Review

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Microstructural characterization of snow, firn and ice

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This paper provides an overview of techniques used to characterize the microstructure of snow, firn and ice. These range from traditional optical microscopy techniques such as examining thin sections between crossed polarizers to various electron-optical and X-ray techniques. Techniques that could have an impact on microstructural characterization of snow, firn and ice in the future are briefly outlined.

This article is part of the theme issue 'The physics and chemistry of ice: scaffolding across scales, from the viability of life to the formation of planets'.

1. Introduction

Although ice can adopt many different crystal structures, at the temperatures and pressures that exist on Earth it exists naturally only as Ice I_h . This is simple hexagonal with lattice parameters of $a = 0.45227$ nm and $c = 0.73671$ nm and belongs to the space group $P6_3/mmc$ (figure 1). This paper describes techniques that have been used for the microstructural characterization of four different naturally occurring morphological forms of ice, *viz.*, snow, firn (multi-year snow), freshwater ice and sea ice. These different forms of ice are of interest because their occurrence and extent are both affected by and affect climate change. They are also of interest from an engineering point of view for a number of reasons *viz.*; sea ice can damage structures in the ocean; bridges and other structures are built in the winter from freshwater ice; and an excessive amount of snow on a roof can produce damage, possibly catastrophically. Finally, since it is transparent, ice is sometimes a useful analogue for minerals and rocks.

There is some confusion in the literature over terminology regarding grains and orientation when discussing snow, firn and ice. Here, we will use the

Figure 1. The crystal structure of ice I_h , showing the hydrogen (black) and oxygen (white) atoms in relation to the hexagonal unit cell (dotted lines). The lattice parameters are $c = 0.73671$ nm and $a = 0.45227$ nm, and the space group is $P6_3/mmc$. Courtesy of Victor F. Petrenko.

standard Materials Science terminology: ‘grain’ is simply the name given to a crystal in an aggregate, and texture refers to the orientation of many grains in a polycrystal. The literature on the microstructure of firn is particularly confusing since ‘grain’ has often been used to describe the ‘feature size’, which may be composed of either one crystal or several individual crystals. Again, we use the Materials Science description in which grain only refers to a single crystal in firn.

It is worth noting that there have been previous reviews that covered various aspects of this review. The author has previously provided an overview of various techniques for observing dislocations in ice such as etch-pitting/replication, diffraction contrast imaging in a transmission electron microscope (TEM) and X-ray topography (XT) [1–3], while Blackford [4] discussed imaging using both an optical microscope (OM), a scanning electron microscope (SEM) and micro-computed X-ray tomograph (micro-CT) for studying sintering in ice. She also briefly reviewed the use of energy dispersive X-ray spectroscopy (EDS) and electron backscattered patterns (EBSPs) in an SEM to study ice: EDS uses the fluorescent characteristic X-rays emitted when inner shell electrons are knocked out of material by the impinging high-energy electron beam and can be used to produce a semi-quantitative analysis of the composition of the material, while EBSPs use Bragg diffraction of impinging high energy electrons to provide three-dimensional (3D) information on crystal orientation. Pielmeier & Schneebeli [5] provided a history of the methods (mainly optical) used to study snow, while Sazaki *et al.* [6] provided a brief summary of the use of optical interference techniques for examining steps on ice crystal surfaces. Wilen *et al.* [7] presented an overview of early automated fabric analyzers for determining the c -axes of grains in polycrystalline ice, while Wilson *et al.* [8] provided an overview of *in situ* OM observations, via the use of automated fabric analyzers, for understanding the deformation and annealing processes in ice. The latter also presented brief reviews of the use of EBSPs in an SEM, neutron diffraction and XT to study ice. Very recently, the author briefly reviewed the use of OM, SEM, TEM, micro-CT and XT for examining both sea ice and freshwater ice [9]. Finally, Petrenko [10] provided an overview of techniques for studying the quasi-liquid-like layer on the surface of ice, a subject that we will only briefly touch on in this review.

2. Snow

Characterizing snow is important for a number of reasons. First, the albedo or reflectance of snow depends on the shape and size of the snow crystals, which both evolve over time [11]. Knowing

the albedo of the Earth, which has an average of approximately 0.3, is fundamental for modelling Earth's climate, snow greatly influences the Earth's albedo with fresh snow having an albedo of around 0.9. Understanding the relationship between the mechanical properties and the structure of snow has also been of interest for many years [12–17].

Snowflakes evolve in three ways. First, the dendrite arms on snowflakes are broken off as the snow blows around or as the snow is compacted due to the weight of snow above it in a snowpack. Second, the morphology of the snowflakes becomes coarser in order to minimize their surface area and, hence, surface energy. Third, snow undergoes changes because it usually resides in a temperature gradient in a snowpack since the sun heats the surface during the day while during the cold night air cools the surface. In fact, snow often experiences diurnal alternating temperature gradients. In a temperature gradient, dry snow undergoes much more rapid microstructural changes than under isothermal conditions [18]. A temperature gradient in snow produces a water vapour pressure gradient. This produces a flux of water vapour from the warmer regions to the colder regions. This water vapour flow to colder regions can cause very large so-called hoar crystals to grow that are only weakly bonded to the surrounding snow crystals. The processes involved in dry snow metamorphism are of great interest for understanding the conditions that lead to avalanches since avalanches can originate in weak layers in the snowpack. Ice lenses, regions of solid ice, within a snowpack, formed on the surface due to either freezing rain or a melt event, are also significant causes of avalanches [19].

Several methods have been used to image snow. No one knows who first photographed snowflakes, but the most famous photographer is Wilson A. Bentley (1865–1931), who was afforded the moniker Snowflake Bentley. Bentley, who lived in Jericho, VT, first photographed a snowflake in 1885 using a large tabletop bellows camera attached to an OM. He developed the technique of capturing the snowflakes on a black-painted board and quickly transferred them to a slide, all of which was performed outdoors during the long Vermont winters. Bentley went on to capture more than 5000 images of snowflakes, of which over 2400 were published in the book 'Snow Crystals' co-authored with William J. Humphreys of the US Weather Bureau in 1931, the year of Bentley's death. Some of Bentley's images can be found in the Snowflake Bentley exhibit at the Jericho Historical Society in Jericho, VT, while Bentley's glass-plates used for recording the photomicrographs are archived in the 'Bentley Snow Crystal Collection' at the Buffalo Museum of Science. The websites of these two institutions show numerous examples of Bentley's snowflake, see [20,21].

Together with George H. Perkins of the University of Vermont, Bentley determined that no two snowflakes are identical. Snowflakes take many forms. As well as the dendritic forms typically depicted on Christmas cards, they can be plates, hollow columns, capped columns, prisms and many other shapes. Several classification schemes have been developed for snowflakes, one of which included 80 different groupings [22]. Temperature and humidity determine which structure forms.

