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Strain localization during high temperature creep of marble: The effect of inclusions

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Abstract

The deformation of rocks in the Earth's middle and lower crust is often localized in ductile shear zones. To better understand the initiation and propagation of high-temperature shear zones induced by the presence of structural and material heterogeneities, we performed deformation experiments in the dislocation creep regime on Carrara marble samples containing weak (limestone) or strong (novaculite) second phase inclusions. The samples were mostly deformed in torsion at a bulk shear strain rate of $\approx 1.9 \times 10^{-4} \text{ s}^{-1}$ to bulk shear strains γ between 0.02 and 2.9 using a Paterson-type gas deformation apparatus at 900°C temperature and 400 MPa confining pressure. At low strain, twisted specimens with weak inclusions show minor strain hardening that is replaced by strain weakening at $\gamma > 0.1 - 0.2$. Peak shear stress at the imposed conditions is about 20 MPa, which is $\approx 8\%$ lower than the strength of intact samples. Strain progressively localized within the matrix with increasing bulk strain, but decayed rapidly with increasing distance from the inclusion tip. Microstructural analysis shows twinning and recrystallization within this process zone, with a strong crystallographic preferred orientation, dominated by $\{r\}$ and (c) slip in $\langle a \rangle$. Recrystallization-induced weakening starts at local shear strain of about 1 in the process zone, corresponding to a bulk shear strain of about 0.1. In contrast, torsion of a sample containing strong inclusions deformed at similar stress as inclusion-free samples but do not show localization. The experiments demonstrate that the presence of weak heterogeneities initiates

localized creep at local stress concentrations around the inclusion tips. Recrystallization-induced grain size reduction may only locally promote grain boundary diffusion creep. Accordingly, the bulk strength of the twisted aggregate is close to or slightly below the lower (isostress) strength bound, determined from the flow strength and volume fraction of matrix and inclusions.

1. Introduction

Strain localization in rocks deformed in the ductile field has been investigated in a plethora of field and experimental studies (e.g., Barnhoorn et al., 2005; Burlini and Bruhn, 2005; Paterson, 2007; Rutter and Brodie, 1992). The strength and deformation behavior of the continental and oceanic crust is generally assumed to depend on observation scale (Paterson, 2001). Localized heterogeneities like fault zones control the development of the brittle upper crust (Ben-Zion and Sammis, 2013; Faulkner et al., 2010), whereas high temperature shear zones impose constraints on the mechanical behavior of the lower ductile crust (Bürgmann and Dresen, 2008; Handy et al., 2007; Platt and Behr, 2011; Vauchez et al., 2012). Strain localization in the brittle regime may be described using damage rheology, continuum models or fracture mechanics-based approaches (Ashby and Sammis, 1990; Lyakhovsky and Ben Zion, 2014; Rudnicki and Rice, 1975). In the ductile field, localization may occur under varying thermodynamic boundary conditions and it may be associated with strain-dependent processes such as shear heating and melting, a change in the constitutive behavior by grain size reduction, enhanced water activity, or by the formation of a layered fabric in polyphase rocks (e.g., Montési, 2013; Getsinger et al., 2013).

The evolution of a high temperature shear zone by strain localization involves a nucleation and growth stage. In the brittle-ductile transition regime and at lower temperatures, shear zone formation often occurs at brittle precursors (Brander et al., 2012; Fousseis and Handy, 2008; Pennacchioni and Mancktelow, 2007; Pittarello et al., 2012), or by ductile fracturing (Dimanov et al., 2007; Menegon et al., 2013; Rybacki et al., 2008; Rybacki et al., 2010; Shigematsu et al., 2009; Spiess et al., 2012; White, 2012). Ultimately, strain weakening associated with localization may result from (e.g., Burlini and Bruhn, 2005): (1) grain size reduction by dynamic recrystallization or metamorphic reaction that produce new stress-free grains and/or a switch to grain size-sensitive deformation mechanisms, (2) structural softening in polyphase materials induced by the formation of interconnected weak layers at high strain, (3) geometric softening resulting from shape and crystallographic preferred orientation, (4) chemical weakening resulting in a change of point defects concentration, (5) fluid-induced dissolution-precipitation creep or reduction of the effective pressure causing embrittlement, (6) partial melting, leading to mechanical weakening and fast diffusion along wet grain boundaries, (7) transformation plasticity caused by mineral phase change, and (8) shear heating. In nature, the formation of a shear zone may be associated with a combination of these mechanisms, which holds also for the evolution of shear zones in the uppermost mantle (Kruckenberg et al., 2013; Newman and Drury, 2010; Precigout et al., 2007; Skemer et al., 2010).

In numerous experimental studies, such as high strain torsion and split-cylinder experiments, on quartz (Schmocker et al., 2003), olivine (Bystricky et al., 2000), feldspar (Rybacki et al., 2008), marble and limestone (Barnhoorn et al., 2004; Rybacki et al., 2003), it was found that single-phase rocks generally show minor localization, independent of the dominant deformation mechanism. Localization, however, appears to be more easily initiated at constant stress rather than at constant creep rate (Hansen et al., 2012a; Paterson, 2007). Two-

phase aggregates also show minor localization if deformed in the dislocation creep regime, for example in olivine-magnesiowüstite (Bystricky et al., 2006) and calcite-quartz aggregates (Rybacki et al., 2003). In contrast, multiphase rocks develop pronounced shear zones usually at a shear strain of $\gamma > 1$ if (1) deformed by linear viscous creep, particularly at low stress (Rybacki and Dresen, 2006), (2) in the presence of reaction products (Barberini et al., 2005; de Ronde et al., 2005; Delle Piane et al., 2007), and (3) through formation of weak interconnected layers formed by recrystallization and grain size reduction (Barnhoorn et al., 2005; Delle Piane et al., 2009; Holyoke III and Tullis, 2006; Ji et al., 2004). Compared to homogeneous two phase mixtures, layered polyphase can be, however, substantially stronger if compressed loaded normal to layering (Ji et al., 2000). Localized deformation may also result from formation of amorphous material at low temperature and high stresses (Pec et al., 2012) and by stress-driven segregation of melt at high temperatures (Holtzman et al., 2012; Holtzman and Kohlstedt, 2007; King et al., 2010; Kohlstedt et al., 2010; Misra et al., 2009).

In polyphase rocks, the evolution of localized shear zones is commonly observed in nature and in experiments. However, studies on the initiation of localization induced by single heterogeneities are rare and in natural shear zones appropriate locations are seldom exposed or difficult to identify. Here, we performed a systematic experimental and microstructural study on Carrara marble containing weak and strong inclusions to explore the initiation and growth of shear zones by heterogeneities in the ductile deformation regime.

2. Sample material and preparation

As starting material we used samples of Carrara marble, which is a low porosity (<0.5%), nearly pure (>99% CaCO_3) calcite rock. The mean grain size is $220 \pm 40 \mu\text{m}$. Little undulose extinction, some twins, a dislocation density of about 10^{12} m^{-2} , slightly sutured grain

boundaries, and a random crystallographic preferred orientation (CPO) indicate minor stored internal deformation. For torsion experiments, we used hollow cylinders of 10 mm length and 15 mm outer diameter with a 6.1 mm inner borehole filled with a gold cylinder. This procedure ensures a rather homogeneous stress distribution in the hollow sample cylinder (Paterson and Olgaard, 2000). For triaxial deformation tests, we prepared solid cylindrical samples with 10 mm diameter and 20 mm length. As inclusion material we used ≈ 0.75 mm thin slices of Solnhofen limestone and Arkansas novaculite (1 sample). The inclusions were glued into previously sawed slots in the marble cylinders. Solnhofen limestone is a very fine-grained (6 ± 2 μm) calcite rock with less than 2 vol % impurities. At the experimental conditions, Solnhofen limestone acts as a soft inclusion material (see below). Arkansas novaculite is a homogeneous, fine-grained (≈ 10 μm) and dense quartz rock (Wenk et al., 2006) that is considerably stronger than Carrara marble.

