Synthesis and Crystal Chemistry of $BaNd_2Ti_3O_{10}$, $BaNd_2Ti_5O_{14}$, and $Nd_4Ti_9O_{24}$

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Two new ternary compounds $BaNd_2Ti_3O_{10}$ (1:1:3) and $BaNd_2Ti_5O_{14}$ (1:1:5) have been identified in the $BaO-Nd_2O_3-TiO_2$ system. Single crystals of the compounds were grown and unit cell dimensions and space group symmetry were determined. $BaNd_2Ti_3O_{10}$ is orthorhombic with $a = 3.8655 \pm 0.0003$, $b = 28.156 \pm 0.003$ and $c = 7.6221 \pm 0.0007$ Å and possible space groups are *Cmcm* or *Cmc*2. The compound melts congruently at $1640 \pm 20°C$. $BaNd_2Ti_5O_{14}$ is also orthorhombic with $a = 22.346 \pm 0.002$, $b = 12.201 \pm 0.001$ and $c = 3.8404 \pm 0.0003$ Å and possible space groups are *Pbam* and *Pba*2. This compound melts congruently at $1540 \pm 20°C$. Single crystals of the binary compound $Nd_4Ti_9O_{24}$ were also grown and found to be orthorhombic with $a = 35.289 \pm 0.003$, $b = 13.991 \pm 0.001$, $c = 14.479 \pm 0.001$ Å, space group *Fddd*.

1. Introduction

Dielectric ceramics based on the $BaO–Nd_2O_3–TiO_2$ system are characterized by relatively high permittivity, high temperature stability, and low dielectric losses. As such, they have found important applications in modern electronic practice (*1*). During an investigation of these ceramics, the existence of two hitherto unknown ternary compounds was noted. Consequently, a detailed study of the TiO_2-rich part of the $BaO–Nd_2O_3–TiO_2$ system has been carried out. The work reported in this paper is concerned with the identification and characterization of compounds and their stability relations in the subsystem $BaTiO_3–Nd_2Ti_2O_7–TiO_2$.

2. Experimental

The starting materials[1] were reagent-grade barium carbonate, titanium dioxide (99.5% pure), neodymium oxide (>99.9% pure), and reagent grade barium titanate. Appropriate proportions of the powdered materials were mixed under alcohol, dried, and pressed into pellets. Samples were calcined at 1300–1400°C for prolonged periods with intermittent cooling, crushing, mixing, and pressing to ensure homogeneity and to attain equilibrium. The fired samples were examined by X-ray powder diffraction. For routine phase identification, X-ray powder patterns were ob-

[1] Used at J. Stefan Institute, Ljubljana, Yugoslavia.

tained in a Guinier-type focusing camera using Ni-filtered Cu$K\alpha$ radiation.

Single crystals of the compounds were grown from the melt in small sealed Pt tubes or from the BaO-rich flux. Precision lattice parameters measurements were obtained by least-squares refinement of diffraction data collected with a high-angle recording diffractometer using Ni-filtered Cu$K\alpha$ radiation. Indexing of the powder patterns was accomplished by reference to the single-crystal diffraction data obtained with a Buerger precession camera.

Reflected-light microscopy was used extensively throughout the phase analysis and for the determination of the correct compositions of the compounds by observation of small amounts of secondary phases in the microstructure. The correctness of the composition of the new compounds was also confirmed by scanning electron microscopy. Melting behavior was investigated by a hot-stage microscope. Density of the compounds was determined by pycnometric method using hexane as the immersion liquid.

3. Results and Discussion

Two new ternary compounds were identified in the system. Chemical compositions as ascertained by the combination of metallography, electron microanalysis, and X-ray investigations were found to be BaNd$_2$Ti$_3$O$_{10}$ and BaNd$_2$Ti$_5$O$_{14}$, respectively.