Artificial snowflakes can also be made. Prof. Kenneth Libbrecht at the California Institute of Technology grows snow crystals and photographs them under different lighting conditions. His beautiful images have been published in several popular books and can be seen on his website <http://www.snowcrystals.com/>.

OM can be used both in reflection and transmission modes, depending on the specimen, and both methods can be used with polarized light. The problem with using OM to image snow crystals is the same issue with using an OM to image any material at high magnification, that is, the shallow depth of field (DOF): for a magnification of around 60 times the DOF is only around 0.4 μm ; and the DOF decreases as the magnification increases. This means that for an object with significant topography, the whole object cannot be in focus at the same time. One way to circumvent this problem would be to use a confocal scanning optical microscope (CSOM) [23,24], in which the out of focus regions are prevented from contributing to the image by an aperture, although to the author's knowledge this has not been done with snowflakes.

Another approach is to use an electron microscope. The first electron microscope observation of snow appears to have been done in 1972 by Urwin and Muguruma using a TEM [25]. They

Figure 2. Secondary electron image of snowflakes with water droplets frozen on, which are referred to as rime ice. Imaging was performed at -200°C using 1 keV electrons. Courtesy of Si Chen.

cooled snowflakes to -80°C in a holder and then inserted the specimen into the TEM where it was maintained at the same temperature. This low temperature enabled the specimen to persist for a reasonable time even in the high vacuum of the TEM. They obtained little useful information on the snowflake, but the authors showed that radiation damage from the electron beam rapidly produced cavities of 2–50 nm.

An SEM also provides both much higher resolution (less than 1 nm versus approx. 500 nm) and greater DOF than an OM. A downside to the use of the SEM, as with the TEM, is that it has to operate in a vacuum, which can lead to rapid sublimation of the water in the snowflakes. Another problem is that ice, having very low electrical conductivity, will rapidly charge under the scanning electron beam. One solution is to coat the snowflakes with a metal and use a cold stage to keep them cold enough to minimize sublimation [26,27]. Wolff and Reid in 1994 were the first to use an SEM to image snowflakes, which they coated with aluminium [26]. Another solution is to examine snowflakes at -200°C using 1 KeV electrons [28]. Ice sublimation is negligible at that temperature and the use of the low-voltage electron beam produces surface conduction that enables imaging of the snowflakes at high resolution without the need for a metal coating. Figure 2 shows a secondary electron image of snowflakes taken in an SEM, in which spherical rime ice droplets are present on the snowflakes. The rime ice arises from water droplets that freeze onto the snowflakes as they fall through the air. Image acquisition times for the SEM are typically a couple of minutes.

OM observations can be performed with the microscope located in a cold room where the humidity is very low and so frost formation on a cold sample is not generally a problem. However, SEMs and TEMs generally do not reside in cold rooms and, thus, an issue for observation of snow, firn or ice using these instruments is the transfer of the specimens from a cold room to the electron microscope. This has been undertaken in different ways. At Dartmouth College, a commercial cold stage and airlock is used. The specimen stage is not cooled in the airlock and, thus, specimens are cooled with liquid nitrogen under a nitrogen atmosphere and a polyethylene cap is placed over the specimen holder trapping dry nitrogen gas over the specimen in a cold room. Once in the airlock of the SEM the specimen is pumped down. When an adequate vacuum is achieved the specimen is turned upside down and the specimen cap falls off into the airlock,

Figure 3. (a) A nitrogen gas-filled glove box for transferring ice specimens to a scanning electron microscope (SEM), and (b) copper braid cooling system for the cold stage inside the SEM. The systems were developed by Prof. David Prior and co-workers for an SEM at the U. Otago, Dunedin, NZ.

and the specimen can be inserted into the SEM onto the pre-cooled cold stage. This cold stage is cooled via cold nitrogen-gas and has a heater so the temperature can be precisely controlled. This system works in the winter but not so well in the humid New England summers. A better alternative, developed by David Prior's group at the University of Otago, Dunedin, NZ is to build a glovebox around the SEM door (figure 3a). This set-up also uses a home-built cold stage, which is cooled by a copper braid (figure 3b), one end of which is connected to a liquid nitrogen-filled container. In this set-up, an airlock is used to introduce the specimen into the glovebox, which remains under a nitrogen atmosphere.

While optical microscopy and scanning electron microscopy are useful for imaging an individual snowflake or a small group of snowflakes, these techniques examine only surfaces; dynamic studies of snow metamorphism using these techniques are unlikely to accurately reflect the real behaviour of snowflakes within a snowpack. Thus, most recent research has used a micro-CT to study temperature-gradient snow metamorphism and the effects of an overburden on a

Figure 4. Three-dimensional micro-CT reconstructions of snow (a) immediately after collection when the ice fraction is only 12% and the surface to volume ratio was 56 mm^{-1} , and (b) the same snow after 1 year stored at -20°C when the ice fraction had increased to 35% and the surface to volume ratio was 11 mm^{-1} . In these reconstructions, the ice is shown rather than the pores, and an accelerating voltage of 40 keV was used. Courtesy of Si Chen.

snowpack. The technique was pioneered by Lundy and Adams in 1998 [29] at Montana State University for the examination of snow. Since then a micro-CT has been used in many studies of snow metamorphism [30–44]. The principle of the micro-CT is relatively straightforward: X-ray absorption images are collected at various angles as the specimen is rotated from 0 to 180° —typically 270 images are collected, which takes upwards of 15 min—and assembled in a computer to produce a 3D image. Accelerating voltages of 40–60 keV are typically used to produce the X-rays for imaging snow and firn. Figure 4 shows examples of micro-CT reconstructions of snow before and after it has been stored at -20°C for a year in order to simulate natural coarsening of snow, which is clearly evident. The storage also led to a significant increase in ice fraction and a reduction in surface-to-volume ratio.

A micro-CT was first used to study snow metamorphism *in situ* under isothermal conditions by Chen and Baker in 2010 [36]. While the resolution typically used in micro-CT imaging of snow (5–15 μm) is less than that of both an OM (approx. 500 nm) and an SEM (less than 1 nm), the ability to see individual snowflakes inside a snowpack and characterize the snow during *in situ* experiments more than makes up for this shortcoming. For example, at Dartmouth, a desktop micro-CT sits inside a cold room at -10°C and stages that can either impose a temperature gradient or apply a compressive load to the snow are used to understand the effects of these two parameters on the evolution of the snow microstructure.

