

The mechanical and microstructural behaviour of calcite-dolomite composites: An experimental investigation



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ABSTRACT

The styles and mechanisms of deformation associated with many variably dolomitized limestone shear systems are strongly controlled by strain partitioning between dolomite and calcite. Here, we present experimental results from the deformation of four composite materials designed to address the role of dolomite on the strength of limestone. Composites were synthesized by hot isostatic pressing mixtures of dolomite (Dm) and calcite powders (% Dm: 25%-Dm, 35%-Dm, 51%-Dm, and 75%-Dm). In all composites, calcite is finer grained than dolomite. The synthesized materials were deformed in torsion at constant strain rate (3×10^{-4} and $1 \times 10^{-4} \text{ s}^{-1}$), high effective pressure (262 MPa), and high temperature (750 °C) to variable finite shear strains. Mechanical data show an increase in yield strength with increasing dolomite content. Composites with <75% dolomite (the remaining being calcite), accommodate significant shear strain at much lower shear stresses than pure dolomite but have significantly higher yield strengths than anticipated for 100% calcite. The microstructure of the fine-grained calcite suggests grain boundary sliding, accommodated by diffusion creep and dislocation glide. At low dolomite concentrations (i.e. 25%), the presence of coarse-grained dolomite in a micritic calcite matrix has a profound effect on the strength of composite materials as dolomite grains inhibit the superplastic flow of calcite aggregates. In high (>50%) dolomite content samples, the addition of 25% fine-grained calcite significantly weakens dolomite, such that strain can be partially localized along narrow ribbons of fine-grained calcite. Deformation of dolomite grains by shear fracture is observed; there is no intracrystalline deformation in dolomite irrespective of its relative abundance and finite shear strain.

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1. Introduction

The styles and mechanisms of deformation associated with many variably dolomitized limestone shear systems are strongly controlled by strain partitioning between dolomite and calcite. Furthermore, the mechanical behaviour of shear zones that form in calcite–dolomite composites is likely a function of external parameters (e.g. P_c , P_p , T , and $\dot{\gamma}$), the mineralogy (calcite/dolomite content; (Delle Piane et al., 2009a)), and texture (e.g. grain size and

porosity) of the rock. Carbonate fault rocks can have heterogeneous distributions and variable contents of calcite and dolomite. For instance, fluid flow during thrusting can result in partial de-dolomitization (i.e. calcite formation) of carbonates resulting in heterogeneous distribution of calcite and dolomite in fault rocks (Erikson, 1994). Conversely, shear strain, in tandem with fluid flow, may result in a more dolomite-rich fault rock than the protolith due to the dissolution of calcite and subsequent passive enrichment of dolomite along thrust faults (Kennedy and Logan, 1997). Fault rocks derived from carbonate rocks can therefore be composed of variable amounts of dolomite and calcite, and grain size distributions within these rocks can be heterogeneous. In many shear zones, dolomite is demonstrably stronger than calcite, but the amount of dolomite required to significantly change the rheological behaviour of carbonate shear zones is poorly understood. Field observations suggest that dolomite may lead to the embrittlement of limestone

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(Viola et al., 2006). However, despite the common occurrence of calcite–dolomite composites, the influence of dolomite content on the strength of limestone under both ambient and high temperature conditions is poorly understood.

The deformation response of pure calcite and, to a lesser extent, pure dolomite under a variety of crustal conditions is well understood. Field observations suggest that under similar conditions of deformation, below amphibolite facies metamorphism, dolomitic rocks are stronger than limestone of similar grain size and porosity. During deformation, dolomite generally becomes highly fractured whereas calcite undergoes dislocation creep and dynamic recrystallization (Bestmann et al., 2000; Erikson, 1994; Woodward et al., 1988). Under similar experimental deformation conditions, dolomite rock is stronger and less ductile than limestone (Davis et al., 2008; Griggs et al., 1951, 1953; Handin and Fairburn, 1955; Higgs and Handin, 1959; Holyoke et al., 2013). At high temperatures (>700 °C), coarse grained dolomite is still stronger than calcite; however, fine-grained dolomite rocks (grains less than 15 µm in diameter) weaken significantly and can be weaker than calcite-rich rocks deformed under the same conditions (Davis et al., 2008; Delle Piane et al., 2009a; Delle Piane et al., 2008; Holyoke et al., 2013).

In this study, we address the role of coarse-grained dolomite on the strength and microstructural evolution of calcite–dolomite composites. Synthetic, hot isostatically pressed (HIP) calcite–dolomite (Cc–Dm) composites of four unique compositions – 1) 25%Dm:75%Cc, 2) 35%Dm:65%Cc, 3) 51%Dm:49%Cc, 4) 75%Dm:25%

Cc (hereafter designated by their dolomite content (%): Dm25, Dm35, Dm51, and Dm75) – were deformed in a torsion apparatus at elevated temperature and confining pressure to determine their rheological behaviour and to evaluate the effect of dolomite content and grain size on rock strength. A total of 13 rock deformation experiments were conducted at the following conditions: temperature (T) of 750°C, effective pressure (P_{eff}) of 262 MPa, imposed maximum shear strain rates ($\dot{\gamma}$) of $1 \times 10^{-4} \text{ s}^{-1}$ and $3 \times 10^{-4} \text{ s}^{-1}$, and total shear strains (γ) between 0.16 and 5.5. We observe that 1) in carbonate composites, even low dolomite contents greatly affect rock strength; 2) coarse-grained dolomite accommodates strain by brittle deformation in high dolomite content samples; and 3) calcite deforms by dislocation glide and diffusion creep assisted grain boundary sliding. Finally, we compare the experimental results to other studies and comment on their application to natural deformation environments.

