THE EPITAXIAL GROWTH OF ICE ON SINGLE-CRYSTALLINE SUBSTRATES

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Abstract—Epitaxial deposits of ice crystals have been formed on the basal (0001) faces of hexagonal AgI, PbI₂, CuS, CdI₂ and brucite, on the (001) face of freshly-cleaved muscovite, on the (010) face of orthorhombic HgI₂, on the (001) face of orthorhombic iodine, on the (100) face of V₂O₅ and on the rhombohedral faces of calcite. The temperature at which ice crystals first appear on a particular substance is very close to the threshold temperatures at which an aerosol of the same substance nucleates a cloud of supercooled water droplets.

The crystals appear preferentially at cleavage and growth steps on the substrate surface, provided that these exceed ~ 0.1μ in height and preserve their parallel orientation irrespective of the direction of the step.

Thin, hexagonal ice plates, growing on the basal plane of covellite (CuS), show interference colours which give a measure of their thickness. Commonly, the ice crystals increase appreciably in diameter with no discernible change of thickness, suggesting that molecules arriving on the upper surface are not assimilated but migrate over the surface and are built in at the crystal edges. Crystals usually thicken on touching a neighbouring crystal; coloured growth fronts are then seen spreading out across the crystal surface from the point of contact.

Detailed measurements on AgI, PbI₂, CuS and CdI₂ show that, for a given substance, there is a well-defined critical temperature above which ice crystals can appear only if the air exceeds saturation relative to liquid water, but below which ice forms directly from the vapour phase provided that the supersaturation relative to ice exceeds 12 per cent for silver iodide and slightly different values for the other substrates. These critical supersaturations are required for growth at the edges of steps; ice appears on the flat parts of the substrate only at much higher supersaturations (> 100 per cent).

1. INTRODUCTION

Interest in the epitaxy of ice on foreign substrates stems mainly from the ability of crystals of certain natural minerals and of some inorganic compounds to nucleate supercooled water and to initiate ice-crystal formation in clouds of supercooled water droplets. The results of very careful tests on the ice-nucleating ability of finely-dispersed aerosols, carried out in both diffusion- and mixing-cloud chambers by MASON and HALLETT(1,2), MASON and MAYBANK(3), and MASON and VAN DEN HEUVEL(4), are summarized in Table 1. In each case the quoted threshold temperature is that at which about one particle in 10⁴ produces an ice crystal; for all the substances listed the activity gradually increases as the temperature falls below the threshold value.

It will be seen from Table 1 that, although there is a tendency for the more effective nucleators to possess hexagonal crystalline symmetry and lattice parameters reasonably close to those of ice, there are a number of exceptions; but for all the substances which nucleate at temperatures above -15°C, it is possible to find a low-index face on which the atomic spacings differ from those in either the basal or prism faces of ice by not more than a few per cent. If, in calculating the minimum misfit, one permits the matching of atomic rows in the ice and substrate lattices in ratios of up to 3:1, the values shown in heavy type in Table 1 are the misfits in the two most favoured of the three mutually perpendicular directions, (1210), (1010), (0001), of the ice lattice.

Although, in the literature, epitaxy is usually discussed in terms of the misfit in only the most favourable direction, it seems reasonable to regard the formation of two-/three-dimensional nuclei...
Table 1. Data on substances active as ice nuclei at temperatures above 