The composition BaO:Nd$_2$O$_3$:3TiO$_2$ (1:1:3) (prepared using a less pure Nd$_2$O$_3$) was calcined at 1425°C for 183 hr with periodic grindings to yield a product consisting primarily of 1:1:3 with only small amounts of BaTiO$_3$, TiO$_2$, and an unidentified phase remaining. A sample of this calcined material, heated at 1625°C, was completely melted. Small single crystals of the 1:1:3 compound were selected from the melted material. Crystals of

Nd$_2$O$_3 \cdot$ 2TiO$_2$ were also found in this melted sample. X-Ray diffraction of a similar completely melted specimen indicated the presence of 1:1:3 as the major phase with minor amounts of BaTiO$_3$ and several other phases. The 1:1:3 phase was found to be orthorhombic with cell parameters[2] obtained from an unmelted specimen prepared with more pure Nd$_2$O$_3$ of $a - 3.8655 \pm 0.0003$, $b = 28.156 \pm 0.003$, $c = 7.6221 \pm 0.0007$ Å with a C-centered cell, $hkl{:}h + k = 2n$ and $h0l{:}l = 2n$. The possible space groups are thus No. 63 $Cmcm$ or No. 36 $Cmc2$. Calculated density $\rho = 5.845$ g/cm^3 compared reasonably well with the experimentally determined powder density of $\rho = 5.66$ g/cm^3. The compound melts congruently at $1640 \pm 20°$C. The indexed X-ray diffraction powder pattern is listed in Table I.

An essentially single-phase composition of BaO:Nd$_2$O$_3$:5TiO$_2$ (1:1:5), prepared by calcining the constituent oxides, using the less pure Nd$_2$O$_3$, for 60 hr at 1350°C and 12 hr at 1375°C with periodic grindings, was used for the preparation of small single crystals of this compound. Heating the calcined material for 25 hr at 1400°C in a small sealed platinum tube followed by quenching in water resulted in a product showing traces of melting, primarily composed of small, acicular crystals of 1:1:5. X-Ray diffraction showed only the 1:1:5 phase, identical to the 1375°C calcined material. The compound is orthorhombic with unit cell parameters obtained from an unmelted specimen prepared from more pure Nd$_2$O$_3$ of $a = 22.346 \pm 0.002$, $b = 12.201 \pm 0.001$ and $c = 3.8404 \pm 0.0003$ Å. The extinction rules appear to be $0kl{:}k = 2n$ and $h0l{:}h = 2n$, so the possible space

[2] Electron diffraction patterns made by A. Olsen and R. S. Roth at Arizona State University revealed that the a axis is really doubled ($a = 7.722$) although the extra spots requiring this doubled cell are very weak. Results of this electron diffraction and high-resolution lattice image study will be published in the near future.

TABLE I

INDEXED X-RAY DIFFRACTION POWDER PATTERN FOR THE COMPOUND
$BaO \cdot Nd_2O_3 \cdot 3TiO_2$

h	k	ℓ [1]	d_{obs}	$2\theta_{calc}$	$2\theta_{obs}$	I/I_o
0	2	0	14.09	6.27	6.27	10
0	6	0	4.687	18.90	18.92	33
1	1	0	3.829	23.21	23.21	11
0	0	2	3.815	23.32	23.30	15
1	3	0	3.574	24.89	24.89	6
1	1	1	3.422	26.02	26.02	18
1	3	1	3.236	27.54	27.54	12
1	5	0	3.186	27.97	27.98	24
0	6	2	2.959	30.19	30.18	100
1	5	1	2.939	30.38	30.39	20
1	7	0	2.786	32.09	32.10	60
1	1	2	2.700	33.14	33.15	57
1	7	1	2.617	34.23	34.23	7
0	8	2	2.5860	34.67	34.66	7
1	5	2	2.4436	36.73	36.75	5
0	12	0	2.3458	38.33	38.34	18
1	9	1	2.3168	38.84	38.84	6
1	7	2	2.2495	40.05	40.05	45
1	1	3	2.1168	42.67	42.68	8
1	3	3	2.0715	43.68	43.66	6
1	11	1	2.0554	44.03	44.02	8
1	9	2	2.0496	44.14	44.15	7
0	14	0	2.0108	45.04	45.05	18
0	12	2	1.9986	45.35	45.34	12
1	5	3	1.9870	45.63	45.62	3
2	0	0	1.9333	46.98	46.96	21
2	2	0	1.9149	47.44	47.44	3
0	0	4	1.9058	47.69	47.68	18
1	13	0	1.8901	48.12	48.10	5
0	2	4	1.8883	48.15	48.15	5
2	6	0	1.7873	51.07	51.06	5
0	6	4	1.7651	51.74	51.75	6
0	16	0	1.7603	51.92	51.90	6
2	6	1	1.7401	52.56	52.55	2
2	0	2	1.7239	53.09	53.08	4
1	13	2	1.6930	54.13	54.13	32
1	15	0	1.6886	54.29	54.28	22
1	5	4	1.6354	56.20	56.20	5
2	6	2	1.6180	56.86	56.86	22
1	7	4	1.5730	58.64	58.64	18
2	8	2	1.5476	59.68	59.70	3
1	15	2	1.5432	59.86	59.89	6

