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A composite viscoelastic model for incorporating grain boundary sliding and transient diffusion creep; correlating creep and attenuation responses for materials with a fine grain size

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A new viscoelastic creep function that incorporates both the effects of elastically-accommodated grain boundary sliding (GBS) and transient diffusion creep is proposed. It is demonstrated that this model can simultaneously describe both the transient microcreep curves and the shear attenuation/modulus dispersion in a fine-grained (d ~ 5 μm) peridotite (olivine + 39 vol. % orthopyroxene) specimen. Low-frequency shear attenuation, Q\(_{-1}(\omega)\), and modulus dispersion, G(\(\omega\)), spectra were measured in a one-atmosphere reciprocating torsion apparatus at temperatures of 1200 ≤ T ≤ 1300°C and frequencies of 10\(^{-2.25}\) ≤ f ≤ 10\(^{6}\) Hz. Reciprocating tests were complemented by a series of small stress (τ ~ 90 kPa) microcreep experiments at the same temperatures. In contrast to previous models where the parameters of viscoelastic models are derived by fitting the Laplace transform of the creep function to measured attenuation spectra, the parameters are derived solely from the fit of the creep function to the experimental microcreep curves using different published expressions for the relaxation strength of elastically-accommodated GBS. This approach may allow future studies to better link the large dataset of steady-state creep response to the dynamic attenuation behavior.

Keywords: anelasticity; internal friction; grain boundary sliding; diffusion creep

1. Introduction

1.1. Creep functions

Low-frequency attenuation is the frequency-domain manifestation of creep (i.e. time-dependent inelastic deformation at constant stress). This similarity principle suggests that a complete and physically meaningful description of either a creep curve or an attenuation spectrum is sufficient, in principle, to calculate the other. The time-dependent mechanical properties of a material are described by a creep function.

High-temperature transient creep curves typically show three distinct features. Upon the application of a step-function increase in stress, a sample typically exhibits...
an instantaneous elastic strain whose magnitude is proportional to that elastic modulus appropriate to the loading mode. Elastic strain is typically followed by a regime of transient flow where the strain-rate changes over time and ultimately leads to the steady-state strain rate, which is characteristic of the large-strain viscosity. Therefore, a creep function that describes the time-dependent compliance of a material in shear-mode loading may take the following form:

\[ J(t) = J_U + \Gamma(t) + \frac{t}{\eta_{ss}}, \]  

(1)

where \( J_U \) represents unrelaxed compliance, \( \eta_{ss} \) represents the steady-state shear viscosity, and \( \Gamma(t) \) is a function of time. The first term, \( J_U \), is equivalent to the reciprocal of the unrelaxed shear modulus and describes the near-instantaneous elastic distortion associated with loading. The second term, \( \Gamma(t) \), represents a transient wherein the strain-rate varies with time. The final term, \( t/\eta_{ss} \), represents the steady-state creep rate.

The relationship between transient creep and attenuation can be obtained by subjecting the creep function to an oscillating shear stress (\( \tau(t) = \tau_0 \exp(i\omega t) \)) of the form \( \tau_0 \exp(i\omega t) \), where \( \omega \) represents angular frequency (\( \omega = 2\pi f \), where \( f \) is frequency in Hz) and \( \tau_0 \) represents the magnitude of the shear stress. The resulting shear strain (\( \gamma(t) \)) will lag behind the shear stress by some phase angle \( \delta \) and take the form \( \gamma(t) = \gamma_0 \exp(i\omega t - \delta) \). The complex compliance, \( J^* \), can then be written:

\[ J^* = \frac{\gamma_0}{\tau_0} (\cos \delta - i \sin \delta), \]  

(2a)

or

\[ J^* = J_1 + iJ_2, \]  

(2b)

where \( J_1 \) and \( J_2 \) are the real and imaginary components of the dynamic compliance, respectively. Attenuation (\( Q^{-1}(\omega) \)) and shear modulus dispersion (\( G(\omega) \)) are then related to Equation (1) according to

\[ Q^{-1}(\omega) = \frac{J_2(\omega)}{J_1(\omega)} = \tan \delta, \quad \text{and} \]

\[ G(\omega) = [J_1(\omega)^2 + J_2(\omega)^2]^{-1/2}, \]  

(3)

(4)

respectively. A time-domain creep curve (Equation (1)) is thus related to attenuation and modulus dispersion (Equations (3) and (4)) through its Laplace transform.

An appropriate viscoelastic model that relates \( \gamma_0 \) to \( \sigma_0 \) must meet four criteria. It must describe both the attenuation spectra and the shear modulus dispersion spectra given by Equations (3) and (4). Additionally, it must also accurately describe the time-dependent creep behavior of a specimen, given by Equation (1). Ideally, the form of the creep function must be physically meaningful, such that, e.g. extrapolation from laboratory to natural settings can be confidently made.

In much previous experimental work, a creep function is chosen so that its transform can reproduce the form of a measured attenuation spectrum. The parameters of the creep function are then determined by fitting the transform of the creep function to the measured attenuation data [1,2]. Often no attempt is made to relate the creep function to microcreep curves. As discussed in detail below, existing...
models where the attenuation spectrum is based on time- to frequency-domain inversion of creep data do not describe the entirety of features of the attenuation spectrum [3,4]. In this paper we adopt the approach originally taken by Gribb and Cooper [3], but extend the model in order to describe more fully all features of experimentally observed attenuation spectra. We choose this approach for the following reasons. First, an important goal of attenuation studies is to relate the physical mechanisms underlying attenuation to those operative during steady-state creep, such that the large set of experimental creep data can be applied to questions of attenuation. This may be particularly important for addressing the role of extrinsic variables, such as the chemical potential of water, on attenuation where existing experimental techniques make it difficult to address directly. Basing viscoelastic models on creep curves represents an important step in that direction. Second, the requirement that the viscoelastic model describe the transient response in both the time and frequency domains imposes an additional demand on the creep function, which may allow for better discrimination amongst models.

1.2. Previous work
Experimental studies of attenuation in fine-grained (2–100 μm grain size) peridotite have consistently shown a low-frequency regime, the “high-temperature background”, where shear attenuation, \( Q/\mu \), decreases mildly and monotonically with increasing frequency (\( Q/\mu \propto f^{-0.25–0.35}) \). A consensus is emerging that the low-frequency background, specifically in the case of linear viscoelasticity, is due primarily to processes occurring at and along grain (and subgrain) boundaries (e.g. [6–8]).

