

Time to Failure Testing in Shear of Wood–Adhesive Bonds under Elevated Temperatures*

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Abstract

At present, the evaluation of wood–adhesive bonds lacks a method that is able to predict the long-term load carrying capacity in shear of a bond in a comparatively short testing time. For this reason, a new test approach was investigated to determine the time to failure of wood–adhesive bonds. In our research, lap joint specimens were prepared with a melamine-urea-formaldehyde (MUF) adhesive at two mixing ratios (100/100 and 100/20 [resin/hardener]). The specimens were subjected to tensile shear stresses at load levels between 30 and 90 percent of their mean wet short-term strength while being immersed in water at temperatures of 60°C and 90°C. The time to failure and the wood failure percentage were determined. The analysis showed good correlations between time to failure and load level as well as between time to failure and temperature. The adhesive mixing ratio, however, showed no influence on the failure characteristics. The wood failure percentage highly depended on the test duration. With prolonged test duration, the mode of failure increasingly changed from wood failure to adhesion failure. Overall, the test method proved to be promising for a detailed performance evaluation of wood–adhesive bonds.

Structural engineered wood products have to withstand high and fluctuating stresses during service life. Stresses are induced by permanent and variable service loads as well as climate and weather exposure. For wood, the long-term material strength has been investigated intensely in time to failure (TTF) experiments. In particular, long-term bending tests by Wood (1947) resulted in the following equation for time to failure of wood under constant loading:

$$\frac{\sigma}{\sigma_0} = 0.904 - 0.063 \times 10 \log t_f \quad (1)$$

where σ is the specimen load relative to the average short-term strength σ_0 and t_f is the time to failure (given in hours). Leont'ev (1961) investigated the duration of load effect for shear loaded specimens and obtained more pronounced characteristics than for bending:

$$\frac{\sigma}{\sigma_0} = 0.914 - 0.104 \times 10 \log t_f \quad (2)$$

It has to be taken into account that the climate was not controlled in these experiments and moisture content

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variation may have contributed to this behavior. The more pronounced duration of load effect for shear, however, may have important impact for wood–adhesive bonds because loads are mainly transferred by shear. Further investigations on the influence of the load level, moisture content, and temperature on the TTF were performed by, e.g., Hoffmeyer (1990) for wood and van de Kuilen (1999) for timber joints, confirming a steeper curve for joints where loads are transferred mainly in shear using bolts and proprietary fasteners. The long-term strength characteristics are taken into account using strength modification factors in relevant design standards. For example, in EN 1995-1-1 (European Committee for Standardization [CEN] 2010) the factor k_{mod} is applied to calculate design strength values depending on the expected duration of load and moisture content of the structure.

For the present investigation, the TTF approach was transferred to wood–adhesive bonds. This approach was chosen because in engineered products such as glued laminated timber both wood and adhesive are exposed to long-term effects, and the lifelong functionality of wood–adhesive bonds is crucial for the safety of glued laminated timber structures (van de Kuilen and Gard 2016). It is furthermore known that the behavior of wood adhesives is temperature dependent and particularly moisture dependent (e.g., Clauß 2011, Kläusler et al. 2013). In addition, the long-term behavior highly depends on the adhesive chemistry. For example, strength loss of the adhesive bond can occur due to hydrolysis with formaldehyde-based adhesives such as urea-formaldehyde (UF) or melamine-urea-formaldehyde (MUF; Dunky and Niemz 2002).

Over the years, durability assessment of wood–adhesive bonds has been realized with a number of tests, including single exposure tests, cyclic exposure tests, or mechanical tests (Dinwoodie 1983). In structural timber components such as cross-laminated or glued laminated timber, bond durability, e.g., is assessed in cyclic delamination tests according to EN 14080 (CEN 2013a, Knorz et al. 2017). Here, stresses in the wood–adhesive bonds are induced by enforced moisture changes and the material properties of the wood. Other long-term test methods for structural wood products or adhesives mainly aim at the determination of creep behavior of wood–adhesive bonds after application of a shear or bending load (ASTM D3535 [ASTM International 2013], EN 302-8 [CEN 2017a], EN 15416-3 [CEN 2017b]). The test method according to ASTM D4680 (ASTM International 2004) determines both the creep behavior and the TTF of an adhesive bond but requires a rather long test duration (4 mo).

