

MEASUREMENTS OF THE COMPLEX PERMITTIVITY OF ACID-DOPED ICE  
FROM 1 KHZ TO 30 MHZ  
—NEW DATA SET FOR DEVELOPING ICE RADAR AND DIELECTRIC  
ANALYSIS OF ICE CORES—

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**Abstract:** We measured the complex permittivity of pure and acid (HCl, HNO<sub>3</sub> and H<sub>2</sub>SO<sub>4</sub>)-doped ice from 1 kHz to 30 MHz and from -9°C to -33°C. The complex permittivity of acid-doped ice is assumed to depend linearly on the concentration. The frequency and temperature dependence of the relationship between the complex permittivity and the acid concentration are investigated. The tendency of dielectric dispersion of acid-doped ice is discussed. A simple calculation shows that the reflection coefficient due to acidity change in the HF band in ice sheets increases with decreasing frequency. The effect of permittivity changes and loss tangent changes due to acidity changes to reflection coefficient are quantitatively shown, respectively. As a result, a data set which is essential to develop HF ice radar, and to compare radar echoes and ice core signals is established.

## 1. Introduction

Radio Echo Sounding (RES) of ice sheets is a unique method to investigate the three dimensional internal structures and bedrock topography of ice sheets (*e.g.* ROBIN *et al.*, 1969; BOGORODSKY *et al.*, 1985). In order to investigate the internal structure, it is necessary to recognize the causes of internal reflections of radio waves. Many studies on this problem have been carried out.

The most recent discussions have clarified that there are three dominant causes: 1) density fluctuation (PAREN and ROBIN, 1975); 2) ice fabric changes (FUJITA and MAE, 1994); 3) acidity changes (MOORE, 1988; FUJITA and MAE, 1994). Among these three causes, density fluctuation is plausible only for reflections from shallow layers because the density fluctuations are smoothed out by hydrostatic pressure in ice sheets. PAREN and ROBIN (1975) concluded that the dominant cause of the internal reflections above 1500 m was the density fluctuation. On the other hand, the latter two causes are also plausible for reflections from deeper layers. Based on the dielectric anisotropy of ice and dielectric properties of ice which contains acid, the reflection coefficient ( $R_s$ ), when radio waves are normally incident on the layer boundary, due to the fabric changes does not depend on the frequency, and  $R_s$  due to the acidity changes is inversely proportional to the frequency (FUJITA and MAE, 1994). As a result, FUJITA and MAE (1994) concluded

that acidity changes are the dominant cause of the internal reflections at lower frequencies than about 60 MHz and fabric changes are the dominant cause at higher frequencies than about 60 MHz. This result suggests that the HF band (less than 30 MHz) is very effective in detecting acidity changes in the ice sheets.

However, there still remain important unsolved problems. First, there have been no experimental data on the dielectric properties of polycrystalline ice which contains acid impurity in the MHz range. FUJITA and MAE (1994) calculated  $R_s$  based on the comparison between the experimental data at 9.7 GHz and the result of ice core analysis in the LF band (MOORE and FUJITA, 1993). Second, only electrical conductivity changes (or dielectric loss) due to acidity changes have been taken into account (FUJITA and MAE, 1994); *i.e.*, permittivity changes have been neglected, although it is still an open question whether permittivity changes are negligible or not in the MHz range. It is possible that permittivity changes due to acidity changes may play an important role in the internal reflections, at the frequency that is closer to the Debye dispersion. Therefore, measurements of the complex permittivity of acid-doped ice are necessary in the MHz range. Particularly, measurements are strongly required in the HF band, in which permittivity changes due to acidity changes may be larger than at higher frequencies.

On the other hand, there are two AC electrical measurement techniques of ice core analysis. One is DEP (MOORE and PAREN, 1987) and another is AC-ECM (SUGIYAMA *et al.*, 1994), which used frequencies in the kHz range. To compare RES data and electrical measurement data from ice cores, dielectric properties of pure ice and ice which contains acid in the LF and HF are needed.

To clarify the dielectric properties of ice containing acid impurity, a new experiment was carried out. Frequencies between 1 kHz to 30 MHz were used for the experiment. As a result, a new data set, that is essential for obtaining and interpreting data from future HF ice radar, was obtained. This data set is also essential to compare the RES data and the result of ice core analysis.

## 2. Experiment

The complex permittivity (permittivity,  $\epsilon'$  and loss tangent,  $\tan\delta$ ) was derived by measuring the capacitance and dielectric loss of disk-shaped ice specimens. The system was composed of an HP-16451B electrode and precision LCR meters (HP-4284A and HP-4285A). The LCR meters were controlled and data were collected by a computer with GP-IB. We measured them at frequencies from 1 kHz to 30 MHz continuously and at 7 temperatures from  $-8.7^\circ\text{C}$  to  $-32.6^\circ\text{C}$ . Specimens were sandwiched between two flat electrodes which were kept parallel to each other. Thickness of specimens was about 7 mm. Diameters of the electrodes and specimens were 38 mm and about 50 mm, respectively. The electrodes had a guard electrode. From 1 kHz to 75 kHz, HP-4284A was used. From 75 kHz to 30 MHz, HP-4285A was used.

