Visual Observation of Volume Relaxation under Different Storage Temperatures in the Dome Fuji Ice Core, Antarctica

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Abstract: We are keeping Dome Fuji ice core samples at two storage temperatures (−50 and −20°C) after deep ice core drilling reached 2503 m at the Dome Fuji station, Antarctica. Since we aimed to develop new methods for studying physical properties of ice cores, cold storage down to −50°C was necessary to avoid volume relaxation of ice cores during storage. To compare the volume relaxation process of an ice core under different storage temperatures, we captured images using a digital camera. Many tiny bubbles and plate-like inclusions (cleavage cracks) were observed in the ice core kept at the high storage temperature (−20°C). The clear ice seen just after ice core recovery changes to milky coloured cloudy ice because of the many tiny bubbles and cleavage cracks generated. We analyzed the volume relaxation rate of each ice core sample based on the digital images; it was clear that the generation of tiny bubbles and cleavage cracks and the decomposition of air hydrate crystals were avoided effectively at a storage temperature of −50°C. In cold storage down to −50°C, various physical properties of the ice core have been successfully investigated. In the present paper, we emphasize the importance of low-temperature storage to maintain high-quality ice cores.

Key words: ice core, storage temperature, volume relaxation, cleavage crack, air hydrate crystal

1. Introduction

Ice core studies are an important part of climate and environmental change research. To get good data from ice core studies, we have to drill a high-quality ice core and avoid any deterioration after core recovery. The volume relaxation research of ice core, for example, has been carried out by Langway [1], Shoji and Langway [2] and Nakawo [3]. They analyzed bubble pressure, plastic deformation of ice and density of bubbly ice core samples. Theoretical studies of volume relaxation were discussed by Salamatin and Lipenkov [4]. Here, we discuss the influence of the storage temperature on volume relaxation of the ice core by visual observation.

The volume relaxation features of the Byrd deep ice core have been reported by Gow [5], using precisely measured ice core density. This result indicated that the density decreased over time at a temperature of −10°C. The largest volume relaxation rate occurred in ice cores from between 800 and 1100. Around these depths, the ice core volume had increased by at most 0.6% after 27 months. Gow [5] observed cavities, plate-like inclusions and cleavage cracks in the Byrd ice core samples. No such inclusions were observed initially. Gow [5] concluded that these were certainly of secondary origin.

Kipfstuhl *et al.* [6] reported the results of microscopic observation of Antarctic ice cores (Dome C and Dronning Maud Land) and Greenland ice cores [GRIP and North GReenland Ice core Project (NorthGRIP)] made just after core retrieval. The "black dots (micro-bubbles)" transformed first into plate-like inclusions and finally into bubbles. These microstructural changes had already begun in the first hours/days after core retrieval.

We can observe a distinct cloudy band structure consisting of alternating layers of cloudy and clear ice in glacial ice of the Greenland deep ice core (e.g. [7], [8], [9]). This structure was observed just after core retrieval. Svensson *et al.* [9] reported that the North GRIP ice core at a depth of 1751.5 m, which was stored for one year at the NorthGRIP drilling site, shows much more pronounced cloudy bands and many more "white patches" and bubbles than the freshly drilled ice. The annual mean temperature at the NorthGRIP site is −31.5°C [10]. Under such storage condition, the volume relaxation process advanced in only one year. In the Greenland Ice Sheet Project 2 (GISP2) ice core, cavities and cleavage cracks have also been observed [11].

The model calculation of molecular diffusion in polar ice core showed that the effect of gas loss decreases as the storage temperature decreases [12]. At the storage temperature of −53°C, the calculated O_2/N_2 ratio reaches −11.2‰ (initial value is −9.9‰) after the storage for 1000 days. However, this value shows −20.5‰ for the sample stored at −25°C.

