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A new high-pressure technique for the measurement of low frequency seismic attenuation using cyclic torsional loading

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ABSTRACT

We report a new technique for torsional testing of materials under giga-pascal pressures, which uses a shearing module in a large volume Paris-Edinburgh press in combination with high-resolution fast radiographic X-ray imaging. The measurement of the relative amplitude and phase lag between the cyclic displacement in the sample and a standard material (Al_2O_3) provides the effective shear modulus and attenuation factor for the sample. The system can operate in the 0.001 to 0.01 Hz frequency range, and up to 5 GPa and 2000 K; although high-temperature measurements may be affected by grain growth and plastic strain. Preliminary experimental results on San Carlos olivine are in quantitative agreement with previously reported Q^{-1} factors at lower pressure. This cyclic torsional loading method opens new directions to quantify the viscoelastic properties of minerals/rocks at seismic frequencies and in pressure-temperature conditions relevant to the Earth's mantle, for a better interpretation of seismological data.

INTRODUCTION

The Earth's materials are viscoelastic solids characterized by a continuous change from elastic behavior at high frequencies and/or low temperatures to viscous behavior at low frequencies and/or high temperatures (*e.g.* Jackson, 2008). This anelastic behavior of rocks is evidenced by seismic attenuation that manifests through the decrease in amplitude and energy of seismic waves as they propagate inside the Earth (*e.g.* Aki and Richards, 1980). This viscoelastic relaxation is responsible for dispersion, *i.e.* the frequency dependence, of seismic wave velocities. It is generally quantified in seismology by the quality factor Q (or its reciprocal

42 loss factor Q^{-1}) defined as $Q = 2\pi E/\Delta E$, where E is the energy of seismic wave and ΔE is the
43 energy change per cycle (Knopoff, 1964). The relaxation processes in rocks are controlled by
44 the temperature, composition, grain size, melt/volatile content, and a number of other physical
45 and chemical factors of the rocks (*e.g.* Karato and Spetzler, 1990; Cooper, 2002; Jackson,
46 2008). For this reason, seismic attenuation is a potential rich source of information about the
47 Earth's interior, complementary to seismic velocities. Over the last decade, seismologists have
48 made striking progress in mapping seismic attenuation, parametrized in terms of the quality
49 factor Q , either at the regional (Stachnik *et al.*, 2004; Pozgay *et al.*, 2009) or global scale (Gung
50 and Romanowicz, 2004; Adenis *et al.*, 2017). However, attempts to translate Q values into
51 meaningful quantitative physico-chemical parameters have been hampered by the lack of
52 experimental data on the anelasticity of rocks at the relevant high pressure-temperature (P-T)
53 conditions and low frequencies of teleseismic waves (mHz-Hz).

54 Laboratory measurements of attenuation Q^{-1} involves cyclic-loading methods that
55 measure the dynamic mechanical response of a material to an applied stress (*e.g.* Guéguen and
56 Palciauskas, 1994). These involve either axial or torsional forced-oscillation tests, where the
57 sample and an elastic standard of known modulus and negligible dissipation are connected in
58 series and subjected to a low-frequency sinusoidal time varying stress. For the elastic standard
59 the resulting strain is in phase with the applied stress with an amplitude inversely proportional
60 to its elastic modulus. Similarly, for the investigated sample, except that its viscoelastic
61 behaviour is manifest in a phase lag ($\Delta\delta$) of strain behind stress. Thus the measurement of the
62 relative amplitudes and phases of the cyclic displacements in standard and sample provides the
63 elastic modulus, and the strain energy dissipation ($Q^{-1} = \tan(\Delta\delta)$) respectively, for the sample.
64 The measurements must be performed in the linear domain (*i.e.* where the stress response is
65 linearly proportional to strain) so that the elastic modulus and internal friction Q^{-1} are
66 independent of strain amplitude. The upper limit of strain in the linear region depends on
67 materials, for example, 10^{-2} for alumina (Ota and Pezzotti, 1996). Therefore, for mm-size
68 sample, the displacement strain to be observed is usually of the order of few microns.

69 Torsion pendulum devices have been successfully applied at high-temperature and room
70 pressure to explore the anelastic properties of minerals (Guéguen *et al.*, 1981, Gribb and
71 Copper, 1998), and solid or partially molten rocks (Berckhemer *et al.* 1982, Bagdassarov and
72 Dingwell, 1993). However, these early measurements suffered from the creation of porosity
73 through thermal microcracking and the complex behavior of interfaces within the experimental
74 assembly. To overcome these limitations, Jackson and Paterson (1993) implemented the forced-
75 oscillation technique in an internally heated gas-charged pressure vessel. In this device, a pair
76 of electromagnetic drivers housed within the pressure vessel provides for the generation of the

77 torque, and the twist of the assembly is measured above and below the elastic standard by pairs
78 of parallel plate capacitance transducers. The frequency-dependence of the shear modulus and
79 dissipation has been determined in this device in a variety of polycrystalline materials like
80 olivine (Jackson *et al.*, 2002), MgO (Webb and Jackson, 2003) or titanate perovskites (Webb
81 *et al.*, 1999). However, the maximum accessible pressure within this device is 0.3 GPa, which
82 is a major limitation to the application of attenuation data to the Earth's mantle.

83 The effect of pressure on a general anelastic response is manifested by an activation
84 volume that might be significant at giga-pascal pressures. Furthermore, pressure commonly
85 induces structural changes in minerals which act to modify mechanical properties, both elastic
86 and anelastic. Such properties clearly need to be understood at the high P-T conditions of
87 stability of the phases involved. The challenge in applying the cyclic loading method in the
88 giga-pascal pressure domain is the need of a technique for measuring stress and strain with
89 sufficient precision, and with sufficiently short measurement time, while the sample is
90 embedded in a high-pressure vessel. Recently, several studies demonstrated these difficulties
91 can be overcome by the use of synchrotron radiography. Li and Weidner (2008) and Yoshino
92 *et al.* (2016) reported axial-forced oscillation measurements of Q^{-1} for San Carlos olivine
93 specimen up to 5 GPa and 2000 K in the D-DIA, a large-volume press with capabilities of
94 advancing / withdrawing the top and bottom rams. Change of strain and elastic properties with
95 time is directly observed by X-ray radiography at a specific oscillation frequency. Use of
96 synchrotron X-ray gives an opportunity to determine not only strain but also confining pressure
97 and phase identification. A limitation of this axial-loading method is that it provides the
98 Young's modulus (E). The shear modulus (G) must be converted from the measured Young's
99 modulus using a value for the bulk modulus (K) or the Poisson's ratio (ν) either assumed or
100 from literature data, and the following equations: $G = 3.K.E / (9K-E)$ or $G = E / 2.(1+\nu)$,
101 respectively.

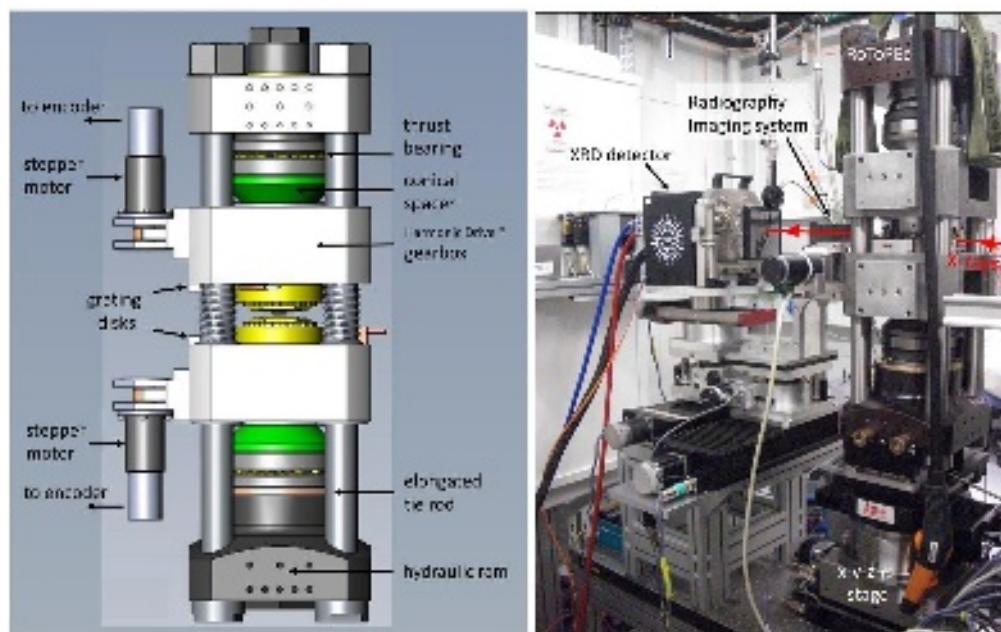
102 As an alternative, we developed a new high-pressure technique to directly measure the
103 shear modulus (G) dispersion and shear attenuation (Q_G^{-1}) by cyclic torsional loadings (Jackson,
104 1993). In this paper, we fully describe the experimental set-up that combines a Paris-Edinburgh
105 press with rotating anvils capabilities and synchrotron X-rays. We detail the technical and
106 analytical protocol to determine the anelastic properties from the X-ray monitoring of the
107 sample/standard strain, and discuss the attenuation factor resolution this method can resolve at
108 high P-T and low frequencies. To demonstrate the potential of this new technique, we present
109 a pilot study on strain energy dissipation in polycrystalline San Carlos olivine.

