Rheology of synthetic anorthite-diopside aggregates: Implications for ductile shear zones

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Received 14 September 2004; revised 2 March 2005; accepted 18 March 2005; published 27 July 2005.

[1] We investigated the high-temperature creep strength of fine-grained anorthite-diopside rocks at temperatures ranging from 1323 K to 1523 K and at 300 MPa confining pressure in a Paterson-type gas-medium deformation apparatus. Flow stress varied between 20 and 450 MPa resulting in strain rates between $6.1 \times 10^{-7} \text{ s}^{-1}$ and $7.5 \times 10^{-4} \text{ s}^{-1}$. Pure diopside and anorthite samples were hot pressed from crushed natural single crystals and glass powders, respectively. Two-phase samples were produced by hot isostatic pressing of mechanically mixed powders of anorthite glass with 25, 50 and 75 vol % diopside particles. Arithmetic mean grain size of the anorthite matrix is $d_{An} \approx 3.5 \ \mu m$. Three different ranges of diopside particle size were used: $d_{Di} < 25 \ \mu m$, $<35 \ \mu m$, and $<45 \ \mu m$. Water content of as is samples was about 0.05 ± 0.02 wt % H₂O, and predried samples contain about 0.004 ± 0.001 wt % H₂O. At experimental conditions, as is samples are assumed to be water saturated. Water content of predried samples is about 3 times less than that of starting diopside single crystals. The specimens contain about 1 vol % glass located at fluid inclusions and some multiple grain junctions. Two-grain boundaries examined by high-resolution transmission electron microscopy did not show amorphous layers to a resolution of 1 nm. At experimental conditions, pure diopside aggregates are about 2-3 orders of magnitude stronger than pure anorthite samples for as is and predried specimens, respectively. In general, strength of the two-phase aggregates increases with increasing diopside content but remains between isostress and isostrain rate bounds. Aggregate strengths predicted from continuum mechanics models are in good agreement with the experimental data for dilute diopside particle mixtures and highvolume fractions, when diopside particles form a load-bearing framework. At low stresses (<100-200 MPa) the stress exponent is $n \approx 1$, suggesting diffusion-controlled creep. At higher stresses, mechanical data and microstructures suggest that samples deformed in the transition region between diffusion-controlled creep and dislocation creep. For pure anorthite and diopside aggregates deforming in dislocation creep we estimated stress exponents of $n \approx 3$ and $n \approx 5.5$, respectively. For the two-phase aggregates, n is between n \approx 3 and n \approx 5, depending on diopside content. At low stresses, deformation microstructures indicate load transfer from a weak anorthite matrix to stronger diopside particles. Creep activation energies for pure diopside and anorthite mixtures range from 286 kJ mol⁻¹ for wet anorthite deformed at low stresses to 691 kJ mol⁻¹ for dry diopside deformed at high stresses. Activation energies of two-phase mixtures are between or close to those of the end-members. As is samples have significantly lower activation energies than predried samples.

Citation: Dimanov, A., and G. Dresen (2005), Rheology of synthetic anorthite-diopside aggregates: Implications for ductile shear zones, *J. Geophys. Res.*, *110*, B07203, doi:10.1029/2004JB003431.

1. Introduction

[2] The prevailing view of the upper limit strength of the continental lithosphere assumes a relatively weak lower crust situated in between strong upper crust and mantle. This view is largely based on earthquake depth distribution and predictions from laboratory measurements of rock

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strength [Brace and Kohlstedt, 1980; Kirby, 1980; Chen and Molnar, 1983; Kohlstedt et al., 1995]. Relatively low viscosities (about 10^{18} – 10^{19} Pa) for the lower continental crust are also inferred, for example, from relating the topographic relief of mountain belts and orogenic plateaus to crustal strength [Royden, 1996; Royden et al., 1997]. However, recent reassessment of earthquake focal depth and elastic thickness in some continental areas suggest that the lower crust may indeed be stronger than the mantle lithosphere [Maggi et al., 2000; Jackson, 2002]. This contention is supported by independent evidence from modeling postseismic surface deformation [Vergnolle et al., 2003; Pollitz et al., 2001]. The current controversy highlights that lower crustal viscosities may show significant spatial-temporal variations depending, for example, on transient changes in strain rate or temperature distribution. In particular, recent laboratory studies of synthetic rocks suggest that variations in mineralogical composition, water and impurity content will significantly affect rock strength allowing for a broad range in viscosities at given thermodynamic boundary conditions [Dimanov et al., 1999; Rybacki and Dresen, 2000; Mei and Kohlstedt, 2000a, 2000b; Xiao et al., 2002; Herwegh et al., 2003; Chen and Kohlstedt, 2003; Rybacki et al., 2003].

[3] Field studies of exposed sections of the continental lower crust frequently show highly localized shear zones accommodating most of the deformation [Rutter and Brodie, 1988, 1992; Kruse and Stünitz, 1999; Kenkmann and Dresen, 2002]. Progressive strain partitioning into mylonite zones is typically associated with a significant grain size reduction from millimeter-scale in the host rock to micron-scale in ultramylonites [White et al., 1980; Austrheim et al., 1997]. The dominant deformation mechanism operating in these very fine-grained (\sim 5-50 µm) shear zone rocks may be diffusion-controlled grain size-sensitive flow as often suggested from field and laboratory studies [Boullier and Gueguen, 1975; Allison et al., 1979; Jensen and Starkey, 1985; White and Mawer, 1986; Behrmann and Mainprice, 1987; Rutter and Brodie, 1992; Kenkmann and Dresen, 2002; Dimanov et al., 1999; Rybacki and Dresen, 2000].

[4] In the lower continental crust feldspars and pyroxenes are the most abundant minerals [Deer et al., 1976]. On the basis of microstructural observations, deformation regimes of feldspar-bearing rocks are well investigated for a broad range in pressures and temperatures in experiments and in nature [Tullis and Yund, 1985; Dell'Angelo et al., 1987; Tullis and Yund, 1987; Tullis, 1990]. Fewer studies exist on deformation microstructures in pyroxenites [Kirby and Kronenberg, 1984; Mauler et al., 2000]. Constitutive equations are now available for high-temperature creep of synthetic feldspar aggregates at nominally dry conditions, in the presence of water, and for partially molten rocks [Dimanov et al., 1998, 1999, 2000; Rybacki and Dresen, 2000]. Flow laws also exist for pure synthetic and natural pyroxene aggregates [Bystricky and Mackwell, 2001; Chen and Kohlstedt, 2003; Dimanov et al., 2003] and dry diabase [Mackwell et al., 1998].

[5] Experimental studies of polyphase rocks are still scarce. Two-phase aggregates show a rich mechanical behavior depending on second-phase content and thermodynamic conditions, but in most cases their strength is

rigorously bounded by the uniform stress and uniform strain rate bounds [Tullis et al., 1991]. This fact is supported by laboratory findings on mixtures of calcite-quartz [Siddiqi, 1997; Dresen et al., 1998; Rybacki et al., 2003], anorthitequartz [Xiao et al., 2002], and forsterite-enstatite [Ji et al., 2001]. However, in addition to the purely mechanical interactions of the individual phases, other factors may dominate deformation of polyphase rocks. For example, metamorphic reactions between different minerals leading to fine-grained reaction products may significantly reduce rock strength [White and Knipe, 1978; Stünitz and Tullis, 2001]. Second-phase particles or fluid inclusions may stabilize a small grain size and promote weakening of the rock matrix [Olgaard, 1990] and in some cases polyphase aggregates are effectively weaker than predicted by the uniform stress bound [Wheeler, 1992; Bruhn et al., 1999; McDonnell et al., 2000].

[6] No robust constitutive laws are available for feldsparpyroxene rocks that commonly occur in granulite-facies metabasites at the base of the Earth's crust. The purpose of this study is to investigate the mechanical behavior of feldspar-pyroxene rocks deforming in the diffusion and dislocation creep regimes. We study the effect of mineralogical volume fractions, grain size and water trace content on viscosity of the aggregates. We present constitutive laws for the polyphase mixtures and compare our results to the aggregate strength predicted from continuum mechanics and mixing models.

2. Experimental Procedures

2.1. Starting Materials

[7] Pure anorthite (An) specimens were fabricated from a $CaAl_2Si_2O_8$ glass powder (Schott Glaswerke, Mainz). The impurity content was <0.1 wt %. Grain size of the starting powder was <60 μ m. Pure diopside (Di) samples were prepared from crystalline powders obtained by crushing clear, gem-quality single crystals (Gebrüder Bank GmbH, Idar-Oberstein). The diopside chemical composition in oxide wt % was determined using electron microprobe (CAMECA SX-100). The corresponding structural formula is:

Ca_{1.001} Na_{0.025} Mg_{0.885} Fe_{0.051} Mn_{0.026} Al_{0.021} Ti_{0.002} Si_{1.989} O₆,

Fe is the major impurity with 2.6 at%. Crushed single crystal powders were immersed in alcohol, ground in an agate mortar and subsequently sieved. The diopside powders were separated into three different grain size ranges: $d_{Di} < 25 \ \mu m$, $<35 \ \mu m$ and $<45 \ \mu m$. Anorthite and diopside powders were mechanically mixed in alcohol using an agate mortar producing two-phase mixtures with diopside fractions of 25, 50 and 75 vol %, respectively. These two-phase mixtures are referred to as An25Di_d, An50Di_d and An75Di_d, where d indicates the maximum diopside grain size (i.e., 25, 35 or 45 μm).

[8] All powders were first cold pressed in steel cans 20 mm in length and 10 mm in diameter [*Dimanov et al.*, 1999]. To fabricate samples with varying water content, green bodies were hot isostatically pressed as is or after drying in CO/CO_2 for 72 hours at 0.1 MPa and 1223 K. Pure anorthite specimens and two-phase aggregates were



Figure 1. Scanning electron microscope (SEM) micrographs of thermally etched samples. (a) Close view of sample An25Di₃₅. The central diopside particle is labeled Di, and anorthite matrix is labeled An. The anorthite grains are lath-shaped with a mean grain size $d_{An} = 3.5 \mu m$. The largest diopside particles are elongated, but the finer-grained diopside particles are equant. Samples (b) An25Di₃₅ (coarser particles, $d_{Di} < 35 \mu m$) and (c) An25Di₂₅ (finer particles, $d_{Di} < 25 \mu m$). The larger diopside grains have higher aspect ratios and are less closely spaced than fine particles. (d) Close view of diopside particle cluster in An50Di₃₅. At high diopside fractions such clusters or frameworks of contacting grains are frequently observed.

hot isostatically pressed for 5 hours in a gas-medium Paterson-type apparatus at a confining pressure of 300 MPa. Densification of the glass powders was allowed for 30 min at 1123 K. Temperature was raised to 1373 K and 1423 K at a rate of 20 K min⁻¹ for as is and predried samples, respectively. Densities of hot-pressed samples were within 1.5% of theoretical. Producing low-porosity pure diopside samples required hot pressing for 24 hours at 1423 K and 300 MPa pressure. The resulting single- and two-phase aggregates are referred to as AnY and Di_dY and AnXDi_dY. In this sample labeling convention Y denotes dry (D) or wet (W) samples, X indicates diopside content (i.e., 25, 50 or 75 vol %), and d indicates the maximum diopside grain size (i.e., 25, 35 or 45 μ m).

2.2. Grain Size

[9] Thick sections of specimens were ground and polished to a $0.3 \ \mu m$ finish. To reveal grain boundaries, samples were thermally etched for 48 hours at 1373–1423 K at atmospheric pressure (Figure 1). Grain size distribution was determined from scanning electron microscope (SEM) (ZEISS DSM 962) micrographs using the linear intercept method.

