

## Applications

# Observations of Snow Crystals Using Low-Temperature Scanning Electron Microscopy

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**Summary:** Low-temperature scanning electron microscopy (SEM) was used to observe precipitation particles commonly known as "snowflakes." The snowflakes were collected in Beltsville, Maryland, at temperatures ranging from  $-6^{\circ}$  to  $+1^{\circ}\text{C}$ , mounted on SEM stubs, frozen in liquid nitrogen ( $\text{LN}_2$ ), and then transferred to a cryosystem mounted on a field-emission SEM. Neither sputter coating with platinum nor irradiation by the electron beam affected their delicate fine structure. SEM observations revealed that snowflakes consisted of aggregations of snow crystals that occurred as hexagonal plates, prismatic columns, needles, and dendrites. In some cases, the snow crystals contained minute surface structures that consisted of rime, microdroplets, short prismatic columns, and amorphous films. Snow crystals from wet snow, which were collected at temperatures of  $0^{\circ}$  and  $+1^{\circ}\text{C}$ , exhibited varying degrees of metamorphism or melting. The discrete crystalline faces and their sharp intersecting angles were gradually replaced by sinuous surfaces that tended to exhibit more spherical shapes. This study indicates that low-temperature SEM is a valuable technique for studying the formation and metamorphosis of snow crystals. The results suggest that combining low-temperature SEM and x-ray analysis could also provide qualitative elemental information on the nucleation particles of snow crystals as well as on the composition of acid snow.

**Key words:** low temperature, SEM, snowflake, snow crystal

## Introduction

Snow, which may cover up to 53% of the land surface in the northern hemisphere (Foster and Rango 1982) and up to 44% of the world's land areas at any one time, supplies at least one third of the water that is used for irrigation and the growth of crops (Gray and Male 1981). For this reason, estimating the quantity of water that is present in the winter snowpack is an extremely important forecast activity that attempts to predict the amount of moisture that will be available for the following growing season. Remote sensing approaches, using microwave data, have been successfully tested in certain situations to calculate areal water equivalent of the snowpack prior to melting (Goodison *et al.* 1990, Rango *et al.* 1989). Unfortunately, these estimates can be easily confounded by the sizes and shapes of the snow crystals or grains in the snowpack.

A snow crystal is a single frozen ice grain that generally results from a process known as ice nucleation in which atmospheric water vapor condenses or freezes on a solid particle or "nucleus" at temperatures below  $0^{\circ}\text{C}$ . When nucleation occurs, the water molecules form a hexagonal crystal lattice resulting from the specific orientation and binding that occurs between the oxygen and hydrogen atoms. Depending on the temperature and moisture that prevails during formation and descent of snow crystals, the crystal shape may take the form of plates, stellar crystals, columns, needles, or dendrites, all of which are based on the hexagonal lattice structure. An individual snow crystal may range in size from  $50\ \mu\text{m}$  to 5 mm (Gray and Male 1981); aggregations of two or more of these crystals form a snowflake, which may range in size from 0.1 mm to several cm (Hobbs 1974).

The shapes of snow crystals have been extensively studied and photographed with the light microscope (Bentley 1904, 1923; Bentley and Humphreys 1931; Nakaya 1954). Although these studies have resulted in several classification systems that recognize as many as nine distinct classes of snow crystals and over 30 subclasses (Hobbs 1974), detailed examinations have been hampered by the difficulty of working with a frozen specimen, which is susceptible to sublimation and melting, and by the limiting resolution of the light microscope. In our laboratory, preliminary studies indicated that low-temperature scanning electron microscopy (SEM) could be used to examine snow crystals (Wergin and Erbe 1994a, b). Encouraged by these investigations, this report describes the technique for sampling

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snow, the feasibility of storing snow samples, and the use of low-temperature SEM to observe a full range of snow crystals.

## Materials and Methods

Samples of snowflakes and snow crystals were collected in Beltsville, Maryland, on six different occasions. The samples, which were obtained when the air temperatures ranged from  $-6^{\circ}\text{C}$  to  $+1^{\circ}\text{C}$ , consisted of freshly fallen snowflakes as well as snow that had fallen up to 30 h earlier.

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. A successful procedure consisted of placing a thin layer of methyl cellulose solution (Tissue Tek) on a holder. The holder was then placed in a Petri dish, taken outside, and precooled to the ambient outdoor temperature. Next, the lid of the Petri dish was removed and snowflakes were either allowed to settle on the surface of the methyl cellulose solution or lightly brushed onto its surface. After a visible sample of snowflakes was obtained in this manner, the holder was plunged into a styrofoam cup containing liquid nitrogen ( $\text{LN}_2$ ) at  $-196^{\circ}\text{C}$ , transferred to the laboratory, and placed into the slush chamber of an Oxford CT 1500 HF Cryotrans system. The holder was then attached to the transfer rod of the Oxford cryosystem, moved under vacuum into the prechamber for sputter coating with platinum (Pt), and then inserted into a Hitachi S-4100 field emission SEM equipped with a cold stage that was maintained at  $-185^{\circ}\text{C}$ .

