

Elasticity of pyrope and majorite–pyrope solid solutions to high temperatures

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Received 11 February 2002; received in revised form 8 July 2002; accepted 9 July 2002

Abstract

Majorite–garnet solid solutions are major mineral phases in the Earth's upper mantle and transition zone. Here we present the first Brillouin scattering measurements of the elasticity of majorite (Mj, $\text{Mg}_4\text{Si}_4\text{O}_{12}$)–pyrope (Py, $\text{Mg}_3\text{Al}_2\text{Si}_3\text{O}_{12}$) solid solutions ($\text{Mj}_{50}\text{Py}_{50}$ and $\text{Mj}_{80}\text{Py}_{20}$) and single-crystal elasticity of pure synthetic pyrope at temperatures up to 800°C. The temperature derivatives of the adiabatic bulk (K_S) and shear (μ) moduli for all compositions along the Mj–Py join are the same within the experimental uncertainties ($-\partial K_S/\partial T = 14.0\text{--}14.5(20)$ MPa/K, $-\partial\mu/\partial T = 8.3\text{--}9.2(10)$ MPa/K). The temperature dependence of the acoustic velocities for Mj–Py solid solutions is about half that of other major transition zone minerals. This implies that temperature variations in the transition zone, inferred from lateral velocity heterogeneity, can be significantly underestimated if the properties of majoritic garnet are not taken into account.

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Keywords: majorite; pyrope; crystal structure; elasticity; temperature; elastic properties; transition zones

1. Introduction

Garnet is a major rock-forming mineral stable over a wide range of *PT* conditions: from the Earth's crust down to the lower mantle. Although most natural garnets are complex solid solutions, the most significant component in garnets of mantle origin is likely pyrope. In the depth range from ~300 to ~500 km (depending upon the bulk composition), pyroxenes progressively dissolve into garnet with increasing pressure, forming ma-

ajorite–garnet solid solutions (equivalently, an aluminum-deficient garnet) [1–5]. Because the majorite:garnet ratio varies with depth, and a CaSiO_3 perovskite phase exsolves from majorite–garnet at depths > 520 km, majoritic garnet in the Earth's mantle has a highly variable chemical composition which changes through the transition zone.

Majoritic garnet is volumetrically the single most abundant mineral phase in the transition zone, even for an olivine-rich 'pyrolite' composition, because other important phases (e.g. β - and γ - $(\text{Mg,Fe})_2\text{SiO}_4$) have significantly more limited *PT* stability fields. Therefore, knowledge of its elasticity as a function of composition, pressure and temperature is essential for constructing reliable mineralogical models of the Earth's mantle, interpreting tomographic observations, and

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understanding the behavior of subducted crustal material (which transforms to an assemblage dominated by majoritic garnet in the transition zone). The high-pressure elasticity of some garnets [6–8] and majoritic garnets (e.g. [9–11]) has been measured. Although the elasticity of several garnets has been measured at high temperatures (e.g. [12–17]), these measurements were performed either on garnets that are not representative of mantle compositions (e.g. grossular [14], almandine–spessartine [13]), and/or over a very moderate temperature interval (e.g. [13,15,16]). Some high-temperature elasticity measurements were performed on natural pyrope–almandine garnets, which are the most relevant to mantle mineralogy [12,15–17], but these garnets were very Fe rich with $X_{\text{Fe}} \geq 16\%$.

The effect of temperature on the acoustic properties of majorite–garnet solid solutions has not been studied before. The only high-temperature elasticity measurements for pure pyrope and majorite–pyrope solid solutions are static compression measurements performed in a large volume press using synchrotron radiation [18,19]. While these studies present similar temperature derivatives of the isothermal bulk modulus, they do not provide any information on the temperature dependence of the shear modulus, wave velocities, or single-crystal elastic moduli. Here we present the first acoustic (Brillouin spectroscopy) measurements on the elasticity of Fe-free synthetic pyrope and majorite–pyrope solid solutions.

