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ADHESIVE TESTING PROCEDURES AND BONDING STRENGTH TESTING EQUIPMENT[1]

by

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Contents

1. Testing of glues

Tests on uncured glues can be made according to various physical and chemical procedures. Such tests are carried out either by glue manufacturers or by users to guarantee constant quality of the products. Many of these tests are standardized, whereas others are applied only by individual manufacturers or users for testing special properties with regard to their own requirements. Some of these tests can be used for all kinds of glues, others only for a special type of glue or a certain group of glues.

1.1 Testing of glue viscosity

Viscosity is the most important property and of fundamental importance to practice. For this reason measurements of viscosity were subject of intensive studies which led to numerous testing methods.

For routine control and comparison measurements are carried out with rather simple testing equipment. In general it is not necessary to evaluate the absolute value of viscosity; to compare different products it is sufficient to measure the glue viscosity by the same method, i.e. to evaluate an equivalent viscosity.

1.1.1 Flow cup viscosimeter

The flow cup viscosimeter is small and cheap, but it is only suitable for adhesives with low viscosity (Fig. 1a). Viscosity is quantified by the time a certain quantity of glue needs to pass through a nozzle of a certain diameter. For nozzles diameters of 2, 4, 6 or 8 mm are generally used. The specified temperature for the glue and the surrounding air should be kept on a constant level. As glues of high viscosity, as for instance with filler additives, do not efflux constantly but tend to drop-forming flow cup viscosimeters are not suitable for general glue-testing.

1.1.2 Falling ball type viscosimeter

The viscosity of the glue is calculated from the time taken by a steel ball to fall through a certain distance in a glass-tube filled with glue. Equipment of this type is well suited for measuring high viscosity glues; but with simple glass-tubes only transparent liquids can be measured. With more expensive equipment it is possible to measure the drop time by electronic measuring device. With this testing equipment also testing of opaque liquids is possible. During the tests special care should be taken that the steel ball does not touch the tube wall and a constant temperature is kept. Measuring errors are rather small after training; when using calibrated testing equipment absolute viscosity can easily be calculated in centipoise from falling time.

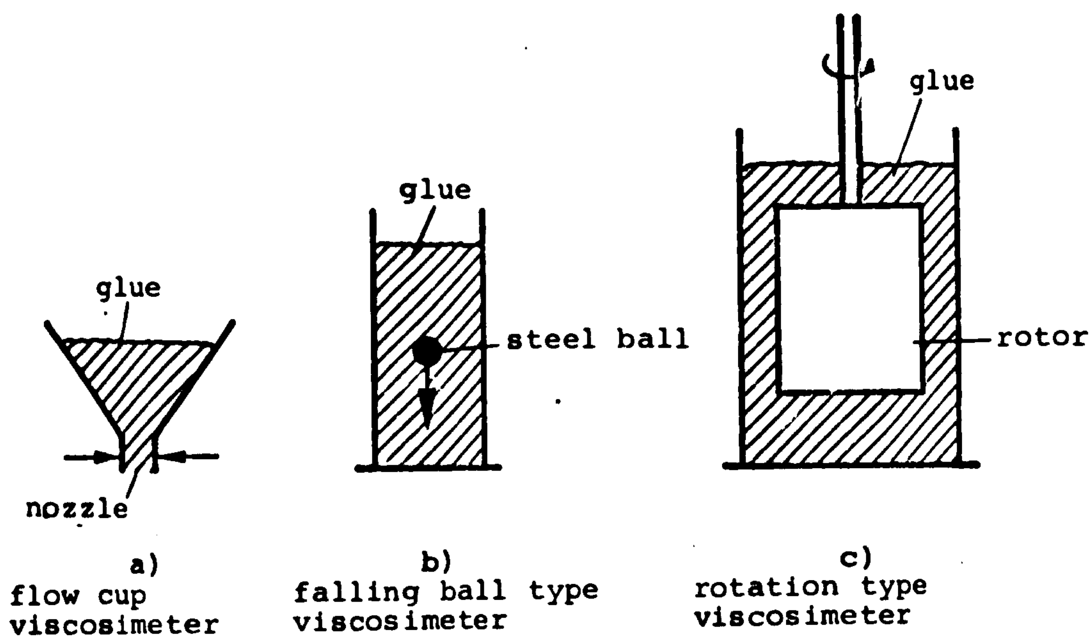


Fig. 1: Normal equipment for the determination of glue viscosity (diagrammic)

1.1.3 Rotation type viscosimeter

With the rotation type viscosimeter (Fig. 1c) in principle the frictional force of a cylinder rotating in the glue is determined. When using calibrated measuring equipment the results can easily be converted into centipoise. Rotation type viscosimeters are rather expensive and complicated and require well trained operators. This type of viscosimeter is used only for precise viscosity measurements; it is suitable for liquids of almost any viscosity.

Like all kinds of viscosity measurements the rotation type viscosimeter requires an accurate testing temperature (in general 20 °C) as viscosity depends on the temperature to a great extend (Fig. 2). If this dependance on temperature is not considered, it is possible that, during the cold season of the year, a correction of viscosity is performed by adding water to the liquid glue. The result is that in normal temperatures (processing temperatures) the glues are of a viscosity which is too low.

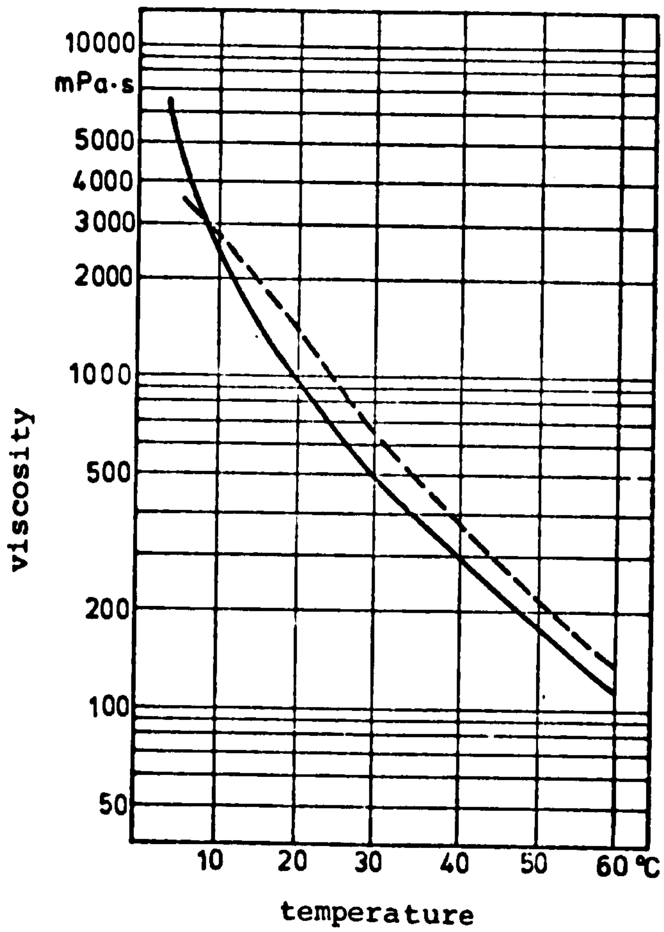


Fig. 2: Dependence of the viscosity upon the glue temperature

———— Urea-formaldehyde

-------- Phenolic-formaldehyde

1.1.4 The importance of viscosity for wood bonding

The viscosity of a glue particularly influences the spread on wood. With roll spreaders the rate of glue per surface unit will be too high, if viscosity is too high.
With curtain coating spreaders the coat will not be uniform, either too low or too high.

With blenders in particleboard production glue will not properly be sprayed; the consequence is a coat which is not uniform and generally too low.

The quality of the glue-bonds is influenced by viscosity too. When viscosity is low, the absorption on wood will be high which results in rather low bond qualities, especially with thick glue-lines or low pressure. When viscosity is too high the penetration of the glue into the wood structure is not sufficient and there is no interlinking between the glue and the wood surface (mechanical adhesion) which also results in low bond qualities.

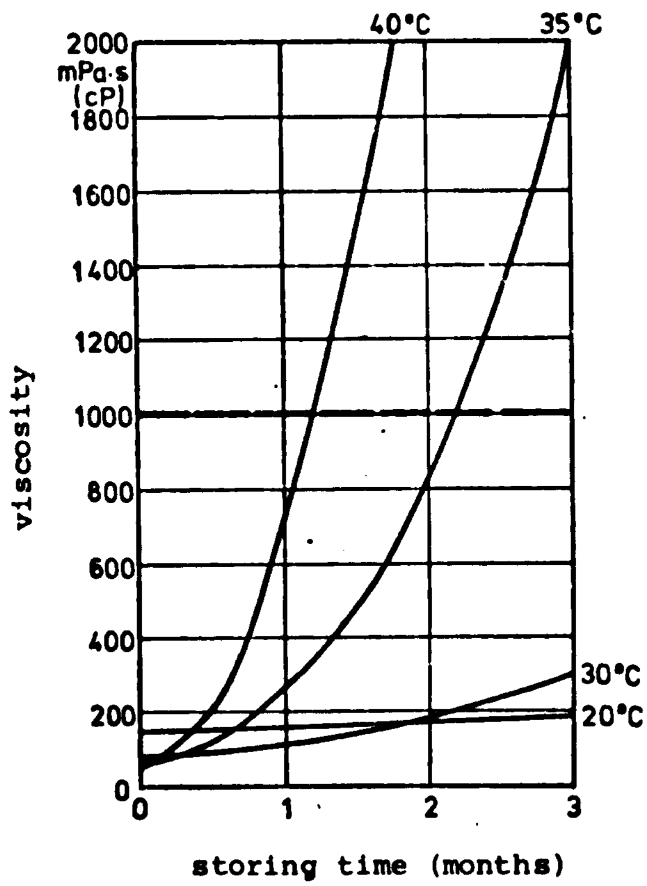


Fig. 3: Dependence of the viscosity of phenolic glues upon the storing time

When bonding wood the viscosity should be kept within a rather small range. Deviations have negative influence on the bond quality. Most of the condensation resins used in wood-working industries, like melamine- or phenolic-formaldehyde type adhesives, are condensating during storage. The consequence is an increase in viscosity. Therefore, this type of adhesives can only be stored for a limited time. The duration is largely dependent on the temperature during storage.

1.2 Gelation time tests

Gelation time is defined as the time which is needed for the transition from the liquid to the gel-state after the addition of the hardener. For thermo-setting adhesives the gelation time is calculated from the point when the glue is mixed with water. Gelation time depends on temperature as well as on the type and quantity of hardener. Gelation time measurements are carried out mostly at temperatures up to 40 °C according to the following procedure:

For instance, a plastic cup is filled with 50 g of glue and heated up by a thermostat to the proper testing temperature (e.g. 20 °C). Then the necessary amount of hardener (which has been heated up to the testing temperature) is added. Glue and hardener are well mixed and after certain time intervals the glue-hardener-mix is well stirred with a glass-stirrer. The transition point is reached when the glue sample is not behaving stringy when lifting the glass-stirrer, but is breaking rubber-like.

For temperatures above 40 °C in general gelation time is relatively short. The glue-hardener-mix should be stirred continuously in this case to follow the transition into the gelation state.

