Hydrostatic Compression of γ-Mg$_2$SiO$_4$ to Mantle Pressures and 700 K: Thermal Equation of State and Related Thermoelastic Properties

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Abstract. P-V-T equations of state for the γ phase of Mg$_2$SiO$_4$ have been fitted to unit cell volumes measured under simultaneous high pressure (up 30 GPa) and high temperature (up to 700 K) conditions. The measurements were conducted in an externally heated diamond anvil cell using synchrotron x-ray diffraction. Neon was used as a pressure medium to provide a more hydrostatic pressure environment. The P-V-T data include 300 K-isothermal compression to 30 GPa, 700 K-compression to 25 GPa and some additional data in P-T space in the region 15 to 30 GPa and 300 to 700 K. The isothermal bulk modulus and its pressure derivative, determined from the isothermal compression data, are 182(3) GPa and 4.2(0.3) at $T = 300$ K, and 171(4) GPa and 4.4(0.5) at $T = 700$ K. Fitting all the P-V-T data to a high-temperature Murnaghan equation of state yields: $K_{T0} = 182(3.0)$ GPa, $K'_{T0} = 4.0(0.3)$, $(\partial K_T/\partial T)_0 = -2.7(0.5) \times 10^{-2}$ GPa/K and $(\partial^2 K_T/\partial P\partial T)_0 = 5.5(5.2) \times 10^{-4}$/K at the ambient condition.

Introduction

(Mg, Fe)$_2$SiO$_4$, a major mantle mineral, transforms to the spinel structure (the γ phase) in the Earth's transition zone. In conjunction with seismic observations, the physical properties of γ-(Mg, Fe)$_2$SiO$_4$ under transition zone P-T conditions yield information about the mineralogical composition of the mantle. Due to its geophysical importance, the γ phase has been widely studied: thermochemical properties (Watanabe 1982), elastic properties at ambient conditions (Weidner et al. 1984), lattice parameters and thermal expansion at high temperatures (Suzuki et al. 1979; Ming et al. 1992), acoustic velocities of polycrystal to 3 GPa (Rigden et al. 1991) and single crystal compression study at ambient temperature to

4.79 GPa (Hazen 1993). However, studies under simultaneous high P-T conditions are limited.

In a previous study (Meng et al. 1993a), we measured the unit cell volumes of the γ phase in the temperature range from 759 to 962 K at $P = 9.8$ GPa using synchrotron x-ray in a cubic anvil apparatus (SAM 85). These isobaric P-V-T data provided a direct determination of the thermal expansion at high pressure and high temperature. They also provide a constraint on the effect of temperature on the isothermal bulk modulus which is related to the thermal expansion at high pressure by thermodynamic relations.

In the present paper, we report the unit cell volume measurements on γ-Mg$_2$SiO$_4$ under simultaneous high P-T conditions in an externally heated diamond anvil cell. The pressure range of this experiment (up to 30 GPa at 300 K and up to 25 GPa at high temperatures) covers that of the transition zone, although the temperature is limited to 700 K. The data of this study, which are mostly isothermal, provide a more direct determination of the temperature effect on the isothermal bulk modulus.

Experimental

The specimen of γ-Mg$_2$SiO$_4$ was synthesized at 19.5 GPa and 900° C, in a uniaxial split-sphere apparatus (USSA-2000) located in the High Pressure Laboratory at Stony Brook. The experimental procedures for synthesizing high-pressure polymorphs have been described in detail by Gwanmesia et al. (1990) and by Gwanmesia and Liebermann (1992).