2. Experiment

Majorite samples with compositions $\text{Py}_{50}\text{Mj}_{50}$ and $\text{Py}_{20}\text{Mj}_{80}$ (with densities of $\rho = 3.567 \text{ g/cm}^3$ and $\rho = 3.556 \text{ g/cm}^3$, respectively) were synthesized by T. Gasparik from homogeneous glasses (95% ^{29}Si enriched) at 17.7 GPa and 2000°C with the uniaxial split-sphere apparatus at the Stony Brook Center for High-Pressure Research. The samples were fine-grained ($< 1 \mu\text{m}$) with a random orientation of crystals, which was confirmed by angle-dispersive synchrotron X-ray measurements. Further details on sample synthesis and characterization are given by Sinogeikin et al.

[20]. Large ($\sim 1 \text{ mm}$) euhedral flawless single crystals of pure pyrope ($\rho = 3.567 \text{ g/cm}^3$) were synthesized from an oxide mix at 1000°C and $P_{\text{H}_2\text{O}} = 23.5 \text{ kbar}$ [21]. The single-crystal elasticity of these pyrope samples at ambient conditions and at high pressure to 20 GPa was reported elsewhere [21,22].

High-temperature Brillouin measurements were performed in a ceramic high-temperature cell of our design [23]. In some of the experiments an inconel cell body was used instead of ceramic. Grains of polycrystalline majorite–garnet solid solutions with lateral dimensions of $\sim 100 \mu\text{m}$ were embedded in platinum foil and polished to a thickness of 15–20 μm . The samples then were placed inside a relatively massive platinum sample holder with dimensions of approximately $3 \times 4 \times 0.25 \text{ mm}$ and secured with a platinum cap. The large ($\sim 1 \text{ mm}$) crystals of pyrope were polished parallel to a (110) growth face, placed directly into a platinum holder, and secured with high-temperature magnesium-based cement. Two K-type thermocouples, embedded into the platinum sample holder but separated from a holder body with fine-grained high-temperature zirconia cement, provided temperature readings with a precision of about 0.5°C. A temperature calibration against the α – β quartz transition shows that the temperature in the sample is within 2–3°C of the thermocouple reading.

The Brillouin measurements were performed using an argon ion laser ($\lambda = 514.5 \text{ nm}$) as a light source, and a Sandercock-type six-pass Fabry-Perot interferometer. A detailed description of the Brillouin system is given in [24]. We employed an 80° scattering angle with a platelet scattering geometry, which is insensitive to sample refractive index [22,25]. Geometric uncertainties were reduced by collecting the data in several different orientations. First, the elasticity of our samples was characterized at ambient conditions (STP) outside the high-temperature cell. After a sample was loaded in the high-temperature cell, its room temperature elastic properties were again measured for comparison with the accurate measurements made without the high-temperature cell. We made sure that the results with and without the temperature cell agreed within experimental

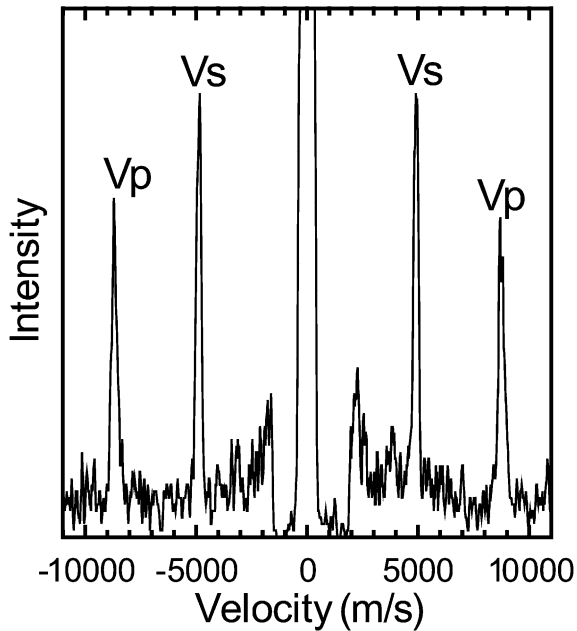


Fig. 1. Brillouin spectrum of $Mj_{50}Py_{50}$ at $800^{\circ}C$.

uncertainty ($\sim 0.5\%$ in velocities), indicating that the temperature cell was not introducing significant geometrical errors in velocity determinations (for example, due to the insulating windows used at high temperatures). Before the STP and high-temperature measurements the Brillouin system was calibrated against a single-crystal MgO standard to reduce systematic uncertainties in the velocity measurements.