[10] Arithmetic mean intercept length was converted to average grain size using correction factors of 1.9 and 1.78 for anorthite and diopside, respectively, to account for the difference in grain shape [*Dimanov et al.*, 2003] (Figure 1). Grain size distribution of pure anorthite aggregates is narrow and lognormal (Figure 2a). The mean grain size is about 3.5 μ m. For hot-pressed pure diopside aggregates, we obtained broad grain size distributions (Figure 2b), reflecting the initial starting particle fractions <25 μ m, <35 μ m and <45 μ m and subsequent particle crushing during cold and hot isostatic pressing. For the two-phase mixtures, average grain size of the anorthite matrix remained unaffected by the diopside



Figure 2. Grain size distribution of pure anorthite and diopside. (a) Anorthite grain size distribution and average grain size of 3.8 μ m for one specific measurement. The mean grain size for anorthite obtained from several measurements is d_{An} = 3.5 μ m. (b) Grain size distribution of diopside particles, broad and bimodal. Maximum diopside grain size is d_{Di} < 45 μ m for this sample.

particle volume fraction. We suggest that the anorthite grain size is stabilized by abundant small pores [*Olgaard* and Evans, 1988; Rybacki and Dresen, 2000].

[11] Distribution of diopside inclusions in the anorthite matrix is relatively homogeneous. However, for the finergrained diopside particles ($<25 \mu$ m,), average spacing is smaller and clustering was more frequently observed than for the larger-grained particles (Figures 1b and 1c). For diopside volume fractions of 50 vol % and 75 vol % numerous particles are interconnected and clusters are common (Figure 1d).

2.3. Water Content

[12] The glass and crystal powders are hygroscopic adsorbing significant quantities of water. To determine bulk water content, Fourier-transformed infrared (FTIR) absorbance spectra were measured from polished 150 µm thick sections of hot-pressed samples. The spectrometer (Bruker IFS-66v) is equipped with a microscope with minimum spot size of about 60 µm. Line scans oriented parallel and perpendicular to the long specimen axis suggest that the water content is homogeneous on the sample scale. For quantitative estimates Beer-Lambert's law was used with molar absorptivities for plagioclase [Beran, 1987] and diopside [Bell et al., 1995]. For the two-phase aggregates water content is estimated using a volume-weighted average of the absorptivities. In general, as is (wet, W) samples contain about 0.05 ± 0.02 wt % H₂O, but predried (dry, D) samples contain about 0.004 ± 0.001 wt % H₂O. The absorbance spectra show broad bands superimposed on distinct peaks. The broad absorption bands display a maximum between wave numbers 3400–3600 cm⁻¹ indicating molecular water or hydroxyl possibly contained in fluid inclusions [Hofmeister and Rossman, 1985]. The sharp peak located at wave number 3640 cm⁻¹ is characteristic of substitutional OH⁻ groups in the crystalline structure of diopside [Wilkins and Sabine, 1973].

[13] The water content of the diopside starting crystals was determined prior to crushing from doubly polished 1 mm thick sections. We observed two absorption peaks at 3640 cm⁻¹ and 3530 cm⁻¹. A total water content of 0.012 wt % H₂O was estimated using the calibration of Bell et al. [1995], indicating that the diopside crystals are fully saturated at room pressure before crushing [Ingrin et al., 1995]. Recent studies of hydrogen solubility in natural diopside at high pressure (500 MPa) and temperature (1273-1373 K) [Bromiley et al., 2004] suggest that at our experimental conditions (300 MPa and 1323-1523 K) as is (wet) samples may be considered water saturated. Although some diffusive dehydration may be expected in the gas apparatus, FTIR measurements of as is hot pressed and deformed samples do not evidence significant water loss during deformation runs of up to 2 days.

2.4. Microstructures of Hot Isostatically Pressed Samples

[14] Microstructures were investigated using transmission electron microscopy (TEM, Philips CM 200 Twin) equipped with an energy dispersive X-ray (EDX) detector. Dislocation densities in hot-pressed anorthite are typically less than 10^{11} m⁻² and never exceed 10^{12} m⁻². Multiple growth twins are very common. In hot-pressed diopside, dislocation densities vary significantly between grains but are essentially between $<10^{12}-10^{13}$ m⁻². In diopside mechanical twins are observed in a few samples. Dislocation densities in two-phase aggregates are about $\sim 10^{12}$ m⁻² and very heterogeneous with higher densities typically in and around the diopside inclusions. Intracrystalline fluid inclusions (20-50 nm diameter) are present in most samples, but are more common in wet specimens. Intercrystalline fluid inclusions are also frequent in wet diopside samples (Figure 3a). We suggest that the presence of abundant fluid inclusions in wet hot-pressed samples indicates water saturation and a water activity $a_{H,O} \approx 1$.



Figure 3. Bright-field transmission electron microscopy (TEM) micrographs of hot-pressed samples. (a) Grain boundary in wet pure diopside sample decorated with numerous fluid inclusions. Amorphous film results from radiation damage. Several faceted inclusions indicate formation in a crystalline structure. We found grain boundaries in wet specimens containing fluid inclusions to be more readily beam damaged than those in dry samples, suggesting high concentration of hydrous defects in grain boundaries. (b) Phase boundary between anorthite and diopside free of melt to a resolution of <1 nm.

[15] Fluid inclusions in anorthite are often surrounded by a 10-50 nm thick shell of amorphous material, possibly indicating synthetic glass relicts or local melting [*Dimanov et al.*, 2000]. EDX analysis shows that the glass is depleted in aluminium and enriched in silicon with respect to pure anorthite. Glass with similar composition is also frequently found in triple junctions. Irrespective of diopside content of the specimens, two-grain boundaries and phase boundaries are melt-free to a resolution of <1 nm (Figure 3b). Total melt content of wet samples is estimated to be $\ll 1$ vol % and dry samples are free of melt.

2.5. Deformation Experiments

[16] Some 450 creep tests were performed stepwise on 24 samples at constant temperature and constant load in a Paterson-type gas medium deformation apparatus. Samples contained in an iron can used for cold pressing were placed in an iron sleeve between alumina spacers. Samples were separated from spacers by thin iron foils to avoid direct contact. Differential stresses varied between 20 MPa and about 500 MPa resulting in strain rates between 6.14 \times $10^{-8}~s^{-1}$ and 7.45 \times $10^{-4}~s^{-1}$. Confining pressure was kept constant at 300 MPa. For single steps the load was maintained constant within ±0.02 kN resulting in a variation of axial stress of <1 MPa. Displacement was measured using an internal linear-variable displacement transducer (LVDT) with an accuracy of 2 µm. Finite strain for individual steps was <3% and steady state secondary creep was reached at about 0.5-1% axial strain. The maximum axial strain for a specimen was 25%. Temperature was monitored using a Pt/Pt-10%Rh thermocouple within 5 mm of the specimen top end. The temperature gradient along the sample axis was <5 K.

[17] In general, samples were deformed following hot pressing. However, some specimens were removed from the pressure vessel after hot pressing and sample dimensions determined while still jacketed. Diameter of cylindrical samples was 9.2-9.5 mm and constant within 0.2 mm. Sample length was between 16.5 to 18 mm. The resulting uncertainties in differential stress and strain rate are <7% and <5%, respectively.

3. Results

3.1. Mechanical Data

[18] We assume that total creep strain of the specimens is accommodated by different deformation mechanisms operating in parallel and the mechanical data (Tables 1 and 2) were fit to a power law equation of the form:

$$\frac{d\varepsilon}{dt} = A_{gss}\sigma^{n_{gss}}d^{-m}e^{-\frac{Q_{gss}}{RT}} + A_{gsi}\sigma^{n_{gsi}}e^{-\frac{Q_{gss}}{RT}}$$
(1)

where $\frac{d\varepsilon}{dt}$ is strain rate, A is a constant, σ is differential stress, n is stress sensitivity of the strain rate, d is grain size, m is grain size sensitivity, Q is activation energy, R is molar gas constant and T is absolute temperature. The subscripts gss and gsi indicate the grain size–sensitive and grain size–insensitive components of the total strain rate $\frac{d\varepsilon}{dt}$. To fit the data d^{-m} was incorporated in A_{gss} and n_{gss} was set equal to 1. The results are reported in Tables 3a and 3b for wet and dry materials, respectively. For reference we include the results for pure anorthite aggregates from *Rybacki and Dresen* [2000] corresponding to our experimental conditions.

3.1.1. Stress Exponents and Particle Strengthening

[19] For the range of stresses and strain rates covered with our experiments, we find two dominant deformation mechanisms indicated by stress exponents of $n \approx 1$ at stresses <100-200 MPa and $n \approx 3-5.5$ at elevated stresses (Figures 4 and 5 and Tables 3a and 3b). The data indicate dominantly linear-viscous diffusion-controlled creep at low stresses and power law or dislocation creep at high stresses, respectively. The transition stress is substantially higher for

 Table 1. Creep Data for Dry Samples

Table 1. (continued)