As noted earlier, the evolution of individual snowflakes within a snowpack can also be followed as a function of time *in situ* in the micro-CT [36] (figure 5). The micro-CT can provide a large amount of information on the microstructure of a snowpack as it evolves either under a temperature gradient or isothermally *in situ* in the micro-CT including the specific surface area, density, the fraction of the volume that is ice, and the dimensions of the ice crystals and pores including frequency distribution plots [36] (figure 6). It is worth noting that Hagenmuller *et al.* [40] showed that the values of the specific surface area and density of snow can vary by up to 5% when different algorithms are used to analyze the micro-CT reconstructions.

Both Wang & Baker [41] and Krol & Lowe [39] compared four-dimensional *in situ* micro-CT observations of snow under a temperature gradient with modelling of the microstructural evolution of the snow. A micro-CT has been used to study *in situ* how the thermo-physical properties of snow containing an ice wedge change under a temperature gradient [42].

In situ compression tests have also been performed on snow in a micro-CT using a displacement-controlled loading system that measures the resulting load [37,38,43]. In some of these studies, the loading rate could be varied enabling the stress exponent to be determined

Figure 5. Micro-CT reconstructions of an individual snowflake from the middle of an isothermal snowpack during an *in situ* study in which the snow was held at -5°C for 70 days: (a) initial snowflake, and after (b) 10 days, (c) 20 days, (d) 30 days, (e) 48 days and (f) 70 days. The snowflake image was extracted from a reconstruction of the whole snowpack. The micro-CT was operated at an accelerating voltage of 40 keV. Adapted from [36].

[37,38]. More recently, Wiese & Schneebeli [43] developed a device that enables both a load *and* a temperature gradient to be imposed on a specimen while being observed in a micro-CT.

It is often necessary to use multiple techniques to fully characterize a material. Riche, Montagnat & Schneebeli [44] used both a micro-CT and optical methods to characterize the evolution of snow during snow metamorphism. The micro-CT provided the detailed microstructural features of the snow noted above, while the optical microscopy, using an automated ice fabric (texture) analyzer (see later), provided information on the *c*-axes of the crystals. Unfortunately, while providing useful information, this approach is not able to follow the same volume of snow as function of time since the optical microscopy technique is destructive.

In situ compression experiments have also been performed on snow while being imaged using synchrotron-based *diffraction-contrast* tomography (DCT), in which not only the size and shape but also the orientation of the grains can be observed (figure 7) [45]. The latter is a very powerful technique for understanding deformation of a material containing many crystals, and importantly is a non-destructive technique so that the same volume of snow can be imaged over time. DCT requires not only recording of the X-ray absorption images but also the collection of X-ray diffraction data for each absorption image (to determine crystal orientation), and the correlation of these two datasets.

Since every sample of snow includes a unique combination of snowflake forms, performing reproducible experiments on snow is problematic. One way to get at coarsening and sintering mechanisms in a reproducible fashion is to use artificial snowflakes or snow spheres. After performing such temperature gradient metamorphism experiments on snow spheres using a micro-CT, Chen and Baker subsequently examined the samples in an SEM, thus demonstrating

Figure 6. Parameters derived from an *in situ* micro-CT study of a snowpack held at -5°C for 70 days: (a) the solid ice fraction, which increases from 7.7% to 12.9%, (b) specific surface area, (c) structural thickness of both the ice matrix and the pores between them and (d) the frequency of different ice structure thickness values for four different times. Adapted from [36]. (Online version in colour.)

the ability to obtain 3D images during *in situ* experiments in the micro-CT and high-resolution images in the SEM on the same sample [46].

3. Firn

Firn in temperate regions can be defined as snow that has survived more than one melt season. In the Greenland and Antarctic ice sheets, seasonal melting only occurs over part of the ice sheet and the snow undergoes temperature gradient metamorphism and sintering to form a firn column that is 80–100 m deep, depending on the snow accumulation rate and temperature. Below that depth is ice containing unconnected air bubbles. The processes that turn the porous firn into the bubbly ice are of interest because it is by using the various gases trapped in the air bubbles that paleoclimate can be reconstructed.

The firn column can be divided into regions in which different processes are taking place. At the top of the firn column, the air in the pores between the ice crystals is exchanged with the atmosphere by convection and wind pumping—imagine blowing over the top of a bottle. This region, which is called the convective zone, is where younger air is incorporated into the ice. Thus, unlike a layer of the firn itself, the air in a pore is clearly not from a single year but has a spread of ages. As an aside, the age of the firn and the ice below it can be obtained by counting the annual winter and summer layers (where clearly visible) whereas the age of the bubbles can be

Figure 7. Synchrotron-based *diffraction-contrast* tomograph reconstruction of an approximately 10 mm high $\times$ 10 mm dia. cylinder of snow. Imaging took approximately 60 min. Not only the size and shape but also the orientation of the grains can be determined as indicated by the different colours and intensity: red, green, blue correspond to the azimuth angle ϕ of the c -axis of $\phi = 0, 120, 240^\circ$, while the intensity (from light to dark) corresponds to the c -axis zenith angle $\theta = 0-90^\circ$. The image reconstruction requires the simultaneous collection and correlation of absorption and diffraction data. Courtesy S. Rolland du Roscoat [45].

obtained by measuring trace atmospheric gases. Below the convective zone is the diffusive zone, in which diffusion within the firn occurs but communication with the atmosphere is no longer possible. Knowing the depth at which this transition occurs between these two zones is essential for determining the difference between the age of the ice and the age of the air. Below this zone is the non-diffusive zone or lock-in zone, in which only limited diffusion is possible. Once the pores are completely closed off, we have ice.

The earliest method used to characterize firn was optical imaging. For this, the firn is typically infiltrated with a supercooled liquid, frozen and then sectioned, as reviewed by Perla [47]. The resulting surface can be treated with a dye to enhance the contrast between the ice and the filler, or the ice could be sublimated away and the holes filled with a contrast agent [48,49], and then photographed. The liquid frequently used to infiltrate the firn is dimethyl phthalate, which freezes at -4°C . This technique has been used to study how firn structure changes with depth (figure 8). A significant advantage of this technique is that the infiltrating chemical holds shallow fragile shallow firn together, thus enabling its transport to remain intact. The technique has the downside that the liquid can't infiltrate very narrow channels and can't enter closed-off pores at all. However, these issues do not matter greatly since this is a low-resolution technique that typically uses a camera to record the microstructure.

