Effect of Low Temperature Cyclic Treatments on Modulus of Elasticity of Birch Wood

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The modulus of elasticity (MOE) of birch (*Betula platyphylla*) wood specimens with four different moisture content (MC) levels, *i.e.,* water-saturated, green, air-dried, and oven-dried, were examined under a low temperature condition ranging from -196 °C (liquid nitrogen temperature) to +20 °C (room temperature). Dynamic mechanical analysis (DMA) was used to evaluate the dynamic viscoelastic properties before and after the low temperature treatment, while X-ray diffraction (XRD) was used to analyze the crystal structure. The results showed that MOE with different MC increased after the low temperature treatment. Specimens with higher MCs were more affected by the treatment than specimens with lower MCs. However, the effect of low temperature treatment (within four times) on MOE was not significant (P = 0.053 - 0.225). Cyclic treatments of liquid nitrogen did not decrease wood MOE. As a structural material, wood has a better residence to low temperatures compared to concrete, in which mechanical properties decreased dramatically after one cycle of low to room temperature.

Keywords: Low temperature treatment; Cyclic treatments; Modulus of elasticity; Viscoelastic properties; Crystallinity

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INTRODUCTION

Temperature plays a crucial role in affecting the mechanical properties of wood. With respect to the relationship between temperature and wood mechanical properties, most previous studies merely focused on the effect of high temperature (>140 °C) treatments (Kubojima *et al*. 2000; Moraes *et al*. 2004; Awoyemi and Jones 2011). However, there are many wood applications below 0 °C, such as for the body of a liquefied natural gas (LNG) ship to contain LNG at -163 °C (Zhang *et al.* 2009), and for the roof of wooden-structural housing to withstand the snow pressure and low temperature impact (Ayrilmis *et al.* 2010). Research studies of wood properties under low temperatures have been rarely reported.

The effect of low temperatures on the flexural properties of wood was first investigated in the 1930s, when pine (*Pinus cembra*) wood strength was examined from -70 °C to +70 °C, which indicated that the strength of wood increased with the decreasing temperature (волна 1957). Similar results were found by Kollmann and Cote (1968). A negative linear relationship was found between MOE and temperature from -18 °C to 66 °C for lumber with 12% moisture content (MC) (Green *et al.* 1999). Jiang *et al.* (2014) furthermore discovered that when temperature was decreased from 23 °C to -196 °C, the compression strength and compression modulus of elasticity of oak (*Quercus mongolica*) wood were increased by 283.91% and 146.30%; both changing trends of compression

strength and compression modulus of elasticity as a function of temperature were regressed by two models, a linear model and a polynomial model, respectively. The finding of DeGeer and Bach (1995) showed that MOE and modulus of rupture (MOR) of spruce-pine-fir (*Picea glauca - Pinus cembra - Abies lasiocarpa*) increased 0.13% and 0.34%, respectively, as the temperature was decreased by 1 °C. Kendra and Cortez (2010) found that the MOR of a wooden baseball bat was increased by 26% after it was treated at -190 ºC for 24 h.

There are three aspects in terms of low-temperature applications for wood products, including being in a low temperature environment (Ayrilmis *et al.* 2010), undergoing a process increasing from low temperature to room temperature, and cyclic repeating from low to room temperatures (Li 2005; Kendra and Cortez 2010). When timber is used as the body of liquefied natural gas (LNG) ship to contain LNG, it experiences the circulations between low and room temperatures related to the amount of LNG. Under this circumstance, timbers require adequate strength to endure the pressure from LNG (Zhang *et al.* 2009).

The intracellular moisture state would affect mechanical properties of wood together with the temperature. Previous studies showed that when the green wood was cooled below the freezing point, ice formed in the cell lumens, which made the cell walls shrink as the expanding of ice lens in the lumen; consequently, the physical damage probably occurred in the wood cell wall, which would affect the mechanical property of wood (Choong *et al.* 1973; Jiang and Lu 2008). The objective of this study was to determine the effect of low temperature treatment and cyclic treatments on wood MOE with different MC. Four MCs were selected in this study to characterize the inner moisture state of wood. Results were compared to concrete in order to show the excellent residence to low temperatures when wood was used as a structural material. Dynamic mechanical analysis (DMA) was used to evaluate the dynamic viscoelastic properties, and X-ray diffraction (XRD) was used to analyze the crystal structure in order to study the effect of low temperature treatment. The innovations of this study are as follows: (1) wood materials were cyclically treated at an ultra-low temperature (-196 °C), which can provide technical support of wood application at low temperatures; and (2) DMA and XRD were used to characterize the property change.