Several samples of snowflakes were stored in  $\text{LN}_2$  for future study. To store samples of snow, a flat metal plate ( $15 \times 27$  mm) was substituted for the Oxford holder. After the samples had been collected and frozen they were stored in an  $\text{LN}_2$  for periods up to 1 month before being attached to a modified Oxford holder and processed for examination as described above.

Accelerating voltages of either 2 kV or 5 kV were used to observe and record images onto Polaroid Type 55 P/N film. To obtain stereo pairs, the first image was recorded, the stage was tilted  $5$  to  $10^{\circ}$ , the specimen was recentered, and a second image was recorded.

## Results

### Samples Obtained at Subfreezing Temperatures

At  $-5^{\circ}\text{C}$  air temperature, the samples consist of snowflakes that are composed of several types of snow crystals. In Figures 1 and 2, the majority of the snow crystals, which are irregularly shaped, fall into a class known as simple flat hexagonal plates. The largest plates, which are frequently associated in complex assemblages, measure 0.7 to 0.8 mm in diameter and 0.07 to 0.08 mm in thickness. The edges of the thicker plates frequently reveal an indentation between the two broad narrow surfaces (Fig. 1, arrow). This type of plate appears to correspond to the double sheet structure described by Nakaya (1954). Single flat hexagonal sheets occasionally appear as caps on short columns that measure 0.4 to 0.5 mm in length (Fig. 2, arrow).

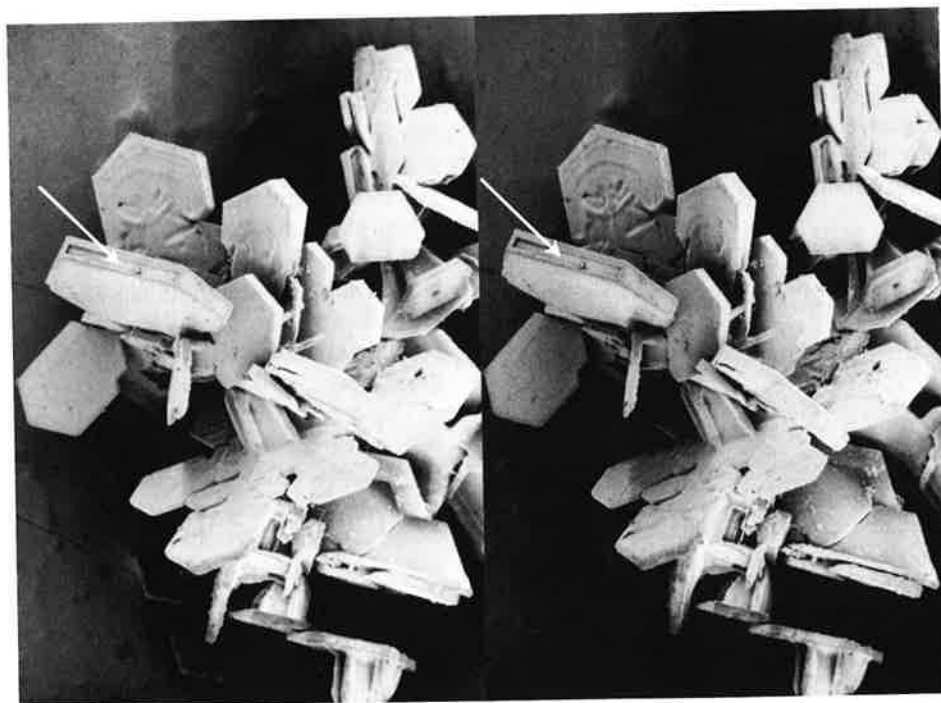


FIG. 1 Stereo pair illustrating a snowflake composed of numerous snow crystals, which largely consist of hexagonal plates. The thicker plates have two apposed thin hexagonal sheets and generally exhibit an indentation between the two layers (arrow). (Collected at  $-5^{\circ}\text{C}$ , imaged at 5 kV.) Horizontal field width = 2.0 mm.

Other types of individual snow crystals consist of variations of the hexagonal plates. A single hexagonal snow crystal having six broad branches is illustrated in Figure 3. The hole that appears in the center of this crystal probably represents the nucleation center from which crystallization was initiated. Although the nucleation particle is not apparent, the hole measures about  $30 \times 50 \mu\text{m}$  and the crystal has an edge to edge span of 1.5 mm.

Irregularly shaped snow crystals are by far more common than the symmetrical forms. However, even the irregular forms of the crystalline plates have a basic hexagonal structure (Fig. 4). In Figure 4, the center of the crystal probably began as a hexagonal plate; however, further growth resulted in six dissimilar branches that also exhibit hexagonal patterns.

A sample collected at  $-6^\circ\text{C}$  reveals snowflakes whose surface consists of aggregations of numerous short prismatic crystals (Fig. 5). These individual crystals, which are 0.5 to 0.7 mm in diameter and 0.3 to 0.8 mm in height, are well defined on the surface of the snowflake, but the aggregate seems to be held together by an amorphous, noncrystalline matrix (Figs. 5 and 6). This matrix, which appears continuous with the outer surface of the crystal (Fig. 6), may represent a water droplet that collided with the prismatic crystals either during formation or while descending. No obvious secondary surface structures could be observed on the surface of the prismatic snow crystals (Fig. 6).

