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Crystal structure and compressibility of a two-layer polytype of pseudowollastonite ($CaSiO_3$)

HEXIONG YANG* AND CHARLES T. PREWITT

Geophysical Laboratory and Center for High Pressure Research, Carnegie Institution of Washington, 5251 Broad Branch Road, N.W.,
Washington, D.C. 20015-1305, U.S.A.

ABSTRACT

The crystal structure of a synthetic two-layer polytype of $CaSiO_3$ pseudowollastonite was determined using single-crystal X-ray diffraction data. It is monoclinic with space group $C2/c$ and unit-cell parameters a = 11.8322(6), b = 6.8624(8), c = 10.5297(5) Å, β = 111.245(8)°, and V = 796.9(1) $Å^3$. The material is isostructural with two-layer $SrSiO_3$ and $SrGeO_3$ and has basic structural features similar to those found in four-layer $CaSiO_3$ pseudowollastonite, except for the stacking sequence of layers of CaO_6 octahedra and Si_3O_9 ternary rings. The compressibility of the structure was measured up to 9.94 GPa and no phase transition was observed. With increasing pressure, all unit-cell parameters decrease nonlinearly with a positive curvature. The axial compression ratios at room pressure are $\beta_a:\beta_b:\beta_c$ = 2.25:2.36:1.00 and the bulk modulus, K_T, is 86(1) GPa with $K' = \partial K_T/\partial P$ = 3.8(4).

INTRODUCTION

Pseudowollastonite, the stable form of $CaSiO_3$ at temperatures above ~1125 °C, is an important phase in experimental petrology and commonly occurs in slags, cement, and ceramic materials. Its crystal structure is characterized by alternate stacking of layers of ternary (Si_3O_9) tetrahedral rings and distorted-bicapped Ca octahedra along [001]. A distorted-bicapped octahedron is formed by eight oxygens, with six of them in an octahedral arrangement plus two from bridging oxygens of the Si_3O_9 ternary rings—one above and one below the octahedron. At high pressure and temperature (~3.0 GPa and 1100 °C), $CaSiO_3$ transforms to a walstromite-II phase (Trojer 1969) and further transforms to perovskite above 10 GPa (Ringwood and Major 1967; Liu and Ringwood 1975). Owing to different stacking sequence of octahedral layers and ternary rings, various polytypes could exist in pseudowollastonite (Yamanaka and Mori 1981; Ingrin 1993). However, because of the difficulties in preparation of good quality single crystals (Jeffery and Heller 1953; Yamanaka and Mori 1981; Matteson 1983; Ingrin 1993; Richet et al. 1998), few investigations have been performed to characterize polytype structures in pseudowollastonite.

From X-ray precession photographs, Yamanaka and Mori (1981) observed three polytypes of pseudowollastonite: a dominant four-layer phase, plus a six-layer and a disordered structure. The layers referred to here are composed of one layer of ternary rings plus one layer of CaO_6 octahedra (Yamanaka and Mori 1981). The six-layer polytype was thought to be isostructural with $SrGeO_3$ described by Dornberger-Schiff (1962) and Hilmer (1963). For the four-layer polytype, Yamanaka and Mori (1981) obtained a triclinic unit cell and solved its structure in space group $C\bar{1}$. However, a re-determination of the four-layer pseudowollastonite structure by Yang and Prewitt (1999) suggests that its real symmetry is monoclinic $C2/c$, rather than triclinic $C\bar{1}$. Ingrin (1993) investigated polytypism in $CaSiO_3$ using transmission electron microscopy and observed the coexistence of two-, four-, and six-layer polytypes in the form of lamellae parallel to (001). Trojer (1969) determined the structure of a three-layer $CaSiO_3$ phase synthesized at 6.5 GPa and 1300 °C. This paper reports the structure of a two-layer pseudowollastonite polytype and presents the compressibility data on this phase up to 9.94 GPa.