EXPERIMENTAL

Materials

Green birch (*Betula platyphylla*) wood specimens with a MC of 67.0% and a basic density of 0.57g/cm^3 were cut from a 1-m-long quarter-sawn board from a 4-m-long log, which grew in a plantation stand located in Dunhua, Jilin province, China. Specimens without knots and defects were cut to a size of 300 mm (length) × 20 mm (radial) × 20 mm (tangential) for the determination of MOE, and cut to 60 mm (L) ×12 mm (R) × 2.5 mm (T) for determination of DMA. Specimens for XRD analysis were milled to a 40 to 60 mesh powder.

Methods

Conditioning moisture content

Four MCs of birch wood specimens including water-saturated, green (*i.e.*, specimens directly cut from fresh felled timber), air-dried, and oven-dried MCs were

obtained. The water-saturated wood specimens were prepared by immersing green wood specimens in water for 2 months, while the air-dried wood specimens were prepared by placing the green wood specimens in an air-drying shed for 2 months followed by equilibration in a temperature-humidity chamber at 20 °C and 65% relative humidity (RH) for 2 months. The oven-dried wood specimens were prepared by placing the green wood specimens in an air-drying shed for 2 months, and then in an oven at 103 °C for at least 48 h.

Cyclic Treatments

Specimens (water-saturated, green, air-dried, and oven-dried) sealed with plastic bags were soaked in liquid nitrogen (-196 °C) for 72 h, and then placed at room temperature for 24 h, when the average core temperature of the specimen was 23.2 °C measured by an infrared thermometer by cutting additional treated specimens from the center (three duplications). Above steps were designated as one treatment cycle. Specimens were treated by four treatment cycles successively. Untreated specimens were defined as controls.

Measurement of MOE

Eighty specimens were selected and randomly sorted into four groups consisting of 20 specimens at each MC level for static bending test. Before cyclic treatment, MOE was determined for the control (MOE_0) according to the standard GB/T 1936.2 (2009). The MOEs were examined after each cycle on the same specimen (*i.e.*, 1-4), as MOE_1, MOE_2, MOE_3, and MOE_4, respectively. The measuring instrument was a Universal Mechanical Testing Machine (Instron 5582, USA) with the maximum load of 100 KN. Specimens were loaded in the tangential direction of birch wood.

In order to monitor the change of MC during the entire experiment, specimens were weighed before the determination of MOE of the untreated controls and from one to four cycles marked as M_0, M_1, M_2, M_3, and M_4. At the end of the entire experiment, specimens were placed in an oven at 103 °C for at least 48 h, until no weight change was found, when the weight was marked as M. The MC of MC_1, MC_2, MC_3, and MC_4 corresponding to one to four treatments, and the change of MC, were then calculated.

The MOE, change of MOE (ΔMOE), MC, and change of MC (ΔMC) of the specimens were calculated in Eqs. 1 to 4,

$$MOE = \frac{23FL^3}{108bh^3s} \quad (1)$$

$$\Delta MOE = \frac{MOE_n - MOE_0}{MOE_0} \times 100\% \quad (2)$$

$$MC = \frac{M_n - M}{M} \times 100\% \quad (3)$$

$$\Delta MC = \frac{MC_n - MC_0}{MC_0} \times 100\% \quad (4)$$

where F is the increment of load on the straight line portion of the load-deformation curve (N), L is the length of span (mm), b is the width of the specimen (mm), h is the

thickness of the specimen (mm), *s* is the increment of deformation corresponding to *F* (mm), *n* is the number of cycles (*i.e.*, 1-4), and *ΔMOE* and *ΔMC* are the change proportion of MOEs and MCs of specimens after one to four treatments compared to the untreated one.

Measurements of viscoelastic properties

The oven-dried wood specimens were soaked in liquid nitrogen (-196 °C) for 72 h, then placed into a glass desiccator with phosphorous pentoxide (P_2O_5) as a drying medium until reaching a constant weight. Above treatment was repeated four times. After the fourth treatment, the viscoelastic properties were measured using dynamic mechanical analysis (DMA 2980, TA Instruments, USA). A dual-cantilever bending mode with a span of 35 mm was used in the temperature range from -120 °C to 40 °C. The heating scans of 1 Hz, heating rate of 2 °C/min, and displacement amplitude of 15 mm were carried out. Three replicates were used for all the experiments.

