

Review of *Journaux, Baptiste et al*: “Microstructure and texture evolution in polycrystalline ice during hot torsion. Impact of intragranular strain and recrystallization processes.”

By *Dave Prior*, University of Otago. 9th December 2018.

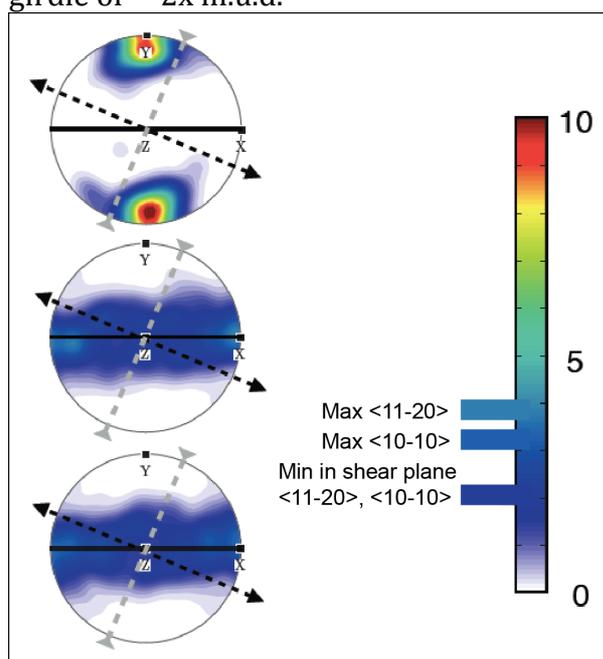
General Comments:

This is an excellent contribution that presents new data on the evolution of microstructure and crystallographic preferred orientation (CPOs) of polycrystalline ice during simple shear up to a shear strain of ~ 2 . The paper is reasonably well-written and is well-illustrated. I enjoyed reading it. The wmv analysis is particularly nice and provides a good analytical template for other researchers (including me!). I have some significant scientific discussion points that I would like the authors to consider and I have some suggested modifications. In addition to the comments in this document, I have annotated a pdf of the paper. I hope that my comments are useful.

There is significant complementarity between this paper and a paper that we also have in Cryosphere Discussions (<https://www.the-cryosphere-discuss.net/tc-2018-140/>), with Chao Qi as first author. I will refer to this paper as (Qi et al., 2018) in this review. I hope that the authors can relate some of their observations to the ones that we have made; we will endeavour to do the same.

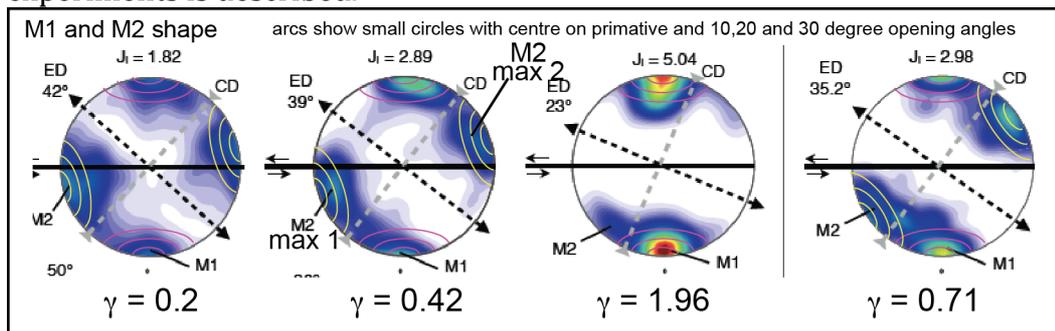
Scientific Discussion:

1. Some of the description of the CPOS is misleading or incorrect.
 - a. The **$\langle 11-20 \rangle$ and $\langle 10-10 \rangle$** in the high strain sample ($\gamma=1.96$) are not randomly distributed within the girdle. The $\langle 11-20 \rangle$ and $\langle 10-10 \rangle$ both have broad maxima, parallel to the shear direction, of $\sim 4x$ m.u.d. and $\sim 3x$ m.u.d. respectively. These compare to minima within the girdle of $\sim 2x$ m.u.d.



