

## ***Interactive comment on “Crystallographic preferred orientations of ice deformed in direct-shear experiments at low temperatures” by Chao Qi et al.***

**Chao Qi et al.**

qichao1qc@gmail.com

Received and published: 13 November 2018

Response to Reviewer 2

We thank Reviewer 2 for their thoughtful review. We present our detailed responses (shown in black text) to the Reviewer's comments (shown in blue).

1. During the sample preparation, when the samples are cooled to  $-60^{\circ}\text{C}$  for the welding of the indium jacket, is there any possibility for the thermal/confinement stresses to alter the microstructure as it would relate to the grown-in dislocation density?

Each experimental sample, including the undeformed one published in Qi et al. (2017),

C1

were stored in liquid nitrogen before microstructural characterization in the SEM. No thermal-stress-related microstructure was observed in these samples. Transferring the sample from the  $-30^{\circ}\text{C}$  freezer to the  $-60^{\circ}\text{C}$  alcohol bath for welding is a modest temperature change compared to immersing a sample from the freezer in liquid nitrogen. Thus, transferring the sample from the  $-30^{\circ}\text{C}$  freezer to  $-60^{\circ}\text{C}$  alcohol should not induce any observable alterations to the microstructure.

Moreover, after welding the jacket, each sample was placed into the apparatus and maintained at the temperature and pressure of the deformation experiment for  $>1\text{h}$ , so that if any dislocations were produced during welding, those dislocations would be relaxed before deformation started.

2. From looking at Figure 1, I am perplexed as to how the piston is able to translate laterally while also remaining rigid and in-line with the axis of compression? Could you please explain?

As illustrated in the figure below, as the driving piston moves vertically up, the bottom  $45^{\circ}$ -cut piston moves sideways, because the ice sample is weaker than the pistons and the boundary between the assembly and the driving piston allows lateral movement. This design for shear deformation is widely used in rock deformation studies (e.g., Schmid et al., 1987, JSG; Dell'angelo and Tullis, 1989, Tectonophysics). We have modified Figure 1 to include this information.

3. Could you include the data (via personal communication) related to the flow law of the indium jacket and perhaps also the company/supplier that is used? Such that these experiments could be repeated?

We can share W. Durham's indium data with you. But as the data belong to Durham, we think it is not appropriate to publish it in our paper.

4. Please add a citation for Line 1-2, page 5, regarding using the minimum strain rate in creep tests.

C2

We added a citation (Jacka and Maccagnan, 1984) here as suggested.

5. In Section 3.4, please comment on the skewed distribution of grain sizes. Is this log-normal? As would be expected? Was this distribution used for calculating the mean? How was the anisotropy in the elongated grains accounted for?

For most samples, the distribution is lognormal. A lognormal distribution of grain size was expected, because our previous compression experiments found roughly the same distribution. Moreover log normal distributions of dynamically recrystallized grain size in rocks and metals are very common. The distribution was not used to calculate the mean grain size, but was used to identify the “peak” grain size. Using an equivalent radius to calculate grain size, the influence of anisotropic grain shapes is minimized.

6. Discussion Section. Although I appreciate the detail of this section, it seems to me that it could be more concise, such that the most relevant findings and results are more impactful.

We have shortened the discussion section by about half a page.

7. In Section 4.1, should any consideration be given to the recrystallized grains experiencing primary creep in this scenario?

The terms “primary creep”, “secondary creep” and “tertiary creep” are used to describe the behavior of the bulk of a material. In tertiary (approximately steady-state) creep, dynamic recrystallization and grain growth are balanced. Recrystallized grains with lower dislocation densities than other grains are produced at all times. But researchers do not generally consider these recrystallized grains to be under primary creep, because the bulk material is in the tertiary creep stage.

8. Page 11, line 30, replace “in” with “are”.

Changed as suggested.

9. Line 13-14, Page 13, please add a citation for this statement.

C3

We added a citation (Steinbach et al., 2017) as suggested.

10. Regarding GBS mentioned in Section 4.6, was there any evidence of this in the observed microstructure? Quadruple points? If not, how would this be incorporated into models for ice if it has yet to be directly observed?

