

Lithiomarsturite, a new member of the pyroxenoid group, from North Carolina*

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ABSTRACT

Lithiomarsturite, ideally $\text{LiCa}_2\text{Mn}_2\text{HSi}_5\text{O}_{15}$, is a new member of the p-p (pectolite-pyroxene) series of pyroxenoids (hydropyroxenoids). It has a structure based on *fünferketten*, but chain-periodicity faults are common, causing a refinement of the structure to terminate with $R = 18\%$. It has space group $P\bar{1}$, $a = 7.652(3)$, $b = 12.119(3)$, $c = 6.805(2)$ Å, $\alpha = 85.41(2)$, $\beta = 94.42(3)$, $\gamma = 111.51(2)^\circ$, $Z = 2$. The strongest lines in the powder-diffraction pattern are (d , I/I_0) 3.19, 90; 3.08, 50; 3.01, 100; 2.913, 90; 2.744, 60. It is transparent, light pinkish brown to light yellow in color with a vitreous luster and good {100} and {001} cleavages. $D_{\text{meas}} = 3.32$ and $D_{\text{calc}} = 3.27$ g/cm³. It is biaxial, negative; $2V = 59.9(8)^\circ$ (meas.); $\alpha = 1.645(1)$, $\beta = 1.660(1)$, $\gamma = 1.666(1)$; dispersion is moderate, $r > v$; $X \approx \mathbf{b}$, $Y \approx [100]$, $Z \approx \mathbf{c}$. Lithiomarsturite occurs at the Foote mine in North Carolina as 1- to 3-mm-diameter euhedral crystals (forms {100}, {010}, and {001}) in vugs in pegmatite with albite, fluorapatite, bavenite, brannockite, parsettensite, and tetrawickmanite crystals.

INTRODUCTION

The mineral described here from the Foote mine in North Carolina was called to our attention by Mr. Emory McDanal of Clover, South Carolina, in 1987. Our preliminary study suggested it was unique and closely related to the Mn pyroxenoids, a finding later supported by more detailed investigation. We have named this new mineral *lithiomarsturite* in order to emphasize the relation to marsturite (Peacor et al., 1978), with which it is isostructural. The mineral and the name were approved by the Commission on New Minerals and Mineral Names, IMA. The holotype and cotype specimens are deposited in the Smithsonian Institution under catalogue numbers NMNH 165268 and NMNH 165460, respectively.

OCCURRENCE

The mineral assemblage of the Foote Mineral Company's spodumene mine (commonly called the Foote mine), Kings Mountain, North Carolina, is quite remarkable, both in its complexity and in its unique character. The conditions that led to the generation of four distinct stages of mineral formation, each having a characteristic suite of associated species, were reviewed by White (1981).

The number of species thus far recognized at the Foote mine exceeds 100 and is still growing. Of these there are at least 40 phosphates.

Lithiomarsturite is found in small vugs in fresh pegmatite consisting principally of up to 2-cm microcline crystals in a groundmass of granular quartz. The bulk chemistry of the assemblage of minerals in the vugs is typical of what one normally finds in the "tertiary stage" paragenesis at the Foote mine. The elements that are most prevalent, in addition to Si, are K, Na, Ca, Be, Li, Mn, Fe, and Sn. The crystals of all of the observed species in association with lithiomarsturite are a few millimeters in diameter. Each vug examined is lined with euhedral white albite crystals, upon which have grown transparent prismatic fluorapatite crystals, usually pale violet in color, and pyrite in simple cubes. Sprays of bavenite occur commonly, either in clusters of delicate fibers or as radiating blades that are virtually colorless. In one specimen lithiomarsturite is accompanied by numerous yellow tetrawickmanite pseudooctahedra; in another it is found with several clusters of brannockite, and most of the pocket is heavily dusted with tiny hemispheres of parsettensite. Lithiomarsturite is found in these specimens in two distinctly different habits, but both have the same sherrylike color. The more typical habit is rhombic with sharp edges that tend to be markedly lighter in color than the rest of the crystal. Another habit shows slightly divergent crystal

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Fig. 1. Photograph of lithiomarsturite crystals on albite (provided by D. Behnke).

bundles with irregular terminations that looked as if they were etched and that are lighter in color than the main body of the crystals.

