Matsubaraite, Sr₄Ti₅(Si₂O₇)₂O₈, a new mineral, the Sr-Ti analogue of perrierite in jadeitite from the Itoigawa-Ohmi district, Niigata Prefecture, Japan

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Abstract: Matsubaraite, $Sr_4Ti_5(Si_2O_7)_2O_8$, is a new member of the perrierite-chevkinite group found in the jadeitite from the Itoigawa-Ohmi district, central Japan. It is monoclinic, $P2_1/a$ (pseudo C2/m), a = 13.848(7), b = 5.626(2), c = 11.878(6) Å, $\beta = 114.19(4)^\circ$, V = 844 Å³, and Z = 2. The six strongest lines in the X-ray powder diffraction pattern are 3.16 (70) (400), 3.09 (95) (-403), 3.01 (90) (-313), 2.96 (95) (311), 2.71 (100) (004), 2.17 (90) (-421). Electron microprobe analysis gave SiO₂ 22.60, TiO₂ 39.06, SrO 38.84 total 100.50 wt.%, corresponding to $Sr_{3.92}Ti_{5.11}Si_{3.93}O_{22}$ on the basis of O = 22. The crystal structure analysis (R = 0.0372) revealed that the space group is pseudo C2/m, and that matsubaraite is Sr and Ti-analogue of perrierite-(Ce), Ce₄Fe₂Ti₃(Si₂O₇)₂O₈, or Zr-free analogue of rengeite, Sr₄ZrTi₄(Si₂O₇)₂O₈. It is transparent, grey with blue tint with adamantine luster. Streak is white, cleavage not observed. The hardness is VHN₁₀₀ 681-743 kg mm⁻² (Mohs' 5.5). The calculated density is 4.13 g cm⁻³. It occurs as long prismatic euhedral to subhedral crystal with interstitial natrolite in a boulder of lavender-colored Tibearing jadeitite from the bed of the Kotaki-gawa river, Itoigawa City, Niigata Prefecture, central Japan. Matsubaraite is considered to have crystallized during later stage activity of high-*P/T* metamorphism. The mineral is named after Dr Satoshi Matsubara

Key-words: matsubaraite, new mineral, strontium, perrierite group, jadeite, Itoigawa-Ohmi district, crystal structure.

Introduction

The Itoigawa-Ohmi district is the most famous locality for jade in Japan (Kawano, 1939; Ohmori, 1939; Iwao, 1953; Chihara, 1971). During mineralogical and petrological research on jade from the Itoigawa-Ohmi district, we have found some Sr-dominant minerals such as two new minerals, itoigawaite SrAl₂Si₂O₇(OH)₂•H₂O (Miyajima et al., 1999) and rengeite Sr₄ZrTi₄(Si₂O₇)₂O₈ (Miyajima et al., 2001), and rare minerals such as stronalsite SrNa₂Al₄Si₄O₁₆, lamprophyllite Na₂(Sr, Ba)₂Ti₃(SiO₄)₄ (OH, F)₂, slawsonite SrAl₂Si₂O₈, tausonite SrTiO₃ and strontium-apatite (Sr, Ca)₅(PO₄)₃(OH, F). The new mineral described here, matsubaraite, found as euhedral to subhedral prismatic crystals with high refractive indices within natrolite, which is interfillings of jadeite, is named in honour of Dr Satoshi Matsubara of the National Science Museum. Tokyo, for his significant contributions on mineralogy of strontium dominant minerals. The mineral data and the name have been approved by the Commission on New

Mineral and Mineral Names, International Mineralogical Association (#2000-027). The type specimens of matsubaraite are deposited at the National Science Museum, Tokyo, under the registered number NSM-M28084 and at Fossa Magna Museum, Itoigawa, Niigata, under the catalogue number FMM01309.

Occurrence

The Itoigawa-Ohmi district of the Renge belt (Nishimura, 1998) is characterized by a serpentinite melange with high-P/T type schists, jade, albitite, rodingite and metagabbro, and various fragments of Palaeozoic accretionary complexes composed of greenstone, lime-stone and chert (Nakajima *et al.*, 1992). The Itoigawa-Ohmi district is located in the easternmost part of the Renge belt, which is the oldest of the high-P/T metamorphic belts in the Japanese Islands (Fig. 1).

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Fig. 1. Geotectonic subdivision of Southwest Japan (modified from Nishimiura, 1998). The Renge Belt is typically associated with metaophiolite sequences (470-340 Ma) including serpentinite. It is also characterized by association of glaucophane-schist.

Physical and optical properties

The matsubaraite is transparent grey with blue tint with adamantine lustre. No cleavage was observed. The Vickers microhardness is 681-743 kg mm⁻² (100g load), corresponding to 5.5 on the Mohs scale. The density could not be measured due to insufficiency of material, but the calculated density is 4.13 g cm⁻³. There is no fluorescence in the either long- or short-wave ultraviolet radiation. Matsubaraite is optically biaxial positive. The refractive indices are higher than titanite, however, these could not be measured because these are too high for normal liquid. Matsubaraite can be distinguished by the defferent colour and paragenesis from rengeite. Rengeite is transparent greenish brown with adamantine lustre and often shows close association with pinkish zircon.

Chemical composition

Chemical analyses of matsubaraite were performed on a JEOL JXA-8800 Superprobe using the wavelength-dispersive mode. A conventional ZAF correction routine was used for data reduction. The accelerating voltage was 15 kV, the beam current was 20 nA, and the beam was focused to diameter 2 to 3 μ m. The standards used were wollastonite (Si), TiO₂ (Ti), SrF₂ (Sr). Backscattered electron images and chemical analysis show that the composition of matsubaraite does not vary grain-by-grain and within individual grains. The average of 6 electron microprobe measurements of matsubaraite gave SiO₂ 22.60, TiO₂ 39.06, SrO 38.84 total 100.50 wt.% (Table 1). It leads to the empirical formula, Sr_{3.92}Ti_{5.11}Si_{3.93}O₂₂ on the basis of O = 22. Consequently, the ideal formula of matsubaraite is Sr₄Ti₅Si₄O₂₂.

