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Dislocation creep of polycrystalline dolomite

Caleb W. Holyoke III *, Andreas K. Kronenberg, Julie Newman

Department of Geology and Geophysics, Texas A&M University, College Station, TX 77843, United States

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ABSTRACT

The field of dislocation creep and rheological parameters for coarse-grained (d = 240 μm) natural dolomite has been determined through experiments performed at temperatures of 700–1000 °C, effective pressures of 300–900 MPa and strain rates of 10^{-4}/s to 10^{-7}/s. At low strain (<7%), dolomite aggregates deform homogeneously and define a power law between strain rate and differential stress with a stress exponent of 3.0 +/− 0.1, but at higher strains, through-going, fine-grained (<10 μm) shear zones develop in the dolomite aggregates concomitant with strain weakening. Recrystallization is limited at low strain and microstructures observed in the low strain samples include undulatory extinction, twins, grain boundary bulging, limited recrystallization along twins and fluid inclusion trails. These same microstructures are present outside of the narrow, through-going shear zones in high strain samples; however, within the shear zones the grain size is small (<10 μm) with some larger porphyroclasts (20–50 μm). Shear zones nucleate at fine-grained zones formed at twin boundaries, twin–twin intersections and fluid inclusion trails and is likely due to a switch in deformation mechanism due to the large strength contrast between the fine-grained zones deforming by diffusion creep and the coarse-grained protolith. The activation energy (Q) for creep of coarse-grained dolomite at low strain is 145 kJ/mol. In contrast to other activation energies for dislocation and diffusion creep of minerals, Q for dislocation creep of dolomite is considerably less than that for diffusion creep (248 kJ/mol). The results of this study indicate that coarse-grained dolomite will initially deform by dislocation creep at natural strain rates and temperatures between 200 and 550 °C, but due to limited recovery mechanisms, fine-grained shear zones will nucleate and diffusion creep may control the rheology of these fine-grained shear zones in nature at temperatures above ~300 °C.

1. Introduction

Carbonates, primarily calcite and dolomite, are commonly found in shear zones in the middle and upper crust and likely control the rheology of these zones. The deformation mechanisms and rheology of calcite aggregates have been explored extensively (Austin and Evans, 2009; de Bresser, 2002; Griggs and Miller, 1951; Griggs et al., 1953; Handin and Griggs, 1951; Heard and Raleigh, 1972; Herwegh et al., 2005; Renner et al., 2002; Rutter, 1972, 1974; Schmid et al., 1977; Schmid et al., 1980; Turner et al., 1956; Walker et al., 1990). However, deformation mechanisms and associated rheologies of dolomite aggregates have only recently been explored (Davis et al., 2008; Delle Piane et al., 2008); these studies provide a mechanical relationship for crystal plasticity and twinning at low temperatures and diffusion creep at high temperatures when grain size is small.

Davis et al. (2008) observed a temperature-dependent transition in the rheology of coarse-grained (240 μm) dolomite aggregates between low temperature plasticity and the onset of dislocation creep between 700 and 800 °C. However, due to decomposition of the dolomite aggregates at the maximum temperature (850 °C) and pressure limitations (400 MPa) of the Heard-type gas confining medium rock deformation apparatus (gas apparatus) used in their study, they were unable to explore this deformation mechanism in detail and mechanical data are transitional between plasticity and dislocation creep fields. Earlier experiments by Neumann (1969) provide microstructural evidence of dislocation creep in coarse-grained (700 μm) dolomite aggregates at T = 1000 °C, but these experiments were performed using strong solid media (pyrophyllite) assemblies in the Griggs piston-cylinder rock deformation apparatus (Griggs apparatus) that do not allow accurate stress measurements (e.g. Stewart et al., 2013) and mechanical data were not reported. In this study, we performed an experimental investigation using the molten salt cell (MSC) and solid salt assembly (SSA) in a Griggs apparatus to characterize the rheology of coarse-grained dolomite aggregates deformed by dislocation creep.

2. Experimental study

We performed a series of experiments to characterize the strain rate ($\dot{\varepsilon}$) and temperature dependence of flow strength of a coarse-grained natural dolomite aggregate (Madoc dolomite).

* Corresponding author. Tel.: +1 979 458 3924.
E-mail address: holyoke@geo.tamu.edu (C.W. Holyoke).

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2.1. Sample preparation and jacketing

The Madoc dolomite is a coarse- and equant-grained (d ~ 240 μm) dolomite aggregate with straight extinction when viewed optically with crossed Nicols, twins, fluid inclusion trails and few inclusions of quartz (≪1%). Cores of Madoc dolomite (5 mm diameter) were collected from the same block of dolomite used as the starting material by Davis et al. (2008). These cores were cut to the approximate final length (10 mm) and cylinder ends were ground perpendicular to the cylinder axis. The cylinder ends of the alumina pistons were also ground perpendicular to the piston length and the lower piston length was adjusted so that the center of the dolomite cylinder would be at the same location as the thermocouple (Pt–Pt/10% Rh) weld. The dolomite cylinders were placed within Pt jackets, each with a layer (0.6 mm wall thickness) of Ag or Ni between the Pt jacket and annulus of the cylinder, for experiments performed below or above 850 °C, respectively. The conductive Ag or Ni layer is used to reduce temperature gradients along the insulating sample (+/−5–10 °C, Holyoke, unpublished data), which are +/−20 °C without a thick metal layer (Tingle et al., 1993). Ni was not used at low temperatures because its strength becomes significant (40 MPa at 800 °C) and Ag was not used at higher temperatures due to its low melting point (~960 °C). One Ag or Ni disc was placed at each cylinder end in each experiment. Pt cups, which fit within the Pt jacket, were weld-sealed in all MSC experiments. In SSA experiments, a Pt disc was placed at the end of the cylinder end, outside of the inner Ag or Ni, and the ends of the Pt jacket were crimped over the cylinder end, which produces a mechanical seal at high pressure. After experiments performed using welded and mechanically sealed jackets, the Pt at the cylinder ends was always bowed out, indicating that both of these methods effectively prevented loss of CO_2 from the jacket and the CO_2 pore pressure could be determined from the dolomite decomposition reaction at equilibrium.