[1] Indexed, with the aid of single crystal precession patterns, on the
basis of an orthorhombic unit cell with $\underline{a}$ = 3.8655 $\pm$ .0003,
$\underline{b}$ = 28.156 $\pm$ .003, $\underline{c}$ = 7.6221 $\pm$ .0007A, although electron diffraction
indicates the true unit cell as twice the listed $\underline{a}$ value.

groups are No. 55, *Pbam* and No. 32, *Pba*2. Calculated density $\rho = 5.62$ g/cm³ compares reasonably well with the pycnometrically determined powder density of 5.44 g/cm³. The compound, prepared from a more pure Nd_2O_3, melts congruently at 1540 $\pm$ 20°C. The indexed X-ray diffraction powder pattern is listed in Table II.

To establish the subsolidus phase relations in the $BaTiO_3$–TiO_2–Nd_2TiO_5 subsystem, the pseudobinary joins had to be known. The $BaTiO_3$–TiO_2 join has been reexamined recently (2, 3) and the existence of $Ba_6Ti_{17}O_{40}$, $Ba_4Ti_{13}O_{30}$, $BaTi_4O_9$, and $Ba_2Ti_9O_{20}$ were reported. In the join $BaTiO_3$–Nd_2TiO_5 no binary compound was

TABLE II

INDEXED X-RAY DIFFRACTION POWDER PATTERN FOR THE COMPOUND
$BaO \cdot Nd_2O_3 \cdot 5TiO_2$

h	k	λ [1]/	d_{obs}	$2\theta_{calc}$	$2\theta_{obs}$	I/I_o
2	0	0	11.20	7.91	7.89	10
1	1	0	10.72	8.25	8.24	3
0	2	0	6.09	14.51	14.53	3
4	1	0	5.08	17.45	17.43	16
5	1	0	4.197	21.15	21.15	14
1	3	0	4.003	22.20	22.19	10
0	0	1	3.841	23.14	23.14	12
2	3	0	3.819	23.26	23.27	15
2	0	1	3.633	24.49	24.48	5
5	2	0	3.607	24.67	24.66	4
2	1	1	3.482	25.57	25.56	7
0	2	1	3.251	27.42	27.41	18
6	2	0	3.180	28.05	28.04	22
4	1	1	3.064	29.13	29.12	45
3	2	1	2.978	29.97	29.98	16
2	4	0	2.943	30.35	30.35	20
5	1	1	2.833	31.55	31.56	100
1	3	1	2.770	32.28	32.29	28
8	1	0	2.723	32.87	32.87	12
2	3	1	2.710	33.04	33.03	58
4	4	0	2.677	33.44	33.45	14
5	2	1	2.629	34.08	34.08	17
3	3	1	2.614	34.27	34.28	30
6	1	1	2.611	34.31	34.32	17
8	2	0	2.5392	35.31	35.32	5
2	5	0	2.3835	37.70	37.71	3
2	4	1	2.3364	38.51	38.50	6
9	2	0	2.3003	39.14	39.13	9
7	2	1	2.2768	39.54	39.55	29
3	4	1	2.2735	39.59	39.61	24
8	1	1	2.2213	40.58	40.58	4
{10	1	0}	2.1970	{41.03 41.07}	41.05	12
4	4	1				
5	5	0	2.1422	42.16	42.15	12
{9	3	0}	2.1187	{42.63 42.65}	42.64	14
8	2	1				
5	4	1	2.1074	42.90	42.88	5
10	2	0	2.0990	43.08	43.06	24
0	6	0	2.0348	44.52	44.49	4
11	1	0	2.0049	45.21	45.19	8
2	6	0	2.0011	45.29	45.28	8
9	2	1	1.9726	45.96	45.97	2
3	6	0	1.9610	46.24	46.26	8
7	5	0	1.9388	46.82	46.82	12
0	0	2	1.9202	47.30	47.30	35
{5	5	1}	1.8708	{48.64 48.65}	48.63	5
2	1	2				
5	6	0	1.8504	49.19	49.20	8
12	1	0	1.8403	49.47	49.49	5
11	3	0	1.8169	50.16	50.17	3
4	1	2	1.7965	50.79	50.78	6
