

## Review of the paper by Fan et al.

### **“Temperature and strain controls on ice deformation mechanisms: insights from the microstructures of samples deformed to progressively higher strains at -10, -20 and -30°C.**

#### **The Cryosphere**

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In this report, I first provide specific comments following the course of the text, and I finish with some more general comments.

P3 l. 28: why should the sample be cooled in liquid nitrogen? Couldn't that induce some thermal stress due to the fast and strong temperature gradient? Changes in local microstructure, dislocation arrangements, etc. are expected to occur in the first minutes after the test... so this quenching should not avoid it.

P7 l. 8-10: in the paragraph just before it is mentioned that the grain size distributions are mostly bimodals, and therefore not gaussians... The mean and STD parameters are therefore not suited to described them, since they do not represent well the given statistics. I therefore suggest the authors to provide medians and quartile data instead to better fit the type of distributions observed.

P7 l. 30: couldn't it exist a bias link to the lack of resolution in step size and misorientation when getting toward smaller subgrains?

Part 3.2.3: this is not clear to me how is the subgrain size defined and calculated. Although we observe a clear grain boundary structure in the figures, there appears no clear subgrain structure (as one could observe in some minerals or metals for instance). On the contrary, subgrains appear more like straight tilt bands or kink bands, with, in some places, some variations around the straight shape.

I would be curious to see, for instance, how was measured this subgrain size in the sample deformed at 3% at -10°C, or at 12% at -20°C. I think that the author should clarify this technical aspect as they make a lot of explanation rely on such “average” parameters.

Furthermore, provided it is calculated properly, I doubt the distribution is normal, and I think that a metric other than the average would fit best.

In particular, the following assertion is questionable:

“because subgrain rotation recrystallization should produce grains that have similar sizes with subgrains, while bulging nucleation should produce grains that have smaller sizes than subgrains” that rely on a parameter (subgrain size) that is ill measured here, since subgrain structure does not resemble at all the one observe in quartz (Halfpenny et al 2012).

On top of that, this expected hierarchy of grain size depending from the nucleation mechanism comes from one study on quartz and should not be taken as granted, see for instance Humphreys 2004 (Materials Science Forum) that shows clearly a bulged grain much larger that the surrounding subgrains. It will therefore depend on the material and its anisotropy, and on the resolution of the observations (ability to distinguish between a grain resulting from a bulge and one resulting from subgrain rotation...)

p8 l. 8-9: To consider  $\langle D_{\text{small}} \rangle$  as a good representative of the mean recrystallized grain size is also a strong hypothesis that should be justified (either by some specific observations or by references from previous work). It will, in particular, depend on the relative effect of grain boundary migration compare to nucleation during recrystallization (and therefore on the temperature of the test) since an apparent small grain size at high GBM rate could well be a 2D cut of a strongly lobated grain, while, at lower temperature (lower GBM rate), small grains indeed are newly

recrystallized grains (see for instance the sample deformed at 8% at -10°C, could one certify that small grains observed on the 2D sections are indeed small grains?).

Here again appears the necessity of statistic metrics adapted to the observed distributions.

For the sake of clarity, I would suggest the authors not to mix result presentations and interpretations, and keep interpretations for the discussion part. In particular when interpretation requires additional hypotheses on top of direct observations and results.

P9 l. 13 “At -10 °C, the CPO intensity of “small grains” is lower than “big grains”, and this contrast becomes strengthened as the temperature decreases.”

This could also be related with the fact that it is less straightforward to distinguish small grains from big grains for these tests, this should be mentioned here.

P11 l. 26

The authors mention “much of the stress increase prior to peak stress relates to elastic strain”, and, as they notice just after, this is not coherent with the known Young modulus of ice of 9 Gpa... There is a broad literature, dating back to the 70’s and 80’s (Duval et al. 1983, Jacka 1984 for instance, and review by Schulson and Duval 2009) explaining that the transient behavior of ice is not elastic, but anelastic, and is related to the built of an internal stress field related to strain incompatibilities between grains. **I am therefore very astonished to read this sentence here, and I think that this should be corrected before publication.**

The “dissipative deformation” mentioned here is indeed plastic deformation related to intracrystalline dislocation slip, the porosity loss being very likely negligible.

Part 4.1.1: Discussion about GBS. The experimental results shown here present no evidence of a grain size sensitive mechanisms, since there is no initial grain size variation, no study of the influence of grain size on the stress – strain-rate relation. I therefore don’t understand why GBS is mentioned here, since it is not necessary at all to explain the observations performed. Indeed, all results presented here can be explained by intracrystalline dislocation slip accommodated by dynamic recrystallization mechanisms, as very well illustrated in the high quality EBSD observations performed.

