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ORIGINAL ARTICLE

Performance of Sugi lamina impregnated with low-molecular weight phenolic resin

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Abstract This study was conducted to evaluate the performance of Sugi lamina impregnated with low-molecular weight phenolic (LMWP) resin using the full cell process followed by curing at high temperature. In this study, penetration of LMWP resin into finger-jointed lamina was examined. Physical and mechanical properties, such as surface hardness, dimensional stability, bending and shear strength of LMWP-resin-treated and untreated lamina were investigated. In addition, the bonding quality and nail-withdrawal resistance of 3-ply assembly specimen made from LMWP-resin-treated and untreated lamina bonded using resorcinol–phenol formaldehyde resin adhesive were also investigated. The main results were as follows: LMWP resin was found to have penetrated sufficiently into finger-jointed lamina. The physical properties of LMWP-resin-treated lamina were found to have improved significantly in comparison with untreated lamina. However, no significant difference was found between LMWP-resin-treated and untreated lamina in terms of their mechanical properties. There was an improvement in bonding quality of the assembly made from LMWP-resin-treated lamina when compared with that made from untreated lamina. In the assembly made from untreated lamina, a significant decrease in nail-withdrawal resistance was observed between dry conditions test and after humidity conditioning test. However, the same tendency was not found in the assembly made from LMWP-resin-treated lamina.

Keywords Lamina · Low-molecular weight phenolic resin · Physical and mechanical properties · Bonding quality · Nail-withdrawal resistance

Introduction

Sugi (*Cryptomeria japonica* D. Don) is one of the most abundant planted wood species in Japan. Traditionally, solid sawn timber of Sugi and glue-laminated products made from Sugi were developed for both structural and non-structural use in conventional Japanese wooden houses and buildings. However, the Japanese Agricultural Standard (JAS) for large dimension of structural glue-laminated timber (glulam) was established in 1986, and the building code was revised in 1987. After these developments, large timber construction was permitted and the supply of glue-laminated products increased remarkably [1].

Recently, the development of new methods of utilizing glulam, such as for outdoor purposes, has received considerable attention. For such applications, the timber needs to be treated with preservatives to prevent biodeterioration occurring. Previously, CCA (chromated copper arsenate) preservatives were extensively used in all parts of the world, preventing the timber from biodeteriorating. However,

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environmental and health concerns about the release of arsenic from the timber led to the development of alternative copper compounds preservatives, such as alkaline copper quaternary (ACQ) and copper azoles (CuAZ). In general, the bonding quality of timber treated with preservatives is recognized to be lower than that of untreated timber. The lower bond quality of treated timber has been attributed to multiple causes, including chemical interference by the preservative with the adhesive cure; reduction in the timber wettability; and physical blockage of the surface, where the adhesive attaches to the timber [2].

On the other hand, it has been reported that phenolic resin treatment is effective in preventing biodeterioration, improving the mechanical properties, as well as the dimensional stability of the timber [3–6]. Although much of the research regarding wood impregnation with phenolic resin has been reported, most of the impregnation was conducted into a thin wood (veneer). Not much research has been conducted to investigate the effect of phenolic resin impregnation into wood with a thickness greater than veneer, such as lamina. Therefore, it is important to understand the effect of phenolic resin impregnation into wood with a thickness greater than veneer, as it was predicted that the use of phenolic resin treatment to improve the properties of timber would increase rapidly in the near future [7].

The objective of this study was to evaluate the performance of Sugi lamina impregnated with low-molecular weight phenolic (LMWP) resin. In this study, physical and mechanical properties, such as surface hardness, dimensional stability, bending and shear strength of LMWP-resin-treated and untreated lamina were investigated. In addition, bonding quality and nail-withdrawal resistance of 3-ply assembly specimen made from LMWP-resin-treated and untreated lamina bonded using resorcinol–phenol formaldehyde resin adhesive were also investigated. Moreover, because the manufacture of glulam involves applying adhesive to the prepared end-joint, and it is thought that this inhibits the penetration of preservatives because penetration would otherwise take place much further in the longitudinal direction of the lamina. Therefore, the effect on the penetration of LMWP resin in the finger-jointed lamina was also examined in this study.