1	6	1	1.7916	50.94	50.93	3
12	2	0	1.7808	51.25	51.26	4
11	1	1	1.7763	51.39	51.40	12
2	6	1	1.7740	51.46	51.47	9
3	6	1	1.7472	52.33	52.32	5
{1	3	2}	1.7309	{52.84 52.86}	52.85	20
7	5	1				
9	4	1	1.7212	53.17	53.17	12
12	0	1	1.6758	54.74	54.73	7
12	1	1	1.6599	55.30	55.30	7
4	3	2	1.6585	55.36	55.35	5
{6	2	2}	1.6429	{55.90 55.93}	55.92	4
11	3	1				
5	7	0	1.6240	56.64	56.63	5
2	4	2	1.6076	57.24	57.26	8
3	4	2	1.5876	58.05	58.05	22
9	5	1	1.5849	58.15	58.16	34
1	7	1	1.5834	58.23	58.22	25
2	7	1	1.5708	58.71	58.73	11
8	1	2	1.5691	58.80	58.80	5
4	4	2	1.5600	59.17	59.18	4

[1]/ Indexed, with the aid of single crystal precession patterns, on
an orthorhombic unit cell with $\underline{a}$ = 22.346 ± .002, b = 12.201 ± .001,
$\underline{c}$ = 3.8404 ± .0003A.

found to exist. In the join TiO_2–Nd_2TiO_5 the existence of orthorhombic Nd_2TiO_5 had been reported (4) and monoclinic unit cell dimensions are reported for $Nd_2Ti_2O_7$ (5, 6). Recently, a binary compound with a molar ratio TiO_2 : Nd_2O_3 4 : 1 has been reported however, the homogeneity range of the compound was found to extend up to the composition TiO_2 : Nd_2O_3 = 9 : 2 (7).

In the present investigation, single crystals of the TiO_2-rich phase in the Nd_2O_3–TiO_2 binary system were grown from a BaO-containing flux. The addition of small amounts of BaO, to act as a "flux" and also shift the composition to a point where the compound was the primary phase, allowed the growth of small crystals of the compound. A composition of $2.5BaO$: $17.5Nd_2O_3$: $80TiO_2$ was heated at 1450°C for 91 hr in a small sealed platinum tube and quenched into water. The X-ray diffraction pattern showed the crystals to be identical with the compound described in Ref. (7). Analysis of the single-crystal X-ray data confirmed that the unit cell had orthorhombic symmetry as suggested in Ref. (7), but with a considerably larger unit cell. Precision unit cell dimensions obtained from a single-phase unmelted specimen using more pure Nd_2O_3 are a = 35.289 ± 0.003, b = 13.991 ± 0.001, c = 14.479 ± 0.001 A. The extinction rules found were hkl : $h + k$, $k + l$, $l + h$ = $2n$; $0kl$: $k + l$ = $4n$; $h0l$: $l + h$ = $4n$; $hk0$: $h + k$ = $4n$. The space group was thus determined as No. 70 $Fddd$. On the basis of the indexing of powder data it can be concluded that the compound is better described as a 2 : 9 phase rather than a 1 : 4 phase, indicating the correct formula as $Nd_4Ti_9O_{24}$. The indexed X-ray diffraction powder pattern is listed in Table III.