PROPERTIES

Lithiomarsturite occurs as 1- to 3-mm euhedral crystals, as shown in Figure 1, that vary in habit from nearly equant to pseudoprismatic. Composite aggregates of crystals have markedly elongate pseudoprismatic habits, and parallel growth is common. For the orientation adopted here, the forms present are {100}, {010}, and {001}; {100} is dominant.

The crystals in most observed aggregates are uniform in appearance, but some show a semiopaque whitish druse of dull-lustered microcrystals of lithiomarsturite coating the more common transparent material. The color of lithiomarsturite varies from light pinkish brown through intermediate hues to light yellow; the streak is very light brown; the luster is vitreous on fresh cleavages. The hardness (Mohs) is approximately 6, and the mineral is brittle. Cleavages are good on {100} and {001} and easily developed. The density, measured using heavy-liquid techniques, is 3.32 g/cm³, compared with the calculated value of 3.27 g/cm³. There is no discernible fluorescence in ultraviolet radiation.

Optically, lithiomarsturite is biaxial, negative, $2V = 59.9(8)^\circ$ (meas.), 64° (calc.), with the following indices of refraction, determined in Na light: $\alpha = 1.645(1)$, $\beta = 1.660(1)$, and $\gamma = 1.666(1)$. Dispersion is moderate, $r > v$; the orientation is $X \wedge \mathbf{b} = -7^\circ$ in γ acute, $\phi = 331^\circ$, $\rho = 95^\circ$, $Y \wedge \mathbf{a} = +28^\circ$ in γ obtuse, $\phi = 62^\circ$, $\rho = 90^\circ$, $Z \wedge \mathbf{c} = 6^\circ$ in α acute, $\phi = 0^\circ$, $\rho = 6^\circ$, or in general terms, $X \approx \mathbf{b}$, $Y \approx [100]$, $Z \approx \mathbf{c}$.

CHEMICAL COMPOSITION

Lithiomarsturite was analyzed using electron and ion microprobes. No elements other than those indicated here were detected using wavelength-dispersive microprobe

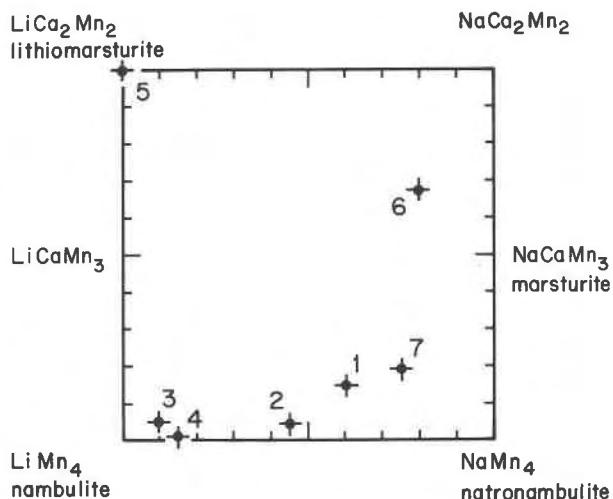


Fig. 2. Plot of compositions of hydroxyroxenoids having *fünferketten* in terms of large cations. Fe and Mg are included with Mn. (1) Kombat, Namibia (von Knorring et al., 1978) (2) Funakozawa mine, Japan (Yoshii et al., 1972) (3) Gozaisho mine, Japan (Ohashi, personal communication) (4) India (Ito, 1972) (5) Foote mine, North Carolina (this paper) (6) Franklin, New Jersey (Peacor et al., 1978) (7) Tanohata mine, Japan (Matsubara et al., 1985).

