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Influence of stress, temperature, and strain on calcite twins constrained by deformation experiments

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ABSTRACT

A series of low-strain triaxial compression and high-strain torsion experiments were performed on marble and limestone samples to examine the influence of stress, temperature, and strain on the evolution of twin density, the percentage of grains with 1, 2, or 3 twin sets, and the twin width—all parameters that have been suggested as either paleopiezometers or paleothermometers. Cylindrical and dog-bone-shaped samples were deformed in the semibrittle regime between 20 °C and 350 °C, under confining pressures of 50–400 MPa, and at strain rates of ~10^{-4}–10^{-6} s^{-1}. The samples sustained shear stresses, τ, up to ~280 MPa, failing when deformed to shear strains $\gamma > 1$. The mean width of calcite twins increased with both temperature and strain, and thus, measurement of twin width provides only a rough estimation of peak temperature, unless additional constraints on deformation are known. In Carrara marble, the twin density, N_L (no of twins/mm), increased as the rock hardened with strain and was approximately related to the peak differential stress, σ (MPa), by the relation $\sigma = (19.5 \pm 9.8)\sqrt{N_L}$. Dislocation tangles occurred along twin boundaries, resulting in a complicated cell structure, which also evolved with stress. As previously established, the square root of dislocation density, observed after quench, also correlated with peak stress. Apparently, both twin density and dislocation cell structure are important state variables for describing the strength of these rocks.

1. Introduction

Twinning on e-planes $\{01\bar{1}8\}$ (using the hexagonal structural cell) is common during natural deformation of calcite rocks at temperatures, $T < 300$ °C (Barber and Wenk, 1979). At higher T, twin intensity is reduced, while glide on r-, f-, and c-planes is favored, owing to strong decreases in the critical resolved shear stress (CRSS) for dislocation glide (Barber et al., 2007). Because twins are visually striking and easily measured, they are often used to estimate deformation conditions in rocks containing quartz, calcite, dolomite, or pyroxene (Blenkinsop, 2000; de Bresser and Spiers, 1997; Masuda et al., 2011; Molli et al., 2011; Passchier and Trouw, 1996; Tullis, 1980; Turner, 1953; Wenk et al., 1983, 2006).

Paleopiezometers use twin microstructure to infer either the complete deviatoric stress tensor (Lacombe, 2007; Lacombe and Laurent, 1992; Laurent et al., 1981, 2000), or the magnitude of the difference of the greatest and least principal stresses (Jamison and Spang, 1976; Rowe and Rutter, 1990). The former class uses numerical inversion of twin orientations with constraints from failure or friction criteria, while the latter uses experimentally calibrated relations between twin frequency and stress magnitude. For example, Jamison and Spang (1976) estimate differential stress, σ, by computing the frequency of grains oriented such that the resolved stress on a given plane exceeds that necessary for twinning, τ_c. That critical value is given by the product of σ and a 'resolved shear stress coefficient', S_1, a function of grain orientation analogous to the Schmid-factor. For randomly oriented grains, Jamison and Spang (1976) calculated the frequency of grains for which $\sigma S_i > \tau_c$ where i indicates 1, 2, or 3 twin sets. In addition to assuming a random orientation distribution, this piezometer treats τ_c as constant and neglects stress inhomogeneities within the solid. In fact, τ_c depends on temperature and strain, ranging from $\approx$15 MPa to less than 5 MPa (de Bresser and Spiers, 1997; Laurent et al., 2000).

Rowe and Rutter (1990) proposed piezometers using three different measures: twin density (N_L = # twins/length), incidence (I_τ = # twinned grains/# grains total), and volume fraction of twins (V_τ = volume of twins/total volume). Over the temperature range from 200–800 °C, they found that all three varied directly with stress and were less dependent on temperature, strain, or strain rate; but the latter two also depended on grain size. Their twin density piezometer was

$$\sigma = -52.0 + 171.1 \cdot \log(N_L) \tag{1}$$

where stress was in MPa and twin density in # twins/mm.

