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Evaluation of a Boron-Nitrogen, Phosphate-Free Fire-Retardant Treatment. Part I. Testing of Douglas-Fir Plywood per ASTM Standard D 5516-96*

ABSTRACT: The objective of this work was to evaluate (a) the effects of a new boron–nitrogen, phosphate-free fire-retardant (FR) formulation on the initial strength of Douglas-fir AB-grade plywood and (b) the potential of this FR treatment to experience subsequent thermal degradation In-service when exposed to elevated temperatures. Test Method ASTM D 5516 was generally followed The results of our analysis indicated that treatment and post-treatment redrying with the new boron-nitrogen, phosphate-free FR had a significant negative effect of about 7% on bending strength and a significant negative effect of 26% on energy-related properties, such as work to maximum load. The properties of modulus of elasticity and maximum moment carrying capacity were not significantly reduced. Our results indicate that the likelihood of FR-treated plywood to experience in-service reduction in mechanical properties when exposed to elevated temperatures is no different than that of matched untreated plywood. As a result of our analysis, we recommend a revision to the cutting procedure specified by D 55 16-96 We recommend that users first cut the specimens, then inspect and cull specimens with defects, and finally allocate specimens to experimental groups prior to FR treatment and redrying We also recommend the substitution of commercially available grades of higher quality plywood such as AA or AB, for the custom-made N-grade plywood currently recommended by D 5516-96.

KEYWORDS: plywood, roof sheathing. thermal degradation. fire-retardant treatment

Wood is an environmentally desirable material for fiber and structural use. It is efficient in both economic and environmental costs to the user. To extend its utility into new markets, wood is sometimes treated with chemicals. In North America, fire-retardant (FR)-treated plywood is sometimes permitted as an alternative to noncombustible materials in structures where increased fire safety is required [1]. However, in the middle to late 1980s, some commercial FR treatments failed to perform as expected when used as roof-sheathing plywood and roof truss lumber [1–4]. Elevated roof temperatures caused by solar radiation in combination with FR chemicals and moisture prematurely caused some FRs to initiate hydrolytic reactions that often caused the plywood to darken, become brittle, experience cross-grain checking. and crumble easily. This problem often required costly roof replacement [1].

Extensive research over the past decade has defined the mechanism of the problem. In an intensive ten-year research program, methodologies were developed to determine the current condition of FR-treated plywood roof sheathing arid to predict its residual serviceability [1]. The results of that program clearly showed that today's generation of phosphate-based FR systems without pH buffers had problem [1]. Some inorganic phosphate-based formulations have experienced significant thermal degradation [2–4]. Borate-based phosphate-free systems seemed to be less likely to experience in-service related thermal degradation [3–5]. Inorganic or organic phosphate systems using borate buffers were intermediary in their response [3–5].

To assess the effects of FR treatment on mechanical properties and the potential of various FRs to undergo thermal degradation on extended exposure to elevated temperatures, two new ASTM Standard Test Methods, D 5516 [6] and D 5664 [7] were developed. A standard practice for deriving adjustment factors for FR-treated plywood was also developed, ASTM D 6305 [8]. Only a few minor changes have been made to these ASTM standards since they were adopted. These new ASTM test methods and design practices have been incorporated in mandatory performance requirements for AWPA Standard P17 for FR formulations [9] and for Standards C20 and C27 for FR-treated lumber and plywood, respectively [10, 11], to preclude future serviceability problems when FR-treated wood is used in engineered wood systems.

We know of no published reports of performing the ASTM Standard D 5516-96 Test Method in the version approved. The D 5516-96 standard evolved after several iterations from nonstandardized test methods originally and arbitrarily developed by code bodies or industrial associations as building product evaluation criteria [2]. Although the eventual ASTM standard was similar to the original evaluation methods used to qualify current commercially available FRs, this standard is certainly not identical to the original evaluation methods.

