Evaluation of a method for testing adhesive-preservative compatibility

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Abstract

The current method for testing the compatibility of adhesive-preservative systems used in glulam timbers has not recently been correlated with actual performance. The historically used two-cycle soak-dry delamination method is compared here to a proposed multiple-cycle soak-dry method. To evaluate these, penta-treated and CCA-treated southern pine boards were bonded with phenol-resorcinol at room temperature to represent compatible and incompatible model systems, respectively. The amount of deep wood failure measured with the new method shows great sensitivity to differences in system compatibility. Deep wood failure of the two adhesive-preservative systems differed only slightly before exposure to the new method, but this difference increased after one cycle and continued to increase gradually with increasing cycles. Shear strength measured with the new method was less sensitive to differences in adhesive-preservative compatibility, while delamination measured with the old method was far less sensitive.

No test method can precisely predict the service life of bonded wood products in the myriad environments in which they may be expected to serve. Yet widespread confidence in the long-term integrity of glued-laminated structural timbers is attributable to the effectiveness of a cyclic soak-dry delamination test of whole beam sections.

The cyclic delamination test method was originally developed to assess weathering performance. It has since been modified many times — each dramatically reducing the time required to perform each test. These modifications have effectively changed the objective of the test from one of predicting exterior weather resistance (with substantial collaborative field data) to one of measuring quality control (with little or no field data). Accordingly, we feel that the current cyclic delamination test is not sensitive enough to predict adhesive-preservative compatibility.

The current test is not comprehensive in that it defines only one point of a delamination-cycles function, but not the function itself. For example, joints prepared with two different adhesive-preservative systems could have the same percentage of delamination at one point in time but very different percentages at other points in time. Present methods do not reveal these differences. There are other weaknesses as well. Neither the extent of delamination in the interior of large beam sections used in the test nor the extent of the deterioration of the remaining intact bondline can be ascertained by visual inspection or by probing the bondline with a feeler gauge as prescribed in the standard delamination test.

To overcome these problems, we wanted a test with a rigorous exposure based on the principal factors causing degradation in service (water and swell-shrink stress) that would reveal relative rates of degradation of bonded wood joints. We believe that such a test would provide greater sensitivity than the cyclic delamination test. The objective of this study was to evaluate an accelerated laboratory method needed for assessing the compatibility of new adhesives and preservatives intended for use in preservative-treated glued-laminated products.

Background

The cyclic soak-dry delamination test of whole beam sections was developed in 1943 to evaluate the weathering resistance of laminated white oak ship timbers bonded with the then-new melamine, resorcinol, phenol-resorcinol, and low-temperature-setting phenol resin adhesives (11, 19). Truax and Selbo (19) found good correlation between the amount of delamination in laminated beams exposed to their accelerated aging test and those exposed in actual service.

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Their original test consisted of three soak-dry cycles and required 180 days to complete. Under wartime pressures, the required time was shortened to 21 days by soaking with alternating vacuum and pressure, elevating the drying temperature, and introducing forced air circulation during drying.

After several years, the industry gained confidence in the new waterproof adhesives and there was a continuing need for a quicker quality control test. This prompted further shortening of the test time. In 1950 the test was shortened to a 12-day, three-cycle test by eliminating a period of atmospheric-pressure soak and reducing the drying time in each cycle from 6 days to 3-2/3 days (4). In 1959 the test was shortened to 8 days (5).

In 1981 the delamination test was shortened again for expeditious quality control. The present cyclic delamination test includes two alternative methods: a 3-day, three-cycle test and a 1-day, one-cycle test (6).

Unfortunately, from the standpoint of evaluating the bonding quality of new adhesives and the compatibility of new adhesive-preservative systems, there is no evidence that the results of these shortened test methods correlate with the results of actual weathering that established the level of acceptable performance for laminated timbers 40 years ago. In our opinion the test in its present form may have lost a good measure of its credibility as a means to evaluate the weathering resistance of laminated beams especially when evaluating new adhesives or combinations of adhesive and preservative.

The current cyclic delamination test is indeed rigorous, but the resistance of an adhesive joint to load or delamination is a function of time, the number of swell-shrink cycles, and other variables (10). When large sections of beam or even standard ASTM shear blocks (7) are exposed to severe cyclic swelling and shrinking, progressive checking and delamination reduce the maximum stress that the laminate can withstand after each succeeding cycle. Wilkie and Wellons (20) found that at least five swell-shrink cycles were required to reveal deficient bonds in exterior plywood. Thus, a desirable test would involve many cycles, would induce strong swell-shrink stresses to reveal poor bonds, and would then sensitively measure bond quality. One such test, a cyclic swell-shrink procedure used at FPL (9, 15) for evaluating the relative durabilities of phenolic-bonded flake and hardboards, offered these attributes.