scans. Electron-microprobe operating conditions included 15-kV operating voltage and 0.025- μ A sample current measured on brass; the standards used were rhodonite (Si, Mn) and hornblende (Fe, Mg, Ca). The Li content was measured by the Cameca IMS-3F ion microscope using nambulite as a standard. The primary beam consisted of negative oxygen ions with 100-nA current and net impact energy of 14.0 keV on the specimen. The beam was focused and rastered on a $250 \times 250 \mu\text{m}$ area. Positive secondary ions of ^7Li in both specimen and standard were collected from a circular area of 20- μm diameter at the center of the square raster. Water was not determined directly because of the paucity of material. However, the calculated water content is implied by the fact that lithiomarsturite is isostructural with minerals such as nambulite, as described below. The resultant analytical values are SiO₂ 51.6, FeO 6.9, MgO 0.7, CaO 19.1, MnO 16.4, Li₂O 2.6, H₂O (calculated) 1.55, total = 98.9 wt%. Because pyroxenoids based on *fünferketten* units have 15 oxygen atoms per formula, the analytical values were normalized to that value, giving rise to the empirical formula $\text{Li}_{1.01}\text{Ca}_{1.98}\text{Mn}_{1.35}\text{Fe}_{0.56}\text{Mg}_{0.10}\text{H}_{1.00}\text{Si}_{5.00}\text{O}_{15}$. The idealized formula is $\text{LiCa}(\text{Mn,Fe})_2(\text{Ca,Mn,Fe})\text{HSi}_5\text{O}_{15}$, based in part on the X-ray diffraction results described below; i.e., there is 1 Ca ordered on M(4), Mn (plus Fe) ordered on M(1) and M(3), and M(2) is occupied at least in part by Ca. The composition of lithiomarsturite is plotted in Figure 2 with those of other members of the hydroxyroxenoids having *fünferketten* in order to illustrate the compositional range of the group.

TABLE 1. X-ray powder-diffraction data for lithiomarsturite

d_{obs}	d_{calc}	hkl	$ l_0$	d_{obs}	d_{calc}	hkl	$ l_0$
7.05	7.11	100	10	2.659	2.653	13 $\bar{1}$	10
6.79	6.77	001	20	2.635	2.630	24 $\bar{1}$	20
5.25	5.22	110	2		2.630	2 $\bar{1}$ 2	
4.47	5.21	11 $\bar{1}$	2				
	4.45	021		2.539	2.540	122	2
4.16	4.16	11 $\bar{1}$	2		2.538	320	
3.79	3.81	2 $\bar{1}$ 0	2	2.510	2.518	202	5
	3.79	120			2.514	122	
					2.510	3 $\bar{1}$ 0	
3.55(D)	3.56	13 $\bar{1}$	10	2.475	2.471	132	10
	3.55	200					
3.39	3.43	2 $\bar{1}$ $\bar{1}$	5	2.451	2.457	23 $\bar{2}$	2
	3.39	002			2.447	032	
	3.36	031			2.444	22 $\bar{1}$	
					2.443	330	
3.31	3.32	121	5		2.442	32 $\bar{1}$	
	3.30	131					
	3.30	12 $\bar{1}$		2.262	2.262	340	20
	3.29	012			2.259	25 $\bar{1}$	
3.19	3.22	2 $\bar{1}$ 1	90		2.258	003	
	3.22	20 $\bar{1}$					
	3.21	031		2.217	2.220	151	50
	3.19	012			2.214	113	
	3.19	112			2.211	230	
					2.208	34 $\bar{1}$	
3.08	3.11	22 $\bar{1}$	50	2.102			5
	3.08	210		2.089			10
	3.08	201		2.012(D)			2
				1.943(D)			2
3.01	3.03	23 $\bar{1}$	100	1.894(D)			5
	3.02	140					
	3.01	122		1.833(D)			5
	3.00	102		1.805			2
				1.776			5
2.913	2.911	130	90	1.752			2
2.811	2.826	112	20	1.710			20
	2.815	231					
	2.814	040		1.692			2
2.744	2.743	240	60	1.681			5
2.710	2.697	131	2	1.609			10
	2.695	132		1.596			10
				1.573			2
				1.556			2
				1.542			2
				1.515			5
				1.492			5
				1.446			10
				1.428			20
				1.394			5

Note: Data obtained using a 114.6-mm-diameter Gandolfi camera with NIST Si standard and FeK α (Mn-filtered) radiation. Intensities were estimated visually. D = diffuse.

X-RAY CRYSTALLOGRAPHY

Single crystals were studied using the precession and Weissenberg methods, giving rise to lattice parameters similar to those of pyroxenoids having *fünferketten* (see below for discussion), and determination of the space group as $P\bar{1}$ or $P1$. The space group is inferred to be $P\bar{1}$ by comparison with isostructural minerals. Powder X-ray diffraction data were obtained using a 114.6-mm diameter Gandolfi camera, polycrystalline sample, FeK α (Mn-filtered) radiation and Si as an internal standard. Data are

listed in Table 1. Because an attempt to refine the lattice parameters using powder-diffraction data by least-squares was unsuccessful primarily because of ambiguities in indexing, refinement was carried out using a 4-circle automated diffractometer. The unit-cell parameters are $a = 7.652(3)$, $b = 12.119(3)$, $c = 6.805(2)$ Å, $\alpha = 85.41(2)$, $\beta = 94.42(3)$, and $\gamma = 111.51(2)^\circ$.

