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A refinement of the crystal structure of ohmilite, $Sr_3(Ti,Fe^{3+})(O,OH)(Si_2O_6)_2 \cdot 2–3H_2O$

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Abstract

The monoclinic unit cell of ohmilite, $Sr_3(Ti,Fe^{3+})(O,OH)(Si_2O_6)_2 \cdot 2–3H_2O$, has dimensions $a = 10.979(6)$, $b = 7.799(5)$, $c = 7.818(4)$Å, $\beta = 100.90\ (3)°$, and space group $P2_1/m$; $Z = 2$, $V = 657.4(6)$Å^3. The crystal structure has been refined to an R-value of 5.9% for 2121 independent reflections. Vertex-sharing Ti–O octahedra form chains parallel to b. Each of the octahedral chains is flanked by a pair of $[Si_4O_{12}]$ chains, which are similar to those found in batisite and haradaite, thus forming a composite chain of composition $[Si_4O_{12}\ (Ti_2O_2)Si_4O_{12}]^{12-}$. These composite chains are joined together by Sr atoms and water molecules to form the structure. Of the three independent Sr atoms, Sr(1) and Sr(3) are 9-coordinated, with average Sr–O distances of 2.675Å. Sr(2) is 8-coordinated, with average bond length of 2.586Å. The average Ti(Fe)–O bond length is 1.992Å. Average Si–O distances are 1.643 and 1.606Å for bridging and nonbridging bonds, respectively. As consistent with the IR data, hydrogen is present as both H_2O and OH. The former forms hydrogen bonds with the oxygen atoms of the composite chains, with O–O distances from 2.69 to 2.83Å. The latter is associated with the octahedral cations in accordance with the substitution scheme, $Ti^{4+}O^{2-} \rightarrow Fe^{3+}OH^-$.

Introduction

Ohmilite, $Sr_3(Ti,Fe^{3+})(O,OH)(Si_2O_6)_2 \cdot 2–3H_2O$, was found in riebekite–albite units in serpentinite from Ohmi, Niigata Prefecture, Central Japan and was described as a new mineral by Komatsu *et al.* (1973). It occurs as spherulites composed of radially arranged fine needles or fiber-like crystals which are generally less than 10 μm in diameter. The chemical analysis (Komatsu *et al.*, 1973) gave SiO_2 34.79, TiO_2 10.27, Fe_2O_3 0.20, SrO 47.37, H_2O_+ 6.68 with a total of 99.31 weight percent. The chemical formula $Sr_3TiSi_4O_{12}(OH) \cdot 2H_2O$ was proposed assuming that titanium is trivalent. However, according to a suggestion made by Dr. A. Kato the formula has been revised to $Sr_3Ti[O|Si_4O_{12}] \cdot 2H_2O$ or $Sr_3(Ti,Fe^{3+})[(O,OH)|Si_4O_{12}] \cdot 2H_2O$ such that the titanium is present as Ti^{4+}.

The crystal structure of ohmilite has been determined and refined to an R-value of 14% (Mizota *et al.*, 1973), showing that ohmilite has a chain structure similar to that of haradaite (Takéuchi and Joswig, 1967). The relatively high value of the R-factor was due to the poor quality of the crystal used. Because we have recently found a crystal fragment of high quality, we have undertaken the refinement of the crystal structure. The present paper describes the detailed structure of ohmilite and discusses the configuration of the silicate chains and the role of the hydrogen atoms in conjunction with data from infrared absorption spectrometry and DSC-TG.

Experimental

X-ray diffraction

A crystal fragment having dimensions of approximately $0.6 \times 0.1 \times 0.04$ mm was used for the present study. Unit cell dimensions were determined utilizing a Philips PW1100 single-crystal diffractometer using graphite-monochromated Mo$K\alpha$ radiation ($\lambda = 0.71069$Å). The cell dimensions, obtained by least-squares refinement with 25 independent reflections, are $a = 10.979(6)$, $b = 7.799(5)$, $c = 7.818(4)$Å, $\beta = 100.90(3)°$, $V = 657.4(6)$Å.