The polycrystalline majorite–pyrope samples possess poor optical quality and a somewhat milky appearance. Nevertheless by using thin samples with a platelet geometry and a vertical-slit aperture mask we were able to obtain high-quality Brillouin spectra with well-defined peaks and a high signal-to-noise ratio (Fig. 1). Moreover, the quality of the Brillouin peaks increased with increasing temperature. The Brillouin spectra of single-crystal pyrope were of superior quality with virtually zero background.

The high-temperature measurements on pyrope were performed on two different crystals. Below $800^{\circ}C$ no change in optical quality of the samples was observed. Above this temperature the pyrope samples deteriorated, possibly due to interaction

of the sample with volatile components of the high-temperature cement, or the onset of decomposition of pyrope [26]. Even though we were able to collect some Brillouin spectra of pyrope at $1000^{\circ}C$, the poor quality of these spectra did not warrant their use.

Brillouin measurements were performed on $Mj_{50}Py_{50}$ and $Mj_{80}Py_{20}$ samples to $800^{\circ}C$ and $670^{\circ}C$, respectively. Above these temperatures the optical quality of the samples degraded and the quality of the Brillouin spectra became poor, although a separate set of high-temperature angle-dispersive synchrotron X-ray experiments demonstrated that $Mj_{50}Py_{50}$ persists metastably to at least $950^{\circ}C$.

The pyrope crystals were oriented using crystal morphology (growth faces). The acoustic velocities were measured as a function of phonon direction in the (110) plane, and these were inverted for the single-crystal elastic moduli using a linearized least-square inversion algorithm [27] (Table 1, Fig. 2). The densities of both majorite–pyrope solid solutions and pure pyrope at high temper-

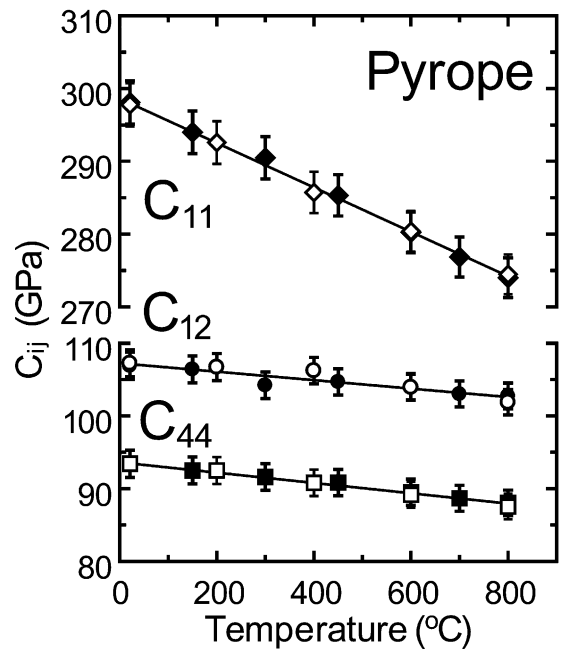


Fig. 2. Single-crystal elastic moduli of pyrope as a function of temperature. Open and closed symbols correspond to different sets of measurements performed on two samples.

ature were calculated using the thermal expansion data of Skinner [28] and Thieblot et al. [26], who showed that the thermal expansion coefficients of garnets are relatively insensitive to composition.

The fine-grain size and extremely low elastic anisotropy of Py, Mj, and Mj–Py solid solutions [20,22,29] allows us to measure the aggregate acoustic velocities of polycrystalline samples directly. No significant broadening of the Brillouin peaks was observed with increasing temperature.

