Supplementary Materials for

Dislocation Damping and Anisotropic Seismic Wave Attenuation in Earth's Upper Mantle

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Materials and Methods

Preparation

Dense polycrystalline olivine specimens of Fo90 composition were fabricated from a synthetic precursor, prepared by solution-gelation (sol-gel) methods (29,30). Pelletised sol-gel powders were fired at 1400°C under controlled oxygen fugacity conditions (ca. 5 \times 10^{-4} \text{ Pa for } \frac{\text{pCO}}{\text{pCO}_2} = 1) for 16 hours and subsequently hot-pressed at 1300°C and 300 MPa Ar confining pressure for 24 hours. After hot-pressing, the resulting dense olivine polycrystals (e.g. H6585 in Table 1) were precisely ground to a typical length of 30 mm and 11.5 mm in diameter.

Deformation in compression and torsion

Selected specimens (D6618, D6646, T0436, see Table 1) were deformed in either triaxial compression or torsion to increase the dislocation density further. All experiments were carried out with the olivine specimen surrounded by Ni\textsubscript{0.7}Fe\textsubscript{0.3} foil in the stability field of olivine at high temperature. Compression experiments at 1250°C were conducted at ANU using a Paterson-type gas apparatus with a differential stress just below the gas confining pressure (300 MPa) to avoid brittle behaviour. Under these conditions, dislocation creep was expected even for a specimen of 3 µm grain size (31). The maximum strain rate achieved was about 6 \times 10^{-5} \text{ s}^{-1} with a maximum flow stress of 290 MPa. The specimens were deformed by 2% (D6618) and 22% (D6646) total strain introducing a moderate and high dislocation density respectively.
A torsional deformation experiment (T0436) was carried out at the University of Minnesota. The twist rate, rather than the internal torque, was controlled during the experiment at 1300°C and 300 MPa Ar gas confining pressure. The specimen was sandwiched between porous alumina discs in order to minimise slip at the alumina – specimen interfaces. The specimen was successfully deformed to a maximum shear strain of $\gamma = 0.8$ at a maximum shear stress of 155 MPa. The maximum shear strain rate was $2.6 \times 10^{-5}$ s$^{-1}$. Note that 'maximum' here corresponds to deformation at the cylindrical surface of the specimen. The dislocation density adjacent to this outer surface was $2.4 \mu$m$^2$, and up to a factor 2 lower radially inwards.

**Torsional forced-oscillation testing**

Micro-creep forced-oscillation tests were carried out on the hot-pressed and pre-deformed sol-gel specimens in a compound pressure vessel capable of holding 200 MPa of argon gas pressure. Many aspects of the attenuation apparatus and its operation have been described previously (20,32). A summary is given here regarding the current standardised mode of operation and any specifics pertaining to current experiments. The cylindrical specimens were sandwiched between two tapered Lucalox™ torsion rods. Each assembly was wrapped in a mild steel jacket, flared at the bottom to mate with the experimental assembly inside the apparatus. The lower half of the assembly is composed primarily of a steel elastic standard of known elastic modulus and negligible internal friction and connects to three horizontal lever arms. Two lever arms extend to four sensitive capacitance displacement transducers and the bottom lever arm connects to two opposing electromagnetic drivers that generate an oscillating torque. The upper pair of
transducers measure the displacement caused by the twisting of the specimen assembly \(d_1(t)\) and the lower pair of transducers measure the difference in displacement between the upper and lower pairs of transducers \((d_{12}(t) = d_2(t) - d_1(t))\) reflecting the twist of the elastic standard. During experimentation, these displacements were recorded and converted to frequency domain via a Fourier transform function to determine the relative amplitudes and phase of \(d_1(t)\) and \(d_{12}(t)\).