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112 EXPERIMENTAL METHODS

113 The forced-oscillation high P-T device

114 To perform the forced-oscillation mechanical testing under high P-T conditions we
 115 modified a Paris-Edinburgh cell, a large-volume press based on opposed anvil geometry
 116 (Besson *et al.*, 1992), to allow for the controlled rotation of the top or bottom anvil under load.
 117 Using an X-ray transparent gasket, this geometry offers a large window in the equatorial plane
 118 for the *in situ* radiography and diffraction measurements. A detailed mechanical description of
 119 this so-called RoToPEc device (for Rotational and Tomography Paris–Edinburgh cell) has been
 120 given by Philippe *et al.* (2016). A schematic drawing is shown in **Figure 1**. In brief, the two
 121 opposed anvils can rotate independently under load, with no limitation in the rotation angle,
 122 through two sets of gear reducers and thrust bearings located at the end of each anvil. The
 123 accurate rotation of the anvils is monitored by stepper motors and encoders, with an angular
 124 resolution of 0.02°. After pressurization, a sinusoidal stress and strain can be produced on the
 125 assembly by rotating the top anvil while the bottom one is held fixed. A Labview software
 126 allows to configure the desire sine wave profile by defining the rotation amplitude (in degrees),
 127 period (in seconds) and number of cycles. A frequency range from 0.001 to 0.01 Hz and a
 128 rotation amplitude from 1 to 5 degrees have been tested so far, and produced resolvable strain
 129 on the sample. This cyclic torsional loading system can operate up to 5 GPa and 2000 K for
 130 millimeter-size sample (using 10 mm boron-epoxy gasket with tungsten carbide anvils), the
 131 main limitation to operate at higher pressure being the reduction of the X-ray window due to
 132 the gasket flow under high P-T.



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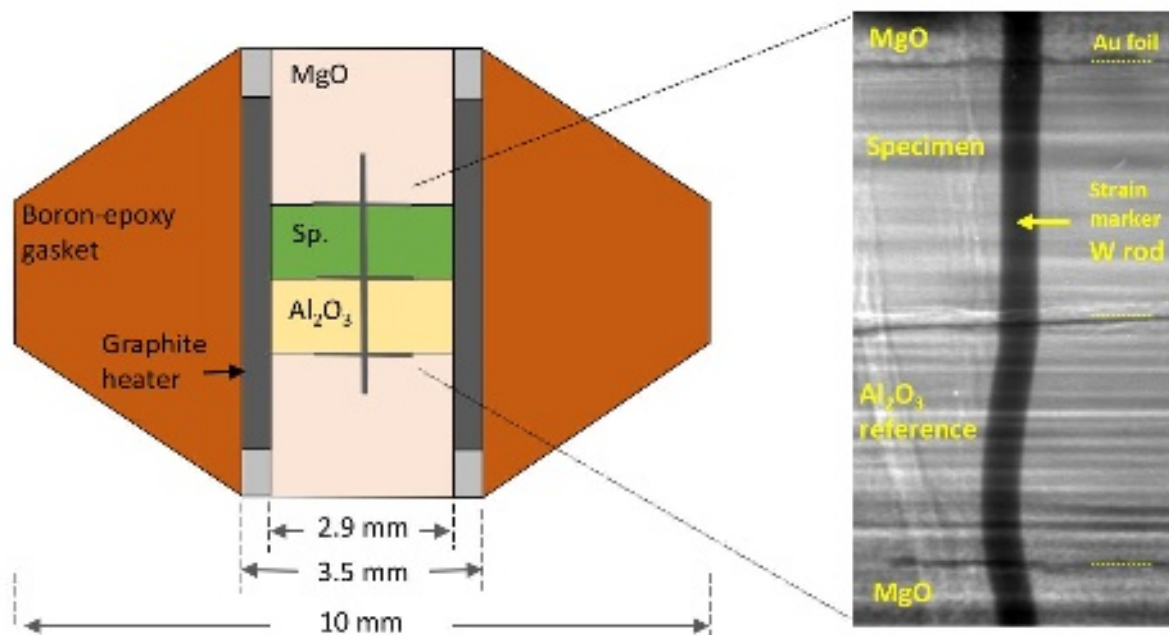
134 **Figure 1** – 3D view of the 450 ton V7 Paris-Edinburgh press with the RoToPEc module, and
 135 picture of its installation on beamline ID27 of the ESRF.

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Cell assembly

The configuration of the cell assembly used for the forced-oscillation measurements is shown in **Figure 2**. It consists of a biconical boron epoxy gasket of outer diameter of 10 mm and a cylindrical graphite furnace, both transparent to high-energy X-rays. To ensure a transmission of the torque and avoid slippage at interfaces, the sample and elastic standard are directly stacked in between two MgO rods. The sintered polycrystalline specimen and the elastic reference, a dense Al₂O₃ rod (Alfa Aesar™ Al23), are 0.5-0.8 mm in length and 2.9 mm in diameter, both prepared with flat faces polished to mirror quality to be perfectly parallel. The length of MgO rods is adjusted to place the sample and reference material in the center of the X-ray window. A tungsten rod ($\varnothing=90\ \mu\text{m}$) is positioned vertically along the outer diameter of the sample/Al₂O₃ for measuring the twist angle by X-ray absorption. The tungsten rod is actually inserted in a thin groove, made with a diamond file, along the sample/Al₂O₃ cylinder, to ensure a good mechanical coupling between the rod, the sample and the elastic standard. Gold wires ($\varnothing=25\ \mu\text{m}$) are also placed on the top and bottom sides of the sample and alumina reference to measure their lengths during the cycling torsional loadings. The pressure on the sample is derived from the MgO unit-cell volume measured using X-ray diffraction along with the thermoelastic data of Speziale *et al.* (2001). Temperature is estimated by a power-temperature relation curve determined in a previous experiment using a thermocouple and identical cell configuration.



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Figure 2 – (left) Cell assembly used for the forced-oscillation measurements, and (right) representative X-ray radiograph through the sample and alumina standard. Highly absorbing features are the gold and tungsten strain markers.

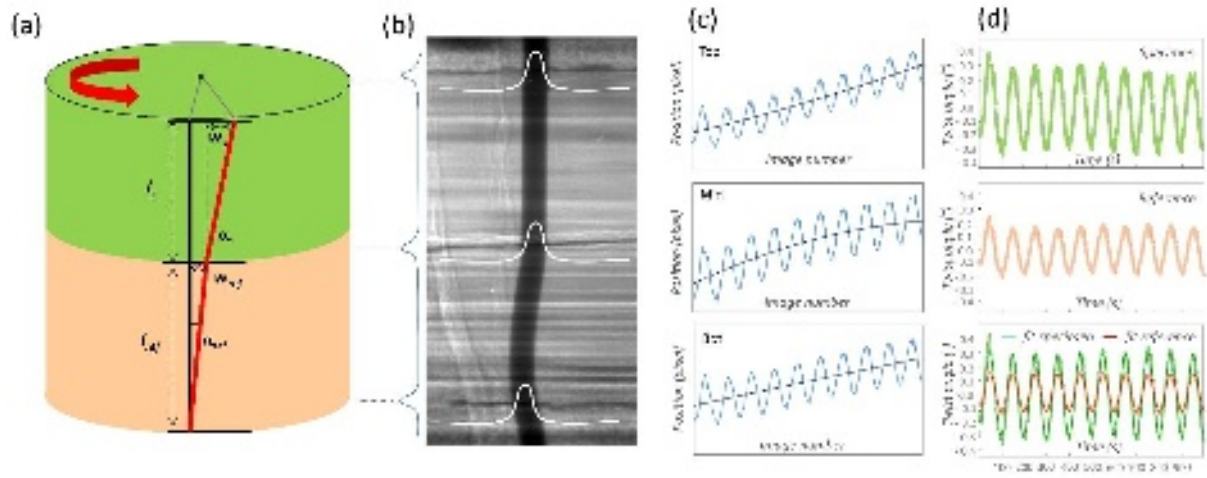
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In situ strain measurements

The force-oscillation technique relies on the time-resolved measurement of the twist angles of the sample and elastic reference in response to the sinusoidal imposed torque. The attenuation factor (Q_G^{-1}) of the sample is determined from the phase lag of strain relative to the standard, while the strain amplitude ratio provides a comparative determination of the effective shear modulus (G). A difficulty of the technique is that, in order to be independent of strain amplitude (*i.e.* in the linear viscoelastic domain), extremely small strain variations as 10^{-3} to 10^{-6} depending on the investigated material need to be detected. The use of synchrotron X-ray radiography gives the opportunity to monitor such small displacements, with high precision (micron to sub-micron) and at short periods, by following the motions of strain markers.

