Review of Christopher, Wilson et al.'s: "Partial melting in polycrystalline ice: Pathways identified in 3D neutron tomographic images."

General comment on the manuscript:

The preprint article by Wilson et al. presents an innovative approach for characterizing the microstructure of polycrystalline ice using small-angle neutron scattering (SANS) measurements. The authors successfully obtained high-resolution images of the 3D ice microstructure, providing valuable insights into the crystallographic structure of the ice, the distribution of pores, and the connectivity of the pore network. Notably, this study is unique in its use of deuterium ice, which allowed the authors to obtain higher-quality data than previously possible. They explore the role of crystal orientation and pore geometry on the deformation of polycrystalline ice and investigate the effect of stress and strain rate on the microstructural pore evolution of ice resulting from deformation. The paper is well-written and presents a clear and concise overview of the methodology and results. However, there are some sections that may benefit from further clarification or expansion, as outlined below.

We thank this reviewer for these valuable comments and suggestions which have helped to improve the manuscript.

Specific comments for the authors' consideration:

- I'm curious why 90% deuterium ice was chosen. Does this suggest 10% liquid water content when samples deform above the H_2O melting temperature? If so, why not use a higher fraction of deuterium solid to produce water contents more in the range expected of glacier ice? (< 3% liquid water i.e., Vallon et al., 1976). Moreover, assuming liquid water contents are high, I would be hesitant to infer known ice deformation mechanisms in unexplored water-content regimes.

- Because of the sample size, the small quantities of partial melt generated coupled with the resolution of the neutron diffraction tomographic images any lesser amount of H_2O would have made identification of melting sites very difficult. The value of <3% described by Vallon et al. (1976) is in Firn ice and is not applicable to these experiments. In our initial and final experimental samples, you cannot distinguish differences between H_2O vs D_2O both have a polycrystalline grain microstructure and similar grain sizes.

- It remains unclear why calcite powder was used in some layered samples (or at all). Consider including the objective/ relevance of the calcite layer with regard to the overarching research questions.

- A new sentence has been added in Methods section (at line 114) to explain the calcite helped to provide a rheological contrast in the sample.

- When labeling "pores" (i.e., Line 161), consider being explicit as to whether you refer to liquid water interstices (i.e., veins) or bubble inclusions. Further, are you able to get a sense of the volumetric air content in the samples using tomography?

- A volumetric count of bubbles was attempted, but results were highly variable. Instead, we are only presenting data we are confident about such as the data portrayed in fig. 4d,e.

- Following Nye and Mae (1972), the authors may consider clarifying the differences between their deuterium–H_2O samples and pure ice with regard to textural and thermo-mechanical equilibrium at the melting temperature. My (perhaps limited) understanding is that melt evolution, migration, and distribution during compression will be driven by grain-scale stress heterogeneity and a tendency for liquid in a polycrystal to be drawn from warm to cold temperatures as a result. In a system with two distinct melting temperatures, I'm unsure how applicable this paper's results on molten phase migration will be to glacier ice systems.
- As we point out in this paper there is a major structural factor that controls the redistribution of the melts, namely shear bands and the deformation bands and these exist in other experimental studies (e.g. Rist & Murrell, 1994) and in natural ice masses. From a set of complimentary unpublished in situ studies there is definitely no warm to cold transfer, instead it is controlled on activity of the different slip systems between adjacent ice grains and the degree of grain boundary migration.
- I didn't catch how the textural characteristics (including grain size) were measured and what the errors were. Perhaps you could elaborate? I apologize if I overlooked it.
- All the textural characteristics came from a fabric analyser. Therefore, an additional sentence has been added to line 106 in 'Methods Section'.
- It remains unclear to me how the coordination number was measured (and what the errors are) in the mean CNs (Table 1). Could you elaborate? And do you think the resolution is sufficient to adequately characterize the connectivity of pores in the samples? (i.e., if your voxel resolution is 20 microns, are melt channels smaller than 20 microns overlooked and/or deemed insignificant?)
- Two additional references have been added to the paper (Andrew, 2018; and Berg et al., 2016) plus a sentence has been added at line 145 in methods section and to the caption to Table 1 to clarify the procedure used. In the deformed samples we have images of total porosity showing pores smaller than 20 microns and distribution of melts. These we have not used in this paper as their resolution does not show clear details.
- Were you able to examine the general melt channel shape in your samples? I'm curious whether the mean dihedral angle is greater for deuterium ice (possibly producing more spherical pores), causing the pore connectivity and melt migration rates to be lower than pure polycrystalline ice.
- During the processing of the data an examination of the 3D channel shape was not undertaken, However, as we point out in Figures 1-3, on the margins of all the deformed samples the melt accumulates as circular patches that correspond with the apex of deformation bands indentified in the relevant 2D slices. Whereas, in adjacent slices no melt is identified. Regarding the dihedral angles, determining these was beyond the resolution of our tomographic images. However, we are currently writing up complimentary in situ experiments undertaken on a fabric analyser (similar to those described in Peternell et al., 2019) in which we record melting occurring and redistributed in a matrix of H_2O ice where dihedral angles control melt migration on a localised scale, however, shear bands again control the overall distribution of melt.
- I think the conclusion could be strengthened by summarizing the main findings of this study and their significance in a more succinct way, as well as highlighting the key areas for future work that emerge from the study.
- A short conclusion has now been added to the manuscript.