Approach

To evaluate this new method, it was necessary to test it on a compatible and an incompatible system, and then compare its sensitivity to that of the standard cyclic test (5).

The new method uses a cyclic vacuum-pressure soak-dry treatment (VPSD) similar to that of the standard cyclic delamination tests, but the VPSD treatment is repeated for up to 20 cycles. At periodic intervals throughout the VPSD procedure, specimens are withdrawn and evaluated.

This method provides 1) the shear strength, 2) the wood failure characteristics, 3) the rate of strength loss with respect to the number of VPSD cycles, 4) the rate of change in failure characteristics caused by swelling and shrinking stresses, also with respect to the number of VPSD cycles, and 5) an indication whether these rates increase or decrease as the number of swell-shrink cycles increase.

In our proposed method we combine the swell-shrink cycling used by Baker and Gillespie (9) with a small shear block specimen developed for this type of cyclic swell-shrink durability test (18). The small shear block specimen minimizes stress relief due to check formation that occurs in many accelerated cyclic swell-shrink tests. The specimen has been used with good results at FPL in constant condition aging studies (14); however, we have not had any experience with the specimen in cyclic aging conditions.

We chose a phenol-resorcinol adhesive and two widely used preservatives to evaluate the proposed test method. Twenty years of laboratory aging experience (17) and another 20 years of industrial experience have shown that phenol-resorcinol and pentachlorophenol (penta) preservative form a compatible system. Over the years this has been the dominant adhesive-preservative system for laminated beams for general exterior use. In recent years, however, there has been a trend away from penta in favor of waterborne treatments like chromated copper arsenate (CCA).

Although early indications were that phenol-resorcinol adhesive and CCA (salt) preservative were compatible (16), industrial experience seems to indicate otherwise. Recent laboratory studies have shown mixed results (12, 13). CCA-treated laminations appear especially sensitive to the glueline curing temperature, with elevated temperature being desirable.

Based on these experiences, we chose phenol-resorcinol and penta as a compatible adhesive-preservative system and phenol-resorcinol and CCA (cured at ambient temperature) as an incompatible system in this preliminary evaluation for the proposed test method of adhesive-preservative compatibility.

The experiment was of a 2 × 3 factorial design having two types of chemical (penta and CCA) and three levels of treatment (solvent only, low retention, and high retention). The VPSD method then used five levels of cycling (0, 1, 5, 10, and 20). The cyclic delamination method used only the two specified cycles. A detailed description of our experimental procedures is presented in the Appendix.

Results

VPSD-shear tests

Before VPSD treatment there is little significant difference in shear strengths between the different types or levels of preservative treatment (Table 1). However, VPSD treatment dramatically affects mean shear strength (Fig. 1), and causes many significant differences to appear (Table 1). Differences during the first few VPSD cycles are short-term effects and are, in part, related to the water-excluding properties of the different solvent systems, particularly the mineral spirits. After five VPSD cycles, the treatments assume and

TABLE 1. — *Shear strength after cyclic soak-dry (VPSD) exposure.*

Exposure cycles	Ordered rank of each preservative and treatment level, by shear strength[a,b,c]					
	(psi)					
0	HP (2615)	HC (2405)	NC (2400)	LP (2340)	LC (2245)	NP (2210)
1	LP (2610)	HP (2590)	NP (2340)	HC (2240)	NC (2085)	LC (1660)
5	LP (1510)	HP (1470)	NP (1435)	NC (1420)	HC (1395)	LC (1225)
10	LP (1490)	HP (1360)	NP (1035)	NC (995)	HC (740)	LC (670)
20	LP (1150)	HP (1040)	NP (880)	NC (725)	HC (685)	LC (575)
Summed exposure	LP (1820)	HP (1815)	NP (1580)	NC (1525)	HC (1495)	LC (1275)

[a] LP = low-retention penta; HP = high-retention penta; NP = mineral spirits-treated (no penta) control; LC = low-retention CCA; HC = high-retention CCA; NC = water-treated (no CCA) control.
[b] Mean shear strength (psi) of 25 specimens.
[c] Means with the same underline are not significantly different from each other at the 95% probability level as tested by Tukey's Studentized Range Test.

