



## **STANDARDIZATION OF LABORATORY EXPERIMENTAL TECHNIQUES WITH A COLD ROOM IN KOREA**

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### **ABSTRACT**

The first Korean cold room facility for ice mechanics experiments was assembled in 2004. Since then, the 4 m x 6 m cold room facility has been extensively used under various different environmental and loading conditions. After reviewing published references on cold room testing methods and also by trial and error, the standard procedures for testing and preparing laboratory ice material were established for the measurement of basic ice properties. In this paper, laboratory experimental techniques with the cold room facility and standard procedures established for ice material properties are introduced. Test specimens include laboratory-grown fresh water ice and frozen soils. Tests are carried out for unconfined compressive strength. Preparation and dimension of the specimen are the most important issues arising in cold room tests. The details of specimen preparation, testing procedure and strength test results are also discussed.

**KEY WORDS:** Cold room; Standard experimental procedures; Fresh water ice; Frozen soil; Uniaxial compressive strength

### **INTRODUCTION**

The cold room is an essential part of the laboratory facilities for ice research and cold regions engineering. The experiment with a cold room includes various tests of materials at low temperature, whose range is often encountered in the Arctic. As a non-Arctic country, the first cold room for ice mechanics research was recently built in the Arctic Research Laboratory (ARL), at the Korea Maritime University (KMU), in Busan, Korea. The 4 m×6 m×2.6 m cold

room has served as a basic tool for ice and cold regions study in Korea. Since its opening in late 2004, ARL-KMU has been conducting a year-long study for developing standardized experimental techniques utilizing the new cold room. This paper introduces laboratory experimental techniques with the cold room facility and standard procedures established for observing ice properties. Unconfined compression tests were carried out for measuring the strength of laboratory-grown fresh water ice and frozen soils. Preparation and dimension of specimen were the most important issues during cold room tests. Present work deals with specimen preparation, testing procedures and uniaxial strength test results under constant deformation rate compression. It is the intent of this paper to set up a standard method for creating repetitive environmental conditions through cold room experiments and it is not our aim to obtain and to make a quantitative comparison. As of this writing, additional tests are being performed to further obtain the refinement of test procedures.

## **APPARATUS AND TEST METHODS**

### **Cold Room Facility and Specimen Preparation**

The ARL-KMU cold room is built on the ground floor inside a laboratory building. The chamber is surrounded by insulated panels and the air temperature inside the chamber can be controlled by an outside digital controller within the range of  $-30^{\circ}\text{C} \sim +15^{\circ}\text{C}$ . Accuracy level of the temperature controller is  $0.1^{\circ}\text{C}$ . Inside the cold room, a 20 ton capacity UTM(universal testing machine) is located for compression and flexural tests. Crosshead speed of the UTM is controlled within the range of  $0 \sim 200$  mm/min. Other equipments inside the chamber include a microtome and a band saw for thin section preparation from ice specimen, and a photography apparatus kit using crossed polarizing light.

The most difficult part was to prepare specimens for the cold room experiment. Without prior knowledge on particular freezing sequences and procedures, we reviewed available references and articles on cold room experiments and making of ice specimens. Because no tests with iceberg or sea ice sample brought from Arctic fields were planned yet, we first focused on laboratory-grown ice and frozen soil specimens for uniaxial compression test. For the case of brittle materials such as concrete, the length of a cylindrical specimen for compression tests is usually 1.0-1.5 times the diameter. The size of ice specimens used for test suitable to a UTM in the cold room was chosen to be 70 mm and 100 mm in diameter and 110 mm, 140 mm in length. Molds used for freezing water inside are made of transparent acryl cylinders of 5 mm in thickness. An acryl mold is 100 mm longer than ice specimen produced for cutting away and trimming. Photo. 1 shows ice specimens prepared for the compression test.

