ACCELERATED AGING OF PHENOLIC-BONDED FLAKEBOARDS

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Abstract

Specimens of phenolic-bonded flakeboard, vertical-grain southern pine and Douglas-fir, and marine-grade Douglas-fir plywood were exposed to four accelerated aging situations. These consisted of: 1) Multiple cycles of boiling and elevated-temperature drying, 2) multiple cycles of vacuum-pressure soaking and intermediate-temperature drying, 3) the six-cycle ASTM D-1037 exposure and, 4) continuous exposure to weathering at the Madison, Wis. site. Thickness change and bending strength, bending stiffness, and internal bond strength retention were measured after various numbers of exposure cycles, or time of exposure. The phenolic-bonded flakeboards retained enough strength and stiffness during accelerated aging to indicate they might be used satisfactorily in structural applications where conventional wood products are now used.

Introduction

The acceptance of new types of structural grades of flakeboard would be stimulated if suitable long-term service life can be demonstrated. The purpose of this study was to develop methods for assessing such service life and to apply these methods to evaluate selected prototypes of flakeboard from forest residues. This work was to determine the life-expectancy of phenolic-bonded flakeboard using accelerated aging by comparing its response with that of exterior-type plywood and of solid lumber.

There is much misunderstanding about accelerated-aging tests--the experimental techniques, analysis, interpretation of results, correlations, and objectives. But accelerated-aging tests are an essential step in the process of developing new products for use in critical, long-term applications. They can provide insight into degradation that could occur in service and thereby reduce the risk that an inadequate product might appear in the marketplace.

Evaluating a phenolic-bonded flakeboard for long-term performance is a different problem from that of evaluating a new adhesive system for its potential use in wood products. Structural flakeboard is made with an adhesive known to have excellent resistance to thermal degradation, to hydrolysis by moisture, and to the swelling and shrinking stresses a variety of acceptable wood products impose on the gluelines. These facts were responsible for selecting phenolics as the binder system in structural grades of flakeboard. The problem then, was to devise accelerated-aging tests to evaluate a bonded wood product for the retention of properties that are important in the intended end use.

The principal intended end use for structural flakeboard is for wall, roof, and floor sheathing where bending strength and stiffness are the important properties governing design and application. Because of this, bending strength and bending stiffness were the primary propertiesevaluated.

The intended end use also requires the product to resist the changing moisture conditions that arise in service.. Because of this, phenolic-bonded flakeboard will be considered under requirements similar to those of exterior-type softwood plywood and not of the intermediate or interior types that are often used for sheathing purposes.

Background Review

Cyclic boil-dry treatments are most often used for evaluating weather-resistant products. This treatment maximizes the effect of three important influences that cause degradation--heat, moisture, and swelling and shrinking stresses. Boil-dry cycles are an empirical mixture of the three influences rather than any logical or intended simulation of a real-life situation. But repeated boil cycles have the potential to degrade weather-resistant products in the laboratory at a relatively rapid rate so that comparison between products can be made in a short time.

The accelerated-aging procedures in common use today for evaluating wood-based materials were developed primarily for quality-control purposes. In fact, ASTM D 1037 cyclic test for accelerated aging (1), which is a mixture of soaking, steaming, freezing, and drying through 6 cycles requiring 12 days, is the prescribed test in the product standard for Type 2 mat-formed wood particleboard (CS 236-66) (12). The West Coast Adhesive Manufacturers Association (WCAMA) (15,16) attempted to reduce the tine required for this test by a 6-cycle procedure of vacuum-pressure soaking, boiling, and drying to be completed in 6 days. This exposure and the D 1037 treatment are much more complicated and time-consuming than the single boil-dry-boil used for quality control in the manufacture of interior-type softwood plywood (PS-1-74) (13).

Shen and Wrangham (11) more recently reported on the development of a rapid accelerated-aging test also for quality-control purposes. They evaluated the 2-hour boil followed by wet testing, described in the German Standard DIN 68761 (5) for change in internal bond (IB) strength. This exposure was used in the Canadian Standard (CSA 0188-68) (3) but applied to bending specimens. Shen and Wrangham then correlated torsion shear testing with the more traditional IB test. But any of these quality-control tests provide only limited information--a two-point, before-and-after condition. They cannot be used for estimating the long-term performance.

