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Dilatant plasticity in high-strain experiments on calcite–muscovite aggregates

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ABSTRACT

Torsion experiments were performed on synthetic aggregates of calcite with a 50% volume of muscovite. The tests were performed at 627–727 °C with a confining pressure of 300 MPa at constant shear strain rates of 3×10^{-5}–3×10^{-4} s^{-1} on cylindrical samples with the starting foliation parallel and perpendicular to the cylinder axis. Both the foliation parallel and the foliation perpendicular experiments show similar stress–strain patterns, with an initial hardening stage followed by significant strain weakening (>60%) before a catastrophic rupture. Microstructural analysis shows that in low-strain experiments calcite grains are intensely twinned while muscovite grains appear slightly bent and kinked. Higher strains promote a segregation of the two phases with calcite forming thin layers of fine, dynamically recrystallized grains, which act as localized shear bands, while muscovite grains keep their original size and rotate assuming a strong shape preferred orientation. This strain localization of the calcite from an initially homogeneous rock produced catastrophic failure at moderate bulk shear strains ($\gamma \sim 3$). Localization of the strain first involved ductile deformation to produce a new calcite layering with fine dynamically recrystallized grains along which cavities nucleated. The orientation and kinematics of the cavities are comparable to R1 Riedel structures. All experiments on calcite–muscovite mixtures resulted in heterogeneous strain. In these torsion experiments chemical changes and crystallization of new phases (anorthite and kalsilite) are observed at 627 °C. Whereas, samples hot pressed or deformed in compression at 670 °C did not show such reactions or any localization. The effect of stress-field geometry and pore pressure upon mineral reactions is discussed. It is concluded that deformation-induced heterogeneous phase distributions caused local strength differences initiating strain localization in the calcite–muscovite mixtures, eventually leading to plastic failure.

1. Introduction

In naturally deformed marbles, sheet silicates are the predominant minerals that form the foliation (Herwegh and Jenni, 2001; Leiss and Molli, 2003) but in many cases marbles are dominated by a strong compositional layering where the mineral phases are differentiated into a fine compositional layering (Molli et al., 2000) through a process of strain localization. The strength of these composite marbles also depends on the strengths of the constituent phases and their geometric arrangement, all of which may change with progressive strain. Experimental studies suggest that strain localization rarely occurs in a mono-phase material even after deformation to very large shear strains (e.g. $\gamma_{max} = 50$, in the case of Carrara marble Barnhoorn et al., 2004). Only few experimental deformation studies have been performed on two-phase aggregates, for example those on quartz–muscovite (Tullis and Wenk, 1994) and on calcite–anhydrite (Bruhn and Casey, 1997; Bruhn et al., 1999) showed that there is no strain localization at the sample scale in small strain, compression experiments. In contrast large-strain experiments on calcite–halite (Jordan, 1985), calcite–quartz aggregates (Rybacki et al., 2003), and calcite–anhydrite aggregates (Barnhoorn et al., 2005) strain localization from initially homogeneous rocks has been described.

There have been numerous experimental studies of the deformation of single crystals of various mica compositions (Etheridge et al., 1973; Mares and Kronenberg, 1993), demonstrating that they are able to undergo basal slip. Mariani et al. (2006) experimentally deformed fine-grained aggregates of muscovite up to shear strain of 2 at temperatures between 300 and 700 °C at pore pressure conditions suppressing the onset of dehydroxylation reaction. The authors inferred deformation to have occurred by a mixture of brittle/frictional and crystal-plastic

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processes with a distinct switch of mechanical response from strain rate independent to linearly viscous, below a shear strain rate of $1.4 \times 10^{-5}\ s^{-1}$. In addition, there have been several experimental studies of the effect of the mica volume percentage and orientation on the deformation of schists and gneisses under conditions where the micas are crystal-plastic but the other minerals (mostly quartz and feldspar) are brittle (Gottschalk et al., 1990; Shea and Kronenberg, 1993; Holyoke and Tullis, 2006). For instance the experiments of Shea and Kronenberg (1993) are all coaxial, at room temperature, 200 MPa confining pressure, and showed that after small amounts of axial shortening strain was localized in the mica rich zones with cataclasis of the quartzo-felspathic stronger phases. Similar features are described in the shear experiments of Holyoke and Tullis (2006) undertaken on gneiss at high temperatures (745 °C and 800 °C) and high confining pressures (1500 MPa). This produced an interconnection of the weaker biotites and stress concentrations and brittle failure in the stronger framework grains of quartz and feldspar. However, there is a paucity of experiments of two-phase aggregates that identifies the behaviour of the individual phases at high strains.