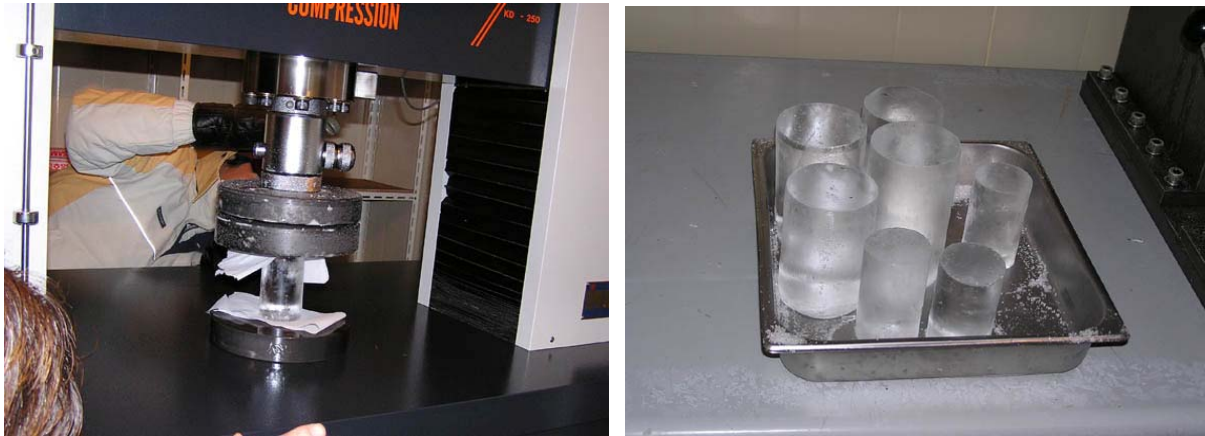


Photo. 1 Testing of fresh water ice specimens prepared for the compression test

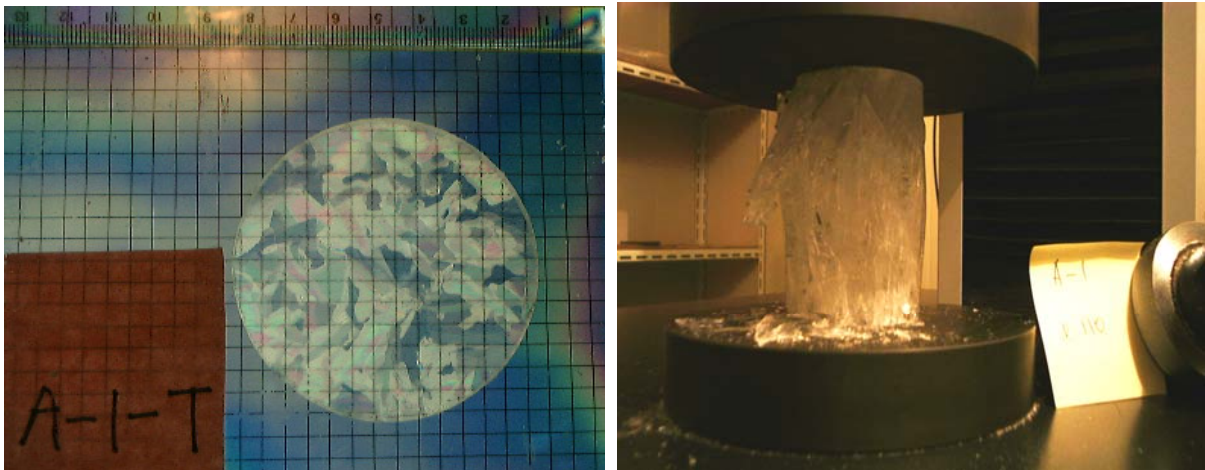


Photo. 2 Thin section photograph of typical untested ice specimen (diameter = 70 mm) and photograph of failed specimen after compression test

Ice specimens were made by using the temperature-controlled method. Some methods, such as the one described by Cole(1979), prefer to use seed crystals for controlling the specimen grain size. Here the temperature-controlled method is used to remove air bubbles from water inside the mold and to obtain bubble-free transparent ice specimens. Since air bubbles are easily trapped inside ice when freezing action is fast, it is necessary to cool down the entire mold very slowly. Five-fold cloths served as an insulating material on the side and bottom parts of the mold. Filtered tap water around  $4^{\circ}\text{C}$  was poured in the mold and was stirred by a stick to eliminate air bubbles on the surface of the mold. Then the molds were kept in the cold room for approximately 3~5 days at  $-15^{\circ}\text{C}$ . Ice grows from top to bottom uni-directionally and air bubbles are trapped at the bottom part of ice specimen, which is later cut away before test begins. The ice specimens produced were stored in the freezer at desired test temperatures.

This method produced ice specimens with large grain size. A thin section was cut from each

ice specimen used in the tests, and photographed (Photo. 2). The grain size was determined by measuring the number of grain intersections on a diameter of thin section photograph. The average grain size was 12.7 mm.