The determination of the gelation time can be carried out with relatively cheap and simple device but requires well trained operators. CLAD (1961) describes an automatic testing device for routine viscosity determination. As the transition into the gelation state is related to a rather strong increase in viscosity, a rotor in the glue-hardener-mix will be relayed when the gelation transition takes place. This automatic "gelation timer" is rather expensive, but mostly such high accuracy is not necessary.

1.3 Pot-life

The pot-life of a glue is usually defined as the duration time between the addition of the hardener or catalyst and the point when reaching the maximum suitable viscosity for application. In this case the pot-life can be a few minutes up to many hours, which depends on kind and quantity of hardener and on the temperatire, but is always shorter than the gelation time for the same temperature. The pot-life is influenced to a great extend by external influences, for example by the evaporation of water when using glue spreaders or by addition of extenders and fillers. Pot-life is normally specified by the manufacturer for different temperatures. For different spreading techniques there is a variation in maximum suitable viscosity (Fig. 4, Fig. 5).

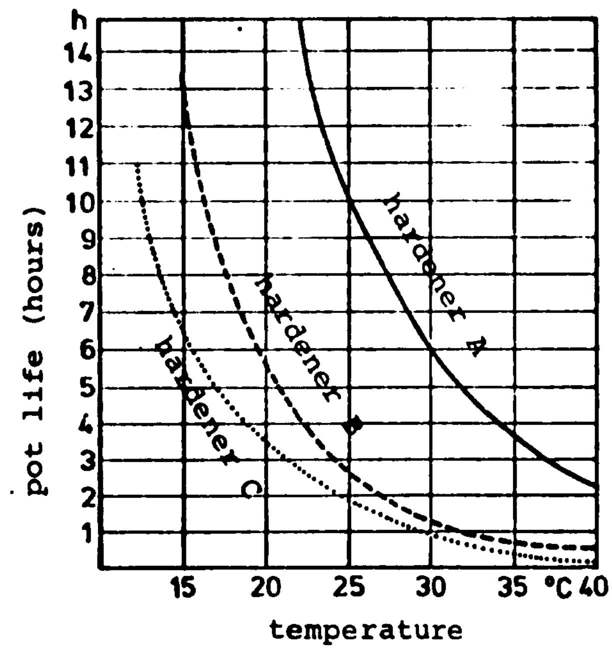


Fig. 4: Pot life of urea-formaldehyde resin with different hardeners

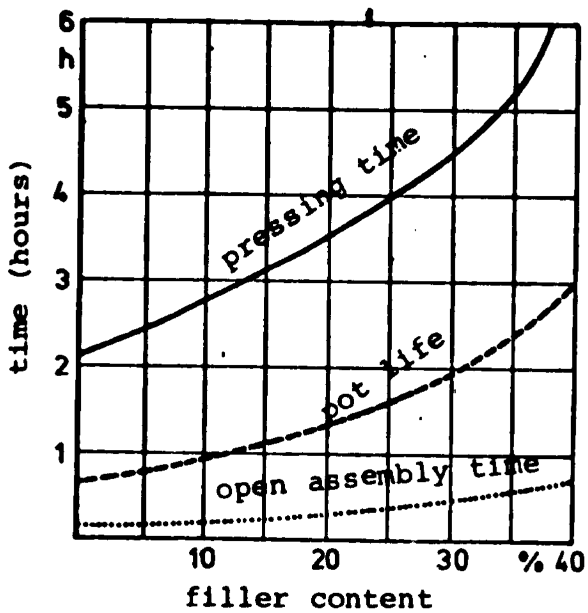


Fig. 5: Pressing time, pot life and open assembly time of urea-formaldehyde resin with different filler contents

1.4 Check-up of pH-values

The cure of most wood glues is a physico-chemical reaction, which can be controlled by the pH-value. For this purpose a certain pH-value can be adjusted or the pH-value can be altered by temperature and different kinds of hardener. To get information on the curing time the pH-value of liquid adhesives or the pH-value of the cured film must be checked. The values for liquid glues are generally between pH = 2.5 and pH = 11, lower and higher pH-values are causing wood fibre destruction which results in lower bond quality.

For routine control measurements with pH-indicator paper is sufficient. By this method the pH-value can be determined with an accuracy of pH ± 0.5. More precise results can be obtained by electric measuring equipment.

1.5 Solid content of glues

As the solid content of glues is rather important for the bond quality, every shipment has to be checked. For the determination of solid content 2 to 3 g of glue are weighed in a petri or similar dish. For this purpose the glue should cover the dish with a thin layer. The glue is oven dried to constant weight with a temperature in the range of 140 - 150 °C. Later the dry glue is cooled in a desiccator and the solid content can be determined. The solid content is defined in per cent of liquid gluemix.

1.6 Other tests

Apart from the above mentioned tests other glue tests can be conducted as for instance the determination of the flash point, the alkali content, the ash-content, the water stability, the content of free formaldehyde and the content of NCO-groups.
Sometimes fillers and extenders (e.g. flour) are tested by glue users and manufacturers. In this connection the granular size, the water absorption, the swelling behaviour, the pH-value of a solution and the ash-content are very important. A detailed description of these test methods should not be a subject of this study.

2. Testing of bonded wood products (mechanical tests)

2.1 General information

The utilization of bonded wood products and building elements, like plywood, particleboard gluelam or other elements, is decisively affected by the glue-line quality. Therefore, particular regulations for the estimation of bonding strength are existing in different countries.
Some of the regulations might be:

Product standards,
commercial standards,
industry standards,
national standards,
military standards.

Apart from minimum requirements on the bonding strength, the testing methods and the necessary quantity of samples are prescribed in these regulations. As an example the American,

British, and German standards are listed. (Appendix: Summary of the German, British, and American standards for bonded wood product.).
These standards are rather differentiated according to the different fields of application of wood species and adhesives.
Thus, the national standards are often not systemized.
, because standards for special fields are not available.

The quality of wood bonds depends on the kind of joining, the the wood specie used, the properties of the glue, and the bonding conditions. These influences should be considered when testing adhesives. . Therefore, many different specimens and testing methods are being applied. As generally wood specimens are used for testing wood glues, rather great difficulties might arise. The variability of wood properties, like density and strength, have an additional influence on the variation of the results when testing the bond quality.

2.2 Evaluation of bond quality

Until now the quality of bonded wood products has only been determined by destructive tests. In this connection two different methods are distinguished:
- The determination of bonding strength and
- the determination of wood failure.

In this case the determination of wood failure is a comparing estimation of bond and wood strength.
For the evaluation of bonding strength three different testing methods are applied:
- Tension loading perpendicular to the glue-line,
- cleave tests,
- shearing tests (tension and compression).

Absolute results which can easily be interpreted are the advantage of strength tests. However, the variation of the test results caused by different wood species used are of great disadvantage, thus special quality levels for each wood species are required in the standards.
Of further influence is the specimen shape, because unequivocal tension or shearing stresses are rather seldom, especially when using easy to handle specimens. For this reason in some standards the additional determination of wood failure was adopted. When testing the bond strength of plywood in France or hardwood bond strength in the U.S.A. according to the relevant specifications, wood failure is determined in addition to the strength properties. For bonds of coniferous wood products the failure only characterises the bond quality in the U.S.A.
In Germany and Great Britain in addition to strength tests knife tests are carried out with the purpose to determine wood failure.
The determination of wood failure in tension tests is very important according to the American standards. Wood failure

percentage depends on the interlinking of the glue and the wood to a great extent and can only be influenced to a limited extent by fillers. It can only be considered as a criteria for the proportion of mechanical adhesion compared to the total bond strength. The proportion of mechanical adhesion contributes with 20 - 30 per cent to the total bonding strength. Through specific adhesion a rather high strength of the glue-lines is possible which depends on the type of glue, the wood species and bonding conditions. Thus a direct relation of wood failure and shearing strength does not exist. For example, if high solid content resins are used for dense wood species the glue does not penetrate the wood structure to the necessary extent to insure a sufficient interlinking between glue and fibre structure. Consequently the percentage of wood failure and mechanical adhesion is lower than of species with good penetrability for the glue. Therefore the evaluation of the bonding strength through wood failure only is impossible. For a better evaluation it is necessary to determine wood failure as well as bonding strength.

Special difficulties arise when determining the bonding quality of plywood. In this case where tangential plains are bonded it would a a nonsense to apply the wood failure percentage or the shearing strength as criteria for the bond quality. Differences in wood density and surface quality are of rather great influence and the shearing strength as well as wood failure are effected by the weakest material in the tangential plain, e.g. by summer wood. The definition of the wood failure is rather difficult. The plywood over its cross-section consists of a glue coat, a layer of resin soaked wood and unsoaked wood (Fig.6).

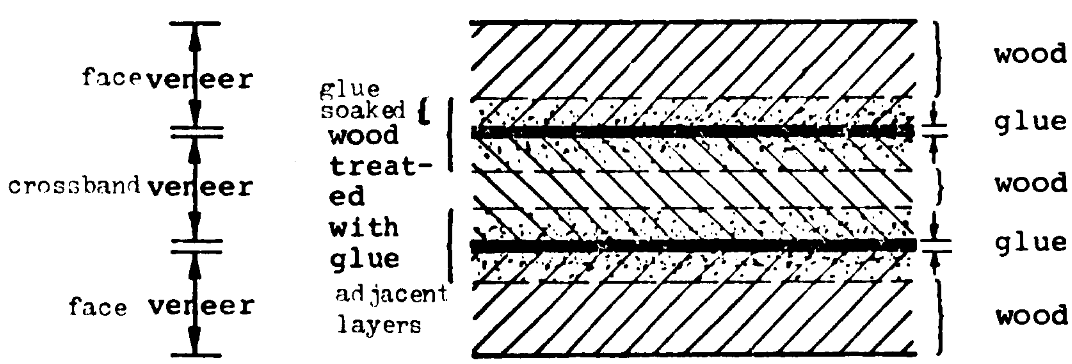


Fig. 6: The different layers of plywood

After testing the shearing strength the percentage of the glue coat or the percentage of resin soaked wood (the percentage of unsoaked layer) is determined in steps of 5 %. This estimation requires a well trained staff and often it can happen that the same glue-lines are classified in a different way by different persons. Therefore, CHUDZINSKI (1976) proposed the following method for a better classification of the wood failure:
The surface of fracture is covered by a net of for instance 100 divisions and the number of sections indicating wood failure are counted. This way is rather time consuming and it should be checked whether a net of only 20 divisions (this means 5 % of the fracture surface per division) would fulfil the required accuracy. This would be of great advantage when training laboratory staff.
The results of the evaluation for each specimen as well as the mean values of all samples are recorded in steps of 5 %. In this connection CHUDZINSKI (1976) classifies the quality of glue-lines in 4 groups:

75 - 100 % wood failure - very good bonds
50 - 75 % wood failure - good bonds
25 - 50 % wood failure - sufficient bonds
0 - 25 % wood failure - unsatisfactory bonds