In practice, a large X-ray beam illuminates the cell assembly through the transparent gasket. The absorption contrast between the assembly parts, sample, Al_2O_3 reference, and strain markers is converted into visible light by a phosphorous YAG single-crystal set in the transmitted beam on the downstream side of the press. A 45° mirror reflects the visible contrast into a high-speed CCD camera, and the image is focused by an objective lens (**Figure 1**). In this study, measurements have been obtained during two sessions performed at beamline ID27 of ESRF (Mezouar *et al.*, 2005) and beamline PSICHE of Synchrotron SOLEIL (King *et al.*, 2016). At ID27, the monochromatic 33 keV beam has a field of view of 2.0 and 2.7 mm in the vertical and horizontal directions, respectively. The CCD camera is a CoolSNAP HQ2 (Photometrics) which allows the acquisition of 745 x 413 pixel images at time intervals down to 100 ms. The magnification of the imaging system leads to a pixel size of 2.2 μm . At PSICHE, a pink beam with an average energy of 45 keV is collimated to 2.5 mm and 4.5 mm in the vertical and horizontal directions, respectively. The objective lens of a Hamamatsu ORCA Flash4.0 sCMOS camera gives an effective pixel size of 2.45 μm . For a region of interest of 1500 x 900 pixels the acquisition time can be achieved 10 ms.

The experimental protocol is first to cold compress the cell to the target pressure. Due to compaction of the assembly parts, the strain markers may then appear deformed or tilted on the X-ray image, but without effect on the further strain monitoring (**Figure 2**). Afterward, temperature is raised by steps, and oscillations are applied according to the sine wave profile configuration. The usual settings for torsional loadings are 1 to 5 degrees for the top anvil rotation, over 100 to 1000 s sine wave period, and between 5 to 20 successive cycles. Meanwhile, radiographic images are recorded at high frequencies, producing an image sequence of thousands of frames that traces the motion of the sample/alumina strain markers as a function of time.



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 198 **Figure 3** – Principle of the forced-oscillation technique to measure strain energy dissipation.
 199 (a) Schematic drawing of the motion of the strain markers during oscillation, and (b)
 200 corresponding X-ray radiograph from which the position of the gold foils and tungsten rod are
 201 monitored over time. (c) Position of the W rod marker as a function of time (i.e. image number)
 202 at the sample and alumina interfaces, and (d) estimated twist angle in degrees for the alumina
 203 reference and sample. Represented data are from an olivine specimen and alumina reference at
 204 0.9 GPa and 300 K (see below).
 205

206 Estimation of anelastic properties

207 The image sequence is analyzed with an automated ImageJ routine that outputs the
 208 sinusoidal curves of the twist angle of the sample (α_s) and reference (α_{ref}) as a function of time,
 209 during the cyclic loading. The image processing scheme is depicted in **Figure 3**. It first includes,
 210 for each frame, the determination of the horizontal offset of the tungsten rod in the sample (w_s)
 211 and reference (w_{ref}) obtained by fitting a Gaussian function along the absorption profile. The
 212 round shape and the diameter of 90 μm of the tungsten generate a strong absorption signal on
 213 the X-ray images which helps this profile fitting. The offset defines sine waves of few pixels
 214 amplitude superimposed to a non-linear baseline that is further subtracted by a second order
 215 polynomial fit. This non-linear motion comes from a translation of the whole assembly relative
 216 to the X-ray beam as a result of gasket extrusion. The lengths of the sample (l_s) and reference
 217 (l_{ref}) as defined by the location of the gold wires markers are also measured. Actually, we did
 218 not observed any resolvable change in lengths whatever the rotation angle of the anvils,
 219 meaning that the applied torque generates only simple shear. The twist angles of the sample and
 220 reference are then calculated according to $\alpha_s = \text{atan}(w_s/l_s)$, and $\alpha_{ref} = \text{atan}(w_{ref}/l_{ref})$, respectively.
 221 These two curves are finally fitted to a sinusoidal equation $\alpha_{s(ref)} = A_{s(ref)} \sin(\omega t + \delta_{s(ref)})$ where
 222 A is the amplitude, t is time, and δ is a phase angle. The shear attenuation factor (Q_G^{-1}) is
 223 determined from the phase angle lag $\Delta\delta_{s-ref}$ between the sample and reference according to Q_G^{-1}
 224 $= \tan(\Delta\delta)$. The shear modulus is obtained from the A_{ref}/A_s amplitude ratio and the elastic
 225 modulus of the reference material.

226 **RESULTS AND DISCUSSION**227 **Calibration of the forced-oscillation apparatus**

228 In order to check the integrity of the present forced oscillation technique, it is necessary
229 to investigate any contamination of the relative amplitudes and phases of the twist angle
230 sinusoids. Potential sources of errors include (i) any mechanical interaction between the
231 specimen/standard set and the boron-epoxy pressure medium, (ii) sliding along the MgO rods,
232 specimen and standard interfaces, or (iii) some image artefacts affecting the location of strain
233 markers. We therefore conducted initial tests at the ESRF synchrotron in which the specimen
234 was replaced by a cylinder of Al₂O₃ of the same composition and dimensions as the one used
235 for the elastic standard. We carried out two sets of measurements under 3.1 GPa pressure, at
236 ambient temperature and 800°C, respectively. For each P-T point, the mechanical testing was
237 performed at conditions of 2 degrees rotation angle - 200 s period, and 1 degree rotation angle
238 – 100 s period.