Other examples of snowflakes are composed of snow crystals that are aggregated as an assemblage of hexagonal plates and, as "bullets," capped with these plates (Fig. 7). The assem-

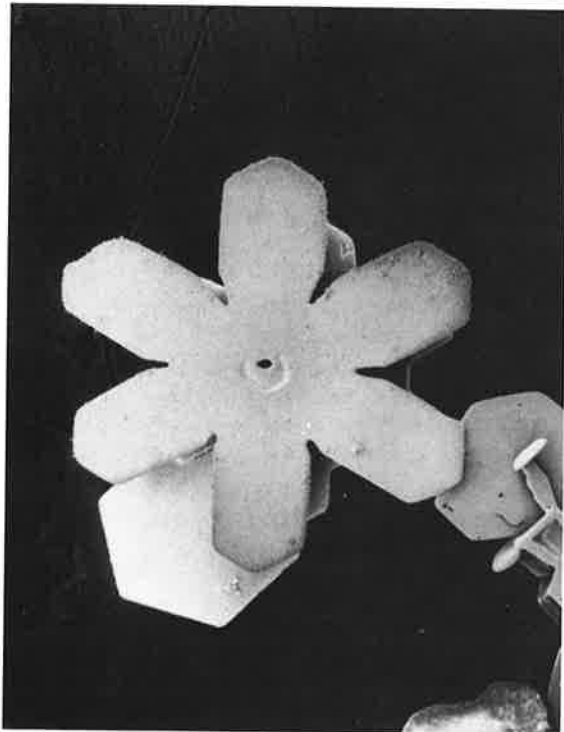


FIG. 3 A snow crystal with broad hexagonal branches is a variation of the hexagonal plate. In the center of the plate is a hole that may have contained the nucleation particle at the time of formation. (Collected at  $-5^\circ\text{C}$ , imaged at 5 kV.) Horizontal field width = 2.0 mm.



FIG. 2 Snowflake composed of numerous snow crystals. Crystals consisting of short columns are occasionally capped with the hexagonal plates (arrow). (Collected at  $-5^\circ\text{C}$ , imaged at 5 kV.) Horizontal field width = 2.3 mm.



FIG. 4 Irregularly shaped snow crystals are far more common than the symmetrical forms. However, even though the branches are nonsymmetrical, they do exhibit variations of the hexagonal plates. (Collected at  $-5^\circ\text{C}$ , imaged at 5 kV.) Horizontal field width = 1.8 mm.

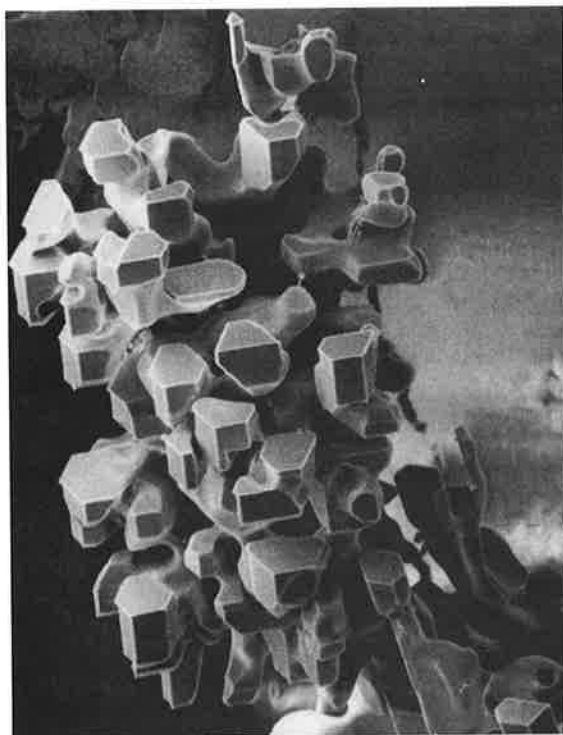


FIG. 5 Snowflake composed of short prismatic snow crystals. This aggregate of crystals appears to be held together by an amorphous non-crystalline matrix. (Collected at  $-6^{\circ}\text{C}$ , imaged at 5 kV.) Horizontal field width =  $600\ \mu\text{m}$ .