The experiment of this study was carried out in an externally heated Mao-Bell type diamond anvil cell using energy-dispersive synchrotron x-ray radiation at beam line X-17C (a superconductor wiggler beam line) of the National Synchrotron Light Source, Brookhaven National Laboratory. The technical details of the experiment have been described previously (Mao et al. 1991; Fei et al. 1992). The finely ground powder of γ-Mg$_2$SiO$_4$ sample, a small piece of thin gold foil and a few ruby grains were loaded in a 250 micron diameter sample chamber with neon as a pressure medium. The sample chamber was made from a nickel alloy gasket (Réne 41) pre-indented to 15 GPa. Sample heating was achieved

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by a Ni80Cr20-wire resistance heater mounted on the outside of the cylinder of the diamond anvil cell. The large size of the heater assured a uniform heating of the specimen and the diamonds. The temperatures were recorded by a chromel-alumel thermocouple placed on the diamond near the sample chamber. In this experiment, the incident x-ray beam was collimated to 25×35 micron. The diffracted x-rays were collected using a solid-state germanium detector at a fixed 2θ angle $14.996°$ ($\pm 0.005°$), which was calibrated by diffraction lines of gold. The energy calibration of the multichannel analyzer was made by x-ray emission lines (K_α and K_β) of Mn, Cu, Rb, Mo, Ag, Ba and Tb.

Results

The unit cell volumes of the γ-Mg$_2$SiO$_4$ were determined from diffraction lines 311, 400 and 440, with an average precision of 0.05%. The pressure calibrations at 300 K were achieved using the ruby fluorescence line, R_1, (Mao et al. 1986) and the neon x-ray diffraction line, 220, (Hemley et al. 1989; Finger et al. 1981). The pressures at high temperature were calibrated by the gold x-ray diffraction lines (Anderson et al. 1989) with a typical uncertainty of ± 0.2 GPa. At each P-T condition, at least two spectra were collected: one was from the sample only, and the other was from both the sample and the gold. There were some overlap between the γ phase diffraction line, 440, and the gold diffraction line, 220, also the γ phase 400 and the gold 200; therefore we used a peak-fitting program to locate the peak positions accurately.

We have found that the effect of deviatoric stress (i.e. departures from hydrostaticity) on the unit cell volume measurement and further on the volume-based pressure calibration is directly proportional to K/μ of the measured material (Meng et al. 1993 b). As gold has an unusually high value of K/μ (6.2) compared with most samples (1 to 2), the measurement of unit cell volume of gold is very sensitive to the existence of even a small amount of deviatoric stress. The pressure environment of this experiment has been characterized based on gold diffraction peaks. At room temperature, the deviatoric stress in gold increases with increasing pressure above 15 GPa. By 30 GPa, the calculated deviatoric stress is about 0.6 GPa. The deviatoric stress decreases dramatically with increasing temperature and disappears by a temperature of about 650 K. Even though such a small amount of deviatoric stress is insignificant for the γ-Mg$_2$SiO$_4$ sample, it causes a systematic error in the pressure calibration based on the unit cell volume of gold if the deviatoric stress effect is not taken into account. The method for determining the deviatoric stress in a diamond anvil cell, the deviatoric stress in gold as a function of pressure and temperature for this experiment, as well as the correction of the deviatoric stress effect on the volume-based pressure calibration have been discussed in a separate paper by Meng et al. (1993 b). We also demonstrated in the paper that, for the isothermal compression of γ-Mg$_2$SiO$_4$ at 300 K, the pressure calibration using gold after correcting the deviatoric stress effect was consistent with the ruby calibration. The unit cell volumes of γ-Mg$_2$SiO$_4$ and the corresponding P-T conditions are listed in Table 1.

