

On the Absence of Shear Mode Softening in Single-Crystal Fayalite Fe_2SiO_4 at High Pressure and Room Temperature

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Abstract. The geophysical importance of the kinetics of the olivine \rightarrow spinel phase transformation has stimulated considerable interest in the transformation mechanism. Both nucleation-and-growth and diffusionless martensitic models have been proposed. It has recently been suggested that a martensitic transformation (effected by partial dislocations associated with the (100) [001] slip system) would probably be accompanied by premonitory pressure-induced softening of the shear moduli c_{55} and c_{66} . We have explored this possibility by measurement of the modulus c_{55} for a single crystal of fayalite over a pressure range of 3 GPa (at 295 K) by ultrasonic interferometry. The variation of c_{55} with pressure is described by a quadratic with the parameters $(c_{55})_0 = 46.90 \pm 0.04$ GPa, $(\partial c_{55}/\partial P)_0 = 1.715 \pm 0.004$ and $(\partial^2 c_{55}/\partial P^2)_0 = -(0.136 \pm 0.003)$ GPa⁻¹ where the subscript '0' refers to atmospheric pressure and 295 K. The first pressure derivative is comparable with those for forsterite and $\sim \text{Fo}_{90}$ olivine in spite of the much greater proximity of fayalite to the olivine \rightleftharpoons spinel phase boundary. The absence of pronounced pressure-induced shear mode softening in this study, along with similar results from a recent ultrasonic study of polycrystalline fayalite under conditions of simultaneous high pressure and high temperature, weakens the case for a martensitic olivine \rightleftharpoons spinel transformation mechanism.

Introduction

Olivine \rightleftharpoons spinel phase equilibria are generally considered to exert a major influence not only on the mineralogy, but also on the physical properties and dynamics, of the transition zone of the Earth's mantle. For olivine of upper mantle composition ($\sim \text{Fo}_{89}$), the olivine (α) \rightarrow spinel (γ) transformation is complicated by the stability at intermediate pressures of the β -phase. Inspection of published phase diagrams (Ringwood and Major 1970; Akimoto et al. 1976) reveals the following sequence of reactions in the pressure-induced phase transformation of Fo_{89} : $(\alpha) \rightarrow (\alpha + \gamma) \rightarrow (\alpha + \beta) \rightarrow (\beta) \rightarrow (\beta + \gamma) \rightarrow (\gamma)$. Most of the associated increase in density and elastic wave velocities is achieved by the reactions $(\alpha + \gamma) \rightarrow (\alpha + \beta) \rightarrow (\beta)$ which occur within a 1 GPa pressure interval centred near 12 GPa at 1,000–1,200°C (Ringwood and Major 1970; Akimoto et al. 1976; Liebermann 1975). Accordingly, the $(\alpha) \rightarrow (\beta)$ transformation is generally associated with the major discontinuity in seismic wave velocities near 400 km depth in the

Earth's mantle, while the $(\beta) \rightarrow (\gamma)$ transformation may explain a more minor discontinuity sometimes observed in the depth range 500–550 km (e.g. Ringwood 1975; Akimoto et al. 1976).

Elevation of these equilibria within relatively cool subducting lithosphere (by virtue of positive Clapeyron slopes dP/dT) may contribute substantially to the driving force for plate tectonics (Schubert and Turcotte 1971; Toksöz et al. 1971; Ringwood 1975). Alternatively, sluggish transformation kinetics in the neighbourhood of the equilibrium boundary could result in significant olivine metastability, with potentially important implications not only for the dynamics of the lithospheric slab but also for its stress state and for the origin of deep-focus earthquakes (e.g. Sung and Burns 1976). Any role for transformation plasticity in the rheology of subducting lithosphere or surrounding mantle is also dependent upon the kinetics of transformation relative to those for dynamic recovery (Paterson 1983).