Materials and methods

Materials

In total, 14 Sugi laminas with dimensions of 27 mm thick, 120 mm wide and 3000 mm long were prepared. LMWP resin with average molecular weight of 200 provided by Kyushu Mokuzai Kougyou Co., Ltd. (SP-400) was impregnated into the lamina using a wood treatment cylinder. A vacuum of 8 kPa was applied for 20 min followed by air pressure of 200–1200 kPa for 220 min at a constant temperature of 5 °C. The average retention measured immediately after impregnation was 412 kg/m^3. After impregnation, the LMWP-resin-treated lamina were air dried for 30 days and then cured at elevated temperature from 60 to 130 °C for 114 h using kiln drying. The experiment also included untreated lamina of the same species and size, used as controls.

Evaluation of LMWP resin penetration into finger-jointed lamina

Five Sugi finger-jointed laminas with dimensions of 27-mm thick, 120 mm wide and 3000 mm long were used. Each lamina had three finger-joint connections, located at the middle, and at 300 and 600 mm from the right and left end of the lamina, respectively. LMWP resin impregnation was conducted using the same method described before. After impregnation, each lamina was cut sequentially into pieces by cross-sectional cutting at 100 mm intervals in a longitudinal direction. To make the LMWP resin visible, ferric chloride solution was applied on one side of cross-section of each piece and the penetration area of LMWP resin into finger-jointed lamina was calculated using image analysis software.

Evaluation of physical and mechanical properties of lamina

The physical and mechanical properties of the lamina were evaluated using Methods of Test for Woods, Japanese Industrial Standard (JIS Z2101-1994) [8]. A total of 30 specimens with dimensions of 27 mm thick, 120 mm wide and 500 mm long obtained from LMWP-resin-treated and untreated lamina were prepared for a static bending test under dry conditions using the universal testing machine. Three-point bending was applied at a loading speed of 5 mm/min. In the bending test, span was set at 460, 82 mm longer than the standard. From load–deflection curve, modulus of rupture (MOR) and modulus of elasticity (MOE) were obtained. In addition, work to maximum load (*W*) of each specimen was also calculated from the load–deflection curve [9]. A total of 96 shear block specimens with a shear plane area of 25 × 25 mm obtained from LMWP-resin-treated and untreated lamina were prepared for a shear strength test parallel to the grain. A load was applied at a speed of 1 mm/min using the universal testing machine and the maximum load was recorded. Following the static bending test, the specimen was cross-sectionally cut to a length of 100 mm, to be tested for surface hardness. A total of 30 specimens obtained from the

LMWP-resin-treated and untreated lamina were prepared. A Brinell hardness test was conducted at 12 points on the tangential surface of each specimen using the universal testing machine at a loading speed of 1 mm/min. A steel ball with a diameter of 10 mm was dropped on to the specimen so as to indent it, and an applied load at an indenting depth of 1/π mm (about 0.32 mm) was recorded.

Vertical density profile measurement

Following the Brinell hardness test, LMWP-resin-treated and untreated specimen obtained from the same lamina was cross-sectionally cut to a length of 5 mm. Cross-section of the 5-mm-length-specimens and reference wood specimens with known density were scanned together using soft X-ray. X-ray image was then reconstructed and grey value of the 5-mm-length specimens and the reference wood specimens was measured using image analysis software. Using regression equation resulted from relationship between grey values of the reference wood specimens and their densities, the vertical density profiles of the 5-mm-length specimens were then calculated.

Evaluation of dimensional stability of lamina

Following the static bending test, the specimen was cross-sectionally cut to a length of 30 mm. A total of 90 specimens obtained from LMWP-resin-treated and untreated lamina were prepared. The changes in weight, thickness and width of each specimen were measured during humidity conditioning at 90 % relative humidity (RH) for 10 days followed by 40 % RH for 10 days at a temperature of 20 °C. Before the test, cross-sections of each specimen were sealed with epoxy resin and the specimen was conditioned at 20 °C and 65 % RH.