Table 1. P-V-T data of γ-Mg$_2$SiO$_4$ and the results of least-squares fits

T(K)	$P_{obs.}$(GPa)	$V_{obs.}$(Å^3)	$P_{cal.}$[a]	ΔP	$V_{cal.}$[b]	ΔV
300	0.0	526.7 (3)	0.0	0.0	526.7	0.0
300	5.7	511.4 (2)	5.7	0.0	511.4	0.0
300	6.6	509.0 (4)	6.7	0.1	509.2	0.2
300	8.6	504.7 (2)	8.5	−0.1	504.4	−0.3
300	11.1	498.9 (1)	11.1	0.0	498.8	−0.1
300	14.8	491.0 (0)	14.8	0.0	491.0	0.0
300	17.9	485.1 (2)	17.8	−0.1	484.9	−0.2
300	20.8	479.1 (5)	21.0	0.2	479.5	0.4
300	23.1	475.4 (8)	23.1	0.0	475.5	0.1
300	25.1	471.8 (12)	25.2	0.1	472.1	0.3
300	29.4	464.9 (5)	29.6	0.2	465.2	0.3
300	30.8	463.7 (4)	30.3	−0.5	463.1	−0.6
355	30.0	464.6 (2)			464.3	−0.3
401	29.2	466.0 (4)			465.6	−0.4
450	28.5	467.8 (3)			466.8	−1.0
501	26.3	470.2 (0)			470.5	0.3
501	27.8	468.5 (2)			468.1	−0.4
550	27.2	468.9 (3)			469.3	0.4
600	26.0	471.0 (0)			471.4	0.4
645	24.2	474.5 (2)			474.7	0.2
646	23.9	474.9 (1)			475.2	0.3
650	21.0	480.2 (1)			480.4	0.2
650	25.6	472.3 (2)			472.2	−0.1
700	4.5	517.2 (0)	4.6	0.1	517.5	0.3
700	8.2	508.7 (0)	7.9	−0.3	507.9	−0.8
700	9.7	504.0 (5)	9.8	0.1	504.2	0.2
700	13.0	496.3 (1)	13.2	0.2	496.7	0.4
700	15.4	491.5 (3)	15.5	0.1	491.5	0.0
700	18.5	486.1 (2)	18.2	−0.3	485.4	−0.7
700	20.2	481.5 (4)	20.6	0.4	482.0	0.5
700	22.2	478.6 (2)	22.2	0.0	478.4	−0.2
700	23.1	477.0 (3)	23.0	−0.1	476.8	−0.2

[a] Birch-Murnaghan EOS fitted to isothermal compression data
[b] P-V-T EOS, equation (7) fitted to all P-V-T data
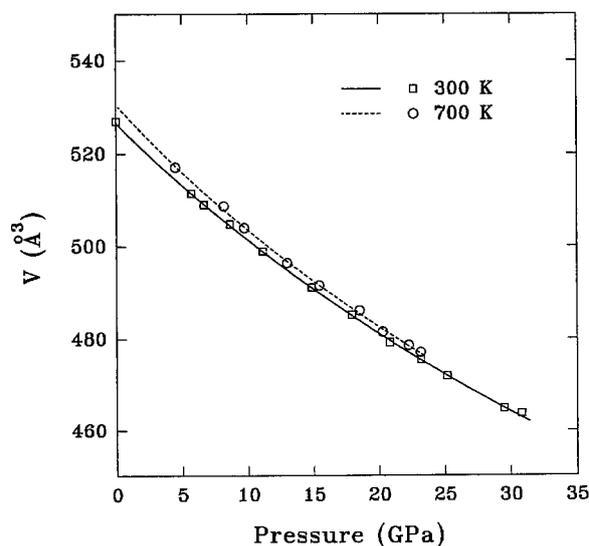
Fig. 1. Isothermal P-V relations of γ-Mg$_2$SiO$_4$ at 300 K (*squares*) and at 700 K (*circles*). The *solid* and *dashed* lines represent the least-squares fits of the compression data to a third order Birch-Murnaghan equation of state
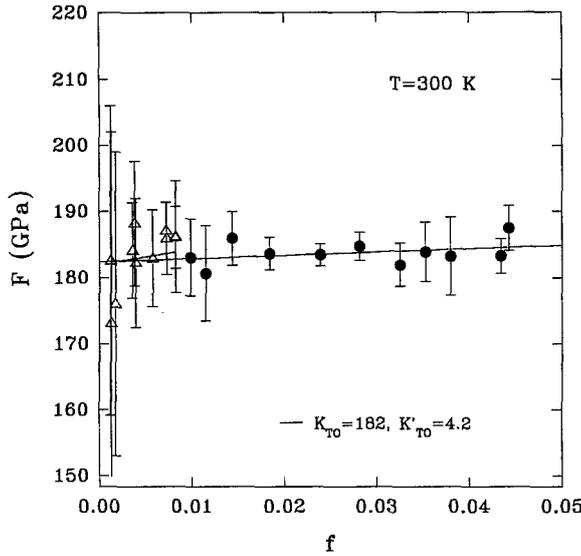
Fig. 2. Isothermal compression data of γ-Mg$_2$SiO$_4$ at 300 K plotted as normalized pressure, F, versus Eulerian finite strain, f. The *solid line* represents the best fit of the data to a linearized third order Birch-Murnaghan equation of state, equation (4). The parameters from the best fit are also listed in the figure. The single crystal data by Hazen (1993) are also plotted for comparison (*open triangles*)

