

Dear Dr Craw,

We appreciate your helpful comments. Here we present our responses to the comments. Our responses are in black, while your comments are in blue. We will make necessary revisions to address the questions.

Response to general comments

Generally, I think it's important to acknowledge in the manuscript that these results concern a specific scenario (relatively high concentrations of two soluble impurities), and can't be easily generalised to describe the behaviour of all natural ice. So I would stop short of recommending an 'established impurity factor', as in lines 16 and 483. The results are still very valuable because they expose the relationships between grain size, temperature and concentration of these specific impurities.

Reply: Thank you very much for your suggestion, we will remove these statements in the abstract and conclusion sections. We will also add statements that underscore the limit of our experiments, i.e., high concentrations of two soluble impurities, that cannot accurately mirror natural conditions, i.e., low concentrations of a diverse range of soluble and insoluble impurities.

In a similar vein, it would be good to see some justification of why these particular ionic species and concentrations were chosen for the study. The concentrations are high compared to what is usually seen in ice sheets (Cuffey and Paterson [2010] give a range of $1e^{-7}$ to $1e^{-5}$ mol/L total dissolved impurity concentration in most polar ice sheets, with higher values closer to the bed). While overall it seems like concentration is not the most important factor, there must be a lower limit where there is no longer an effect on grain growth? There should be some more discussion of how these results might or might not scale to natural ice masses.

The same goes for the choice of KCl and MgSO₄. It's clear that different ions have different effects on grain size, so how might the results of this study translate to describing ice with both of these together, along with other impurities as well?

Reply: In response to your first question concerning the presence of a lower limit for the impact of impurity concentration on grain growth, we suggest that it can be evaluated in two distinct scenarios: those above the eutectic point and those below the eutectic point.

- (1) **Above the eutectic point.** In scenarios above the eutectic point, our dataset is relatively limited, because this study only includes data related to KCl-doped ice annealed at 268 K (-5°C). Results from our experiments indicate that, even at the minimum concentration of KCl utilized in this study, specifically at 1×10^{-5} mol/L, there remains a notable promotion of grain growth. It is crucial to acknowledge that these minimum concentrations utilized in our experiments are still higher than ice sheets, specifically at 1×10^{-7} to 1×10^{-5} mol/L (Cuffey and Paterson, 2010). Hence, in the revised manuscript, we will add statements to emphasize that results from our experiments do not fully represent natural

scenarios; caution is needed when extrapolating our experimental results to warmer glacial regions. The bed of glacier is characterized by relatively high temperatures and impurity content (close to melting point in some ice cores and total impurity concentration of $\sim 8 \times 10^{-4}$ mol/L, according to Cuffey and Paterson (2010)). Thus, the potential enhancement of grain growth due to chemical impurities could be considered. However, the height range of the glacier bed (Cuffey, 2000) is limited and is not the focus of glaciologists.

(2) **Below the eutectic point.** In this scenario, our experiments encompass two sets of KCl-doped ice, each maintained at least 2 K below the eutectic point, and three sets of MgSO₄-doped ice maintained at least 5 K below the eutectic point. For KCl, despite the rejection of ions by ice crystals, previous experimental observations and simulations have indicated that Cl⁻ and K⁺ can be incorporated into the crystal lattice with low concentrations under conditions below the eutectic point. It is only when the concentration surpasses the solubility threshold (approximately 2 to 3×10^{-5} mol/L, according to Yashima et al., (2021)) that hydrates precipitate at the grain boundaries, thereby impeding grain growth. This solubility value may be above the average content range of Earth's glaciers (1×10^{-7} - 1×10^{-5} mol/L), suggesting that the impact of chloride salts on grain growth might be small in cold glacial regions.