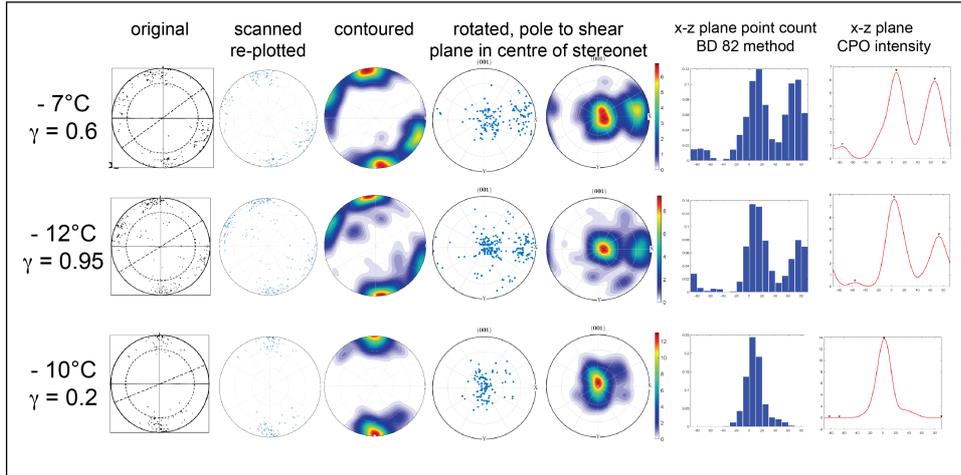
This level of $\langle a \rangle$ and $\langle m \rangle$ alignment is comparable to that shown for the highest shear strain data at -5C in fig 4 of (Qi et al., 2018). Additionally the ratio to the $\langle c \rangle$ axis maximum ($\max \langle a \rangle \sim \max \langle c \rangle / y$ where y is between 2 and 4) is very similar to the highest shear strain data at -5C and all data at -20C and -30C in fig 4 of (Qi et al., 2018). The alignment of $\langle a \rangle$ and $\langle m \rangle$ orientations is important. This might provide a cool tool for assessing shear directions in the analysis of naturally deformed ice so it needs to be documented. $\langle a \rangle$ and $\langle m \rangle$ being co-aligned matches our data and is intriguing. At present I do not have a coherent explanation for this. I'd be interested to hear your views on this.

- b. You have not commented on the **shape of the M1 and M2** maxima. In virtually all experimentally sheared polycrystalline ice samples these maxima are elongated in a direction perpendicular to the shear direction (see discussion in (Qi et al., 2018) and in our response to a Maurine Montagnat comment on this in the discussion section). Sometimes the elongated maxima (both M1 and M2) are actually each double maxima, with the profile plane as a mirror plane. The vast majority of naturally sheared ice samples do not have elongated maxima, the contours of the maxima match small circle distributions (e.g. (Hudleston, 1977)). This point of difference between experiment and nature is important and as such it is important that the shape of the M1 and M2 maxima from experiments is described.

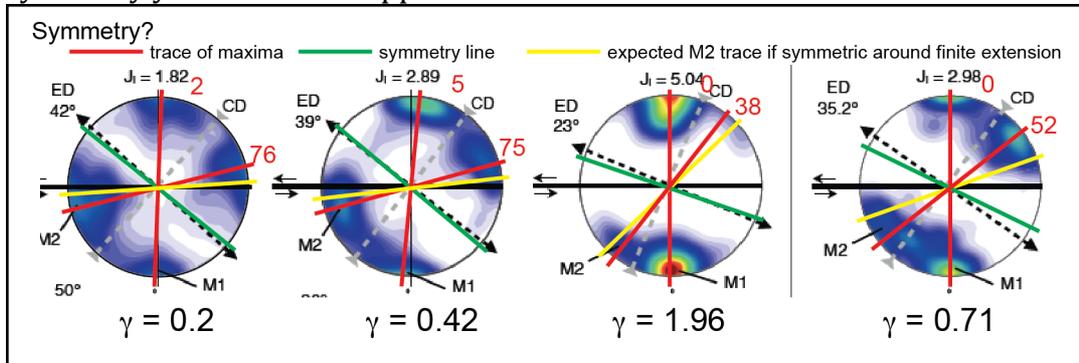


The high strain ($\gamma=1.96$) M1 is clearly elongated in the direction perpendicular to shear. I have superposed small circles, with their cone axes on the primitive, on the figure above to emphasise this point. M1 in the lower strain experiments is not so clearly elongated. In the annealed experiment the contours match the small circles, and it looks like this is the case for the lower strain experiments. In our experiments (Qi et al., 2018) elongation increases with shear strain.

