

06 Oct 2023

Editor decision: Publish subject to revisions (further review by editor and referees)

by [Kaitlin Keegan](#)

Public justification (visible to the public if the article is accepted and published):

Thank you for your updated manuscript. The additional descriptions of the methods and the addition of Table 2 indeed help to address some of the main points raised by Referee #2. Please find my additional comments below, which aim to improve the readability of the manuscript and further address the comments raised by the referees.

All editorial suggestions have received attention. Additional figures have been added, namely Fig. 2, which illustrates how the slices were generated. Supplementary Figures 4, 5, and 6 with appropriate wording in text have been added to section 3.2. An additional three references have been added with appropriate text.

General comments:

- Are there images of undeformed sample DHC-23 to put in Supplementary Figure 2? If so, including them in this figure would help to compare the results to the DHC-06 samples and would show if similar results for the location of melt-enriched regions were found. That would certainly strengthen the statements about these melt-enriched regions rotating towards the XY-plane. It appears the editor is referring to Figure 3 and not Figure 2. As shown in Table 1 we did not undertake segmentation of the tomography data from the undeformed sample DHC-23. Therefore, the undeformed segmentation was not included in Figure 3b.

- More generally, the manuscript would benefit from a discussion of how representative these results are given that only five deformed samples are used in the analyses, with only two sets of replicate samples (it is never directly mentioned if DHC-06/DHC-23 and LDH-20/LDH35 can be considered replicate experiments). This is likely due to difficulty in generating samples, deforming them, and measurement techniques. Still, a brief explanation would be helpful. At the end of the sentence on line 79 there is the wording, experiments are "hard to perform".

In this manuscript we are definitely not going to provide an explanation for the enormous technical difficulties experienced. They first had to be overcome during sample preparation. Problems then surfaced during our allocated two weeks of beam time to undertake the deformation experiments and tomography. During this time, we were locked out of ANSTO for four days, because of a bushfire in the neighbourhood of the nuclear reactor, and had to control experiments remotely. However, we were compensated with an additional week of beam time on both the Kowari and Dingo beam lines. The total number of 3D experiments started were greater than ten, with different elements of success. This included samples melting, failure during loading of samples, and with excessive meltwater in compaction band regions resulting in failures prior to unloading. All the partially successful experimental data supports the results described in this paper, as does a set of complimentary 2D experiments (to be published elsewhere). It was decided not to include incomplete mechanical data and tomography results from failed or unused samples as data needed to be linked to successful experiments. Only the five most successful 3D experiments are described, together with results from previous pure D₂O experiments.

- A description of why some samples are included in Table 1 but are not mentioned in the results or discussion sections would be helpful. Otherwise, consider removing the

extraneous samples. Additionally, an explanation for why so many samples do not have values for Mean or Maximum Coordination Numbers would be beneficial in the Methods or Results section. All samples discussed in the paper are summarized in a modified Table 1. Only DC-01 has been removed, as it had appeared in an earlier version of Supplementary Figure 2. An addition to the caption identifies why coordination numbers don't exist in some samples.

- A clearer description of what Mix-1, Mix-2, and Mix-3 represent in the images presented in Figures 1-3 and Supplementary Figure 2 would be really helpful for the reader when considering the results, and ensuing discussion. A modification has been made to legend and captions in Figures 1-3. This does not apply to the non-segmented Figure 2.
- I respectfully disagree with the authors' comment to Referee #2 about Figure 2D, where the authors claim that it's clear to see the pores situated on grain boundaries. This is likely due to a difference in familiarity with these tomographs between the authors and general readers. I agree with the referees that it is hard to determine pores and grains in the figures presented. If the image resolution is not sufficient for including lines demarcating grains/pores, consider including a description of how the reader should interpret the structure from the colors in Figure 2d. Modification have been made to caption of old Figure 2 pointing our that pores are attached to the Mix-2 phase.

Technical corrections:

L45-46: '...occur as viscous forces that dominate over capillary forces...' **Changed**
L56: commas needed before 'which' and after the parenthesis **Changed**
L62: should be 'suggests' here **Changed**
L63: do you mean 'attributing'? The phrase 'attributed to meltwater segregations' seems out of place here **Changed**
L72: should be 'enhances' **Changed**
L92: you use 'mold' in Supplementary Fig. 1, so be consistent in the text with 'mold' instead of 'mould' (or vice versa) **Changed to proper English usage 'mould'**
L131: consider breaking this run-on sentence up to something like: '...and CPOs analysed (Hunter et al., 2022). The final microstructures...' **Changed**
L132: remove the 'and' **Changed**
L133: remove the extra '(' in the citation **Changed**
L145-146: this sentence is confusing. Perhaps: 'It was experimentally determined that the neutron beam spectrum has a Maxwellian distribution with a peak at approximately 1.5 Å.' **Changed**
L147: '...is about 2.4 cm-1 for H2O, 0.35 cm-1 for D2O, and intermediate values for mixtures of HDO.' **Changed**
L157: '...are shown in Supplementary Fig. 2c,f in red, while the yellow...' **Changed**
L168: '...and different concentrations of hydrogen in the D2O, which are henceforth referred to as...' **Changed and also added DHO in following sentence**
L171: '...to cm-scale, correlating it...' **Changed**
L177: 'The locations of former meltwaters are identified...' **Changed**
L178: '...over a sample. This distribution suggests...' **Changed**
L179: comma needed after 'sample' **Changed**
L188: 'shows' **Changed**
L196: 'Figs. 1b-c' **Changed**
Lines 196-197- it's unclear where the reader can see evidence of this result and is an example of where a quantified result could be reported (how much larger are the pores

between the deformed and undeformed slices?). A reference has been made to Fig. 4.