There is little background information about how particleboard responds to exterior exposure--none of it extends for longer than 8 years. The WCAMA tests with board of unknown particle geometry showed MOR losses of over 50 percent In 5 years. The most extensive information on flakeboard durability was that by Hann, Black, and Blomquist (7,8) and Jokerst (9) at the Forest Products Laboratory. Weathering of phenolic-bonded flakeboards showed bending strength losses of 50 to 63 percent in 8 years at the Madison site. Clad and Schmidt-Hellcrau (4) reported data for 3-year exposures on particleboards produced in Germany with alkaline-type phenolic adhesives. All of this work shows a general pattern of rapid loss of strength and stiffness during the first year or two of exposure with a much slower rate of loss in subsequent years, at least through 8 years.

Repeated boil-dry cycles to follow strength losses throughout a product's useful life are difficult to perform in the laboratory. A limited attempt to do this was made during the early development of the rate-process method of analysis (6). Specimens were carried through as many as 35 standard boil-dry-boil cycles (PS-1-74), but this was time-consuming and difficult to carry out manually during normal working hours. Attempts to develop machines to carry out cyclic tests automatically have been described in the literature from time to time with the most successful initiated at the Canadian Forest Products Laboratory in Vancouver (14). This equipment was further refined and extensively evaluated by the Weyerhaeuser Company (10). The developed method has been adopted as an ASTM Standard (D 3434, Multiple-Cycle Accelerated-Aging Test (Automatic Boil Test) for Exterior Wet-Use Wood Adhesives) (2). While the equipment was designed and used for evaluating standard plywood shear specimens, small lap specimens, and finger-joint specimens, modifications in design would adapt the unit to evaluate larger sized specimens such as flakeboard bending specimens. Any extended program of research involving cyclic accelerated aging of either products or adhesives would benefit from a machine of the Weyerhaeuser type.

Experimental Procedures

Materials

Flakeboard, plywood, and wood samples were selected for comparison. Each was assigned an alphabet identification that is referred to in the Results and Discussion and all tables and figures. These materials are described in Table 1.

Specimen Selection

Each material was cut into specimens 1/2 by 2 by 12 inches. Enough specimens were cut to carry out the planned treatments and sampling schedules. All specimens were conditioned to 80°F (65 percent relative humidity (RH)) prior to and after accelerated-aging exposures to achieve comparable moisture conditions at time of test. Specimens were mounted on racks designed to maintain a 1/4-inch space between them during the aging treatments.

Accelerated-Aging Exposures

Boil-dry exposure

Thirty specimens from each material were saturated with water by vacuum-pressure soaking and immediately placed in boiling water. There was sufficient stored heat to maintain boiling conditions when cold wood samples were placed in the bath. Distilled water was used in the bath to reduce scaling problems. After 10 minutes, the specimens were removed, allowed to drain momentarily, and then placed in an air circulating oven at 225°F for drying. At the end of 3-3/4 hours, the specimens were removed and weighed. The drying time was selected so that moisture content of the slowest drying material was reduced to 6 to 8 percent.

The vacuum-pressure soak was applied only before the first boiling treatment and not applied to subsequent cycles. Five randomly selected specimens were removed from each set for test after each of the prescribed cycles--1, 5, 10, 20, 40, and 80. Two cycles per day could be accomplished.

Vacuum-pressure-soak-dry exposure

Replicates of five specimens for each material were subjected to 1, 5, 10, 20, and 40 cycles of vacuum-pressure-soak-dry cycles. Each cycle consisted of 30 minutes at 29 inches Hg vacuum and 30 minutes of pressure at 60 psi submerged in tap water and 23 hours of drying in a forced-draft oven at 180°F. Weighing specimen sets before and after cycling assured that the slowest drying material was reduced to 6 to 8 percent moisture content during drying. One cycle per day could be accomplished.

Accelerated aging by ASTM D 1037

Five specimens for each material were subjected to accelerated aging by ASTM D 1037 accelerated-aging test procedures consisting of 6 cycles of the following sequence:

1. Soaking in water at 120°+3°F--1 hour.

2. Spraying with steam and water vapor at 200°±5°F--3 hours.

3. Storing at 10°±5°F--20 hours.

4. Drying at 210°±3°F--3 hours.

5. Repeating 2 above--3 hours.

6. Repeating 4 above--18 hours.

This procedure requires a minimum of 12 days and was carried on for each material, yielding only before and after values.

