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High-temperature viscoelasticity of fine-grained polycrystalline olivine

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Abstract Torsional forced-oscillation and microcreep methods have been employed in a study of the viscoelastic behaviour of fine-grained polycrystalline olivine at high temperatures (to 1300 °C), seismic frequencies and low strain amplitudes. The FeO90 specimens are of low porosity and low dislocation density. They vary in mean grain size from 8 to 150 μm and contain only trace amounts (≪0.1 vol%) of quenched melt glass. For \( T \leq 900 \) °C, their behaviour is essentially elastic and the shear modulus \( G \) closely approaches that expected for a dense polycrystal from single-crystal elasticity data – confirming the suppression of thermal microcracking in this study. At higher temperatures, pronounced absorption-band dissipation and associated dispersion of the shear modulus provide evidence of linear viscoelastic behaviour. Both recoverable (anelastic) and permanent (viscous) strains are involved and the proportion of the latter increases with increasing temperature and decreasing frequency. Comparison of the results for the three specimens provides a clear indication that the viscoelastic behaviour, attributed to diffusional processes, is grain-size-sensitive with the dissipation and associated dispersion increasing with decreasing grain size. Both elastically accommodated and diffusional accommodated grain-boundary sliding appear to be implicated.

Keywords Olivine · Viscoelastic behaviour · Andrade model · Burgers model · Seismic wave attenuation · Anelastic behaviour · Grain boundary · Diffusion

Introduction

The attenuation of seismic waves in the Earth’s mantle has been intensively studied since the 1960’s. A recent review by Romanowicz and Durek (2000; see also Jackson and Rigden 1998) highlights the following major findings. The dissipation \( Q^{-1} \) is associated predominantly with shear deformation (governed by the shear modulus \( G \)) rather than hydrostatic compression (described by the bulk modulus \( K \)). The radial variation of \( Q_G^{-1} \) in the upper mantle and transition zone is relatively well established. The attenuation is most pronounced at depths of about 80–220 km in the upper mantle, where \( Q^{-1} \) exceeds 0.01. This zone of strong attenuation is overlain by a low-loss lid, and underlain by a region within which \( Q^{-1} \) decreases markedly with increasing depth to values of 0.0025–0.003 in the upper part of the lower mantle. \( Q_G^{-1} \) appears to be mildly frequency-dependent, varying as \( \omega^{-2} \) with \( \omega \sim 0.1–0.4 \).

The upper mantle zone of high attenuation is also characterised by shear wave speeds which are generally relatively low (e.g. Dziewonski and Anderson 1981; Grand and Helmerber 1984) and highly variable laterally (Grand 1994; Zielhuis and Nolet 1994; Su et al. 1994; Ritzwoller and Lavely 1995; Li and Romanowicz 1996; Su and Dziewonski 1997; van der Hilst et al. 1998). Indeed, both the radial models and the superimposed lateral variability revealed by tomographic studies (Durek et al. 1993; Romanowicz 1995) exhibit a strong spatial correlation between high attenuation and relatively low shear wave speeds. This association is suggestive of thermally activated viscoelastic behaviour in the solid state (e.g. Goetze 1977) and, at least locally (e.g. beneath mid-ocean ridges), with the effects of partial melting (e.g. Toomey et al. 1998; Webb and Forsyth 1998).

Interpretation of these seismological observations requires an understanding of the viscoelastic behaviour of ultramafic materials at high temperature and seismic frequency established through laboratory experiments conducted under carefully controlled conditions. The torsional forced-oscillation/microcreep methods best suited to such measurements have been developed and applied to natural ultramafic rocks by Berckhemer et al. (1982) and Jackson et al. (1992). These studies revealed
pronounced dissipation and associated shear modulus dispersion at high subsolidus temperatures, and a frequency dependence of $Q^{-1}$ given by $\omega^{-z}$ with exponent $z \sim 0.1$–0.3.

However, detailed interpretation of the observed behaviour in terms of defect-related relaxation mechanisms was significantly complicated by the occurrence of thermal microcracking, by the progressive dehydration of hydrous layer-silicate minerals, and in the study of Aheim dunite by Berckhemer et al. by melting, which may account for their very high activation energy (800 kJ mol$^{-1}$). Dissipation has also been measured at high temperatures and seismic frequencies on untreated and predeformed specimens of single-crystal San Carlos olivine (Gueguen et al. 1989). This sensitivity of attenuation to prior deformation (and thus dislocation density), along with the approximate consistency between the results for natural rock specimens and single-crystal olivine, suggest that grain-size-insensitive (dislocation-related) processes may be responsible for the viscoelastic relaxation.

