Compression Strength and Modulus of Elasticity Parallel to the Grain of Oak Wood at Ultra-low and High Temperatures

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The influence of temperature on the compression strength (f_{c0}) in the range of -196 ºC to +220 ºC, and compression modulus of elasticity (E_{c0}) parallel to the grain of oak (*Quercus mongolica* Fisch et Turcz.) wood in the range of -196 ºC to +23 ºC were studied. Five specimens were prepared for each temperature level. The specimens were kept at each temperature level for 30 min before a mechanical test was performed in an adjustable-temperature chamber. The results indicated that there were four different failure patterns, depending on the temperature range. When the temperature was decreased from +23 ºC to -196 ºC, the f_{c0} and E_{c0} of wood increased by 283.91% and 146.30%, respectively. The relationships between f_{c0} and temperature and between E_{c0} and temperature could be described by a linear and a polynomial model, respectively. Moreover, the E_{c0} could be used to predict f_{c0} using a polynomial model. However, when the temperature was increased from +23 ºC to +220 ºC, the f_{c0} decreased by 67%, indicating a non-linear relationship.

Keywords: Compression strength; Compression modulus of elasticity; Ultra-low temperature; High temperature

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INTRODUCTION

Temperature plays an important role in the mechanical strength of wood. When wood is heated, its mechanical properties generally decrease. In terms of heat-treated wood, it has been reported that the modulus of rupture (MOR) of spruce (*Picea abies*) decreased by 44 to 50% when the treatment temperature was raised from +100 ºC to +200 ºC, while the modulus of elasticity (MOE) decreased by only 4 to 9% (Bekhta and Niemz 2003). It was found by Manríquez and Moraes (2010) that the average compression strengths parallel to grain of paricá (*Schizolobium amazonicum*) were 32 MPa and 11 MPa at +20 ºC and +230 ºC, respectively. In contrast, as wood is cooled below room temperature, its mechanical properties tend to increase (Cheng 1985; Green *et al.* 1999; Yamada 1971). It has been reported that the MOE values of Swedish pine (*Pinus sylvestris*) wood having a moisture content of 12% were 14.2 GPa and 11.6 GPa at -20 ºC and +20 ºC, respectively (Kollmann and Cote 1968). Kendra and Cortez (2010) found that the MOR of a wooden baseball bat increased by 26% when it was subjected to a temperature of -190 ºC for 24 h. Kollmann and Cote (1968) observed a straight-line relationship between crushing strength of oven-dry wood and temperature in the range of temperature between -191 ºC and +200 ºC. However, until this study, the strength and compression modulus of elasticity of kiln-dried wood have not been measured in an ultra-low temperature environment such as that between -100 ºC and -196 ºC.

Wood consists of cellulose, hemicelluloses, and lignin. Cellulose and hemicelluloses are carbohydrates that are structural components in wood. Cellulose constitutes 40 to 50%, and hemicelluloses 25 to 35%, of wood. The decomposition temperature for hemicelluloses is about +150 to +260 ºC, and the corresponding temperature for cellulose is about +240 to +350 ºC. The mass of lignin starts to decrease only when the temperature exceeds +200 ºC. At a temperature of +150 to +250 ºC, major changes in the hemicelluloses occur, which causes them to degrade (Finnish Thermowood Association 2003). The decrease in wood strength as a result of heat treatment is mostly due to the degradation of the hemicelluloses (Brito *et al.* 2008). However, when wood is cryogenically treated in an ultra-low temperature environment and then mechanically tested, the wood strength increases and the moisture content of wood remains unchanged. Because the water in the specimens forms ice at the freezing point, Merkel (2004) established the following relationship between compression strength (σ) and temperature (T):

$$\sigma = 9.4 \times 10^5 \{(\text{crystal size})^{-1/2} + 3|T|^{0.78}\} \tag{1}$$

As temperature decreases, the ice increases in strength, which may partially explain the increase in wood strength (Michael 1978).

