

# Experimental Elasticity of Earth's Deep Mantle

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## Abstract (200 words):

Remote sensing by seismology provides increasingly detailed views on the seismic wave velocity structure of Earth's deep mantle. The interpretation of these observations critically relies on quantitative knowledge of the elastic properties of Earth's mantle minerals. This knowledge comes largely from experimental *in-situ* measurements of sound wave velocities of mantle minerals at high-pressure/-temperature, a field that has significantly evolved within the past decade. In this Technical Review, we highlight the major methodologies employed and discuss their advantages, limitations and future potential. We focus on light scattering techniques in the diamond-anvil cell and ultrasonic methods in large volume presses since these techniques have provided – and likely will provide – the majority of elasticity data on deep mantle minerals in the foreseeable future. We summarize the current state of knowledge with respect to the elastic properties of the minerals making up the transition zone and lower mantle, where substantial advances have been recently made, and highlight major gaps in published data.

## 1. Introduction

Physical, chemical and geodynamic properties and processes occurring throughout the Earth's mantle govern the behaviour of our planet's interior, and in turn exert strong controls on the nature of surface plate tectonics. Thus, to successfully model the evolution of Earth, or indeed other planets, on billion-year timescales we must have an accurate and quantitative understanding of the physical and chemical properties of the planet's mantle. The major challenge with understanding the deep Earth has been, and always will remain, the lack of any direct access. Thus, it has been information from remote sensing, largely seismological observations, that has driven some of the most significant insights into our planet's deep, and otherwise hidden, interior over the last decades. Whether analysing receiver functions, precursor arrivals, or reporting tomographic models or maps of mantle anisotropy, the quantity and quality of seismological observations of Earth's interior are continuously growing at an ever-increasing pace. Indeed, the number of high-quality observations has risen to such an extent that arguably the current challenge is not a lack of observations of Earth's deep mantle, but rather their interpretation. Figure 1 shows an illustrative summary of the range of mantle seismic observations that still need to be fully understood, including the large low shear velocity provinces, velocity anomalies in the transition zone velocity and pervasive scatterers throughout the mid-mantle.

Successful inversion of seismic observations requires knowledge of the physical properties of all possible Earth materials, at any and all conditions that occur throughout the mantle. To a first-order approximation, the seismic wave velocities in any material (which are responsible for the observations seismologists can make) are governed by a few key isotropic elastic properties; the bulk modulus ( $K$ ), shear modulus ( $G$ ), and density ( $\rho$ ) (Box 1). In the Earth's mantle, the materials in question are rock assemblages which themselves are composed from a handful of mineral species (Fig. 2). Thus, knowledge of a few elastic parameters for this handful of mineral species ought to be enough to allow full inversion of global seismological data. However, the elastic properties of each constituent phase change substantially as a function of pressure ( $P$ ), temperature ( $T$ ), and chemical composition ( $X$ ). Consequently, whilst it has been decades since researchers first attempted to utilise seismic inversions, success has continued to seem evasive due to the lack of knowledge of elastic properties of mantle constituents in appropriate parameter space. Even the elastic properties of olivine, which is the most abundant and most heavily studied upper mantle mineral, have only been measured at temperatures up to 1300 K at pressures below 13 GPa<sup>4</sup>. These data are several hundred degrees below the mantle geotherm, and require significant extrapolation to directly interpret seismic observations. Data for other mantle phases are even more sparse, normally limited to low pressure and/or

54 temperature and very few constrain elastic anisotropy at all. Thus, reliable seismic inversions remain a  
55 “dream”. It is only with the advent of new in-situ techniques over the last few years that measurements of  
56 minerals’ elasticity at pressure and/or temperature conditions approaching those relevant for the mantle<sup>5-17</sup>  
57 have become widely possible. These developments now provide increased hope that the long-realised  
58 potential of the elasticity-based approach to understanding the Earth’s interior may begin to flourish.

59  
60 However, recent steps forward have also revealed additional hurdles. The emerging consensus is that the  
61 interpretation of seismic observations is far more complicated than previously assumed, with other changes  
62 in mineral properties (in addition to changes in  $P$ ,  $T$  and  $X$ ) known to affect, or even dominate, seismic  
63 velocities. These extra degrees of freedom range from the spin state of valence elements to the presence  
64 and concentration of defects and the oxygen fugacity. Some of these processes are summarised in Fig. 2. As  
65 a result, the traditional interpretation of seismic tomography, which assigns velocity variations to changes in  
66 temperature, might not always be sufficient to explain the origin of the variations observed. While the  
67 traditional interpretation likely holds in some localities, it might completely fail in others. One potential  
68 example are regions where ferropericlase may undergo its iron spin transition<sup>19</sup>. Here, compressional wave  
69 velocities are theorised to increase with temperature – not decrease as traditionally expected<sup>16,20,21</sup> (Fig. 2b).  
70 This is just one of several hypotheses that is awaiting verification from high- $PT$  elasticity measurements.  
71 Importantly, since many of the predicted mineralogical changes occur over extended depth ranges (Fig. 2a),  
72 established equation of state (EoS) extrapolations<sup>22</sup> that have been the backbone of any mineral physics  
73 based interpretations for decades will fail as they cannot easily replicate these non-linear mineral properties.  
74 Recent studies have made it clear that more and better measurements of elastic parameters are required.  
75 Specifically, these measurements must provide improved  $PT$ -resolution that will allow compilation of a more  
76 complete database that minimises the need to inter- and extrapolate elastic properties.

77  
78 In this Technical Review we provide the perspective of two experimentalists on the major techniques  
79 currently being used to determine elastic properties at deep mantle conditions. We summarise current  
80 knowledge and highlight some of the major gaps that exist in experiments and studied materials relevant to  
81 the transition zone (TZ) and lower mantle (LM). We particularly discuss the techniques employed in light of  
82 their potential, their limitations and their uncertainties, and hope that this will provide a reference piece that  
83 is accessible for a variety of researchers.

84

## 85 **2. Measurement techniques**

86 In principal, seismic wave velocities can be determined either by measuring them (or their related elastic  
87 moduli) using experiments, or by calculating them with atomistic theory. Whilst we focus almost entirely on  
88 methods for the experimental determination of velocities here, it is clear that computational methods  
89 provide a complementary approach<sup>e.g.23</sup>. Computational techniques can range from classical mechanics to  
90 molecular dynamics to full ab-initio. They can already predict the elastic properties of mantle phases with  
91 uncertainties smaller than 2-5 % across the entire range of mantle  $PT$ -conditions and are continually evolving.  
92 However, although these computational uncertainties may seem small in an absolute sense, they remain  
93 sufficiently large compared with the seismic anomalies observed in the mantle. These discrepancies derive  
94 from the approximations required in calculations, and the only way of significantly improving them is via  
95 iteration using accurate experimental measurements as the benchmark.

96  
97 Experimental techniques that study elastic properties of deep Earth materials are numerous (see also refs.  
98 <sup>24,25</sup>), and include a range of X-ray inelastic scattering, (visible) light inelastic scattering and ultrasonic  
99 techniques. Some of these techniques that can provide accurate and insightful data, not only for mineral  
100 velocities but also elastic anisotropy, phonon density of states (PDOS), phonon lifetimes etc. are limited to  
101 ambient pressures, e.g. Resonant Ultrasound Spectroscopy, RUS<sup>26,27</sup>, or require sufficiently large samples that  
102 their application at high pressure is impractical, e.g. Inelastic Neutron Scattering, INS<sup>28</sup>. However, many  
103 others can principally be applied to study the properties of Earth materials at mantle pressures.

104  
105 Both Inelastic X-ray Scattering<sup>29,30</sup> (IXS) and Nuclear Resonant Inelastic X-ray scattering<sup>31</sup> (NRIXS) utilise X-  
106 rays to probe the phonon properties of crystalline materials, which can be related to their elastic

107 properties. Both techniques, exploiting the high-intensity and fine focus of synchrotron X-ray sources, can  
108 be applied in the diamond anvil cell (DAC) at lower mantle pressures. Whilst this makes them well-suited  
109 for investigating mineral properties at mantle pressures they rely on X-ray scattering ability of the sample;  
110 samples composed of relatively heavy atoms, in geological terms, are the most ideal. IXS provides  
111 measurements of individual points on the (linear part of the) phonon dispersion curve, from which  
112 compressional and shear wave velocities can be derived<sup>12</sup>, and can therefore give a direct constraint of  
113 geophysically relevant velocities. NRIXS, on the other hand, allows determination of the partial PDOS, which  
114 can directly constrain the Debye velocity ( $v_D$ )<sup>32</sup>. The Debye velocity, however, is not a geophysical property  
115 and indirect estimates for  $v_p$  and  $v_s$  must be extracted from  $v_D$  using an averaging scheme applied alongside  
116 independent constraints on the bulk modulus. Both techniques are “slow” when studying high-pressure  
117 samples in a DAC, where individual velocity measurements take several hours. These time constraints,  
118 partially arising from the poor X-ray scattering of silicate samples, combined with the need for dedicated  
119 synchrotron facilities explains the small number of studies applying these techniques to mantle  
120 minerals<sup>12,32-37</sup>.