We have an ice core facility equipped with cold storage at −50°C at the Institute of Low Temperature Science (ILTS), Hokkaido University. This cold storage was constructed in 1997 just after deep ice core drilling by Japanese Antarctic Research Expedition (JARE) parties reached 2503 m at the Dome Fuji station, Antarctica. About three-quarters of the Dome Fuji ice core samples were stored at ILTS and used for physical analyses. The rest of the core was stored at −20°C at the National Institute of Polar Research (NIPR) for chemical analyses. After we had stored ice core samples in each cold storage for about 2500 days, we took a digital image of each sample to compare the condition of the samples and the extent of volume relaxation. It is

evident that the appearance of samples preserved at −50°C is excellent. In addition, we also tried to examine the volume relaxation rate by processing the digital ice core images.

2. Samples

After the ice core was drilled at the Dome Fuji station, samples that were cut lengthwise were shipped to two institutes in Japan. One side of the sample was transported to NIPR in Tokyo, which had cold storage at −20°C for chemical analysis. The other side of the sample was used to study the physical properties of ice core. To avoid relaxation of the ice core during storage, it was transported to ILTS in Sapporo, which had cold storage down to −50°C. Thus, Dome Fuji ice core samples from precisely the same depths were kept at two different storage temperatures.

The shipping temperature was kept at less than about −20°C, although there was a possibility that temperature rose to around −10°C for about one day at some relay points. Therefore, it was assumed that the temperature in the ice core box did not rise greatly during the shipping of ice core samples.

We chose five samples from five depths (999.30, 1196.00, 1502.00, 1999.00 and 2485.41 m) to compare the volume relaxation process as shown in Table 1. The shallowest sample, which was located in the transition zone, is from 999.30 m. The air hydrate crystals start appearing below 450 m in the Dome Fuji ice core [13]. Deeper than this and down to about 1200 m, air bubbles and air hydrates coexist in the Dome Fuji ice core. Therefore, we call this depth rage the transition zone. This sample included many air bubbles in the ice sheet. The second sample is also from the transition zone at a depth of 1196.00 m. Because this depth range is quite close to the boundary between the transition zone and the bubble-free zone, most air bubbles are transformed to air hydrate crystals; in other words, the number concentration of air bubbles was quite low in the ice sheet at these depths.

The third sample is from the bubble-free zone at a depth of 1502.00 m, which is located in the middle of the ice thickness at the Dome Fuji station. Theoretically, below 1200 m, all bubbles are transformed into air hydrate crystals. Indeed, we could not see prominent air bubbles in the Dome Fuji ice just after the cores were recovered. The fourth and fifth samples are also from the bubble-free zone at a depth of 1999.00 m (the middle of the bubble-free zone) and 2485.41 m (the lowest depth reached by the first drilling project at the Dome Fuji station).

About 2500 days after the ice core was recovered at the Dome Fuji station, we photographed the samples for this study. The details are given in Table 1.

Table 1: Sampling depth and storage period of the Dome Fuji ice core samples.

Sample No.	Depth, m (top of sample)	Storage period, days
1	999.30	2656
2	1196.00	2631
3	1502.00	2596
4	1999.00	2538
5	2485.41	2435

3. Observations and analysis

To compare the volume relaxation process of the Dome Fuji ice core under different storage temperatures, we carried out visual observation of ice core samples. Volume relaxation causes micro-bubbles, cleavage cracks and bubbles from decomposed air hydrate crystals to appear in the Dome Fuji ice core. We can recognize these air bubbles and cleavage cracks as multiple tiny white dots on grey-scale photographs. We assumed that the amount of grey value on whole area of the acquired ice core images, which we can measure using the image processor application ImageJ [14], to be an index of the degree of volume relaxation.

When we took the photographs, ice core samples at NIPR were transported to the cold room of ILTS. The photographic apparatus was set in a cold room of −20°C at ILTS. Each section of ice core sample was about 50 cm long, and the thickness was about 3 cm. Figure 1 shows a schematic illustration of the setup. The ice core samples were put on a matte black background (black velvet cloth) and exposed to light from one side from a fluorescent light. All images were taken using a Nikon E4300 digital camera under the same conditions at ILTS. Each image captured about 18 cm length; therefore, for each sample, we connected several images in order to image the 50-cm-long sample. The digital images of each sample are shown in Figure 2.

