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The synthesis of potassium titanate fibres by flux evaporation methods

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The synthesis of potassium titanate fibres was tried by evaporation of a flux such as Na_2O–K_2O–B_2O_3. Also, the reaction was carried out with different weight ratios of the flux to $K_2Ti_6O_{13}$ crystal powder. Potassium titanate fibres with maximum length and the minimum diameter were prepared using the weight ratio of the flux to potassium titanate powder of 0.88 and Na_2O–$3K_2O$–$5B_2O_3$ flux. Also, the diameter of the fibres tended to increase with the eliminated volume of the flux.

1. Introduction

Gier [1] reported that potassium titanate fibres ($K_2Ti_6O_{13}$) were obtained by a hydrothermal method. Berry [2] and Saito [3] described the preparation of potassium titanate from flux such as KCl–KF and $3K_2O$–$5B_2O_3$. However, they were not so successful, as the KCl–KF flux readily corroded the container and the $3K_2O$–$5B_2O_3$ flux was easily changed by heating.

Kajiwara [4] also described the production of potassium titanate from fluxes such as PbO, Bi_2O_3, K_2CO_3–$K_4P_2O_7$, K_2CO_3–V_2O_3 and PbO–$K_2P_2O_7$, respectively. In this report, we discuss the synthesis of potassium titanate fibres from fluxes such as K_2O–B_2O_3–Na_2O in order to avoid corrosion of the container.

2. Experimental procedure

2.1. Preparation of anatase (TiO_2) and potassium titanate ($K_2Ti_6O_{13}$)

100 g of $TiCl_4$ was slowly added dropwise to 1 l of water including 30 g of concentrated H_2SO_4 and then the solution was heated at 80–85° C for three hours and cooled to room temperature. The product was then separated by filtration and washed several times with the distilled water, before being dried at 110° C for 24 h. Finally, it was calcinated at 650° C for 5 h and the product determined by X-ray diffraction analysis.

A mixture of 2.1 g of potassium carbonate and 4.8 g of anatase, ground in a mortar with a pestle for 30 min, was placed into a platinum crucible heated at 1050° C for 5 h. When the reaction had taken place, the product was removed from the crucible and placed in a beaker with 100 ml of water and the alkaline material extracted with boiling water. The remainder of the product obtained by filtration was dried at 110° C for 24 h. The product was a grey-blue colour and had needle-like crystals. The X-ray diffraction analysis showed that the product was $K_2Ti_6O_{13}$.

2.2. Formation of potassium titanate fibres from fluxes such as K_2O–B_2O_3 and Na_2O–K_2O–B_2O_3

3 g of a mixture of the flux and the needle-like potassium titanate was placed in a 3 cm diameter platinum crucible and heated at 1050° C for 2 h. After the reaction was complete, the mixture was cooled by air to room temperature.

2.3. Analysis

The weight loss of the flux was determined with a balance. The amount of sodium and potassium in the flux was also determined by the methods of Koening [5] and Magaux [6], respectively.

The products were determined by X-ray diffraction analysis and by electron microscopy.

3. Results and discussion

3.1. The crystal growth of potassium titanate with Na_2O–K_2O–B_2O_3 flux

0–5 wt % of Na_2O is mixed with $3K_2O$–$5B_2O_3$ flux. This mixture and fluxes such as Na_2O–K_2O–B_2O_3

TABLE I Crystal growth of potassium titanate fibre using Na_2O–K_2O–B_2O_3 flux

Flux	Na_2O addition wt (%)	Elimination (%)	Fibre diameter (μm)	Length (mm)
$3K_2O$–$5B_2O_3$	0	18.2	10	0.3–1.0
	1	20.3	80–100	4
	2	21.1		4
	5	21.0		5
Na_2O–$3K_2O$–$5B_2O_3$		23.4		8
$2Na_2O$–$3K_2O$–$5B_2O_3$		10.3		Non crystalline
Na_2O–$2K_2O$–$5B_2O_3$		5.6		1–1.5
$2Na_2O$–$3K_2O$–$5B_2O_3$		9.2		3–4

