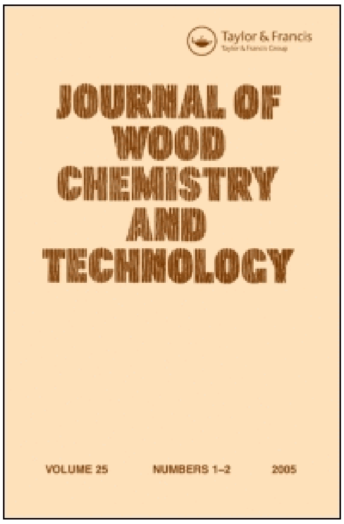

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Development of Fire Resistant Laminated Wood Using Concentrated Sodium Polyborate Aqueous Solution

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Abstract: A highly concentrated aqueous solution of sodium polyborate (SPB) was used to treat laminated Japan cedar (*Cryptomeria japonica*) in order to first develop a fire resistant laminated wood. After the pressure-impregnation of a water borne solution containing 9.78 mol/L of boron, the fire properties were evaluated by cone calorimeter (CCM) and weight loss (WL). The resorcinol formaldehyde resin (RF) laminated wood did not fracture and the adhesive interfaces kept the pieces together even after heating at 750°C for 20 min or with the CCM test using the heat flux of 50 kW/m^2 for 20 min. Heat emission for 20 min in the CCM test below 8 MJ/m^2 was achieved by a WPG of 53% or more.

Keywords: Borate, cedar, cone calorimeter, fire resistant, laminated wood, pressure impregnation

INTRODUCTION

Boric acid and borates have long been used to make wood more fire resistant.[1–3] However, the solubilities of the conventional boric acid and borates at room temperature are insufficient for meeting fire standards (e.g., boric acid (H_3BO_3), 3.99; borax ($Na_2B_4O_5(OH)_4{\cdot}8H_2O$), 2.58 at 20°C). A highly

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concentrated borate solution is desirable to give sufficient fire resistant properties to wood.

We have successfully prepared a highly concentrated aqueous solution of sodium polyborate with 5.24 mol/kg of boron at 20°C by adjusting the Na/B ratio to 0.22.[4] This is markedly more concentrated than the most soluble borate solution ($Na_2O{\cdot}B_2O_3{\cdot}8H_2O$, 2.49 mol/kg at 20°C).[5] Evaporation of the solution at 5.24 mol/kg yielded amorphous solid foam with a density of 0.1–0.3 g/cm^3. This highly concentrated polyborate solution has been applied to a fire resistant treatment of wood specimens of Japan cedar (*Cryptomeria japonica*), and the specimens pressure-impregnated with the solution (14 mol/L as to boron) at 150°C (474 kPa) for 1 h had a weight percent gain of 124% by uptake of polyborate with a weight loss of 38% when heated at 750°C for 20 min in air.[6]

In the present study, we applied this concentrated polyborate solution to laminated wood of Japan cedar (*Cryptomeria japonica*). In recent years, laminated wood produced by gluing lumber boards is widely used for both fixture and structural materials of buildings such as schools, houses, and domes. It is highly desirable to develop fire resistant laminated wood from the viewpoint of disaster prevention. Compared to solid wood, possible poor thermal stability of the adhesive interfaces was crucial to the fire resistance. We solved this problem by selecting a glue and prepared laminated wood that does not fracture by heating in air at 750°C, for 20 min. We present here findings in fire resistant treatment of laminated wood using the polyborate solution and heat release tests using the cone calorimeter.

EXPERIMENTAL SECTION

Materials

Specimens of both solid and laminated wood of Japan cedar (*Cryptomeria japonica*) were pressure-impregnated to compare permeability. The size was 100 (R) mm × 100 (L) mm × 10–50 mm, and their butt ends were arranged at the side of the specimens. All the specimens were dried at 105°C for more than 24 h before pressure-impregnation. This drying process did not affect the adhesives. The adhesive interfaces of the laminated wood were located every 30 mm perpendicularly to the surface and the butt end. The wide surface of the laminated wood was quartersawn. To laminate the wood, two kinds of adhesives, aqueous vinyl polymer-isocyanate resin (API) and resorcinol-formaldehyde resin (RF), were investigated.

