

Characterization of a New Potassium Titanate, $\text{KTiO}_2(\text{OH})$ Synthesized via Hydrothermal Method

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A new potassium titanate, $\text{KTiO}_2(\text{OH})$, synthesized by hydrothermal oxidation of Ti metal powder in KOH solution was characterized. The X-ray photospectroscopy revealed that titanium contained in this compound consists of only Ti^{4+} , and FT-IR spectrum supported the existence of hydroxyl group in the structure. It was found that the titanate has a hexagonal tunnel structure with the space group $P6_322$ and lattice parameters $a = 9.8889 \text{ \AA}$ and $c = 28.6711 \text{ \AA}$, by crystal structure analysis with the use of powder XRD pattern. This agrees with the results of electron diffraction and morphology observed by SEM. The heating of $\text{KTiO}_2(\text{OH})$ above 528°C led to dehydration and phase transformation to give $\text{K}_2\text{Ti}_2\text{O}_5$.

Introduction

It is well-known that various alkali titanates represented by $\text{A}_2\text{O} \cdot n\text{TiO}_2$ (A is the alkali metal ion, $n = 2\text{--}9$) in chemical formula have their own crystal structure such as layered or tunnel type. Since these titanates show excellent ion-exchange ability,^{1–5} they have been used in applications as ion exchangers for fixing radioactive metal ions,^{6–8} as host materials for intercalation of organic compounds,^{9–13} or in the fabrication of porous materials.^{14–18} Among the alkali titanates, potassium titanates such as $\text{K}_2\text{Ti}_2\text{O}_5$, $\text{K}_2\text{Ti}_4\text{O}_9$, and $\text{K}_2\text{Ti}_6\text{O}_{13}$ have been an attraction due to their specific photochemical properties or their artificial cage type structure. The structures of $\text{K}_2\text{Ti}_2\text{O}_5$ and $\text{K}_2\text{Ti}_4\text{O}_9$ consist of layered sheets made of TiO_5 pentahedra or TiO_6 octahedra

linked with the K atoms.^{19,20} These titanates show photocatalytic activities for H_2 evolution by the water cleavage reaction even without Pt cocatalysts.^{21–23} Potassium hexatitanate $\text{K}_2\text{Ti}_6\text{O}_{13}$ is also a well-known material used as reinforcement in plastics and acts as wear resistant or heat insulator material.^{24–26}

Despite having a wide variety of applications, these potassium titanates were synthesized by a conventional solid-state and flux method. In these methods, titania powder and alkali carbonate or alkali peroxide were used as raw materials. The particle size and morphology of the products are difficult to control. With this point of view, hydrothermal synthesis is a promising method due to a large no. of parameters for operation such as temperature, time, concentration of the solution, pH, liquid–solid ratio, and additives (oxidant, chelating reagents, etc.).

Moreover, hydrothermal synthesis has more advantages as it yields highly pure, homogeneous, fine crystalline powders, or metastable phases (for example, TeI_2 ,²⁷ $\gamma\text{-CuI}$,²⁸ cubic ZrO_2 ,²⁹ etc.) at moderate conditions and at a considerably lower temperature when compared to the solid-state reaction.

In the previous work, we have reported that various potassium titanates could be produced under hydrothermal conditions by using metallic Ti powder as the raw material in short reaction time without a second-

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phase product.^{30,31} Here, in this study, the new compound $\text{KTiO}_2(\text{OH})$ was focused upon for the study of its crystal structure and physical and chemical properties.

Experimental Section

Reagents and Materials. All chemicals used were of reagent grade. Ti powder (Wako Pure Chemical Industries, Ltd.) that was used as the starting material was of 95% purity (min) with a diameter of $<45\ \mu\text{m}$. The chemical analysis of potassium hydroxide was potassium carbonate (K_2CO_3) $<1.5\%$, chloride (Cl) $<0.002\%$, phosphate (PO_4) $<0.001\%$, and sulfate (SO_4) $<0.001\%$. Water was deionized by the ion exchanger after the distillation.