This is the first report of a three-part evaluation of the effects of a new boron-nitrogen, phosphate-free FR treatment on wood strength and its potential to experience strength loss when exposed

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to high temperature. Associated work reported on the testing and evaluation of small clear specimens of Douglas-fir, southern pine, and white spruce as specified in Methods A and B of ASTM Standard D 5664-95 [12] and nominal 2 by 4 (standard 38- by 89-mm) southern pine lumber as specified in Method C of ASTM D 5664-95 [13]. This report presents work on Douglas-fir plywood as outlined in ASTM D 5516-96 [6].

Objectives

The primary objective of this study was to evaluate the effects of treatment with a new boron-nitrogen, phosphate-free FR formulation on the initial strength of Douglas-fir plywood and its potential for thermal degradation in service when exposed to elevated temperatures according to ASTM D 5516-96, Standard Test Method for Evaluating the Flexural Properties of Fire-Retardant Treated Softwood Plywood Exposed to Elevated Temperatures.

Secondarily, the data and experience gained through this work may be used to improve the experimental design, precision, accuracy, and economy of ASTM Standard D 5516. Also, before this project was undertaken, no phosphate-free FR formulation existed in the commercial market. The research on this project was thought to provide direct benefits to consumers with respect to safety and long-term serviceability once the new phosphate-free FR was eventually accepted by model building codes and if standardized through the American Wood Preservers' Association (AWPA).

Experimental Design

The experimental design included two treatments (FR-treated and untreated) and six temperature exposure durations. They included no exposure and exposure to five progressively longer environmental exposure times (14, 32, 50, 68, or 86 days) at 170°F (71°C) and 67% RH. Prior fire testing had established the minimum chemical retention level required for meeting the flame-spread and flame-progression limits set forth in model building codes and in AWPA Standard C27 [10]. A four-step kiln temperature and humidity schedule was developed to provide a post-treatment kiln-drying schedule applicable to relatively small volumes of plywood in experimental-sized kilns and within the temperature limitations of AWPA Standard C27 (Table 1).

Method and Materials

We obtained ten sheets of AB-grade, 0.5-in. (12-mm)-thick, five-ply Douglas-fir plywood from a commercial manufacturer in western Oregon. All plies were Grade B or better Douglas-fir veneer. The N-grade plywood recommended by ASTM D 5516 was not used because no commercial plywood manufacturer could be found to provide custom lay-up of an all-N-grade plywood product. Four 2- by 4-ft (0.6- by 1.2-m) sections were cut from each sheet of AB-grade plywood for a total of 40 sections. Two 2- by 4-ft (0.6-by 1.2-m) sections from each sheet were assigned as untreated specimens, and the remaining two sections were assigned as FR-treated specimens. A 2-in. (50-mm)-wide edge was then removed from the 24-in. (600-mm) edge of each section; 14 individual 0.5-by 3- by 24-in. (12- by 75- by 600-mm) experimental specimens were ripped from each section. Each specimen was numbered to ensure that its original panel and section location were traceable. This specimen assignment technique allowed us to examine visually each individual 0.5- by 3- by 24-in. (12- by 75- by 600-m) specimen for defects (recall that AB-grade rather than all-N-grade plywood was used) prior to its assignment to an experimental group. This deviation in specimen processing and group assignment ensured greater between-group matching and also ensured virtually defect-free plywood specimens, as intended for use in the ASTM D 55 16 standard.

One defect-free 0.5- by 3- by 24-in. (12- by 75- by 600-mm) experimental specimen from each 2- by 4-ft (0.6- by 1.2-m) section was randomly allotted to one of the twelve experimental groups (two treatments by six exposures). This allotment method evenly distributed the within-sheet and between-sheet variability in the original plywood and allowed for a blocked analysis of the data that could account for within- and between-sheet variability in the original plywood.

Specimens assigned to groups requiring FR treatment were treated using a pressure-treating process including a final vacuum.

The FR-treating formulation[3] was supplied by Osmose, Inc.[4] (Buffalo, NY). Prior fire testing had established the minimum chemical retention levels to be used in these experiments for each species to meet the required flame spread and flame progression limits set forth in national building codes and in AWPA Standard C27 [11].

