Time to failure analysis of wood adhesives: a non-linear approach based on chemical reaction kinetics

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Similar to wood, adhesives may exhibit duration of load effects. When loaded for longer periods of time, damage processes in the material may develop, eventually leading to failure. From wood research it is known that load level, temperature and relative humidity have an important influence on this behaviour. In general, higher stress levels, temperatures, and moisture content will lead to shorter times to failure and these effects may be more pronounced in loading directions such as shear or tension perpendicular to the grain. It is shown that the reaction kinetics based approach for damage accumulation effects in polyurethane based adhesives can be described using the same non-linear damage accumulation expression as used for wood. The relationship between the time to failure and load-level as influenced by for instance temperature is determined for lap joints, immersed in hot water with temperature of 60°C and 90°C, and at load levels varying between 30 and 90% of the mean short term shear strength.

It is shown that a non-linear damage accumulation expression as used for wood, can also be used for damage accumulation effects in melamine-urea-formaldehyde adhesives. The relationship between the time to failure and load-level as influenced by temperature is determined for beech lap joints loaded in tensile shear. The specimens have been immersed in hot water with temperatures of 60°C and 90°C respectively, and at load levels varying between 30 and 90% of the mean short term shear strength.

1 Introduction

Similar to wood, wood adhesives may exhibit 'duration of load effects'. When loaded for longer periods, creep will occur and damage may develop in the material at sufficiently high stresses, eventually leading to failure. From wood research it is known that load level, generally expressed as ratio between applied stress and short term strength, temperature and relative humidity (or wood moisture content), influence the time-to-failure (TTF). In general, higher stress levels, temperatures and moisture contents lead to shorter time to failure (p.e. Hoffmeyer, 1990, Fridley et al. 1989, 1990, 1991, 1992) and effects may be more pronounced in load directions such as shear or tension perpendicular to the grain (Leontev, 1961).

For the present investigation, the time to failure approach was applied to wood-adhesives and wood adhesive bonds in particular. 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. In Van de Kuilen and Gard (2013), the concept of damage accumulation effects was applied to cracked glulam beams. It is furthermore known that also the behavior of wood adhesives is temperature 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 or melamine-urea-formaldehyde (Dunky and Niemz 2002), but historically used bioadhesives are known to age in similar ways in art and cultural heritage objects (Poulis et al. 2022), (Mosleh et al. 2023).

Research into the duration of load effects of adhesives is important for a variety of reasons. First of all, failures of structures may occur by the time dependent deterioration of the mechanical properties of the adhesives. A reduction of adhesives' shear strength may lead to an increased stress ratio in structural members, partial delamination of members and even crack formation. Therefore, already at the manufacturing stage of engineered wood products, it is important to match the moisture content of the wood as much as possible with the use phase, avoiding crack development and early delamination due to moisture induced stresses when adapting to the environment (Van der Velden and Kuipers, 1976). Also, developments of block glulam with relatively thick gluelines or structures where structural safety is also governed by the glueline itself such as the Swiss TS3.0 system, require information on the long term reliability of the adhesives. Therefore, in this paper, a more profound time to failure analysis is performed on the long term strength of beech lap joints with Melamine-Urea-Formaldehyde adhesive (MUF). This type of adhesive is used in glulam members for applications in Service Class 1 and 2 as specified in Eurocode 5 Timber Structures.

In this paper, it will be shown that the time-to-failure effects in melamine urea formaldehyde (MUF)adhesives, as measured in long term load tests, can be described using a similar duration of load approach as used for wood. A chemical reaction kinetics based approach can be used to describe the relationship between the time to failure and load-level as influenced by temperature. Prior research has shown that a reaction kinetics based approach to duration of load effects (creep, relaxation, damage accumulation, time-to-failure) can be applied to wood, as shown by research works on static loading by Caulfield (1985) and Van der Put (1986, 1989), and timber joints by Van de Kuilen (1999). Clerc et al. (2020) showed that this approach can also be applied to bonded wood lap joints loaded in shear-fatigue at various moisture content and load levels. Wood lap joints with various adhesives types were loaded in shear-fatigue at various moisture content and load levels. In this paper, time-to-failure data is obtained at various load levels and two distinct temperatures, 60° and 90°C, and the relationship between load level and time-to-failure of these lap joints will be described using a non-linear kinetic approach.