Overall, this paper presents original, high-quality data on the deformation behavior of laboratory-made ice samples under uniaxial compression tests. The novel use of

neutron imaging allows for non-destructive 3D visualization of the internal ice structure during and after the deformation, providing unique insights into the deformation mechanisms of ice. The results have implications for a range of applications, including ice mechanics, ice sheet modeling, glacier dynamics, and englacial hydrology. Therefore, I believe this paper is well-suited for publication in The Cryosphere.

We thank the reviewer for an excellent set of comments and suggestions.

Editorial comments keyed to line numbers:

28 – Insert the word “to” before “suggest” Changed to “suggesting”

43–44 – Consider adding a comma after “masses” and some rephrasing, as the meaning in this sentence I find unclear. Comma added. We believe it is clear.

59 – Change the word “occurs” to “occur” changed

149 – Consider changing adopting to “as they adopt” as it reads a bit awkward otherwise. Thanks now changed

154 – Missing first parenthesis in “Supplementary Fig. 3). Added

164–166 – This reads a bit awkward. Consider changing “its correlation” to “correlating it” perhaps? changed

172 – Consider adding a comma after “sample” for clarity Added

174–175 – “Mix-3 occurs as a fine rim (Fig. 1d-e) and Mix-2 and Mix-3 at the outer rims of the sample (Fig. 2a-c)” reads a bit awkwardly; consider rephrasing for clarity. This has been rephrased.

193 – Change “concentration” to “concentrations” (for agreement with “are”) changed

194 – Consider adding comma after “(Fig. 2e, Supplementary Fig. 3a)“ added

211 – Add “and” before “blind” added

219 – Change “relative” to “relatively” changed

220 – Add a comma after “samples” Added

224 – Change the word “was” to “were” Changed

232 – Consider adding a comma after “(Kronenberg et al., 2020)” Added

233 – Add a hyphen between “meltwater” and “free” added

243 – Move the hyphen position to be between “dry” and “compacted” undertaken

250 – Hyphenate “quasi steady” added

266 – Add the word “and” after “boundaries,”added

276 – Change the word “are” to “is”changed

278 – Consider changing the word "shears" to "shear bands" for consistency with later usage added “bands”

281 – Hyphenate “dry compacted”; this is a bit inconsistent throughout the paper, so check occurrences elsewhere for consistency.changed

283 – Add the word “and” after “shapes,”added

292 – Consider changing “which preceded” to “that precede” for grammatical correctness.changed

312 – Add a comma after “stresses” added

330 – Add a comma after viscosity, or change “reaching” to “reaches.” With the current phrasing, the meaning of the sentence is unclear. Comma added

356 – Add a comma after “(Fig. 10e)”. added

389 – Remove the hyphen in “ice-sheet” changed

392 – Remove the comma and change “is” to “are.” Otherwise, I think it reads awkwardly. changed

405 – Change the word “control” to “controls” and change “on” to “of.” changed

423 – Consider bracketing “more commonly” with commas on either side. added

559 – I would suggest explaining what is meant by “pore fluid factor” and, additionally, consider adding a hyphen between “pore” and “fluid” here.