Evaluation of bonding quality

A total of 27 and 9 specimens from LMWP-resin-treated and untreated lamina, respectively, with dimensions of 27 mm thick, 120 mm wide and 500 mm long were prepared. All the specimens were planed to produce a smooth surface and uniform thickness. After they were planed, additional surface planing of the LMWP-resin-treated lamina to thicknesses of 0, 2 and 4 mm was conducted to investigate the effect of surface removal on the bonding quality of LMWP-resin-treated lamina. Finally, one group of untreated lamina specimens with a thickness of 26 mm and three groups of LMWP-resin-treated lamina specimens with thicknesses of 26, 22 and 18 mm were used for a bonding quality test. A spread rate of 300 g/m^2 was applied to one surface of the specimen. Three 3-ply assemblies made from each group of lamina specimen were bonded using resorcinol-phenol formaldehyde resin adhesive (Dianol D-40) provided by Oshika Shinko Co., Ltd. Viscosity, non-volatile content and specific gravity of the adhesive were 0.30 Pa s, 58.9 % and 1.14, respectively. The assemblies were then pressed in a cold press at 980 kPa for 24 h. The bonding quality test consisted of a shear bond strength test and a test for durability of the bonded interface. These tests were conducted according to the Japanese Agriculture Standard for Structural Glulam [10]. A total of 16 shear block specimens with a shear plane area of 25 × 25 mm were prepared from the assembly made from each group of lamina for the shear bond strength test. A load was applied at a speed of 1 mm/min using the universal testing machine, and the maximum load and wood failure percentage were recorded. The test for durability of the bonded interface was conducted using the delamination test. The 3-ply assembly was cross-sectionally cut to a length of 75 mm for the delamination test. The delamination test took the form of two types of test, which were a cyclic-boiling and a cyclic-dipping test. A total of eight specimens were prepared from the assembly made from each group of lamina for both cyclic-boiling and cyclic-dipping test. The method used for the cyclic-boiling test has two cycles. In each cycle, the specimen was immersed in boiling water for 5 h and then cooled in water at a temperature of 25 °C for 1 h. After that the specimen was dried at a temperature of 70 °C until its weight reached 100–110 % of its initial weight. The ratio of delamination to bonded interface length was then calculated. Like the cyclic-boiling test, the cyclic-dipping test also has two cycles. In each cycle, the specimen was immersed in water at a temperature of 25 °C for 24 h. After that the specimen was dried at a temperature of 70 °C until the weight of the specimen reached 100–110 % of its initial weight. The ratio of delamination to bonded interface length was then calculated.

Evaluation of nail-withdrawal resistance

The 3-ply assembly made from 26-mm-thick LMWP-resin-treated and untreated lamina specimen was cut to a length of 75 mm for the nail-withdrawal resistant test. The test was conducted under dry conditions, and after humidity conditioning at 90 % RH for 7 days followed by 40 % RH for 7 days at a temperature of 20 °C. A total of eight specimens made from LMWP-resin-treated and untreated lamina and nails measuring 65 mm in length and 3.05 mm in diameter were used for each test condition. On the tangential surface of each specimen six holes measuring 2 mm in diameter were drilled, to act as guide holes; a nail was then driven to a depth of 30 mm into each guide hole. The specimen was then placed in a jig on the universal testing machine that allowed for withdrawal of the nails from the specimen at a loading speed of 2 mm/min. The maximum load required for withdrawal of each nail was recorded.

Results and discussion

LMWP resin penetration into finger-jointed lamina

Figure 1 shows the average penetration area of LMWP resin into finger-jointed lamina. The average penetration area of LMWP resin was 100 % at both ends of the lamina, and this decreased gradually as toward the middle of the lamina. However, the overall average penetration area of LMWP resin into finger-jointed lamina was very high, which was above 90 %. Therefore, the effect of the finger-joint on penetration of the LMWP resin was not found to be significant. It is thought that sufficient penetration of the LMWP resin occurred not only in a longitudinal direction, but also in transverse directions on the lamina.