3. Results and discussion

The temperature derivatives of elastic moduli for all of our samples were found to be the same within the experimental uncertainties (Table 2, Fig. 3), indicating that the Al/Si ratio (or garnet/majorite ratio) does not exert a strong influence on these properties. This conclusion is supported by volume compression studies of pure pyrope–majorite [18,19]. These studies reported values of $\partial K_T/\partial T$ which are in excellent agreement with the values calculated from our results (Table 2). A comparison of our results with those for natural pyrope–almandine garnets suggests that the presence of Fe increases $\delta K/\delta T$ [12,16]. In contrast, comparisons with prior results for grossular suggest that the primary effect of Ca is to increase $\delta\mu/\delta T$ [14]. Thus, the substitution of different cat-

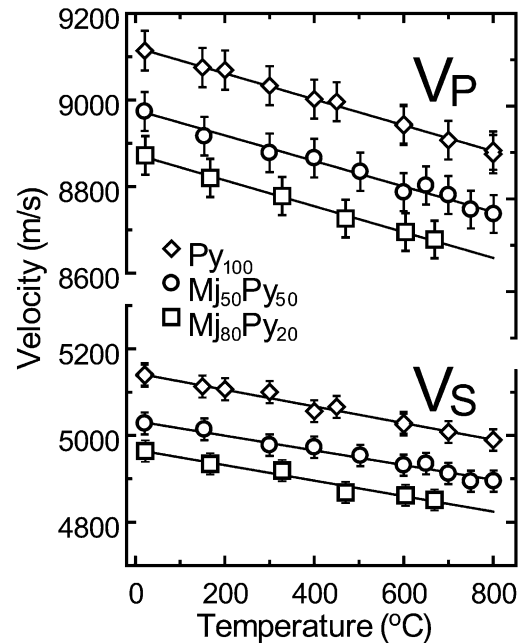


Fig. 3. Aggregate acoustic velocities of pyrope and majorite–pyrope solid solutions as a function of temperature. The error bars are 0.5%. The uncertainty in temperature (5 K) is smaller than the size of the symbols.

ions for Mg will increase either $\delta K/\delta T$ or $\delta\mu/\delta T$, depending on the specific cationic substitution. The temperature derivatives of the elastic moduli of chemically complex majoritic garnets in the

Table 1
Single-crystal elastic moduli of garnets as a function of temperature

Reference	Composition	Max. T (°C)	C_{11}	$\partial C_{11}/\partial T$	C_{12}	$\partial C_{12}/\partial T$	C_{44}	$\partial C_{44}/\partial T$
[15]	Py ₇₃ Alm ₁₆ And ₄ Uv ₆	80	296.6(16)	−36.8(16)	108.4(17)	−15.4(14)	91.7(2)	−7.4(3)
[15]	Py ₅₀ Alm ₄₆ Gr ₂ And ₁	80	301.5(15)	−38.8(32)	109.6(17)	−14.6(28)	95.0(1)	−9.7(4)
[15]	Py ₃₉ Alm ₅₄ Gr ₅ And ₁	80	302.5(11)	−40.2(27)	109.0(12)	−14.0(25)	94.7(1)	−9.4(3)
[12]	Py ₇₃ Alm ₁₆ And ₄ Uv ₆	727	296.6(15)	−36.0 ^a	108.5(16)	−11.1 ^a	91.6(15)	−8.7 ^a
[14]	Py ₁ Gr ₉₇ And ₂	1077	318.8(8)	−36.1 ^b	92.1(7)	−3.3	102.9(2)	−10.2 ^b
[17]	Py ₂₁ Alm ₇₆ Gr ₃	40	306.2	−34.8	112.5	−12.8	92.7	−10.4
[16]	Py ₆₁ Alm ₃₆ Gr ₂	100	292.2(4)	−32.9	106.2(5)	−11.8	91.6(1)	−7.5
[13]	Alm ₅₂ Sp ₄₆ Gr ₁	206	306.5(2)	−33.3	111.2(2)	−9.1	94.4(1)	−8.7
This study	Py ₁₀₀	800	298(3)	−30.5(30)	107(2)	−5.8(20)	93(2)	−7.1(20)

The compositions are approximate, with components < 1 mol% ignored. All single-crystal elastic moduli are in units of GPa, and their temperature derivatives are in units of MPa/K (GPa/1000 K).