The matsubaraite is found in lavender-coloured jade (FMM01309), which was collected in the bed of the Kotaki-gawa river (Fig. 2). It is a boulder ~ 50 cm in diameter. The mineral occurs as a tiny prismatic crystal ~ 0.3 mm long in jadeitite (Fig. 3a). Under the microscope, the host jadeitie is composed of essentially pure jadeite, Tibearing jadeite and natrolite with minor constituents of lamprophyllite, titanite, zircon, rutile, tausonite, rengeite and matsubaraite. The specimen is characterized by the existence of nearly euhedral jadeite crystals in the natrolite matrix. Matsubaraite as euhedral prismatic crystals occurs interstitially within jadeite grains (Fig. 3b). The matsubaraite also closely associates with titanite, lamprophyllite, tausonite, rengeite and zircon (Fig. 3c), and often occurs as hollow crystal (Fig. 3d).

The chemical compositions of other members of perrierite-chevkinite group minerals are also given in Table 1.

Table 1. Chemical composition of the perrierite-chevkinite group minerals.

	1	2	3	4	5	6		
Wt.%								
SiO ₂	22.60	22.58	22.04	19.54	20.64	20.45		
TiO ₂	39.06	29.88	23.60	15.33	19.15	23.16		
ZrO ₂	0.00	9.49	10.11	0.00	0.00	10.30		
Nb ₂ O ₅	0.00 0.24		1.33	3.56	0.00	0.00		
Ta ₂ O ₅	0.00 0.07		0.01	0.00	0.00	0.00		
AI_2O_3	0.00 0.20		1.70	0.17	0.38	0.11		
FeO	0.00 0.10		1.19	11.87	8.17	6.02		
MnO	0.00	0.00	0.00	0.23	1.70	0.08		
MgO	0.00	0.00	0.00	0.29	0.40	0.00		
CaO	0.00	0.43	0.52	3.40	3.94	2.05		
SrO	38.84	34.32	28.24	0.00	1.28	19.60		
BaO	0.00	0.13	0.16	0.00	0.00	0.38		
Na ₂ O	0.00	0.00	0.00	0.00	0.47	0.02		
K ₂ O	0.00	0.00	0.00	0.06	0.00	0.00		
ThO ₂	0.00	0.00	0.00	4.84	0.68	0.00		
Y201	0.00	0.00	0.00	0.00	0.40	0.05		
La ₂ O ₃	0.00	0.00	2.73	13.08	21.01	9.18		
Ce ₂ O ₃	0.00	0.38	4.81	22.09	18.24	9.35		
Pr ₂ O ₃	0.00	0.10	0.63	0.00	2.16	0.00		
Nd ₂ O ₂	0.00	0.29	1.40	4.01	2.25	0.00		
Sm ₂ O ₂	0.00	0.04	0.06	0.00	0.00	0.00		
Tb ₂ O ₂	0.00	0.00	0.00	0.00	0.13	0.00		
Yb ₂ O ₂	0.00	0.00	0.00	0.00	0.11	0.00		
	0.00	0.00	0.00	0.00	0.11	0.00		
Total	100.50	98.25	98.53	98.47	101.11	100.75		
Number o	f cations on	the basis of	0=22					
Si	3.93	4.10	4.10 4.13 4.11			3.91		
Ti	5 11	4 08	3 33	2 43	2 89	3.33		
Zr	0.00	0.84	0.92	0.00	0.00	0.96		
Nb	0.00	0.01	0.11	0.33	0.00	0.00		
Та	0.00	0.00	0.00	0.00	0.00	0.00		
Al	0.00	0.04	0.37	0.04	0.09	0.02		
Fe	0.00	0.01	0.18	2 09	1.37	0.96		
Mn	0.00	0.00	0.00	0.02	0.18	0.00		
Ma	0.00	0.00	0.00	0.02	0.10	0.00		
Ca	0.00	0.08	0.00	0.76	0.84	0.42		
Sr	3.91	3.61	3.07	0.00	0.14	2 17		
Ba	0.00	0.00	0.01	0.00	0.00	0.02		
Na	0.00	0.00	0.00	0.00	0.00	0.02		
K	0.00	0.00	0.00	0.00	0.00	0.00		
Th	0.00	0.00	0.00	0.23	0.00	0.00		
v	0.00	0.00	0.00	0.20	0.00	0.00		
a	0.00	0.00	0.00	1 01	1 55	0.00		
Co	0.00	0.00	0.10	1.01	1.30	0.65		
Pr	0.00	0.02	0.00	0.00	0.15	0.00		
Nd	0.00	0.00	0.04	0.00	0.15	0.00		
Sm	0.00	0.01	0.09	0.50	0.10	0.00		
Th	0.00	0.00	0.00	0.00	0.00	0.00		
Vb	0.00	0.00	0.00	0.00	0.00	0.00		
TD I	0.00	0.00	0.00	0.00	0.00	0.00		
Total	12.95	12.88	12.91	13.18	13.32	13.13		
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1: matsubaraite		1	(nis study) Mixelime et al. (2004)					

1. matsabarance	this study
2: rengeite	Miyajima et. al. (2001)
3: rengeite	Miyajima et. al. (2001)
4: chevkinite	Imaoka & Nakashima (1994)
5: perrierite	Segalstad & Larsen (1978)
6: strontio-chevkinite	Haggerty & Mariano (1983)



Fig. 2. Index map of the Itoigawa-Ohmi district (modified from Komatsu, 1990).