Experimental treatment concentrations were established on the basis of results of preliminary treatment. Six groups of 20 specimens were treated at one time. An initial vacuum of just over 25 in. Hg (85 kPa) was held for 45 min, after which a 9% solution concentration of FR in water was introduced into a 1.5-ft (0.46-m)-diameter, 6-ft (1.8-m) long treating cylinder. Immediately afterwards, 150 lb/in.^{2} (1.03 MPa) of pressure was held for 90 min. The treating solution was drained off at the end of the pressure period, and a final vacuum of just over 25 in. Hg (85 kPa) was held for 10 min. Each specimen was weighed before and after treatment to de-

[3] Fire-retardant formulation used as described in U.S. Patent No. 6,306,317; it has recently been introduced in U.S. Markets as "FirePRO™".

[4] The use of trade or firm names in this publication is for reader information and does not imply endorsement by the U.S. Department of Agriculture of any product or service. Osmose, Inc. is not associated with the Federal government. The information given in this section should not be construed as an endorsement or approval of the chemical or processes reported.

TABLE 1—*Schedule for kiln drying after treatment of FR-treated specimens.*

Drying Time, h	Dry-Bulb Temp., °F (°C)	Wet-Bulb Temp., °F (°C)	Wet-Bulb Depression Temp.,°F (°C)	Relative Humidity, %	Equilibrium Moisture Content, %
0 to 4	130 (54)	Vent	NA	NA	NA
4 to 24	130 (54)	115 (46)	15 (8)	62.0	9.7
24	150 (66)	125 (52)	25 (14)	48.0	6.9
48 to dry	160 (71)	130 (54)	30 (17)	43.0	5.8

TABLE 2—*Treating data for Douglas-fir plywood.*

Group	Net Absorption, lb/ft³ (kg/m³)	Dry Chemical Retention, lb/ft³ (kg/m³)
T1	35.4 (567.0)	3.19 (51.0)
T2	35.8 (572.6)	3.22 (51.5)
T3	362 (580.3)	3.26 (52.2)
T4	36.4 (583.7)	3.28 (52.5)
T5	363 (581.5)	3.27 (52.3)
T6	35.8 (572.6)	3.22 (51.5)
Average	36.0 (575.9)	3.24 (51.8)
Standard deviation	2.0 (32.6)	0.18 (2.9)

termine net solution absorption. The absorption and retention results for the six groups of 20 specimens and the overall results are given in Table 2.

The FR-treated plywood specimens were kiln dried after treatment using a 2000-board foot (15.6-m^3) steam-heated brick kiln, and the kiln schedule shown in Table 1. A four-stage temperature-humidity regime was employed. The first two stages were intended to facilitate heat absorption by the plywood; the latter two stages facilitated drying. The maximum kiln temperature of 160°F (71°C) was achieved at 48 h into the kiln-drying process. Total time in the kiln was 72 h. Initial plywood moisture content was 110 to 125%, and final moisture content was approximately 15 to 18%. Throughout kiln drying, an average air speed of 200 to 220 ft (61 to 67 m) per second was maintained through the load, with fan reversal every 3 h. Although this schedule was appropriate for small kiln loads, different schedules using similar maximum temperature limits will be needed for commencial kilns.

After treatment and redrying, all specimens (both untreated and FR-treated) were conditioned to a constant weight at 74°F (23°C) and 65% RH. After conditioning, each defect-free 0.5- by 3- by 24-in. 12- by (75- by 600-mm) specimen intended for high-temperature exposure was exposed at dry-bulb/wet-bulb temperatures of 170°F (77°C)/161°F (72°C). yielding 67% RH for 14, 32, 50, 68, and 86 days, using a 33-ft^3 (0-9-m^3) Hotpack (model 417532, SP Industries, Buena, NJ) envhnmental chamber. These high-temperature exposure periods are recommended in ASTM D 5516 [6]. The ASTM D07.07 subcommittee selected these exposure durations based on their evaluation of available roof sheathing temperature data. Later they further explained their reasoning in ASTM D 6305 [8].

