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Discussions

Interactive comment on "Uniaxial compression of calcite single crystals at room temperature: insights into twinning activation and development" *by* Camille Parlangeau et al.

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First of all, we would like to thank you for the time you have taken to read and write down your comments about this article.

First comment: "I find the number of samples used, namely 3, really too low to make conclusions with confidence about the effect of orientation and the effect of grain size. One would at least want to see duplication tests. But prefarably more" Actually, it was not only 3 samples that have been tested in this study. We have chosen to present only the results of 3 representative ones, because we find it clearer for the main message. But we did test 4 synthetic "perfect samples" (purchased from SurfaceNet supplier).

The respective loading curves provided in the paper (and now in the supplementary material) show that the results are qualitatively (and quantitatively) reproducible with respect to the different stages characteristic of the elastic and plastic phases. Two samples have dimensions of 3x3x6 mm3 and 2 other samples of 4x4x8 mm3. For both sizes there are two different lattice orientations, which are chosen in order to favour twinning. In addition to these samples, we also used several specimens cut from optically clear Iceland Spaths. They were used to establish the appropriate deformation protocol to be applied to the synthetic strain-free samples, in terms of sample preparation, loading rates, and conditions for in the situ optical/SEM monitoring. This preparation phase allowed for instance to select the most suitable CCD cameras and to establish the SEM imaging conditions. Unfortunately, because of the natural origin of the samples pre-existing cleavage cracks, twin lamellae and micro-fluid inclusions preclude the extraction of perfectly similar samples with respect to size, crystal orientation and pre-existing defects which can cause local stress concentration and the possibility to precisely infer the CRSS value (Critical Resolved Shear Stress) of interest. Hence, Iceland Spaths are not appropriate for this study, but only for training. However, interestingly the corresponding results showed qualitatively the same patterns for the loading curves as for the "perfect samples", which further demonstrates the reproducibility of the results. A supplementary data has now been added to this article to show the results of some of these preliminary tests on Iceland Spaths, as well as the result for the fourth "perfect sample". Concerning the relationship between the grain size and the CRSS value, as said in the discussion part and showed in the Figure 5 of the article, no reliable conclusion can be drawn, and we discuss the reasons why. We did not report any link between the lattice orientation and the CRSS value.

Changes have been made in the text between line 30 page 2 until line 5 page 3, line 11 to 12 page 3 and line 4-6 page 5 + information has been added about the camera and software used line 18 page 3.

Second comment: "The CRSS in the current paper are based on taking the overall

stress value at the moment of the first twin, but perhaps there are local stress concentrations that play a role. The different strains at which the first twin develops (0.3-0.7%) and the fact that microcracks develop already at low strains might be an indication for such stress concentrations. The authors do bring up this point in the discussion (p.7), but without a clear inference." We have chosen to work on synthetic material precisely in order to guarantee the quality of the single-crystal: limited dislocation density, fluid inclusion-, cleavage- and twin-free, which would undoubtedly influence the calculated CRSS through inherited work hardened state and local potential stress concentrations. We disagree with what the reviewer wrote about the appearance of the first twins at different strains. The total strains considered by the referee (0.3 – 0.7%) are only apparent values, which are not necessarily representative of the plastic yielding, because there is an important emplacement phase (stage I) during which the full contact between pistons and samples is progressively established. The latter is not the same for each specimen, because it depends on the exact sample geometry (parallelism of loaded faces).Moreover, eventual stress concentrations would expectedly be more pronounced during this emplacement stage, however the micro-cracks did not appear at this stage. Micro-cracks essentially developed latter on, during the pseudo-elastic and plastic phases. This may indicate that micro-cracking is a complementary and necessary deformation mechanism, allowing for accommodation of local incompatibilities of strain, for instance at sample-piston interfaces when contact have been made (stage II and III). The latter are actually expected, because twinning is not isotropic and the pistons have not enough degrees of freedom to fit the inhomogeneous sample strain if solely resulting from twinning. Conversely, it is in practice impossible to completely prevent local stress concentration near the crystals ends, due to the piston-sample frictional contacts. The early fractures are likely due to the friction between the sample and the pistons and the fact that the loading geometry is axial, but twinning related strain is not axi-symmetric. The calcite crystal lattice stereo-diagram (Figure 1 in the main text) show that the deformation systems are not symmetrical along the stress axes. Therefore, when only twinning deformation mechanism is activated the deforma-