In order to avoid the difficulties arising from the chemical complexity of natural rocks and the inevitable microcracking during thermal cycling of relatively coarse-grained materials, the emphasis has switched in recent studies to fine-grained synthetic ultramafic materials. Tan et al. (1997a) demonstrated the viability of this approach, reporting measurements of the temperature and frequency dependence of both shear modulus $G$ and associated strain energy dissipation $Q^{-1}$ for an Fo$_{90}$ olivine polycrystal of high purity and $\sim$50 μm grain size which was prepared by hot-isostatic pressing of grain-size-sorted powder derived from carefully selected crystals of San Carlos (Arizona) olivine. Subsequently, Gribb and Cooper (1998) reported torsional forced-oscillation and microcreep data for a very fine-grained synthetic dunite produced by hot-isostatic pressing powder obtained by pulverising Balsam Gap (North Carolina) dunite.

Here, we extend the preliminary work of Tan et al. (1997a) by reporting results for a suite of three synthetic olivine polycrystals of varying grain size and microstructure. These specimens remain essentially melt-free and uncracked throughout the 20–1300 °C range of the measurements, and therefore provide a robust subsolidus baseline, at least at relatively small grain size, with which seismological data can eventually be compared.

and 180–125 μm grain-size fractions – these ranges being chosen to allow optimal purification by subsequent heavy liquid and magnetic separation – and by physical removal of individual impurity grains. The fraction of the crushed powder recovered from suspension in methyl iodide (specific gravity 3.3) was significantly depleted in lighter pyroxene and heavier magnetic impurities, the latter being further reduced in concentration by magnetic separation. The residual level of impurities (<1%) was substantially reduced by manual removal of any impurity grains recognised through a binocular microscope.

The purified powder was then ground in an agate mill – the speed and duration of milling being chosen to achieve effective comminution with minimal contamination. The milled powder was passed through a set of brass sieves reserved for use with Fo$_{90}$ olivine, yielding batches of powder of 38–63 μm and <38-μm particle size. Finally, sedimentation from suspension in ethanol over time intervals of order 1 h, 1 day and 1 month, repeated for the 1-h and 1-day intervals, yielded powders with relatively narrow particle-size distributions of 10–38, 2–10 and <2 μm, respectively.

**Cold-pressing and preliminary heat treatment**

Pellets of 15 mm diameter and 7–8 mm length, containing ~30% porosity, were produced by cold pressing in a hardened steel die under 300 MPa uniaxial pressure. Prior to hot-pressing (described below), such pellets were either simply oven-dried (150 °C) for several days or prefired at 1200 °C under inert atmosphere to remove strongly adsorbed water and any residues from organic solvent. For temperatures from 100°C gas mixture comprising equal partial pressures of CO and CO$_2$ was used, yielding log $f_0$ (Pa) $\sim$ -6 at 1200 °C (Muan and Osborn 1965). In order to prevent the precipitation of graphite from gas of this composition (Muan and Osborn 1965), argon was substituted for the CO/CO$_2$ gas mix for temperatures below 700 °C. The pellets emerge from this treatment cream to pale green in colour.

**Hot-isostatic pressing**

Dense polycrystalline olivine compacts were prepared from stacks consisting of several cold-pressed pellets by hot-isostatic pressing within an internally heated gas-medium high-pressure apparatus (Paterson 1990). Hot-pressing conditions, optimised through extensive prior exploratory experiments, for the various specimens mechanically tested in this study are displayed in Table 1. The stack of pellets was located between blind alumina pistons within a mild steel tube of 15 mm internal diameter, sealed at either end with an O ring. Thin layers of iron foil inserted at either end of the stack of pellets served to isolate the specimen from the alumina pistons and also to reduce thermal stresses caused during cooling by the mismatch in thermal contraction between the alumina pistons and olivine specimen. The Fe foils and mild steel jacket enclosing the specimen impose a chemical environment that is strongly reducing (Raterron et al. 1998), leading to the precipitation within the olivine polycryst of some small Ni-rich metallic blebs.

Following transverse sectioning of the steel-jacketed hot-pressed compacts for microstructural characterisation, the steel jackets were removed from the dense hot-pressed olivine compacts either by acid (HNO$_3$/HCl) dissolution or, on the later-tested specimens (nos. 6261 and 6328), by mechanical grinding. A cylindrical specimen typically 12 mm in diameter and 30 mm in length was then prepared by precision grinding. The specimen was cleaned successively in water, ethanol and acetone and then oven-dried in anticipation of mechanical testing described below.

**Microstructural characterisation**

**Light microscopy**

Microstructures were examined by polarised light microscopy in both transmission and reflection. Thin sections were epoxy-