121  
122 Brillouin Spectroscopy (BS) and Impulsive stimulated light scattering (ISLS) are light scattering techniques  
123 that utilise the inelastic interactions of coherent laser sources and sample phonons, and thus do not require  
124 synchrotron sources. These light scattering techniques, provide direct measurements of  $v_p$  and  $v_s$ , and due  
125 to their ability to be performed in university laboratories around the world have become widespread in  
126 Earth Science applications (see section 3).

127  
128 Ultrasonic techniques do not exploit inelastic interactions, but instead study the elasticity of deep Earth  
129 materials at high pressure by literally sending “seismic waves”, albeit at extremely high-frequency (kHz-GHz  
130 acoustic waves), into high-pressure samples. Techniques are based on interferometry, and can be performed  
131 using either piezoelectric materials or laser sources to generate the acoustic waves. Techniques using  
132 piezoelectric transducers typically operate at MHz-GHz frequencies, and are employed in conjunction with  
133 the large-volume press (LVP) or DAC to directly measure  $v_p$  and  $v_s$ . Whilst both devices have been used to  
134 study the acoustic velocities of mantle silicate minerals, the DAC-based GHz-ultrasonics technique has, until  
135 now, been limited to  $\sim 10$  GPa<sup>38,39</sup> and temperatures of only a few hundred degrees<sup>40</sup>. LVP-based ultrasonics  
136 can be readily applied at conditions  $> 20$  GPa and 2000K<sup>41</sup> (section 3).

137 Laser-based ultrasonic interferometry, which is commonly named picosecond ultrasonics (PSU), is an  
138 ultrafast pulse-probe optical technique<sup>42</sup>. Stress waves are generated by interaction of the pulse laser with  
139 the sample surface, and their arrival at the near (after reflection) or far side is detected using laser-based  
140 interferometry. As PSU measurements can be performed in the DAC<sup>42</sup>, achieving high-pressure is relatively  
141 straightforward. PSU is mostly limited to opaque materials, and if transparent samples (including silicates)  
142 are studied experiments are essentially analogous to time-domain Brillouin Scattering<sup>43</sup>. Applications of PSU  
143 have so far been restricted to relatively low temperatures, but extension to mantle temperatures appears  
144 possible<sup>44</sup>.

145  
146 Whilst the experimental toolbox available for studying mineral elasticity is clearly diverse, Brillouin Scattering  
147 in DACs and Ultrasonic Interferometry in the LVP (Fig. 3) have, until now, provided the majority of  
148 experimental elasticity data for mantle silicates. Additionally, their widespread development means they also  
149 provide the best hope for expanding these datasets further in the near future. We therefore provide more  
150 extensive overviews of these two techniques in the subsequent sections, deliberately highlighting their  
151 limitations and current application in Earth Science research. Additionally, we also briefly outline the use of  
152 X-ray diffraction on mantle minerals, because whilst it does not directly constrain velocities, it does play a  
153 central role in the determination of the compressibility of mantle minerals and their high-*PT* densities. The  
154 other techniques that we have briefly mentioned above might become more widely applied to deep mantle  
155 minerals in the future, but have so-far mostly been used to study materials that may be present in the Earth’s  
156 core. Table 1 provides a summary of references to datasets for deep Earth minerals produced using each of  
157 these techniques.

158  
159

### 160 **3. Laser light scattering techniques**

161

#### 162 3.1. Brillouin Spectroscopy

163 Brillouin Spectroscopy (BS) is an experimental technique capable of measuring mineral elasticity in the  
164 diamond anvil cell (DAC). Its use has become far more widespread in recent years; the number of high-  
165 pressure Brillouin Scattering systems actively used for Earth Science research has increased from 3 systems  
166 in 2000 to > 10 systems in 2020. A comprehensive discussion of the BS methodology can be found in ref. <sup>2</sup>.

167

168 BS works by measuring a frequency shift between a probing laser and scattered light from the sample, which  
169 is generated during inelastic interactions with sound waves (acoustic phonons) in the studied material. High-  
170 pressure BS experiments on Earth mantle minerals are almost exclusively conducted in the symmetric  
171 platelet forward scattering geometry (Fig. 3a), where a direct relation exists between the measured  
172 frequency shift and the sound wave velocities<sup>45</sup> (Fig. 3b). These measurements require a double-side polished  
173 and transparent sample, where all sample and diamond interfaces are parallel. Sound wave velocities at high  
174 pressure can be measured with an accuracy and precision better than 1% and 0.5%, respectively, in the  
175 DAC<sup>2,46</sup>. Larger errors can be introduced if the scattering geometry deviates from the ideal case<sup>46</sup>, which can  
176 be evaluated by collecting data over a large angular range<sup>5</sup>, however this is not common practice in individual  
177 studies. BS can be performed on both single crystal samples and polycrystalline aggregates. The Brillouin  
178 signal is strongly dependent on the thickness of the sample. In experiments aimed at deep mantle pressures,  
179 samples have to be very thin (<15  $\mu\text{m}$ ), this leads to signal collection times of several tenths of minutes to  
180 hours.

181

182 In single-crystal BS experiments, which constrain the full elastic tensor (Box 2), sound velocities are measured  
183 along several different propagation directions in the crystal. Subsequently, the entire data set is inverted to  
184 derive the elastic constants, from which values for the elastic moduli  $K$  and  $G$  can be calculated using  
185 appropriate averaging schemes (Box 2). Datasets often contain tens or hundreds of individual measurements,  
186 which, when inverted, provide well-constrained elastic properties. Elastic constants can principally be  
187 determined with an uncertainty < 1% (refs. <sup>2,47</sup>). As a generalisation, it is expected that the larger the number  
188 of measured velocities, the smaller the uncertainties in the derived constants. However, actual uncertainties  
189 depend on numerous factors including the data analysis strategy<sup>48,49</sup>.

190

191 The price of low uncertainties, however, is time; even cubic-symmetry crystals, such as ringwoodite, majoritic  
192 garnet, ferropericase, where measurements are only required in one plane, require very long experimental  
193 times. The situation is compounded for minerals with lower crystallographic symmetries, including  
194 orthorhombic wadsleyite and bridgmanite, which require measurements from multiple orientated samples  
195 to provide access to additional lattice directions. Recent developments have focused on facilitating the  
196 loading of multiple crystals into one diamond anvil cell<sup>5,13,50-52</sup>. This allows constraint of the entire elastic  
197 tensor of low symmetry crystals in a single experiment<sup>5,50,51</sup> or direct constraint of the effect of chemical  
198 variations on velocities<sup>13</sup>.

199

200 It is now possible to perform single-crystal Brillouin measurements to almost 100 GPa<sup>16,53</sup>. But the large time  
201 requirements, often weeks for one pressure point, have mostly limited experiments to minerals with cubic  
202 symmetry; majoritic garnets<sup>54-56</sup>, ringwoodite<sup>13,57,58</sup> and ferropericase<sup>16,17,53,59</sup>. Only few data sets have been  
203 measured on wadsleyite<sup>50,60-63</sup> and bridgmanite<sup>5,9</sup>, which are orthorhombic. Single-crystal BS measurements  
204 on CaSiO<sub>3</sub> perovskite, post-perovskite or seifertite are not possible, since none of these phases can be  
205 recovered from the high- $PT$  synthesis. A major limitation of all BS experiments is that at pressures beyond ~  
206 40 GPa the measurement of compressional velocities becomes challenging. This is because the  $v_p$  signal is  
207 obscured by a peak from the diamond anvils (Fig. 3b), which occurs above ~ 20-40 GPa at room temperature  
208 for typical mantle minerals<sup>5</sup>.

209

210 Single-crystal results at high  $PT$  using resistive-heated DACs on TZ and LM minerals are so-far limited to  
211 temperatures < 1000K, and have only been reported for ringwoodite<sup>13,64</sup>, majoritic garnet<sup>55</sup>, and (ferro-)  
212 )pericase<sup>65,66</sup>. To-date, measurements using resistive-heated DACs are, therefore, not directly applicable to

213 mantle *PT* conditions. The use of laser-heating can possibly overcome the current obstacles in achieving  
214 mantle temperatures. However, Brillouin spectroscopy measurements demand optically transparent  
215 samples, which commonly do not absorb infrared wavelengths ( $\sim 1\mu\text{m}$ ) most widely used for laser-heating.  
216 A common solution is to add a laser-absorbing material, such as platinum to the sample. This, however, is  
217 not possible in combination with Brillouin spectroscopy as samples need to remain transparent. An  
218 alternative solution is to use  $\text{CO}_2$  lasers that operate at wavelengths of  $\sim 10\mu\text{m}$  to heat the sample. The  
219 longer wavelength, and absence of fibre  $\text{CO}_2$  lasers has, however, complicated the experimental setup as  
220 they are noticeably more difficult to handle, guide and focus. We note that the recent development of wave-  
221 guide-based  $\text{CO}_2$  lasers might improve the situation<sup>67,68</sup>. To-date, first single-crystal Brillouin scattering  
222 experiments have been successfully performed with laser-heating at more realistic mantle temperatures<sup>4,68-</sup>  
223 <sup>71</sup>, but no complete data set has yet been published for any TZ or LM mineral.