Physical and mechanical properties

The physical and mechanical properties of LMWP-resin-treated and untreated lamina are summarized in Table 1. A slight increase in the density of LMWP-resin-treated lamina was observed in comparison with the untreated lamina. This result is considered good because there was only a small increase in weight. In addition, the LMWP-resin-treated lamina has a lower MOR compared with the untreated lamina. This is probably because the LMWP-resin-treated lamina was exposed to a high temperature during curing. It is well known that MOR is more sensitive to heat, and many studies have reported that treatment in a high temperature decreased the MOR of the timber [11, 12]. On the other hand, it was found that the MOE of LMWP-resin-treated lamina was slightly larger than that of the untreated lamina. However, no significant difference was found in either the MOR and or the MOE between LMWP-resin-treated and untreated lamina. It was also found that the work to maximum load of LMWP-resin-treated lamina was significantly lower than that of the untreated lamina. From these results, it seems that the LMWP-resin-treated lamina became more brittle than the untreated lamina. As shown in Table 1, the shear strength of the LMWP-resin-treated lamina increased significantly in comparison with the untreated lamina. The increase in shear strength was the result of resin penetrating into the cell wall followed by bulking of the cell structure [5, 13]. Bulking of the cell structure resulted in an increase in cooperation between the wood fibers, thus ensuring a resistance to the force that tends to cause one section to slip over another along the grain. In addition, the Brinell hardness of the LMWP-resin-treated lamina was significantly increased compared with that of the untreated lamina. The improvement in hardness was caused by an increase in density, especially the density on the surface part of the lamina. Figure 2 shows vertical density profile of LMWP-resin-treated and untreated lamina. From Fig. 2, the density of both early and late wood of LMWP-resin-treated lamina was found to be higher than that of untreated lamina. Moreover, the increase in early wood density on the surface part of LMWP-resin-treated lamina was found relatively higher than that on the inner part. This is because impregnation using the full cell process makes resin penetration and bulking of the cell structure to take place mainly on the surface part of the lamina.

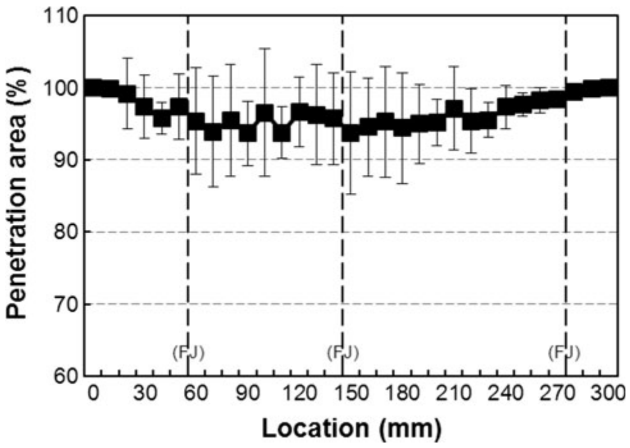

Fig. 1 Average penetration area of LMWP resin. *FJ* finger-joint position. *Error bar* standard deviation

Table 1 Average and standard deviation of physical and mechanical properties of LMWP-resin-treated and untreated lamina

Properties	Lamina type			
	n	Untreated	*n*	LMWP-resin-treated
Density (kg/m^3)	15	393 (36)	15	454 (30)
MOR (MPa)	15	53.29 (8.25)	15	49.71 (5.99)
MOE (GPa)	15	6.25 (1.68)	15	6.45 (1.62)
W (J)[b]	15	7.49 (2.22)	15	4.26 (1.06)
Shear strength (MPa)[a]	48	9.29 (1.36)	48	10.09 (1.77)
Brinell hardness (MPa)[b]	180	9.26 (4.70)	180	16.49 (7.27)

Numbers in parentheses indicate standard deviations

n number of samples, *MOR* modulus of rupture, *MOE* modulus of elasticity, *W* work to maximum load

[a] Significant different between untreated and LMWP-resin-treated lamina at 5 % level

[b] Significant different between untreated and LMWP-resin-treated lamina at 1 % level

