BREAKEVEN POINT IN ULTIMATE THICKNESS BETWEEN MOISTURE-REDUCED SHRINKAGE AND THICKNESS RECOVERY OF DENSIFIED SOFTWOOD SPECIES: PART 1: AT ROOM TEMPERATURE

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Abstract. Thermo-hygro-mechanical (THM) densification technique has been used to increase the density and mechanical properties of underutilized softwoods. One potential application of the densified softwoods is the substitute of hardwood flooring. This technique is usually employed to mechanically compress the softened wood in the radial direction with the assistance of a high temperature greater than 100°C. Use of high temperature shows that the THM densification process consumes a lot of thermal energy, losing the economic competitivity in comparison with the hardwood floorings. From the energy-saving point of view, the authors tried to densify two softwood species at three temperatures of 20°C, 50°C, and 90°C with various compression ratios (CRs), followed by air-drying (AD) the densified wood specimens at room temperature. It was discovered that the decrease of MC would result, during the AD conditioning, in shrinkage of densified wood, which could offset part of the thickness recovery of densified wood. However, this phenomenon was not well addressed. This study was aimed at discussing the effect of shrinkage on the thickness recovery ratio (TRR) of densified softwoods. To ignore the influence of temperature on the shrinkage capability of wood because of the decomposition of hemicellulose, only the data of densified wood specimens compressed and posttreated at room temperature were analyzed in this study. Two softwood species used were eastern white pine (*Pinus strobus*) and balsam fir (*Abies balsamea*). The CRs ranged from 0.05 CR and 0.65 CR. The major findings were: 1) the final thickness of densified wood was a result of "recovery" containing elastic deformation and visco-elastic deformation, and "shrinkage" of cell walls; 2) the relationship of TRR of densified pine or fir and CR followed an exponential increase trend at 20°C; 3) after AD conditioning, the breakeven point in thickness between moisture-reduced shrinkage and visco-elastic recovery when the CRs of densified pine and fir at 20°C were 0.12 and 0.13, respectively.

Keywords: Air-drying conditioning, thermos-hydro-mechanical densification, softwood, thickness recovery, wood shrinkage.

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INTRODUCTION

Thermo-hydro-mechanical (THM) densification technique has been used to increase the density and mechanical properties of low-density wood. This technique normally consists of three processes: softening, densifying, and posttreatment. For softening, wood is usually soaked into water, which can effectively minimize the damage of the cellular structure and decrease the energy required to initiate chain mobility because of plasticization of cell wall compounds by water (Kelley et al 1987). After softening, wood cell walls and lumens are full of bound water and free water, at which the wood has a high MC exceeding FSP (FSP ≈ 25%). During the following densifying process, external loads apply in the transverse direction, most likely in radial one, of the wood with the assistance of a temperature that is higher than 100°C, causing wood cell walls to buckle and thereby the volume of lumens decreases to different degrees depending on the compression ratio (CR) defined as the ratio of reduced thickness and initial thickness (Scharfetter 1990; Norimoto 1993). Kamke and Kutnar (2010) described the wood buckling includes 1) initial linear elastic deformation, 2) nonlinear visco-elastic deformation, and 3) permanent plastic deformation. After releasing the loads/stresses applied, the elastic deformation is instantaneously released (ie springback), followed by the slow recovery of visco-elastic deformation with time. A good densified wood product should be of a uniform thickness. Various posttreatment methods were investigated to minimize the thickness recovery of the THM densified wood, including steam-, heat-, or oil-treatment, at various temperatures and time periods on the decrease of thickness recovery (Navi and Gigardet 2000; Welzbacher et al 2008; Gong et al 2010; Rautkari et al 2010; Fang et al 2012; Kutnar and Kamke 2012; Li et al 2012; Popescu et al 2014; Rautkari et al 2014). It was widely agreed from these studies that the thickness recovery could be almost minimized if the densified wood was treated at a high temperature ranging from 180°C to 230°C.