Frozen soils are important in view of the application of cold room facility beyond ice test. Preparation of frozen soil specimen is somewhat different from that for ice itself. Frozen soil specimen constitutes standard sand and kaolinite, a sort of clay. First sand and kaolinite were mixed at different component ratios, 10%, 20%, 30% and water was added to give different water content ratios, i.e., 7%, 15% and 20%. A total of 9 specimens were made for the frozen soil test. The size of each specimen was  $\phi=100$  mm in diameter and  $l=200$  mm in length. By hammering 25 times at a height of 30 cm (standard hardening procedure), about 1/3 of the specimen in the mold, containing a mixture of soil and water was hardened. This procedure was repeated 3 times to make the standard frozen soil specimen. Molds were kept at  $-15^{\circ}\text{C}$  in the cold room for approximately 7 days. The soil specimen was drawn from the mold using an oil jack extractor and was stored in a freezer at a desired test temperature.

### **Uniaxial Compression Tests for Fresh Water Ice**

The uniaxial compressive strength is an important ice property that changes much according to temperature, strain-rate, grain size, impurity content and crystallographic orientation, and so on. Regarding the compression tests of ice material, there are many references and test data available so far. The most relevant previous works are the papers of Hawkes and Mellor (1972), Hooke et al.(1980), Mellor and Cole(1982, 1983), Jones(1982), Timco and Frederking(1982), Schulson and Cannon(1984), Cole(1985) and so on.

In this study, only the uniaxial unconfined compressive strength was considered. The peak stress was recorded during the compression tests of ice specimen. Table 1 summarizes the test result. The strength data is organized as a function of varying loading rates converted to strain-rates. Measured compressive strengths were 9.4MPa at average. There are two distinctive responses, i.e., brittle and ductile behavior as shown in Fig. 1, typical stress-strain curves for different strain-rates. Even though the exact transition point from one characteristics to the other is not known, the strain-rate seem to be a key factor for brittle-to-ductile transition phenomena as many researchers pointed out (For example, refer to Jones et al., 2003). In Fig. 2 peak compressive stresses are plotted against strain-rates. When the strain-rates were low enough, it was found that the compressive strengths varied much depending on strain-rates. However, because of the lack of data especially in the lower strain-rates region, it was not enough to say whether it followed the well-known “power-law”.

Table 1 Strength data of fresh water ice versus strain-rates

Specimen	T (°C)	Strain-rate (s <sup>-1</sup> )	$\sigma_c$ (MPa)	Specimen	T(°C)	Strain-rate (s <sup>-1</sup> )	$\sigma_c$ (MPa)
4-A9	-15	3.15E-04	9.982	2-A1	-15	7.58E-05	5.364
3-A1	-15	3.03E-04	13.862	2-A2	-15	7.58E-05	6.358
3-D1	-15	3.03E-04	13.194	4-A3	-15	7.58E-05	10.167
2-A6	-15	3.03E-04	11.735	3-B2	-15	5.95E-05	10.932
4-A8	-15	2.85E-04	10.831	2-B6	-15	5.95E-05	9.734
4-A7	-15	2.60E-04	9.712	4-A5	-15	5.01E-05	6.214
3-B1	-15	2.30E-04	9.441	3-C2	-15	4.20E-05	9.097
4-B1	-15	2.30E-04	7.899	2-B1	-15	1.21E-05	10.193
4-A2	-15	1.80E-04	10.065	2-B2	-15	1.21E-05	9.11
3-C1	-15	1.70E-04	11.582	2-B3	-15	1.21E-05	8.83
2-A3	-15	1.52E-04	9.67	2-B4	-15	1.21E-05	11.059
2-A5	-15	1.52E-04	9.161	2-B5	-15	1.21E-05	11.862
4-A1	-15	1.50E-04	9.887	3-A4	-15	7.58E-06	6.621
4-B2	-15	1.20E-04	9.174	4-A4	-15	7.58E-06	7.925
4-A6	-15	1.00E-04	9.012	3-B3	-15	5.95E-06	5.952
3-D3	-15	7.58E-05	11.666	4-B3	-15	5.90E-06	7.976

