Hydrological Sciences Journal

ISSN: 0262-6667 (Print) 2150-3435 (Online) Journal homepage: https://www.tandfonline.com/loi/thsj20

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To cite this article: A. RANGO , W. P. WERGIN & E. F. ERBE (1996) Snow crystal imaging using scanning electron microscopy: I. Precipitated snow, Hydrological Sciences Journal, 41:2, 219-233, DOI: 10.1080/02626669609491494

To link to this article: https://doi.org/10.1080/02626669609491494

Published online: 24 Dec 2009.

Snow crystal imaging using scanning electron microscopy: I. Precipitated snow

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Abstract Low-temperature scanning electron microscopy (SEM) was used to observe precipitated snow crystals. The newly-fallen snow crystals were obtained in storms at Beltsville, Maryland, and at Bearden Mountain near Davis, West Virginia, USA. The snow samples were mounted on modified SEM stubs, frozen in liquid nitrogen, sputter coated with platinum, and imaged with an electron beam. Many types of precipitated snow crystals were observed including hexagonal plates, columns, needles, stellar dendrites, bullets, graupel, and rimed crystals. The SEM techniques that were developed can be used for quantitative measurements of size, shape and structure of crystals. SEM of precipitated snow appears to have direct application for the inference of atmospheric and cloud conditions where the snow crystals formed and travelled to the ground and for the development of a relationship between snow crystal type and snowfall intensity and amount. The SEM technique provides a new procedure to record permanently snow crystal conditions during winter storms.

Images de cristaux de neige obtenues par microscopie électronique à balayage: I Neige fraîche

Résumé La microscopie à balayage à basse-température a été utilisée pour observer des cristaux de neige fraîchement tombés. La neige fraîche a été recueillie au cours de tempêtes de neige ayant eu lieu à Beltsville dans le Maryland et Bearden en Virginie Occidentale. Les échantillons de neige ont été montés sur des supports pour microscope à balayage, plongés dans de l'azote liquide, recouverts d'un film de platine, et observés sous un faisceau d'électrons. Plusieurs types de cristaux de neige ont pu être identifiés, tels que des plaques hexagonales, des colonnes, des aiguilles, des dendrites stellaires, des boules, des fusions et des cristaux ourlés. Les méthodes d'observations en microscopie à balayage que nous avons développées permettent d'évaluer quantitativement la taille, la forme et les liaisons des cristaux de neige. Cette technique semble avoir de grandes potentialités pour estimer les conditions atmosphériques et nuageuses conduisant à des chutes de neige importantes et pour établir une relation entre le type de cristal de neige et l'intensité de la chute de neige. La technique de microscopie à balayage à basse température offre une nouvelle approche pour enregistrer les conditions de formation des cristaux de neige durant les tempêtes de neige.

Open for discussion until 1 October 1996

INTRODUCTION

Snow may cover up to 53% of the land surface in the northern hemisphere (Foster & Rango, 1982) and up to 44% of the world's land areas at any one time. It supplies at least one third of the water that is used for irrigation and the growth of crops worldwide (Steppuhn, 1981). In high mountain snowmelt basins of the Rocky Mountains, USA, as much as 75% of the total annual precipitation is in the form of snow (Storr, 1967), and 90% of the annual runoff is from snowmelt (Goodell, 1966).

A snow crystal is a single frozen ice grain that generally results from a process known as ice nucleation in which atmospheric water vapour condenses or freezes on solid particles or nuclei at temperatures below 0°C. When nucleation occurs, the water molecules form a hexagonal crystal lattice resulting from the specific orientation and bonding that occurs between the oxygen and hydrogen atoms. Depending on the temperature and moisture conditions that prevail during the formation and descent of snow crystals, the shapes may be in the form of plates, stellar crystals, columns, needles, dendrites and other variations, all of which are based on the hexagonal lattice structure. An individual snow crystal may range in size from 50 μm to 5 mm (Gray & Male, 1981); aggregations of two or more of these crystals form a snowflake, which may range in size from 0.1 mm to several centimetres (Hobbs, 1974).

After falling snow reaches the ground, changes in the fine structure of the snow crystal begin and a second type of snow results. LaChapelle (1969) separates precipitated snow in the atmosphere and metamorphosed snow on the ground. However, metamorphic changes can sometimes be observed in precipitated snow which indicates that there is not always a strong contrast between precipitated snow crystals and metamorphosed snow grains.