In general, it can be summarized that there is a lack of reliable test methods that determine the durability of wood–adhesive bonds in a reasonable testing time. All established test methods have determined that it is difficult or simply not possible to draw quantifiable conclusions about the expected service life of a bond in practice. Therefore, a new test approach was developed to experimentally determine the TTF of wood–adhesive bonds under tensile shear stress with a test duration of up to 2 weeks by applying higher than usual temperature and moisture content.

Materials and Methods

An established test method (EN 14292 [CEN 2005]) was used as a basis for our research. The test method requires

the preparation of lap shear test specimens following EN 302-1 (CEN 2013b). For this, 5-mm-thick boards were produced from beech wood with a mean density of 717 kg/m³ (± 27 kg/m³ SD) and a mean moisture content of 10.8 percent ($\pm 0.6\%$ SD). Two boards with freshly planed surfaces were bonded with a commercially available MUF adhesive. The curing of the MUF adhesive is set in motion by the acidity of the hardener, with formic acid and formaldehyde being important components of the hardener. Important adhesive properties are given in Table 1. The adhesive was applied on one side using two mixing ratios (100/100 and 100/20 [resin/hardener]). An overview of the bonding parameters is given in Table 2. The minimum pressing time for the adhesive is specified as 6.5 hours (100/20) and 4 hours (100/100). A pressing time of 14 hours was chosen to ensure a high initial strength when removing the bonded members from the press. From the bonded members, lap joint test specimens 90 mm long and notches to generate a shear plane with a 10-mm length (Fig. 1) were cut following EN 302-1 (CEN 2013b). The specimen width for short-time strength testing was 20 mm, for the TTF experiments the specimen width depended on the load level. Twelve test specimens were prepared for each combination of mixing ratio, temperature, and load level.

First, it was required to determine the short-term strength to obtain reference values for the TTF experiments. For this, specimens were stored in a water bath at temperatures (T) of 60°C and 90°C, respectively, for 3 hours. The short-term strength tests were then performed by means of a universal testing machine (TesT 112; TesT GmbH) using a constant

Table 1.—Physical and chemical properties of resin and hardener (according to technical datasheet; pH, density, and viscosity measured at 20°C).

	Solid content (%)	pH	Density (g/cm ³)	Viscosity (mPa s)
Resin	68.5 \pm 1	9–10.5	1.37	2,000–4,000
Hardener	38.5 \pm 1	1–2	1.06	2,500–4,500

Table 2.—Bonding parameters for preparation of the test specimens.

Mixing ratio (resin/hardener)	Open assembly time (min)	Closed assembly time (min)	Pressing time (h)	Pressure (N/mm ²)	Adhesive spread (g/m ²)
100/20	<5	60	14	1.2	400
100/100	<5	20	14	1.2	400

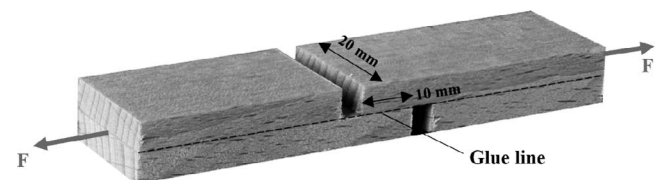


Figure 1.—Lap shear test specimen with a 10 by 20-mm shear plane for short-term strength testing; to increase the relative load level in the shear plane, the specimen width is varied.

displacement rate. The tensile shear strength $f_{v,t}$ was calculated as follows:

$$f_{v,t} = \frac{F_{\max}}{A} \text{ (MPa)}$$

where F_{\max} is the load at failure and A is the area of the shear plane. In addition, the wood failure (WF) percentage on the sheared plane was estimated visually to the nearest 10 percent.

For the TTF tests, the EN 14292 (CEN 2005) test apparatus (which was originally defined for bending tests) was modified to allow testing under tensile shear load. In addition, small containers were integrated in the test apparatus so that specimens could be immersed in water at elevated temperatures during the experiment (Fig. 2). Similar to short-term strength testing, the specimens were immersed in water at temperatures of 60°C or 90°C before load application. Then, the specimens were subjected to constant shear stress at load levels between 30 and 90 percent of their wet short-term strength using a lever arm. The load was kept constant, and required stress level was obtained by varying the width of the shear plane, but not the length, so as not to influence peak stresses at the beginning and end of the glue line (see Table 3). The TTF was automatically detected by means of a switch in the test apparatus and a measurement software (LabVIEW; National Instruments). After failure, the specimens were dried at 20°C and 65 percent relative humidity and WF was estimated.