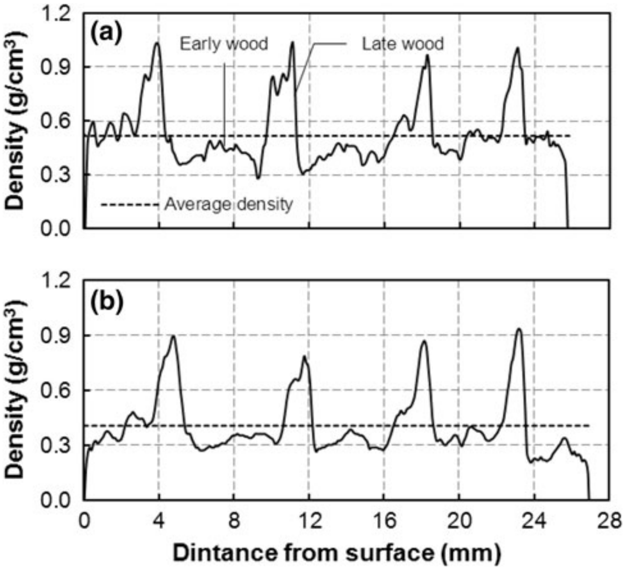

Fig. 2 Typical vertical density profile of **a** LMWP-resin-treated and **b** untreated lamina

Dimensional stability

Figure 3 shows the hygroscopic change for both LMWP-resin-treated and untreated lamina. It was found that LMWP-resin-treated lamina absorbed and desorbed less moisture than the untreated lamina. As expected, LMWP-resin-treated lamina also showed more reduction in swelling and shrinkage in both thickness and width direction. The bulking of the cell structure and the interaction of the LMWP resin with the wood through its hydroxyl groups might be responsible for decreasing the hygroscopicity of the LMWP-resin-treated lamina. The average anti-swelling efficiency (ASE) of LMWP resin-treated lamina in thickness and width direction was 55.4 and 26.4 %, respectively.

Bonding quality

The shear bond strength and wood failure percentage of the assembly made from each group of lamina is shown in Fig. 4. The results showed that significant difference in the shear bond strength was found between assemblies made from LMWP-resin-treated and untreated lamina with the thickness of 26 mm. The shear bond strength of the assembly made from the LMWP-resin-treated lamina was significantly larger than that of the assembly made from untreated lamina. As described before, LMWP resin mainly penetrated into the cell wall and then bulked the cell structure, resulting in an increase in the shear strength of LMWP-resin-treated lamina. In addition, the adhesive could flow deeply to fill the cell lumens of the LMWP-resin-treated lamina, as shown in Fig. 5. Therefore, it can be concluded that the improvement in shear strength and the ability of the adhesive to penetrate into the cell lumens lead to an increase in the shear bond strength of the assembly made from LMWP-resin-treated lamina. Moreover, Fig. 4 also shows the shear bond strength was found to decrease as the thickness of the surface removal of LMWP-resin-treated lamina increased. From the results described before, it was found that the density on the surface part of the LMWP-resin-treated lamina was higher than that on the inner part. As the thickness of the surface removal increased, the surface density of the LMWP-resin-treated lamina would gradually decrease. We suggested