The objective of the study was to determine the effects of temperature on the compression strength parallel to grain of oak in the temperature range from -196 ºC to +220 ºC. The findings may help to explain the behavior of wood contractures during fires as well as to expand the application range of wood in ultra-low temperature environments, such as under liquid natural gas or liquid nitrogen conditions. In the previous studies (Ayrilmis *et al*. 2010; Bekhta and Marutzky 2007; Kendra and Cortez 2010), the specimens were placed for several hours in a climatic chamber set at the desired temperature but then tested outside the chamber at room temperature. Thus, the values that were determined using the previous methods did not report the exact properties of the specimens at those chamber temperatures because the specimens were exposed to room temperature in the few seconds following. The present research is unique because the specimens were tested in the climatic chamber while at the desired temperature.

EXPERIMENTAL

Materials

Oak (*Quercus mongolica* Fisch et Turcz.) board with dimensions 120 (R) x 30 (T) x 800 (L) mm, air-dry density 0.819g/cm^3, and moisture content (MC) 12.18% were sourced from a natural forest. All specimens, each having the dimensions 20 (R) x 20 (T) x 30 (L) mm, were cut from the board. Nineteen temperature levels, -196 ºC (liquid nitrogen), -170 ºC, -150 ºC, -130 ºC, -110 ºC, -90 ºC, -70 ºC, -50 ºC, -30 ºC, -10 ºC, 0 ºC, +23 ºC, +50 ºC, +80 ºC, +110 ºC, +140 ºC, +170 ºC, +200 ºC, and +220 ºC were used for the experiments. The specimens were sorted into 19 groups based on their average weights to average the coefficient of variation for each group. Five specimens were prepared for each temperature level.

Methods

The specimens were cryogenically treated with liquid nitrogen for 2 h in an adjustable-temperature chamber of a universal mechanical testing machine. A 3 mm diameter end-perforation was made in each specimen of size 20 (R) x 20 (T) x 50 (L) mm. A thermocouple was inserted in the hole to attain a measurement of the temperature at the geometrical center of the specimen. This allowed for the determination of the time required for the specimen to reach the desired temperature. The compression strength and modulus of elasticity were measured at the low end of the temperature range.

The specimen-clamping head of the universal mechanical testing machine was pre-set in an adjustable-temperature chamber. The specimens were heated in the chamber for 30 min. The compression strength was measured at the high end of the temperature range.

The mechanical tests were performed with a MTS-SANS CMT5000 universal testing machine (Shenzhen, China) with a max load of 100 kN and a temperature-controlled chamber. The experimental device was placed inside the chamber. The compression load was applied at a speed of 1.0 mm/min during a period of 1 to 5 min. The test was considered completed when the specimen failed.

The f_{c0} values of the specimens were calculated as follows,

$$f_{c0} = \frac{f_{c0\,max}}{a \times b} \tag{2}$$

where f_{c0max} is max load, a is width of cross section, and b is thickness of cross section.

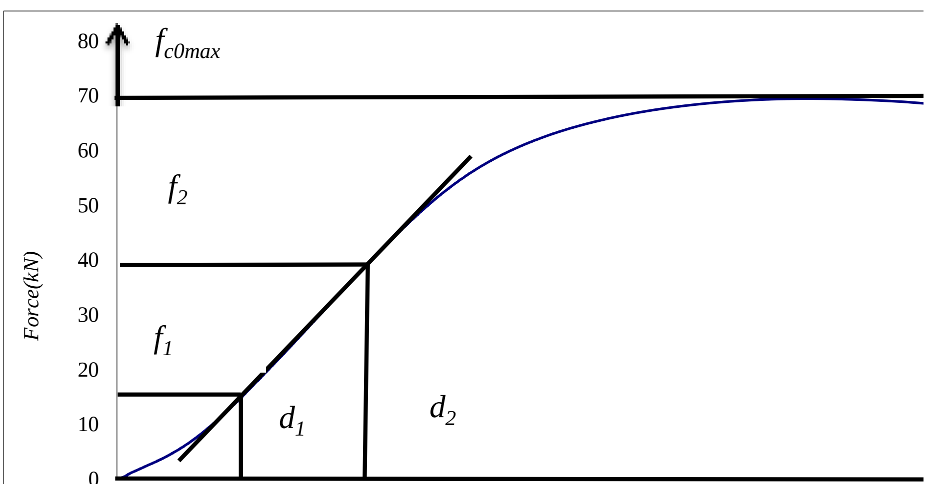


Fig. 1. The curve of force and deformation

The compression modulus of elasticity (E_{c0}) values were calculated using the 10% and 40% values of the failure load (f_{c0max}) (Fig. 1) from the load and deformation curve under proportional limits using the following formula,