EXPERIMENTAL PROCEDURES

A $Ca_{0.75}Mg_{1.25}Si_2O_6$ gel was first prepared from stoichiometric amounts of puratronic $CaCO_3$, MgO, and tetraethyl orthosilicate for the synthesis of the Di-En solid solution. The dried gel was crystallized at 1400 °C for five days and then mixed with K_2WO_4 as flux in a weight ratio of 1:1. The mixture was heated at 1300 °C for 24 days and then slowly cooled to 1200 °C, at which the charge was held for 7 days and quickly quenched into liquid mercury. Microprobe analysis and X-ray diffractometry revealed the existence of three different, transparent phases: diopside ($CaMgSi_2O_6$), an unknown K-Mg silicate, and a $CaSiO_3$ phase. The flux apparently reacted with the starting material, resulting in the loss of Mg to the K-Mg silicate and formation of $CaMgSi_2O_6$ diopside and the $CaSiO_3$ phase, instead of the intended Di-En solid solution. Three $CaSiO_3$ crystals were analyzed and all were homogeneous giving a nearly stoichiometric composition (average of 11 points): $Ca_{0.995}Si_{1.005}O_3$. Based on optical examination, an elongated single crystal (0.09 mm × 0.06 mm × 0.05 mm) was selected for precession photography. It was found to be monoclinic with possible space group Cc or $C2/c$; no diffuse streaks or other polytypes were observed. The crystal was then placed on a Picker four-circle diffractometer equipped with a Mo X-ray

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*E-mail: yang@gl.ciw.edu

tube (β-filtered) for X-ray diffraction measurements. All examined reflections showed sharp peak profiles. Unit-cell parameters (Table 1) were determined by fitting the positions of 16 reflections with 20° < 2θ < 35° following the procedure of King and Finger (1979).

X-ray diffraction intensity data from a quadrant of reciprocal space with 0° ≤ 2θ ≤ 60° were collected on the basis of the *C*-centered lattice using ω scans of 1° width in step increments of 0.025° and 2s per step counting time. In order to check if any reflections violated the *C*-centered symmetry, a data set with 0° ≤ 2θ ≤ 30° was first collected in a 1s per step counting time based on the primitive lattice; no such reflections were detected. Digitized step data were integrated by the method of Lehmann and Larsen (1974) with background manually reset when necessary. Corrections were made for Lorentz and polarization effects, and for X-ray absorption by the crystal (μ = 25.0 cm^{1}). The total number of measured reflections was 1056, out of which 813 reflections had intensities greater than $2\sigma_I$ and were included in the structure determination and refinement. Measured reflections confirmed the possible space group as *Cc* or *C*2/*c*.

Because the E-value statistics of the X-ray intensity data showed that the structure is probably centric, we began solution of the structure with space group *C*2/*c*. The structure was solved using program SHELX97 and refined with RFINE90. Neutral atomic scattering factors, including anomalous dispersion corrections for Ca, Si, and O, were taken from Ibers and Hamilton (1974). Weighting schemes were based on $w = [\sigma^2(F) + (pF)^2]^{-1}$, where *p* is adjusted to ensure that the errors were normally distributed through probability plot analysis (Ibers and Hamilton 1974). Type II isotropic extinction corrections (Becker and Coppens 1975) were applied in the refinements. The anisotropic refinement for all atoms converged after six cycles (R_w = 0.028 and *R* = 0.025) and resulted in featureless difference Fourier maps. A model based on the *Cc* symmetry was also attempted, but it did not improve the refinement statistics significantly. Thus, the *C*2/*c* symmetry was adopted for all refinements. Final atomic coordinates and displacement parameters are presented in Table 2 and selected interatomic distances in Table 3.

TABLE 1. Unit-cell parameters of two-layer $CaSiO_3$ pseudowollastonite at various pressures

P (GPa)	*a* (Å)	*b* (Å)	*c* (Å)	β (°)	*V* (Å^3)
0.00*	11.8322(6)	6.8624(8)	10.5297(5)	111.245(8)	796.9(1)
0.10	11.8241(6)	6.8598(8)	10.5273(6)	111.238(4)	795.9(1)
2.26	11.7128(5)	6.7830(9)	10.4839(4)	111.155(3)	777.9(1)
4.03	11.6264(4)	6.7402(8)	10.4510(4)	111.086(2)	764.1(1)
6.20	11.5152(4)	6.6743(7)	10.4103(3)	110.991(2)	747.0(1)
8.22	11.4260(5)	6.6216(9)	10.3790(4)	110.917(3)	733.5(1)
9.94	11.3674(5)	6.5853(9)	10.3604(5)	110.879(3)	724.6(1)

* Data at room pressure were measured without the diamond-anvil cell.

After the X-ray diffraction data collection at room pressure, the same crystal used for the structure determination was polished to ~0.035 mm in thickness and mounted in a modified Merrill-Basset diamond-anvil cell for the compressibility measurement of the structure. The detailed procedures of the high-pressure experiment were similar to those described by Yang and Hazen (1999). No phase transition was detected up to 9.94 GPa. Measured unit-cell parameters as a function of pressure were listed in Table 1.