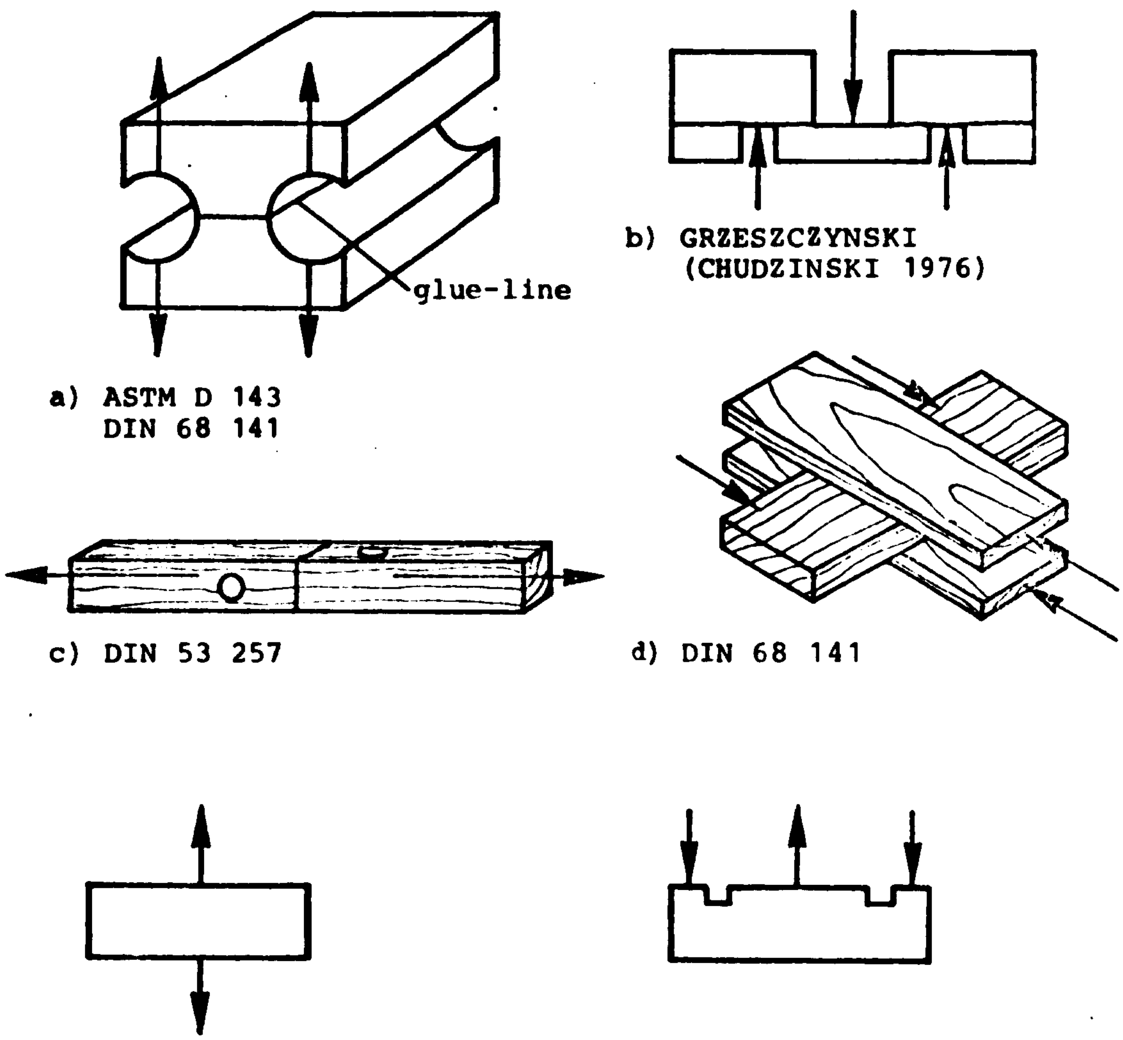
2.3 Specimens for testing bond strength

For testing glue bonds the standard testing regulations of the different countries show a variety of different specimens. In this connection the most important specimens, their advantages and disadvantages shall be explained briefly.

2.3.1 Specimens for tension tests

For the determination of the tension strength of bonds perpendicular to the glue-line different specimens are used (Fig. 7).
As a consequence of the relatively complex stress distribution in all specimens the real tension strength perpendicular to the glue-line cannot be evaluated, but the determined strength properties are lower. One disadvantage is the rather difficult preparation of some specimens, like ASTM D 143 (Fig. 7a) or Fig. 7b specimens . The sample for transverse tension testing according to Fig. 7e is used only for internal tension tests of particleboards . Testing of plain bonds, like plywood, is not possible because in most cases it cannot be assured that the bonds between the surface of the specimens and the clamping device is better than the glue-line to be tested. A modification is the specimen according to DIN 53 266 (Fig. 7f) for the determination of the resistance against ply delamination, like veneers or wood based panels. The strength of the glue-line can relatively well be determined by these tests. The specimen for internal tension

testing according to DIN 68 141 (Fig.7d) is not suitable for general testing of bonds because of its rather large sizes. It is used only for testing bonds in gluelam beam production.



a) ASTM D 143
DIN 68 141

b) GRZESZCZYNSKI
(CHUDZINSKI 1976)

c) DIN 53 257

d) DIN 68 141

e) DIN 52 365

f) DIN 52 366

Fig. 7: Specimens for testing tension strength of glue-bonds when load is applied perpendicular to the glue-line

Testing cross-grained wood specimens according to DIN 53 257 (Fig. 7c) gives relatively high values for the ultimate stress of bonds in tension because of its small dimensions. Therefore, this cross-grained wood specimen is used in Germany only for testing the suitability of protein glues. In general specimens for tension testing (loading perpendicular to the glue-line) of glue bonds are rather unimportant.

2.3.2 Specimens for cleave tests

CHUDZINSKI (1976) describes a specimen for cleavage testing which was developed especially for structural laminated beams (Fig. 8). Relatively large dimensions and difficult preparation together with undefined stress distributions might be the reason why this type of specimen is seldom used. Especially specimens for cleavage testing of solid wood are not useful for testing the glue bonding strength, because the influence of wood inhomogenities is rather apparent in this connection.

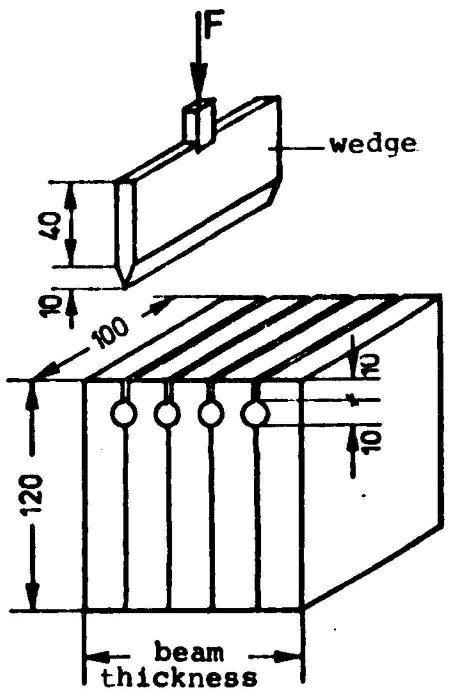


Fig. 8: Sample for testing glue-lines by cleavage test (CHUDZINSKI 1976)

2.3.3 Samples for shearing tests

With regard to shearing tests two methods should be distinguished: tension and compression loading. Both are taking the shearing strength (ultimate load to shearing area) and in some cases wood failure percentage in the glue-line as a measure for the bond quality.

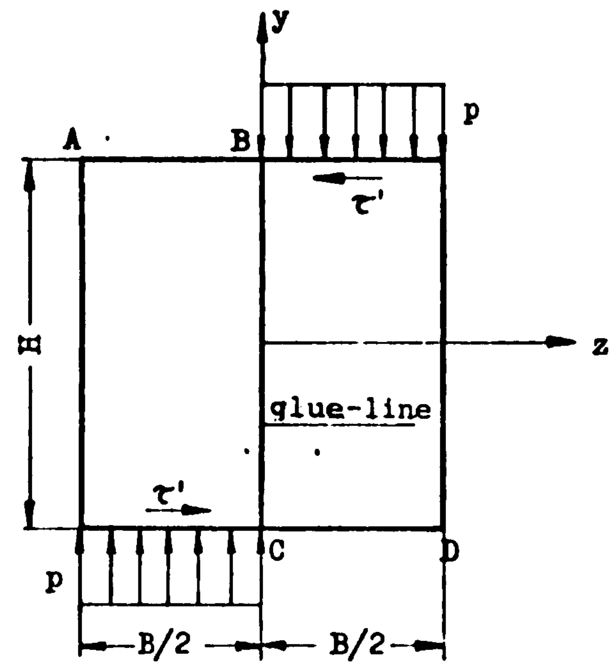
2.3.3.1 Specimens for shear tests in compression

Compression test shear specimens of different shapes are used to determine the shearing strength of solid wood (like BS 373 - 1938, ASTM D 143-52, DIN 52 187). The suitability of these specimens is rather different because of the complex stress distribution in the shearing plain and because of difficulties when testing and preparing the samples.

Cube shear block specimens

The cube type shear block (Fig. 9) is the simplest of all kinds of specimens. The cube has an edge length of 50 mm according to the British standard BS 373 - 1938. The way of loading is demonstrated in Fig. 9. NISKANEN (1955, 1957) has examined the stress distribution for cube type shear block specimens in the shearing plain. He takes a uniform loading P for the total area of bearing AB and CD respectively.

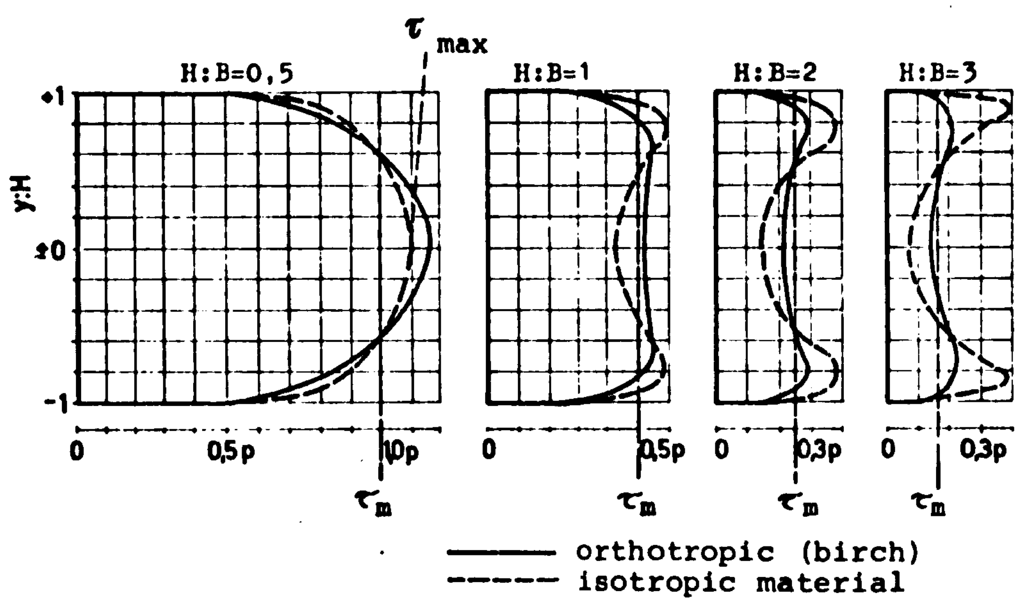
This block type shear specimen, although not standardized, was introduced into testing of glue bonds wood based materials. NOACK and SCHWAB (1972) propose a square shear specimen of the size of 5 x 5 cm for particleboard testing. GRESSEL (1975) follows up this proposal with a height of 2.5 cm for thin particleboards. NOACK and SCHWAB as well as GRESSEL found in agreement with KUFNER (1975) a strong correlation between shearing strength and ultimate internal strength of particleboards. In particleboard testing the ultimate internal strength is regarded as a good indicator for the bond quality. But this specimen is not well suited for routine tests because it is difficult to clamp in the testing machine. The use of shear type specimen means for this reason a remarkable simplification when testing particleboards.