Two pure ice specimens and five acid-doped ice specimens were made from ion-exchanged water, and a mixture of the water and acid, respectively. Three types of acid, HCl,  $\text{HNO}_3$  and  $\text{H}_2\text{SO}_4$ , were doped respectively. We made two acid-doped specimens for HCl and for  $\text{HNO}_3$ , and one for  $\text{H}_2\text{SO}_4$ . They were frozen in a cold room at  $-20^\circ\text{C}$ . Densities of specimens ranged from 898 to 903 ( $\text{kg/m}^3$ ) at  $-20^\circ\text{C}$ . Air bubbles were ob-

served under the microscope, hence, density variation was considered to be induced by air bubbles. We determined the acid concentration from the electrical conductivity of melted specimens. The electrical conductivity was measured by a conductivity meter manufactured by Horiba Co. The acid concentration was between 3.9 ( $\mu\text{mol/l}$ ) and 10.5 ( $\mu\text{mol/l}$ ). These concentrations are similar to the acid concentration in ice cores (*e.g.* CLAUSEN and LANGWAY, 1989). We regarded conductivity of ion-exchanged water which was measured just before measuring that of melted specimens as a background noise. In order to prevent contamination, samples were handled with tweezers and measurements were carried out after acetone with which the electrodes were cleaned before the experimentation, was evaporated.

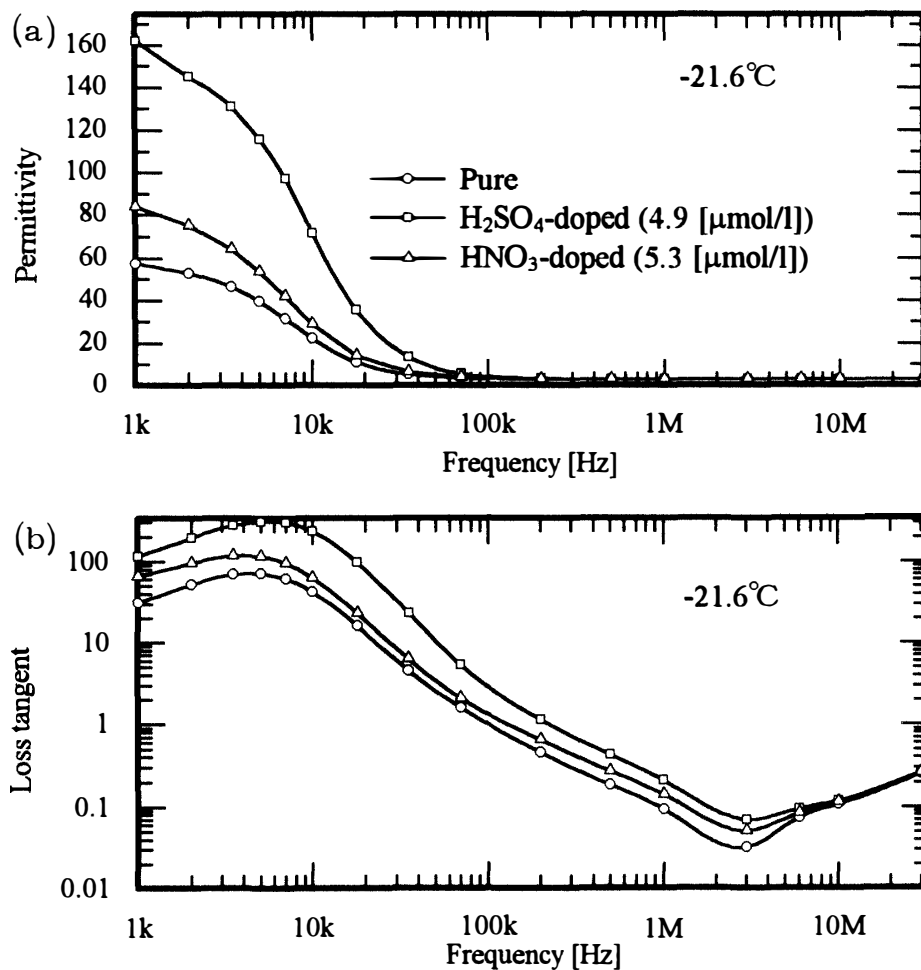


Fig. 1. The complex permittivity of pure and acid-doped ice at  $-21.6^\circ\text{C}$ . Data at 16 frequencies are chosen from 66 frequencies. The legends in (b) are the same as in (a).  $\epsilon'$  shown in this figure were corrected to that of ice of density  $917 \text{ (kg/m}^3\text{)}$  with the Looyenga equation (GLEN and PAREN, 1975), to eliminate the effect of the density.  $\tan\delta$  at frequencies higher than 5 MHz include the systematic error so that  $\tan\delta$  increases with frequency. However, the data were not rejected because increases of the complex permittivity arising from the presence of acidity in ice can be analyzed with these data, *i.e.* relative comparison between data at the same frequency and same temperature were not affected by the systematic error. We confirmed the validity of this by data comparison.

