Experimental study on the rate dependent strength of ice-silica mixture with silica volume fractions up to 0.63

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We conducted deformation experiments of ice-1μm silica beads mixture to clarify the effect of silica beads volume fraction and temperature on the strength. The silica beads volume fraction was changed from 0 to 0.63 to simulate the surface of icy bodies. Unconfined uniaxial compression tests were made in a cold room at the temperatures from -10°C to -25°C and the constant strain rates ranged from 2.9×10^{-3} to 8.5×10^{-7}s^{-1}. We determined the rate dependent strength of the mixture written by $\dot{\varepsilon} = A \cdot {\sigma_{\max}}^{n}$ from the relationship between the maximum stress, $\sigma_{\max}$, on the stress-strain curve and the applied strain rate, $\dot{\varepsilon}$. The mixture with the silica volume fractions of 0.004-0.04 had almost the same strength with pure ice and the stress exponent, n, is about 3. On the other hands, at the silica volume fractions more than 0.15, the mixture became harder as the beads were more included, and it had the same stress exponent, about 6. This high stress exponent might be caused by crack generation. Also, we found that the A for the silica volume fractions more than 0.15 at -10°C was written by an exponential equation related to the silica volume fraction, ϕ, $A = 6.86 \times 10^{-8} \exp(-6.35\phi)$. Furthermore, we found that the n of ϕ=0.15 was independent of the temperature, and the brittle-ductile boundary of ϕ=0.29 and 0.63 was more than 30°C higher than that of pure ice.

1. Introduction

Water ice is distributed on the surfaces of Earth and Mars, and in many icy satellites of the outer solar system. It is revealed by recent planetary explorations that polar caps and layered deposits on Mars, and most of icy satellites have a rocky component in water ice, that is, they are composed of ice-rock mixture with various rock contents. Therefore, we must study the rheology of ice-rock mixtures to clarify the surface structures found on these extra-terrestrial bodies.

Brittle-ductile (B/D) boundary of ice-rock mixture is one of the most important rheological properties to determine the tectonic style on the surface of icy bodies: various tectonic forces have deformed their surfaces to develop the geological structures related to brittle failure and ductile deformation such as faults and glacial flows. The experimental study on the B/D boundary of pure ice was made by *Schulson*[1990] and *Arakawa and Maeno* [1997], but that of the mixture has not been studied yet.

The rate dependent strength of ice-rock mixture has been studied in relation to ice engineering, glaciological interests and planetological implications. Previous studies reported that the mixtures of ice and solid particles became softer or harder than pure ice at small solid particles content, while did simply harder with increasing the content at large content. *Hooke et al.* [1972] and *Durham et al.* [1992] studied the strength or the viscosity of ice-sand mixture at the volume fractions from 0.0 to 0.6, and revealed that the mixture became weaker than pure ice as the volume fraction increased from 0 to 0.1-0.2, and above that, the mixture became harder.

Mangold et al. [2002] confirmed that the strength of ice-sand mixture at the volume fractions from 0.5 to 0.7 was about ten times larger than that of pure ice. They experimentally derived a flow law of ice-sand mixture, and the stress exponent showed 2.7, which was almost the same as that of polycrystalline water ice. The stress exponent is one of the most important factor to control the strength of the mixture. However, *Hooke et al.* [1972] and *Durham et al.* [1992] assumed the stress exponent, 3 or 4, to derive an enhancement factor of the flow law of ice-sand mixture. Thus, we must determine the stress exponent of the equation showing the rate dependent strength of ice-sand mixture experimentally, particularly we should study the dependence of sand content.

In this study, we carried out deformation experiments of ice-solid particles mixture systematically to investigate the effects of solid particles on the B/D boundary and the stress exponent of the equation showing the rate dependent strength near the B/D boundary.