Testing a cube shear block

p [kp/cm] = compression load per unit

τ' [kp/cm] = friction force per unit



H : B	0.5	1.0	2.0	3.0	
isotropic K ≈	1.18	1.16	1.5	2.4	$K = \frac{\tau_{max}}{\tau_m}$
orthotropic K ≈	1.10	1.08	1.14	1.4	

Fig. 9: Distribution of shearing stress for isotropic and orthotropic cube shear block in elastic range according to NISKANEN (1955)

Shear block according to ASTM D 143-52 (Fig. 10a)

This type of specimen is used in the U.S.A. for testing shearing strength of solid wood. If the glue-line is identical with the shearing plain it might be used for testing glue-lines strength too. Lately it could be observed that efforts have been made to introduce this type of specimen. The sizes of the specimens and the stress distributions in the shearing plain are shown in Fig. 10a and 10b. In comparison to the cube shear block specimen a rather ununiform stress distribution with peaks near the notches are remarkable. Therefore, this type of shear test specimen might not be suited for testing solid wood and glue-lines. More useful is the block type specimen.

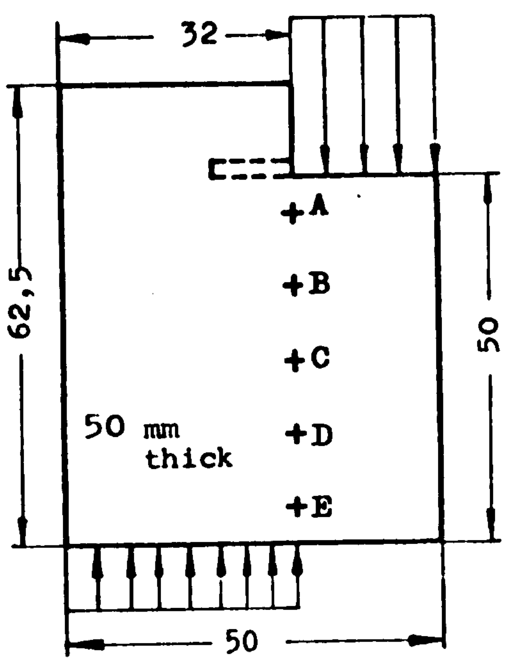
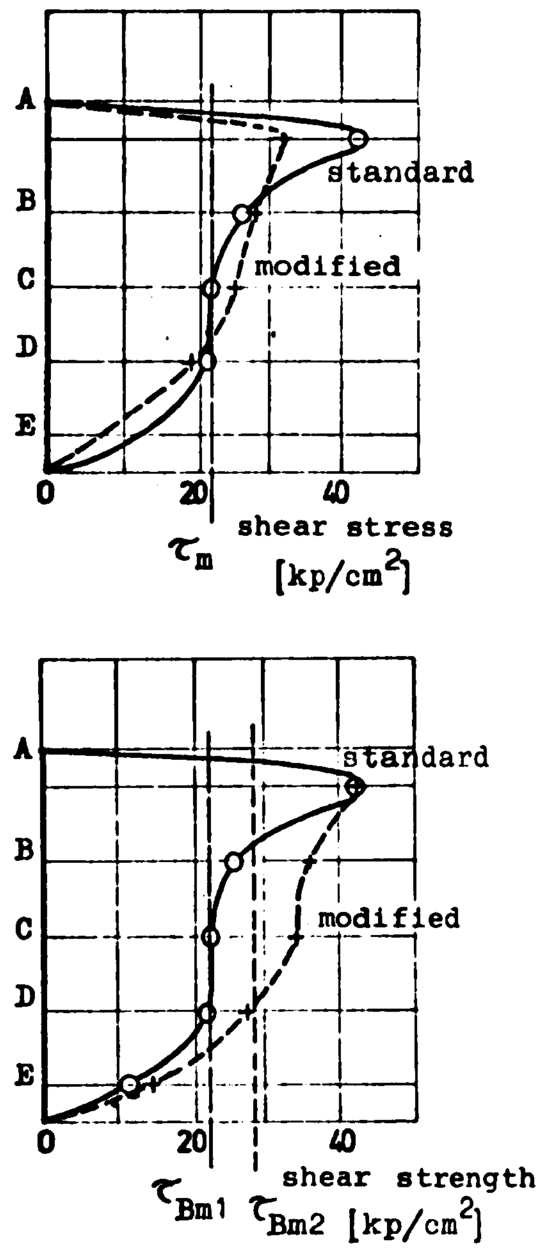


Fig. 10a: Normal shear block according to ASTM D 143-52 and modified shear specimen



$k_{standard}$ = 1.89

$k_{modified}$ = 1.43

Fig. 10b: Distribution of shearing stress for standard and modified specimens of Douglas fir in elastic range (above) and in the state of shear failure (below) according to RADCLIFFE and SUDDARTH (1955)

Shear block according to ASTM D 805 (Fig. 11)

This specimen is chosen according to ASTM: Testing of bond- The stress distributions, as for instance for the shear specimen according to ASTM D 143, has not yet been determined. However, YAVORSKI and CUNNINGHAM (1955) reported of stress concentrations in the notch area. On loading the specimen more heavy cracks were observed than in the centre. Shown that, besides the transverse strains of the glue-lines, also tension and compression stresses as well as the cleavage of the glue-line can be considered as additional loadings.

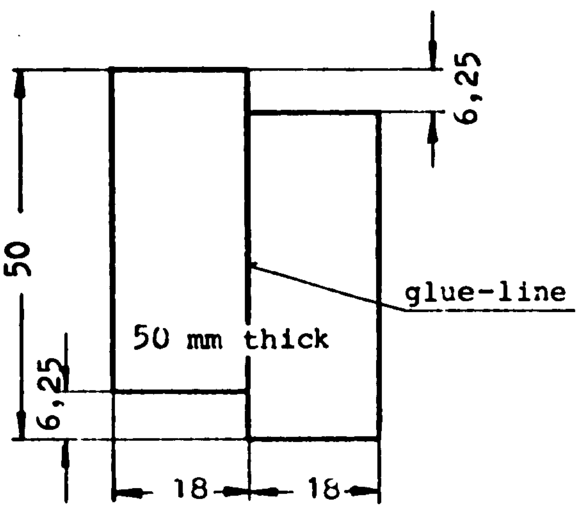


Fig. 11: Shear block according to ASTM D 805-52

In a lot of tests this shear specimen has proved to be useful for testing glues in compression. Preparing the specimen is relatively simple and above all can easily be tested in a special testing equipment (Fig. 12). However, care should be taken that the angle of the growth rings to the glue-line is about 45 - 75° (Fig. 13). If the angle is about 0°, which means that the tangential plain of the wood is glued, the samples mostly break in the summer wood which is the weakest area. If the angle is 90° the rays are parallel to the glue-line and the fracture is determined by the strength of the rays. Attention should be paid to the correct direction of the growth rings not only in testing glue-lines, but also in the manufacture of building elements, which are loaded perpendicular to the glue-line, as for instance laminated beams.

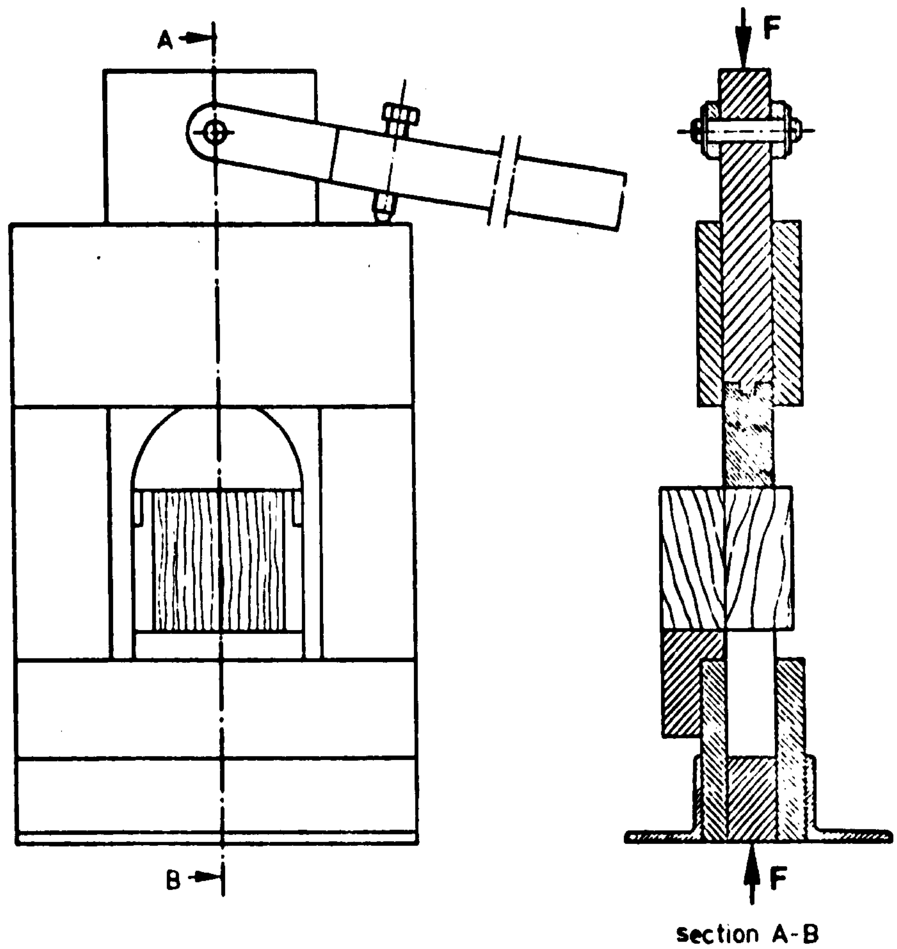


Fig. 12: Equipment for testing shear blocks according to ASTM

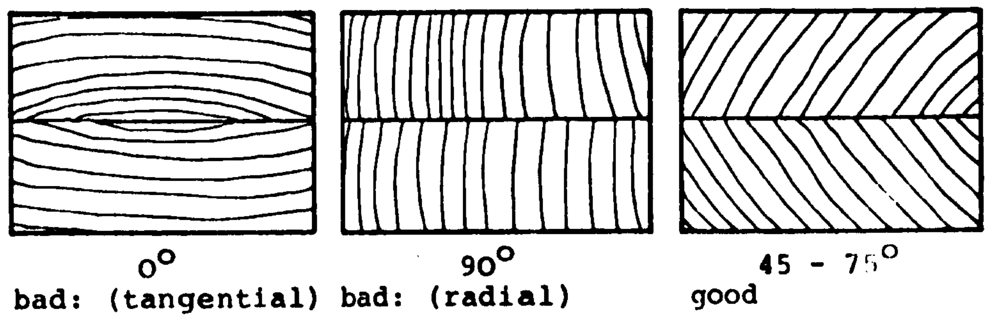


Fig. 13: Direction of growth rings and glue-lines in shear specimens

According to ASTM D 2559 a special type of specimen (the stair-step shear specimen) is prescribed for testing the glue-lines of structural laminated beams (Fig. 14a). The specimens are drawn in such a way that normally 5 glue-lines can be tested. As there are routine testings, the loading rate of 1.27 cm/min is significantly higher than in ASTM D 805, limited to 0.37 mm/min only. Besides testing of the bonding strength in the dry state this standard covers also testing of the bonding strength after accelerated aging of the glue-lines.