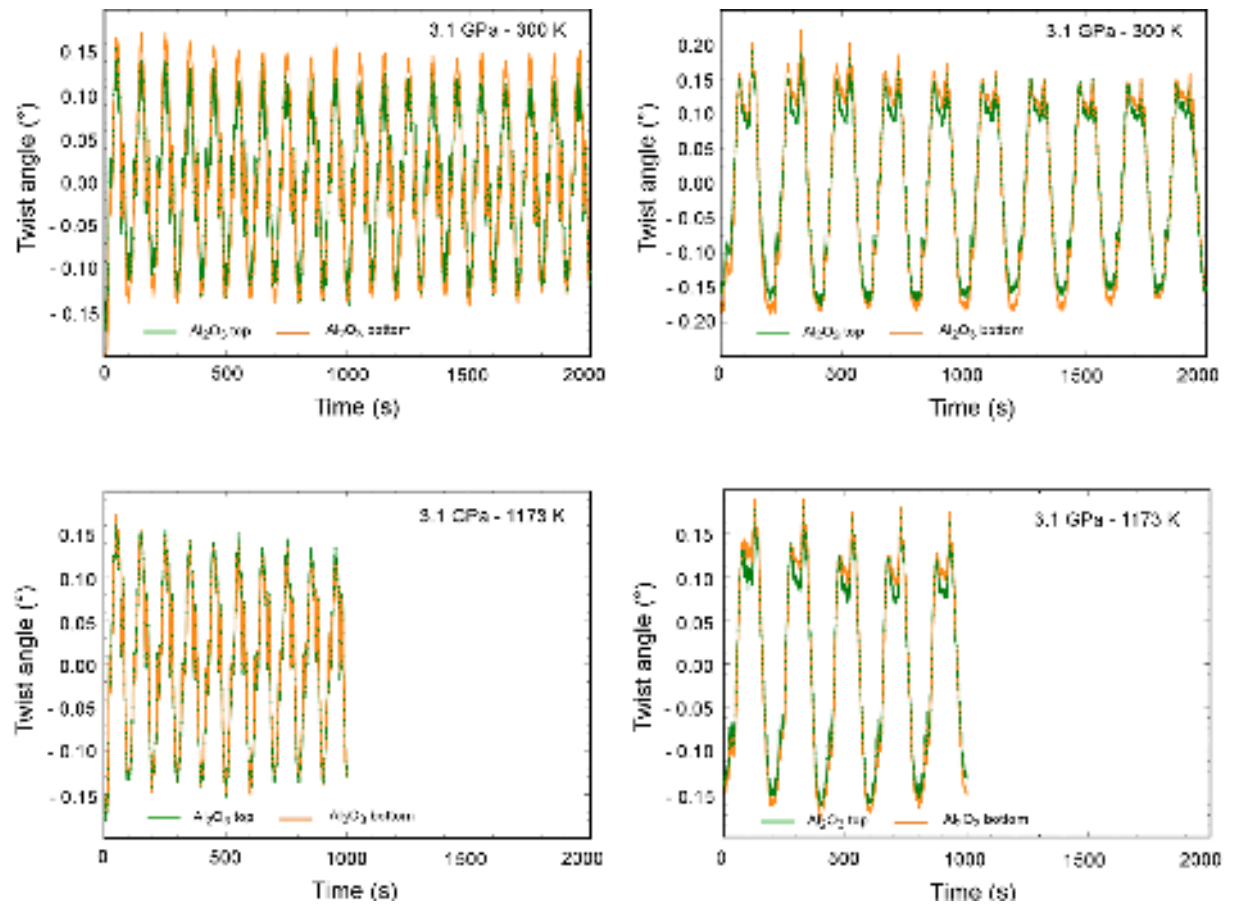
239 The twist angle versus time curves are shown in **Figure 4**. The values of the twist angles
240 for the top and bottom Al₂O₃ rods correspond to a $\sim 10^{-3}$ magnitude of strain. These twist angles
241 are consistent with the expected amount for the 2 degrees torsion of a 6.5 mm long assembly
242 (*i.e.* the length of the sample plus reference and MgO rods); meaning that the rotation of the
243 anvils is transmitted to the sample assembly with no loss at the anvil-gasket interface. For some
244 runs, the position of both the top, middle and lower interfaces exhibit some non-sinusoidal
245 spikes (see **Figure 3c**) that translate into similar artefacts on the twist angle curves (**Figure 4**).
246 These features are reproducible over cycles which seems to rule out an origin from sliding on
247 an interface. Instead, they appear to be somehow associated with reversing direction of rotation,
248 being more pronounced when increasing load, when the rotation angle is larger or when the
249 oscillating period is longer, so that we suspect some slack in the gearbox to be their cause.
250 Being reproducible, these parasitic motions do not significantly affect the sine wave fitting of
251 the twist angle curves, and hence the determination of the Q_G^{-1} and G parameters. In all runs,
252 the top and bottom Al₂O₃ sinusoids are in phase with amplitude ratio close to one, providing
253 compelling evidence that there is no compliance associated with the interfaces, nor any
254 significant effect of the pressure medium and/or graphite furnace. These findings validate the
255 synchrotron imaging method to quantify oscillation strain and the image processing scheme to
256 retrieve the mechanical properties.

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262 **Figure 4** – Twist angle vs. time curves for Al₂O₃-Al₂O₃ test experiments at 3.1 GPa and 300
263 K-1173 K, and 100 - 200 s oscillation periods, respectively

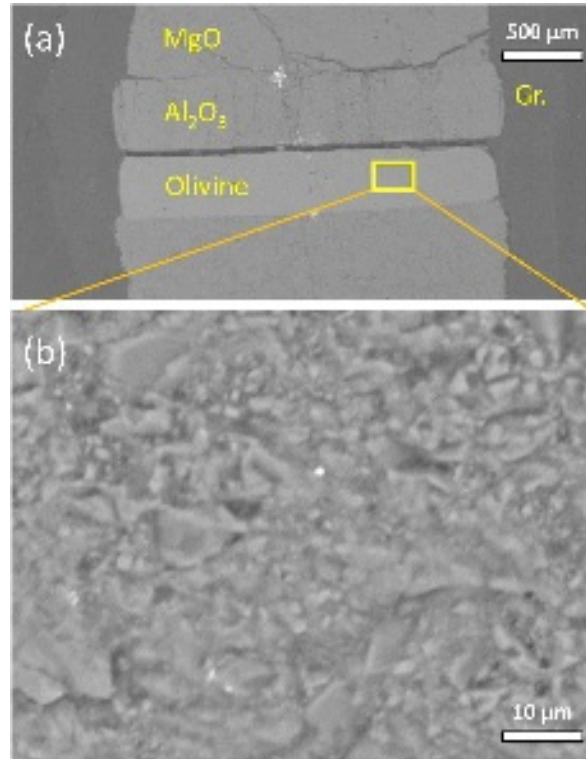
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265 **Preliminary measurements on San Carlos olivine aggregates**

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267 To demonstrate the capabilities of the present forced-oscillation technique, we carried
268 out experiments on San Carlos polycrystalline olivine. This phase is one of the most abundant
269 minerals in the mantle, so that the knowledge of its elastic and anelastic properties are critical
270 parameters to understand and model the Earth's interior. Several previous studies have
271 investigated the viscoelastic behavior of olivine both under ambient (Guéguen *et al.*, 1989;
272 Gribb and Copper, 1998) or high pressures (Jackson *et al.*, 2002; Li and Weidner, 2007;
273 Yoshino *et al.*, 2016) to compare with our preliminary measurements. The starting material
274 consisted of natural San Carlos (Fe_{0.1}Mg_{0.9})₂SiO₄ crystals, powdered and hot-pressed at 4 GPa
275 – 1000°C for 24 hours to achieve a fine 1-10 μm grain size and a low porosity. Cylindrical
276 samples, respectively 0.48 mm in length and 2.9 mm in diameter were loaded in series with the
277 Al₂O₃ (Alfa Aesar™ -Al23) reference. In a first loading, the cell assembly was cold compressed
278 and then heated by steps at 1020 K, 1380 K and 1490 K for the mechanical tests. In a second
279 loading, pressure was first increase to 1.6 GPa at 300 K, with subsequent heating at 1130 K,
1430 K and 1560 K. For all P-T points, the forced-oscillation measurements were performed at

280 conditions of 2 degrees rotation angle – 200 s period, and X-ray images were recorded at 1 Hz.
 281 A SEM image of the recovered sample is shown in **Figure 5**.

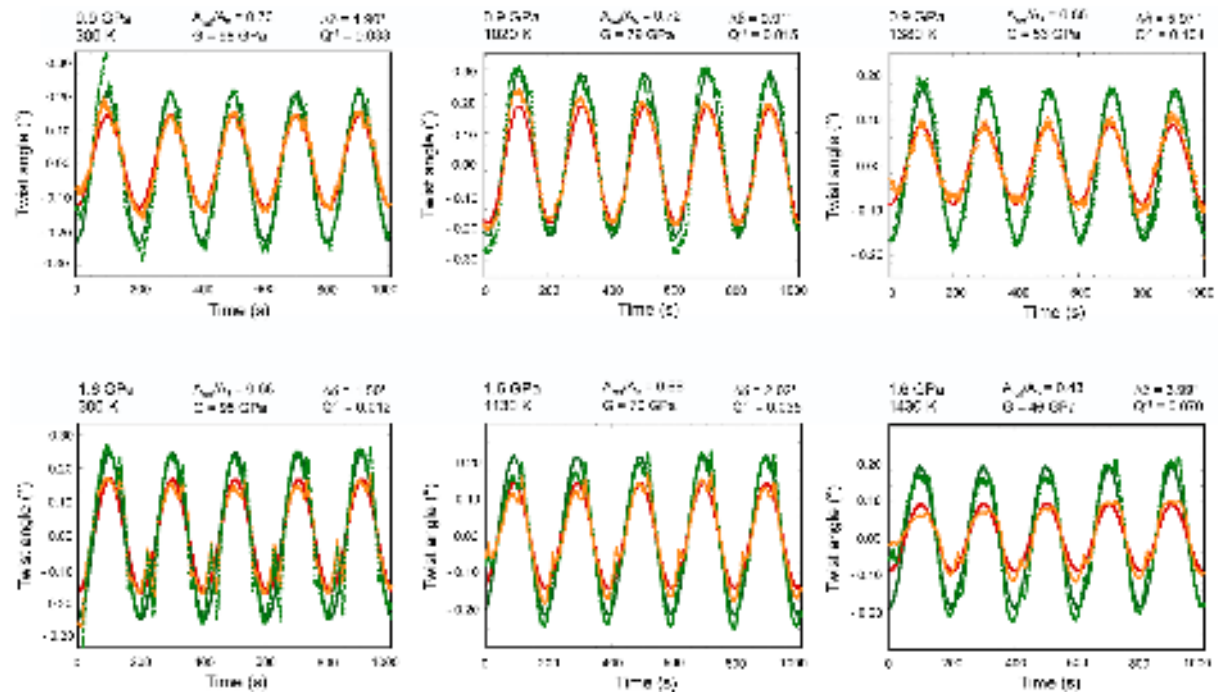


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 283 **Figure 5** – Electron backscattered images of the sample charge after experiment showing (a)
 284 the assembly parts: MgO rod, Al₂O₃ reference, Olivine specimen, Gr. Graphite furnace; and (b)
 285 the microstructure of the olivine specimen with 1 – 10 μm grain sizes.
 286