Much higher resolution images can be obtained by sectioning firn samples and examining them in the SEM. A flat surface can be obtained by microtoming or scraping with a razor blade. While firn samples could be imaged at -200°C using 1 KeV electrons like snow, the disadvantage of this approach is that X-ray spectroscopy and electron diffraction information cannot be obtained with such a low energy probe. Thus, firn is imaged in the SEM at temperatures around -100°C typically using 15 KeV electrons. At -100°C some sublimation of the ice occurs

Figure 8. Optical photomicrographs of the pore spaces (black regions) in firn imaged by infiltrating with a liquid, e.g. dimethyl phthalate, and then sublimating away the firn. Courtesy of M. R. Albert.

and this dissipates the charge build-up from the higher energy electrons. (Snow cannot be easily imaged at this higher temperature since the much higher specific surface area of snow means that it sublimates very rapidly.) The use of higher energy electrons means that not only can the firn be imaged but also that energy dispersive X-ray spectroscopy (EDS) can be used to identify dust particles and some soluble impurities and their spatial locations, and the 3D orientations of the ice crystals can be determined using EBSPs. SEM observations of firn were undertaken by Baker *et al.* on firn from different depths acquired during the 1999 US Trans-Antarctic Scientific Expedition, see, for example (figure 9) [50]. The SEM approach provides high-resolution imaging, orientation information and microchemical information. However, again, it is difficult to get a 3D picture of the structure from an image of a surface. One feature that the SEM imaging does provide is the clear differentiation of grain boundaries and, hence, it clearly differentiates one grain or crystal from another (figure 9).

Three-dimensional characterization of firn microstructure is best undertaken, as for snow, with a micro-CT. The pores in firn are from a few to tens of microns in size and, thus, the relatively low resolution of the micro-CT (5–15 μm) is not a problem. As an aside, a nano CT can provide a resolution as low as 50 nm, but at the expense of a very small specimen size (a few tens of microns), which is not useful for examining snow or firn. Even with a micro-CT, the limited sample size in some commercial systems (20–40 mm) can be an issue when examining near-surface firn due to the very large pores, which are not well captured. The first application of micro-CT to firn was by Freitag *et al.* in 2004 [51]. As with snow, numerous parameters can be extracted from the micro-CT data, and 3D models can be reconstructed of the ice or the pores. Two-dimensional sections from the micro-CT reconstructions are also very useful for visualizing the changes in structure with depth (figure 10) [52].

A concern with the micro-CT for producing reconstructions from the X-ray data is properly setting threshold parameters for the reconstructions. One solution is to obtain a micro-CT image and then obtain a secondary electron image in the SEM from the same specimen. The SEM image can be binarized and the threshold parameters for the micro-CT reconstruction from the same layer adjusted until it matches the SEM image (figure 11) [53]. The combination of the non-destructive micro-CT imaging followed by SEM imaging means that a 3D view of the pore structure can be obtained while the use of EBSPs in the SEM provides the orientation of the crystals, a combination that has been demonstrated by Lomonaco, Baker and Chen but little used at this point [53].

Figure 9. Secondary electron image of firn from a depth of 9.71 m taken from the US ITASE. The flat areas arise because the firn was shaved to be flat before imaging. GB, grain boundary; D, debris from specimen preparation. The grain boundaries can be observed due to the grooves that typically form there. The image was acquired using 15 keV electrons while being held at -100°C . Adapted from [50].

Figure 10. Vertical cross-sections from micro-CT reconstructions from Summit, Greenland at the depths shown. The white areas are ice and the black areas are pores. The micro-CT was operated at an accelerating voltage of 40 keV. Each square is from a region $8\text{ mm} \times 8\text{ mm}$. Adapted from [52].

4. Ice

Interest in characterizing the microstructure of ice, as with many materials, is to relate its microstructure to its mechanical properties and to its microstructural evolution. An unusual feature of ice compared to many materials is that it is always at a large fraction of its melting point. This means that grain growth in ice is rapid and grain sizes are large compared to many materials, and are typically greater than 1 mm.

Figure 11. (a) Binarized secondary electron image from firn, and (b) micro-CT image from the same layer in the firn. The secondary electron image can be used to set threshold parameters for the micro-CT reconstruction. The micro-CT was operated at an accelerating voltage of 40 keV, while the SEM was operated at an accelerating voltage of 15 keV with the specimen held at -100°C . Adapted from [53].

We can subdivide ice into two forms: freshwater ice and sea ice.

Freshwater ice forms through a number of processes, i.e. by compaction of snow, by freezing the water in rivers or lakes, by accretion onto the bottom of ice sheets that overlie subglacial lakes, by freezing water droplets in the air to form ice pellets or hail, and by the accretion of water droplets in fog that freeze onto cold surfaces to form rime ice. Ice is unusual, although not unique, among materials in that at its melting point its density (917 kg m^{-3}) is less than at its liquid form (1000 kg m^{-3}), which is why it floats. Microstructural characterization of freshwater ice has mostly focused on polar ice and laboratory-grown ice. The aim is generally to relate the grain size and grain orientation, soluble impurities, particles and dislocation density to the flow and microstructural evolution in ice sheets. The development of a physics-based constitutive model would enable the flow of ice sheets to be modelled on a fundamental physics-based level rather than on the semi-empirical basis that is now performed. Two key features of the behaviour of freshwater ice under load are that the critical resolved shear stress for non-basal slip is over an order of magnitude greater than for basal slip [54], and that dynamic recrystallization occurs at strains as low as 5%. Being able to model the flow of ice sheets feeds into the interpretation of paleoclimate derived from the gases in the air bubbles in the ice sheets.

For sea ice, the interest is to relate the mechanical properties under various loading conditions to the microstructure so that the interaction of the sea ice with ships and static structures in the ocean can be modelled. Determining the brine network connectivity is essential for determining the permeability of sea ice, which is itself important for understanding air-ice-ocean interactions, which again relates to understanding climate change.

As for snow, firn and ice has been characterized using both OM and SEM techniques. Ice specimens have also been examined in the TEM by Unwin and Muguruma in 1972 [25], in which the ice was held at -80°C in a cooling holder. Using the heating from the electron beam and by controlling the temperature of the stage they were able to thin specimens *in situ*. They showed that cavities rapidly formed under the electron beam, but little other useful information was obtained.

Using a thin section (0.5–1 mm thick) of ice viewed in transmission between crossed polarized gratings both the grain structure and the *c*-axis directions can be determined. In the absence of a specimen, no light passes through the crossed gratings. Ice is birefringent and, thus, rotates the light. Thus, differently oriented grains exhibit different colours; and the ice appears black when viewed directly along the *c*-axis. The latter feature is used to determine the *c*-axis direction using a so-called Rigby or Universal Stage on which the sample is rotated and tilted until each grain appears black: unfortunately the *a*-axis orientations cannot be determined using this method. By logging all the rotations and tilts for different grains a pole figure of the *c*-axis of the crystals can

Figure 12. Optical thin-sections from (a) seasonal river ice near the surface of the Connecticut river near Hanover, NH, (b) thin sections from three different orientations in a laboratory-grown sea ice specimen assembled into a three-dimensional image, (c) horizontal section of sea ice from bottom of ice sheet near McMurdo Sound, Antarctica taken at -3°C showing brine pockets and (d) sea ice from near Barrow, Alaska at -15°C showing brine pockets. For (a), the field of view is $21.7\text{ cm} \times 16\text{ cm}$. For (c,d), the field of view is $10\text{ mm} \times 15\text{ mm}$. (a,b) Were taken with the ice between cross-polarizers. Image (b) is courtesy of E. M. Schulson. Images (c,d) are courtesy of D. M. Cole.

be constructed. Figure 12 presents optical images of thin sections of both freshwater ice and sea ice. The sea ice consists of columnar grains with brine pockets, which are formed when salt is rejected from the freezing water between them.