Outdoor exposure

Thirty individual specimens from each material were placed on an exposure rack at the Madison site. The specimens were attached with brass screws and exposed at an angle of 60° to the horizontal facing south. At various intervals of time, five randomly selected specimens from each set are planned for removal for test. After the first year of exposure, subsequent exposure periods will depend upon the extent of degradation each material already experienced, with the view to test all specimens of each type while specimen integrity is still maintained and reasonable rates of property loss can be measured.

Measurements and Physical Testing

Specimens were measured for thickness and width after conditioning at 80°F, 65 percent RH. The weight of specimen sets was measured before and after accelerated aging exposure and after conditioning. conditioning at 80°F; 65 percent RH.

Each specimen was tested for bending strength, bending stiffness,, internal bond strength, and thickness swell. A span of 10 inches was used in the bending measurements.

Measurements of tensile strength perpendicular to the face (internal bond) were carried out on the ends of the specimens broken in the bending strength tests, selecting portions that received the least strain during bending.

Data Analysis

Bending strength and bending stiffness were calculated on the basis of original dimensions measured at 80°F, 65% RH, before exposure to accelerated aging. For unexposed materials, the mean and standard deviation were calculated for the replicates tested to provide an original property value and an indication of the variability in the unexposed materials. The mean value for replicates of exposed specimens was calculated and converted to a percentage of the property retained by comparison with the original mean value. Any change in variability that might have taken place during aging was found not to influence the general conclusions reached in this study.

The percentage change in each property obtained during accelerated aging was plotted against the number of cycles exposed. Inspection of a large number of these plots revealed a rapid property loss in the first cycle followed by a generally uniform slower rate of loss in subsequent cycles. Consequently, a linear regression line was calculated by the least squares method to provide an average rate of change of all specimens of each material subjected to accelerated aging. The values for unexposed specimens was not included in the calculations of the regression lines. Because of the limited number of replicates, the variability among specimens, and the limited objectives of this study, more sophisticated analysis of the data was unwarranted.

Results and Discussion

The properties of the unexposed materials evaluated are given in Table 2. The flakeboards at 0.60 to 0.64 specific gravity were generally more dense than plywood and solid wood at 0.47 to 0.50 specific gravity. Flakeboard bending strength ranged from about 2,658 psi to 4,927 psi, in contrast with plywood at 6,663 psi, Douglas-fir lumber at 14,346 psi, and southern pine lumber at 16,176 psi. Bending stiffness for the flakeboards was between 508,000 and 677,000 psi; plywood, 965,000 psi; Douglas-fir wood, 2,007,000 psi; and southern pine, 1,990,000psi. The internal bond value of 127 psi for plywood was within the range of the values of 69 to 164 psi for the flakeboards, as compared with the much higher values of 372 psi for Douglas-fir wood and 612 psi for southern pine wood.

[1]The terms "bending strength" and "bending stiffness" are used because the specimens used in this study are smaller than the specimen specified in ASTM D 1037 (3 by 14 in.) for determination of modulus of rupture and modulus of elasticity of 1/2-inch-thick specimens.

The results of the accelerated-aging tests are shown in Figure 1 for bending strength response, in Figure 2 for bending stiffness, in Figure 3 for internal bond strength, and in Figure 4 for changes in thickness swelling. The figures include regression lines for each material subjected to the cyclic boil-dry and vacuum-pressure soak-dry treatments, each marked with the appropriate letter code. The results of the D 1037 test and the 1-year test fence exposure, providing single values for each material, are shown in the figures on a separate "percent retained" axis with the individual letter codes placed at the point of retentionobtained.

With regard to bending strength, Figure 1, all accelerated-aging tests appeared to differentiate among the materials in the same way. The highest bending strength retention was obtained with solid wood and plywood. The four laboratory-prepared flakeboards were similar in their bending strength retention, which was generally at somewhat lower level than solid wood and plywood. The lowest bending strength retention was shown by the commercially prepared waferboard, but it still retained 30 to 40 percent of its strength after the D 1037 test and after 40 cycles of vacuum-pressure-soak-dry or 50 cycles of the boil-dry test The commercially prepared waferboard is believed to have a lower binder content than the laboratory-prepared flakeboards evaluatedhere. One year of test fence exposure reduced bending strength no more than 25 percent in all cases. For the flakeboards, 10 boil-dry cycles reduced strength more than 1 year on the test fence, while 10 cycles of the vacuun-pressure-soak dry treatment reduced strength less than 1 year on the test fence.