Several viscoelastic models have been put forth to describe the high-temperature background and its associated shear-modulus dispersion. The first approach describes attenuation in terms of transient diffusion creep. In this formulation, grain boundaries are assumed inviscid at low frequency; dissipation is due to chemical diffusion in the presence of a diminishing potential. An analytically-derived creep function based on these principles was presented by Raj [9]. This model accurately describes the high-temperature background in fine-grained olivine aggregates and takes the form of an Andrade viscoelastic model [3,10].

Gribb and Cooper [3] conducted low-frequency reciprocating torsion measurements of \( Q_G^{-1} \) on fine-grained (\( d \sim 3 \mu m \)), essentially dislocation-free, olivine aggregates at temperatures of 1200 ≤ T ≤ 1285°C and frequencies ranging from 10^{-2.25} ≤ f ≤ 10^{-0.5} Hz. They complemented these experiments with a series of microcreep tests on the same samples at the same temperatures. The salient results of their study were: (1) the activation energy determined for the transient portion of the creep curves was identical to that determined for the steady-state state portion suggesting that the same physical mechanism was operative in both cases, (2) the Andrade viscoelastic model provided an excellent description of their transient creep curves, and moreover, that the transform of the Andrade model provided an excellent fit to their measured \( Q_G^{-1} \) values. The transient term of the Andrade model takes the form of a power law where \( \Gamma(t) = At^n \). \( n \) is a constant with a value typically close to \( \frac{1}{3} \).
They interpreted their results in the context of the transient creep model proposed by Raj [9], in which the time evolution of the normal traction along grain boundaries in a hexagonal network was characterized assuming the grain boundaries were nearly shear inviscid. At the onset of loading (though actually following elastically-accommodated sliding; the inviscid boundary assumption makes this step instantaneous) singularities in normal stresses exist in the vicinity of triple junctions. Steady-state diffusion creep requires a nominally parabolic (paraboloidal in three dimensions) distribution of normal stresses along grain boundaries. Transient creep is thus a manifestation of the diffusion-effected evolution of normal tractions from the initial condition to the steady-state distribution. By solving the coupled elasticity and diffusion equations using a Fourier series description of the boundary shape, Raj [9] determined that the transient term during grain boundary diffusion (Coble) creep takes the form of an Andrade model.

A second approach to describing the transient term has taken the form of an extension of the classic Burgers solid model. The transient term in the Burgers model is an exponential decay with a discrete relaxation time (e.g. [11]). The “extended” Burgers model broadens the transient term by summing many exponential decays with different relaxation times according to

$$\Gamma(t) = \Delta \int_{t_0}^{t} D(\tau)(1 - \exp(-t/T_B))d\tau, \quad (5a)$$

where

$$D(\tau) = \frac{\alpha Q T_B^{\alpha-1}}{T^{\alpha-1}_h - T^{\alpha-1}_l} \quad (5b)$$

is chosen such that the resulting transient mimics the form $Q^{-1}_G \propto f^{-\alpha}$ over a finite, and somewhat arbitrary range of relaxation times, $T_h - T_l$ [2]. In these expressions, $\alpha$ imposes the frequency dependence of attenuation and $\Delta$ is termed the “relaxation strength” and describes the magnitude of the transient. Both models have been utilized to successfully model the high-temperature (1000 $\leq T \leq 1300^\circ$C) background observed in low-frequency (10$^{-3}$ $\leq f \leq 10^0$ Hz) reciprocating torsion experiments on fine-grained peridotite samples.

At higher frequencies, however, the high-temperature background behavior commonly gives way to an attenuation plateau, where $Q^{-1}_G$ is nominally constant over several decades in frequency. Neither the Andrade model nor the extended Burgers model alone provides the flexibility to model an attenuation “peak” superimposed over a monotonically diminishing background. Consequently, in the subsequent section, we develop a simple analytical function that incorporates both elastically-accommodated GBS and transient diffusion creep. We then demonstrate that this simple model is sufficient to model both attenuation and microcreep in an internally consistent way.

2. Composite viscoelastic model

Whereas both the Andrade model and the extended Burgers model successfully describe the high-temperature background, neither provides an explanation for an
attenuation plateau at high-frequencies and/or low temperatures. This plateau has been attributed to the superposition of a Debye peak on top of the high temperature background. An attenuation peak of this nature was originally observed experimentally by Kê [12] in polycrystalline aluminum and has commonly been attributed to “elastically-accommodated grain boundary sliding (GBS)”. In this model, an instantaneous application of stress leads to flow along grain boundaries that is time-dependent and ultimately halted by the build-up of elastic stresses at triple-junctions or other asperities along the interface. Sliding may proceed until the shear stress across the boundary falls to zero. The flow is anelastic in that upon the release of the load, the triple junctions “push back” and the strain produced by sliding is recovered. Several workers have hypothesized that the high-frequency “plateau” observed in polycrystalline peridotite is due to the superposition of this Debye peak on top of the high-temperature background [6,13]. Raj [9] made the assumption that elastically-accommodated GBS occurs rapidly, that is, the boundaries are essentially inviscid in shear, thus the distribution of stresses along grain boundaries following the completion of sliding is an initial condition in his model and not considered directly. The onset of an attenuation plateau in the dissipation spectrum therefore points to the frequency whereby the shear-inviscid boundary assumption fails.