After the appropriate high-temperature exposures were completed, all specimens (untreated and FR-treated, exposed and unexposed) were again conditioned to constant weight at 74°F (23°C) and 65% RH. After conditioning, each defect-free 0.5- by 3- by 24-in. (12- by 75- by 600-mm) plywood specimen was tested to failure in a three-point bending test using a center-point load. The test span was 22 in. (559 mm) and the rate of loading was 0.2 in. (5 mm) per min, which caused failure in 3 to 5 min. Load and center-point deflection were measured using a load cell and a linear variable displacement transducer (LVDT), respectively, both of which were interfaced to a computer that recorded load and deflection. From these data, modulus of elasticity (MOE), modulus of rupture (*MOR*), maximum load (P_{max}), maximum moment carrying capacity (*MM*), and work to maximum load (*WML*) were calculated. Modulus of rupture is a measure of strength, whereas P_{max} and *MM* are measures of load-carrying capacity. Modulus of elasticity is a measure of stiffness (resistance to deflection) and *WML* a measure of energy absorption up to failure. P_{max} and *MM* are directly related by a constant ($MM = P_{max} \cdot \text{span})/4$). Thus, the results of statistical significance tests on either variable will give identical results with the other.

After mechanical testing of each plywood specimen to failure, a 3-in. (75-mm) long section of full width and thickness was cut from near the point of failure and used to calculate specific gravity and moisture content at time of test.

For each mechanical property, the average property for each panel and treatment group at each exposure period was determined. Then, for each property for each panel and treatment group, a simple regression was fit separately with exposure time (time) as the independent variable as follows:

$y_{iU} = \beta_{0U} + \beta_{1U}(time)$ for panel i in the untreated condition
$y_{iT} = \beta_{0T} + \beta_{1T}(time)$ for panel i in the treated condition

This gave parameter estimates of the initial property b_1 and rate of property change (slope) b_1 for each panel i by treatment group (U, untreated; T, treated). Since each panel had both treated and untreated estimates, differences of the estimates within the panel were then compared using the multivariate Hotelling's T^2,

$$H_0 \left|\frac{\beta_{0U} - \beta_{0T}}{\beta_{1U} - \beta_{1T}}\right| = \left|\frac{0}{0}\right|$$

The data analysis took into account the original panel as a blocking variable. Because two specimens from each of the original ten Douglas-fir plywood panels were in each group of 20 variously treated and exposed specimens, the results from tests on the two matched specimens were averaged and these "panel"averages were compared. This is an effective method of increasing the power and accuracy of statistical analysis because it allows separation of within- and between-panel variability. Between-panel variability is used for tests of significant differences because each group has effectively been adjusted to have the same influence from within-panel variability by the specimen assignment procedure.

Two levels of statistical testing were carried out in the analysis of the data derived using the ASTM D 5516-96 Standard Test Method. At the first level of analysis (a £ 0.05), the blocked experimental design of D 5516, which specifies the use of ten observations per exposure-treatment combination (that is, two replicates from each of ten panels), could detect about a 17.5% loss in a property as significantly different about 75% of the time when that property had a coefficient of variation of 14%. At the second level of analysis (a £ 0.10), this experiment was designedto determine a 15% loss in a property as significantly different about 75% of the time. Thus, real differences of less than 14% may truly exist, but these tests could not distinguish those differences as significant.

For the analysis of trends (reduction in mechanical properties) of thermal exposure over time, a simple linear relationship between mechanical properties and exposure time was assumed. In this analysis, regression parameter estimates were obtained for both treated and untreated portions of each panel. For each panel, the differences between the untreated and treated parameter estimates were calculated and compared. These multipanel comparisons were performed using a multivariate version of a *t*-test, called Hotelling's T^2.