The abbreviations for garnet end members are: Py, pyrope (Mg₃Al₂Si₃O₁₂); Alm, almandine (Fe₃Al₂Si₃O₁₂); And, andradite (Ca₃Fe₂Si₃O₁₂); Uv, uvarovite (Ca₃Cr₂Si₃O₁₂); Gr, grossular (Ca₃Al₂Si₃O₁₂); Sp, spessartine (Mn₃Al₂Si₃O₁₂).

^a Linear fits to reported data.

^b Only the first temperature derivatives are given.

mantle are expected to be slightly higher than the values obtained in this study due to the presence of Fe and Ca.

The values of $\partial K_S/\partial T$ and $\partial\mu/\partial T$ which have often been used in mantle modeling are those of Suzuki and Anderson [12] for a natural sample containing 16 mol% of almandine ($\text{Fe}_3\text{Al}_2\text{Si}_3\text{O}_{12}$). Such a high Fe content in garnets could possibly be found in the upper mantle because Fe preferably partitions into garnet in olivine–pyroxene–garnet assemblages. In the transition zone the partitioning relations between garnet and the olivine polymorphs change significantly. The partitioning of Fe between β -phase and majoritic garnet is subequal or only slightly shifted toward majoritic garnet, whereas in an assemblage of γ -phase and majoritic garnet Fe partitions preferentially into γ -phase (e.g. [3,5]). Therefore, taking the average $[\text{Fe}]/([\text{Fe}]+[\text{Mg}])$ content in the mantle to be 10–12%, majoritic garnet in the transition zone will be Fe-poor compared with garnets at shallower levels, and the value of $\partial K_S/\partial T$ for transition zone majoritic garnet is expected to be

somewhere between our new value and those of Suzuki and Anderson [12].

To illustrate how the choice of elastic modulus temperature derivatives affects the extrapolation of elastic properties to mantle conditions, we calculated the acoustic velocities for majoritic garnet along a 1673 K adiabat at 660 km depth using the formalism of Duffy and Anderson [30] and the results of this study, as well as those of Suzuki and Anderson [12]. Our temperature derivatives result in 1.5–2.0% higher velocities, which could significantly affect mineralogical interpretations of the 660 km discontinuity.

The velocity variations obtained from seismic tomography are often interpreted as temperature variations in the Earth (e.g. [31,32]), but the inferred temperatures are often based on outdated measurements or velocity systematics that involve questionable assumptions (e.g. [33]). Our results from this and previous recent work on γ - $(\text{Mg,Fe})_2\text{SiO}_4$ [34] allow us to calculate more accurate temperature derivatives of velocities for the actual high-pressure transition zone phases,

Table 2
Elastic moduli of garnets and majorite–pyrope solid solutions and their temperature derivatives

Reference	Composition	K_S (GPa)	$\partial K_S/\partial T$ (MPa/K)	K_T (GPa)	$\partial K_T/\partial T$ (MPa/K)	μ (GPa)	$\partial\mu/\partial T$ (MPa/K)
[15]	Py ₇₃ Alm ₁₆ And ₄ Uv ₆	171.2(16)	−22.5(12)			92.7(1)	−8.7(3)
[15]	Py ₅₀ Alm ₄₆ Gr ₂ And ₁	173.6(16)	−22.7(25)			95.3(1)	−10.7(3)
[15]	Py ₃₉ Alm ₅₄ Gr ₅ And ₁	173.5(12)	−22.7(23)			95.5(1)	−10.9(3)
[14]	Gr ₇₆ And ₂₂ Sp ₁	166.2(12)	−14.7 ^a (2)	165(1)	−20.9(2)	103.4(2)	−12.5(1) ^a
[14]	Py ₁ Gr ₉₇ And ₂	167.8(7)	−14.9(1)	166.4(7)	−20.0(1)	107.0(2)	−12.5(1) ^a
[12]	Py ₇₃ Alm ₁₆ And ₄ Uv ₆	171.2(8)	−19.4 ^b	169.4	−25.6 ^b	92.6(3)	−10.2 ^b
[17]	Py ₂₁ Alm ₇₆ Gr ₃	177	−20.1			94.3	−10.6
[13]	Alm ₅₂ Sp ₄₆ Gr ₁	176.3(2)	−17.2(5)				
[18]	Py ₁₀₀			170(2)	−20(3)		
[18]	En ₃₈ Py ₆₂			160(3)	−20(1)		
[19]	En ₈₀ Py ₂₀			156	−19.0 (30)		
This study ^c	Py ₁₀₀	171(2)	−14.0 (20)	169.4(20)	−19.4(30)	94(2)	−9.2 (10)
This study ^c	En ₅₀ Py ₅₀	167(3)	−14.5 (20)	165.5(20)	−19.6(30)	90(2)	−8.2 (10)
This study ^c	En ₈₀ Py ₂₀	163(3)	−14.3 (20)	161.7(20)	−19.1(30)	88(2)	−8.3 (10)