| Temperature, K | Log σ, MPa | $\text{Log }\epsilon',s^{-1}$ | Temperature, K | Log σ, MPa | $\text{Log }\epsilon', \text{s}^{-1}$ |
|----------------|-------------------------------|-------------------------------|----------------|--------------------------------|--|
| | Sample Di ₃₅ #1 | | 1523 | 1.491 | -5.675 |
| 1423 | 1.881 | -5.848 | 1523 | 1.699 | -5.453 |
| 1423 | 2.068 | -5.815 | 1523 | 1.908 | -5.222 |
| 1423 | 2.204 | -5.757 | 1523 | 2.086 | -5.050 |
| 1423 | 2.299 | -5.719 | 1523 | 2.207 | -4.873 |
| 1423 | 2.444 | -5.488 | 1523 | 2.307 | -4.716 |
| 1423 | 2.303 | -5.879 | 1523 | 2.384 | -4.552 |
| 1423 | 1.763 | -6.943 | 1523 | 2.430 | -4.286 |
| 1423 | 1.949 | -6.936 | | | |
| 1423 | 2.352 | -6.418 | | Sample An75Di ₃₅ #2 | |
| 1473 | 1.813 | -6.300 | 1423 | 1.778 | -6.631 |
| 1473 | 2.009 | -6.094 | 1423 | 2.000 | -6.377 |
| 1473 | 2.161 | -5.924 | 1423 | 2.152 | -6.146 |
| 1473 | 2.350 | -5.558 | 1423 | 2.369 | -5.830 |
| 1473 | 2.474 | -5.231 | 1423 | 2.450 | -5.577 |
| 1523 | 1.763 | -5.987 | 1423 | 2.522 | -5.358 |
| 1523 | 1.978 | -5.783 | 1423 | 1.447 | -6.827 |
| 1523 | 2.130 | -5.584 | 1473 | 1.462 | -6.199 |
| 1523 | 2.281 | -5.236 | 1473 | 1.914 | -5.744 |
| 1523 | 2.425 | -4.822 | 1473 | 2.083 | -5.495 |
| 1523 | 1.914 | -5.812 | 1473 | 2.199 | -5.322 |
| 1523 | 2.124 | -5.590 | 1473 | 1.996 | -5.742 |
| 1523 | 2.294 | -5.260 | 1473 | 2.303 | -5.197 |
| 1523 | 2.442 | -4.759 | 1473 | 2.417 | -4.972 |
| 1523 | 2.525 | -4.442 | 1523 | 1.301 | -5.799 |
| 1523 | 2.360 | -5.061 | 1523 | 1.785 | -5.266 |
| 1523 | 2.493 | -4.562 | 1523 | 2.004 | -5.033 |
| | | | 1523 | 2.143 | -4.824 |
| | Sample Di35#2 | | 1523 | 2.279 | -4.542 |
| 1423 | 2.127 | -6.676 | 1523 | 2.378 | -4.407 |
| 1423 | 2.288 | -6.456 | 1523 | 2.400 | -4.258 |
| 1423 | 2.446 | -6.196 | | | |
| 1423 | 2.547 | -5.842 | | Sample An50Di ₃₅ #1 | |
| 1423 | 2.601 | -5.703 | 1423 | 1.301 | -5.682 |
| 1423 | 1.875 | -7.212 | 1423 | 1.602 | -5.246 |
| 1473 | 1.892 | -6.372 | 1423 | 1.778 | -5.059 |
| 1473 | 2.057 | -6.149 | 1423 | 1.903 | -4.939 |
| 1473 | 2.193 | -5.979 | 1423 | 2.000 | -4.818 |
| 1473 | 2.326 | -5.652 | 1423 | 2.086 | -4.708 |
| 1473 | 1.362 | -6.914 | 1423 | 1.491 | -5.386 |
| 1473 | 2.413 | -5.440 | 1373 | 1.892 | -5.575 |
| 1473 | 2.502 | -5.136 | 1373 | 2.072 | -5.397 |
| 1473 | 2.574 | -4.848 | 1373 | 1.301 | -6.224 |
| 1473 | 2.618 | -4.684 | 1373 | 1.591 | -5.967 |
| 1523 | 1.763 | -5.851 | 1473 | 1.462 | -4.883 |
| 1523 | 1.903 | -5.780 | 1473 | 1.255 | -5.194 |
| 1523 | 1.978 | -5.652 | 1473 | 1.699 | -4.747 |
| 1523 | 2.140 | -5.526 | 1473 | 1.839 | -4.524 |
| 1523 | 2.255 | -5.312 | 1473 | 1.987 | -4.485 |
| 1523 | 2.344 | -5.253 | 1473 | 2.143 | -4.305 |
| 1523 | 2.447 | -4.780 | 1473 | 2.301 | -4.040 |
| 1523 | 2.500 | -4.580 | 1473 | 2.356 | -3.827 |
| 1523 | 2.507 | -4.368 | 1473 | 2.389 | -3.684 |
| 1523 | 2.551 | -4.191 | | | |
| | | | | Sample An50Di ₃₅ #2 | |
| | Sample An75Di ₃₅ # | | 1423 | 1.591 | -5.265 |
| 1423 | 1.602 | -6.593 | 1423 | 1.892 | -4.963 |
| 1423 | 1.903 | -6.242 | 1423 | 1.255 | -5.788 |
| 1423 | 2.079 | -6.066 | 1423 | 1.681 | -5.228 |
| 1423 | 2.255 | -5.812 | 1423 | 1.959 | -4.967 |
| 1423 | 2.380 | -5.580 | 1423 | 2.090 | -4.804 |
| 1423 | 2.484 | -5.380 | 1423 | 2.204 | -4.629 |
| 1423 | 1.792 | -6.402 | 1423 | 2.301 | -4.491 |
| 1423 | 1.301 | -6.992 | 1423 | 2.393 | -4.353 |
| 1473 | 1.491 | -6.197 | 1373 | 1.908 | -5.606 |
| 1473 | 1.778 | -5.710 | 1373 | 2.149 | -5.334 |
| 1473 | 1.903 | -5.599 | 1373 | 2.310 | -5.104 |
| 1473 | 2.009 | -5.517 | 1373 | 1.415 | -6.368 |
| 1473 | 2.149 | -5.349 | 1373 | 1.568 | -5.963 |
| 1473 | 1.623 | -6.106 | 1373 | 1.763 | -5.772 |
| 14/3 | 2.262 | -5.212 | 1473 | 1.447 | -4.848 |
| 14/3 | 2.386 | -4.954 | 1473 | 1.690 | -4.648 |
| 14/3 | 2.48 | -4.754 | 14/3 | 1.176 | -5.367 |

| Table 1. (continue) | d) | |
|---------------------|--------------------------------|------------|
| Temperature, K | Log σ, MPa | Log ε′, s⁻ |
| 1473 | 1.908 | -4.483 |
| 1473 | 2.097 | -4.341 |
| 1473 | 2.220 | -4.168 |
| 1473 | 2.303 | -3.924 |
| | Sample An50Di ₄₅ #1 | |
| 1373 | 1.531 | -5.967 |
| 1373 | 1.763 | -5.923 |
| 1373 | 2.017 | -5.813 |
| 1373 | 2.017 | -6.029 |
| 1373 | 2.146 | -5.867 |
| 13/3 | 2.279 | -5.730 |
| 13/3 | 2.354 | -5.581 |
| 1373 | 2.441 | -5.585 |
| 1423 | 1.602 | -5.197 |
| 1423 | 1.002 | -5.613 |
| 1423 | 1 903 | -5 461 |
| 1423 | 2.079 | -5.260 |
| 1423 | 2.204 | -5.035 |
| 1423 | 2.265 | -4.954 |
| 1423 | 2.310 | -4.894 |
| 1423 | 2.358 | -4.628 |
| 1423 | 2.394 | -4.481 |
| 1473 | 1.477 | -5.332 |
| 1473 | 1.699 | -5.051 |
| 1473 | 1.845 | -4.975 |
| 14/3 | 2.000 | -4.704 |
| 14/3 | 2.079 | -4.308 |
| 1473 | 2.140 | -4.477 |
| 1473 | 2.204 | -4.395 |
| 1473 | 2.200 | -4 158 |
| 1175 | 2.501 | |
| | Sample An25Di ₃₅ #1 | |
| 1423 | 1.477 | -5.071 |
| 1423 | 1.602 | -4.943 |
| 1423 | 1.699 | -4.845 |
| 1423 | 2 000 | -4.072 |
| 1423 | 2.000 | -4 363 |
| 1373 | 1.477 | -5.697 |
| 1373 | 1.699 | -5.401 |
| 1373 | 1.903 | -5.185 |
| 1373 | 2.083 | -5.022 |
| 1373 | 2.207 | -4.893 |
| 1473 | 1.477 | -4.419 |
| 1473 | 1.699 | -4.264 |
| 1473 | 1.892 | -4.089 |
| 1473 | 2.079 | -3.955 |
| | Sample An25Di35#2 | |
| 1423 | 1.477 | -5.052 |
| 1423 | 1.602 | -4.932 |
| 1423 | 1.699 | -4.799 |
| 1423 | 1.778 | -4.690 |
| 1423 | 1.903 | -4.578 |
| 1423 | 2.079 | -4.399 |
| 1373 | 1.002 | - 3.490 |
| 1373 | 1.778 | -5.284 |
| 1373 | 2 000 | -5.057 |
| 1373 | 2.079 | -4.967 |
| 1473 | 1.602 | -4.180 |
| 1473 | 1.301 | -4.559 |
| 1473 | 1.778 | -4.121 |
| 1473 | 1.845 | -4.079 |
| 1473 | 1.954 | -4.012 |
| 1473 | 2.000 | -3.936 |
| | Sample An25Dise#3 | |
| 1423 | 1.477 | -5.135 |
| 1423 | 2.303 | -3.951 |

| 7 | of 24 | |
|---|-------|--|

| Temperature, K | Log σ, MPa | $\text{Log } \varepsilon', \text{s}^{-1}$ |
|----------------|--------------------------------|--|
| 1423 | 2.401 | -3.836 |
| 1423 | 2.490 | -3.697 |
| 1373 | 1.903 | -5.283 |
| 1373 | 2.146 | -5.015 |
| 1373 | 2.258 | -4.860 |
| 1373 | 2.301 | -4.783 |
| 1373 | 2.342 | -4.715 |
| 1373 | 2.398 | -4.593 |
| 1373 | 2.450 | -4.488 |
| 1373 | 2.508 | -4.352 |
| 1373 | 2.545 | -4.293 |
| 1373 | 2.610 | -4.112 |
| | Sample An25Di ₄₅ #1 | |
| 1373 | 1.580 | -5.210 |
| 1373 | 1.996 | -4.688 |
| 1373 | 2.215 | -4.465 |
| 1373 | 2.394 | -4.258 |
| 1373 | 2.529 | -4.078 |
| 1373 | 1.342 | -5.533 |
| 1423 | 1.491 | -4.762 |
| 1423 | 1.690 | -4.562 |
| 1423 | 1.892 | -4.370 |
| 1423 | 2.083 | -4.184 |
| 1423 | 2.210 | -4.019 |
| 1423 | 2.294 | -3.873 |
| 1423 | 2.468 | -3.587 |
| 1473 | 1.290 | -4.167 |
| 1473 | 1.491 | -4.031 |
| 1473 | 1.623 | -3.967 |
| 1473 | 1.699 | -3.917 |
| 1473 | 1.775 | -3.903 |
| 1473 | 1.851 | -3.807 |
| 1473 | 1.906 | -3.767 |

diopside than for anorthite in both wet and dry conditions (Figures 4d and 5d). The transition stresses for the twophase aggregates are between those for the end-members. At low stresses, the strength of two-phase aggregates with high diopside content and pure diopside samples depends on diopside grain size (Figures 4c and 5b). For pure diopside larger grain size results in substantially lower strain rates. However, as in the starting material, the grain size distribution of diopside is relatively broad. Therefore we did not explicitly determine *m*. The effect of a broad grain size distribution on grain size sensitive creep is still poorly understood [Dimanov et al., 2003; Ter Heege et al., 2004]. For pure diopside and pure anorthite with narrow grain size distributions grain size exponents m of 2.9 ± 0.3 and 2.7 ± 0.2 were estimated, respectively, indicating grain boundary diffusion-controlled creep operating at low stresses [Dimanov et al., 1999; Xiao, 1999; Dimanov et al., 2003]. Accordingly, we assume m = 3 (i.e., grain boundary diffusion-controlled creep) for the pure end-member phases and the two-phase aggregates. In the diffusion creep regime aggregates containing 50 vol % diopside deformed with a 3 times lower strain rate when maximum particle size increased from 35 μ m to 45 μ m. However, at diopside volume fractions of 25%, samples show nearly similar strengths irrespective of particle size (Figure 4b). No substantial effect of diopside particle size was found in the dislocation creep field.

[20] For pure diopside samples deformed at high stresses we estimate the stress exponent $n \approx 5.5$. This value is in

