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SYNTHESIS OF SCANDIUM PSEUDOBROOKITE, Sc_2TiO_5 ¹

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ABSTRACT

Synthesis of small crystals ($1 \times 1 \times 2$ mm) of Sc_2TiO_5 , scandium pseudobrookite, has been effected by slow cooling from $1,460^\circ\text{C}$ - $1,000^\circ\text{C}$ using sodium tungstate, Na_2WO_4 , as a flux. Complete solid-solution series were obtained between pseudobrookite, Fe_2TiO_5 , and scandium pseudobrookite, Sc_2TiO_5 , by heating at $1,150^\circ\text{C}$ gels in air.

Isostructural compounds R_2^{3+} with ortho-rhombic space group *Bbmm* include Al_2TiO_5 (Yamaguchi, 1944; Lang, Filmore, and Maxwell, 1952; Kim and Hummel, 1960; Goldberg, 1968), Ga_2TiO_5 (Goldberg, 1968), and $\text{Fe}^{3+}\text{TiO}_5$, pseudobrookite (Akimoto, Nagata and Katsura, 1957; Karkhanavala, 1959; McChesney and Muan, 1959; Goldberg, 1968; Buddington and Lindsley, 1964).

Synthesis of small crystals of Sc_2TiO_5 , scandium pseudobrookite, has been effected by slow cooling (2°C/hr.) from $1,460$ - $1,000^\circ\text{C}$ in Pt crucible of Sc_2O_3 and TiO_2 in sodium tungstate, Na_2WO_4 , as a solvent. Optimum charge ratio of the oxides and flux was found to be in the vicinity of $\text{Sc}_2\text{O}_3 = 22.0$. $\text{TiO}_2 = 22.0$ and $\text{Na}_2\text{WO}_4 = 56.0$ (in mole per cent).

The tabular to prismatic crystals are faintly yellow due to Fe_2O_3 (0.17 percent). They are elongated on $[001]$, measure approx. $1 \times 1 \times 2$ mm and often show terminal faces. At the end of the runs, only Sc_2TiO_5 remained after dissolving the flux in hot water. Previously reported

¹ Mineralogical Contribution No. 480.

TABLE 1. X-RAY POWDER DATA FOR SYNTHETIC SC-PSEUDOBROOKITE
 Sc_2TiO_5 , *Bbmm*, *a* 10.127(1), *b* 10.274(1), *c* 3.8509(5).
 Cu radiation (Ni filtered) $K\alpha_1 = 1.54051$. All *d*-values calculated with
 computer program (IBM 7094) and are given in Angstrom. Sc_2TiO_5
 contains 0.17% of Fe_2O_3 ; specific gravity is 3.62 ± 0.01 .

<i>h k l</i>	<i>I/I</i> ₀	<i>d</i> (obs.)	<i>d</i> (calc.)
0 2 0	20	5.13	5.14
2 0 0	50	5.06	5.06
1 0 1	100	3.60	3.60
1 1 1	8	3.397	3.397
1 2 1	5	2.946	2.948
2 3 0	100	2.838	2.837
3 0 1	30	2.537	2.538
1 3 1	25	2.480	2.481
2 4 0	20	2.290	2.290
4 2 0	25	2.271	2.271
4 3 0	30	2.037	2.036
0 0 2	20	1.926	1.925
2 5 0	10	1.905	1.904
3 4 1	5	1.806	1.805
2 0 2	5	1.801	1.800
0 6 0	15	1.713	1.712
5 2 1	20	1.693	1.692
6 1 0	5	1.666	1.666
2 6 0	5	1.623	1.622
3 5 1	5	1.597	1.597
2 3 2	25	1.593	1.593
5 3 1	20	1.588	1.588
1 6 1	5	1.547	1.546
2 4 2	5	1.474	1.474
5 4 1	10	1.469	1.470
6 4 0	3	1.410	1.410
4 3 2	8	1.399	1.399
1 7 1	5	1.359	1.360
7 0 1	8	1.354	1.354
7 1 1	3	1.343	1.343
7 2 1	15	1.309	1.310
0 6 2	5	1.280	1.280
1 3 3	5	1.194	1.194

$2\text{Sc}_2\text{O}_3 \cdot 3\text{TiO}_2$ and $3\text{Sc}_2\text{O}_3 \cdot 2\text{TiO}_2$ have not been found as products (Kommissarova and Pokrovskii, 1966).