In this study, ice doped with MgSO₄ has a lowest concentration of 1×10^{-5} mol/L. At this concentration the grain growth was still impeded. The solubility of MgSO₄ in ice may be lower than this value. Currently, there is limited research on MgSO₄ solubility in ice. We assume it approximates the actual solubility, which may be lower than 2×10^{-6} mol/L (Iliescu and Baker, 2008). Additionally, the eutectic point of sulfate is generally high, placing glaciers below this temperature. In ice deposited during Pleistocene glacial climates with higher impurity concentrations (10^{-6} to 10^{-5} mol/l, according to Cuffey and Paterson, 2010), sulfate may moderately hinder grain growth. This conclusion appears to establish a connection between reduced ice core grain size and elevated concentrations of soluble impurities (sulfate and chloride ions) (e.g., Alley 1986, Langway 1988, Paterson 1991).

Next, we explore how the results of this study can be applied to describing ice containing other impurities. In this research, KCl was chosen to exemplify common alkali metal chloride, while MgSO₄ was selected to represent prevalent alkali metal sulfates, found in glaciers (Cuffey and Paterson, 2010). As mentioned in the Introduction, above the eutectic point, soluble impurities manifest as saturated solutions at grain boundaries, facilitating grain boundary migration. Consequently, when extrapolating our outcomes to the impact of other salts on ice grain growth, their respective eutectic points become a crucial consideration. In conditions below the eutectic point, soluble impurities exceeding the solubility range reside at grain boundaries in the form of hydrates, impeding grain boundary migration. Therefore, when extending our findings to ice grain growth influenced by other salts, reliance on the solubility of the salt in the ice lattice may be pertinent.

We will add the above discussion in Section 5.5: Implications for natural ice.

“Cl⁻ and SO₄²⁻ constitute two of the most prevalent anions in polar glaciers, while Na⁺, Mg²⁺, Ca²⁺, and K⁺ are the predominant alkali metal cations in Earth's glaciers (Obbard and Baker, 2007). In this investigation, we have chosen KCl and MgSO₄ as representatives of chloride salts (hereinafter specifically referred to as chloride salts of the above four ions) and sulfates (hereinafter specifically referred to as sulfates of the above four ions), respectively.

The impacts of KCl and MgSO₄ on ice grain growth can be attributed to their eutectic points and solubility within the crystal lattice of ice. In extrapolating our results to the influence of chloride or sulfate on ice grain growth, it is imperative to consider these two aspects. We suggest that it can be evaluated in two scenarios: above the eutectic point and below the eutectic point.

(1) In scenarios above the eutectic point, our dataset is relatively limited, because this study only includes data related to KCl-doped ice annealed at 268 K (-5°C). Our results reveal a significant promotion of grain growth by soluble impurities at temperatures above the eutectic point. This phenomenon is also observed in NaCl-doped ice (De Achaval et al. 1987). Except for KCl, other chloride salts exhibit relatively low eutectic points (approximately 223 K to 252 K, according to Baumgartner (2009)), potentially nearing or even falling below the temperature of some glaciers (Cuffey et al., 2000). In other words, most glaciers have temperatures above the eutectic point of chloride salts. Nevertheless, the lowest concentration utilized in our experiments (1×10^{-5} mol/L) remains higher than the total impurity concentration in the ice sheet, estimated at about 1×10^{-7} to 1×10^{-5} mol/L (Cuffey and Paterson, 2010, pp.33-34). Applying our experimental results directly to glaciers with low impurity concentrations poses challenges. Consequently, extra caution should be paid when extrapolating our results to lower impurity concentrations in glaciers.

(2) At temperatures below the eutectic point, we hypothesize that salt hydrates will precipitate at grain boundaries, impeding grain growth only when the concentration surpasses the solubility in the crystal lattice of ice. Given that Cl⁻ ions can integrate into the interior of the ice crystal lattice, chloride salts exhibit slightly greater solubility in ice than sulfate salts. Specifically, the solubility of KCl is approximately 2 to 3×10^{-5} mol/L (Yashima et al., 2021), while NaCl's solubility ranges from 3×10^{-5} to 1×10^{-4} mol/L (Mongnant et al., 2001, and Gross, 1977). For Ca²⁺ and Mg²⁺ with smaller ionic radii, we assume that the solubility of their corresponding chlorine salts (CaCl₂ and MgCl₂) in the ice lattice is of the same order as KCl and NaCl, that is, at least greater than 1×10^{-5} mol/L. Thus, the solubility of common chloride salts may generally exceed the average content range of glaciers (1×10^{-7} to 1×10^{-5} mol/L). Therefore, the effect of chloride salts on grain growth in colder glaciers is minimal.