This relates to a change made in the caption to Figure 10. In addition, a sentence has been added in text at line 360 explaining the nature of the other factors.

655 – Consider explaining what is meant by a “capped yield surface” as I, and perhaps others, will be unfamiliar with that terminology.

An explanation regarding the use of a capped Mohr-Coulomb diagram has been added to section 4.1.

References:

Vallon, M., Petit, J., & Fabre, B. (1976). Study of an Ice Core to the Bedrock in the Accumulation zone of an Alpine Glacier. *Journal of Glaciology, 17*(75), 13-28. doi: 10.3189/S0022143000030677

Nye, J., & Mae, S. (1972). The Effect of Non-Hydrostatic Stress on Intergranular Water Veins and Lenses in Ice. *Journal of Glaciology, 11*(61), 81-101. doi:10.3189/S0022143000022528

Additional changes

Figure 6. An additional statement has been added to caption to explain stress increase.

Figure 10. An important detail was changed in (b) the shear bands are now inclined at less than 45deg to the compression axis. For (c) this was correct. For (d) the angle had to be greater than 45deg and this modification has also been undertaken.

Reply

RC2: 'Comment on egusphere-2023-70', Anonymous Referee #2, 12 Jul 2023

This paper deals with the effect of melt on the rheology and permeability of ice sheets and glaciers ices. The authors make use of ice specimens made of a mixture of deuterium and water ices. This is a very interesting idea as both have different melting point, and neutron scattering can distinguish between both allowing neutron tomography to be carried out.

The topic of the paper is relevant and very interesting, however I am asking for a rejection of this contribution as the authors largely over-interpret the experimental results that are shown. Many times in the paper, the figures do not provide any support to the text and interpretation, and therefore, after completing the reading, I am not convinced at all by the robustness of the results. Sometimes, the text is completely disconnected with the figures (experimental evidences) provided.

We completely reject the above statement. This reviewer has failed to question any of our scientific aims, techniques, arguments or the manner of presentation. In comparison the comments of RC1, which were well structured and form the backbone for the revised manuscript. RC2 has presented an incomplete hurried review, as there are sweeping assertions with no solid backing, the reviewer's sentence construction is poor and the review is full of typos. All comments have been carefully evaluated and we have ended up making minor additional changes to the text in response to RC2, as highlighted in our response below.

- The paper severely lacks of a quantitative analysis. For example, the sample porosity and its evolution with strain, which has a central position in the paper, is never given quantitatively, although it can be estimated from neutron tomography. Porosity might affect the effective behaviour even more than the melt content (the melt content is also never quantify !). ex. line 193 state an 'increase in porosity', but no evaluation is provided.
- Unfortunately, this is a very misleading and highly inaccurate comment. Numerous quantitative steps have been necessary to process all the data sets even before the results were presented e.g. Visualisations (e.g. Figs 1-3); Pore densities (Fig. 4), Textural changes (Fig. 5), Stress-strain relationships (Fig. 6), Microstructures (Fig. 7); Grain numbers (Fig. 9).
- As for porosity and its influence on strain, well this is illustrated in Fig. 8b, and discussed in the text.
- It is not clear to me how the author can distinguish between water ice and melt water with tomography… Is this possible ? How much of the specimen really melts during the experiment ?
- This is clearly pointed out in the manuscript in sections 2.3 and 3.1. The manuscript points out how this was undertaken with appropriate references to the processes that we have used, especially during segmentation e.g. Kahn, et al., 2012; Wang et al. 2015; Andrew, 2018). This could not have been undertaken unless there were different attenuation coefficients for H2O (2.4 cm-1) and D2O (0.35 cm-1) as pointed out on line 135-136 in the original manuscript.
- The amount of melt is difficult to define, because of the degree of proton exchange between deuterium and hydrogen, and depends on the where strain is localised and varies from slice to slice but will always be significantly less that the initial 10% H2O.