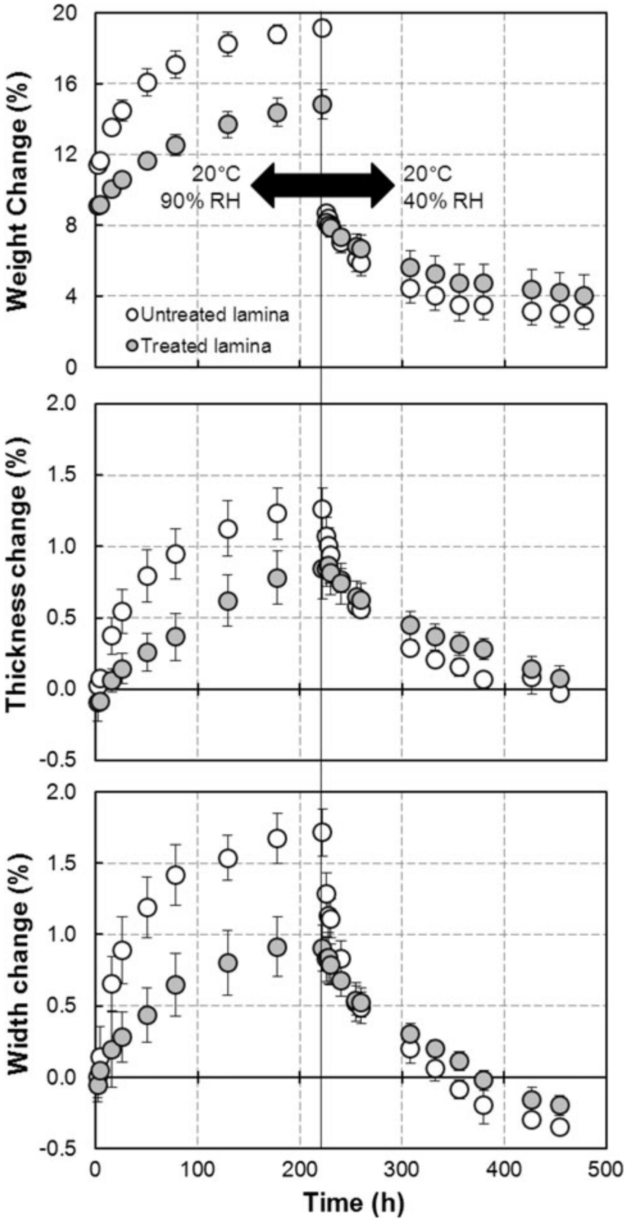

Fig. 3 Hygroscopic change of LMWP-resin-treated and untreated lamina. *Error bar* standard deviation

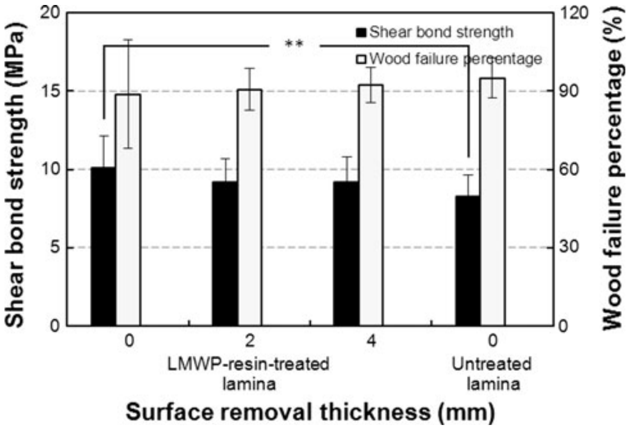

Fig. 4 Average shear bond strength and wood failure percentage of the assembly made from each group of lamina. *Error bar* standard deviation. *Double asterisk* significant different at 1% level

that a decrease in surface density caused a reduction in shear strength of the lamina, which in turn would decrease the shear bond strength of the assembly. However, the effect of surface removal on the shear bond strength of the assembly made from LMWP-resin-treated lamina was not significant. In addition, the results also showed that wood failure percentage between assemblies made from LMWP-resin-treated and untreated lamina was not significant. On the other hand, Fig. 6 shows the average delamination percentage during the cyclic-boiling and cyclic-dipping tests carried out on the assembly made from each group of lamina. In both cyclic-boiling and cyclic-dipping test, the delamination percentage of the assembly made from LMWP-resin-treated lamina has tendency to have lower value than that of the assembly made from untreated lamina. This is probably because LMWP-resin-treated lamina has a better dimensional stability than the untreated lamina. When moisture fluctuates, an assembly with poor dimensional stability would induce larger stress in each bonded interface. This condition would cause delamination when bonding between lamina could not resist the applied stress.

Nail-withdrawal resistance

Figure 7 shows the average nail-withdrawal resistance of the assembly made from LMWP-resin-treated and untreated lamina under dry conditions and after humidity conditioning test. In the dry conditions test, the nail-withdrawal resistance generally increased as the density of the lamina increased. However, the results show that the difference in the withdrawal resistance between assemblies made from LMWP-resin-treated and those made from untreated lamina was not significant. The reason for this may be attributed to the vertical density profile of LMWP-resin-treated lamina, which has a higher density on the surface part and a lower density on the inner part of the lamina. In case of after humidity conditioning test, the