To establish the subsolidus phase relations in the $BaTiO_3$–TiO_2–Nd_2TiO_5 system, 57 ternary compositions were fired and examined by X-ray powder diffraction. The phase diagram $BaTiO_3$–TiO_2–Nd_2TiO_5 con-
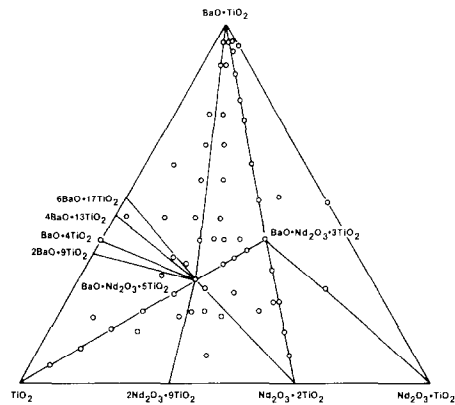

FIG. 1. Subsolidus equilibria in the system $BaTiO_3$–TiO_2–Nd_2TiO_5 showing various experimental compositions and tie lines. Scale in mole%.

structed on the basis of this analysis and previously known data is shown in Fig. 1.

The binary joins $BaTiO_3$–$Nd_2Ti_2O_7$ and TiO_2–$BaNd_2Ti_3O_{10}$ were also examined by metallography. The data obtained were consistent with those obtained by X-ray and scanning electron microscopy. Figure 2 shows the microstructure of a sample with molar ratio $BaTiO_3$: $Nd_2Ti_2O_7$ = 70 : 30. Elongated grains of $BaNd_2Ti_3O_{10}$ were observed in the $BaTiO_3$ matrix. Figure 3 shows the elongated grains of pure $BaNd_2Ti_3O_{10}$ compound under higher

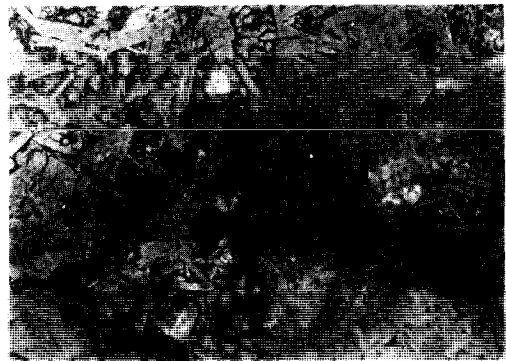FIG. 2. Microstructure of sintered ceramic with the composition 70 mole% $BaTiO_3$–30 mole% $Nd_2Ti_2O_7$. Needlelike crystals of $BaNd_2Ti_3O_{10}$ and $BaTiO_3$ grains may be distinguished (300×).

TABLE III

Indexed X-Ray Diffraction Powder Pattern for the Compound $2Nd_2O_3 \cdot 9TiO_2$