Thermal expansion data of pyrope ([26,28]) were used to calculate elastic moduli from acoustic velocities at high temperature. The uncertainties in temperature derivatives take into account errors in linear regression as well as uncertainty in velocity measurements.

^a Only the first reported temperature derivatives are given.

^b Linear fits to reported data.

^c The isothermal bulk moduli were calculated from the relations: $K_T = K_S/(1+\alpha\gamma T)$, $\gamma = \alpha K_S/\rho C_P$. The values of α for pyrope were taken from [38]. The values for C_P are from [39]. α and γ for majorite–pyrope solid solutions are assumed to be equal to those of pyrope.

Table 3

Temperature derivatives of the acoustic velocities of some mantle minerals extrapolated to 700 km depth along 1673 K adiabat

Mineral	$\partial V_p/\partial T$ (km/s)/100 K	$\partial V_s/\partial T$ (km/s)/100 K	Reference
Majorite–pyrope	−0.016	−0.011	this study
β -(Mg,Fe) ₂ SiO ₄	−0.035	−0.030	[35,36]
γ -(Mg,Fe) ₂ SiO ₄	−0.038	−0.029	[34]

accounting for the effects of P and T . Unfortunately the cross pressure–temperature derivatives of the elastic moduli are not known. However, we can assess the importance of these parameters by comparing our ambient pressure–temperature derivatives of the pyrope bulk modulus with those of Wang et al. [18] who reported the average temperature derivative of the isothermal bulk modulus of pyrope in the pressure range of 0–11 GPa and over a similar temperature range as in the present experiments. The resultant values of $\partial K_T/\partial T$ agree within the experimental uncertainties, indicating that to first order, the effect of pressure on the temperature dependence of the isothermal bulk modulus is not large.

Acoustic velocities of majorite–pyrope solid solutions and their temperature dependencies along high-temperature adiabats were calculated yielding the results given in Table 3. Also listed are the temperature derivatives of the acoustic velocities for β -(Mg,Fe)₂SiO₄ and γ -(Mg,Fe)₂SiO₄ calculated from data in [34–36]. Compared with values commonly cited in the literature (e.g. −0.050 (km/s)/100 K, [31]), our new values are significantly lower. This result suggests that temperature variations within the transition zone may be far greater than previously thought, assuming that velocity variations are due to temperature alone. Unfortunately, at this point it is not possible to take into account the effects of anelasticity (which is presumably more important for shallower mantle) [37], or possible nonlinearity in temperature derivatives at higher temperatures. Nevertheless, the large difference in temperature derivatives of the acoustic velocities between majoritic garnet and olivine polymorphs strongly suggests that the thermal structure of the transition zone may need to be re-evaluated using more realistic tem-

perature derivatives of the elastic properties of mantle rocks.

Acknowledgements

We thank K. Langer, T. Gasparik and R.J. Kirkpatrick for providing samples. We are grateful to W.A. Bassett, F. Schilling, and H.J. Reichmann for comments and constructive reviews. This research was supported by NSF (EAR 0003383). [SK]

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