Results and Discussion

Short-term strength tests

The results of the short-time strength tests are displayed in Table 3. The mean shear strength $f_{v,t,\text{mean}}$ was between 6.68 and 7.27 N/mm² with coefficients of variation (CoV) ranging between 7.9 and 20.2 percent. The WF was consistently 100 percent, which indicates that varying CoV can be attributed to the raw material. Furthermore, the strength values exceed the requirement of $f_{v,t,\text{mean}} \geq 6.0$ N/mm², which is specified in EN 301 (CEN 2013c) for wet shear strength of structural adhesives. However, it should be noted that this requirement is given for different pretreatments than used in this investigation. However, the strength values in combination with high WF indicate a good quality of the MUF bond.

Table 3.—Short-term strength of lap shear specimens.^a

Temperature (°C)	MR (resin/hardener)	$f_{v,t,\text{mean}}$ (N/mm ²)	CoV (%)	WF _{mean} (%)
60	100/20	6.71	7.9	100
	100/100	7.27	18.3	100
90	100/20	6.68	20.2	100
	100/100	7.04	8.4	100
Total		6.85	14.6	100

^a MR = mixing ratio; CoV = coefficient of variation; WF = wood failure.

The strength values of the samples were statistically compared by means of variance analysis. This did not reveal statistically significant differences between the samples tested at 60°C and 90°C or between mixing ratios. Therefore, the mean shear strength of all samples, $f_{v,t,\text{mean}} = 6.85$ N/mm², was used as a reference (100%) load level for the TTF tests. Based on this reference value, the required shear stress for load levels between 30 and 90 percent was calculated, and the required specimen widths were determined (Table 4).

Duration of load performance

General analysis of TTF results.—In Figure 3, results of the TTF tests are shown both for 60°C and 90°C as well as for mixing ratios 100/20 and 100/100. As expected, longer TTF values are obtained at lower load levels. This general picture is true for both temperatures and mixing ratios. The scatter of the TTF results strongly depends on the load level. By trend, higher scatter in TTF is obtained for lower load levels. For example, for 60°C and 100/100 (Fig. 3B) the TTF at 60 percent varies between 2 minutes and 51 hours 20 minutes, whereas the spread is between 4 hours 41 minutes and 164 hours at 40 percent. To a large extent, this can be explained by the fact that the actual load level in specimens deviates from the specified load level owing to variation of the actual strength of the wood. As a result, the distribution of the short-time material properties leads to an even higher spread of TTF for the individual load levels.

The results at 60°C for the two mixing ratios (Figs. 3A and 3B) agree very well. The regression lines show high coefficients of determination of 0.75 and 0.80, which indicates a good TTF prediction for bonded elements. Furthermore, both the axis intercept values (chosen at $t = 0.01$ min: 100/20, 76.3; 100/100, 77.7) and the rates of strength loss (100/20, 9.26; 100/100, 9.16) are very similar.

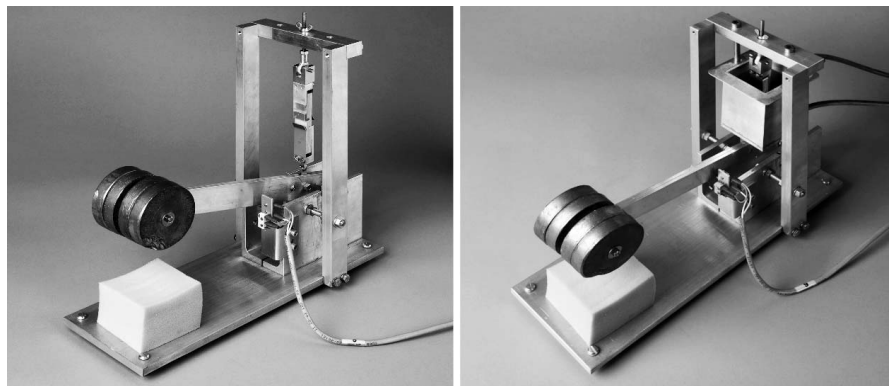


Figure 2.—Test apparatus: (left) application of the shear load with a lever; (right) test setup with container for water immersion tests.