2. Starting material

2.1. Starting powders and sample preparation

Two end member powders (coarse-grained dolomite and fine-grained calcite, described below) were mixed in varying proportions to produce four distinct compositions: Dm25, Dm35, Dm51, and Dm75.

Reagent-grade calcite powder (Minema 1™) (supplied by Alberto Luisoni AG, Mineral- & Kunststoffe) is characterized by equiaxed calcite grains exhibiting rare growth twins. The powder has a modal grain size of 9 µm (Fig. 1A), as measured with a Mastersizer 2000 laser diffraction particle size analyser (Malvern Instruments Ltd.). Rietveld refinement of XRD spectra (Raudsepp et al., 1999) of the calcite powder confirms its composition to be 99% CaCO_3 ; the remaining constituents are Mg, Al, Fe, and Si oxides.

A 4 kg block of Badshot marble, a natural dolomite marble from the Selkirk Mountains of British Columbia, was crushed to produce a powder with a broad grain size distribution and modal grain size of ~120 µm (Fig. 1A). Badshot dolomite is characterized by coarse dolomite grains (mean grain size of 477 µm, (Austin and Kennedy, 2005)) featuring lobate grain boundaries and fine, polygonal grains. Cleavage and twinning are prevalent in most grains (Austin, 2003; Austin and Kennedy, 2005; Austin et al., 2005). XRD analysis of the powder indicates a mineralogy that is ~99.8% dolomite. Thin section analysis reveals trace quantities (<<1%) of pyrite, apatite, calcite, tremolite, and white mica; these accessory phases are sufficiently low in abundance to be undetected by XRD analysis.

The powder mixtures were mechanically shaken to create homogeneous mixtures; the grain size distributions of the mixed powders are shown in Fig. 1B. The mixed starting powders have a bimodal grain size distribution, reflecting the dolomite proportion. The powder mixtures were then dried at 120°C for a minimum of 24 hours before being cold pressed into stainless steel, cylindrical canisters. The canisters were filled and pressed in 20g increments to produce homogenous packing of the powder along the canister length. This was done to avoid pressure shadow development during heat treatment. Pressing was done with an Enerpac-H-Frame 50 ton press up to a load of 40 tons, corresponding to a vertical stress of 200 MPa. A small volume of alumina powder with a porosity of ~30% was placed at the top and bottom of the canisters to act as a CO_2 sink for decarbonating dolomite. This ensured the migration of the emitted CO_2 to the storage areas, allowing the porosity to remain reduced in the rest of the canister.

All canisters were welded shut and, subsequently, hot isostatically pressed (HIP) to produce synthetic composite rock samples. The HIP was performed in a large volume, internally heated, argon gas apparatus under a confining pressure of 170 MPa (Delle Piane et al.,

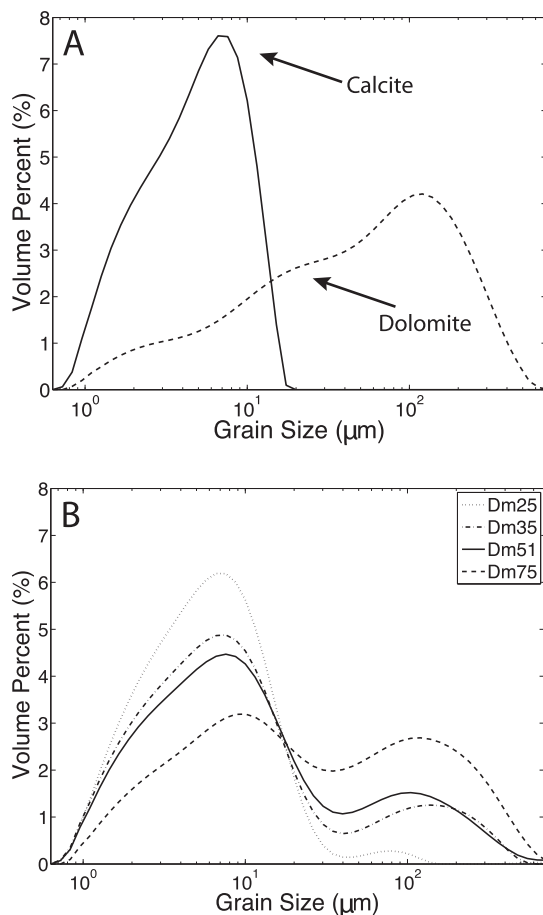


Fig. 1. Grain size distributions (vol.%) of starting material powders. A. Grain size distributions of pure calcite and pure dolomite powders. Modal grain sizes of the calcite and dolomite powders are 9 µm and 120 µm, respectively. B. Grain size distributions of calcite–dolomite powder mixtures used to fabricate the synthetic composites.

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