The creep function for elastically-accommodated GBS takes the form of the standard anelastic solid with a characteristic relaxation time \( T_{gbs} \) specified as

\[
J_c(t) = J_U + (J_R - J_U)[1 - \exp(-t/T_{gbs})]
\]

and

\[
T_{gbs} = \frac{\eta_{gbd}}{\delta G_U},
\]

where \( \eta_{gb} \) is the grain boundary viscosity, \( d \) is the grain size, \( \delta \) is the structural width of the grain boundary, and \( G_U \) is the unrelaxed shear modulus of the material [14]. Subjecting this model to a periodic stress leads to real and imaginary components of the dynamic compliance given by

\[
J_1(\omega) = J_U + \frac{J_R - J_U}{1 + \omega^2 T_{gbs}^2}
\]

and

\[
J_2(\omega) = (J_R - J_U) \frac{\omega T_c}{1 + \omega^2 T_{gbs}^2},
\]

respectively, where \( J_R \) is the relaxed compliance of the material. \( Q_G^{-1} \) and \( G \) can then be calculated according to Equations (3) and (4), respectively. Expressed in terms of moduli, attenuation for this model is then expressed as

\[
Q_G^{-1}(\omega) = \frac{G_U - G_R}{G_R} \frac{\omega T_{gbs}}{1 + \omega^2 T_{gbs}^2},
\]

where \( G_R \) and \( G_U \) are, respectively, the relaxed and unrelaxed moduli of the material. This expression yields a “Debye” peak of width approximately one decade of frequency, symmetrical about \( \omega T_c = 1 \) [15]. The magnitude of the relaxation, \( (G_U - G_R)/G_R \), is termed the “relaxation strength” \( \Delta \) (cf. Equation (5)).
Several workers have developed models for the relaxation strength of elastically-accommodated GBS. For polycrystalline materials, the relaxation strength associated with elastically-accommodated GBS is fixed and independent of grain size [16]. The relaxation strength is solely a function of Poisson’s ratio and the shape of the grains. For shear modulus loading of spherical grains, Kê [12] expressed the relaxation magnitude solely in terms of the Poisson’s ratio \( \nu \) according to

\[
\frac{G_R}{G_U} = \left( \frac{2}{5} \right)^\frac{7 + 5\nu}{7 - 4\nu}.
\]  

(11)

The Poisson’s ratio for polycrystalline olivine at high temperatures is roughly \( \frac{1}{4} \) [17]. According to Equation (11), for \( \nu = \frac{1}{4} \), \( G_R/G_U = 0.55 \) and thus \( \Delta = 0.82 \). Kê [12] obtained \( \Delta = 0.67 \) in his experiments on polycrystalline aluminum (\( \nu = \frac{1}{4} \) for aluminum and thus \( \Delta = 0.64 \)). Consequently, \( J_R \) in Equations (8) and (9) can be expressed in terms of the unrelaxed compliance according to \( J_U(1 + \Delta) \).

In contrast, Ghahremani [18] conducted finite-element simulations of GBS in a hexagonal grain network and parameterized the results according to

\[
\frac{G_R}{G_U} = \frac{E'}{E_R} = \frac{0.83\nu - 0.86}{\nu - 1},
\]  

(13)

where \( \nu_R \) and \( \nu_U \) are the unrelaxed and relaxed Poisson’s ratios respectively and \( E_U \) and \( E_R \) describe the Young’s modulus dispersion. From isotropic elasticity,

\[
\frac{G_R}{G_U} = \frac{E'}{2(1 + \nu')},
\]  

(14)

and thus \( \nu = \frac{1}{4} \) yields values of \( \Delta = 0.23 \) for the finite element model. Ghahremani [18] also derived the relaxation strength of spherical grains using a self-consistent approach. In this formulation, the grain behaves as a spherical inclusion with unrelaxed elastic properties within a homogenous and continuous matrix that has the relaxed elastic properties. Across the interface between matrix and inclusion, the shear stress vanishes and the normal stress is continuous. This approach yields

\[
\frac{G_R}{G_U} = \frac{7 + 5\nu}{4(7 - 4\nu)}.
\]  

(15)

This modulus relaxation is exactly \% that of Equation (11). Equation (15) yields \( \Delta = 1.91 \) for \( \nu = \frac{1}{4} \).

Finally, Raj and Ashby [19] used a Fourier series approximation for the grain boundary morphology to derive

\[
\frac{G_R}{G_U} = (0.57(1 - \nu) + 1)^{-1}.
\]  

(16)

Consequently, for \( \nu = \frac{1}{4} \), \( \Delta = 0.43 \). As recognized and emphasized by Ghahremani [18], the randomly oriented, viscous crack model of O’Connell and
Budiansky [20] yields results that are indistinguishable from the Raj and Ashby [19] model, and thus will not be considered separately here.

A recent reanalysis of the coupling between transient diffusion creep and elastically-accommodated GBS has been presented by Morris and Jackson [8]. These authors used a perturbation approach to solve the coupled elasticity and diffusion problems for an infinitesimal boundary slope. While their analytical functions differ substantially from the Fourier series approximation proposed by Raj [9], the form of the attenuation spectra are similar to those hypothesized by Cooper [6]: a Debye-like peak at high-frequency associated with elastically-accommodated GBS, followed by a broad region of smoothly increasing attenuation with decreasing frequency. Direct utilization of this model awaits numerical solutions with finite interfacial slopes.

To model the combined effects of the diffusional background and the effect of elastically-accommodated GBS we introduce the composite creep function:

\[ J(t) = J_U + (J_U \Delta)[1 - \exp(-t/T_{gbs})] + At^n + \frac{t}{\eta_{ss}}. \]  

A spring and dashpot model of the composite creep function is presented in Figure 1. We chose to base our model for the high-temperature background on the Andrade viscoelastic model due to its parametric simplicity and the uncertainty associated with choosing cutoff frequencies associated with the extended Burgers model. Furthermore, the Andrade model is physically meaningful in that it describes chemical diffusion in the context of a diminishing potential. As demonstrated by Gribb and Cooper [3], this relaxation scales according to a non-exponential relaxation time:

\[ T_{an} \approx \frac{4\eta_{ss}}{G_U}. \]  

A simple combination (Boltzmann superposition) of the two previous creep functions is justifiable for the following reasons. First, due to the very-fine grain size in our experimental specimens combined with the very-small shear stresses employed in our experiments (as well as those of other investigators), nonlinearity associated with many dislocation-based creep mechanisms is suppressed (e.g. [21]). Second, if elastically-accommodated GBS is sufficiently rapid \((T_{gbs} \ll T_{an})\), then the long-range chemical diffusion mechanism described by the Andrade model will not “feel” the unrelaxed compliance \(J_U\). The apparent elastic displacement at the onset of loading will be larger than expected by \(J_U\) and will rather reflect the relaxed compliance, \(J_R\), which prevails following the sliding step. In contrast, if the time scales for GBS and for grain boundary diffusion are not widely separated, then there may be coupling between the stress fields associated with the two relaxation processes such that Equation (17) may not hold. A schematic representation of the anticipated attenuation and shear modulus dispersion spectrum for the composite creep function is presented as Figure 2 for the case where \(T_{gbs} \ll T_{an}\). We now evaluate this model against our new experimental data using the different models for the relaxation strength discussed above and demonstrate that Equation (17), besides being logical physically, provides an acceptable to fit to both our creep data and our measured attenuation data.
3. Experimental methods