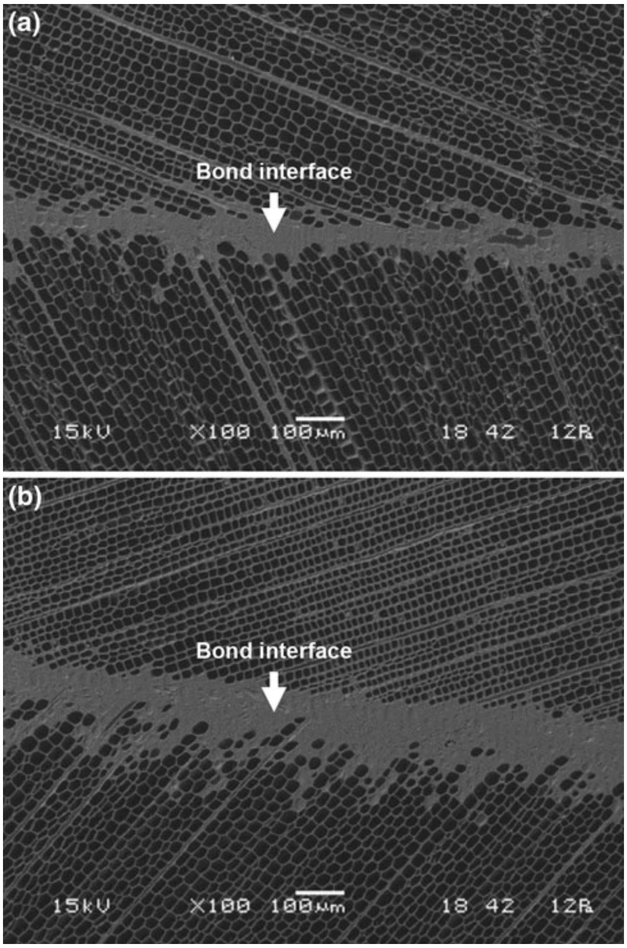

Fig. 5 SEM micrograph of bonded interface of the assembly made from LMWP-resin-treated (**a**) and untreated lamina (**b**)

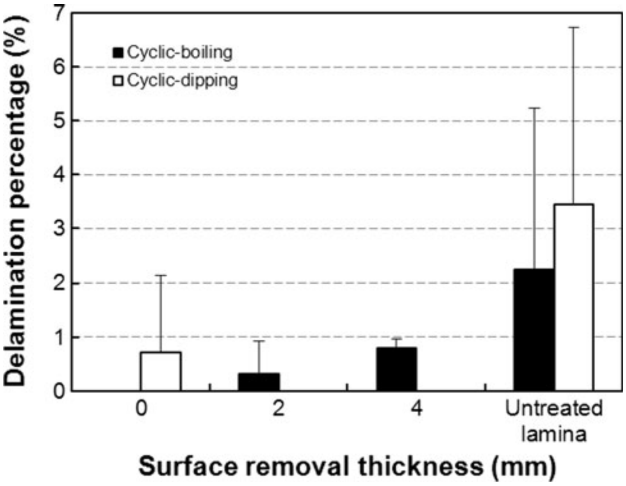

Fig. 6 Average delamination percentage of the assembly made from each group of lamina. *Error bar* standard deviation

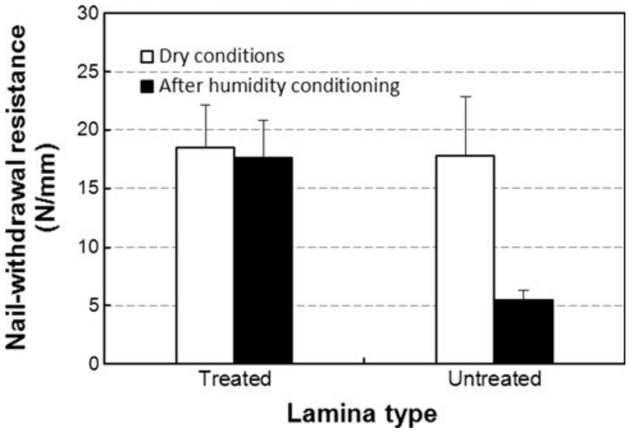

Fig. 7 Average nail-withdrawal resistance of the assembly made from LMWP-resin-treated and untreated lamina. *Error bar* standard deviation