Hydrothermal Synthesis of Potassium Titanate Powder. Initially, 1.0 g of Ti powder was put into a poly(tetrafluoroethylene) (Teflon) cup, then certain amounts of KOH and water were added. The cup was placed inside an autoclave of 200 cm^3 internal volume made of SUS 316 type stainless steel. After being sealed, the autoclave was set in an electric furnace at a desired temperature for 2 h. During the reaction period, the autoclave was kept at the autogenous saturation vapor pressure of the solution with stirring at 500 rpm. After the reaction, the product was separated from the solution by centrifugation and then rinsed with methanol. Finally, the sample was dried in an oven at 105 $^\circ\text{C}$ for 12 h.

Characterization. The FT-IR spectrum of the sample in a KBr pellet was measured by a JASCO FT/IR-8300 spectrophotometer with a resolution of 4 cm^{-1} . Chemical analysis was carried out by ICP-AES (SEIKO SPS-1200A) and AAS (Shimadzu AA-6500S) after dissolving the sample in aqua regia. The morphology of the product was observed by SEM (Hitachi S-430). Powder XRD data were collected on a Rigaku RINT-2500 diffractometer with graphite-monochromatized $\text{Cu K}\alpha$ radiation ($\lambda = 1.5405\ \text{\AA}$) in a step-scan mode (step interval of 0.03 $^\circ$ in 2θ) over an angular range of 10–120 $^\circ$. The structural models of the new compound were constructed with the program EXPO³² by the direct method from powder XRD patterns, and cell parameters were refined by the Rietveld method with the use of the program RIETAN-2000.³³ JEOL JEM-2000EXII was employed to carry out electron diffraction and TEM observation. Thermal analysis of the compound was performed in air at a heating rate of 10 $^\circ\text{C}/\text{min}$ with the use of a Rigaku TAS-200 thermogravimetric analyzer.

Results and Discussion

Synthesis and Determination of the Chemical Formula. The region of formation of a new potassium titanate $\text{KTiO}_2(\text{OH})$ is shown in Figure 1. The formation conditions of KOH concentration and temperature were at around 30–60 mol kg^{-1} of H_2O and 150–250 $^\circ\text{C}$, respectively. A single phase was obtained at KOH concentration of 30–40 mol kg^{-1} of H_2O and temperature of 200 $^\circ\text{C}$ or at KOH concentration of 35 mol kg^{-1} of H_2O and temperature of 150–250 $^\circ\text{C}$. The sample obtained at KOH concentration of 40 mol kg^{-1} of H_2O and 200 $^\circ\text{C}$ was used for further characterization. At first, chemical analysis of the sample was carried out. The K/Ti atomic ratio of the new potassium titanate was about 1, which showed the stoichiometric composition of this compound.

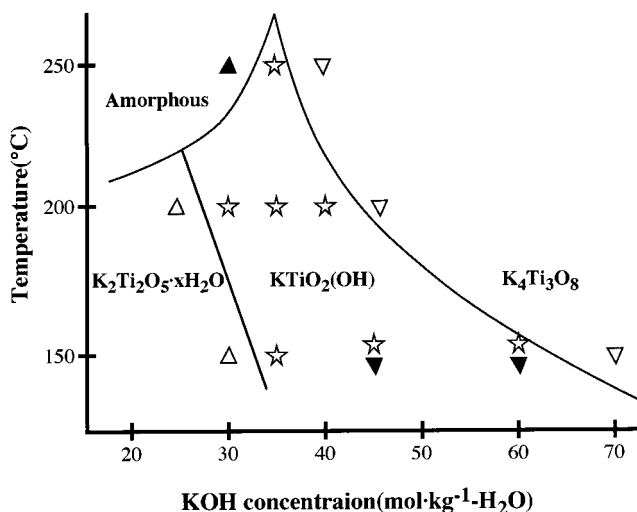


Figure 1. Diagram of the formation of $\text{KTiO}_2(\text{OH})$ in the Ti–KOH– H_2O system: ▼, Ti; ▲, amorphous; ☆, $\text{KTiO}_2(\text{OH})$; △, $\text{K}_2\text{Ti}_2\text{O}_5 \cdot x\text{H}_2\text{O}$; ∇, $\text{K}_4\text{Ti}_3\text{O}_8$.