2.2. Apparatus and differential stress calibrations

All experiments were performed using a Griggs-type triaxial piston cylinder rock deformation apparatus (Holyoke and Kronenberg, 2010). The MSC technique was developed to eliminate contributions in load due to solid confining media surrounding cylindrical samples in a triaxial piston-cylinder apparatus (Green and Borch, 1989; Gleason and Tullis, 1993; Rybacki et al., 1998). A salt mixture (KCl/LiCl) is chosen that melts eutectically at experimental conditions, to replace the solid NaCl that normally surrounds the sample. The MSC technique also removes the Pb from the load column, which sharpens hit points in load records, in comparison with rounded hit points determined in SSA experiments associated with driving the load piston through the Pb. However, the removal of the Pb at the top of the load column by squeezing Pb from between the upper alumina piston and WC load piston in MSC experiments also leads to sample shortening of ~3–5%. Because of the strain sensitivity of dislocation creep in dolomite (discussed later), one experiment (D-5) was performed using the MSC assembly without performing the Pb removal step. This procedure caused a rounded hit point and decreases the precision of stress determination during this experiment, but it allowed us to compare the mechanical data of samples with liquid or solid confining media at identical shortening strains.

2.2.1. Hit points in the MSC

In order to demonstrate that this modification of the MSC technique does not affect the accuracy of the force measurements collected in experiments performed without the pre-hit deformation stage, we performed an experiment on a nickel cylinder at 600 °C, $\dot{\varepsilon} = 1.6*10^{-4}$/s, and $P_c = 1000$ MPa (Table 1). Three deformation steps were performed in this experiment; the first deformation step was performed by advancing the load piston through the Pb and deforming the Ni cylinder to 12% strain. The hit point during this deformation step is rounded, similar to those in SSA experiments, but the Ni cylinder was surrounded by molten salt. After reaching the desired strain, the deformation piston was retracted. The first deformation step removes the Pb in the load column and couples the upper alumina piston to the WC load piston. While the load piston is retracted, molten salt is injected between the upper alumina piston and middle alumina piston. When the second and third deformation steps were performed, only molten salt was removed from the load column and the hit points were sharp, as is the case in a normal MSC experiment. Differential stresses increased slowly over the first 5% strain in the first deformation step and then the sample deformed at a nearly constant differential stress of ~150 MPa for the remainder of the experiment (Fig. 1a). Differential stresses in the second and third deformation steps increase rapidly in the first percent strain and then deform at a nearly constant differential stress (145 and 135 MPa in steps 2 and 3, respectively) for the remainder of each deformation step (Fig. 1a). The constant differential stress deformation in each of these experiments is the same within experimental error, but the slope of the elastic response of Ni to loading during the first deformation step is considerably lower than the elastic slopes determined during the second and third deformation steps, which had sharp, well-defined MSC hit points. The difference in apparent elastic behavior during the first step and the latter steps indicates complex behavior of the load column of the Griggs apparatus during the removal of Pb. We expect that simple, linear elastic response is best determined once Pb has been removed. Nevertheless, the constant stress deformation portion of all experiments is consistent, irrespective of these initial loading procedures.

2.2.2. Pressure sensitivity of the MSC and SSA load calibrations

Holyoke and Kronenberg (2010) demonstrated that differential stresses calculated from forces measured using the MSC and SSA in the Griggs apparatus are not accurate relative to those measured in the gas apparatus without correction. They reported calibrations for MSC and SSA experiments that correct for an increase in friction of the load column due to elastic strain of the load column during loading.

Table 1
List of metal deformation experiments.

Experiment	Assembly	Material	Temperature (°C)	Confining pressure (MPa)	Strain rate (s^{-1})	Strain (%)	Strength at $\varepsilon = 5\%$[a,b] (MPa)	Final strength[b] (MPa)
D-4	MSC	nickel	600	1000	$1.5*10^{-4}$	12	116	130
					$1.5*10^{-4}$	12	125	120
					$1.5*10^{-4}$	12	125	125
D-21	SSA	Mo	900	1500	$1.6*10^{-5}$	4	160	160
			800	1500	$1.7*10^{-4}$	8	245	260
			700	1480	$1.7*10^{-4}$	9	270	295

[a] Unless total strain is less than 5%, then differential stress at the final strain is listed.
[b] Corrected using methods described in Holyoke and Kronenberg (2010).