The lattices of silicates having *fünferketten* structure units all have approximately the same dimensions, but they have been referred to a variety of different choices of unit cell. Peacor and Niizeki (1963) pointed out that

TABLE 2. Lattice parameters of selected pyroxenoids with *fünferketten* units

Mineral	Reference	Cell type*	a (Å)	b (Å)	c (Å)	α	β	γ
Nambulite	Narita et al., 1975	R	7.621	11.761	6.731	92°46'	95°05'	106°52'
Natronambulite	Matsubara et al., 1985	R	7.620	11.762	6.737	92.81°	94.55°	106.87°
Marsturite	Peacor et al., 1978	S	7.70	12.03	6.78	85.26°	94.10°	111.04°
Babingtonite	Araki and Zoltai, 1972	R	7.509	11.697	6.719	91.433°	93.886°	104.255°
"Lithium-hydrorhodonite"	Murakami et al., 1977	R	7.530	11.736	6.710	92°58'	95°14'	106°16'
Rhodonite	Peacor and Niizeki, 1963	R	7.682	11.818	6.707	92.36°	93.95°	105.67°
Rhodonite	Peacor and Niizeki, 1963	S	6.707	7.682	12.234	111.54°	85.25°	93.95°
Lithiomarsturite		S	7.652	12.119	6.805	85.41°	94.42°	111.51°

* R = reduced cell; S = "structure cell," with one translation parallel to chains of tetrahedra.

the cell can conveniently be chosen with one translation parallel to the chains of tetrahedra, giving rise to a translation magnitude greater than 12 Å. This cell is referred to as the "structure cell." On the other hand, a translation can be chosen such that the "reduced cell" is defined, giving rise to a translation slightly smaller than 12 Å. As shown in Table 2, the unit cells of most silicates having *fünferketten* have been chosen as the reduced cell. However, because directional properties of silicates with *fünferketten* units are related to the orientation of the chains, the choice of the "structure cell" leads to simplicity in indexing (e.g., cleavages are {100} and {010}); indeed, forms, cleavages, and other units have been misindexed because of confusion resulting from the choice of the reduced cell. We have therefore referred lithiomarsturite to the "structure cell." A third setting of the unit cells, giving rise to C-centering, has been utilized by Ohashi and Finger (1975). This also utilizes a translation parallel to chains, but such a cell is little used.

The similarity in lattice parameters of lithiomarsturite and silicates known to have *fünferketten* units of structure implied that lithiomarsturite also has *fünferketten* units. This was confirmed by the chemical data, as shown below. In addition, direct comparison of single-crystal diffraction photographs of lithiomarsturite with those of marsturite, rhodonite, and babingtonite showed a general similarity in diffraction intensities of all phases, further confirming the analogy. However, Takéuchi and Koto (1977) showed that *fünferketten* structures fall into either of two categories: (1) those, like rhodonite, that contain no hydrogen and have a three-tetrahedron unit of the chain, each tetrahedron of which has a vertex in common with the vertices of a triangle that is the face of an octahedron, or (2) those, like marsturite, that contain essential hydrogen and have a three-tetrahedron unit of the chain, each tetrahedron of which has a vertex in common with a triangle with vertices common to two octahedra. The former belong to the w-p (wollastonite-pyroxene) series and the latter to the p-p (pectolite-pyroxene) series. The p-p series is marked by the presence of monovalent cations such as Li or Na. The *fünferketten* members of the p-p series typically have compositions approximating $M_2^{2+}M^{1+}HSi_5O_{15}$, where M^{1+} is a cation that occupies sites between bands of octahedra.