Many of the previous experimental studies on calcite aggregates, particularly Carrara marble, were obtained from coaxial compression and extension deformation experiments (e.g. Rutter, 1974, 1995; Schmid et al., 1980) where the maximum strains reached in the experiments are too small to result in steady-state deformation behaviour. Whereas, Pieri et al. (2001) and Barnhoorn et al. (2004), in large-strain torsion experiments, have achieved mechanical steady-state flow in Carrara marble. This involved deformation in the dislocation creep field with dynamic recrystallization starting at moderate strains ($\gamma \sim 1$). The accompanying strain weakening involves cycles of both subgrain rotation and grain boundary migration at approximately constant stress levels.

The important mechanical influence of a weak phase that may control creep during dynamic recrystallization has also been suggested as a mechanism for flow localization in crustal rocks (Rutter and Brodie, 1992; Handy, 1994; Handy et al., 1999). In this study we will investigate the mechanical influence and microstructural aspects related to strain localization using two-phase samples prepared from Carrara marble and a muscovite powder. Another mechanism that has been identified in this investigation is dilatant plasticity. Dilatant plasticity is known from deformation of metals and ceramics (Kassner and Hayes, 2003; Chokshi, 2005), where it limits the life of components that are subjected to high-temperature creep. The micromechanism for the generation of high-temperature dilatant pore spaces, which, in the ceramic literature are called microcavities, is related to diffusional grain boundary sliding. Here we present evidence for cavity nucleation, also known as cavitation, which accompanies a chemical reaction involving the muscovite and calcite.

This paper focuses on the rheology and microstructural evolution of a synthetic calcite rock where there is the addition of muscovite deformed to moderate temperatures of 600–727 °C and 300 MPa. Maximum bulk shear strains of about 4.5 were achieved before sudden shear failure and abrupt stress drop. Prior to failure strain localization and mechanical softening can be correlated to the formation of zones of reduced grain size and ductile deformation in the calcite. Using microstructural observations it will be shown that calcite is the weak phase. However, the rheology of the polyphase aggregate is significantly influenced by the addition of the muscovite and at large strains, phase transformations and void formation produce a mechanism of dilatant plastic flow. Finally, we address the question as to the role of muscovite in producing the mineralogical segregations that we see in many natural marbles. For it is often inferred, that the presence of sheet silicate minerals is one of the factors that contribute to the localization of deformation in zones of high-strain in natural rocks.

2. Experimental procedures

2.1. Starting material

We performed deformation experiments on 10 synthetic two-phase aggregates containing 50% volume muscovite and 50% volume calcite prepared following the procedure described by Schmidt et al. (2008). The calcite powder had a grain size ranging from 0.3 to 60 μm (average 10 μm); it was obtained from a block of Carrara marble described by Pieri et al. (2001). The muscovite, obtained from Minas Gerais, Brazil, was purchased as fine-grained platelets with average lengths ranging from 50 to 100 μm and with length to width aspect ratios of approximately 10:1. The powders were mechanically mixed and oven dried at 120 °C for 24 h and then cold pressed in stainless steel cylinders by stepwise filling and pressing of small portions (ca. 20 g) at the same load to achieve a homogeneous compaction. The pressing was done with an Enerpac-H-Frame 50-t-press up to a maximum stress of 400 MPa. The cold pressed samples were then annealed by hot-isostatically pressing (HIPing) at 170 MPa and 670 °C for 3 h to generate firm samples. The initial porosity of the HIPped material was calculated by difference between the geometrical volume of each sample and the volume measured with a helium pycnometer (AccuPyc 1330) and resulted in an average value of porosity of 20% (see Fig. 1(c)). The specimens used in the experiments were precision-ground from the larger sized HIPed sample (S27) to produce cylinders 12 mm in diameter and approximately 10 mm in length.

The calcite fraction in the starting material shows a wide grain size distribution ranging from ∼10 μm to 80 μm. The different grain size fractions are homogeneously distributed. The larger calcite grains are frequently strongly twinned, and overall no significant shape preferred orientation can be observed (Fig. 1(e)). The muscovite fraction also shows a wide grain size distribution with maximum grain sizes of 100 μm. The grains have their basal planes oriented normal to the compression direction and are often bent and kinked (Fig. 1(a)). The texture of the muscovite fraction was probably produced from rigid body rotation during compaction prior to the HIPing process.

2.2. Deformation experiments

Deformation experiments were performed in a Paterson-type gas medium apparatus (Paterson and Olgaard, 2000) in torsion and in compression testing at 300 MPa confining pressure, temperatures between $T = 627–727$ °C (Table 1) under undrained conditions. The samples were constrained between alumina spacers and zirconia end pistons within an iron jacket of 0.25 mm wall thickness and 15 mm diameter. The temperature during an experiment was monitored using a K-type thermocouple placed at about 3 mm from the top of the sample and the gradient along the sample axis was ±1 °C. Two type of stress configuration were tested in order to assess the role of the three principal stresses orientation on the high temperature, high pressure chemical stability of the aggregate.

2.2.1. Torsion experiments

Torsion tests allow simple shear deformation configuration with σ_1 and σ_3 inclined at 45° to the axis of the cylindrical sample