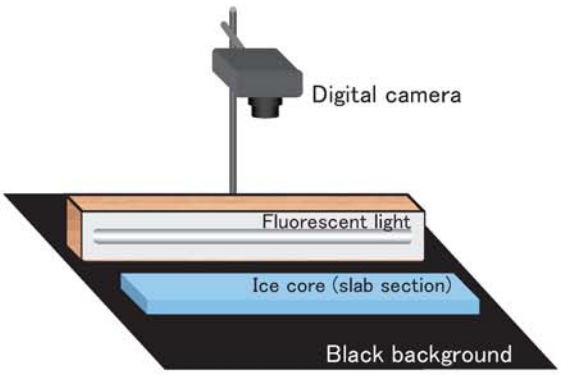

Figure 1: Photographic setup in a cold room of −20°C.

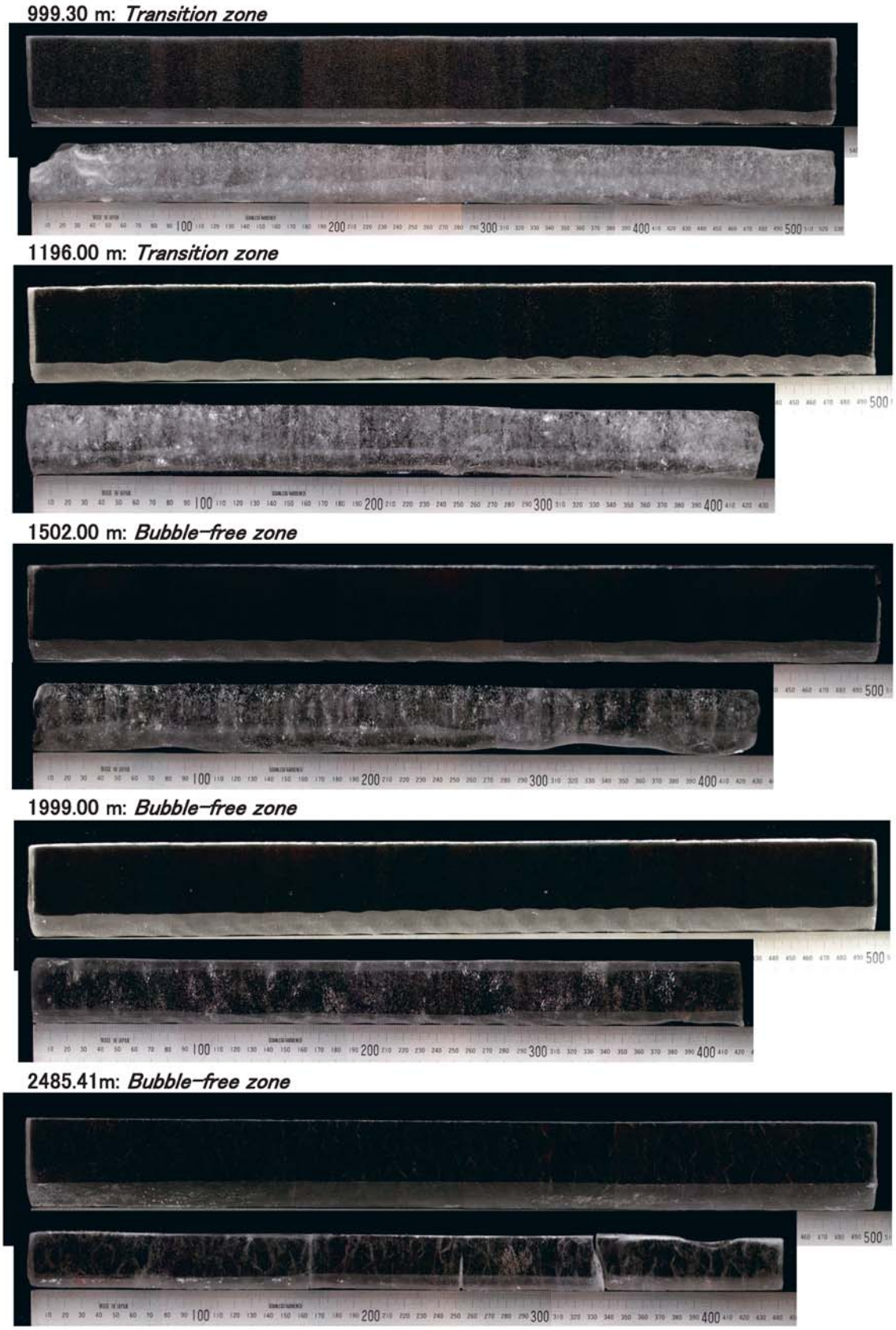

Figure 2: Photograph of the Dome Fuji ice core samples. In each depth, the upper photograph shows the sample stored at −50℃, and the lower one shows the sample stored at −20℃. The scale below the samples indicates values in mm. In the samples stored at −20℃, we can observe many white dots and white patches. These are bubbles and cleavage cracks generated secondarily by volume relaxation during the approximately 2500 days since ice core recovery.

All digital colour images were converted into black-and-white monochromatic images with an 8 bit grey scale. Each pixel has one of 256 different intensities between black and white. Grey values of 0 and 255 represent completely black and completely white, respectively. If the entire area of an ice core image is filled with white dots, the mean grey value will be close to 255. We measured the grey value of the velvet cloth to determine the background level. The average grey value of the velvet, measured at 10 places, was 0.9. This means that the background was almost black.