Furthermore, there exist a large number of studies showing that GBS occurs significantly only in fine-grained materials (see Boullier and Gueguen 1975, Goldsby and Kohlstedt 1997) where grain boundary diffusion can play a role (Ashby 1973). Diffusion in ice is known to be very slow, that renders the hypothesis of a diffusion-controlled mechanism quite unlikely, especially for large grains, and high strain-rate conditions as encountered here.

The authors could try to calculate the strain-rate expected based on a GBS diffusion flow law (Nabarro-Coble for instance) for similar level of stress as the one of their experiences. They would likely see that the stress – strain-rate curves they obtained are not compatible with a GBS-influencing mechanism.

Part 4.1.2: In this part, the authors use the subgrain size measurements to estimate the role of subgrain rotation in the recrystallization mechanisms.

Once again, the subgrain structure observed here is very far from the ones in quartz, to enable using the paper mentioned here as a reference (Trimby et al. 1998), and I think the authors should be much clearer about the way they evaluate the subgrain size before getting to strong an interpretation from this parameter.

Ice behavior, and in particular in the experiments presented here, is very different from the one of more isotropic materials in the sense that the dislocation substructures are not characterized by subgrain cells as observed in Al or Quartz for instance. This is due to the fact that subgrain substructures as observed in Quartz results from equivalent activity of several slip systems.

Although one observe some c-dislocations in the microstructure, slip system activity in ice remains dominated by basal slip, and resulting subgrains have mostly the shape of large tilt and kink bands. Only close to GB and triple junctions will we find more complex substructures. Is it enough to evaluate an “average” subgrain size? Care must therefore be taken before using interpretations coming from these more isotropic materials. And explanation should be given about how is this subgrain size measured here.

P13 l. 1-2: Indeed, Jacka and Li Jun 1994 evidenced a linear relationship between grain size and stress during dynamic recrystallization of polycrystalline ice (creep experiments, tertiary creep). I think that this should be mentioned here.

P13. l. 5: here again, caution must be taken with making use of the subgrain size as it is still ill defined... and the ice case can not be compared straightforward to Halpenny et al. studies! Indeed, observation given l. 16-17 goes in the direction of my remark... Subgrain size, if measurable here, can not be used similarly as in the other studies mentioned since there is no clear subgrain substructure. But, still, subgrain rotation could explain part of the recrystallization by, for instance, closing the bulges (see Chauve et al. 2017, Phil Trans), or by separating grains via highly misoriented tilt or kink bands. But, indeed, one can not talk about “continuous” recrystallization as observed in Al for instance (see Sakai et al. Progress in Materials Science, 60(0):130–207, 3 2014 for a review).

#### Part 4.1.3

p 14 l. 11: “Because grains with hard slip orientations should have greater internal distortions”, there is absolutely no proof of that in ice, and some recent work tend to show that there is no systematic relation between orientation and strain localisation (see Grennerat et a. 2012 for instance) or between orientation and subgrains density (see Journaux et al. 2019 for instance). I think it should not be considered as granted, in particular when not shown directly in your experiments.

Have you tried, for instance, to measure the density of GNDs as a function of grain orientation?

#### P14 l. 20: GBM instead of GMB

About GBS and apparent texture weakening in small grains: to my point of view, this apparent texture weakening could be related to the nucleation process itself, and the fact that close to GBs, local misorientation can be high, and induce nucleation orientations varying from parent grains orientations (by bulging or subgrain rotation). This process would be enough to justify the small difference in texture concentration in small grains (that could also be due to more spread in data as there are less pixels measured in small grains, since GBs are interfering with the measurement, reducing its quality in small grain areas ?). See for instance the work of Falus et al. 2011 about Olivine for rotation recrystallization or Chauve et al. 2017 for the orientation of nucleus formed by bulging.

The work of Qi et al. 2017 mentioned several times in this part concluded that “the dominant mechanism of CPO development occurs with increasing stress, from GBM, which consumes grains with low Schmid factors, at low stress, to the rotation of basal slip planes to an orientation normal to the compression axis at high stress, due to dislocation glide.” I didn’t find any mention of “grain size sensitive mechanism” as certified l. 25...

Such a grain size sensitive mechanism should be verified by varying grain size during the experiments and evaluate its effect on a given parameter, such as peak stress, strain-rate or so.

I maintain that there is no proof of such a GSS mechanism in the experiments presented here, and therefore the interpretation should be cleared about that.

That GBS is more active in smaller grains is well known since Boullier and Gueguen work! It does not mean that it should occur in the specific case here, unless otherwise proven...

The hypothesis that GBM being less active at low temperature, the impact of grain rotation driven by intracrystalline slip prevails is much clearer, especially since it is very coherent with the observations that the cone angle is reduced, and more orientations are found close to the vertical. This assertion is, indeed, justified by the experimental observations.