RESULTS AND DISCUSSION

Crystal structure of two-layer pseudowollastonite

The $CaSiO_3$ phase contains two unit layers parallel to (001) in a unit cell, which should be compared to the four-layer pseudowollastonite structure studied by Yamanaka and Mori (1981) and Yang and Prewitt (1999). In the two-layer structure, the second layer of ternary rings is displaced one-sixth of the *a* dimension with respect to the first layer of ternary rings (Fig. 1a). In the four-layer structure, the first and second layers of ternary rings are stacked directly on top of one another, whereas the third layer of ternary rings is displaced one-sixth of the *b* dimension with respect to the second layer. The fourth layer lies directly on top of the third layer (Fig. 1b). Note that the *a* and *b* axes in the two-layer structure are reversed with respect to those in the four-layer structure. Nevertheless, the two structures closely compare in many other aspects. For example, the average Ca-O and Si-O distances are 2.532(2) and 1.624(2) Å, respectively, in the two-layer structure and 2.529(1) and 1.625(1) Å in the four-layer structure; the average Si-O-Si angles within a ternary ring are identical [134.7(1)°] in the two structures. Moreover, the CaO_6 octahedra in both structures are compressed in the *c* direction, whereas the SiO_4 tetrahedra are elongated in the same direction. These similarities give an identical density of 2.90 g/cm^3 for the two structures. Compared to the average Ca-O bond length in the two- or four-layer pseudowollastonite, that (2.491 Å) in the three-layer high-pressure $CaSiO_3$ phase (Trojer 1969) is significantly short, accounting for the greater density (3.07 g/mm^3) of the high-pressure form. The short average Ca-O distance and greater density of the high-pressure phase is principally achieved through the distortion of Si_3O_9 ternary rings: The average Si-O-Si angle within a ternary ring is only 123.4° in the high-pressure structure, 11° smaller than that in the two- or four-layer strucuture. Similar

TABLE 2. Atomic coordinates and anisotropic displacement factors of two-layer $CaSiO_3$ pseudowollastonite

	x	*y*	*z*	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Ca1	0.0870(1)	0.2403(1)	0.4989(1)	0.0011(1)	0.0035(1)	0.0007(1)	0.0003(1)	0.0001(1)	0.0001(1)
Ca2	0.25	0.25	0	0.0010(1)	0.0058(1)	0.0009(1)	0.0008(1)	0.0003(1)	–0.0004(1)
Si1	0.1278(1)	0.4530(1)	0.2445(1)	0.0007(1)	0.0032(1)	0.0005(1)	0.0002(1)	0.0002(1)	0.0001(1)
Si2	0	0.8400(1)	0.25	0.0010(1)	0.0023(1)	0.0006(1)	0	0.0001(1)	0
O1	0.1275(1)	0.4045(2)	0.0965(2)	0.0018(1)	0.0063(3)	0.0009(2)	0.0006(1)	0.0006(1)	–0.0001(2)
O2	0.2290(1)	0.3991(2)	0.3863(2)	0.0014(1)	0.0080(3)	0.0014(2)	0.0013(2)	0.0001(1)	0.0003(2)
O3	0.1109(1)	0.6929(2)	0.2478(2)	0.0014(1)	0.0033(3)	0.0021(2)	0.0001(1)	0.0011(1)	–0.0001(2)
O4	0.0497(1)	0.9436(2)	0.3939(2)	0.0023(1)	0.0050(3)	0.0010(2)	–0.0002(2)	0.0004(1)	–0.0010(2)
O5	0	0.3592(3)	0.25	0.0013(1)	0.0041(4)	0.0020(3)	0	0.0009(2)	0

TABLE 3. Selected interatomic distances (Å) in two-layer $CaSiO_3$ pseudowollastonite

Ca1-O1	2.622(1)	Si1-O1	1.592(2)
Ca1-O1'	2.621(2)	Si1-O2	1.583(2)
Ca1-O2	2.619(2)	Si1-O3	1.660(1)
Ca1-O2'	2.282(1)	Si1-O5	1.664(1)
Ca1-O3	2.573(2)	Avg	1.625
Ca1-O4	2.282(2)		
Ca1-O4	2.609(2)	Si2-O3 (×2)	1.662(1)
Ca1-O5	2.577(1)	Si2-O4 (×2)	1.582(2)
Avg	2.523	Avg	1.622
Ca2-O1 (×2)	2.308(2)		
Ca2-O2 (×2)	2.661(2)		
Ca2-O3 (×2)	2.598(2)		
Ca2-O4 (×2)	2.591(1)		
Avg	2.540		

results have been observed in $BaSi_4O_9$ (Hazen et al. 1999), in which the average Si-O-Si angle within a ternary ring in the denser $P3$ polymorph is 5.5° smaller than that (133.6°) in the less dense benitoite-type $P\bar{6}c2$ polymorph.