287 **Figure 6** displays the twist angle vs. time curves for each P-T point, and the fits to
 288 sinusoids for the olivine and alumina reference. **Figure 7** shows an enlarged view of these
 289 curves for the 0.9 GPa – 1380 K data, as well as the corresponding plot for the specimen twist
 290 angle against the alumina reference. In such a graph, the deviation from linearity, *i.e.* the
 291 appearance of an elliptic shape, is a manifestation of anelasticity, and the surface of this loop
 292 represents the amount of energy dissipated (*e.g.* Tisato and Madonna, 2012). The twist angle
 293 vs. time curves show the presence of a visible phase lag, and hence attenuation, for temperatures
 294 above about 1300 K, along with a marked decrease of the shear modulus. This dispersion of the
 295 modulus results from the increase of shear attenuation with temperature.

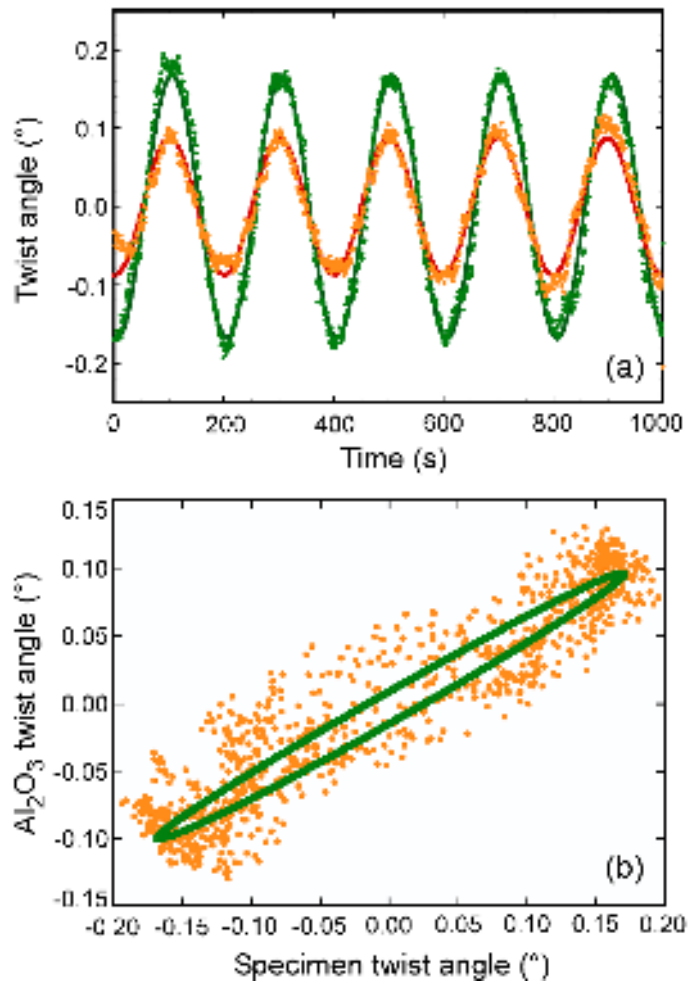
296 The attenuation factors (Q_G^{-1}) obtained at 0.9 GPa – 1380 K and 1.6 GPa – 1430 K fall
 297 within the range of previous estimates for olivine at ambient and high pressures (**Figure 8**)
 298 using either axial or torsional loading techniques (Gribb and Copper, 1998; Tan *et al.*, 2001;
 299 Jackson *et al.*, 2002; Yoshino *et al.*, 2016). The temperature dependence of attenuation for the
 300 present 0.005 Hz measurements is in broad agreement with literature data obtained at 0.01 Hz
 301 on polycrystalline olivine; although a more precise comparison would require to investigate in
 302 details the grain size and frequency dependence of the attenuation. We could not determine the

303 attenuation for temperature beyond 1430 K since the strain curves did not follow anymore the
 304 sinusoidal torsion imposed by the anvils. This may point to the onset of plastic strain in the
 305 sample or alumina, or any sliding at interfaces. Grain growth is also efficient at high
 306 temperature and may affect the attenuation factor for samples where the strain energy
 307 dissipation is controlled by grain-boundary sliding processes.



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Figure 6 – Twist angle vs. time curves for the San Carlos olivine sample (green dots) and Al_2O_3 reference (orange dots) at specified PT conditions. The forced-oscillation were performed at conditions of 2 degrees rotation angle – 200 s period. Attenuation factors (Q^{-1}) and shear moduli (G) are determined from the phase lag and amplitude ratio estimated from the sinusoid fits to strain curves (dark green and red lines for the olivine sample and alumina reference fits, respectively).



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318 **Figure 7** – (a) Detailed view of the twist angle vs. time curve for the olivine-Al₂O₃ reference
319 at conditions of 0.9 GPa – 1380 K and sinusoidal loading with period of 200 s; and (b) the
320 corresponding hysteresis loop. The elliptic shape is a manifestation of anelasticity, and results
321 in a Q^{-1} factor of 0.104.
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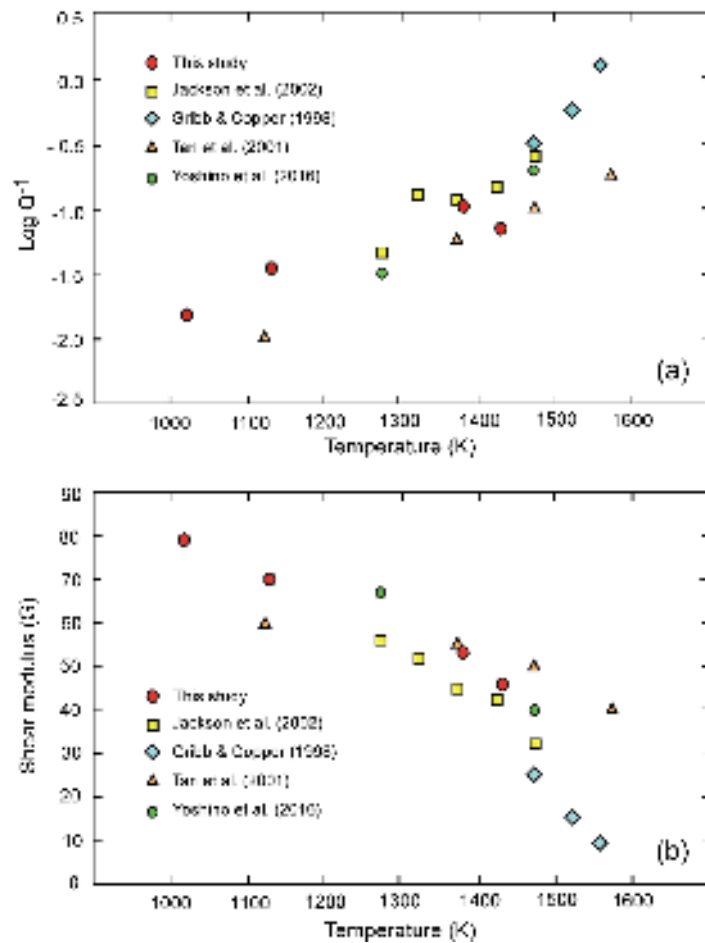
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These preliminary data demonstrate that forced-oscillations in the RoToPEc device combined with synchrotron X-ray imaging is an effective method to study the anelastic properties of materials under extreme conditions, and quantify the effect of temperature, pressure, frequency and grain size on energy dissipation. To date, this method can operate up to 5 GPa within a frequency range from 0.001 to 0.1 Hz. The magnitude of strain achieved is about 10^{-3} while the upper limit of strain to stay in the linear regime domain may be as low as 10^{-4} for some minerals (Guéguen *et al.*, 1989). Further developments to achieve 10^{-4} strain will involve the increase of sample length, the reduction of rotation angle and improvement of X-ray radiography resolution. The use of sintered diamond anvils and/or toroidal gaskets (Kono *et al.*, 2015) will also enlarge the X-ray window available for CT imaging in order to expand the technique to higher pressure.