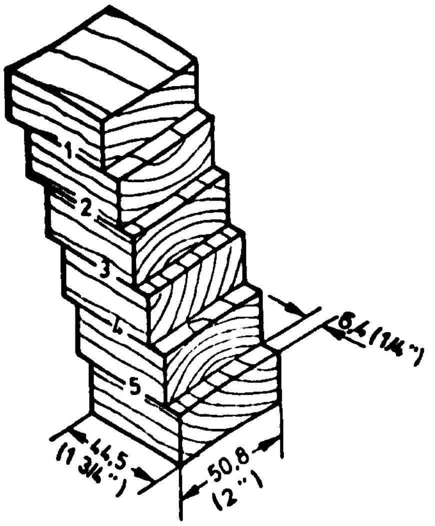


Fig. 14a: Stair-step specimen according to ASTM D 2559

A similar shear specimen is used for quality control in the European timber laminating industry (Fig. 14b). In this case from the cross cut end of each manufactured beam a slice is taken. The shearing strength of each glue-line is tested by a hand operated hydraulic press. In the standards minimum values for shearing strength are stated. If the values of shearing strength are below these minimum values the wood failure is considered as a further criteria.

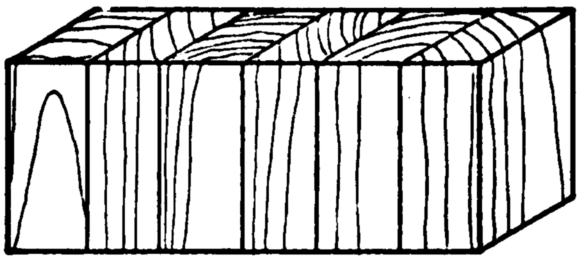


Fig. 14b: Specimen for quality control used in the German timber laminating industry

2.3.3.2 Specimens for shear tests in tension

Simple specimens for shear tests in tension are used to classify adhesives suitable for special kinds of application. Especially when testing the bonding strength in the wet state after different moisture treatments these specimens show good results. Various types of specimens are proposed by the different standards.
The American and British standards included simple specimens consisting of laminated veneers of 1.6 mm (Fig.15a).
DIN 53 254 specific the type of specimen shown in Fig. 15b.
If necessary special specimens for thicker glue-lines can be prepared (Fig. 15c). Thicker glue-lines are of special interest for wooden building constructions.

In general there is again the problem of ununiform stress distributions in the plain of the glue-line for overlapped and incised specimens for shear tests in tension. Therefore, the absolute shearing strength cannot be determined by these specimens, they are mostly used for comparison tests.

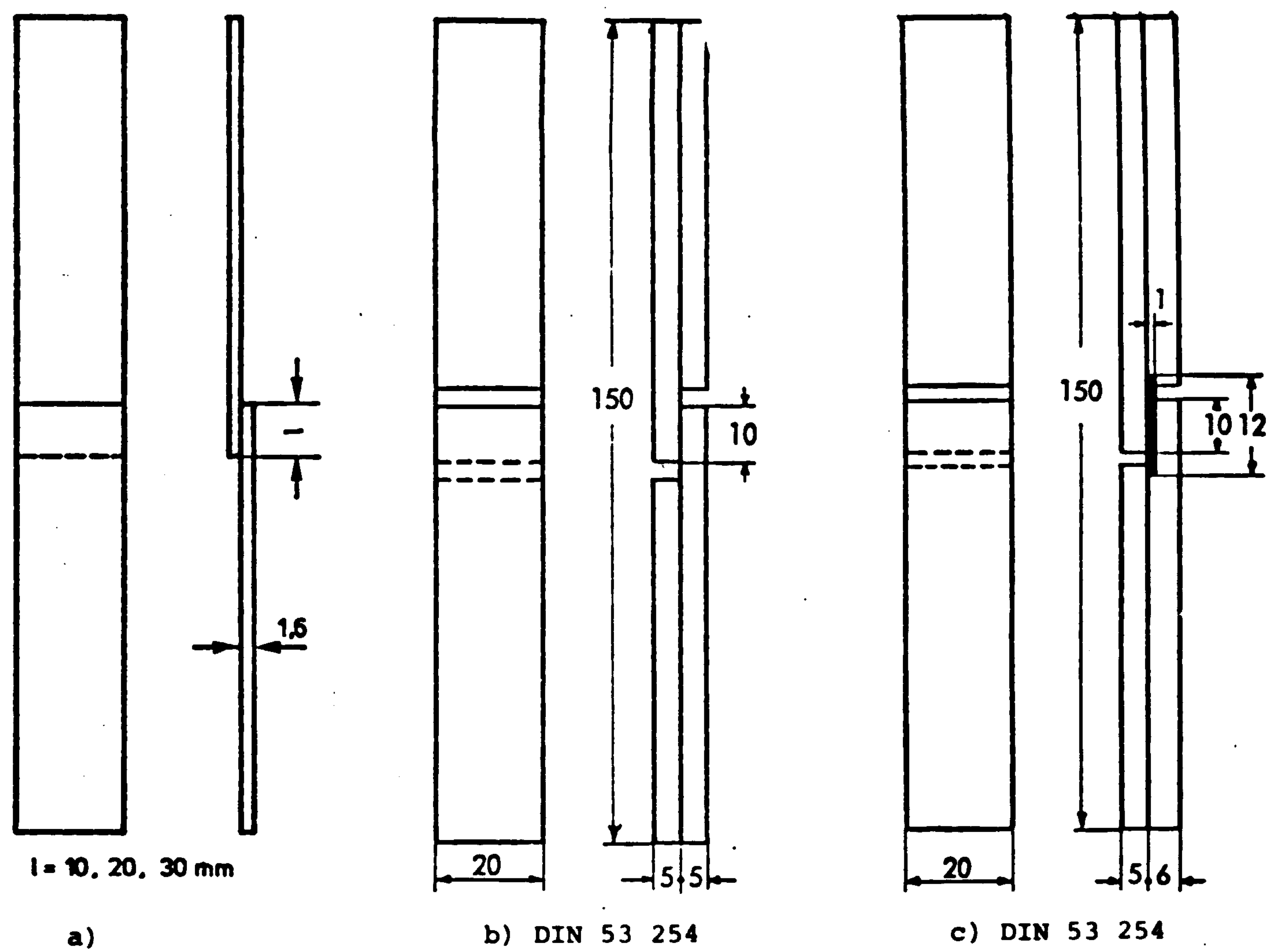


Fig. 15: Different specimens for lap shear tests

2.3.3.3 Specimens for testing plywood

In nearly all national standards the bonding strength of plywood is defined by lap shear tests (Fig. 16). The specimens are basically similar and nearly always consist of a width of 25 mm. There are only essential differences in the overlap region, which is 10 mm in Germany, 20 mm in France, and 25mm and 12.7 mm in Great Britain and the U.S.A. respectively. The longer the overlap region, the more important is the influence of the stress peaks in the notch-area and the smaller becomes the apparent shearing strength calculated from the ultimate strength and the shearing area, which is called the bonding strength.
When testing the influence of the type of specimen on the bonding strength the test results showed less varying values with small overlapping areas than with the long ones. When applying small overlapping areas the influence of wood anisotropy is eliminated and stress peaks are reduced. Similar tests were carried out to show the influence of using different wood species. Beech has proven to be suited best.

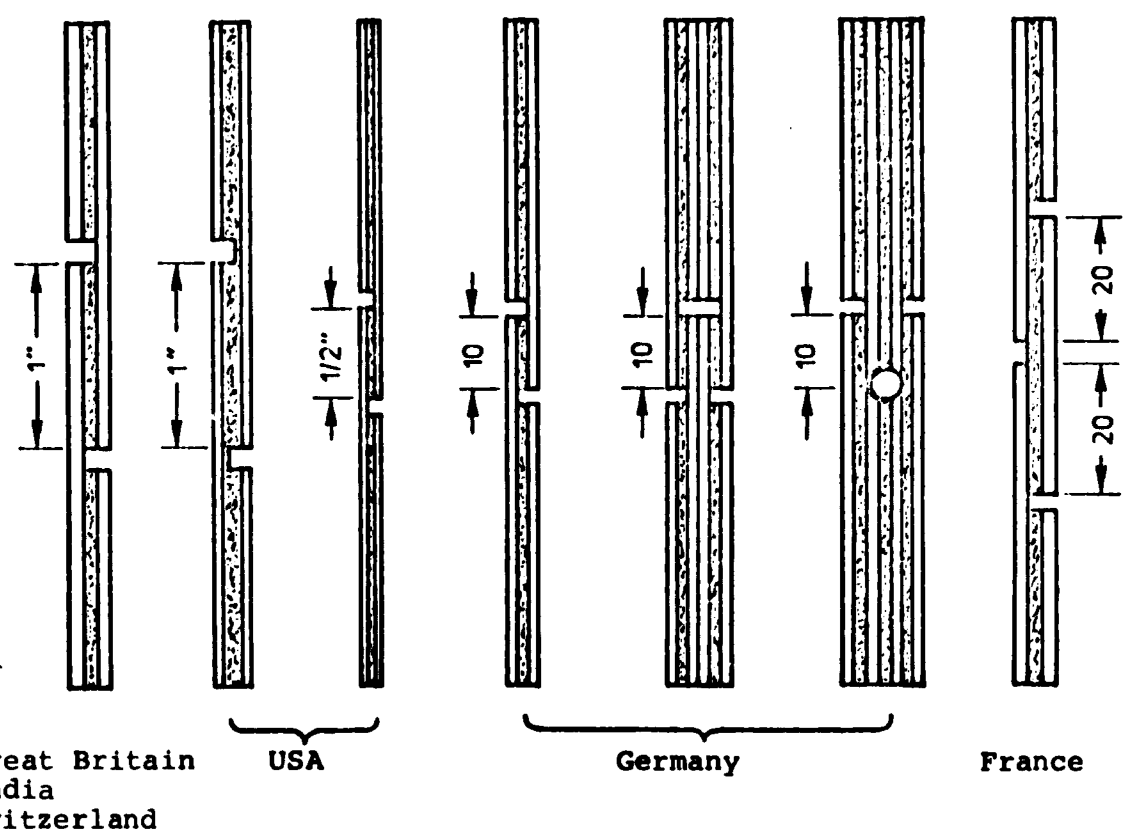


Fig. 16: Different specimens for testing plywood

The amount of random sampling when testing glues for plywood products can only be fixed by statistical methods. It is apparent that the variation between the mean values of the panels is bigger than the variation within the panels. This inhomogenity is caused by the wood species.
Because of this inhomogenity a lot of panels must be examined. The German standard DIN 68 705 , therefore, specifies to take from 12 panels 10 specimens each, which means 120 specimens. Another difficulty is caused by the fact that the veneers used for the plywood production contain peeling checks in different amounts. When drawing samples from the panels care should be taken that the peeling checks are either close or open when testing (Fig. 17).

peeling ckecks close

peeling checks open

Fig. 17: The importance of peeling checks in lap shear specimens

Half of the samples should contain cutting checks which are open whereas the other half should contain those which are close. A possibility to achieve this is given in ASTM D 805. For the evaluation of the bonding strength of plywood bond normally the shearing strength is used. The standards of the different countries give minimum values for the bond strength in the dry as well as in the wet state after a moisture or temperature treatment. In addition to the shearing strength the wood failure percentage is used for the evaluation if the bonding strength in some countries, as for instance Great Britain and the U.S.A.