Classification systems for precipitated and metamorphosed snow have been devised based on observations of falling snow (Nakaya, 1954; Magono & Lee, 1966) and snow on the ground (Sommerfeld, 1969; LaChapelle, 1969; Sommerfeld & LaChapelle, 1970). Classification systems covering both precipitated and metamorphosed snow have also been established (ICSI, 1954; Colbeck *et al.*, 1990). The observation and permanent recording of the various categories of snow in the classification systems in the field has been difficult. The most frequently used approaches employ microscopes (photomicrography) and cameras (photomacrography) (LaChapelle, 1969).

LaChapelle (1969) has provided an excellent description and discussion of the techniques used in photomicrography and photomacrography. The use of reflected light yields a white image against a black background; transmitted illumination produces a white background and an image which yields more information about internal structure; low oblique transmitted illumination provides information on both surface and internal features. Most of the famous snow crystal photographs made by Bentley in the early 1900s (Bentley & Humphreys, 1962) were made with a microscope and transmitted illumination.

To obtain photographs of white snow crystals on black backgrounds, Bentley had to cut out the snow crystal image meticulously from its negative, place it on a clear background, and print the image a second time. Nakaya's (1954) snow crystal photographs were done with a microscope in a cold laboratory and used low oblique transmitted illumination. The photographs of LaChapelle (1969) were all taken with 35 mm cameras using reflected, transmitted and low oblique transmitted illumination (sometimes with additional transmitted light).

The problems with either the light microscope or 35 mm camera approaches are numerous. The snow crystals must be protected from melting before photographing them. Body heat, heat from slide mounts, warmer ambient air and especially heat from strong illumination melts the snow crystals. Low cold room temperatures and high humidities sometimes result in condensation on the crystals and in frost on optics. The snow crystal samples are also susceptible to metamorphism and evaporation. Because of these problems, this paper describes studies to find a new technology that could be used for imaging snow crystals or grains.

SCANNING ELECTRON MICROSCOPY

Although the first scanning electron microscope was built about 60 years ago (Black, 1974), commercially available instruments were not available until 1965 (Wergin, 1981). Since then, many advances for applying scanning electron microscopy (SEM) in biological fields have been made. The SEM has several important advantages over the light microscope. First, the images obtained are generally easy to interpret because they consist of visual information only about surface characteristics (as opposed to a mix of surface and internal characteristics obtained from the light microscope). Second, with magnifications of 20-100 000×, SEM resolution is 100 times greater than that of a light microscope, while its depth of field is 300-500 times greater than that of the light microscope. Third, relatively large samples can be examined with a SEM (up to 4 cm^3). Fourth, a wide range of stage movements is possible with the SEM thereby allowing one to photograph samples from numerous angles. The ability to tilt a sample allows the investigator to record stereo images that enable a viewer to perceive a three-dimensional image of the specimen. As a result, accurate spatial relationships and precise measurements can be obtained.

In the SEM, a stream of electrons in an intense beam (5-50 nm in diameter) is focused on the surface of the sample in the vacuum of the instrument column. The focused beam is deflected (scanned) across the sample in a manner analogous to an electron gun producing an image on a TV screen. The primary electron beam of the SEM excites the surface electrons of the sample. These electrons, which are called secondary electrons, escape from the surface of the sample and are collected by a detector. In the detector, the electrons hit a scintillator producing photons that are photoamplified and converted to an electrical signal used to generate an image on the viewing screen.

Recently, the Beltsville USDA laboratory has been equipped with a new generation of SEMs, namely, a Hitachi S-4100 field emission SEM, with an Oxford CT 1500 HF Cryosystem. This combination of instrumentation, known as low temperature scanning electron microscopy, allows the use of an SEM at low accelerating voltages, normally 1-10 kV, to observe and record images of delicate, frozen, hydrated samples that are maintained at temperatures of about −185°C. In addition to applying this technique successfully to biological studies, application has recently been made to the imaging of snow crystals.

Snow falling outside the SEM facility in Beltsville was sampled on six different occasions during the winter of 1993-1994. Because of the geographic location, the storms were relatively warm with air temperatures ranging from −6°C to +1°C during the snow events. Additional data were acquired from the mountains of West Virginia during storms in the Winter of 1994-1995 with air temperatures ranging from −11°C to −8°C.

MATERIALS AND METHODS

The initial attempts to allow snowflakes to settle onto a precooled specimen holder were unsuccessful; the snowflakes tended to "bounce" off the holder and those that did alight did not remain attached during subsequent handling. These problems were solved (Wergin & Erbe, 1994; Wergin *et al.*, 1995) by placing a thin layer of methyl cellulose solution (Tissue Tek) onto a precooled copper plate placed in a Petri plate at ambient outdoor temperatures. For collection, snowflakes were allowed to fall or were lightly brushed onto the methyl cellulose solution. After a visible sample of snowflakes had accumulated in this manner, the copper plate with sample was frozen by being plunged into a styrofoam cup containing liquid nitrogen (LN_2) at −196°C. Frozen samples were transferred directly to the laboratory or stored in a LN_2 Dewar flask for future analysis. From here, the sample was vacuum transferred to the preparation chamber of an Oxford CT 1500 HF Cryotrans system for sputter coating with platinum (Pt) metal. Finally, samples were placed on the precooled (−185°C) stage of a Hitachi S-4100 field emission SEM.