The amount of material available for this study was too small to permit an analysis for H. However, the presence of eight $(Ca + Mn)^{2+}$ and two $(Li + Na)^{1+}$ per unit cell gives rise to a charge imbalance of -2 and by analogy with the formulae of members of the p-p series, implies that lithiomarsturite contains essential H. These relations collectively show that it is isostructural with the minerals marsturite (Peacor et al., 1978), nambulite (Yoshii et al., 1972), natronambulite (Matsubara et al., 1985), and babingtonite (Araki and Zoltai, 1972). The crystal structures of babingtonite (Araki and Zoltai, 1972), nambulite (Narita et al., 1975) and a phase identified as "lithium-hydrorhodonite" (Murakami et al., 1977) have been refined. These structures have bands of octahedra that are constructed of parallel columns of eight octahedra joined through edge-sharing in an offset manner. The octahedra at opposite ends of the column [M(4)] are significantly larger than the others and are occupied preferentially by Ca. As two of the four M^{2+} cations per formula in lithiomarsturite are Ca, whose radius is significantly greater than that of Mn, the analogy implies that one of the two Ca atoms is ordered in M(4).

In addition, babingtonite has two Ca per formula $(Ca,Fe^{3+}Fe^{2+}HSi_5O_{15})$, as does lithiomarsturite. In babingtonite the second Ca is also ordered in a single site [M(2)] that is substantially larger than those for Fe. The equivalent sites in nambulite and "lithium-hydrorhodonite" are also slightly larger relative to the remaining two. Although M(1), M(2), and M(3) all are occupied by Mn in the latter two structures, the slightly larger M-O distances for M(2) further imply that it is preferred by larger cations, as in babingtonite. As the numbers of Ca and Mn per formula of lithiomarsturite are close to 2, these relations collectively imply that all cations are ordered in lithiomarsturite, with Ca in M(4) and M(2). The Li is assumed to occupy sites between bands of octahedra as in the analogous structures.

Because the cation scheme described above required confirmation, we attempted to carry out a refinement of the structure. However, the results are largely unsatisfactory owing to the presence of chain periodicity faults (see below) and are therefore only briefly described here. The intensities of 2338 reflections were measured using an automated Weissenberg (Supper) system, MoK α radi-

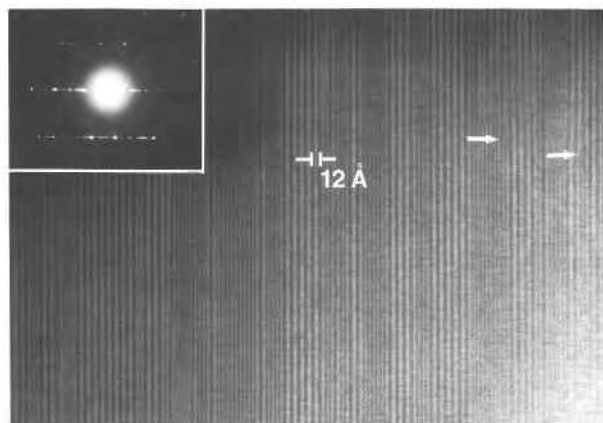


Fig. 3. Lattice-fringe image (001 fringes) of lithiomarsturite showing chain-periodicity faults. The selected-area electron-diffraction pattern shows diffuseness parallel to c^* . The larger fringes (12 Å in width) correspond to *fünferketten* units. The fringes having a smaller spacing correspond to *dreierketten* units. The arrows point to faults where *fünferketten* and *dreierketten* units are interchanged parallel to (001).

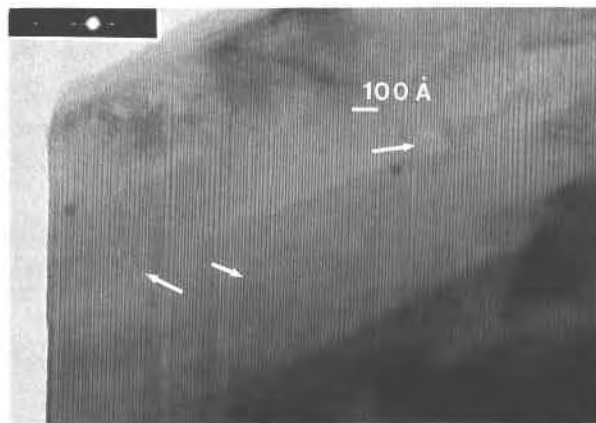


Fig. 4. Lattice-fringe image of lithiomarsturite (001 fringes) dominated by fringes 12 Å in width (spanned by scale bar) and fringes with 7-Å spacing. The latter fringes are not resolved, but units composed of them occur as broad featurless lamellae. Numerous faults occur, with three designated by arrows.