GBS is always a tricky process to infer because, unlike dislocation creep, it does not leave clear microstructural signatures, or its characteristic microstructures have not yet been identified. Furthermore it is unlikely that basal-slip accommodated GBS dominates the deformation at the high stresses explored in our experiments, in which dislocation creep is likely also occurring. Our EBSD maps contain numerous examples of quadruple junctions and near-quadruple junctions. Similar observations have been used to infer GBS in other materials (Negrini et al., 2018) but the issue is complex (Kellermann-Slotemaker and De Bresser, 2006) and needs a more systematic investigation to be useful in these experiments. The microstructures of the samples deformed at -20°C and -30°C are very similar to those in the recrystallized portions of samples deformed by Craw et al. (2018). In the Craw et al. work, the (natural) samples had much larger original grains, and explaining the CPOs of the porphyroclasts and recrystallized grains is much, much easier if GBS is allowed. We cannot prove that GBS is occurring in our experiments, but we can infer it is active based on extrapolation of existing flow laws for GBS to the conditions of our experiments, and that it will then influence the evolution of the CPO. Modelling GBS allows us to explore its potential effects on CPO in a more rigorous way. Another indication that GBS is likely is that peak stresses at a given strain rate are grain size-dependent (Qi et al. 2017). Grain size sensitivity, with strain rate increasing with decreasing grain size, requires grain boundary sliding (Langdon, 2006) for kinematic reasons, irrespective of whether sliding is accommodated by diffusional or dislocation processes. As we have evidence that there is grain size sensitivity (and thus a component of GBS) at the (larger) starting grain size (Qi et al. 2017), it is likely that GBS becomes even more important as the grain size is reduced with strain.

C4

11. Conclusions Section. Could also be more concise. (e.g. no need to summarize the method and/or results before presenting a conclusion)

We consolidated this section as suggested. The summary of the experimental methods a was removed.

12. Figure 3b – and with regard to Question 2: :Am I correct to interpret the increase in the shear stress in these tests as related to the piston becoming displaced and the onset of frictional effects? If not, could you further explain the cause of the increase in the shear stress?

The raw data are illustrated in Figure 3a. The stress is roughly stable with increasing strain in the raw data, which suggests there is no onset of an additional frictional force. In Figure 3b, the increase in the stress is due to the application of the area correction. As shear strain increases, the piston becomes displaced (see Figure R1 above), and the area of the sample that is in contact with both top and bottom pistons decreases.

However, after consulting with experimentalists who are more familiar with the direct shear method (Greg Hirth and Leif Tokle), we decide to remove panel b from Figure 3. They have concluded for a large data set on other earth materials that stress is transferred across the whole cross sectional area up to high strain and that the area correction is not needed. The observation that the grain sizes of our samples do not change much with increasing strain, especially in the -20 and -30°C experiments, suggests that the stresses in our experiments are roughly constant with increasing strain at strains > 0.2. This confirms that an area correction is not necessary.

13. Figures 5,6,7 – It's not clear to me what is being indicated with the blocky black arrow on the left of these maps. Is this a transverse view/map?

These black arrows mark the shear direction. These figures are transverse view, which we called it shear plane, as illustrated in Figure 1. You can see the black arrows in Figures 1 and 2. In the captions of Figures 5, 6 and 7, we mentioned that: "The shear

C5

direction on the top side is up, as shown by the black arrow."

14. Figure 10 – Is it possible to also quantify the Key Processes related to the Final microstructure? Such that these 2-D characteristics could be identified with an automated algorithm? Perhaps see Lehto et al. 2016, Characterization of local grain size variation of welded structural steel, as a good starting point. It seems that there needs to be a better method of identifying and/or quantifying the differences in these microstructural regimes.

Thank you for suggesting Lehto et al. 2016. It is a very interesting paper, but beyond the scope of the discussion in our paper. In our paper, we are focusing on the observed transition in the CPO patterns. We use microstructural evidence to support our explanation of CPO formation, but at the current stage it is very difficult to quantify the contributions from different recrystallization processes. We have considered kernel average misorientation and subgrain boundary density, but neither of them is a good proxy for a recrystallization mechanism. This is an area of future research for our groups.

15. Lastly, after reading Maurine Montagnat's insightful comments pertaining to this manuscript, I would have to agree that it is difficult to ascertain with any certainty the nucleation mechanisms responsible for recrystallization from a 2-D post-mortem analysis alone.

Yes. There are difficulties in determining the nucleation mechanism from a 2D section. We have removed the sentence related to the nucleation process in the conclusion section.

Dell'angelo, L.N. and Tullis, J., 1989. Fabric development in experimentally sheared quartzites. *Tectonophysics*, 169(1-3), pp.1-21.

Jacka, T.H. and Maccagnan, M., 1984. Ice crystallographic and strain rate changes with strain in compression and extension. *Cold Regions Science and Technology*, 8(3),

C6

pp.269-286.

Langdon, T.G., 2006. Grain boundary sliding revisited: Developments in sliding over four decades. *Journal of Materials Science*, 41(3), pp.597-609.