Sodium polyborate (SPB) was prepared according to our previous article[4] and used for fire resistant agent of wood. The pH of the SPB solution (6.68 mol/L as to B) was 6.86 at 20°C. In the present study, 150 kg of amorphous SPB (Na/B ratio 0.22) was dissolved into 100 kg of water at 60°C. The boron concentration of the obtained SPB aqueous solution was 9.78 mol/ L, and the density of the solution was 1.25 g/cm^3. The SPB solution at 9.78 mol/L was used in all the experiments in the present study, though the boron concentration can be raised to 14 mol/L. This is because our previous study indicated 9.78 mol/L was sufficient to satisfy the fire resistant properties.

Pressure-Impregnation Treatment

The specimens were pressure-impregnated at 1 MPa, 60°C for 1 h using an autoclave. The pressure was directly increased from ambient pressure without an initial vacuum. The weight percent gain (WPG, %) was calculated from the mass increase of the specimen after drying at 105°C for 24 h. Most of the laminated wood boards were accumulated using spacers and sunk in the solution in the autoclave. Some specimens were accumulated without spacers and one specimen was floated in the solution to investigate how the arrangement of specimens in the solution affected the WPGs and to establish techniques to control WPGs using one standard concentration (9.78 mol/L). Bending and compressive strengths after pressure-impregnation have been found to be almost the same as those before the impregnation, suggesting that the polyborate has no or only a minor effect on the strength properties.

Heat Release Test

The cone calorimeter (CCM) test was performed to evaluate the fire resistance properties (Fire Testing Technology Ltd). The CCM monitors heat release from a specimen with surface area of 99 mm × 99 mm when it is irradiated with infrared in air at a heat flux of 50 kW/m^2. The amount of heat release from the specimen is calculated from O_2 consumption in the exhaust gas and recorded for 20 min. A total amount of heat release below 8 MJ/m^2 at a heat flux of 50 kW/m^2 for 20 min is the necessary criterion for “non-combustible materials” by the Japanese Building Standards.[7] Weight loss (WL, %) in the CCM test at 20 min was also recorded.

RESULTS AND DISCUSSION

Choice of Adhesives

In order to investigate heat resistance preliminarily, the pressure-impregnated laminated wood (WPG, 50%) was heated in an electric furnace at 750°C for 20 min. The laminated wood glued with API adhesive was found to separate from all its adhesive interfaces and to break into its constituent pieces, although the pieces were only carbonized and maintained their shapes. In contrast, the laminated wood with RF adhesive did not break, and the adhesive interfaces kept gluing the carbonized pieces together even after heating at 750°C for 20 min. The RF adhesives bore at 750°C for 20 min, although the ignition point of the RF resin is 607°C. This is probably because the temperature inside wood was lower than the wood surface due to the heat-insulating effect of the fire resistant wood.

Figure 1 shows SEM images of the butt-end surface including the cross-section of the adhesive interface after: (a) impregnation and before heating and (b) heating at 750°C, for 20 min. The adhesive interfaces kept solid after impregnation (WPG, 50%), that is, the impregnation did not bring about the destruction of adhesive interfaces (Figure 1(a)). After heating at 750°C, the adhesive interfaces still glued the carbonized pieces despite their small partial transformation as much as 0.1 mm (Figure 1(b)). Laminated wood that did not separate at 750°C in air was first achieved for our developed SPB-treated composite with a RF adhesive. Thus, experiments later were performed using the RF adhesive.

Permeability of Laminated Wood and Solid Wood

The WPGs of the laminated wood and the solid wood were plotted as a function of thickness in Figure 2. The laminated wood showed higher WPGs than the solid wood, although the impregnation condition was the same. This is probably because the wide surface of the laminated wood was quartersawn and quartersawn lumber has better permeability than flatsawn lumber.