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337 **Figure 8** – (a) Attenuation factor ($\log Q_G^{-1}$) vs. temperature and (b) shear modulus (G) vs.
 338 temperature for olivine aggregates at frequency of 10 - 5 mHz and pressures between 1 atm to
 339 1.6 GPa, as determined by Gribb & Cooper (1998) for 3 μm grain-size Balsam Gap olivine,
 340 Tan et al. (2001) for 30 μm grain-size San Carlos olivine, Jackson et al. (2002) for 3-12 μm
 341 grain-size Fo90 olivine, Yoshino et al. (2016) for 3 μm grain size San Carlos olivine, and the
 342 present study for 1-10 μm grain-size San Carlos olivine
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344 DATA AVAILABILITY

345 The data that support the findings of this study are available from the corresponding author
 346 upon reasonable request.
 347

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FIGURES CAPTIONS

Figure 1 – 3D view of the 450 ton V7 Paris-Edinburgh press with the RoToPEc module, and picture of its installation on beamline ID27 of the ESRF.

Figure 2 – (left) Cell assembly used for the forced-oscillation measurements, and (right) representative X-ray radiograph through the sample and alumina standard. Highly absorbing features are the gold and tungsten strain markers.

Figure 3 – Principle of the forced-oscillation technique to measure strain energy dissipation. (a) Schematic drawing of the motion of the strain markers during oscillation, and (b) corresponding X-ray radiograph from which the position of the gold foils and tungsten rod are monitored over time. (c) Position of the W rod marker as a function of time (i.e. image number) at the sample and alumina interfaces, and (d) estimated twist angle in degrees for the alumina reference and sample. Represented data are from an olivine specimen and alumina reference at 0.9 GPa and 300 K (see below).

Figure 4 – Twist angle vs. time curves for Al_2O_3 - Al_2O_3 test experiments at 3.1 GPa and 300 K-1173 K, and 100 - 200 s oscillation periods, respectively.

Figure 5 – Electron backscattered images of the sample charge after experiment showing (a) the assembly parts: MgO rod, Al_2O_3 reference, Olivine specimen, Gr. Graphite furnace; and (b) the microstructure of the olivine specimen with 1 – 10 μm grain sizes.

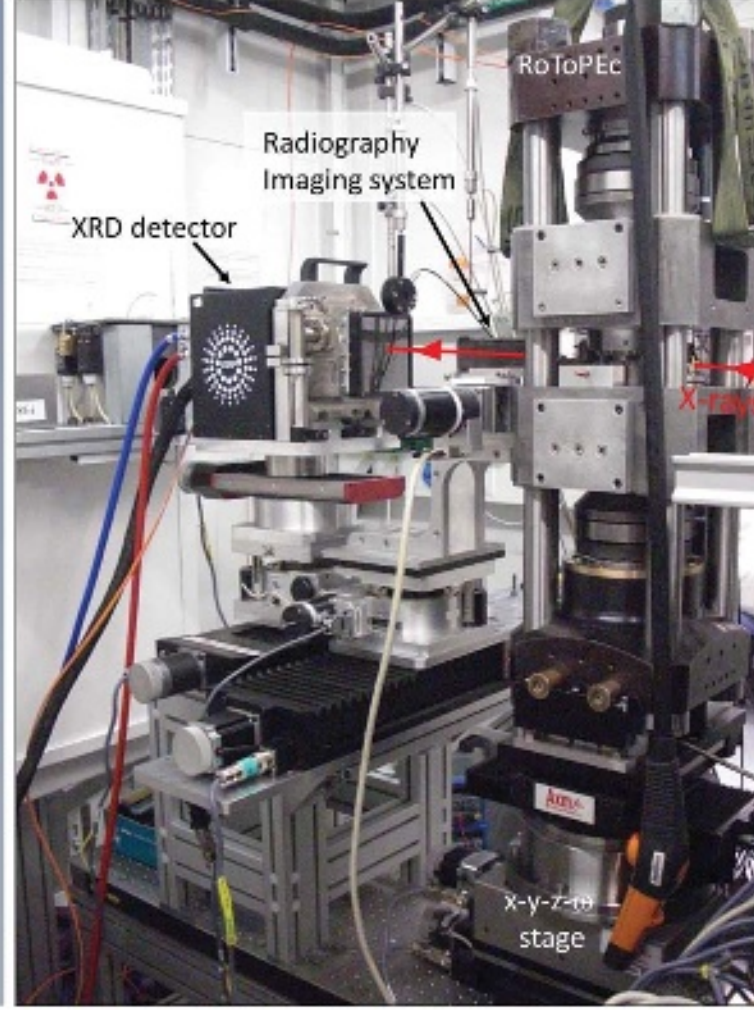
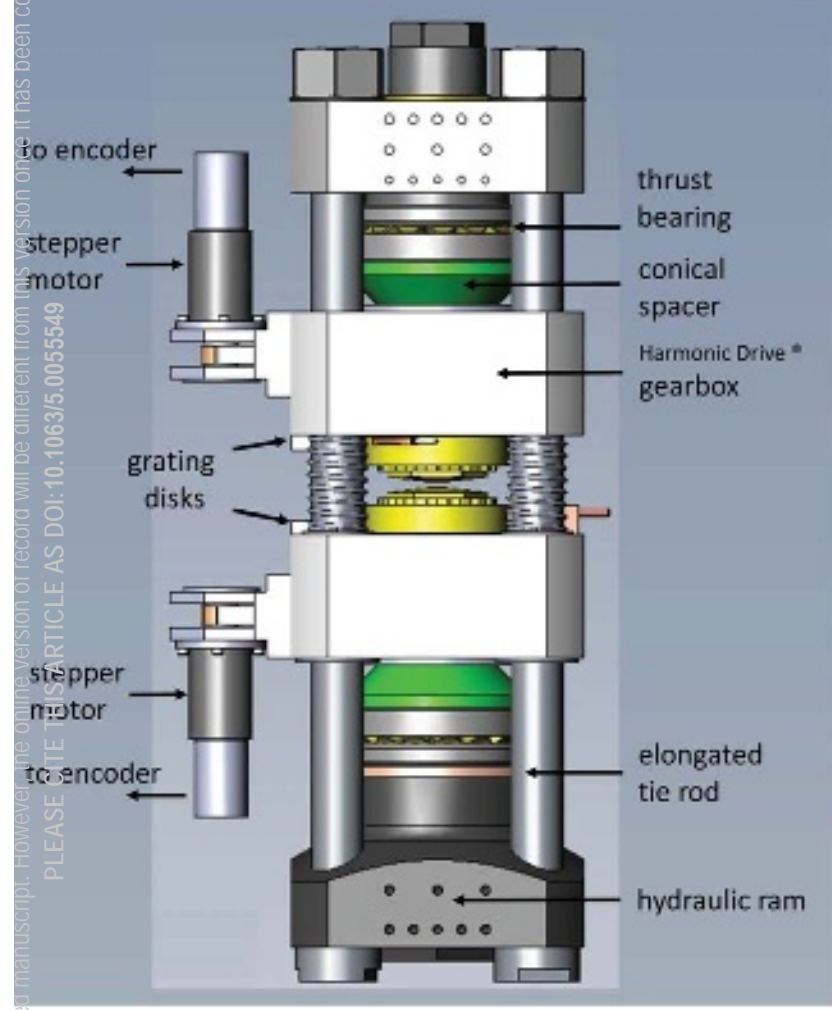
Figure 6 – Twist angle vs. time curves for the San Carlos olivine sample (green dots) and Al_2O_3 reference (orange dots) at specified PT conditions. The forced-oscillation were performed at conditions of 2 degrees rotation angle – 200 s period. Attenuation factors (Q^{-1}) and shear moduli (G) are determined from the phase lag and amplitude ratio estimated from the sinusoid fits to strain curves (dark green and red lines for the olivine sample and alumina reference fits, respectively).

Figure 7 – (a) Detailed view of the twist angle vs. time curve for the olivine- Al_2O_3 reference at conditions of 0.9 GPa – 1380 K and sinusoidal loading with period of 200 s; and (b) the corresponding hysteresis loop. The elliptic shape is a manifestation of anelasticity, and results in a Q^{-1} factor of 0.104.