### 3. Results

Figure 1 is an example of the experimental data at  $-21.6^\circ\text{C}$ . At frequencies above 5 MHz, the measured data were affected by systematic error with which the circuit in the precision LCR meter changed. Therefore, absolute values of the data at frequencies above 5 MHz are not reliable. However, the data were not rejected because increases of the complex permittivity arising from the presence of acidity in ice can be analyzed with these data, *i.e.* relative comparison between data at the same frequency and at the same temperature was not affected by the systematic error. We confirmed this by comparison of data. Figure 1 shows that the dielectric properties of acid-doped ice at frequencies lower than several hundred kHz is dominated by the orientation polarization of ice molecules.

Figure 2 shows an example of relationships between the acid concentration and  $\epsilon'$  (a), and between the acid concentration and  $\tan\delta$  (b), at  $-21.6^\circ\text{C}$  and 2 MHz. A linear relationship was assumed as follows:

$$\epsilon' = \epsilon'_0 + \left(\frac{d\epsilon'}{dC}\right)C, \quad (1a)$$

and

$$\tan\delta = \tan\delta_0 + \left(\frac{d\tan\delta}{dC}\right)C, \quad (1b)$$

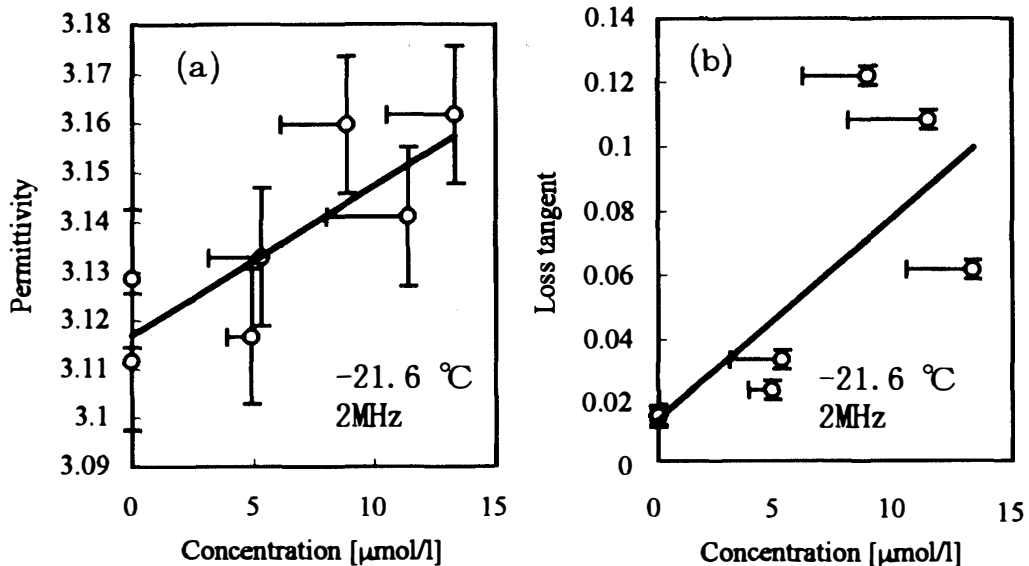


Fig. 2. An example of the relation between acid concentration and permittivity (a), and of loss tangent (b), at  $-21.6^\circ\text{C}$  and 2 MHz. Each symbol corresponds to a specimen, and lines are regression lines. Note that the unit of the concentration is micro molarity. Error bars of  $\epsilon'$  and  $\tan\delta$  indicate the dielectric anisotropy (FUJITA *et al.*, 1993). Error bars of concentration indicate the background noise in the concentration measurement. At  $-21.6^\circ\text{C}$  and 2 MHz,  $d\epsilon'/dC$  and  $d\tan\delta/dC$  are  $3.0 (\pm 0.1) \times 10^{-3}$  ( $l/\mu\text{mol}$ ) and  $6.4 (\pm 2.5) \times 10^{-3}$  ( $l/\mu\text{mol}$ );  $\epsilon'_0$  and  $\tan\delta_0$  are  $3.12 (\pm 0.01)$  and  $1.44 (\pm 1.97) \times 10^{-2}$ , respectively.

where  $\epsilon'_0$  and  $\tan\delta_0$  are  $\epsilon'$  and  $\tan\delta$  of pure ice. This assumption is based on the fact that previous studies have shown that the increase of  $\epsilon'$  and  $\tan\delta$  arising from the presence of acidity is a linear function of the molarity of acid (*e.g.* WOLFF and PAREN, 1984; FUJITA *et al.*, 1992; MOORE and FUJITA, 1993). No experimental data have contradicted this tendency. The proportional coefficients were denoted molar permittivity ( $d\epsilon'/dC$ ) and molar loss tangent ( $d\tan\delta/dC$ ).

