

## BRITTLE ZONE AND AIR-HYDRATE FORMATION IN POLAR ICE SHEETS

Tsutomu UCHIDA<sup>1</sup>, Paul DUVAL<sup>2</sup>, Volodya Ya. LIPENKOV<sup>3</sup>,  
Takeo HONDOH<sup>4</sup>, Shinji MAE<sup>5</sup> and Hitoshi SHOJI<sup>6</sup>

<sup>1</sup>*Hokkaido National Industrial Research Institute, Sapporo 062*

<sup>2</sup>*Laboratoire de Glaciologie et Géophysique de l'Environnement,  
38402 St Martin d'Hères Cedex, France*

<sup>3</sup>*The Arctic and Antarctic Research Institute, St. Petersburg 199 226, Russia*

<sup>4</sup>*The Institute of Low Temperature Science, Hokkaido University, Sapporo 060*

<sup>5</sup>*Faculty of Engineering, Hokkaido University, Sapporo 060*

<sup>6</sup>*Kitami Institute of Technology, Koen-cho 165, Kitami 090*

**Abstract:** Microscopic observations of air bubbles and air-hydrate crystals included in the Vostok ice cores, Antarctica, revealed that badly fractured ices were observed in the ice cores having high concentration of air bubbles. They also showed that the transformation of air bubbles to air-hydrate crystals reduced the chance of ice core fracture. In this paper, we suggest a fracture model of an ice core including air bubbles to discuss the depth dependence of ice core brittleness.

## 1. Introduction

Ice core fracture is one of the most serious problems in deep drilling of polar ice sheets. Brittle ice cores are observed in the depth region of several hundred meters, which is called the brittle zone. For example, the brittle zone of Vostok ice cores, Antarctica, was observed between depths of about 250 and 750 m (LIPENKOV, personal communication), and that of Dye-3 ice cores, Greenland, was between 600 and 1100 m (SHOJI and LANGWAY, 1982), as shown in Fig. 1. Soon after the ice cores in the brittle zone were taken, their quality became lower, with many cracks forming. This property changes the physical characteristics of the ice core, such as bulk density, with loss of the ancient air included in it.

The bottom of a brittle zone is observed above the depth of air bubble disappearance which is about 1250 m for the Vostok ice core (BARKOV and LIPENKOV, 1984) and about 1640 m for the Dye-3 ice core (SHOJI and LANGWAY, 1982). The disappearance of air bubbles is caused by their transformation into air-hydrate crystals.

The existence of air-hydrate crystals in deep ice sheets was predicted by MILLER (1969) who calculated its dissociation pressure theoretically. SHOJI and LANGWAY (1982) first found the air-hydrate crystals in Dye-3 ice cores deeper than 1280 m. Later they revealed that a significant number of air-hydrate crystals existed in other deep ice cores, and showed that the brittle zone terminated in the depth region where air-hydrate crystals were observed (Fig. 1).

In the present study, we observed air-hydrate crystals in Vostok ice cores shallower than 1400 m depth with a microscope. This depth region covered the whole transition zone from air bubbles to air-hydrate crystals. Then we compared depth profiles of both air bubbles and air-hydrate crystals with those of crack densities to discuss the physical