The very considerable geophysical importance of the kinetics of the $(\alpha) \rightarrow (\alpha + \gamma) \rightarrow (\alpha + \beta) \rightarrow (\beta)$ transformation has stimulated interest in the transformation mechanism. The observation that the $(\alpha) \rightarrow (\beta)$ transformation of Fo_{89} is initiated by the formation of spinel (γ , composition $\sim (\text{Mg}_{0.5}\text{Fe}_{0.5})_2\text{SiO}_4$), and the contraction of the β -phase field with decreasing temperature, have been cited as justification for emphasis of the olivine \rightleftharpoons spinel transformation mechanism (Sung and Burns 1976; Poirier 1981a), for which two radically different alternatives have been proposed. The first is a nucleation and growth mechanism (Sung and Burns 1976) in which the olivine/spinel boundary propagates by a generally uncoordinated, thermally activated movement of atoms which is rate-controlled by the diffusion of oxygen ions.

The second possibility is a martensitic diffusionless mechanism (Poirier 1981a) which achieves the conversion of the hexagonal-close-packed anion array of olivine into the face-centred-cubic geometry of spinel by the action of dislocations. Specifically, it is suggested by Poirier that partial dislocations associated with the (100) [001] slip system sweep across the basal plane of olivine creating layers of stacking faults with the spinel structure, which become more stable as the transition is approached. It is envisaged that the necessary cation rearrangement over atomic distances is accomplished by a coordinated shuffle known as synchroshear.

It is further suggested (Poirier 1981a) that such a transformation would probably be accompanied by premonitory

softening of the elastic stiffness moduli (c_{55} and c_{66}) governing shear on the (100) plane. In this connection, Poirier draws attention to the fact that pressure derivatives of the elastic moduli for single-crystal forsterite and $\sim\text{Fo}_{90}$ olivine are least for c_{55} and c_{66} . The availability of single-crystal fayalite, along with the (room-temperature) 3 GPa pressure capability of our ultrasonic interferometer, affords an opportunity for study of the pressure sensitivity of the key shear moduli of an olivine to pressures beyond its metastable olivine \rightleftharpoons spinel transition pressure (~ 2 GPa for Fe_2SiO_4 , Ringwood 1975).

Experimental Procedure

The original objective of this study was measurement by ultrasonic interferometry of the pressure dependence of all nine single-crystal elastic stiffness moduli of a large crystal of fayalite (Fe_2SiO_4 , space group $Pbnm$) grown by the floating-zone technique (Takei 1978). However, minor cracks which developed along the (010) cleavage plane during preparation of the crystal were found to seriously affect the propagation of certain modes (especially those with propagation vector $\mathbf{k} \parallel [010]$, thereby eliminating the possibility of a comprehensive study (see also Sumino 1979). It was possible, however, to obtain high quality data for the shear mode with $\mathbf{k} \parallel [001]$ and polarization $\mathbf{s} \parallel [100]$, and thus measure the pressure dependence of the key shear modulus, c_{55} . Accordingly, a pair of parallel (001) faces was cut, ground and polished flat within $\lambda/2$. The density of the virgin (uncracked) crystal boule, measured by the Archimedeon technique, was 4.400 ± 0.003 g/cm³ which compares with an X-ray density of 4.397 g/cm³ (Sumino 1979). The orientation of the polished faces, determined by Laue X-ray diffraction, was within 1° of (001) and their spacing was 6.375 ± 0.002 mm.

The techniques of ultrasonic interferometry and pressure generation and calibration employed in this study have been described in detail elsewhere (Jackson et al. 1981; Jackson and Niesler 1982) and need only a brief review here. Round-trip travel times through the crystal are measured by observation of the interference between echo trains resulting from the application of a pair of appropriately spaced phase-coherent RF pulses to a piezoelectric transducer. The effects of transducer-bond phase shifts on the observed travel-times are eliminated by performing comparative one- and two-transducer experiments which allow the phase shift to be calculated and removed from the observed travel-times. Any errors in this procedure are further minimized by high-frequency operation (around the third harmonic of the 20 MHz transducer fundamental frequency) which reduces the contribution of transducer-bond phase shifts to the measured travel times.