The process of determining the c -axes manually is rather tedious, and automated ice fabric (texture) analyzers have been developed that use this process and can rapidly (approx. 4 grains per minute) produce c -axis pole figures [8,55–57] with a spatial resolution of around $6\text{ }\mu\text{m}$ and an angular resolution of around 3° [58].

There have been quite a few studies on two-dimensional plane strain deformation of polycrystalline ice while viewing ice between crossed polarizers, in which slip planes, kinking and recrystallization can be observed during deformation. The results of these studies have been reviewed by Wilson *et al.* [8]. The latter workers extended these earlier OM studies by performing *in situ* deformation and annealing experiments in a fabric analyzer [8,58], thus allowing both the grains and their c -axis orientations to be determined during deformation and annealing. Although the results pertain to two-dimensional deformation, a number of interesting observations are possible using this approach.

Another way to look at the surface of ice is to use atomic force microscopy, also sometimes called scanning force microscopy. In this technique, information on a surface is obtained by

Figure 13. Micro-CT reconstructions of a sea ice specimen. The sea ice is viewed from above. The brine pockets (green) and air (red) are shown separately, and then together. The ice appears black. The volume is $7.4 \text{ mm} \times 7.4 \text{ mm} \times 14.5 \text{ mm}$. Adapted from [67].

touching the surface with a scanning probe, whose tip is a few nanometres in radius. The technique can be used to study the quasi-liquid-like layer that appears to exist on the surface of ice [59–63]. The temperature at which the quasi-liquid-like layer forms and its thickness at any given temperature are still open questions [64].

CSOM, which has 3D imaging capability, has been used to examine grain boundaries in ice [23]. A more sophisticated use of CSOM is to incorporate differential interference contrast as demonstrated by Sazaki *et al.* for imaging the surface of ice [24,65,66]. In this technique, a polarized light beam is split into two and one of the beams is scanned over the surface of the specimen. The reflected beam from the sample is recombined with the reference beam, with which it will likely have a phase difference, and passed through a polarizing analyzer that is at a right angle to the polarization of the initial polarization direction. The small difference in phase of the two waves enables sub-nanometre steps on the surface to be observed. Since the technique is non-destructive, it has been used to show the motion of steps on the surface of ice as a function of time [24].

In order to get a view of the 3D distribution of brine pockets, it is necessary to use a micro-CT. Fortunately, the X-ray attenuation between ice and brine is sufficiently different to allow the two phases to be differentiated. Figure 13 presents a micro-CT reconstruction of sea ice showing ice, brine pockets and air pockets [67,68].

The micro-CT can also be used to show the distribution of dust particles, tephra and air bubbles in specimens taken from ice sheets and glaciers as long as they are greater than $5 \mu\text{m}$. The micro-CT can also be used to image cracks in ice [23,69]. Unfortunately, the micro-CT cannot be used to image the individual ice crystals since the X-ray attenuation is independent of grain orientation. This is an area where DCT using a synchrotron has the potential to relate the grain structure and orientation directly to the mechanical properties of ice via *in situ* deformation experiments.

To the author's knowledge, the first successful use of electron microscopy to examine ice dates back to the work of Cross in 1969 [70], who used surface evaporation to keep cold the pre-cooled (to -20°C) specimen, which was mounted on thermally insulating supports in an SEM. He observed thermal etching of the surface and the formation of the fine whiskers ($0.5 \mu\text{m}$ dia.) on polycrystalline ice. Later, Schulson *et al.* also used evaporative cooling of a pre-cooled specimen (to -50°C) to examine the fracture surfaces of strained ice in an SEM [71]. Mulvaney, Wolff and Oates were the first to use EDS to determine the location of soluble impurities in ice, which was maintained at -160°C in an SEM [72]. They coated the ice with aluminium to prevent charging and were able to identify several elements at the grain boundaries. Besides the difficulty of the aluminium coating itself, the problem with this approach is that the aluminium absorbs some of the X-rays generated in the specimen. Later, Cullen and Baker demonstrated

Figure 14. Secondary electron image showing three grains and a triple junction in ice from a depth of 2563 m from the GISP2 core, which was drilled at Summit, Greenland. The X-ray spectroscopy data (right) are from the white filament that was present along the grain boundaries and show that it was largely composed of Na and Cl. The oxygen peak in the spectrum is from the ice. Courtesy of D. Cullen. (Online version in colour.)

that X-ray spectroscopy data could be obtained from uncoated ice in the SEM by maintaining the ice at around -100°C [73]. Fortuitously, the sublimation that occurs at this temperature also concentrates the soluble impurities locally to levels where they can be detected, although quantification using this approach is problematic (figure 14). Another downside with using EDS to study impurities is that it only shows what elements are present and does not provide information on the compounds present. This is particularly a problem when examining soluble impurities or small particles in ice since the EDS data always contains a strong oxygen peak from the ice (figure 14) (there is no peak for hydrogen since when the sole electron is knocked out by an impinging high energy electron there are no electron transitions to produce characteristic X-rays). Thus, it is not possible to differentiate between, say, MgSO_4 and MgS .

Subsequently, X-ray fluorescence has been used for a number of applications in the SEM to study ice: to identify the elements present in $10\text{--}800\text{ }\mu\text{m}$ particles; to examine the chemistry of grain boundaries; to examine $3\text{--}10\text{ }\mu\text{m}$ frozen brine inclusions in Vostok accretion ice (ice frozen onto the bottom of the core from the underlying Lake Vostok, Antarctica). X-ray Absorption Near Edge Structure (XANES) spectroscopy has also been used to distinguish between sulfides, sulfites and sulfates in ice cores [74,75]. X-rays from a synchrotron are used in XANES, which is also referred to as Near Edge X-ray Absorption Fine Structure (NEXAFS). The fine structure within 50 eV of an absorption edge provides information on the oxidation state of an element and can, thus, be used to distinguish between different compounds. Similar to EDS in an SEM, Synchrotron-based X-ray fluorescence not only allows chemistry determination but also mapping of elemental distributions (figure 15).