Figure 3 shows the frequency dependence of  $d\epsilon'/dC$  (a) and  $d\tan\delta/dC$  (b) at  $-21.6^\circ\text{C}$ , as an example. As shown in Fig. 3 (a), from about 100 kHz (depends on the temperature) to 5 MHz,  $d\epsilon'/dC$  decreases with decreasing frequency. At higher frequencies than 5 MHz, the error in the linear regression is large and we can not determine whether  $d\epsilon'/dC$  is constant or not. As shown in Fig 3 (b), at higher frequencies than about 100 kHz (depends on the temperature),  $d\tan\delta/dC$  is almost inversely proportional to the frequency; for example, at  $-21.6^\circ\text{C}$ ,  $d\tan\delta/dC \propto f^{-0.95 \pm 0.03}$  with the standard error. This feature of the frequency dependence can be characterized by the high frequency tail of the Debye dis-

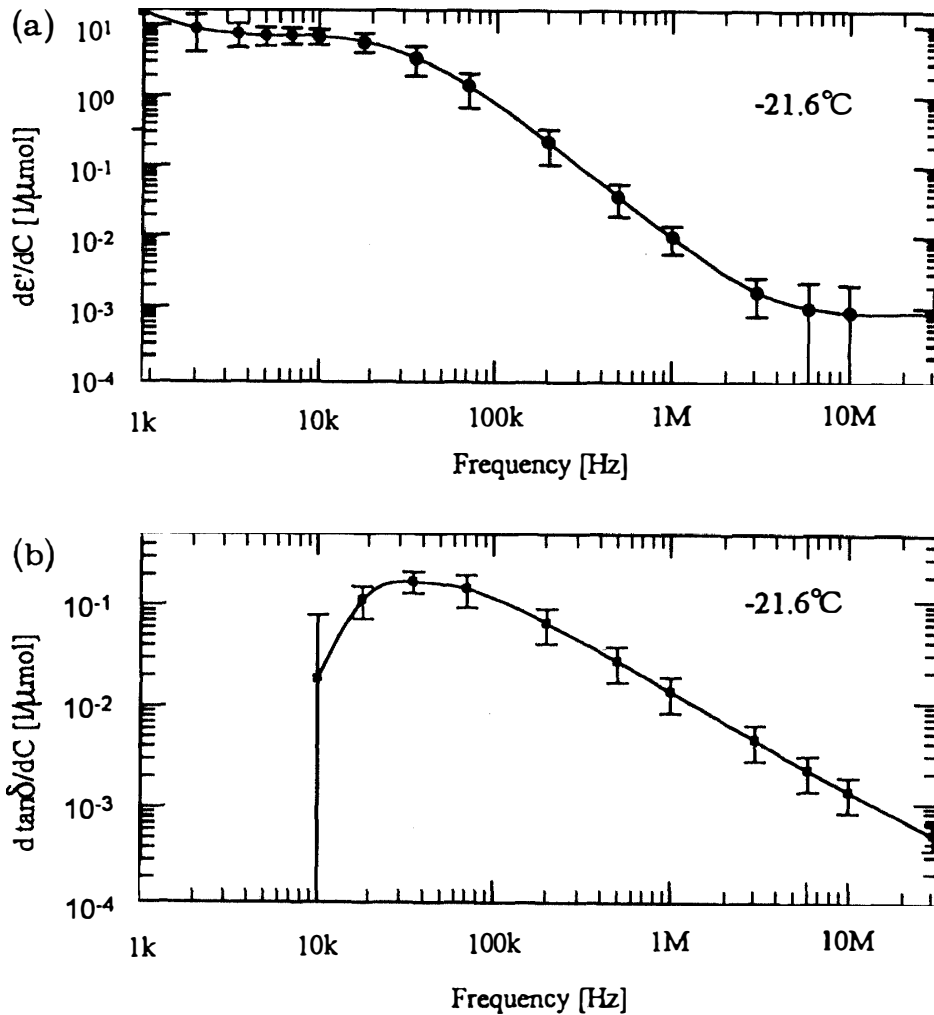


Fig. 3. Frequency dependence of molar permittivity ( $d\epsilon'/dC$ ) (a) and molar loss tangent ( $d\tan\delta/dC$ ) (b) at  $-21.6^\circ\text{C}$ . Error bars show the standard error in the linear regression.