224  
225 Brillouin Spectroscopy experiments can also be performed on polycrystalline aggregates, which provide  
226 direct constraints on the average elastic bulk and shear moduli if the density of the sample is known (see Box  
227 1). Measurements can be performed on pre-synthesised samples, or on those synthesised *in-situ*. Generally,  
228 measurements on polycrystalline aggregates are “easier” as compared to those on single crystals, because  
229 no directional dependence of velocities is expected. This significantly reduces the number of measurements  
230 required making polycrystalline experiments much faster.

231  
232 Time gains in polycrystalline experiments do come at the expense of information on the elastic tensor and  
233 elastic anisotropy. Measured polycrystalline velocities fall somewhere in the spectrum of average velocities,  
234 and it is not straightforward to know what kind of average they represent (see Box 2). Further considerations  
235 for polycrystalline data include that apparent velocities are sensitive to samples’ microstructures, such as  
236 grain size<sup>72</sup>, the shape of grains, and/or a shape preferred orientation. Measured velocities can further be  
237 affected by lattice preferred orientations if the materials are elastically anisotropic<sup>73</sup>. Such elastic anisotropy  
238 might also lead to inhomogeneous stress distributions in the sample that can affect the elastic response and  
239 wave propagation<sup>2,74</sup>. A final consideration is that the strength of the Brillouin signal is a function of the  
240 coupling efficiency between the probing laser and the propagation direction of the acoustic phonons<sup>e.g. 75</sup>,  
241 which implies that the measured velocities could be weighted towards certain propagation directions. Whilst  
242 this is negligible for mantle minerals with low elastic anisotropy, it can significantly bias results in cases with  
243 significant anisotropy. Proof-of-concept work, comparing polycrystalline and single-crystal BS, has been  
244 reported for bridgmanite at room pressure<sup>76</sup>, but no systematic high-pressure studies have yet been  
245 performed.

246  
247 Despite the above limitations, the major advantages of polycrystalline BS are, as already mentioned, the  
248 significantly reduced experimental times which facilitates high-*T* experiments, and the possibility of  
249 measuring samples not available as single-crystals<sup>e.g. 77</sup>. These advantages have driven several studies;  
250 measurements have now been reported for bridgmanite<sup>6,15,78,79</sup>, majoritic garnets<sup>80</sup>, ferropervicase<sup>6</sup>, (post-)  
251 stishovite<sup>81</sup>, calcium perovskite<sup>82</sup> and post-perovskite<sup>77</sup>. Moreover, high-pressure Brillouin spectroscopy  
252 measurements on laser-heated polycrystalline aggregates of Al-bearing  $\text{MgSiO}_3$  bridgmanite and MgO have  
253 been successfully performed at a temperature of 2700 K<sup>6</sup>.

### 254 255 3.2. Impulsive Stimulated Light Scattering

256 Impulsive Stimulated Light Scattering (ISLS)<sup>83</sup> is a similar technique to BS, but there is no diamond  
257 interference as in BS. The major difference is that a second (pulsed) laser is used to induce the acoustic waves  
258 in the sample, and it is these induced sound waves that are measured, whereas sound waves in BS are of  
259 purely thermal origin. In almost all aspects ISLS has the same advantages and limitations of BS. The most  
260 significant differences are that ISLS is capable of measuring opaque samples as well as constraining  
261 compressional wave velocities at all pressures. However, ISLS does not provide a comparably reliable  
262 measurement of bulk shear wave velocities; these have to be inferred from an interfacial wave travelling  
263 between the sample and the pressure-transmitting medium<sup>8</sup>. An additional unique complication in ISLS  
264 measurements in the DAC is that the pulsed laser used to excite the acoustic waves (1064 nm wavelength)  
265 can potentially damage the diamond anvils, in particular when they contain defects and/or are strained<sup>53</sup>. So

266 far, only ferropericlase and bridgmanite have been measured by ISLS at lower mantle pressures<sup>8,9,15</sup>, with  
267 ISLS otherwise largely employed to study materials of Earth's core. A recent exciting development is the  
268 combined use of BS (to measure shear wave velocities) and ISLS (to measure compressional wave velocities)  
269 to reliably determine the velocities of bridgmanite and ferropericlase<sup>9,53,15</sup>.

#### 270 271 **4. Ultrasonic techniques**

272 Ultrasonic interferometry (US), most regularly performed in the large volume press (LVP), is an alternative  
273 technique for the measurement of mineral elasticity at high pressures (Fig. 3c). The principal is to pass sound  
274 pulses into a sample and detect returning echoes. By measuring the time-delay ( $\Delta t$ ) between echoes  
275 returning from the upper and lower sample surfaces ( $R_2$  and  $R_3$  in Fig. 3c), and assuming the sample behaves  
276 like an infinite thin parallel plate of thickness ( $L$ ), the velocity ( $v$ ) of sound travelling through the sample is  
277 simply  $2L/\Delta t$ . Conceptually this approach is simple. In reality the high-pressure environment (either the LVP  
278 or DAC) complicates the measurements by enforcing strict geometrical constraints on, and the inability to  
279 visually see, the sample's length. Despite challenges, a well-tested and fairly standardised approach,  
280 summarised briefly here, is now used around the world such that simultaneous  $v_p$  and  $v_s$  measurements with  
281 uncertainties at the 1% level or better are possible in the LVP<sup>14,41,84-87</sup> or DAC<sup>39</sup>. Up until now the DAC-based  
282 approach has been limited to low temperature and pressure conditions of  $< 10$  GPa, which are the limit of  
283 the silica aerosol gel that provides an acoustic bond in the DAC<sup>38,39</sup>. Thus, the discussion below focusses on  
284 LVP-based ultrasonic experiments (mostly performed in the multi-anvil), because at the present time this  
285 approach can actually achieve conditions of the transition zone and/or lower mantle. If future innovations  
286 extend the DAC technique beyond 10 GPa, all descriptions given here can be applied to this DAC-based  
287 ultrasonic approach, only samples are significantly smaller ( $< 50 \mu\text{m}$  thick) and probing frequencies higher  
288 (250+ MHz).

#### 289 290 **4.1. Large Volume Press Ultrasonic Interferometry**

291 The most stringent constraints for LVP-US experiments are those placed on the sample. Polycrystalline  
292 samples must be parallel-faced media, that are free from porosity, cracks or any other macroscopic defects,  
293 whilst remaining fine-grained and not possessing lattice preferred orientation. Single crystals can also be  
294 measured, although to date such studies remain fairly limited in the LVP<sup>88</sup>, likely due to difficulties associated  
295 with producing high-quality samples of sufficient size. Single crystal studies of high-pressure phases, although  
296 at  $< 10$  GPa, have more commonly been performed using US in the DAC<sup>39,89</sup>. Whether poly- or  
297 monocrystalline, the sample's surfaces must be flat, and its aspect ratio should be such that its length is  
298 smaller than its radius to avoid edge effects affecting wave propagation. In practice these requirements mean  
299 that samples are usually pre-synthesised (normally by pre-sintering) and manipulated into cylinders of  
300 appropriate dimensions; for studies at  $> 20$  GPa samples will be smaller than 2mm in diameter. Provided  
301 compression proceeds as expected and the parallel-faced sample geometry is maintained, high-quality  
302 velocity measurements are possible. The combined time requirement for one synthesis and ultrasonic  
303 experiment, including all preparation, is around 1 week; this is one of the largest downsides of this approach.  
304 A few recent studies have successfully avoided pre-synthesising samples<sup>7,11</sup>, instead combining synthesis and  
305 ultrasonic measurements in one experiment. Whilst this approach sounds attractive, approximately halving  
306 the time requirement per ultrasonic experiment, these studies discarded data from a significant number of  
307 experimental runs due to imperfect sample geometry at target conditions (pers. comm. Thomson and  
308 Gréaux) so are not significantly more economical.

309  
310 Once at high- $PT$  conditions two measurements are required; (i) the time delay between returning echoes ( $\Delta t$ )  
311 and, (ii) the sample length ( $L$ ). The former is more trivial. Sound pulses, with a wavelength in the sample of  $<$   
312  $L/2$ , are generated by a dual mode  $\text{LiNbO}_3$  or other suitable piezoelectric transducer (e.g.  $\text{ZnO}$  is used in DAC-  
313 based US experiments<sup>38,39</sup>) and passed into the sample. Typical frequencies used in LVP experiments are 20-  
314 80 MHz. Returning echoes, with or without amplification, are measured using an oscilloscope (Fig. 3d). The  
315 time delay between these echoes can be determined with an uncertainty  $\ll 0.5\%$  (normally  $< 0.1\%$ ). The  
316 main difficulty occurs if samples shorten sufficiently at high pressure, such that the two echoes interfere with  
317 one-another, this requires more complex signal processing. Compared with travel time measurements, the  
318 accurate determination of sample length is more challenging. In laboratory experiments, the inability to

319 directly image the sample means that the sample length can only be estimated; normally using “the Cook  
320 Method” (ref. <sup>90</sup>). Whilst the accuracy of this approach depends on the material being studied (as it relies on  
321 an equation of state for the sample), uncertainties will typically be > 3-5%. These directly translate into  
322 uncertainties of 3-5% in derived sound wave velocities.

