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Equation of state of antigorite, stability field of serpentines, and seismicity in subduction zones

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[1] Antigorite, the high-pressure variety of serpentine, is a major water carrier in subduction zones. Its equation of state, which controls its stability field at high pressure, was determined at ambient temperature up to 10 GPa by in situ synchrotron X-Ray diffraction in a diamond-anvil cell. No amorphization, phase transition or hysteresis were detected during compression or decompression. Compression is anisotropic, with the *c* axis twice as compressible as the others. A fit to the second order Birch-Murnaghan equation of state gave $V_0 = 2926.23(50) \text{ \AA}^3$ and $K_0 = 67.27(123) \text{ GPa}$. In antigorite the atomic assemblage is denser than in lizardite, which may influence phase stability. The antigorite K_0 value obtained here is significantly higher than currently assumed in thermodynamic databases. Antigorite *P-T* stability field is re-evaluated, with major dehydration reaction (and associated potential earthquake) loci occurring at lower pressures, and is found to be consistent with the latest experimental results. **Citation:** Hilaiet, N., I. Daniel, and B. Reynard (2006), Equation of state of antigorite, stability field of serpentines, and seismicity in subduction zones, *Geophys. Res. Lett.*, 33, L02302, doi:10.1029/2005GL024728.

1. Introduction

[2] Serpentines are hydrous phyllosilicates formed from anhydrous Fe-Mg minerals in ultrabasic rocks, either (1) during hydrothermal alteration of the oceanic lithosphere or (2) by hydration of the peridotitic mantle wedge above the subducting dehydrating slab [Bebout and Barton, 1989; Guillot *et al.*, 2001]. Containing up to 13 wt % water, serpentines are among the most hydrated minerals going down to the mantle during subduction. Their dehydration is believed to be one of the major causes of mantle wedge hydration and related partial melting processes [Ulmer and Trommsdorf, 1995], and of deep focus earthquakes [Dobson *et al.*, 2002; Raleigh and Paterson, 1965].

[3] Antigorite is the predominant structural variety in rocks showing high pressure mineralogical assemblages [Mellini and Zanazzi, 1987]. It has been shown experimentally to be the stable variety at high pressure and high temperature (*HP-HT*), although its exact stability field is a matter of debate [e.g., Evans, 1976; Ulmer and Trommsdorf, 1995; Wunder and Schreyer, 1997].

[4] Antigorite structure is based upon the stacking of corrugated layers comprised of alternating octahedral and tetrahedral sheets [Wicks and O'Hanley, 1988]. It displays periodic reversals of the layer's polarity, characterized by the number *m* of tetrahedra in one modulation length.

Polytypism and polysomatism (variation of *m*), and chemical variability, result in a great structural variability, within a given sample and from one sample to the other.

[5] In order to model the *P-T* stability field of antigorite, to quantify the role of serpentine in subduction zone dynamics and water recycling into the mantle, a reliable Equation of State (EoS) for antigorite is required. For that purpose, we measured *P-V* EoS for antigorite using in situ synchrotron X-Ray Diffraction (XRD) in a membrane diamond anvil cell (DAC), up to $\sim 10 \text{ GPa}$ at ambient temperature.

2. Experimental Methods

[6] We used a natural sample Cu12, from the Escambray massif (Central Cuba), with a structural formula $(\text{Mg}_{2.62}\text{Fe}_{0.16}\text{Al}_{0.15})_{\Sigma=2.93}(\text{Si}_{1.96}\text{Al}_{0.04})_{\Sigma=2}\text{O}_5(\text{OH})_{3.57}$ [Auzende *et al.*, 2004]. It is a one layer polytype with an average *m* = 14 superperiodicity [Auzende *et al.*, 2002]. Sample Cu12 contains less than 5 wt% chrysotile and exhibits small variations in the structural parameter *m*, both of which are common features of antigorite.

[7] The sample was finely ground (grain size $\sim 2\text{--}5 \mu\text{m}$) in an agate mortar, and pressurized in a membrane type diamond anvil-cell [Chervin *et al.*, 1995]. Pressure was measured with the ruby fluorescence technique [Mao *et al.*, 1986], before and after each diffraction exposure. The mean value is considered to be the pressure at which *V* is measured. Pressure transmitting medium was a 16:4:1 methanol-ethanol-water mixture, which ensures hydrostatic conditions up to the highest pressures reached here of 10 GPa.