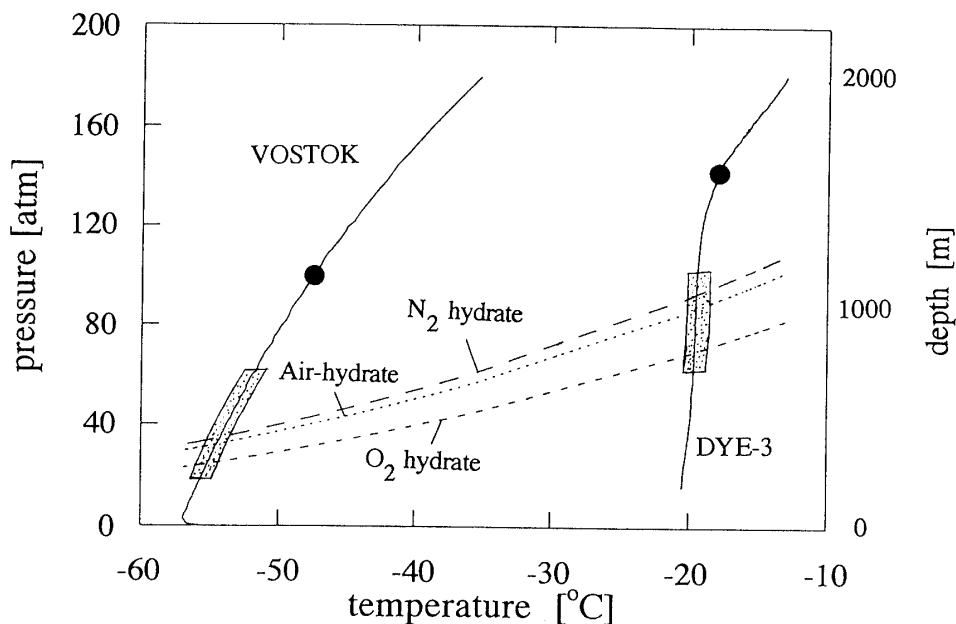


Fig. 1. Composite diagram of the air-hydrate experiment on deep ice core samples.

▨ : brittle zone.

● : air bubble disappearance in ice cores.

$N_2$ ,  $O_2$  and air-hydrate curves are represented by MILLER (1969).

properties associated with ice core brittleness. The results indicated that ice core samples stabilized with decreasing number concentration of air bubbles, or with increase of air-hydrate crystals. We suggest a model of the brittleness of ice cores which contains many air bubbles.

## 2. Experimental Procedures and Results

The drilling at Vostok Station reached a depth of 2083 m in 1982, with later extension to 2202 m (3 $\Gamma$  ice core). The sampling of ice for core analyses was done in the field during the 1982–83 austral summer, and the ice cores were sent to France. They have been stored at about  $-20^{\circ}\text{C}$  for about ten years.

Twelve samples were selected for the present study from the depth range between 218 and 1303 m, that is, 218, 404, 548, 605, 706, 804, 899, 954, 1005, 1108, 1196 and 1303 m. All of them contained some cracks. We chose crack-free sections of the samples for the air-hydrate crystal observations.

The planar faces of a thin section for each sample were cut roughly from the ice cores by a band saw and finished with a plane to about 5 mm thick. The preparation procedure of the thin sections was the same as that described in detail in a previous paper (UCHIDA *et al.*, 1994).

Air-hydrate crystals were observed with a microscope two or three times in each thin section. All air-hydrate crystals included in the observed area (about  $10 \times 10 \times 5 \text{ mm}^3$ ) were measured for each observation. The air-hydrate crystals were identified by using the Becke test which was used by SHOJI and LANGWAY (1982). These experiments were done in a

cold laboratory at  $-14^{\circ}\text{C}$ .

Air-hydrate crystals were observed in specimens below 548 m. Some typically shaped crystals are shown in Fig. 2. These crystal shapes were similar to those observed below 1050 m (UCHIDA *et al.*, 1994), such as (a) spherical, (b) irregular, (c) rod-like and (d) polyhedral. We could not find any air-hydrate crystals in 218 and 404 m ice samples even if we repeated the observation more than three times.

