.66 mass%, and the iron and nickel content was 0.01 mass%. In some cases in this study titanium powder with 0.28 mass% oxygen was obtained (see exp. C in Table 3). It was found that the preform reduction process was effective in producing titanium powder with 99% purity. This method can be applied to the production of various metals and alloys because it is based on a very simple metallothermic reduction.

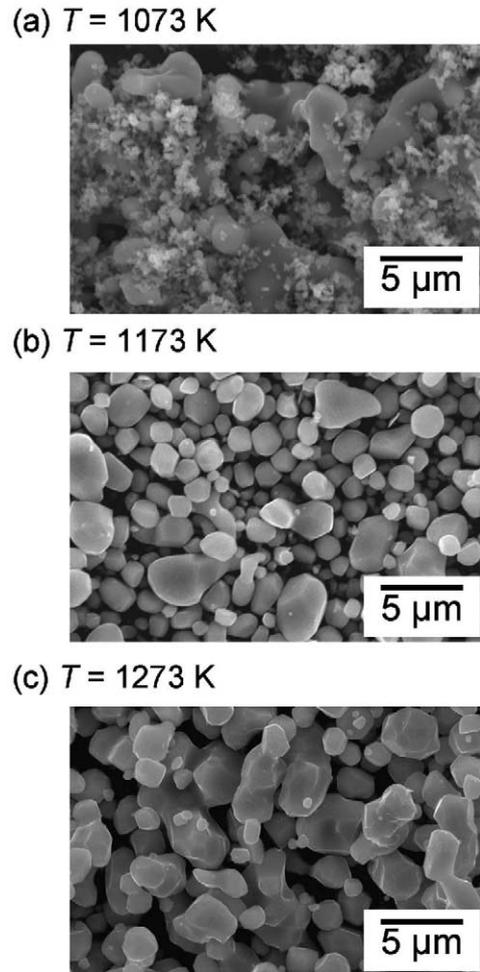


Fig. 9. SEM images of the titanium powder obtained after various reduction temperatures (reduction conditions: flux = CaCl<sub>2</sub>, cationic molar ratio,  $X_{C/Ti} = 0.5$ , reduction temp.,  $T = 1073, 1173, 1273$  K, Holding time,  $t' = 6$  h; exp. no. K, L, M).

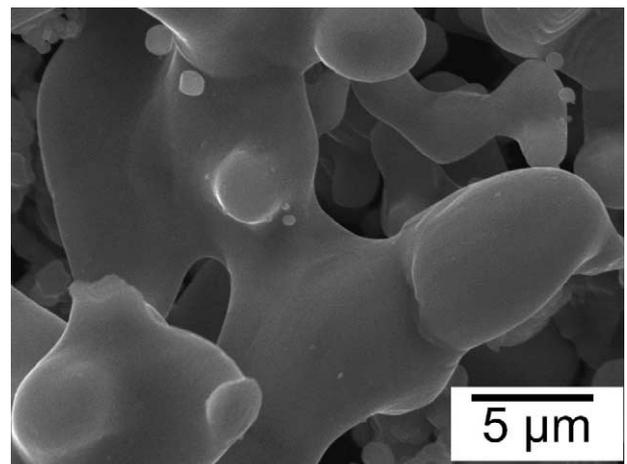


Fig. 10. SEM image of the titanium powder obtained by means of the preform reduction process (PRP). (Reduction conditions: flux = CaCl<sub>2</sub>, cationic molar ratio,  $X_{C/Ti} = 0.5$ , reduction temp.,  $T = 1273$  K, holding time,  $t' = 6$  h; exp. no. M).

## 5. Conclusion

A new reduction process for producing fine titanium powder by the metallothermic reduction of preform containing feed material was developed. Various types of preforms in the form of plates, spheres, or tubes were prepared using a conventional technique and were supplied as feed material for metallothermic reduction. The slurry obtained by mixing  $\text{TiO}_2$  powder, flux (e.g.  $\text{CaCl}_2$ ,  $\text{CaO}$  . . .) and binder (e.g. collodion), was cast into a mold and then dried to obtain the preform. Before the reduction process, the fabricated preform was heated at 1073 K in order to remove the binder and water present in it. The sintered solid preform containing  $\text{TiO}_2$  was then placed in a stainless steel container, and reacted with calcium vapor at constant temperatures ranging from 1073 to 1273 K for 6 h. The titanium powder was recovered from the reduced preform by leaching it with acid. As a result, pure titanium powder in sponge morphology with 99 mass% purity was obtained. This process was found to be suitable for producing fine, homogeneous powder when the composition of flux and the size of the preform are controlled. Another advantage of this process is purity control. Contamination from the reaction container and reductant, can easily be avoided because the feed material in the self-supporting preform is suitable for minimizing physical contact with the reaction container and reductant. Furthermore, the developed process is highly scalable as demonstrated by its ability to simultaneously treat multiple pieces of preform in a single reduction chamber.

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