In contrast to KCl, grain growth remains impeded in the ice sample doped with MgSO₄ even at the lowest concentration (1×10^{-5} mol/L). We assume the solubilities of sulfates are similar to the solubility of sulfate ion, which may be lower than 2×10^{-6} mol/L (Iliescu and Baker, 2008). Additionally, the eutectic points of sulfates are generally high

(>268 K), placing glaciers below these temperatures. In ice deposited during Pleistocene glacial climates with higher impurity concentrations (10^{-6} to 10^{-5} mol/L, according to Cuffey and Paterson, 2010), sulfates may hinder grain growth. This conclusion could establish a connection between reduced grain size in ice cores and elevated concentrations of soluble impurities (sulfate and chloride ions) (e.g., Alley 1986, Langway 1988, Paterson 1991).”

In the Results, some grain size distributions are described as having a 'bimodal component' (lines 172, 185). It's not obvious to me exactly what this means. E.g., the 10-100hr annealed KCl sample in Figure 2(b) does have some higher frequencies of larger grains, but this second 'mode' isn't any larger than one seen in the smaller grain sizes of the starting material in Figure 2(d), which is described as normally distributed. The starting materials in general look to me to have a more skewed distribution, compared to the annealed samples which are more normally distributed. I don't think this is important to the conclusions, but I would recommend either quantifying the skewness and bimodality of the distributions, or removing these descriptions.

Reply: Upon a thorough review of our results, we found that the presence of a bimodal distribution is not apparent. Furthermore, we omitted any discussion of bimodal distributions in the subsequent sections. So, we have chosen to exclude these descriptions from our analysis. Now the sentences are changed to the following:

Line 157: *“The grain size distribution remains log-normal, and in the sample annealed for 100 hours, there is higher frequencies at larger grain sizes (500 to 1000 μ m).”*

Line 173: *“while a few samples (such as the sample with 10^{-5} mol/L KCl annealed for 10 to 100 hours, Figure 2(b)) exhibit higher frequencies at grain sizes larger than the peak.”*

Line 186: *“The grain size distribution remains lognormal after annealing. In the sample with 10^{-5} mol/L KCl annealed to 100 hours, there is a slightly lower peak frequency and the longer tail towards larger grain sizes compared to other samples.”*

Line 177: We decided to delete the following sentence.

“The increase in σ_d/d usually suggests some grains grow faster than others, resulting a bimodal distribution in grain size.”

Response to Specific comments

line 75: ‘Overestimation of the value of n can occur if grain size distribution not in steady state...’: are the grain size distributions in this study in steady-state? Could that bias the n value estimates?

Reply: The statement came from the citation Breithaupt et al., (2021). They assess whether the distribution of grain size remains steady. The grain size distribution at the steady state follows a log-normal pattern. After normalization, the grain size distributions at various annealing times exhibit similarity, that is, they have the same or

similar normalized variance. The steady-state distribution appears independent of the initial grain size distribution and eventually converges to a distinctive normalized pattern. As such, we consider that our grain size distribution is in a steady state.

Not all of our grain-size distributions display characteristics of a steady-state distribution. However, upon a thorough review of Breithaupt et al., (2021), I did not find explicit information linking the steady-state distribution to the value of n . Therefore, we removed the description of the relationship between the steady-state distribution and the value of n .

line 6: For glaciologists with poor mental maths (like me), it would be good to include what these K values correspond to in °C.

