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Measurement and Evaluation of Natural Frequencies of Bulk Ice Plate Using Scanning Laser Doppler Vibrometer

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Abstract

The structure of water in its solid state (ice) is complex. A number of process parameters and the chemistry of the freezing aqueous solution influence ice crystal growth and ice characteristics such as crystalline structure, impurity inclusions, and appearance. The resulting variation in ice properties makes determining mechanical properties challenging. This study examines the performance of a Scanning Laser Doppler Vibrometer (SLDV) used to measure the natural frequency of an ice sample and consequently evaluate its modulus of elasticity. Analytical and numerical methods were used, and the results have been compared to values found in the literature. The modulus of elasticity for the measured ice plate, with a porosity of 8.8 \%, was found to be 6.7 GPa, which is 2.7 \% lower than previously published values.

Keywords: Bulk ice, modulus of elasticity, natural frequency, non-destructive

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1. Introduction

The structure of water in its solid state (ice) is complex. Variations in freezing process parameters and the chemistry of the freezing aqueous solution influence ice crystal growth and contribute to the characteristics of the resulting ice. For example, crystal structure, impurity inclusions, and appearance can all be affected. From the mechanical perspective, ice can be considered fragile and viscoelastic. Determining its mechanical behaviors is complicated by the onset of melting in areas where temperatures rise above freezing. For low stress-strain rates, viscous behavior dominates. However, elastic behaviors become dominant over other mechanical behaviors for higher stress-strain rates [1].

Modulus of elasticity (also known as Young’s modulus) is a measure of a solid material’s stiffness. It describes the relationship between the material’s stresses and strains. A number of methods can be used to define the modulus of elasticity. It can be determined by stretching or compressing a sample material using a known load while measuring displacement (strain) [2]. It can also be determined by measuring the speed of sound through the material and relating it to material density [3]. Finally, modulus of elasticity can be determined based on natural frequency, which varies with material stiffness, and which can be expressed through the material’s modulus of elasticity, geometric dimensions, mass, and inertia.

In this study, a Scanning Laser Doppler Vibrometer (SLDV) was used to examine the natural frequency of a plate ice sample. Knowing the natural frequency makes it possible to determine the plate’s modulus of elasticity. The modulus of elasticity can be employed e.g. in structural analyses. Once
known, moreover, excitation at the natural frequency can be applied to the plate to break it up into smaller pieces.

When its temperature is relatively close to the freezing point of water (minimal undercooling), water in an aqueous solution freezes and forms pure ice. Impurities migrate to and accumulate in the remaining unfrozen aqueous solution [4] This makes it possible to purify wastewater by freezing it in controlled conditions. An SLDV, which can detect the increasing mechanical rigidity of forming ice, can be used to determine when to break up the ice layer. It can also be used to measure ice layer thickness or to determine mechanical properties. Depending on the conditions of the freezing process and the initial chemistry of the aqueous solution, freeze purification efficiency varies [5]. Some suspended impurities (e.g., salt) can be trapped within the bulk ice as it forms, which structurally weakens it. [6] Weaker ice has a lower natural frequency. Therefore, natural frequency measurement is one way to estimate the purity of ice. [7] As part of a wastewater purification process, impurities could be estimated by measuring ice mechanical rigidity using the SLDV.

2. Materials and methods

This study focuses on examining a plate ice sample formed from ultrapure water in a controlled environment. To ensure measurement accuracy and repeatability, natural frequency was measured four times at room temperature as the sample melted. Each sample was measured at fifteen different locations. As the ice melted, the size of the sample decreased and the effect on natural frequency was recorded.
To measure natural frequency using the SLDV, the subject item must be excited. Often, excitation is accomplished using a hammer or a piezo exciter. The suitability of using these methods to excite plate ice for vibration measurement was tested in this study. The ice sample was clear and transparent, so the signal reflection back to the scanning head was not sufficient for accurate measurement. Typically, retroreflective tapes or reflective sprays are utilized to overcome this problem. In this study, the piezo exciter worked best to provide excitation, and the best signal was obtained using the retroreflective tape. This combination yielded the largest velocity responses.

2.1. The ice sample

To make the natural frequency measurements, an ice sample was formed in a crystallizer from ultrapure water under controlled freezing conditions. In air-cooled natural freeze layer crystallization, ice crystals form a layer on the water surface, which grows downwards into the liquid. Natural ice crystallization due to forced heat convection was simulated with a wind tunnel-like apparatus introduced in more detail in previous studies by Hasan et al. [8].