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K	FO	FC
0	K	0
2	35	-31
4	302	307
6	145	-138
8	84	80
10	145	-138
12	27	30
1	K	0
6	29	26
5	13	-10
4	33	27
2	52	51
1	10	-12
2	K	0
0	64	-72
1	8	6
2	47	-50
3	51	51
4	21	-14
5	36	33
6	30	28
7	45	42
8	35	31
9	14	13
10	45	44
3	K	0
11	70	68
9	69	-63
7	126	123
6	12	9
5	86	-83
4	23	20
3	178	191
2	24	27
1	135	-149
0	40	44
4	K	0
0	74	81
1	9	8
2	35	34
3	91	-97
4	32	32
5	39	-40
7	74	-73
10	11	-11
11	12	-15
5	K	0
11	23	-18
9	20	21
8	20	-16
7	29	-28
6	32	30
5	30	31
3	42	-44
2	62	65
1	43	46
6	K	0
0	75	-78
1	40	41
2	109	114
3	36	36
4	69	-69
5	70	72
6	86	83
7	36	33
8	58	-55
9	26	29
10	52	52
7	K	0
10	42	-35
9	18	16
8	49	48
7	20	-18
6	46	-46
5	19	19
4	77	78
3	29	-28
2	49	-49
1	23	25
0	97	100
8	K	0
0	7	7
2	70	-73
3	27	-27
4	24	25
6	23	-24
7	24	-22
8	47	46
9	K	0
9	36	36
7	54	-51
5	52	50
3	68	-67
1	65	66
0	9	11
10	K	0
0	94	91
1	79	-76
2	26	24
3	71	68
4	50	50
5	68	-68
6	24	-24
7	50	48
9	54	-53
10	42	-37
11	K	0
9	27	-28
5	38	-37
3	19	18
1	34	-32
12	K	0
0	18	14
2	25	-25
4	24	25
5	15	13
8	37	34
13	K	0
8	46	-46
6	62	64
4	42	-44
2	95	95
0	41	-39
-13	K	1
0	72	79
1	22	-25
2	6	9
3	16	14
4	58	55
5	33	-28
6	21	-19
8	19	18
-12	K	1
9	49	42
8	41	37
5	75	70
4	41	37
3	27	-26
2	27	-26
1	55	60
0	31	32
-11	K	1
0	29	28
1	19	-21
2	9	-11
3	46	44
4	29	26
7	43	38
8	22	21
-10	K	1
9	32	-33
8	29	-25
7	24	21
6	37	35
5	70	-64
4	13	-15
3	30	27
2	67	65
1	57	-57
-9	K	1
0	44	48
1	22	-24
2	44	-43
3	14	-14
4	51	48
5	45	-42
6	27	-26
8	46	41
9	16	-19
10	18	-15
-8	K	1
10	28	28
9	21	20
8	49	-45
7	25	22
6	58	57
5	51	51
4	41	-39
3	25	25
2	102	99
1	29	28
0	27	-29
-7	K	1
0	15	15
1	87	87
2	96	95
3	26	-24
4	12	-15
5	99	97
6	48	47
7	8	-11
8	39	-36
9	55	54
10	17	17
11	19	-22
-6	K	1
11	20	23
10	18	18
9	28	-29
8	21	20
6	21	17
5	68	-65
4	9	7
2	10	8
1	51	-50
-5	K	1
0	27	-30
1	141	138
2	70	-68
3	119	-117
4	9	9
5	97	96
6	16	-10
7	75	-75
8	36	36
9	59	64
11	37	-43
-4	K	1
11	20	-20
10	48	-49
9	35	36
8	25	27
7	29	-27
6	61	-62
5	60	60
4	77	72
3	45	-42
2	76	-77
1	76	72
0	95	92
-3	K	1
0	88	84
1	46	-42
2	117	106
3	32	29
4	15	11
5	44	-41
6	24	21
7	16	19
8	35	-40
9	23	-28
11	13	14
12	26	-30