2.3.3.4 Importance of testing speed for the strength of glue-lines

For all shearing tests of glue-lines either in tension or in compression the rupture of the specimens is induced after a certain time. The resultant rate of loading is fixed in some national standards.
For example for plywood testing in tension:

Germany	100 kp/cm^2 loading speed per minute and cm^2 glue-line
Great Britain	6 or 12 mm speed per minute 136 or 272 kp loading speed per minute
U.S.A.	0,6 mm speed per minute
Norway	in practice 10 mm speed per minute
Poland	in practice 10 - 15 mm speed per minute

In testing solid wood it is well known that different rates of loading cause different test results. The influence of the testing time on the strength is shown in the American standard ASTM D 2555 (Fig. 18).

The diagram of bending strength (ASTM D 2555) shows that continuous application of 60 percent of the load is required to break the specimens on a universal testing machine in 3 to 5 minutes would result in rupture in 50 years. Theory as well as experiment prove that the load required for rupture for viscoelastic materials (like wood or adhesives) is a linear function of the time. Similar behaviour curves, like the bending strength of wood, should be obtained for adhesives being evaluated or for other building materials.

Especially for elasto-plastic adhesives, like polyvenylacetat or hot melts, a rather strong dependence of the test results on the loading time is well known. Therefore, bond strengths measured in the U.S.A. with a rate of loading of 0.6 mm per minute can hardly be compared with results from Norway measured with a loading rate of 10 mm per minute. However, condensation type resins are rather insentitive in this connection. Detailed statements on the influence of the rate of loading are not possible because of dependency on the type of glue, glue-modification, amount of fillers, degree of condensation, and other factors.
SIMON (1975) pointed out that for high rates of loading adhesion fractures in the layer between glue and wood are more often than for low rates of loading, where the tendency for cohesion fractures is predominant. This is apparent especially when elasto plastic glues are used. Consequently low rate loading would result in lower wood failure percentages than in high rate loading. Because of the above

mentioned influence on the ultimate strength as well as on the percentage of wood failure the rate of loading has to be observed precisely

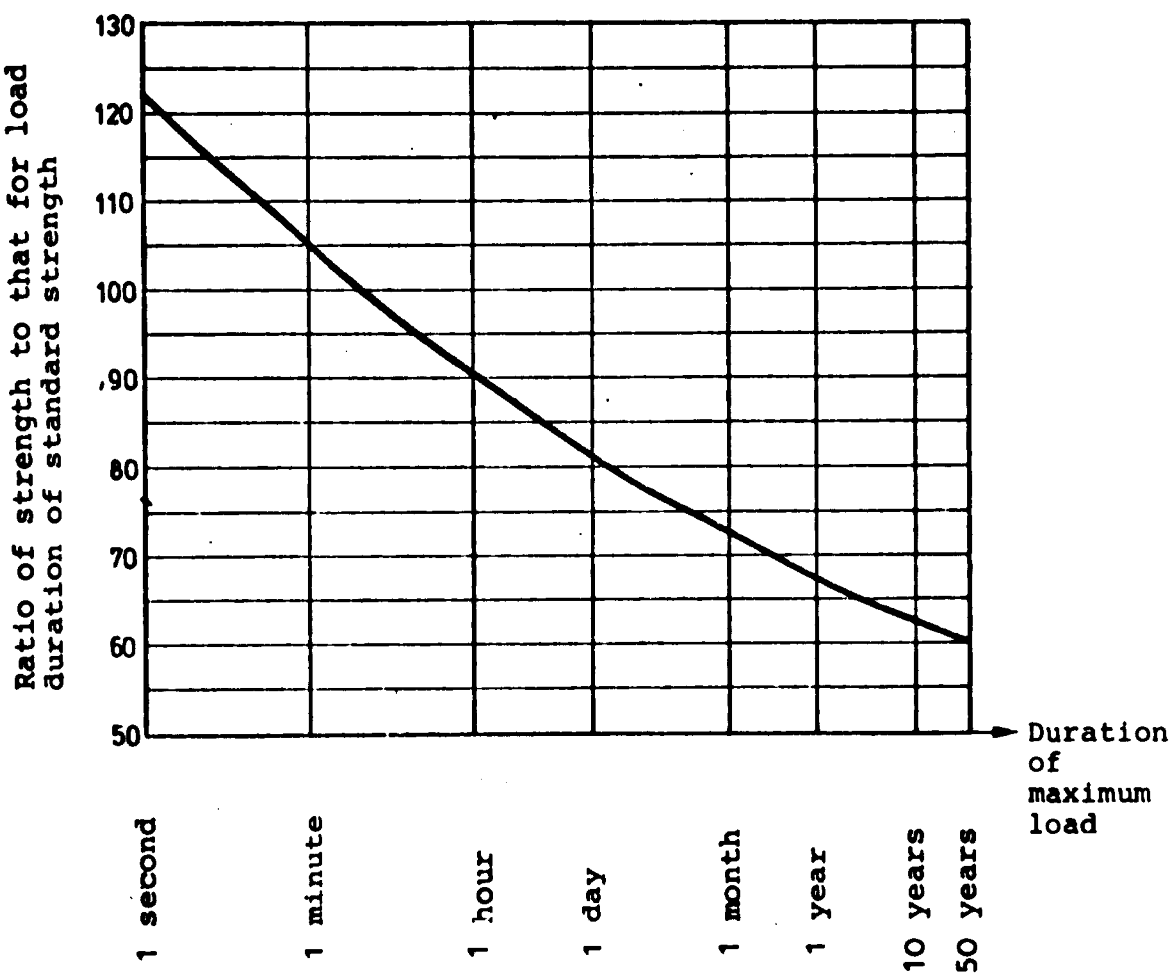


Fig. 18: Relation of strength to duration of load (ASTM D 2555)

2.3.4 Knife test for plywood

The knife test, as standardized in Germany and Great Britain (DIN 53 255 and BS 1455:1963 respectively), for the evaluation of the glue bonding strength of plywood can be regarded as a mixture between cleavage- and internal bond-test. With regard to plywood bonding quality the knife test is more useful than shear tests because it allows a better assessment of the interlinking of the glue and the wood surface. This is apparent when exposing the panels to moisture.

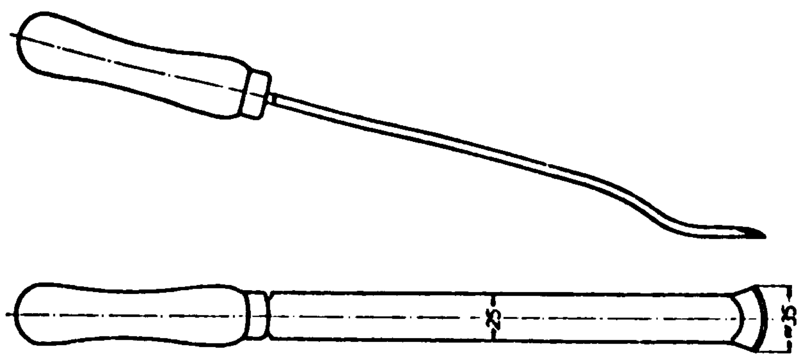


Fig. 19: Tool for the knife test of plywood (DIN 53 255 and BS 1455:1963)

For testing the knife edge (Fig.19) is applied parallel to the grain of the veneer. It is pushed downward through the face veneer to the plain of the glue line for testing the bond strength. A slight side to side motion will ease the test process. When the knife is forced approximately 25-50 mm along the line the handle will be pressed downwards to lift the veneer. The process can be repeated several times but any test has to follow the same procedure.

When the operator is satisfied that he has examined a sufficient area of the joint the test piece is ready for comparison with the master scale. If the knife has gone through a glue-line

and made a clean cut in the adjacent ply the test spot should be disregarded for an assessment.
DIN 53 255 includes a scale of four bond qualities:

First quality: (1)	The highest bond strength (the separation occurs only through the breaking of the wood itself and the glue-line is entirely covered by adhering wood - there is only wood failure)
Second quality: (2)	Good bond strength (the glue-line is covered by adhering fibre)
Third quality: (3)	Low bond strength (the glue-line is mostly covered by adhering fibre - in some cases there is a small "glue failure" percentage)
Fourth quality: (4)	Bad bond strength (the veneer parts from the surface below by separation along the glue-line, to which few or no wood fibres are left adhering - the wood failure percentage is low).

The British standard BS 1455:1963 contains a scale of six bond qualities; the highest bond quality numbered 10, the lowest 0, with intermediates of 8, 6, 4 and 2. The master scale is stepped by 2, but intermolations to the next number is possible
Each glue-line is tested for itself and no glue-line shall have a bond quality of less than 3 (DIN 68 705); according to BS 1455:1963 no glue-line shall have a bond quality of less than 2 and the average value for all specimens shall not be less than 5.

Both procedures, the German and the British knife test are similar in principle and in performance. There is an operators classification, and therefore it is necessary that the operator is well trained.
Care must be taken for interpretation of the knife test results. When plywood made from low density or rotten wood the force necessary to separate the veneers is low but excessive amounts of fibre may be left on the glue-line. With high density wood, the veneers may be more difficult to separate, but less fibre is left on the glue-line, indicating lower ratings on the bond quality scale than lower density wood species.
Good results can be obtained by the knife test if plywood is tested after the wet test at different temperatures. LAIDLAW (1975) did some research about the connection between natural weathering and cyclic wet test in the laboratory (Fig. 20). For both, natural and laboratory tests phenolic glues showed the best results. Ureas, fortified by phenol-resorcinol or melamin showed good results, and pure ureas showed the lowest bond qualities.

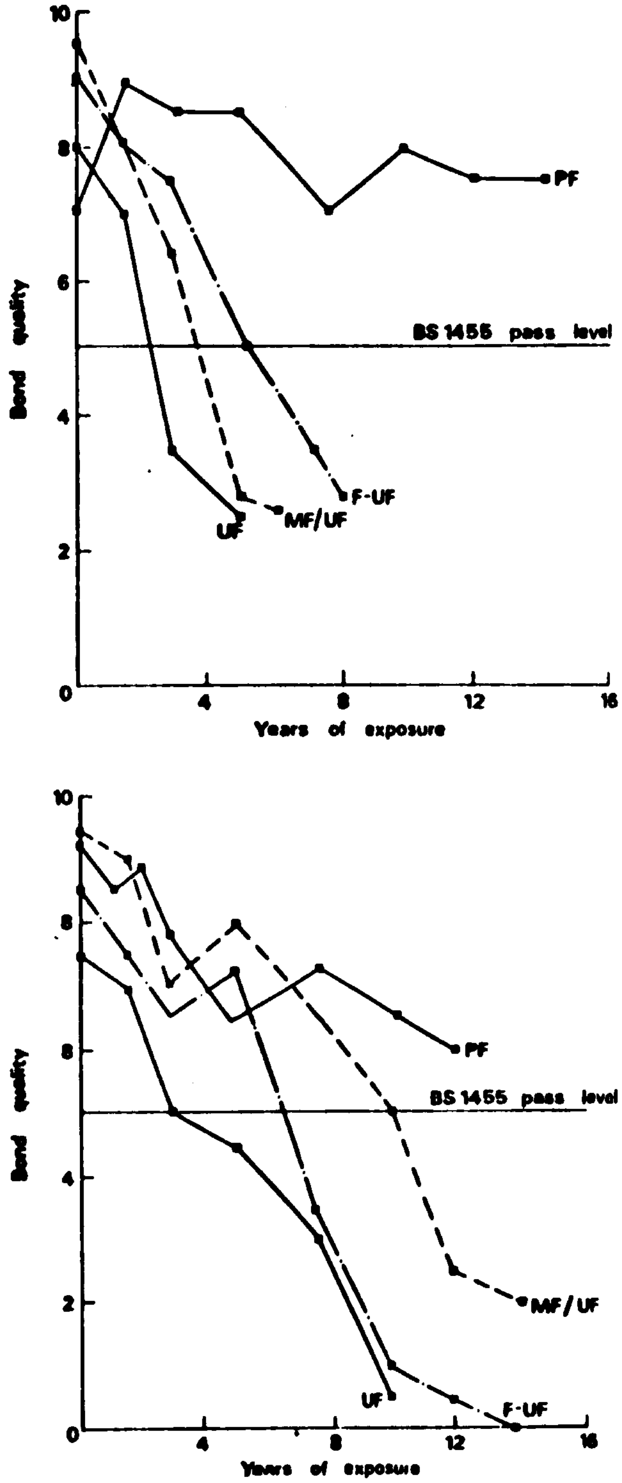


Fig. 20: Plywood made with different glues exposed to weather (above) and cyclic humidity conditions (below) (according to LAIDLAW 1975)