Many samples of snowflakes were stored in LN_2 for future study utilizing flat copper plates (15 mm × 27 mm) inserted in long, square profile brass tubes. The copper plates were subsequently attached to a modified Oxford holder for analysis. Samples have been stored in LN_2 storage Dewars for periods of up to six months before observation by SEM.

Accelerating voltages from 1 to 10 kV were used to record stereo images photographically onto Polaroid Type 55 P/N film. To obtain stereo pairs, a first image was recorded, the stage was tilted 6°, the specimen was recentered, and a second image was recorded.

A similar field methodology was attempted by Wolff & Reid (1994) for observing snow crystals collected in Greenland. However, they experienced difficulty in collecting and retaining new snow crystals and this resulted in only a few useable images. Methods developed in this study seem to have solved these problems.

RESULTS

Samples obtained at sub-freezing temperatures

At −5°C air temperature, samples consist of snowflakes composed of several types of snow crystals. In Fig. 1, the majority of the snow crystals making up the snowflake can be categorized as simple flat hexagonal plates (classified "P1a" by Magono & Lee, 1966), although some are irregularly shaped. The largest plates, which are frequently associated in complex or radiating assemblages of plates (P7a), measure 0.7-0.8 mm in diameter and 0.07-0.08 mm thick. The edges of the thicker plates frequently reveal an indentation between the two narrow surfaces (Fig. 1). This type of plate appears to correspond to the double sheet structure described by Nakaya (1954). Figure 2 shows an even more complex association of crystals from the Beltsville storms than Fig. 1. In Fig. 2 the following crystal types are represented: solid columns (C1e), long solid columns (N1e), elementary sheaths (N1c) and needles (N1a).

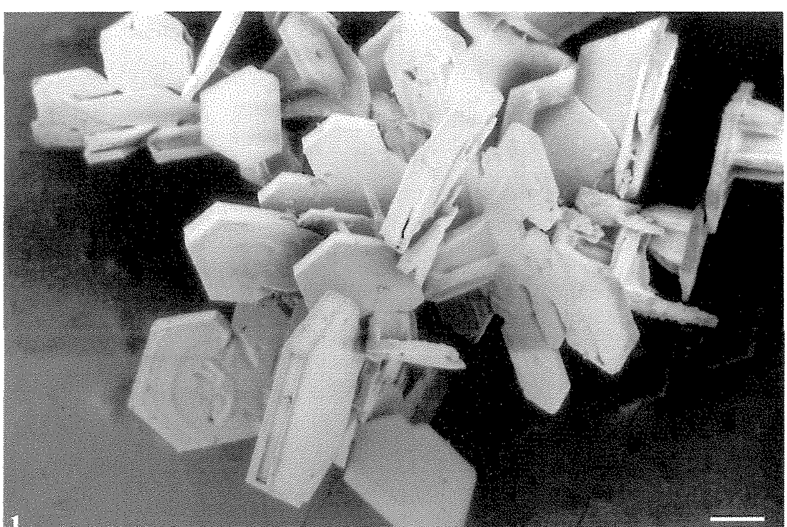

Fig. 1 SEM photograph illustrating a snowflake composed of numerous snow crystals, which largely consist of hexagonal plates. The thicker plates have two opposed thin hexagonal sheets and generally exhibit an indentation between the two layers. (Collected at −5°C; magnification 40×; bar = 0.2 mm).