All these methods contain assumptions that may limit their applicability (Blenkinsop, 2000; Burkhard, 1993; Ferrill, 1998). Most notably, the approach of Lacombe (2007) and Lacombe and Laurent (1992)

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assumes a constant CRSS for twinning and requires knowledge of rupture and friction laws or of the magnitude of at least one principal stress (e.g., from overburden). Moreover, to get stable results, this piezometer method also rejects incompatible twin sets. Although easier to use, the measures proposed by Jamison and Spang (1976) and Rowe and Rutter (1990) are supposed to be strain-independent, even though calibrations are limited to coaxial mechanical tests done only to low strains <20%. Additionally, most of Rowe and Rutter's experiments were performed in the range 400 °C < T < 800 °C, where deformation mechanisms other than twinning (e.g., dislocation glide) contribute substantial strain. The approaches of Jamison and Spang (1976), Lacombe (2007) and Lacombe and Laurent (1992) assume that τ_c is constant and neglects any dependence on grain-size, temperature, or strain (de Bresser and Spiers, 1997; Laurent et al., 2000). In fact, Ferrill (1998) suggested that stresses inferred for highly strained rocks may be overestimated by a factor of 20.

Twin morphology has also been proposed as a geothermometer for low-grade metamorphic conditions (Burkhard, 1993; Ferrill, 1991; Ferrill et al., 2004). From observations of carbonates deformed along the Swiss Helvetic nappes, Burkhard (1993) proposed a 4-fold classification of twin types to estimate formation temperature. Type I twins are thin (<1 μm), straight, and evolve at T < 170–200 °C; thick (>1–5 μm) type II twins are slightly lensoid, formed at T = 150–300 °C; type III twins are thick, curved, tapered, possibly re-twinned, accompanied by substantial dislocation glide, and occur at T > 200 °C; finally, type IV twins are thick, irregular, accompanied by grain boundary migration, and indicate T > 250 °C (Burkhard, 1993; Ferrill et al., 2004). The classification assumes that twin morphology depends largely on T and only to a minor degree on stress, strain, or strain rate. Hence, twin morphology is often used as a quick and easy geothermometer for the time of deformation (e.g., Janssen et al., 2004).

In the paper, we examined twin production under an extended range of temperatures, stress, and strain by observing the microstructure in carbonate samples from two series of deformation experiments at T < 350 °C to compare the temperatures and stresses in the experiments with those inferred from twin morphology and density, respectively. One set was deformed in coaxial compression to low axial strain, ε < 0.12; the second set was deformed in torsion to shear strains γ of up to 1.8.

2. Experimental and analytical techniques

For most experiments, the starting material was Lorrano Bianco marble from Carrara (Italy), a rock composed of >99% calcite with porosity <0.5%, and a homogeneous microstructure (Coli, 1989). The mean grain size, 220 ± 40 μm, was determined by the line-intercept method, corrected by a factor of 1.5 (Underwood, 1970). Grains have slightly sutured boundaries, but, when viewed in thin section, show little undulose extinction and random crystallographic-preferred orientation verified by electron-backscatter diffraction (EBSD) imaging (L. Morales, pers. communication, 2013). Grains larger than ≈150 μm contain twins >1 μm wide, with a mean twin density of 32 ± 12/mm. Transmission electron microscopy (TEM) shows straight or gently curved dislocations with a density of about 10^{12} m^{-2}. Two additional experiments were done on two other carbonates: fine-grained (7 ± 1 μm) almost untwinned Solnhofen limestone and coarse-grained (720 ± 240 μm) Kunzendorfer marble, which is often twinned in 2 sets with an initial twin density that is about half that of Carrara marble. See Fig. 1 for micrographs of the starting materials.