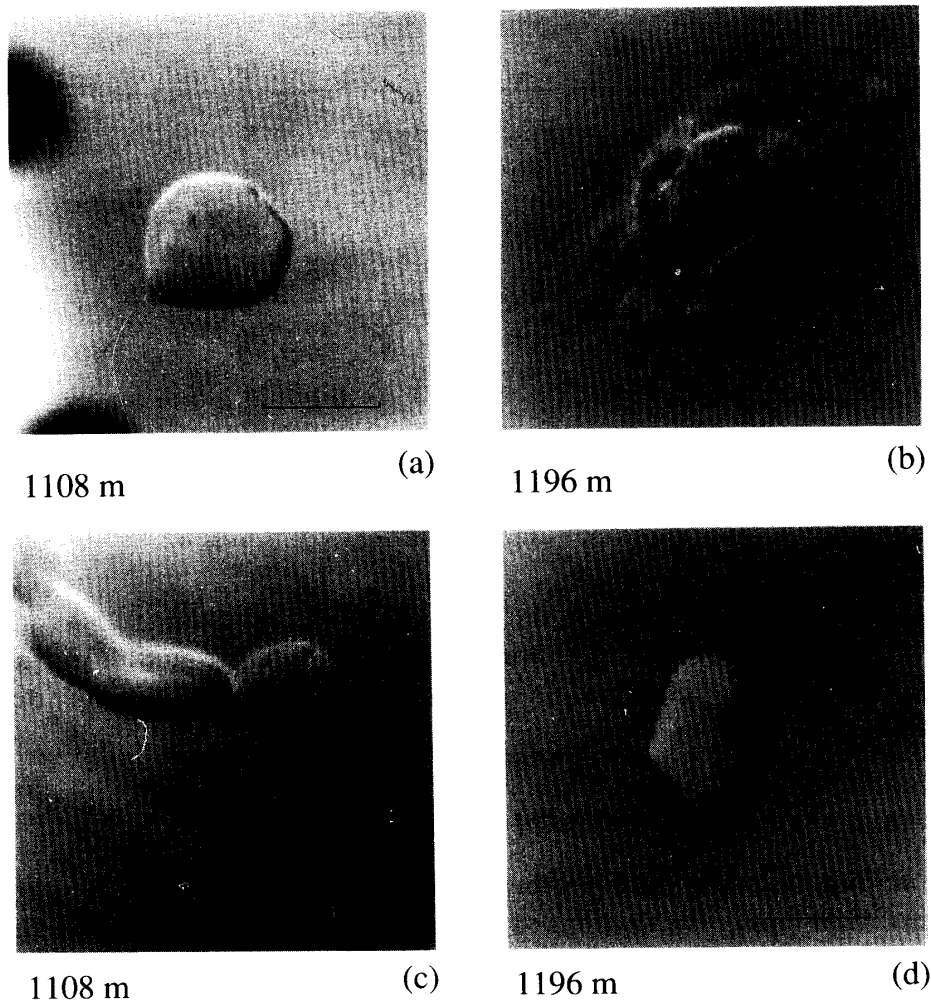


Fig. 2. Typically shaped air-hydrate crystals observed in the Vostok ice cores: (a) spherical, (b) irregular, (c) rod-like and (d) polyhedral. The scale bar in each photo is  $100\ \mu\text{m}$ .

The depth profile of the total number of air-hydrate crystals per unit volume of each ice sample (number concentration,  $N_{\text{ah}}\ \text{m}^{-3}$ ) is illustrated by the solid line in Fig. 3. This figure shows that  $N_{\text{ah}}$  gradually increases with depth from about  $1 \times 10^7\ \text{m}^{-3}$  (548 m) to about  $5 \times 10^8\ \text{m}^{-3}$  (1196 m). The ends of the error bar for  $N_{\text{ah}}$  represent the deviation of measurements performed on the same sample. The transformation rate of air bubbles to air-hydrate crystals is represented by the inclination of the  $N_{\text{ah}}$  profile because the abscissa of Fig. 3 can be considered to be the age of ice. Figure 3 shows, then, that the transformation rate reaches its maximum at about 800 m. This depth approximately corresponds to