The high ultrasonic attenuation of fayalite dictated the use of LiNbO_3 (41° rotated X-cut) transducers as this shear mode transducer has a higher electromechanical coupling factor and a higher impedance than AC-cut quartz transducers, resulting in a correspondingly higher signal/noise ratio. Unfortunately, this gain in signal level is at the expense of mode purity (Warner et al. 1967) and care must be exercised in the positioning of the receiver gate to avoid compressional mode interference (Jackson et al. 1981). For the high-pressure measurements, transducers were bonded to the crystal with 9:1 (molar) mixture of glycerine and phthalic anhydride (Jackson and Niesler 1982). Although

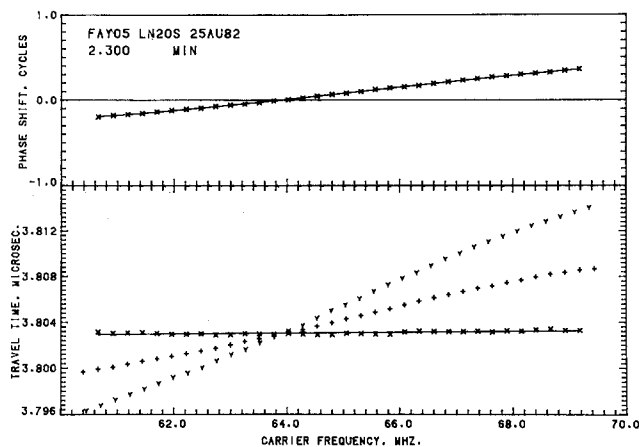


Fig. 1. Phase comparison data at 2.30 GPa for the shear mode c_{55} with $\mathbf{k} \parallel [001]$ and polarization $\mathbf{s} \parallel [100]$. The plotting symbols (+), (Y), and (X) in the lower part of the figure denote, respectively, apparent travel times for one- and two-transducer configurations, and travel times corrected for transducer-bond phase shifts, for all interference minima in the displayed frequency range. The lines superimposed on the phase shift and corrected travel times are, respectively, the best fitting quadratic and linear representations of the data. The corrected travel times are characterized by a standard deviation of 0.10 ns about a mean of 3.80305 μs

this bonding material minimizes the deviatoric stresses between the transducer and the crystal at high pressures, it lacks the rigidity at lower pressures which is required to propagate a shear wave of measurable amplitude through a material as highly attenuating as fayalite. This resulted in a lack of data below ~ 0.9 GPa; a 1:1 mixture was used to bond the transducer to the crystal at room pressure.

The hydrostatic pressure was determined using a coil of manganin wire which had been calibrated with respect to the pressures associated with the melting of mercury during pressure release (the freezing point may be affected by super-cooling) and the centre of the region of indifference of the I \rightleftharpoons II transition of bismuth (Zeto and Vanfleter 1971). These two fixed points along with the known zero-pressure resistance define a quadratic

$$R = R_0(1 + \alpha_1 P + \alpha_2 P^2)$$

which describes the dependence of the manganin gauge resistance with pressure; for $R_0 = 42.376 \Omega$, $\alpha_1 = (2.654 \pm 0.006) \times 10^{-2} \text{ GPa}^{-1}$, and $\alpha_2 = -(5.76 \pm 0.26) \times 10^{-4} \text{ GPa}^{-2}$.

The presence of some internal cracks probably dominantly on the imperfect (010) cleavage plane was revealed by a series of low-amplitude echoes returning to the transducer even earlier than the round-trip compressional wave arising from LiNbO_3 mode impurity. Most of these spurious echoes were suppressed by the application of less than 0.5 GPa pressure, which is suggestive of closure of internal cracks. A very narrow gate width (0.1 μs) was used to minimize the influence of any such spurious echoes upon the interference between the desired shear mode echoes (Jackson et al. 1981). A typical set of such travel-time data from the comparative one- and two-transducer experiments is displayed in Figure 1. The very small scatter among the corrected travel times (e.g. $\pm 0.0005 \mu\text{s}$ at 2.3 GPa) provides clear evidence that the c_{55} mode measurements are not complicated by interference with spurious echoes. The good agreement between zero-pressure values of c_{55} deriving

from the present study (46.90 GPa) and that (46.7 GPa) of Sumino (1979) is a further indication that c_{55} mode travel-times are being reliably measured.