The previously known mineral with composition close to matsubaraite is rengeite, which is a member of the perrieritechevkinite group found in the jadeitite from the Itoigawa-Ohmi district. Rengeite is characterized by very high Sr, Ti and Zr, and very low *REE* and Fe compared to perrierite, chevkinite and strontiochevkinite (Miyajima *et al.*, 2001). Matsubaraite has a very simpler composition than rengeite, with a very high content of Sr and Ti, and no *REEs* and other components. The polymorphs perrierite and chevkinite have the ideal stoichiometry $A_4^{3+}B^{2+}C_2^{3+}Ti_2Si_4O_{22}$ (Ito, 1967) where A = REE, B = (Fe, Mg), C = (Fe, Al). In matsubaraite, it is though that the A, B and C sites are occupied by Sr²⁺, Ti⁴⁺ and Ti⁴⁺ cations, respectively.

X-ray crystallography and crystal structure

The X-ray powder diffraction pattern for matsubaraite was obtained using a Gandolfi camera of 114.6 mm diameter employing Ni-filtered CuK α radiation. A fragment of the single crystal of matsubaraite was picked up from the thin section, which was analysed for chemical composition, under a binocular microscope with an effort to reduce the contamination of associate minerals such as jadeite and



Fig. 3. (a) Close up photograph of matsubaraite showing fan shape aggregate of long prismatic crystals in the lavender-coloured jadeitite (NSM-M 28084 and FMM01309) from the Kotaki-gawa river, the Itoigawa-Ohmi district.

(b) Photomicrograph (PPL) of matsubaraite showing euhedral prismatic crystal with high refractive indices.

(c) Back-scattered electron image of aggregate composed of matsubaraite, titanite, zircon and some Sr dominant minerals.

(d) Back-scattered electron image of matsubaraite showing hollow crystal in interstitial natrolite.

Abbreviations: Jad = jadeite, Lam = lamprophyllite, Mat = matsubaraite, Nat = natrolite, Ren = rengeite, Ttn = titanite and Zir = zircon.

natrolite, and was put on a glass fiber (10 μ m in diameter). Because matsubaraite is free from uranium and thorium, it is nonmetamict and gives excellent diffraction profile. The powder X-ray diffraction data of matsubaraite are given in Table 2. The unit cell parameters were refined from the powder X-ray diffraction data with internal Si standard (NBS, #640b) using a computer program by Toraya (1993); a = 13.848(7), b = 5.626(2), c = 11.878(6) Å, $\beta = 114.19(4)^{\circ}$ and V = 844 Å³.

The single crystal fragment was investigated with a precession camera and a Rigaku RASA-7R four-circle diffractometer. The intensity data were collected with the diffractometer using graphite monochromatized MoK α radiation (50 kV, 250 mA). Experimental details of the data collection are given in Table 3. Several very weak reflections, which violate the extinction rule for C2/m, h + k =2n+1 for *hkl*, were observed in the single crystal investigation on matsubaraite. Therefore the true space group of matsubaraite is $P2_1/a$, as for rengeite (Miyajima *et al.*, 2001; Miyawaki *et al.*, 2002) and synthetic material,

 $La_4Mg_2Ti_3(Si_2O_7)_2O_8$ with the perrierite type structure (Calvo & Faggiani, 1974). Two models with the space groups of $P2_1/a$ and C2/m were examined in the present calculation of refinements. The result of the crystal structure analysis of perrierite-(Ce) by Gottardi (1960) and that of a related synthetic material by Calvo & Faggiani (1974) were used as the initial parameters of C2/m and $P2_1/a$ models, respectively. The computer program package for crystal structure analysis 'teXsan' (1993) recommended the C-lattice, and the refinement converged with R = 0.025, $R_{\rm w} = 0.028$ for 1490 observed reflections with I > 3σ (I), whereas the calculation with the $P2_1/a$ model was unstable. Then, the data reductions to F_0^2 with corrections for Lorentz, polarization and absorption (ψ -scan procedure) were made with a computer program by Dr. Kazumasa Sugiyama of the University of Tokyo (personal communication). The computer program, SHELXL-97 (Sheldrick, 1997), was employed for the further refinement of crystal structure. Scattering factors for neutral atoms and anomalous dispersion factors were taken from the International

Table 2. Powder X-ray diffraction data and lattice parameters of matsubaraite and related minerals.