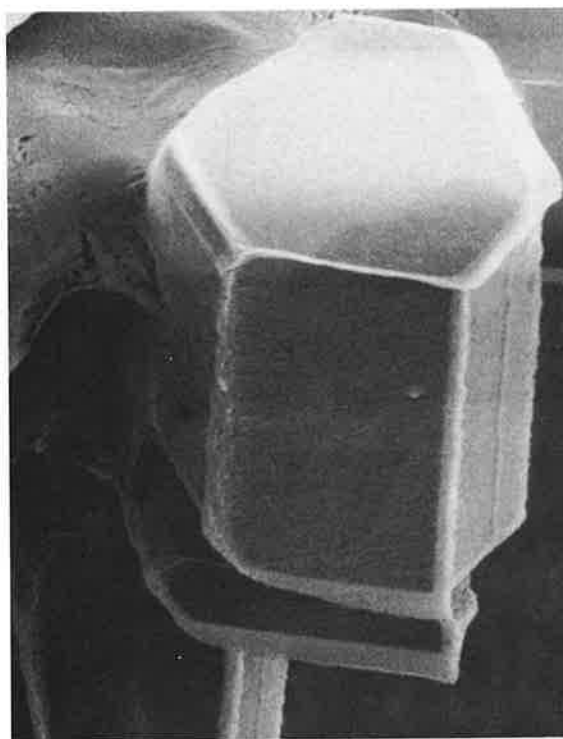


FIG. 6 Single short prismatic snow crystal and its continuity with the noncrystalline matrix illustrated in Figure 5. This figure illustrates that crystals can be observed and imaged at magnifications of several hundred times. (Collected at  $-6^{\circ}\text{C}$ , imaged at 5 kV.) Horizontal field width =  $90\ \mu\text{m}$ .

blage of plates is a nonsymmetrical aggregation in which two or three interconnected hexagonal plates appear to alternate at approximate right angles with other interconnected plates. The bullets consist of short columns that are tapered at one end. Three or four bullets are frequently joined at their tapered ends. The opposite end of the bullet, which is flat, is capped with an hexagonal plate that is centered on the top of the bullet and whose planer axis is perpendicular to that of the bullet. In the hexagonal plates, small holes, which may correspond to the nucleating particle, suggest that a single nucleation center may have given rise to the hexagonal plate as well as to its associated bullet.

Four distinct types of surface structures can be observed on snow crystals. At low magnification, the most common type of surface structure, which generally occurs along an edge of a snow crystal, appears as a fuzzy coating (Fig. 8). When this coating is resolved at higher magnifications, it appears as a fine, nonhexagonal crystalline covering, heterogeneous in structure and distribution, predominantly attached to the edge of a snow crystal (Fig. 9). This coating appears to correspond to "rime." 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 (Figs. 8 and 9) as well as on the edges of the columnar crystals, branched arms (Fig. 10), and needles (Fig. 11). The needles represent another subclass of snow crystals that consist of long, slender columns, frequently hollow and generally associated in irregular bundles (Fig. 11).

The dominant snow crystal illustrated in Figure 12 is a hexagonal plate with six radiating arms. However, associated with the surface of the central plate and extending into the arms are numerous spherical "droplets." These particles, which are about 0.03 to 0.05 mm in diameter, are consistent with the descriptions of cloud particles or supercooled water droplets that have been described in light microscopic examinations of snow crystals (Bentley and Humphreys 1931, Nakaya 1954). The radiating arms contain another type of surface structure, namely small microcrystals that consist of short hexagonal columns most of which are only 0.05 to 0.10  $\mu\text{m}$  in diameter. Some of these structures appear to be transitional stages that arise from the droplets.

The fourth type of surface structure that is observed on snow crystals appears as a thin amorphous coating (Fig. 13, arrows). This type of coating is observed on snow crystals that appear to have undergone some degree of "melting" because they do not exhibit the sharply delineated crystalline edges that are generally associated with snow crystals that were collected at sub-zero temperatures.

### 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 of the crystals in a snowflake, for example, needles,

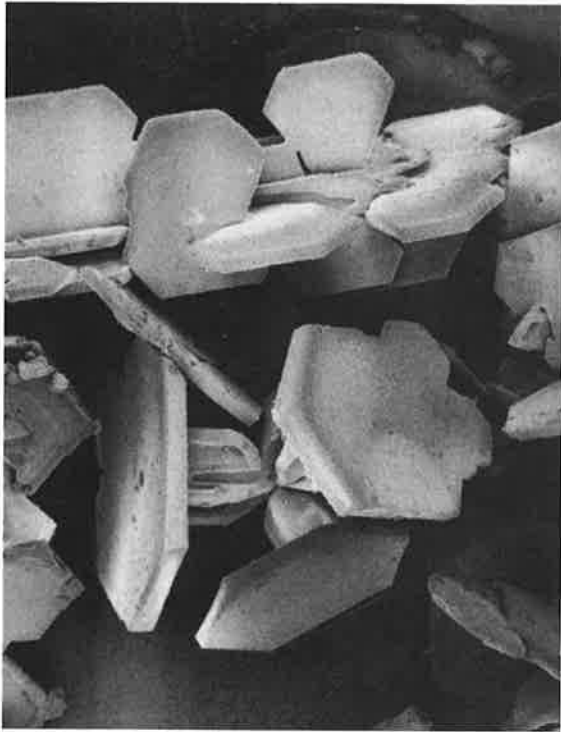


FIG. 7 Snowflakes consisting of an assemblage of hexagonal plates and several "bullets" capped with plates. The bullets appear to be joined at their tapered ends. (Collected at  $-5^{\circ}\text{C}$ , imaged at 5 kV.) Horizontal field width = 1.6 mm.