We conducted five measurements of the mean grey value of the entire area of each ice core sample. On the image processing, we selected the entire area of microtomed surface on each ice core images by rectangular shape, and the mean grey value in this rectangular area was calculated by the image processor application. The average of these five measurements was defined as the mean grey value of that sample. A layered structure was observed in the ice core, and the degree of volume relaxation varied with location in the structure; however, its average value was defined as the grey value of that ice core sample in this paper.

4. Discussion

The grey values of ice core samples kept at −50°C were lower than those of ice cores kept at −20°C for all depth ranges (Figure 3). It is clear that the volume relaxation is restrained for all depths when the ice was kept at −50°C. However, a large difference was seen in the degree of volume relaxation, depending on the depth range.

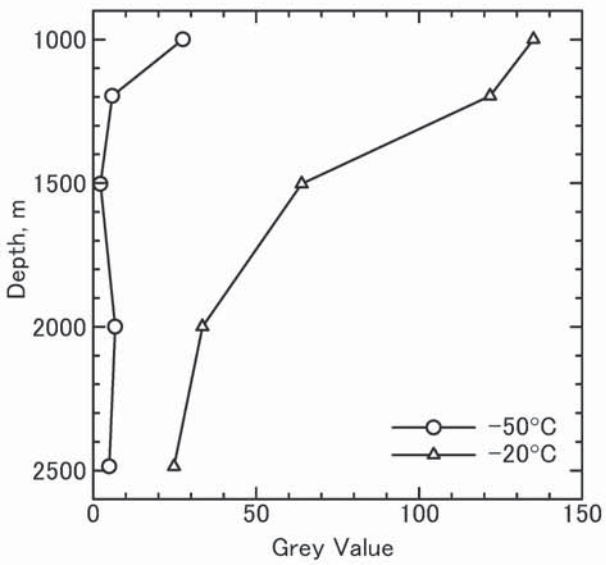

Figure 3: Measured grey values with depth for the Dome Fuji ice core samples. The grey values of −50℃ storage samples are less than 10, except for sample No. 1 (999.30 m). The −50℃ storage samples were almost transparent, even about 2500 days after core retrieval.