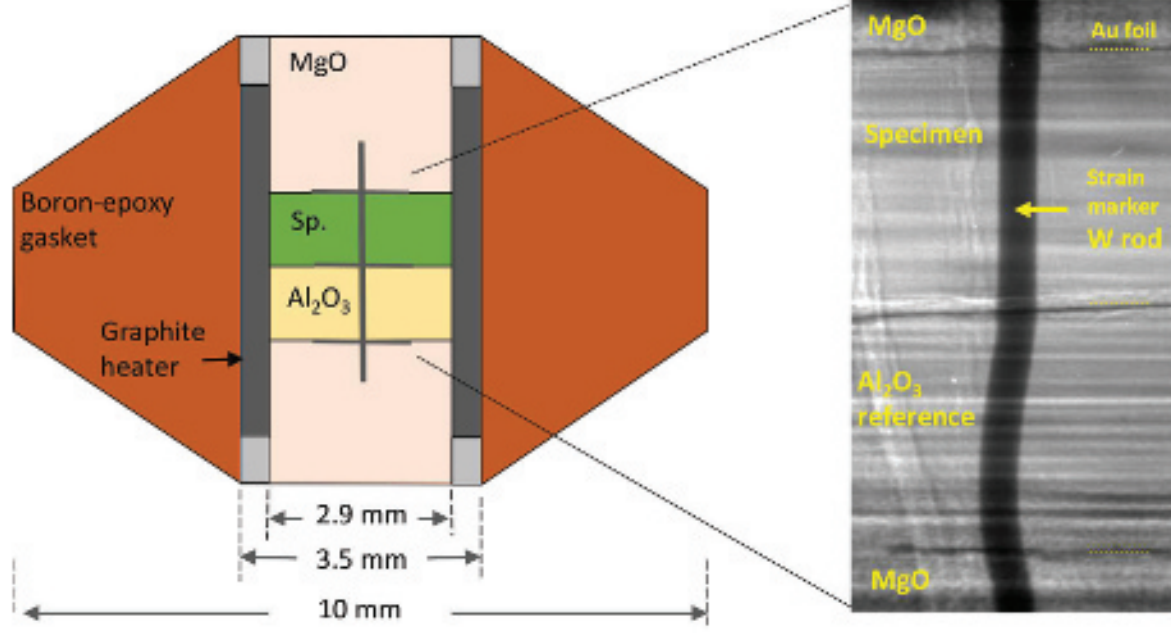
Figure 8 – (a) Attenuation factor ($\log Q_G^{-1}$) vs. temperature and (b) shear modulus (G) vs. temperature for olivine aggregates at frequency of 10 - 5 mHz and pressures between 1 atm to 1.6 GPa, as determined by Gribb & Copper (1998) for 3 μm grain-size Balsam Gap olivine, Tan et al. (2001) for 30 μm grain-size San Carlos olivine, Jackson et al. (2002) for 3-12 μm grain-size Fo90 olivine, Yoshino et al. (2016) for 3 μm grain size San Carlos olivine, and the present study for 1-10 μm grain-size San Carlos olivine.

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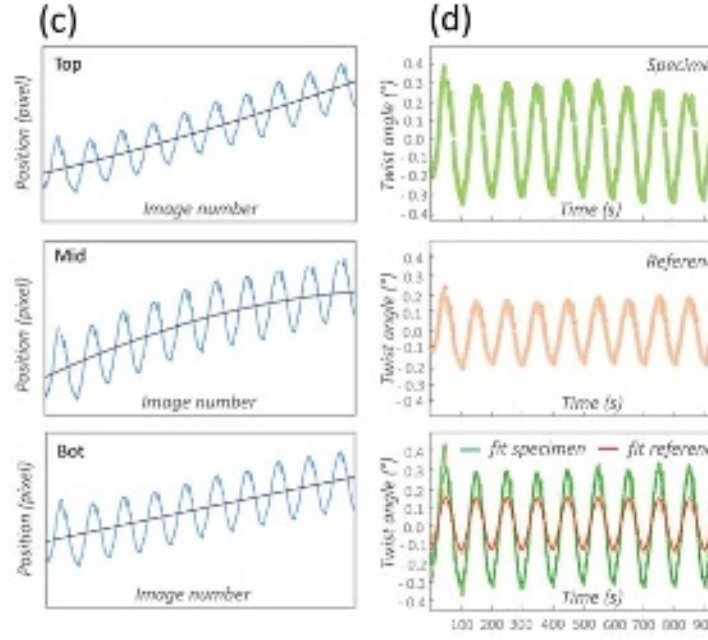
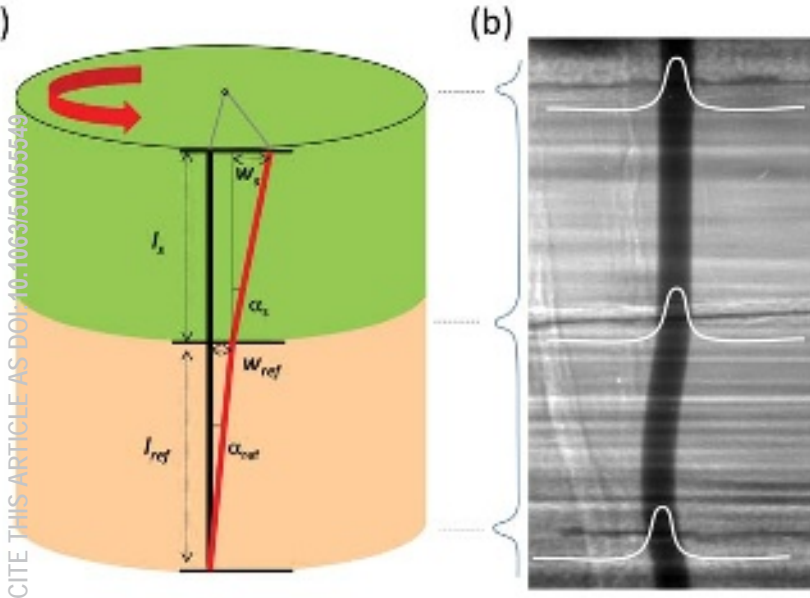
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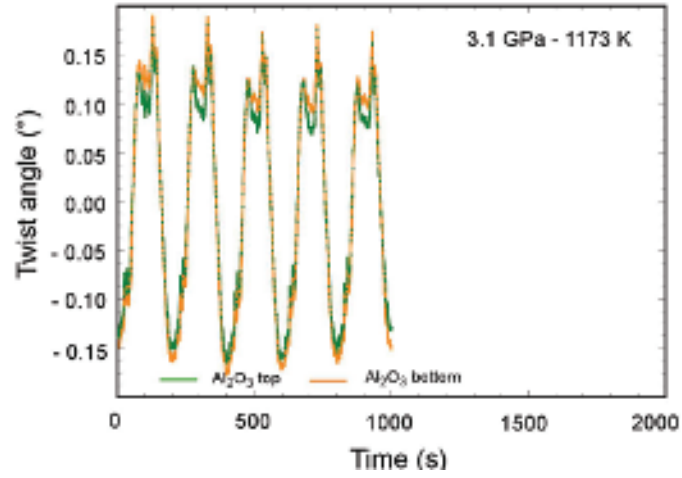
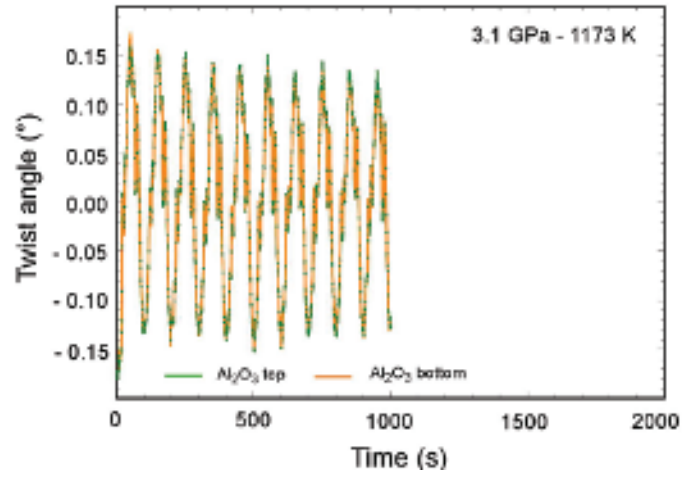
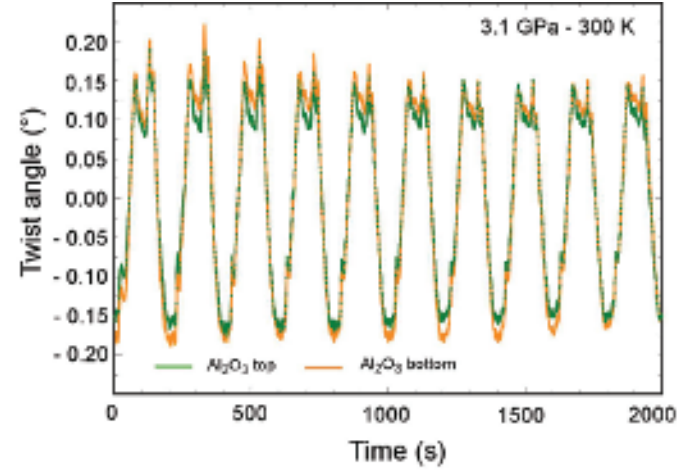
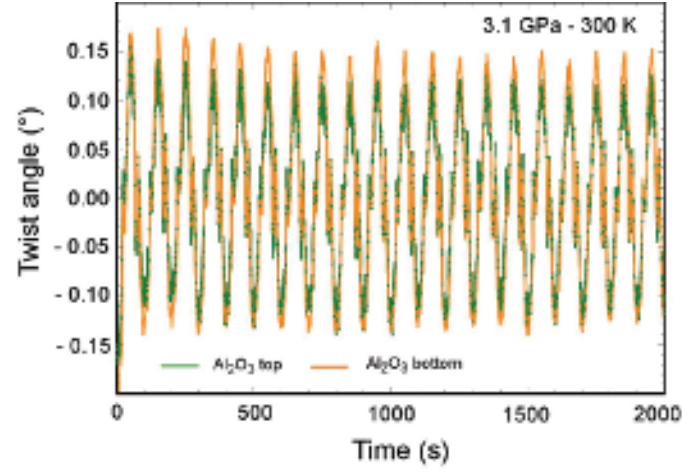
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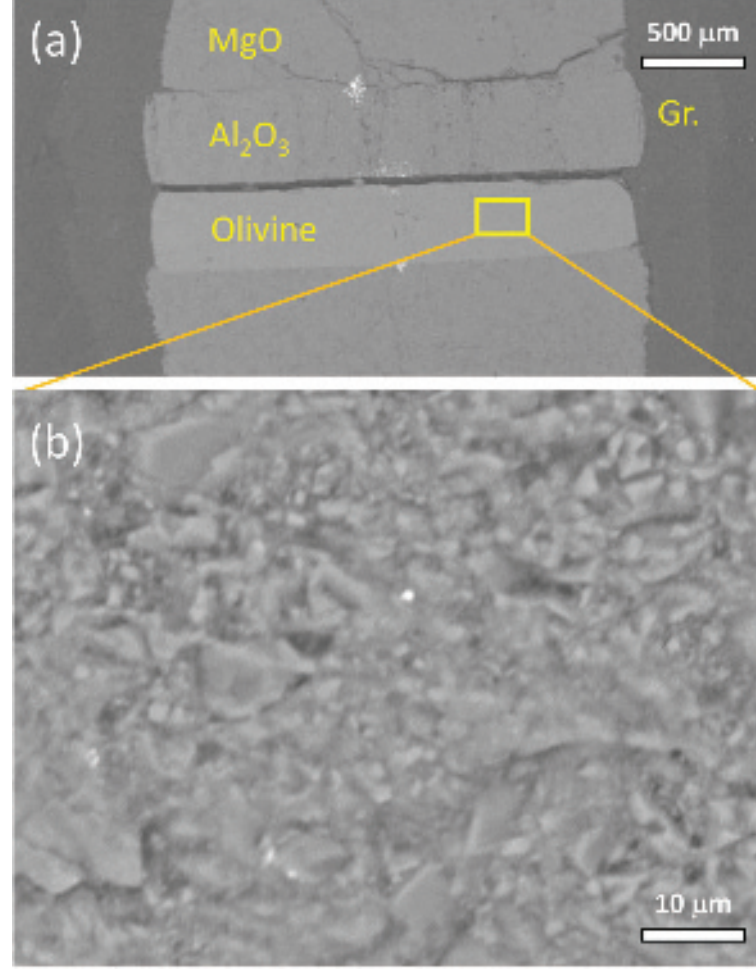