<table>
<thead>
<tr>
<th>Substance</th>
<th>Crystal symmetry</th>
<th>Lattice constants (Å)</th>
<th>Substrate plane</th>
<th>Misfit between substrate lattice in directions (per cent)*</th>
<th>Threshold temp. (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>a</td>
<td>b</td>
<td>c</td>
<td>(1210)</td>
</tr>
<tr>
<td>Ice</td>
<td>Hex.</td>
<td>4.52</td>
<td>7.36</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td></td>
<td>(a) Natural substances</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Covellite</td>
<td>Hex.</td>
<td>3.80</td>
<td>16.43</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Vaterite</td>
<td>Hex.</td>
<td>4.12</td>
<td>8.56</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>β-Tridymite</td>
<td>Hex.</td>
<td>5.03</td>
<td>8.22</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Kaolinite</td>
<td>Pseudo-hex. (OH groups)</td>
<td>2.98</td>
<td>7.08</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Hematite</td>
<td>Hex.</td>
<td>5.03</td>
<td>17.35</td>
<td>(001)</td>
<td>—</td>
</tr>
<tr>
<td>Brucite</td>
<td>Hex.</td>
<td>3.11</td>
<td>4.74</td>
<td>(001)</td>
<td>—</td>
</tr>
<tr>
<td>Cinnabar</td>
<td>Hex.</td>
<td>4.14</td>
<td>9.49</td>
<td>(001)</td>
<td>+</td>
</tr>
<tr>
<td>Graphite</td>
<td>Hex.</td>
<td>2.46</td>
<td>6.70</td>
<td>(001)</td>
<td>+</td>
</tr>
<tr>
<td>(b) Inorganic crystals</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>AgI</td>
<td>Hex.</td>
<td>4.58</td>
<td>7.49</td>
<td>(001)</td>
<td>+</td>
</tr>
<tr>
<td>PbI₂</td>
<td>Hex.</td>
<td>4.54</td>
<td>6.86</td>
<td>(001)</td>
<td>+</td>
</tr>
<tr>
<td>CuS</td>
<td>Hex.</td>
<td>3.80</td>
<td>16.43</td>
<td>(001)</td>
<td>—</td>
</tr>
<tr>
<td>HgI₂</td>
<td>Tetr.</td>
<td>4.36</td>
<td>4.36</td>
<td>12.34</td>
<td>(001)</td>
</tr>
<tr>
<td>O. Rhombic</td>
<td>4.67 13.76</td>
<td>7.32</td>
<td>(010)</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>Ag₂S</td>
<td>Monoclinic β = 55°</td>
<td>4.20</td>
<td>6.93</td>
<td>9.50</td>
<td>(010)</td>
</tr>
<tr>
<td>NH₄F</td>
<td>Hex.</td>
<td>4.39</td>
<td>7.02</td>
<td>(001)</td>
<td>-</td>
</tr>
<tr>
<td>V₂O₅</td>
<td>O. Rhombic 11.48</td>
<td>4.36</td>
<td>3.55</td>
<td>(001)</td>
<td>-</td>
</tr>
<tr>
<td>Ag₂O</td>
<td>Cubic</td>
<td>4.72</td>
<td>4.36</td>
<td>3.55</td>
<td>(100)</td>
</tr>
<tr>
<td>CdI₂</td>
<td>Hex.</td>
<td>4.24</td>
<td>6.84</td>
<td>(001)</td>
<td>-</td>
</tr>
<tr>
<td>MnO₂</td>
<td>Tetr.</td>
<td>4.44</td>
<td>4.44</td>
<td>2.89</td>
<td>(010)</td>
</tr>
<tr>
<td>T₉</td>
<td>O. Rhombic 4.78</td>
<td>7.25</td>
<td>9.77</td>
<td>(001)</td>
<td>+</td>
</tr>
</tbody>
</table>

* The percentage misfit \( \Delta = ((\delta_r - \delta_i)/\delta_i) \times 100 \), where \( \delta_i \), \( \delta_s \) are the spacings of those atomic rows, in ice and the substrate which most nearly coincide, with the restriction that \( \delta_i \) does not exceed three atomic spacings. If, for example, alternate rows in the substrate are matched to every row in ice, this is shown by the subscript (2:1).

in terms of the misfit in two/three mutually perpendicular directions. The misfit in the third direction is therefore given in lighter type in Table 1. For the rather complex mineral structures, the geometrical similarity between the ice and substrate lattice, as expressed by the misfit in one or more directions combined, is not highly correlated with the nucleating ability as indicated by the threshold temperature. In the case of the simple inorganic structures, however, a reasonable correlation appears to exist between the threshold nucleation temperature (which enters explicitly into the expression for the critical size of the ice nucleus) and the sum of the misfits in the three mutually perpendicular directions. Thus it appears that, as for most other substances, the epitaxial growth of ice on foreign substrates is determined only partly by geometrical factors and overall by the configuration and intensity of the surface force field of the substrate and its interaction with the first layers of the deposit.