The sample assembly is shown in Figure 1. For torsion tests, the center angle of the slices is about 90° , corresponding to an arc length of ≈ 11.8 mm and a segment height of ≈ 2.2 mm (Figure 1a). For large strain tests we used samples with one inclusion, whereas for small strain experiments we used cylinders containing two inclusions on opposite sides. For the triaxial compression experiment, the single slice was oriented at 45° to the sample axis, extending from the cylindrical surface to the center (Figure 1b).

3. Experimental and analytical techniques

3.1 Deformation experiments

The samples were jacketed in copper sleeves of about 0.2 mm and 0.34 mm thickness for torsion and triaxial compression experiments, respectively. Subsequently, specimens were

deformed in a Paterson-type gas deformation apparatus at 400 and 300 MPa confining pressure in torsion and compression experiments, respectively (Paterson, 1970; Paterson and Olgaard, 2000). Temperature was $900^{\circ}\text{C} \pm 2^{\circ}$ along the sample axis. Heating and cooling rates were $\approx 30^{\circ}\text{C}/\text{min}$. All samples were kept under load during cooling to preserve the deformation microstructures.

In torsion tests the measured torque, M , was corrected for jacket strength and converted to maximum shear stress, τ , at the sample periphery using a hollow cylinder approximation (Paterson and Olgaard, 2000)

$$\tau = M \frac{4 \left(3 + \frac{1}{n} \right)}{\pi} \frac{D_o^{\frac{1}{n}}}{D_o^{3+\frac{1}{n}} - D_i^{3+\frac{1}{n}}} \quad (1)$$

assuming a power law behavior of the form

$$\dot{\gamma} = A \tau^n \exp \left(-\frac{Q}{RT} \right) d^{-m} \quad (2)$$

where M is torque (Nm), τ is shear stress (MPa), D_o is the outer cylinder diameter and D_i is the diameter of the inner hole, $\dot{\gamma}$ is shear strain rate (s^{-1}), A is a material dependent constant ($\text{MPa}^{-n} \mu\text{m}^m \text{s}^{-1}$), n is the stress exponent, Q is the activation energy (kJmol^{-1}), R is the molar gas constant ($\text{kJmol}^{-1} \text{K}^{-1}$), T is absolute temperature (K), d is grain size (μm), and m is the grain size exponent. The maximum shear strain was determined from measured angular displacement and sample dimensions after correcting for system compliance.

In triaxial compression tests the measured force was corrected for the jacket strength and converted to axial stress assuming constant volume deformation. The axial strain was determined from measured axial displacement and corrected for system compliance. For both test types, the error in bulk stress and strain is less than 4%. From geometrical constraints, the

shear stress τ and strain γ in the planar inclusion of the triaxial sample assembly can be estimated from axial stress, σ , using the relations

$$\tau = \frac{1}{2} \sigma \sin(2\alpha) \quad (3)$$

and

$$\gamma = \frac{\varepsilon L}{d \cos(\alpha)} \quad (4)$$

where L ($= 20$ mm) is the initial sample length, d ($= 0.75$ mm) is the thickness of the slice, and α ($= 45^\circ$) is the angle between inclusion and sample axis. Application of equations (3) and (4) assumes a full (laterally unconstrained) split cylinder sample assembly configuration with constant inclusion thickness and contact area (Tembe et al., 2010).

3.2 Microstructure analysis

Microstructures were inspected using optical and electron microscopy on thin sections, prepared close to the highly strained rim of the twisted samples and oriented parallel to the axis of the deformed cylinders (longitudinal tangential section, Paterson and Olgaard, 2000). The approximate shear strain γ_{sec} in any section cut at radius r_{sec} can be calculated from the measured maximum shear strain γ_{max} at the outer surface of the sample cylinder with radius r from the equation

$$\gamma_{sec} = \frac{r_{sec}}{r} \gamma_{max} . \quad (5)$$

The radius r_{sec} at the centre of a section of width s is given by

$$r_{sec} = \frac{1}{2} \sqrt{4r^2 - s^2} . \quad (6)$$

All thin sections were polished, using first diamond paste to $0.25 \mu\text{m}$ finish and then using an alkaline solution of colloidal silica for two hours. Thin sections were first analyzed optically.

For seven thin sections, crystallographic orientations of calcite grains were determined via automated indexation of electron backscatter patterns (EBSD) in a scanning electron microscope (SEM). The EBSD measurements were conducted on uncoated thin sections using a FEI Quanta 3D FEG dual beam machine equipped with an EDAX-TSL Digiview IV EBSD detector and the TSL software OIM 5.31 operated at GFZ in Potsdam. The SEM parameters were: accelerating voltage of 15 kV, beam current of 8 nA, working distance of 15 mm, and step size of 5 μm . Average indexation rates for all samples were always above 90% without filtering. Post-acquisition treatment included standardization of the confidence index (CI) of different points and CI correlation between neighbour points, assuming a CI of 0.2. One step of grain dilation was carried out in each dataset considering a minimum grain tolerance angle of 10° and a minimum grain size of 10 correctly indexed pixels. After processing, all remaining points with CI values lower than 0.2 were removed to avoid incorrect measurements.

Calculations of the orientation distribution function and pole figure contouring were carried out using the MTEX toolbox for Matlab (Hielscher and Schaebe, 2008; Bachmann et al., 2010), using a Gaussian half-width of 10° and a maximum harmonic expansion factor (L_{max}) of 32. The fabric strength was also characterized, represented by the dimensionless J-index ($J = \int f(\mathbf{g})^2 d\mathbf{g}$), where $f(\mathbf{g})$ is the volume fraction of orientations with an orientation between $\mathbf{g}$ and $\mathbf{g}+d\mathbf{g}$ and $d\mathbf{g}$ is the volume of the region of integration in orientation space. For calcite, J-index values vary from 1 for a random fabric to a maximum of ~ 95 for a single crystal. The E-W vertical shear plane and the E-W horizontal shear direction, marked on each pole figure, define the external reference frame for the pole figures. Pole figures, misorientation angle distribution and grain size histograms were calculated for grains in the process zone (adjacent to the tip of the inclusion, see below) and not for the whole orientation maps. Grain boundaries were defined by comparing misorientation angle between neighbouring points in

the EBSD maps. High-angle grain boundaries include all grain boundaries with misorientation angles $> 15^\circ$, whereas for low angle grain boundaries the misorientation varies between 1 and 15° . Grain size was determined via mean intercept length, considering a scaling factor of 1.5 (Hansen et al., 2012b; Underwood, 1970). Transmission electron microscopy (TEM) analyses were performed on $0.15\text{ }\mu\text{m}$ thick foils prepared by the focused ion beam technique (Wirth, 2004) using a FEI Tecnai G2 F20 X-Twin TEM.

4. Results

4.1 Mechanical behavior

In total, we performed 7 torsion tests on inclusion-bearing samples using hollow cylinders and one compression test using a solid cylinder. To evaluate the strength of matrix and inclusion materials, we conducted 4 torsion tests and 3 triaxial compression experiments on pure Carrara marble (CM) and Solnhofen limestone (SH) samples (Table 1). Except for constant load (creep) and strain rate stepping experiments, axial tests were done at constant axial strain rate of $\approx 7.2 \times 10^{-5} \text{ s}^{-1}$. Torsion tests were performed at constant twist rate yielding a maximum shear strain rate of $1.9 \pm 0.1 \times 10^{-4} \text{ s}^{-1}$ at the sample cylinder surface.