Table 4.—Shear stress and specimen width for time to failure tests in dependence of the load level.

	Load level (%)							
	100	90	80	70	60	50	40	30
Shear stress (N/mm ²)	6.85 ^a	6.16	5.48	4.79	4.11	3.42	2.74	2.05
Specimen width (mm)	—	6.6	7.42	8.48	9.90	11.88	14.85	19.79

^a Reference strength value as determined in short-term shear tests.

This leads to the conclusion that the mixing ratio has hardly any influence on the TTF at 60°C. Moreover, it is noticeable from Figures 3A and 3B that the TTF at the 30 percent load level is shorter than what can be expected from the regression model. A shift of the damage pattern from creep rupture in the wood to bond failure could be noticed with increasing load level, which may have contributed to the lower TTF at 30 percent load level.

For the 90°C tests, the results for the two mixing ratios (Figs. 3C and 3D) also compare well. The regression model shows similar axis intercept values of 65.3 (100/20) and 65.4 (100/100). The rate of strength loss is slightly higher for the mixing ratio 100/100 (12.9) than for 100/20 (11.7). In addition, the variation in the data can be better explained with the regression model for mixing ratio 100/100 (coefficient of determination 0.74) than for 100/20 (0.57).

It is noticeable that most specimens failed within a short time (1 min) after applying the load at load levels between 60 and 90 percent, similar to the short-term tests. The visual inspection of the specimens showed almost exclusively WF, which indicates that failure is determined by the creep rupture characteristics of the wood. Given a similar time to

failure, it is interesting that short-term tests with a constant displacement rate and increasing stress result in strength values of $f_{v,t,mean} = 6.85 \text{ N/mm}^2$, whereas the application of a constant load leads to significantly lower strength values of 4.79 N/mm^2 (70%) or even 4.11 N/mm^2 (60%). If the test specimens with load levels between 60 and 90 percent had not been taken into account, this would have led to different regression models with lower rates of strength loss. This would have resulted in models with presumably longer TTF for low load levels and, at the same time, could mean that the current models underestimate the TTF for low load levels.

The comparison of the results at 60°C and 90°C shows a clear influence of the temperature on the TTF. In particular, the rates of strength loss (60°C, 9.16 and 9.26; 90°C, 11.7 and 12.9) vary significantly. Furthermore, the rates of strength loss obtained in our investigation are significantly higher than the value from Equation 1. The rate of strength loss determined by Leont'ev (1961) for shear load, on the other hand, lies between the values determined in our study.

Influence of the mixing ratio on TTF.—In Figure 4, the influence of the mixing ratio on the TTF is shown. For this