The storage modulus (E') is defined as the elastic response proportional to the stored energy, while the loss modulus (E'') is the viscous response proportional to the dissipated energy (Jiang and Lu 2008). Since wood is anisotropy, the relative storage modulus (E'/E_0') and the relative loss modulus (E''/E_0'') were used instead of the real values of E' and E'' to reduce the variation, where E_0' and E_0'' were values at -120 °C.

Crystallinity analysis

Wood powder, weighing 1.00 g, was soaked in liquid nitrogen (-196 °C) for 72 h, then placed into the temperature-humidity chamber at 20 °C and 65% RH to equilibrate until the constant weight was reached. Above treatment was repeated four times. Then the wood powder before and after the treatment was pressed into a sheet. The crystallinity analysis was performed by XRD with a 2θ intensity curve, a scanning range of 5° to 40°, and a scanning speed of 2°/min. The degree of crystallinity (C_r) of wood cellulose was calculated using Eq. 5 (Park *et al.* 2010),

$$C_r = \frac{I_{002} - I_{am}}{I_{002}} \times 100\% \quad (5)$$

where I_{002}- I_{am} is the intensity of the crystalline peak, I_{002} is the total intensity of amorphous and crystalline regions.

Data analysis

The statistical software, SPSS version 17.0 (SPSS Inc, Chicago, IL, USA), was used for data analysis. By assuming that MOE of the specimens was a normal distribution, Duncan's multiple comparison tests (P = 0.05) were carried out to analyze the significant effect of the low temperature treatment.

RESULTS AND DISCUSSION

MOE of One to Four Cyclic Treatments

The results of MOE after each cycle of treatment are presented in Table 1. The MOE of all birch wood specimens increased compared to the untreated one after the low temperature treatment for all specimens with different MC. The largest increments after four cycles of treatments were 0.80, 0.79, 0.64, and 0.25 GPa for the water-saturated,

green, air-dried, and oven-dried specimens, respectively, which meant 6.81%, 6.21%, 4.77%, and 2.98% increase, respectively. Based on Duncan's multiple comparison tests, at each MC level, there were no significant differences in MOE after each cycle of low temperature treatment, while the effect of treatment times (within four times) on MOE was not significant (P = 0.053 – 0.225).

It is confirmed that at room temperature, when MC is over the fiber saturation point (FSP), the MC variation has little influence on wood MOE; however, when it is below the FSP, an increase in MC caused a decrease in wood MOE, the relationship between which was that MOE increased 1.5% when the MC decreased by 1% (Ishimaru *et al*. 2001; GB/T 1936.2 2009). Thus, when MC change was considered, the increases in MOE of air-dried one after each treatment were 3.52%, 5.62%, 1.98%, and 3.00%, respectively, while that of the oven-dried one were 4.70%, 3.63%, 5.68%, and 5.53%, respectively. The largest increase for water-saturated, green, air-dried, and oven-dried specimens were 6.81%, 6.21%, 5.62%, and 5.68%, respectively. Specimens with higher MCs, *i.e.*, water-saturated and green specimens, were more affected than the air-dried and oven-dried one.

Table 1. MOE after Four Cyclic Treatments from -196 °C to +20 °C

Moisture Status	MOE	Untreated	Treatment (1 cycle)	Treatment (2 cycles)	Treatment (3 cycles)	Treatment (4 cycles)
Water-saturated specimen (136.0 ± 3.38%)	Mean (GPa)	11.75 (1.05)	11.87 (1.02)	12.55 (1.42)	11.81 (1.17)	11.62 (1.57)
	ΔMOE (%)	--	1.02	6.81	0.51	-1.11
	ΔMC (%)	--	-0.23	-1.37	-2.17	-2.93
Green specimen (67.0 ± 2.04%)	Mean(GPa)	12.73 (1.46)	12.85 (1.22)	13.52 (1.48)	12.89 (1.57)	12.87 (1.58)
	ΔMOE (%)	--	0.94	6.21	1.26	1.10
	ΔMC (%)	--	-0.65	-1.50	-2.21	-2.64
Air-dried specimen (12.3 ± 0.56%)	Mean(GPa)	13.42 (1.03)	13.78 (1.18)	14.06 (1.65)	13.51 (1.09)	13.64 (1.66)
	ΔMOE (%)	--	2.68	4.77	0.67	1.64
	ΔMC (%)	--	0.56	0.57	0.87	0.91
Oven-dried specimen (0.6 ± 0.05%)	Mean(GPa)	14.77 (1.62)	15.21 (1.12)	14.85 (1.59)	15.02 (1.33)	14.92 (1.36)
	ΔMOE (%)	--	2.98	0.54	1.69	1.02
	ΔMC (%)	--	1.15	2.06	2.66	3.01