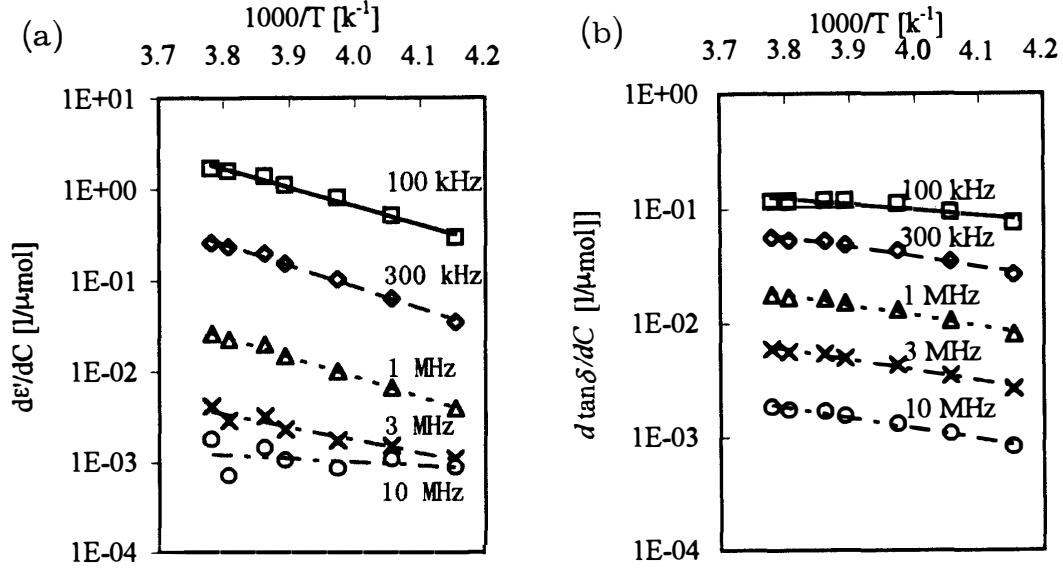


Fig. 4. Arrhenius plot of molar permittivity,  $d\epsilon'/dC$ , (a) and molar loss tangent,  $d\tan\delta/dC$ , (b). At 1 MHz,  $A$  and  $A'$  are  $7.82 \times 10^6$  and  $54.3$  ( $1/\mu\text{mol}$ );  $E_A$  and  $E_{A'}$  are  $4.28(\pm 0.12) \times 10^4$  and  $1.76(\pm 0.12) \times 10^4$  ( $\text{J/mol}$ ) with the standard error in the regression, respectively.

person. Detailed analysis of this dispersion will advance understanding of the mechanisms of increase of  $\epsilon'$  and  $\tan\delta$  arising from the presence of acidity.

Figure 4 shows the temperature dependences of  $d\epsilon'/dC$  (a) and  $d\tan\delta/dC$  (b) which are assumed to fit to the Arrhenius equations:

$$\frac{d\epsilon'}{dC} = A \exp\left(-\frac{E_A}{kT}\right), \quad (2a)$$

and

$$\frac{d\tan\delta}{dC} = A' \exp\left(-\frac{E_{A'}}{kT}\right), \quad (2b)$$

where  $k$  is the Boltzmann constant and  $T$  is the absolute temperature.  $E_A$  and  $E_{A'}$  do not depend on the frequency at higher frequencies than 100 kHz;  $E_A$  and  $E_{A'}$  are  $4.28(\pm 0.12) \times 10^4$  and  $1.76(\pm 0.12) \times 10^4$  ( $\text{J/mol}$ ) at 1 MHz with the standard error in the regression, respectively. Physical meanings of these assumptions have not been clear, however, eqs. (2a) and (2b) are used empirically in the following analysis. Table 1 shows  $d\epsilon'/dC$  and  $d\tan\delta/dC$  at some frequencies.

The temperature dependences of  $\epsilon'_0$  and  $\tan\delta_0$  also fit well to the Arrhenius equations. This fitting was done with corrected values to that of ice of density  $917$  ( $\text{kg/m}^3$ ) with the Looyenga equation (GLEN and PAREN, 1975). The physical meaning of fitting the temperature dependence of  $\epsilon'_0$  with the Arrhenius equation is not clear; however, we use this assumption empirically in the following discussion. Comparison of  $\epsilon'_0$  in the HF band with  $\epsilon'_0$  at 35 MHz and 60 MHz measured by JOHARI and CHARETTE (1975) showed that  $\epsilon'_0$  at the frequencies of this study is a little bit higher. JOHARI and CHARETTE (1975)

Table 1.  $d\epsilon'/dC$  and  $d\tan\delta/dC$ .

