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Contrasting viscoelastic behavior of melt-free and melt-bearing olivine: Implications for the nature of grain-boundary sliding

Ian Jackson [a,*], Ulrich H. Faul [a], John D. Fitz Gerald [a], S.J.S. Morris [b]

[a] *Research School of Earth Sciences, Australian National University, Canberra, ACT, Australia*
[b] *Department of Mechanical Engineering, University of California, Berkeley, CA, USA*

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Abstract

Melt-free and basaltic (complex alumino-silicate) melt-bearing specimens of fine-grained polycrystalline olivine $(Mg_{0.9}Fe_{0.1})_2SiO_4$, tested at high temperature and low frequency in torsional forced oscillation and microcreep, display markedly different behavior. For the melt-bearing materials, superimposed upon the high-temperature background is a dissipation peak whose height varies systematically with melt fraction that is attributed to elastically accommodated grain-boundary sliding facilitated by the rounding of grain edges at melt-filled triple junctions. The melt-free materials display only the high-temperature background dissipation associated with transient diffusional creep—elastically accommodated sliding evidently being inhibited by their tight grain-edge intersections. These and similar observations for other ceramic materials require that the classic theory of grain-boundary sliding be revisited and suitably modified.

Keywords: Grain boundaries; Grain-boundary sliding; Mechanical spectroscopy; Partial melting

1. Introduction

The need to interpret pronounced variations in seismic wave speeds and attenuation in the Earth's upper mantle, dominated by the mineral olivine $(Mg,Fe)_2SiO_4$, motivated a recent laboratory study of the mechanical behavior of this material. We have fabricated fine-grained olivine polycrystals by hot-isostatic-pressing of both natural and synthetic (sol–gel) olivine precursors with and without added basaltic melt glass. The resulting specimens have been mechanically tested with torsional forced-oscillation and microcreep methods under conditions of simultaneous high pressure (200 MPa) and temperature (to 1300 °C).

The melt-free and melt-bearing specimens display qualitatively different behavior [1,2]. For the melt-free materials there is no evidence of a strain-energy dissipation peak. The behavior is that described as 'high-temperature background' [3]—a viscoelastic absorption band within which dissipation varies monotonically with temperature and oscillation period with concomitant dispersion of the shear modulus. In marked contrast, the melt-bearing materials invariably display a broad dissipation peak superimposed upon a melt-enhanced background.

These findings, which have close parallels in recent work on other fine-grained ceramic materials, help clarify the nature of the progressive transition at high temperature from elastic through anelastic to viscoelastic behavior and invite re-examination of the classic literature on grain-boundary sliding.

2. Mechanical spectroscopy of melt-free and melt-bearing polycrystalline olivine

2.1. Experimental and analytical procedures

Specimens recovered after hot-isostatic pressing at 200 MPa and 1200–1300 °C were precision ground to cylindrical shape, enclosed within metal foil (Fe or $Ni_{70}Fe_{30}$), and mounted between torsion rods of high-grade polycrystalline alumina for the torsional forced-oscillation and microcreep tests. The entire assembly is enclosed within a mild-steel sleeve sealed with O-rings at either end in order to exclude the argon pressure medium; this arrangement ensures that the optically flat interfaces within the assembly, across which the torque is transmitted, experience a normal stress equal to the confining pressure. The experimental

* Corresponding author. Tel.: +61 2 61252498; fax: +61 2 61258253.
E-mail address: ian.jackson@anu.edu.au (I. Jackson).

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apparatus and procedures have been described in detail elsewhere [4,5].

Both hot-pressed and mechanically tested specimens have been characterized by light and electron microscopy and infrared spectroscopy. In particular, grain-size distributions have been determined from lattice orientation maps derived by electron-backscattered diffraction in scanning electron microscope and grain-boundary regions have been imaged and subjected to energy-dispersive chemical analysis in a 300 kV transmission electron microscope.

Mechanical testing of each specimen started with annealing at the highest temperature (usually either 1200 or 1300 °C) and 200 MPa pressure—in order to heal any thermal microcracks and optimize mechanical coupling between torsion rods and specimen. A progressive reduction in compliance and dissipation towards stable values was usually observed over several tens of hours. Once such asymptotic behavior had been established, the assembly was slowly cooled through 50 or 100 °C intervals to room temperature. At each stage, isothermal forced-oscillation tests at selected periods of 1–100 (or 1000 s) and microcreep measurements were performed. Maximum stress amplitudes < 0.3 MPa correspond to maximum shear strains $<5 \times 10^{-5}$—demonstrated to be within the realm of linear viscoelasticity.