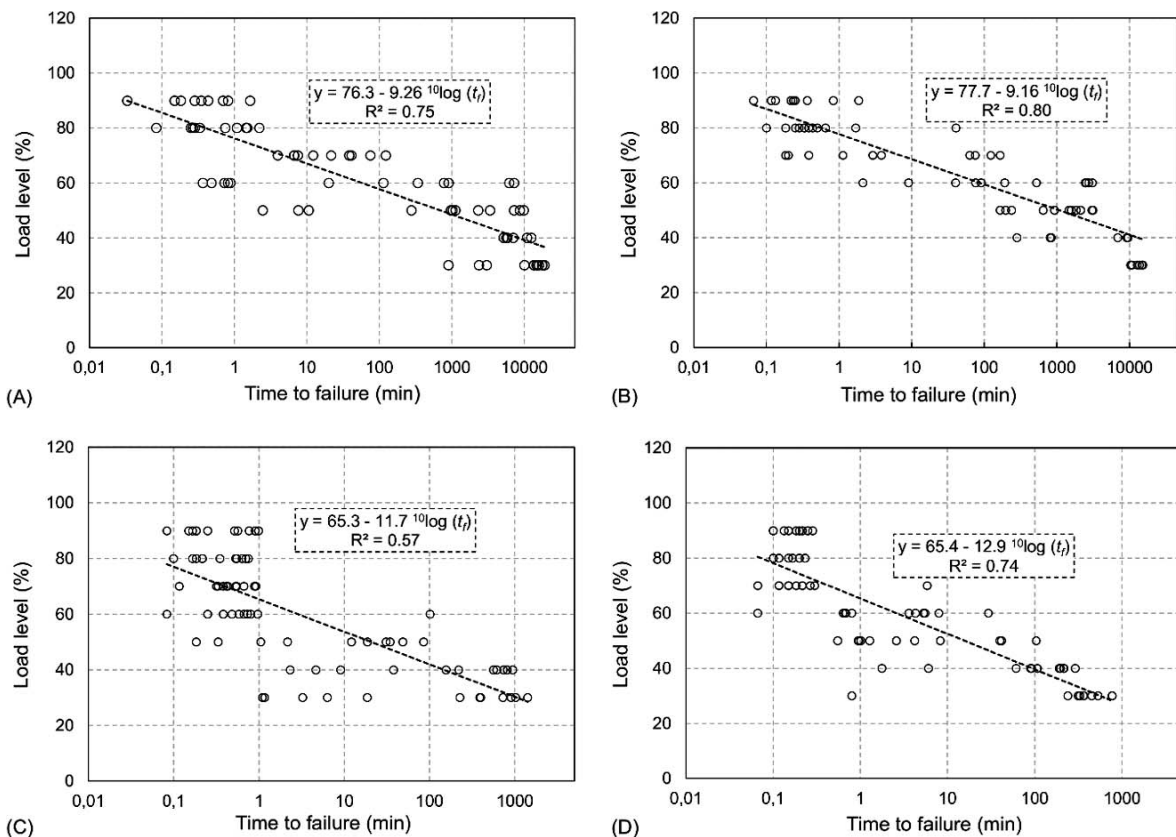


Figure 3.—Results of the time to failure tests: (A) 60°C, 100/20; (B) 60°C, 100/100; (C) 90°C, 100/20; (D) 90°C, 100/100.

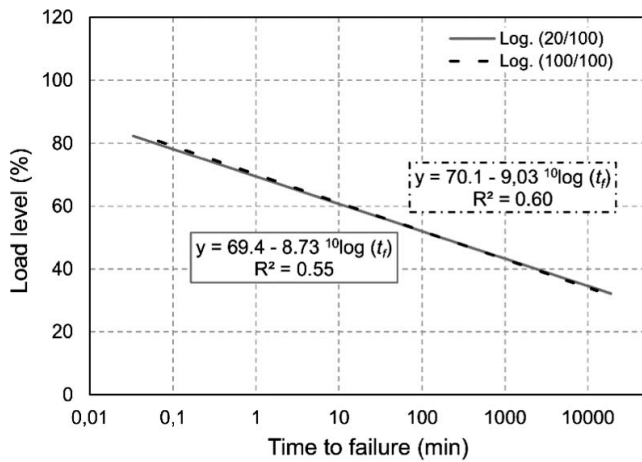


Figure 4.—Influence of mixing ratio on the time to failure.

analysis, the subsamples 60°C and 90°C were combined and a comparison was only made between mixing ratios. As can be seen in Figure 4, with axis intercept values of 69.4 (100/20) and 70.1 (100/100) as well as rates of strength loss of 8.73 (100/20) and 9.03 (100/100) being very similar, the regression lines are very close to each other. Therefore, it can be assumed that the mixing ratio has a negligible influence on the time to failure.

The different mixing ratios had been included in the design of the experiments for two reasons. On the one hand, we assumed that the hardener percentage might influence the creep behavior of the wood–adhesive bond. This is because the shear modulus of the cured adhesive depends significantly on the hardener quantity (Bruder 2017). On the other hand, a high hardener content could impair the moisture resistance of the polymer adhesive network. Both assumptions can be explained, among other things, by the fact that the hardener contains high amounts of polyvinyl acetate (PVAc). PVAc adhesives, if not integrated in a two-component adhesive system, tend to creep, and bond strength is reduced when exposed to high moisture and temperature (Dunky and Niemz 2002). In the present study, however, the hardener ratio obviously had no influence on the TTF of wood–adhesive bonds. One explanation for this might be that the creep behavior of the adhesive is lower than that of the wood and, therefore, the material properties of the hardened adhesive have negligible influence on TTF. It can also be stated that even though higher percentages of PVAc are embedded in the adhesive, this obviously does not impair the moisture resistance of the duroplastic adhesive network.

