

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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Author's answers to the interactive comment of Anonymous (referee) Thank you for the time you spent on reading and writing comments about this article.

First comment: “A question I still have is how can these results be extrapolated to natural conditions in which grain size distribution is obviously much more complex? Starting with the simplest situation seems to be an interesting idea but they could maybe discuss in more details the implications of this (over?)simplification.” The single-crystal approach does not intend to be directly applied to natural aggregates. It follows a classical methodology in determining the intrinsic mechanical properties (elastic moduli,

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thermal expansion, flow strength, resolved shear stresses, anisotropy. . .) of minerals and crystalline materials. Such fundamental data are the basic input for homogenization models (such for example, mean field elasto-visco-plastic self-consistent schemes or full field fast Fourier transform ones), which intend to calculate the average mechanical properties of aggregates and rocks. The aggregate behaviour is the final target, and it largely depends on phase proportions and microstructures, such as spatial distribution of phases and porosity, crystallographic texture. . . , which complexity has to be specifically addressed in the homogenization type of model. As you highlighted in your review it has to take into account the grain size (and its distribution), texture, porosity, etc. . . which impact the local interactions and dictate each grain boundary conditions (deformation compatibility between grains and local stress state resulting from these grain interactions). So, we do not pretend there is simple direct extrapolation of measurements performed on single-crystal to aggregates. However, our aim is specifically to provide the critical resolved shear stress (CRSS) value needed to activate the twinning phenomenon. The result can be considered as the basic input for further homogenization modelling. Most importantly, it is an intrinsic crystal related parameter, which is not depending on microstructure of the aggregates. From the point of view of the CRSS value, the differential stress is the prime variable, whilst confining pressure (and possibly temperature) is a second order one. The role of the grain size, which is still being debated, enters in the category of microstructure parameters, important to be considered for aggregates, but not for single crystals.

Information have been added in the main text line 9-12 page 9 + the review from the previous reviewer line 26-27 page 9, line 31 page 9 to line 2 page 10.

Second comment: “Why not compare their own different experiments first? Side question: their results seem to compare to previous results obtained by Turner et al (1954) on unconfined samples: Do the authors think that confinement might play a role on the development of twinning, contrary to what is claimed in the introduction? One idea might be that twinning is actually slightly associated with microcracking which is

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highly dependent on confining pressure.” According to Covey-Crump et al., (2017) the CRSS value is supposed to follow the Hall-Petch equation, with power law decreasing of CRSS for increasing grain size. But, as shown in the figure 5 of the main text, the crystal sizes of our experiments correspond to the asymptotic part of the curves, where experimental uncertainties may obscure the possibly small differences of CRSS with respect to the small differences in crystal sizes. Considering only our results did not show any clear trend in CRSS evolution, with a mean value of 0.90 MPa and a standard deviation of 0.35 MPa, so that we do not extend further the discussion in comparing only our data. Comparing our data with those from other similar studies is not easy as well: - Turner et al. (1954) used natural Iceland spaths. We show in the complementary data that we also used Island spaths for establishing the experimental protocol. The results are not directly comparable with those obtained from the synthetic (optical quality) crystals. As said in the main text, the natural single-crystals of calcite are already slightly strained (some twins are visible), or cleaved, and most contain micro-fluid inclusions. All these imperfections induce strain hardening, and hence increased CRSS values for twinning activation. (see fig. 2, 3 and 4 as in the supplementary data added after the first review). This is why our, synthetic samples provide a CRSS value slightly lower than that of Turner et al. (1954) (as expected). Using natural samples implies undefined initial state, which questions the validity of the retrieved CRSS values, when applying inversion techniques in order to determine paleo differential stresses. - De Bresser and Spiers (1997) annealed their Iceland spath specimens in order to restore the previous cumulated strain. Unfortunately, as explained in the main text, this procedure leads to the formation of sub-grain boundaries, that affect the propagation and spreading of twins and cause non-uniform stress distribution at the grain scale, which is potentially biasing the results. Also for both of these previous studies, the CRSS determination was based on post mortem observations, which precluded accounting for the loading history and probably the detection of the earliest twinning events. The lack of confinement is certainly responsible to some extent of micro-cracking. Though, micro-cracking is probably unavoidable in compression loading geometry, with laterally

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bound pistons. Indeed, twinning is anisotropic and strain incompatibilities must arise at the sample pistons interfaces. The latter would necessarily result in crystal rotation and frictions in the vicinity of the interface. Micro-cracking could actually be a necessary local accommodation mechanism. Conversely, confining pressure is unlikely to be directly involved in twinning activation, which is mostly dependent on differential stress. However, we must admit that confining the samples with a fluid, or leaving an unconfined free surface during uniaxial compression could affect twin thickening, with respect to confinement by crystalline grains. In the latter case, within a polycrystal, the crystallographic orientation of the neighbours will greatly condition the amount of crystal shear that can be transmitted across the grain boundary. The crystallographic constraints, in terms of shear strain compatibility, along the grain boundary would favour the occurrence of numerous distributed thin twins. Conversely, the presence of fluid confined free surface could favour the localized development of thick twins.

Information have been added in the main text line 26 page 7 to line 2 page 8 and 14-29 page 8.

Third comment: “The results obtained in this study would allow the authors to draw the evolution of total twin thickness as a function of axial strain. Did they have a look at this correlation? It may help them decipher whether twinning is associated with microcracking or not (and even maybe try to quantify strain due to each of these two micromechanisms if they are associated).” Unfortunately, it is not possible to simultaneously monitor both the scale of the sample and the scale of individual twin lamellae. The whole sample surface is observed by optical microscopy in order to detect where and how many twins occur. But, optical resolution does not allow to follow very precisely the thickening of the latter. In order to be able to follow the thickening of each twin lamellae we would have been obliged to dismount the specimens from the loading stage for closer observations in the SEM. However, such a step-by-step approach would have induced cyclic loading, with a priori unknown effects on twin activity, but with serious risks of enhancing micro-fracturing. Besides, each emplacement on the

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loading stage could also modify the boundary conditions at piston-sample interfaces.

Fourth comment: "Interestingly, the authors mention that the duration of stress application has a great impact on twin lamellae thickness (line 30, page 6). To me, this may imply that making a creep experiment would be of interest, also since natural conditions may be closer to constant stress conditions rather than constant strain rate deformation." We are actually not aware of any creep experiment where the macroscopic flow is ensured solely by twinning. The experiment is certainly interesting to do. But, it is clearly not the purpose of this work. On the other hand, it is not clear if constant stress or strain rate is more representative of natural conditions. It might actually depend on the geodynamical context.

Fifth comment: "Finally, stress-strain curves show multiple small stress drops. Are these stress drops associated with microcracking or twin nucleation or not?." We are absolutely clear about the fact that the stress drops are associated with twinning. Unfortunately, it is very difficult to draw a figure showing the entire loading curve, but that is also able to clearly highlight the fact that the stress drops relate to twinning. However, it is clear that the micro-cracking events are not numerous enough to account for all the serrations (stress drops). Besides, the first loading curve corresponds to a crack-free sample and still shows the dense serrations.

Please also note the supplement to this comment:

<https://www.solid-earth-discuss.net/se-2018-80/se-2018-80-AC2-supplement.zip>

Interactive comment on Solid Earth Discuss., <https://doi.org/10.5194/se-2018-80>, 2018.