Interestingly the two-layer $CaSiO_3$ phase reported here is isostructural with $SrSiO_3$ studied by Machida et al. (1982a) and Nishi (1997a) and with $SrGeO_3$ by Matteson (1983) and Nishi (1997b). However, Machida et al. (1982a) described their $SrSiO_3$ structure in space group $C2$. Marsh and Herbstein (1983) reanalyzed the structure factors of Machida et al. (1982a) based on space group $C2/c$ and obtained in a substantial improvement in all categories of refinement statistics, indicating that the real space group of the $SrSiO_3$ structure studied by Machida et al. (1982) should be $C2/c$, rather than $C2$. This conclusion was confirmed by Nishi (1997a).

By assuming that all ternary rings and octahedra in pseudowollastonite were ideal with $3/m$ and $\bar{3}$ symmetry, respectively, Yamanaka and Mori (1981) derived 12 possible polytypes, including one two-layer, two four-layer, and nine six-layer structures. This model should also be applicable to all structural analogs of $CaSiO_3$ pseudowollastonite, such as $SrSiO_3$ and $SrGeO_3$ phases, as it only deals with ideal polyhedra. The symmetry of the two-layer polytype predicted by Yamanaka and Mori (1982) is $Ccmm$, differing from the $C2/c$ symmetry observed in this study and in $SrSiO_3$ and $SrGeO_3$ (Machida et al. 1982; Matteson 1983; Nishi 1997a, 1997b). The failure of the model of Yamanaka and Mori (1981) in prediction of the space group of pseudowollastonite polytypes is due to the fact that no ideal tetrahedra or octahedra have been observed in pseudowollastonite-type structures.

Compressibility of two-layer pseudowollastonite

With increasing pressure, all unit-cell parameters decrease nonlinearly with a positive curvature:

$$\begin{aligned}
a\ (\text{Å}) &= 11.8320 - 0.0560P + 0.000885P^2 & \gamma &= 0.9997,\\
b\ (\text{Å}) &= 6.8621 - 0.0341P + 0.000615P^2 & \gamma &= 0.9996,\\
c\ (\text{Å}) &= 10.5300 - 0.0221P + 0.000488P^2 & \gamma &= 0.9997,\\
b\ (^\circ) &= 111.25 - 0.0444P + 0.000671P^2 & \gamma &= 0.9989,\\
V\ (\text{Å}^3) &= 797.05 - 9.0335P + 0.16956P^2 & \gamma &= 0.9997,
\end{aligned}$$

where P is in GPa and γ is the correlation coefficient. The linear compressibilities of the a, b, and c dimensions at room pressure are 0.00473(6), 0.00496(8), and 0.00210(5) GPa^1, respectively, with the axial compression ratios of $\beta_a:\beta_b:\beta_c$ = 2.25:2.36:1.00. The greater compressibility of the a and b dimensions is a direct consequence of the structure topology and