2 Materials

Beech lap shear joints were produced and tested in accordance with EN 302-1 (2013). 5 mm thick boards were produced from beech wood with a mean density of 717 kg/m^3 (± 27 kg/m^3 standard deviation (std)) and a mean moisture content of 10.8% (± 0.6% std). Two boards with freshly planed surfaces were bonded with a commercially available melamine-urea-formaldehyde (MUF) adhesive. Series of lap joints were prepared, for testing at 60°C and 90°C respectively and a comprehensive overview can be found in Knorz et al (2018). The short term strength results did not reveal any differences between the samples tested at 60°C and 90°C nor between mixing ratios. Therefore, the mean shear strength of all samples $f_{v,t,\mathrm{mean}}$ = 6.85 N/mm^2 could be used as the reference (100%) load level for the TTF tests. Based on this reference value, the required shear stress for load levels between

30% and 90% were calculated and the required specimen widths were determined. No differences in short term strength could be identified. The mean shear strength was determined at a mean *f* of 6.85 N/mm^2 and series values between 6.68 N/mm^2 and 7.27 N/mm^2 with coefficients of variation (CoV) ranging between 7.9% and 20.2%. The wood failure was consistently 100%, which indicates that varying CoV can be attributed to the raw material. Furthermore, the strength values exceed the requirement of $f_{v,t,\mathrm{mean}} \geq 6.0$ N/mm^2 which is specified in EN 301 (2013) 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. Even though, the strength values in combination with high wood failure indicate a good quality of the MUF bond at these high temperatures.

3 Experimental tests for long term strength of lap joints

Knorz et al. (2018) determined linear time to failure lines for beech lap joints made with melamine urea formaldehyde adhesive. Specimens were immersed in hot water with temperature of 60°C and 90°C, and at load levels varying between 30 and 90% of the mean short term shear strength of 6.85 N/mm^2. The test method and procedure had been modified from the European test method given in EN 14292, for long term testing of adhesive lap joints. There, a continuous increase in air temperature is prescribed until a specimen fails. (For full details, reference to EN 14292 and Knorz et al. 2018). The modifications allow for testing in saturated condition and constant high temperatures, both expecting to contribute to a shortening of the time to failure. An image of a standard lap joint specimen is shown in Figure 1a, while the test set-up for water immersed specimens in shown in Figure 1b.

4 Theoretical approach to the long term strength

The data in Knorz et al. (2018) was used for a linear TTF analysis, meaning the relationship between log(time-to-failure) and stress level is represented by a straight line, as is done in most literature for wood. However, such a linear approach is not always representative for the data that is obtained from experimental measurements. Often this data is difficult to obtain and analyse. In time to failure analysis, a number of issues need to be addressed, especially when time-to-failure times are relatively short, or, in other words, when the relative load level is high. The relative load level is generally expressed as a ratio between

Figure 1a. Standard lap joint specimen (Beech, Adhesive surface l × w = 10×20 mm, width w adapted per series to the required load level)