 Table 2.
 Creep Data for Wet Samples

Table 2. (continued)

| Temperature, K | Log σ, MPa | Log ε' , s ⁻¹ | Temperature, K | Log σ, MPa | Log ε' , s ⁻¹ |
|----------------|----------------------------|--------------------------------------|----------------|--------------------------------|--------------------------------------|
| | Sample Di25# | | 1373 | 2.644 | -4.895 |
| 1373 | 2.000 | -6.207 | 1373 | 2.697 | -4.703 |
| 1373 | 2.146 | -6.146 | 1423 | 2.463 | -4.932 |
| 1373 | 2.255 | -5.959 | 1423 | 2.184 | -5.361 |
| 1373 | 2.380 | -5.757 | 1423 | 2.598 | -4.682 |
| 1373 | 2.447 | -5.664 | 1423 | 2.649 | -4.479 |
| 1373 | 2.505 | -5.541 | | | |
| 1373 | 2.556 | -5.410 | | Sample An75Di ₄₅ #1 | |
| 1373 | 2.643 | -4.889 | 1323 | 2.410 | -6.306 |
| 1373 | 2.699 | -4.735 | 1323 | 2.595 | -5.625 |
| 1373 | 2.176 | -6.055 | 1323 | 2.660 | -5.293 |
| 1423 | 1.778 | -5.921 | 1323 | 2.467 | -6.240 |
| 1423 | 2.000 | -5.873 | 1373 | 2.290 | -5.861 |
| 1423 | 2.079 | -5.757 | 1373 | 2.161 | -6.220 |
| 1423 | 2.204 | -5.712 | 1373 | 2.468 | -5.574 |
| 1423 | 2.255 | -5.587 | 1373 | 2.549 | -5.022 |
| 1423 | 2.342 | -5.397 | 1373 | 2.387 | -5.762 |
| 1423 | 2.491 | -5.18/ | 13/3 | 2.297 | -5.983 |
| 1423 | 2.556 | -4.815 | 1423 | 2.318 | -5.229 |
| 1423 | 2.146 | -5./42 | 1423 | 2.164 | -5.570 |
| | S | | 1423 | 2.430 | -4.983 |
| 1422 | Sample D135#2 | 5 020 | 1423 | 2.393 | -5.211 |
| 1423 | 1./40 | -5.959 | 1425 | 1.982 | -3.935 |
| 1423 | 1.001 | -5.850 | | Sample An75Di #1 | |
| 1423 | 2 246 | -3.007 | 1373 | 1 1 1 1 1 | 6 132 |
| 1423 | 2.240 | -5.447 | 1373 | 1.447 | -0.432 |
| 1423 | 2.441 | -3.172 | 1373 | 2 201 | -0.043 |
| 1423 | 2.54 | -4.386 | 1373 | 2.201 | -5.156 |
| 1423 | 1 663 | -5.613 | 1373 | 2.507 | -4 523 |
| 1473 | 1.820 | -5 521 | 1373 | 2.040 | -3 959 |
| 1473 | 1 903 | -5437 | 1423 | 1 462 | -6200 |
| 1473 | 1 732 | -5 561 | 1423 | 1 763 | -5.851 |
| 1473 | 2.025 | -5.406 | 1423 | 2.009 | -5.607 |
| 1473 | 2.176 | -5.208 | 1423 | 2.204 | -5.408 |
| 1473 | 2.279 | -5.045 | 1423 | 2.279 | -5.243 |
| 1473 | 2.369 | -4.876 | 1423 | 2.435 | -4.979 |
| 1473 | 2.444 | -4.666 | 1423 | 2.497 | -4.703 |
| 1473 | 2.477 | -4.511 | 1473 | 1.477 | -5.439 |
| 1473 | 2.537 | -4.298 | 1473 | 1.785 | -5.246 |
| 1473 | 2.589 | -4.070 | 1473 | 2.107 | -4.951 |
| | | | 1473 | 2.246 | -4.644 |
| | Sample Di ₄₅ #1 | | 1473 | 2.479 | -4.162 |
| 1373 | 2.009 | -6.625 | | | |
| 1373 | 2.303 | -6.381 | | Sample An75Di ₃₅ #1 | |
| 1373 | 2.477 | -6.007 | 1373 | 1.756 | -6.024 |
| 1373 | 2.643 | -5.135 | 1373 | 1.987 | -5.839 |
| 1373 | 2.600 | -5.450 | 1373 | 2.146 | -5.777 |
| 1423 | 2.004 | -6.157 | 1373 | 2.305 | -5.498 |
| 1423 | 2.173 | -5.907 | 1373 | 2.412 | -5.285 |
| 1423 | 2.301 | -5.690 | 13/3 | 2.476 | -5.19/ |
| 1423 | 2.396 | -5.507 | 13/3 | 2.507 | -5.049 |
| 1423 | 2.484 | -5.324 | 1423 | 1.681 | -5.695 |
| 1423 | 2.542 | -5.095 | 1423 | 1.820 | -5.609 |
| 1423 | 2.602 | -4./50 | 1423 | 1.982 | -5.450 |
| 14/3 | 1.740 | -5.921 | 1425 | 2.140 | -5.239 |
| 14/3 | 2.015 | -5.025 | 1423 | 2.070 | -5.801 |
| 14/3 | 2.173 | -5.595 | 1423 | 2.079 | -5.399 |
| 1473 | 2.303 | -4 963 | 1423 | 2.230 | -4.943 |
| 1473 | 2.403 | -4.903 | 1423 | 2.320 | -4.945 |
| 1473 | 2.405 | -4.507 | 1423 | 2.580 | _4 712 |
| 1473 | 2.545 | -4.307 -4.274 | 1423 | 2.451 | -4.570 |
| 14/5 | 2.300 | 7.277 | 1423 | 2.408 | -4 413 |
| | Sample Diss#1 | | 1423 | 2.530 | _4 337 |
| 1323 | 2.466 | -5000 | | 2.550 | 1.557 |
| 1323 | 2.284 | -5.636 | | Sample An50Di,#1 | |
| 1323 | 2.155 | -6.284 | 1373 | 1.763 | -5.093 |
| 1323 | 1.968 | -6.627 | 1373 | 1.505 | -5.448 |
| 1323 | 2.411 | -6.121 | 1373 | 1.763 | -5.114 |
| 1323 | 2.700 | -5.561 | 1373 | 1.663 | -5.359 |
| 1373 | 2.461 | -5.398 | 1373 | 1.839 | -5.063 |
| 1373 | 2.182 | -5.900 | 1373 | 1.908 | -5.049 |

1373

1373

1373

1373

1373

2.017

2.079

2.204

2.301

2.380

 Table 2. (continued)

 Table 2. (continued)
 $Log \varepsilon', s^{-1}$ <u>Log</u> ε' , s⁻¹ Temperature, K Log σ, MPa Temperature, K Log σ, MPa 1373 1 964 -4991373 2.447 -3.8621373 2.000 -4.9151373 2.534 -3.6101373 2.097 -4.8211373 2.601 -3.3801373 2.164 -47051373 2.217 Sample An25Di35#2 -4.6362.267 -4.5501423 -4.8541373 1.301 1 505 1373 2.312 -44391423 -4.6831373 2.352 -4.3531423 1.623 -4.5771373 2.423 -4.2441423 1.716 -4.4841423 1 477 -5.0371423 1 792 -43931423 1.580 -4.9501423 1.857 -4.3351423 1.699 -4.7751423 1.924 -4.2651423 1.908 -4.6861423 2.017 -4.1441423 2.025 -4.5791423 2.093 -4.0541423 2.079 -4.4971423 2.158 -3.929-4.3381423 2.176 1423 2 292 -3.6611473 1.279 -4.8921473 1.301 -4.5231473 1.447 -4.7471473 1.477 -4.4101473 1.580 -4.6441473 1.580 -4.3181473 -4.499 1473 1.778 -4.132 1.643 1473 1.699 -4.4851473 1.903 -4.0401473 1.756 -4.4621473 2.000 -3.9001473 1.857 -4.3711473 2.146 -3.6741473 1.944 -4.2901473 2.199 -3.544-43451523 1.265 1473 2 2 5 5 -3.4391523 1.447 -4.211473 2.301 -3.3061523 1.568 -4.1331523 1 681 -4009Sample An#1 1523 1323 1.362 -5.5031.146 -4.5271323 1.556 -5.207Sample An50Di35#3 1323 1 740 -5.1101373 1.415 1323 1.863 -4.807-5.6562.017 1373 1 613 -53641323 -4670-5220-4.5001373 1.732 1323 2.188 1373 1.973 -4.9001323 2.364 -4.1971373 2.207 -4.5731373 1.491 -4.955-4.4321373 1.623 1373 2.291 -4.8511373 2.340 -4.3461373 1.785 -4.6561373 2.382 -4.2431373 1.964 -4.5131373 2.428 -4.1411373 2.149 -4.2521373 2.500 -3.996 1373 2.276 -4.0811373 2.549 -3.8701373 2.375 -3.8931423 2.146 -4.2741423 1.431 -4.4931423 2 2 9 0 -3.8991423 1.633 -4.3021423 2.396 -3.7141423 1.785 -4.1891423 2.471 -3.4211423 1 9 5 9 -4.0431423 2.143 -3.845Sample An50Di35#2 -3.622 1423 2.303 -5.535 1373 1 4 4 7 1373 -5.2531.663 1373 1.903 -4.7571373 2.265 -4460good agreement with stress exponents estimated for creep of 1373 2.401 -4.161diopside single crystals of similar chemical composition (n =1373 2.505 -3.793 6.5 ± 0.5 [Raterron and Jaoul, 1991]. When extrapolated to 1373 2.602 -3570lower stresses our data for power law creep at T = 1423 K 1473 1.431 -4.5701473 1.643 -4.428closely agrees with that observed for single crystal defor-1.903 -4.1571473 mation accommodated by the strongest slip systems 1473 2.064 -3.932(100)[010], (010)[101] and (010)[100]) (star in Figure 4a). 1473 2.241 -3.460The stress exponent is also similar to that suggested by 1473 2.362 -3.128Dimanov et al. [2003] for uniaxial creep of coarse-grained Sample An25Di35#1 diopside aggregates at high stresses and at T = 1413 K 1373 1.204 -5.470(Figure 4a, n = 5.1, thick solid line). Stress exponents 1373 1.477 -5.227 estimated for dislocation creep of dry and wet specimens -5.1041373 1.602 do not differ significantly (Tables 3a and 3b). Dislocation 1373 1.778 -4.8231373 1.903 -4.707creep of pure synthetic anorthite aggregates similar to our

report n = 3 for both wet and dry conditions. [21] The addition of diopside decreases the strain rate of the anorthite-diopside aggregates at fixed differential stress

material was investigated by Rybacki et al. [2000], who

-4.577

-4.456

-4.267

-4.125

-4.016

Table 3a. Flow Laws ($d\varepsilon/dt = A\sigma^n e^{-Q/RT}$) for Wet Materials

| | · · · | | |
|------------------------|--|---------------|-------------------------|
| Material | A, $Pa^{-n} s^{-1}$ | n | Q, kJ mol ⁻¹ |
| Di ₂₅ W | $1.26 	imes 10^{-1} {}^{+9.58 	imes 10^{-1}}_{-1.12 	imes 10^{-1}}$ | 1 | 345 ± 25 |
| Di ₃₅ W | $1.08 	imes 10^{-1} {}^{+6.45 	imes 10^{-1}}_{-0.93 	imes 10^{-1}}$ | 1 | 349 ± 23 |
| Di ₄₅ W | $3.07 \times 10^{-3} {}^{+2.45 \times 10^{-2}}_{-2.73 \times 10^{-3}}$ | 1 | 318 ± 26 |
| An75Di ₃₅ W | $8.47 \times 10^{-3} {}^{+5.58 \times 10^{-2}}_{-7.35 \times 10^{-3}}$ | 1 | 310 ± 24 |
| An75Di ₄₅ W | $9.98 \times 10^{-3} {}^{+5.28 \times 10^{-2}}_{-8.39 \times 10^{-3}}$ | 1 | 322 ± 21 |
| An50Di35W | $1.28 \times 10^{-1} {}^{+4.28 \times 10^{-1}}_{-9.85 \times 10^{-2}}$ | 1 | 316 ± 18 |
| An25Di ₃₅ W | $2.89 \times 10^{-2^{+1.56 \times 10^{-1}}}_{-2.44 \times 10^{-2}}$ | 1 | 291 ± 22 |
| AnW | $3.21\times 10^{-2}^{+1.37\times 10^{-1}}_{-2.60\times 10^{-2}}$ | 1 | 286 ± 19 |
| AnW ^a | $1.995 \times 10^{5+4.31 \times 10^{5}}_{-1.364 \times 10^{5}}$ | 1 | 267 ± 13 |
| DiW | $5.16 \times 10^{-33+7.98 \times 10^{-32}}_{-4.85 \times 10^{-33}}$ | 5.52 ± 0.09 | 534 ± 32 |
| An75DiW | $3.16 \times 10^{-28+5.56 \times 10^{-27}}_{-2.99 \times 10^{-28}}$ | 5.03 ± 0.07 | 533 ± 34 |
| An50DiW | $1.54 \times 10^{-17+4.00 \times 10^{-16}}_{-1.48 \times 10^{-17}}$ | 3.97 ± 0.10 | 556 ± 39 |
| An25DiW | $5.25\times 10^{-15+5.56\times 10^{-14}}_{}}$ | 3.01 ± 0.08 | 391 ± 29 |
| AnW ^b | $3.981 \times 10^{2+3.962 \times 10^2}_{-1.986 \times 10^2}$ | 3.00 ± 0.00 | 356 ± 9 |
| | | | |

^aThe diffusion creep flow law for AnW is from *Rybacki and Dresen* [2000], where the preexponential parameter A is given in MPa⁻ⁿ m⁻³ s⁻¹ and the grain size is 3.4 μ m.