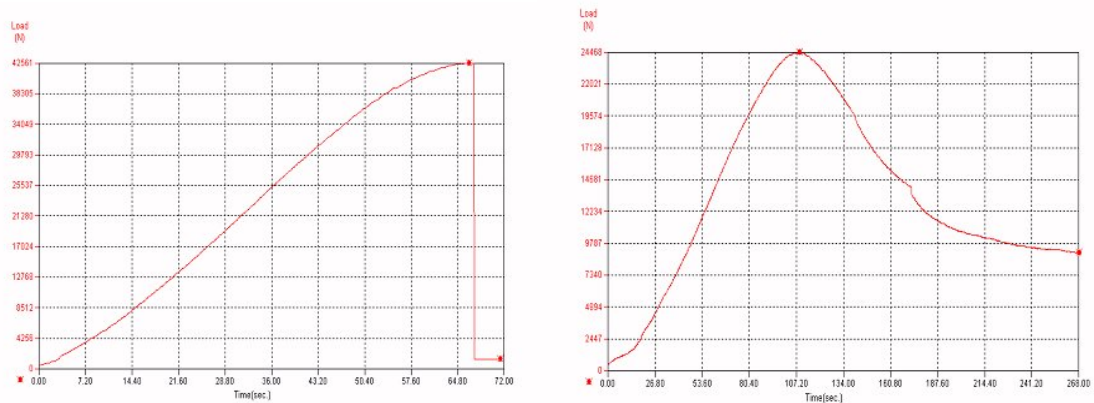


Fig. 1 Typical stress-strain curves for brittle and ductile failure modes

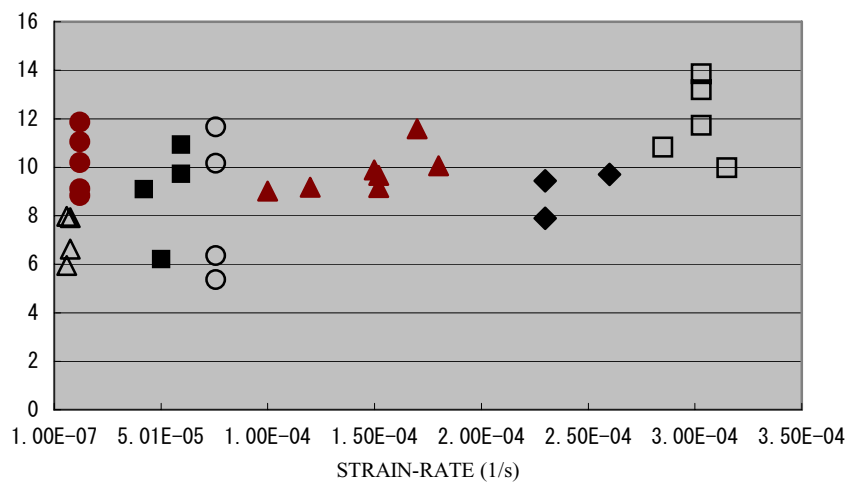


Fig. 2 Compressive strength (MPa) versus strain-rates (s<sup>-1</sup>)

## Uniaxial Compression Test for Frozen Soil

Frozen soils have a characteristic of ice when water is frozen in the pore of the soil. When the water is frozen in thick gravel, the strength gets very high such as the concrete. The strength of frozen pore water has a close relationship with temperature and type of soil. In case of soils of high porosity, their strengths are comparable to that of pure ice (Freitag and Mcfadden, 1997). Compression test can be performed for studying experimental techniques on frozen soils using cold room facility. It is useful since the compression test on frozen soils gives an insight of material behaviors like ice. In this study unconfined uniaxial compression test was performed on frozen soils by varying water content ratios and mixing component ratios. The test was done at the loading rate of 4 mm/min, i.e., 2% of specimen length.

Fig. 3 depicts stress-strain curves recorded during the test for soil specimens. In Fig. 3(a), the stress-strain curves are shown when the kaolinite contents are 10%, 20%, 30% respectively. As water content ratio increases, the compressive strength of the soil increases. When the water content ratio increases from 7% to 15%, the compressive strength increases approximately 2 times. However when the water content ratio increases from 15% to 20%, the strength increment is small. It seems that the compressive strength increases in general with increasing the water content ratio for the frozen soil, but after saturation the compressive strength does not increase any more (Cho and Sohn, 1993). The compressive strength measured for the laboratory-prepared frozen soil specimens was within the range, 4~11MPa.

Fig. 3(b) shows stress-strain curves when the water content ratio are 7%, 15%, 20% respectively. When the kaolinite content decreases, the compressive strength of soil increases. However, when the water content ratio is 7%, the compressive strength reaches maximum at 20% of kaolinite content, which means the soil reaches super-saturation at 7% water content and 20% kaolinite content. As shown in the figures, the compressive strengths of frozen soil depend on various factors such as water content and kaolinite content, however, the observed failure modes are all plastic as if ductile deformation in fresh water ice.

## CONCLUSIONS

In this paper, unconfined uniaxial compression tests were carried out for measuring the strength of laboratory-grown fresh water ice and frozen soils. Preparation and dimension of specimen were the most important issues during the cold room tests. It is the intent of this paper to set up a standard method for creating repetitive environmental conditions through cold room experiments.