[8] Measurements were carried out with a monochromatic synchrotron beam ($\lambda = 0.3706 \text{ \AA}$), at the ID30 beamline of the European Synchrotron Radiation Facility (Grenoble, France), during compression and decompression. Diffraction patterns were collected with a MAR[®]345 detector. The time for diffraction exposure was 54 s. The sample to detector distance was calibrated against a Silicon standard. The Fit2D software [Hammersley *et al.*, 1996] was used to apply tilt and distortion correction, and to integrate the 2D patterns.

[9] Lattice parameters were refined in Le Bail [Le Bail *et al.*, 1988] procedure, with the GSAS package [Larson and Von Dreele, 2004]. Since no refinement for unit cell atomic positions in the *m* = 14 polysome is available, atomic positions in the cell used for the refinements were from antigorite-1*T* (*m* = 17, space group Pm [Capitani and Mellini, 2004]). Using the *m* = 17 structure to index diffraction peaks of our sample with *m* = 14 has negligible effect on refined *m* = 1 volumes. (*P,V*) data were fitted with

Figure 1. Typical XRD pattern of antigorite at 4.94 GPa, refined in Le Bail procedure with the GSAS package. Main peaks are indexed. Calculated peak position marks are omitted for clarity. The residual is shown below the pattern; it is mostly due to intensity miscalculations due to texture effects, not to peak position mismatch.

a second and third order Birch-Murnaghan EoS using the EoSfit5.2 software [Angel, 2001]. Data points were weighted by an estimated 3% P uncertainty, and by estimated standard deviation on V ($\text{esd}(V)$, 1σ) from diffraction peak fitting.

3. Results

[10] An example of refined antigorite pattern is given in Figure 1. The small residues between refined and observed spectra are mostly due to intensity mismatch (possible texture effect which was not refined), and not to peak position mismatch, ensuring reliable volume determinations. Peaks remain sharp over the whole P range, indicating no amorphization, and all spectra could be fitted with the same space group. The hydrostatic compression of this material to 10 GPa is reversible with no hysteresis between compression and decompression.

[11] Table 1 gives the lattice parameters and volume for each pressure point. In the following, subscript 0 on V

and K stands for standard condition $P_0 = 10^5$ Pa and $T_0 = 298$ K.

[12] Antigorite exhibits pronounced anisotropy under compression. The c axis is twice as compressible as the a and b axes, with compressibilities at P_0 of 0.0083, 0.0037 and 0.0033 GPa^{-1} , respectively. The a axis is slightly more compressible than the b axis, which may be explained by an increase in the sheet curvature. Indeed, tilt of the tetrahedra along a should be easier than along b if we assume incompressible tetrahedra. The β angle slightly decreases with increasing P .

[13] The compression curve (Figure 2) was fitted with second and third order EoS. The best fit is obtained with a second order EoS, yielding $V_0 = 2926.23(50)$ $\text{\AA}^3$ and $K_0 = 67.27(123)$ GPa ($K'_0 = 4$). A fit to the third order yielded $V_0 = 2926.65(47)$ $\text{\AA}^3$, $K_0 = 62.03(223)$ GPa and $K'_0 = 6.39(98)$. A F - f plot [Angel, 2001] confirms a second order EoS for antigorite, and shows that V_0 estimation is correct. The K_0 value estimated from this F - f plot is coherent with results from the second order EoS fit.

4. Discussion

[14] The measured standard state unit cell parameters and volume are specific to this antigorite sample and integrated over its structural variability. Indeed, the bulk modulus might vary between different polysomes and polytypes. However, as the compression curves are similar for different structural varieties [Mellini and Zanazzi, 1989; Hilairet et al., manuscript in preparation, 2005], it is unlikely that polytypism and polysomatism in serpentines affect significantly bulk compressibility. The bulk compressibility of antigorite is dominated by the compressibility of the c axis [Mellini and Zanazzi, 1989], which is consistent with the weaker interlayer interactions, when compared with the strong ionic bonds in the TO layer.

[15] The V_0 value corresponding to $m = 1$ for antigorite is 172 $\text{\AA}^3$, lower than the value of $V_0 \sim 179$ $\text{\AA}^3$ for lizardite [Mellini and Zanazzi, 1989]. Since the bulk modulus of these two varieties is similar, as discussed above, their

Table 1. Antigorite Unit Cell Parameters During Compression and Decompression From Pattern Refinements With GSAS^a

