Review of "Frictional properties and microstructural evolution of dry and wet calcite-dolomite gouges" by M. Demurtas et al.
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General comments

The manuscript (ms) by Demurtas et al. reports on an experimental investigation of the frictional behavior of simulated calcite-dolomite faults. Experiments are carried out using the well-known SHIVA rotary shear apparatus, covering a wide range of slip rates ($3 \cdot 10^{-5}$ to 1 m/s) under room humidity and water-dampened conditions (normal stresses 17 to 26 MPa). Much attention is given to post-mortem microstructural analysis, using SEM, EDX, XRD as well as EBSD. The results show marked differences in the frictional strength change with increasing displacement between room humidity and water-dampened samples, specifically in (the velocity dependence of) the onset of dynamic weakening. Based on microstructural evidence, the authors interpret that gouge fluidization played a role in water-dampened experiments, regardless of shearing rate, and that fault rock foliation may develop at co-seismic slip rates.

The authors are well-known for a long list of strong publications in experimental and microstructural fault friction research, and this manuscript seems to be another addition to that. The experiments are technically challenging, but appear to be carried out well using a proven method and a logical, systematic approach. I especially appreciate the emphasis on post-mortem microstructural analyses using a range of different techniques. That said, I do have a few general and technical comments, in particular on the interpretation of EBSD data and inferences on grain size, which I think should be addressed or at least satisfactorily rebutted. I am confident that this ms will come to represent a strong contribution to Solid Earth.

Bart Verberne
20 Oct 2020

Specific comments

Lines 27-33: The work summarized here represents shear experiments on gouges as well as compression and/ or torsion tests on dense polycrystals, and even single crystals, deformed under an enormous range of experimental conditions. Elsewhere in the ms (such as in the next paragraph) particular attention is given to a comparison with torsion tests on dense polycrystals. I understand that data on dolomite gouges are sparse, however, I'm not sure if such this comparison is warranted given the major differences in experimental conditions and physical processes at play (in particular the role of porosity and frictional heating). At the very least, a very clear notion of this should be made.
I recall Boneh et al. (2013) published data on dolomite friction, have the authors considered this?

Line 37: Calcite can deform plastically at room temperature, by twinning.

Line 39-40: "For example…..strain localization". Why? How? Some more argumentation is needed here.

Lines 63-65: This interpretation for a CPO formation mechanism is similar to that by Toy et al. 2015, who deserve credit here.

Lines 105-106: Was there pore fluid pressure build-up in the experiment? If not, why not? Some discussion on this, perhaps later on in the ms, where this is explicitly mentioned would be highly topical, I think.

Line 174: Its always a challenge to report microstructural observations in an objective way. While I recognize the importance of Figure 7, I strongly suggest to begin with reporting the observations instead of this highly interpretative sketch. The summarizing sketch should be presented after the key observations have been convincingly demonstrated (i.e., after the present Figs. 8-11).

Line 197: Why not refer to the observations reported in *this* paper (Fig10)? As it is written now, it seems like the reader is referred to other papers by the same authors.

Lines 206-209: Individual grains within the PSZ (which is, I presume, the ultra-comminuted zone immediately adjacent to the PSS as identified by the authors in Fig. 10) are impossible to distinguish from the present BSE images. Furthermore, I contest that the domain highlighted in Fig 10e, and analyzed using EBSD, represents grains from *within* the PSZ. This domain is surrounded by what seem to be coarse fragmented grains, and therefore it is more likely that these represent grains *adjacent* to the PSZ.
At the very least, we need a very clear definition of what represents the PSZ.
The authors indicate that they have prepared thin sections (line 116). Have they tried to image the samples using a polarizing light microscope? This is a cheap and easy way of getting more evidence for a CPO, incl. from within the PSZ, and at a much larger scale than can be achieved using SEM-EBSD (see Verberne et al. 2013, 2019, Niemeijer 2018, Smith et al., 2015). Also, extremely fine grain sizes (<100 nm) are practically impossible to measure, including using t-EBSD.