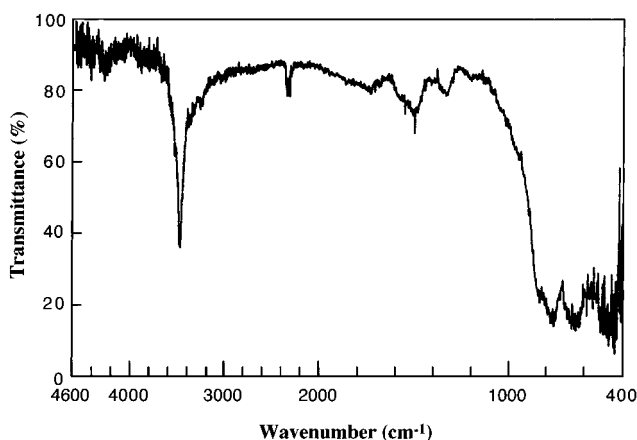


Figure 2. IR spectrum of $\text{KTiO}_2(\text{OH})$ after drying at 105 $^\circ\text{C}$ for 12 h.

The FT-IR spectrum of this compound was measured. As shown in Figure 2, a sharp peak originated from the O–H stretching vibration and appeared at 3490 cm^{-1} . By contrast, the O–H bending vibration at 1500 cm^{-1} was relatively broad. This may suggest that the absorption does not result from adsorbed water. In other words, the OH group is considered to be contained in the crystal structure and its vibration mode is restricted.

Because the sample shows slightly gray color, it was doubtful that low valency titanium ions exist in the compound. Therefore XPS was employed to evaluate the bonding condition of titanium and oxygen. The XPS spectra of Ti 2p and O 1s of the sample are shown in Figure 3. The binding energy of the Ti 2p_{3/2} orbital was 458.2 eV which is the same as TiO_2 (Figure 3a).³⁴ This result means that all titanium contained in the compound exists as tetravalent Ti^{4+} . The XPS spectrum of O 1s shown in Figure 3b has a shoulder at higher binding energy. The main peak and the shoulder peak were 529.9 and 531.7 eV respectively. The former agreed very well with the binding energy of oxygen in TiO_2 .³⁵ Thus, it is considered that the existence of the OH group

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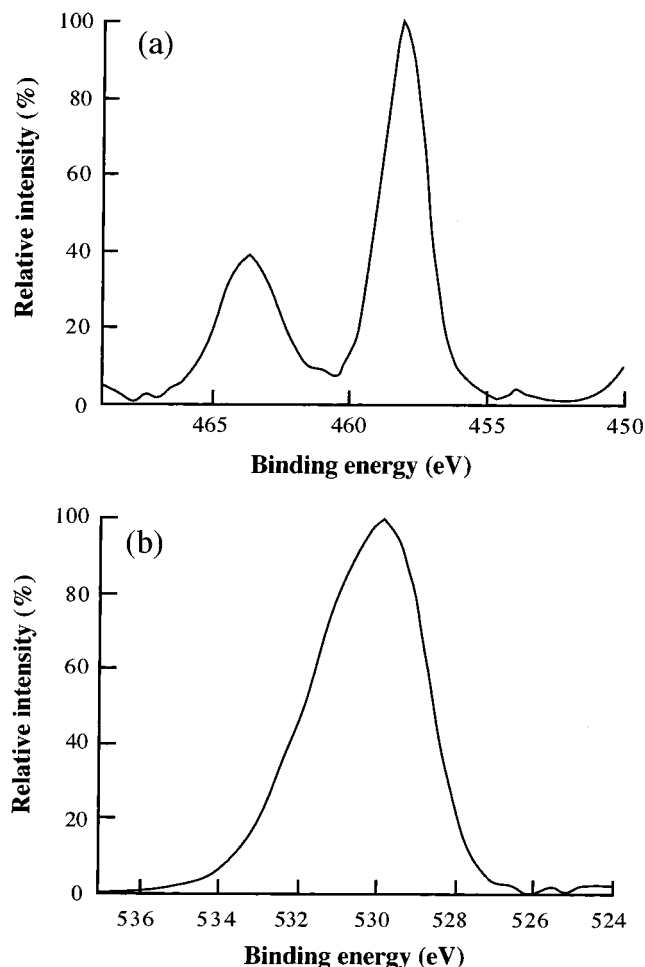