Fig. 3. Isothermal compression data of γ-Mg$_2$SiO$_4$ at 700 K plotted as normalized pressure, F, versus Eulerian finite strain, f. The *solid line* represents the best fit of the data to a linearized third order Birch-Murnaghan equation of state, equation (4). The parameters from the best fit are also listed in the figure

Isothermal Compressions at 300 K and 700 K

The unit cell volumes as a function of pressure at 300 K and 700 K are presented in Fig. 1. We use a third-order Birch-Murnaghan equation of state to derive the isothermal bulk modulus and its pressure derivative from the isothermal compression data:

$$P = \left(\frac{3}{2}\right) K_{T0} \left[\left(\frac{V_0}{V}\right)^{\frac{7}{3}} - \left(\frac{V_0}{V}\right)^{\frac{5}{3}}\right]$$
$$\cdot \left\{1 - \left(\frac{3}{4}\right)(4 - K'_{T0})\left[\left(\frac{V_0}{V}\right)^{\frac{2}{3}} - 1\right]\right\}, \tag{1}$$

where V_0, K_{T0} and K'_{T0} are the unit cell volume, the isothermal bulk modulus and its pressure derivative, respectively, at ambient pressure. With the normalized pressure

$$F = \frac{P}{[3f(1+2f)^{\frac{5}{2}}]} \tag{2}$$

and the Eulerian finite strain

$$f = \frac{1}{2}\left[\left(\frac{V_0}{V}\right)^{\frac{2}{3}} - 1\right], \tag{3}$$

a third order Birch-Murnaghan equation of state, equation (1), is a straight line on a F-f plot:

$$F = K_{T0}[1 - \tfrac{3}{2}f(4 - K'_{T0})] \tag{4}$$

(Birch 1978). The intercept of the straight line defines K_{T0} and the slope represents the deviation of K'_{T0} from 4.0. The isothermal compression data expressed as the normalized pressure (F) versus the Eulerian finite strain

(f) are shown in Figs. 2 and 3. The best fit of the F-f data to equation (4) (K_{T0} and K'_{T0} refined simultaneously using an unweighted least-squares method) yields: 182 (3) GPa for K_{T0} and 4.2 (0.3) for K'_{T0} at 300 K with $V(0,300 \text{ K}) = 526.7 (0.3)$ Å^3, and 171 (4) GPa and 4.4 (0.5) at 700 K with $V(0,700 \text{ K}) = 530.5 (0.5)$ Å^3.

The room temperature value of K_{T0} is in good agreement with the value, 182 (1) GPa, calculated from K_{S0} measured in Brillouin scattering experiment by Weidner et al. (1984). However, K'_{T0} is lower than the value based on the ultrasonic measurement up to 3 GPa by Rigden et al. (1991). The dependence of K_{T0} on the assumed K'_{T0} is demonstrated in Fig. 4. Furthermore, the best fit values of K_{T0} and K'_{T0} are sensitive to $V(0, T)$ (Fig. 4); an increase in $V(0, T)$ will increase K'_{T0}, but decrease K_{T0}. Given these uncertainties, K_{T0} and K'_{T0} at $T = 300$ K of this study are in reasonable agreement with the ultrasonic result ($K'_{S0} = 5.0$, Rigden et al. 1991) and that of the single-crystal compression study ($K_{T0} = 184$ GPa with K'_{T0} fixed at 4.8, Hazen 1993).