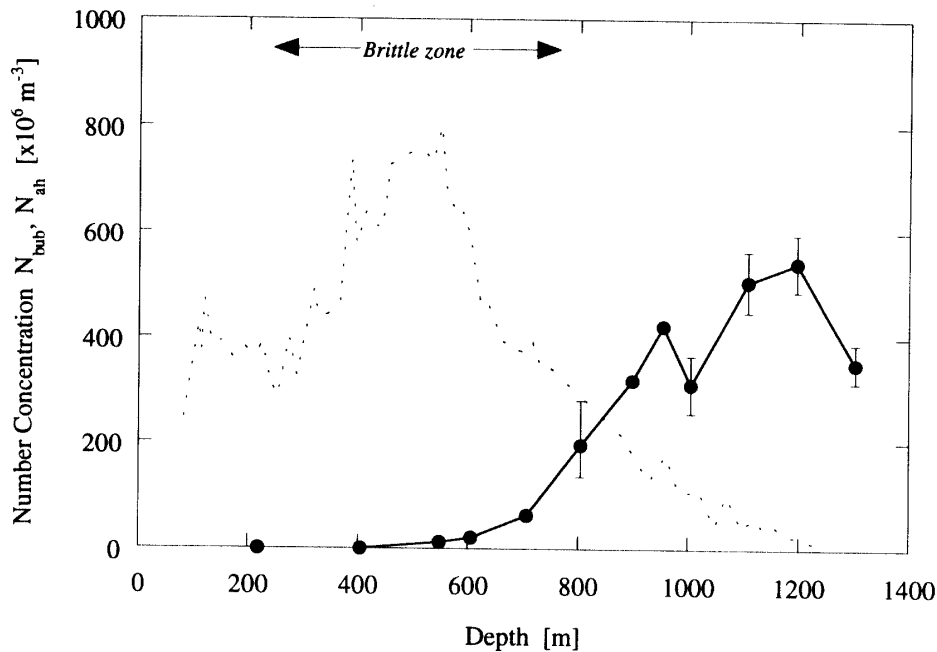


Fig. 3. Depth variation of the number concentration of air-hydrate crystals,  $N_{\text{ah}}$   $\text{m}^{-3}$ , in Vostok ice cores shown by solid line (this work), and that of air bubbles,  $N_{\text{bub}}$   $\text{m}^{-3}$ , by dotted line (BARKOV and LIPENKOV, 1984). The depth range of the brittle zone is also pointed out by arrows.

the center of the transition zone from air bubbles to air-hydrate crystals.

The new  $N_{\text{ah}}$  data in the fresh Vostok ice core (LIPENKOV, personal communication) showed that the depth profile of  $N_{\text{ah}}$  obtained in the present study was qualitatively the same. This means that the maximum transformation rate was observed at the same depth, about 800 m. The quantity  $N_{\text{ah}}$  was, however, approximately half that in the fresh ice core.

The depth profiles of both number concentration ( $N_{\text{bub}}$   $\text{m}^{-3}$ ) and mean diameter ( $d_{\text{bub}}$  m) of air bubbles in Vostok ice cores were obtained by BARKOV and LIPENKOV (1984). The depth profile of  $N_{\text{bub}}$  illustrated by the dotted line in Fig. 3 showed that  $N_{\text{bub}}$  increased with depth from the depth of the pores closed off at about 90 m to its maximum at 550 m and, subsequently, decreased to zero at about 1250 m. The larger value of  $N_{\text{bub}}$  below 250 m is considered to be affected by the colder environment, and its decrease below 550 m is caused by transformation of air bubbles into air-hydrate crystals.

The crack density of Vostok ice cores was measured soon after their recovery from the drill hole (LIPENKOV, personal communication). The depth profile of the crack density showed that the ice core became brittle with increasing depth below 100 m. The ice core was badly fractured between 250 and 750 m, which we call the brittle zone (shown by arrows in Fig. 3). Subsequently, the physical quality of the core progressively improved; below 900 m, it was of excellent quality.

Figure 3 shows that the brittle zone corresponds to the depth from where  $N_{\text{bub}}$  becomes larger to where the transformation rate of air bubbles to air-hydrate crystals reaches its maximum. It also shows that the ice core quality improves below about 800 m with depth where  $N_{\text{ah}}$  increases and  $N_{\text{bub}}$  decreases. It is, therefore, concluded that  $N_{\text{bub}}$  affects the brittleness of the ice core, and that the quality of the ice core improves with increasing  $N_{\text{ah}}$ .