3.1. Specimen fabrication

Peridotite, i.e. olivine–orthopyroxene (ol–opx), specimens were fabricated from Balsam Gap (Jackson Co., NC) dunite (a rock composed almost exclusively of olivine) and Bamble (Norway) orthopyroxene. Optically clean specimens of both phases were conventionally ground to millimeter sized particles and then reduced to \(10^{-2} \, \text{mm}\) using fluid jet energy mill pulverization. Balsam Gap dunite includes a small volume fraction of talc (\(\leq 1\text{ vol. }\%\)), which forms a minor amount of additional opx during processing. The olivine and orthopyroxene powders were then mixed in the desired proportion under ethanol and subsequently baked in a horizontal furnace at 1000°C for 4 h inside a Ni-foil-lined alumina tray to dry (adsorbed \(\text{H}_2\text{O}\)) and

\[ J(t) = J_U + \frac{t}{\eta_{ss}} + \left( \frac{J_U \Delta}{(1 - e^{-\frac{t}{T_{gb}}})} \right) + At^n \]

Figure 1. Spring and dashpot model of the composite creep function given in Equation (17). The spring, \(J_U\), represents the unrelaxed compliancy of the material \((J_U = 1/G_U)\). The term \(\eta_{ss}\) represents the steady state viscosity of the material, which prevails at \(t > T_{ss}\). \(T_{gb}\) represents the exponential-type relaxation time associated with elastically-accommodated GBS. For small \(T_{gb}\), the anelastic strain associated with elastically-accommodated GBS is difficult to distinguish from the elastic strain associated with \(J_U\). \(T_{ss}\) represents the non-exponential relaxation described by the Andrade viscoelastic model that gives rise to the high-temperature background, as discussed by Gribb and Cooper [3]. The form of the transient in the Andrade model, \(At^n\), represents a continuous distribution of relaxation times [10,11].
Figure 2. Schematic representation of (a) a composite attenuation spectrum and (b) shear modulus dispersion incorporating both diffusion-effected relaxation as the high-temperature background (Andrade model) and elastically accommodated GBS. The short-dash curve illustrates the continuously diminishing attenuation predicted by the Andrade viscoelastic model [3], which incorporates three regimes: (1) a steep frequency dependence at low frequencies characteristic of steady state creep, (2) an intermediate frequency range where $Q^{-1}_G$ varies mildly with frequency, and (3) a high-frequency regime where attenuation steepens again. The second dashed curve shows the Debye peak associated with elastically accommodated GBS. The solid curve illustrates the behavior of the composite mechanism, in this case a simple sum of the two mechanisms as allowed by Boltzmann superposition. The composite curve shows an attenuation “plateau”—perhaps a peak, depending on the absorption strength of the Debye peak—similar to the features observed in experiments.
dehydrate (decompose minor hydroxyl-bearing phases like talc) the mineral powders. At these conditions, the diffusion of ionic hydrogen (protons) in silicate minerals is sufficiently rapid to completely dehydrate the material [22]; our experimental specimens can be considered anhydrous. The oxygen partial pressure within the furnace was held near the Ni–NiO solid-state reaction by a continuous flow of an appropriate mixture of CO and CO$_2$ gas at a rate of 50 cm$^3$ min$^{-1}$. The dried powder was then mixed with 25 wt % acetone (which acts as a binder) and measured amounts of the mixture pressed into a steel die to 90 MPa at ambient temperature. The pressed cylindrical plug displayed a “green-state” density of roughly 1.7 g cm$^{-3}$ as measured by the mass and dimensions of the sample. The samples were then sintered in a horizontal furnace, under rough vacuum ($P \approx 10$ Pa), for 4 h at 1390°C. The long sintering time (i.e., in excess of that required to densify the specimen) acts to stabilize the sample against further grain growth during mechanical testing.

Microstructural characterization was done using a Leo 1530VP field emission gun scanning electron microscope (FEG-SEM). The grain size of both olivine and pyroxene were measured using the linear intercept method and employing a geometrical correction factor of 1.5 [23]. The aggregate, in the experiment reported here, of olivine and 39 vol. % orthopyroxene (ol–39 vol. % opx) is nearly fully dense. A backscatter electron micrograph of the sample (in this case, post-mechanical testing) is shown as Figure 3. The grain sizes of olivine and orthopyroxene in this sample are nearly identical at 5.2 and 5.4 μm, respectively. The sample also displayed a modest melt fraction of 0.015 as evidenced by glass concentrated at grain triple junctions visible in part in Figure 3. Grain and solid-state phase boundaries in this system remain melt-free, as evidenced by careful electron microscopy studies (e.g. [24,25]) and measurements of dynamic mechanical response (e.g. [26]).

(For completeness’ sake, we note that samples compacted and sintered without the initial 1000°C, 4 h drying/dehydration step displayed dramatic grain growth during sintering, which is likely related to the release of H$_2$O, from the breakdown of the talc component from the Balsam Gap dunite, and its effect on grain- and phase-boundary mobility.)

Following sintering, the sample was cut and ground conventionally, for mechanical testing, into a rectangular parallelepiped with dimensions of 3.5 × 3.5 × 15 mm.

### 3.2. Mechanical testing

All experiments, both reciprocating torsion and unidirectional creep tests, were conducted using a 1 atm torsion apparatus whose design is described elsewhere [27]. Subsequent modification of the original design has included: (1) an upgrade to the digital control electronics, (2) improved differential impedance transducers (model DIT-5200, Kaman Instrumentation) which increase the displacement resolution of the apparatus by roughly one order of magnitude, and (3) a new gage conditioner for the original torque cell [4]. The improved electronics bring the resolution in shear stress to approximately 2 kPa and the resolution in shear strain to <1 × 10$^{-7}$ (for samples with the dimensions listed above) to temperatures of 1400°C. Specimen gripping is accomplished through the mismatch in the coefficient of thermal expansion between the peridotite sample and the molybdenum alloy load train.
The oxygen fugacity during the experiment is controlled by introducing a slow
(50 cm³ min⁻¹) but continuous feed of CO + CO₂ gas maintained at a ratio of 7:1.
This ratio maintains an oxygen fugacity that is (1) sufficiently low to prevent
oxidation of the high-temperature refractory-metal components of the apparatus
yet (2) safely within the redox stability field of the silicate phases of interest [27,28].