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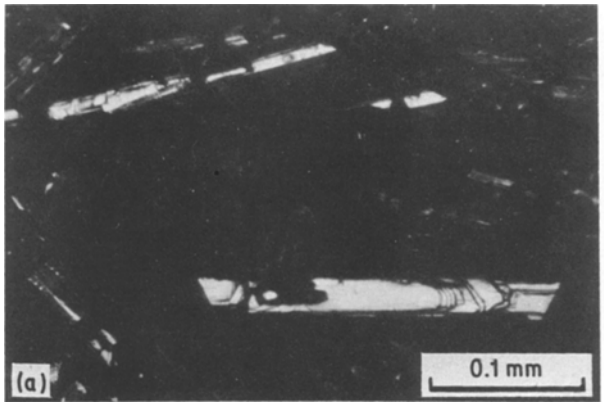
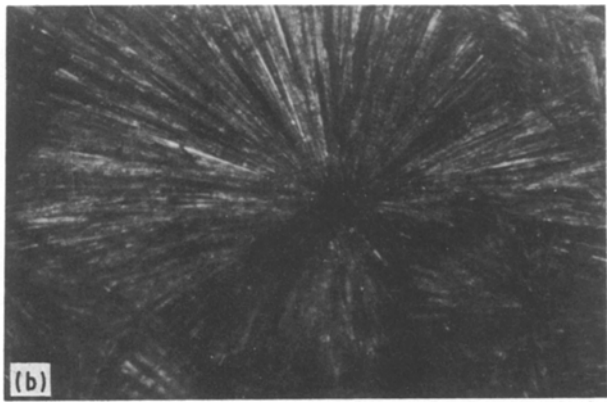




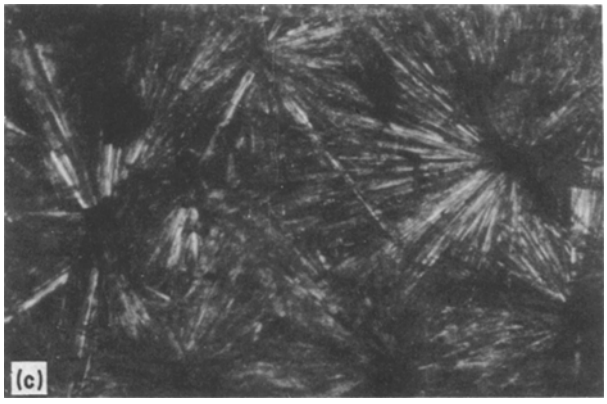


Figure 1 Electron micrography of the fibres formed using the weight ratio flux to $K_2Ti_6O_{13}$ (a) 0.5, (b) 0.88 (c) 1.0.

were used to prepare potassium titanate fibres. As the total amount of the flux and potassium titanate is about 3 g and the weight ratio of the flux to potassium titanate (F/P) is 1, crystal growth is carried out 1050° C for 2 h. The diameter and length of the fibres prepared under the experimental conditions are summarized in Table I.

The amount eliminated from the fluxes, the diameter and length of the fibres increased with increasing the addition percentage of Na_2O. Using four types of flux, the amount eliminated, the diameter and length of the fibres decreased with increasing Na_2O and decreasing K_2O. However, any fibre is formed in the case of $2Na_2O$–$3K_2O$–$3B_2O_3$. As the longest length fibre is formed using Na_2O–$3K_2O$–$5B_2O_3$, the crystal growth of potassium titanate fibres was investigated by changing F/P. The results are summarized in Table II.

It is found from Table II that the minimum amount of elimination appears in the vicinity on $F/P = 0.88$, however, the fibre having the smallest diameter and the longest length is prepared with $F/P = 0.88$. On the other hand, using $F/P = 1.30$, no crystalline material is obtained, and a glass is formed as the potassium titanate is dissolved in the flux.