Freq. [MHz]	$d\epsilon'/dC \times 10^{-3} (//\mu\text{mol})$					$d\tan\delta/dC \times 10^{-3} (//\mu\text{mol})$				
	-10°C	-15°C	-20°C	-25°C	-30°C	-10°C	-15°C	-20°C	-25°C	-30°C
0.1	1710	1210	841	578	391	122	113	103	94.7	86.4
0.2	540	366	245	161	104	78.7	69.1	60.3	52.4	45.3
0.5	96.3	64.5	42.5	27.6	17.6	34.8	30.0	25.6	21.7	18.3
1	25.0	17.1	11.5	7.65	5.00	17.7	15.2	12.9	10.9	9.17
2	6.76	4.99	3.64	2.62	1.86	8.91	7.62	6.48	5.47	4.59
5	1.73	1.51	1.30	1.12	0.953	3.57	3.06	2.60	2.19	1.84
10	1.20	1.12	1.05	0.976	0.905	1.80	1.54	1.31	1.10	0.927
20	1.13	1.05	0.974	0.898	0.828	0.914	0.780	0.661	0.556	0.465
30	0.951	0.892	0.835	0.780	0.726	0.569	0.487	0.415	0.351	0.294

showed that, from  $-1^\circ\text{C}$  to  $-25^\circ\text{C}$ ,  $\epsilon'_0$  at 60 MHz is lower than  $\epsilon'_0$  at 35 MHz. For example, at  $-10^\circ\text{C}$ ,  $\epsilon'_0$  of this study at 1 MHz and 10 MHz are 3.22 and 3.19, respectively, and  $\epsilon'_0$  of JOHARI and CHARETTE (1975) at 35 MHz and 60 MHz are 3.187 and 3.176, respectively. It is possible that  $\epsilon'_0$  gradually decreases with frequency, in the MHz range.  $\tan\delta_0$  comparison at 35 MHz between an extrapolation of our measurement (assumed to be  $\tan\delta_0 \propto 1/f$ ) and that measured by JOHARI and CHARETTE (1975) showed that our  $\tan\delta_0$  is lower than  $\tan\delta_0$  measured by JOHARI and CHARETTE (1975). For example, at  $-10^\circ\text{C}$ ,  $\tan\delta_0$  extrapolated from our measurements is 0.0028 and  $\tan\delta_0$  of JOHARI and CHARETTE (1975) is 0.00403. It is possible that these differences are due to other dielectric dispersions in the MHz range which were suggested by JOHARI and CHARETTE (1975).

## 4. Discussion

### 4.1. Tendency of the dielectric dispersion arising from the presence of acidity

$\epsilon'$ ,  $\tan\delta$  and the relaxation frequency ( $f_r$ ) increase when ice contains acid. These phenomena can be explained as follows. Acid contained in the ice is at the three-grain boundaries as liquid phase (MULVANEY *et al.*, 1988). Therefore, it is plausible that a mixture of acid and water at the three-grain boundaries makes ice molecules around the boundaries rotate easier than pure ice molecules. Hence,  $f_r$  and  $\epsilon'$  at frequencies close to  $f_r$  increases. It is also plausible that  $\epsilon'$  of the mixture of acid and water at temperatures below  $0^\circ\text{C}$  is higher than  $\epsilon'$  of ice, since  $\epsilon'$  of the liquid water has a higher value than  $\epsilon'$  of ice in the MHz range. Both should contribute to the increases of  $\epsilon'$ ,  $\tan\delta$  and  $f_r$ . The latter contributes  $\epsilon'$  increases at frequencies not only close to  $f_r$  but much higher than  $f_r$ .

In this study, it was observed that  $d\epsilon'/dC$  decreases with increasing frequency. There are two possibilities to explain this:  $\epsilon'$  of the mixture depends on the frequency; the dielectric dispersion of acid-doped ice is broader than that of pure ice, *i.e.* there are other dispersions. It is required to separate both effects and to measure  $f_r$  and its dispersion strength. To clarify the effect of the mixture itself, it is necessary to measure  $\epsilon'$  of the mixture of acid and water at temperatures below  $0^\circ\text{C}$ . If we obtain  $\epsilon'$  of the mixture, we can calculate the effect of the mixture itself by calculating the mass of the mixture as WOLFF and PAREN (1984) did.