### 3. Discussion

#### 3.1. Bottom depth of a brittle zone and formation of air-hydrate crystals

As ice cores in the brittle zone include many air bubbles, they fracture soon after the recovery from the bore hole. The fracture of ice cores results from volume expansion driven by the pressure difference between air bubbles and the atmosphere. The volume expansion of ice will induce the formation and propagation of cracks. Moreover, the larger the porosity of ice, the easier the cracks interact with each other. Therefore, the brittleness of the ice core depends both on the pressure of air bubbles included in it and on the porosity of ice.

Here we assume that the air bubble pressure is almost the same as the hydrostatic pressure at its original depth. On the other hand, the porosity of an ice core depends on both the size and number of air bubbles included in it. The size distribution of air bubbles in Vostok ice was obtained by BARKOV and LIPENKOV (1984). The distribution indicated that  $d_{\text{bub}}$  decreased markedly with depth to about 200 m. However, the variation of  $d_{\text{bub}}$  with depth became smaller below that. Since the depth variation of porosity is related to that of  $N_{\text{bub}}$ , the depth profile of  $N_{\text{bub}}$  is useful to discuss that of ice core brittleness.

The comparison of depth profiles between  $N_{\text{bub}}$  and the crack density shows that ice cores, which have larger values of  $N_{\text{bub}}$ , are badly fractured in the depth range between about 250 and 750 m. However, below 750 m depth, the quality of ice cores improves with decreasing  $N_{\text{bub}}$  even though the depth increases. This result suggests that the porosity of ice is the dominant factor determining its brittleness.

The improvement of ice core quality is also accompanied by increasing  $N_{\text{ah}}$  with depth below about 500 m. The result indicates that the transformation of air-hydrate crystal from air bubbles results in the stabilization of ice cores. Two processes can explain these phenomena considering that an air-hydrate crystal is a solid which has similar density to ice. The density of air-hydrate crystals is estimated from structure data of air-hydrate crystals (HONDOH *et al.*, 1990). First, the transformation of air bubbles to air-hydrate crystals makes the porosity of the ice core smaller. Second, the transformation will reduce the internal pressure of the ice core, which is the driving force of its volume expansion.

It is, therefore, concluded that the formation of air-hydrate crystals avoids the fracture of ice cores. The difficulty of dissociation of air-hydrate crystals may also be one of the reasons why ice core quality does not deteriorate.

Moreover, the bottom depth of a brittle zone is almost the same depth where the transformation rate reaches its maximum (about 800 m). This result suggests that  $N_{\text{bub}}$  has a critical value which determines the brittleness of ice cores. The bottom depth of the brittle zone should have the smaller value of  $N_{\text{bub}}$  than the critical one. This process will be discussed in the following section.

#### 3.2. Brittleness of ice core containing air bubbles

The resistance of ice cores to brittle failure may be characterized by fracture toughness. Microcracks are initiated from pressurized bubbles and grow as long as the mode I stress intensity factor  $K_I$  is higher than the fracture toughness of ice  $K_{\text{IC}}$ . For an isolated crack stressed by uniaxial tension,  $K_I$  has the form:

$$K_I = \alpha \sigma (\pi L)^{1/2}, \quad (1)$$

where  $L$  is the crack length,  $\sigma$  the applied tensile stress and  $\alpha$  is a coefficient which depends on the sample geometry.

For polar ice, the growth of microcracks makes the air pressure within bubbles lower because the total amount of air in the bubbles is constant. In consequence, the stress  $\sigma$  decrease when the crack length increase and microcracks become stable when  $K_I$  is lower than  $K_{IC}$ .