The temperature was increased at 250°C h⁻¹ and allowed to stabilize for 30 min
prior to each segment of mechanical testing. Each sample was tested at temperatures
between 1000°C and 1300°C in 50°C intervals, typically beginning with the lowest
temperature to minimize grip losses associated with creep of the specimen within
the sample grips. The typical experiment consists of reciprocating torsion tests in the
frequency range between 10⁻².25 and 10⁰ Hz in increments of 10⁻⁰.25 Hz. At least six
complete periods were recorded for each frequency step.

The torque and displacement record were then fit to a waveform using a
Levenberg–Marquardt nonlinear least-squares algorithm, implemented in
MATLAB, and the shear attenuation (Q⁻¹ G ≡ tan δ, where δ is the phase lag between
stress and strain) and shear modulus determined directly from these fits. The
procedure for converting torque into shear stress and angular displacement into
shear strain is given by Blake [29]. Additionally, several small-strain unidirectional
constant-torque (microcreep) tests were conducted on the material at each

Figure 3. Backscattered electron micrograph of the synthesized peridotite (ol–39 vol. % opx)
material used in the experiments. The micrograph was taken from a sectioned, post-
mechanical-testing specimen. Olivine is the light phase and orthopyroxene is the darker phase.
The dispersed black “phase” is porosity left over from sintering. This sample contains a melt
fraction of 0.015 (as measured by point counting), manifest as the finest-scale dark gray
material, located primarily at grain triple junctions.
temperature step, employing a maximum shear stress of \( \sim 90 \text{kPa} \) (unless otherwise noted), to characterize the transient and steady-state creep responses of the sample and to ensure the effectiveness of our sample-gripping procedure.

Following mechanical testing, the sample microstructure was again characterized. Little grain growth was noted; the microstructure was, for all practical purposes, identical to that of the as-sintered specimen.

3.3. Mechanical data analysis

The initial elastic displacement should correspond to the shear modulus of the material (~60 GPa at temperatures of 1200–1300°C). However, the initial elastic displacement measured in the creep data often yields much smaller values in the range of 20–40 GPa. These low apparent shear moduli have been observed previously, both by Gribb and Cooper [3] and Jackson et al. [1]. This phenomenon could be attributed to the inability of the apparatus to precisely reproduce a step function in stress (either sample grip-related or related to lag in the control electronics). Alternatively, the formation of microcracks due to differential thermal expansion between adjacent grains (or phases), while unlikely in materials with such fine grain size [30], could also yield a lower shear modulus.

However, the high shear moduli calculated from the low-temperature reciprocating torsion experiments and the lack of visible cracks in the post-mortem electron micrographs suggest that microcracking is not responsible for the low apparent shear modulus at high temperature. Our protocol for inverting the creep record with the Andrade model is similar to that employed by Gribb and Cooper [3] and Bunton [4]. We truncate the initial “instantaneous” displacement and invert for attenuation based on the unrelaxed shear modulus of olivine. However, the composite creep function suggested in Equation (17) suggests that elastically-accommodated GBS could account for the very fast creep rates if the relaxation time \( T_{gbs} \) (Figure 1) is sufficiently small.

The grain boundary viscosity is often assumed to be small compared to the steady-state creep viscosity, but large relative to an intergranular melt phase [31]. Jackson et al. [1] fit their attenuation data to a Gaussian peak superimposed over the Laplace transform of a modified Andrade viscoelastic model and determined that the grain boundary viscosity ranged from \( 10^9 \) to \( 10^4 \text{Pa s} \) between 1000°C and 1300°C. They did not, however, fit their data based on results of their unidirectional microcreep data. Nonetheless, at high-temperatures where creep testing is tractable \( (T \geq 1200°C) \), and for grain sizes in the range of 5–20 \( \mu \text{m} \), their data suggests relaxation times for creep in their experiments in the range of \( 10^{-3} \) to \( 10^{-1} \text{s} \). These short times are consistent with our observation that the apparent elastic displacement is not entirely elastic, that is, the displacement combines instantaneous elastic distortion associated with the unrelaxed modulus with some anelastic, time-dependent distortion.

We fit the raw attenuation data to the Laplace transform of the composite creep function in a manner similar to that of Jackson et al. [1] to determine the relaxation time directly. This approach successfully modeled the background portion of the attenuation spectrum, however the fitted-values for the peak were very sensitive to
the initial parameters of the fitting algorithm. This behavior is most likely related to the limited frequency-temperature window available to us in these experiments.

The time scale for creep is related to the oscillation frequency of the reciprocating tests as $2\pi f = t^{-1}$. Our data typically show the beginning of a peak at or around $\sim 0.18$ Hz, depending on the temperature. Consequently, the information in the creep curve that pertains to the attenuation peak is found within the first $\sim 0.9$ s after loading. For these creep experiments we used a logging interval of 0.08 s, thus all of the information in the creep curve that pertains to anelastic elastically-accommodated GBS is contained within the first twelve data points. Thus this analysis is very sensitive to systematic experimental error and the nonlinear least-squares fitting algorithm is usually unable to resolve $T_{gbs}$ in the fits to the creep curves. Consequently, we force a reasonable value of $T_{gbs} = 0.1$ s in our fits to the creep curve and our subsequent modeling.