Once a specimen was loaded into the compound pressure vessel, a series of routine steps were carried out in preparation for experimentation. These steps include first time pressurization to 150 MPa to cause jacket collapse of the top half of the assembly onto the fixed lower half. The entire assembly was then raised at room pressure by 1.5 mm to account for thermal expansion during heating. Each set of outer transducer plates at every station were aligned relative to the central fixed plate and offset to account for predictable displacements at high pressure and temperature. Ideal alignment under experimental conditions ensures optimal sensitivity for torsion and discrimination against flexure. Each experimental run at the highest desirable temperature conditions and 200 MPa confining pressure was preceded by roughly 50 hours of annealing whilst forced-oscillation tests were run to monitor mechanical stability of the specimen and assembly.

Once repeated exploratory forced-oscillation tests indicated stable mechanical behaviour, extensive forced-oscillation tests were started for periods between 1.28 s and 999.68 s (chosen to avoid mains-frequency aliasing effects). The voltage-time signals from the displacement transducers were converted into actual displacements with calibration
factors interpolated between calibrations conducted before and after each phase of forced-oscillation testing. Temperature was progressively lowered at 25°C, 50°C and finally 200°C increments whilst forced-oscillation tests were carried out at each temperature. Reproducibility of data was confirmed upon re-heating to the highest temperature in that experiment for a final forced-oscillation test. The raw displacement-time data was recalculated in terms of compliance and phase lag of the assembly. Further data processing progressively removed all factors contributing to dynamic compliance and phase lag from the twisting column until at last only the properties of the specimen remained. These were expressed as dissipation (Q⁻¹) and shear modulus dispersion (G).

The data can be fitted by a constitutive viscoelastic equation such as the Andrade creep function (12) or the Burgers creep function, evaluated for temperature, frequency and grain size (16).

Characterisation
Grain size and dislocation density of each specimen were determined from oxidative decoration of the grain boundaries and dislocation lines (except specimen T0436, for which grain size was determined via Electron Back-Scatter Diffraction, see Figs. S1 and S2). Selected polished sections were oxidised in air at 900°C for 45 minutes (33). The surfaces were carefully re-polished to remove surface oxidation. Large regions (ca. 5000 µm²) were imaged at high magnification (e.g. ×8000) using a field emission scanning electron microscope equipped with a back-scattered electron detector. Images of these regions (typically 3 to 4 per specimen) were processed to highlight either the grain boundaries or dislocation lines. For each domain enclosed by the imaged grain
boundaries, the diameter of the equivalent circle, corrected for sectioning effects, was used as a proxy for grain size. Dislocation density was determined by measuring the apparent length of each dislocation in a micrograph. This value was corrected for projection of plunging dislocations to a flat surface, by knowing the depth of information at a given acceleration voltage (i.e. 5 kV, see Appendix of (19)). Dislocation density was then calculated as the true cumulative length divided by the product of imaged area and the depth of information. Uncertainty in dislocation density was obtained by subdividing existing images into 16 equal parts of about 180 µm² from which dislocation density was estimated. The mean dislocation density corresponded to the whole and a standard deviation among the 16 areas was obtained. Water contents measured by infrared spectrometry are listed in Table 1 and representative FTIR spectra are found in Figure S3.