		matsubaraite Itoigawa, Japan (Present study)		rengeite Itoigawa-Ohmi, Japan (Miyajima <i>et al .</i> , 2001)		perrierite Oslo, Norway (Segaistad and Larsen, 1978)			perrierite Amherst, Virginia, USA (Mitchell, 1966)					
h	k	1		d (obs)	d (calc)		d (obs)	d (calc)		d (obs)	d (calc)		d (obs)	d (calc)
0	0	1	50	10.81	10.83	~~~	10.92	10.92				~~	10.92	10.76
-2 2	0	1 0	10	6.36	6.80 6.32	vw vw	6.86 6.37	6.87 6.37						6.68 6.24
0 -2	0	2			5.42 5.33	w	5.45	5.46	vw	5.40	5.41	w	5.41	5.38 5.24
-1	1	0	30 20	5.16 5.00	5.14 4.98	Ŵ	5.19 5.00	5.18 5.02	w	5.16 5.00	5.16 4.99	Ŵ	5.19 5.01	5.17 4.99
1	1	1	30	4.39	4.68	~~~~	4.72	4.72		4.00	4.00	vw	4.40	4.65
-2	0	233	40 10	4.09 3.91 3.62	4.00 3.90 3.61	m	4.15 3.94 3.65	4.12 3.93 3.64	w	4.09	4.09	vvw	3.85	4.08 3.84 3.59
-3	1	1	20	3.57	3.57	Ŵ	3.60	3.60	w	3.01	3.01	m	3.56	3.54
1	1	2	30	3.45	3.45	w	3.49	3.48	w	3.47	3.46	w	3.47	3.45
-3	1	2			3.41	w	3.44	3.44	~~	5.55	5.40		3 3 1	3.38
3	1	03			3.37							1000	3.27	3.36
4 -4	0 0	0 3	70 95	3.16 3.09	3.16	m	3.20 3.12	3.19 3.11	w	3.14	3.13	Ŵ	3.14	3.12
-3 -2	1	3 4	90	3.01	3.00 2.97	vvs	3.05	3.03	s	2.99	2.99	s	2.99	2.97
3	1 2	1	95 50	2.96 2.81	2.96 2.81	vs s	2.99 2.84	2.98 2.84	s m	2.96 2.85	2.65 2.83	s m	2.96 2.84	2.95
1	1 0	3	50	2.75	2.75	m	2.78	2.77	w	2.75	2.75	w	2.74	2.74
0	2	1	100	2.71	2.72	s	2.74	2.73	m	2,70	271	mw	2 6 9	2.75
2 -4	0	3			2.70 2.67	w	2.69	2.69	vw	2.64	2.64	w	2.63	2.68
-2 -1	2 1	1	20	2.58	2.60 2.58	vw vvw	2.62 2.59	2.62 2.60				vw	2.56	2.61 2.57
2 -3	2 1	0 4			2.57 2.56				vw	2.58	2.58			2.58 2.53
3 0	1 2	2 2	20 45	2.52 2.50	2.52 2.50	vw m	2.54 2.51	2.54 2.52	w vw	2.52 2.50	2.51 2.51	w	2.51	2.51 2.51
-2 -5	2 1	2 2	10	2.45	2.49 2.48			2.50	vw	2.47	2.46	~~~~	2.47	2.50
2 4	2 0	1 2	20 25	2.41 2.35	2.41 2.34	Ŵ	2.44 2.38	2.43 2.36						
-6 -2	0 2	2 3	15 20	2.32 2.30	2.31 2.28	vw	2.33	2.33				~~~~	2.28	
-4 1	0 1	5 4	30	2.28	2.27 2.25	m	2.30	2.29	vw	2.25	2.25	vw	2.24	
-6 0	0 2	1 3	15 45	2.25 2.22	2.25 2.22	vw	2.27	2.27						
-5 -3	1	4 5			2.21 2.18	s	2.20	2.19				m	2.18	
-4 -4	2	1 2	90	2.17	2.18 2.17			2.20 2.19	w	2.18	2.17			
-6 3	0	4	35	2.14	2.14 2.14	vw	2.16	2.16 2.16	vw	2.14	2.14			
-1	1	5	25	2.11	2.13 2.11	w	2.13	2.14 2.13				~~~~	2.11	
-4	2	3			2.10 2.08	vw	2.10	2.13 2.10	vw	2.08	2.10	vw	2.07	
-2	2	4	95	1.950	1.962	m	1.978	1.978	w	1.96	1.96			
2	2	3			1.949		4.029	1.040				m	1.95	
5	1	2	15	1.883	1.882	Ŵ	1.896	1.898						
-7	1	3	15	1.836	1.858	w	1.077	1.875						
2	02	5	25	1.000	1.832			1 8 2 7						
-7 -7	1	1	20	1.012	1.810	wwb	1 8 1 8	1.821						
-1 -1	1	6 2	30	1.802	1.800	w	1.802	1.815						
-6 -5	2	2	25	1 782	1.784		1.002	1.800						
-6 -6	0	6 3			1.777	w	1 784	1 781						
-6 6	20	1	15 40	1.756 1.737	1.755	w	1.769 1.751	1.771 1.751						
4 -8	0	4 3	60	1.731	1.734 1.730			1.746						
-8 -4	0 0	2 7	25	1.720	1.717 1.690	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	1.732 1.707	1.732 1.705						
-8 -3	0 3	1 3	30	1.660	1.663 1.659	w	1.674	1.677 1.673						
3 -3	3 1	1 7	35 20	1.652 1.625	1.650 1.623	Ŵ	1.665 1.635	1.664 1.636						
7 3	1 1	1 5	25	1.603	1.603 1.600	w	1.613	1.617 1.612						
-7 -5	1	6 7	20	1.590	1.593 1.589	w	1.604	1.608 1.603						
6 0	2	1 7	25	1.550	1.588 1.548	w	1.562	1.560						
-4 8	0	8			1.484 1.478	√√wb	1.490	1.491						
-7	2	4			1.476 1.470	vvwb	1.482	1.488 1.483						
-3 -2	3	5	15	1.436	1.468 1.436	w	1.444	1.481 1.448						
-8	2	1 7	15	1.433	1.431	w	1.428	1.444						
-8	4	507	20	1.408	1.413	w	1.419	1.426						
-6	2	3	25	4 35 4	1.390	~~~	1.401	1.402						
-6	2	8	20	1.354	1.354									
0	4	4	25	1.249	1.248									
0 -11	2	8 5	15	1.219	1.220									

Table 3. Crystallographic data and experimental details*

a (Å)	13.850(2)
b (Å)	5.631(2)
c (Å)	11.8920(10)
β (°)	114.143(7)
V (Å ³)	846.3(3)
Space group	C2/m*
Z	2
Formula	Sr ₄ Ti ₅ (Si ₂ O ₇) ₂ O ₈
D _{calc} (g/cm ³)	4.137
μ (cm ⁻¹)	15.137
Crystal dimension (mm)	0.10 x 0.08 x 0.03
Diffractometer	Rigaku AFC-7R
Radiation	Mo $K\alpha$ (graphite)
Scan mode, rate (°/ min in ω)	2θ-ω, 1
2θ range	6.0 - 70.0
Reflection range	-22 = h = 22,
	-9= k = 0,
	-19 = = 19
No. of measured reflections	4032
No. unique reflections	2018
No. of observed reflections $[I > 2\sigma(I)]$	1549
R _{int}	0.0334
Variable parameters	97
R1 [$F_o > 4\sigma(F_o)$], R1(all reflections)	0.0316, 0.0556
wR2 (all reflections)	0.1058
Weighting parameters, a, b	0.1, 0
Goodness of Fit	0.781
Final Δρ _{min} (e/ų)	3.247
Final ∆ρ _{max} (e/ų)	-2.367