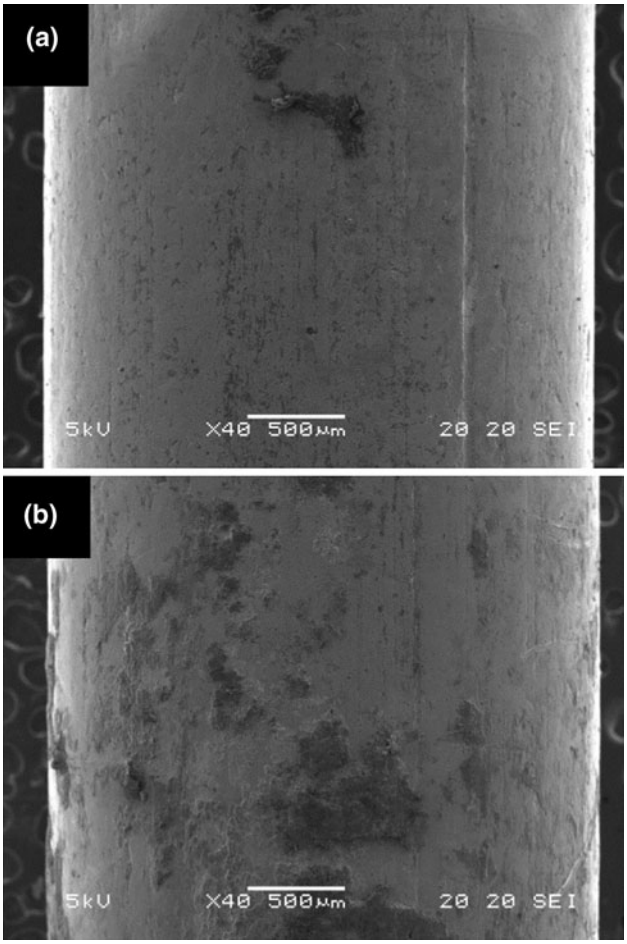

Fig. 8 SEM micrograph of surface conditions of nail shank before (**a**) and after being driven (**b**) into the assembly made from LMWP-resin-treated lamina

withdrawal resistance of the assembly made from LMWP-resin-treated lamina decreased slightly when compared with the dry conditions test. However, a significant difference between the dry conditions test and after humidity conditioning test was observed in the assembly made from untreated lamina. Generally, nails resist withdrawal forces through the frictional forces between the wood fibers and the nail shank. Frictional forces are greatest just after the nail is driven in, but eventually the wood fibers surrounding the nail relax, causing the withdrawal strength to decrease. The relaxation of the wood fibers may be compounded if the timber dries and shrinks over time as a result of changing moisture conditions. Therefore, the withdrawal resistance of the assembly made from untreated lamina decreased drastically after humidity conditioning at temperature of 20 °C. On the other hand, in the assembly made from LMWP-resin-treated lamina, after humidity conditioning, corrosion occurred in the nail driven into the assembly, as shown in Fig. 8. Although the relaxation of the wood fibers led to a decrease in the withdrawal resistance, excellent dimensional stability and frictional force between the wood fibers and the rusted nail shank made the withdrawal resistance decrease slightly.

Conclusions

This study was conducted to evaluate the performance of Sugi lamina impregnated with LMWP resin using the full cell process followed by curing at high temperature. In this study, penetration of the LMWP resin into finger-jointed lamina was examined. Physical and mechanical properties, such as surface hardness, dimensional stability, bending and shear strength of LMWP-resin-treated and untreated lamina were investigated. In addition, bonding quality and nail-withdrawal resistance of 3-ply assembly specimen made from LMWP-resin-treated and untreated lamina bonded using resorcinol–phenol formaldehyde resin adhesive were also investigated. The main results were as follows:

1. LMWP resin penetrated sufficiently into finger-jointed lamina.
2. The physical properties of LMWP-resin-treated lamina were found to have improved significantly compared with untreated lamina. However, no significant difference was found between LMWP-resin-treated and untreated lamina in terms of their mechanical properties.
3. There was an improvement in bonding quality for the assembly made from LMWP-resin-treated lamina compared with that made from untreated lamina.
4. A significant decrease in nail-withdrawal resistance between dry conditions test and after humidity conditioning test was observed in the assembly made from untreated lamina. However, the same tendency was not found in the assembly made from LMWP-resin-treated lamina.

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