Results

The two-way travel-time $t(P)$, corrected in the manner outlined above for the effects of transducer-bond phase shifts, was measured as a function of hydrostatic pressure to 3 GPa. The modulus c_{55} may then be calculated from the expression

$$c_{55}(P) = \rho(P) v^2(P) = 4\rho(P) L^2(P)/t^2(P)$$

where ρ , L and v are respectively the density, length and wave velocity. $\rho(P)$ and $L(P)$ were calculated from Murnaghan expressions of the form

$$\rho/\rho_0 = (L_0/L)^3 = (1 + K'_0 P/K_0)^{1/K_0}$$

In the case of $L(P)$, K_0 and K'_0 are not the fayalite single-crystal incompressibility and its pressure derivative, but rather, the corresponding quantities for a hypothetical cubic crystal with the same linear compressibility as fayalite along [001] (see Nye 1957, p. 146 for details). The values of K_0 needed for the density and length calculations were based on the fayalite zero-pressure moduli reported by Sumino (1979); the pressure derivatives K'_0 were estimated from data for forsterite and olivine (Graham and Barsch 1969; Kumazawa and Anderson 1969).

The modulus versus pressure data ($c_{55}(P)$, Fig. 2) were fitted in a least squares sense to polynomials of successively higher order with the statistical significance of the added term being tested at each step (see Jackson and Niesler, 1982, for details). For the present data set addition of a quadratic term was significant at the 99.9 percent confidence level. The parameters of the best fitting quadratic are

$$c_{55} = 46.90 \pm 0.04 \text{ GPa}$$

$$\left(\frac{\partial c_{55}}{\partial P}\right)_{P=0, T} = 1.715 \pm 0.004$$

and

$$\left(\frac{\partial^2 c_{55}}{\partial P^2}\right)_T = -(0.136 \pm 0.003) \text{ GPa}^{-1}$$

The uncertainties assigned to these parameters, which reflect errors in measurement of travel-times and zero-pressure length and density and errors in pressure calibration, are significantly smaller than those reported by Jackson and Niesler (1982). This is a consequence of improved pressure calibration resulting from more precise determination of the manganin gauge resistances which correspond to the fixed points on the pressure scale.

The modulus c_{55} and its pressure derivatives measured in the present study are compared with data for other ferromagnesian olivines in Table 1. As previously mentioned, there is good agreement between the value of c_{55} deriving from the present study and that of Sumino (1979) for Fe_2SiO_4 . Among the transition-metal silicate olivines, c_{55} decreases across the sequence Ni, Co, Mn, Fe in accord with the variation of c_{44} among the transition-metal monoxides with the BI structure (Bass 1982). Our measured first pressure derivative is 'normal' in the sense that it is positive and comparable in magnitude with $\partial c_{55}/\partial P$ for

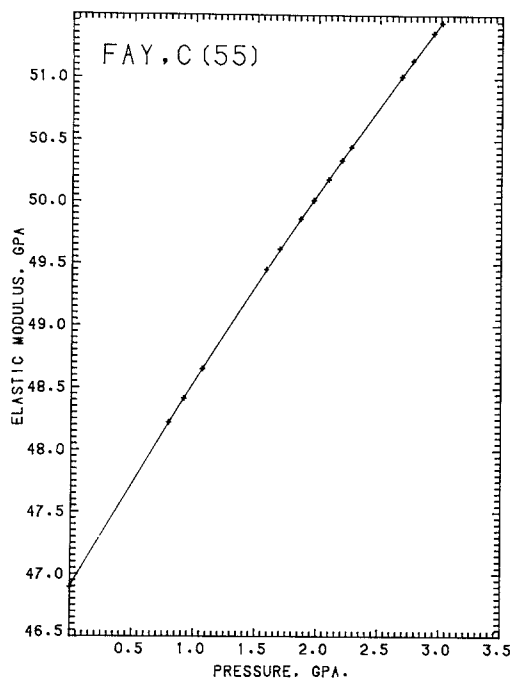


Fig. 2. The variation of the modulus c_{55} with pressure. The line of best fit is a quadratic characterized by the parameters $(c_{55})_0 = 46.90$ GPa, $(\partial c_{55}/\partial P)_0 = 1.715$, and $(\partial^2 c_{55}/\partial P^2)_0 = -0.136$ GPa^{-1} . The uncertainties in modulus are everywhere less than the size of the plotting symbols