Negrini, M., Smith, S.A., Scott, J.M. and Tarling, M.S., 2018. Microstructural and rheological evolution of calcite mylonites during shear zone thinning: Constraints from the Mount Irene shear zone, Fiordland, New Zealand. *Journal of Structural Geology*, 106, pp.86-102.

Qi, C., Goldsby, D.L. and Prior, D.J., 2017. The down-stress transition from cluster to cone fabrics in experimentally deformed ice. *Earth and Planetary Science Letters*, 471, pp.136-147.

Schmid, S.M., Panozzo, R. and Bauer, S., 1987. Simple shear experiments on calcite rocks: rheology and microfabric. *Journal of structural Geology*, 9(5-6), pp.747-778.

Slotemaker, A.K. and De Bresser, J.H.P., 2006. On the role of grain topology in dynamic grain growth—2D microstructural modeling. *Tectonophysics*, 427(1-4), pp.73-93.

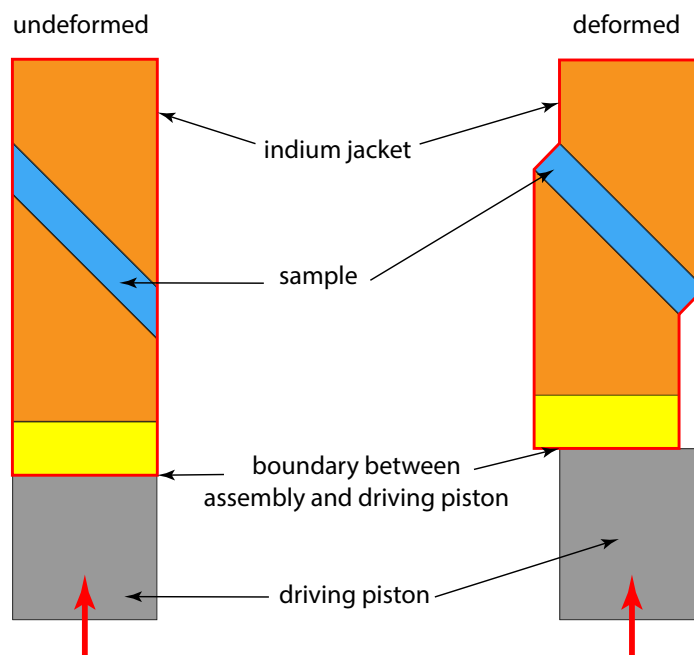
Steinbach, F., Kuiper, E.J.N., Eichler, J., Bons, P.D., Drury, M.R., Giera, A., Pennock, G.M. and Weikusat, I., 2017. The relevance of grain dissection for grain size reduction in polar ice: insights from numerical models and ice core microstructure analysis. *Frontiers in Earth Science*, 5, p.66.

Please also note the supplement to this comment:

<https://www.the-cryosphere-discuss.net/tc-2018-140/tc-2018-140-AC2-supplement.pdf>

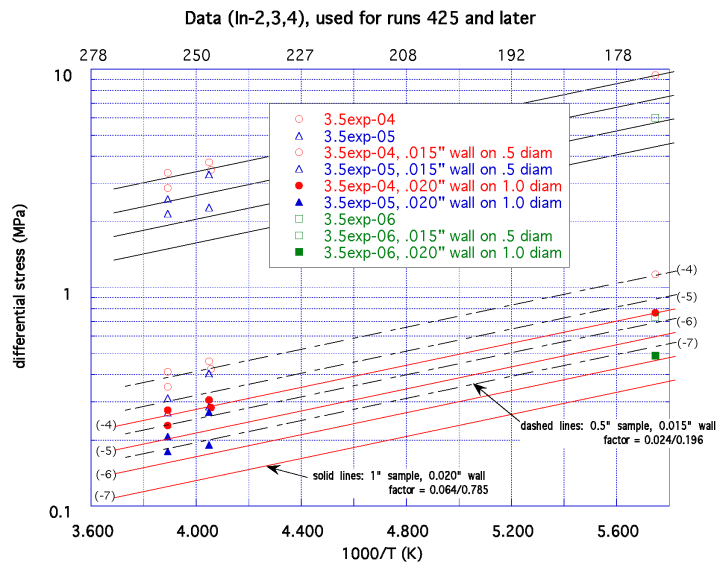
Interactive comment on The Cryosphere Discuss., <https://doi.org/10.5194/tc-2018-140>, 2018.

C7



**Fig. 1.** Figure R1. Schematic drawing illustrating the movement of the pistons during deformation.

C8



**Fig. 2.** Figure R2. Plot of differential stress vs. temperature for indium. Data from W. Durham.