$$E_{c0} = \frac{(f_2 - f_1) \times L}{a \times b \times (d_2 - d_1)} \tag{3}$$

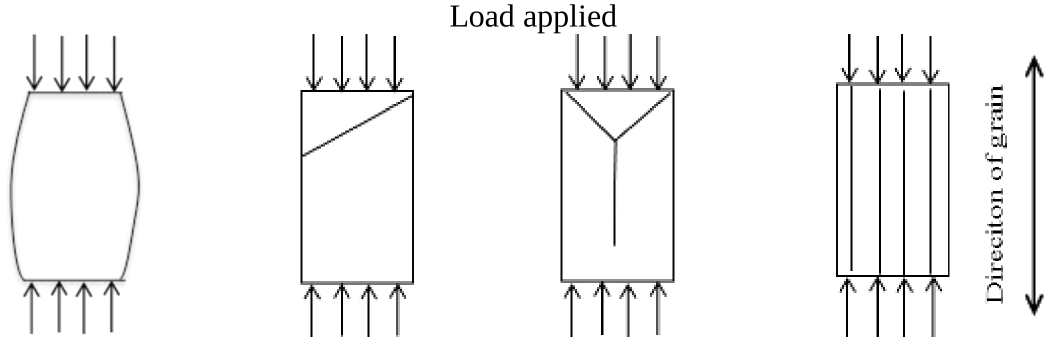
where L is length of specimen, $(f_2\text{-}f_1)$ is increment of load on the straight line portion of the load deformation curve, a is width of cross section, b is thickness of cross section, and $(d_2\text{-}d_1)$ is increment of deformation corresponding to $F_2\text{-}F_1$.

The Duncan's multiple comparison tests were performed with IBM SPSS Statistics 17.0 software.

RESULTS AND DISCUSSION

Specimen Failure

Four patterns of failure were shown by the specimens: pear-shaping, shearing, wedge-splitting, and splitting, all of which are illustrated in Fig. 2. In the range from +220 ºC to +50 ºC (Fig. 2a), the boards showed a pear-shaped pattern of failure. In the range from +23 ºC to -30 ºC (Fig. 2b), the failure occurred between the middle and the end of the specimens, at an angle ranging from 30° to 60°. In the temperature range from -50 ºC to -110 ºC (Fig. 2c), the boards underwent a wedge-splitting type of failure, in which the direction of the split was either radial or tangential. In a range of -130 ºC to -196 ºC (Fig. 2d), the specimens were ruptured parallel to the grain, which is a failure pattern known as splitting.



(a) Pear shaping (+220—+50 ℃) (b) Shearing (+23—-30 ℃) (c) Wedge splitting (-50—-110 ℃) (d) Splitting (-130—196 ℃)

Fig. 2. Different failure patterns

f_{c0} and E_{c0} under Ultra-low Temperatures

The determined values for f_{c0} and E_{c0} parallel to the grain of the wood at ultra-low temperatures are presented in Table 1. The table shows that the f_{c0} and E_{c0} increased as the temperature was reduced. The average f_{c0} and E_{c0} values at +23 ºC were 57.17 MPa and 7.83 GPa, respectively, while the average f_{c0} and E_{c0} values at -196 ºC were 219.49 MPa and 19.28 GPa, respectively. When the temperature was reduced from +23 ºC to -196 ºC, the average f_{c0} and E_{c0} values were increased by 283.91% and 146.30%, respectively. Several researchers also reported similar increases in the f_{c0} of wood with decreasing temperatures (Chang 1985; Green *et al.* 1999; Yamada 1971). According to the Duncan's multiple comparison tests that were run, significant differences ($p<0.05$) were observed in both the f_{c0} and E_{c0} by SPSS statistics 17.0 software. The letters in Table 1 show the results of Duncan's multiple range tests. The analysis of variance showed that there were statistically significant differences between the f_{c0} at +23 ºC and those at other temperature levels. No significant differences in E_{c0} were found between +23 ºC and -50 ºC, or between -70 ºC and -150 ºC. However, a significant difference in E_{c0} was observed between -170 ºC and the liquid nitrogen temperature of -196 ºC.