The standard deviations ($n = 12$) of the WPGs of the laminated wood were smaller than that of the solid wood, likely because the wood parts comprising the laminated wood reduced the density variation. The dispersion of the WPGs is one of the difficulties in quality control of fire resistant wood, so quality control in fire resistance of laminated wood is easier than solid wood. In the present laminated wood study, the adhesive interfaces are arranged every ca. 30 mm and the specimens consist of 3 or 4 parts. The standard deviations of the laminated wood are statistically predicted to be 0.50–0.58 times of the solid wood, which was similar to that obtained for the specimens thicker than 25 mm.

Impregnation and CCM Tests

Heat release (MJ/m^2) for 10 and 20 min CCM tests are summarized in Table 1 along with the thickness of specimen (mm), dry density (g/cm^3), WPG (%), and WL (%). Heat releases for specimens with WPGs over ca. 50% were consistent,

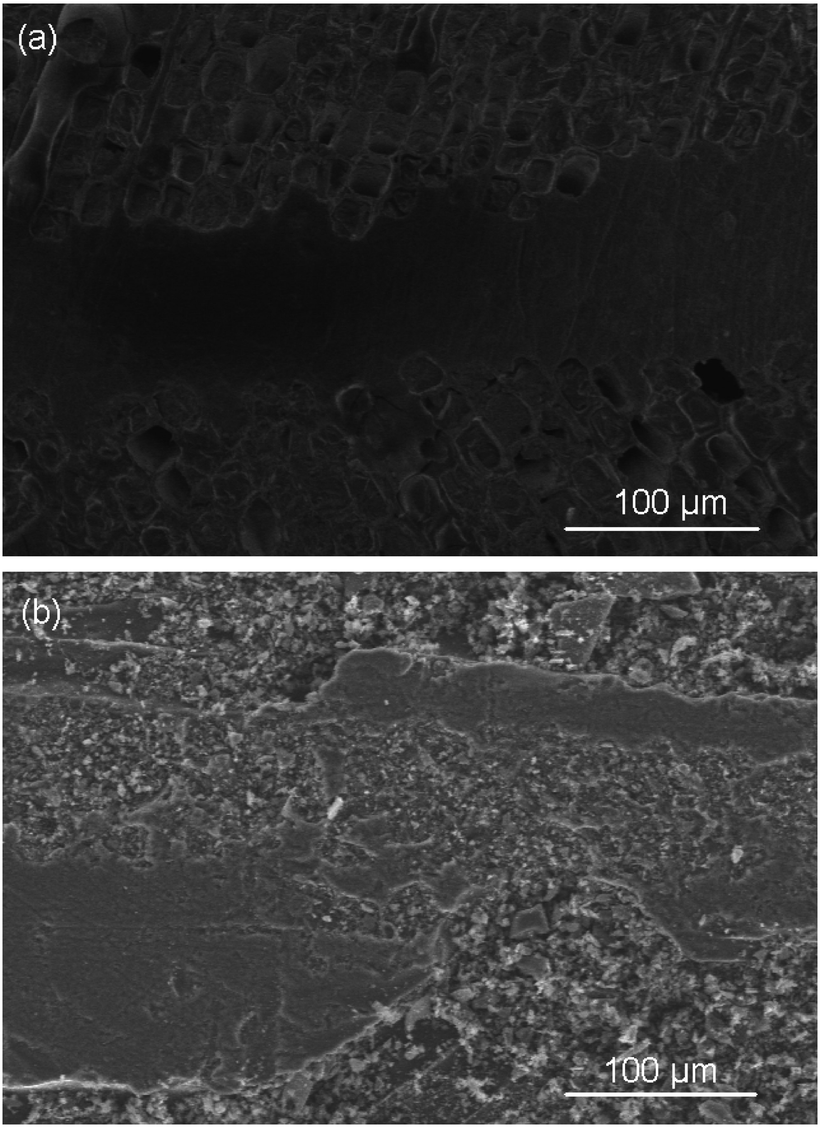

Figure 1. SEM images of the butt-end surfaces: (a) after impregnation and before heating, and (b) after heating at 750°C, for 20 min. The observed surfaces are perpendicular to the adhesive interfaces, and the cross-sections of the adhesive interface are observed as plain thick lines traversing the images.