A first attempt at defining stress intensity factors,  $K_I$ , associated with a spherical void circumferentially cracked (see Fig. 4) and loaded by a tensile stress normal to the crack plane has been presented by BARATTA and PARKER (1981). In considering the growth of cracks from air bubbles, there are two contributions to the free energy of the system in isothermal changes, in addition to the surface energy: the change in elastic energy of the body; and the change in the potential energy which is borrowed from pressurized bubbles. By assuming that air is a perfect gas, the energy balance can be given for a bubble circumferentially cracked:

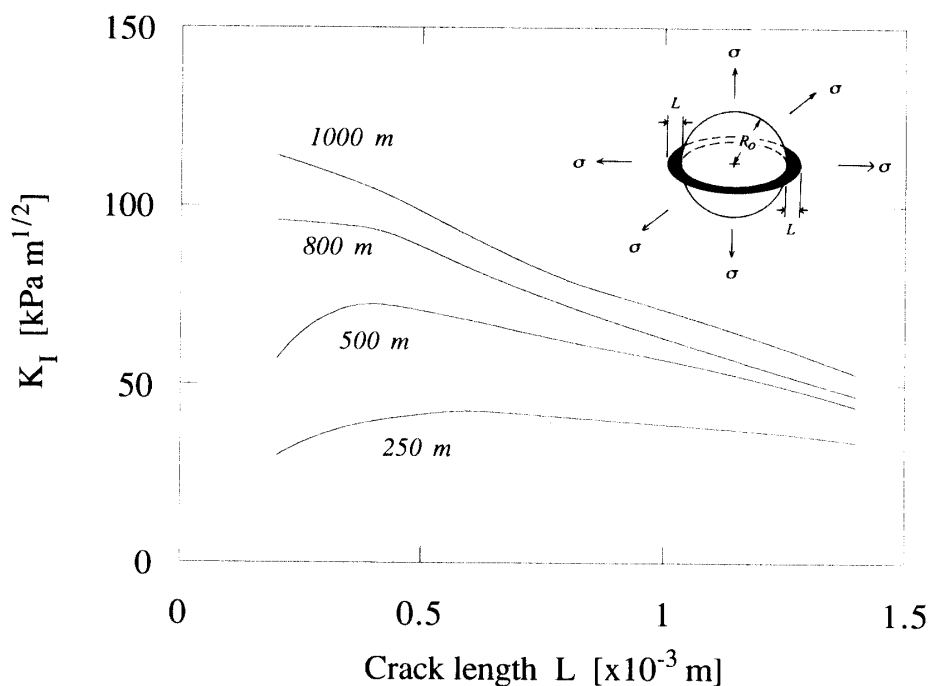


Fig. 4. Stress intensity  $K_I$   $\text{kPa m}^{1/2}$  for Vostok ice cores of four different depths as a function of the crack length  $L$ .

$$P_0 V_0 \ln \frac{P}{P_0} = \frac{(3 \pi^2 L^2 P^2)}{E} (R_0 + L) + 2 \gamma \pi (L^2 + 2 R_0 L). \quad (2)$$

The first term represents the energy associated with a decrease of air pressure ( $P_0$  and  $V_0$  are respectively the initial pressure and volume of one bubble); the second term corresponds to the elastic energy of the crack ( $R_0$  is the radius of bubbles and  $E$  the elastic

modulus of ice); the third term represents the surface energy of cracks ( $\gamma$  is the specific surface energy).

This equation was used to determine the pressure within the bubble  $P$  as a function of the crack length  $L$ . We have assumed that the pressure  $P_0$  at different depths was exactly the overburden pressure. The stress intensity factor  $K_I$  was calculated with  $\alpha = 0.63$  (BARATTA and PARKER, 1981) and  $\sigma = P$ , and results are given in Fig. 4. This figure shows, for example, that a crack on an air bubble in ice at 800 m depth will grow when  $L$  is larger than the critical length for which  $K_I = K_{IC}$ . If  $K_I = 70 \text{ kPa m}^{1/2}$ , cracks smaller than 0.8 mm will grow.

