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APPLICATION OF COMMON PLASTIC SOLUTIONS FOR ICE CRYSTAL REPLICATION

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Résumé - L'utilisation de plastiques usuels tels que polystyrène, plexiglass (polymethyl methacrylate) et Lescan (polycarbonate) a été étudiée pour faire des répliques de cristaux de glace. Ces résultats suggèrent que tous les plastiques testés sont utilisables pour cela avec des combinaisons appropriées de solvants. Les associations de plexiglass et de dichloro-éthylène ainsi que de polystyrène et de 1,1,1,-trichloroethane sont préférables aux autres. La méthode par vaporisation n'est pas appropriée pour reproduire les détails de très petits cristaux de glace.

Abstract - Use of common plastics, i.e., polystyrene, Plexiglas (polymethyl methacrylate) and Lexan (polycarbonate), was investigated for ice crystal replication. The results suggest that all common plastics tested are usable for ice crystal replication with proper combinations of solvents. The combinations of Plexiglas and ethylene dichloride, and polystyrene and 1,1,1-trichloroethane are advantageous over others. The vapor method is found, in general, not suitable for replicating details of minute ice crystals.

I - INTRODUCTION

The ethylene dichloride or chloroform solution of Formvar (polyvinyl formal) has been useful for replicating snow crystals in laboratory or field for later analysis /1/. This plastic film would reproduce detailed features of snow crystal surfaces. The basic idea behind this method is to let a polymer solution, both the solvent and solute insoluble in water, surround the crystals and the solvent to evaporate and harden well before the crystals dissappear. The Formvar method is common not only in cloud physics but also in electron microscopy. The replication method is used for studying electron opaque materials, such as metals or glass fibers which are unsuitable for direct or thin section evaluation with an electron microscope /2/.

With the Formvar method, there are several disadvantages known. As Formvar contains polyvinyl alcohol (>5%) and polyvinyl acetate (>9%) /3/, which are soluble in water and insoluble in ethylene dichloride, and is ordinarily powdery, it absorbs water vapor and on occasion the solution gets murky at low temperatures. Ethylene dichloride cannot be used as the solvent below its melting point (-35.7°C) and chloroform is known to be carcinogenic. Spurious ice crystals may sometimes form due to the crystallization of impurity water in ethylene dichloride /4/. For replication of small crystals, methyl 2-cyanoacrylate monomer, an instant adhesive, is also available /5/, but exposure of an ice crystal surface to this vapor gives rise to the growth of secondary fibers in the direction of the c-axis of the crystal /6/.

To surmount the disadvantages of the Formvar method, we examined common plastics produced commercially with popular solvents.

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II-EXPERIMENT

Four plastics, i.e., polystyrene, Plexiglas (polymethyl methacrylate), Lexan (polycarbonate) and Formvar, were tested for ice crystal replication by the following three methods: The first is the liquid method /1/ in which snow crystals are received on a glass slide coated with a cold, 1 to 15 percent plastic solution. After the solvent has evaporated, snow crystals leave the replicas in the thin plastic film. The slide is kept cold for more than a half day to allow the ice to sublime. In the case of a larger crystal or an aggregate of snow crystals, a few drops of the solution are placed on it after a sample is received on a glass slide.

The second is the vapor method /4/ believed to be useful for ice crystals up to about 500 µm in size. After crystals have been deposited on a glass slide precoated with a dry plastic film, the glass slide is exposed for a few seconds to the warm saturated vapor of the proper solvent which dissolves the film by condensation to surround the crystals and evaporates to harden the film which leaves the crystal imprints.

The third is a direct observation of ice crystals received on a glass slide under an optical microscope with subsequent photomicrography.

Solvents applied in combination with the four plastics were methylene chloride, 1,1,1-trichloroethane, carbon tetrachloride, ethylene dichloride, trichloroethylene, and toluene. Table 1 shows the compatibility between the plastics and the solvents, and solvent properties: the melting point, the water solubility, and the toxicity of each solvent /7/. Vapor pressure of each solvent, based on /8/, is shown in Fig. 1. Polystyrene easily dissolves in all solvents examined although its prolonged exposure to sunlight causes yellowing /9/. Plexiglas possesses excellent fastness to light and resistance to weathering. Lexan is only affected by ultraviolet light. Addition of water by placing small pieces of ice in these solutions hardly makes them murky in contrast to the Formvar solution because these plastics

Table 1. Compatibility between the plastics and the solvents, and solvent properties