It should be pointed out that such a THM densification process consumes a lot of energy, especially thermal energy, which reduces the economic competitivity of the densified low-density wood products in comparison with high-quality hardwood ones, for example, wood floorings. From the energy-saving point of view, the authors made their efforts to densify two softwood species at three temperatures of 20°C, 50°C, and 90°C with various CRs, followed by air-drying (AD) the densified wood specimens at room temperature and at 65% relative humidity (RH) until its MC stabilized and reached a constant value. It was discovered that the decrease of MC would result, during this AD conditioning, in shrinkage of densified wood, which could offset part of thickness recovery of densified wood. The shrinkage and thickness recovery occurred interactively at the same time. However, this phenomenon has not been well discussed so far.

This study was aimed at elucidating the effect of shrinkage that occurred during the posttreatment process on the thickness recovery of THM-densified softwoods. To ignore the influence of temperature on the shrinkage capability of wood because of the decomposition of hemicellulose, only the data of densified wood specimens compressed and posttreated at room temperature were analyzed in this study. The effects of the other two temperatures, 50°C and 90°C, on the ultimate thickness of two densified wood species are addressed in Part II of this study and will be published elsewhere. It is pointed out that the densified wood specimens compressed at room temperature were also named as THM-densified wood specimens to keep in a consistence with another two groups of specimens discussed in Part II. It is expected to provide some useful information for designing a low energy consuming THM densification process.

MATERIALS AND METHODS

Materials

The species tested were eastern white pine (*Pinus strobus* L.) and balsam fir (*Abies balsamea* L. Mill.). The average specific gravity values at oven-dry (SG_{od}) of pine and fir boards used were 0.412 with a standard deviation (SD) of 0.020 and

0.375 with a SD of 0.037, respectively. The nominal dimensions of a specimen were 30 mm (L) × 30 mm (T) × 25 mm (R). Specimens with density located in mean ± 2 SD, ie a 95% confidence level was used for grouping. There were five groups for each species to produce densified wood with CRs of 0.05, 0.10, 0.25, 0.50, and 0.60/0.65, as shown in Table 1. Each group had five replicates, which were selected based on the SG_{od} with an interval of 0.02 (white pine) and 0.037 (balsam fir), respectively. This could ensure that each group had a similar average SG_{od} and SD: 0.412 ± 0.031 and 0.375 ± 0.058 for white pine and balsam fir, respectively.

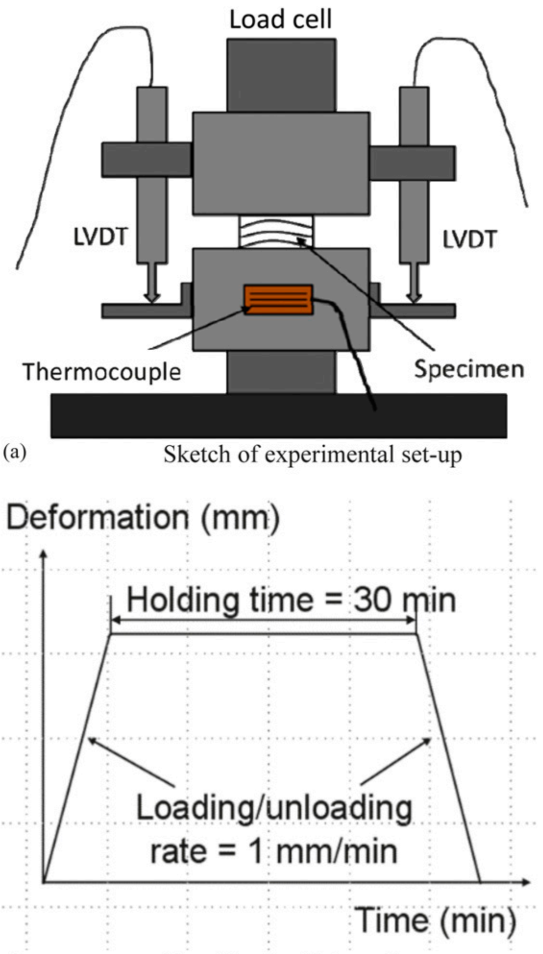
THM Densification Processing Technique

The THM densification procedure used in this study was: 1) soaking all wood specimens in water in a pressure vessel, followed by an 85-kPa vacuum for 45 min and then the 517-kPa pressure for another 45 min; 2) densifying the specimens in the radial direction to a preset deformation, ie nominal CR, as shown in Table 1, and holding for 30 min at room temperature; 3) unloading and then removing the specimens from the jig; and 4) AD the specimens in a conditioning chamber at 20 ± 2°C and 65 ± 5% RH until their MCs were stabilized, which was determined when the weight change of each specimen was less than 0.02 g. It should be pointed out here that the room temperature used in the densification process, ie step 2, was aimed at minimizing the effect of high temperature on the strength of wood.