h	k	l [1/]	d_{obs}	$2\theta_{calc}$	$2\theta_{obs}$	I/I_0
5	1	1	5.78	15.32	15.32	12
0	2	2	5.03	17.62	17.63	6
3	3	1	4.149	21.38	21.40	10
9	1	1	3.658	24.35	24.31	4
0	0	4	3.622	24.59	24.56	2
0	4	0	3.497	25.45	25.45	5
4	0	4	3.345	26.61	26.63	85
8	2	2	3.317	26.86	26.86	47
4	2	0	3.250	27.41	27.42	100
3	3	3	3.222	27.64	27.66	10
10	0	2	3.172	28.11	28.11	18
10	2	0	3.149	28.30	28.32	11
2	4	2	3.100	28.78	28.78	3
5	3	1	3.028	29.47	29.48	20
9	1	3	2.972	30.03	30.04	33
6	0	2	2.938	30.39	30.40	22
7	3	1	2.817	31.71	31.74	12
1	1	5	2.790	32.03	32.06	15
3	5	1	2.736	32.67	32.71	6
11	1	1	2.676	33.47	33.46	3
13	1	1	2.621	34.15	34.18	13
11	3	1	2.619	34.19	34.21	18
12	2	2	2.601	34.47	34.46	7
11	3	2	2.5378	35.33	35.34	15
2	4	4	2.5144	35.68	35.68	35
10	4	4	2.3890	37.61	37.62	11
10	0	6	2.3476	38.28	38.31	6
11	4	2	2.3156	38.82	38.86	6
9	3	5	2.2986	39.19	39.16	5
12	0	4	2.2823	39.46	39.45	17
12 / 12	4 / 0	0 / 1	2.2500	40.04 / 40.04	40.04	10
9	5	6	2.2319	40.39	40.38	10
6	0	6	2.2068	40.85	40.86	11
8	2	4	2.1843	41.30	41.30	9
6	6	0	2.1682	41.63	41.62	9
4	6	2	2.1529	41.94	41.93	8
13	3	3	2.1092	42.83	42.84	12
15	1	1	2.0916	43.24	43.22	3
9	5	5	2.0824	43.40	43.42	3
15	3	1	2.0792	43.50	43.49	3
10	4	4	2.0474	44.19	44.20	3
8	2	6	2.0248	44.71	44.72	4
16	2	2	2.0206	44.84	44.82	4
10	2	6	1.9907	45.52	45.53	3
14 / 8	4 / 6	2 / 1	1.9828	45.71 / 45.73	45.72	7
1	7	1	1.9779	45.87	45.84	7
14	4	2	1.9686	46.09	46.07	15
5	1	7	1.9646	46.18	46.17	14
2	6	4	1.9470	46.59	46.61	10
15	3	3	1.9264	47.16	47.14	3
5	7	1	1.9066	47.67	47.66	7
7	1	7	1.8950	47.97	47.97	7
18	0	2	1.8927	48.04	48.03	8
17	1	3 / 0	1.8901	48.12 / 48.17 / 48.18	48.10	10
18	1	1	1.8876	48.17 / 48.18	48.17	9
1	3	7	1.8832	48.29	48.29	5
16	0	4	1.8817	48.34	48.33	6
6	5	7	1.8683	48.70	48.70	2
3	3	3 / 16	1.8657	48.77 / 48.77	48.77	2
16	4	4	1.8600	48.95	48.93	2
6	6	3	1.8445	49.39	49.37	2
1	7	7	1.8254	49.92	49.92	4
5	3	3	1.8237	49.96	49.97	4
9	1	9	1.8129	50.28	50.29	3
0	2	8	1.8088	50.41	50.41	16
12	6	6	1.8021	50.62	50.61	25
14	8	2	1.7711	51.55	51.56	30
0	0	10	1.7494	52.27	52.25	8
14	4	6	1.7435	52.47	52.44	5
10	4	6	1.7303	52.87	52.87	4
10	6	1 / 7	1.7137	53.43 / 53.43	53.42	18
19	3	7	1.7019	53.81	53.82	8
9	5	3	1.6871	54.34 / 54.33	54.23	18
0	4	6	1.6761	54.71	54.72	6
17	1	5	1.6750	54.77	54.76	6
9	7	3	1.6710	54.92	54.90	6
14	6	2	1.6663	55.09	55.07	4
17	5	1	1.6563	55.43	55.43	4
13	1	7 / 2	1.6333	56.28 / 56.28	56.28	3
6	8	7	1.6285	56.47	56.46	7
11	3	3	1.6248	56.61	56.60	7
13	5	5	1.6156	56.93	56.95	4
11	7	3	1.6002	57.55	57.55	4
8 / 17	8 / 3	8 / 5	1.5861	58.11 / 58.11 / 58.12	58.11	3
20	0	4	1.5807	58.33	58.33	6
4	4	8	1.5755	58.53	58.54	9
17	5	3	1.5745	58.58	58.58	6
0	8	4	1.5694	58.82	58.79	9
8	2	8	1.5672	58.89	58.88	12
16	6	6	1.5648	59.00	58.98	7
22	2	2	1.5633	59.04	59.04	8
14	4	0	1.5597	59.19	59.19	2
13	7	6	1.5507	59.56	59.57	6
14 / 6	8 / 4	4 / 8	1.5500	59.60 / 59.61	59.60	4
14	6	4	1.5469	59.72	59.73	8
3 / 10	9 / 8	1 / 2	1.5320	60.35 / 60.40	60.37	2
22	2	2	1.5281	60.54	60.54	2
12	8	0	1.5026	61.66	61.68	2
21	3	3	1.5019	61.70	61.71	2