The measured stress-strain curves of shortened and twisted inclusion-free samples are shown in Fig 2a and 2b, respectively. At low strain (< 0.2), the samples show nearly steady state strength, followed by weakening for CM up to a shear strain of ≈ 2 . The reproducibility of measured stresses is $< 5\%$ as indicated by triaxial compression of solid samples CMAS4 and CMAS5 (Fig. 2a) and by torsion of samples CMTT6 (solid cylinder) and CMHT2 (hollow cylinder, Fig. 2b). At the imposed experimental conditions and a strain γ of ≈ 0.05 , CM is about 23 times stronger than SH in compression and about 9 times stronger in torsion,

respectively. Fig. 2c shows a log-log plot of the maximum shear stress strain-rate data measured in compression and torsion, including results of constant stress (sample CMTT7) and constant strain rate (sample SHTT2) stepping tests (Table 1). Axial stress and strain rate data were converted to similar equivalent stress and strain rate data using (Paterson and Olgaard, 2000):

$$\tau = \frac{1}{\sqrt{3}}\sigma \text{ and } \dot{\gamma} = \sqrt{3}\dot{\epsilon}, \text{ respectively.} \quad (7)$$

The best-fit slopes yield stress exponents of $n = 1.4$ for SH and $n = 7.6$ for CM, indicating diffusion and dislocation-controlled creep, respectively. For comparison, torsion tests on Solnhofen limestone at $T = 927^\circ\text{C}$ yielded $n = 1.7$ (Rybacki et al., 2003) and $n \approx 11$ and 7.4 for Carrara marble twisted at $T = 927^\circ\text{C}$ and 727°C , respectively (Barnhoorn et al., 2004; Pieri et al., 2001a). At a shear stress of about 20 MPa (vertical dotted line in Fig. 2c), which corresponds to the average bulk shear stress measured in inclusion-bearing hollow samples (Table 1), the creep rate of CM is about 1.8% of SH and $\approx 10^4$ times higher than of Novaculite, estimated from the flow law for wet flint (Mainprice and Paterson, 2005). For the applied bulk shear strain rate of about $1.9 \times 10^{-4} \text{ s}^{-1}$, CM is ≈ 9 times stronger than SH and ≈ 10 times weaker than NC (horizontal dotted line in Fig. 2c).

The influence of a weak SH-inclusion on the bulk stress strain behavior of a solid CM sample deformed in triaxial compression is shown in Fig. 2a (sample CMAS1). With respect to the strength of inclusion-free samples (CMAS4, 5), it appears to have lower yield strength and a more pronounced initial hardening behavior.

In torsion, the mechanical behavior of SH-bearing samples deformed at constant strain (twist) rate is also marked by a smaller creep strength compared to inclusion-free samples (represented by sample CMHT2 in Fig. 3a, cf. Fig. 2b), but with almost similar strain

hardening/weakening behavior. The shear stress level of intact samples is on the order of 20 MPa, quite similar to that measured by Pieri et al. (2001a) on Carrara marble in high strain torsion tests at 927°C temperature and a strain rate of $3 \times 10^{-4} \text{ s}^{-1}$. One sample (CMHT1) showed an oscillating stress at high strain. This is likely related to some offset between the two sample parts above and below the sheared region containing the inclusion. The sample to sample variation of strength at $\gamma = 0.05$ is up to 8% with mostly lower (but overlapping) values for samples containing 2 SH-inclusions (CMHT9, 8, 4) compared to those with 1 inclusion (CMHT1, 3). Compared to inclusion-free marble, the samples containing weak inclusions are 4-12% weaker (average 8%). Therefore, samples with inclusions appear to be slightly weaker than solid specimens, irrespective of reproducibility. In contrast, sample CMHT5 with strong novaculite inclusions is almost as strong as the inclusion-free rocks (Fig. 3a).

The measured deformation of sample CMHT6, twisted at a constant stress (torque) of ≈ 17 MPa (cf. Fig. 3a) is shown in Figure 3b. The initial shear strain rate of about $2.4 \times 10^{-4} \text{ s}^{-1}$ first slightly decreased to a bulk shear strain of ≈ 0.1 -0.2. Subsequently shear strain rate rapidly increased up to $\approx 6.6 \times 10^{-4} \text{ s}^{-1}$ approaching a constant value at high strain (>0.9). This change in strain rate with increasing bulk strain is remarkably similar to the transition of strain hardening to weakening in constant strain rate tests that also occurred at $\gamma \approx 0.1$ -0.2 (Fig. 3a). The weakening occurs irrespective of the presence of inclusions (compare samples CMHT2 and CMTH3 in Fig. 3a).

4.2 Strain localization

The distorted shape of the (initially rectangular) inclusions in deformed samples mimics the imposed intense shear deformation in the plane of the inclusions (Fig. 1 c, d). The

macroscopic strain distribution of twisted samples within the weak inclusion and in the adjacent regions above and below were estimated from the inclination of initially vertical scratches on the jacket surface, which served as markers. At experimental conditions, shear strain of the weak inclusions is considerably larger than the bulk strain (Fig. 4a). In contrast, the strain within the matrix above and below the inclusions is less than the bulk strain (dotted line in Fig. 4b). Interestingly, at high bulk deformation the measured matrix strain ($\gamma = (\tan \alpha)^{-1}$), estimated from the inclination angle α of the jacket scratches, is less than the difference of measured bulk and inclusion twist. This discrepancy is attributed to strain localization at the inclusion tip. The ratio between inclusion and matrix strain increases to about 24 at a bulk strain of 2.9 (Fig. 4c). It should be noted that in torsion testing the torque (mean stress) is similar for all sections perpendicular to the cylinder axis of a homogeneous material. Therefore, for the section containing the inclusions stresses are locally enhanced in the matrix and reduced in the inclusion in order to maintain the total torque in this section equal to any other section.

At low bulk strain the measured strain ratio is considerably lower than the calculated strain ratio, which is close to the expected ratio of 56 predicted by the contrast in viscosity between the pure phases (cf., Fig. 2c).

Strain localization as indicated by sheared markers decreases rapidly within about 1-3 mm distance ahead of the inclusion tip (Fig. 5; sample CMHT8, bulk shear strain of 0.05). In addition, the width of the localization zone broadens approximately 5 times from 0.75 mm at the inclusion tip to about 3.7 mm at about 5 mm distance. As indicated by the stippled border in Fig. 5a, the shape of the localized zone is nearly elliptical between the 2 inclusions (the second inclusion is not shown here). The maximum strain ratio between the highest strain in the center of this zone and the residual strain in the matrix outside the stippled area decreases

approximately 4 times from about 13 at the inclusion tip towards a value of ≈ 3 about 5 mm apart (Fig. 5b). For samples with two inclusions, deformed tips will start to interact at some finite shear strain.

4.3 Microstructures

Localized deformation in front of the tip of soft inclusions resulted in enhanced undulose extinction, twinning, subgrain formation and recrystallization with progressive shear strain (Fig. 6a). This localization-related region of enhanced microstructural modification compared to the remaining matrix is called the process zone. Twin orientation is quite variable and some grains developed two conjugate twin sets with an orientation difference of $\approx 60^\circ$ between twin planes which are bent in places. With increasing bulk strain, development of subgrain boundaries in twinned and non-twinned crystals and intense grain size reduction becomes more pronounced and the length of the process zone increases (Fig. 6a-b, e-g). Strain values of the shown thin section areas are recalculated from the measured final bulk strain using Eqn (5) (Tab. 1). At $\gamma_{\text{sec}} \approx 0.2$, a second foliation (S_2), defined by the oriented alignment of grain boundaries and subgrain walls at about 40° to the shear plane (S_1) appears (Fig. 6e). Twins are less frequent in the process zone, but when present they are thicker and bent (Fig. 6f). At these strain conditions, subgrains have similar dimensions as the recrystallized grains and the subgrain walls are oriented parallel or at high angles to S_1 . Recrystallized grains are twin free and locally lobate grain boundaries are evident (Fig. 6b, e-f). At the highest imposed bulk shear strain of $\gamma \approx 2.9$, corresponding to a shear strain γ_{sec} of ≈ 1.9 in the inspected thin section, a continuous dextral shear zone evolved, marked by intense grain size reduction.

In comparison, the microstructure resulting from shear deformation of sample CMHT5 containing hard (novaculite) inclusions is different from that of sample CMHT4 with soft

(Solnhofen limestone) inclusions, both deformed to the same bulk strain of $\gamma \approx 0.1$ (Tab. 1, Fig. 6 b-d). Adjacent to the hard inclusions little evidence of plasticity is present, but small wollastonite grains formed at experimental conditions by the reaction of calcite + quartz $\leftrightarrow$ wollastonite + carbon dioxide (cf., Rybacki et al., 2003). The CO₂ production induced abundant pores (Fig. 6d). At larger distance to the tip, the grains are intact and the presence of twinning is common as in the other low strain experiments. If not induced by unloading, the appearance of straight vertical cracks in the hard inclusion (Fig. 6c, d) indicates enhanced internal stresses, which are obviously high enough to induce tensile fracturing irrespective of the high confining pressure.