The experimental set-up and a compressing history diagram are shown in Fig 1(a) and (b), respectively. An Instron machine was used to control a compressing device/jig. The speed of loading and unloading period was 1 mm/min. Two linear variable differential transducers (LVDTs) were used to measure the deformations. The nominal CRs ranged from 0.05 to a value not causing any visual cracks. The actual CRs were just slightly lower than the nominal values in this study as shown in Table 1. To facilitate discussion, only nominal CRs were used in this article.

Table 1. Nominal and actual compression ratios (CRs) used.

		Nominal CR					
	Species	0.05	0.10	0.25	0.50	0.60	0.65
Actual CR	Pine	0.04	0.09	0.24	0.49	0.59	—
	Fir	0.05	0.09	0.24	0.49	—	0.64



(a) Sketch of experimental set-up

(b) Densification history diagram

Figure 1. Experimental setup (a) and densification history (b). LVDT, linear variable differential transducers.

MC, Thickness Recovery Ratio (TRR), and Unrecovered Thickness Ratio (UTR)

To examine the unrecovered thickness of densified wood specimens, all specimens after step 4) were resoaked in cold water for 24 h and then put into boiling water for another 2 h to ensure that the buckled thickness was fully recovered, ie step 5). Then, all specimens were oven-dried at 100 ± 2°C until their MCs decreased to 0%, ie step 6).

The mass of each specimen after steps 1) vacuum-soaking (VS), 3) removal of jig (RJ), 4) AD, and 6) oven-drying (OD) was measured via a digital balance with an accuracy of 0.001 g. The MC of each specimen at each step (ie VS, RJ, and AD) is calculated by using Eq 1.

$$MC_i\ (\%) = \frac{M_i - M_{OD}}{M_{OD}} \times 100 \quad i = VS,\ RJ, \text{and } AD \tag{1}$$

where, M_i is the mass at a given test condition of VS, RJ, or AD in gram; and M_{OD} is the oven-dry mass in gram.

The thickness of each specimen in steps 1) VS, 3) RJ, 4) AD, and 5) after boiling (AB) was measured using a micrometer with an accuracy of 0.001 mm. The TRRs of each specimen after RJ and AD are calculated using Eq 2.

$$TRR_i\ (\%) = \frac{T_i - T_n}{T_n} \times 100 \quad (i = RJ \text{ and } AD) \tag{2}$$

where, T_i is the thickness of a densified wood specimen after RJ or AD in millimeter; and T_n is the thickness of a densified wood specimen reaching a given nominal CR recorded by LVDT (ie the thickness in step 2) of THM densification process) in millimeter.

The UTR of a densified specimen after step 5) AB is calculated using Eq 3.

$$UTR\ (\%) = \frac{T_{VS} - T_{AB}}{T_{VS}} \times 100 \tag{3}$$

where, T_{VS} is the thickness of a densified wood specimen after step 1) VS in millimeter; T_{AB} is the thickness of a densified wood specimen after step 5) AB in millimeter.

RESULTS AND DISCUSSION

Densification and Relaxation Processes

Figure 2 provides two examples showing the load vs deformation curves of pine and fir densified at CRs of 0.60 (pine) and 0.65 (fir). It can be seen that the load vs deformation curves between pine and fir are quite similar, but the load used for densifying pine is slightly greater than that of fir, which might be because of the relatively higher