The water was purified to achieve $18.2 \text{ M}\Omega\text{cm}$ resistivity and $<5$ ppb total organic carbon (TOC), respectively, using an Elga PureLab water system. This ensured that freezing would occur at $0 \degree C$. To prevent undercooling and the need for ice seeding, each 500 ml pure water sample was precooled to nearly $0 \degree C$ in a freezer set to -18 $\degree C$. The pure water was poured into a plastic boxlike vessel with rounded corners and 750 ml total volume. This vessel was then embedded into the floor of the wind tunnel so the bottom and all sides were heat insulated. Only the top of the vessel was open to produce a freezing area on the water surface of approximately $13.4 \cdot 10^{-3} \text{ m}^2$. 
The external freezing conditions, the temperature and velocity of the air flowing inside the tunnel, were set to -2 °C and 2 ms\(^{-1}\) to produce a suitable ice layer thickness in preparation for further frequency measurements.

A 22 mm plate ice layer formed in 22 hours. The freezing ratio of the water in the vessel was 51 \%. The total purified water mass and the mass of the plate ice produced during the freezing process were monitored using a Precisa BJ2200C precision balance (resolution 0.01 g) to determine the ice growth rates \((2.78 \cdot 10^{-7} \text{ ms}^{-1} \text{ and } 850 \text{ gh}^{-1}\text{m}^{-2})\) and the mass loss rate caused by evaporation and sublimation \((75 \text{ gh}^{-1}\text{m}^{-2})\). Water temperature was measured to monitor the thermodynamics of crystallization during the freezing process. The cooling curve shown in Figure 1 depicts the temperature profile of the water.

![Figure 1: Temperature profile](image)

The sample was then stored in a freezer at -18 °C. Because the ice sample formed at -2 °C, its surface began to melt during the handling and when taking measurements at room temperature. This resulted in a small amount of melt water being refrozen on the left bottom corner of the ice sample during freeze storing.
2.2. Mechanical properties of ice plate

For an undamped single degree of freedom system, e.g. a spring-mass system, the natural frequency \( f \) relation to mass \( M \), kg and stiffness \( K \), N/m can be expressed [9] as

\[
f = \frac{1}{2 \pi} \sqrt{\frac{K}{M}}. \tag{1}
\]

An analytical approximation for the flexural frequency in the longitudinal direction can be estimated using beam theory per the following equation.

\[
f = ((2n + 1)\pi/2)^2 \sqrt{\frac{EI}{A\rho L^4}}, \tag{2}
\]

where \( n \) is the critical frequency number (1 for first flexural frequency), \( E \) is the modulus of elasticity (Pa), \( I \) is the area moment of inertia (m\(^4\)), \( A \) is the cross-sectional area (m\(^2\)), \( \rho \) is the density (kg/m\(^3\)), and \( L \) is the plate length (m). Because the SLDV can measure flexural frequency, it can be used to determine modulus of elasticity if the density and physical dimensions of the subject object are known as expressed by Eq. 2 [10].

The mechanical properties of ice have been extensively studied. Its modulus of elasticity varies depending on freezing process conditions and the aqueous solution from which the ice is formed. In the literature, early studies report modulus of elasticity values for ice from 0.3 to 11.2 GPa [11]. Later research recommends utilization values for the modulus of elasticity of 8.83 GPa and a Poisson’s ratio of 0.36 ± 0.13 [12]. For freshwater lake ice, the literature reported variations in modulus of elasticity values from 9.7 GPa to 11.2 GPa and a Poisson’s ratio of 0.33 [13, 14]. For freshwater lake ice, the reported modulus is 8.7 GPa at 0 °C and 10.0 GPa at -20 °C, and the theoretical density of bubble-free ice is 917 kg/m\(^3\) with a Poisson’s ratio of
0.33 [2]. The structure of ice, *e.g.* porosity and impurities captured in the ice, affects its modulus of elasticity; therefore, values found from the literature should not be considered absolute. For example, nonporous ice has a 10 GPa modulus of elasticity. With 10 % porosity, the modulus is 6.5 GPa [15].

2.3. *Scanning laser Doppler vibrometer (SLDV)*

The Polytec Scanning Vibrometer (PSV) is a commercial SLDV. An optical system, it determines surface velocities by measuring movement with nanometer accuracy. The hardware can operate within a $+5 \degree \text{C}$ to $+40 \degree \text{C}$ temperature range [16]. Figure 2 depicts the measurement setup. The Polytec PSV-500 is able to measure one-directional surface velocities and thus can capture out-of-plane vibrations. In the figure, A is the scanning head (PSV-500), B is the reference laser (OFV-505), C is the measured ice sample, and D is the exciter (Alpha Solution AS-1220).