 $\begin{aligned} \mathsf{R1} &= \Sigma \mid |\mathsf{F}_{o}| - |\mathsf{F}_{o}| \mid / \Sigma \mid \mathsf{F}_{o}| \\ \mathsf{wR2} &= \Sigma \left[\mathsf{w}(\mathsf{F}_{o}^{\ 2} - \mathsf{F}_{o}^{\ 2})^{2}\right] / \Sigma \left[\mathsf{w}(\mathsf{F}_{o}^{\ 2})^{2}\right]^{0.5} \\ \mathsf{w} &= 1 / \left[\sigma^{2}(\mathsf{F}_{o}^{\ 2}) + (\mathsf{aP})^{2} + \mathsf{bP}\right] \\ \mathsf{P} &= \left[2\mathsf{F}_{o}^{\ 2} + \mathsf{F}_{o}^{\ 2}\right] / 3 \end{aligned}$

* The result of refinement with the space group of C2/m is basically consistent that with $P2_1/a$, but is better, *i.e.*, positive anisotropic displacement parameters for all the sites, than $P2_1/a$. See the text.

Tables for X-ray Crystallography, Volume C (1992). Fullmatrix least-squares refinement was performed by refining positional parameters, scale factor, and displacement parameters. The site occupancy factors (*sof*) were not refined because this specimen does not contain any detectable substituents. The refinement with C2/m model using anisotropic displacements converged successfully. Strong correlations among parameters were found, and many of the equivalent isotropic displacement parameters had non-positive values in the calculation with $P2_1/a$ model with anisotropic displacement parameters. The calculation

Table 4. Final atomic coordinates and equivalent isotropic displacement parameters.

	x	У	z	Ueg
Sr(1)	0.76487(3)	0.0000	0.73287(4)	0.00758(10)
Sr(2)	0.54607(3)	0.5000	0.73841(4)	0.00788(11)
Ti(1)	0.0000	0.5000	0.5000	0.00488(19)
Ti(2)	0.5000	0.74369(15)	0.0000	0.00573(14)
Ti(3)	0.27126(6)	0.0000	0.00074(8)	0.00616(15)
Si(1)	0.40654(9)	0.0000	0.73070(11)	0.0045(2)
Si(2)	0.84194(10)	0.0000	0.45339(12)	0.0052(2)
O(1)	0.06287(18)	0.2652(4)	0.1824(2)	0.0069(4)
O(2)	0.29210(18)	0.2491(4)	0.1185(2)	0.0064(4)
O(3)	0.39456(18)	0.2601(5)	0.4189(2)	0.0083(4)
O(4)	0.0937(3)	0.0000	0.9998(3)	0.0076(6)
O(5)	0.4046(3)	0.0000	-0.0007(3)	0.0076(6)
O(6)	0.4709(3)	0.0000	0.6442(3)	0.0087(6)
0(7)	0.2791(3)	0.0000	0.6563(3)	0.0100(6)
O(8)	0.1478(3)	0.0000	0.4109(3)	0.0103(6)

with the $P2_1/a$ model with isotropic displacements converged to R1 [F_o > 4 σ (F_o)] = 0.0372, wR2 (all reflections) = 0.1514, Goodness of Fit = 0.816. The result of refinement with the *C2/m* model is summarized in Table 3. The final positional parameters and equivalent isotropic displacement parameters are given in Table 4. Table 5 shows anisotropic displacement parameters. Selected interatomic distances and bond angles are summarized in Table 6.

The atomic positions in $P2_1/a$ model with the isotropic displacement parameters are basically consistent with those in C2/m model. The deviations of atomic positions in $P2_1/a$ model from the individually corresponding positions in the C2/m model are negligible small, except for x of Ti(2) and y of O(7), O(8) and O(9). In the $P2_1/a$ model with lower symmetry, the imaginary pseudo-mirror planes at y = 0 and 0.5 are sustained, generally.

Discussion

Paragenesis

The occurrence of jadeite as euhedral to subhedral crystal with interstitial natrolite (Fig. 3b) implies that jadeite have been crystallized in the free space such as in metamorphic fluid. Matsubaraite occurs as isolated long prismatic crystal and fan-shape aggregates with euhedral to subhedral jadeite in the natrolite matrix (Fig. 3a, b). It is clear that matsubaraite and jadeite were formed before natrolite. The matsubaraite may be formed in the same stage of the crystallization of jadeite. The matsubaraite closely associates with titanite, lamprophyllite, tausonite, rengeite and zircon (Fig. 3c). Titanium may be relatively immobile element during metamorphism (e.g. Tatsumi & Kogiso, 1995). Therefore titanite may have been the sources for the Ti of matsubaraite. Matsubaraite never shows a direct contact with zircon. In contrast, rengeite usually shows close association contact with zircon directly. These facts support the idea that matsubaraite could not be crystallized at near side of zircon under the existence of

Table 5. Final anisotropic displacement parameters.

	U11	U ₂₂	U ₃₃	U ₂₃	U ₁₃	U ₁₂
Sr(1)	0.00811(17)	0.00870(19)	0.00640(19)	0.000	0.00344(13)	0.000
Sr(2)	0.00790(17)	0.0100(2)	0.00602(19)	0.000	0.00315(13)	0.000
Ti(1)	0.0069(4)	0.0054(4)	0.0027(4)	0.000	0.0024(3)	0.000
Ti(2)	0.0091(3)	0.0045(3)	0.0032(3)	0.000	0.0021(2)	0.000
Ti(3)	0.0083(3)	0.0048(3)	0.0052(3)	0.000	0.0026(2)	0.000
Si(1)	0.0058(4)	0.0061(5)	0.0019(5)	0.000	0.0020(4)	0.000
Si(2)	0.0059(4)	0.0056(5)	0.0039(5)	0.000	0.0018(4)	0.000
O(1)	0.0098(8)	0.0066(9)	0.0041(9)	0.0008(8)	0.0025(7)	-0.0010(8)
O(2)	0.0104(9)	0.0055(9)	0.0034(9)	-0.0002(8)	0.0029(7)	0.0000(8)
O(3)	0.0095(9)	0.0077(9)	0.0077(10)	0.0004(8)	0.0033(7)	0.0017(8)
O(4)	0.0067(12)	0.0054(13)	0.0114(15)	0.000	0.0044(11)	0.000
O(5)	0.0062(11)	0.0075(14)	0.0098(15)	0.000	0.0038(11)	0.000
O(6)	0.0101(13)	0.0117(15)	0.0050(14)	0.000	0.0038(11)	0.000
0(7)	0.0065(12)	0.0140(16)	0.0069(14)	0.000	0.0000(11)	0.000
O(8)	0.0127(14)	0.0131(16)	0.0058(14)	0.000	0.0044(12)	0.000