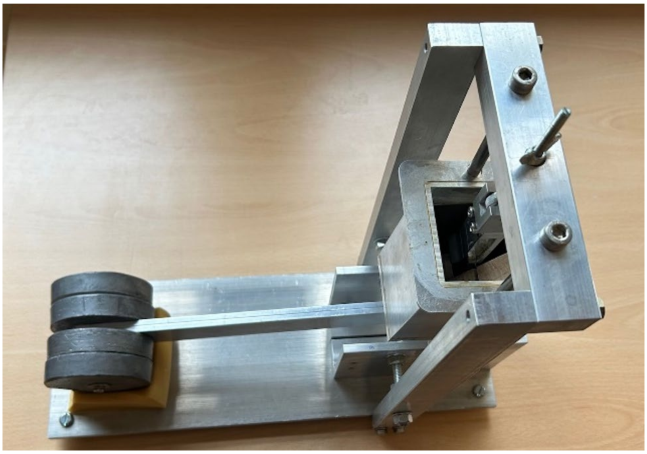

Figure 1b. Tensile testing apparatus with reservoir and level arm

the applied stress σ in a creep test, divided by the short term strength f_s . As the strength cannot be determined on the same specimen as used in the creep test, the stress ratio has to be related to an 'equivalent' short term strength. This value is generally obtained by testing a series of specimens of a single type, and use the mean value of that series as the reference value f_s . Generally, the reference strength is given such that the stress ratio for each creep test series is based on the mean value of the short term strength determined for that specific series tested in short term. Sometimes this reference level is not clearly defined, for instance when different moisture contents are tested, and as a consequence long term strength data becomes difficult to compare between test series and literature. Clerc et al. (2020), who tested fatigue of lap joint specimens, determined a separate short term strength value for each type of specimen, obtaining a well-defined normalized dataset. In this paper, as no differences in short term strength data of the separate series could be (statistically) identified, all TTF data was related to the stress level based on the mean strength of 6.85 N/mm^2.

From the data representation of Knorz et al. (2018) and shown in Figure 1, a certain non-linearity can be observed. This is quite similar to observations made on wood, which can be regarded in this context as a mixture of polymers. Load levels above 80% may lead to data that is influenced by specimens that fail during uploading to the creep level, or very shortly after it. At such a high load level, fracture processes will take place during uploading already and failure will occur during or shortly after uploading. In Figure 1, this can be seen at 70% load level, where specimens at 60°C especially, already show long survival times. At the lower relative load levels ($\sigma/f_s <\approx 0.5$), the time to failure increases more than linearly and consequently merits a non-linear approach. In order to describe the time to failure as a function of the load level, the basic reaction kinetics approach of Krausz and Eyring (1975) is used, which describes deformation kinetics processes in solids.

The non-linear relationship is attributed to multiple energy barriers in a chemical reaction kinetics approach. A single barrier as used by Caulfield (1985) is often sufficient and leads to a linear time to failure equations. In that case, the equivalent exponential damage accumulation equation is covered by the model of Gerhards (1977). However, as wood is more complex, a non-linear solution (assuming two or more types of damage accumulation processes (≈plastic strain and cracking processes) seems more appropriate. Also in the case of bonded assemblies, damage processes at high loads can occur in the adhesive itself, as well as in the bonded assembly (adhesive-wood interface). The shear stresses in lap joint tests are about the same order of magnitude as the shear strength parallel to the grain of wood loaded in tensile-shear, and consequently damage in creep testing is expected to occur both in the wood components as well as in the adhesive layer. Consequently, failure in long term loading of a bonded assembly will be governed by several damage processes running in parallel.

The chemical reaction kinetics approach for multiple barrier systems leads to a non-linear strain rate equation as follows from Krausz and Eyring (1975). This was later applied to wood by Caulfield (1985) assuming a single barrier system would be sufficient to describe the long term behaviour. Around the same time, Van der Put (1986) developed the theory for multiple barrier systems like wood, represented by a parallel series of Maxwell elements with non-linear dashpots, accounting also for plastic strains, i.e. damage development (Eq. 1). The terms $B_i\varepsilon$ account for the plastic strain developing for each creep process (material damage). Other background information can be found in Tang Engelund and Svensson (2011) and Clerc et al. (2019):

$$\frac{d\varepsilon}{dt} = C_1(1+B_1\varepsilon)\left(\frac{\sigma(t)-\sigma_0}{f_s}\right)^n + C_2(1+B_2\varepsilon)\left(\frac{\sigma(t)-\sigma_0}{f_s}\right)^m \tag{1}$$

Integration of Equation 1 gives a non-linear strain development result at the stress level applied. Equation 1 can be written as:

$$\frac{d\varepsilon}{dt} = C_1\left(\frac{\sigma(t)-\sigma_0}{f_s}\right)^n + C_1B_1\varepsilon\left(\frac{\sigma(t)-\sigma_0}{f_s}\right)^n + C_2\left(\frac{\sigma(t)-\sigma_0}{f_s}\right)^m + C_2B_2\varepsilon\left(\frac{\sigma(t)-\sigma_0}{f_s}\right)^m \tag{2}$$