![Figure 2: Illustrative measuring setup](image)
In the scanning head, a laser signal of 670 nm (+/-5 nm (red)) wavelength is directed through a Mach-Zehnder interferometer. Part of the signal is directed to the measured section with scanning mirrors, and part is directed to the photo detector. The reflected signal from the measured section is compared to the input signal, and from the Doppler frequency shift, the analogue signal proportional to the velocity is produced. The scanning mirrors make it possible to measure several scan points at predefined locations without repositioning the head [17]. Figure 3 illustrates the optical measuring procedure.

![Figure 3: Optical measuring procedure](image)

2.4. Signal reflection

SLDV operation is based on the Doppler Effect; therefore, part of the signal directed at the object being measured must be reflected back to the scanning head. A laser signal is projected at a single point, and the reflected signal is recorded. Based the difference between the projected and reflected signals, surface velocity can be determined. The plate ice sample used in this
study was relatively non-reflective, so a method was needed to improve the percentage of projected signal that was reflected back to the scanning head.

To enhance the reflectivity of the subject ice sample, both ARDROX reflective spray and ACOUTRONIC retroreflective tape (A-RET-T010) were tested. The reflective spray did not improve reflectivity. The retroreflective tape gave good results. For this study, the tape was placed on the top surface of the ice to reflect the projected signal. The additional stiffness added by the tape was negligible compared to the stiffness of the ice plate; therefore, its effect on natural frequency can also be considered negligible. With this setup, it was possible to record the high frequency vibration of the ice and to determine its natural frequencies.

2.5. Excitation of the ice sample

Two traditional excitation methods were tested. The ice sample was excited with an automatic hammer (Alpha Solution AS1220) and a piezo exciter (PI Ceramic P-844.10). The automatic hammer could not excite the sample ice to sufficiently high frequencies, so the piezo exciter was used for the study. To approximate its natural frequency regime, an estimate of the ice sample’s first flexural natural frequency was calculated using Eq. 2 with a modulus of elasticity value taken from the literature (8.7 GPa) and a theoretical ice density of 917 kg/m$^3$. The calculated result was 3554 Hz. This predicted natural frequency is high, because the plate ice sample was small (140 x 105 x 22 mm) and formed from ultrapure water. The piezo exciter had no problem providing excitation in this frequency range. To take the measurements in this study, the exciter was set to provide burst chirps from 800 to 10 000 Hz. A burst chirp generates a constant amplitude sine
wave signal in the time domain with variable frequency. The piezo exciter was positioned behind and at the middle of the plate ice sample.

2.6. Measurement setup

To make the natural frequency measurements, the plate ice sample was removed from a storage freezer. The measurement procedure was conducted at room temperature. Because the sample began and continued to melt during the measurement procedure, the measurements were also able to capture any changes in vibration response (and therefore natural frequency) as melting progressed. Figure 4 shows the 140 x 105 x 22 mm ice sample with retroreflective tape hanging from two metal wires passing through two six-millimeter holes. The metal wires will have some effect on the ice plate rigid body modes. However, as the interest is on the flexural frequencies, and those are estimated with Eq. 2 to be near 3554 Hz, the effect of rigid body modes is negligible.

Figure 4: Ice sample hang from two six millimeter holes
As measurements were being taken, melting continuously reduced the physical dimensions of the plate ice sample. At the end of the 28-minute measurement procedure, the ice plate measured 137 x 102 x 19 mm, 3 mm smaller in each direction. The first measurement was performed three minutes after the sample was taken from the storage freezer. There was a 194 s delay between the first and the second measurement and a 177 s delay between the second and third. Between the third and fourth measurement, there was a 954 s delay. Figure 5 shows the ice sample and the 15 measurement locations.

Figure 5: Measuring points and the piezo excitation location

Four separate measurements were taken and the effect of ice melting on the natural frequencies was studied. The initial mass (without the retroreflective tape) of the ice sample was 247 grams. At the conclusion of the measurement procedure, the ice weighed 224 grams. Three measurements were taken at each measurement location. The average response spectrum from 15 measuring points was used to determine the natural frequencies.
Sample frequency of 25 000 Hz were used in the measurements. Each measurement used 12800 FFT lines giving a resolution of 0.78125 Hz (0 Hz to 10 000 Hz). Polytec scanning vibrometer software version 9.2.2 was used to interpret the results. To evaluate the measurement uncertainty, reproducibility and representativeness of results, the measurement was repeated four times for a melting ice sample with 15 individual measurements.