The isothermal compression data are also analyzed using the Murnaghan equation of state,

$$\frac{V}{V_0} = \left(1 + \frac{K'_{T0}}{K_{T0}}P\right)^{\frac{-1}{K'_{T0}}}. \tag{5}$$

The two equations of state have different assumptions concerning the second order pressure derivative of the bulk modulus K''_{T0}. K''_{T0} is zero in the Murnaghan equation of state, while it has a non-zero, negative value in the Birch-Murnaghan equation of state. The assumptions on K''_{T0} have more effect on the refinement of K'_{T0}; however in the pressure range of this study, such an effect is insignificant. A non-linear least-squares fit of
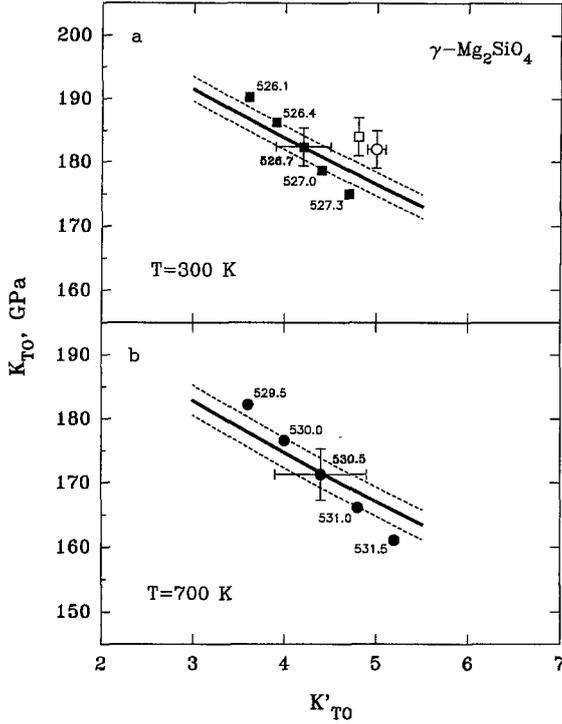
Fig. 4a, b. Dependence of K_{T0} on fixed K'_{T0} and dependence of K_{T0} and K'_{T0} on $V(0, T)$ for the 300 K isotherm (a) and the 700 K isotherm (b) of γ-Mg$_2$SiO$_4$. The *solid lines* represent the dependencies of K_{T0} on fixed K'_{T0} at fixed $V(0,300\,\mathrm{K})=526.7\,\text{Å}^3$ and $V(0,700\,\mathrm{K})=530.5\,\text{Å}^3$. The *filled symbols* are the best fitted K_{T0} and K'_{T0} at fixed $V(0, T)$ whose values are indicated by the numbers. The single crystal data (*open square*) by Hazen (1993) and the ultrasonic data (*open circle*) by Rigden et al. (1991) are also plotted for comparison

Fig. 5. The P-V-T relations of γ-Mg$_2$SiO$_4$. The isotherms are calculated using equation (7) with $K_{T0}=182\,\mathrm{GPa}$, $K'_{T0}=4.0$, $(\partial K_T/\partial T)_0 = -2.7 \times 10^{-2}\,\mathrm{GPa/K}$, $(\partial^2 K_T/\partial P\,\partial T)_0 = 5.5 \times 10^{-4}/\mathrm{K}$ and $\alpha_{av}=17.7 \times 10^{-6}/\mathrm{K}$. Symbols 3, 7 and cross represent the isothermal compression data at 300 K, at 700 K and the data at the random P-T conditions

the Murnaghan equation of state yields: 182 (3) GPa for K_{T0} and 4.0 (0.3) for K'_{T0} at 300 K, and 171 (4) GPa and 4.3 (0.5) at 700 K. Within the uncertainties of the measurements, they are consistent with the results using the Birch-Murnaghan equation of state.