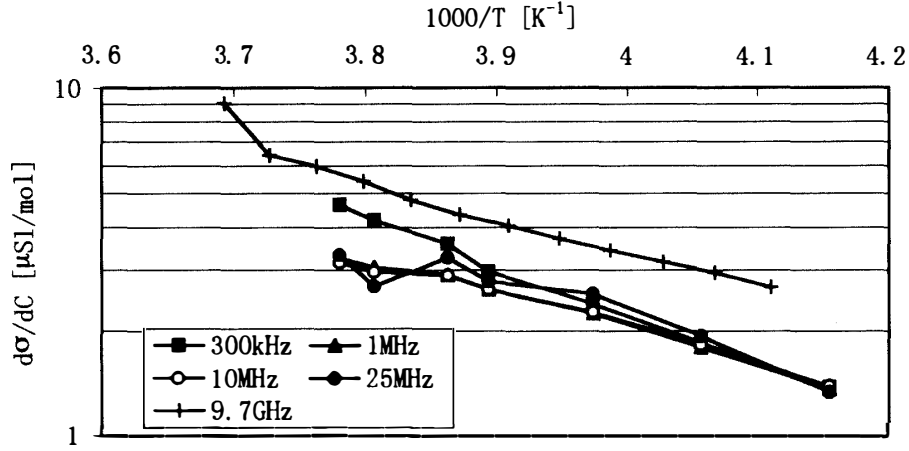


Fig. 5. Arrhenius plot of molar conductivity,  $d\sigma/dC$ . Data at 9.7 GHz are after MOORE and FUJITA (1993).

To clarify the frequency dependence of the dielectric properties of acid-doped ice in the wide frequency range, the results of this study and a previous study at microwave frequency are compared. Using the experimental data of this study, molar conductivity,  $d\sigma/dC$ , are derived with the following equation:

$$\frac{d\sigma}{dC} = 2\pi f \epsilon \left( \tan \delta_0 \frac{d\epsilon'}{dC} + \epsilon'_0 \frac{d \tan \delta}{dC} \right), \quad (3)$$

where  $\epsilon$  is the permittivity of free space. In Fig. 5,  $d\sigma/dC$  of this study and at 9.7 GHz (FUJITA *et al.*, 1992) are shown. At frequencies lower than about 100 kHz, the errors of  $\epsilon'$  are large and  $d\sigma/dC$  values are not reliable. Figure 5 indicates that  $d\sigma/dC$  in the HF band is smaller than that at 9.7 GHz. This suggests that ice which contains acid has other small dispersions in the MHz range. To clarify this point, it is necessary to measure the dielectric properties of ice which contains acid in the VHF and UHF.

#### 4.2. Reflection coefficient due to acidity changes in ice sheets

We assume a simple horizontal plane boundary within the ice sheets. Denote the permittivity and loss tangent of upper and lower layers by  $\epsilon'$  and  $\tan \delta$ , and  $\epsilon' + d\epsilon'$  and  $\tan(\delta + d\delta)$ , respectively. When radio waves are normally incident on the boundary, a reflection coefficient ( $R_s$ ) on the boundary is given by PAREN (1981) as

$$R_s = \frac{1}{16} \left[ \left( \frac{d\epsilon'}{\epsilon'} + \tan \delta d\delta \right)^2 + (d\delta)^2 \right]. \quad (4)$$

Using the differential relationship,  $d\delta = d \tan \delta / (\tan^2 \delta + 1)$ , eq. (4) becomes

$$R_s = \frac{1}{16} \left[ \left( \frac{d\epsilon'}{\epsilon'} + \frac{\tan \delta}{\tan^2 \delta + 1} d \tan \delta \right)^2 + \left( \frac{d \tan \delta}{\tan^2 \delta + 1} \right)^2 \right]. \quad (5)$$

From the experimental results, we obtain  $d\epsilon'$  and  $d \tan \delta$  as follows:



$$d\varepsilon' = \left( \frac{d\varepsilon'}{dC} \right)_{f,T} C, \quad (6a)$$

and

$$d\tan\delta = \left( \frac{d\tan\delta}{dC} \right)_{f,T} C, \quad (6b)$$

where  $C$  is the acidity change between layers expressed in micro molarity. The subscripts  $f$  and  $T$  of  $d\varepsilon'/dC$  and  $d\tan\delta/dC$  mean that  $d\varepsilon'/dC$  and  $d\tan\delta/dC$  are functions of the frequency and temperature. We put eqs. (6a) and (6b) into eq. (5) and we obtain

$$R_s = \frac{1}{16} \left[ \left| \frac{\left( \frac{d\varepsilon'}{dC} \right)_{f,T}}{\varepsilon'} + \frac{\tan\delta \cdot \left( \frac{d\tan\delta}{dC} \right)_{f,T}}{\tan^2\delta + 1} \right|^2 + \left| \frac{\left( \frac{d\tan\delta}{dC} \right)_{f,T}}{\tan^2\delta + 1} \right|^2 \right] C^2. \quad (7)$$

We assume that  $\varepsilon'$  and  $\tan\delta$  in eq. (7) are approximately those of pure ice. FUJITA and MAE (1994) estimated the maximum  $C$  in Antarctica as 7 ( $\mu\text{mol/l}$ ) from the results of ice core analysis. Substituting 7 for  $C$ , we obtain the dependence of the maximum  $R_s$  due to acidity changes in Antarctica on the frequency and temperature as shown by the solid lines in Fig. 6. Figure 6 shows that  $R_s$  increases with decreasing frequency.