Pressure $\pm$ 3%, GPa	V , $\text{\AA}^3$	a , $\text{\AA}$	b , $\text{\AA}$	c , $\text{\AA}$	β , $^\circ$
0.0001 $\pm$ 0.00003	2927.200(249)	43.5590(15)	9.2597(3)	7.2590(6)	91.264(5)
0.25 $\pm$ 0.01	2912.922(266)	43.5195(21)	9.2521(5)	7.2362(5)	91.264(6)
0.68 $\pm$ 0.02	2897.092(217)	43.4692(17)	9.2467(3)	7.2094(4)	91.257(6)
1.95 $\pm$ 0.06	2842.292(237)	43.2608(24)	9.2126(6)	7.1332(5)	91.183(7)
2.86 $\pm$ 0.09	2814.499(246)	43.1591(28)	9.1890(5)	7.0980(5)	91.090(7)
3.81 $\pm$ 0.11	2783.744(269)	43.0156(26)	9.1597(4)	7.0665(6)	91.122(9)
4.94 $\pm$ 0.15	2752.398(213)	42.8450(22)	9.1279(3)	7.0389(4)	90.992(6)
5.87 $\pm$ 0.18	2718.505(034)	42.7178(33)	9.1023(4)	6.9922(7)	90.803(9)
7.03 $\pm$ 0.21	2689.081(352)	42.5941(04)	9.0727(4)	6.9592(7)	90.800(10)
8.05 $\pm$ 0.24	2650.289(182)	42.4336(25)	9.0252(6)	6.9213(3)	90.979(10)
8.49 $\pm$ 0.25	2645.977(286)	42.3631(26)	9.0280(4)	6.9191(5)	90.809(7)
9.00 $\pm$ 0.27	2628.157(231)	42.2930(27)	9.0121(5)	6.8962(4)	90.886(10)
9.45 $\pm$ 0.28	2623.060(244)	42.2413(18)	9.0061(4)	6.8958(5)	90.850(6)
9.98 $\pm$ 0.30	2610.730(272)	42.2876(23)	8.9783(5)	6.8772(6)	90.943(5)
7.82 $\pm$ 0.23	2661.707(264)	42.4364(21)	9.0436(5)	6.9363(5)	90.863(7)
4.98 $\pm$ 0.15	2755.339(233)	42.8703(16)	9.1376(3)	7.0345(5)	90.863(6)
3.10 $\pm$ 0.09	2810.150(366)	43.1104(23)	9.1833(7)	7.0995(7)	91.080(9)
1.16 $\pm$ 0.03	2873.300(254)	43.4110(16)	9.2252(5)	7.1766(5)	91.278(6)

^aUncertainty on pressure is estimated to 3%; $\text{Esd}(1\sigma)$ are given in parentheses on the last decimals.

Figure 2. Normalized compression curve at ambient temperature for antigorite. Filled and empty symbols represent data collected during compression and decompression respectively. Standard deviation (1σ) on volume is smaller than data points, and errors bars on P correspond to a 3% uncertainty. The fit obtained with a second order Birch-Murnaghan EoS (full line) is better than a fit to the third order (dotted line).

relative stability is controlled by their V_0 , antigorite being the high-pressure form.

[16] Little data exist on P - V - T of antigorite [Bose and Navrotsky, 1998], yielding, from a fit with a Birch-Murnaghan EoS, $K_0 = 49.6(7)$ GPa and $K'_0 = 6.14(43)$. The discrepancies with our EoS cannot be explained by sample chemistry and structure differences. They might be due to differences in pressure and temperature measurements. Indeed, Bose and Navrotsky [1998] conducted their experiments in a DIA-type apparatus, known to generate significant deviatoric stress at low/medium T , which may lead to inaccuracies in pressure calibration using NaCl EoS. Because they do not present their diffraction patterns, we cannot discuss further this issue.

[17] The present antigorite sample is quite rich in Al (3.45 wt % Al_2O_3), which extends the antigorite stability field toward higher temperatures, and slightly toward higher pressures with respect to pure antigorite [Bromiley and Pawley, 2003]. Antigorite with low m values and high Al content (up to 5%), these two being probably correlated [Uehara and Shirozu, 1985; Wunder *et al.*, 2001], are considered to be those of the highest grade in the subduction processes. Hence, the K_0 value proposed in this study for an Al-rich antigorite sample with $m = 14$ is directly applicable to models of subduction zone processes.