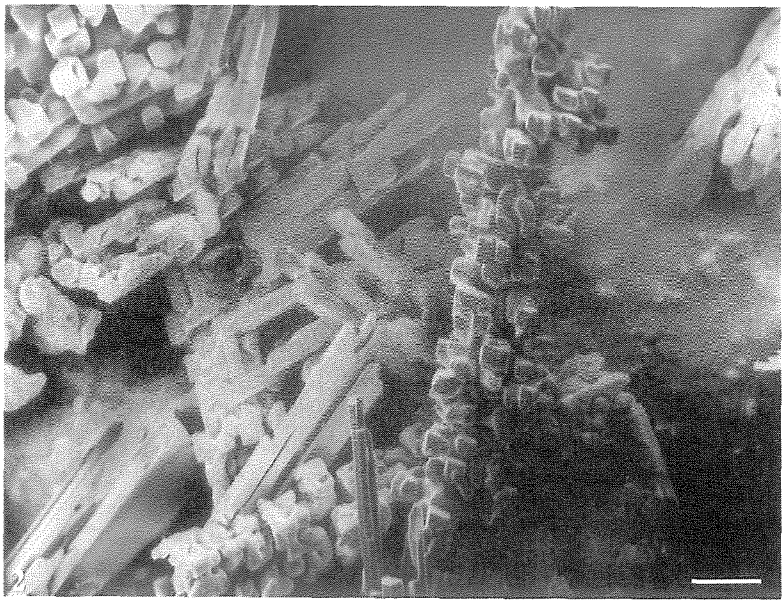

Fig. 2 Snowflakes composed of numerous snow crystals including solid columns, long solid columns, elementary sheaths and needles. (Collected at −5°C; magnification 55×; bar = 0.2 mm).

Depending on the snowfall rate or collection technique, individual snow crystals are easily captured. Many types of individual snow crystals consist of variations of the hexagonal plates. A single snow crystal having six broad branches (P1c) is illustrated in Fig. 3. The hole that appears in the centre of this crystal probably represents the nucleation centre from which crystallization was initiated. Although the nucleation particle is not apparent, the hole measures about 30 x 50 μm across and the crystal has an edge to edge span of 1.5 mm.

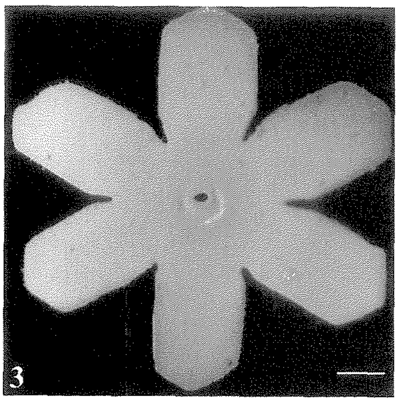

Fig. 3 A snow crystal with broad branches which is a variation of the hexagonal plate. In the centre of the plate is a hole that may have contained the nucleation particle at the time of formation. (Collected at −5°C; magnification 45×; bar = 0.2 mm).

The crystal shown in Fig. 4 is about 1 mm in diameter and probably began as a hexagonal plate and then subsequently developed dendritic extensions (P2g). Figure 5 shows a crystal with sector-like branches (P1b) that measures 0.75 mm in diameter and has another hexagonal plate shaped like a six-pointed star on the edge of one arm. This star shape, which is about 250 μm in diameter, is not shown in the classification of Magono & Lee (1966). The centre of the star is a hexagonal plate with a diameter of only about 40 μm.

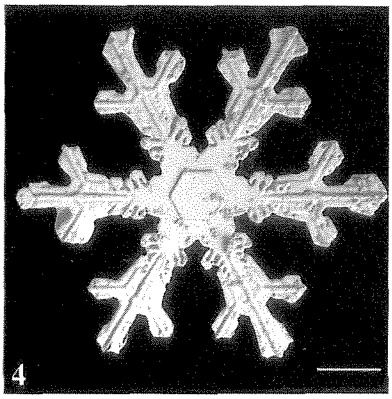

Fig. 4 SEM example of a "classic" snow crystal – a plate with dendritic extensions. (Collected at $-10°C$; magnification 50×; bar = 0.2 mm).

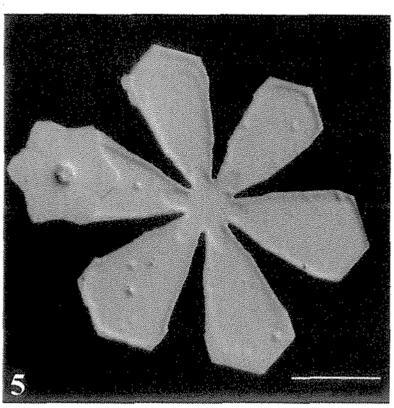

Fig. 5 Example of a combination crystal imaged with SEM – a crystal with sector-like branches with another hexagonal plate in the form of a six-pointed star on the edge of one branch. A smaller hexagonal plate is at the centre of the star. (Collected at $-10°C$; magnification 70×; bar = 0.2 mm).

The snowflake in Fig. 6 is composed of bullets capped with hexagonal plates (CP2a). The bullets consist of short columns that are tapered at one end. The three bullets are joined at their tapered ends and have an overall diameter of about 1 mm. The opposite end of the bullet, which is flat, is capped with a hexagonal plate that is centred on the top of the bullet and whose planer axis is perpendicular to that of the bullet.