Results

The mechanical properties of FR-treated and untreated plywood are presented in Table 3. The results of these comparisons can be found in Figs. 1 to 3 for *MOR*, *MOE*, and *WML*, respectively. Ta-

TABLE 3—*Mechanical testing data for untreated and FR-treated Douglas-fir plywood tested per ASTM D 5516.*

Treatment	Exposure (days)	P_{max} (lb)	*MOR* ($\times 10^3$ lb/in.^{2})	*MOE* ($\times 10^6$ lb/in. 2)	*WML* (lb/in.^{2})	Moisture Content (%)	Specific Gravity
Untreated	0	240.2[a]	10.0	1.69	5.63	9.5	0.51
		43.10	1.74	0.26	2.04	0.19	0.03
Treated	0	238.5	9.44	1.67	4.04	12.2	0.53
		43.52	1.71	0.24	1.39	0.22	0.02
Untreated	14	232.3	9.56	1.73	4.76	10.20	0.51
		41.45	1.69	0.27	1.62	0.24	0.02
Treated	14	221.0	8.72	1.61	3.40	12.3	0.52
		36.07	1.38	0.24	1.09	0.27	0.02
Untreated	32	227.1	9.37	1.68	4.75	10.4	0.51
		44.70	1.84	0.24	1.76	0.27	0.02
Treated	32	226.1	8.99	1.66	3.75	12.3	0.52
		31.18	1.23	0.23	1.26	0.47	0.02
Untreated	50	228.3	9.43	1.66	4.80	10.3	0.51
		40.09	1.63	0.25	1.59	0.28	0.02
Treated	50	221.5	8.82	1.63	4.01	12.3	0.52
		43.29	I.72	0.27	1.64	0.35	0.02
Untreated	68	221.4	9.42	1.68	5.21	10.3	0.52
		36.38	1.51	0.23	1.97	0.22	0.02
Treated	68	21 3.7	8.53	1.65	3.34	12.2	0.52
		42.84	1.69	0.27	1.01	0.33	0.02
Untreated	86	234.3	9.7 I	1.71	5.13	10.5	0.5 I
		43.84	1.75	0.25	1.85	0.29	0.02
Treated	86	227.3	9.05	1.71	3.78	12.2	0.52
		41.49	1.63	0.24	1.35	0.27	0.02

[a] Average and standard deviation for all values.

P_{max} is maximum load: *MOR,* modulus of rupture: *MOE,* modulus of elasticity: and *WML,* work to maximum load.

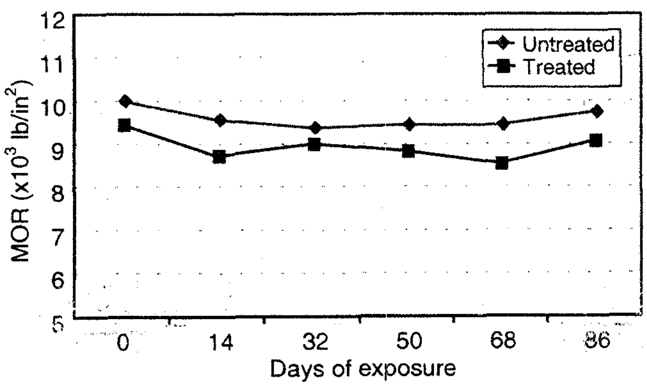

FIG. 1 —*Modulus of rupture of FR-treated and untreated Douglas-fir plywood 1 lb/in.* ^{2} *= 6.895 kPa.*

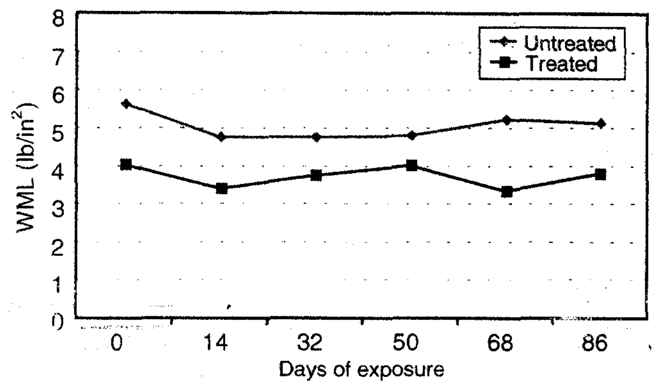

FIG. 3—*Work to maximum load of FR-treated and untreated Douglas-fir plywood. 1 in. ·lb/in.* ^{3} *= 6.895 kj/m*^{3}