Values in parentheses are standard deviations
-- denotes that the value does not exist
When the MC variation was considered, the increases in MOE of air-dried wood specimens after each treatment were 3.52%, 5.62%, 1.98%, and 3.00%, while that of the oven-dried one were 4.70%, 3.63%, 5.68%, and 5.53%, respectively.

The increase in MOE is likely caused by two reasons. The first is the reducing distance of molecules as decreasing temperature enhanced the intermolecular forces of specimens, which caused the increase in wood MOE after the low temperature treatment compared to the untreated one (Jiang *et al*. 2006; Ayrilmis 2007; Lower 2009). Second, it was discovered that materials such as metals, alloys, plastics, and composites showed a

stronger and more durable property after low temperature treatment. The reason was that the treatment refined and stabilized the crystal structure and reduced residual stress (Kalia 2010). According to which we concluded that the increase in MOE might be due to the partial release of wood internal residual stress.

Viscoelastic Properties and Crystallinity

Figure 1 shows the temperature dependencies of E'/E_0' (Fig. 1a) and E''/E_0'' (Fig. 1b) for treated and untreated wood specimens. The E'/E_0' ratio decreased with increasing temperature due to the softening of the materials (Jiang and Lu 2008). From the E''/E_0'' curve, two mechanical relaxation processes (α and β) were observed. The α process occurred at approximately 30 °C, and the β process was at nearly -50 °C. According to previous studies, α relaxation was attributed to a similar glass transition of hemicellulose with a low molecular weight (Mano 2002; Jiang and Lu 2008), and the β relaxation process was attributed to both the motions of methyl groups in amorphous of wood cell wall and adsorbed water molecules (Obataya *et al.* 1996; Sugiyama and Norimoto 1996). It was found that dynamic viscoelastic properties of treated wood specimens were similar to the untreated one, and there were no obvious changes in the content of hemicellulose and the crystal structure before and after the low temperature treatment.

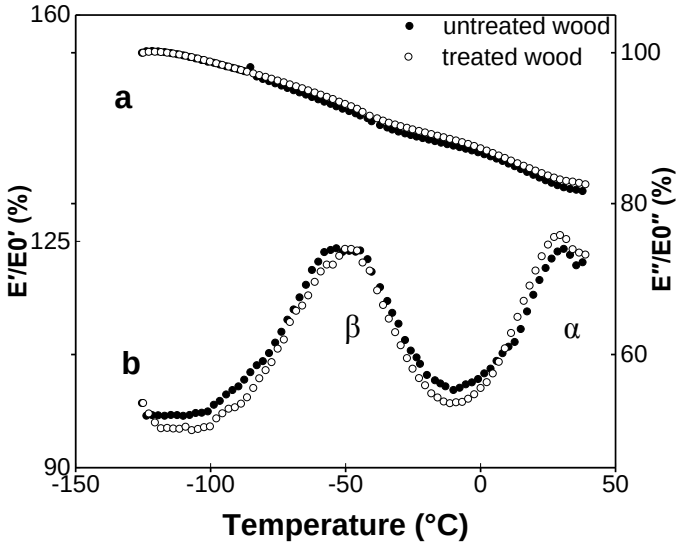

Fig. 1. Temperature dependencies of (a) E'/E_0' and (b) E''/E_0'' for treated and untreated specimens

Figure 2 is the diffraction curve of wood before and after the treatment. The three diffraction peaks of cellulose still remained after the treatment, which indicated that the crystal structure did not change, and the crystallinity was essentially the same through calculation with a crystallinity of 37.7% (treated specimens) and 37.9% (untreated specimens). The results showed that the crystal structure and crystallinity after the low temperature treatment was unchanged.