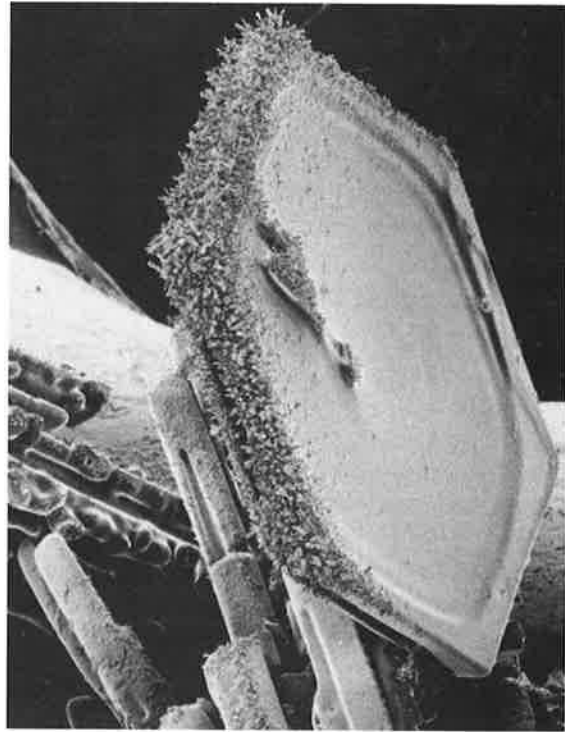


FIG. 8 Single hexagonal plate containing random accumulation of "rime" along its edge. (Collected at  $-6^{\circ}\text{C}$ , imaged at 5 kV.) Horizontal field width = 850  $\mu\text{m}$ .

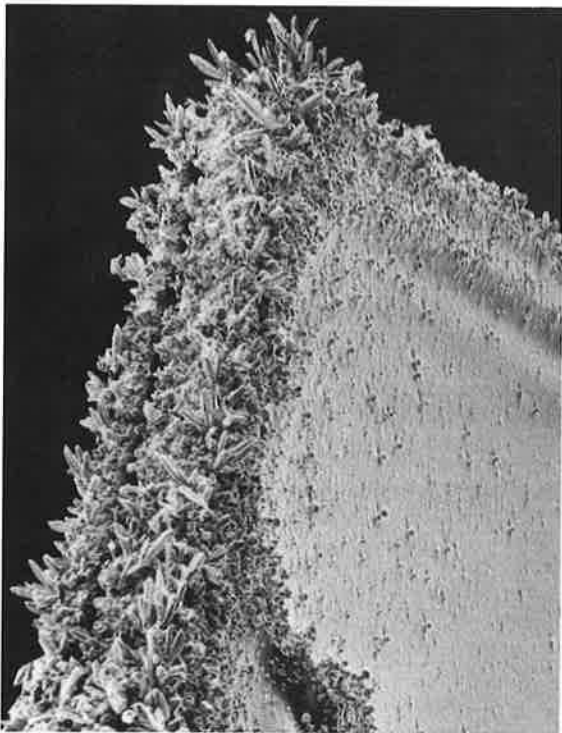


FIG. 9 Edge of the hexagonal plate illustrated in Figure 8. Rime appears as a fine, nonhexagonal, heterogeneous crystalline accumulation that generally collects along one edge of a snow crystal. (Collected at  $-6^{\circ}\text{C}$ , imaged at 5 kV.) Horizontal field width = 280  $\mu\text{m}$ .

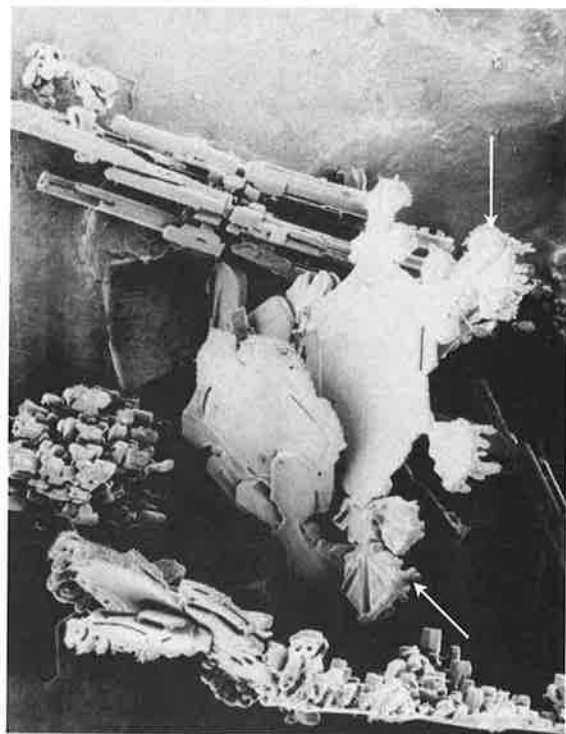


FIG. 10 Snowflakes consisting of needles, short columns, and branched hexagonal plates. Area showing rime is indicated by arrow. (Collected at  $-6^{\circ}\text{C}$ , imaged at 5 kV.) Horizontal field width = 3.0 mm.





FIG. 11 Needles consist of long slender columns that frequently have a hollow core. The needles generally are associated in irregular bundles. (Collected at  $-6^{\circ}\text{C}$ , imaged at 5 kV.) Horizontal field width = 1.1 mm.