tion cannot be axisymmetric. Twinning activation leads to the formation of shear bands composed of several twin lamellae which result in a shortening component along the stress axis, but also in extensional transverse components. The latter are perpendicular to the stress orientation, but are not equivalent (not axisymmetric). However, the pistons remain aligned and cannot accommodate a later motion during the shearing of the crystal imposed by twinning. Similarly to uniaxial compression of a sample activating crystal slip onto a single slip system, the sample must either rotate, or the pistons must follow the lateral motion. If impossible, there occurs frictions and stress concentrations at the sample – piston interface, with possibly local micro-fracturing. The fact we use samples with a length of twice the width (3 x 3 x 6 and 4 x 4 x 8 mm3) allows however to keep the central part of the crystal free of these edge effects, so one can safely consider that our results and inference are reliable.

Information have been added in the main text between line 10 and 17 of the page 6.

Third comment: "One other question I have is: are you sure that no other mechanism than twinning plays a role in straining? Does the volume of twins fit the amount of axial strain?" We do not have any monitoring of what happens in the inner part of the crystal but only on one surface. Fortunately however, it is possible to have a reliable guess of what is going on within the crystal by transparency (pure calcite crystals are transparent). There is no optically observable deformation mechanism other than fracturing and twinning, but since the tests are performed at room temperature and at relatively high strain rate we do not expect any noticeable crystal slip plasticity. Once fracturing impacts a large portion of the crystal it is difficult to quantify the partitioning of the shortening along the stress axis due respectively to fracturing and twinning. Therefore, we measured the shortening after the EBSD analysis performed between the two consecutive loading phases. For the example of the crystal of 3 x 3 x 6 mm3 compressed along [21 ÌĚ1 ÌĚ0], the whole shortening after the first loading was of about 151 μm. High resolution SEM micrographs were used to measure twin lamellae thicknesses, which allows to determine that twin is responsible of the axial deformation for 116 μm.

Optically we can see by transparency a few twin lamellae which are not crossing the whole crystal to the surface and this can explain for part of the difference we calculate with the prescribed shortening. The optical error of measurement of the individual twin lamellae thicknesses on pictures may also contribute to the difference.

Information has been added line 17 to 22 of the page 4.

Fourth comment: “the possible role of (the lack of) confining pressure; relevant when extrapolating to nature.” The lack of confining pressure probably leads to easier micro-fracturing (with some contribution to the whole deformation), but it does not affect the differential stress required to activate twinning. But the main issue for extrapolation of the data collected from single crystals to natural aggregates resides in having free surfaces instead of neighbour grains, which presence necessarily modifies the local stress state. The problem consists in extrapolating the uniaxial state to a grain within a polycrystal (see next question too). The twin lamellae probably thicken up to 90 μm wide also due to the free surfaces. So we acknowledge that the results from single-crystal experiment cannot be extrapolated directly and easily to natural aggregates.

Information has been added line 20 to 22 of the page 7.

Fifth comment: “the effect of multiple grain boundaries in an aggregate, i.o.w. how to relate single crystal observations to polycrystals.” The choice of single crystals instead of aggregates is motivated by the aim of obtaining the purely intrinsic, material dependent CRSS value of the twin systems. For that, we need the actual stress state on the considered crystal, which can only be achieved with single crystals. The referee is perfectly aware of the problem, since he has himself extensively worked with the same methodology on crystal slip plasticity of calcite. The application of mechanical data gathered on single crystals to a polycrystal is another topic. It is known for instance that grain boundaries act as barriers for the propagation of twins (Barber and Wenk, 1979) and that some additional deformation mechanisms (pressure-solution, ...) are required to maintain geometric and kinematic consistency between grain within the

aggregates and/or to release elastic strain due to these local geometrical incompatibilities. For that purpose, analytical mean field, or computational full field homogenization models must be applied but this is much beyond the scope of the present work.

Information has been added line 26 to 33 of the page 8.

Sixth comment: “In terms of presentation: it would be useful to clearly indicate the stages I, II, IIIa en IIIb in Figures 2, 3 and 4.”

Done as suggested.

Please also note the supplement to this comment:
https://www.solid-earth-discuss.net/se-2018-80/se-2018-80-AC1-supplement.zip

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