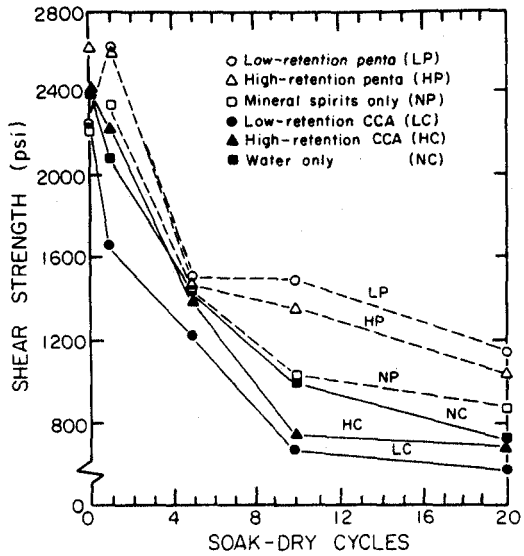

Figure 1. — Decline of the mean shear strength of each treatment group over an increasing number of cyclic soak-dry (VPSD) cycles.

maintain the rank from 1 (highest) to 6 (lowest) based on shear strength:

1 LP = low-retention penta
2 HP = high-retention penta
3 NP = penta control (mineral spirits)
4 NC = CCA control (water)
5 HC = high-retention CCA
6 LC = low-retention CCA

The differences between the mineral spirits and water-treated control specimens are not significant (Table 1). Thus the significant differences between the penta- and CCA-treated specimens' strengths after multiple VPSD cycles (Table 1) are apparently due to the preservatives and not to the solvent systems.

There is no significant overall difference between the various retention levels of each preservative. The only individual differences in performance occurred between the low- and high-penta specimens before VPSD treatment and the low- and high-CCA specimens after one VPSD cycle (Table 1). Apparently our range of retention levels (penta, 0.3 to 0.6; CCA, 0.25 to 0.4 lb./ft.) was not great enough to have a differential effect on bond strength. Though contrary to some general experience, this lack of a differential effect has been reconfirmed. Subsequent research indicates that the eventual bond strength with southern pine is far more sensitive to changes in bonding conditions, such as assembly time and pressure, than it is to changes in CCA retention (River, to be published).

An important feature to note from this discussion and from both Figure 1 and Table 1 is that these differences (or lack thereof) in compatibility between CCA-phenol-resorcinol and between penta-phenol-resorcinol preservative-adhesive systems (as indicated by changes in shear strength) were not readily apparent until after five VPSD cycles. In comparison, the current cyclic delamination test, which employs only two cycles before determining the percent delamination, does not detect this large loss in performance that occurs after more than two cycles.

Wood failure characteristics are more sensitive to the differences between penta and CCA treatments than are shear strength characteristics. In comparing the percentage of wood failure based on the average of high- and low-retention specimens from each preservative, there is little difference between the two treatments before the first VPSD cycle (Table 2). However, after one

TABLE 2. — *Total wood failure after cyclic soak-dry (VPSD) exposure.*

Exposure cycles	Ordered rank of each preservative and treatment level, by total wood failure[a,b,c]					
	(%)					
0	LP (89)	NP (80)	LC (86)	NC (82)	HP (82)	HC (77)
1	LP (97)	NP (95)	HP (95)	NC (87)	HC (84)	LC (79)
5	LP (95)	NP (95)	HP (92)	HC (82)	NC (79)	LC (76)
10	LP (97)	HP (96)	NP (92)	HC (80)	NC (71)	LC (68)
20	LP (93)	HP (91)	NP (88)	NC (78)	LC (70)	HC (70)
Summed exposures	LP (94)	NP (92)	HP (91)	NC (79)	HC (79)	LC (76)

[a] LP = low-retention penta, HP = high-retention penta; NP = mineral spirits-treated (no penta) control; LC = low-retention CCA; HC = high-retention CCA; NC = water-treated (no CCA) control.
[b] Total wood failure (%) includes both deep and shallow failure as defined in the text.
Weans with the same underline are not significantly different at the 95% probability level as tested by Tukey's Studentized Range Test.

VPSD the percentage of wood failure increased by approximately 10 percent in the penta treatments (average of the high and low retention and the mineral spirits controls) compared to the precycle result. The percentage of wood failure in the penta treatments (average of LP, HP, and NP) was about 14 percent higher after one cycle than the percent failure in the CCA treatments (average of LC, HC, and NC). This significant difference increased to about 22 percent after 20 cycles, as the percentage of wood failure in CCA specimens gradually declined.