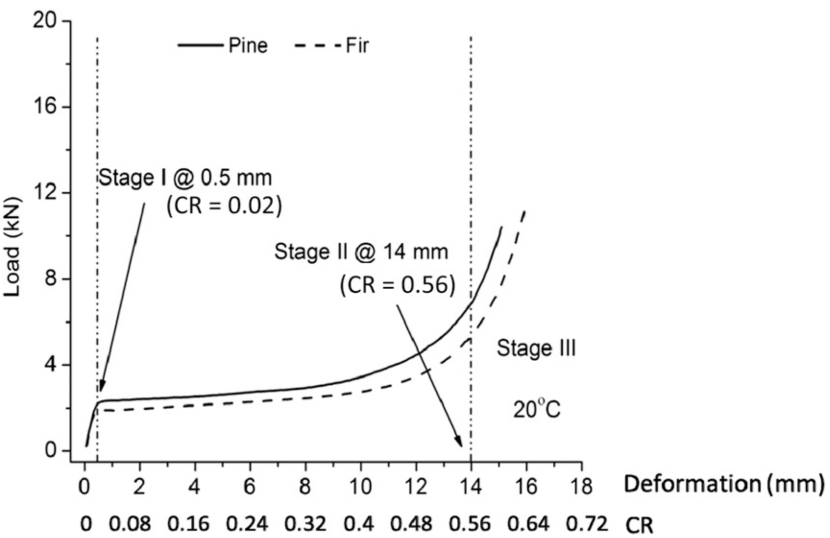


Figure 2. Examples of load–deformation curves of pine (compression ratio [CR] = 0.60) and fir (CR = 0.65) during densification.

density of pine providing more wood substances than fir (Gong et al 2006). In Fig 2, stage I is a linear elastic region, at the end of which the deformations of both pine and fir were about 0.5 mm, generating a CR of 0.02. It indicates that the smallest CR of 0.05 used in this study included elastic deformation and visco-elastic deformation. stage II, as a plateau region, where the load increases in a nonlinear way and almost keeps constant covers the region until the deformation reaches about 14 mm (CR of 0.56). After passing 14 mm, in stage III, the load increases abruptly. CRs of 0.60 and 0.65 are located in stage III.

Figure 3 shows two examples of the load/stress relaxation processes of pine and fir during 30-min holding time at CR of 0.50. The relaxation curves of pine and fir are also very similar, but the loads are different. In the first 3 min, the load dropped very quickly to about 80% of the initial load. Then the load decreased slowly to about 60% of the initial load in the remaining 27 min.

Moisture Changes during Densifying and AD Processes

Figure 4 plots the MC changes (mean values) of pine and fir at VS (ie the initial MC), RJ, and AD. In Fig 4, the initial MCs of undensified pine and

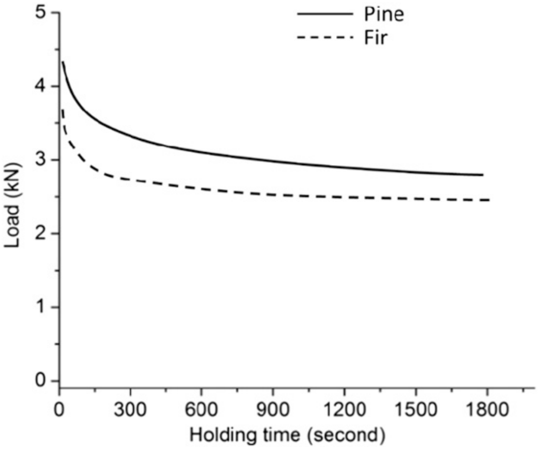


Figure 3. Examples of load/stress relaxation of pine and fir during densification (compression ratio = 0.50).

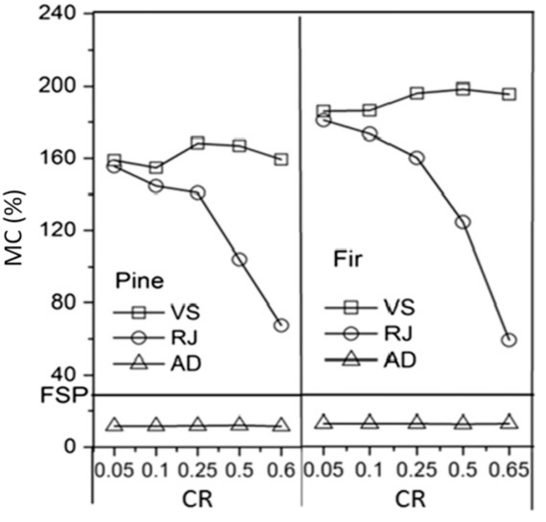


Figure 4. Change in average MC of pine (left) and fir (right) at vacuum-soaking (VS), removal of jig (RJ), and air-drying (AD) at 20°C. CR, compression ratio.

fir are greater than 160% and 180% at VS, respectively. At RJ, the MCs of densified pine and fir significantly decrease with increasing CR because of the volume decrease of the lumens of wood cells. The MCs of densified pine and fir at each CR are greater than FSP, indicating no wood shrinkage occurring at RJ. At the end of AD, the MCs of densified pine and fir specimens are low to about 12% and 9%, respectively, which suggests that the densified pine and fir specimens underwent shrinkage.