both Ti and Zr. The rengeite may be formed instead of matsubaraite under such a condition.

Some strontium dominant minerals such as matsubaraite, rengeite, lamprophyllite and tausonite have been found in the host jadeitite. These minerals have been crystallized at the same or the later stage of formation of jadeite. Both Sr and Ca are member of alkali-earth elements, however, the chemical affinity of these elements for clinopyroxene is quite different. Matsui et al. (1977) shows trace element partitions between phenocrysts and groundmass. According to their study, the partition coefficients of Sr and Ca between augite phenocryst and groundmass are 0.1 and 2.0, respectively. This difference is attributed to their different ionic radii, which are 1.21 and 1.08 Å, respectively (Shannon & Prewitt, 1969). Strontium was not detected in the present chemical analysis of jadeite. The ionic radius of Sr might be too large to be accepted in the six-fold coordinated M2 site in jadeite. Therefore Sr is not distributed into jadeite, and Sr content in residual metamorphic fluid is significantly increased after crystallization of jadeite. Consequently, the Sr is distributed into Sr-dominant minerals such as matsubaraite, rengeite, tausonite and lamprophyllite. Matsubaraite is considered to have crystallized by interaction between pre-existing titanite and Srrich residual metamorphic fluid during high-P/T metamorphism in the subduction zone.

Structure of matsubaraite

Perrierite and chevkinite are dimorphous. They are distinguished by their different monoclinic β angle (113û and 100û respectively), associated with different cation-oxygen bond length patterns in their structures (Calvo & Faggiani, 1974). Two space groups, *C2/m* and *P2*₁/*a* are reported for the chevkinite- and perrierite-type materials with general formula of $A_4BC_2\text{Ti}_2(\text{Si}_2\text{O}_7)_2\text{O}_8$, where $A = \text{Ce}^{3+}$, La³⁺, Th⁴⁺, Ca²⁺, Na⁺ and the other rare earths, B =

Fe²⁺, Mg²⁺, Ca²⁺ and the other transition metals, $C = Ti^{4+}$, Fe³⁺, Fe²⁺, Mg²⁺ (Pen & Pan, 1964; Gottardi, 1960; Calvo & Faggiani, 1974; Yang *et al.*, 1991; Miyawaki *et al.*, 2002). The true space groups for minerals and synthetic materials in the chevkinite-perierite group are under discussion.

The crystal structure of matsubaraite could be refined with the space group of C2/m with a better result, *i.e.*, positive anisotropic displacement parameters for all the sites, in comparison with $P2_1/a$. The atomic coordinates are basically consistent with each other in the two models, C2/mand $P2_1/a$. Therefore, the diffractions out of keeping with C2/m are so weak. The space group of rengeite can be regarded as pseudo-C2/m, though the true space group of rengeite is $P2_1/a$.

The coodination polyhedra of Sr^{2+} ions in matsubaraite is as large as those of Sr^{2+} ions in rengeite, and is larger than those of lanthanides in perrierite and its synthetic La-Mg-analogue (Table 6). On the other hand, the Ti(1)O₆octahedron in matsubaraite is smaller than the corresponding octahedra in rengeite (ZrO₆), perrierite (FeO₆) and the synthetic La-Mg-analogue (MgO₆), respectively. According to Ito & Arem (1971), the perrierite structure is stabilized with larger cations in the *A* site and smaller cations in the octahedral *B* and *C* sites, relative to the chevkinite structure. The crystal structure of matsubaraite may be the most stable among the structures of perrierite-(Ce), rengeite, La₄Mg₂Ti₃(Si₂O₇)₂O₈, and matsubaraite.

The Si(1)-O(7)-Si(2) angle, through the bridging oxygen atom of the Si₂O₇ disilicate group, in the crystal structure of matsubaraite, 163.7°, is similar to that of La₄Mg₂Ti₃(Si₂O₇)₂O₈ [perrierite type], 165.6°, and is slightly bended than that of rengeite, 170.0° (Table 6). Calvo & Faggiani (1974) pointed out that the displacement parameter for the bridging O(7) is the largest among the O sites in the perrierite or chevkinite structures. Such a fea-

Table 6. Interatomic distances (Å	A) and bond angles (û) of matsubara	ite. Those of rengeite, perrier	rite-(Ce), and Mg ₂ La ₄ T	i ₃ Si ₄ O ₂₂ are giver
for comparison. The mean distan	nce is given in the last of each data	set of polyhedron.		