The differences between the two treatments are even more pronounced when deep wood failure is distinguished from shallow wood failure (failure in the wood only a few cells below the surface—i.e., at or about the limit of adhesive penetration (Fig. 2). The amount of deep wood failure is directly related to bond quality. The penta-treated wood joints produce 20 to 25 percent more deep wood failure than do the CCA-treated wood joints (Fig. 3). If joints of the same treatment level are compared, between 5 to 20 VPSD cycles, differences of 20 to 32 percent in the amount of deep wood failure are found (Fig. 3, bottom curve).

The top curve in Figure 3 shows a slight upward trend in the deep wood failure of penta-treated specimens with increasing number of cycles and a slight downward trend of CCA-treated specimens (middle curve). The bottom curve shows that after five cycles the difference between mineral spirits- and water-treated controls remains constant at about 20 percent. The implication is that the solvent system has a lot to do with the initial difference between the performance of the two adhesive-preservative-solvent systems but that after five cycles the increasing difference between the systems is due to the preservative. This implication, in part, contradicts the observation based on the strength tests—that preservative chemical was more important than the solvent. This contradiction bears further exploration.

Cyclic delamination tests

The cyclic delamination tests showed slight differences between the performance of penta- and CCA-treated bondlines, but these differences were not statistically significant (Table 3). The four low-retention penta-treated specimens exhibited no delamination, while the four equivalent CCA specimens averaged 5.1 percent delamination—0.1 percent over the maximum allowed by industry specifications (2,3). Only one of the four high-retention penta-treated specimens exhibited any delamination (0.7%) while the equivalent CCA-treated specimens averaged 2.5 percent delamination—well below the 5 percent maximum allowed. One mineral spirits-treated control specimen had 2.6 percent

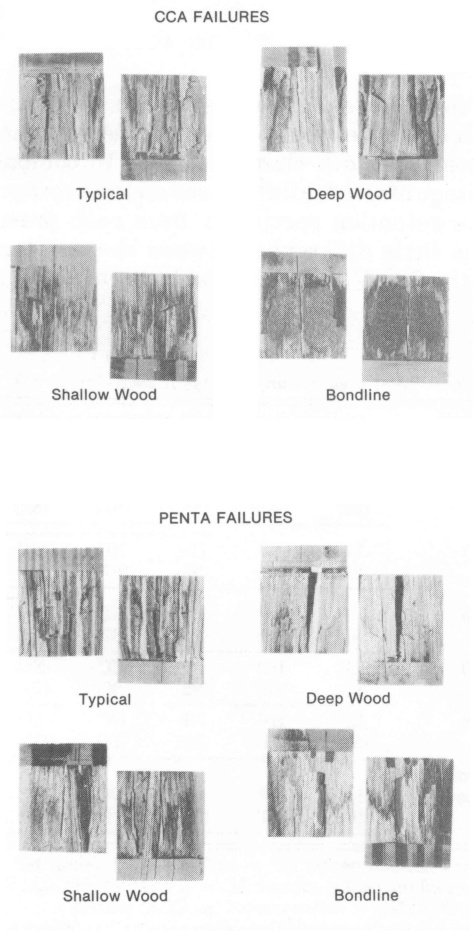

Figure 2. — Examples of some characteristic failures for both CCA- and penta-treated shear blocks.

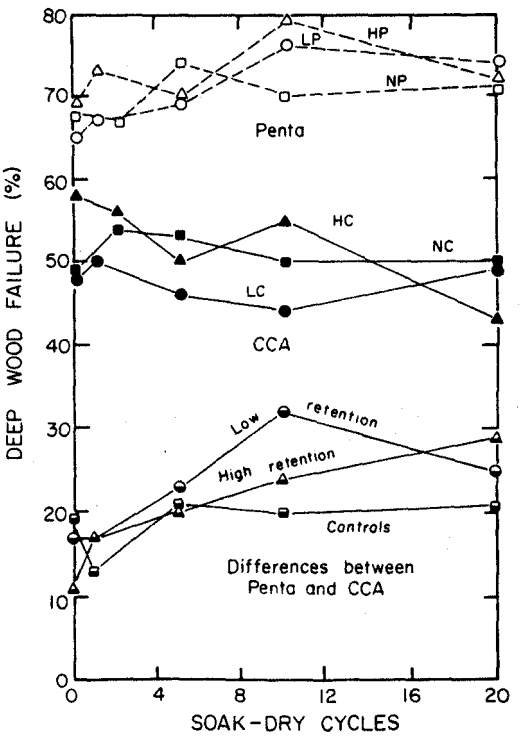

Figure 3. — Percentage of deep wood failure with respect to the numbers of soak-dry cycles (VPSD) in penta treatments and mineral spirits controls (top) and in CCA treatments and water controls (middle), and the differences between the percentages for the two treatments and controls (bottom).