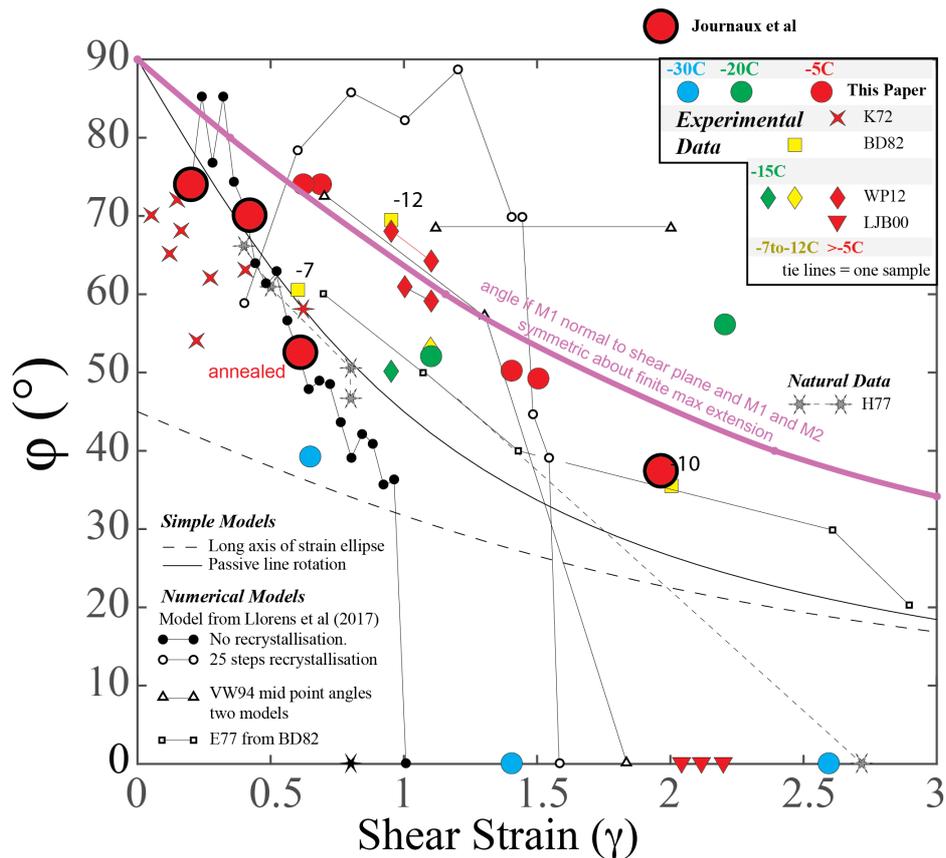
M2 in the $\gamma=0.42$ experiment is elongated, with a double maximum (labeled above max1, max2), with the profile plane as a mirror plane. The $\gamma=0.42$ experiment may also show this but I can't tell from the figure. Interestingly M2 in the annealed sample does not look elongated. This could be an important point. Does annealing remove the cluster elongation? One of the reasons we adopted a different reference frame in (Qi et al., 2018), with the pole to shear plane in the middle of the stereonet, is that it makes it easier to see cluster shapes, as shown below in a re-analysis of the (Bouchez and Duval, 1982) data. The highest and lowest strain samples in these data have elongated M1, the medium strain sample does not.



- c. I think you need to be a little more precise in description of the symmetry of the M1, M2 maxima pair with respect to the finite elongation direction. I think this is a cool observation and potentially of some value, but the symmetry is far from perfect. Below I have plotted up some traces for M1 and M2 (red lines), with angles measured from the top of the stereonet. The green line has equal angles to the two red traces. Superficially this green line is close to the finite extension direction (ED), but if I plot the expected M2 trace (yellow line) assuming it has the same angle to ED as M1 (and adjusting ED for for M1 not being at 0 degrees in the two lowest strains) then the observed M2 is anticlockwise of the yellow line for the three lowest strain, most markedly for the annealed sample. The symmetry you describe is approximate.