Influence of the temperature on TTF.—The influence of the temperature on the TTF is shown in Figure 5. For this analysis, the subsamples with mixing ratios 100/20 and 100/100 were combined, and a comparison was only made between temperatures. As already indicated in Figure 3, the significant influence of the temperature can be confirmed with the regression models in Figure 5. The higher temperature of 90°C led to reduced TTFs when tested at the same load level and caused a shift of the regression line. In addition, the different rates of strength loss resulted in continuously diverging regression lines. For example, at 80 percent load level and 60°C, the TTF is 5.5 times the TTF at 90°C; at 60 percent load level, the factor between 60°C and

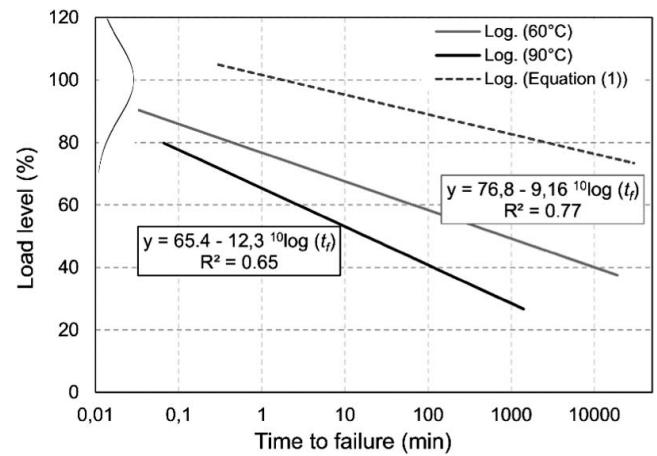


Figure 5.—Time to failure regression models for 60°C and 90°C, together with the regression line according to Equation 1.

90°C increases to 25; and at 40 percent load level, the TTF at 60°C is 100 times the TTF at 90°C.

In comparison with Equation 1, our investigation shows significantly reduced relative failure stress levels. Reasons for this are very likely a combination of temperature, water immersion, the direction of load application, and bond and adhesive degradation. The influence of temperature and moisture content on the duration of load effect of wood was examined, e.g., by Fridley et al. (1989, 1991). In these investigations, the authors showed that both increasing temperature and moisture reduce the failure stress level of wood. The influence of temperature and humidity could be modeled using parallel shifted regression lines. The rate of strength loss, however, was different for temperature and moisture examinations. These findings from Fridley et al. (1989, 1991) can partly explain the results obtained in our survey. For duration of load testing of wood–adhesive bonds, very little can be found in the literature. One study by Uysal et al. (2010) showed that a long-term storage at elevated temperature can decrease the bonding strength.

Interestingly, the significantly different results for 60°C and 90°C that were found in the long-term tests are in contrast to the short-term tests where no statistical difference in strength was determined. Accordingly, the type of load application (continuously increasing load vs. constantly high load) possibly influences the weakening or dissolving of cohesive bonds in wood and, thus, the behavior of the lap shear specimens.

Analysis of the TTF together with wood failure.—In Figure 6, a comparison of WF and TTF development with increasing load level at 60°C and 90°C is shown. By trend, lower load levels are associated with an increase in TTF and decreasing WF at both temperatures.

At 90°C and load levels of 90, 80, 70 (apart from a few exceptions), and 100 percent, WF is determined, which indicates no damage to the glued joint. With a load level of 60 percent and lower, the WF percentage decreases, and the proportion of failure in wood–adhesive bond increases. This characteristic is even more pronounced for the results at 60°C. In this case, the WF percentage decreases continuously until at 30 percent load level only the wood–adhesive bond fails and no WF occurs anymore. Within the individual load levels, the WF frequently spreads widely, which is