Table 1 also shows the effect of temperature on the f_{c0} and E_{c0} results. The f_{c0} and E_{c0} values of the wood increased with decreasing temperature, which was mainly attributed to the formation of ice crystals in the wood cell walls at ultra-low temperatures, especially at the liquid nitrogen temperature (-196 ºC). The liquid nitrogen condition gave rise to the maximum f_{c0} and E_{c0}, which was attributed to the 10% increase in weight that the specimen incurred as a result of having absorbed and frozen some extra moisture. The previous study reported that the MOE of Swedish pine was 14.2 GPa at -20 ºC and 11.6 GPa at +20 ºC (Kollmann and Cote 1968). Kendra and Cortez (2010) found that the MOR of a wooden baseball bat was increased by 26% at -190 ºC. Several researchers reported similar increases in the MOE and MOR of wood-based panels with decreasing temperatures (Ayrilmis *et al*. 2010; Bekhta and Marutzky 2007; Suzuki and Saito 1987; Yu and Östman 1983). However, in those previous studies, the specimens were placed into a climatic chamber at the desired temperature for several hours and then tested in a separate room temperature environment. Thus, the values determined using the previous methods did not report the exact properties of the specimen at the chamber temperatures because the specimens were equalized to room temperatures in the few seconds following. The present research is unique because the specimens were tested in the climatic chamber while at the desired temperature.

Zhang *et al.* (2010) investigated and characterized the strength and fracturing of wood cells through a uniaxial micro-compression test and reported that the compressive strengths of the loblolly pine (*Pinus taeda* L.) and the Keranji (*Dialium* spp.) cell walls were 125 MPa and 160 MPa, respectively. Water is in contact with hydrophilic hydroxyl groups on the cellulose chains. In one study, when the specimens were treated under the ultra-low temperatures, the water in the specimens turned into ice. However, the compressive strength of the freshwater ice depended on the crystal size, the strain rate, and the ice temperature. When the ice temperature was -170 ºC, the compressive strength of ice can be as high as 155 MPa (Merkel 2004). The strength of adhesion of ice to substrate surfaces, such as wood and concrete, may exceed the strength of the substrate material and cause the substrate to break or spall (Ayrilmis *et al*. 2010). Moreover, the longitudinal modulus of the oak cell wall was determined to be 18.4 GPa by the method of nanoindentation (Wu *et al*. 2009). It was concluded that at low moisture contents such as 12.18%, the presence of frozen water molecules between the cellulose fibrils improved f_{c0} and E_{c0} of the wood due to the fact that the water molecules stiffened the cellulose fibrils in the same manner as an adhesive.

Table 1. f_{c0} and $\underline{E}_{c0}$ of Oak wood at a temperature of +23 to -196 ºC

Temperature (ºC)	+23	0	-10	-30	-50	-70	-90	-110	-130	-150	-170	-196
f_{c0} (MPa)	57.17 J	75.85 I	74.26 I	91.85 H	100.22 H	121.92 G	144.12 F	159.92 E	187.76 DC	213.07 AB	200.80 BC	219.49 A
	(2.32)	(2.11)	(4.73)	(5.12)	(3.90)	(3.76)	(9.07)	(9.44)	(9.01)	(13.47)	(18.13)	(19.23)
E_{c0} (GPa)	7.83 E	8.73 ED	7.22 E	8.72 ED	9.59 ED	10.97 CD	11.76 CD	11.04 CD	10.94 CD	13.48 C	16.38 B	19.28 A
	(0.90)	(0.97)	(1.28)	(2.11)	(1.55)	(0.87)	(1.04)	(1.56)	(0.85)	(1.75)	(3.51)	(5.27)