[18] Most thermodynamic calculations use self-consistent databases [Berman, 1988; Holland and Powell, 1998]. In their latest estimations, Holland and Powell [1998] gave the same value of $K_0 = 52.5$ GPa ($K'_0 = 4$) to both ideal Mg end-member compositions of antigorite and chrysotile. Our bulk modulus of antigorite is much higher than this value. Figure 3 shows thermodynamic calculations for P - T stability field of antigorite in a H_2O saturated MSH system of ideal antigorite composition using (1) Holland and Powell [1998] database values and (2) our new bulk modulus and using the thermal expansivity of Holland and Powell [1998]. Our larger bulk modulus results in a reduced stability field for antigorite, by up to 1 GPa and over 100°C , with respect to that obtained with Holland and Powell [1998] value. Therefore the most important dehydration reactions of

antigorite would occur at lower P and T . The new calculated invariant point in a MSH system for antigorite, enstatite and phase A, lies at ~ 5 GPa and 550°C instead of ~ 5.9 GPa and 600°C . This is actually identical to the experimental results in a MSH water saturated system from Komabayashi *et al.* [2005], and calculations by Wunder and Schreyer [1997]. Hence, the antigorite EoS proposed here gives thermodynamic predictions in agreement with the latest in situ phase equilibrium results from Komabayashi *et al.* [2005], and confirms the need to revise the antigorite EoS parameters in current databases.

[19] Thermodynamic calculations in MASH systems would shift the dehydration reaction loci toward higher pressure and temperature [Bromiley and Pawley, 2003]. When calculating the phase diagram in the MFSH system, a multivariant assemblage with antigorite and minor orthopyroxene appears at ~ 3 GPa at 550°C , but antigorite remains stable up to the invariant point in MSH at 5 GPa and 550°C . In a more realistic MFASH system with a non-antigorite stoichiometry, antigorite breakdown to phase A or Mg-sursassite [Bromiley and Pawley, 2002], could occur at

Figure 3. Ideal antigorite stability field in a water saturated system, computed with the PeRple_X package [Connolly and Petrini, 2002], with the bulk modulus obtained in this study, $K_0 = 67.27$ GPa (full lines), and with Holland and Powell's [1998] database value $K_0 = 54.5$ GPa (dotted lines). A larger value for the bulk modulus reduces significantly the computed stability field, and particularly shifts the location of the dehydration reaction $\text{Atg} = \text{En phA}$ toward lower P . The star corresponds to the invariant point found by Komabayashi *et al.* [2005] for water saturated experiments in the MSH system. The grey arrow represents a P - T trajectory for lithospheric mantle in a cold slab. The numbers are the compressional waves velocities (km s^{-1}) in antigorite calculated with the K_0 obtained in this study (in bold letters), and K_0 [Holland and Powell, 1998] (light letters). The shaded area corresponds to the P - T locations for intermediate-depth events in the lower seismicity plane of the North-East Japan subduction zone [Peacock, 2001]. It is limited at high-pressure and low-temperature by the serpentine dehydration curve from our refined EoS. Atg, antigorite; En, enstatite; phA, phase A; Fo, Forsterite; ta, talc.

lower P than the invariant point calculated here in a simplified MSH system. Phase diagram calculations such chemistries would require the inclusion of Mg-sursassite in thermodynamic databases which is beyond the scope of this study.

[20] A higher value for the bulk modulus of antigorite, using existing data on thermal expansivity [Holland and Powell, 1998], has consequences on computed seismic velocities and densities (Figure 3). At realistic conditions for cold slabs sinking into the mantle, for example at ~ 5.7 GPa and 470°C , antigorite density calculated with our new bulk modulus is 2765 kg.m^{-3} approximately 1.6 % lower than values obtained from Holland and Powell [1998]. Similarly, V_p in pure antigorite is 5.8 % higher than with Holland and Powell [1998] K_0 value, which should lead to a significant effect in seismic models of serpentinite layers or of extensively serpentinized peridotites.

[21] With the new EoS we provide, the predicted depth of serpentine dehydration in the subducting lithospheric mantle will change for subduction zones which are sufficiently cold for the dehydration reaction $\text{Atg} = \text{En} + \text{phA}$ to occur, such as North East Japan. This subduction zone has a double-plane structure, which lower plane is attributed to dehydration reactions involving serpentine or chlorite [Peacock, 2001; Hacker et al., 2003]. Intermediate depth earthquakes cannot be related to antigorite dehydration and are rather due to chlorite [Pawley et al., 2002].

[22] Figure 3 shows the P - T locations reported for the lower plane seismicity of the North East Japan subduction zone [Peacock, 2001; from Hasegawa et al., 1994, seismic data]. We note that in the coldest zone of the slab, the deepest events occur at least at 1 GPa lower than the dehydration reaction $\text{Atg} = \text{En} + \text{phA}$ loci calculated with Holland and Powell [1998], whereas their maximum P - T locations are indeed consistent with the dehydration loci we recalculate here.

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