Figure 3. X-ray photoelectron spectra of KTiO₂(OH): (a) Ti 2p; (b) O 1s.

in the structure causes the shoulder peak. This phenomenon was also reported in the oxidation of titanium by water vapor.³⁶

From the results described above, we assumed the chemical composition of the new potassium titanate to be KTiO₂(OH).

Crystal Structure Analysis and Superstructure.

Morphology of the compound was observed by SEM as is shown in Figure 4. The new potassium titanate showed hexagonal columnar representation, which is 24 μm in average length.

Table 1 shows X-ray powder diffraction data of the new compound. The indexing to the data revealed that this compound belongs to a hexagonal system and space group of *P6₃/mmc* with lattice parameter about *a* = 5.71 and *c* = 9.56 Å. The result of the indexing also indicated that the sample was highly oriented along the *c*-axis and this agrees with its hexagonal columnar morphology.

Because almost all potassium titanates (K₂Ti₂O₅, K₂Ti₄O₉, K₂Ti₆O₁₃, etc.) and their derivatives take the monoclinic system, we could not find a related compound which takes the hexagonal system among the K–Ti–O compounds. Therefore, the structural model of this compound was constructed ab initio by using the direct method. Refined crystallographic and positional

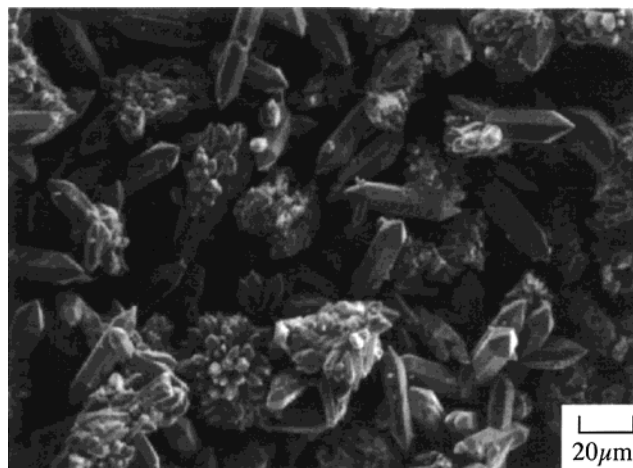


Figure 4. SEM photograph of KTiO₂(OH).

Table 1. Powder X-ray Diffraction Data of KTiO₂(OH)

<i>d</i> (Å)	<i>I</i> / <i>I</i> ₀	<i>d</i> (Å)	<i>I</i> / <i>I</i> ₀
4.95	42	1.95	44
4.79	4	1.83	6
4.40	28	1.72	6
3.44	6	1.65	14
2.85	100	1.61	22
2.68	48	1.59	2
2.45	6	1.56	4
2.39	10	1.47	4
2.35	4	1.42	22
2.26	4	1.39	6
2.20	44	1.37	4
2.15	4	1.36	4
2.12	2	1.34	4
2.05	2	1.26	8

Table 2. Crystallographic Data and Results of Refinement for KTiO₂(OH)^a

crystal system	hexagonal
space group	<i>P6₃/mmc</i> (no. 194)
<i>Z</i> (moles/unit)	4
lattice constant	<i>a</i> = 5.7096(3) Å <i>c</i> = 9.5574(4) Å
<i>R</i> _{wp}	0.162
<i>R</i> _p	0.114
<i>R</i> _i	0.067
<i>R</i> _F	0.056
<i>R</i> _{exp}	0.103

^a *R* factors are defined in ref 33.