The microstructures of the triaxial compression experiment CMAS1 (Fig. 6h, i) with the weak inclusion orientated at 45° to σ_1 are to some extent similar to the torsion experiments at very low shear strains <0.05 (Fig. 6a). Using Eqn (4) the estimated local shear strain of sample CMAS1 is 0.58, comparable to local shear strain $\gamma = 0.44$ determined from the change of the inclusion shape (Tab. 1). Twinning is a common feature all over the sample, but twin density, defined as the number of twins per unit length, is relatively low, whereas subgrain boundaries predominantly occur in the process zone. Initial grain size reduction is observed in the region around the inclusion and grain-scale fractures parallel to the inclusion tip of the inclusion occur (Fig. 6i), locally displacing twin planes.

4.4 Crystallographic preferred orientations

The development of a clear CPO of calcite grains in front of the weak inclusion starts at bulk strains of ≈ 0.05 (Fig. 7). At these conditions, calcite [0001] axes are aligned at $\approx 20^\circ$ to the shear plane pole, whereas the poles of {11-20} develop weak incomplete girdles parallel to the shear plane (Fig. 7b). With increasing strain the [0001] pole maximum becomes stronger,

which is not observed for the other crystallographic axes (Fig. 7c, d). At a shear strain γ_{sec} of ≈ 0.2 in the thin section a pronounced CPO is observed. The [0001] axes are distributed along a single continuous girdle normal to the shear plane with two maxima, inclined by $\approx 40^\circ$ with the pole of the shear plane (Fig. 7d). The poles of {11-20} are predominantly parallel to the shear direction and the poles of {10-14} are predominantly parallel to the shear plane pole. The {01-12} poles are aligned in symmetrical maxima, oriented with an angle of $\approx 45^\circ$ to the shear direction.

In sample CMHT1, deformed to $\gamma = 2.9$ ($\gamma_{sec} \approx 1.8$), the CPO is different and marked by a single maximum of [0001] axes oriented at low angle with the shear plane pole, with slightly curved contours lines (Fig. 7e). The poles of {11-20} are distributed along a continuous girdle parallel to the shear plane, with a maximum at $\approx 25^\circ$ with the shear direction. {10-14} poles are distributed in a complex way and the {01-12} poles are distributed in three maxima, two of them forming an angle of $\approx 35^\circ$ with the shear plane. The fabric strength of the calcite grains in front to the inclusion tip increases progressively with increasing strain from $J \approx 2$ to $J \approx 8$, which is substantially higher than the values measured for solid Carrara marble deformed in torsion at high temperature and similar bulk shear strains (Barnhoorn et al., 2004; Barnhoorn et al., 2005; Bruijn et al., 2011). The difference is attributed to the high local strains in front of the inclusion (Fig. 5) that enhances the CPO strength compared to solid samples deformed to similar bulk strain.

The CPO of sample CMAS1, deformed under triaxial compression, is very weak. It is characterized by a weak girdle of [0001] axes oblique to the shear plane, defined by the initial plane of the inclusion, {11-20} poles varying from normal to oblique to the shear plane and poles of {10-14} parallel to the shear direction, which is parallel to the shear plane (Fig. 7f).

Reoriented to similar reference frame orientation, this distribution is similar to the one observed in sample CMHT9 (Fig. 7a), twisted to low shear strain of 0.015.

5. Discussion

The observed microstructures suggest that stresses are locally enhanced in front of weak SH inclusions leading to progressive strain localization and recrystallization of the Carrara marble matrix grains with increasing strain (Fig. 6). This is accompanied by brittle fracturing in triaxial deformation (Fig. 6h), which may probably activate grain size sensitive deformation processes without need of high (local) strain to initiate recrystallization. In contrast, the microstructures of the sample containing strong NC inclusion suggest a strong stress concentration inside the inclusion and possibly reduced stresses ahead of the inclusion tip (Fig 6b, c). In the following, we briefly discuss the inclusion-related texture evolution, the associated stress and strain enhancement in the process zone, where changes of microstructures and local strain occur, and the bulk aggregate strength.

5.1 Deformation mechanisms and fabric evolution at the tip of weak inclusions

At low shear strains ($\gamma_{sec} < 0.06$) the microstructures are dominated by twinning, mainly in the process zone in front of the weak inclusions. Twin lamellae are predominantly thick and the twin density is relatively low (Fig. 6a), which is typical for twinning at high temperature and low stress conditions (Burkhard, 1993).

At low strain, the misorientation angles between calcite grains show a peak at 75°-80° and misorientation axes are oriented predominantly parallel to equivalent [0-221] and [2-201] directions of calcite by cyclic permutation of [uvt] in [uvtw] (Fig. 8a). Together with the angle between the [0001] axes of host and twinned crystal of $51 \pm 1^\circ$, the misorientation suggests that

e-twinning is predominant (Barber and Wenk, 1979). At low strain in the process zone we also observed intense undulose extinction and the presence of subgrain boundaries in some crystals. The EBSD data-derived Kernel average misorientation maps show that internal misorientation angles of grains within the process zone are higher than in grains outside this zone (Fig. 9a, b). In Kernel average misorientation maps, the local misorientation at a given point is determined by the average misorientation of that point with all of its neighbors, excluding grains that exceed a certain misorientation angle. Here, we used a Kernel considering up to the third nearest neighbor points. The regions with intense undulose extinction are marked by enhanced dislocation density (Fig. 10a), and partially subgrain boundaries formed by arrays of dislocations (Fig. 10b).

With increasing strain, intense grain size reduction at the contact between inclusions and host rock increasingly affects a larger fraction of the matrix (Figs. 6, 9). In addition, a secondary foliation evolves (Fig. 6e), associated with dynamic recrystallization. The development of secondary foliation planes were also observed in experimentally twisted monophase anorthite aggregates, forming localized shear zones that may have induced strain softening (Ji et al., 2004). In the process zone, progressive recrystallization with increasing strain at the expense of initially twinned grains shifts the peak of misorientation angles between grains from 75-80° to 5-10° (Fig. 8b). Abundant subgrain walls, recrystallized grains of similar size as subgrains and predominantly low misorientation angles (Figs. 6e-g) suggest the activation of subgrain rotation recrystallization (e.g., Guillopé and Poirier, 1979), whereas the presence of lobate/sutured grain boundaries (Fig. 6e, f) indicates localized grain boundary migration recrystallization (Urai et al., 1986). The occurrence of both processes may indicate cyclic dynamic recrystallization (Lloyd and Freeman, 1991, 1994; Pieri et al. 2001b). This is supported by average intragranular misorientation in low strain samples, showing intracrystalline misorientation of 1-4° in front of the inclusion (Fig. 9a, b). Dynamic

recrystallization at higher strain reduces intragranular misorientation angles to 0-1° (Fig. 9c), likely associated with decreasing dislocation density. Progressive deformation then results in a new cycle of dislocation nucleation, increasing dislocation density, development of subgrain boundaries and recrystallization, leading to further grain refinement, as observed in the highly deformed sample CMHT1 (Fig. 9d). In this sample, grains with high misorientation angles are more widespread. Cyclic dynamic recrystallization may be also indicated by the presence of a secondary oblique foliation plane (Fig. 6e), marked by aligned recrystallized grain boundaries (Bruijn et al., 2011).