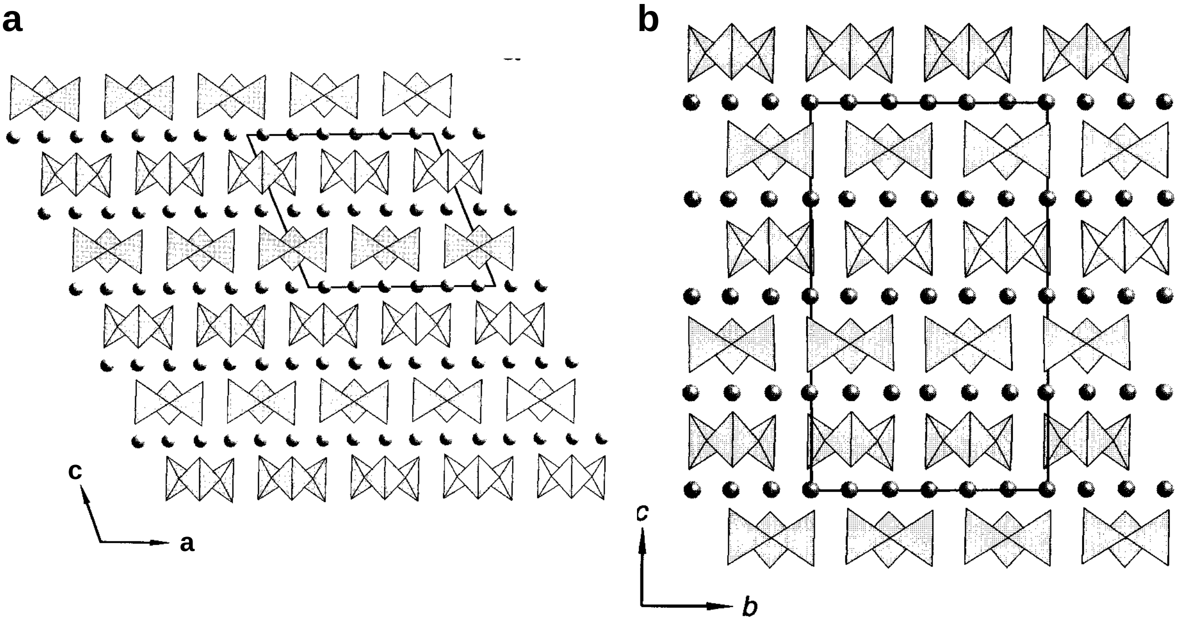

FIGURE 1. Comparison of stacking sequence of layers of Si_3O_9 ternary rings in (**a**) two- and (**b**) four-layer $CaSiO_3$ pseudowollastonite. Note that the a and b axes in the two-layer structure are reversed with respect to those in the four-layer structure. The solid spheres represent Ca atoms.

distortion of CaO_6 octahedra. In the two-layer structure, not only all CaO_6 octahedra forming layers parallel to the *a*-*b* plane are compressed along [001], but there also exists considerable space among Si_3O_9 ternary rings that are sandwiched by CaO_6 octahedral layers. Hence, the structure is much less packed along the *a* and *b* axes than along the *c* axis, giving rise to the apparent anisotropy of the structure as it is compressed.

Weighted volume and pressure data fit to a second-order Birch-Murnaghan equation of state yields V_0 = 796.9(1) Å^3, K_0 = 86(1) GPa, and K' = 3.8(4). Because of the great similarities between the two- and four-layer structures, we would expect a similar bulk modulus for four-layer pseudowollastonite. If we assume that the bulk modulus-volume relationship that holds for isostructural phases (see Hazen and Finger 1982) is also true for polytypes, then we could have an estimated bulk modulus of ~91 GPa for the three-layer high-pressure form. Using the energy minimization technique, Taniguchi et al. (1995) simulated the structures and physical properties of three- and four-layer $CaSiO_3$. Without any particular assumptions about the nature of the bonding or interatomic forces, they obtained a bulk modulus of 76 GPa for the four-layer structure and 74 GPa for the three-layer high-pressure phase. Considering the fact that the molar volumes calculated by Taniguchi et al. (1995) for the three- and four-layer structures are 5.7 and 3.8% larger than the experimentally determined values, respectively, we think that Taniguchi et al. (1995) were likely to have underestimated their bulk moduli for these two phases.

From in-situ X-ray powder diffraction, Tamai and Yagi (1989) reported a new, unquenchable phase ($CaSiO_3$-III) intermediate between $CaSiO_3$-II and perovskite-type $CaSiO_3$ in the pressure range from 10 to 13.8 GPa. However, based on their examination on quenched high-pressure phases in the $CaSiO_3$ system by X-ray diffraction and ^{29}Si MAS NMR, Kanzaki et al. (1991) suspected that the new phase reported by Tamai and Yagi (1989) might be a mixture of two phases. In contrast, Machida et al. (1982a, 1982b) synthesized three high-pressure polymorphs of $SrSiO_3$ with space groups $C2/c$, $P\bar{1}$, and $P2_1/c$ at 3.5, 4.0, and 5.5 GPa, respectively, and solved their structures. Intriguingly, the $C2/c$ $SrSiO_3$ phase is isostructural to the two-layer $CaSiO_3$ phase determined in this study, whereas the $P\bar{1}$ $SrSiO_3$ phase is isostructural to the three-layer high-pressure $CaSiO_3$ phase studied by Trojer (1969). By analogy, we could, therefore, postulate the existence of another $CaSiO_3$ phase that would be stable at pressure higher than that of the three-layer $CaSiO_3$-II phase described here. Further high-pressure investigations would be required to clarify the phase relations and polymorphism, as well as polytypism, in the $CaSiO_3$ system.

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