Reply: We have modified the descriptions in the Section Abstract and Methods (lines 6 and lines 100).

Line 6: *“Samples were annealed for a maximum of 100 h at a hydrostatic confining pressure of 20 MPa (corresponding to a depth of about 2 km) and different constant temperatures of 268, 263, 258 and 253 K (corresponding to -5, -10, -15 and -20°C, respectively).”*

Line 100: *“Annealing was conducted at a hydrostatic pressure of 20 MPa, corresponding to the pressure at the base of a 2-km glacier, and temperatures of 268, 263, 258 and 253 K (corresponding to -5, -10, -15 and -20°C, respectively), to a maximum duration of 100 h.”*

line 25: ‘Demonstrate weaker mechanical properties’ seems like a roundabout way of saying ‘are mechanically weaker’.

Reply: we have revised the statement accordingly. (lines 25)

“In the grain-size sensitive deformation regime, ice samples with smaller grain sizes are mechanically weaker, resulting in reduced stresses under constant strain rates or increased strain rates under constant stresses (Goldsby and Kohlstedt, 2001).”

line 33: ‘concentration’ is probably more accurate than ‘content’.

Reply: We have replaced 'content' with 'concentration' in the text (lines 33).

“However, some other studies reported that a high concentration of soluble impurities inferred from electrical conductivity measurements does not necessarily result in a slowdown of grain growth (Durand et al., 2006).”

line 76: ‘transition in grain boundary structure’: it’s not clear what this means exactly.

Reply: In the review by Cantwell et al., (2014), facet coarsening, changes in orientation, changes in grain boundary thickness, and amorphous phase grain boundaries are all classified as complexion transitions in grain boundaries. These transformations significantly impact the rate of grain boundary migration. However, due to the absence of TEM evidence in our experiments, an accurate assessment of our grain boundary structure is challenging. Moreover, as this information is not clear linked to the

subsequent results and discussions, and considering its potential to mislead readers, we have decided to delete the relevant content.

line 83: What is the 'prepared solution', is it the solution from the previous sentence?

Reply: We have revised the statement (lines 83).

“Second, the prepared KCl (or MgSO₄) solution was added to the container of a medical ultrasonic nebulizer, which generated a fine mist of droplets with a diameter less than 50 μm.”

line 100: Briefly mention which time periods were chosen up to 100 hrs (i.e. 3.2hrs, 10 hrs, 32 hrs) and why (to capture rapid change in grain size early in the annealing process?)

Reply: We have modified the descriptions in the Section Methods (lines 100).

“Furthermore, annealing experiments were conducted with durations of 3.2 hours, 10 hours, and 32 hours to capture the rapid changes in microstructure and grain size in the early stage of annealing.”

line 147-149: The starting material grain size distribution in (c) of each of those figures looks shorter and broader than the others. Are they all cut from the same starting sample? I doubt it would make much difference, but it's worth making a brief comment on how much variation there is between the starting material samples.

Reply: We did not conduct repeated observations on the 0-h sample, i.e., annealing experiments at different temperatures use the same 0-h samples. In panel (c), the 0-h sample corresponds to the one doped with 10⁻² mol/L MgSO₄. The higher proportion of small-sized grains in this sample gives its initial grain size distribution a broader appearance compared to the distributions of the other samples 0-h samples. The larger molecular mass of MgSO₄ (120), compared to KCl (molecular mass 74.5), contribute to this difference. Vetráková et al., (2019) observed that higher-concentration solution result in smaller average ice particle sizes when frozen in liquid nitrogen. Therefore, we speculate that the ice particles formed in liquid nitrogen by doping a 10⁻² mol/L MgSO₄ solution are generally smaller, leading to a broader and wider particle size distribution towards the finer sizes.