Table 3. Fractional Coordinates and Isotropic Temperature Factors for KTiO₂(OH)^a

atom	position	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> (Å ²)
K(1)	2a	0	0	0	3.40(10)
K(2)	2d	2/3	1/3	1/4	3.28(9)
Ti	4f	1/3	2/3	0.0955(2)	0.68(4)
O(1)	6g	1/2	0	0	0.87(10)
O(2)	6h	0.1829(4)	0.3658(8)	1/4	1.11(11)

^a All occupancies are equal to 1.0.

parameters for KTiO₂(OH) are listed in Tables 2 and 3. The unit-cell structure is also visualized in Figure 5.

The resultant *R* factors converged satisfactorily (*R*_{wp} = 0.161, *R*_e = 0.103) for the oriented powder XRD pattern. Ti coordination in the unit cell consisted of TiO₆ octahedral coordination. It is seen that two octahedrons share their faces and each of the two hedrons is three-dimensionally linked with corner-sharing. Potassium ions take two kinds of position. One is located in the center of the hexagonal tunnel and the other is sur-

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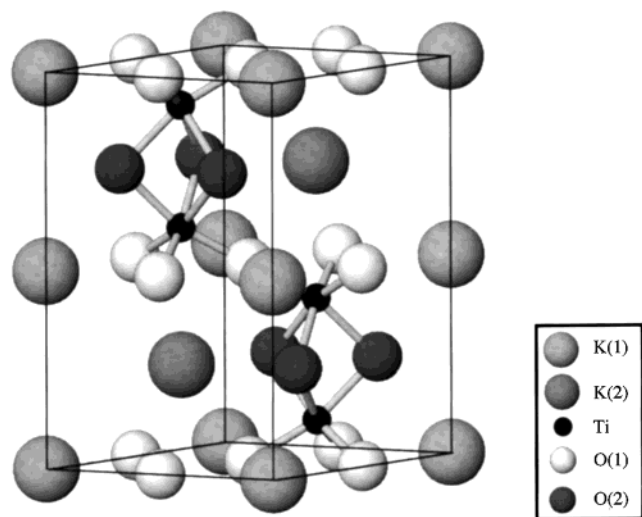


Figure 5. Unit-cell structure of the basic cell of $\text{KTiO}_2(\text{OH})$.

Table 4. Crystallographic Data and Results of Refinement for the Structure of $\text{KTiO}_2(\text{OH})^a$

crystal system	hexagonal
space group	$P6_122$ (no. 176)
Z (moles/unit)	36
lattice constant	$a = 9.8889(5) \text{ \AA}$ $c = 28.671(1) \text{ \AA}$
R_{wp}	0.141
R_p	0.104
R_i	0.076
R_F	0.064
R_{exp}	0.103

^a R factors are defined in ref 33.

Table 5. Fractional Coordinates and Isotropic Temperature Factors for the Superstructure of $\text{KTiO}_2(\text{OH})^a$

atom	position	x	y	z	$B (\text{\AA}^2)$
K(1)	6b	1.320(2)	0.6597(12)	1/12	2.98(8)
K(2)	6b	-0.0026(25)	-0.0013(13)	1/12	2.98(8)
K(3)	12c	0.3405(20)	0.3333(15)	-0.0044(4)	2.98(8)
K(4)	6a	0	0.6699(22)	1/3	2.98(8)
K(5)	6b	0.6861(23)	0.3431(12)	1/12	2.98(8)
Ti(1)	12c	-0.0018(17)	0.3349(12)	0.0955(2)	0.31(5)
Ti(2)	12c	0.9884(15)	0.3197(14)	0.2826(3)	0.31(5)
Ti(3)	12c	0.6786(13)	0.6658(14)	0.0530(4)	0.31(5)
O(1)	6b	0.3374(46)	0.1687(23)	1/12	0.04(1)
O(2)	6b	0.9931(48)	0.4965(24)	1/12	0.04(1)
O(3)	6b	1.6398(51)	0.8199(26)	1/12	0.04(1)
O(4)	12c	0.1998(29)	0.3574(28)	0.0834(13)	0.04(1)
O(5)	12c	0.0021(31)	0.1995(36)	0.0046(14)	0.04(1)
O(6)	12c	0.6693(38)	0.4905(30)	-0.0041(12)	0.04(1)
O(7)	12c	0.3448(34)	0.4891(31)	0.1696(19)	0.04(1)
O(8)	12c	0.5018(41)	0.4988(44)	0.0817(13)	0.04(1)
O(9)	6a	0.1671(42)	0.1671(42)	1/6	0.04(1)
O(10)	12c	0.5381(33)	0.6713(33)	-0.0010(13)	0.04(1)
O(11)	12c	0.8327(35)	0.1472(38)	0.0831(14)	0.04(1)