TABLE 3. — *Delamination of 3- by 3- by 3-inch four-ply blocks after two soak-dry cycles (ASTM D 1101-59) (6).*

Treatment	Specimen	Delamination at each treatment level		
		Control	Low retention	High retention
		(%)		
Penta				
	1	0	0	0
	2	0	0	0
	3	2.6	0	0.7
	4	0	0	0
	Avg.	0.65	0	0.18
CCA				
	1	0	6.1	2.2
	2	0	3.9	1.8
	3	0	6.0	2.4
	4	0	4.6	3.7
	Avg.	0	5.1	2.5

delamination, while water-treated control specimens exhibited no delamination.

The cyclic delamination test which we used has less severe drying conditions than the current industry standard.' This difference may explain in part the lack of sensitivity which we observed. In addition, specimens wider than those used here would have tended to create higher internal stresses and may have increased the sensitivity of the test to bond quality. [2] However, laminae width is not specified in either current cyclic delamination test method (2,6).

Conclusions

The proposed VPSD-shear test with evaluations of shear strength and wood failure characteristics after multiple VPSD cycles is an effective test for determining the compatibility between an adhesive and preservative-treated wood. The standard cyclic delamination test method is less informative and less effective.

Although few significant differences in shear strength were detected with respect to the level of each preservative treatment, striking differences appear between the types of preservative treatments, especially after 10 cycles. When phenol-resorcinol bondlines are cured at room temperature:

- CCA-treated specimens are significantly lower in bondline shear strength than penta-treated specimens.
- Wood failure and, in particular, deep wood failure (failure well beyond the depth of adhesive penetration) is significantly higher in the penta-treated specimens than in the CCA-treated specimens.

No significant differences are detected in delamination as a result of the type or retention level of preservative treatment. The greatest percent delamination occurs in the low-level CCA-treated specimens; however, the percentage exceeds the maximum permissible in the industry specification (3) by only 0.1 percent. A new version of the standard cyclic delamination test (2,6) uses more severe drying conditions; this, together with the use of wider specimens (which would create greater internal stresses), will undoubtedly improve the sensitivity of the delamination test to preservative-influenced differences.

The VPSD-shear method yields information on bondline shear strength, wood failure characteristics, rates of change in these two criteria as swell-shrink processing accumulates, and the change in these rates. The cyclic delamination test yields only one delamination value after two swell-shrink cycles.

Future plans

We plan further work with the cyclic VPSD-shear test method and with the cyclic delamination test, using wider laminae and more severe drying conditions. This work will concentrate on 1) refining the new cyclic VPSD-shear test, 2) exploring solvent versus chemical effects, and 3) developing an understanding of the correlation between laboratory results and outdoor performance.

[1] The test used for this study, ASTM D 1101-59 (reapproved 1976) (5), specifies 91-1/2 hours of air-dryin at 27° 29°C and 25%-30% RH and was still in effect at the time of these experiments. The current standard cyclic delamination test (ASTM 1101-81) (6) specifies 10 hours of ovendrying at 71°C and 8%-10% RH. Both methods specify 500 ft./min. air speed.

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Appendix: materials and methods

Materials, preservatives, and treatments

One hundred and forty-four pieces of southern pine lumber, 25 mm (radial) by 89 mm (tangential) by 381 mm (longitudinal) (1 by 3.5 by 15 in.), were prepared for this study. Each piece had specific gravity of at least 0.59 at 12 percent moisture content (ovendry basis) and satisfied the requirements of ASTM D 905 (7).

Twenty-four pieces were randomly assigned to each of six treatment groups:

- Penta, low-retention (LP), 0.3 pcf
- Penta, high-retention. (HP), 0.6 pcf
- Control, mineral spirits-solvent only (NP)
- CCA, low-retention (LC), 0.25 pcf
- CCA, high-retention (HC), 0.4 pcf
- Control, water-solvent only (NC)

This material was pressure treated with the assigned treatment to retention levels specified for aboveground (low-retention) or ground-contact (high-retention) service by C28-79 (8).