As described in our results, the grain size distribution of samples which doped with 10⁻² mol/L MgSO₄ (corresponding to Figure 2 (c) to Figure 5 (c)) follow log-normal distribution. And after normalization, the grain size distributions at different annealing times exhibit similarities, indicating a steady-state distribution. The steady-state distribution appears to be independent of the initial grain size distribution and eventually converges to a distinct normalized pattern. Consequently, we consider that the initial grain-size distribution does not have a significant impact on the grain size distribution of the annealed sample.

line 178: 'Given an annealing time...' does this refer to any length of time, or a specific one?

Reply: We have modified the descriptions in the text.

Line 178: *“At a given time, grain sizes in all KCl-doped ice are coarser than those in pure ice, as illustrated in Figure 6(a).”*

Line 189: *“At any given time during the 100-h annealing, grain sizes of ice samples doped with different concentrations of KCl are similar to each other and also similar to the grain size in pure ice, as illustrated in Figure 6(b).”*

Line 196: *“At any given time, grain sizes in all KCl-doped ice are slightly finer than those in pure ice, as illustrated in Figure 6(c).”*

Line 206: *“At a given time, grain sizes in ice doped with higher concentrations (10^{-2} and 10^{-3} mol/L) are significantly finer than those in ice doped with lower concentrations (10^{-4} and 10^{-5} mol/L), as illustrated in Figure 6(c).”*

Line 223: *“At a given time, grain sizes in $MgSO_4$ -doped ice are always finer than those in pure ice, as illustrated in Figure 7(b), (c) & (d).”*

line 195: ‘the growth of grains displays self-similarity’; does this just mean they are similar to one another?

Reply: Based on Faul and Scott (2006), ‘self-similarity’ entails ‘grain size distribution normalized by the mean grain size is time invariant’. We apologize for the misleading writing; we have revised related description in the text.

Lines 130: *“The normalized standard deviation (σ_d/d) can be utilized to evaluate the self-similarity of the grain size distribution, i.e., the grain size distributions are similar to each other (Faul and Scott, 2006).”*

lines 195: *“The grain size distributions among the samples are similar to each other, suggested by the values of σ_d/d , keeping in the range between 0.4 and 0.6 for all samples.”*

Lines 204: *“The growth of grains are similar to each other, as the values of (σ_d/d) vary within the range between 0.4 and 0.6 for all samples.”*

Lines 221: *“The growth of grains are similar to each other, as the values of (σ_d/d) vary within the range between 0.4 and 0.55 for all samples.”*

line 223: Isn’t this contradictory? Or there is a difference in grain size between different concentrations, but it’s not statistically significant?

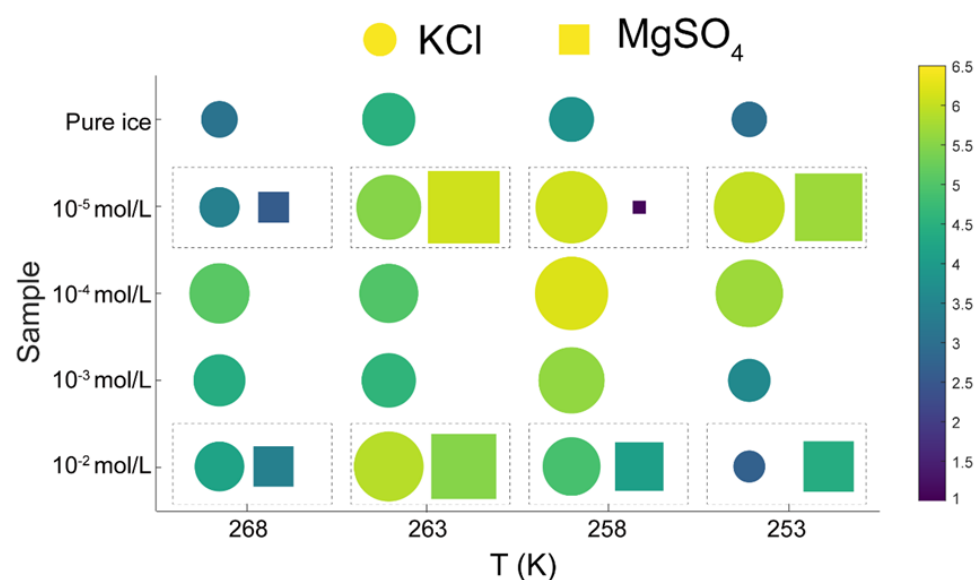
Reply: To prevent any potential confusion among readers, we have revised the sentence (lines 223).