Within the process zone ahead of the weak inclusions a CPO indicating shear deformation starts to develop at a bulk shear strain of ~ 0.05 ($\gamma_{sec} < 0.045$), with maxima normal to the shear plane and at 45° to the shear zone boundary (Fig. 7b) (Barnhoorn et al., 2004; Pieri et al., 2001a). However, CPO patterns do not allow identifying active slip systems. With increasing local strain in front of the inclusion, the CPO becomes stronger. The CPO pattern of the sample CMHT3 suggest the activation of predominant $\{r\}\langle a \rangle$ slip, accompanied by activation of other slip systems in a common $\langle a \rangle$ direction, resulting in the girdle development of [0001] axis normal to the shear plane (Fig. 7d). Similar CPO patterns were obtained by Pieri et al. (2001a) in torsion tests on solid Carrara marble at similar temperature conditions. Bulk shear strains in the Pieri et al. experiments that were more than one order of magnitude larger but comparable to the local shear strains at inclusion tips. The CPO measured ahead of the inclusions in highly deformed samples shows a typical pattern for fine-grained dynamically recrystallized calcite as observed in many studies of naturally and experimentally deformed calcite mylonites (e.g., Austin et al., 2008, Behrmann, 1983; Barnhoorn et al., 2004; Bestmann and Prior, 2003; Bruijn et al., 2011; Ebert et al., 2007a; Herwegh and Kunze, 2002; Pieri et al., 2001a; Pieri et al., 2001b; Schmid et al., 1987). These patterns have been attributed to grain boundary migration recrystallization (GBMR) (Schmid

et al., 1987, Pieri et al., 2001a; Pieri et al., 2001b). Based on modeling, we show in the following that these patterns may result from high strain deformation alone without pervasive dynamic recrystallization.

To simulate the CPO of sample CMHT3 ($\gamma_{sec} \approx 0.2$), we modeled the fabric development during simple shear of calcite aggregates using the viscoplastic self-consistent (VPSC) approach (Lebensohn and Tome, 1993), adopting the relative critical resolved shear stresses for basal, f-slip and r-slip used established by de Bresser and Spiers (1990) and Pieri et al. (2001b), respectively. For the simulations we considered an aggregate consisting of 1000 randomly orientated spherical grains, which was deformed in strain increments of 2.5% each up to a von Mises equivalent strain of 250% ($\gamma = 4.33$). Further, we assumed a stress exponent of 9 and a hardening factor of 0.01 for all slip systems (e.g., Pieri et al., 2001b).

Our modeling produced very similar CPO patterns although the measured CPO was slightly stronger (Figs. 7d and 11a). In addition, the results are similar to those obtained by Pieri et al. (2001b, their Fig. 9) with some variations of the pole figure scales due to different parameters used for the calculation of the orientation distribution functions. In our high-strain model ($\gamma=4.3$) the poles of (0001) are concentrated at low angle to the pole of the shear plane, the poles of {11-20} are predominantly parallel to the shear direction, the {10-14} planes are parallel to the shear plane, and the poles of {01-12} form two maxima at about 45° with the shear direction (Fig. 11b). Successful simulation of the CPO pattern requires relatively high strain and dominantly {r}<a> slip, whereas the {f}<a> slip system becomes less important (Fig. 11c), which is in accordance with the high local strain (maximum 2.8, Tab. 1) in front the inclusion of sample CMHT3. Although Pieri et al. (2001b) argued that the simulated CPO requires recrystallization to occur, we successfully simulated the same pattern at high strain without accounting for recrystallization. The difference may be explained by the

recrystallization model used by Pieri et al. (2001b) for CPO modeling. In this nucleation recrystallization model, grains in “soft orientation” (easy-slip positions) start to nucleate if the “recrystallization” is initiated. These nuclei grow at the expense of grains in harder orientations. The “recrystallization step” affects the intensity of the preferred orientations, but not the CPO patterns that remain similar with or without recrystallization. However, at higher strains (e.g., sample CMHT1 with a local shear strain up to ≈ 15) modeling of the CPO was not successful unless we assume that the (c)<a> slip system is slightly weaker and the {r}<a> system slightly stronger than used for modeling of sample CMHT3. This strongly suggests that at high strains dynamic recrystallization is important for the CPO development and should not be neglected in modeling.

5.2 Stress concentration

Twinning is associated with strain hardening of the samples (e.g., Barnhoorn et al., 2004; Rybacki et al., 2013). The average twin densities (number of twins/mm), measured on thin sections of samples CMHT8, 3, 4 and CMHT5 in successive 1 mm steps in front of the inclusions, asymptotically decrease from ≈ 67 twins/mm directly adjacent to the inclusion to ≈ 35 twins/mm 5 mm apart, irrespective of the imposed bulk shear strain. Increased twin densities in the process zone and the presence of conjugate twin families in some grains in front of the inclusion (Fig. 6d) suggest local stress concentration. To further estimate the stress concentration at the weak inclusion tip, we applied existing paleo-piezometers for calcite rocks based on dislocation density and recrystallized grain size.

To our knowledge, this study presents the first experimental data on stress and strain distribution around single inclusions in creeping natural materials. So far, several studies exist, which estimate the effective elastic and flow properties of composite materials, based

on the properties of the constituents and their relative amount and distribution (Covey-Crump et al., 2013; Dimanov and Dresen, 2005; Dimanov et al., 1997; Takeda and Griera, 2006; Zhao and Ji, 1997; Zhou et al., 2012). Also, the strain-induced rotation and motion of individual or multiple clasts within a deforming matrix was investigated in analytical and numerical studies (Jiang, 2013; Mancktelow, 2011, 2013; Treagus, 2002; Treagus and Treagus, 2001). Numerical approaches were used to study localization induced by shear heating or inclusions (Kaus and Podladchikov, 2006; Mancktelow, 2002; Mandal et al., 2004; Mandal et al., 2003; Misra and Mandal, 2007; Regenauer-Lieb et al., 2006; Takeda and Griera, 2006).

Estimates of stresses in the process zone may be obtained by applying calibrated piezometers based on dislocation density and recrystallized grain size. Application of these piezometers assumes that the microstructures are in equilibrium with the thermodynamic boundary conditions, which is probably not the case in the process zone. Therefore, the stress estimate using the recrystallized grain size may give a lower limit if the measured grain size is still larger than the equilibrium grain size since the piezometer-derived stress is roughly inversely proportional to grain size. Using densities of free dislocations, ρ , to estimate flow stress, ($\sigma \sim \sqrt{\rho}$), may give at least an upper bound.

Dislocation densities within calcite grains were estimated from transmission electron microscope images (Fig. 10). The dislocation densities (total length of free dislocation lines per unit volume measured by the line intercept method, for details see Rybacki et al., 2011) in calcite in front of the soft inclusions in samples CMHT1, CMHT8 and CMAS1 yield locally high values ranging from $2.9 \times 10^{13} \text{ m}^{-2}$ close to the inclusion tip to $1.7 \times 10^{13} \text{ m}^{-2}$ and $0.8 \times 10^{13} \text{ m}^{-2}$ measured about 0.5 and 1.5 mm in front of the tip, respectively. These densities correspond to differential stresses of 127, 98, and 78 MPa when applying the piezometer

calibrated by de Bresser (1996) on calcite single crystals during compression at temperatures of 550-800°C. The calculated far field (bulk) equivalent stress is about 33 MPa (corresponding to a shear stress of 19.1 MPa, cf. Eqn 7) at the thin section position ((Paterson and Olgaard, 2000, Eqn. (31), assuming a stress exponent of $n = 7.6$)). Accordingly, the estimated stress in the process zone is about 3.8-2.4 times higher than in the bulk, decaying with increasing distance from the tip. In front of the hard inclusion of sample CMHT5 the estimated free dislocation density is $\approx(0.5-1.7) \times 10^{13} \text{ m}^{-2}$, yielding stresses between 44 and 98 MPa, i.e. 1.3 – 3.0 times higher than the far field stress. Note that a stress enhancement of a factor of 2 requires just a 3 times higher dislocation density, which is just above the uncertainty of dislocation density measurements.

We also estimated the size of recrystallized grains on polished thin sections using an optical microscope by measuring the mean planar diameter of at least 5 neighboring grains without stereological corrections. Since samples deformed at low strain (e.g., CMHT8, CMHT4, CMHT5, CMAS1) did not develop recrystallized grains that are clearly discernable, we only considered samples CMHT3 and CMHT1 with the highest shear strain. The mean recrystallized grain size in Carrara marble close to the inclusion tip is $12 \pm 3 \text{ }\mu\text{m}$ and $10.4 \pm 1.9 \text{ }\mu\text{m}$ for the two samples, respectively. For reference, the mean recrystallized grain size of the high strain inclusion-free sample CMHT2 is $23.3 \pm 4.3 \text{ }\mu\text{m}$. The presence of numerous dislocation walls observed in TEM (Fig. 10b) and the low-angle misorientation peak in the misorientation histogram (Fig. 8b) suggest that subgrain rotation recrystallization is dominant.