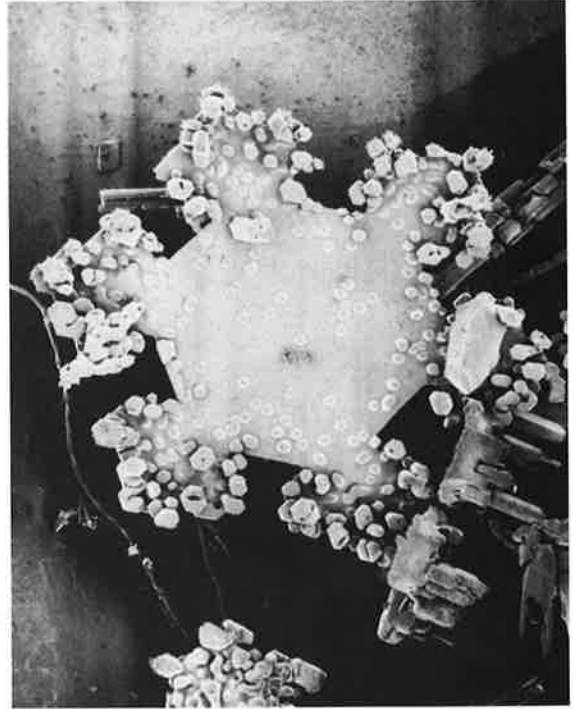


FIG. 12 Snow crystal consisting of a hexagonal plate with branches. The central portion of the crystal exhibits surface structures consisting of spherical "droplets," whereas the branches contain microcrystals that consist of short hexagonal columns, which may arise from the droplets. (Collected at  $-6^{\circ}\text{C}$ , imaged at 5 kV.) Horizontal field width = 2.3 mm.

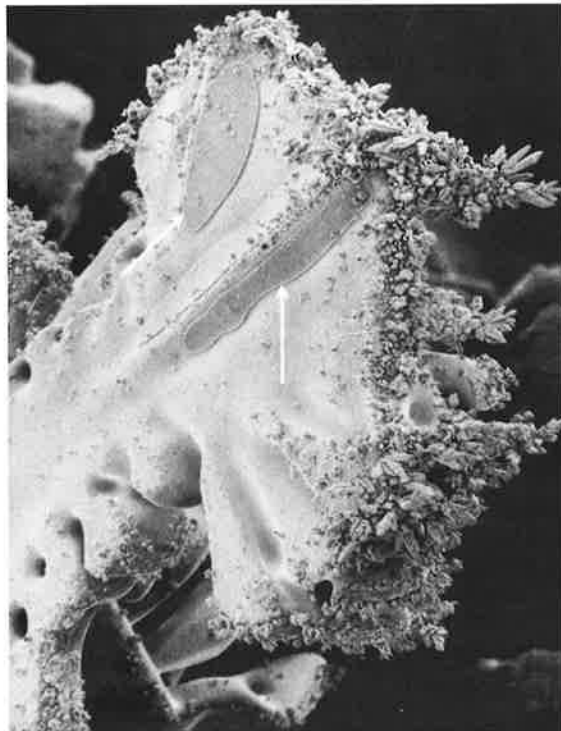


FIG. 13 Rimmed edge of hexagonal plate that also exhibits areas that contain a thin amorphous coating (arrows). Rounded edges of this snow crystal suggest that some melting may have occurred at some time during formation or descent. (Collected at  $-6^{\circ}\text{C}$ , imaged at 5 kV.) Horizontal field width = 420  $\mu\text{m}$ .



FIG. 14 Dendritic snow crystal 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  $0^{\circ}\text{C}$ , stored for 1 month in  $\text{LN}_2$ , imaged at 2 kV.) Horizontal field width = 2.9 mm.

columns, and dendrites, exhibit this phenomenon (Fig. 14). Melt-freeze metamorphism is characterized by a general lack of the sharply defined faces and angles that characterize a snow crystal (Fig. 14 and 15). Alternatively, the angular corners of the crystal become rounded and the surface flows from one face to the next (Fig. 15). Occasionally, a snow crystal is captured when melt metamorphism appears to be actively occurring in a unidirectional manner (Fig. 16). In this case, the same snow crystal will exhibit 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 the crystalline features. As the melting process continues, the snow crystal, which is gradually engulfed in an amorphous liquid matrix, loses its identity (Fig. 17).

Snow crystals illustrated in Figures 14 through 17 were examined after the samples had been collected, frozen, and stored in liquid nitrogen for 1 month. No indication of secondary formation of ice crystals or any other destructive structural changes could be associated with the storage procedure.

At 0°C, "wet snow" consists of snowflakes that are composed of aggregations of partly melted snow crystals or decomposed precipitation particles that are held together in what appears to be an amorphous liquid matrix (Fig. 18). At more advanced stages, the individual snow crystals are no longer recognizable, and the "snowflake" consists of an amorphous mass in which the angular intersects of individual crystalline faces are replaced by rounded protrusions of melting crystals (Fig. 19). The most advanced stage of this phenomenon consists of "wet snow" that was collected at 1°C and shows continuous spherical masses with no crystalline characteristics (Fig. 20).