Groups with the same letters in each column are those in which there is no statistical difference (at the 0.05 level) between the samples according to the Duncan's multiple range tests. The values in parentheses are standard deviations

f_{c0} at High Temperature

The results of the tests of compression strength parallel to grain at high temperatures are presented in Table 2. With increase in temperature, the f_{c0} values were initially reduced, then increased, and finally reduced again. The results were similar to those obtained by previous researchers (Cao *et al*. 2012; Kubojima *et al*. 2000; Manríquez and Moraes 2010; Millett and Gerhards 1972; Moraes *et al*. 2004; Shi *et al*. 2007). The moisture content of the specimens decreased with the increasing temperature and was near to zero by +170 ºC (Table 2). Several researchers have reported that moisture content and temperature are important factors affecting the strength of wood. A reduction in moisture content causes an increase in wood strength, while an increase in temperature produces a decrease in strength. According to Schaffer (1973), the compression strength depends strongly on the lignin located at the exterior of the wood fibers, which was softened at +110 ºC. In this study, the f_{c0} value at +110 ºC was lower than that at +140 ºC. Jiang (2013) observed that when the temperature was increased to +140 ºC, the hemicellulose content began to decrease, the α-cellulose content began to increase, and the cellulose crystallinity, which was higher than it had been at +23 ºC, increased. As the temperature continuously increased, the hemicelluloses and cellulose were degraded, resulting in the loss of mass and the reduction in wood strength. This degradation was probably the reason that the f_{c0} value was the lowest at +220 ºC.

Table 2. f_{c0} of Oak Wood at a Temperature of +23 to +220 ºC

Temperature (ºC)	+23	+50	+80	+110	+140	+170	+200	+220
f_{c0} (MPa)	57.17	44.89	34.16	33.47	44.36	36.86	28.86	18.86
	A	B	C	C	B	C	D	E
	(2.32)	(3.30)	(2.08)	(4.80)	(5.39)	(2.46)	(0.85)	(1.21)
After-test MC (%)	12.16	9.71	8.32	5.17	1.81	0.59	0.02	0.00
	(0.16)	(0.30)	(0.90)	(1.32)	(0.71)	(0.25)	(0.06)	0.00

Groups with the same letters in each column are those in which there is no statistical difference (at the 0.05 level) between the samples according to the Duncan's multiple range tests. The values in parentheses are standard deviations

The Regression Model

From Fig. 3, the relationships between f_{c0} and temperature (T) were obtained as follows:

$$f_{c0} = -0.813\times T + 70.649,\ -196\ ºC \leq T \leq +23\ ºC,\ R^2=0.974\ (F<0.001) \quad (4)$$

$$f_{c0} = -3.0\times 10\text{-}5\times T^3 + 0.011\times T^2 - 1.264\times T + 81.529,\ +23\ ºC \leq T \leq +220\ ºC,\ R^2 = 0.942\ (F=0.006) \quad (5)$$

The coefficients of determination for linear and nonlinear modes were 0.974 and 0.942, respectively, at a significance level of 0.01. It can be noted that there were two separate temperature ranges, which is the reason for the qualitatively different changes in the strength properties of wood, and those ranges were:

Range 1: -196 ºC to +23 ºC. In this range, the moisture content did not change except for under the liquid nitrogen -196 ºC, but the f_{c0} increased with the decreasing temperature, which was captured by a linear model. A similar result was obtained by

Ayrilmis *et al.* (2010), who reported that the increase in temperature from -30 ºC to +30 ºC adversely influenced the flexural properties of plywood, medium density fiberboard, and oriented strand board. Bekhta and Marutzky (2007) found that the relationships between the MOR/MOE and temperature from -40 ºC to +40 ºC were described by a linear model. Cheng (1985) also discovered that the relationship between f_{c0} and temperature was described by a linear model.

Range 2: +23 ºC to +220 ºC. In this range, the f_{c0} values initially decreased in correspondence with the increase in temperature, increased, and then decreased again. A polynomial function was more suitable than a linear or exponential function to describing the relationship between f_{c0} and temperature.