2.3.5 Impact tests

The ultimate strength of high polymeres - to which class wood and glue belongs - for any given geometric stress situation includes time effects. Impact tests are tests of a very short duration, i.e. 1/1000 of a second. The essential difference between dynamic (impact) and static loads is that dynamic loads always produces a peak stress higher than slowly static loads. The property measured in an impact test is not the maximum stress supported by the glue-line, but rather the amount of energy necessary to cause failure.

Mostly static loading has to be considered in normal use of glued wood, impact tests are not so important. Only in some cases, i.e. in building construction and machine parts impact load occurs. Therefore, impact tests were made in special cases only. This test is standardized in the U.S.A. (ASTM D 950). The specimen is like an impact bending one (Fig. 21). For this purpose the absorbed work and wood failure percentage is determined.

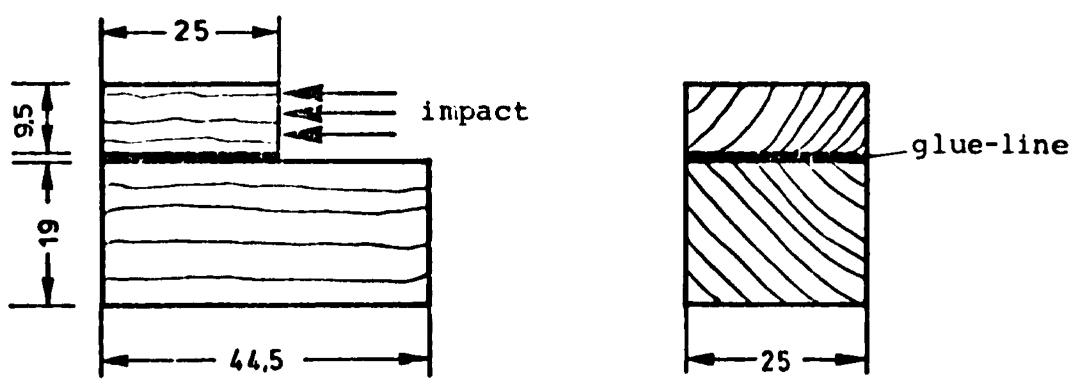


Fig. 21: Block shear impact test specimens (ASTM D 950)

2.3.6 Testing the long-term durability of bonds through accelerated aging

One of the most important properties of glued structures is the long-term durability of the bond. Research work should help to get information about the performance of a structural member and to obtain details for the determination of safe loading.
The testing of bonding quality on specimens exposed frequently to stresses in building constructions or in boat-building are closely related to fatigue resistance of glue-wood bonds compared to bonding quality of unloaded constructions. Consequently structural members should be tested immediately after fabrication and at regular intervals up to the supposed durability. According to the durability of structural members, experiments would have to be extended over many years. For this reason it was necessary to find some way to get information about the fatigue resistance behaviour of glued bonds by accelerated aging within a period shorter than the expected time of service. But a rather big difficulty is the recording of all possible influences that bear upon a structural member during its intended use.

One important influence is heat. Generally condensation resins are applied, which cure after glue spreading. The grade of age hardening is increased in the course of time, whereby this increase is dependent on temperature.
W.V. HANCOCK and S.Z. CHOW (1969) did some research work on the bonding strength of plywood bondings in relation to the grade of age hardening of the phenol-formaldehyde resin. They detected an increase in bonding strength and wood failure with growing age hardening. But they did their tests only on dry bonding strength up to 75 % age hardening. For the wet bonding strength at an age hardening degree of more than 75 %, a slight decrease in the adhesive strength is possibly caused by an embrittlement of the glue-bond. For this reason the same results can also be expected for the dry bonding strength.

In addition to the above mentioned embrittlement of the glueline, other climatic influences affect the fatigue resistance of gluings. Condensation glues are more or less insensitive to moisture. Changes in humidity cause swelling and shrinking of wood. A loosening of the compound at the interface "wood-glue" by internal stresses is the consequence.
A testing procedure for the accelerated aging of glue-bonds (short-time test) should comprise elevated temperatures and alternating humidities. On the other hand, apart from climatic influences, short-time tests must include probable mechanical stress. But in many cases it is almost impossible to foresee all longterm stresses.

In several studies different short-time tests for an accelerated aging of glue-bonds were applied. P.L. NORTHCOTT and coworkers (1968) compared 11 different short-time tests. In the course of their tests on 10 different glues they found out that none of the aging procedures is sufficient to enable a precise statement of the glue-bond's later behaviour. M.D. STRICKLER (1958) agrees fairly well with this and he thinks the development of a universal accelerated testing method rather impracticable. The results of work performed at the Ottawa Forest Products Laboratory (AONONYMOUS, 1967) are rather different. In this study the durability of bands which were subject to outdoor exposure up to 15 years have been compared with the results of a 72hour permanent boiling test. The results generated by weathering and by accelerated tests are corresponding.

While the influence of weathering on the durability can be determined sufficiently well by accelerated tests under different conditions, the influence of mechanical stress cannot be reproduced through normal accelerated tests as fixed under ASTM D 1101, ASTM D 1037, and DIN 53 254 which only comprise the effect of humidity and termperature. To estimate the durability of bonds in building elements exposed to mechanical stress, it is necessary either to alter the samples to adequate mechanical loading or to compare the glueline bonding strength of stressed building elements with unstressed samples. This method cannot be applied in practice because destruction of the building elemtns cannot be avoided.

Especially for testing wood based panels the standards of different countries contain accelerated aging tests (wet tests). Good examples are the different aging tests (specified in the British and German plywood standard).

The standard BS 1455:1963 includes three different wet tests (Tab.1).

Type	Condition	Time (hours)	Glue
WBP	a) water boiling under normal atmospheric condition, or	72	PF, RF PF/RF
	b) steaming in a closed vessel at 2 kp/cm^2 gauge pressure, the test pieces being above the level of condensate or free water, or	12	
	c) as for b), but at 1 kp/cm^2 gauge pressure	24	
BR	water boiling under normal atmospheric conditions	3	MF or fortified UF
MR	water at 67 $\pm$ 2 °C	3	UF

Tab. 1: Wet tests in hot water or steam for plywood (BS 1455:1963)

Type of tests:

WBP - Weather and boil proof

Adhesives of the types which by systematic tests and by their records in service over many years make joints which are highly resistant to weather, micro-organisms, cold and boiling water, steam and dry heat. At present phenolic and resorcinol resin adhesives meet with these requirements.

BR - Boil-resistant

Joints made with these adhesives have good resistance to weather and the boiling water test, but fail under the very prolonged exposure to weather. They will withstand cold water for many years and are highly resistant to attack by micro-organisms.
At present phenolic, resorcinol, melamine, melamine/urea and fortified urea resin adhesives withstand these requirements.

MR - Moisture resistant and moderately weather-resistant. Joints made with these adhesives will withstand full exposure to weather for only a few years and will withstand cold water for a long period, but hot water only for a limited time and fail under the boiling water test. They are resistant to attack by micro-organisms. Also urea resin adhesives fulfill these requirements.

INT - Interior

Joints made with these adhesives are resistant to cold water, but are not required to withstand attack by micro-organisms.
Also animal glues (like blood albumin, blood/casein mixes and soya bean derivates) used outside of Great Britain have shown these requirements.

The German standard DIN 68 705 contains four accelerated test methods, but only two of these tests, IF 20 and AW 100, are applied (Tab. 2).

Type	Condition	Time (hours)	Glue
IF 20	water at 20 ± 2 °C	24	HF
IW 67	water at 67 ± 0,5 °C water at 20 ± 5 °C	3 2	HF, MF/HF
A 100	water boiling under normal atmospheric condition water at 20 ± 5 °C	6 2	MF/HF, MF
AW 100	water at 20 ± 2 °C water boiling under normal atmospheric condition air-drying 60 ± 2 °C water boiling under normal atmospheric condition water at 20 ± 5 °C	24 4 16 - 20 4 2 - 3	PF, RF, PF/RF

Tab. 2: Wet tests for plywood (DIN 68 705)

Type of tests:

AW 100 - Bonds resist natural weathering and wet tests. Phenolic, resorcinolic and in particleboard production melamin and isocyamate adhesives meet the test requirements.

A 100 - Bonds resist cold and hot water. Also melamin-ureas pass this test.

IW 67 - Bonds resist higher humidity and cold water up to 67°C but not against hot water and full exposure to weather. Some pure ureas and melamin fortified ureas meet the requirements of this test.

IF 20 - Bonds resist normal humidity and normal temperature only, not water or weathering. All ureas pss this test.

All different wet tests are performed to get information on the glue-line and not on the material.

Plywood made from unprotected non-durable timbers will be unsuitable for exposure of permanent humidity whatever glue applied. However, if durable or preservative-treated wood species are used the performance of the plywood will be governed by the properties of the adhesive.

Plywood may meet a wide variety of exposure conditions in service, ranging from indoor to outdoor exposures; adhesive formulations are known to fulfill a wide variation in their resistance to moisture and heat. In order to gain results on glue performance, a series of exposure trials and cyclic laboratory aging tests are necessary.

2.3.7. Determination of evaporation of formaldehyde

For urea bonds it is known that different amounts of formaldehyde are set free during production, storage and use of the products. In some cases the evaporation of formaldehyde is decreasing very slowly which results in a rather high concentration of formaldehyde in the environmental atmosphere over a long period. This might be a disadvantage when using particleboards for interior decoration applied to prefabricated wooden houses.

Because of the above mentioned problems the disposal of formaldehyde has been a subject of intensive research for a long period.
In this connexion PLATH (1968) examined the influence of the chip moisture content, the pressing temperature, the

pressing time, the hardener components, and the acceleration of the hardening process. PETERSEN, REUTHER, EISELE and WITTMANN (1972, 1973, 1974), too, did extensive research on the influence of chip moisture content, the molar concentration, the glue pick up, the pressing temperature, the pressing time, the kind of chips and hardener as well as on the amount of hardener and the effect of storage time.