Each piece was weighed before and after treatment to determine net preservative retention. The eight pieces with the greatest deviation from the mean retention at each treatment level were discarded in an effort to reduce variability in bondline quality resulting from retention variability.

The remaining pieces were stickered and air-dried at »23°C (»73°F, uncontrolled relative humidity) to approximately 20 percent moisture content. They were then equilibrated in a room controlled at 27°C (80°F) and 65 percent relative humidity (approximately 12% EMC) for not less than 30 days before bonding and testing.

Shear strength tests

Specimen preparation.—For shear strength tests, 12 randomly selected pieces from each group of 16 were machined to 22 by 63 by 305 mm (7/8 by 2.5 by 12 in.) and then sawn into two thinner pieces 9.5 mm (3/8 in.) thick. The thinner pieces were knife-jointed on one side (the side to be bonded) and then reduced to the final thickness of 8 mm (5/16 in.) by knife-planing the opposite side. Each thin piece was randomly assigned to 1 of 12 pairs in its retention level. The pairs were bonded with phenol-resorcinol adhesive according to the adhesive manufacturer's directions. The conditions for bonding were:

- Spread: 300 g/m^2 (60 lb./1,000 ft.^{2}) evenly divided between the two pieces
- Open assembly time: 30 seconds
- Closed assembly time: 70 minutes CCA panels, 20 minutes penta panels
- Pressure: 1,200 kPa (175 psi)
- Temperature: 23°C (73°F)
- Press time: 24 hours

After pressing, each panel cured for another 7 days at 23°C (73°F) and 65 percent RH before being cut into 16- by 25- by 38-mm-long (5/8 in. by 1 in. by 1-1/2 in. long) compression shear block specimens, as developed by Strickler (18) for durability testing. Two preservative treatments, three retention levels (including control), 12 panels per level, and 14 specimens per panel produced 1,008 specimens. Each specimen was carefully inspected for defects and discarded if any were found. Subsequently, 750 were randomly selected and assigned to the 30 groups.

Cyclic soak-dry (VPSD) exposure.—The 25 replicate specimens for each treatment and retention level combination were subjected to either 0, 1, 5 10, or 20 VPSD cycles. Each cycle consisted of subjecting the samples to 30 minutes of vacuum at 737 mm (29 in.) H while submerged in tapwater at room temperature, followed by 30 minutes of pressure at 413 kpa (60 psi) while submerged, and finally, drying for 16 hours at 43°C (110°F) in a forced draft oven. After drying, the moisture content was less than 6 percent. After each group of 25 specimens had been subjected to the required number of VPSD cycles, the specimens were conditioned to equilibrium moisture content in a room at 27°C and 65 percent RH before testing.

Specimen testing.—Shear testing was conducted as prescribed in ASTM D 905 (7) with the exception of the specimen size (18).

Cyclic delamination tests

Specimen preparation.—The remaining four pieces (25 by 89 by 381 mm) from each of the six treatment groups were planed to 19-mm (3/4 in.) thickness. The four pieces for each treatment group were bonded together under the same conditions used or the shear specimens to produce a laminate >76 mm (3 in.) thick. After cure, the six laminates were planed to 76 mm wide and cut into four 76-mm-cubed specimens.

Exposure and evaluation.—The cyclic delamination exposure and evaluation were conducted as specified in ASTM D 1101-59 (5). Each specimen is placed in an autoclave, completely immersed in water 18° to 27°C (65° to 80°F), exposed to a vacuum of 508 to 635 mm (20 to 25 in.) Hg for 15 minutes, and finally exposed to a pressure of 1,034 kPa (150 psi) for 2 hours. Then the entire vacuum-pressure cycle is repeated. After the two vacuum-pressure cycles, the specimens are dried at 27° to 29°C (80° to 85°F), 25 to 30 percent RH with a controlled airspeed of 150 m/min. (500 ft./min.) for 91-1/2 hours. This entire 96-hour vacuum-pressure dry cycle is repeated a second time before delamination is measured. Delamination is visually measured using magnification and a 0.08- to 0.10-mm (0.003 to 0.004 in.) feeler gauge.

This standard was revised in 1981 after these experiments had been completed. The two current versions of the cyclic delamination tests are ASTM D 1101-81 (6) and AITC 201-81 (2). Both versions use increased drying temperatures, decreased RH, and decreased drying duration. It is anticipated that these revised methods would be more severe than the older method we used because of the more severe drying conditions, but these changes would not significantly affect the results reported in this paper or the reasons for these experiments.

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