L201: please report that 'discrete decrease in number' **Wording changed here.**

L211-212: I'm not sure what the authors are trying to say in this sentence. Please reword to clarify. **Two sentences have been reworded here.**

L214: should be 'shows' **Changed**

L226-227: where are the images of the thin sections of the undeformed samples to support his statement? **This is supported by the data in Figure 4. Thin section data is not necessary to support this statement.**

L233: 'This suggests that the pores have...' **Changed**

L250: do you mean Supplementary Figure 4 here? **Changed to 7, as three other supplementary Figs added.**

L252: references here should be to Figure 5, not Figure 6 **because a new Fig. 2 has been added, this is now correct. Subsequent Fig. numbers changed in revised text.**

L259: comma needed after 'curves' **Changed**

L277-278: need a comma or connecting word in phrase 'are free of undulose extinction have diffuse low-angle boundaries' **Changed**

L283: do you mean 'red areas' here? I don't see specific red lines in Fig. 8c. Additionally, it would be helpful to define what green and red represent in the figure caption. **Wording modified in both the text and figure caption to point out they are the fine red and yellow lines on green background.**

L290: I don't see any white lines in Fig. 8c as mentioned here **They were originally white but had been changed to black during a previous revision of manuscript. Now rectified.**

L361: need parentheses after '2004' **Changed**

L366: change 'and' to 'where' **Changed**

L380: remove comma after 'deformed' **Changed**

L385: change 'and' to a comma **Changed**

L397: should be 'break down' here **Changed**

L399: do you mean 'dominant' here? **Changed**

L401: 'The change to weaker CPOs...' **Changed**

L407-408: this is a sentence fragment. Combine with the previous sentence. **Changed**

L414-415: this is a sentence fragment. Combine with the previous sentence. **Changed**

L420: change semicolon to comma **Changed**

L447-448: commas need: '...instabilities, namely...and compaction bands, emerge...' **Changed**

L453: change 'is' to 'are' **Changed**

Figure 1 caption: Explain in the caption how (b) and (c) differ as they're showing the same sample after deformation but with different color distributions; L628- '...sample, where ellipses outline the...'; L629: should be concentrations here; L632: '...X478, where ellipses show the distribution of... the compression direction. Water can be seen concentrated on the margin of the sample.' **Changes made to caption**

Figure 2 caption: L642: should be 'black circle' **Changed. In addition the caption has been expanded to describe distribution of pores.**

Figure 4: why are the lines in the legends for (d) and (e) not straight? Does that indicate that the trends depicted in Figure 4d,e plots are more 'wiggly' than they should be?

What an inappropriate comment. It is obvious the lines are not straight.

Figure 5: it appears that the image for panel (a) is missing; Panels (b) and (c) are described as the 'top' and 'bottom' halves of the LDH-20 deformed sample- which way has the sample been halved? Is the top ♦ bottom of each half going left to right in the

image, or top to bottom? **There is no image missing it is an explanation.**

Caption: L674- remove 'e' before 'Fabric' **Done**

Figure 7: is there a legend for the colors in the polarized images in panels (b) and (c)? **Definitely not required, they represent normal cross polarized birefringence colours and you never provide a legend for these as they depend on grain orientation with respect to polarizers.**

Figure 8: In panel (a), it appears that the total strain value is missing for the DH-layer ('=' %') **25% added**

Caption: L694: should be '...of DHC-23. The finer-grained...'; what do the colors represent in panels (b) and (c)? In panel (c), how are the orientations of the shear bands (black lines) determined? Including some text in caption or text would be helpful for the reader to better understand how these were determined. **Changes made to caption. No explanation is necessary as they are based on 2D observations of the microstructures in thin sections from sample.**

Table 1: Should the row for 'LDH-35def' be unshaded (part of the Deformed samples) here? **Changed**

Table 2: Put '%' in parentheses in the column titles to indicate that those are the units, then eliminate the extra '%'s in some of the data cells of the table. **Changed** Caption: I don't understand the phrase '..., and representing the former liquid phase during the 2C part of the deformation.' here. Consider removing. Also, shouldn't this be volume fraction if all values are reported as percentages? **It has been changed not removed.**

*The text describes Mix-1, Mix-2, and Mix-3 as gradations of HDO, but Figures 1, 2, 3, and Supplementary Figure 3 describe them as 'DHO' in the legend. The references to them in the text and figure legends should agree. **Figs 1-3 have been changed to reflect this and in a couple of places in the text.**

* It'd be helpful to indicate somewhere the labeling scheme for the slice numbers for the tomographic images. For example, in Figure 2, are the slices shown in (d-f) roughly the top, bottom, and middle, given their slice names? **A sentence has been added at line 179 together with a new Fig. 2 illustrating the nature of a slice.**