“At 263 K and 253 K, samples with high impurity concentration (10^{-2} mol/L) exhibited slightly smaller grain sizes than the low-concentration sample (10^{-5} mol/L). Overall, the diversity in grain size between different concentrations is not evident.”

Figure 8: This is a great visualisation. The square symbols are a little hard to interpret, it would help to have the line width the same between all of them (rather than scale the

line width with the size of the square). It might even be better to have a separate panel for the MgSO_4 values, so that they can be viewed side-by-side.

Reply: We apologize for the unclear illustration. We have updated Figure 8 to include separate value panels for KCl and MgSO_4 , respectively. The updated Figure 8 and caption is presented below.



“Figure 8. Plots of grain size exponent, n , versus temperature and sample type. Round symbols are KCl-doped ice, and square symbols are MgSO_4 -doped ice. The symbols within the dashed rectangle represent ice doped with the same concentration and annealed at the same temperature. Symbols are colored with respect to the value of n . Larger size and warmer color represent a larger value of n .”

lines 232-233: ‘could be deemed as’; I think you can just say that it is relatively independent, and therefore you have decided to treat the average value as representative of all of the samples going forward.

Reply: We have modified the descriptions in the text (lines 232).

“The value of n is relatively independent of both temperature and concentration. Therefore, we will treat the average value of n as representative for all samples.”

line 384: Hammonds and Baker [2016] also found the same with Ca^{2+} -doped ice... maybe worth mentioning.

Reply: Hammonds and Baker (2016) conducted an analysis of grain size following deformation tests with CaSO_4 -doped ice. They observed that, in compressive deformation experiments with the lowest applied strain rate of $1 \times 10^{-6} \text{ s}^{-1}$, the average grain size of CaSO_4 -doped samples was larger than that of pure water ice. While no difference in grain size was noted between compression and tensile tests at higher deformation rates, intergranular cracks were evident in these samples. Similarly, Hammonds and Baker (2018) observed, in the deformation test with H_2SO_4 -doped ice,

that several groups of samples developed intergranular microcracks, with no discernible difference in grain size between doped ice and pure water ice. Notably, in samples without intergranular microcracks, the grain size of doped ice was significantly larger than that of pure ice. As a result, we cautiously speculate that the presence of intergranular microcracks observed by Hammonds and Baker (2016) might impede the migration of grain boundaries rather than being promoted by doped impurities. Consequently, we suggest comparing Hammonds & Baker (2016) with our study may mislead readers.

line 472: There was one case with $n = 1.1$ (10^{-5} mol/L MgSO_4 annealed at 258 K) . Was this discarded as an outlier?

Reply: The observed value of $n = 1.1$ is lower than the theoretical value of $n = 2$, indicating an outlier in our data. We speculate that this lower n value may be attributed to a slow grain growth rate, resulting in difficulties in fitting. We added a sentence in Section 5.1 to address this.

“Fitting of data from this sample suggests $n = 1.1$, which is lower than the theoretical value of 2. We think this low value of n may be attributed to the slow growth in the first 10 h. Because experiments are not perfect, such variations in the fitted values of n were also reported in previous studies (Azuma et al., 2012).”

Appendix B: It would be useful to have the full grain size datasets also included in the figshare repository, so that others don't have to repeat the Cellpose and ImageJ analysis to use the data.

Reply: Thank you for bringing this up. We have uploaded the full grain size datasets to the Figshare repository.

Response to Typos

Thank you very much for identifying grammatical errors and spelling issues. We have addressed each of them individually in the revised text (as indicated below).

line 25: In the grain-size sensitive...