FIG. 15 Branched end of metamorphosed dendritic crystal shown in Figure 14. The surface of the "crystal" appears to flow from one face to the next rather than forming sharp angular intersections. (Collected at 0°C, stored for 1 month in LN<sub>2</sub>, imaged at 5 kV). Horizontal field width = 1.0 mm.

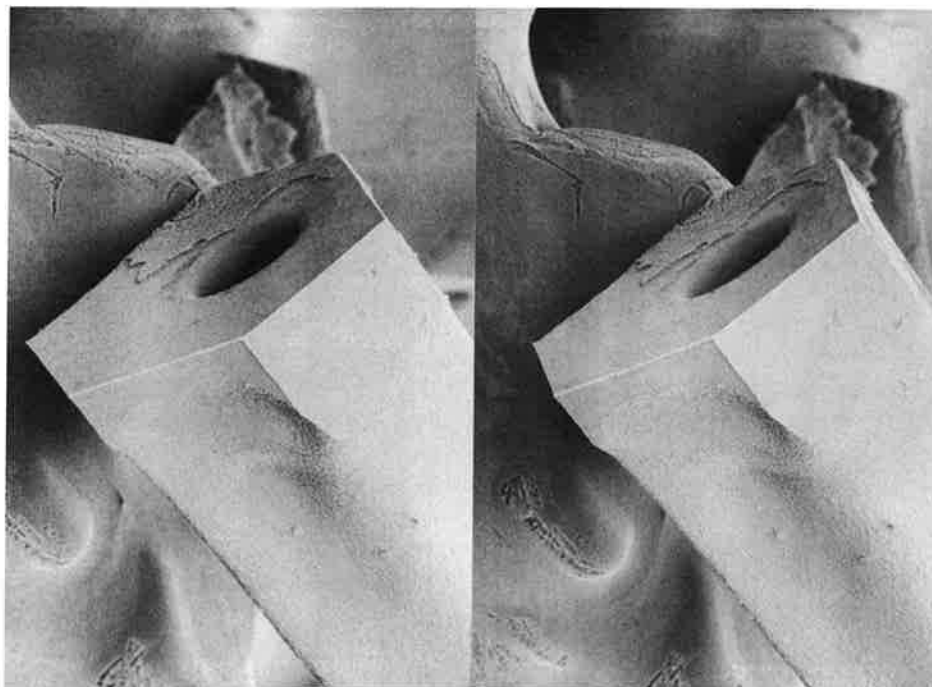


FIG. 16 Stereo pair illustrating the end of a hexagonal column. The column has round cylindrical core that is asymmetrically positioned in the crystal. Upper portion of the column shows the effects of unidirectional melt-freeze metamorphism while the end continues to exhibit the sharply defined crystalline characteristics of a snow crystal. (Collected at 0°C, stored for 1 month in LN<sub>2</sub>, imaged at 2 kV). Horizontal field width = 400 μm.

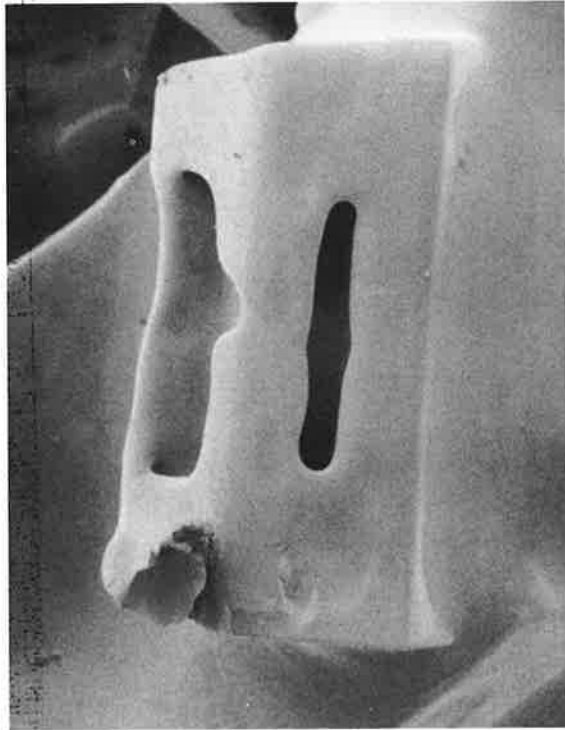


FIG. 17 Wet snow that consists of a column undergoing melt-freeze metamorphism. (Collected at 0°C, stored for 1 month in LN<sub>2</sub>, imaged at 2 kV.) Horizontal field width = 650 μm.



FIG. 18 Snowflakes consisting of aggregations of partly melted snow crystals held together in an amorphous matrix. Several microspheres (arrows) are associated with the surface of the melting snowflake. (Collected at 0°C, imaged at 5 kV.) Horizontal field width = 4.0 mm.



FIG. 19 At more advanced stages of melting, the individual snow crystals are no longer distinguished. The particle consists of a highly amorphous mass without any crystalline faces. (Collected at 0°C, imaged at 5 kV.) Horizontal field width = 1.6 mm.