Fukazawa *et al.* [76] were the first to use micro-Raman spectroscopy to study impurities in ice. They identified several acids [HSO_4^- , HNO_3 and H_2SO_4] at the triple junctions in ice from two locations in Antarctica [Nansen and South Yamoto]. An advantage of Raman spectroscopy compared to EDS is that it detects chemical species (by Raman shifts in bond vibrational frequency) rather than simply elements. More recently, Hammonds & Baker [23] used Raman spectroscopy in a scanning CSOM to show that the grain boundaries in H_2SO_4 -doped ice held at -6°C contained water.

Iliescu, Baker and Chang demonstrated in 2004 that both EBSPs and selected area channelling patterns (SACPs) could be obtained from uncoated ice held at -100°C (figure 16a) [77]. Both EBSPs and SACPs provide complete 3D orientation information, i.e. unlike optical thin sections which provide only the c -axis orientation, EBSPs and SACPs provide both c -axis and a -axis information, and, thus, the exact misorientation between grains or crystals can be calculated.

Figure 15. Map of Br K_{α} X-ray fluorescence (11.924 keV) using synchrotron radiation from a 2 mm $\times$ 2 mm sample of snow ice from 10 cm depth on the Ross Sea. Brightness indicates relative Br concentrations, which are higher in grain boundaries and brine channels. Courtesy of R. W. Obbard. (Online version in colour.)

Although the production of both EBSPs and SACP's rely on Bragg diffraction, there are significant differences in the geometry of the two techniques. SACP's are produced by rocking a parallel electron beam on the specimen. A significant advantage is that the specimen remains horizontal allowing EDS to easily be obtained from the same area as the SACP. The major downside is that the electron beam wanders during rocking due to the spherical aberration of the objection lens so that the region from which the SACP information is obtained is many microns in diameter. The large area from which the SACP information is obtained also means that the surface has to be flat over a large area. By contrast, EBSP's are simply obtained by stopping the rastering of the electron beam on the specimen; the convergent electron beam produces diffraction from many planes. The disadvantage of the technique is that the specimen has to be tilted (typically to 70°) in order to maximize the diffracted electron signal. The advantage is that information can be obtained from a region a few nanometres in diameter. The stationary beam can be stepped across the specimen and analyzed automatically. The resulting EBSP's can be used to produce a false colour image of the grain structure based on orientation (figure 16).

Since the work of Iliescu *et al.* [77], an increasing number of studies have used EBSP's to study ice [78–87], including to produce pole figures (both *c*- and *a*-axis) and grain boundary misorientation frequency data for ice, particularly from Polar ice cores [86,87]. EBSP's can be used to observe low angle boundaries and kink bands in ice [78,80,82–85]. Regions of local stress can be determined and the weighted Burgers vector, defined as the '(density of intersections of dislocation lines with a map) $\times$ (Burgers vector)' [88], can be obtained from EBSP's in order to estimate the geometrically necessary dislocation density [84]. Combining optical thin section micrographs with EBSP's is a useful way to study low-angle sub-boundaries in ice [86]. The combination of EBSP and EDS has also proved useful for identifying dust particles in ice [89].

5. Dislocations

There has been a significant body of work concerned with characterizing dislocations in ice. In ice, dislocations glide on the basal (0001) plane either as a 60° or screw dislocations or as edge

Figure 16. (a) Secondary electron image of three grains meeting at a triple junction in polycrystalline ice. (b) EBSPs obtained from the three grains in (a). Note that high-quality patterns could be obtained as indicated by the third order lines (arrowed). The Miller indices indicate different poles in the pattern. (c) Orientation image corresponding to (a) with different colours used to visualize regions of different lattice orientation. The orientations of the hexagonal unit cells in each of the three grains, computed from the EBSPs, are inset. The ice was held at around -100°C in a vacuum of 5×10^{-4} Pa, and 15 keV electrons electron were used. Adapted from [77]. (Online version in colour.)

dislocations on the prismatic or pyramidal planes; in either case, they have a $1/3$ $[11\bar{2}0]$ Burgers' vector. The critical resolved shear stress for non-basal slip is much higher than that for basal slip [54]. Two types of basal planes exist: the glide set, in which the packing of the oxygen atoms resembles that of a h.c.p. metal; and the more widely separated shuffle set (figure 17). In principle, the $1/3$ $[11\bar{2}0]$ dislocation can dissociate into partials on the glide set, whereas such a dissociation would not occur on the shuffle set. Thus, observations of dislocations dissociated into partials would definitively show that the glide set is the operative slip plane. At this point, it has not been demonstrated on which type of basal plane slip occurs [18].

Three techniques have been used to 'image' dislocations in ice, etch-pitting replication, TEM and XT.

Etch-pitting was the first technique used to examine dislocations in ice [90–95]. Typically, the surface of the ice is coated with formvar and an etch pit forms where a dislocation intersects the surface. Either the etch pit itself or a plastic replica of the etch pit can be examined (figure 18). Etch pitting has the advantage that it provides an overview of the dislocation distribution over a large area. However, it has two major disadvantages. First, a typical etch pit is greater than $3\text{ }\mu\text{m}$ in diameter, which poses an upper limit to the observable dislocation density of approximately $1 \times 10^{11}\text{ m}^{-2}$. Second, one cannot observe dislocations that don't intersect the surface. Another problem is that it is difficult to generate etch pits from the common basal dislocations, which emerge on a non-basal surface. Sinha refined the etch pitting technique in 1977 and produced

Figure 17. Section of the (110) plane showing both ionic (H_3O^+ , OH^-) and Bjerrum (L, D) point defects and a dislocation on the shuffle set of (0001) basal planes in the ice Ih structure. The oxygen atoms are indicated by the large open circles and the hydrogen atoms by the small black circles. The short unconnected lines containing hydrogen atoms indicate out-of-plane bonds. The Burgers' vector of the dislocation is $1/3 [11\bar{2}0]$. Courtesy of E. M. Schulson. (Online version in colour.)

Figure 18. Optical micrograph showing etch pits (corresponding to dislocations) and the orientations of two grains (labelled 6 and 7) in a polycrystalline ice specimen. The left-hand grain is close to (0001), while the right-hand grain is close to (8085). Adapted from [95]. (Online version in colour.)

a 'whisker' replica that traces the line of the dislocation [96,97]. Both basal and non-basal dislocations can be observed using this approach, and it provides a somewhat 3D view of the dislocations. Using this approach with $0.25\ \mu\text{m}$ diameter whiskers, dislocation densities up to $1 \times 10^{13}\ \text{m}^{-2}$ can be observed, which is a very high dislocation density for ice.