4. Results

4.1. Results of reciprocating torsion tests

A plot of $\log_{10}(Q^1_G)$ vs. $\log_{10}(f)$ for temperatures of 1200, 1250, and 1300°C is presented in Figure 4a. Two distinct features are evident in the attenuation spectra. Over the temperature range of 1200 to 1300°C and the frequency range of $10^{-2.25}$ to $10^0$ Hz, $\log_{10}(Q^{-1}_G)$ ranges from −0.8 to 0.25 (Table 1). For fixed frequency, an increase in temperature is associated with an increase in attenuation. At $f < 10^{-1}$ Hz, attenuation spectra for each of the three temperatures show high-temperature background behavior where $Q^{-1}_G \propto f^{-1/3}$. At the highest temperature tested, 1300°C, the attenuation spectra show positive curvature at $f < 10^{-1.5}$ Hz. All attenuation spectra deviate from high-temperature background behavior at high frequencies. The frequency at which the spectra deviate from high-temperature background behavior moves to progressively higher frequencies at higher temperatures, ranging from $f \sim 10^{-0.75}$ Hz at $T = 1200°C$ to $f \sim 10^{-0.25}$ Hz at $T = 1300°C$. Shear moduli range from $\sim 5$ GPa to $\sim 35$ GPa over this range of temperature and frequency (Figure 4b). As expected, lower shear moduli are associated with higher values of $Q^{-1}_G$. The onset of positive curvature in the $Q^{-1}_G$ data is also associated with a kink in the modulus dispersion spectra.

4.2. Results of microcreep tests

Unidirectional creep testing shows the three distinct features commonly observed in viscoelasticity: an initial, nearly instantaneous elastic displacement, followed by a continuously diminishing transient strain-rate that ultimately decays to a steady state (Figure 5). The time-to-strain duration of the transient diminishes with increasing temperature. At $T = 1300°C$ the transient is complete in approximately 50 s, whereas at $T = 1200°C$ the transient lasts approximately 200 s. The steady-state viscosity ranges from $3.4 \times 10^{12}$ Pa s at $T = 1200°C$ to $1.9 \times 10^{11}$ Pa s at $T = 1300°C$ with an activation energy of $560 \pm 15$ kJ mol$^{-1}$. 
Figure 4. Dynamic responses of the ol–39 vol. % opx specimen. (a) A plot of \( \log_{10}(Q^{-1}/\xi) \) vs. \( \log_{10}(f) \) for \( T = 1200, 1250, \) and \( 1300^\circ C \). (b) A plot of \( \log_{10}(f) \) vs. shear moduli for the same sample at the same temperatures. Attenuation spectra (a) reveal three distinct features. First, at moderate frequencies all three curves show a broad region of high temperature background behavior where \( Q^{-1}_G \propto f^{-0.3} \). At the lowest frequencies, particularly evident at \( T = 1300^\circ C \), the spectra begin to deviate toward a steeper curvature. At higher frequencies, \( f > 10^{-0.5} \) Hz, the data show an incipient dissipation peak. The frequency at which this peak becomes evident shifts to higher frequency with higher temperature. The shear modulus data mirror the attenuation spectra: lower frequencies are associated with lower shear moduli. Note the kinks in the shear modulus spectra at the frequencies associated with the peak in the attenuation spectra.
Table 1. Observed shear attenuation and dispersion for the peridotite (ol–39 vol. % opx) sample.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>$Q_{G}^{-1}$ (GPa)</th>
<th>Frequency (Hz)</th>
<th>$G$ (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1200</td>
<td>0.4301</td>
<td>0.0056</td>
<td>21.2</td>
</tr>
<tr>
<td></td>
<td>0.3523</td>
<td>0.01</td>
<td>23.8</td>
</tr>
<tr>
<td></td>
<td>0.2816</td>
<td>0.0178</td>
<td>25.3</td>
</tr>
<tr>
<td></td>
<td>0.2183</td>
<td>0.0316</td>
<td>27.3</td>
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<td></td>
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<td>0.0562</td>
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<td>0.1649</td>
<td>0.1</td>
<td>30.6</td>
</tr>
<tr>
<td></td>
<td>0.1469</td>
<td>0.1778</td>
<td>32.0</td>
</tr>
<tr>
<td></td>
<td>0.1737</td>
<td>0.3162</td>
<td>32.9</td>
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<td></td>
<td>0.1861</td>
<td>0.5623</td>
<td>34.6</td>
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<td>1</td>
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<td>0.01</td>
<td>14.9</td>
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<tr>
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<td>0.4964</td>
<td>0.0178</td>
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<tr>
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<td>0.0316</td>
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<td></td>
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<td>0.1</td>
<td>24.7</td>
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<tr>
<td></td>
<td>0.2333</td>
<td>0.1778</td>
<td>26.6</td>
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<tr>
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<td>0.2124</td>
<td>0.3162</td>
<td>28.3</td>
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<tr>
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<td>0.2175</td>
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<td>1.2105</td>
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<td>0.0562</td>
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<tr>
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<td>0.3001</td>
<td>1</td>
<td>26.0</td>
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4.3. Viscoelastic modeling

We begin by fitting our measured creep curves to the Andrade viscoelastic model and comparing the inversion of the fit to our measured attenuation and shear modulus dispersion spectra, now plotted against angular frequency. We force an anelastic-strain time exponent of $n = \frac{1}{3}$ as it is: (1) appropriate to the high-temperature background portion of our attenuation spectra, and (2) is consistent with chemical diffusion in the presence of a continuously diminishing potential (e.g. grain growth). We present the results of these calculations in Figure 6. Each figure shows the fit of the Andrade model to the transient creep curve in the upper right corner, the parameters of the fit, and the comparison to the attenuation spectra (center), and shear modulus dispersion spectra (lower left). Consistent with past analyses, the Andrade model does a reasonable job of fitting the lower-frequency portion of the attenuation spectra. However, as described previously, the Andrade model is unable to reproduce the positive curvature in the high-frequency attenuation data. Additionally, the predicted shear moduli are much higher than the measured values, in some cases by a factor of two. Finally, as described in the previous section, the Andrade model is unable to describe the very low apparent shear modulus present in the “instantaneous” displacement.