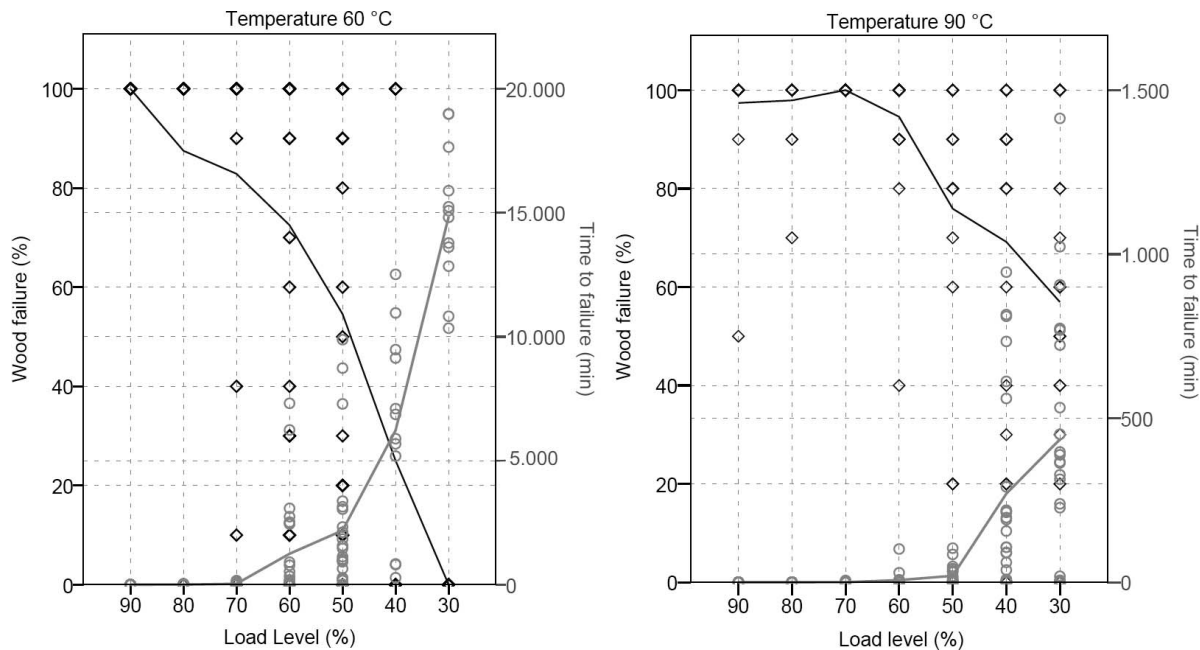


Figure 6.—Relation between wood failure (◇) and time to failure (○) at 60°C (left) and 90°C (right).

presumably mainly due to the strength of the individual specimens.

As can be seen in Figure 6, the WF percentage highly depends on the test duration. With prolonged test duration—caused either by lower temperature or lower load level—the mode of failure increasingly changed from WF to failure in the wood–adhesive bond. Because no further analyses have been carried out, it is not possible to make a quantitative statement as to whether the failure in the wood–adhesive bond is caused by hydrolysis of the adhesive or the weakening of physical bonds between wood and adhesive. However, it has been shown that strength loss in wood–adhesive bonds can occur due to hydrolysis, in particular with formaldehyde-based adhesives such as UF or MUF (Dunky and Niemz 2002). In addition, it should be noted that the hydrolysis is temperature dependent (Ginzel 1971) as well as time dependent, i.e., hydrolytic degradation increases with longer exposure (Freeman and Kreibich 1968, Yamaguchi et al. 1980).

This shift in failure mode from WF to failure in the wood–adhesive bond is considered a relevant indication for a further assessment of adhesive performance and degradation mechanisms in a bondline. However, as different factors such as creep of wood, hydrolysis, and deterioration of physical bonds between wood and adhesive interact with each other, it is difficult to clearly divide between causes of failure without further investigation.

Conclusions

By means of duration of load testing, the influence of the load level, the test temperature, and the resin/hardener ratio on the time to failure of MUF bonded specimens was investigated under water immersion. A significant impact of the temperature on the time to failure could be determined; the mixing ratio, however, did not show any influence. In regression models, high coefficients of determination were obtained, which indicates a good TTF prediction. In addition, the shift from wood failure to adhesion failure

with increasing test duration gives valuable indications for bond deterioration. Overall, the methodological approach and the results are considered to be promising and a valuable basis for future TTF testing. Therefore, it is recommended to further develop the test method for the evaluation of the durability of wood–adhesive bonds.

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