The bending stiffness (Figure 2) of solid wood and plywood did not change appreciably during any of the accelerated-aging treatments. Stiffness appeared to increase slightly during the cyclic wetting and drying treatments with the lumber and plywood, and only 10 percent or less was lost by the D 1037 test or 1-year test fence exposure. The four laboratory-prepared flakeboards lost stiffness with wet-dry cycling but still retained 70 to 80 percent after 80 boil-dry cycles or 80 to 90 percent after 40 VPS-dry cycles. The D 1037 test and 1 year of test fence exposure produced bending stiffness losses about equivalent to those caused by 50 to 60 boil-dry cycles and 40 VPS-dry cycles. Again, the commercially prepared waferboard lost bending stiffness to a greater extent than did the other materials evaluated, but still retained more than 40 percent under the most severe treatment, 80 boil-dry cycles.

The internal bond strength (Figure 3) was the strength property undergoing the most change during accelerated aging. Even the D 1037 test produced a 20 percent loss in internal bond with solid wood and plywood. This loss was equivalent to the loss after 10 to 20 boil-dry and VPS-dry cycles. Continued cycling produced drastic reductions in internal bond strength with all materials evaluated, with the greatest loss being exhibited by the commercially prepared waferboard. One-year exposure on the test fence produced only moderate losses of internal bond strength with the flakeboards (60percent or better retention) and little change in Douglas-fir and plywood.

The southern pine lumber, after exposure to the D 1037 conditions, yielded lower internal bond strength than expected. Southern pine lumber became noticeably checked in all accelerated tests and posed experimental problems during testing, especially in internal bond strength measurements.

There appeared to be some differences among the four laboratory-prepared flakeboards with regard to internal bond retention, variances which were not differentiated by either bending strength or stiffness measurements. The residue flakeboard B lost internal bond strength to a greater extent than did flakeboard A which had been prepared with a higher binder content. Flakeboard L, similar to flakeboard B but prepared from a different residue mix, lost internal bond strength at a rate intermediate to that exhibited by flakeboards A and B. The removal of fines from flakes to produce flakeboard 0 appeared to have little effect on internal bond retention in either the boil-dry cycles or the D 1037 test. There was a hint of improvement caused by fines removal with the VPS-dry treatment, but it is highly unlikely that the noted difference is statistically significant considering the variability in the data.

As expected, there was little residual thickness swelling (Figure 4) with the two solid wood samples or plywood in any of the accelerated-aging treatments. Of these, the 4 percent thickness swelling of plywood was the greatest. All laboratory-prepared flakeboards exhibited thickness swelling ranging from about 14 percent to 27 percent, and the commercially prepared waferboard swelled over 40 percent after 80 boil-dry cycles. The boil-dry cycles caused most of the thickness swelling during the first few cycles, with only minimal changes thereafter. The VPS-dry cycles caused less swelling during the first few cycles. The swelling gradually increased to about the same swelling after 40 VPS-dry cycles that was obtained after 1 to 5 cycles of the boil-dry treatment. The D 1037 produced about the same thickness swelling as 1 to 5 boil dry cycles or 40 VPS-dry cycles, with the exception of the waferboard.

Thickness swelling after 1-year test fence exposure was no greater than 10 percent, in all cases.

The test fence exposures were considered to be an accelerated treatment be-Cause individual test specimens, rather than panels were exposed in a position to provide maximum opportunity for wetting and drying. After 1-year exposure, two laboratory-prepared flakeboards (A and B) retained 83 and 91 percent bending strength and 90 and 71 percent internal bond strength, respectively. Previous outdoor exposure tests (7,8,9) had involved panel exposure in a vertical position facing south and strength test specimens were cut from panel centers with the edges discarded--a much less rugged exposure. A flakeboard bonded with 6 percent phenolic resin and no wax, when exposed in this fashion for 1 year, retained 52 percent bending strength and 47 percent internal bond strength. After 8 years' exposure these values decreased only to 48 percent bending strength retention and 33 percent internal bond strength retention. Comparing these results with those from the present study where fence exposure degradation was accelerated suggests that the phenolic-bonded flakeboards should perform well for many years during outdoor weathering. Results of strength tests after additional years of exposure will be needed to verify thisindication.