tion, and graphite monochromator. Data were corrected for Lorentz-polarization and absorption ($\mu_1 = 37 \text{ cm}^{-1}$, crystal 0.2 mm in diameter). Refinement was carried out using the program SHLEX (Sheldrick, 1976), utilizing isotropic temperature factors, starting coordinates as given for the nambulite structure (Narita et al., 1975), and anomalous scattering factors. Refinement was initiated assuming the complete ordering scheme described above, with refinement of occupancy factors in the latter stages. Refinement only of coordinates proceeded satisfactorily to an R factor of 18%. However, further refinement with isotropic temperature factors and occupancy factors of M(1) through M(5) varying gave rise to no further decrease in the R value, and to many negative temperature factors. Because such factors may be caused by the presence of positional disorder, the crystals serving as a source of intensity data were reexamined.

Examination of single-crystal diffraction patterns revealed that some reflections displayed broadening and diffuseness parallel to c^* . Chain-periodicity faults that occur in pyroxenoids (e.g., Ried and Korekawa, 1980; Aikawa, 1984) may be manifested by such diffuseness. Crushed fragments distributed on a holey carbon-coated Cu grid were therefore examined by transmission electron microscopy (TEM) in order to directly detect the presence of such faults. The instrument used is a Philips CM-12 STEM, operated at 120 kV. Selected-area diffraction patterns including 001 reflections verified the presence of diffuseness parallel to c^* . This diffuseness was extreme as compared to the subtle effect displayed by only a few reflections in X-ray diffraction photographs. Figures 3 and 4 show lattice-fringe images obtained using only 001 reflections. They show that chain-periodicity faults are common, with units having *dreierketten* being especially abundant, although *fünferketten* units are dominant. Al-

though not shown, *siebenerketten* units are observed rarely. The (010) and (100) crystal faces commonly appear to be curved, with the curvature in the form of a number of discrete steps, as displayed through $[hk0]$ striations. Such steps may well be the surficial manifestation of the chain periodicity faults, with steps corresponding to changes in chain periodicity.

The presence of abundant chain periodicity faults provides an explanation of the anomalous temperature factors and high R -factor of the structure refinement. Nevertheless, the occupancy factors appear to have some significance. The occupancies are $\text{Mn}_{1.00}\text{Ca}_{0.00}$, $\text{Mn}_{0.77}\text{Ca}_{0.23}$, $\text{Mn}_{1.00}\text{Ca}_{0.00}$, and $\text{Mn}_{-0.01}\text{Ca}_{1.01}$ for M(1) through M(4), respectively, with standard errors of site occupancies equal to 0.03. These values are consistent with predicted ordering of Mn on M(1) and M(3) and Ca on M(4). However, the predicted preference of Ca for M(2) is matched by only 23% Ca relative to Mn; the occupancies collectively imply a composition with significantly more Mn relative to Ca, as compared with the nearly equal amounts shown by direct analysis. Nevertheless, the combination of interatomic distances, occupancies, and analogies with site occupancies in other pyroxenoids are consistent with the ordering of Mn in M(1) and M(3) and Ca in M(4). Some preference of Ca for M(2) is indicated, with the results being not inconsistent with M(2) being largely occupied

TABLE 3. Occupancies of octahedral sites for *fünferketten* hydopyroxenoids

	M(1)	M(2)	M(3)	M(4)	M(5)
Nambulite	Mn	Mn	Mn	Mn	Li,Na
Natronambulite	Mn	Mn	Mn	Mn	Na
Marsturite	Mn	Mn	Mn	Ca	Na
Lithiomarsturite	Mn	Mn,Ca	Mn	Ca	Li

by Ca. Table 3 shows that lithiomarsturite therefore effectively has the same cation distribution as marsturite, but with Li rather than Na in M(5).

ACKNOWLEDGMENTS

We are grateful to D. Behnke for photographs of the crystals and to Y. Takéuchi, Y. Ohashi, and F. F. Foit, Jr., for helpful reviews. Y. Ohashi kindly supplied unpublished data on the composition of nambulite.

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MANUSCRIPT RECEIVED FEBRUARY 10, 1989

MANUSCRIPT ACCEPTED NOVEMBER 29, 1989