^bThe dislocation creep flow law for AnW is from *Rybacki and Dresen* [2000], where the preexponential parameter A is given in MPa⁻ⁿ s⁻¹.

for both wet and dry specimens (Figures 4d and 5d). Note that aggregate creep rates at constant load remain between creep rates of the pure anorthite and diopside end-members. At experimental conditions the observed strengthening of the two-phase aggregates is relatively similar in the dislocation and diffusion creep regimes. However, if extrapolated to larger grain size and lower stresses, the difference in strength between the diopside and anorthite end-member phases will become larger in the dislocation creep regime than in the diffusion creep regime, because of the difference in stress exponents.

3.1.2. Activation Energy

[22] The activation energy Q for diffusion creep for pure anorthite and diopside aggregates ranges from 286 \pm 19 kJ mol⁻¹ for wet anorthite to 528 ± 42 kJ mol⁻¹ for dry diopside specimens (Tables 3a and 3b). In the presence of <0.1 wt % water creep strength and creep activation energy are significantly reduced for pure anorthite and diopside end-members and the two-phase aggregates (Tables 3a and 3b and Figure 6). The activation energy for diffusioncontrolled creep of anorthite-diopside two-phase mixtures is between those for end-members for dry and wet samples (Tables 3a and 3b and Figures 6a and 6c). In the dislocation creep regime activation energies range from $356 \pm 9 \text{ kJ mol}^{-1}$ for wet anorthite aggregates to 691 ± 1000 46 kJ mol⁻¹ for dry diopside samples (Tables 3a and 3b). Activation energies for power law creep of anorthitediopside aggregates are close to or between those of the end-members (Figures 6b and 6d).

[23] The ratios of activation energies for dislocation and diffusion creep for wet and dry samples are about 1.3 irrespective of diopside volume fraction. This ratio is similar to values found for other rock types; for example for dry dunites and synthetic olivine aggregates the ratios are 1.4 and 1.6, respectively [*Karato et al.*, 1986; *Mei and* *Kohlstedt*, 2000a, 2000b], and for Solnhofen limestone the ratio is 1.4 [*Schmid et al.*, 1977]. The activation energy of grain boundary diffusion controlled creep is assumed to be close to the activation energy for grain boundary diffusion. The activation energy of dislocation (climb controlled) creep is assumed to be close to the activation energy for volume diffusion. The activation energy for volume diffusion. The activation energy for volume diffusion is commonly assumed to be a factor ≤ 2 larger than that for grain boundary diffusion [*Haasen*, 1986; *Gottstein and Shvindlerman*, 1999].

3.2. Microstructures of Deformed Samples

[24] For pure diopside samples deformed in the highstress regime (\geq 200 MPa) dislocation densities are up to 10¹⁴m⁻². Curved dislocations, dislocation walls, cell structures and subgrain boundaries are frequently found indicating dislocation climb and subgrain formation (Figures 7a and 7b). Average dislocation densities of deformed wet and dry specimens were similar, but cell structures and subgrain boundaries were more frequently found in wet samples (Figure 7b). For two-phase samples deformed only in the low-stress regime (An25Di₃₅D#1 and An25Di₃₅D#2, Table 1 and Figure 4b), the average dislocation density remains unchanged. Dislocation microstructures are similar to those observed after hot pressing except within the rim of the diopside inclusions, where high dislocation densities were often observed (Figure 7d). A significant increase in average dislocation density by about 1 order of magnitude was only found in a specimen deformed at differential stresses ≥ 200 MPa (AnDi₃₅25D#3, Table 1, Figure 4b) and dislocations remain very heterogeneously distributed. In particular, the highest dislocation densities were found in diopside inclusion rims close to the boundary and in the surrounding anorthite matrix where fine, recrystallized grains are commonly observed (Figure 7c). The recrystallization mechanism could not be clearly identified. After deformation glassy pockets are still present at some multiple

Table 3b. Flow Laws $(d\varepsilon/dt = A\sigma^n e^{-Q/RT})$ for Dry Materials

| Material | A, $Pa^{-n} s^{-1}$ | n | Q, kJ mol ⁻¹ |
|-----------------------------------|---|---------------|-------------------------|
| Di ₃₅ D | $3.19 \times 10^{4+9.50 \times 10^{5}}_{-3.08 \times 10^{4}}$ | 1 | 528 ± 42 |
| An75Di35D | $3.81 \times 10^{3+6.81 \times 10^{4}}_{-3.61 \times 10^{3}}$ | 1 | 485 ± 36 |
| An50Di35D | $1.21 	imes 10^{3} {}^{+2.63 	imes 10^{4}}_{-1.16 	imes 10^{3}}$ | 1 | 436 ± 37 |
| An50Di ₄₅ D | $6.79 \times 10^{4^{+1.13 \times 10^6}}_{-6.41 \times 10^4}$ | 1 | 496 ± 34 |
| An25Di35D | $1.32\times 10^{4+2.63\times 10^5}_{-1.26\times 10^4}$ | 1 | 453 ± 36 |
| An25Di ₄₅ D | $2.95 \times 10^{4+4.92 \times 10^{5}}_{-2.78 \times 10^{4}}$ | 1 | 454 ± 34 |
| AnD ^a | $1.259 \times 10^{12+3.753\times 10^{12}}_{-9.427\times 10^{11}}$ | 1 | 467 ± 16 |
| DiD | $3.01 	imes 10^{-28+1.25 	imes 10^{-26}}_{-2.94 	imes 10^{-28}}$ | 5.47 ± 0.13 | 691 ± 46 |
| An75DiD | $3.81 \times 10^{-24 + 1.46 \times 10^{-22}}_{-3.71 \times 10^{-24}}$ | 4.96 ± 0.19 | 666 ± 45 |
| An50DiD | $2.71 \times 10^{-12} {}^{+2.16 \times 10^{-10}}_{-2.68 \times 10^{-12}}$ | 4.08 ± 0.24 | 723 ± 52 |
| An25DiD | $6.15\times10^{-4^{+1.46\times10^{-2}}}_{-5.90\times10^{-4}}$ | 3.03 ± 0.15 | 701 ± 38 |
| $\operatorname{AnD}^{\mathrm{b}}$ | $5.012 \times 10^{12+1.494 \times 10^{13}}_{-3.753 \times 10^{12}}$ | 3.00 ± 0.00 | 648 ± 20 |

^aThe diffusion creep flow law for AnD is from *Rybacki and Dresen* [2000], where the preexponential parameter A is given in MPa⁻ⁿ μ m⁻³ s⁻¹ and the grain size is 2.7 μ m.

^bThe dislocation creep flow law for AnD is from *Rybacki and Dresen* [2000], where the preexponential parameter A is given in MPa⁻ⁿ s⁻¹.



Figure 4. Mechanical data (log stress-log strain rate) of dry samples showing two deformation regimes at low and high stresses with stress exponents of n = 1 and $n \approx 3-5.5$, respectively. (a) Data for pure diopside aggregates. Circles, diamonds, and squares represent data at 1523, 1473, and 1423 K, respectively. Data are fit with equation (1) (solid line) using flow law parameters from Table 3b. Extrapolation of the fit of high-stress data (squares) to lower stresses (dashed line) is in good agreement with previous studies on dislocation creep in single crystals (star, 1423 K) from Raterron et al. [1994] and diopside aggregates (thick line, 1413 K) from *Dimanov et al.* [2003]. Triangles represent data for creep of dry pure diopside aggregates (average grain size <10 µm) at 1473 K from *Bystricky and Mackwell* [2001]. (b) Two-phase aggregates (An25Di) showing little or no strength difference between samples with different diopside particle size (<35 µm and <45 µm). Solid circles correspond to sample deformed only at low stresses in the diffusion creep regime. Open circles represent a sample also deformed at high stresses in the dislocation creep regime. (c) At low stresses, samples containing 50 vol % of coarsegrained ($<45 \,\mu$ m) particles, significantly stronger than aggregates containing particles with $<35 \,\mu$ m grain size. No strength difference is found at stresses >200 MPa. (d) Data at T = 1423 K and flow laws from Table 3. Strength of two-phase aggregates increases with increasing volume fraction of diopside particle (25%, diamonds; 50%, circles; and 75%, squares) but remains between strengths of the pure anorthite (crosses) and diopside (inverted triangles) samples. In the dislocation creep regime at high stresses the stress exponents increase with increasing particle content. Crosses represent data for dry pure anorthite (similar grain size as in our study) from Rybacki and Dresen [2000].



Figure 5. Mechanical data (log stress–log strain rate) of wet samples. Stress exponent is n = 1 indicating diffusion creep at low stresses and $n \approx 3-5.5$ at higher stresses indicating dislocation creep. (a) Data for pure diopside aggregates. Circles, diamonds, and squares represent data at 1473, 1423, and 1373 K, respectively. Data are fit with equation (1) using the flow law parameters from Table 3a. Inverted triangles represent data for creep of wet pure diopside aggregates (average grain size <10 µm) at 1423 K from *Bystricky and Mackwell* [2001]. (b) Strength of pure diopside aggregates with grain size d_{Di} < 25, 35, and 45 µm, respectively. In diffusion creep, strength of samples increases with increasing grain size. At high stresses, no difference in strength is observed between samples with different grain size. (c) Two-phase aggregates containing 50 vol % diopside. At low stresses n = 1; at high stresses $n \approx 4$. (d) Strength of two-phase aggregates increases with increasing volume fraction of diopside particles but remains between those of pure anorthite and diopside samples. In the dislocation creep regime at high stresses the stress exponents increase with increasing diopside particle content. Crosses represent data for wet pure anorthite (similar grain size as in our study) from *Rybacki and Dresen* [2000].



Figure 6. Plots of log strain rate versus inverse temperature for (a and b) dry and (c and d) wet samples with diopside particle size of $<35 \ \mu m$ deformed in the diffusion (Figures 6a and 6c) and dislocation (Figures 6b and 6d) creep regimes. Flow law parameters and data (crosses) for pure anorthite aggregates are from *Rybacki and Dresen* [2000]. In addition, Figure 6c contains a flow law (dashed line) and data (triangles) for pure wet anorthite aggregates from this study. Data are normalized to stresses of 10 MPa and 100 MPa for diffusion and dislocation creep, respectively. Strength of the aggregates increases with increasing diopside content but is between the strength of the pure anorthite and diopside aggregates. The activation energies for diffusion and dislocation creep of two-phase aggregates also increase with increasing diopside content but are bounded by (or close to) those of the end-member values (see Tables 3a and 3b and Figure 9).

grain junctions (Figure 7c), but two-grain boundaries remained free of melt.