Lines 265-275: I am puzzled how the authors can connect the grain size observed in the recovered samples to that relevant during the test. If the temperature at the PSS reached up to hundreds of degrees (here 621degC), post-test static recrystallization must have played a role. In fact, with a few simple assumptions for calcite grain growth (which is well constrained), we showed that a grain size of 300-400 nm will be reached within seconds after a test (see Verberne et al. 2017). The implication is that post-test grain size data must be taken with extreme caution. In fact, the grain size likely was much smaller during shear deformation, in the <100 nm range, which has profound implications for their physical properties.
I doubt that the “significant decrease of the activation energy for creep”, as claimed in line 273, applies to the grains observed here, or that there is any evidence for this. A dramatic change in the physical properties of nanograins is well-known to occur for grains <100 nm in size, in particular for metals. For calcite, there is evidence for a decrease of the decomposition temperature for a grain size below 50 nm (Wang et al. 2014; see also Verberne et al., 2019). However, the present grains are hundreds of nm’s in size, and suggesting that these behave any differently because of their size is a shot in the dark.

Line 287-300: The authors should look at the work of Chen et al. (2017a, b). As I am sure the authors are aware, the presence of water has a buffering effect on temperature, with profound mechanical implications. I think this is highly relevant to the discussion here.

Lines 323-325: Are the authors inferring a low transient permeability during shear deformation? Also, I am curious how this compares with the "dilatancy strengthening" mechanism hypothesized in the 90's (see Marone et al., 1990; Segall and Rice, 1995; Beeler et al., 1996; Samuelson et al., 2009). This may help to broaden the impact of the discussion a bit, I feel. The authors may also consider the role of grain size as well. When grains are extremely tiny, compaction is fast, and transient low permeability or "water-saturated patches" may be readily envisioned.

Line 346: I realize that the authors are probably tired of me pointing this out, but I will continue to express major concerns on claims that MSS's are indicators of co-seismic slip – truncated clasts or not. Although we did never explicitly make a point of this in any of our papers, in my experiments at sub-seismic displacement rates on calcite, I have *also* observed calcite clasts that are truncated by PSZ's (e.g., Fig 8D Verberne et al., Pageoph, or Fig2A Verberne et al, Science). I am not trying to make an of our lives more difficult, but please, reconsider these claims because they are just not consistent with experimental observations.

Lines 366-371: We have also observed this in grains adjacent to the principal slip zone in experiments on calcite gouge at 550°C. Admittedly, the conditions are different, but least the shear rate is closer to what is achieved in here. See Verberne et al. (2017) and Chen et al. (2020).

<u>Technical corrections/ suggestions</u>

The manuscript is generally well written.

Throughout the manuscript I noticed an inconsistent use of the hyphen (e.g., water-dampened and water dampened, slip rates and slip-rates, grain size and grain-size). Please check.

Line 7: I suggest to mention the starting layer thickness of 3 mm somewhere within the abstract as well, to be able to put the quoted slip zone width into perspective.
Line 73: "in to" → "into"
Line 153: "cm" → plural
Line 370: I suggest to write out in full what is meant with the range of shear rates here. Mathematically this suggests $10^{0.8}$ s^{-1}, which surely is not what is intended.

Discussion

The discussion now consists of two, rather long sections. Perhaps the authors can consider to separate out an 'implications' section, from which the reader can readily take away the more general, geological importance of this work. In my view, one of the nicest results points to the potential role of fluidization over a wide range of slip rates, and on foliation development at co-seismic slip rates.

Figure 8, caption.

"Slip zone thickness evolution with slip rate and ambient conditions". I don't quite follow the last part of this sentence. What is meant by, 'and ambient conditions'?

Figures 9-11
I suggest to print as-large-as-reasonably-possible images at high resolution. It may be the quality of the pre-print that affects the figure quality here, but certainly in Fig 9 there is not an optimal use of space in the rectangular area that is available.

Table 1
Line 106 states that the normal stress was 17.5 ± 0.1 MPa. It's a bit strange then, to list 17.4 MPa for each experiment in table 1. Also, I think a typo may have slipped in for experiment s1234. Shouldn't this read 26 MPa?

References cited which are NOT already listed in the ms:

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