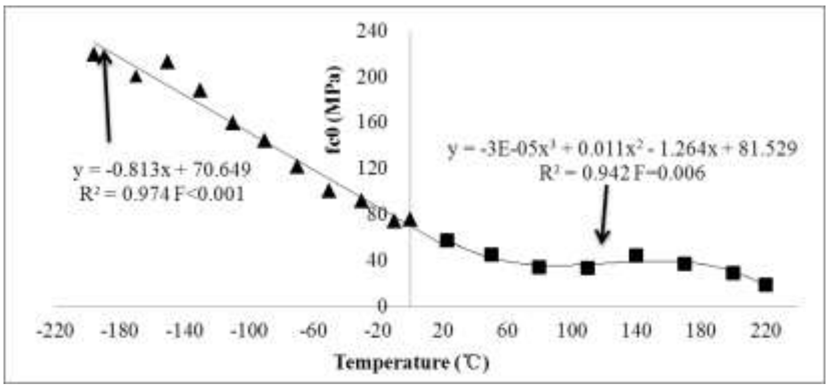


Fig. 3. Influence of temperature on the $\boldsymbol{f_{c0}}$ of wood

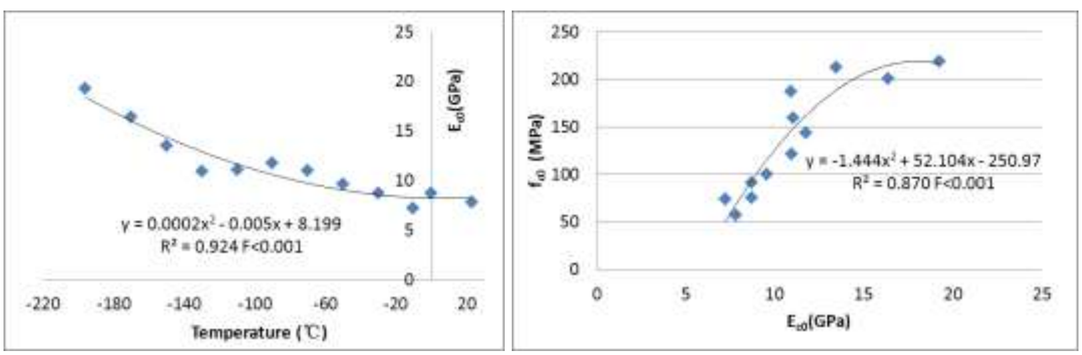


Fig. 4. The relationships between $\boldsymbol{E_{c0}}$ and temperature (a), and $\boldsymbol{f_{c0}}$ and $\boldsymbol{E_{c0}}$ (b)

In Fig. 4, the relationships between E_{c0} and temperature (T), and E_{c0} and f_{c0} were established using a polynomial model with the following form:

$$E_{c0} = 2.0\times10\text{-}4\times T^2 - 0.005\times T + 8.199,\ -196\ ºC \leq T \leq +23\ ºC,\ R^2 = 0.924\ (F<0.001) \tag{6}$$

$$f_{c0} = -1.444\times E_{c0}^2 + 52.104\times E_{c0} - 250.97,\ -196\ ºC \leq T \leq +23\ ºC,\ R^2 = 0.870\ (F<0.001) \tag{7}$$

The coefficients of determination for these two relationships were 0.924 and 0.870, respectively, at the significance level of **0.001.** The E_{c0} values could be estimated based on temperature, which could be used for predicting the f_{c0} of the wood.

CONCLUSIONS

This study evaluated the influence of temperature, in the range of -196 to +220 ºC, on the f_{c0} and E_{c0} parallel to the grain of oak wood. The results are summarized as follows:

1. Four failure patterns, known as pear-shaping, shearing, wedge-splitting, and splitting, were observed within the temperature ranges +220 to +50 ºC, +23 to -30 ºC, -50 to -110 ºC, and -130 to -196 ºC, respectively.

2. When the temperature was decreased from +23 ºC to -196 ºC, the average f_{c0} and E_{c0} were increased by 283.91% and 146.30%, respectively. The relationships between f_{c0} and temperature and E_{c0} and temperature constituted a linear model and a polynomial model, respectively. Moreover, it was found that a polynomial model could be used to predict f_{c0} from E_{c0} at temperatures lower than 23 ºC.

3. When the temperature was increased from +23 ºC to +220 ºC, the average f_{c0} values **decreased** from 57.17 MPa at +23 ºC to 18.86 MPa +220 ºC. The f_{c0} value at the highest temperature level corresponded to 32.99% of the f_{c0} value at room temperatures. The relationship between f_{c0} and temperature could be described by a polynomial model.

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