In order to study in more detail the orientation of ice crystals on well-defined faces of single-crystalline substrates, their growth has been
directly observed under carefully controlled conditions of temperature and supersaturation of the vapour. Although epitaxial deposits of ice have been observed on AgI, PbI₂ and mica by Montmory(5), and Jaffray and Montmory(6,7), on AgI and PbI₂ by Mason(8) and on AgI, PbI₂, CdI₂ and biorite by Kleber and Weis(9) the conditions under which they appeared were not studied in detail.

In the present experiments, orientation of ice has been observed on the basal (0001) faces of hexagonal AgI, PbI₂, CuS, CdI₂ and brucite, on the (001) face of freshly-cleaved muscovite, on the (010) face of orthorhombic HgI₂, on the (001) plane of orthorhombic iodine, on the (100) plane of V₂O₅ and on the surfaces of rhombohedral calcite.

2. EXPERIMENTAL TECHNIQUES

The apparatus, shown in Fig. 1, is a modified version of that used by Shaw and Mason(10) to study the growth of ice crystals on a polycrystalline metal surface. The metal surface supporting the substrate crystal is situated in the centre of a lagged, cylindrical metal chamber, the hollow walls of which may be cooled down to −50°C by circulating petrol chilled with solid carbon dioxide through the annular space. The temperature of the chamber walls is controlled by a heating coil wound on the copper tube carrying the coolant where it leaves the cooling bath. The base of the chamber is coated with ice, so that the air in the experimental space (about 7 cm diameter × 1 cm deep), which is stirred by a small electric fan, is uniformly saturated with respect to ice at a temperature T₁ indicated by a thermocouple attached to the surface of the ice layer. The temperature over the chamber floor does not vary by more than 0.2°C and the thermocouple is placed half-way out along the radius.

The substrate crystal rests on the end surface of a long copper rod which is insulated from the chamber and dips into a Dewar flask containing liquid air. The crystal is thus cooled to a lower temperature, T₂, than that of the surrounding air, and this is controlled by varying the current through the heating coil wound on the copper rod. The temperature T₂ of the thin substrate crystal is recorded by a fine thermocouple immediately below the surface of the copper rod, and the saturation ratio of the air in the neighbourhood of the growing ice crystals is altered by adjusting the temperatures T₁, T₂, and is measured by the ratio of the equilibrium vapour pressures of ice at these two temperatures.

The reliability of the supersaturation measurement was checked by using the apparatus as a dew-point hygrometer; for a fixed value of T₁, the temperature T₂ of the copper surface was adjusted until a deposit of very small water droplets began to form or disappear, and it was confirmed that, at this point, the measured temperatures showed the air to be just saturated relative to liquid water. Moreover, ice crystals grown as hexagonal plates on the substrate began to evaporate at the corners when the indicated temperatures showed the air to be just saturated with respect to a plane surface of ice.

The crystals were viewed through a microscope fitted with a vertical illuminator, an 8-mm objective and ×20 eyepiece being used to give a linear magnification of 350.

3. OBSERVATIONS OF EPITAXIAL GROWTH

(a) On Silver iodide

The specimens were prepared by Pashley's (see Newman and Pashley(11)) technique of evaporating silver on freshly-cleaved mica in vacuo,
the (111) faces of silver then being attacked, in darkness, by the vapour from a weak solution of iodine. This produces a very thin layer in which (0001) faces of hexagonal silver iodide and (111) faces of the cubic form are parallel to the surface, both forms giving an identical hexagonal array of iodine ions.

Epitaxial deposits of ice crystals growing on their basal planes were obtained at temperatures below \(-4^\circ\mathrm{C}\); at higher temperatures only water droplets appeared. At temperatures between \(-8^\circ\mathrm{C}\) and about \(-25^\circ\mathrm{C}\) the crystals grew preferentially along the secondary axes to form thin plates (Fig. 2), but at temperatures between \(-4^\circ\mathrm{C}\) and \(-8^\circ\mathrm{C}\), and also below \(-25^\circ\mathrm{C}\), growth proceeded preferentially in the direction of the \(c\)-axis, normal to the surface (Fig. 3), in accordance with variation of habit with temperature observed by Hallett and Mason for freely-growing ice crystals. The crystals showed a strong tendency to form on specific sites (see Fig. 4), which at this stage of development darkened with increasing exposure to light. They appear to be preferred centres for the decomposition of silver iodide to metallic silver and may therefore mark the sites of emergent dislocations.