Having established the limitations of the Andrade viscoelastic model, we now evaluate the composite viscoelastic model, Equation (17), against our experimental data. We fit the creep curve, now including the large initial jump, to Equation (17) and compare the predicted attenuation values to those observed in the dynamic tests using the different relaxation strengths as discussed in Section 2. The viscoelastic parameters corresponding to each fit are shown in Table 2. Figure 7 shows the result of inverting the fit of the composite creep function to the creep data obtained from our ol–39 vol.
Figure 6. Comparison of measured attenuation data (discrete points) to the predicted attenuation from the Andrade-model fits to the creep curve (cf. [3]) using a transient time exponent of $n = \frac{1}{2}$ at (a) 1300°C, (b) 1250°C, and (c) 1200°C, and evaluated using an unrelaxed shear modulus of 60 GPa. The Andrade model clearly provides an adequate description for the high-temperature background behavior in this specimen. The model predicts that the transition to steady-state creep occurs at slightly higher frequencies than observed in the dynamic tests (particularly well resolved in the 1250°C curve). However, the Andrade model is unable to reproduce the positive curvature and associated higher $Q_G^{-1}$ observed at the highest frequencies and so predicts shear moduli well in excess of those observed experimentally.
% opx sample at temperatures of 1200°C using $T_{ghs} = 0.1$ s using (a) $\Delta = 0.23$, (b) $\Delta = 0.82$, and (c) $\Delta = 1.91$ as appropriate to (a) the finite-element model of hexagonal grains, (b) Zener’s relaxation strength, and (c) the self-consistent model, respectively. For the sake of brevity we have not plotted the fits for $T = 1250°C$ and $T = 1300°C$. The viscoelastic parameters corresponding to each fit are shown in the bottom of each figure. In addition to the predicted attenuation spectra, each figure includes the time-domain fit of Equation (17) to the creep curve in the top right and the predicted shear modulus dispersion in the bottom right. The results of these analyses demonstrate that (1) the composite creep function can reproduce the larger-than-expected initial elastic strain, and (2) can yield low shear moduli values closer to those observed experimentally.

### Table 2. Results of fits to viscoelastic models for the peridotite (ol–39 vol. % opx) sample.

<table>
<thead>
<tr>
<th>Viscoelastic model</th>
<th>$T$ (°C)</th>
<th>$\Delta$</th>
<th>$A$ (Pa$^{-1}$ s$^{-15}$)</th>
<th>$\eta_{ss}$ (Pa s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Andrade model</td>
<td>1200</td>
<td>n/a</td>
<td>$5.10 \times 10^{-12}$</td>
<td>$3.42 \times 10^{12}$</td>
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<tr>
<td></td>
<td>1250</td>
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<td></td>
<td>1300</td>
<td></td>
<td>$2.7 \times 10^{-11}$</td>
<td>$1.87 \times 10^{11}$</td>
</tr>
<tr>
<td>Composite model</td>
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<td>0.23</td>
<td>$6.53 \times 10^{-12}$</td>
<td>$3.30 \times 10^{12}$</td>
</tr>
<tr>
<td>Finite-element</td>
<td>1250</td>
<td></td>
<td>$1.34 \times 10^{-11}$</td>
<td>$8.0 \times 10^{11}$</td>
</tr>
<tr>
<td>Ghahremani [18]; Equations (12) and (13)</td>
<td>1300</td>
<td></td>
<td>$2.92 \times 10^{-11}$</td>
<td>$1.86 \times 10^{11}$</td>
</tr>
<tr>
<td>Composite model</td>
<td>1200</td>
<td>0.82</td>
<td>$4.85 \times 10^{-11}$</td>
<td>$3.32 \times 10^{12}$</td>
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<td>Spherical grains</td>
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<td>$1.12 \times 10^{-11}$</td>
<td>$8.06 \times 10^{11}$</td>
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<td>$1.88 \times 10^{11}$</td>
</tr>
<tr>
<td>Composite model</td>
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<td>1.91</td>
<td>$5.97 \times 10^{-12}$</td>
<td>$3.36 \times 10^{12}$</td>
</tr>
<tr>
<td>Self-consistent model</td>
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<td>$1.30 \times 10^{-11}$</td>
<td>$8.10 \times 10^{11}$</td>
</tr>
<tr>
<td>Ghahremani [18]; Equation (15)</td>
<td>1300</td>
<td></td>
<td>$1.30 \times 10^{-11}$</td>
<td>$1.86 \times 10^{11}$</td>
</tr>
<tr>
<td>Composite model</td>
<td>1200</td>
<td>0.43</td>
<td>$6.37 \times 10^{-12}$</td>
<td>$3.38 \times 10^{12}$</td>
</tr>
<tr>
<td>Fourier series approx.</td>
<td>1250</td>
<td></td>
<td>$1.34 \times 10^{-11}$</td>
<td>$8.17 \times 10^{11}$</td>
</tr>
<tr>
<td>Raj and Ashby [19]; Equation (16)</td>
<td>1300</td>
<td></td>
<td>$2.80 \times 10^{-11}$</td>
<td>$1.86 \times 10^{11}$</td>
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</tbody>
</table>
Figure 7. Predictions of the composite viscoelastic model (Equation (17)) that includes both the high-temperature background and elastically accommodated GBS to our measured attenuation data at $T = 1200^\circ$C, for (a) $\Delta = 0.23$, (b) $\Delta = 0.82$, and (c) $\Delta = 1.91$. Each of these models successfully: (1) reproduce a larger-than-expected “instantaneous” strain, (2) produce shear moduli much lower than those of the Andrade model, and (3) produce a Debye peak. However, none of these choices for the relaxation strength successfully model the height of the Debye peak or successfully reproduce the shear moduli observed in the experiments.
Figure 8. Predictions of the composite viscoelastic model (Equation (17)) that includes both the high-temperature background and elastically accommodated GBS to our measured attenuation data at $T = (a) 1300\,^\circ C$, (b) $1250\,^\circ C$, and (c) $1200\,^\circ C$. The relaxation strength for elastically-accommodated GBS is taken as $\Delta = 0.43$ from [19]. The fit of Equation (17) to the creep data is shown at the top right. The predicted shear moduli are shown as an inset in the bottom left. $\Delta = 0.43$ yields attenuation and shear modulus dispersion that are largely consistent with those observed experimentally for the high-temperature background at moderate frequencies, the transition to steady state creep at low frequencies, and the incipient Debye peak at the highest frequencies.
case, the model provides both an excellent description of the attenuation spectra and an acceptable fit to the shear modulus dispersion.