Matsubaraite present study		Rengeite (Miyawaki et al., 2001)		Perrierite-(Ce) (Gottardi, 1960)		Mg₂La₄Ti₃Si₄O₂₂ (Calvo & Faggiani, 1974)	
Sr(1)-O(8) -O(2) -O(2) -O(1) -O(3) -O(3) -O(3) -O(4) -O(7) -O(7) -Sr(1)-O> ₈ <sr(1)-o>₁₀</sr(1)-o>	2.465(4) 2.619(2) 2.649(2) 2.640(2) 2.640(2) 2.649(3) 2.649(3) 2.975(4) 2.9903(16) 2.9903(16) 2.9903(16) 2.9903(16)	$\begin{tabular}{ c c c c c c } \hline Sr(1)-O(8) & & & -O(1) & & & \\ -O(1) & -O(2) & & & & & \\ -O(2) & -O(2) & & & & & \\ -O(3) & -O(3) & & & & & \\ -O(3) & -O(3) & & & & & \\ -O(7) & -O(7) & & & & & \\ -Sr(1)-O>_8 & & & & \\ _{10} & & & & \\ \hline \end{tabular}$	2.505(5) 2.591(3) 2.691(3) 2.637(3) 2.637(3) 2.667(4) 2.667(4) 2.977(5) 3.043(2) 3.043(2) 2.659 2.736	$\begin{array}{c} \hline Ce(1){-}O(3) \\ -O(3) \\ -O(1) \\ -O(1) \\ -O(8) \\ -O(2) \\ -O(2) \\ -O(2) \\ -O(7) \\ -O(7) \\ $	2.36 2.36 2.37 2.37 2.44 2.48 2.48 2.48 2.93 3.02 3.02 2.47 2.58	$\begin{matrix} \text{La}(1)\text{-}O(3) \\ -O(2) \\ -O(8) \\ -O(2') \\ -O(1) \\ -O(1') \\ -O(3') \\ -O(7) \\ -O(5) \\ <\text{La}(1)\text{-}O_8 \\ <\text{La}(1)\text{-}O_9 \end{matrix}$	2.468 2.503 2.511 2.549 2.553 2.556 2.591 2.860 3.452 2.574 2.671
Sr(2)-O(8) -O(1) -O(2) -O(2) -O(3) -O(3) -O(4) -O(6) -O(6) -Sr(2)-O> ₈ <sr(1)-o>₁₀</sr(1)-o>	2.542(4) 2.560(2) 2.560(2) 2.607(2) 2.607(2) 2.694(3) 2.694(3) 2.901(4) 3.0517(17) 3.0517(17) 2.646 2.727	Sr(2)-O(8) -O(1) -O(2) -O(2) -O(3) -O(3) -O(3) -O(4) -O(6) -O(6) -Sr(2)-O> ₈ <sr(2)-o>₁₀</sr(2)-o>	2.580(5) 2.607(3) 2.625(3) 2.625(3) 2.625(3) 2.738(4) 2.738(4) 2.861(5) 3.0343(19) 3.0343(19) 3.0343(18) 2.673 2.745	Ce(2)-O(8) -O(2) -O(2) -O(1) -O(1) -O(3) -O(3) -O(4) -O(6) -O(6) <ce(2)-o>₈ <ce(2)-o>₁₀</ce(2)-o></ce(2)-o>	2.40 2.55 2.55 2.6 2.70 2.70 2.70 2.76 2.96 2.96 2.96 2.61 2.68	La(2)-O(8) -O(2') -O(1) -O(3') -O(1') -O(2) -O(6) -O(4) -O(3) <la(2)-o>₈ <la(2)-o>₉</la(2)-o></la(2)-o>	2.470 2.544 2.603 2.610 2.612 2.624 2.67 2.707 2.707 2.711 2.605 2.616
Ti(1) -O(6) -O(6) -O(3) -O(3) -O(3) -O(3) <ti(1)-o>₆</ti(1)-o>	1.915(3) 1.915(3) 2.014(3) 2.014(3) 2.014(3) 2.014(3) 1.981	Zr -O(6) -O(6) -O(3) -O(3) -O(3) -O(3) -O(3) <zr-o>₆</zr-o>	2.044(5) 2.044(5) 2.107(3) 2.107(3) 2.107(3) 2.107(3) 2.07(3) 2.086	Fe ²⁺ -O(6) -O(6) -O(3) -O(3) -O(3) -O(3) < Fe ²⁺ -O> ₆	1.91 1.91 2.15 2.15 2.15 2.15 2.07	Mg -O(6) -O(6) -O(3) -O(3) -O(3') -O(3') <mg-o>₆</mg-o>	2.014 2.014 2.179 2.179 2.179 2.179 2.179 2.124
Ti(2) -O(4) -O(4) -O(5) -O(5) -O(1) -O(1) <ti(2)-o>₆</ti(2)-o>	1.889(2) 1.889(2) 1.955(2) 1.955(2) 1.984(2) 1.984(2) 1.984(2) 1.943	Ti(1) -O(4) -O(4) -O(1) -O(1) -O(5) -O(5) <ti(1)-o>₆</ti(1)-o>	1.893(3) 1.893(3) 1.965(3) 1.965(3) 1.976(3) 1.976(3) 1.976(3) 1.945	Ti -O(4) -O(4) -O(5) -O(5) -O(1) -O(1) <ti-o>₆</ti-o>	1.99 1.99 1.99 2.08 2.08 2.02	C(1) -O(4) -O(4) -O(5) -O(1') -O(5) -O(1) <c(1)-o>₆</c(1)-o>	1.924 1.942 1.978 2.002 2.018 2.023 1.981
Ti(3) -O(5) -O(2) -O(2) -O(2) -O(2) -O(2) -O(4) <ti(3)-o>₆</ti(3)-o>	1.853(3) 1.919(2) 1.919(2) 1.938(2) 1.938(2) 2.455(3) 2.004	Ti(2) -O(5) -O(2) -O(2) -O(2) -O(2) -O(2) -O(4) <ti(2)-o>₆</ti(2)-o>	1.848(5) 1.931(3) 1.931(3) 1.947(3) 1.947(3) 2.513(5) 2.020	Me ³⁺ -O(5) -O(2) -O(2) -O(2) -O(2) -O(2) -O(4) < Me ³⁺ -O> ₆	1.72 1.95 1.95 1.95 1.95 2.21 1.96	C(2) -O(5) -O(2') -O(2) -O(2) -O(2') -O(2') -O(4) <c(2)-o>₆</c(2)-o>	1.789 1.959 1.963 1.979 1.982 2.421 2.016
Si(1) -O(6) -O(7) -O(1) -O(1) <si(1)-o>4</si(1)-o>	1.613(4) 1.619(4) 1.624(3) 1.624(3) 1.620	Si(1) -O(6) -O(7) -O(1) -O(1) <si(1)-o>₄</si(1)-o>	1.604(5) 1.609(5) 1.63(3) 1.63(3) 1.618	Si(1) -O(7) -O(1) -O(1) -O(6) <si(1)-o>₄</si(1)-o>	1.53 1.62 1.62 1.67 1.61	Si(1) -O(6) -O(7) -O(1) -O(1') <si(1)-o>₄</si(1)-o>	1.605 1.636 1.645 1.646 1.633
Si(2) -O(8) -O(7) -O(3) -O(3) <si(2)-o>4</si(2)-o>	1.562(4) 1.650(4) 1.664(3) 1.664(3) 1.635	Si(2) -O(8) -O(7) -O(3) -O(3) <si(2)-o>4</si(2)-o>	1.568(5) 1.641(5) 1.654(4) 1.654(4) 1.629	Si(2) -O(8) -O(7) -O(3) -O(3) <si(2)-o>₄</si(2)-o>	1.61 1.67 1.68 1.68 1.66	Si(2) -O(8) -O(3') -O(7) -O(3) <si(2)-o>₄</si(2)-o>	1.601 1.635 1.639 1.643 1.630
Si(1)-O(7)-Si(2) 163.7(3)		Si(1)-O(7)-Si	(2) 170.0(4)			Si(1)-O(7)-Si	(2) 165.6