Another way of looking at this is to plot the angle between M1 and M2 against shear strain. Below is a modified version of fig 8 from (Qi et al., 2018) with the addition of your data (big red dots) and a line (pink) that predicts the position of M2 if it has the same angle to the finite extension direction as M1. This is quite an interesting addition to the plot as very broadly the red data points (high T experiments: not just yours) do follow the path of the pink line, but at slightly lower angles? Is M2 at high T and low shear strain ($\leq \sim 2$) related to the orientation of the finite strain ellipsoid?

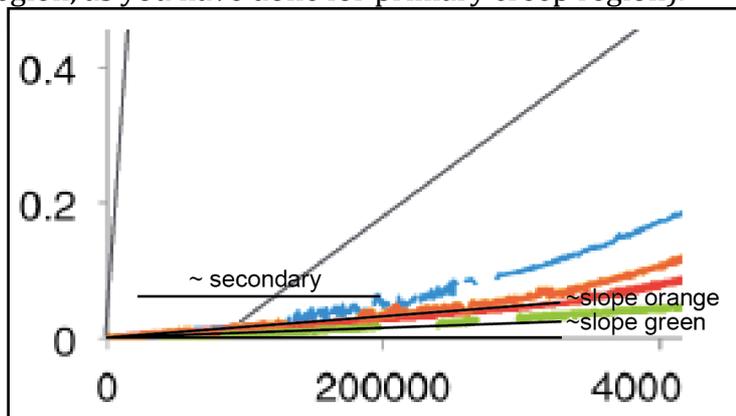


2. The **description/ documentation of the experimental set up** needs to be improved. Please provide some key diagrams that show the experimental set up. Torsion is an important deformation kinematic and the torsion experiments you show here and the classic work of (Bouchez and Duval, 1982) represent significant contributions to our understanding of ice with direct application to polar ice sheets and glaciers. I believe that torsion is an important deformation kinematic to explore more fully in the future. The picture in (Duval, 1976) and the words in (Bouchez and Duval, 1982), (Duval, 1976) and presented here are insufficient for someone to reproduce the experimental set up. It would be great if you could present (maybe in supplementary information) some diagrams that show the mechanics of the deformation apparatus. There is one particular aspect that I think is of paramount importance. I think that this apparatus is constrained to deliver simple shear, with no shortening or extension normal to the shear plane. If this is the case I presume that the “platens”, that deliver the torque, are fixed so that they cannot move normal to the shear plane. This is important so that we can be clear which experiments are simple shear only, and which comprise simple shear with a component of shortening (or extension). This is not necessarily the same as having zero normal stress on the shear plane. (Li et al., 2000) (a key paper that is not cited in your work) point out that direct shear experiments using a “Jacka” rig, with the normal load set as zero still experience shortening/ extension normal to the shear plane (and that the magnitude depends on sample geometry). Furthermore they suggest that an experiment with fixed platens will generate shear plane normal stresses of 0.1 to 0.2MPa. In my view a constrained (by fixed platens) simple shear

experiment is great - it's a clear kinematic end member. We do need to be absolutely clear about the experimental kinematics and the implications the kinematics have for stress, rheology and microstructure. What are the kinematics and dynamics of naturally deforming ice systems is yet another matter. I can imagine some scenarios (e.g. ice stream margins) where perfect simple shear may occur and others (e.g. basal zones) where shear with shortening parallel to the shear plane occurs.

3. The **mechanical data** are a bit puzzling. The focus of this paper is the microstructure, and I don't think the questions about the mechanical data affects substantially the microstructural observations and interpretations, but I would like to see a bit more analysis of the data. The key problem for me is that the applied shear stress should be the dominant control of the shear strain rate (whether secondary, tertiary or at a \sim given strain in transient creep), given that your temperature and starting materials were nominally the same for all experiments. A shear stress of 0.6MPa vs 0.50.5MPa should give a \sim doubling of strain rate (for n between 3 and 4). The secondary creep rate for TG10.42 (0.6MPa) is slower than that for TG10.71 (0.5MPa) and faster than TG10.2 (also 0.5MPa). In the text this is attributed to "variability of grain size and textures". This could be true, but it needs to be unpicked in a bit more detail.