2.1. Deformation experiments

Using a Paterson deformation apparatus, samples were loaded in triaxial compression resulting in coaxial deformation to low shortening strains, or in confined torsion, resulting in simple shear to large shear strains (Fig. 2 and Tables 1–2). Details of the apparatus are given by Paterson (1970) and Paterson and Olgaard (2000). For the triaxial compression experiments, we prepared cylindrical samples 20 mm long and 10 mm wide. Samples were jacketed in copper sleeves with wall thickness ≈ 0.3 mm, and deformed without a pore fluid. We performed twelve constant strain rate (velocity) tests to 12% axial strain at confining pressures, P_c = 50–300 MPa, at strain rates, $\dot{\varepsilon} = 10^{-4}$–$10^{-6}$ s^{-1} and 20 < T < 300 °C (Fig. 3a). Axial force was measured inside the pressure vessel using an internal load cell. Jacket strength was determined by testing non-annealed solid copper samples at similar conditions and subtracted from the measured total force ignoring jacket hardening below about 3% strain. Axial force was converted to true axial stress accounting for sample distortion and assuming constant volume deformation. True (natural) strain and strain rates were determined from axial displacement corrected for system compliance. Error propagation suggests that uncertainties of stress and strain rate are <7% and 3%, respectively. In both the triaxial compression and the torsion tests, temperatures were measured by a thermocouple about 3 mm above the sample and kept constant within ≈2 °C. The temperature gradient along the sample was less than 2–3 °C, confirmed by calibration runs. The precision of the temperature measurements is about 0.5%. One sample was loaded isostatically to P_c = 300 MPa at 200 °C for several hours to examine the effect of heating/cooling and pressurization/depressurization on microstructure.

Torsion tests were performed at P_c = 400 and 370 MPa and T = 50 °C–350 °C (Fig. 3b). Nine samples were deformed at constant twist rate, yielding maximum shear strain rates of 10^{-4} s^{-1} at the periphery of the sample. Two samples were twisted at constant torque, producing a maximum shear stress of about 260 MPa. The applied torque is transmitted from the actuator to the sample only by friction, and, thus, the maximum transferable torque is limited by frictional slippage between the sample and pistons. The yield strength of the samples at low-temperatures is high, so we used cylindrical 'dog-bone'-shaped samples with a reduced diameter in the central part (Fig. 2b). In total, the samples were 30 mm long; the top and bottom sections (s1 and s5) were 15 mm in diameter, and the central section (s3) was 9.2 mm long and either 8 or 10 mm in diameter. Because the total torque applied to a transverse section is constant along the sample axis, and because shear stress increases with an inverse power of diameter, stresses within s3 are much larger than those elsewhere (see Appendix A and Paterson and Olgaard, 2000).

Torque was measured internally by means of an internal load/torque cell, and twist was measured by an external rotary velocity transducer. Samples were jacketed with copper sleeves with wall thicknesses of 0.2–0.4 mm, depending on diameter. The measured twist was corrected for apparatus compliance. Torques were corrected for jacket strength, as determined by calibration tests on low-strength Teflon (polytetrafluorethylene) and Nylon (polyamide) samples, jacketed with copper; by triaxial compression tests on solid copper and using data for the strength of copper (Copper Development Association Inc., Deutsches Kupferinstitut). The calculated strength of copper is between 150 and 80 MPa at T = 50° and 350 °C, respectively, with an uncertainty of 40% that accounts for jacket-hardening persisting at low shear strain (<0.1) and the pre-treatment of Cu-jackets by down-spinning in a lathe at a low rate of feed to ensure that they fit onto dog-bone shaped samples. Due to the relatively low experimental temperatures, jacket strength amounts to about 15% of the measured torque with an uncertainty of about 7%.

To determine the local shear stress, τ, and strain, γ, from measured torque, M, and twist, θ, in torsion tests, we used a simple power law as an approximate description of the mechanical behavior of the rock:

$$\dot{\gamma} = A\sigma^n e^{(-Q)/RT} \quad (2)$$

where $\dot{\gamma}$ is shear strain rate; n and Q are the stress sensitivity (stress exponent) and temperature sensitivity (activation energy), respectively.