^a All occupancies are equal to 1.0, and B factors of each element are fixed as the same.

rounded by the four face-sharing octahedrons. It is expected to apply to an ion exchanger since it has a tunnel structure. The structure was classified as a 4L-type³⁶ of perovskite-like compound and isostructural to alkaline earth metamanganates.^{37,38}

Because of difficulty in the determination of the position of a light atom like hydrogen by powder X-ray

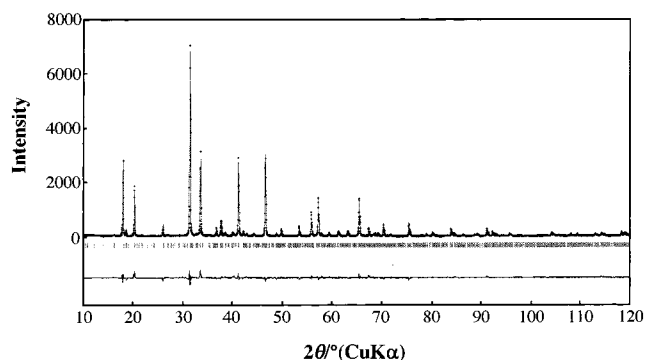


Figure 6. Observed, calculated, and difference X-ray diffraction profiles for the super structure of $\text{KTiO}_2(\text{OH})$.

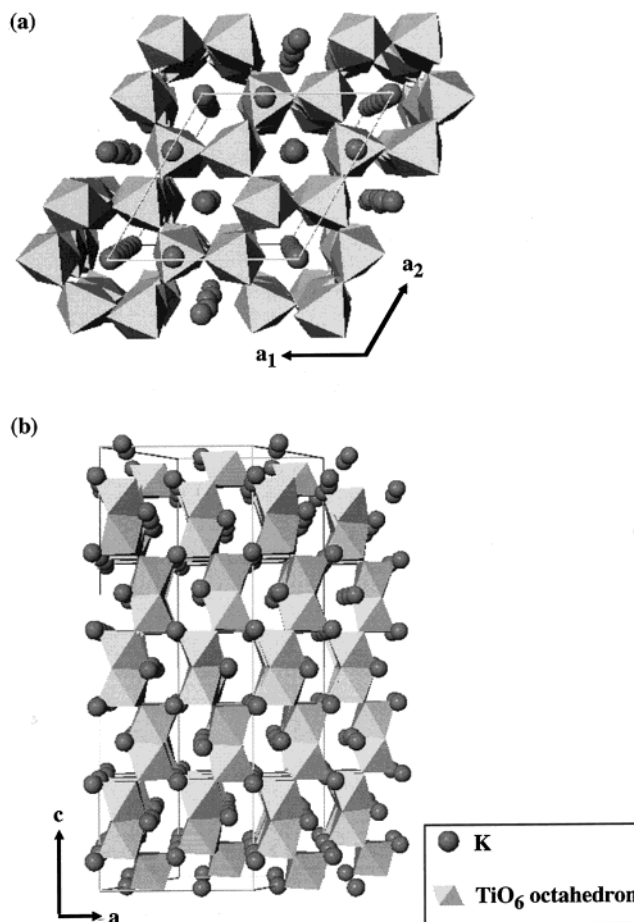


Figure 7. Perspective representation of the refined crystal structure of the super structure of $\text{KTiO}_2(\text{OH})$: (a) projection on a_1 -plane; (b) projection on ac -plane. Solid lines show the unit cell.