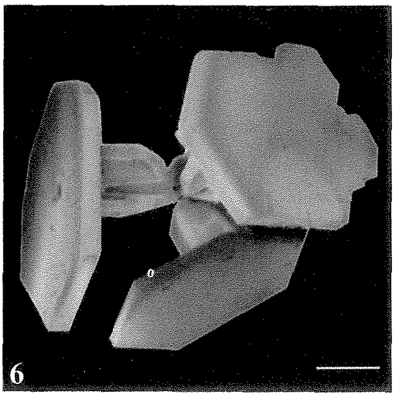

Fig. 6 SEM image of crystal in the form of bullets capped with plates. The bullets appear to be joined at their tapered ends. (Collected at −5°C; magnification 50×; bar = 0.2 mm).

Figure 7 shows another crystal that can be classified as a plate with simple extensions (P2e). This crystal, which is only 0.6 mm in diameter, has some surface deposits, perhaps collected while falling through clouds containing supercooled water droplets. This crystal also has a central circular hole where a possible freezing nucleus existed. The hole itself is less than 8μm across.

The dominant snow crystal illustrated in Fig. 8 is a hexagonal plate with six sector-like extensions (P2f). Associated with the surface of the central plate and extending into the extensions or arms are numerous spherical "droplets". These particles, which are about 0.03-0.06 mm in diameter, are consistent with descriptions of cloud particles or supercooled water droplets observed in light microscopic examinations of snow crystals (Bentley & Humphreys, 1962; Nakaya, 1954). The radiating arms contain concentrations of another type of surface structure, namely small microcrystals that consist of minute hexagonal plates (G3) most of which are only 0.05-0.10 mm in diameter. Some of these structures appear to be transitional stages that arise from the droplets.

Rime is another type of surface structure that can be observed on snow crystals. At lower magnification (110×), the rime appears as a fuzzy deposit on the edge of a hexagonal plate (Fig. 9). When this deposit is resolved at

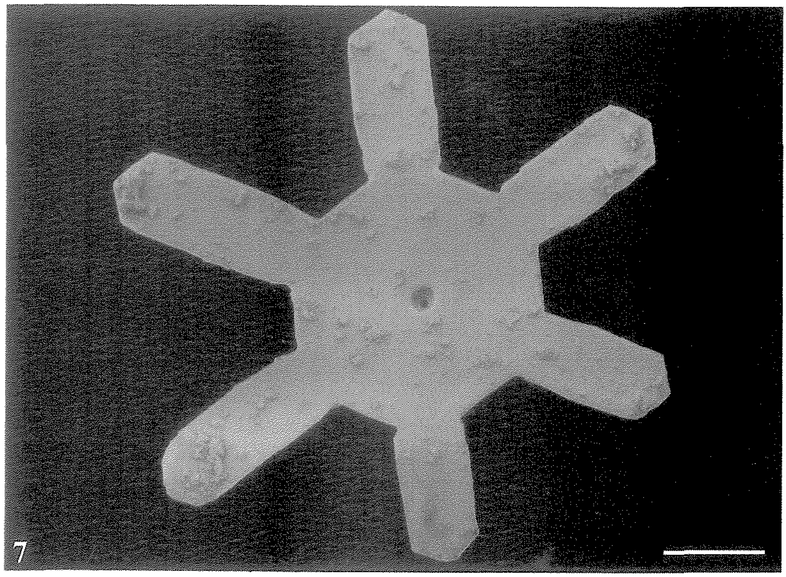

Fig. 7 SEM image of a plate with simple extensions collected from a mixture of other crystals. The centre hole may have contained the crystal nucleus. Deposits on the surface probably accumulated while falling through the atmosphere. (Collected at −10°C; magnification 160×; bar = 0.1 mm).