2. Experimental Methods

The sample of ice-solid particle mixtures was prepared by mixing ice grains with silica beads. Ice grains were produced by crashing commercial ice blocks into small pieces by using a blender. They were sieved to sort the grain size between 0.3 to 1mm. The silica beads have a smooth spherical shape with the diameter of 1μm to exclude the effects of the shape and the size distribution of solid particle. It is widely accepted that the micron sized silicate dusts were very common in the solar nebula to be incorporated into icy bodies. Moreover, the surface of Mars is covered with micron sized dust that could be included in PLDs [*Neakrase et al.*, 2006]. We prepared the samples with the silica beads volume

fraction, ϕ, of 0.004, 0.04, 0.15, 0.29, 0.49, and 0.63, and also did the polycrystalline ice sample without silica beads, ϕ=0.

The sample preparation method is described as follows. At first, the ice grains are mixed with silica beads homogeneously. Then, a few amount of this mixed grains are put into a cylindrical mold and it is filled with liquid water at the temperature of 0°C. We repeat this process until the mixture fills the mold, and then freeze it in a cold room of -10°C for more than a day. We observed the sample texture by a microscope, and found that ice matrix which was the frozen water with silica beads was distributed among the ice grain boundaries. In the case of ϕ=0.49 and 0.63, the preparation method is different from others: we pour the suspension of silica beads into the mold and then, freeze it. The sample has a cylindrical shape with the diameter of ∼30mm and the height of ∼60mm. There are unavoidable pores in the sample The porosity before deformation was about 3±2%, and after deformation, it changed less than a few %, so the porosity has little effect on the strength.

We conducted unconfined uniaxial compression tests under constant strain rates by using the universal testing machine (TENSILON UCT-2.5). To examine the temperature dependence of the strength and the B/D boundary, we changed the cold room temperature from -10 to -25°C. The strain rates that we conducted in this experiment ranged from 2.9×10^{-3} to $8.5\times10^{-7}s^{-1}$. We are especially interested in the stress exponent of the rate dependent strength, so that we made intensive tests at -10°C to derive the dependence of the stress exponent on the silica volume fraction. Moreover, this temperature of -10°C and the unconfined condition are suitable for comparing our results with those of

Mangold et al.[2002] in order to study the effect of confining pressure, and these strain rates are suitable for comparing our results with that of *Durham et al.*[1992] obtained at low temperatures and high confining pressures.

3. Results and Discussions

3.1. Deformation features

The stress-strain curves of the mixtures with different silica volume fractions at the same strain rate show the same trajectory before the stress reaches the maximum (auxiliary material Figure S1). Firstly, the stress linearly increases with the strain, and then the slope of the curve gradually approaches to zero, and the stress indicates the maximum. After the maximum, the stress decreases with increasing the strain, and then it becomes constant at small silica volume fractions, while it remains to decrease at large silica volume fractions. Moreover, at ϕ=0.63, the stress decreases similarly but rises again at large strain. The rate dependent strength of ice was found to be expressed by the relationship between $\dot{\varepsilon}$ and $\sigma_{\max}$ as follows [*Arakawa and Maeno*, 1997],

$$\dot{\varepsilon} = A \cdot {\sigma_{\max}}^{n}, \tag{1}$$

where $\dot{\varepsilon}$ is the strain rate, $\sigma_{\max}$ is the maximum stress, and A in the unit of $\mathrm{s}^{-1}(\mathrm{MPa})^{-n}$ and n are called as the flow parameters in this study. The rate dependent strength of ice in the ductile deformation without any crack generation is described by a creep flow law at low strain rates, however, near the B/D boundary, the deformation causes micro-cracks among grain boundaries even when ice is still ductile, and they might affect the strength.