**Dislocation glide in torsional forced oscillation of specimens pre-deformed in either compression or torsion**

The anelastic strain expected from reversible dislocation motion in a polycrystalline specimen tested in torsional oscillation requires consideration of the resolved shear stress \( \sigma \), or Schmid factor \( S \), for dislocation glide within individual crystallites (34). During prior deformation by dislocation creep, the resolved shear stress determines the dislocation density \( \rho \) through a relationship of the form \( \rho \propto \sigma^m \), with exponent \( m \) in the range \( \sim1-2 \) (31,35,36). During subsequent testing in microstrain torsional oscillation, it is envisaged that segments of existing dislocations glide reversibly in response to the prevailing resolved shear stress for the relevant slip system. The shear strain associated with such dislocation glide contributes to the torsional strain to a degree also specified by
the Schmid factor. For a crystallite oriented with \([\alpha,\beta,\gamma]_c\) parallel to the axis of a cylindrical specimen, the Schmid factor for \([100](010)\) glide in axial compression is \(S_c = \alpha\beta\) where \(\alpha\) and \(\beta\) are direction cosines (34). The corresponding Schmid factor for torsion around the same axis is \(S_t = T(\alpha,\beta) \sin \lambda\), where \(T^2(\alpha,\beta) = \alpha^2 + \beta^2 - (2\alpha\beta)^2\), and the angle \(\lambda\) relates the azimuthal orientation around \([\alpha,\beta,\gamma]_c\) to the direction of the local radius vector. Accordingly, the anelastic strain expected of dislocation glide in such a crystallite in a specimen pre-deformed in torsion is proportional to \(S_t^{2+m}\), whereas for prior compressive deformation, the corresponding quantity is \(S_c^mS_t^2\). With the simplifying assumptions of uniform stress throughout the polycrystal and \(m = 2\), average values \(\langle S_t^{2+m}\rangle\) and \(\langle S_c^mS_t^2\rangle\) for an aggregate of randomly oriented crystallites were determined. The intuitively reasonable result is that \(\langle S_t^{2+m}\rangle/\langle S_c^mS_t^2\rangle = 9\) – suggesting that prior torsional deformation is much more effective than prior compressive deformation in generating a population of dislocations suitably oriented for glide during the subsequent torsional oscillation testing.
Fig. S1.

Electron Back-Scatter Diffraction maps of the torsionally pre-deformed sol-gel specimen T0436. (a) Grains are colour-coded by their shear directions, summarised in the inverse pole figure. Grains also exhibit a shape preferred orientation. Shear sense in the shear plane is dextral as indicated by the pair of arrows. (b) Band contrast map (in grey-scale) and sub-grain boundaries highlighted. Coloured lines correspond to the following mis-orientation angles: > 2° yellow, > 5° lime green, > 8° purple, and grain boundaries with high mis-orientation angles > 10° blue, > 20° aqua and > 30° black.
Fig. S2

Specimen T0436 after forced-oscillation testing. Other details as for Fig. S1.
Fig. S3

Fourier transform infrared spectroscopy measurements on specimens recovered from forced-oscillation testing (H6585, D6618, D6646 and T0436) normalised to 1 cm thickness and each offset on the y-axis by 1 cm⁻¹. Note the near-zero absorption coefficients of the sol-gel specimens (flat lines) suggesting they are essentially dry in comparison with ‘dry’ (PI-181) and ‘wet’ (PI-333) olivine polycrystals synthesised from a natural precursor (San Carlos) by Mei and Kohlstedt (37).
Fig. S4

Pole figures of the torsionally pre-deformed specimen T0436 before (a) and after (b) forced-oscillation testing for the principal directions in olivine ([100], [010] and [001]).

The colour coding refers to the density of data points as multiples of uniform distribution (MUD) normalised to 3 to facilitate comparison. The pole figures are presented as lower hemisphere equal area projections with a half-scatter width of 15° and cluster size of 3°. The sense of shear is indicated by the arrows. The north (south) poles correspond to the shear plane normal. The pole figures demonstrate a weak CPO prior to forced-oscillation testing (a) for a one-point-per-grain analysis, which is enhanced for a grain-size weighted
analysis. Final measurements (b) do not repeat this result. The histograms show uncorrelated mis-orientations for olivine between 0 – 120° and the insets show the fabric strength indicators: M- and J-indices, demonstrating progressively low values from prolonged high temperature annealing and thus close to uniform fabrics. An M-index of one corresponds to a single crystal orientation, and an M-index of zero to a uniform fabric.
**Database S1**

*Raw forced oscillation data*

*Not included in model fit*

†Extrapolated between temperatures
Database S2
JF Burgers creep model oscillation data – based on grain size of individual specimens
Database S3

Dislocation density data (in $\mu$m$^2$):

*Regions are sized at about 180 micrometres squared
Database S4
Grain size information
Database S5
Raw FTIR data
References