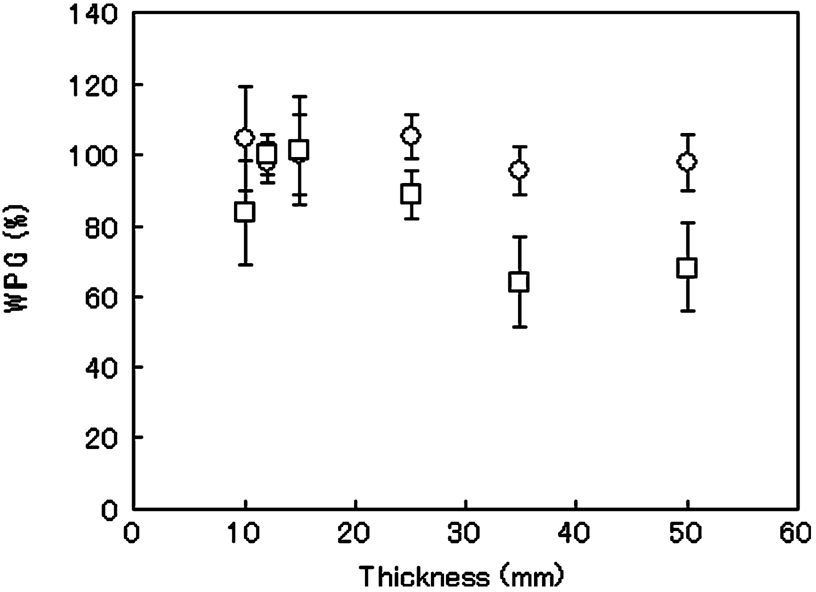

Figure 2. Comparison of permeability of laminated wood ○, and solid wood □, with different thickness. The error bars denote standard deviation (n = 12).

Table 1. Heat release (MJ/m^2) for 10 and 20 min in the CCM tests along with the thickness (mm^2), dry density (g/cm^3), WPG (%), and WL (%) of the specimens. Specimens No. 2–4 and 8 were accumulated without spacers, and Specimen No. 6 was floated in the solution during the pressure-impregnation

Specimen no.	Thickness (mm)	Dry density (g/cm^3)	WPG (%)	Heat emission (MJ/m^2) 10 min	Heat emission (MJ/m^2) 20 min	WL (%)
1	9.5	0.32	48.5	5.4	12.0	60.4
2	10.7	0.31	38.2	0.4	8.4	61.2
3	12.1	0.32	35.6	18.0	28.4	63.6
4	12.2	0.35	39.5	11.6	26.6	61.5
5	12.9	0.34	44.8	1.2	13.0	58.0
6	15.8	0.38	21.1	10.0	29.2	63.2
7	15.8	0.33	53.0	0.9	1.9	45.4
8	16.0	0.38	43.7	0.5	1.0	43.2
9	16.0	0.35	54.0	0.0	0.9	42.2
10	25.1	0.35	55.5	0.8	2.2	24.0
11	35.4	0.35	53.1	1.2	2.0	13.2
12	51.0	0.35	48.0	0.2	0.3	0.0

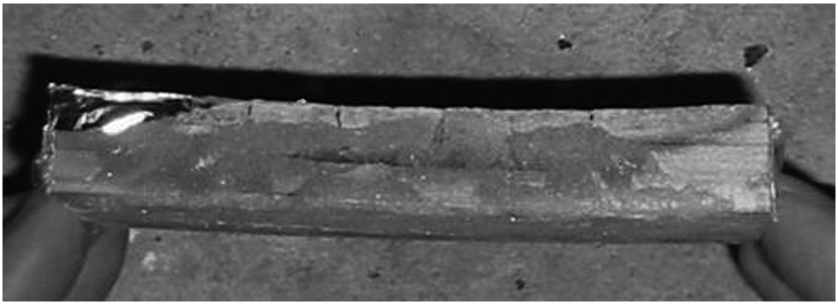

Figure 3. Specimen of fire resistant laminated wood with thickness of 16 mm after the CCM test. The bottom half remained unchanged, although the upper half was carbonized.