5. Discussion
We contend, thus, that the Fourier series expression developed by Raj and Ashby [19] yields a relaxation strength that most accurately describes elastically-accommodated GBS in our samples. However, this conclusion should be approached cautiously for two reasons. First, our data are collected over a limited range of temperature and frequency and only reveal approximately half of the peak associated with elastically-accommodated GBS. Therefore, we cannot uniquely determine both the relaxation strength and relaxation time simultaneously. The attenuation spectra presented in Figure 4 and the fits presented in Figure 8 suggest that $T_{gb}$ decreases with increasing temperature as would be anticipated if GBS is a thermally activated process (i.e. $n_{gb} \propto \exp(-E/RT)$), where $E$ is an activation energy, and $R$ is the gas constant). The inability of our apparatus to directly determine $T_{gb}$ thus limits our ability to resolve the frequency shift associated with this process. However, for $T_{gb} = 0.10$ s, an unrelaxed shear modulus of 60 GPa, a grain size of 5 μm, and a grain boundary width of 1 nm, we estimate a grain boundary viscosity of $\sim 10^6$ Pa s [cf. Equation (7)] in our peridotite samples. This value is comparable to the grain boundary viscosities inferred by Jackson et al. [1] and by Xu et al. [32]. Note, however, that we do not allow $T_{gb}$ to vary with temperature in our analysis.

Secondly, a recent reanalysis of Raj and Ashby [19] has demonstrated that their Fourier series representation of the relaxation strength does not converge with increasing number of terms [33]. This observation has led to the speculation that the relaxation strength for elastically-accommodated GBS is related to the sharpness of multi-grain contacts. This speculation can be critiqued by experimental observations and analyses.

Xu et al. [32] conducted fixed-frequency, free-oscillation torsion pendulum experiments on melt-bearing olivine aggregates. At $f \sim 1$ Hz they observed a distinct Debye peak in their data which they attributed to elastically-accommodated GBS centered on $T \sim 1200^\circ$C whose height increased with higher melt fractions. Extrapolation to $\varphi = 0$ suggested a peak height of $\sim 0.1$ in their experiments, thus yielding $\Delta \sim 0.2$. This result is similar to the relaxation strength predicted from the finite element simulations of Ghahremani [18].

Jackson et al. [1] suggested that the emergence of a peak due to elastically-accommodated GBS is due to the presence of melt which rounds triple junctions, facilitates sliding, and yields a higher relaxation strength. They based this argument on the observation that their synthetically derived and melt-free olivine aggregates did not display a dissipation peak within the frequency band of their experiments. In contrast, samples fabricated from reconstituted San Carlos olivine always developed a small melt fraction and displayed a dissipation peak whose magnitude increased with increasing melt fraction. This phenomenon may also be attributable, however, to an increase in grain boundary viscosity effected by grain boundary segregation of incompatible elements (e.g. [25,34]), which pushes the Debye-peak associated with elastically-accommodated GBS into the frequency/temperature
regimes where experiments are tenable. Nevertheless, our experiment contains a melt fraction of 0.015; thus our experiments neither confirm nor refute the suggestion of Jackson et al. [1].

Subsequent work by Morris and Jackson [8] and have demonstrated that the effect of boundary sharpness on $\Delta$ is small. That being said, the model proposed here requires no melt and is based solely on solid-state phenomena. Also, peaks similar to those we observe have also been observed in unquestionably melt-free, single-phase aggregates of $\beta$-In$_3$Sn tested at ambient temperature [35]. Furthermore, the Zener model (high $\Delta$) accurately reproduced the relaxation strengths observed by Kê [12] in melt-free tests on polycrystalline aluminum.

Pezzotti [36] conducted free-oscillation torsion pendulum experiments on high-purity MgO and Al$_2$O$_3$ aggregates of varying grain sizes and observed dissipation peaks characteristic of elastically-accommodated GBS. He further observed a decrease in the relaxation strength with increasing grain size. At present, the relaxation strength for elastically-accommodated GBS is not thought to depend on grain size. However this observation suggests the possibility that the grain size in these experiments could serve as a proxy for some other microstructural change associated with annealing such as boundary curvature or boundary roughness. This suggestion is broadly consistent with the analyses of Jackson et al. [33] and Morris and Jackson [8] and may be responsible for the wide range of relaxation strengths observed experimentally. If this hypothesis is correct, our composite viscoelastic model is sufficiently flexible to incorporate this process.

We therefore suggest that the large initial transient in the creep curve is not due to sample gripping or electronics artifacts, but is rather a feature associated with elastically-accommodated GBS along a low-to-modest viscosity interface. While the limitations of the apparatus introduce considerable uncertainty into the measurement, the close correspondence between the measured data and the predicted data for both the creep curve and the attenuation/modulus-dispersion measurements suggest the model accurately describes the physical process of GBS as it contributes to the dynamic response.

6. Conclusions
We have developed a composite viscoelastic creep function that incorporates the combined effects of elastically-accommodated grain boundary sliding and transient diffusion creep. We demonstrate that this model can accurately reproduce the form of microcreep curves in a fine-grained peridotite specimen. Based on the parameters of the fit to the creep curves, we demonstrate that the transform of the model can accurately predict the measured shear attenuation and shear modulus dispersion spectra we observe in our material given an appropriate choice for the relaxation strength associated with elastically-accommodated GBS.

Acknowledgements
This work was supported financially, in part, by the National Science Foundation Division of Earth Sciences Program in Geophysics, Grant EAR-0609869; that support is
gratefully acknowledged. Additionally, the authors would like to thank Joe Bunton for his help with reconditioning the reciprocating torsion apparatus and David Goldsby for numerous stimulating discussions about the dynamics of grain boundary sliding.

Note

1. “Peridotite” is a rock name for the refractory mineral assemblage that constitutes the upper mantle of Earth (under ocean basins, ~10–400 km depth). The primary mineral is a ferromagnesian orthosilicate, olivine (“ol:” [Mg,Fe]2SiO4); less abundant minerals, but important mechanically (e.g. [5]), are a ferromagnesian metasilicate, orthopyroxene (“opx:” [Mg,Fe]SiO3), and a calcium-ferromagnesian metasilicate, clinopyroxene (“cpx:” [Ca,Mg,Fe]SiO3). Interpreting seismological data re the structure of the mantle is first-order dependent on the viscoelastic response of peridotite; developing responsible energetics models of plate tectonics is first-order dependent on the plastic response of peridotite. Application of these experimental rheology data to natural conditions requires, at minimum, extrapolation in grain size from the micrometer grain sizes used in laboratory studies to the millimeter-to-centimeter grain sizes observed in natural peridotite.

References