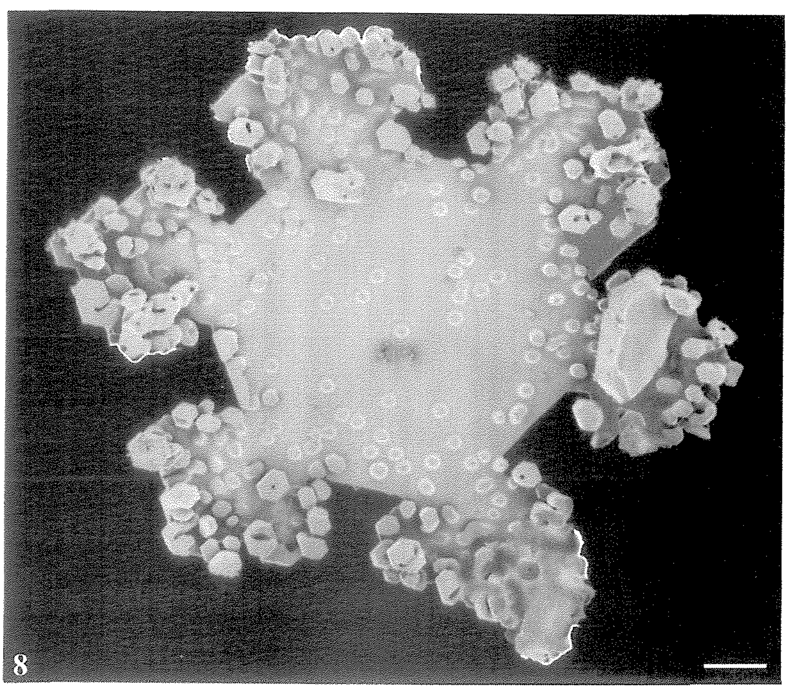

Fig. 8 Snow crystal in the form of a hexagonal plate with sector-like extensions. The central portion of the crystal exhibits surface structures consisting of spherical "droplets" whereas the branches contain microcrystals that consist of small hexagonal plates, which may arise from the droplets. (Collected at −6°C; magnification 50×; bar = 0.2 mm).

higher magnifications (320×), it appears as a multitude of fine, rounded non-hexagonal crystals about 5 μm in diameter and 20 μm long, predominantly attached to the edge of the plate crystal (Fig. 10). Rime is a formation that results when a growing crystal falls through a cloud composed of supercooled water droplets that freeze on its surface. Rime, which is not restricted to any one type of snow crystal, can be observed on the edges of the hexagonal plates (R2a, Figs 9 and 10) as well as on the edges of the columnar crystals, branched arms and needles.

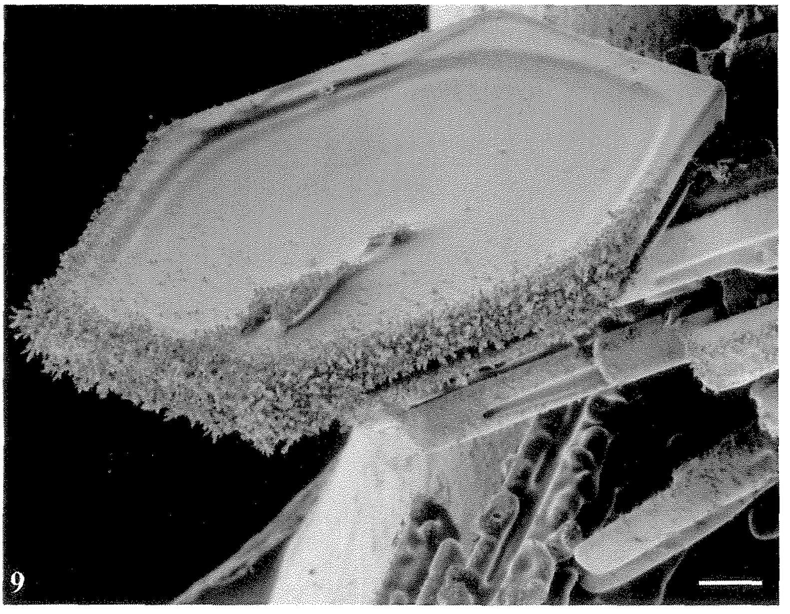

Fig. 9 Single hexagonal plate containing preferential accumulation of "rime" along its edge. (Collected at −6°C; magnification 110×; bar = 0.1 mm).

Snow samples obtained at or above freezing temperatures

Snow that is sampled when the air temperature close to the ground is at or above freezing exhibits snow crystals that appear to be undergoing melt-freeze metamorphism. In general, all crystals, e.g. plates, needles, columns and dendrites, in a snowflake, exhibit this phenomenon when collected near 0°C. Figure 11 shows a plate with dendritic extensions (P2g) which seems to have undergone some melt and freeze. The angular corners of the crystal become rounded as soon as the melt is initiated. Occasionally, a snow crystal, in this case a hollow column (C1f, Fig. 12), is captured when melt metamorphism appears to be actively occurring. The hollow column exhibits both an area where the crystalline faces and their angular intersects have melted as well as adjacent areas that have not been affected and continue to exhibit sharp