There has only been one published TEM study of dislocations in ice [98], and in that study only grown-in dislocations were examined. In principle, one could strain an ice specimen and then make a TEM thin foil as is typically performed for metals, but for ice specimen preparation from the bulk is quite challenging, as the author can attest from personal experience. Even if an ice-thin foil can be produced and successfully transferred to the TEM, ionization damage in the electron beam is a major problem as demonstrated by Falls *et al.* [98]. In their 1982 study, as they attempted to analyze the grown-in dislocations, numerous voids formed in the thin foil and the dislocations rearranged themselves substantially (figure 19) [98].

Hayes & Webb [99] performed the first XT study of ice in 1965. Since then, there have been numerous XT studies of both dislocations and stacking faults in ice [100–112]. XT has some

Figure 19. Transmission electron microscope (TEM) imaging of dislocations in hexagonal ice frozen onto a TEM grid. The thin ice film orientation is $[2\bar{2}03]$. (a) Bright-field image of dislocations (dark lines), (b) $(\bar{3}032)$ dark-field image of (a) in which the dislocations are labelled A and B, (c) $(\bar{1}2\bar{1}2)$ dark-field image of dislocations when they display double images—radiation from the electron beam has induced point defects in the ice (arrowed). (d) $(\bar{1}120)$ dark-field image of dislocations at longer time in the TEM in which radiation-induced defects (arrowed) have increased in number and strongly diffract the electrons. Adapted from [98].

similarities to TEM observations in that a dislocation can be observed due to its strain field and its Burgers' vector can be determined using invisibility criteria (figure 20) [105]. Stacking faults can be observed due to the change in atomic stacking sequence across the fault and, again similar to TEM, the fault vector can be determined using invisibility criteria. Finally, grain boundaries can be observed due to the difference in diffraction conditions across the grain boundary.

While XT is somewhat similar to TEM, there are several advantages of XT compared to using the TEM for examining dislocations in ice: (i) the low X-ray absorption allows samples 1–2 mm thick specimens to be studied; (ii) no vacuum is required as in the TEM, avoiding sublimation problems; (iii) the ice can be examined close to the melting point; and (iv) the radiation damage is much less than in the TEM. However, a major disadvantage is that dislocation image widths are very large (approx. $20\ \mu\text{m}$). This sets a limit to the maximum dislocation density that is observable of $1 \times 10^9\ \text{m}^{-2}$. It also means that small dislocation loops cannot be resolved, and dislocation dissociation into partials, if it occurs, would be unobservable since the separation is estimated to be only approximately 20 nm [102]. Fortunately, dislocation densities in ice are very low compared to metals, hence this technique is usable for ice but not very useful for metals in which dislocation densities are much higher.

All early studies used a Lang camera and a conventional X-ray source. In this technique, a well-collimated monochromatic line X-ray beam is scanned over a single crystal (the crystal is moved rather than the X-ray beam), which is oriented at the exact Bragg condition. The resulting single diffracted beam is collected on a photographic plate (figure 21). Any dislocations present

Figure 20. Synchrotron X-ray topographs of the same region in an ice crystal imaged using three different diffraction vectors (indicated right). Some dislocations (dark lines) are invisible in each image because they satisfy the $g \cdot b$ invisibility criterion. Adapted from [105]. (Online version in colour.)

tilt the lattice planes locally with the result that intensity is redirected away from the diffracted beam. Thus, dislocations appear as dark lines on the photographic plate. The Lang technique is no longer used for a number of reasons: (i) the difficulty of precisely setting the exact Bragg condition for a crystal; (ii) it is only useful for single crystals; and (iii) the very long exposure times (from several minutes to hours). Even with these limitations, there have been numerous studies of ice using the conventional Lang camera. Such studies have shown several important features, i.e. the slip systems in ice were determined; interstitials were shown to be the common point defects; and large stacking faults were shown to occur in as-grown ice crystals [100–106].

The key problem with using conventional XT to study ice was revealed by the first use of synchrotron XT in 1986 for this purpose [107]. Synchrotron-based XT differs from the conventional Lang camera method in that polychromatic radiation is used. A highly collimated, area-filling,

Figure 21. Schematic of a Lang Camera in which a single crystal in the Bragg condition is translated through a monochromatic X-ray beam. The diffracted beam is recorded on a photographic plate. S1 is a slit that collimates the incident X-ray beam, while S2 is a slit that allows only the diffracted X-ray beam to impinge on the photographic plate. See text for details.

Figure 22. Set-up for transmission Laue synchrotron-based white-beam X-ray topography. See text for details.

high-intensity beam impinges on the ice specimen resulting in a transmission Laue pattern as each set of planes selects the appropriate wavelength to satisfy Bragg's Law (figure 22). Each of the resulting diffracted beams contains an image of the dislocations. A key feature with using the synchrotron is that exposure times are only 1–2 s, which enables both imaging of dislocation structures that have not relaxed and *in situ* deformation studies. Whitworth's group, which performed the first synchrotron-based studies on ice, showed that both Frank-Read Sources and pole mechanisms lead to dislocation multiplication in single crystal ice (figure 23) [107,108]. They also showed that dislocations in ice *under load* are aligned along 60° and screw orientations on the basal plane, i.e. along Peierls valleys, whereas prior conventional XT studies had shown curvy dislocations. Thus, it is evident that such curvy arrangements were due to relaxation as dislocations minimized their line length. Whitworth and co-workers also demonstrated that short

Figure 23. Synchrotron X-ray topographs of a Frank-Read source operating in an ice single crystal. Plane of projection (0001). The Burgers' vector, the diffraction vector and the shear stress direction are all the same and shown by the arrow. The point marked by the white arrow remains fixed as the two dislocation segments spiral around it. Adapted from [107]. (Online version in colour.)

Figure 24. Synchrotron X-ray topographs taken from an ice bicrystal at -12°C : (a) at no load and (b,c) at increasing load in the direction $\mathbf{F}$: ($10\bar{1}0$) image of left grain ($\lambda = 0.076\text{ nm}$) and ($11\bar{2}0$) image of right grain. Slip starts in the right-hand grain (the slip plane is almost edge on to the viewing direction), producing pile-ups at the grain boundary (GB), which leads to slip in the left-hand grain. Adapted from [110].

edge dislocation segments can move rapidly on the $\{1010\}$ prismatic planes, leaving behind sessile screw segments [109]. Thus, they demonstrated that the prismatic dislocations contribute little to the overall strain. Further, they found no evidence of basal screw dislocation cross-slip. This suggests that the screws are dissociated on that plane and, that the dislocations move on the glide (0001) set, see earlier.

The use of white radiation in synchrotron-based XT means that multi-grained samples can be examined. Thus, starting in 1992, Baker and co-workers performed *in situ* deformation studies on ice specimens that were one-grain thick perpendicular to the X-ray beam but contained several grains [95,105,110]. Each grain produces a Laue pattern with the shape of the diffraction spots corresponding to the shape (or part) of the grain that is illuminated. Their studies demonstrated that in polycrystalline samples, dislocations nucleated at grain boundary facets rather than by Frank-Read sources or by a pole mechanism, as observed in single crystals. Once nucleated, the dislocations move across the grain and produce pile-ups at the opposite grain boundaries (figure 24) [110]. Non-basal slip could occur but only when a grain was oriented such that the resolved shear stress on the basal plane was close to zero.