-2	K	1
12	48	56
10	22	-21
9	24	-23
8	93	90
7	22	18
6	60	-55
5	27	-24
4	97	87
3	23	16
2	151	-137
1	31	-26
0	66	68
-1	K	1
0	51	50
1	56	-54
2	26	-22
3	38	32
4	48	43
5	50	-47
7	33	30
8	36	34
9	30	-32
11	11	16
12	20	19
0	K	1
12	42	-38
8	51	-53
7	39	-42
5	73	76
4	38	-38
3	44	-39
2	183	177
1	20	18
0	19	-13
1	K	1
0	8	7
1	119	-128
2	76	80
3	59	62
4	7	-10
5	106	-112
6	39	42
7	35	39
8	16	-19
9	53	-61
10	18	20
11	34	40
12	19	-15
2	K	1
12	25	-24
11	17	-15
10	12	15
9	26	28
8	29	-35
7	17	20
6	42	45
5	70	75
4	18	-21
3	15	17
2	108	120
1	55	61
0	8	9
3	K	1
0	97	104
1	109	119
2	20	-22
3	81	-93
4	68	73
5	84	94
6	23	-24
7	54	-61
8	33	36
9	49	55
10	25	-22
11	43	-48
4	K	1
12	18	-18
10	51	59
9	11	-16
8	23	-23
7	46	-48
6	76	85
5	46	-53
4	48	-54
3	51	-58
2	103	116
1	23	-27
0	71	-73
5	K	1
0	68	74
1	25	28
2	32	-36
3	9	-10
4	59	64
5	25	26
6	16	-17
8	47	49
9	17	16
10	5	-9
6	K	1
11	27	29
10	77	-75
9	13	-15
8	39	41
7	51	53
6	61	-66
5	19	-20
4	123	131
3	63	68
2	11	-13
1	42	-47
0	188	207
7	K	1
0	56	60
1	31	31
2	54	57
3	24	-24
4	24	24
5	32	32
7	18	-17
8	15	-13
9	27	26
8	K	1
10	34	40
9	29	-32
8	23	24
7	52	52
6	18	19
5	41	-43
4	28	-30
3	67	69
2	38	-40
1	61	-63
0	77	-78
9	K	1
0	42	-44
1	68	-69
2	32	33
3	80	81
4	30	-33
5	47	-47
6	36	35
7	63	63
8	11	-17
9	36	-36
10	26	29
10	K	1
10	30	-26
7	32	-30
6	14	-14
4	38	40
3	34	-36
2	20	19
1	12	10
0	69	66
11	K	1
0	30	-28
2	68	68
3	25	-24
4	32	-32
5	20	-22
6	45	46
7	24	-23
8	34	-33
12	K	1
8	24	-24
6	38	39
5	54	57
4	32	-34
2	47	47
1	44	41
0	40	-38
13	K	1
0	45	47
2	33	-34
3	28	27
4	38	42
6	26	-28
7	24	23
-13	K	2
7	10	-13
5	25	-22
4	21	20
3	18	-14
2	20	16
0	25	28
-12	K	2
0	55	-61
1	31	-33
2	89	88
3	43	39
4	62	-58
5	28	-24
6	73	67
7	35	30
8	52	-43
9	18	-22
-11	K	2
5	17	17
4	20	18
3	25	26
2	21	19
0	34	34
-10	K	2
1	15	14
2	11	8
3	14	-12
6	22	18
8	8	10
10	18	17
-9	K	2
10	46	44
9	49	46
8	23	-19
7	99	-91
6	69	64
5	61	56
4	40	-37
3	123	-115
2	74	70
1	91	90
0	48	-50
-8	K	2
0	90	93
1	13	-14
2	37	34
3	14	9
4	61	57
5	15	-16
8	10	10
9	15	-13
10	23	-23
-7	K	2
10	27	-27
8	12	11
7	13	12
6	15	-13
5	12	-9
4	64	57
3	12	15
2	29	29
1	19	-16
0	95	91
-6	K	2
0	151	147
1	81	77
2	89	-81