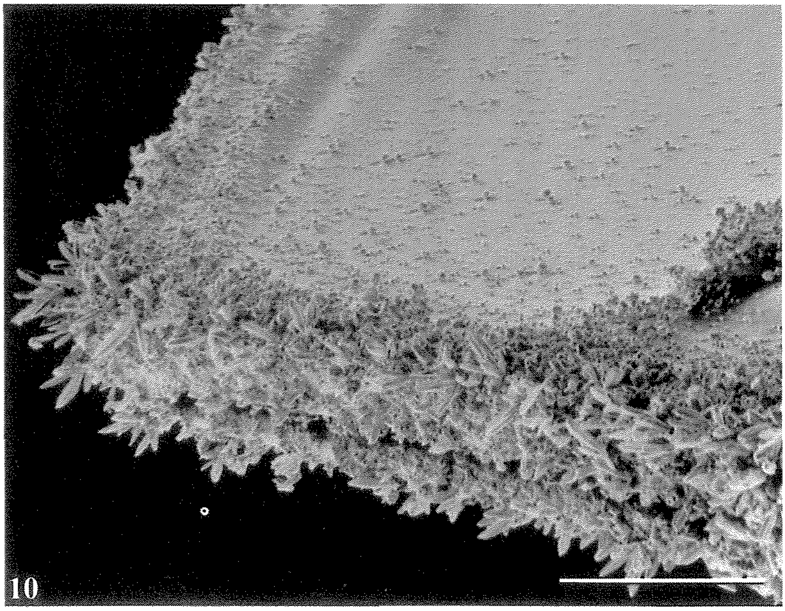

Fig. 10 Edge of the hexagonal plate illustrated in Fig. 9. Rime appears as a fine, non-hexagonal, heterogeneous crystalline accumulation that collected along one edge of a snow crystal. (Collected at −6°C; magnification 320×; bar = 0.1 mm).

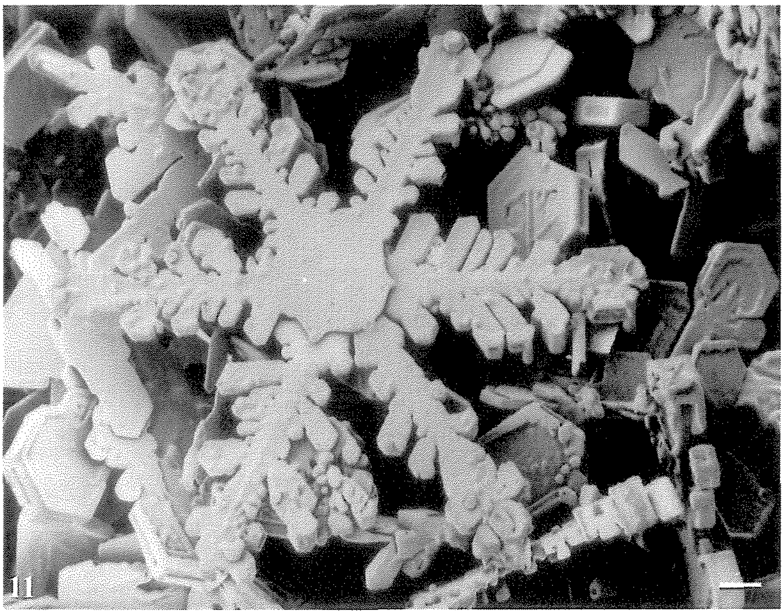

Fig. 11 A snow crystal in the form of a hexagonal plate with dendritic extensions showing effects of melt-freeze metamorphism. The apices of the crystalline faces do not form sharp angles. Other types of snow crystals are apparent in the background. (Collected at 0°C; stored for 1 month in LN_2; magnification 35×; bar = 0.2 mm).

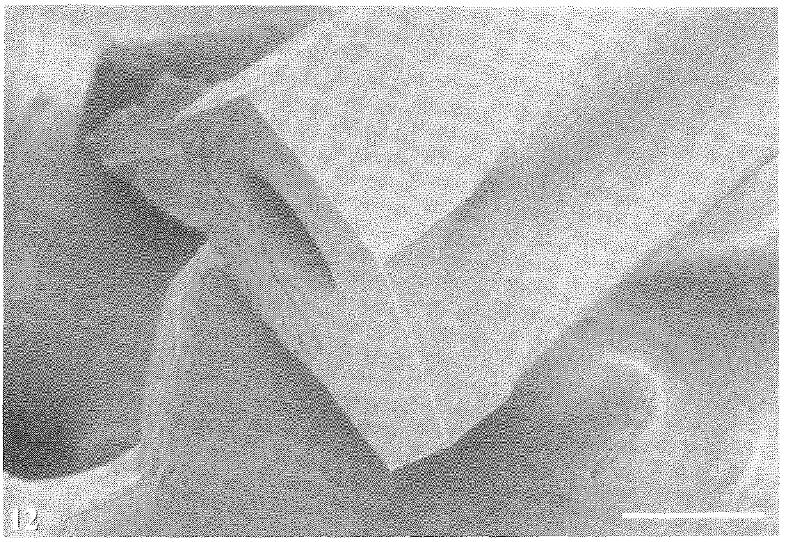

Fig. 12 A hexagonal column with a round cylindrical core that is asymmetrically positioned in the column. Upper portion of the column shows the effects of melt-freeze metamorphism while the end continues to exhibit the sharply defined crystalline characteristics of a snow crystal. (Collected at 0°C; stored for 1 month in LN_2; magnification 225×; bar = 0.1 mm).

crystalline features. As the melting process continues, the snow crystal, which is gradually engulfed in an amorphous liquid matrix, starts to lose its identity.