“In grain-size sensitive deformation regime, ice samples with smaller grain sizes demonstrate weaker mechanical properties, resulting in reduced stresses under constant strain rates or increased strain rates under constant stresses (Goldsby and Kohlstedt, 2001).”

line 41: Replace the semicolon with a full stop

“For instance, sulfates may form hydrates at grain boundaries and within grains (Ohno et al., 2005, 2006; Sakurai et al., 2011).”

line 47: Highly concentrated NaCl solutions

“De Achaval et al. (1987) conducted experiments on columnar ice samples made from highly concentrated NaCl solutions (10^{-4} to 10^{-2} mol/L).”

line 53: 'including' makes it sound as though there were other impurities as well. I'd go with 'soluble impurities (potassium chloride and magnesium sulfate)...

"In this contribution, we performed annealing experiments at different temperatures on polycrystalline ice with and without the presence of soluble impurities (potassium chloride and magnesium sulfate), and investigated the grain growth kinetics in these systems."

line 94: ice particles were pressed

"During this high-pressure pressing, a phase transition from amorphous ice to ice I_h occurred, and ice particles were pressed into a fully densified ice sample."

line 104: was done

"Temperature-calibration was done by measuring the temperatures inside the cold chamber (where the CIP was placed) and inside the antifreeze in the non-pressurized CIP (see Appendix A for details)."

line 110: within a few minutes

"In the freezer, samples were taken out from the press and the plastic bag, and then placed into a long-term storage dewar filled with liquid nitrogen within a few minutes."

line 121: missing full stop

"For each sample, the images were merged into a large mosaic image covering an area of at least 1.5×3.0 mm."

line 129: and the standard deviation?

"The average grain size, d , and its standard deviation, σ_d , were then determined as the arithmetic mean and its standard deviation of the equivalent diameters, respectively."

line 143: remove 'is'

Therefore, we conducted the entire fitting procedure again with fixed n from 2 to 6, corresponding to the distribution of results of n , and calculated the goodness of these fits represented by the coefficient of determination, R^2 .

line 167: microstructures and line 168: temperature

"Here we describe the microstructures, grain-size distributions (Figures 2 to 5), average grain sizes (Table 1) and grain-growth curves (Figure 6) of annealed samples with respect to temperature."

line 183: 'for' rather than 'to' 100 hours

"In samples annealed for 100 hours, grain sizes vary greatly, showing many coarse grains (often >600 μm) surrounded by finer grains (<200 μm)."

line 187: 'increase' is repeated

"For the two higher-concentration samples (10⁻² and 10⁻³ mol/L), values of σ_d/d

increase from <0.5 and stabilize at ~0.6 after a 10-hour annealing.”

line 212: microstructures

“Here we describe the microstructures, grain-size distributions (Figures 2 to 5), average grain sizes (Table 1) and grain-growth curves (Figure 7) of annealed samples with respect to temperatures.”

line 229: is illustrated

“The variation of n with temperature and impurity concentration is illustrated in Figure 8.”

line 256: remove ‘obviously’

“In most samples, the grain size increases accompanied by a deceleration in growth rate over time.”

line 391: separately

“Ice doped with KCl and MgSO₄ exhibit slightly different behaviors, which will be examined separately.”

line 428: impurities

“A factor for the effect of soluble impurities”

line 481: soluble impurities

“When the concentration of soluble impurities exceeds the limit that can be accommodated in the ice lattice, the effect of soluble impurities can be described by a factor, F , in the following equation...”

line 525: illustrated

“The ‘outlines’ data were then imported into FIJI (ImageJ) and overlaid on the input image for visualizing grain boundaries, as illustrated in Figure A2(b).”

line 539: to directly correlate

“Due to a higher temperature, increased ice solubility in ethanol modified the sample surface, making it difficult to directly correlate microstructures within the same area between Figure A3(a) and (b).”

Reference

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