323  
324 A more reliable way to quantify the sample length is by measuring it using synchrotron X-rays that penetrate  
325 through the sample environment sufficiently to allow radiographic imaging of the sample<sup>86</sup>. By positioning  
326 the cylindrical axis of the sample perpendicular to the X-ray beam, sample lengths can be determined to 1-2  
327  $\mu\text{m}$  precision. This typically corresponds to uncertainties in length measurements of  $\sim 0.2\text{-}0.4\%$  (ref. <sup>86</sup>).  
328 However, it remains difficult to ascertain if samples are truly cylindrical, or if they have “barrelled” during  
329 compression, which might affect velocity propagation. Thus, it is reasonable to assume that combined  
330 geometrical and travel time errors combine to overall uncertainties of 0.25 - 0.5% in best-case conditions for  
331 synchrotron-based experiments. At synchrotron sources, density is additionally determined using X-ray  
332 diffraction (see below), allowing for direct determination of  $K_s$  and  $G$  (see Box 1 for relationship with  $v_p$  and  
333  $v_s$ ). The limitations of synchrotron-based ultrasonic experiments are mostly logistical and financial.  
334 Synchrotron experiments require access to beamline instruments, restricted via competitive proposal  
335 systems. Experimental time allocations are infrequent, typically a few days twice a year, permitting few  
336 repeated measurements. Combined with individual experiment success rates of  $\sim 75\%$ , these time and  
337 financial restrictions mean that experimental datasets from LVP-ultrasonics produced at synchrotron sources  
338 are sparse, and will likely remain sparse for some time.

339  
340 However, huge advances have been made over the last few years, and are still ongoing. The routinely  
341 accessible range of  $PT$  conditions achievable using the LVP is ever expanding (Figure 4) due to the advent of  
342 new commercial tungsten carbide and sintered diamond anvil materials<sup>91,92</sup>. LVP-based ultrasonic  
343 experiments have now been performed at conditions of  $\sim 27\text{ GPa}$  (ref. <sup>93</sup>) and  $2400\text{ K}$  (ref. <sup>94</sup>), and there is no  
344 reason to believe this field will not continue to expand. Additionally, the time required for individual  
345 ultrasonic measurements has reduced from > 5 minutes to < 10 seconds<sup>87</sup> as oscilloscope performance has  
346 rapidly improved. Up until now the LVP ultrasonic approach has predominantly been used to study  
347 polycrystalline solids and their isotropic bulk properties, but recently there has been early success employing  
348 it to measure the thermoplastic properties and compressional wave velocity of liquids and other amorphous  
349 materials<sup>94</sup>. With further developments there is no obvious reason, aside from time constraints and sample  
350 synthesis, why single crystals could not also be targeted more frequently in future studies, following  
351 approaches used in Brillouin Spectroscopy of studying oriented single crystal samples. To our knowledge,  
352 such measurements are currently limited to a single study of MgO to 8 GPa and 1600 K<sup>88</sup>, but future work  
353 could aim to constrain anisotropy at high temperatures.

354  
355 **5. Measurements of density and compressibility by X-ray diffraction**  
356 X-ray diffraction (XRD) is the established and traditional approach for determining the density and  
357 compression behaviour of materials. High- $P$  XRD experiments can be performed in either LVPs or DACs, and  
358 in recent years are usually carried out at synchrotron sources. Compared with laboratory sources,  
359 synchrotrons provide high-energy X-rays that allow for short acquisition times. XRD measurements  
360 principally constrain the sample’s lattice parameters and crystal structure. Lattice parameters can be used to  
361 calculate the (unit cell) volume  $V$  of the sample and, if the mass is known, its density. Measurements of unit  
362 cell volumes as a function of pressure are now routine, and resulting datasets can be fitted using an  
363 appropriate equation of state formalism. The room pressure isothermal bulk modulus  $K_{0,T}$  and its pressure  
364 derivative at ambient conditions ( $K'_0$ ) can be extracted from the resulting models. These data alone provide  
365 an estimate of a material’s bulk sound velocity ( $v_\phi$ , Box 1).

366  
367 Diffraction experiments are significantly quicker and easier to perform compared with any direct sound wave  
368 velocity measurements. The relative ease of the experiments and the long-standing implementation of the  
369 technique at global synchrotron sources have generated large datasets for several minerals at high  $PT$ , largely  
370 by conducting x-ray diffraction in laser-heated DACs. Recent increases in synchrotrons’ photon fluxes,  
371 combined with developments in detector technology have made XRD experiments even faster than before.

372 Ten years ago, the collection of a single powder diffraction pattern at a state-of-the-art synchrotron facility  
373 took a few minutes; the same information can now be derived substantially faster. For example, using novel  
374 single photon counting detectors with very high quantum efficiency individual diffraction patterns of  
375 (Mg,Fe)O can be collected in a few milliseconds e.g. <sup>10,95,96</sup>. These developments allow for collection of quasi-  
376 continuous volume-compression data, from which the elastic isothermal bulk modulus (eq. 4) can be directly  
377 determined at any pressure without relying on an equation of state formalism. These developments of time-  
378 resolved XRD, in particular when combined with finely controlled compression paths or stress cycles at  
379 tailored frequencies facilitated through membrane- or (resistive-heated) piezo-driven DACs<sup>10,96</sup>, will no doubt  
380 lead to new innovations in the use of synchrotron XRD in high-*P* experiments. However, XRD measurements  
381 do not provide information on the shear modulus of the sample.

382  
383 Both BS and US setups have been coupled with x-ray diffraction at Synchrotron sources<sup>97,98</sup>, whilst XRD-BS  
384 has additionally been performed in university laboratories<sup>5,99</sup>, to measure sound wave velocities and  
385 densities simultaneously. This becomes particularly useful for high-*PT* measurements where the density of  
386 the minerals is often poorly known. Moreover, the combination of the Brillouin or ultrasonic results ( $K_s$ ) and  
387 x-ray diffraction allows for calculating an “absolute pressure”, i.e. a pressure that is independent of the used  
388 pressure standard<sup>5,50,55,97,99,100</sup>. The combination therefore has the potential to eliminate uncertainties in  
389 experimental results related to the use of a secondary pressure standard.

390  
391 **6. Outstanding challenges and future directions:**  
392 Light scattering methods in the DAC and LVP-ultrasonic interferometry have emerged as the dominant  
393 techniques to measure high-*PT* elastic properties of deep mantle phases. While light scattering in the DAC at  
394 pressure of the lower mantle is now routine, the simultaneous generation of high temperature remains highly  
395 challenging. Equivalently, LVP experiments can reach temperatures of the mantle adiabat but have not been  
396 used to achieve *PT* conditions beyond the very upper portions of the lower mantle when combined with  
397 elasticity measurements. Across both DAC and LVP techniques, experimental data on the seismic properties  
398 of most phases now exists at simultaneously high-*PT* conditions. However, in almost all cases these data are  
399 limited to one, or in some cases two, study(ies) that measured polycrystalline seismic properties.  
400 Ringwoodite, calcium perovskite, bridgmanite, stishovite and majoritic garnet have been studied using LVP  
401 ultrasonics to temperatures of > 1500 K – but data are for a single composition, at limited *PT* conditions and  
402 to date there have been few duplicate measurements. High-*PT* polycrystalline BS has been used to measure  
403 ferropericlase and bridgmanite at 2700 K and ~95 GPa. However, the entirety of these data are limited to 7  
404 data points (4 for fp and 3 for bm), each of which is likely to have large uncertainties on the temperatures at  
405 experimental conditions. Furthermore, there are no measurements of the seismic properties of NAL-phase,  
406 CF-phase, post-stishovite, seifertite or post-perovskite at temperatures above 300 K, and those for wadsleyite  
407 are limited to ~ 1000 K. Even for the most-studied lower mantle phase ferropericlase, uncertainties remain  
408 regarding the effect of the iron spin transition on the elastic response and the shape of the bulk modulus  
409 softening with pressure<sup>8,12,16,53</sup>. And there are still no experimental reported measurements of sound wave  
410 velocities across the spin transition region at high *T* (Fig. 2b). Moreover, our experimental knowledge on the  
411 elastic properties of minor phases in the mantle, such as carbonates<sup>101</sup> or hydrous mantle phases<sup>102</sup>, as well  
412 as melts<sup>103</sup> remains poor or almost non-existent. Examples of some of the high-pressure hydrous mantle  
413 phases that have so-far been studied include single-crystal superhydrous phase B, which has been measured  
414 by Brillouin spectroscopy at 12 GPa and 700 K<sup>104</sup>, whilst Phase D and Phase E have still only been studied at  
415 ambient conditions<sup>105-107</sup>. Elastic properties of polycrystalline Ice VII and  $\delta$ -AlOOH have been studied to much  
416 higher pressures extending above 100 GPa<sup>108-110</sup>.