K	FO	FC
3	19	-16
4	128	114
5	99	87
6	70	-64
8	81	77
9	50	50
10	45	-44
11	18	-16

-5	K	2
11	27	25
10	44	43
7	75	69
6	53	47
4	47	-43
3	101	91
2	32	29
1	48	-44
0	81	-76

-4	K	2
0	26	26
1	82	-74
2	26	23
3	15	11
4	12	11
5	95	-82
9	42	-39
11	25	25

-3	K	2
11	44	45
10	18	-14
9	68	-65
8	25	22
7	47	44
6	14	-12
5	134	-121
4	49	42
3	92	85
1	154	-144
0	58	56

-2	K	2
0	45	-43
1	116	113
2	101	92
3	70	-66
4	48	-43
5	105	98
6	72	64
7	39	-35
8	43	-40
9	56	53
10	37	37
11	37	-37
12	31	-27

-1	K	2
12	27	-23

K	FO	FC
9	33	31
8	32	-31
7	40	38
6	28	26
5	79	74
4	9	5
3	55	54
2	95	90
1	37	38
0	52	53

0	K	2
0	87	-87
1	44	-44
2	25	23
3	18	18
4	45	-46
5	42	-41
6	47	46
7	12	8
8	11	-11
9	21	-18
10	37	42
11	17	18

1	K	2
12	44	54
11	39	-42
10	26	-25
9	31	32
8	76	81
7	93	-96
6	61	-63
5	27	25
4	78	79
3	134	-138
2	136	-141
1	67	70
0	70	71