Plastic	Solvent					
	Methylene chloride	1,1,1-Trichloroethane	Carbon tetrachloride	Ethylene dichloride	Trichloroethylene	Toluene
Polystyrene	0	0	0	0	0	0
Plexiglas (Polymethyl methacrylate)	0	x	x	0	0	x
Lexan (Polycarbonate)	0	x	x	x	x	x
Formvar (Polyvinyl formal)	0	x	x	0	x	x
Freezing point (°C)	-95.1	-30.4	-23.0	-35.7	-86.4	-95.0
Water solubility (%w)	$0.20^{25°C}$	$0.034^{25°C}$	$0.01^{24°C}$	$0.15^{20°C}$	$0.03^{25°C}$	$0.033^{25°C}$
Threshold limit value in air (ppm)	500	350	10	50	100	200

0: suitable combination x: unsuitable combination

do not include a water soluble ingredient. Methylene chloride is suitable as the solvent of the vapor method because the vapor pressure is 5 times higher than that of ethylene dichloride. Therefore, ice crystals are not affected by a shorter time exposure under a relatively high temperature. However, it is not suitable to prepare a plastic film on a glass slide for the vapor method because the thickness of the film is not uniform due to water vapor condensation on the surface which is cooled down by its evaporation. 1,1,1-trichloroethane has less toxicity than other solvents and is commercially available as a correcting fluid thinner of Liquid Paper Corporation, (U.S.A.). Carbon tetrachloride is a toxic substance. Trichloroethylene is affected by sunlight. As the water solubility in 1,1,1-trichloroethane or carbon tetrachloride is less than 1/5 of that in ethylene dichloride and the vapor pressure of each solvent is nearly equal to or larger than that of ethylene dichloride, snow crystals can hardly dissolve into the solution and spurious ice crystal growth is prevented. As the melting point of methylene chloride and toluene are -95.1 and -95°C, respectively, these solvents can be used in a wide temperature range.

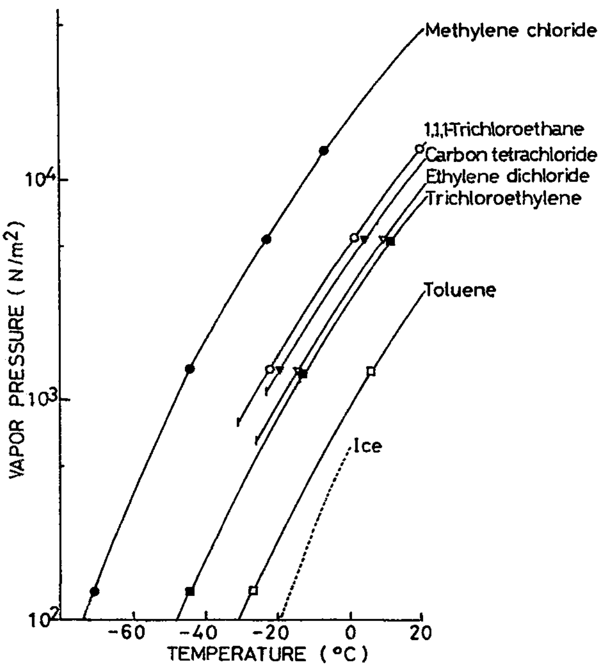

Fig. 1 Vapor pressure of solvents

III-RESULTS

Figure 2 shows a replica of a natural snow crystal: (a) before replication, (b) after replication by the liquid method using 10 percent solution of Plexiglas in ethylene dichloride. The replica preserves the surface structures of the snow crystal excellently. 5 to 10 parts of Plexiglas dissolved in 100 parts of ethylene dichloride made a proper solution. The solution above 10 percent was too viscous.

In case of the solution below 5 percent deformation of crystals was observed during replication (see Fig. 3). Figure 4 also shows examples using other plastics by the vapor method: (a) polystyrene (carbon tetrachloride), (b) Lexan (methylene chloride), and (c) Formvar (methylene chloride). These plastics compare favorably With Formvar for making replicas of ice crystals.

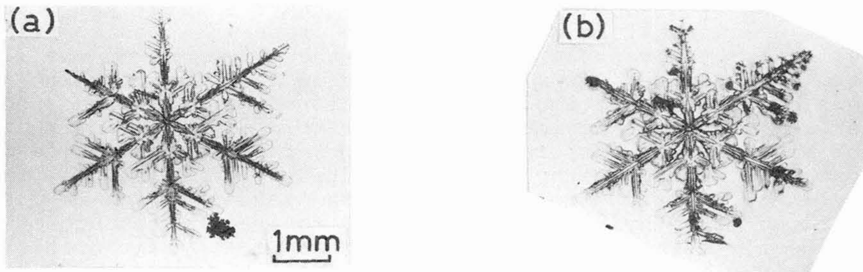

Fig. 2. Snow crystal before (a) and after (b) replication by the liquid method using 10 percent solution of Plexiglas dissolved in ethylene dichloride.