diffraction, the OH group was treated as an oxygen atom. The R factors were satisfactorily small to be able to suggest that the model is the crystal structure of this compound. Nevertheless, according to chemical formula of the compound, there should be 4 OH groups per 12 O atoms contained in the unit cell. In Figure 4, the O(1) atoms are involved in Ti–O–Ti corner-sharing and the O(2) atoms are involved in face-sharing. Four of 6h site oxygen atoms are considered as OH groups since the O(1) atoms are restrained in a stronger manner by the titanium ions. And it is possible that $\text{KTiO}_2(\text{OH})$ has the superstructure obtained from the long period ordering of OH groups.

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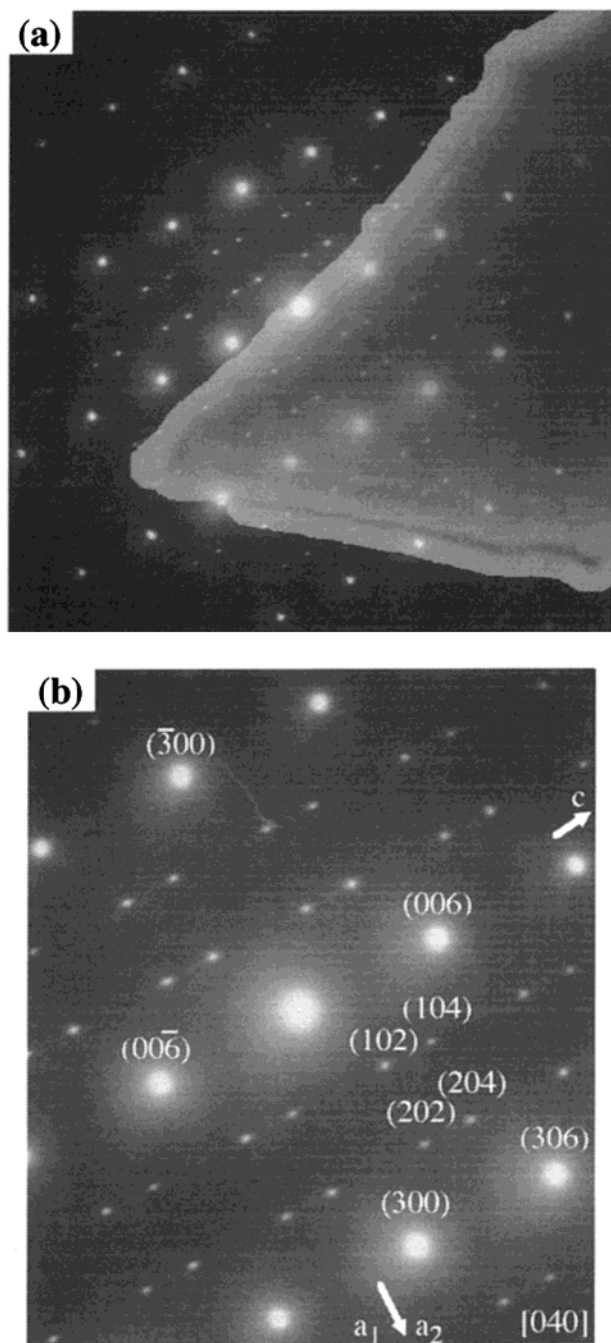


Figure 8. Typical selected area electron diffraction pattern of $\text{KTiO}_2(\text{OH})$ irradiated along $[040]$: (a) overlapped TEM image of the area; (b) image after indexing.

Therefore, structural analysis of the superstructure was carried out. Since the superstructure has a larger unit cell, the space group was set to $P6_122$ which has the lowest symmetry in the same Laue class ($6/mmm$) of $P6_3/mmc$. The superstructure was constructed by crystallographically corresponding atomic positions of the refined basic structure to the other. Rietveld refinement was also carried out by using the RIETAN-2000 program. Because of a large number of parameters per reflection (199 intensities and 34 parameters), the B_{iso} factors were fixed for each element during the refinement. The final refined cell and positional parameters are listed in Tables 4 and 5. Also, the final fitted pattern and crystal structure of the superstructure are shown in Figures 6 and 7, respectively.