4. Discussion

4.1. Creep Activation Energies and Stress Exponents of Pure Anorthite and Diopside Aggregates

[25] At low stresses, grain boundary diffusion-controlled creep dominates deformation of the fine-grained pure diopside and anorthite aggregates. For a similar range in temperatures (1273–1523 K) and a confining pressure of

300 MPa the data are in good agreement with previous studies (Tables 3a and 3b). For diffusion-controlled creep of wet anorthite we obtained an activation energy $Q = 286 \text{ kJ mol}^{-1}$. *Rybacki and Dresen* [2000] reported $Q = 267 \text{ kJ mol}^{-1}$ for temperatures T > 1273 K. For diffusion creep of dry diopside aggregates we found an activation energy $Q = 528 \text{ kJ mol}^{-1}$, which is very similar to $Q = 540 \text{ kJ mol}^{-1}$ reported by *Bystricky and Mackwell* [2001] for triaxial deformation of dry diopside samples. For diopside aggregates deformed uniaxially at low stresses and similar temperatures *Dimanov et al.* [2003] found activation



Figure 7. TEM micrographs of deformed samples. Bright-field micrographs of (a) dry diopside aggregate, (b) wet diopside aggregate, and (c) dry 25 vol % two-phase aggregate deformed at high stresses in the dislocation creep regime. High density of curved dislocations $(5 \times 10^{13} \text{ m}^{-2}$ (Figure 7a)) indicates climb. Dislocation walls and low-angle grain boundaries are frequently observed (Figures 7a and 7b). Note the formation of subgrains in Figures 7a and 7b. For sample An25Di₃₅D#3 both diopside (Di) inclusions and anorthite (An) matrix show high dislocation densities (Figure 7c). Grain boundary dislocations are observed in the diopside inclusions, and small, recrystallized grains of both phases are observed at the anorthite/diopside interface (Figure 7c). Note the presence of a small glassy pocket labeled gl at a multiple grain junction. (d) Dark field micrograph representative of sample An25Di₃₅D#1 deformed in diffusion creep regime (Figure 4b, solid circles). Dislocation density is low for the anorthite matrix but high in diopside close to the boundary.

energies between 468 kJ mol⁻¹ and 558 kJ mol⁻¹, depending on diopside chemical composition.

[26] Activation energies for dislocation creep of pure anorthite and diopside aggregates are significantly lower in the presence of water. However, the effect is less pronounced for diopside samples. This may suggest that the storage capacity for hydrous defects is significantly higher for plagioclase compared to diopside. Unfortunately, there is no experimental study comparing water solubility and diffusion kinetics in both anorthite and diopside corresponding to the conditions of our study. For comparison, FTIR studies of natural samples indicate that plagioclase contains larger trace amounts of water, when compared to other silicates like olivine and quartz and in particular nonsodic clinopyroxenes [*Wilkins and Sabine*, 1973; *Hofmeister and Rossman*, 1985; *Beran*, 1987; *Ingrin et al.*, 1991; *Bell and Rossman*, 1992].

[27] The effect of <0.1 wt % water on activation energies for grain boundary diffusion-controlled creep of the pure end-members is very similar with a ratio of $Q_{\text{wet}}/Q_{\text{dry}} \approx 0.6$ for both materials. This suggests that water fugacity has a similar effect on defect concentration and defect mobility in anorthite and diopside grain boundaries.

[28] For the deformation of pure anorthite aggregates in the high-stress regime we refer to the data of *Rybacki and Dresen* [2000]. The stress exponent is n = 3 and the creep activation energies Q range from 356 kJ mol⁻¹ to 648 kJ mol⁻¹ for wet and dry anorthite, respectively. We found a stress exponent n = 5.5 and an activation energy Q = 691 kJ mol⁻¹ for dislocation creep of dry diopside aggregates. This value is very similar to those reported for dislocation creep from uniaxial creep tests of diopside single crystals (Q = 740 kJ mol⁻¹, n = 6.5) [*Raterron and Jaoul*, 1991], uniaxial creep tests of diopside polycrystals (Q =719 kJ mol⁻¹, n = 5.1) [*Dimanov et al.*, 2003] and triaxial deformation of dry diopside polycrystals (Q = 760 kJ mol⁻¹, n = 4.7) [*Bystricky and Mackwell*, 2001].

4.2. Particle Strengthening of Anorthite-Diopside Aggregates

[29] Mixing laws and continuum mechanics models are often used to predict particle reinforcement of a ductile matrix [Chen and Argon, 1979; Berveiller and Zaoui, 1984; Cho and Gurland, 1988; Yoon and Chen, 1990; Tanaka et al., 1991; Ravichandran and Seetharaman, 1993; Treagus, 2002]. In these models, load sharing between matrix and particles is a central assumption. Particle size and particle spacing have to be large with respect to matrix grain size and the dislocation length scale. Since nonlocal effects (i.e., interaction of particles) are mostly not captured, the models are best applied to dilute and homogeneous particle-matrix mixtures. However, experimental studies and self-consistent and numerical models have shown that particle size, particle shape and orientation, clustering of particles and interface coherence strongly affect the strengthening of composite materials [Arsenault, 1991; Arsenault et al., 1991; Bao et al., 1991; Corbin and Wilkinson, 1994; Watt et al., 1996; Parashivamurthy et al., 2001; Wilkinson et al., 2001; Kouzeli and Mortensen, 2002].

[30] A salient finding of this study is that the strength of the two-phase mixtures deforming in dislocation and diffusion creep increases with increasing diopside fraction,

irrespective of water content. At experimental conditions, irrespective of the dominant creep mechanism, the viscosity of pure diopside aggregates is about 2 and 3 orders of magnitude higher compared to pure anorthite aggregates, for wet and dry samples, respectively (Figures 5 and 6). When extrapolating the data to reference stresses of 10 MPa and 100 MPa for diffusion and dislocation creep, respectively, the viscosity contrast of the end-members increases for dislocation creep, because of the higher-stress exponent of diopside (Figure 8). The relative strengthening of the two-phase aggregates with 25 vol % and 50 vol % diopside particles is somewhat more pronounced for dry samples compared to wet specimens. Importantly, the strength of the two-phase aggregates remained between the isostress and isostrain rate bounds [Tullis et al., 1991] for all anorthitediopside mixtures (Figures 5, 6, and 8).

[31] Deformation microstructures of the two-phase aggregates indicate significant variation in local stresses and strains already after hot isostatic pressing. For example, fragmentation of large diopside particles, observed after hot isostatic pressing (Figure 1), indicates buildup of high stresses in diopside particles. High dislocation densities as observed locally at the matrix/particle interface in deformed two-phase aggregates may be related to strain gradients at the interface and load transfer between phases [Ashby, 1970; Fischmeister and Karlsson, 1977] and thermal expansion mismatch between matrix and inclusions [Arsenault, 1991]. In aggregates deformed at high stresses in the dislocation creep regime, we found high dislocation densities in the anorthite matrix and the diopside inclusions and small recrystallized grains located at inclusion/matrix interfaces. These observations indicate significant stress and strain gradients and load transfer from the anorthite matrix to the stronger diopside particles (Figure 7). Load transfer from a weak matrix to strong particles is well known, for example, from ceramic materials and metal matrix composites [Clyne and Withers, 1993; He et al., 1999]. Microstructural evidence for load sharing and particle reinforcement has also been reported for geological materials from laboratory and field studies [Prior et al., 1990; Dresen et al., 1998; Kenkmann and Dresen, 1998; Rybacki et al., 2000; Ji et al., 2001; Xiao et al., 2002].

4.2.1. Diffusion Creep Regime

[32] The normalized creep rates of anorthite-diopside aggregates are compared to predictions from the continuum mechanics models of *Hill* [1965] (SC) and *Yoon and Chen* [1990] (YC), and the averaging scheme of *Ravichandran and Seetharaman* [1993] (RS) (Figure 8). In these models, the strong phase is completely surrounded by a weak matrix. This assumption is appropriate for dilute mixtures. The original self-consistent scheme (SC) considers both phases deforming by power law creep. In the simplified self-consistent approach of *Yoon and Chen* [1990] the strong phase is assumed completely rigid. This assumption is appropriate for our anorthite-diopside mixtures, because at the experimental conditions the viscosity of diopside is 2-3 orders of magnitude higher than that of anorthite.

[33] The self-consistent (SC) theory of an isotropic twophase composite proposed by *Hill* [1965] may be applied to linear-viscous phases (see reviews by *Watt* [1976], *Berveiller and Zaoui* [1984], and *Treagus* [2002]). In the case of linear-viscous phases the viscosity of the composite



Figure 8. Log creep rates of two-phase aggregates versus diopside content. Solid circles represent the data of the experimentally obtained flow laws for samples with given diopside fraction ($d_{Di} < 35 \mu m$) and water content. When error bars are missing, they are smaller than the symbol size. The data are compared to the isostress and isostrain rate bounds and to predictions of different models: SC [*Hill*, 1965], T [*Tharp*, 1983], YC [*Yoon and Chen*, 1990], RS [*Ravichandran and Seetharaman*, 1993], RSiwl and RSisl (extended RS model, see Appendix A), and THT [*Tullis et al.*, 1991]. T model is given for different k values between 1 and 2 (equation (5)). Diffusion creep of (a) dry samples and (b) wet samples.

 (μ_c) may be expressed as a function of the viscosity of the matrix (μ_m) , the viscosity of the dispersed particles (μ_p) and the volume fractions of the phases $(V_m \text{ and } V_p)$ by a simple quadratic equation:

$$3\mu_{c}^{2} + \left[2\left(\mu_{m} + \mu_{p}\right) - 5\left(V_{m}\mu_{m} + V_{p}\mu_{p}\right)\right]\mu_{c} - 2\mu_{m}\mu_{p} = 0 \quad (2)$$

[34] The model proposed by *Yoon and Chen* [1990] (YC) is also based on the self-consistent approach [*Chen and Argon*, 1979], but considers the special case of rigid spherical inclusions embedded in nonlinear viscous matrix. The YC composite strain rate $\frac{d\varepsilon_c}{dt}$ normalized by the matrix strain rate $\frac{d\varepsilon_m}{dt}$ is given as a function of volume fraction of

rigid particles (V_P) and the stress sensitivity of the matrix strain rate (n_m) :

$$\frac{\frac{d\varepsilon_c}{dt}}{\frac{d\varepsilon_m}{dt}} = \left(1 - V_p\right)^{\left(2 + \frac{n_m}{2}\right)} \tag{3}$$

[35] The RS mixing model assumes a homogeneous (periodic) distribution of rigid cubic particles embedded in a nonlinear viscous matrix. A specific geometry is defined for the representative composite unit volume, whose subelemental volumes are either in constant stress, or subjected to constant strain rate (see Appendix A). The predicted composite strain rate $\frac{d\varepsilon_c}{dt}$ normalized by the matrix strain rate $\frac{d\varepsilon_m}{dt}$ is given as

$$\frac{\frac{d\varepsilon_c}{dt}}{\frac{d\varepsilon_m}{dt}} = \left[\frac{\left(1+c\right)^2}{f\left[\frac{\left(1+c\right)}{c}\right]^{\frac{1}{n_M}} + \left(1+c\right)^2 - 1}\right]^{\frac{1}{n_m}}$$
(4)

[36] The normalized creep rate of the composite depends mainly on the stress sensitivity of the matrix strain rate (n_m) and the volume fraction of rigid particles (V_P) that is introduced through $c = \frac{1}{\sqrt[3]{V_P}} - 1$. The geometric factor $f \approx 1 + \frac{0.3}{c}$. To account for deformation of both phases and for the spatial distribution of phases we extended the RS model to include deformation of two linear-viscous phases (see Appendix A). In the modified RS models we assume that anorthite forms an interconnected weak layer (RS-iwl), or that diopside forms an interconnected strong skeleton (RS-isl) (Figures 8a and 8b). At some critical volume fraction of strong particles close to the maximum packing fraction (MPF) [*Krieger and Dougherty*, 1959; *Aharonov and Sparks*, 1999] the strength of the aggregate may increase abruptly because of the formation of an interconnected rigid skeleton. However, the MPF depends on particle shape, size distribution and matrix material and is difficult to predict [*Elliott et al.*, 2002].