Significance of This Work

The results of these accelerated-aging tests, provide confidence that phenolic-bonded flakeboards manufactured from forest residues would serve well in many structural applications. They retained a respectable level of bending strength and stiffness when exposed to soaking and drying so severe that even solid lumber and marine-grade plywood, whose performance is well recognized, suffered appreciablelosses,

A comparison of the accelerated aging procedures suggests that multiple cycles of boiling and drying, capable of automation, could detect differences in material behavior in a manner similar to that produced by the standard, but more complicated,method.

References

1. American Society for Testing and Materials. 1977. Standard methods of evaluating the properties of wood-base fiber and particle panel materials. ASTM Desig D 1037-72. ASTM, Philadelphia, Pa.

2. American Society for Testing and Materials. 1977. Standard recommended practice for multiple-cycle accelerated aging test (automatic boil test) for exterior wet-use wood adhesives. ASTM Desig D 3434-75. ASTM, Philadelphia, Pa.

3. Canadian Standards Association. 1975. Mat-formed wood particleboard. CSA-0188-1975. CSA Rexdale, Ontario.

4. Clad, W., and C. Schmidt-Hellerau. 1965. Behavior of amino plastic and phenolic resins in particleboard during outdoor exposure. Hold-Zentralblatt 44/45:349-352.

5. 'GermanStandard. 1973. Particleboards; flat-pressed boards for building; terms, properties, testing, supervision. DIN 68763. Deutsche Normenausschusses, Berlin.

6. Gillespie, R.H. 1965. Accelerated aging of adhesives in plywood-type joints. For. Prod. J. 15(9):369-378.

7. Hann, R.A., J.M. Black, and R.F. Blomquist. 1962 How durable is particleboard? For. Prod. J. 12(12):577-584.

8. Hann, R.A., J.M. Black, and R.F. Blomquist. 1963. How durable is particleboard: Part 11. The effect of temperature and humidity. For. Prod. J. 13(5):169-174

9. Jokerst, R.W. 1968. Long-term durability of laboratory-made Douglas-fir flakeboard. USDA For. Serv. Res. Note FPL-0199. For. Prod. Lab., Madison, Wis.

10. Kreibich, R.E., and H.G. Freeman.1968. Development and design of an accelerated boil machine. For. Prod. J. 18(12):24-26

11. Shen, K.C., and B. Wrangham. 1971. A rapid accelerated-aging test procedure for phenolic particleboard. For. Prod. J. 21(5):30-33.

12. U.S. Department of Commerce, National Bureau of Standards. 1966. Mat-formed wood particleboard. Commercial Standard CS 236-66. U.S. Dept. Comm. Washington, D.C.

13. U. S. Department of Commerce, National Bureau of Standards. 1974. Construction and industrial plywood. Voluntary Product Standard PS-1-74. U.S. Dept Comm. Washington, D.C.

14. Walser, D.C., and H.G.M. Colbeck. 1967. Bond-degrade accelerating machine helps predict bond life. Adhesives Age lO(11):33-35.

15. West Coast Adhesive Manufacturers Association Technical Committee. 1966. A proposed new test for accelerated aging of phenolic resin-bonded particleboard. For. Prod. J. 16(6):19-23.

16. West Coast Adhesive Manufacturers Association Technical Committee. 1970. Accelerated aging of phenolic-resin bonded particleboard. For. Prod. J. 20(10):26-27.

Table 1.--DESCRIPTION OF 1/2-INCH BOARD MATERIALS.

Material	Species	Flake	Board Weight	Phenolic Resin	Wax	Press Conditions: Press Temperature	Press Conditions: Time to Thickness	Press Conditions: Press Time
		In.	Pcf	%	%	°F	Min	Min
A FPL flakeboard	Douglas-fir	0.015 x 1 random width	40	6	1	350	1	10
B FPL residue mix (15-70-15 weight)	Douglas-fir	faces 0.02 x 1 x 2 disk core 0.05 x 2 random width ring	40	5	1	350	1	10
C Commercial waferboard	Aspen			Yes				
L FPL residue mix (15-70-15)	Douglas-fir	faces 0.02 x 1 x 2 disk core 0.05 x 2 random width ring	44	5	1	350	1	10
0 FPL residue mix (15-70-15) fines removed	Douglas-fir	faces 0.02 x 1 x 2 disk core 0.05 x 2 random width ring	41	5	1	350	1	10
P Commercial plywood	Douglas-fir			Yes				
W Heartwood	Douglas-fir							
X Sapwood	Southern pine							

Table 2.--PROPERTIES OF UNEXPOSED MATERIALS.