TRRs of Densified Pine and Fir

The TRRs of all densified pine and fir specimens at all CRs after RJ and AD are plotted in Fig 5. Overall, the TRR of densified pine and fir at RJ and AD dramatically increases with increasing CR. The thickness recovery at RJ is the recovery of elastic deformation. As mentioned previously, the decrease of MC from more than FSP to about 12% or 9% resulted in the shrinkage of densifed wood, so that the thickness change at AD consisted of thickness recovery of visco-elastic deformation and wood shrinkage. The TRR data plotted in Fig 5 show that the difference between two TRRs at RJ and AD varies with CR: at CRs of

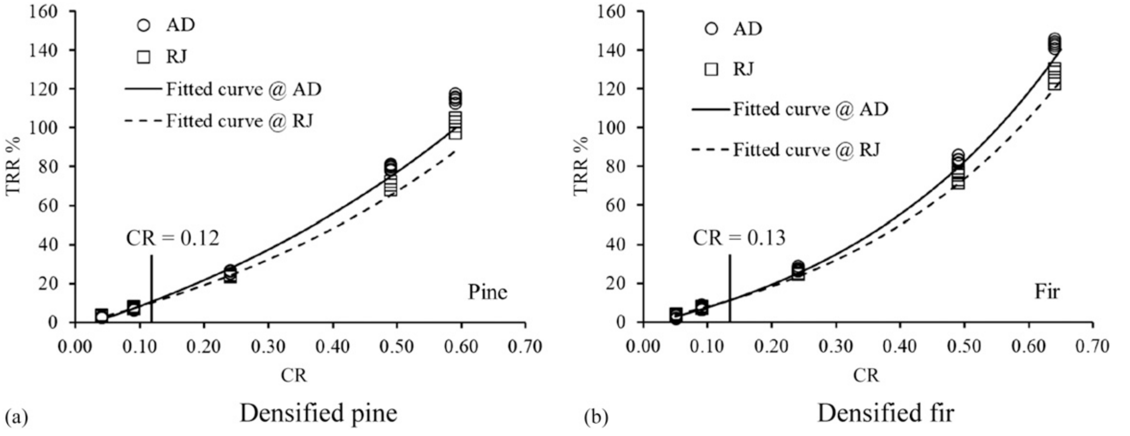


Figure 5. Thickness recovery ratio (TRRs) of densified pine (a) and fir (b) tested. AD, air-drying; RJ, removal of jig; CR, compression ratio.

0.05 and 0.10, the TRRs of densified pine and fir at AD are lower than those at RJ, whereas at CRs of 0.25, 0.50, 0.60, or 0.65, the TRRs at AD are larger than those at RJ. Theoretically, there is a point of interaction at which two TRRs of densified wood at RJ and AD are equal. To find this breakeven point of interaction, a mathematical model was used to fit the experimental results.

The TRRs of densified pine and fir measured at RJ and AD increased exponentially with increasing CR from 0.05 to 0.60 (pine) and to 0.65 (fir). A mathematical model was chosen to describe this exponential growth, as given in Eq 4.

$$\mathrm{TRR}\ (\%) = a e^{\mathrm{CR}/b} + c \quad (0.05 \le \mathrm{CR} \le 0.60 \text{ or } 0.65) \tag{4}$$

where, a, b, and c are empirical constants, which are determined by a curve fitting technique by means of OriginPro 8.0 software (OriginLab Corporation 2010).

The fitted curves of densified pine and fir specimens based on Eq 4 are also drawn in Fig 5. The empirical constants of the TRR model for densified pine and fir at each condition are listed in Table 2. Meanwhile, the breakeven points of two TRR model curves for densified pine and fir are given Table 2 and marked in Fig 5.