[37] *Tharp* [1983] developed a model (T) for the deformation of porous materials. The model may be applied to a power law or linear viscous matrix containing weak inclusions. The normalized flow stress of the composite is expressed as

$$\frac{\sigma_c}{\sigma_m} = 1 - k \left(V_p \right)^{\frac{2}{3}} \tag{5}$$

where σ_m and σ_c are the flow stresses of the strong matrix phase and the composite, respectively, V_p is the volume fraction of weak inclusions (or pores) and k is a material parameter depending on the geometry and the spatial distribution of weak inclusions. For a periodic arrangement of pores k is close to 1. This concept is similar to the RS-isl model and the results of both approaches are nearly identical (Figure 8a). Experimentally derived values for k vary between 1 and 2 [*Tharp*, 1983].

[38] In general, none of the models predict the composite behavior over the whole range of compositions (Figures 8a and 8b). Predictions of the YC and the RS model are in good agreement with the experimental data at low diopside volume fractions $V_p < 0.5$. Since in these models particles are considered rigid, predicted strain rates are outside the isostrain rate bound for very high particle volume fractions (\geq 80–90%). Aggregate strength predicted by the RS-isl model is close to the isostrain rate bound. At high particle volume fractions \geq 75%, the T model can be fitted to the data with a geometrical parameter k = 2. At 50 vol % particles the SC model strongly overestimates the strength of both wet and dry composites. The data for wet samples plots between the isostress bound and creep rates predicted by the YC and RS models, suggesting that load transfer to the inclusions is less effective for wet samples than for dry two-phase aggregates. Similar observations were reported by Xiao et al. [2002]. For particle fractions >0.5 the strengthening of the aggregates is reasonably fit by the T model with k varying between 1.5 and 2.

[39] Samples containing volume fractions ≥ 0.5 of coarse diopside particles are stronger compared to the respective mixtures with smaller particles (Figure 4c). In general, coarse diopside particles have higher aspect ratios than smaller diopside particles (Figures 1b and 1c). The effect of particle aspect ratio on MPF is not well understood. However, modeling results show that the MPF decreases with increasing aspect ratio [*Sherwood*, 1997], suggesting that the formation of strong, load-bearing clusters may occur at lower particle volume fractions for large high–aspect ratio particles than for smaller and more equant particles. In addition, at high-volume fractions of particles grain size–sensitive diffusion creep of diopside (Figure 5b) will affect aggregate strength.

[40] In contrast, we observed that anorthite-diopside aggregates containing well-dispersed coarse diopside particles have a similar strength to mixtures with smaller particles, or are slightly weaker. The strength of composites is well known to depend significantly on interparticle distance $\lambda = d/V_p^{1/3}$ [Fischmeister and Karlsson, 1977; Gustafson et al., 1997; Kouzeli and Mortensen, 2002]. For metals reinforced by ceramic particles, experimental data, numerical modeling, and recently, high-resolution stress measurements have demonstrated that load transfer and composite strength decrease with increasing local particle spacing [Arsenault et al., 1991; Corbin and Wilkinson, 1994; Nan and Clarke, 1996; Watt et al., 1996; He et al., 1999]. Microstructural observations of our samples indicate that the size of the diopside particles is modified by fragmentation (Figure 1) during cold and hot pressing, and possibly by recrystallization (Figure 7) upon deformation. It is likely that a strain-dependent reduction of particle size will also affect aggregate strength.

4.2.2. Dislocation Creep Regime

[41] At high stresses, even for low diopside content both anorthite matrix and diopside inclusions deform by dislocation creep (see section 3.2). Since in the YC and RS models for a nonlinear viscous matrix the particles are assumed rigid the predicted aggregate strengths give an upper bound to the experimental data. To capture the behavior of polyphase aggregates with materials deforming in power law creep [*Tullis et al.*, 1991] suggested an empirical mixing model. Aggregate power law parameters



 n_C , Q_C and A_C are given as volume-averaged parameters of phases 1 and 2, respectively:

$$n_{c} = 10^{(V_{1}\log n_{1}+V_{2}\log n_{2})}$$

$$Q_{c} = \frac{Q_{2}(n_{c}-n_{1}) - Q_{1}(n_{c}-n_{2})}{(n_{2}-n_{1})}$$

$$A_{c} = 10^{\frac{\log A_{2}(n_{c}-n_{1}) - \log A_{1}(n_{c}-n_{2})}{(n_{2}-n_{1})}}$$
(6)

 V_1 and V_2 are volume fractions of phases 1 and 2, respectively. Predictions of aggregate strength of this model are in good agreement with the dislocation creep strength of anorthite-diopside mixtures at high-volume fractions of diopside particles (Figures 8c and 8d). However, the aggregate strength predicted by the different self consistent and mixing models (YC, RS, THT) is not significantly different for particle volume fractions \leq 75%. Also, all models largely overestimate the aggregate strength at low diopside volume fractions.

4.2.3. Stress Exponents and Activation Energy

[42] Stress sensitivities for power law creep and activation energies of two-phase aggregates vary with composition between end-members. For diffusion creep of pure anorthite and diopside samples, the stress exponent is $n \approx 1$ and remains unchanged for all mixtures (Figures 4d and 5d and Tables 3a and 3b). This was also found for linearviscous creep of anorthite-quartz aggregates [*Xiao et al.*, 2002] and superplastic flow of two-phase (zirconia-mullite) ceramics [*Yoon and Chen*, 1990].

[43] In the power law creep regime, stress exponents are between those of the pure end-members, but they increase from n = 3 to 5.5 for wet and dry aggregates. The composite stress exponent increases strongly at a diopside fraction of $V_P \approx 0.5$ (Figure 9a). This behavior is different from

Figure 9. (a) Stress exponents (circles) versus diopside content for wet and dry aggregates deforming in dislocation creep. Triangle indicates stress exponent for diopside single crystal deformation [Raterron and Jaoul, 1991]. Inverted triangle and diamond are stress exponents for dislocation creep of coarse-grained and fine-grained diopside aggregates from Dimanov et al. [2003] and Bystricky and Mackwell [2001], respectively. Progressive increase of the stress exponent with increasing diopside content is predicted by the mixing models of Tullis et al. [1991] (THT) and Ji and Zhao [1993] (JZ). The labels 0.1, 0.5, and 0.9 are different values for the fit parameter in the JZ model. (b) Activation energies for diffusion creep of wet (open symbols) and dry (solid symbols) samples. Triangles represent activation energies for diffusion creep of pure diopside aggregates from *Bystricky and Mackwell* [2001]. The data are in agreement with the increase of activation energy with increasing diopside content predicted by the JZ model. (c) Activation energies for dislocation creep of wet (open symbols) and dry (solid symbols) samples. Triangles and diamond represent activation energies for dislocation creep of pure diopside aggregates from Bystricky and Mackwell [2001] and Dimanov et al. [2003], respectively. Mixing models only qualitatively predict the increase of activation energy with increasing diopside content. In particular, the models do not capture a strong increase of activation energy at 50 vol % diopside.

observations on calcite-quartz aggregates, where the calcite matrix deforms in dislocation creep and stress exponents increase to n > 10 at quartz particle fractions <0.3 [*Siddiqi*, 1997; *Rybacki et al.*, 2003]. The increase of aggregate stress exponent with increasing diopside particle content is only partly captured by the mixing models [*Tullis et al.*, 1991; *Ji and Zhao*, 1993]. For instance, a smooth increase of *n* is predicted for the whole range of diopside fractions (Figure 9a).

[44] The activation energies Q for diffusion and dislocation creep of dry two-phase aggregates vary progressively between those of the end-members. The models for twophase aggregates proposed by *Ji and Zhao* [1993] and *Tullis et al.* [1991] predict a smooth increase of activation energy in good agreement with our data for diffusion creep and dislocation creep of dry anorthite-diopside aggregates (Figures 9b and 9c). However, the observed increase of Q with diopside volume fraction for dislocation creep of wet aggregates occurs abruptly at a diopside volume fraction $V_P \approx 0.5$. This trend is not captured by the averaging schemes (Figure 9c).

[45] For ceramic composites it has been suggested that high-stress exponents and activation energies may be related to a threshold stress varying with applied load and temperature-dependent load transfer, respectively [*Park and Mohamed*, 1995; *Li and Langdon*, 1998]. However, this approach cannot account for changes of flow law parameters of aggregates containing varying amounts of deformable particles.

4.3. Geological Applications

[46] Extrapolation of laboratory data to geological conditions generally involves orders of magnitude changes in stress, strain, strain rate, temperature, fluid pressures, composition and spatial scale of observation [Paterson, 1987, 2001]. A prerequisite for extrapolating mechanical data from experimental conditions to nature is that the dominant deformation mechanisms operating in the field and in the laboratory have to be similar. Commonly, characteristic deformation microstructures are used to relate microphysical processes operating at the laboratory scale to those that may be dominant in the field [Tullis, 1990; Paterson, 2001]. However, this study and previous experimental studies [Xiao et al., 2002; Rybacki et al., 2003] indicate that creep of polyphase aggregates involves significant local variations in stress and strain rate that are recorded in the deformation microstructure. In addition, even at the relatively low macroscopic strains achieved in this study, we find transient changes in the microstructure affecting aggregate strength, like reduction in particle size.

[47] Numerous field studies indicate that high-strain deformation (shear strain $\gamma \gg 5-10$) of the continental lower crust is commonly localized into distinct mylonitic shear zones [*Ji and Mainprice*, 1990; *Rutter and Brodie*, 1992; *Egydio-Silva et al.*, 2002]. Amphibolite to granulite facies ultramylonite layers transecting gabbroic and metabasic wall rocks typically consist of a fine-grained (10–100 µm) matrix containing a low volume fraction (<0.3) of welldispersed porphyroclasts [*Kenkmann and Dresen*, 2002]. Many studies have suggested that diffusion-controlled grain size–sensitive (superplastic) creep may be the dominant deformation mechanism accommodating high shear strains [*Boullier and Gueguen*, 1975; *Behrmann and Mainprice*,

1987; Kruse and Stünitz, 1999]. A significant increase in dislocation density and decrease in matrix grain size is observed close to porphyroclasts, indicating load transfer and concentration of local stresses [Kenkmann and Dresen, 1998]. The deformation microstructures from feldspathic ultramylonites indicating strong local gradients in stress and strain are qualitatively similar to those observed in our experimentally deformed synthetic anorthite-diopside aggregates. This observation indicates that at natural conditions (lower temperatures, stresses and strain rates) the strength contrast between feldspars and pyroxenes is qualitatively similar to the strength contrast between anorthite and diopside at our experimental conditions. Importantly, the isoviscous points for diffusion and dislocation creep for anorthite and diopside are at stresses and temperatures that are higher than in our experiments. Consequently, upon extrapolating the experimental data to lower stresses and temperatures increases the strength contrast between the end-member phases. To our knowledge no sound physical model of two-phase aggregates exists that captures the nonlinear variation of strength, stress sensitivity and activation energy observed in experiments over the whole range of compositions between pure end-members [Cho and Gurland, 1988]. For ceramics, it has been suggested [Clyne and Withers, 1993; Park and Mohamed, 1995; Li and Langdon, 1998] that temperature-dependent threshold stress and load transfer may account for apparent changes in stress exponent and activation energy, but the related models do not incorporate the effect of composition.