417  
418 The bottom line is, experimental datasets are nowhere near suitable. They often remain insufficient for  
419 tuning atomistic modelling results or for developing models of seismic properties that can be used to  
420 interpret the cause of observed seismic reflections, conversions and or anomalies in tomographic models.  
421 We are missing the quantity of data to allow any reliable statistical tests of data quality and/or derived  
422 results, and it remains challenging to quantify the real uncertainties on data reported from any single study.  
423 Likewise, very few deep mantle minerals with similar chemical composition have been measured by different  
424 techniques (e.g. Brillouin and ultrasonics) and comparison of the limited data sets available reveals some

425 inconsistencies, e.g. see discussions for olivine and wadsleyite in refs. <sup>4,50</sup>. Unfortunately, systematically  
426 unravelling the underlying reason is difficult given the small amount of comparable data sets (composition,  
427 pressure-/temperature-range). Overall, uncertainties in velocities for any mantle mineral phase at the *PT*-  
428 conditions of the Earth's deep mantle cannot be assumed to be known to an accuracy better than  $\pm 1$ -2%, or  
429 in many cases  $\pm 2$ -5%. The magnitude of typical observed seismic anomalies in tomographic models reach  
430 maxima of  $\pm 2$ -3% at transition zone conditions, but decrease to  $< 1\%$  in  $v_p$  or  $v_s$  throughout the lower  
431 mantle<sup>e.g.111</sup>. Despite limited tomographic resolution likely masking the true magnitude of anomalies, which  
432 will be somewhat larger and more in line with those observed using full waveform inversion techniques<sup>e.g.</sup>  
433 <sup>112,113</sup>, it very quickly becomes clear that reliable interpretations of observed velocity anomalies are  
434 challenging.

435  
436 The current picture is even more incomplete if we consider the range of measurements available that  
437 constrain the anisotropy of deep Earth phases. At present such high-*P* datasets are generated solely from  
438 DAC-based light-scattering techniques and, aside from olivine, garnet, ringwoodite and ferropericlase, all  
439 such data are currently limited to room temperature conditions. Data for bridgmanite, Earth's most abundant  
440 mineral, come from only two studies<sup>5,9</sup> that are limited to pressures  $< 40$  GPa (corresponding to a depth of  
441 about 1000 km). Moreover, the two studies do not entirely agree in their findings. The chemical complexity  
442 of bridgmanite combined with the absence of any other high-pressure single crystal elasticity data on  
443 bridgmanite make it virtually impossible to unravel the reason for the apparent discrepancy between the  
444 published data. Are the differences related to structural or chemical differences in the studied sample,  
445 differences in the employed laboratory setups, and/or the data analysis procedure? Moreover, even if  
446 knowledge about the elastic tensor of the major mantle minerals at relevant conditions were to become  
447 available, the way that single crystals orient in the mantle – the lattice preferred orientation – needs to be  
448 known to model seismic anisotropy observations in the mantle<sup>114,115</sup> and link them to mantle flow patterns.

449  
450 Additional challenges arise when comparing current experimental results to seismic observations. Elastic  
451 properties of lower mantle and transition zone minerals are measured at very high frequencies (MHz-THz),  
452 while seismic waves propagate at much lower frequencies ( $\sim 0.001$ -100 Hz). It is often assumed that the  
453 mantle behaves like a completely elastic material, in which case wave velocities will be independent of  
454 frequency (there will be no dispersion). However, dispersion of seismic waves might occur in some parts of  
455 the deep mantle. Moreover, seismic attenuation in the mantle<sup>116-118</sup> can occur when processes, such as grain  
456 boundary sliding, chemical diffusion, or phase transitions, take place on the same time-scale (or frequency)  
457 as the pressure (stress) variations induced by a passing seismic wave. In this case, part of the seismic energy  
458 is lost due to coupling to the respective physical/chemical process. In single-phase materials, for example,  
459 processes may include the movement of twin walls in tetragonal  $\text{CaSiO}_3$  or a possible effect of low-spin/high-  
460 spin clustering in  $(\text{Mg,Fe})\text{O}$ <sup>119</sup>. In two-phase regions, associated with phase transitions, this may include  
461 element diffusion processes required to achieve thermodynamic equilibrium<sup>120-123</sup>. Thus, as well as mapping  
462 the first-order effects of elasticity on seismic wave speeds, future experiments should constrain the  
463 frequency- and time-dependence of processes in the mantle. Effort needs to be made to further develop  
464 methodologies to study seismic dispersion and attenuation at transition zone and lower mantle  
465 conditions<sup>122,124</sup>.

466  
467 In summary, it is clear that a significant gap remains between the *PT* conditions that can be routinely accessed  
468 in laboratory elasticity experiments and those that exist throughout the majority of our planet's deep interior  
469 (Fig. 4). However, as hinted above, progress is gathering pace; the pressure regime in the LVP is ever  
470 expanding, with conditions of over 100 GPa achieved using sintered diamond anvils<sup>92</sup>, whilst DAC-based  
471 resistive heating can now be performed up to  $\sim 1500$  K, and possibly higher, for several hours at a time<sup>125,126</sup>.  
472 The latter suffers from practical constraints of how to incorporate bulky vacuum systems or controlled  
473 atmosphere setups with light scattering, but is theoretically feasible. However, achievable temperatures are  
474 still far short of realistic mantle temperatures. Equally, 100 GPa in the LVP is unlikely to ever become  
475 "routine", especially in combination with pulse-echo ultrasonics, but the available pressure regime will  
476 continue to expand. Alternatively, as demonstrated up to 1300 K for single crystals of olivine,  $\text{CO}_2$  laser  
477 heating coupled with BS measurements<sup>4,68</sup> could provide a solution – but these measurements too are far

478 from straightforward. Polycrystalline BS experiments remain the most feasible<sup>6</sup>, but the reliability of these  
479 measurements remains unclear, as discussed above.

480

481 Whichever route(s) forward, it is clear that theoretical calculations will be an essential component of the  
482 attempts to understand the Earth's deep mantle. The challenge is for experiments to provide a sufficient  
483 dataset of measured seismic properties to allow careful and thorough validation of atomistic modelling  
484 results for each, and every, significant mineral phase. Only once these data exist will theoretical  
485 approximations for lower mantle phases be able to be "tuned", and lower the uncertainties on calculated  
486 properties to a level where they can be reliably used to interpret seismic properties. In the near future, we  
487 can also expect first seismic data of Mars' mantle to emerge from the data collected by the InSight space  
488 mission<sup>e.g.127</sup>. Experimental high-*PT* elasticity data will play a pivotal role in the interpretation of the seismic  
489 observations to come, but since the Martian mantle is believed to contain more iron than Earth's mantle,  
490 experimental studies need to broaden the compositional range of studied mantle minerals.

491

492 Finally, mineral physics predictions are still most often compared to one-dimensional, radial averaged,  
493 profiles of Earth's mantle, e.g. PREM<sup>128</sup> or AK135<sup>129</sup>. These comparisons are convenient and relatively simple  
494 to understand, providing a quick evaluation of the properties of any phase relative to a standard velocity  
495 profile. However, one-dimensional radial velocity models are averages of the entire range of mantle  
496 velocities, and do not represent the velocity of any real mantle portion. Seismic tomography clearly reveals  
497 that there are widespread and significant variations of wave speeds throughout the mantle. It is maybe time  
498 that we move away from attempts to compare mineral physics predictions to global 1-D average velocity  
499 profiles<sup>6</sup>. Interpretations of regional velocity models or global tomography with mineral physics models hold  
500 the promise of far more insight into the true nature of our planet's deep interior. No picture about the make-  
501 up of our planet's interior is as detailed as the one emerging from seismic techniques – a lot of information  
502 is encoded, we just need the data to decipher it.

**BOX 1. Seismic Body Waves and Relations to Mineral Physics:**

There are two types of seismic body waves travelling through the Earth. **Compressional** (or **primary**) waves travel via particle motion along the wave propagation direction, with velocity  $v_p$ . They arrive before **shear** (or **secondary**) waves which travel by a transverse particle motion, with velocity  $v_s$ . The propagation velocity of both compressional and shear waves can be related to the isotropic, i.e. direction- and polarization-independent, elastic moduli of the material they are passing through:

$$v_p^2 = \frac{K_s + \frac{4}{3}G}{\rho} \quad (\text{eq. 1})$$

$$v_s^2 = \frac{G}{\rho} \quad (\text{eq. 2})$$

Where  $\rho$ =density,  $K_s$ =adiabatic bulk modulus,  $G$ =shear modulus (also commonly denoted  $\mu$ ).

These two “real” velocities can be combined to define the bulk sound velocity,  $v_\phi$  or  $v_B$ :

$$v_\phi^2 = \frac{K_s}{\rho} \quad (\text{eq. 3})$$

Bulk sound velocity has been widely used because it can be determined from high- $P$  x-ray diffraction experiments as the isothermal bulk modulus ( $K_T$ ) of any material describes how its volume or density responds to a change of hydrostatic pressure at constant temperature within the elastic regime:

$$K_T = -V(\partial P / \partial V)_T \quad (\text{eq. 4})$$

The adiabatic and isothermal bulk moduli are related by the material’s thermal expansion ( $\alpha$ ) and a Grüneisen parameter ( $\gamma$ ):

$$K_S = (1 + \alpha_V \gamma T) K_T \quad (\text{eq. 5})$$

At room temperature, the difference between the isothermal and adiabatic bulk moduli is often about 1%, so many studies simply use  $K_T$  to constrain  $v_\phi$ , but this discrepancy significantly increases at mantle temperatures. Another complication of using the bulk sound velocity is that it is seismologically constrained by combining both P- and S-wave models (substitution of eq. 1 and 2 into 3), but these are usually based on different travel paths and/or periods.

$$v_\phi^2 = v_p^2 - \frac{4}{3}v_s^2 \quad (\text{eq. 6})$$

It is emphasised that the above relations only hold for perfectly elastic and isotropic materials where no seismic energy is lost through dissipation and seismic wave velocities are not dependent on their direction of travel. In such cases, bulk and shear moduli are substituted by the anisotropic elastic tensor, with the tensor elements referred to as elastic constants ( $C_{ij}$ ), Box 2.