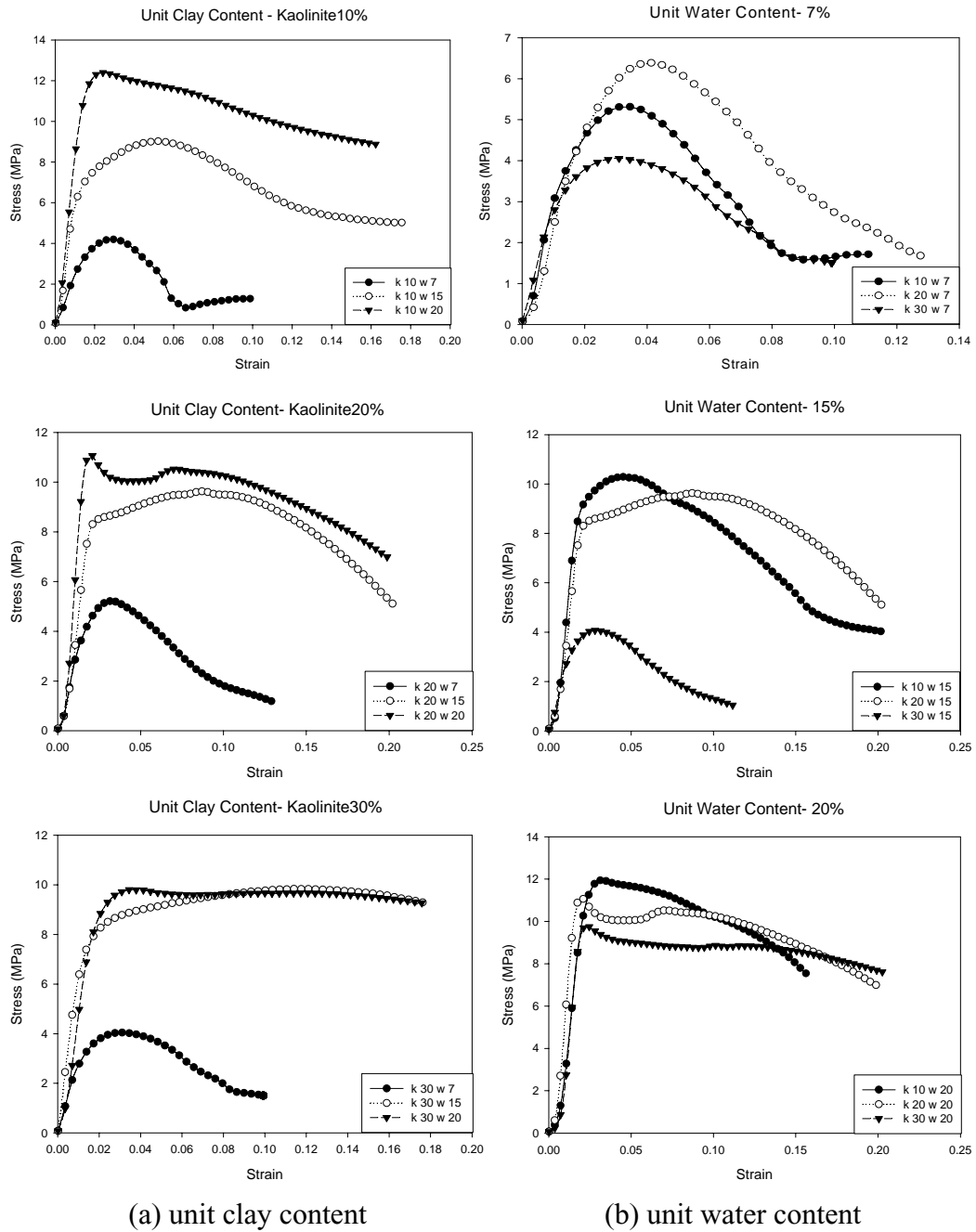


Fig. 3 Stress-strain curves for frozen soil specimens

(1) Stress-strain curves for fresh water ice specimen shows the brittle behavior roughly at strain-rate above  $10^{-4}$  /s, and the ductile behavior roughly at strain-rate below  $10^{-5}$  /s. Even though the exact transition point from one characteristics to the other is not known, the strain-rate seem to be a key factor for brittle-to-ductile transition phenomena as many researchers pointed out.

(2) The compressive strengths of frozen soil depend on various factors such as water content

and kaolinite content. The observed failure modes are all plastic as if the ductile deformation in fresh water ice. As the water content ratio increases, the compressive strength of the soil generally increases. But the compressive strength does not increase any more after the saturation.

## ACKNOWLEDGEMENTS

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