Sinha [1988] theoretically constructed the rheological model of pure ice with micro-crack generation based on the elastic deformation and the creep flow law, and successfully applied the model to the deformation test at constant strain rates. Then, it showed that the stress exponent of Eq.(1) was almost the same as that of the creep flow law. Furthermore, *Mellor and Cole* [1982] proposed that the rate dependent strength shown by Eq.(1) corresponded to the flow law constructed by creep tests when Eq.(1) was obtained by the unconfined compression test under a constant strain rate and near the B/D boundary. These previous studies pointed out the close relationship between the creep flow law and the rate dependent strength of pure ice. Then, we expect the same close relationship between them for the mixture hereafter.

3.2. Effects of silica beads volume fraction on the rate dependent strength

Figure 1 shows the relationships between the maximum stress and the strain rate of ice-silica beads mixtures with various silica volume fractions and pure ice at the constant temperature of -10°C. The each data set with different silica volume fractions is shown in auxiliary material Table S1, and is fitted by Eq.(1). The fitted lines are shown on this figure for the reference.

At the silica volume fractions less than 0.04, the mixtures have almost the same strength as pure ice, that is, the maximum stresses become the same over the whole range of strain rates. However, at the silica volume fractions more than 0.15, the mixtures become harder as more silica beads are included, that is, the maximum stresses become larger as the silica volume fraction increases. As the strain rate increases, the difference of the maximum stress between pure ice and the mixture becomes smaller, in turn, the maximum stresses

of ϕ=0.15-0.49 are close to that of pure ice, about 3-4MPa at the strain rate of about $10^{-4}s^{-1}$. In the case of ϕ=0.63, the maximum stress is larger than that of pure ice over the whole range of strain rates in this study. We notice that the order of the strength changes with the strain rates. This order is determined by the parameters of Eq.(1): they are the maximum stress on the fitted line at a standard strain rate, A, and the slope of the fitted line, n. So next, we examine the dependence of A and n on the silica volume fraction. The relationships of the silica volume fraction vs. the constant A or the power law index n are listed in Table 1.

3.2.1. Constant A

At the silica volume fractions less than 0.04, the A is almost same as that of pure ice. However, the A abruptly drops between 0.04 and 0.15, and then it decreases with increasing the silica volume fraction at more than 0.15. In the previous creep experiments of pure ice, the A represents the effect of temperature on the flow law, and also relates to the physical properties so called as enhancement factor, like crystal orientation, impurity contents and other factors [*Paterson*, 1994]. The enhancement factor, which is proportional to the A, caused by solid particles included in water ice has been measured by *Durham et al.* [1992] and it was found to be expressed by the exponential equation of the solid particles content. In this experiment, we found that the A exponentially decreased with increasing the silica volume fraction at more than 0.15. For example, the A for ϕ=0.49 becomes two orders of magnitude smaller than that of pure ice. So we fit these data by exponential equation and obtain as follows,

$$A = 6.86 \times 10^{-8} \exp(-6.35\phi). \quad (2)$$

The A represents the strain rate at 1MPa, so that we expect that the mixture can deform easily as the A becomes larger, although the actual strength also depends on n.

3.2.2. Power law index n

At the silica volume fractions less than 0.04, the n of the mixtures is almost constant of 3-4, which is same as that of pure ice. On the contrary, the n suddenly rises up from 3 to 6 when the silica volume fraction changes from 0.04 to 0.15, and those of ϕ=0.15-0.63 become almost constant, about 6.5.

These results are consistent with the results of previous works at the silica volume fractions less than 0.04 [*Hooke et al.*, 1972; *Durham et al.* 1992; *Mangold et al.*, 1999]. They also concluded that the values of n with the solid particles volume fractions from 0 to 0.04 were nearly equivalent with that of pure ice, n=3. However, our results are not consistent with previous results at the silica volume fractions more than 0.15. For example, *Li et al.* [2005] reanalyzed the results of *Hooke et al.* [1972] that the values of n with the volume fractions from 0 to 0.6 decreased with increasing the volume fraction according to the equation of $n = 3 - \phi$. However, the n of our results increases with the silica volume fractions from 0.04 to 0.29, so are contrary to previous results. Additionally, the n of ϕ=0.49 of our result is twice as large as that of *Mangold et al.* [2002]: the n they obtained was about 2.7 which is equivalent to pure ice, while that we obtained was about 7.