Single-crystal study using the precession method with $\text{MoK}\alpha_1$ radiation indicates that Sc_2TiO_5 has the same space group as natural pseudobrookite, *Bbmm*, given by Pauling (1930), and the other $R^{3+}_2\text{TiO}_5$ compounds mentioned above. Unambiguously indexed computer refined

TABLE 2. UNIT-CELL DIMENSIONS OF THE SOLID-SOLUTION SERIES OF SC-PSEUDOBROOKITE—PSEUDOBROOKITE (ORTHORHOMBIC *BMM*).

Composition	Temperature (°C)	<i>a</i> (Å)	<i>b</i> (Å)	<i>c</i> (Å)	<i>V</i> (Å ³)
Sc ₂ TiO ₅ ^{1,2} (Fe ₂ O ₃ 0.17%)—Grown in flux 1,300-1,000°C		10.127 (1)	10.274 (1)	3.8509 (5)	400.66
(Sc ₇₅ Fe ₂₅) ₂ TiO ₅	1,150	10.07	10.23	3.810	392.4
(Sc ₅₀ Fe ₅₀) ₂ TiO ₅	1,150	9.979	10.14	3.776	382.1
(Sc ₂₅ Fe ₇₅) ₂ TiO ₅	1,150	9.904	10.06	3.757	374.8
Fe ₂ TiO ₅	1,150	9.813	9.975	3.729	365.0

¹ Spectrographic analysis of the flux grown crystals of Sc₂TiO₅ gave: Si, Mg, Na, Al ≅ 0.01%; Ni, Cu, Ca, Sn ≅ 0.001% and Be, Yb ≅ 0.0001%.

² This compound has been synthesized also by heating the gel at 1,150°C in air, but the flux-grown crystals were chosen for the unit-cell refinement.

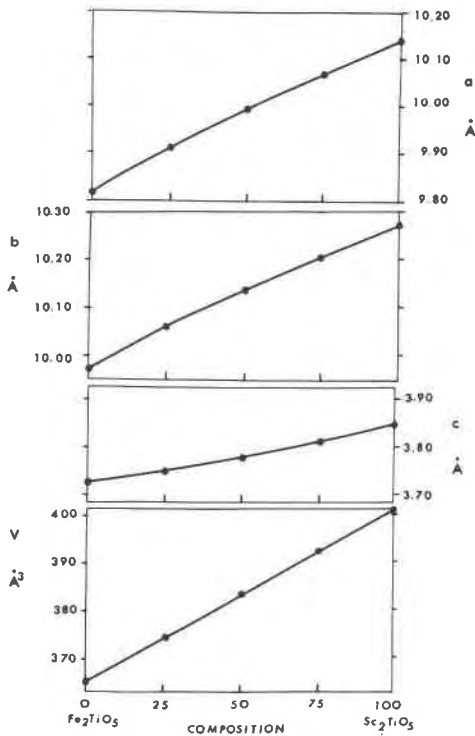


FIG. 1. Unit-cell dimensions of the solid-solution series of Sc-pseudobrookite—pseudobrookite.
Sc₂TiO₅ ————— Fe₂TiO₅

powder data confirmed by the intensities observations on the precession photographs are given in Table 1. Specific gravity determined by floating crystals in Clerici solution gave 3.62 (calculated Sp. Gr. 3.61).

Natural pseudobrookite, $\text{Fe}_2^{3+}\text{TiO}_5$, containing 0.74 percent of Sc_2O_3 was reported from the Thomas Mountains, Utah (Fron del, 1970). Complete solid-solution series were obtained between pseudobrookite, $\text{Fe}_2^{3+}\text{TiO}_5$, and scandium pseudobrookite, Sc_2TiO_5 , by heating at 1,150°C gels containing the exact amount of components precipitated by ammonia.

Unit-cell dimensions of two synthetic end-members and three intermediate compounds were calculated from the powder diffraction data, and are given in Table 2 and Figure 1.

Efforts to synthesize the indium analogue of pseudobrookite failed, $\text{In}_2\text{Ti}_2\text{O}_7$ and In_2O_3 being formed instead under the present experimental conditions.

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