Above might partially explain why there were no significant differences after the low temperature treatment.

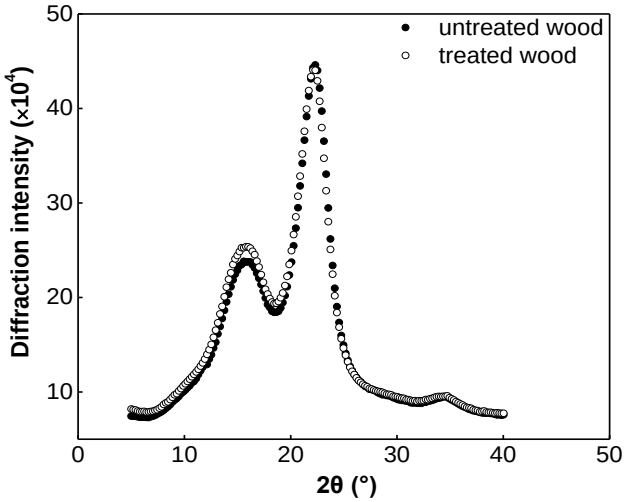

Fig. 2. Diffraction curve of birch wood

Residence to Low Temperatures of Wood and Concrete

As a major structural material, concrete is similar to wood in that its properties are influenced by internal water together with temperature. Table 2 shows the tensile strength (TS), compressive strength (CS), and MOE of concrete after the freeze-thaw cycles (Yamane and Zhao 1980; Ye *et al.* 2010; Shi *et al.* 2012; Xie and Wu 2012).

Table 2. Mechanical Properties of Concrete After the freeze-thaw Cycles (Yamane and Zhao 1980; Ye *et al.* 2010; Shi *et al.* 2012; Xie and Wu 2012)

Methods	Variation (%)	3 cycles (-120 to 20 °C)	3 cycles (-160 to 20 °C)	1 cycles (-196 to 20 °C)	3 cycles (-196 to 20 °C)
Concrete	TS	-33.0	--	--	--
	CS	-3.3	-9.4	-12.2	-50.0
	MOE	--	--	--	-90.0
-- denotes that this was not measured					

It was demonstrated that the CS and MOE of concrete was the maximum at -130 °C, and then showed a linear slow deterioration when it was increased to room temperature. The damage in the concrete was obviously observed after a single cycle from the low temperature to room temperature (Shi *et al.* 2012; Wei *et al.* 2013).

After three cycles of -120 °C to 20 °C, the CS of the C30 concrete decreased by 3.3%, while 9.1% decreases after three cycles of -160 °C to 20 °C (Xie and Wu 2012). When the C60 concrete experienced one cycle of -196 °C to 20 °C, the CS decreased by 12.2% (Ye *et al.* 2010). After three cycles of -196 °C to 20 °C, the CS of C30 concrete decreased by 50%, while the MOE decreased by 90% (Yamane and Zhao 1980). Dahmani *et al.* (2007), furthermore, discovered that the strength and hardness of the water-saturated concrete decreased after one cycle of -170 °C to 20 °C. The higher the water content of the concrete, the greater decrease in strength and hardness was observed.

Unrecoverable damage occurred when the concrete experienced the freeze-thaw cycle which caused the decreased mechanical properties. However, the wood MOE after four treatments increased in this experiment irrespective of the MC. No damage on wood occurred, especially water-saturated wood cell walls, even at -196 °C. Wood has a better

residence to low temperatures compared to the concrete, which will provide a powerful support for the cyclic utilization of wood at low temperatures.

CONCLUSIONS

1. This study evaluated the effect of low temperature treatment on MOE of birch wood. The MOE of all birch wood specimens with four different MCs increased after the treatment, irrespective of MC. The largest increases in water-saturated, green, air-dried, and oven-dried wood specimens were 6.81%, 6.21%, 4.77%, and 2.98%, respectively.
2. Specimens with higher MCs were more affected by the low temperature treatment. However, there were no significant increases in MOE after the treatment at each MC level, and the effect of treatment times (within four times) on MOE was not significant ($P = 0.053 – 0.225$).
3. Liquid nitrogen cyclic treatment did not obviously decrease the flexural property of wood with different MCs. Wood has a better residence to low temperatures compared to concrete.

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