[1/] Indexed with the aid of single crystal precession patterns, on the basis of an orthorhombic unit cell with $\underline{a} = 35.289 \pm .003$, $\underline{b} = 13.991 \pm .001$, $\underline{c} = 14.479 \pm .001$Å.
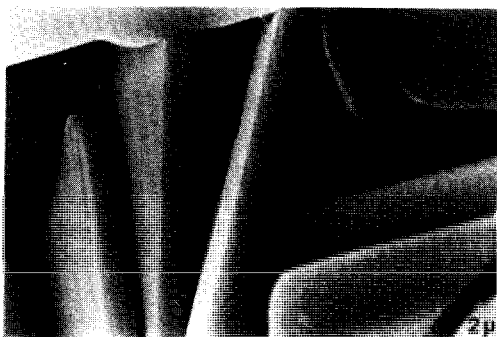
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FIG. 3. SEM picture of needlelike crystals of BaNd$_2$Ti$_3$O$_{10}$ (6000×).

magnification (SEM picture). These elongated grains are actually cross sections through the platy-like structure of BaNd$_2$Ti$_3$O$_{10}$. Figure 4 shows the microstructure of the BaNd$_2$Ti$_5$O$_{14}$ compound.

4. Summary and Conclusions

(1) Subsolidus compatibility relations in the subsystem BaTiO$_3$–TiO$_2$–Nd$_2$TiO$_5$ were established using X-ray powder diffraction.

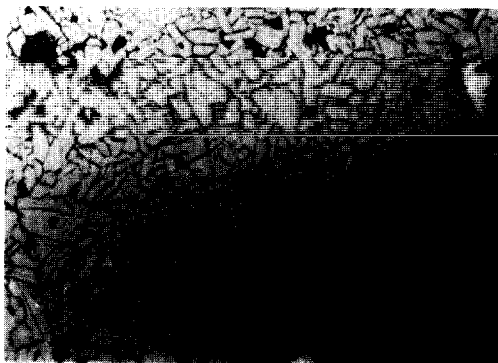(2) Two new ternary compounds, BaNd$_2$Ti$_3$O$_{10}$ and BaNd$_2$Ti$_5$O$_{14}$ were syn-

FIG. 4. Microstructure of sintered ceramic with the composition BaNd$_2$Ti$_5$O$_{14}$ (198×).

thesized. The compounds melt congruently at 1640 ± 20°C and 1540 ± 20°C, respectively.

(3) Single crystals of both the compounds were obtained and unit cell dimensions and space group symmetry determined. BaNd$_2$Ti$_3$O$_{10}$ is orthorhombic with unit cell parameters a = 3.8655 ± 0.0003, b = 28.156 ± 0.003, and c = 7.6221 ± 0.0007 Å. The possible space groups are No. 63 $Cmcm$ or No. 36 $Cmc2$. BaNd$_2$Ti$_5$O$_{14}$ is orthorhombic with a = 22.346 ± 0.002, b = 12.201 ± 0.001, and c = 3.8404 ± 0.0003 Å. Possible space groups are No. 55 $Pbam$ and No. 32 $Pba2$. Indexed X-ray diffraction powder patterns are listed.

(4) Single crystals of the orthorhombic phase in the system TiO$_2$–Nd$_2$Ti$_2$O$_7$ were obtained and the unit cell examined. Analysis of the single-crystal data suggest that the correct composition is Nd$_4$Ti$_9$O$_{24}$. The structure is orthorhombic with a = 35.289 ± 0.003, b = 13.991 ± 0.001 and c = 14.479 ± 0.001 Å. The space group is No. 70 $Fddd$.

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