The fracture of polar ice samples in the brittle zone must be associated with crack-crack interaction and crack linking because these samples include many air bubbles. Then the crack length per unit volume of ice core,  $L_{tot} = L N_{bub}$ , is the critical parameter for final failure.  $N_{bub}$ , therefore, also has a critical value for the brittleness of ice cores.

It is not only necessary for initiating fracture that  $K_I$  must be larger than  $K_{IC}$ , but also that  $L_{tot}$  is longer than a critical value. NIXON and SCHULSON (1987) reported  $K_{IC}$  values from 70 to 90  $\text{kPa m}^{1/2}$  for granular ice. These values are obtained below 500 m at Vostok (Fig. 4). However, the failure of the Vostok core is not observed below about 900 m. This result can be explained by noting that  $L_{tot}$  is higher than the limit value for which  $K_I > K_{IC}$ .

It is impossible to determine this critical length for polar ice due to the uncertainty of the value of  $K_{IC}$ . However, we will estimate the critical value from results obtained in Vostok ice cores. If we assume that  $K_{IC} = 70 \text{ kPa m}^{1/2}$ , the bottom depth of the brittle zone (750 m) implies that  $L > 0.85 \text{ mm}$ . This value corresponds to a critical value of  $L_{tot}$  of about 255 mm ( $0.85 N_{bub(750 \text{ m})}$ ). With this critical value, the crack length for one bubble at 500 m must be  $L > 0.32 \text{ mm}$ . The crack can propagate until 0.45 mm, at which  $K_I < K_{IC}$  (Fig. 4). However, this length corresponds to half of the mean distance between air bubbles ( $0.5 N_{bub(500 \text{ m})}^{-1/3}$ ). The cracks will joint with each other, causing the ice core to fracture.

The calculations done above will be useful to determine external conditions to apply to ice cores in order to avoid brittleness in polar ice cores.

### Acknowledgments

We are very grateful to all Russian and French participants in drilling, field work and ice sampling. We thank Dr. J. OCAMPO, Dr. D. RAYNAUD, Dr. J. R. PETIT and Dr. M. CRESEVEUR for their helpful discussions and technical support. This work was supported financially by the Asahi Glass Foundation, the Science and Technology Agency and the Japan Society for the Promotion of Science.

### References

- BARATTA, F. I. and PARKER, A. P. (1981): Mode I stress intensity factor estimated for various configurations involving single and multiple cracked spherical voids. *Fracture Mechanics of Ceramics*, Vol. 6. New York, Plenum Publ.
- BARKOV, N. I. and LIPENKOV, V. Ya. (1984): Kolichestvennaya kharakteristika struktury l'da do glubiny 1400 m v rayone Stantsii Vostok v Antarktide (Numerical characteristics of ice structure down to a depth of 1400 m in the region of Vostok Station, Antarctica). *Mater. Glyasiol. Issled.*, **51**, 178–186.

- HONDOH, T., ANZAI, H., GOTO, A., MAE, S., HIGASHI, A. and LANGWAY, C. C., Jr. (1990): The crystallographic structure of the natural air-hydrate in Greenland Dye-3 deep ice core. *J. Inclu. Phenom. Molecul. Recogn. Chem.*, **8**, 17-24.
- MILLER, S. L. (1969): Clathrate hydrate of air in Antarctic ice. *Science*, **165**, 489-490.
- NIXON, W. A. and SCHULSON, E. M. (1987): A micromechanical view of the fracture toughness of ice. *J. Phys. (Paris), Colloq. C1*, **48**, 313-318.
- SHOJI, H. and LANGWAY, C. C., Jr. (1982): Air hydrate inclusions in fresh ice core. *Nature*, **298**, 548-550.
- UCHIDA, T., HONDOH, T., MAE, S., LIPENKOV, V. Ya. and DUVAL, P. (1994): Air-hydrate crystals in deep ice core samples from Vostok Station, Antarctica. *J. Glaciol.*, **40**, 79-86.

*(Received May 14, 1993; Revised manuscript received January 14, 1994)*