The method used to fabricate the starting material sounds the same as that we use (except that we do not anneal) as described in (Stern et al., 1997). We have looked at >10 samples of starting materials made by the same methods in four different labs (Otago, MIT, UPenn, UCL) and all have very very similar grain size distributions, mean grain size and random CPO; an example is in fig 1a in (Qi et al., 2017). I cannot see that the annealing will affect the CPO and annealing at consistent T and time should give the same grain size distribution. Do you have initial g size data from more than one sample? We can estimate what grain size differences would be needed to explain the variations in secondary creep rate. The ratio of secondary creep rates of the two samples deformed at 0.5MPa is about 2 (estimated from slopes on fig: would be good to provide an enlargement of secondary creep region, as you have done for primary creep region).



Using the grain size exponent (-1.4) from (Goldsby, 2006; Goldsby and Kohlstedt, 2001) this would require the relative mean grain sizes of the two samples to be \sim 1.7. (e.g. 1.5mm and 0.9mm). This grain size exponent may be a bit large. A more conservative estimate (related to similar starting materials) comes from using the peak stress (= secondary creep) data in (Qi

et al., 2017), fig 3. This gives an \sim grain size exponent of -0.8, requiring a grain size ratio of \sim 2.3 (e.g 1.5mm and 0.65mm) to explain the strain rate differences at 0.5MPa. I am pretty sure that your original grain sizes do not vary by a factor of \sim 2, so grain size is unlikely to provide an explanation for the variability in mechanical data.

Although it seems likely that your bulk CPO is random in all starting materials, it is worth considering whether the sample cross section contains enough grains to give the mechanical properties of a random CPO. This was clearly an issue for us deforming 1 inch diameter samples with a \sim 5mm grain size (Craw et al., 2018): in this case a cross section may contain only 10 or 20 grains and the peak stress (= secondary minimum) data do not have a systematic relationship to strain rate. In your case there should be \sim 500 grains in a 35mm diameter cross-section so I would have thought this effect is unlikely to be significant.

It seems unlikely to me that the variations in strain rate relate to variability in the starting material. In this case it's worth looking back at the experimental set up. How is stress transferred from the rotational drive platens (this needs describing- see point 2) to the sample? Is there a possibility that there is some slippage (frictional loss) or other parameter that varies from one sample to the next so that the torque is not all transferred to shear stress on the sample?

4. The **discussion of modeling** is rather black and white and superficial. Numerical models and physical experiments all have limiting boundary conditions. All models and experiments show us something and none match nature, primarily because we cannot access natural conditions and have uncertainties about natural boundary conditions. Linking physical experiments to numerical models is important as we have much more control on the boundary conditions in both cases: so we learn more about our understanding of processes. However the crucial thing for both experiments and models is that we are clear about what we learn from them. I think having a model that is able to simulate fully CPO and microstructure evolution at high strains is still a way off. All steps on the way to achieving this are valuable and a discussion that implicates that one model is right and another wrong is inappropriate: it demeans what we learn from the models.

I agree that the Etchecopar model as used in (Bouchez and Duval, 1982) matches quite well your data and most of the "hot" shear data (see the red symbols in M1-M2 angle vs shear strain graph posted earlier: Etchecopar model also plotted on this as hollow black squares). The problem is that these are the only data it fits, so if this model is applicable it tells us only part of the story. The model does not predict the drop off to single maxima by shear strain of 2 in (Li et al., 2000); maybe this is a kinematic difference between simple shear and simple shear plus some strain normal to shear. The model does not match the minimal "colder" data we have, most particularly the -30 data from (Qi et al., 2018). The FFT model (Lebensohn, 2001) gives a remarkable match to experimental observations of intragranular deformation at low strain (Grennerat et al., 2012; Lebensohn et al., 2009). This is the code used to simulate shear deformation in the models by (Llorens et al., 2016; Llorens et al., 2017). The fact that the same model works well at low strain and less well at high strain tells us something. The bulk CPOs in

Lloren's models do not have double maxima, but the double maxima are there when only the high strain rate data are used (see Llorens, 2017 fig 5i) and the angle between maxima in the deformation only models evolves in a way that matches the -30 experimental data we have (Qi et al., 2018). Addition of recrystallization into the model changes the result, although not in a way that gives a really clear match to observations. There is no real conclusion here apart from this: both models and experiments are important. Probably most important is to design experiments that enable clear boundary condition matches to numerical models. That is the really beautiful thing about the columnar ice work at low strain e.g. (Grennerat et al., 2012). At high strain and in shear matching of model and experimental boundary conditions is rather harder.