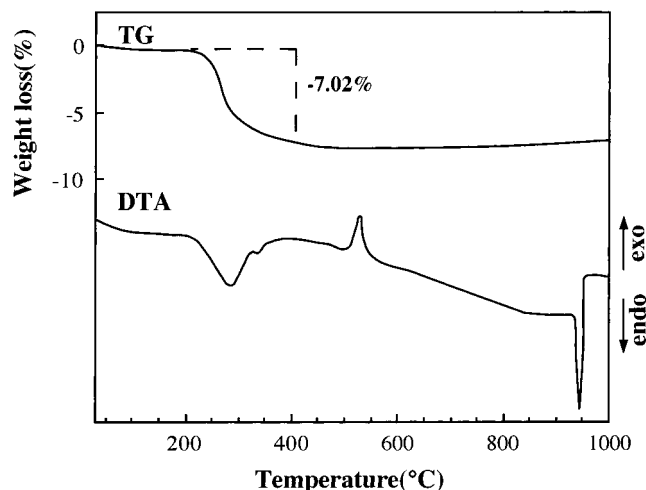


Figure 9. TG-DTA curves of $\text{KTiO}_2(\text{OH})$.

With consideration of the superstructure, the difference between the calculated and observed patterns was decreased, especially in 2θ ranges at $10\text{--}15^\circ$, $35\text{--}45^\circ$, and higher than 80° . The R factors R_{wp} and R_p could also be improved.

The superstructure of $\text{KTiO}_2(\text{OH})$ with hexagonal tunnel type structure is shown in Figure 7. It can be seen that the potassium ions in the two tunnels which are placed on the diagonal of the unit cell show subtle displacement from the center of the tunnel (Figure 7a). The displacement indicates that the oxygen atoms, O(6), O(7), and O(10), surrounding potassium ions are hydroxyl groups. This was supported with the chemical formula in view, but the B_{iso} parameter of the potassium atom was extremely large for it to be discussed in detail.

To confirm the superstructure, the electron diffraction analysis was employed. The selected area electron diffraction (SAED) pattern and the image of the area of $\text{KTiO}_2(\text{OH})$ are shown in Figure 8. The pattern was successfully indexed with the lattice parameters $a = 9.87$ and $c = 28.8 \text{ \AA}$ and agrees with the reflection conditions of $P6_122$ (Figure 8b). It is being specially mentioned that the pattern could not be perfectly indexed with the basic cell.

Thermal Property. The thermal properties of $\text{KTiO}_2(\text{OH})$ were evaluated at temperatures ranging from 20 to 1200 °C by using TG-DTA analysis. The result is shown in Figure 9. The main weight loss was observed at temperatures ranging from 200 to 400 °C. The endothermic peak at 285 °C, observed during the weight loss, corresponds to the dehydration of hydroxyl groups in $\text{KTiO}_2(\text{OH})$. The weight loss of 7.02% almost agrees with the calculated value (6.62%). Additionally, the exothermic peak, which is due to phase transformation to $\text{K}_2\text{Ti}_2\text{O}_5$, was observed at 528 °C. The endothermic peak at 948 °C corresponds to the melting point³⁹ of $\text{K}_2\text{Ti}_2\text{O}_5$, and weight loss by the vaporization of K_2O was also observed above this temperature.

Conclusion

(1) Single phase of a new potassium titanate, $\text{KTiO}_2\text{-(OH)}$, was obtained at a KOH concentration of 30–40 mol kg^{-1} of H_2O and a temperature of 150–250 °C.

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(2) From the results of the crystal structure analysis, it is revealed that $\text{KTiO}_2(\text{OH})$ has a hexagonal tunnel structure with the space group $P6_122$ and lattice parameters $a = 9.8889$ and $c = 28.6711$ Å.

(3) $\text{KTiO}_2(\text{OH})$ was dehydrated at 285 °C and transformed into $\text{K}_2\text{Ti}_2\text{O}_5$ above 528 °C.

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