For the determination of the amount of formaldehyde set free in general two different methods are applied: The FESYP-perforator-method, and the FESYP-gas analysis-method. (FESYP = Federation Europeenne des Syndicats de Fabricants de Panneaux de Particules).

According to the FESYP-perforator-method the free formaldehyde of the particleboard specimen to be tested is extracted by boiling toluol, absorbed by a certain amount of water and determined iodometrically. The result is expressed in percentage in weight of dry particleboards.
For the FESYP-gas analysis-method the specimen is kept in a flow of air or nitrogen of 150 l/hour for a period of four hours. The formaldehyde in the environmental gas is absorbed by water, and it can be determined iodometrically every hour. The result gives the content of formaldehyde in mg per hour for one m^2 or one kg of dry particleboard.
The determination of the formaldehyde content can take place immediately after production when leaving the factory, or after various periods of time after use.

2.3.7.1 Evaporation of formaldehyde during particleboard production

In order to be able to control the process of particleboard production with regard to pressing time and quality more formaldehyde than necessary for condensation only is added to the glue. This is expressed by the molecular equivalent of urea and formaldehyde, which in practice is between 1.4 and 1.8. A rather big amount of the free formaldehyde evaporates already during the press-cycle. The glue coat on the rather big surface of the particles is an advantage for the evaporation of the formaldehyde. Further factors of influence are the particle moisture content, the pressing temperature, and the pressing time.
To reduce the evaporation of formaldehyde during particleboard production the following precautions are possible:

- Use of low formaldehyde content glues (molecular equivalent between 1 and 1.4.
- Rapid curing
- Addition of formaldehyde neutralizing substances, like urea.
- Rather low particle moisture contents.
- Low pressing temperatures.

Because parts of these precautions result in economical disadvantages (increase of pressing time and technical disadvantages, also reduction of bonding strength) it is necessary to find a compromise.

2.3.7.2 Evaporation of formaldehyde during utilization of particleboards

A small part of the gaseous formaldehyde generated during the pressing cycle will escape from the board's cavities at the time of storage or remanufacture. Through reaction of moisture and glue an additional, rather important evaporation of formaldehyde might be caused. This evaporation depends upon the degree of cure. The higher the degree of cure, the lower the evaporation. Higher temperatures cause a higher rate of evaporation by an increased hydrolysis. An increase in temperature of 5°C causes a double or three times higher formaldehyde evaporation. This fact must be considered when using particleboards for interior decoration and for prefabricated wooden houses.

Complaints about unpleasant smell derived from a rather high formaldehyde concentration in the room atmosphere have reduced the importance of urea bonded particleboards for prefabricated housing. A reduction of the later formaldehyde evaporation can be achieved by a surface treatment of the boards. Coatings with plastics and varnishes tighten the boards and are of rather good efficiency if they contair formaldehyde neutralizing substances.

2.3.8 Identification of glues in glue-lines

Identification of glues in glue-lines is of great importance especially in the plywood industry, timber laminating industry and in research institutes. Identification can be made by various physical or chemical techniques. Most of the condensation type glues are different in colour (phenol, urea), resistance to water, temperature, fungi, chemicals. Identification of glues can also be made by infrared-spectro-photometry. For these tests normally expensive equipment and highly trained staff is required. Different test methods were developed. Because a detailed discussion would need too much time a study of the following literature is recommended:

E.PLATH and L. PLATH (1959), BOASSARD and FUTO (1963), FUTO (1973), ROY et al. (1973).

3. Equipment for testing wood adhesives and bonding strength

3.1 Testing wood adhesives

No.	Amount	Equipment	usable for	Cost (approximate) DM Priority I	Priority II	Priority III
1	1	falling ball type viscosimeter	viscosity tests	500		
2	1	flow cup viscosimeter with exchangeable nozzles	viscosity tests		300	
3	1	rotation type viscosimeter	viscosity tests		2,500	
4	2	simple timers	gelation time, tension and compression tests	150		
5	50	beakers with stirrers (250-1000 ml)	gelation time	600		
6	1	thermostat 15 l, up to 150 °C	gelation time storing of specimens	3,000		
7	1	electrical pH-measuring instrument	pH-check-up		2,500	
8	1	oven 100 l, up to 250 °C	solids and filler content, drying of samples (determination of oven dry weight)	1,000		
9	20	petri or similar dish, 50 - 200 mm diameter	solids and filler content	300		
10	1	micro balance, max. 250 g accuracy 10^{-4} g	solids and filler content, density and water content	2,500		
11	1	electrical blunger	glue mixing		1,000	
12	1 set	mesh screens	size grading of fillers and extenders			1,500
			Total	8,050	6,300	1,500

3.2 Testing of bonding strength

No.	Amount	Equipment	usuable for	Cost (approximate) DM Priority I	Priority II	Priority III
1	1	circular saw	preparing specimens	5,000		
2	1	planer (surfacer)	preparing specimens	6,000		
3	1	drilling machine	preparing special specimens			2,500
4	1	tension testing machine, max 30 kN	tension tests	15,000		
5	1	universal testing machine, max. 100 kN	tension-, compression-, bending-tests		50,000 (100,000)	
6	1	equipment for treating specimens: gas burner, pot for boiling specimens, pot for water storage	treating of specimens	600		
7	2	tools for knife tests according DIN 53 255	testing of plywood	150		
8	1	autoclave 50 l, pressure 3 kp/cm^2, electrical heating	boil tests			1,500
9	1	equipment for calculating evaporation of formaldehyde: flask, gas burner, titrating apparatus, different chemicals, nitrogen gas bottle	testing evaporation of formaldehyde			2,000
10	1 set	tools: 3 slide gauges 2 micrometers		800		
11	1	climatic cabinet 200 l, temperature 0 - 100 °C, humidity 20 - 95 %	storing of specimens			15,000
12	1	equipment for testing shear blocks (ASTM D 805)	testing of shear blocks		500	
13	1	hydraulic laboratory press, 50 x 50 cm^2, pressure 3 N/mm^2 (total pressure 750 kN), electrical heating up to 250 °C	preparing specimens (plywood, particleboard)			15.000
			Total	27,550	50,000 (100,000)	36,000

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ASTM Adhesive Standards (in reference to wood gluing)

Specifications for:

D 2558-70	Adhesives for Structural Laminated Wood Products for Use under Exterior (Wet Use) Exposure Conditions

Methods of Test for:

D 1084-63	Viscosity of Adhesives
D 1875-69	Density of Adhesives in Fluid Form
D 1579-60	Filler Content of Phenol, Resorcinol and Melamin Adhesives
D 950-54	Impact Strength of Adhesive Bonds
D 1151-69	Moisture Content of Aqueous Adhesives
D 1490-69	Nonvolatile Content of Urea-Formaldehyde Resin Solutions
D 1582-60	Nonvolatile Content of Phenol, Resorcinol and Melamin Adhesives
D 1877-70	Permanence of Adhesive-Bonded Joints in Plywood under Mold Conditions
D 1183-70	Resistance of Adhesives to Cyclic Laboratory Aging Conditions
E 229-70	Shear Strength and Shear Modulus of Structural Adhesives
D 906-64	Strength Properties of Adhesives in Plywood Type Constructions in Shear by Tension Loading
D 905-49	Strength Properties of Adhesive Bonds in Shear by Compression Loading
D 2339-70	Test for Strength Properties of Adhesives in Two-Ply Wood Constructions in Shear by Tension Loading
D 897-68	Tensile Properties of Adhesive Bonds
D 2556-69	Apparent Viscosity of Adhesives Having Shear-Rate-Dependent Flow Properties
D 1037-64	Evaluating the Properties of Wood-Based Fiber and Particle Panel Materials
D 1759-64	Conducting Shear-Block Test for Quality Control of Gluebonds in Scarf Joints
D 1101-59	Integrity of Glue Joints in Structural Laminated Wood Products for Exterior Use
D 805-63	Testing Veneer, Plywood and Other Glued Veneer Constructions

British Standard Specifications for Adhesives Used with Wood and for Some Bonded Wood Products

Adhesives

BS 647	Sampling and Testing Glues
BS 745	Animal Glues for Wood
BS 1133	Adhesives for Packaging
BS 1203:1963	Synthetic Resin Adhesives (Phenolic and Aminoplastic) for Plywood
BS 1204:1964; Part 1	Synthetic Resin Adhesives, Gap Filling (Phenolic and Aminoplastic) for Constructural Work in Wood
BS 1204:1965; Part 2	Synthetic Resin Adhesives, Close Contact (Phenolic and Aminoplastic) for Wood
BS 1444	Cold Setting Casein Glue for Wood
BS 3544:1962	Methods of Test for Polyvinyl Acetate Adhesives for Wood
BS 4071:1966	Polyvinyl Acetate (PVA) Emulsion Adhesives for Wood

Bonded Products

V 35	Medium and Low-Strength Plywood for Aircraft
6 V3	High-Strength Plywood for Aircraft
BS 1088, BS 4079	Plywood for Marinecraft
BS 1455:1972	Plywood Manufactured from Tropical Hardwoods
BS 3444	Blockboard and Laminboard
BS 3493	Information about Plywood
BS 3583	Information about Blockboard and Laminboard
BS 3842	Treatment of Plywood with Preservatives
BS 4169	Glued Laminated Timber Structural Members
BS 1811	Methods of Test for Wood Chipboard and Other Particle Boards
BS 2604	Resin-Bonded Wood Chipboards

<u>German Standards</u> (in reference to wood gluing)

DIN 4076	Kurzzeichen auf dem Holzgebiet, Blatt 3: Klebstoffe
DIN 16 920	Klebstoffe, Richtlinien für die Einteilung
DIN 16 921	Klebstoff-Verarbeitung
DIN 52 365	Prüfung von Holzspanplatten; Bestimmung der Zugfestigkeit senkrecht zur Plattenebene
DIN 52 366	Prüfung von Spanplatten; Bestimmung der Abgebefestigkeit und der Schichtfestigkeit
DIN 53 251	Prüfung von Holzleimen und Holzverleimungen, - Bestimmung der Bindefestigkeit
DIN 53 252	- Kenndaten des Verleimungsvorganges
DIN 53 253	- Bestimmung von Schaftverleimungen im Zugversuch
DIN 53 254	- Bestimmung von Längsverleimungen im Zugversuch
DIN 53 255	- Bestimmung von Sperrholzverleimungen im Zugversuch und im Aufstechversuch
DIN 53 257	- Bestimmung der Zugfestigkeit von Hirnholzverleimungen
DIN 53 258	- Bestimmung des Verhaltens bei wiederholter kurzzeitiger Wassereinwirkung
DIN 68 141	Holzverbindungen, Prüfung von Leimen und Leimverbindungen für tragende Holzbauteile
DIN 68 601	Holz-Leimverbindungen; Begriffe
DIN 68 602	Holz-Leimverbindungen; Beanspruchungsgruppen
DIN 68 603	Holz-Leimverbindungen; Prüfung
DIN 68 705	Sperrholz, Begriffe, Anforderungen, Prüfung
DIN 68 761	Holzspanplatten; Flachpreßplatten FPY für allgemeine Zwecke; Begriffe, Eigenschaften, Prüfung
DIN 68 762	Spanplatten für Sonderzwecke im Bauwesen; Begriffe, Eigenschaften, Prüfung
DIN 68 763	Spanplatten; Flachpreßplatten für das Bauwesen; Begriffe, Eigenschaften, Prüfung, Überwachung
DIN 68 764	Strangpreßplatten für das Bauwesen

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