For stress estimation we applied the piezometer calibrated by Barnhoorn et al. (2004) in torsion experiments on Carrara marble at temperatures between 500°C and 700°C, involving both subgrain rotation and grain boundary migration recrystallization. In addition, we compared the results to the subgrain rotation recrystallization piezometer established by

Rutter (1995) using triaxial compression and extension tests on Carrara marble at $T = 500^{\circ}\text{C}$ - 1000°C . The piezometer is given by $\sigma = C \cdot d^m$, where σ is axial stress in MPa, d is the recrystallized grain size in μm . The constants are $\log(C) = 2.73$, $m = -0.82$ and $\log(C) = 2.91$, $m = -0.88$ calibrated by Barnhoorn et al. (2004) and Rutter (1995), respectively. The resulting stresses at the inclusion tip, converted to shear stress (Eqn. 7), are 40.4 MPa (sample CMHT3) – 45.6 MPa (sample CMHT1) using the piezometer of Barnhoorn et al. (2004) and 52.5-60.6 MPa applying the piezometer proposed by Rutter (1995). Consequently, stresses in the process zone are a factor of 2.0-2.3 and 2.6-3.0 times higher than the measured bulk shear stress of 19.9 MPa, respectively. For comparison, the estimated shear stress from both piezometers in the intact sample CMHT2 is 23.7 – 29.4 MPa, respectively, which is 12-39% higher than the measured shear strength of 21.2 MPa. Application of the grain boundary migration piezometer for calcite determined by Rutter (1995) with $\log(C) = 3.43$, $m = -0.89$ yields very high stress values of 170-193 MPa at inclusion tips and 95 MPa in the inclusion-free sample, the latter being more than four times higher than the measured values.

As an upper bound stress estimate, we determined also the elastic stress distributions around inclusions for the triaxial and torsion geometry. For the calculations, we used an elastic 3D finite element (FE) modeling simulation (SolidWorks 2013), assuming a friction coefficient of 0.6 between matrix and inclusion and using estimated Young's moduli of 10 GPa and Poisson's ratio of 0.22 for the tested carbonates. Simulation of the experimental stress conditions yields von Mises equivalent stresses at the tip of a weak inclusion, which are roughly 3 times higher than in the matrix, but rapidly decay within a few mm distance. In our experiments, however, the plastic yield strength of Carrara marble certainly limits the spatial extend and the magnitude of a local stress concentration.

For comparison, the stress increases around weak or strong spherical and ellipsoidal inclusions is about a factor of 2.3, inferred from experimental compression of inclusion-bearing analogue materials (PMMA) and analytical calculations (Mandal et al., 2004; Misra and Mandal, 2007). Based on finite element modeling, stress enhancement around strong inclusion in a creeping matrix was estimated to range from 1.1-2.3 (Kenkmann and Dresen, 1998) to 3-8 (Dimanov et al., 2011), depending on the assumed rheology, viscosity contrast, shape of inclusions, and the degree of coupling.

5.3 Strain (-rate) distribution and localization weakening

The zone in front of the weak inclusions shows macroscopic strain localization, defined as the ratio of the maximum strain in the center of the zone to and the strain in the adjacent matrix outside this ‘process’ zone. This ratio gradually decreases with increasing distance (Fig. 5) from the inclusion tip. In accordance with this observation, the intensity of microstructural changes also decreases with increasing distance from the tip (Figs. 6a, 9a). Sample CMHT8, deformed to a bulk shear strain of ≈ 0.05 and a maximum local strain at the inclusion tip of ≈ 0.5 (Tab. 1), shows almost no recrystallized grains. In contrast, samples CMHT4 and CMHT3, with maximum local strains at the inclusion tip of ≈ 0.9 and 2.8, respectively, show recrystallized grains in the process zone. This is in agreement with the observations of Pieri et al. (2001a) and Barnhoorn et al. (2004). These authors report a minimum (local) shear strain of about 1 that is required to initiate recrystallization in calcite at somewhat lower temperatures (500°-770°C). The authors observed the onset of strain weakening for Carrara marble at a shear strain of about 1, associated with an increasing fraction of recrystallized grains and an increase of the CPO strength. In our experiments the maximum stress in constant strain rate tests and the onset of weakening in creep test occurs at a bulk shear strain of about 0.2 (Fig. 3). This difference is likely related to the enhanced local strain in the

process zone, which is up to 10 times higher than the bulk strain (Tab. 1) and to the higher temperature of our experiments (900°C). For example, Schmid et al. (1987) and Pieri et al. (2001b) observed the onset of weakening at strains below $\gamma = 0.5$ on solid Carrara marble deformed at 900 and 927°C, respectively.

After yielding, at first strain hardening occurs and a minimum bulk shear strain of about 0.1 is required to initiate localization, corresponding to local shear strain of about 1 in the weak inclusion (Figs. 3, 4a). Localization is associated with recrystallization and decreasing bulk stresses in constant strain rate tests and with increasing strain rates in constant stress tests (Fig. 3). The increase of the bulk shear strain rate of sample CMHT6 with strain is approximately 3-fold, deformed at constant torque. This strain rate increase agrees with the strain ratio of about 3 observed ahead of the inclusions in sample CMHT8 and deformed at constant strain rate (Fig. 5). The similar strain rate enhancement in different tests may be related to geometry and interaction of the inclusions. The samples contained 2 inclusions of ≈ 11.8 mm arc length, resulting in approximately similar arc distances between inclusions. For comparison, sample CMHT1, containing only one weak inclusion and twisted at constant bulk strain rate, showed a stress decrease from peak stress of 19.6 MPa at $\gamma \approx 0.15$ to 17.9 MPa at $\gamma \approx 1$. Assuming a stress exponent of 7.6, this stress change converts to a roughly two-fold increase in bulk strain rate at constant stress conditions. This suggests that in these experiments the amount of localization weakening for weak inclusions may depend mainly on the inter-inclusion distance and not on the boundary conditions (e.g., constant stress versus constant strain rate).

5.4 Aggregate strength

The measured bulk strength of the NC bearing sample CMTH5 is similar to that of pure Carrara marble (Fig. 3a). For torsion tests, the torque must be the same in all layers along the sample axis. Therefore, the measured torque is limited by the weakest layer, i.e. that of pure marble for sample CMHT5.

Considering weak inclusions, the strength of the aggregates decreases by about 4-15% in triaxial testing and torsion. Here, samples containing two inclusions exhibit the lowest bulk strength (Figs. 2a, 3a). In the following we investigate if the peak strength reduction corresponds just to the volume fraction of weak phase, or if it is caused by the formation of the process zone and associated weakening. We first estimate the aggregate strength bounds using a simple continuum mechanics approach and secondly construct a deformation mechanism map.