- Line 179 is a typical example of over-interpretation of the results, occurring too many times in the paper : “The frozen-in melt-enriched regions or segregations predominantly occupy conjugate shear bands (Fig. 1)”. First of all, the strain field has not been measured (eg. with DIC or DVC) so that I don’t understand how the author can decide whether a specific feature is a shear band or not. Second, the ellipse show ‘concentration of Mix-2’, not really aligned at 35° as indicated; this could be due to the sample preparation, or simply due to some random process, etc… this really needs to check and quantify further.

    - We strongly disagree with the above statement. The shear bands can be clearly identified and located during the segmentation process and this is based on the identification of the different mixes. If we had not undertaken the segmentation, then we would not have identified their location. There is no way they are due to preparation processes. In fact, areas that contain the shear bands will be progressively rotated as the sample is progressively shortened. This is why there are variations in angles as pointed out in the Discussion section of this manuscript and in modifications we have undertaken to Fig 10b-d.

- Legend fig 1, at point A and B it is said that there is an increase in porosity, but one do not see anything in the figure and no quantitative estimation is provided !

    - It is impossible to provide a quantative estimation on the 2D surface. However, with careful examination and our evaluation of all representative slices we stand by our statement. It was impossible to present all the data even as supplementary material, but are available from the AAD Data centre (a comment to this effect has been added to the acknowledgements).

- Line 185 ‘low pressure regions or non-deforming region’ : pressure and deformation field have not been measured, so how can the authors estimated that some regions do not deform, and that some are at lower pressure ? One even don’t know whether the porosity is open or closed (althrougth this might be accessible by tomography).
- This is an unfortunate statement. Anyone who has worked with unconfined samples during deformation recognises that the outer surface of the sample has to be a ‘low pressure region’. We have not inserted strain gauges in samples to measure localised stresses. Therefore, we stand by our text. Similarly, the greater majority of voids within the polycrystalline sample have to be closed.
- Line 203 : “This bimodal distribution into shear and compaction bands are all part of a connected network”. No proof for bimodal distribution, nor shear band / compaction band, nor proof of a connected porosity network in the data …
- The word ‘bimodal” has been removed.
- Line 205 “increasd porosity” but the porosity is not quantified…

    - We completely disagree with this comment. Quantification is clearly shown in Fig. 4.

- Line 223 “(fig 2d). the network consists of pores situated on grain boundaries” => grain structure (and thus grain boundaries) are not indicated in figure 2…
- We disagree with the referee here as they can be clearly identified in area highlighted as melt segregation.

- Line 231 why should quartz (not deforming by basal glide) should be a good analog for ice (deforming mostly by basal glide) ?
- We are not elaborating here as this is clearly explained in Kronenberg et al. (2020) but we have added in text "references therein" as this paper references all the classic papers on this subject.
- Line 251 I find really strange that the stress increases as the temperature is increased up to +2°C, as part of the specimen melts and ice should become softer. What is the reason for this stress increase ?
- A sentence has been added to text to explain this, namely:
- "This modest increase in stress we attribute to an expansion of the aluminium piston as the temperature is increased."
- Line 252-255 "This stress drop we attribute to the softening of the ice with the onset of melting, grain boundary migration and initiation of the deformation bands. This ductile to shear transition can be explained by the competition between different time scales corresponding to the relatively slow melting of the H2O ice, and broken bonds as the HDO mixes were generated" => not clear at all. We agree with the first part of this comment and feel there is no need to elaborate.

  Has gbm been observed (not shown in the figures) ? Grain boundary migration is definitely observed and shown in Figures 7 and 8. In addition on lines 274-275 in the original manuscript, the grain size from ~0.5 to 3-5 mm is described.