FIG. 20 At the most advanced stage of melt-freeze, the wet snow consists of an aggregation of spherical masses that have no crystalline characteristics. (Collected at 1°C, imaged at 5 kV.) Horizontal field width = 1.4 mm.



## Discussion

Drawings of snowflakes based on light microscopic observations were first published by Robert Hooke in his book *Micrographia* in 1665; however, the first comprehensive and systematic light microscopic observations of snow crystals were made by Bentley who secured more than 4000 photomicrographs of snow crystals over a 37-year period (Bentley 1904, 1923; Bentley and Humphreys 1931). Bentley obtained his photomicrographs with transmitted illumination, whereby light passes through the snow crystals. This procedure provided information about the shapes and internal structure of the crystal, however details about the surface structure were limited. To reveal the surface structure of snow crystals, Nakaya (1954) used reflected illumination with a light microscope. The light microscopic studies by Bentley and Nakaya have provided valuable information about snow crystals, however their techniques required rapid collection and handling of snow crystals that could quickly metamorphose. For example, evaporation is a problem when the air is dry, humidity can cause riming, and subtle changes in temperature may result in melting or recrystallization. The approach that we have taken with low-temperature SEM appears to solve many of these limitations. The procedure not only allowed us to observe the same forms of snow crystals that had been previously described in light microscopic studies, but also enabled us to resolve the nucleation centers and other minute surface features easily. The specimens did not appear to be altered by the sputter coating. The snow crystals were stable in the electron beam, did not sublime, and could be magnified several thousand 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, Maryland, during the 1993–1994 winter season, was also capable of preserving melting snow or graupel. Furthermore, alternative holders allowed capture of the snowflakes and their storage in LN<sub>2</sub> until the specimens could be processed for examination in the SEM. Storage of samples in LN<sub>2</sub> had no obvious effects on the structure of the crystals. This observation is consistent with that of Wergin and Erbe (1991), who had used this procedure to store and subsequently observe frozen biological specimens. Finally, the specimen stage of the SEM allowed specimen tilt so that stereo images of the snow crystals could be recorded. This procedure has apparently not been demonstrated with the light microscope.

Through the years, several classification systems have been proposed to categorize snow crystals (Colbeck *et al.* 1990, Magano and Lee 1966, Nakaya 1954, National Research Council 1954, Shedd 1919). Of these, the system that was first proposed by the International Association of Hydrology, Commission on Snow and Ice, in 1951 and was more recently revised (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 the other shapes were observed and appeared to be consistent with the descriptions that were based on light microscopic observations. Therefore, sample preparation and observation with low-temperature SEM do not appear to alter the basic descriptions of snow crystals. The higher resolution that is possible with SEM will allow further detailed structural observations on nucleation, on surface features such as riming, and on changes associated with metamorphism.

The formation of snow crystals most commonly occurs by a process known as heterogeneous nucleation. This process, which occurs when temperatures are  $< -40^{\circ}\text{C}$ , results when aerosol particles (0.01 to 0.1  $\mu\text{m}$ ) cause the formation of ice through either the direct freezing of cloud droplets or the freezing of water deposited as a vapor onto the surface of a particle (LaChapelle 1969). Clay-silicate particles are one of the most common aerosols or ice-nucleating agents. However other particles can arise from industrial plants, forest fires, other organic matter, or extraterrestrial material. Artificial nucleation has also been accomplished by using a seeding agent such as dry ice or silver iodide.

In addition to the nucleation particles, snow can capture and transfer large quantities of other particulate matter from the atmosphere (Gray and Male 1981). Two important processes involved in this phenomenon are washout, in which atmospheric particles are "scavenged" by snow, and snowout in which atmospheric particles become attached or incorporated into the falling/developing snow crystal. Therefore, snow like rain can be used to sample the concentration of particulate matter and aerosols that are present in the atmosphere. Gray and Male (1981) indicate that because a snowflake falls at a slower rate than a rain drop and sweeps out a larger area, the snowflake will have had a greater exposure to pollutants and, therefore, would be a better indicator of their presence. Such particles have not been described with the light microscope. However, if low-temperature SEM were used in conjunction with an x-ray detector, analysis of foreign particles or nucleation centers, which range in size from 0.01  $\mu\text{m}$  to 0.01 mm, could probably be mapped with the SEM; the x-ray data would contribute qualitative elemental composition of these particles, thereby providing an important new tool for studying nucleation and acid snow.

## Conclusion

Low-temperature SEM has considerable potential as a viable technique for examining snow crystals at magnifications that far exceed the resolution of the light microscope. Furthermore, the ability to collect and store snow will enable investigators to accumulate samples from numerous locations or at different time intervals so that detailed observations and comparisons can be made in a convenient and orderly manner. In addition, this technique will allow investigators to compare core samples at different depths as well as to compare and contrast artificially made snow. The ability to add x-ray microanalysis to the low-temperature SEM would further allow one to gain information on the elemental composition of the nucle-

ation particles and any pollutants that were incorporated into the developing snow crystal. Future studies in our laboratory will focus on the shape, size, and structure of snow crystals in a snowpack that has experienced equi-temperature and temperature gradient metamorphism—factors that have a direct effect on the microwave remote sensing of snow.

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