Material	Specific Gravity	Thickness	Bending Strength[1]	Bending Stiffness[1]	Internal Bond[1]
		In.	Psi	Thousand psi	Psi
A FPL flake	0.64	0.51	4,927 (760)	677 (70)	164 (20)
B FPL residue flake	.61	.52	3,799 (650)	608 (57)	83 (21)
C Commercial wafer	.60	.53	2,658 (335)	508 (34)	69 (12)
I FPL residue flake	.63	.52	4,588 (1,117)	711 (104)	107 (16)
0 FPL residue flake	.62	.52	4,886 (667)	670 (26)	125 (21)
P Plywood	.47	.48	6,663 (915)	965 (180)	127 (24)
W DF wood	.48	.50	14,346 (1,900)	2,007 (276)	372 (66)
X SP wood	.52	.50	16,176 (1.937)	1,990 (366)	612 (89)

[1]First value is mean for 10 specimens tested for materials A, B, C, P, W, and X, and for 5 specimens for L and 0. Value in parentheses is standard deviation.

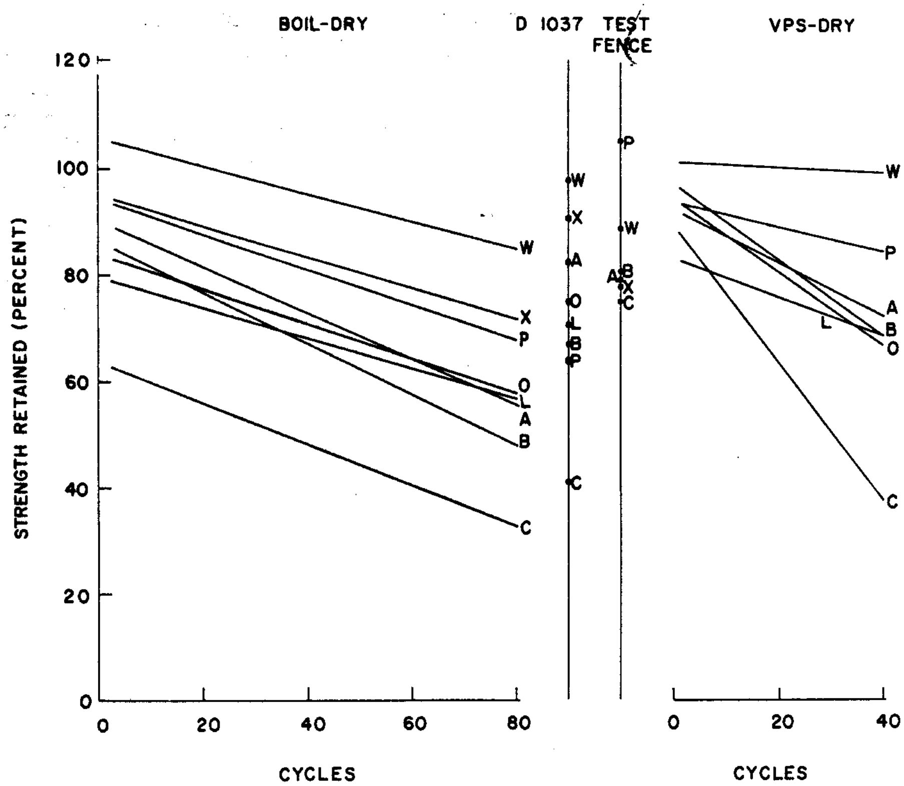


Figure 1.--Bending strength of material. A, B, C, L, and 0 are flakeboards P is plywood, and W, X are from solid wood (see Table 1 for identification).