On the other hand, if only  $\tan\delta$  changes,  $R_s$  is given by FUJITA and MAE (1994) as

$$R_s = \left| \frac{1}{4} \left( \frac{d\tan\delta}{dC} \right)_{f,T} C \right|^2. \quad (8)$$

$R_s$  calculation due to acidity changes has taken into account only for  $\tan\delta$  changes commonly in previous studies (*e.g.* PAREN and ROBIN, 1975; MOORE, 1988; FUJITA and MAE, 1994).  $R_s$  calculated with eq. (8) using the experimental data of this study are also shown

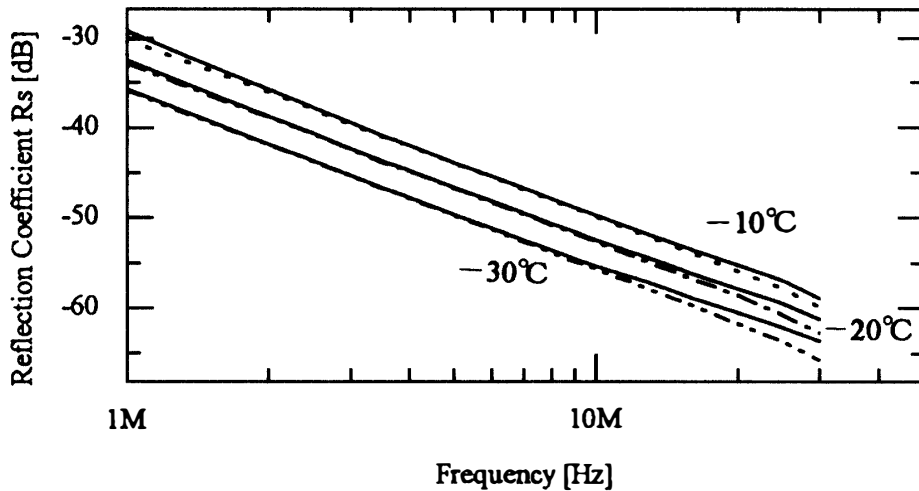


Fig. 6. Reflection coefficient,  $R_s$ , versus frequency. The solid line indicates the  $R_s$  calculated with eq. (7). The dashed line indicates that with eq. (8). Note that  $R_s$  is expressed in dB.

in Fig. 6. The maximum  $R_s$  difference between those calculated with eqs. (7) and (8) is 2.1 dB and the average is 0.4 dB. Therefore, it is valid to calculate  $R_s$  with eq. (8) in the HF band for practical usage. This result indicates that  $R_s$  due to acidity changes is almost inversely proportional to the frequency. This result also indicates that we can decide the cause of the reflection from the frequency dependence of the echoes in the multi-frequency RES.

To find the dominant cause of the reflections in ice sheets, it is required to compare  $R_s$  due to acidity changes with that due to other causes (density fluctuation and ice fabric changes) and to take the thickness of the layer into account. Moreover, to interpret the radar data, it is important to clear the scattering pattern. In this study, we simplified the reflections as reflections at the mirror plane, however, the scattering pattern of radio waves in the ice sheets has not been clear. Therefore it is important to couple the approach from the dielectric studies and the studies on scattering.

## 5. Conclusions

We measured the complex permittivity of pure and acid-doped ice from 1 kHz to 30 MHz and from  $-9^{\circ}\text{C}$  to  $-33^{\circ}\text{C}$ . Permittivity and loss tangent of ice which contains about 10 ( $\mu\text{mol/l}$ ) acid are influenced by the Debye dispersion at frequencies lower than several hundred kHz. Permittivity and loss tangent were assumed to depend linearly on the concentration. The temperature dependence of permittivity and loss tangent increases arising from the presence of acid is expressed empirically by the Arrhenius equation. The tendency for dielectric dispersion arising from the existence of acid impurity was investigated and dielectric measurement on the mixture of acid and water at temperatures below  $0^{\circ}\text{C}$  is required. A simple calculation shows that the reflection coefficient due to the acidity change increases with frequency decreasing in the HF. The effect of permittivity and loss tangent changes on the reflection coefficient due to acidity changes is quantitatively shown, respectively. The effect of permittivity changes on the reflection coefficient was less than +2.1 dB comparing the effect of loss tangent changes in the HF. As a result, a data set was obtained which is essential to develop HF ice radar and to interpret multi-frequency RES data, and to compare the RES data and ice core analysis data.

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