**BOX 2. Anisotropy and averaging schemes**

All minerals, even those with cubic symmetry, are somewhat elastically anisotropic; different directions of the lattice change by different amounts under stress. The full response of any lattice to an external stress ( $\sigma$ ) or strain ( $\epsilon$ ) is described by its elastic tensor (in contracted notation)<sup>1</sup>:

$$\begin{pmatrix} \sigma_1 \\ \sigma_2 \\ \sigma_3 \\ \sigma_4 \\ \sigma_5 \\ \sigma_6 \end{pmatrix} = \begin{pmatrix} c_{11} & c_{12} & c_{13} & c_{14} & c_{15} & c_{16} \\ c_{21} & c_{22} & c_{23} & c_{24} & c_{25} & c_{26} \\ c_{31} & c_{32} & c_{33} & c_{34} & c_{35} & c_{36} \\ c_{41} & c_{42} & c_{43} & c_{44} & c_{45} & c_{46} \\ c_{51} & c_{52} & c_{53} & c_{54} & c_{55} & c_{56} \\ c_{61} & c_{62} & c_{63} & c_{64} & c_{65} & c_{66} \end{pmatrix} \begin{pmatrix} \epsilon_1 \\ \epsilon_2 \\ \epsilon_3 \\ \epsilon_4 \\ \epsilon_5 \\ \epsilon_6 \end{pmatrix} \quad (\text{eq. 7})$$

$\sigma_{1-3}$  and  $\epsilon_{1-3}$  are normal and  $\sigma_{4-6}$ , and  $\epsilon_{4-6}$  are shear stresses/strains respectively the  $c_{ij}$ 's are the mineral's elastic constants.

Since sound waves are actually stress waves, their apparent velocities will be different in different directions. Whilst the full elastic tensor consists of 36 independent elastic constants, this is reduced for many minerals as multiple elastic constants are constrained by symmetry considerations; e.g. cubic minerals have three unique elastic constants<sup>2</sup>:

$$\begin{pmatrix} \sigma_1 \\ \sigma_2 \\ \sigma_3 \\ \sigma_4 \\ \sigma_5 \\ \sigma_6 \end{pmatrix} = \begin{pmatrix} c_{11} & c_{12} & c_{12} & 0 & 0 & 0 \\ c_{12} & c_{11} & c_{12} & 0 & 0 & 0 \\ c_{12} & c_{12} & c_{11} & 0 & 0 & 0 \\ 0 & 0 & 0 & c_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & c_{44} & 0 \\ 0 & 0 & 0 & 0 & 0 & c_{44} \end{pmatrix} \begin{pmatrix} \epsilon_1 \\ \epsilon_2 \\ \epsilon_3 \\ \epsilon_4 \\ \epsilon_5 \\ \epsilon_6 \end{pmatrix} \quad (\text{eq. 8})$$

Thus, the number of  $c_{ij}$ 's requiring definition from single-crystal data is determined by crystal symmetry. Elastic constants are related to the isotropic bulk and shear moduli of a polycrystalline medium of the same mineral by averaging. Such averaging can be performed assuming one of the endmember cases of uniform stress ( $\sigma_1 = \sigma_2 = \sigma_3 = -\delta P$ ; the Reuss bound) or uniform strain ( $\epsilon_1 = \epsilon_2 = \epsilon_3 = -\delta P$ ; the Voigt bound). Whilst for most crystal symmetries the Reuss and Voigt bulk moduli ( $K_R$  and  $K_V$  respectively) are different, they are equal for cubic symmetry minerals:

$$K_R = K_V = \frac{1}{3}(c_{11} + 2c_{12}) \quad (\text{eq. 9 – for cubic minerals only})$$

The same procedure can be undertaken to derive averaged shear moduli, but even for cubic phases this produces two differing values:

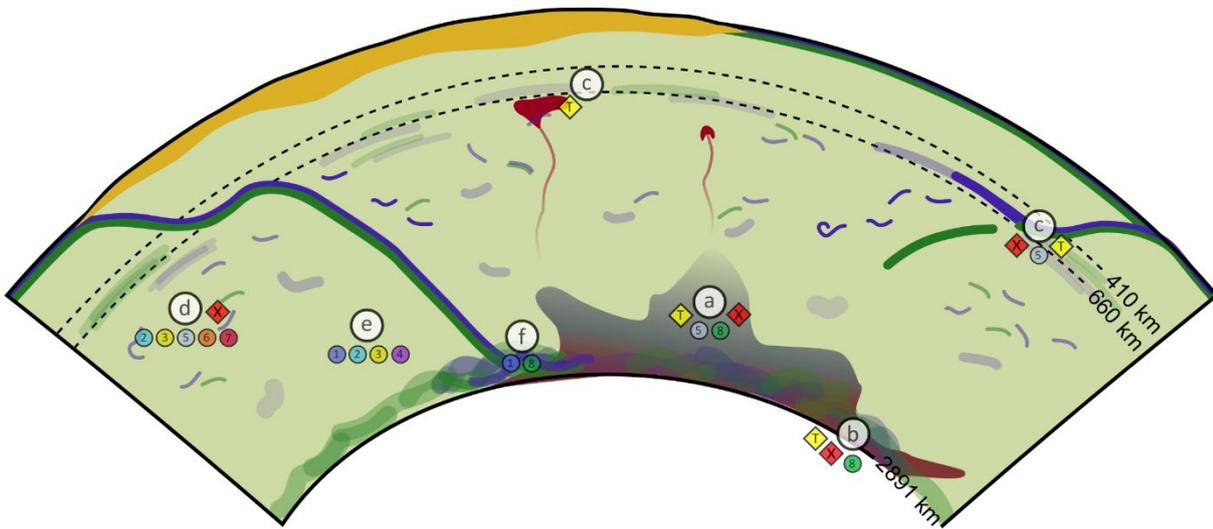
$$G_R = \frac{5(c_{11} - c_{12})c_{44}}{4c_{44} + 3(c_{11} - c_{12})} \quad (\text{eq. 10 – for cubic minerals only})$$

$$G_V = \frac{1}{5}(3c_{44} + c_{11} - c_{12}) \quad (\text{eq. 11 – for cubic minerals only})$$

Whilst the effective moduli must lie between these two bounds, where exactly is unknown. The most common approach when converting  $c_{ij}$ 's into bulk moduli are to use the Voigt-Reuss-Hill<sup>3</sup> average, defined in eq. 12 and 13.

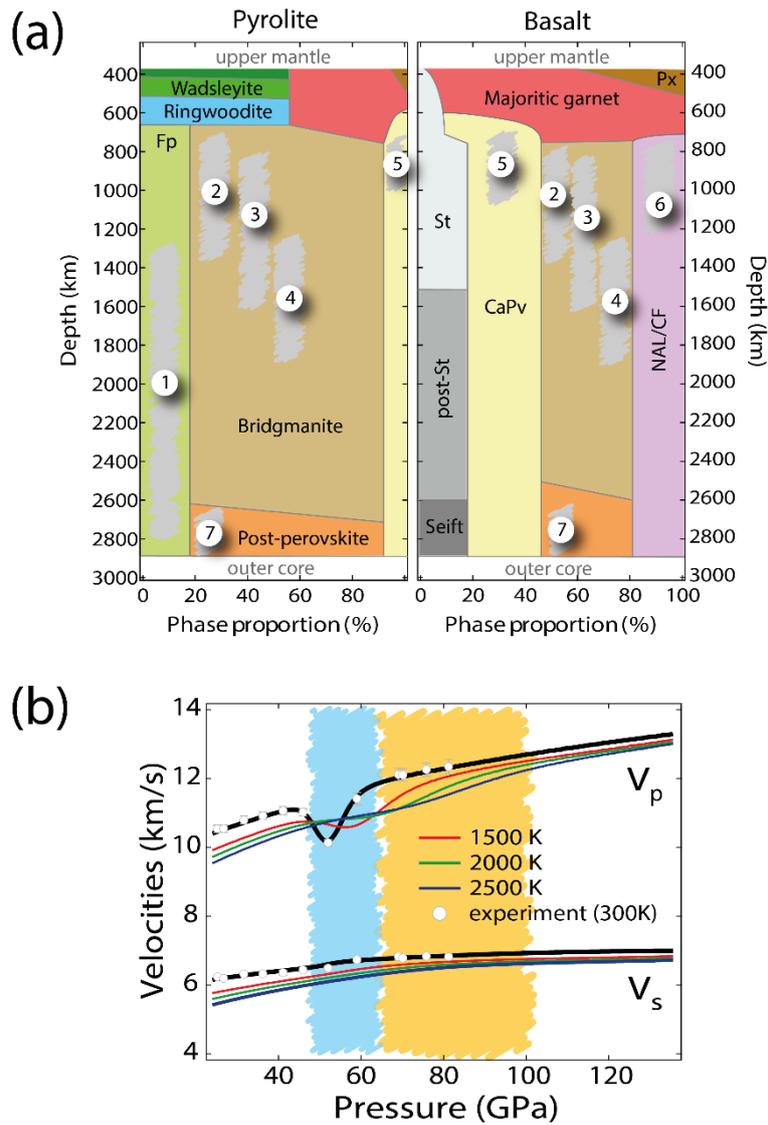
$$K_{VRH} = \frac{1}{2}(K_R + K_V) \text{ or } G_{VRH} = \frac{1}{2}(G_R + G_V) \quad (\text{eq. 12 and 13})$$

For phases with low anisotropy, such as majoritic garnet, the Voigt and the Reuss bounds are effectively indistinguishable. However, for highly anisotropic phases, such as ferropericlase at 80 GPa, they vary by ~ 7% for  $\nu_s$  and 3% for  $\nu_p$  (ref. <sup>16</sup>). Another commonly used averaging scheme is the Hashin-Shtrikman approach that results in tighter boundse.g. <sup>18</sup>.