3. Results

This section presents the results of the measurements taken to determine the natural frequency of the plate ice sample. In addition, using the isotropic material properties of ice, a mathematical model based on physical dimensions, mass, and natural frequency is introduced. The measurement results are compared with values available from the literature, and the analytical results, with the tuned modulus of elasticity, are compared to both the measured and numerical results.

3.1. Measurement

The SLDV measures surface velocity in the time domain, and because it is converted to the frequency domain, the natural frequencies show up as higher peaks. Figure 6 depicts the frequency response of the Pure Water Ice (PWI) sample based on the second set of measurements. Note the higher response peaks corresponding to the natural frequencies.

Four measurements were conducted. They were labeled #1 through #4. The first three were carried out at approximately three-minute intervals, and the fourth was carried out approximately 15 minutes after the third. At the time of the measurement 1, the ice sample had been subject to room
temperature for 355 seconds. For #2, #3, and #4, 549 seconds, 726 seconds, and 1680 seconds, respectively, had elapsed. Natural frequencies up to fifth flexural frequency were considered. Figure 7 depicts the measured natural frequencies for the four measurements.

![Figure 6: Velocity response in frequency domain](image)

Figure 6: Velocity response in frequency domain

![Figure 7: Measured flexural frequencies in the four measurements (PWI #1 to #4).](image)

Figure 7: Measured flexural frequencies in the four measurements (PWI #1 to #4).

3.2. Mathematical model

A three-dimensional geometry was constructed based on the physical dimensions of the plate ice sample. An FEM modal analysis was carried out
for the 3D model of the sample. The mesh consisted of 645 solid elements (SOLID186 elements in ANSYS Workbench™).

To model the ice with an isotropic material approach, three properties needed to be defined for the known geometry. These properties are the modulus of elasticity, \( E \), the equivalent stiffness of the material, the density of the material, and Poisson’s ratio. The first two account for a major part of the dynamics. Poisson’s ratio variation had little effect. The value for Poisson’s ratio was taken from the literature. Because density is known, the modulus of elasticity could be calculated from the model. In the numerical modal analysis, the first six natural frequencies were rigid body modes with a frequency of 0 Hz and rigid mode shapes. The higher modes were flexural; representing the ice sample’s internal flexing modes, which were comparable to results obtained with the SLDV measurements. Since the porosity of the plate ice sample was 8.8 %, its density was 837 kg/m\(^3\), which is lower than the 917.5 kg/m\(^3\) theoretical density published in the literature for pure water ice. With this density, the calculated mass becomes 247 g. Given its geometry and this mass, the modulus of elasticity in the numerical model was tuned so the first flexural frequency fell within 10 Hz of that measured. Table 1 depicts the results for the measured, numerical, and analytical natural frequencies for the first five flexural frequencies in the first (PWI #1) and last measurement (PWI #4).
Table 1: First five measured natural frequencies (PWI #1 and PWI #4) compared to numerical and analytical results