P-V-T Equation of State

We used a high-temperature Murnaghan equation of state to derive the thermoelastic parameters from the experimental P-V-T data. Based on the thermodynamic identity

$$K_T(P, T) = -V\left(\frac{\partial P}{\partial V}\right)_T, \quad (6)$$

and with $K_T(P, T)$ expressed as:

$$K_T(P, T) = K_{T0} + K'_{T0}\,P + \left(\frac{\partial K_T}{\partial T}\right)_0 (T-300) + \left(\frac{\partial^2 K_T}{\partial P\,\partial T}\right)_0 P(T-300),$$

a P-V-T relation is defined by integrating equation (6),

$$V(P, T) = V(0, T)\left(1 + \frac{b}{a}\,P\right)^{-\frac{1}{b}}, \quad (7)$$

where

$$a = K_{T0} + \left(\frac{\partial K_T}{\partial T}\right)_0 (T-300),$$

$$b = K'_{T0} + \left(\frac{\partial^2 K_T}{\partial P\,\partial T}\right)_0 (T-300).$$

To obtain internally consistent parameters, the isothermal compression data at $T=300$ K are analyzed using equation (7) to determine K_{T0} and K'_{T0}. Then, data at high temperatures are fitted to the equation to constrain the temperature derivative, $(\partial K_T/\partial T)_0$ and $(\partial^2 K_T/\partial P\,\partial T)_0$. A non-linear least-squares fit yields: $K_{T0} = 182$ GPa, $K'_{T0}=4.0$, $(\partial K_T/\partial T)_0 = -2.7 \times 10^{-2}$ GPa/K and $(\partial^2 K_T/\partial P\,\partial T)_0 = 5.5 \times 10^{-4}/\mathrm{K}$. The thermal expansion at ambient pressure used to calculate $V(0, T)$ in equation (7) is expressed as $\alpha(0, T) = a_1 + a_2 T + a_3/T^2$. Due to the limited temperature range of this study, the parameters a_2 and a_3, which determine the variation of $\alpha(0, T)$ with temperature, are obtained by averaging the thermal expansion function of Ming et al. (1992) and that of Suzuki et al. (1979). a_1 is refined with a_2 and a_3 fixed in the least-squares fit to the P-V-T data. The thermal expansion as a function of temperature obtained this way is $\alpha(0, T) = 1.872 \times 10^{-5} + 4.210 \times 10^{-9}\,T - 0.6537/T^2$, which gives an average value of 18 $\times 10^{-6}/\mathrm{K}$ in the temperature range from 300 to 700 K. This value is lower than those from the measurements at ambient pressures by Ming et al. ($20 \times 10^{-6}/\mathrm{K}$) and by Suzuki et al. ($23 \times 10^{-6}/\mathrm{K}$). The unit cell volumes from the best fit are listed in Table 1. In Fig. 5, we plot

Table 2. Elastic and thermoelastic parameters of γ-Mg_2SiO_4

	K_0, GPa	K'_0	$\left(\dfrac{\partial K_T}{\partial T}\right)_0$, GPa/K	$\left(\dfrac{\partial^2 K_T}{\partial P \partial T}\right)_0$, K^{-1}
This study				
300 K [a]	182 (3)	4.2 (0.3)		
700 K [a]	171 (4)	4.4 (0.5)		
300 K [b]	182 (3)	4.0 (0.3)		
700 K [b]	171 (4)	4.3 (0.5)		
300 K [c]	182 (3)	4.0 (0.3)	$-2.7\,(0.3)\times 10^{-2}$	$5.5\,(5.2)\times 10^{-4}$
Previous studies:				
183*	5.0*		$-2.8\,(0.3)\times 10^{-2}$	
			P-V-T data, 9.8 GPa and up to 962 K [1]	
184 (1)			Brillouin scattering, ambient condition [2]	
184*	5.0 (0.1)		Ultrasonic, up to 3 GPa [3]	
184 (2)	4.8*		Single crystal P-V data, up to 5 GPa [4]	