Dislocations in cores from a number of glaciers and polar ice sheets have been examined using XT. Dislocation densities from $1 \times 10^6 \text{ m}^{-2}$ to $1 \times 10^8 \text{ m}^{-2}$ were observed in ice from the temperate Mendenhall Glacier in Alaska [106], while ice from cold polar ice sheets generally has dislocation densities that are barely resolvable [111,112].

As an alternative to XT, the distortion of diffraction spots in Laue X-ray diffraction patterns can be used to estimate dislocation densities although no information on spatial arrangements is available. This technique has been used to examine dislocation densities in large grains from the Vostok (Antarctica) ice core (figure 25) [113]. X-ray diffraction is a useful averaging technique and can be used to study higher dislocation densities than observable using X-ray topography. As noted earlier, EBSPs can also be used to observe lattice distortion and rotations and subgrain boundaries in ice and also provides spatial information in order to understand the local dislocation activity, although the technique suffers from the fact that the information is only from the near-surface region (less than $1 \mu\text{m}$) [79].

Neutron diffraction of H_2O ice is not possible due to the very high incoherent scattering; however, it can be performed on D_2O ice, which behaves similarly to H_2O ice. McDaniel *et al.* [114] performed the first *in situ* deformation neutron diffraction experiments on polycrystalline D_2O ice using a specially built deformation apparatus that could apply a constant load while the specimen was kept cold. They showed that a fibre texture was produced in the direction of the applied load. Later, Piazzolo *et al.* [115] performed *in situ* compression experiments on polycrystalline D_2O ice in a neutron diffraction facility equipped with a two-circle Eulerian goniometer. They were, thus, able to track both the texture and grain size as function of strain for different strain rates.

6. Future prospects

Several techniques could be applied to understand the microstructure of snow, firn or ice in the future. Synchrotron-based diffraction contrast tomography (DCT) has been used to image snow crystals [45] and has great potential for dynamic observations of snow, firn, or ice under load in which the orientation, size and shape of individual grains in a polycrystal are tracked as function of time. While a powerful technique, access to a synchrotron can be problematic. Recently, King *et al.* [116] modified a conventional laboratory-based micro-CT and were able to obtain DCT images from metals. This technique has not yet been applied to snow or ice but could prove useful in combining orientation information with structure. Unfortunately, the laboratory-based technique has two disadvantages compared to the synchrotron-based approach. First, data acquisition times are much longer due to the low incident intensity in the laboratory-based systems. Second, laboratory-based systems currently limit specimens to about 2 mm diameter. While this works well for metals, where grain sizes are a few to tens of microns, it clearly will not work for natural ice where grain sizes are typically greater than 1 mm. One way around this problem may be to generate very fine-grained ice using the method of Stern, Durham & Kirby [117], although this may limit studies to low homologous temperatures to prevent grain growth.

High-resolution electron backscatter diffraction has the significant advantage over conventional EBSP data, where lattice rotations of only around 0.5° can be obtained, in that lattice rotations of 0.01° are possible by cross-correlating a reference pattern from a neighbouring region with a pattern from the region of interest. This enables the measurement of much lower

Figure 25. (a,b) Schematic showing how a diffraction pattern from ice can be analysed: (a) horizontal diffraction; the η -axis corresponds to the angular lattice misorientation around the Z -axis. $\Delta\eta$ represents a continuous lattice distortion from the top to the bottom of the sample; (b) vertical diffraction; the η -axis corresponds to the angular lattice misorientation around the X -axis. $\Delta\eta$ represents a combination of mosaicity and special lattice distortion from the left to the right of the sample. (c) Synchrotron X-ray diffraction pattern for the Lake Vostok ice sample V3610, in which diffraction spots from the basal (002), prismatic (100) and several pyramidal planes are present. Adapted from [113]. (Online version in colour.)

geometrically necessary dislocation densities of 10^{-10} – 10^{-12} m^{-2} [118]. The technique has been applied to the mineral olivine [119], but not yet to ice.

The electron channelling contrast imaging (ECCI) method, in which dislocation image widths of less than 100 nm are possible [120], can resolve dislocation densities of up to, at least, $1 \times 10^{13} \text{ m}^{-2}$ in the SEM [121]. Until recently, the technique has been difficult and time-consuming, but recent SEMs with new detectors and controlled small tilt capability have made this significantly easier. In this technique, backscatter electron images are acquired from a specimen that has been tilted to be very close to an exact channelling condition. Any dislocations present affect the local electron channelling and, thus, are observable in the ECCI. By tilting to several different diffraction conditions, the invisibility criterion $\mathbf{g} \cdot \mathbf{b} = 0$, where $\mathbf{g}$ is the diffraction vector and $\mathbf{b}$ the Burgers' vector, can be used to characterize the Burgers vector of the dislocations. The technique is being used increasingly to study metals. It would be very useful for studying ice

and avoiding the difficulties associated with both TEM and X-ray topography examination of dislocations in ice. The challenge is maintaining a relatively smooth, flat surface on the ice since the ECCI contrast is relatively weak and easily overwhelmed by topographic contrast.

One can also anticipate that further automation of data acquisition and analysis will continue in many of the techniques described above. Thus, at some point, ice specimens put into an SEM could have all the particles analyzed using EDS and EBSPs automatically and the results matched with a database to indicate what minerals the particles are composed of, e.g. by using the Mineral Liberation system (<http://www.mintek.co.za/technical-divisions/mineralogy/process-mineralogy/mineral-liberation-analyser-mla/>) or a similar system.

Finally, while data collection and analysis associated with many electron microscopy and X-ray diffraction are automated, specimen preparation can be time-consuming. For serial sectioning metallic materials, automated mechanical polishing systems have been available for many years that can remove as little as approximately 3 μm of material at a time [122]. Similar such automated systems would be useful for ice.

7. Conclusion

This paper has provided an overview of the many microstructural characterization techniques that can be used to examine snow, firn and ice. There are challenges in maintaining the integrity of the material during transport from remote regions and during examination, but these can largely be overcome. Two areas where further developments would prove useful are (i) being able to examine dislocation arrangements at higher dislocation densities by, say, ECCI in an SEM, and (ii) being able to quantify the chemistry of impurities in grain boundaries by using a technique such as synchrotron-based X-ray fluorescence using an X-ray nanoprobe.

Data accessibility. This article has no additional data.

Competing interests. I declare I have no competing interests.

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