Electron micrographs were taken of the crystal formed using Na_2O–$3K_2O$–$5B_2O_3$ and the $F/P = 1$ (Fig. 1a), $F/P = 3$ (Fig. 1b) and $F/P = 4$ (Fig. 1c). It is found that the diameter of the crystal tends to increase with increasing the eliminated volume of the flux.

3.2. The relation between crystal growth and the retention time using Na_2O–$3K_2O$–$5B_2O_3$ flux

By changing the retention time with $F/P = 0.88$, the weight loss percent of the flux, the diameter and length

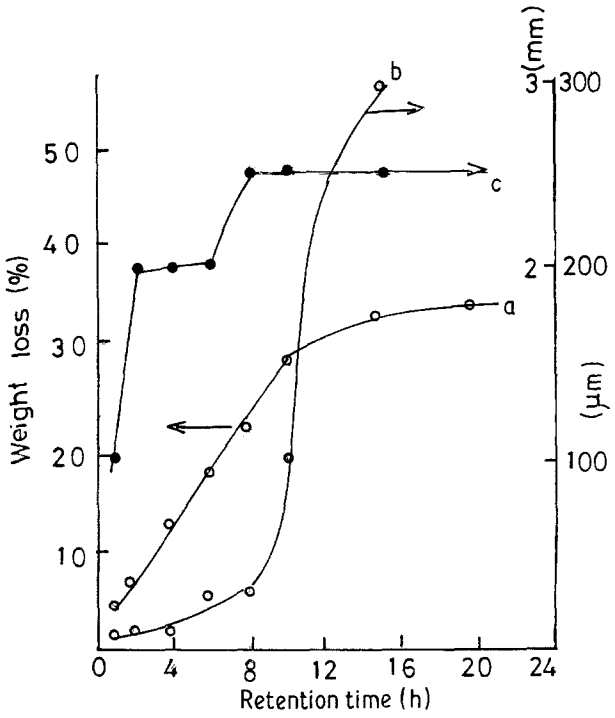


Figure 2 The relation between the weight loss per cent of the (a) flux (b) diameter and (c) length of the fibre.

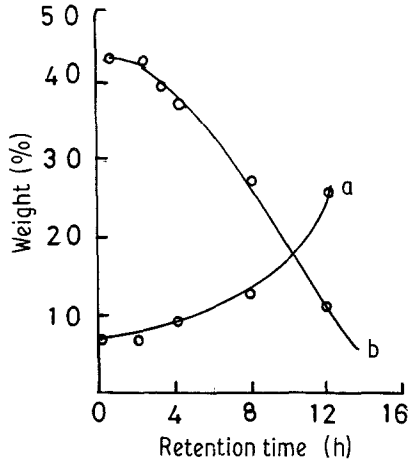
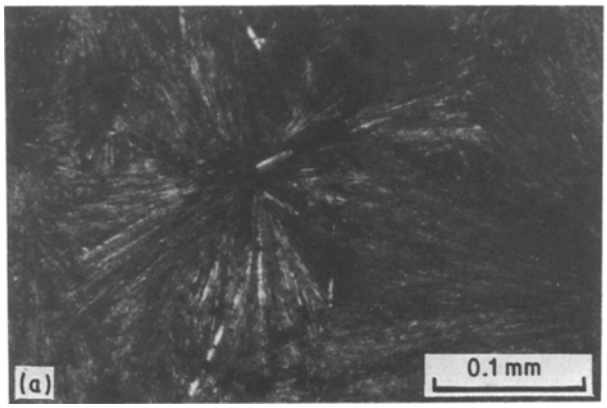
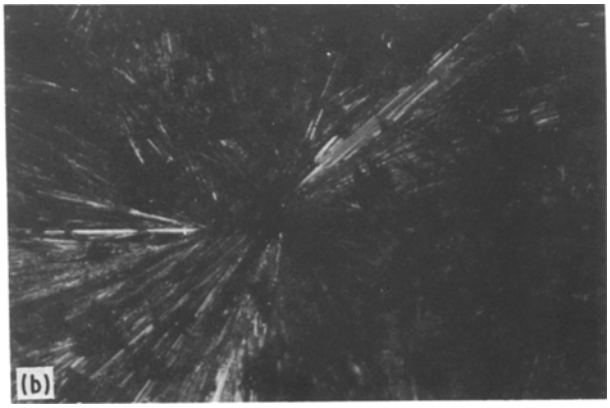


Figure 3 The relation between the weight per cent of (a) Na_2O and (b) K_2O in the flux and the retention time.