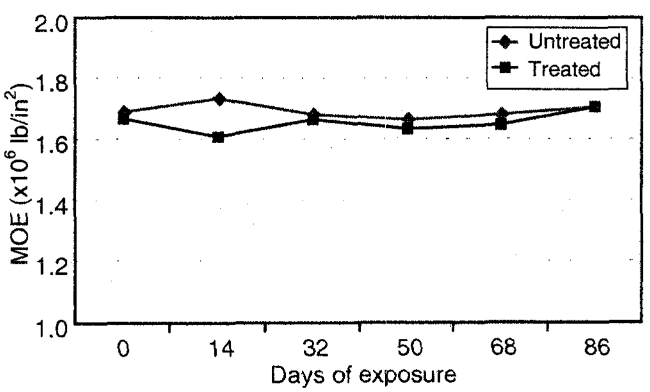

FIG. 2—*Modulus of elasticity of FR-treated and untreated Douglas-fir plywood. lb/in.* ^{2} *= 6.895 kPa.*

bles 4 and 5 show the results of statistical analysis at the 95 and 90% levels of significance. respectively Although a 95% level of significance is often considered the paramount criterion, employing a test at the 90% level of significance can indicate important trends that may influence long-term performance. Thus, both tests were employed. and the results indicated important differences between treated and untreated material The parameter estimates, *p* values, and 95% confidence intervals tor the individual mean parameter differences are shown in Table 4

The original moisture content of the untreated controls was approximately 9%. Note that the untreated and unexposed control group was adjusted from 9 to 10% moisture content for comparative purposes tor MOR (adjusted) and *MM* (adjusted) We believe this difference between untreated and unexposed controls and all the other groups was related to hysteresis. All the other groups and untreated Douglas-fir plywood. Thus, while the reduction in *WML* should be directly accounted for in engineering design. these results of unchanged fracture mechanisms should provide support for the application of simple and direct engineering adjustment factors rather than major alterations in engineering practice because of perceptions of increased brittleness.

In our testing. we deviated from the cutting and specimen allocation procedures mandated in Section 6.1.4 of D 5516. Currently. D 55 16 instructs the user to first cut and treat the 24- by 48-in. (0.6- by 1.2-m) panel sections and then cut individual specimens and allocate them to groups. Instead, we first cut the final sized 3- by 24-in. (75- by 600-mm) plywood specimens and then allocated them to experimental groups prior to FR treatment. We found this revised technique allowed us to better identify specimens with defects prior to carrying them through the treating and drying phases, It also allowed US to avoid the cutting of treated wood. Finally, when directly comparing the variability of measured mechanical properties using our revised technique to previously published data [2,14,16]. we found no increase in the variability of the measured mechanical properties in using the revised cutting and allocation technique.

The ASTM D07.07 subcommittee chose to evaluate N-grade (defect-free) plywood to maximize the yield of defect-free specimens in the cutting procedure. It was thought that this decision would reduce variability and maximize statistical power. Lebow and Winandy [16] later found no significant or practical differences in initial or ongoing FR effects on strength among four grades of FR-treated plywood (CC, AC, AA, or N grades). The consistency in the results of our test methodology using FR-treated AB-grade Douglas-fir plywood when considered in conjunction with the results of Lebow and Winandy [16] leads us to support a modification in ASTM D 5516 to loosen the current recommendation in Note 3 to use only N-grade plywood. We endorse the use of a more commercially available level of plywood quality. such as AA or AB, rather than restricting users of D 5516 to N-grade plywood. Further based on work of Lebow and Winandy [16]. we think that even lower plywood grades could be successfully used if care were taken by the users to inspect for and cull specimens having knots and other strength-reducing defects.