Clarity of writing

The bulk of the text is well-written. The clarity of the writing is not as good in the discussion and not good at all in the conclusions.

The discussion would benefit from some shortening and restructuring. The discussion starts with a reminder of the key observational data and I think it would be very helpful to the reader if you added a schematic diagram to highlight these key observations. This would then give a clear framework for ongoing discussion.

The conclusions needs to have clear statements on what are the new factual observations and what are the interpretations of those observations.

The abstract should be a concise summary of the new findings and some short statement about importance. The abstract contains an extended statement of background that is better placed in the introduction (it is in fact already in the introduction).

I would go for a simpler title: "Evolution in polycrystalline ice microstructure during progressive high temperature shear" ????

Technical/ terminological/ picky things (in no particular order):

5. It would be great if you could show full grain size distributions (frequency plots). You are correct that the mean is not a great scalar to represent recrystallized grain size statistics. Grain size distributions could be represented as an extra row in figs 2 and 4 (it would be nice to compare the AITA and EBSD measures- I don't expect them to be the same: see (Cross et al., 2017))
6. Please put the number of grains that correspond to each pole figure on figs 2 and 4 or in a table. This is important in comparing data sets.
7. If you can, show point stereonets as well as contoured nets. The contouring hides a lot of information.
8. The statement on page 2, line 26 states that the "texture can increase shear strain rate (word "rate" missing) by a factor of ... ". There is a clear correlative relationship of weakening and CPO but a causative relationship is not established. Weakening in ice from secondary to tertiary creep correlates with development of a CPO. It is intuitive that the CPO developed in shear

facilitates further shear. However similar weakening occurs in cold axial shortening where the CPO (cluster of c-axes parallel to shortening) would intuitively make further axial shortening harder e.g. -30 experiments right hand column of fig 3 in (Craw et al., 2018), mechanical data in fig 10. Other changes correlate with weakening, most particularly grain size changes (as documented in your paper and elsewhere). In the geological literature grain size reduction is often thought of as the main cause of weakening. In reality CPO, grain size and other microstructural parameters all change in correlation to change in mechanical behavior. It is unlikely that the mechanical evolution is caused by changes to just one of these sample parameters.

9. I don't think that Kamb's idea that CPO is independent of T, strain rate or stress is confirmed (P3, L11). The data in (Qi et al., 2018) show that in shear the CPO changes with T. (Qi et al., 2017) show that in axial shortening CPO is sensitive to stress or strain rate (the two cannot be separated). It is reasonable that the stress/ rate effect will also apply in shear. Using Huddleston's data in comparison to experiments is complex as both T and rate change. The lower rate has a similar effect to deforming hotter.
10. The statement on page 5, line 8 is incorrect. Cryo EBSD of ice is not (in general) limited to samples of ~10 by 20mm. In terms of published data there is a map in (Prior et al., 2015) (fig 12) of 80 by 30mm, the data in (Wongpan et al., 2018) has maps up to 40 by 40mm etc. Most of the CPO data we publish from experimental samples come from 25.4 by 40mm samples, our shear data CPOs in (Qi et al., 2018) are from elliptical shear surfaces of 25 by ~ 30mm. For natural samples we routinely work on samples of ~60 by 40mm and with suitable cold stage modifications I don't see why 100 by 50mm is not achievable. EBSD maps with the same dimensions as your AITA maps are possible now. If the Montpellier machine has a sample size limitation and this limitation is important to the paper, then link the limitation to that instrument, otherwise just delete the statement about size limitation. I guess if it the Montpellier machine does have a limitation it must be to do with cold stage tethering (gas pipes) or camera position limiting WD, as the sub-stage is designed for very large stages/samples (Seward et al., 2002).
11. Please provide enough information for the reader to understand how surface sublimation is managed. What I mean by this is; how is frost removed from the sample. There will be a frost layer on the sample surface as it goes into the SEM that would prevent EBSD (needs only ~ 10-20nm to do this). The two main ways of removing the frost are to heat the stage (Iliescu et al., 2004; Weikusat et al., 2011) or to cycle through pressure (Prior et al., 2015). I recall Andrea Tommasi telling me that the sample is just put in the SEM and it works. In this case I infer that the sublimation to remove the frost occurs on the down pressure cycle and that the sample is warm enough when put in the SEM to give a path through PT space where the sample goes into the vapour field (see fig7 in (Prior et al., 2015). In this case it would be useful to know the sample temperature on insertion and the pressure sequence: do you go to high vacuum then to controlled gas pressure or directly to controlled gas pressure?