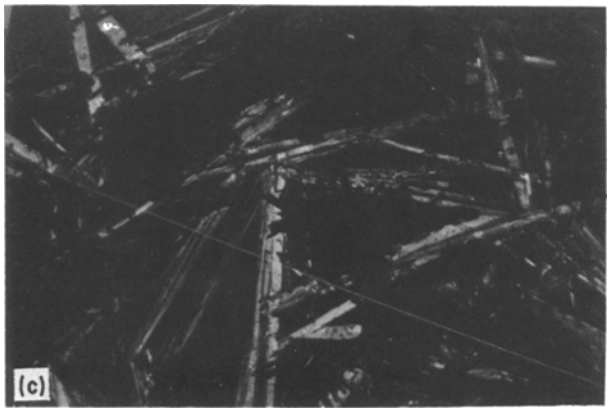


Figure 4 Electron micrography of the fibres for retention time (a) 1 h (b) 4 h and (c) 6 h.

TABLE II Crystal growth of potassium titanate fibre using Na_2O–$3K_2O$–$5B_2O_3$ flux

F/P	Elimination (%)	Fibre diameter (μm)	Length (mm)
0.5	10.9	50–70	2
0.76	8.0	20–40	2
0.88	7.7	10	3
1.0	8.1	20–30	1.5
1.3	14.7	Non crystalline	

F/P = The flux–potassium titanate weight ratio.

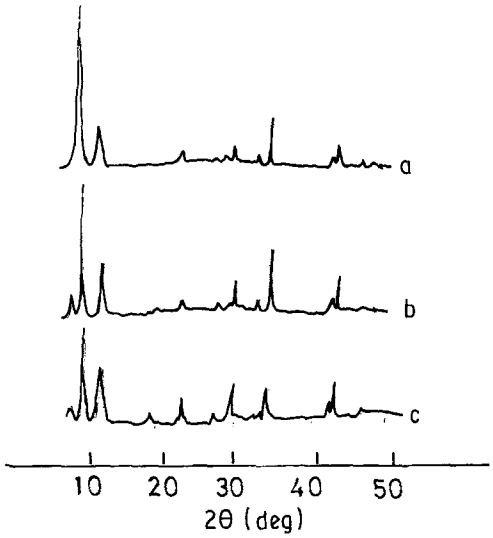


Figure 5 X-ray diffraction of the product for retention time (a) 4 h (b) 2 h and (c) $K_2Ti_6O_{13}$.

of the fibre prepared were determined, and the results shown in Fig. 2.

It is found from Fig. 2 that the weight loss of the flux and the length of the fibre reach a steady state when the retention time is more than 8 h. However, the diameter of the fibre dramatically grows after 8 h. Also, it is assumed that crystal growth of the fibre will be related to the elimination of some substances from the flux as shown in Fig. 2, and Na_2O or K_2O will be eliminated. The amount of Na_2O and K_2O were determined by chemical analysis and the results are shown in Fig. 3.

It is found that K_2O dramatically decreases, and, on the other hand, Na_2O increases after a retention time of 3 h. However, the composition deviation of the flux owing to the retention time does not benefit the crystal growth of potassium titanate fibres. Consequently, using this flux, crystal growth must be finished within 3 h. Electron micrographs of potassium titanate fibres are shown in Fig. 4.

A fibre-like product is formed for a short retention time, however a pillared crystalline product is formed for a long retention time.

The X-ray diffraction analysis of the fibre separated from the reaction mixture by washing with hot water is given in Fig. 5. It is found from Fig. 5 that the product is $K_2Ti_6O_{13}$ because the diffraction pattern is consistent with the appropriate ASTM card.

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