Table 1. c_{55} and its pressure derivatives for $(\text{Mg, Fe})_2\text{SiO}_4$ olivine crystals

Composition	c_{55} (GPa)	$\frac{\partial c_{55}}{\partial P}$	$\frac{\partial^2 c_{55}}{\partial P^2}$ (GPa^{-1})	Reference
Fe_2SiO_4	46.7			Sumino (1979)
Fe_2SiO_4	46.90	1.715	-0.136	Present study
$(\text{Mg}_{0.9}\text{Fe}_{0.1})_2\text{SiO}_4$	78.05	1.64		Kumazawa and Anderson (1969)
Mg_2SiO_4	81.44	1.65		

magnesian olivines (Table 1), in spite of the much closer proximity (at atmospheric pressure) of Fe_2SiO_4 to the high-pressure boundary of its stability field.

A distinctive feature of the present study is the observation of negative curvature ($\partial^2 c_{55}/\partial P^2 = -(0.136 \pm 0.003) \text{ GPa}^{-1}$) in the pressure dependence of c_{55} . Such departures from linear variation of elastic moduli with pressure have generally not been observed in previous studies of relatively incompressible oxide and silicate crystals. However, it has recently been demonstrated that the improved techniques of ultrasonic interferometry and enhanced pressure capability in use in our laboratory, allow resolution of the second pressure derivatives of the elastic moduli of MgO (Jackson and Niesler 1982). The curvature ($\partial^2 c_{55}/\partial P^2$) observed in the present study of fayalite is greater by a factor of 4 to 8 than that associated with the pure shear modes of MgO ($\partial^2 c_{44}/\partial P^2 = -0.016 \text{ GPa}^{-1}$, $\partial^2 [(c_{11} - c_{12})/2]/\partial P^2 = -0.035 \text{ GPa}^{-1}$). This substantial curvature reduces $\partial c_{55}/\partial P$ from 1.72 at zero pressure to 1.31 at 3 GPa.

Table 2. Shear mode softening associated with the B1 → B2 phase transformation

Compound	c_{44} GPa	K GPa	$\partial c_{44}/\partial P$ —	$\partial K/\partial P$ —	c_{44}/K —	$\partial(c_{44}/K)/\partial P$ GPa ⁻¹	P_{TR}^a GPa	Reference
RbI	2.79	11.09	-0.49	5.41	0.25	-0.17	0.4	1, 2
RbBr	3.84	13.66	-0.56	5.30	0.28	-0.15	0.4	1, 2
RbCl	4.75	16.30	-0.61	5.35	0.29	-0.13	0.5	1, 2
KI	3.73	12.15	-0.23	5.10	0.31	-0.15	1.7	3, 4
KBr	5.08	14.86	-0.33	5.38	0.34	-0.15	1.7	5, 4
KCl	6.33	17.89	-0.41	4.78	0.35	-0.12	1.9	6, 4
KF	12.81	31.13	-0.45	5.02	0.41	-0.081	4	7, 8
NaCl	12.80	25.21	0.37	5.26	0.51	-0.091	30	9, 10
NaF	28.22	48.20	0.21	5.18	0.59	-0.059	27	11, 12
SrO	55.87	91.21	-0.21	5.18	0.61	-0.037	36	13, 14
CaO	80.32	112.50	0.20	4.83	0.71	-0.029	65	13, 15
MgO	155.8	162.5	1.11	4.13	0.96	-0.018	>200	16, 17
c.f. Fe ₂ SiO ₄ ^b	46.90	137.9	1.72	(4.8) ^c	0.34	+0.0006	2	18, 19

^a B1 → B2 transformation pressure at room temperature.