The result of *Mangold et al.* [2002] for the mixture of ϕ=0.52 is shown to compare with our result of ϕ=0.63 on Fig.1. The main difference of our experimental condition from that of *Mangold et al.* [2002] is a confining pressure. It is well known that the confining pressure reduces micro-crack generation and enhances the ductile deformation, so that we infer that the micro-cracks reduce the strength to cause the high values of n in our mixtures with large silica volume fractions. However, *Sinha* [1988] and *Arakawa and Maeno* [1997] showed that the micro-crack generation in pure ice did not change the stress exponent so much. According to Sinha's simulation, the number density of micro-crack was quite small until the stress achieved to the maximum, that means the maximum was caused by the ductile creep of ice, then the micro-cracks drastically increased to reduce the strength with increasing the strain. On the other hand, we speculate that at unconfined conditions the micro-cracks in the mixture might start to increase from the beginning of the deformation because of the strong stress concentration on silica inclusions, so that the confining pressure might strongly affect the stress exponent.

3.3. Effect of temperature on the rate dependent strength

The relationship between the maximum stress and the reciprocal of temperature with various silica volume fractions at the constant strain rate of $1.4\times10^{-5}\mathrm{s}^{-1}$ is shown in Fig.2, and auxiliary material Table S1. The maximum stress increases as the temperature becomes lower at each silica volume fraction, so we can fit these data by exponential equation. It is found that the slope of the line becomes larger as the silica volume fraction increases, that is, ice-silica mixture becomes more harder as the silica volume fraction increases. The data of ϕ=0.15 can be fitted by one exponential equation over the whole

range of temperature in this study, and furthermore at other strain rates, these data can be fitted similarly.

We also examined the power law index n of ϕ=0.15 at the temperature lower than -10°C, and the obtained n at different temperatures are listed in Table 1. It is found that the n is almost constant irrespective of the temperature. The fitted lines on Fig.2 are described by the following equation according to the previous study on the rate dependent strength [*Arakawa and Maeno*, 1997],

$$\sigma_{\max} = \left(\frac{\dot{\varepsilon}}{A_0}\right)^{1/n} \exp\left(\frac{Q}{nRT}\right), \tag{3}$$

where R is the gas constant, and T is the absolute temperature. According to Eq.(3), the slopes of these lines are corresponding to Q/nR. Thus, Q/nR is derived to be 6.4×10^3 for ϕ=0.63, 3.9×10^3 for ϕ=0.29 and 2.1×10^3 for ϕ=0.15. We can calculate the activation energy, Q, only for ϕ=0.15 by using the n, which was determined at the corresponding temperature range: the average value was n=6.2. As a result, the Q is calculated to be 129 kJ/mol and is close to that of pure ice at the temperature higher than -8°C [*Barnes et al.*, 1971]. From this previous work, we know that the deformation mechanism of pure ice at higher than -8°C is grain boundary sliding among ice crystals. So, the deformation mechanism of ϕ=0.15 may also be grain boundary sliding even at the temperature lower than -10°C.

We notice that in Fig.2 the deformation type of ϕ=0.29 and 0.63 changes from ductile deformation to brittle failure as the temperature is -25°C. Then, the maximum stress is almost the same when the deformation type shows brittle failure. The deformation types

of ϕ=0.29 and 0.63 are shown on Fig.3, and in auxiliary material Table S1. The B/D boundary of these mixtures is proposed as follows [*Arakawa and Maeno*, 1997],

$$\dot{\varepsilon}_{\rm bd} = A_{\rm bd} \cdot \exp\left(-\frac{E_{\rm bd}}{RT}\right), \tag{4}$$

where $A_{\rm bd}$ is a constant of 1.62×10^{15} s^{-1}, and $E_{\rm bd}$ is a constant of 95.6 kJ/mol. Comparing Eq.(4) with that of pure ice obtained by *Arakawa and Maeno* [1997], we can recognize that the boundary temperature is 30-50°C higher than that of pure ice at the same strain rate. Therefore, we expect that ice-silica beads mixtures with higher silica volume fractions could induce the brittle failure easily in comparison with pure ice, and this feature might correlate with many faults found on the surface of icy bodies such as Europa, Callisto and many Saturnian icy satellites.