For calculation of the upper and lower stress bounds of a two phase aggregate based on their volume fractions, we used the isostrain-rate (or Voigt) and isostress (or Reuss) bounds (e.g., Ji et al., 2001), respectively. At fixed temperature, the upper (isostrain-rate, i.e., $\dot{\gamma}_{bulk} = \dot{\gamma}_1 = \dot{\gamma}_2$) stress bound of the bulk aggregate consisting of the phases 1 and 2 is given by,

$$\tau_{bulk} = f\tau_1 + (1-f)\tau_2 = f\left(\frac{\dot{\gamma}_{bulk}}{B_1}\right)^{\frac{1}{n_1}} + (1-f)\left(\frac{\dot{\gamma}_{bulk}}{B_2}\right)^{\frac{1}{n_2}} \quad (8),$$

where f is the volume fraction of phase 1, n_1 and n_2 is the stress exponent of phase 1 and 2 respectively, and assuming power law creep behavior of phase i , i.e. $\dot{\gamma}_i = B_i\tau_i^{n_i}$. The lower (isostress, i.e., $\tau_{bulk} = \tau_1 = \tau_2$) stress bound can be obtained for the case of a constant deformation experiment by iteratively solving the equation

$$\left[\frac{\dot{\gamma}_{bulk} + (f-1)B_2\tau_{bulk}^{n_2}}{fB_1} \right]^{\frac{1}{n_1}} - \tau_{bulk} = 0 \quad (9).$$

Here, we used power law constants for phase 1 (Carrara marble) of $n_1 = 7.6$, $B_1 = 10^{-13.99}$ (MPa⁻ⁿs⁻¹) and for phase 2 (Solnhofen limestone) $n_2 = 1.4$, $B_2 = 10^{-4.15}$. These values are determined from Fig. 2c. For comparison, Schmid et al. (1977) and Schmid et al. (1980) obtained the constants $n_1 = 7.6$, $B_1 = 10^{-13.51}$ for high temperature of Carrara marble and $n_2 = 1.7$, $B_2 = 10^{-3.10}$ for Solnhofen limestone, assuming a grain size exponent of 3, which are in close agreement.

The resulting bounds are shown in Fig. 12, revealing that the strength of the weak inclusion bearing aggregates is close to the upper strength bound for triaxial compression and close to lower strength bound for the twisted samples. The difference may be attributed to the different loading geometries, i.e., general vs. simple shear, respectively.

It should be noted that calculation of the stress bounds assumes a homogeneous stress distribution within the two phases. However, for hollow cylinder samples deformed in torsion, the stress increases non-linearly from the inner towards the outer radius, depending on the stress exponent (Paterson and Olgaard, 2000). For example, for Carrara marble with $n_1 = 7.6$ the shear stress at the inner cylindrical surface (radius = 3.05 mm) is $\approx 88\%$ of the maximum stress at the outer surface and for Solnhofen limestone with $n_2 = 1.4$ it is $\approx 53\%$. To account for this effect, we used as first approximation the dimensions of an inclusion-bearing hollow cylinder with a larger inner radius (6 mm) equal to the inner radius of the SH inclusion (recalculated from the arc-segment shaped inclusions to a circular ring fraction with equivalent area). This ensures relatively homogeneous stress conditions ($> 85\%$ of the maximum stress at the outer surface for Solnhofen limestone and $> 97\%$ for the Carrara marble matrix). The corresponding fractions f of Carrara marble are 0.981 and 0.963 for one and two inclusions, respectively (Fig. 12).

In contrast, using the inner radius of 3.05 mm of the complete hollow cylinder the corresponding volume fractions of the strong phase (CM) are increased by $\approx 1\text{-}2\%$. Consequently, the measured maximum stresses plot about 10% below the lower strength (Reuss) bound. Alternatively, the bounds may be also calculated assuming constant twist rate (instead of constant strain rate) and constant torque (instead of constant stress). Using this approach, the measured torque values are 4% (1 inclusion) to 8% (2 inclusions) lower than the Reuss (lower torque) bound. In view of the uncertainty associated with the used flow law parameters and inhomogeneous stress distribution, we conclude that in the case of torsion deformation, the measured aggregate strength is close or even below the lower strength, uniform stress or Reuss bound. This indicates that (1) the formation of a shear zone in simple shear poses an upper limit to deviatoric stresses in the neighboring matrix and (2) microstructural changes in the localized shear band may potentially reduce the aggregate strength below the isostress bound.

To validate if early grain size reduction in the process zone is at least partly responsible for the relatively low peak strength of the aggregates with weak inclusions, we determined a deformation mechanism map for calcite rocks that accounts for grain size independent dislocation creep and grain size dependent grain boundary diffusion creep (Fig. 13). For the construction of the map at the experimental temperature of 900°C , the same flow law parameters as for the determination of the stress bounds were used, recalculated for Solnhofen limestone assuming a grain size of $6\text{ }\mu\text{m}$ and a grain size exponent of $m = 3$ (cf., Eqn. (2)).

At the experimental shear strain rate of $\approx 1.9 \times 10^{-4} \text{ s}^{-1}$, the shear strength of Carrara marble with a grain size of $\approx 220\text{ }\mu\text{m}$ is about 22 MPa (solid circle in Fig. 13), consistent with the measured flow strength of the samples deformed to lower strain (CMAS4, CMAS5). Sample CMHT2 without inclusions has a recrystallized grain size of $\approx 23.3 \pm 4.3\text{ }\mu\text{m}$ and a strength of

≈ 21.2 MPa (crossed circle), plotting close to the field boundary. The boundary is well predicted by the paleowattmeter (Austin and Evans, 2009; Austin and Evans, 2007). In the process zone of SH-bearing samples CMHT3 and CMHT2 the recrystallized grain size is 12 ± 3 and 10.4 ± 1.9 μm , respectively, with bulk flow strength of ≈ 20 MPa (open circles). This suggests that enhanced creep rates in the process zone are accommodated by grain boundary diffusion (Fig. 13). However, a stress concentration by factor of about 2 in the process zone may shift the deformation back to the dislocation creep regime, consistent with the paleopiezometer for Carrara marble measured by Barnhoorn et al. (2004). Therefore, the bulk strength of the marble with weak limestone inclusions may be only partially, if at all, influenced by a local switch from dislocation to diffusion creep within the localized process zone. We conclude that the deformation mechanisms operating in a localized process zone are close to the field boundary. In natural carbonate-rich shear zones, localization may be further enhanced by second phase particles and stress or temperature-induced grain size reduction (Ebert et al., 2007a; Ebert et al., 2007b; Herwegh et al., 2008).

6. Conclusions

We investigated the initiation and propagation of localized deformation caused by mm-scale heterogeneities (inclusion) in deforming marble by means of high temperature torsion and compression experiments. The presence of weak inclusions promotes strain localization and associated weakening by stress concentration at the tip of the inclusions. In the process zone of localized deformation zones in front of the inclusions the microstructure is strongly modified by enhanced twinning, dislocation activity and dynamic recrystallization. The strain enhancement in the localized zone appears to be controlled by the viscosity contrast between inclusion and matrix material and inclusion interaction. With increasing strain, a strong, dislocation creep-induced texture evolves, typical for high strain, recrystallization-accommodated creep of marble. In contrast, strong inclusions do not initiate strain

localization at experimental conditions. The evolution of the process zone ahead of the inclusions is very similar in torsion and triaxial compression tests, where we also found evidence for local brittle fracturing at the inclusion tip. The mechanical bulk strength of the twisted aggregates containing weak inclusions is close to the isostress bound, possibly affected by diffusion creep processes occurring locally in front of the inclusions.

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Figures

Figure 1: Schematic drawing of starting samples (a, b) and photographs of deformed samples CMHT4 (c) and CMAS1 (d). For torsion tests (a, c) hollow cylinders with one or two inclusions were used and in triaxial compression a solid cylinder with a single inclusion (b, d). The borehole of the hollow cylinder was filled with a solid gold cylinder. CM is Carrara marble (matrix), SH is Solnhofen limestone (inclusion), and Al_2O_3 denotes alumina spacer.

Figure 2: Strength of starting materials deformed at 900°C temperature. a) Stress strain curves of solid Carrara marble (CM) and Solnhofen limestone (SH), deformed in triaxial compression at a constant strain rate of $7.2 \times 10^{-5} \text{ s}^{-1}$. Sample CMAS1 (CM with SH-inclusion) was deformed at $7.3 \times 10^{-5} \text{ s}^{-1}$. b) Shear stress strain curves of CM and SH, deformed in torsion at a maximum shear strain rate of $(1.9 \pm 0.1) \times 10^{-4} \text{ s}^{-1}$. c) Log-log plot of equivalent maximum stress strain-rate data obtained by torsion and triaxial testing. Horizontal and vertical dotted lines mark the approximate experimental conditions used for inclusion-bearing samples. NC indicates strength of wet flint (Mainprice and Paterson, 2005).