A solvent must be chosen carefully. Toluene is not suitable because the evaporation of toluene is so slow that ice crystals are deformed very frequently (see Fig. 5), even though water solubility in toluene is lower than that in ethylene dichloride. Extreme care must be taken to avoid occurrence of uncontrolled growth of crystals, especially at their corners and edges (see Fig. 6). Protrusions occurred rapidly when sources of water vapor in air coexist with evaporating methylene chloride during replication. These protrusions became quite noticeable when methylene chloride was used as the solvent regardless of the replication methods, i.e., the liquid method and the vapor method. This phenomenon is apparently due to secondary replication. Ice crystal surfaces, especially corners and edges, are cooled down by the evaporating solvent. The continuously depositing water vapor on the ice crystal surface helps the corners grow in the plastic solution. After the solvent evaporates, the replica of spurious growth shows up. The matter may be eased by quickening evaporation of methylene chloride, i.e., shaking a glass slide after replication in a low water vapor environment. Other cautions must also be taken with the vapor replication method. A dry, thick film precoated on a glass slide results in its cracking after vapor supply because of swelling. A more serious problem is that details of ice crystal surfaces may fail to show up in the replication process, as shown in Fig. 7. This is probably because the plastic solution did not creep up sufficiently and the condensed solvent vapor somehow failed to get in between the crystal and the dry film, possibly due to air locking. Thus, the vapor method is not suitable for replicating details of small ice crystals although it is useful for investigating the number and the approximate size of ice crystals. To observe the details, the plastic solution can be dropped on the crystals, but small crystals tend to become round at corners due to some water solubility in the solvent. For this purpose 1,1,1-trichloroethane or carbon tetrachloride is the most suitable solvent because of its low water solubility and proper vapor pressure (see Fig. 8).

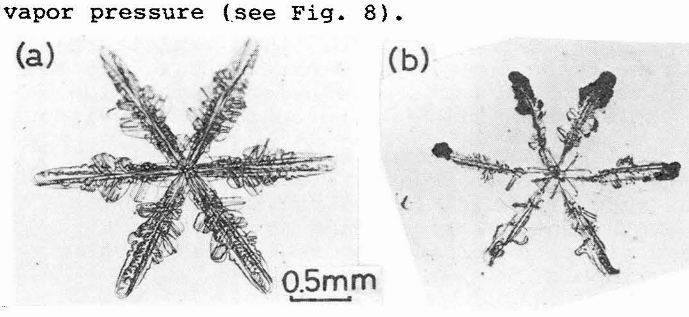

Fig. 3. Snow crystal before (a) and after (b) replication by the liquid method using 1 percent solution of Plexiglas in ethylene dichloride.

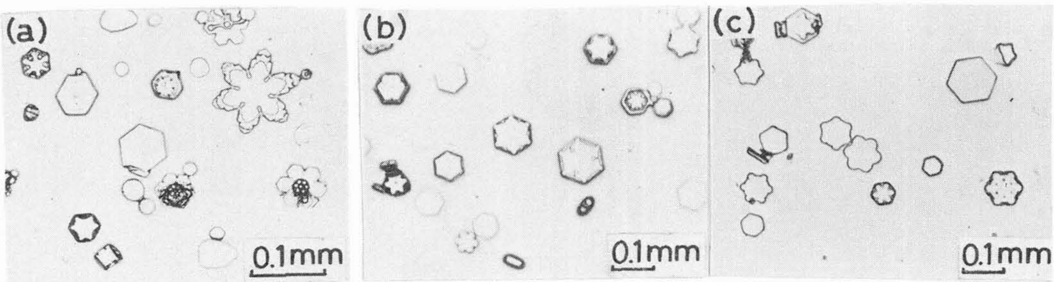

Fig. 4. Ice crystal replicas by the vapor method using (a) polystyrene-carbon tetrachloride, (b) Lexan-methylene chloride, (c) Formvar-methylene chloride.