For small values of C_1B_1 and C_2, Equation 2 reduces to:

$$\frac{d\varepsilon}{dt} = C_1\left(\frac{\sigma(t)-\sigma_0}{f_s}\right)^n + C_2B_2\left(\frac{\sigma(t)-\sigma_0}{f_s}\right)^m \varepsilon \tag{3}$$

If, taking into account that elastic strain and plastic strain can be seen as recoverable (elastic) and non-recoverable (plastic) and the latter strains are considered as microstructural ´damage´, this equation is then equivalent to the Foschi and Yao (1986) non-linear damage accumulation equation:

$$\frac{d\alpha}{dt} = F(\sigma(t),\alpha) = a\,[\sigma(t)-\sigma_0 f_s]^b + c[\sigma(t)-\sigma_0 f_s]^n\,\alpha \tag{4}$$

with:

α = damage parameter with $0 \le \alpha \le 1$

$\sigma(t)$ = the stress

σ_0 = the threshold stress level,

f_s = the ultimate strength,

a, b, c, n = model parameters (see also Eq. 3).

The solution of this equation for the time to failure T_f can be written as:

$$T_f = \frac{1}{c(\sigma-\sigma_0 f_s)^n}\ln\frac{1+c(\sigma-\sigma_0 f_s)^{b-n}}{a_0+c(\sigma-\sigma_0 f_s)^{b-n}} \tag{5}$$

Provided that sufficient test data is available to cover the relevant load levels, the parameters can be determined using regression analysis. However, a reduction of the number of parameters is possible. The model parameters *a* and *c* are the reaction rate constants for the energy barriers of the two processes:

$$a,c = \frac{k_B T}{h} \cdot e^{(-\frac{E_a}{RT})} \tag{6}$$

In which T is the temperature (K), k_B is the Boltzmann constant, h is Plank's constant, R is the gas constant, and E_a the activation energy (Krausz and Eyring, 1975), Caulfield (1985). Model parameters b and n represent the work needed as a function of the external mechanical stress for molecules to pass the energy barrier when shifting into new positions. As the processes studied here lead to failure (high stress), the second part of equation 4 (describing the damage) will be primarily determining the time to failure, reducing the model parameters to be determined to b, c, and n, apart from the threshold level σ_0 .

The problem with run-outs, specimens that have survived the long term tests, can be treated in various ways. Van de Kuilen (1999) in the case of constant load joints (creep-failure) or Clerc et al. (2019a, 2019b) in the case of fatigue failure of bonded wood. Van de Kuilen (1999) assumed that at each load level, the times to failure follow a log-normal distribution, whereas in Clerc et al. run-outs were analysed using the method of Castillo et al. (2009).

The level of threshold stress is set to 0 and 0.05 for the analysis in this paper, as no experimental evidence of such a level is present and failures occur at low load levels and temperatures are high. However, the limit value for $\sigma \rightarrow 0$ will automatically lead to very long TTF values, and it can be seen from Figure 1 that there is a tendency that for very low stresses the TTF values increase more than linear with log(t). For wood, this level is estimated to be between around 0.5 - 0.6 based primarily on experiments with static bending loads and 0.3 - 0.4 for joints in tensile loading with shear failure planes, as well as fatigue loads, see e.g. Foschi and Yao (1986), Foschi et al. (1989), Van der Put (1986), Van de Kuilen (1999), and Clerc et al. (2019).

5 Results

In Figure 1, the results of the non-linear time to failure approach are shown, together with the standard linear TTF-approach of Knorz et al. (2018). The results show that for creep to failure loading, a non-linear approach is able to cover the temperature influence on the time to failure better than the linear approach. The TTF-values at a certain relative load level, shift to the right on the log(t) axis at decreasing temperature from 90° to 60°C. On the other hand, for a linear log(t) regression, that regression line also shifts vertically, which cannot be correct when the vertical axis is given as relative load level. For small values of t, the relative load level must pass the 100% short term strength coordinate.