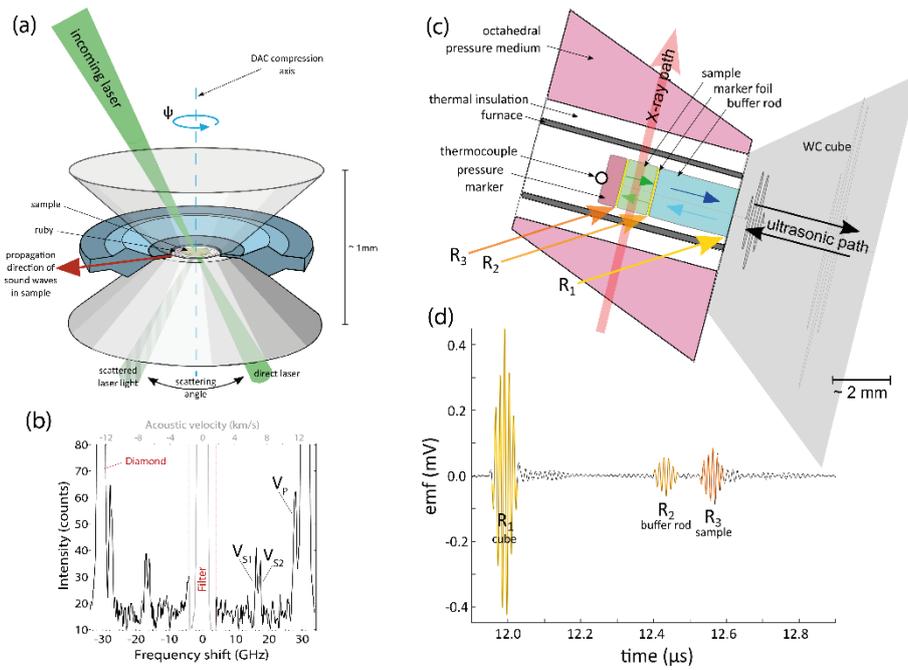
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**Fig. 1: Interpretive cartoon of Earth's deep mantle based on mineral physics interpretations of seismic observations.** Letters in white circles highlight example regions where specific seismic observations have previously been linked to changes in chemistry (X) or temperature (T). Numbers in coloured circles refer to mineralogical features that might provide alternative explanations for the observables, also depicted and labelled in Fig. 2: (a) The seismic signature of Large Low (Shear) Velocity Provinces (LL(S)VPs)<sup>130</sup> has been related to CaPv<sup>11</sup> (5), and post-perovskite<sup>131</sup> (8); (b) Ultra Low Velocity Zones (ULVZs) have been explained by iron-rich (Mg,Fe)O<sup>32,33</sup> or iron-rich post-perovskite<sup>36,103</sup> (8); (c) velocity variations in the transition zone/top of lower mantle have been linked to the low sound wave velocities of cubic CaPv<sup>7</sup> (5); (d) seismic reflectors in the mid-mantle might be related to the spin transition in the NAL-phase<sup>132</sup> (7), the displacive phase transition in stishovite<sup>74,133-135</sup> (6), or the cubic to tetragonal transition in CaSiO<sub>3</sub> perovskite<sup>11,136,137</sup> (5); (e) changes in the one-dimensional velocity structure of the lower mantle, as well as lateral seismic heterogeneity have been attributed to the spin transition in ferropericlase<sup>16,138,139</sup> (1), the spin transition in bridgmanite<sup>15,140</sup> (2), a change of the ferric iron content in bridgmanite<sup>5,141,142</sup> (3), or cation re-distribution within bridgmanite<sup>143</sup> (4); (f) seismic anisotropy in the D'' layer has been linked to post-perovskite<sup>144,145</sup> (8), or low-spin ferropericlase<sup>12,17,125</sup> (1).



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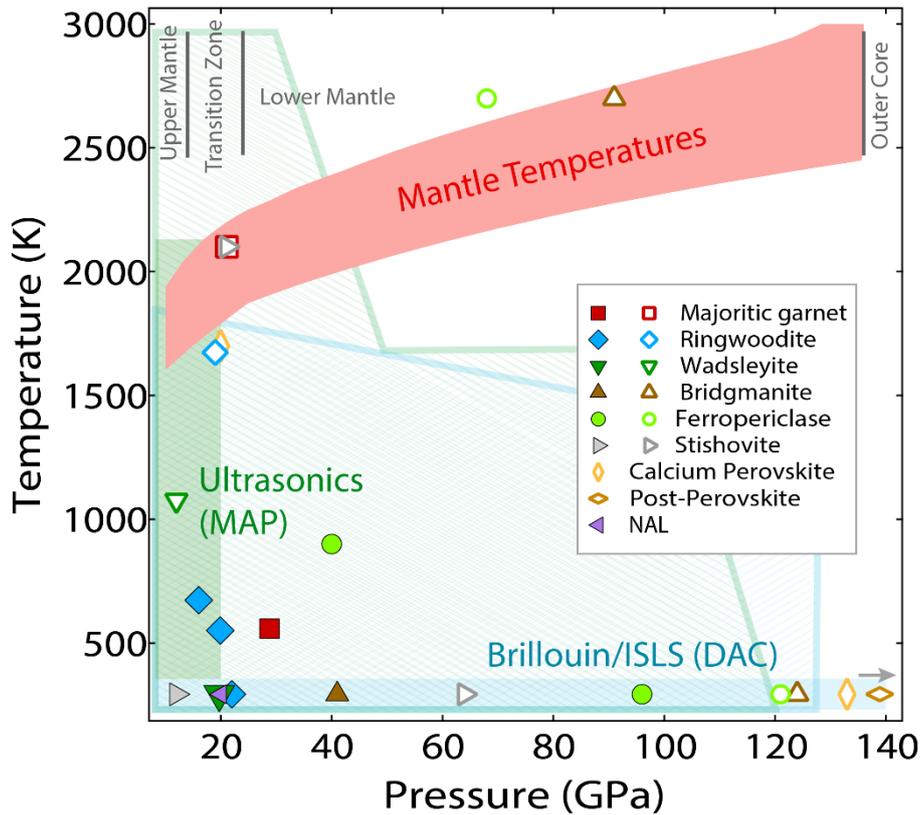
**Fig. 2: Mineralogy of the transition zone and lower mantle for pyrolitic and mid ocean ridge basalt (MORB) bulk compositions and processes that might affect seismic observables.** (a) Mineralogical features that have recently been identified in the lower mantle and have been proposed to explain some of the seismic observables depicted in Fig. 1 are shown: (1) Spin transition of  $\text{Fe}^{2+}$  in ferropericlase<sup>19,146</sup>; (2) Spin transition of B-site  $\text{Fe}^{3+}$  in bridgmanite<sup>15,146,147</sup>; (3) decrease of ferric iron content in bridgmanite<sup>5,141</sup>; (4) cation redistribution in bm<sup>140,142,143</sup>, (5) tetragonal distortion of calcium silicate perovskite<sup>7,11</sup>; (6) displacive phase change in stishovite; (7) Spin transition in the NAL-phase<sup>132</sup>; (8) post-perovskite transition<sup>148-150</sup>. The approximate depth range over which each process/feature extends is illustrated by the scribbled regions. Fp: Ferropericlase; St: Stishovite; Seift: Seifertite; Px: Pyroxene; NAL: New hexagonal Aluminous Phase; CF: Calcium-ferrite phase. (b) The effect of the iron spin transition in (Mg,Fe)O on seismic velocities. Note the marked drop of  $V_p$  at  $\sim 50$  GPa. Previous measurements of this effect are limited to room temperature (circles). Theoretical work predicts that the spin transition range broadens at lower mantle temperatures (coloured curves), but experimental confirmation is still outstanding. This effect could lead to a less pronounced softening of wave velocities that spreads out over a large pressure range. Note that the complex dependence on pressure and temperature can lead to a situation where an increase of temperature causes  $V_p$  to increase (blue scribbled region), an effect that is incompatible with the simple relations currently used to interpret seismic tomography models (slow = hot; fast = cold), as well as a broad region where  $V_p$  is more sensitive to temperature than  $V_s$  (yellow scribbled region). Figure modified from ref. <sup>16</sup>.