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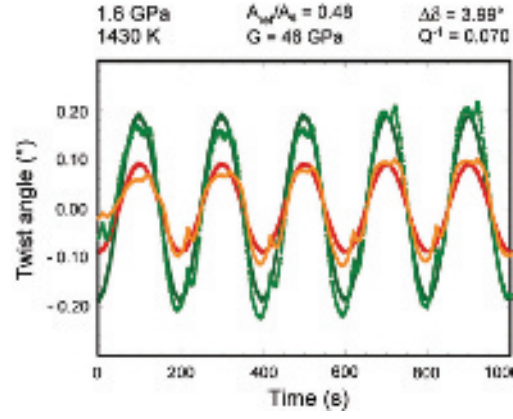
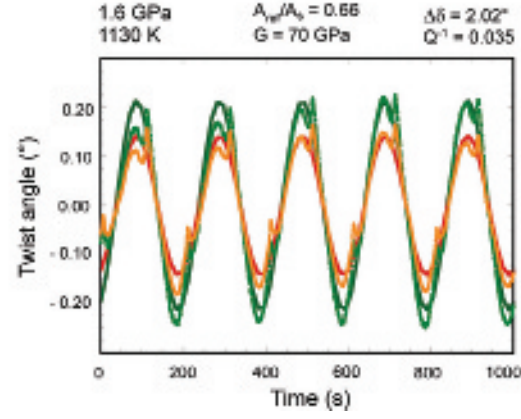
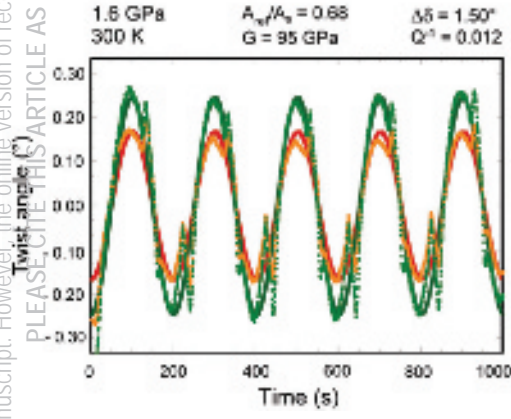
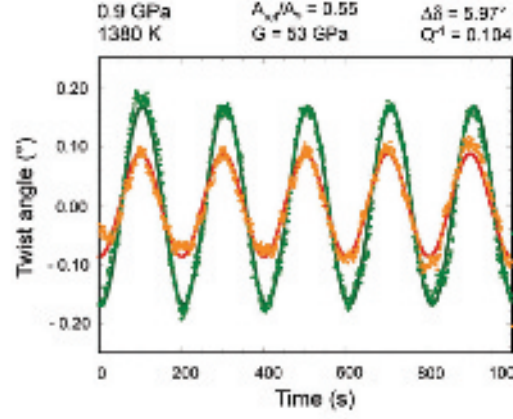
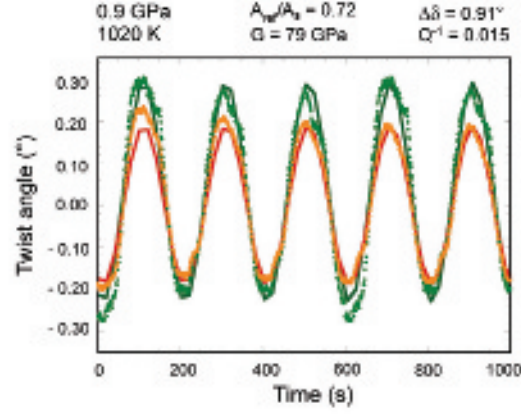
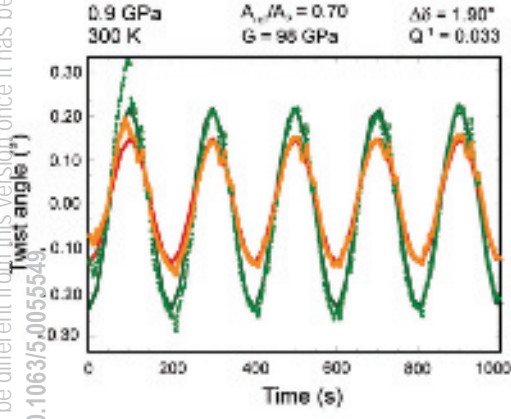
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