Acknowledgments. We thank Prof. N. Azuma of Nagaoka University of Technology, for his useful advices and discussion and S. Nakatsubo of the Contribution Division of the Institute of Low Temperature Science, Hokkaido University, for his technical help. This work was partly supported by a grain-in-aid for scientific research (Grant 17340127) from the Japan Ministry of Education, Science, Sports, and Culture, and the Grant for Joint Research Program of the Institute of Low Temperature Science, Hokkaido University.

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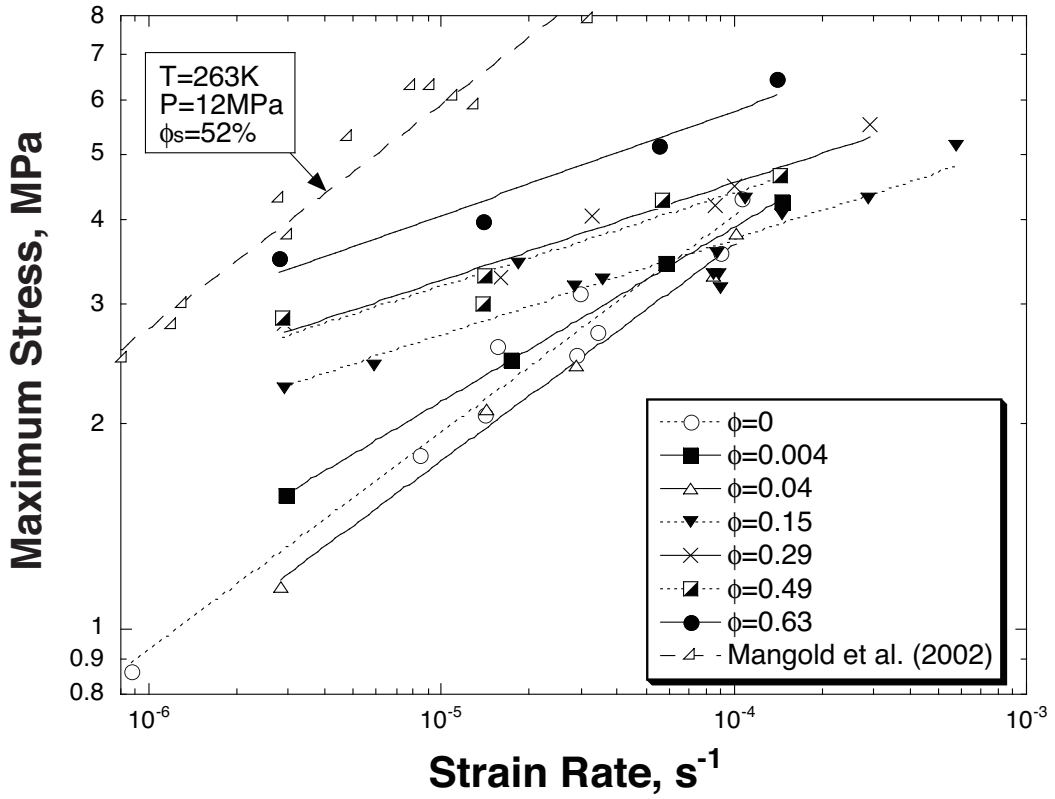

Figure 1. Maximum stress versus strain rate for pure ice sample (open circles) and ice-1μm silica beads mixtures with the silica volume fractions of 0.004 (filled squares), 0.04 (open triangles), 0.15 (filled inverted triangles), 0.29 (X), 0.49 (half-filled squares), and 0.63 (filled circles) at the temperature of -10°C. The lines are fitted by Eq. (1) at each silica volume fraction according to the least square method.