In particular, the grey values of sample Nos. 1 and 2, which are from the transition zone, show a large difference between −20 and −50°C storage. These samples had many tiny high-pressure bubbles in the ice sheet. The high pressures involved are thought to be close to the hydrostatic pressure value at those depths. For example, at a depth of 1196 m (sample No. 2), the hydrostatic pressure value is about 100 bar. After ice core recovery, the expansion of these high pressure bubbles advanced at relatively high temperature (−20°C) and ambient pressure. Therefore, the grey values of samples from the transition zone increased considerably at a storage temperature of −20°C. On the other hand, the grey values are almost the same in all depths at a storage temperature of −50°C, except for sample No. 1. This is because deformation of ice becomes more difficult at lower temperature. Around the depth of sample No. 1 (999.30 m), the volume concentration of air bubbles was higher than that of air hydrate crystals. Around the depth of sample No. 2 (1196.00 m), most air bubbles disappeared [13]. This indicates that there were many high-pressure air bubbles in sample No. 1 just after ice core recovery. Even at a storage temperature of −50°C, such air bubbles expanded during the storage period, and consequently the grey value increased. The volume relaxation processes are controlled by the expansion of pre-existing high-pressure air bubbles from sample taken above about 1000 m.

The grey value of sample No. 3 (1502.00 m) kept in storage temperature at −20°C was much lower than those of samples No. 1 and 2 at that temperature. This sample was chosen from a bubble-free zone. Deeper than about 1200 m, all air bubbles are transformed into air hydrate crystals in the ice sheet; therefore, the volume relaxation processes are controlled by decomposition rate of air hydrate crystals. The high-pressure secondary bubbles generate by decomposition of air hydrate crystals. After that, the volume relaxation rate will be also controlled by expansion of these bubbles. However, as describe later, the number of decomposed air hydrate crystals is important factor for the volume relaxation.

In the deeper parts (sample Nos. 4 and 5), the difference between the grey value after storage at −20 and −50°C tended to decrease (Figure 3). These differences are probably controlled by multiple factors. For example, (1) the number concentration of air hydrate crystals decreases at greater depths (unpublished). This means that the probability of air bubble nucleation on the surface of air hydrate crystals decreases. (2) There is a distinct difference in crystal textures (crystal size, c-axis orientation and others) between the upper and lower layers. The mean crystal size is about 2 and 6 mm at a depth of 1000 and 2500 m, respectively [16]. The c-axis orientation distribution is also completely different: a very weak single maximum at 1000 m and a very strong single maximum at 2500 m [16]. For example, the coefficient of linear expansion is different depend on crystal axis orientation. This means

that volume expansion anisotropy occurs in a very strong single maximum. Such peculiar textures at greater depths may be changed to volume relaxation features in comparison with shallower depths. (3) Impurity content changes on an annual-layer thickness scale. Shimohara *et al.* [8] suggested that the micro-bubbles that cause cloudiness in cloudy bands nucleated around dust particles. The relaxed ice samples also have banding structure, as seen in Figure 2. This indicates that the relaxation rate differs with the distribution of impurities.

Uchida *et al.* [15] predicted the optimized storage conditions for ice core samples from experimental data on the decomposition of air hydrate crystals in the Vostok and Dye-3 ice cores. They counted the number concentration of air hydrate crystals in ice core samples with varying storage periods and temperatures by microscopic observation. From this observation, they estimated the decomposition rate of air hydrate crystals during ice core transportation and/or long-term storage. The result is shown in Figure 4. They suggested relationship between storage temperature and decomposition time for the deep ice core samples. For example, it will take about 34 years for 5% of air hydrate crystals to decompose at a storage temperature of −50°C, but only 115 days at a storage temperature −20°C. Although air hydrate crystals cannot exist alone at ambient pressure because it is in their decomposition pressure, they can exist in an ice core sample after recovery for long time. This is because the surrounding ice acts as a pressure vessel after the ice core is recovered from the ice sheet. The pressure differences become large with temperature on the phase diagram of air hydrate. The high pressure difference between the decomposition pressure of air hydrate and ambient pressure produce a large driving force of the decomposition of air hydrate crystals. Moreover, at a temperature of −50°C, the deformation rate of ice is low in comparison with that at −20°C. After the decomposition of air hydrate, the secondary bubbles expansion is avoided under lower temperature. Thus, decomposition of the air hydrate crystals is slowed in the ice core. We chose to store the Dome Fuji ice core at a temperature of −50°C in ILTS based on the results of Uchida *et al.* [15].