[a] Birch-Murnaghan equation of state applied to isothermal compression data
[b] Murnaghan equation of state applied to isothermal compression data
[c] P-V-T EOS, equation (7), applied to all P-V-T data
* Fixed values
[1] Meng et al. (1993)
[2] Adiabatic bulk modulus K_{S0} (Weidner et al. 1984)
[3] $(\partial K_S/\partial P)_0$ (Rigden et al. 1991), K_{S0} taken from Weidner et al. (1984)
[4] Hazen (1993)

the calculated 300 K and 700 K isotherms, the experimental P-V-T data are also plotted for comparison.

The parameters determined by fitting the P-V-T data to a equation of state are correlated. Since the data are mostly in high pressure and limited temperature region, the thermal expansion $\alpha(0, T)$ is found to correlate with the derivatives of the isothermal bulk modulus. A higher $\alpha(0, T)$ requires that both $(\partial K_T/\partial T)_0$ and $(\partial^2 K_T/\partial P \partial T)_0$ have larger magnitude. If $\alpha(0, T)$ is fixed, a variation of $(\partial^2 K_T/\partial P \partial T)_0$ from 0 to 10×10^{-4}/K results in a change of $(\partial K_T/\partial T)_0$ from -2.2×10^{-2} to -3.1×10^{-2} GPa/K based on the current data.

Summary and Discussion

P-V-T Equation of State

The isothermal bulk modulus, K_{T0}, is 182 (3) GPa at 300 K and 171 (4) GPa at 700 K from the isothermal compression data. The high-temperature Murnaghan equation of state, provides a reasonably good fit to all the P-V-T data of this experiment and yields 182 (3) GPa and 4.0 (0.3) for K_{T0} and K'_{T0} at ambient temperature, $-2.7\,(0.3) \times 10^{-2}$ GPa/K for $(\partial K_T/\partial T)_P$ and 5.5 (5.2) $\times 10^{-4}$/K for $(\partial^2 K_T/\partial P \partial T)_0$. The elastic and thermoelastic parameters of the γ phase derived from this study are summarized in Table 2. The room temperature value of K_{T0} is in good agreement with the previous studies (Weidner et al. 1984; Hazen 1993). $(\partial K_T/\partial T)_P$ from this study agrees with the result of our previous study, $-2.8\,(0.3) \times 10^{-2}$ GPa/K, (Meng et al. 1993a) within the uncertainty of the measurements.

Thermal Pressure as a Function of Volume at 700 K

The temperature derivative of isothermal bulk modulus at constant volume is obtained from the thermodynamic identity:

$$\left(\frac{\partial K_T}{\partial T}\right)_V = \left(\frac{\partial K_T}{\partial T}\right)_P + \left(\frac{\partial K_T}{\partial P}\right)_T \alpha K_T. \tag{8}$$

Using the ambient values of $(\partial K_T/\partial T)_P = -0.027$ GPa/K, $(\partial K_T/\partial P)_T = 4.2$, $K_{T0} = 182.4$ GPa and $\alpha = 17.7 \times 10^{-6}$/K, $(\partial K_T/\partial T)_V$ is -0.013 GPa/K from equation (8). A non-zero value of $(\partial K_T/\partial T)_V$ suggests a volume dependence of thermal pressure. The thermal pressures as a function of volume at a constant temperature of 700 K, calculated from

$$P_{\text{th}}(V, 700 \text{ K}) = P(V, 700 \text{ K}) - P(V, 300 \text{ K}), \tag{9}$$