Prolonged exposure to a strong source of ultra-violet light leads to a rapid decomposition of the silver iodide to metallic silver. After several seconds’ exposure to the beam from a 250-W high-pressure mercury-arc lamp, the surface begins to deteriorate and the oriented crystals become more sparse. After further exposure, on a surface which is now dark and discoloured, it becomes very difficult to form an ice deposit; the condensate appears in the form of droplets, 5–10\(\mu\) in diameter, which may be supercooled down to temperatures as low as \(-30^\circ\mathrm{C}\) and which then freeze to produce needle-like crystals with random orientation. A more detailed account of measurements on the photolytic decomposition of silver iodide is to appear elsewhere.

In this context it is relevant to refer to the claim by Montmory to have observed epitaxy of ice on the (111) face of metallic silver. We are unable to confirm this observation. On clean, fresh surfaces of silver we are able to form only water droplets at temperatures above about \(-25^\circ\mathrm{C}\); at lower temperatures the droplets freeze and develop into randomly oriented prisms, as shown in Fig. 5, or needles, as shown in Fig. 6. But, after exposure for a day or two to room air, the silver surface shows a progressive tendency to produce oriented crystals. At first these take the form of widely separated, irregular, plate-like crystals following the freezing of droplets at temperatures below \(-10^\circ\mathrm{C}\); with even longer exposures denser deposits of regular hexagonal plates appear direct from the vapour phase. Often two distinct orientations are observed, as in Fig. 7. It was suspected that this activation of the silver surface was due to contamination by traces of iodine vapour and the formation of localized patches of silver iodide, because the activity of the latter is easily destroyed by exposure to ultra-violet light. An electron-diffraction examination of the silver surface confirmed the existence of oriented layers of silver iodide, and a second substance which would appear to be silver sulphide.

(b) On lead iodide

Lead iodide crystals, up to 4 mm in diameter, were produced by selection from a commercial preparation, by growth of very thin hexagonal plates from a cooling solution or by selection of crystals from the surface of a solidified melt. At temperatures above \(-6^\circ\mathrm{C}\) the initial deposit consisted of liquid water droplets which, at lower temperatures, froze and developed into ice crystals with \(a\)-axes parallel to those of the lead iodide, their habit varying with temperature as described above for growth on silver iodide.

The crystals grown from solution mostly had flat, featureless faces on which ice crystals rarely appeared except at very high vapour supersaturations exceeding 100 per cent. The tendency was for the ice to appear in irregular aggregates round the edges of these crystals (see Fig. 8), but regular, oriented ice crystals readily appeared along steps which were artificially induced by the overlapping of the thin lead iodide plates. Many of the lead iodide crystals (1 mm in diameter) exhibited trigonal growth spirals whose steps were not usually parallel to the edges of the crystal. Although there was no marked tendency for ice crystals to appear at the edges of the shallow steps (< 500 \(\AA\) high) in Fig. 9, they did show a marked preference for the edges of steps higher than about 0.1\(\mu\) at which they assumed the orientation of the crystal edge rather than that of the step.

After melting and evaporation of the ice-crystal deposit, the nucleating activity of the surface was
greatly reduced—a consequence, no doubt, of lead iodide being slightly soluble in water.

(c) On cadmium iodide

Crystals of up to 5 mm in diameter were produced from a solution of cadmium iodide saturated at 60°C and slowly cooled to room temperature. At temperatures above −12°C the condensate was in droplet form; between −12 and −20°C the drops froze without forming regular ice crystals; well-defined crystals appeared only below −20°C.

Whereas cadmium iodide crystals precipitated from a rapidly-cooled solution exhibited shallow, closely-packed, circular growth steps which did not seem to encourage the nucleation of ice crystals, slow cooling produced higher, more widely-spaced steps of hexagonal symmetry at which ice crystals appeared preferentially, as shown in Fig. 10. The distortion of the ice crystals from their hexagonal shape may be explained by asymmetric growth caused by the anisotropic arrival of water molecules.