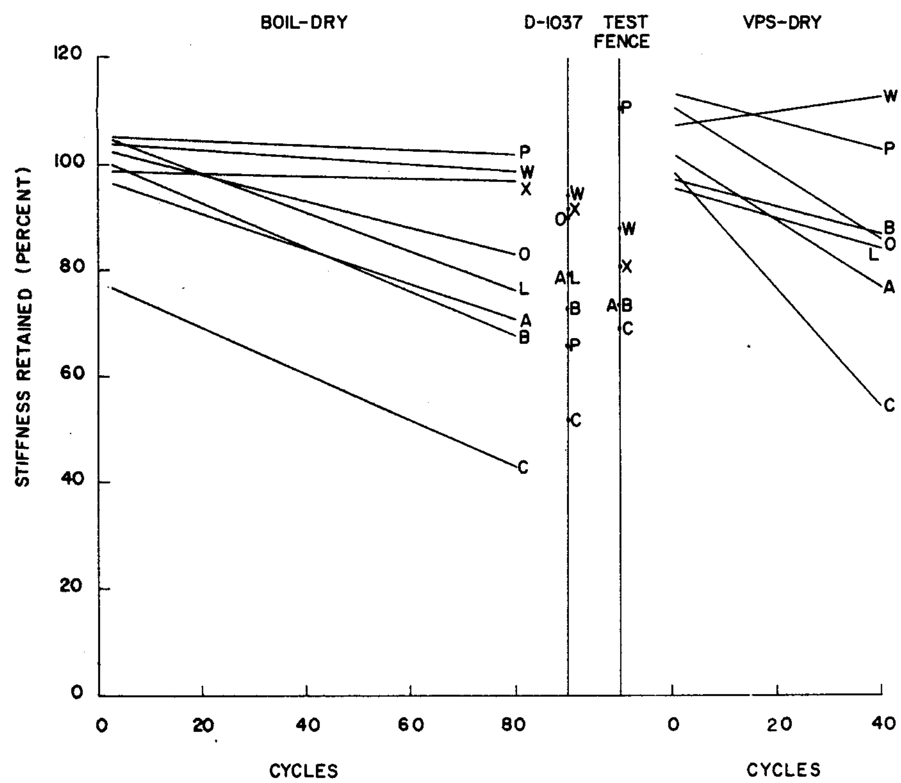


Figure 2.--Bending stiffness of material. A, B, C, L, and 0 are flakeboards, P is plywood, and W, X are from solid wood.

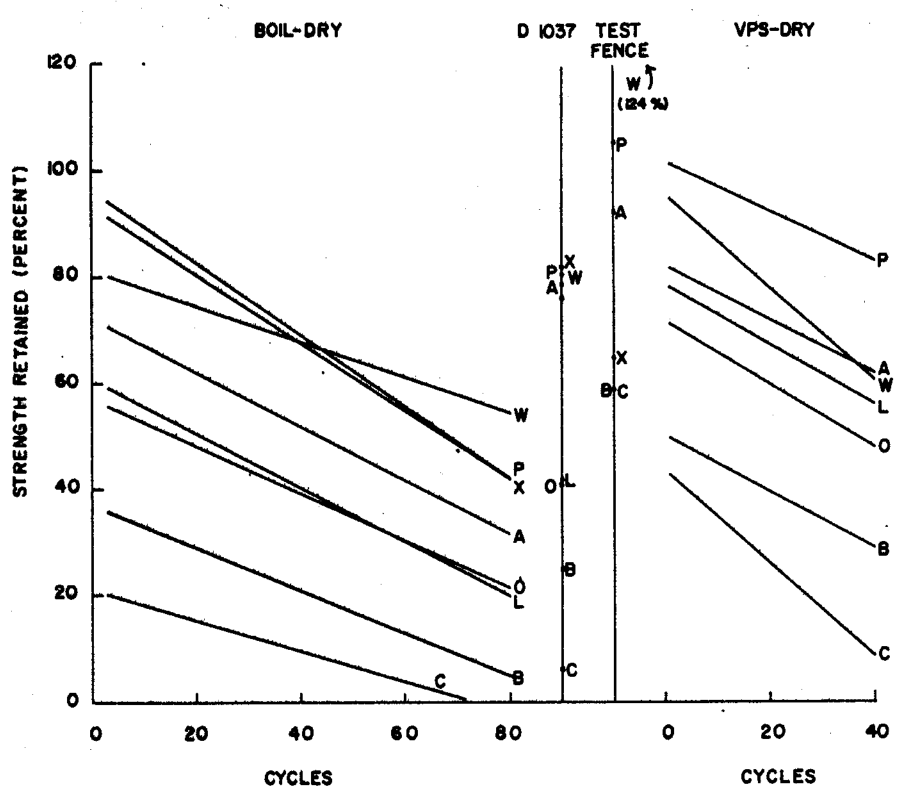


Figure 3.--Internal bond of materials. A, B, C, L, and O are flakeboards, P is plywood, and W, X are from solid wood.