ture was also observed in rengeite. The displacement parameter for the bridging O(7) in matsubaraite is as large as that of O(8), which is one of the vertices of Si(2)-tetrahedron, and is not shared with any Ti-octahedra.

The difference in the ionic radii of the cations can be observed as the difference in the lattice volumes of minerals in perrierite and chevkinite group. The reported unit cell parameters for members of the perrierite-chevkinite group are given in Table 7. The approximate unit cell volumes for chevkinite, perrierite, strontiochevkinite (recalculated as "Sr-bearing perrierite") and rengeite are 829, 842, 863 and 866 Å³, respectively. The unit cell volume for matsubaraite (V = 844 Å³) is following rengeite and the "Sr-bearing perrierite".

Table 7. Unit cell parameters for matsubaraite, rengeite, perrierite, chevkinite and strontiochevkinite.

Locality	<i>a</i> (Å)	b (Å)	<i>c</i> (Å)	β (°)	V (Å ³)	Reference	Remarks
Matsubaraite Itoigawa-Ohmi, Jap	an 13.848	5.626	11.878	114.19	844	This study	unheated
Rengeite	an 12.07	E 69	11.09	114.00	866	Mineime et et (2001)	. un bran et e el
itoigawa-Onmi, Jap	an 13.97	5.68	11.98	114.20	866	Miyajima <i>et al.</i> (2001)	unneated
Perrierite							
Nettuno, Italy	13.59	5.61	11.61	113.28	813	Bonatti (1959)	unheated
Oslo region, Norway	y 13.70	5.66	11.83	113.79	839	Segalstad & Larsen (1978)	heated
Nebraska, USA	13.59	5.62	11.73	113.95	819	Izett & Wilcox (1968)	unheated
Virginia, USA	13.61	5.68	11.73	113.5	832	Mitchell (1966)	unheated
North Baikal, Russi	a 13.95	5.62	11:70	114.53	834	Portnov (1964)	unheated
Urals, Russia	13.52	5.65	11.71	113.3	822	Ito (1967)	recrystalized
La₄Mq₂Ti₃Si₄O ₂₂	13.78	5.67	11.78	113.7	843	lto (1967)	synthetic
average of perrierite	13.68	5.64	11.73	113.72	829		
Chevkinite							
Cape Ashizuri, Japa	in 13.46	5.68	11.35	100.86	852	Imaoka & Nakashima (1994)	unheated
Oslo region, Norway	/ 13.43	5.74	11.07	100.58	839	Segalstad & Larsen (1978)	heated
Oslo region, Norway	/ 13.44	5.736	11.04	100.61	837	Segalstad & Larsen (1978)	heated
Oslo region, Norway	/ 13.40	5.72	11.09	100.53	836	Segalstad & Larsen (1978)	heated
Kansas, USA	13.37	5.66	11.28	100.87	838	Izett & Wilcox (1968)	unheated
Utah, USA	13.37	5.67	11.24	100.72	837	Izett & Wilcox (1968)	unheated
New Mexico, USA	13.32	5.68	11.31	100.72	841	Izett & Wilcox (1968)	unheated
Virginia, USA	13.60	5.82	11.22	100.75	872	Mitchell (1966)	unheated
New Hampshire, US	SA 13.44	5.72	11.10	100.20	840	Jaffe et al. (1956)	heated
Madagascar	13.26	5.75	11.06	100.7	829	Ito (1967)	recrystalized
La ₄ Fe ₂ Ti ₃ Si ₄ O ₂₂	13.50	5.75	11.10	101.0	846	Ito (1967)	synthetic
average of chevkinit	e 13.42	5.72	11.17	100.69	842		·
Strontiochevkinite							
Sarambi, Paraguay	13.56	5.70	11.10	100.32	844	Haggerty & Mariano (1983)	unheated
	13.96	5.68	11.94	114.3	863	Miyawaki et al. (2002)	re-calculated



Fig. 4. The crystal structure of matsubaraite. The walls of $Ti(2)O_6$ and $Ti(3)O_6$ octahedra (medium gray) are connected with the chains of $Ti(1)O_6$ octahedra (dark gray) and Si_2O_7 disilicate groups (light gray). The Sr ions (open circles) are located in the cavities of the framework formed with the walls and chains.

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