This is, in fact, the main “novelty” of the presented work and should be emphasised more. Speculation about GBS tends to lessen this message, and also the interest of the good quality observations performed in this work.

Part 4.2:

During dynamic recrystallization, weakening is classically (see Humphreys and Haterly 2001 or 2004 for instance, Sakai et al. 2014) attributed to the reduction of hardening based on GBM and nucleation of grains, both reducing the stored strain energy associated with dislocation pile-up or dislocation structures. Therefore dynamic recrystallization induced weakening does not require the interplay of CPO or grain-size sensitive mechanism to be explained.

Another point for this consideration about weakening: the relative weakening at about 20% strain is similar for every temperature cases, at about 35% ( $\sigma_p - \sigma_f / \sigma_p$ ). Therefore there is not more weakening with small grains than without... It should rule out the hypothesis of a grain-sensitive mechanism to explain weakening. Nucleation and GBM (each one having different relative influence depending on the temperature) are enough to explain the observed weakening, as expected from the dynamic recrystallization literature.

Part 5:

Point 2: from figure 2, the steady state is not so obviously reached, unless, maybe at  $-10^\circ\text{C}$ . Maybe the authors should be more careful about it, especially about mentioning it in the conclusion.

Point 3: regarding my previous comments concerning the evaluation of a subgrain size, I think that either the authors explain very clearly how they evaluate this subgrain size, and show that it is meaningful based on their experimental observations (that they do observe a subgrain network, although it does not appear clearly in the given figures, from which extracting a subgrain size appears relevant), or this parameter, even if used in the discussion with care, should not appear in the conclusion.

Point 5: once again, this conclusion makes use of the subgrain size which measurement method is not clear, and therefore should not be used in the conclusion unless clarified.

Point 6: I think that there is nothing really new in this point... it has been demonstrated for many materials undergoing dynamic recrystallization, and it is a direct evidence from energy considerations... Should it really come as an important conclusion? At least, the authors should be care to mentioned “as already observed”, or “as expected during dynamic recrystallization”...

Point 7: based on my comments concerning part 4.2, the mention of GBS to explain weakening should be removed. It is also surprising that an hypothesis that is only briefly mentioned in a very short paragraph (4.2), could come to an important conclusion point...

Point 8: same as point 7, and please note that weakening should be measured relatively to the peak stress value (for instance), and it therefore leads to very similar weakening for all temperature conditions (about 35%).

## GENERAL COMMENTS:

- In general, there is a lack of references from the work done on recrystallization (on ice and other materials) by others authors than the authors' team.... this is especially true, for instance, in part 4.1.3, and this should be corrected. In particular when other's work do not come to similar conclusions as the authors...
- Maybe related to this lack of references, some assertions are given with too few justifications, that should come either from experimental observations or from previous works. This should be corrected, and the authors could specify that they are making hypotheses when there is no existing justifications.
- This work does not contain any significant novelty, but provides more detailed and accurate observations at the microstructure scale compared to previous (old!) measurements performed by Jacka and co-authors for instance. Compared to the extensive literature about dynamic recrystallization at hot temperature (see for instance Humphreys and Haterly 2001 or 2004), there is no novelty, and this literature should be mentioned, especially within the discussion, in order to help the interpretation of the results.
- The high quality observations enable to assert more clearly some mechanisms as important in the case of recrystallization in ice as, for instance, the fact that at low temperature, intracrystalline rotation will prevail on GBM and therefore induces texture that are closer to the one observed along deep ice cores.
- It is not clear, all over the text, why the authors want or need to mention GBS as an impacting mechanisms since the experiments performed show absolutely no proof of it, neither in macroscopic data (dependance of peak stress on grain size for instance), nor in microscopic observations. The only observation of small grain necklaces (but limited in number) at the lowest temperature, and a weaker texture in this small grain population is not sufficient, to my point of view, to assert the occurrence of GBS. It could be mentioned as one of the hypothesis among others, but not come to the conclusion as the mechanism at play. In particular, the use of GBS is not necessary to explain stress weakening and does not appear coherent with the results.
- I raise again the point about the lack of proper explanation concerning the measurement of subgrain size in the specific case the presented experiments, since the figures shown do not reveal any proper subgrain structure that could be characterized by a dimension (as a mean size for instance). Since different conclusion are taken out of this subgrain size evaluation, it should be corrected before any publication.
- the authors make no use of their observations from the WBV method neither in the discussion, nor in the conclusion... Should it remain in the paper?

As a conclusion, I suggest that this paper should not be published before the authors first enhance the justifications of some strong hypotheses they are making in order to interpret their observations, and second highlight the novelty of their observations and results regarding already existing work.

Ref:

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