^b The shear modulus data for Fe₂SiO₄ are for c_{55} .

^c Value for forsterite (Graham and Barsch 1969).

References for Table 2

1. Chang and Barsch (1971); 2. Pistorius (1965a); 3. Barsch and Shull (1971); 4. Pistorius (1965b); 5. Reddy and Ruoff (1965); 6. Demarest et al. (1977); 7. Koliwad et al. (1967); 8. Yagi et al. (1978); 9. Spetzler et al. (1972); 10. Bassett et al. (1968); 11. Bartels and Schuele (1965); 12. Yagi et al. (1983); 13. Chang and Graham (1977); 14. Sato and Jeanloz (1981); 15. Jeanloz et al. (1979); 16. Jackson and Niesler (1982); 17. Vassiliou and Ahrens (1981); 18. present study and Sumino (1979); 19. Akimoto et al. (1967)

Discussion

The occurrence of premonitory shear mode softening associated with pressure-induced phase transformations is most clearly documented for the B1 ⇌ B2 transformation in the alkali halides (e.g. Reddy and Ruoff 1965; Anderson 1970; Demarest et al. 1977). The B1 → B2 transformation is accomplished by compression in the [111] direction (B1) which is equivalent to a combination of simple shears associated with the modulus c_{44} . The relevant elasticity data and B1 ⇌ B2 transformation pressures (at room temperature) for alkali halides and alkaline earth oxides are assembled in Table 2. It is noteworthy that the rubidium and potassium halides which transform below 2 GPa, are characterized by markedly negative values of $\partial c_{44}/\partial P$. Furthermore, increasing transformation pressure (down the table) correlates very well with increasing c_{44}/K and with increasing (i.e. less negative) $\partial(c_{44}/K)/\partial P$, in accord with the suggestion by Demarest et al. (1977) that the transformation occurs when c_{44}/K decreases to a critical value of about 0.15–0.20.

At room temperature and pressure, fayalite exhibits a rather low value (0.34) of c_{55}/K which is comparable with c_{44}/K for the potassium halides and suggestive of marginal stability of the olivine phase. There is, however, no comparable evidence for pressure-induced shear instability of fayalite since $\partial c_{55}/\partial P$ is strongly positive and $\partial(c_{55}/K)/\partial P = [\partial c_{55}/\partial P - c_{55}(\partial K/\partial P)/K]/K$ is near zero for all plausible values of $\partial K/\partial P$. The only hint of possible shear mode softening is the negative curvature in the pressure dependence of c_{55} . Such curvature may suggest a mild shear mode softening in the neighbourhood of the olivine-spinel equilibrium boundary, or alternatively, may represent the 'normal' response of the olivine lattice to compression. Further work now in progress on ~Fo₉₀ olivine should resolve this ambiguity.

Although extrapolation of the high-pressure high-tem-

perature olivine ⇌ spinel boundary for Fe₂SiO₄ (Akimoto et al. 1967) suggests an equilibrium pressure of ~2 GPa at room temperature, it is well known that the transformation (unlike the B1 → B2 transition) requires significant thermal activation. Thus although the measurements reported here extend beyond the equilibrium boundary without showing any clearly anomalous behaviour of the shear modulus c_{55} , they are conducted under conditions which preclude the occurrence of the transformation. We note that the need for substantial thermal activation may be readily incorporated into a martensitic model for the olivine → spinel transformation via the temperature dependence of the lattice function stresses acting on dislocations (Poirier 1981 b).

Under these circumstances, it is conceivable that premonitory shear mode softening associated with a martensitic olivine → spinel transformation, like the transformation itself, might require thermal activation. However, Fukizawa and Kinoshita (1982) have recently measured the shear wave velocity of polycrystalline fayalite under conditions of simultaneous high pressure and temperature to 5.2 GPa and 700° C, at which point the olivine → spinel transition began to occur, without observing an anomalous decrease in velocity. There is thus no evidence from either the present single-crystal study, or the work of Fukizawa and Kinoshita on polycrystalline material, for pressure-induced shear mode softening in fayalite premonitory to a martensitic olivine → spinel transformation.

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