Snow crystals illustrated in Figs 11 and 12 were examined after the samples had been collected, frozen, and stored in liquid nitrogen for one month. No indication of secondary formation of ice crystals or any other destructive structural changes could be associated with the storage procedure. The imaging of these crystals was comparable to those imaged directly after collection.

DISCUSSION

The approach taken with low temperature scanning electron microscopy appears to solve many of the limitations of photomicrography and photomacrography. The procedure not only allowed observation of the same forms of snow crystals that had been previously described in light microscopic studies but also easily enabled resolution of the nucleation centres and other minute surface features at very high magnifications with high resolutions. The specimens maintained good thermal contact with the copper plate and did not appear to be altered by the sputter coating procedures. The snow crystals were stable in the electron beam, did not sublime over several hours of observation, and could be magnified many thousands of times to reveal microcrystalline water deposits and rime on the surface of the snow crystals. The procedure, which was used to collect specimens during several snowfalls in Beltsville (40 m a.s.l.) during the 1993-1994 winter season and on Bearden Mountain near Davis, West Virginia (1174 m a.s.l.) during the 1994-1995 winter season, was also capable of preserving precipitated snow that was already melting, and graupel. Furthermore, alternative holders allowed capture of the snowflakes and their storage in LN_2 for periods of up to six months until the specimens could be processed for examination in the SEM. Storage of samples in LN_2 had no obvious affects on the structure of the crystals aside from those already documented in the long term storage of biological samples (Wergin & Erbe, 1991). Finally, the specimen stage of the SEM allowed specimen tilt so that stereo images of the snow crystals could be recorded thus revealing true three-dimensional structure. This procedure has apparently not been demonstrated using light microscopy.

Through the years several classification systems have been proposed to categorize snow crystals (Shedd, 1919; ICSI, 1954; Nakaya, 1954; Magono & Lee, 1966; Colbeck *et al.*, 1990). Of these, the overall system by ICSI (1954) and recently revised by Colbeck *et al.*, (1990) is most widely accepted. In this system, snow crystals or precipitation particles are divided into eight major categories: columns, needles, plates, stellar dendrites, irregular crystals, graupel, hail and ice pellets. Each category can be further subdivided on the basis of shape. Although neither hail nor ice pellets were observed in the present study, examples of all other shapes were observed and appeared to be consistent with the descriptions that were based on light microscopic observations. Of the more detailed classification of precipitated snow crystals by Magono & Lee (1966), 30 different categories have been observed in these early low temperature SEM experiments.

APPLICATIONS OF SNOW CRYSTAL SIZE AND SHAPE OBSERVATIONS

SEM, as well as visual, light microscopy and other camera techniques, can be used to observe important details of snow crystals and grains. Measurements of crystal or grain form, shape, size and bonding can be applied to various types of studies.

Observation of precipitated snow crystal and snowflake forms can be related directly to the conditions in the atmosphere and clouds where the snow crystals formed and along the path they followed to the ground. Both Nakaya (1954) and Magono & Lee (1966) have produced results which relate snow crystal form to the temperature and humidity conditions in the atmosphere. A detailed history of snow crystal form can be used to infer the temperature and humidity history leading to a particular snowfall event. Evidence of riming can be used to document further conditions encountered during the precipitation event. The detailed SEM observations should allow a better understanding of cloud physics and precipitation processes. The addition of X-ray analysis to the SEM technique could provide additional information about the elemental composition and sources of crystal nuclei as well as other pollutants that may have been incorporated into the crystal.

Additionally, crystal type can also be related to snowfall intensity and amount through temperature inference. Data suggest that maximum crystal growth and aggregation of crystals occur when isothermal layers exist at approximately −5°C and −15°C. Hallett & Mason (1958) and Hallett (1965) found that the rate of crystal growth occurring between −4°C and −6°C (needles) and between −14°C and -16°C (dendrites) was in excess of rates at intermediate temperatures by as much as a factor of 100. Cross (1966) observed that when these isothermal layers are 1 km thick or more, heavy snowfall can occur. The SEM observation of crystal type in falling snow could be used to document the conditions involved in heavy snowfalls.

Acknowledgements The authors wish to thank C. Pooley for converting the SEM negatives to the digital images that were used to illustrate this study.

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Received 22 May 1995; accepted 19 November 1995