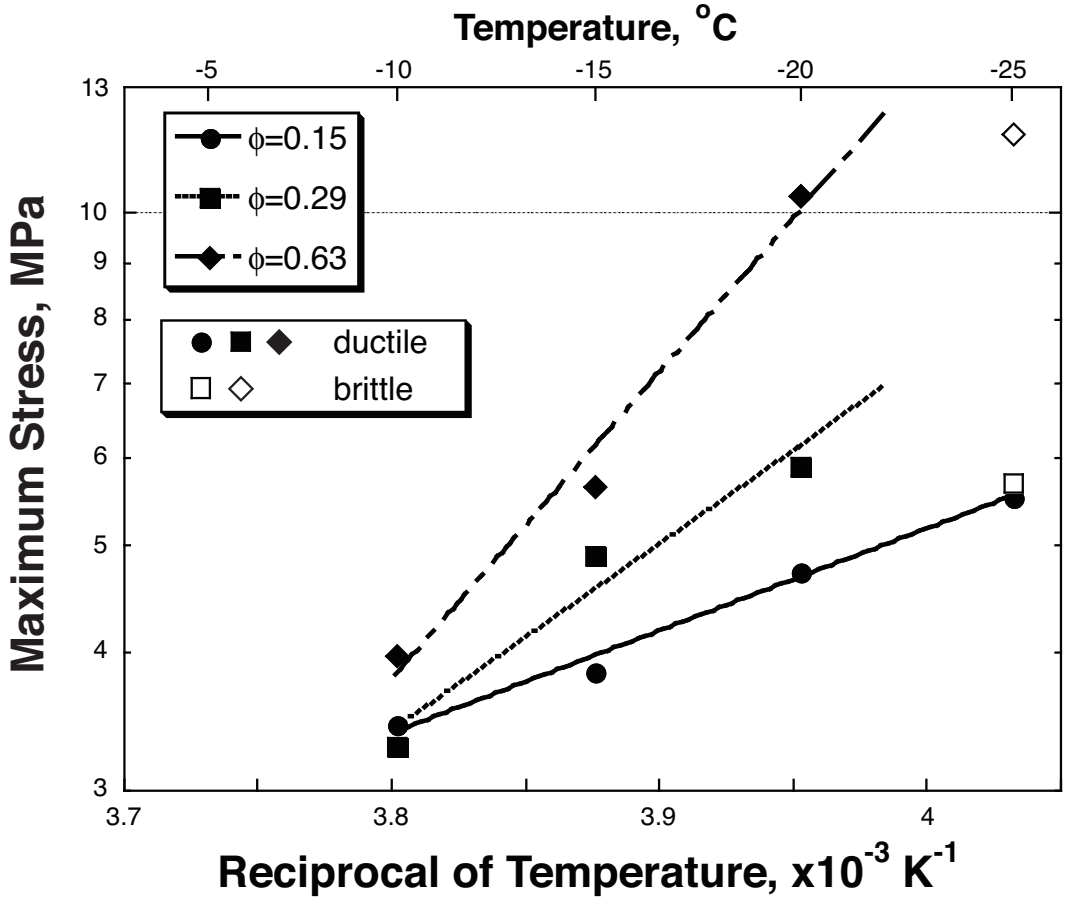

Figure 2. Temperature dependence of the strength for the mixtures with different silica volume fractions of 0.15, 0.29, 0.63. Filled symbols show ductile deformation, and open symbols show brittle failure.