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552 **Fig. 3: Illustration of high-pressure elasticity experiments.** (a) Schematic of a DAC-based Brillouin scattering  
553 experiment, showing a magnified view of the sample chamber. The incoming laser beam is focused on a  
554 platelet sample in the pressure chamber. A small component of this laser light is scattered by the sample and  
555 collected at a defined scattering angle. This scattered light has exchanged energy with sound waves in the  
556 sample, which propagate in the direction marked with the red arrow. (b) The frequency content of the  
557 scattered light is plotted as intensity vs. frequency shift with respect to the incoming laser light. The spectrum  
558 is symmetric since energy can either be gained or lost during the inelastic interaction with sound waves. In  
559 the case of an elastically anisotropic single crystal, three contributions can be identified in the spectrum that  
560 arise from two shear waves ( $V_S$ ) and one compressional wave ( $V_P$ ) propagating in the direction that is probed.  
561 The strong contributions at high frequency shifts relate to the diamond-anvils that are trespassed by the laser  
562 beam. After a measurement is taken, which can takes several hours at high pressures, the DAC is rotated  
563 around the compression direction (blue line in (a)) by a few degrees and another Brillouin spectrum is  
564 measured. All velocities measured along different propagation direction, i.e. rotation angles, in the sample  
565 are inverted for the elastic constants by employing the Christoffel equation; linking phonon propagation  
566 directions, wave velocities, and elastic constants<sup>e.g.2</sup>. Spectrum reproduced from ref. <sup>5</sup>. (c) Schematic of a  
567 high-pressure multi-anvil press ultrasonic experiment, with components labelled. MHz frequency sound  
568 pulses are passed through one of the WC cubes into the sample via a buffer rod, with reflections returning  
569 to the transducer from interfaces  $R_1$  (cube - buffer rod),  $R_2$  (buffer rod - sample) and  $R_3$  (sample - backing  
570 medium). X-ray radiography measures the sample length  $L$  using marker foils at the top and bottom of the  
571 sample. (d) A typical ultrasonic spectrum collected in an experiment, with peaks in intensity resulting from  
572 echoes returning from interfaces  $R_1$ ,  $R_2$  and  $R_3$ . The sound wave velocity in the sample can be calculated from  
573  $2L/\Delta t$ , where  $\Delta t$  is the difference between the arrival time of  $R_2$  and  $R_3$ .

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579 **Fig. 4: *PT*-conditions covered by published light scattering elasticity measurements in the DAC and**  
 580 **ultrasonic experiments in the LVP as compared to the mantle geotherm.** The dark shaded regions illustrate  
 581 the conditions that are routinely achievable for elasticity measurements, whereas the symbols refer to the  
 582 maximum *PT*-conditions reached in elasticity experiments on specific mantle phases. Solid symbols denote  
 583 measurements on single crystals, and open symbols refer to polycrystalline measurements. Single-crystal  
 584 data are plotted for ringwoodite<sup>13,58,64</sup>, wadsleyite<sup>50,60</sup>, majorite<sup>55</sup>, ferropericlase<sup>53,65</sup>, bridgmanite<sup>5</sup>, NAL-  
 585 phase<sup>151</sup>. Polycrystalline data are for wadsleyite<sup>152</sup>, majoritic garnet<sup>153</sup>, ferropericlase (magnesiowüstite)<sup>6,32</sup>,  
 586 bridgmanite<sup>6</sup>, stishovite<sup>81,154</sup>, CaPv<sup>7,11,82</sup>, post-perovskite<sup>77</sup> (measured up to 172 GPa). The scribbled regions  
 587 shows the approximate range of *PT*-conditions that have been reached using the respective high-*PT*  
 588 apparatus, but where few or no elasticity measurements have been performed to-date. Green scribbled  
 589 region: Conditions achievable in the MAP<sup>92</sup>; blue scribbled region: Conditions achievable in the resistive-  
 590 heated DAC<sup>126</sup>. The laser-heated DAC is capable of reproducing the entire range of *PT*-conditions covered by  
 591 the figure.

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Table 1: Overview of techniques to measure high-*PT* elasticity of mantle minerals. BS: Brillouin Spectroscopy; XRD: X-ray diffraction, IXS: Inelastic x-ray scattering; PSA: picosecond acoustics; ISLS: Impulsive stimulated light scattering; NRIXS: Nuclear resonant inelastic x-ray scattering; US: ultrasonics; SC: single crystal; PC: polycrystal; wad: wadsleyite; rw: ringwoodite; mj: majorite; bm: bridgmanite; fp: ferropericlasite; st: stishovite; NAL; CF: calcium ferrite phase; ak: akimotoite.

|           | Method                  | General Principle   | Measured Quantity                       | Probing Frequency   | Derived Property      | Limitations   | Strength                                    | Time-requirement | Where       | Measured TZ/LM minerals (HP or HP/HT)  |
|-----------|-------------------------|---|---|---------------------|-----------------------|---|---|------------------|-------------|--|
| DAC-based | BS (SC)                 | Photon-phonon scattering                                      | Frequency shift of laser light          | 10 <sup>9</sup> Hz  | $V_p, V_s, C_{ij}$    | transparent sample, $V_p$ not detectable at HP                                    | High-quality data, elastic anisotropy       | very high        | laboratory  | Wad <sup>50,60-63</sup> , Rw <sup>57,58,64,155</sup> ; Mj <sup>54-56</sup> ; Bm <sup>5,9</sup> , MgO <sup>66,156,157</sup> , Fp <sup>16,17,53,59,65</sup> , (post-)st <sup>158</sup> , NAL/CF <sup>151</sup> |
|           | BS (PC)                 | Photon-phonon scattering                                      | Frequency shift of laser light          | 10 <sup>9</sup> Hz  | $V_p, V_s, K_s, G$    | same as above, sensitivity to grain size, CPO, laser coupling                     | In-situ synthesis of samples possible       | moderate         | laboratory  | Mj <sup>80</sup> , Bm <sup>5,15,78,79</sup> , Fp <sup>6</sup> ; CaPv <sup>82</sup> ; (post-)st <sup>81</sup> ; Ppv <sup>77</sup>   |
|           | XRD                     | elastic x-ray scattering                                      | lattice parameters, volume              | 0 Hz                | $K_T, \rho$           | no shear modulus  | Fast, HT routine                            | low              | synchrotron | All materials  |
|           | IXS (SC)                | Photon-phonon scattering                                      | points on phonon dispersion curve       | 10 <sup>12</sup> Hz | $V_p, V_s, C_{ij}$    | time-consuming  | opaque single-crystals can be measured      | high             | synchrotron | Fp <sup>12</sup>   |
|           | PSA (SC/PC)             | Optoacoustic generation/detection of acoustic strains         | Time-dependent surface reflectivity     | 10 <sup>9</sup> Hz  | $V_p, V_s, C_{ij}$    | Not yet routine   | Opaque sample, fast                         | low              | laboratory  | No mantle mineral. Metallic samples <sup>42,44</sup>   |
|           | ISLS (SC/PC)            | Photon-phonon inelastic scattering                            | Diffraction intensity in time domain    | 10 <sup>9</sup> Hz  | $V_p, V_s, C_{ij}$    | Limited information for $V_s$   | $V_p$ at HP                                 | moderate         | laboratory  | Bm <sup>9,15</sup> , Fp <sup>8,53</sup>  |
|           | NRIXS (SC/PC)           | Photon scattering from nuclear excitation of <sup>57</sup> Fe | Partial Phonon Density of States (PDOS) | range               | $V_{Debye}, V_p, V_s$ | Derivation of $V_p$ and $V_s$ requires equation of state, <sup>57</sup> Fe needed | Opaque samples can be measured              | moderate         | synchrotron | Fp/Mw <sup>32-35</sup> ; Ppv <sup>36</sup>   |
|           | GHz interferometry (SC) | Generation of sound wave by piezo-actuator                    | Travel time                             | 10 <sup>9</sup> Hz  | $V_p, V_s, C_{ij}$    | Limited to <10 GPa  | Opaque samples can be measured              | moderate (?)     | Laboratory  | Rw <sup>159</sup> , Fp <sup>39</sup>   |
| LVP-based | US (PC)                 | Generation of sound wave by piezo-actuator                    | Travel time, Sample length              | 10 <sup>6</sup> Hz  | $V_p, V_s, K_s, G$    | data sensitive to grain size, preferred orientation, limited P-range              | High-temperatures can be routinely achieved | medium           | synchrotron | Wad <sup>152,160,161</sup> , Rw <sup>14,162</sup> ; Mj <sup>163-165</sup> ; Bm <sup>85,166,167</sup> ; Fp <sup>168</sup> ; CaPv <sup>7,11</sup> ; St <sup>154</sup> , Ak <sup>169</sup>                      |
|           | XRD                     | elastic x-ray scattering                                      | lattice parameters, volume              | 0 Hz                | $K_T, \rho$           | no shear modulus  | Fast, HT routine                            | low              | synchrotron | Most materials   |

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981  
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