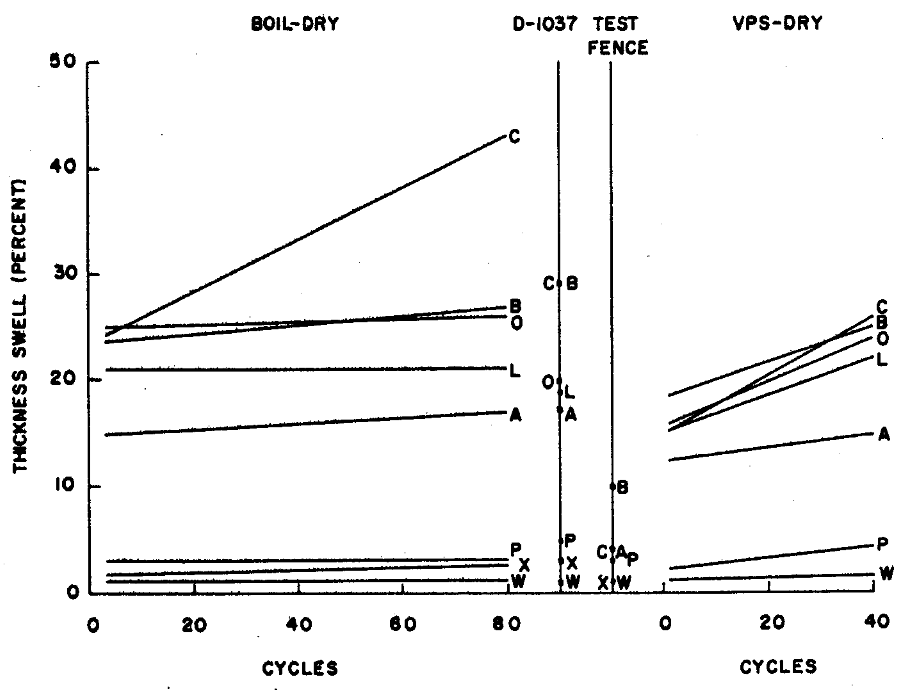
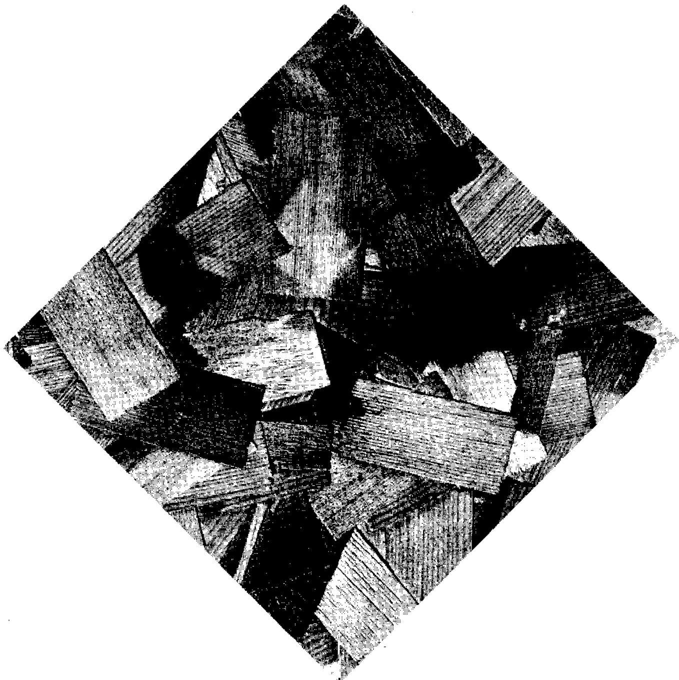


Figure 4.--Thickness swell of material. A, B, C, L, and O are flakeboards, P is plywood, and W, X are from solid wood.



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