Melting and evaporation of the ice deposit again destroyed the nucleating properties of the surface and, in this case, left visible damage because of the high solubility of cadmium iodide.

(d) On cupric sulphide

The substrate was prepared by cleavage of the mineral covellite parallel to the basal plane. At temperatures above −6°C water vapour condensed on the covellite surface as droplets, but at lower temperatures ice crystals appeared with their a-axis at 30° to those of the cupric sulphide, this relative orientation giving a much closer atomic fit between the deposit and the substrate, as shown in Fig. 11. In Fig. 12(a) an oriented deposit of thin, hexagonal ice plates, shown in reflected white light, appear on the blue substrate crystal. In the early stages of growth the crystals, being only a few thousand Angstroms high, show interference colours which give a measure of their thickness. Comparison of Figs. 12(a) and (f) shows that, at first, some crystals (e.g. No. 2 in key) increase noticeably in diameter with no discernible change of thickness. These observations, together with other evidence to be published in detail elsewhere, suggest that molecules arriving on the upper surface of the crystal are not assimilated, but migrate over this surface and are built in at the edges.

Referring to crystals numbered 1–5 in the key, whilst No. 2 remains constant in colour, the others undergo rapid change, showing that thickening is taking place. (One order of colour change is about 0.15μ.) Thickening of a crystal is frequently initiated by contact with another crystal of greater thickness. This is shown in the sequence Fig. 13(a)–(f), where crystals 2, 4, 6 initiate growth layers on crystals 1, 3, 5 as soon as contact occurs.

There is a marked tendency for ice crystals to cluster along cleavage steps on the substrate and to maintain their orientation relative to the substrate lattice irrespective of the direction of the step. A crystal sitting astride a step may be of different thickness on either side, as indicated by the changes of colour in the individual crystals of Fig. 12.

The crystal originating at the large steps in Fig. 12 (crystals No. 7) have grown much more rapidly than those on the flatter areas of the substrate. Here, the crystal thickness having reached several wavelengths, the interference colours have disappeared to be replaced by a uniform pink coloration. Again it is noteworthy that the very shallow steps, only a few hundred Angstroms high, although able to cause a colour change in an overlying ice crystal, do not act as preferred nucleation sites.

(e) On iodine

On the (001) faces of iodine, oriented deposits of ice crystals appeared with both basal and prism faces in contact with the substrate as shown in Fig. 14. The atomic configurations for both orientations are shown in Fig. 15. At temperatures above −7°C the deposit consists entirely of water droplets, among which an occasional ice crystal may appear at temperatures between −7 and −10°C. As the temperature falls below −10°C the number of crystals rapidly increases with no visible liquid deposit. While at higher temperatures the crystals lie preferentially on their prism faces, the other orientation becomes more frequent at lower temperatures and is equally prominent below −20°C. The misfits shown in Table 1 suggest that, of the two orientations, that involving the prism face will be slightly favoured.
(f) *On other substances*

Orientation of ice crystals has also been observed on the (0001) face of brucite, the (010) face of mercuric iodide, the rhombohedral faces of calcite and the (001) face of muscovite, which must be freshly cleaved to avoid deterioration of the surface by the rapid adsorption of impurities.

4. THE ROLE OF SUPERSATURATION

In all the preliminary experiments it was evident that the supersaturation of the water vapour, as well as the temperature, influenced the nucleation and growth of the ice deposit. To investigate this in more detail, careful measurements were made, as a function of temperature, of the minimum supersaturations required to produce oriented ice crystals. The supersaturation was calculated from the measured temperatures as described in Section 2. The temperatures were determined to within ±0.1°C, the corresponding error in supersaturation being ±1 per cent.

The results for silver iodide are shown in Fig. 16. At temperatures above −4°C only water droplets appeared. As the temperature was lowered from −4 to −12°C, increasing numbers of ice crystals formed on selected sites, provided that the air surpassed saturation relative to liquid water. At temperatures below −12°C, however, crystals appeared when the air was subsaturated relative to water but supersaturated with respect to ice by at least 12 per cent. These observations suggest that, at temperatures between −4 and −12°C, the initial deposit may have been liquid water, perhaps in droplets too small to be seen before they froze, while at temperatures below −12°C crystals may appear by sublimation direct from the vapour phase.