In Fig 5(a) and (b), the breakeven points of two fitted curves of densified pine and fir are at a CR of 0.12 and 0.13, respectively. At this point, the TRR of densified wood at AD is equal to that at RJ. As CR exceeds this point, the TRR of densified wood at AD is larger than that at RJ, which means the shrinkage of densified wood could not fully offset the further visco-elastic thickness recovery. When CR is lower than this point, the shrinkage could offset the visco-elastic thickness recovery and a part of elastic deformation released after RJ.

This finding could assist in determining a proper CR to control the thickness recovery of densified wood using the hygro-mechanical densification process. For example, the densification of balsam fir wood at a CR of 0.13 does not require an external force or extra heat to fix the thickness

Table 2. Summary of constants of thickness recovery ratio (TRR) models and compression ratio (CR) values at interaction point.

Species	TRR model at Removal of jig	TRR model at Air-drying	CR at interaction point
Pine	$\mathrm{TRR} = 44.73e^{(\mathrm{CR}/0.54)} - 45.55$ ($R^2 = 0.994$)	$\mathrm{TRR} = 68.06e^{(\mathrm{CR}/0.64)} - 71.34$ ($R^2 = 0.997$)	0.12
Fir	$\mathrm{TRR} = 24.47e^{(\mathrm{CR}/0.36)} - 24.35$ ($R^2 = 0.999$)	$\mathrm{TRR} = 27.88e^{(\mathrm{CR}/0.36)} - 29.27$ ($R^2 = 0.999$)	0.13

recovery contributed by the visco-elastic deformation. Air-conditioning the densified fir at a free state can ensure that the densified fir has a preselected dimension. If the CR is larger than 0.13, the internal shrinkage force is not large enough to counterbalance the force generated by the recovery of visco-elastic deformation, therefore, an external force or extra heat is required to be applied to restrain the thickness recovery during the air-conditioning. The former could be realized by air-conditioning the densified wood in jigs secured by bolts/screws and the latter has been reported by other researchers, as discussed in the introduction section.

The effects of the other two temperatures (50°C and 90°C) on the ultimate thickness of two densified wood species were different with that at 20°C because of the decomposition of hemicellulose to some degree, which will be addressed in Part II.

UTRs of Densified Pine and Fir

The UTRs of all densified pine and fir specimens at different CRs AB are plotted in Fig 6. The UTRs of pine are slightly higher than those of fir. It might be attributed to the about 10% larger density of pine than fir, generating more permanent strains fixed in densified pine than in densified fir. This indicates that almost no damage of cell walls occurred during the densification process. The maximum CR of 0.65 did not exceed the compressive limit of densified pine and fir.

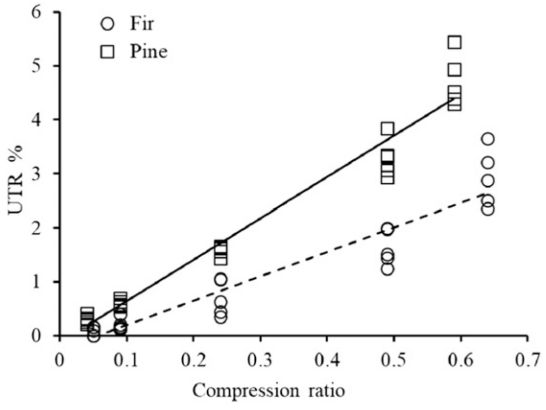


Figure 6. Unrecovered thickness ratio (UTRs) of densified pine and fir tested.

CONCLUSIONS

This study investigated, in terms of TRR, how the shrinkage of densified wood (pine and fir) occurring during the AD (posttreatment) process offset the thickness recovery of THM-densified wood. Meanwhile, this study also examined how the CR and MC change in TRR-influenced UTR of densified wood. The following conclusions could be drawn:

1. After the AD conditioning, the final thickness of densified wood was a result of "recovery" containing elastic deformation and visco-elastic deformation, and "shrinkage" of cell walls;
2. The relationship of TRR and CR could be simulated using an exponential increase model for densified pine or fir at 20°C; and
3. After AD conditioning, the breakeven point in thickness between moisture-reduced shrinkage and visco-elastic recovery when the CRs of densified pine and fir at 20°C were 0.12 and 0.13, respectively.

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