are demonstrated in Fig. 6, where $P(V, 700 \text{ K})$ and $P(V, 300 \text{ K})$ are the fitted isotherms using the Birch-Murnaghan equation of state. Equation (9) is well expressed by

$$P_{\text{th}} = a + b \ln\left(\frac{V_0}{V}\right), \tag{10}$$

where

$$a = \int_{300}^{T} \alpha K_T \, dT = 1.25$$

and

$$b = \left(\frac{\partial K_T}{\partial T}\right)_V (T - 300) = 5.36 \quad \text{at} \quad T = 700 \text{ K}.$$
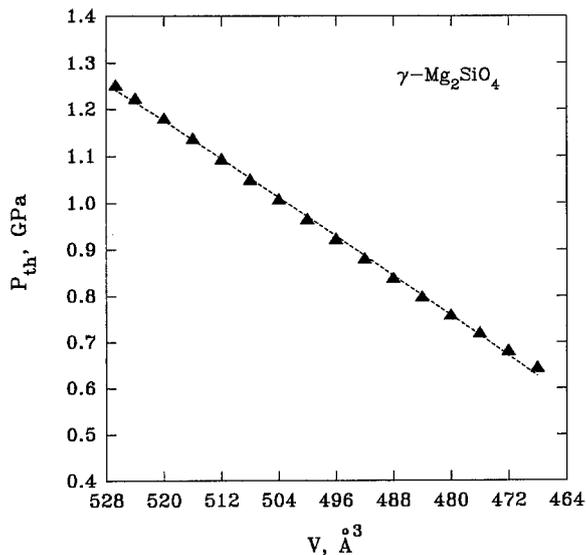
Fig. 6. Thermal pressure as a function of volume at $T= 700$ K. The *triangles* are calculated from equation (9) and the *dash line* is calculated from equation (10)
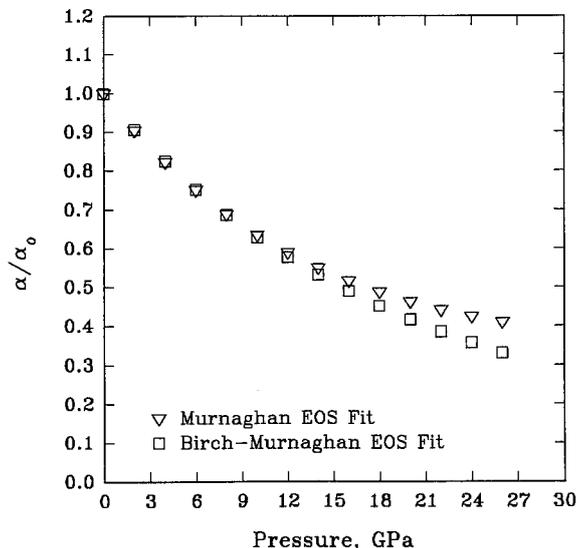
Fig. 7. The normalized average thermal expansion (300 K to 700 K) of γ-Mg_2SiO_4 as a function of pressure. The *open triangles* are calculated from the fits of the Murnaghan equation of state, and the *open squares* are from the Birch-Murnaghan equation of state

Thermal Expansion as a Function of Pressure

The average thermal expansion, α, of the γ phase as a function of pressure is calculated from the isothermal compressions at 300 K and 700 K. The normalized values, (α/α_0), are plotted in Fig. 7. α decreases by a factor of 2.6 from the ambient-pressure value in the pressure range up to 25 GPa.

The Mixed P, T Derivative of the Isothermal Bulk Modulus

The data of this study do not provide a tight constraint on the mixed P, T derivative of the isothermal bulk modulus. However, they are consistent with a positive value of $(\partial^2 K_T/\partial P \partial T)_0$ for γ-Mg_2SiO_4, and the magnitude of $(\partial^2 K_T/\partial P \partial T)_0$ is probably very similar to those for MgO and olivine (Isaak 1993).

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