2.2. Melt-free olivine specimens

The strain-energy dissipation Q^{-1} (and shear modulus G) data from forced-oscillation tests on melt-free specimens display the monotonic variation with oscillation period and temperature characteristic of the high-temperature background (e. g., Fig. 1a). The Q^{-1} data are well described by an Andrade-pseudoperiod model in which the 'pseudoperiod' master variable

$$X_B = T_o\left(\frac{d}{d_r}\right)^{-m} \exp\left[\left(\frac{-E_B}{R}\right)\left(\frac{1}{T} - \frac{1}{T_r}\right)\right], \quad (1)$$

(with $m = 1$, cf. [6] replaces period T_o in the complex compliance given by Laplace transform of the well-known Andrade creep function

$$J(t) = J_U + \beta t^n + \frac{t}{\eta}. \quad (2)$$

The variables T_r and d_r are suitable reference values of temperature and grain size respectively. This approach accommodates both the anelastic and viscous components of the strain (evident in complementary torsional microcreep records). For essentially anelastic conditions, not too far in period-temperature-grain size space beyond the breakdown of elastic behavior, where the Andrade transient creep term dominates over the viscous term, Q^{-1} varies approximately as βT_o^n. For the Andrade-pseudoperiod model this becomes $Q^{-1} \sim [T_o d^{-m} \exp(-E_B/RT)]^n$. Least-squares fitting of data for a suite of three melt-free specimens of mean grain size 3–23 μm with m fixed at 1 yielded optimal values of $n = 0.28$ and $E_B = 400\ \text{kJ mol}^{-1}$ [1].

2.3. Melt-bearing olivine polycrystals

For the melt-bearing specimens the observed dissipation reflects the superposition of a broad dissipation peak upon a monotonically frequency and temperature-dependent background (Fig. 1b). It is evident that the dissipation peak moves systematically to longer period with decreasing temperature—from <1 s at 1300 °C to ∼300 s at 1000 °C.

The broad dissipation peak has been modeled by a Gaussian function of the form

$$Q_P^{-1} = B \exp\left(\frac{-z^2}{2}\right); \quad z = \frac{(\ln X_P - \mu)}{\sigma}; \quad B = B_o \phi^l. \quad (3)$$

The peak pseudoperiod X_P is defined analogously to X_B above and ϕ is the alumino-silicate melt fraction. Dissipation data for temperatures of 1000–1300 °C and oscillation periods of 1–1022 s for a suite of six melt-bearing olivine specimens, of mean grain size 7–52 μm and melt fraction 0.0001–0.037, have been successfully represented as the sum of a Gaussian pseudoperiod peak and the Andrade-pseudoperiod background described above [1].

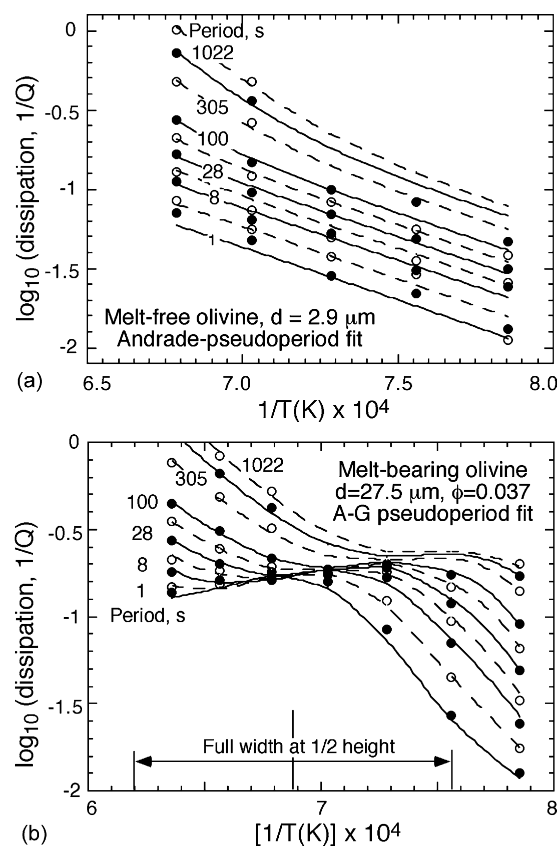

Fig. 1. Contrasting patterns in representative dissipation data (plotting symbols) from torsional forced-oscillation tests on melt-free (a) and melt-bearing (b) olivine polycrystals. The background-only behavior of the melt-free material is fitted to an Andrade-pseudoperiod fit (solid and broken curves for alternate oscillation periods) whereas the background-plus-peak dissipation of the melt-bearing specimen is similarly represented by an Andrade–Gaussian-pseudoperiod model (A–G) as described in the text (redrawn after [1]).