The importance of storing ice cores at lower temperatures is quite obvious from visual observation, which indicated that the grey value of ice core samples was increasing linearly with time. Further, we can roughly estimate the volume relaxation rate from the grey value measurements. We assumed that the ice core sample just after ice core recovery was transparent.

The relaxation factor α was defined as

$$\alpha = (\text{measured grey value})/255. \qquad (1)$$

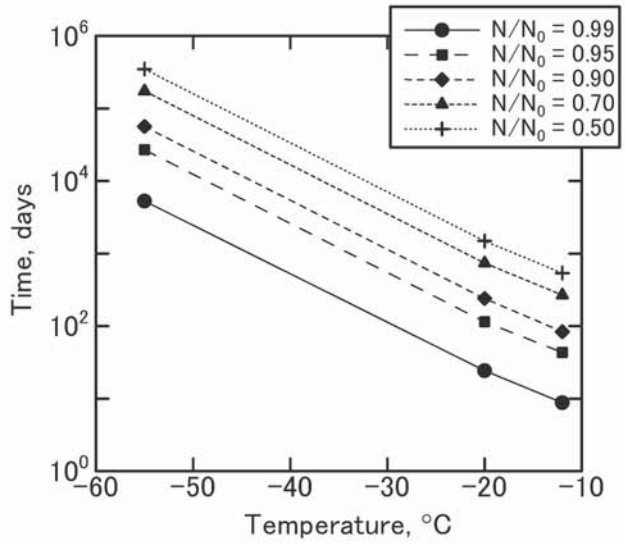

Figure 4: Storage temperature versus time period for air hydrate crystal decomposition in ice from [10]. N [m^{-3}] is the number concentration of air hydrate crystals. N_0 [m^{-3}] is the reference number concentration of air hydrate crystal measured in an excellent-quality ice core sample. For example, "$N/N_0 = 0.95$" indicates how many days it takes for 5% of air hydrate crystals to decompose at each storage temperature.

For example, a relaxation factor of 0.5 means that half of the sample area is covered with white dots. In this analysis, we have only one measurement point for the grey value of each sample about 2500 days after ice core recovery (Figure 5). The computed results of Salamatin and Lipenkov [4] shows that the relaxation process is not a linear with time. However, the average volume relaxation rate for long storage period, v, can be calculated as follows.

$$v = \alpha/(\text{storage period}). \qquad (2)$$

This is the simplest estimation method for the volume relaxation rate. From equations 1 and 2, we estimated the number of days for a volume relaxation of 5%, which was equivalent to a relaxation factor of 0.05. The result is shown in Table 2 and Figure 6. The relaxation time necessary for a volume relaxation of 5% was more than 1200 days at a depth of 1000 m for storage at −50°C. This was the shortest relaxation time. The longest time for a volume relaxation of 5% was more than 15000 days (more than 40 years) at a depth of 1500 m. The relaxation rate at a storage temperature of −50°C does not decrease monotonically with depth. This may be due to artefact errors on the measurement. There are few white dots in samples which storage at -50°C. Therefore, for example, some frost on sample surface will cause a big noise. At a storage temperature of −20°C, the relaxation time for a volume relaxation of 5% was about 250 days to 1200 days. In contrast to these results from the grey value analysis, according to Uchida *et al.* [15], it will take 34 years for about 5% of

air hydrate crystals to decompose at a temperature of −50°C. Uchida *et al.* [15] used the average value of differences in the decomposition rate of air hydrate crystals with depth; however, it was generally the same period as the relaxation time calculated from the grey value. This means that the volume relaxation process of an ice core is mainly controlled by the decomposition rate of air hydrate crystals at the bubble-free zone or deeper. Incidentally, at a temperature of −20°C, about 5% of air hydrate crystals will decompose in about 100 days. This is also almost the same value as that from the grey value analysis. These results show that the grey value analysis gives results comparable with relaxation rates estimated by the decomposition rate of air hydrate crystals [15].