12. Please say in figure captions if pole figures are equal area or equal angle. I think they are equal area from the shapes of maxima (the projection affects shape analysis of maxima).
13. It would be really cool to see a radial section of the sample: to see how microstructure changes with strain in a single sample (e.g. see (King et al., 2011). I'm not suggesting this is needed for this paper- just something cool to do.
14. There are a few key references on experimental shear of ice that are missing and should be cited. These include (Budd et al., 2013; Li et al., 2000; Wilson and Peternell, 2012).
15. There are several published papers that show a lack of CPO change in rocks during annealing. Some of these should be cited.(Augenstein and Burg, 2011; Heilbronner and Tullis, 2002; Ree and Park, 1997). I know there are others in calcite and olivine but can't find them just now.
16. Throughout this paper the term "texture" is used with the meaning common in metallurgy and materials science. There is a very small community of geoscientists who use "texture" in this way and no glaciologists that I know of. For the vast majority of the geoscience community "texture" means the spatial relationships of phases and grains and their internal structures. To most geoscientists, texture is what you would see down a microscope (in a petrographic examination for example) and is broadly synonymous with the term microstructure. The terms "crystallographic preferred orientation" (CPO: which you use in the intro) or "lattice preferred orientation" (LPO) are much better as they are explicit. If you want this paper to have wider readership/ uptake, remove the word texture throughout and replace with CPO. It is also worth (in the intro) relating this terminology to the word "fabric" and/or the acronym "COF" (crystal orientation fabric) as commonly used in glaciology. I avoid using the term fabric (except in explanations of how terminology matches up) as metallurgists use this term to mean microstructure.
17. It is not really clear what are the observations you use to constrain the dimensions of the bulging nucleus.
18. I don't follow the discussion related to nucleation in the section where the annealing is discussed. Grain size increases during the annealing so nucleation is unnecessary. If you are talking about relationships that might be relevant to nucleation prior to the annealing then this needs to be made clear.
19. Bulges cut off by rotation of a subgrain boundary was first suggested (described from see through experiments) by Janos Urai (I think). You should reference (Urai et al., 1986).
20. Spontaneous (random) nucleation? I have a problem with this - it is a bit of magic with no physically realistic explanation.
21. The conditions of your experiments are not close to those in cold glaciers and ice streams (page 18, line 5). Your **slowest** transient strain rate is $2.7E-7s^{-1}$ which corresponds to a 100m thick shear zone having a velocity difference across it of 850m/yr. The tertiary strain rate in your high strain experiment corresponds to $\sim 2700m/yr$ difference across a 100m shear zone. I'm not so familiar with temperate glaciers but such shear rates do not exist in polar ice sheet systems eg (Bons et al., 2018; Rignot et al., 2011). Even fast ice stream shear margins max out below $1E-9s^{-1}$ (Bindschadler et al., 1996; Jackson,

1999; Jackson and Kamb, 1997). The strain rate has a significant effect on the microstructure and the CPO (Hirth and Tullis, 1992; Qi et al., 2017; Tullis, 1972): increasing strain rate has a comparable effect to decreasing temperature. It is not possible to do an experiment to significant strain at natural conditions. Instead experiments need to provide scaling relationships that allow us to predict the effects of T, strain rate (stress) etc on rheology and CPO/microstructure (with the complication that there are feedbacks where CPO/microstructure affect the rheology).

END

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