A similar behaviour has been observed for an aerosol of silver iodide introduced into a cloud chamber. Similar results were obtained for cupric sulphide and lead iodide with critical supersaturations of 13 and 15 per cent at −13 and −15°C, respectively.

![Figure 17](image-url)

*Fig. 17. The conditions of temperature and supersaturation at which oriented ice crystals appeared at steps on the basal plane of a cadmium iodide crystal.*
The behaviour of cadmium iodide was notable in that, at temperatures between -12 and -21°C, the initial deposit was in the form of visible droplets which subsequently froze into irregular crystals. Only at temperatures below -21°C, with the air supersaturated relative to ice by at least 22 per cent (see Fig. 17), did regular oriented ice crystals appear.

In all cases except silver iodide, on which steps were probably present though not visible, the supersaturations of 10–20 per cent were those required to produce ice crystals at the edges of large visible steps; nucleation on the flat areas of the substrate occurred only at much higher supersaturations which could not be accurately determined but certainly exceeded 100 per cent.

5. CONCLUDING REMARKS

Oriented ice crystals deposited from a supersaturated vapour have been observed on the faces of a number of crystals (not necessarily hexagonal) in which the atomic arrangement is similar to that of ice. The nucleating ability of the substrate cannot, however, be completely specified by the degree of misfit, but may be characterized either by the highest temperatures at which it nucleates water droplets or by the minimum supersaturation required for ice crystals to appear direct from the vapour at preferred sites, such as steps. Of course, either the minimum degree of supercooling or the minimum supersaturation, depending upon the operative mechanism, enters explicitly into the expression for the radius of the critical nucleus.

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REFERENCES

Fig. 2. A deposit of oriented, hexagonal, plate-like ice crystals growing at $-15^\circ$C on a substrate of silver iodide.

Fig. 3. Oriented ice crystals growing at $-30^\circ$C on silver iodide; these crystals grew preferentially along the principal axes, normal to the substrate, to form prisms.
Fig. 4. Hexagonal ice plates forming at special sites on the silver iodide surface. Temperature $-15^\circ$C.

Fig. 5. Randomly oriented ice prisms growing on a clean, fresh silver substrate at $-25^\circ$C.
FIG. 6. Randomly oriented ice needles growing along a step on a clean silver surface.

FIG. 7. A sparse deposit of ice crystals, with two different orientations, growing on a contaminated silver surface. Oriented crystals occurred only in patches where silver iodide was produced by adsorption of iodine vapour from the atmosphere.
Fig. 8. Ice crystals growing round the edges of lead iodide crystals but not on the surface which is covered by shallow spiral growth steps 500 Å high.

Fig. 9. Oriented ice crystals originating at the edges of deep steps (> 0.1μ) on the basal plane of lead iodide. Again there is no preferential growth at the shallow growth steps.
Fig. 10. An epitaxial deposit of ice crystals growing at the steps of a hexagonal growth spiral on cadmium iodide. Again a dense deposit forms along the edge of the crystal.
Cu atoms in the (0001) plane of CuS
(H2O molecules in the (0001) plane of ice

Fig. 11. The relative atomic positions in the basal planes of cupric sulphide and ice.

Fig. 12a
Fig. 12. Epitaxial deposit of ice crystals on the basal plane of a covellite (CuS) crystal. The interference colours provide a measure of the crystal thickness. Further details are given in the text and key in Fig. 12a, opposite.
FIG. 13. A time sequence showing the initiation and spread of growth layers across the surfaces of ice crystals growing on a covellite base. Key in Fig. 13a opposite.
FIG. 14. Oriented ice plates and prisms on the (001) face of an iodine crystal.
FIG. 15. The atomic configuration on the (001) face of iodine relative to that in the basal and prism planes of ice.

FIG. 16. The conditions of temperature and supersaturation at which oriented ice crystals appeared at special sites on the surface of a silver iodide crystal.