Conclusions

Using the ASTM D 5516-96 standard test method, our test results indicated that treatment of Douglas-fir plywood with the new boron-nitrogen, phosphate-free fire retardant (FR) had a moderate, but significant. negative initial treatment effect of about 7% on bending strength and a significant negative initial treatment effect of 26% on work to maximum load. There was no significant negative initial treatment effect on modulus of elasticity nor maximum load-carrying capacity. There was no indication of secondary treatment effects related to thermal degradation from in-service exposure to elevated temperatures for the boron-nitrogen, phosphate-free treated plywood we evaluated when compared to matched untreated plywood.

Our results support a revision to D 5516-96 to allow users to first cut the final-sized 3- by 24-in. (75- by 600-mm) plywood specimens, inspect and cull specimens with defects, and then allocate them to experimental groups prior to FR treatment and redrying. This change in specimen-cutting procedure decreases costs by culling defective specimens early in the process, increases the ease of conducting the experiment by eliminating the need to cut treated wood, and has no adverse effect on the variability of measured mechanical properties. We recommend that ASTM Subcommittee D07.07. Fire Performance of Wood, change D 5516-96 to adopt our new cutting and allocation technique as either the primary technique or as an equivalent optional technique to the existing cutting procedure.

Finally, our results show that commercially available grades of higher quality plywood, such as AA or AB, can be successfully substituted for the custom-made N-grade plywood currently recommended for use by ASTM D 5516-96 without increasing specimen variability or the applicability of the tested results. We further recommend a change be adopted in regards to the plywood quality recommended for use in Note 3 and Section 6.1.3 of D 5516-96.

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TABLE 4—*Parameter estimates of initial property* (b_0), *rate of property change* (b_1, *slope), simultaneous probability (p-value) derived using multivariate Hotelling's* T^2 (H_0) *test, and 95% simultaneous confidence intervals (CI) for individual mean parameter differences.*

Property	Treatment Group	Avg b_0 (std dev)	Avg b_1 (std dev)	*p*-value of H_0 test	95% CI for $b_{0U}-b_{0T}$	95% CI for $b_{1U}-b_{1T}$
MOR [a]	Untreated	9,631.61 (1284.99)	–1.71 (8.12)	0.027	(–81.99.1 157.62)	(–8.56,13.25)
	Treated	9,093.79 (1231.07)	–4.05 (10.62)		NS	NS
MOE	Untreated	1.6972 (0.2545)	–0.0002 (0.0011)	0.182	(–0.0305,0.1596)	(–0.0019,0.0006)
	Treated	1.6327 (0.2280)	0.0005 (0.001 8)		NS	NS
WML	Untreated	5.1031 (1.2583)	–0.0013 (0.0083)	0.003	(0.3992.2.2239)	(–0.0136,0.0143)
	Treated	3.7915 (0.8482)	–0.0017 (0.0096)		Significant	NS
MM [a]	Untreated	1,280.46 (178.76)	–0.23 (1.12)	0.396	(–67.03,97.35)	(–1.07,2.01)
	Treated	1,265.30 (174.34)	–0.70 (1.49)		NS	NS

[a] Adjusted values, *MM* is maximum moment-carrying capacity.