- initiation of deformation bands => strain localization into deformation bands starts at the very beginning of the deformation (before 1% strain), see the paper of Grennerat et al. Acta Mater 2012. What is a 'ductile to shear transition' ? shear deformation is not in the ductile regime ? 'different time scales' => could you explain what is meant here ? 'broken bonds' => do you mean atomic bonds ?? any evidences ?

  - Unfortunately, we do not have evidence to support the reviewer's statement that shear bands initiate at 1% strain. We definitely disagree with the referee here – The shear and compression bands observed in our experiments are definitely ductile features. We have added the word "atomic" to the text of the manuscript.

- Line 256-260 : in fig 6, one do not see as written in the text, for LDH-20, a transition from hardening to weakening within the -7°C regime. And one do not see, for LDH-35, a decrease of the flow stress at +2°C (even modest)
- Thanks for comment. We have added "after" to clarify this. In addition, we have added a sentence to explain why there was a subtle increase in stress.
- I really don't understand paragraph 278-284 and what are the supporting informations. One do not see 'white lines' in fig 8c. What do you mean with 'refraction of shear bands' (line 280) ??

  - Thank for the comment "the white lines" should have in fact read "black lines" and has been changed in the caption. The word "refraction' is quite clear in its meaning that there is a change in direction, e.g. as a light ray passes through an object or as a refracted cleavage in rocks.

- Line 287 what is meant with ‘evaluated through statistical analysis of particular angular positions and hkl reflections’ ?? I guess this is related with the neutron diffraction experiment, but this is really not clear
- In the original text, a reference was made to Wilson et al. (2019) which describes the novel method used. This is based on acquiring crystallographic data during the in situ deformation experiments As pointed out in the methods section (line 103) this technique was described in Wilson et al. (2019, 2020). To overcome the referee’s concern the sentence has been modified to:
- “….was evaluated through statistical analysis of particular angular positions and *hkl* crystallographic reflections collected during the in situ deformation (Wilson et al., 2019; 2020).
- Line 294 : one do see, as stated in the text, ‘a strain dependent increase in grain nucleation up to 14.6% strain’ for both DH29, LDH20, and DHC06 (grain size clearly decreases before this strain level). Same for ‘a variable but decreasing number of grains in the final deformation stage (15.4 – 20% strain)’, not observed for dhc06, dh29, dhc23…
- Yes, we agree with this statement. However, to overcome the referees concern we have added “…of new recrystallised grains”
- Line 310 ‘as these experiments have shown, the movement of meltwater’ As far I understand, the meltwater movement has not been observed here. Only few static tomographic images have been acquired

  We thank the referee for this comment. The wording here has been modified to:

  - “Moreover, as these experiments have shown, the diffusion of hydrogen, which is a tracer for the location of meltwater, through conjugate shear zone formation or basal compaction, contributes significantly to overall meltwater transport.”

- Line 315 ‘shear induced failure mode’ => do you have observed any cracks in the specimen (not shown/discussed in the figures) ? why invoking suddenly failure modes ?

  - There are definitely no cracks in any of the samples and this is why it is not mentioned in any figure. The deformation is purely plastic and confined to the ductile and recrystallised deformation bands as illustrated in Figures 7d. and 8. We could have included numerous other e.g. of such microstructures, but because of brevity we have restricted the number of images. Any material scientist would acknowledge these deformation bands represent one form of a failure mode in a deforming material. Our wording here is completely appropriate.

- I find really weird that the word “recrystallization” does not appear even once in such a paper dealing with microstructure evolution at the melting temperature…

  - In many places in the text we describe grain nucleation, grain boundary migration and diffusion processes which create the mixed grains. These all contribute to the vague term recrystallisation and its connotations. Because of the resolution of the tomographic images we are definitely not getting into the debate on whether the microstructure is evolving via sub-grain nucleation vs specific new grain nucleation. For the referees benefit we have added the word recrystallisation in a couple of places in the revised text.

- Etc…