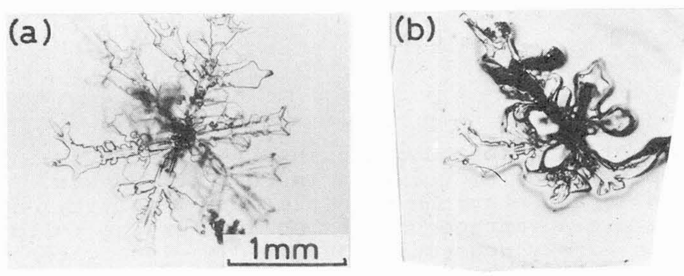

Fig. 5. Snow crystal before (a) and after (b) replication by the liquid method using 10 percent solution of polystyrene in toluene.

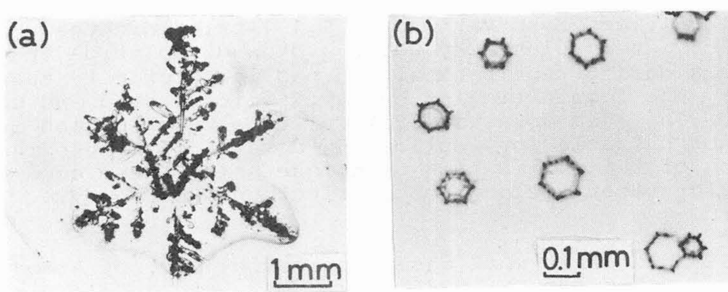

Fig. 6. Secondary protrusions on ice crystals by (a) the liquid method and (b) the vapor method.

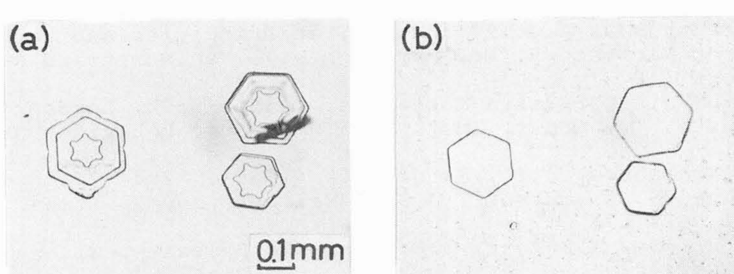

Fig. 7. Ice crystals before (a) and after (b) replication by the vapor method.

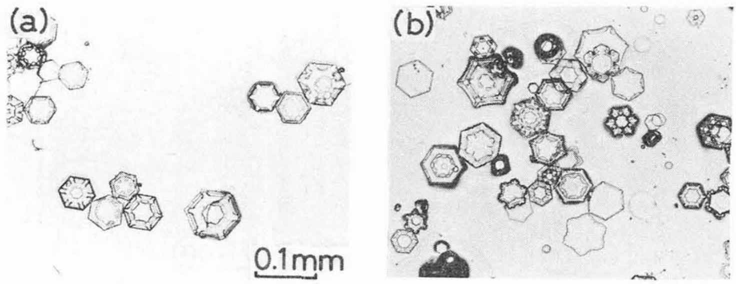

Fig. 8. Small crystals by the liquid method using 5 percent solution of polystyrene dissolved in (a) 1,1,1-trichloroethane and (b) carbon tetrachloride.

IV-CONCLUSION

All common plastic tested are usable for ice crystal replication with proper combinations of solvents instead of Formvar. The combinations of Plexiglas and ethylene dichloride, and polystyrene and 1,1,1-trichloroethane are advantageous over others for the following reasons: 1) each solution hardly gets murky due to dissolved water because these plastics do not include the hydroxyl content as opposed to the Formvar solution, 2) these solvents do not cause the occurrence of protrusion on ice crystals by strong evaporative cooling, or deformation of an ice crystal by slow evaporation of the solvent, 3) Plexiglas appears superior for long-time preservation, 4) 1,1,1-trichloroethane appears superior for low water solubility which avoids dissolution or growth of ice crystals during replication, and has less toxicity than other solvents. The combination of every plastics tested and methylene chloride can be used down to -95.1°C. The vapor method is found, in general, not suitable for replicating details of minute ice crystals. Application of the replication technique using these common plastics is suggested for other fields such as electron microscopy.

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COMMENTS

K. ITAGAKI

Have you tried spray paint ? It is convenient to use ?

Answer :

No, I have not. However, lacquer type point consists of nitrocellulose and ester type solvent and it may work although probably not very nicely.

W. GROSS :

Could the microstructures on the surface of doped and pure ice reported by Truby from formvar replicas be artifacts due to etching ?

Answer:

In my opinion, they may very well be due to dissolving of high energy parts of the surface.

Answer of T.KURODA :

Of course, we should worry about the artificial change of external shapes due to etching. Nevertheless, the surface structures such as lacunar are actual characteristics of growth forms of snow crystals, since such structures are often observed by optical microscope directly without using replica.