TABLE 5—*Parameter estimate of initial property* (b_0), *rate of property change* (b_1, *slope), simultaneous probability (p-value) derived using multivariate Hotelling's* T^2 (H_0) *test, and 90% simultaneous confidence intervals (CI) for individual mean parameter differences.*

Property	Treatment Group	Avg b_0 (std dev)	Avg b_1 (std dev)	*p*-value of H_0 test	90% CI for $b_{0U}-b_{0T}$	90% CI for $b_{1U}-b_{1T}$
MOR [a]	Untreated	9631.61 (1284.99)	–1.71 (8.12)	0.027	(19.93,1055.70)	(–6.77,11.45)
	Treated	9093.79 (1231.07)	–4.05 (10.62)		Significant	NS
MOE	Untreated	1.6972 (0.2545)	–0.0002 (0.0011)	0.182	(–0.0149,0.1440)	(–0.0017,0.0004)
	Treated	1.6327 (0.2280)	0.0005 (0.0018)		NS	NS
WML	Untreated	5.1031 (1.2583)	–0.0013 (0.0083)	0.003	(0.5492,2.0739)	(–0.0113,0.0120)
	Treated	3.7915 (0.8482)	–0.0017 (0.0096)		Significant	NS
MM [a]	Untreated	1280.46 (178.76)	–0.23 (1.12)	0.396	(–53.51,83.83)	(–0.82,1.75)
	Treated	1265.30 (174.34)	–0.70 (1.49)		NS	NS

'Adjusted values.

equilibrated from higher moisture levels in the conditioning rooms to constant weight at 74°F (23°C) and 65% RH, whereas the untreated and unexposed controls equilibrated from the drier condition up and thus stabilized at 9% rather than 10% moisture content. This 1% adjustment in moisture content from 9 to 10% changed the relative values of the untreated and unexposed controls approximately 2 to 4%.

From the analysis conducted at the 95% confidence level and described in Table 4. 'there appears to be a 7% loss in initial bending strength (*MOR*). However this loss was not significant at the 0.05 level of significance. Most important there was no indication of any difference between FR-treated and untreated plywood in its susceptibility to ongoing thermal degradation of *MOR* upon extended exposure to elevated temperatures. This is shown by the lack of any significant difference between the slopes (b_1) of treated or untreated plywood (Table 4). *MOE* was not statistically different at the 0.05 level. Although *WML* showed significant differences in initial strength effects (i.e., the intercept), this property showed no indication of a differential potential for thermal degradation over time (no significant difference in slope at the 0.05 level). Maximum moment, and by default P_{max}, did not show significant differences.

The analysis was repeated with 90% simultaneous confidence intervals for individual mean parameter differences (Table 5). A significant difference for MOR occurred in the intercepts but not the slopes at the 0.10 level of significance (Table 5). *MOE* was not statistically different at the 0.10 level. Although analysis of the *WML* data showed significant differences in the intercepts, it did not indicate a significant difference in the slopes between FR-treated and untreated material at the 0.10 level of significance. Again, maximum moment, and by default P_{max}, did not differ significantly at the 0.10 level.

Discussion

The actual difference in MOE between FR-treated and untreated Douglas-fir plywood was about –4%, and the actual difference in strength was about –7% (Tables 4 and 5). While this difference was not significant at a £ 0.05, it was significant at a £ 0.10 and should thus be considered real. The rate of strength loss (change in strength over time of exposure) did not differ significantly between treated and untreated material. Based on previous experience with the earlier generations of boron-nitrogen FR formulations [3–5] these results on strength effects were not unexpected.

Other borax–boric acid based formulations have not caused significant strength loss, nor have they caused significant changes in strength loss over time of exposure at high temperature when compared to changes in untreated wood [3,14,15]. However, these formulations have caused brashness (embrittlement, for example) in the treated material, which seemingly affects fracture mechanisms and the ductility of material treated to high borate retentions. A basic assumption of wood engineering design is that wood will react more like a ductile material than a brittle material, and this assumption appears to be violated when considering previous boron-nitrogen FRs.

The results for the FR-treated plywood reported here were empirically similar to previously reported results for earlier borax–boric acid based formulations. Work to maximum load of FR-treated plywood was significantly reduced by about 26% when compared to that of matched untreated plywood. However, the rate of loss in WML on an extended duration of high-temperature exposure was not significantly different between FR-treated and untreated plywood at a £ 0.05 or a £ 0.10. Possibly most important, no noticeable changes in the appearance or characteristics of the fracture surfaces were apparent in direct comparisons of FR-treated

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