but for specimens where the WPGs were intentionally decreased were more variable. All the specimens with thickness over 15.8 mm and WPG over 43.7% sufficiently satisfied the criterion for "the non-combustible materials" of the Japanese Building Standards, below 8 MJ/m^2 for 20 min.

Table 1 shows the relationship between the heat release (MJ/m^2) in CCM tests and WPG (%). The heat release decreased with increasing WPG. The correlation coefficients using only specimens with thickness over 15.8 mm were $R = 0.926$ for 20 min and $R = 0.924$ for 10 min. The specimens with thicknesses of 9.5 and 12.9 mm showed a heat release of 12.0 and 13.0 MJ/m^2, although the WPGs were 48.5 and 44.8%. This indicates that the thickness of the board is an important factor in heat release.

The heat release of the specimens in the WPG range of 35.6–55.5% decreased at greater WPGs at thickness of ca. 15 mm or greater. Figure 3 shows the side view of the specimen with 16 mm thickness after CCM test. Although the upper half was carbonized, the bottom half remained unchanged. However, the specimen with 9.5 mm thickness, a little thicker than the carbonized depth, did not satisfy the criterion. Our results suggest that the unchanged bottom half is essential for the suppression of the heat release. The unchanged bottom half may suppress the development of cracks in the carbonized layer and, subsequently, contributes to the protection of the inside from the heat flux. In a word, we developed the technique to satisfy the criterion below 8.0 MJ/m^2 for 20 min by using the board with thickness over 15.8 mm and by pressure-impregnating up to the WPG 44%.

In Table 1, the relationship between the heat release (MJ/m^2) and the WL (weight loss, %) in the CCM test showed a good correlation. Thus, measurement of the WL at a heat flux of 50 kW/m^2 can be a useful preliminary screening test before measuring heat release, as an expensive apparatus is needed for the heat release measurement in the CCM test.

Fire Resistance Mechanism

As we have reported previously,[6] the fire resistance effect of SPB is due to the interception of both oxygen gas and heat conduction by the generation of SPB foam. The SPB impregnated in wood transforms into glass foam barrier upon heating, which enfolds the wood and blocks the contact with oxygen gas. This brings about the carbonization of wood, and the composite of the glass foam and the carbonized wood blocks heat conduction.

CONCLUSIONS

Fire resistant laminated wood of Japan cedar (*Cryptomeria japonica*) satisfying the criterion that the total amount of heat release at a heat flux of 50 kW/m^2 for 20 min is below 8 MJ/m^2 was successfully prepared through a process of pressure-impregnation for 1 h using our developed SPB aqueous solution (9.78 mol/L) and the RF adhesive. The heated specimens were carbonized, but the adhesive interfaces glued the carbonized pieces together even after heating at 750°C for 20 min in an electric furnace or after the CCM test using the 50 kW/m^2 heat flux for 20 min. In the present study, the criterion was achieved when the thickness and the WPG were over 15.8 mm and 43.7%, respectively. There remains a possibility that the specimens thinner than 15.8 mm satisfy the criterion by increasing the WPG, because the saturated amount of the WPG with regard to the Japan cedar was ca. 150%. The fire resistant laminated wood developed in the present study will be useful for fixture and structural materials in buildings where fire prevention is absolutely essential such as high-rise buildings, stations, theaters, museums, and domes. Fire damage with regard to wood materials is expected to be diminished by using the present fire resistance technique. Furthermore, our results suggest that the SPB has a fire resistance effect on the RF resin, and it is also expected that the SPB will be useful for fire retardance of polymers because it does not contain heavy metals or phosphorus.

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