Figure 3: Mechanical shear stress-strain curves of torsion experiments performed at a) constant twist rate and b) constant torque (sample CMHT6). All samples contained weak

inclusions, except sample CMHT5 that contained a strong inclusion and sample CMTH2 without inclusions. Temperature was 900°C and confining pressure was 400 MPa.

Figure 4: Change of strain distribution with the imposed bulk strain for samples with weak inclusion. (a) Measured inclusion strain, (b) measured and calculated matrix strain adjacent to the inclusions, (c) ratio of inclusion to measured and calculated matrix strain. Dotted lines in a) and b) represent equal strain.

Figure 5: a) Scanned jacket surface with markers (scratches) of deformed sample CMHT8. The localization (stippled) region extends few mm in front of the SH-inclusion tips. The weak Solnhofen limestone inclusion is marked by broken lines. b) Average ratio of maximum local strain in the localized area to the residual matrix strain versus distance in front of the inclusion tip. The shown error bar represents the average uncertainty related to repeated measurements of the inclination angle.

Figure 6: Thin section micrographs (crossed nicols) of deformed Carrara marble samples in front of weak (a-b, e-i) and strong (c, d) inclusions. With increasing final bulk shear strain γ (a-b, e-g) the length of the process zone in front of weak inclusions, determined by grain size reduction, subgrain formation and high twin density, increases in length and width. In front of strong novaculite inclusions, which exhibit vertical cracks, twinning and undulose extinction are less pronounced (c) and mineral reaction induces the formation of wollastonite (d, BSE-image). At a bulk shear strain of ≈ 0.2 , a secondary foliation plane (S_2) oblique to the shear plane (S_1) evolves in front of weak inclusions, marked by the alignment of calcite grain boundaries (e). In the process zone, grains with 2 twin sets are common and lobate grain boundaries indicate partial grain boundary migration (arrows in b, e-f). At higher strain, twin density in front of the inclusions decreases, twin width tends to increase and twin boundaries

are sometimes bent, suggesting active crystal plasticity activity (arrow in f). In front of the weak Solnhofen limestone inclusion in the triaxially deformed sample the calcite grains show undulose extinction and subgrain boundaries (h), and fractures parallel to the tip occur in close vicinity to the inclusion (i). Indicated approximate shear strains are recalculated from the measured maximum bulk strain using Eqn (5). Approximate areas used for EBSD mapping are indicated.

Figure 7: Crystallographic preferred orientation of calcite in the process zone for the samples deformed in torsion (a-e) and axial compression (f), for the poles of basal (0001), prismatic $\{11\cdot20\}$, and rhomb $\{10\cdot14\}/\{01\cdot12\}$ planes. Pole figures in the lower hemisphere of equal-area net where the shear plane is vertical E-W (black line) and the shear direction (SD) is horizontal E-W. In the axial compression experiments, we assume a shear plane oriented at 45° to the vertical direction (f). Multiples of uniform distribution, confidence index >0.2 , orientation distribution function assuming a Gaussian half-width of 10° and a maximum harmonic expansion factor ($L_{\max}$) of 32. Indicated approximate shear strains are recalculated from the measured maximum bulk strain using Eqn (5). The top row shows the CPO of undeformed Carrara marble for comparison.

Figure 8: Histograms showing the misorientation angle distribution between calcite grains in front of the inclusion of sample CMHT8 (a), deformed to low strain and sample CMHT1 (b), deformed to high strain. Correlated versus uncorrelated nearest-neighbor misorientations ($>1^\circ$) are shown by red and blue bars, respectively, and the theoretical random distribution for trigonal minerals is shown in green. At low strain, the misorientation angles show a peak at $75\text{-}80^\circ$ with axis parallel to equivalent $[0\cdot221]$ and $[20\cdot21]$ directions, related to *e*-twinning (see inset). At high strain, the peak is at $5\text{-}10^\circ$ with random orientation in relation to the crystal reference frame, related to the widespread occurrence of subgrains boundaries.

Figure 9: Kernel average misorientation maps of 4 samples twisted to increasing strains (a-d). Spatial resolution is $1\mu\text{m}$. The color code indicates the local intracrystalline misorientation, where the average misorientation of each point in the map is calculated in relation to the neighbor points (up to 5 grains apart) considering only misorientation angles less than 10° . Even at very low strains, lattice distortion of calcite grains up to 4° in front of the inclusions is evident, in particular around the grain boundaries (a, b). With increasing bulk shear strain the misorientation angle increases, affecting a broader region (c). At high strain many grains are recrystallized and misorientations exceed the predefined limit of 10° , indicated by abundant average misorientation angles $>8^\circ$. Note the development of lenses of less deformed grains in between completely recrystallized areas (d). Indicated shear strains are recalculated from the measured maximum bulk strain using Eqn (5).

Figure 10: TEM bright field micrographs of the calcite matrix adjacent to the SH inclusion in sample CMAS1, showing areas with numerous free dislocations (a) and areas with abundant dislocation walls (b). Scale bar is $0.2\mu\text{m}$.

Figure 11: Viscoplastic self-consistent modeling of CPO evolution for simple shear of calcite at $\gamma \approx 1.7$ (a) and $\gamma \approx 4.3$ (c), neglecting recrystallization. Pole figures are shown for the poles of basal (0001), prismatic $\{11-20\}$, and rhomb $\{10-14\}/\{01-12\}$ planes. (b) Evolution of activity of different slip systems with increasing strain. $\langle\text{SD}\rangle$ is slip direction, e.g. $\langle 2021 \rangle$ for f-slip in calcite. (d) Evolution of Von Mises equivalent stress with increasing strain. Note a smooth weakening for $\gamma > 3$, corresponding to the peak activity of the $\{r\}\langle a \rangle$ slip system. Pole figures are plotted for 1000 grains in lower hemisphere, equal-area projections, half-width = 10° . East–west horizontal line marks the shear plane, and SD the shear direction. The

oblique line marks the flow plane and X the flow direction. . Contours are multiples of uniform distribution.

Figure 12: Aggregate strength of weak Solnhofen inclusion-bearing samples versus fraction of the strong (Carrara marble) phase. The equivalent stress is normalized to the creep strength of pure Carrara marble. USB and LSB denote (Voigt) upper strength bound and lower (Reuss) strength bound, respectively.

Figure 13: Deformation mechanism map for high (900°C) temperature creep of calcite aggregates. Dotted lines are contours of constant strain rate. The grain size of the starting (Carrara marble) material and mean flow stress of low strain samples is indicated by the solid circle. Measured bulk stress and recrystallized grain size of deformed inclusion-free marble is given by the crossed circle and of weak inclusion-bearing samples within the process zone by open circles. The field boundary is calculated assuming equal shear strain rate of dislocation and grain boundary diffusion creep. The piezometer was calibrated by Barnhoorn et al. (2004) and the wattmeter by Austin and Evans (2007, 2009).

a) b)

c)

d)

a)

b)

CMHT5
 $\gamma=0.065$

CMHT3
 $\gamma=0.195$

CMAS-1
 $\varepsilon=0.016$

(0001)

{11 $\bar{2}$ 0}

{10 $\bar{1}$ 4}

{01 $\bar{1}$ 2}

Starting
material
J=1.15

Max:
1.57

Max:
1.49

Max:
1.36

Max:
1.36

a

Max:
3.03

Max:
1.97

Max:
1.88

Max:
1.76

b

Max:
3.09

Max:
2.00

Max:
1.96

Max:
1.90

c

Max:
4.44

Max:
1.89

Max:
2.3

Max:
2.17

d

Max:
6.7

Max:
4.73

Max:
3.64

Max:
4.41

e

Max:
11.63

Max:
5.29

Max:
3.23

Max:
3.54

f

Max:
2.45

Max:
2.02

Max:
1.63

Max:
1.97

g

CMHT-09
 $\gamma=0.014$
J=2.01

CMHT-08
 $\gamma=0.045$
J=2.19

CMHT-04
 $\gamma=0.062$
J=2.43

CMHT-03
 $\gamma=0.195$
J=6.03

CMHT-01
 $\gamma=1.76$
J=7.97

CMAS-01
 $\varepsilon=0.015$
J=2.02