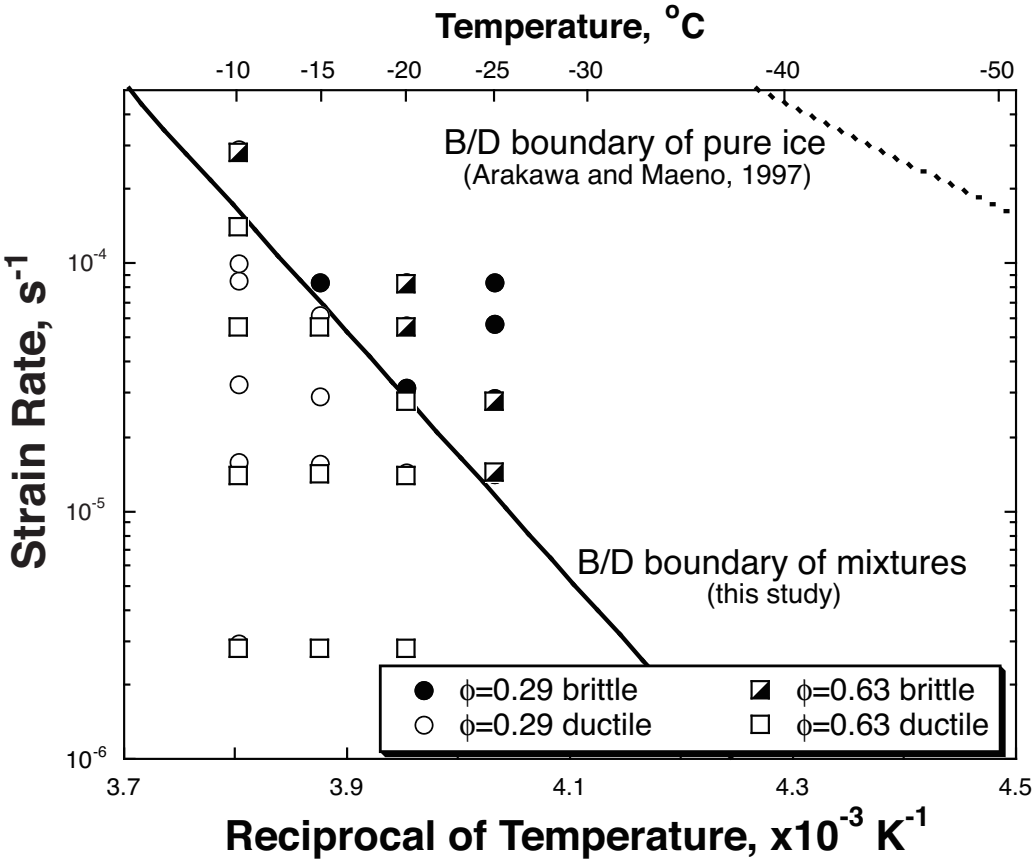

Figure 3. Deformation types of ϕ=0.29 and 0.63. The previous result of the B/D boundary of pure ice is plotted by the dashed line [*Arakawa and Maeno,* 1997].

Table 1. Constant A and power law index n of Eq.(1)

Silica volume fraction, ϕ	Temperature (°C)	A ($s^{-1}(MPa)^{-n}$)	n
0	-10	1.35×10^{-6}	2.84 ($\pm$0.25)
0.004	-10	4.97×10^{-7}	3.90 ($\pm$0.05)
0.04	-10	1.70×10^{-6}	3.12 ($\pm$0.18)
0.15	-10	3.11×10^{-8}	6.05 ($\pm$0.51)
0.15	-15	1.24×10^{-9}	6.84 ($\pm$1.21)
0.15	-20	9.81×10^{-10}	6.37 ($\pm$0.78)
0.15	-25	8.98×10^{-10}	5.62 ($\pm$0.45)
0.29	-10	4.00×10^{-9}	6.67 ($\pm$0.45)
0.49	-10	5.02×10^{-9}	6.60 ($\pm$0.84)
0.63	-10	1.88×10^{-9}	6.15 ($\pm$0.71)