2	K	2
0	80	80
1	19	20
2	56	-63
4	54	56
5	16	18
6	33	-37
7	8	-10
8	14	17
10	29	-27

3	K	2
12	20	-21
10	19	-18
8	20	-21
7	6	-9
6	11	10
5	14	-15
4	36	37
3	4	-5

K	FO	FC
2	75	81
1	16	-15
0	94	95

4	K	2
0	160	170
1	100	-108
2	44	-48
3	93	103
4	109	117
5	71	-78
6	43	-48
7	63	71
8	66	72
9	54	-58
10	37	-36
11	36	38

5	K	2
10	23	30
8	20	22
7	16	15
6	27	28
5	7	-10
3	20	22
1	20	-22
0	26	-24

6	K	2
0	48	52
2	36	38
3	17	-20
4	23	25
5	13	-13
6	13	10
7	15	-16

7	K	2
11	32	35
10	36	38
9	38	-42
8	48	-52
7	34	40
6	65	70
5	64	-71
4	45	-52
3	51	55
2	106	112
1	80	-73
0	42	-46

8	K	2
0	39	40
1	45	47
2	15	18
3	26	-28
4	25	26
5	43	47
7	24	-21
9	22	25

K	FO	FC
9	K	2
10	15	-12
9	26	22
8	14	14
5	46	44
4	32	35
2	5	9
1	29	32
0	48	50

10	K	2
0	33	-33
1	40	42
2	38	38
3	60	-62
4	21	-22
5	23	24
6	41	42
7	45	-48
9	26	25

11	K	2
8	38	39
6	22	-25
5	18	-18
4	49	48
2	36	-35
1	15	-16
0	53	50

12	K	2
0	56	53
3	29	27
4	35	36
6	22	-21
7	28	25

13	K	2
7	18	-14
5	20	17
4	28	32
3	16	-19
1	26	19
0	52	49

-13	K	3
0	49	-57
1	25	26
3	17	-15
4	36	-32
5	32	29
6	35	30

-12	K	3
8	29	27
6	35	-33
4	52	48
2	28	-28
0	53	61

K	FO	FC
-11	K	3
0	33	33
1	61	63
2	51	49
3	83	-78
5	44	42
6	13	7
7	70	-64
8	30	-24
9	33	32

-10	K	3
8	32	31
7	14	-13
6	20	-18
4	36	33
3	12	-12
2	34	-34
0	30	31

-9	K	3
0	29	25
1	45	-46
3	89	83
4	30	26
5	23	-19
7	70	64
8	31	27
9	27	-28

-8	K	3
10	45	-39
8	65	58
7	20	-13
6	55	-49
4	100	91
3	17	-15
2	59	-54
1	12	13
0	113	112

-7	K	3
0	17	17
1	53	-51
2	67	61
3	18	-13
5	86	-77
6	30	27
7	13	-12
8	18	-16
9	37	-34
11	21	20

-6	K	3
10	48	46
8	60	-53
7	19	16
6	88	78
4	65	-59

K	FO	FC
3	20	21
2	128	116
0	59	-57

-5	K	3
0	97	94
1	36	-34
2	9	-10
3	68	63
4	68	62
5	11	-10
6	24	-22
7	56	52
8	25	23
9	19	-22
10	27	-25

-4	K	3
11	33	-27
10	23	24
9	40	34
8	20	-14
7	39	-37
6	48	43
5	60	55
4	12	-9
3	56	-51
2	76	71
1	73	69
0	7	8

-3	K	3
0	10	9
1	153	150
2	19	18
3	148	-141
4	19	16
5	99	94
6	33	31
7	99	-93
8	31	29
9	70	67
10	27	29
11	51	-47

-2	K	3
12	20	-19
11	26	24
9	24	-22
8	15	-14
7	26	26
6	27	24
5	44	-44
4	40	38
3	32	32
2	99	96
1	57	-55
0	105	107

-1	K	3

K	FO	FC
0	175	173
1	63	61
2	78	-79
3	27	-26
4	122	118
5	54	52
6	78	-76
7	12	-13
8	45	44
9	33	32
10	71	-66
12	21	26

0	K	3
12	38	47
10	35	-33
8	71	71
7	28	26
6	66	-65
5	24	23
4	84	83
3	33	33
2	128	-127
1	7	8
0	84	80

1	K	3
0	69	-66
1	46	46
2	63	62
3	21	22
4	52	-53
5	55	58
6	56	57
7	26	23
8	22	-22
9	20	20
10	44	47
12	20	-15

2	K	3
11	33	37
10	7	-11
9	64	-69
8	20	22
7	34	38
6	12	-16
5	115	-120
4	34	35
3	61	61
2	27	-27
1	129	-131
0	41	39

3	K	3
0	16	16
1	81	-83
2	50	52
3	9	11
5	96	-103

K	FO	FC
6	23	22
8	18	-15
9	48	-51
11	26	29

4	K	3
11	37	-40
9	57	61
7	36	-38
6	28	28
5	98	106
3	51	-54
2	41	42
1	103	107

5	K	3
0	26	-25
2	92	99
3	33	37
4	36	-38
5	25	27
6	50	53
7	30	32
8	43	-47
10	21	25

6	K	3
10	18	21
9	13	6
8	15	-13
7	16	-22
6	30	28
4	22	-24
3	23	-26
2	32	33
1	9	13
0	28	-31

7	K	3
0	55	54
1	16	14
2	96	-103
3	55	-58
4	65	70
5	19	-17
6	46	-50
7	46	-49
8	71	78
10	20	-20

8	K	3
8	15	-12
7	32	-34
6	21	18
5	22	26
4	11	11
3	37	-39
2	48	52
1	35	36
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2	30	30
1	24	-25

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OHMILITE FROM OHMI, NIIGATA PREF.

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5	17	18
6	23	-25
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8	24	24
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9	18	19
8	46	-46
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OHMILITE FROM OHMI, NIIGATA PREF.

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7	27	-28

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7	14	11
6	11	9
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5	68	67
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9	51	50

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8	9	13
6	16	-16
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7	29	27

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2	62	-59
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4	11	-11
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OHMILITE FROM OHMI, NIIGATA PREF.

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