<table>
<thead>
<tr>
<th>Natural frequency</th>
<th>Measured (SLDV) (Hz)</th>
<th>Numerical (FEM) (Hz) (^1)</th>
<th>Difference to measured</th>
<th>Analytical (Eq. 2) (Hz) (^1)</th>
<th>Difference to measured</th>
</tr>
</thead>
<tbody>
<tr>
<td>PWI #1 #1(^2)</td>
<td>2923.4</td>
<td>2927.9</td>
<td>0.15 %</td>
<td>3264.4</td>
<td>11.7 %</td>
</tr>
<tr>
<td>PWI #1 #2(^2)</td>
<td>3364.1</td>
<td>3182.8</td>
<td>-5.39 %</td>
<td>3264.4</td>
<td>-3.0 %</td>
</tr>
<tr>
<td>PWI #1 #3(^2)</td>
<td>5955.5</td>
<td>5985.8</td>
<td>0.51 %</td>
<td>8998.9</td>
<td>51.1 %</td>
</tr>
<tr>
<td>PWI #1 #4(^2)</td>
<td>6545.3</td>
<td>6387.4</td>
<td>-2.41 %</td>
<td>8998.9</td>
<td>37.5 %</td>
</tr>
<tr>
<td>PWI #1 #5(^2)</td>
<td>7092.2</td>
<td>6751.9</td>
<td>-4.80 %</td>
<td>17642.7</td>
<td>148.8 %</td>
</tr>
<tr>
<td>PWI #4 #1(^3)</td>
<td>2803.1</td>
<td>2805.9</td>
<td>0.10 %</td>
<td>2944.1</td>
<td>5.0 %</td>
</tr>
<tr>
<td>PWI #4 #2(^3)</td>
<td>3213.3</td>
<td>3051.3</td>
<td>-5.04 %</td>
<td>2944.1</td>
<td>-8.4 %</td>
</tr>
<tr>
<td>PWI #4 #3(^3)</td>
<td>5658.6</td>
<td>5846.0</td>
<td>3.31 %</td>
<td>8115.0</td>
<td>43.4 %</td>
</tr>
<tr>
<td>PWI #4 #4(^3)</td>
<td>6321.9</td>
<td>6247.6</td>
<td>-1.18 %</td>
<td>8115.0</td>
<td>28.4 %</td>
</tr>
<tr>
<td>PWI #4 #5(^3)</td>
<td>6692.2</td>
<td>6604.6</td>
<td>-1.31 %</td>
<td>15911.6</td>
<td>137.8 %</td>
</tr>
</tbody>
</table>

\(^1\) Young’s modulus 6.7 GPa and Poisson’s ratio 0.33.

\(^2\) First measurement, thickness 22 mm, width 105 mm and height 140 mm.

\(^3\) Last measurement, thickness 19 mm, width 102 mm and height 137 mm.

Figure 8 shows the first flexural mode shapes for PWI #1.

The left bottom corner shows the largest amplitude. Because the largest movement is in the corners, in the future, the retroreflective tapes should be placed closer to the sample edges to improve the mode shape determined from the measurements. The similarity between the measured and calculated
mode shapes validate the accuracy of the numerically predicted dynamic behaviors.

4. Discussion

Modeling the ice sample as an isotropic material yields a lower modulus of elasticity than can be found from the literature. However, considering its porosity and lower density, the modulus of elasticity should be about 6.9 GPa [15]. Given the 6.7 GPa modulus of elasticity used in the numerical model, the first flexural frequency is 0.15 % higher than measured (PWI #1) and 0.1 % higher with the last measurement (PWI #4). The numerical results for the first five natural frequencies are within +3.5 % and -5.4 %. This suggests that even higher flexural frequencies can be estimated with reasonable accuracy. Comparing the modulus of elasticity to the values found from literature considering that the ice sample included porosity, the measurement procedure
was conducted at 22 °C, and the geometry was approximate, the difference seems reasonable. The analytical results estimated an 11.7 % higher natural frequency for the first measurement (PWI #1) and a 5.0 % higher natural frequency for the last measurement (PWI #4) when compared to the measured results. To perform the analytical calculation, the geometry was considered rectangular. This simplification resulted in inaccurate estimates for the higher flexural modes. Nonetheless, the analytical equation gave a useful rough estimate of where to expect the first flexural frequency. The SLDV proved capable of capturing the natural frequencies of the ice sample. As the plate ice sample melted, its natural frequencies dropped. The rate of natural frequency drop-off was visibly consistent with the measurements results. The measured natural frequency drop-off as a function of ice volume seemed to be linear. Measurements were conducted at room temperature. The retroreflective tape used to enhance reflected signals remained in place even as the plate ice sample melted. It was held via the surface tension of the liquid water on the ice’s surface. To conduct similar measurement at subzero temperatures, a different means of attached the retroreflective tape will be needed. For example, the tape could be frozen to the ice using water as was done in John et al. [7].

5. Conclusions

This research employed the SLDV optical method to evaluate the natural frequencies and elastic modulus of plate ice. This information can be used to prepare a structural model and use it to analyze ice dynamic behaviors. The study showed that the SLDV consistently and accurately estimated the
natural frequencies of the sample ice. It also revealed that to get a good signal to the scanning head, enhancing the reflectivity of the ice surface is necessary. Retroreflective tape was used. In addition, because the plate ice sample measured was small, its lowest flexural frequencies were over 2800 Hz. A piezo exciter was needed to generate these higher frequency excitations. Burst chirps from 800 to 10,000 Hz were used. In the future, it would be interesting to study the effect of the ice’s crystalline structure on its natural frequencies.

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