[48] Experimental studies of the mechanical behavior of synthetic polyphase rocks with varying composition are still few [Jordan, 1988; Tullis and Wenk, 1994; Siddiqi, 1997; Dresen et al., 1998; Ji et al., 2000, 2001; McDonnell et al., 2000; Xiao et al., 2002; Rybacki et al., 2003]. However, from the existing data it appears that the constitutive behavior is quite variable and, in addition to composition is dependent on the strength difference of the phases, their topology, and the dominant deformation mechanisms. At this point it seems impossible to suggest a suitable continuum model that captures the material behavior at all conditions offering more than a purely phenomenological or qualitative description. Existing models, however, may give some guidance as to how constitutive parameters and strength of pure end-member phases vary for small volume fractions of weak or strong inclusions dispersed in a strong or weak matrix, respectively. For example, from our quantitative estimates of the strength of anorthite-diopside aggregates it appears that changes in activation energy with mineralogical composition remain small for diopside particle volume fractions <0.4. This indicates that the normalized strength of the different two-phase mixtures does not change significantly when extrapolated to lower (geological) temperatures. The experimental data and the predictions from simple continuum mechanics models suggest that the strength of an ultramylonite will not substantially decrease with reduction of the porphyroclast content for volume fractions less than 0.4 (Figure 10).

5. Conclusions

[49] At high temperatures, anorthite-diopside aggregates and pure end-member phases show linear-viscous creep at



Figure 10. Normalized creep rate of (a) dry and (b) wet two-phase aggregates deforming in diffusion creep. Open circles represent data from this study. Open squares represent data from *Xiao et al.* [2002] on anorthite (matrix)-quartz (particles) aggregates. The normalized aggregate strength is compared to predictions from the YC model [*Yoon and Chen*, 1990]. For dry anorthite-quartz and anorthite-diopside aggregates and for particle fractions <50 vol %, creep rates are in good agreement with predictions of the YC model. For wet samples, YC only defines a lower bound on the creep rate (upper bound on strength). For particle fractions <50 vol % the data for anorthite-quartz are very close to the Reuss bound.

low stresses and power law creep at high stresses. Depending on water content and dominant deformation mechanism pure diopside aggregates are 2-3 orders of magnitude stronger than pure anorthite samples at experimental conditions. Strength, activation energy and power law stress exponent of two-phase aggregates increase with increasing volume fraction of diopside particles. Strength remains between the isostrain rate and isostress bounds. Stress exponents and activation energies are close to or between those of the end-members. Microstructures of deformed two-phase aggregates with dispersed particles indicate strong stress and strain gradients close to the matrix/inclusion interfaces. This observation suggests significant load transfer from the weaker anorthite matrix to the stronger diopside particles. The aggregate strengths predicted by simple continuum and mixing models are in good agreement with experimental data for dilute mixtures of strong diopside particles as well as mixtures with high diopside particle content, forming a load-bearing framework. Strength and flow law parameters of aggregates with intermediate composition are typically not well described by the existing mixing and continuum mechanics models.

Appendix A

[50] First, we give the assumed geometry of the representative unit cell of the composite material and the corresponding notations used by *Ravichandran and Seetharaman* [1993]. Second, we derive the extended equations for a two-phase composite with two creeping phases.

A1. Aggregate With Interconnected Weak Layer Microstructure: RS-iwl Model

A1.1. Nomenclature

[51] Figure A1 shows the representative unit cell, as defined by *Ravichandran and Seetharaman* [1993]. A strong cubic particle of length *d* is half embedded in a soft matrix of thickness λ . The strong phase *Di* has a volume fraction V_{Di} . The soft phase An has a volume fraction V_{An} . $V_{Di} + V_{An} = 1$. Stress is applied vertically. The creep behavior of the unit cell is assumed to be representative of the creep behavior of a composite with periodically dispersed strong cubic *Di* particles in a soft *An* matrix.



Figure A1. Representative unit cell for two-phase composite. Element 3 is a cubic inclusion, elements 2 and 4 are matrix, elements 3 and 4 form element 1 [*Ravichandran and Seetharaman*, 1993].

[52]

The unit cell is further subdivided into four subelements with their corresponding volume fractions $(V_1, V_2, V_3 \text{ and } V_4)$ defined as shown in Figure A1. Element 3 represents the strong inclusion, thus $V_3 = V_{Di} = d^3$. Element 4 represents a matrix layer, thus $V_4 = \lambda d^2$. Elements 3 and 4 are combined to form element 1. The rest of the unit cell is composed of matrix, material represented by element 2. The idealized configuration of the RS model does not include interactions between particles. This arrangement of phases represents the interconnected weak layer (IWL) model RS-iwl.

Defining
$$c = \frac{\lambda}{d} = \frac{1}{\sqrt[3]{V_{Di}}} - 1$$
, it can be shown that
 $V_1 = \frac{1}{(1+c)^2}$
 $V_2 = 1 - \frac{1}{(1+c)^2}$
 $V_3 = \frac{1}{(1+c)}$
 $V_4 = \frac{c}{(1+c)}$
(A1)

The flow laws of the strong particles and the soft matrix are respectively defined by

$$\frac{d\varepsilon_{An}}{dt} = K_{An}\sigma^{n_{An}}$$

$$\frac{d\varepsilon_{Di}}{dt} = K_{Di}\sigma^{n_{Di}}$$
(A2)

where K_{Di} and K_{An} are stress independent exponential functions of temperature ($K = A_0 e^{-Q/RT}$).

[53] During deformation the unit cell experiences the applied stress σ_{comp} , that partitions between elements 1 to 4. Each elemental volume V_i sustains a stress σ_i . Similarly, the strain rate of the unit cell, $\frac{d\varepsilon_{comp}}{dt}$, partitions between the elements 1 to 4. Each elemental volume V_i creeps at a strain rate $\frac{d\varepsilon_i}{dt}$.

A1.2. Elements in Isostrain Rate Configuration

[54] The composite is formed by elements 1 and 2, which are oriented in parallel with respect to the applied stress (σ_{comp}). This configuration corresponds to the isostrain rate bound for a composite. Therefore the creep of the composite equals that of elements 1 and 2:

$$\frac{d\varepsilon_1}{dt} = \frac{d\varepsilon_2}{dt} = \frac{d\varepsilon_{comp}}{dt}$$
(A3)

The applied stress σ_{comp} results in component stresses σ_1 and σ_2 in elements 1 and 2, with respective volume fractions V_1 and V_2 :

$$\sigma_{comp} = V_1 \sigma_1 + V_2 \sigma_2 \tag{A4}$$

Using (A1), (A4) can be expressed as

$$\sigma_{comp} = \frac{\sigma_1}{(1+c)^2} + \frac{\left((1+c)^2 - 1\right)\sigma_2}{(1+c)^2}.$$
 (A5)

A1.3. Elements in Isostress Configuration

[55] Element 1 supports the component stress σ_1 . It is subdivided in elements 3 and 4, which are in series with

respect to the applied stress σ_1 . This configuration corresponds to the isostress bound. Therefore the creep rate of element 1 is the volumetric average of the creep rates of elements 3 and 4:

$$\frac{d\varepsilon_1}{dt} = V_3 \frac{d\varepsilon_3}{dt} + V_4 \frac{d\varepsilon_4}{dt}$$
(A6)

with (see (A2))

$$\frac{d\varepsilon_3}{dt} = \frac{d\varepsilon_{Di}}{dt} = K_{Di}\sigma_1^{n_{Di}}$$
$$\frac{d\varepsilon_4}{dt} = \frac{d\varepsilon_{An}}{dt} = K_{An}\sigma_1^{n_{An}}$$

Ravichandran and Seetharaman [1993] assumed rigid particles, and thus set $\frac{d\varepsilon_3}{dt} = 0$. In the general case of two deforming phases (A6) be rewritten using (A1) and (A2):

$$\frac{d\varepsilon_1}{dt} = \frac{K_{Di}\sigma_1^{n_{Di}}}{(1+c)} + \frac{cK_{An}\sigma_1^{n_{An}}}{(1+c)}$$
(A7)

Ravichandran and Seetharaman [1993] considered that element 4 does support $\frac{\sigma_1}{f}$, *f* being a constraint coefficient accounting for the stress enhancement needed to maintain the flow of a weak layer (element 4) sandwiched between strong particles (the elements 3 of two adjacent unit cells).

Ravichandran and Seetharaman [1993] derived $f \approx 1 + \frac{0.3}{c}$ (see the original paper for details):

$$\frac{d\varepsilon_1}{dt} = \frac{K_{Di}\sigma_1^{n_{Di}}}{(1+c)} + \frac{cK_{An}\left(\frac{\sigma_1}{f}\right)^{n_{An}}}{(1+c)}$$
(A8)

From equation (A3) we have $\frac{d\varepsilon_1}{dt} = \frac{d\varepsilon_2}{dt}$ and from (A2) we have $\frac{d\varepsilon_2}{dt} = \frac{d\varepsilon_{An}}{dt} = K_{An}\sigma^{n_{An}}$. Then (A8) provides the following relation between σ_1 and σ_2 :

$$(1+c)\sigma_2^{n_{An}} = \frac{K_{Di}\sigma_1^{n_{Di}}}{K_{An}} + \frac{c\sigma_1^{n_{An}}}{(f)^{n_{An}}}$$
(A9)

Using (A9) in (A5) provides σ_{comp} as a function of σ_2 and the flow law parameters of the two phases. Then, because of (A3) it is possible to express $\frac{d\varepsilon_{comp}}{dt}$ as a function of σ_{comp} , which defines the flow law of the composite. In the specific cases, where $n_{Di} = n_{An}$, an analytical solution exists. For the general case the equations can only be solved numerically.

A1.4. Solution for Two-Phase Aggregate With Linear-Viscous Rheologies

[56] When $n_{Di} = n_{An} = 1$ equation (A9) becomes

$$\sigma_1 = \frac{(1+c)}{\left[\frac{K_{Di}}{K_{An}} + \frac{c}{f}\right]} \sigma_2 \tag{A10}$$

Inserting (A10) into (A5) yields

$$\sigma_{2} = \frac{(1+c)^{2}}{\left[\frac{(1+c)}{\left[\frac{K_{Di}}{K_{An}} + \frac{c}{f}\right]} + (1+c)^{2} - 1\right]} \sigma_{comp}$$
(A11)

Finally, combining (A3) and (A11) provides the flow law of the composite:

$$\frac{d\varepsilon_{comp}}{dt} = K_{An} \left[\frac{(1+c)^2}{\frac{(1+c)}{\left[\frac{K_{Di}}{K_{An}} + \frac{c}{f}\right]} + (1+c)^2 - 1} \right] \sigma_{comp}$$
(A12)

Equation (A12) describes the RS-iwl model (Figures 8a and 8b). It allows to plot the strain rate of the composite at a given temperature as a function of c, and thus of the particle fraction V_{Di} .

A2. Aggregate With Load Bearing Framework Microstructure: RS-isl Model

[57] In this model the overall geometry of the unit cell remains the same, but the strength contrast between phases is reversed. The mathematical analysis is exactly the same as in 1, with subscripts Di and An reversed and the constraint factor f, being omitted. Then, the flow law of the composite becomes:

$$\frac{d\varepsilon_{comp}}{dt} = K_{Di} \left[\frac{(1+c)^2}{\frac{(1+c)}{\left[\frac{K_{An}}{K_{Di}} + c\right]} + (1+c)^2 - 1} \right] \sigma_{comp}$$
(A13)

However, the parameter c is now a function of V_{An} . For convenience it is useful to redefine c as a function of the volume fraction $1 - V_{Di}$.

$$c = \frac{\lambda}{d} = \frac{1}{\sqrt[3]{V_{An}}} - 1\frac{1}{\sqrt[3]{1 - V_{Di}}} - 1$$

Then, the expression (A13) allows to plot the strain rate of the composite at a given temperature as a function of c, and thus of the particle fraction V_{Di} in the framework of the extended RS-isl model (Figure 8a).

[58] Acknowledgments. We thank Richard Wirth for his help with the TEM, Michael Naumann for keeping the gas apparatus running, and Stefan Germann and Karin Paech for preparation of samples for SEM and TEM. We benefited from constructive discussions with Eric Rybacki and Jörg Renner. Reviewers Jan Tullis and John FitzGerald, and Associate Editor Ian Jackson provided very constructive comments that improved the manuscript.

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