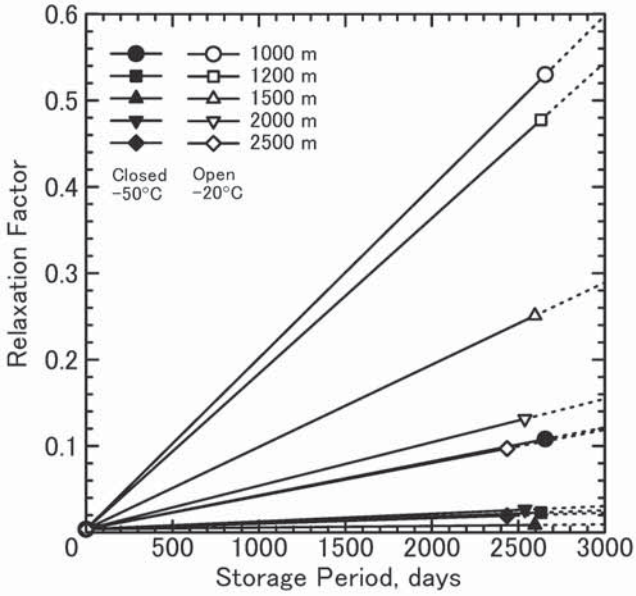

Figure 5: Relaxation factors of each ice core samples at the storage period of about 2500 days. The slope of each line shows the average relaxation rate at each depth and storage temperature during 2500 days.

Table 2: Time period required for 5% of volume relaxation at each storage temperature, estimated from grey value analysis.

Sample No.	−20°C, days	−50°C, days
1	251	1230
2	275	5757
3	517	15212
4	966	4819
5	1254	6339

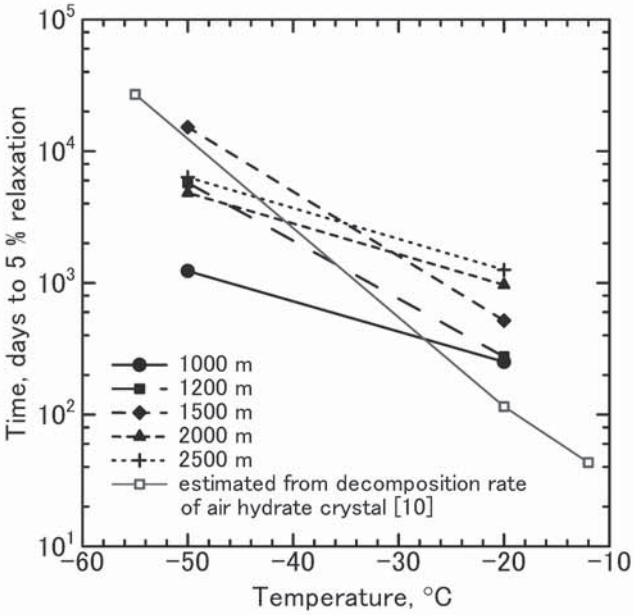

Figure 6: Time period for 5% of volume relaxation at each storage temperature. Grey open rectangles indicate the results of [10] estimated from the time period for decomposition rate of air hydrate crystals at each storage temperature. This is the same as "N/N_0 = 0.95" in Figure 4.

The internal structure of an ice core undoubtedly changes due to volume relaxation. The occurrence of cleavage cracks and air hydrate crystal decomposition have a dramatic effect on the diffusion processes of air molecules in the ice core. In particular, the decomposition of air hydrate crystals changes the existing state of air molecules in the ice. If we do not avoid volume relaxation of ice cores, correct analyses and detailed discussions of ice core studies are not possible. We can keep ice cores in high-quality for more than several years to a few decades, if we store them around −50°C. This is highly important for future ice core research.

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