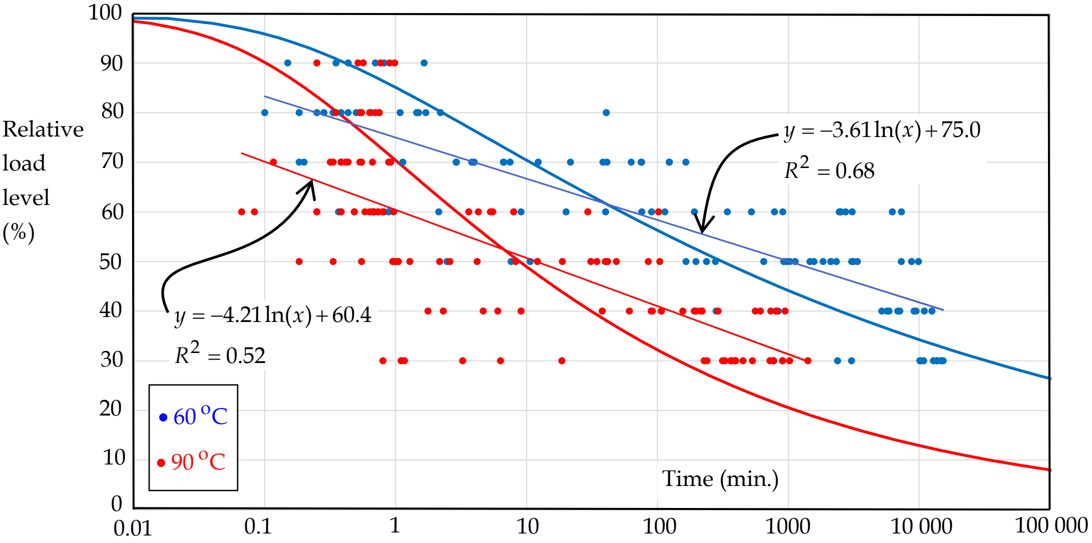

Figure 1. Time to failure results of MUF adhesives under wet creep-failure loading at 60° and 90°C

The parameter estimation is summarized in Table 1 for relative stress threshold levels of 0 and 0.05. For the adhesives analysed here under warm and saturated conditions, it can be seen that at 90°C the threshold stress will be close to zero, but for 60°C a threshold level of around 0.1 could be realistic, but this is still much lower than the threshold level for wood or wood fatigue. The values of the parameters are given in Table 1.

A number of advantages of the chosen non-linear approach can be observed:

- the regression equation passes through the short term strength (absolute value or relative value) of the material at time t equivalent to a short term tests (3 - 5 minutes);
- the short survival times of test specimens at load levels above 80%, and consequently short times to failure, is better accounted for than with a linear approach;
- the model has a threshold stress level, below which no failure will occur.

The non-linear relationship is attributed to multiple energy barriers in a chemical reaction kinetics approach. A single barrier as used by Caulfield (1985) is often sufficient, and leads to a linear time to failure equations. The solution of the damage accumulation equation is then equal to the exponential damage equation of Gerhards (1983). However, as wood and wood adhesive bonding are more complex, a non-linear solution (assuming two or more types of damage processes) seems more realistic, as has been shown on wood in static loading (Van der Put, 1986), (Foschi and Yao, 1986), in fatigue loading (Clerc et al, 2020) and also for timber joints (Van de Kuilen, 1999). It is now also shown to be applicable to adhesives. In the case of bonded assemblies, a damage process in the adhesive itself, as well as a damage process in the bond assembly (adhesive-wood interphase) can be present. The shear stresses on lap joints are relatively low for the wood components that are loaded in tension, and consequently no damage is occurring in the wood components.

Table 1. Estimated parameters

Temperature	T	60°C	90°C	60°C	90°C
Threshold	σ_0	0	0	0.05 N/mm^2	0.05 N/mm^2
Parameters (Eq. 5)	b	77.395	35.802	35.352	4.389
	n	8.081	4.392	7.142	3.904
	c	8.240E-06	0.012911	2.98E-05	5.78E-03

Another issue relates to the quality of fit of the regression and the fitted parameters. From Foschi et al. (1989), it can be seen that the scatter in values (b, c, n) can be quite high for bending tests on three different wood species. On the one hand, this can be attributed to the sensitivity of the model to logarithmic processes and the difficulty of regression when having data covering such long time-scales, on the other hand to the fact that obtaining a large dataset from time to failure tests is costly and time consuming, so the amount of available data is nearly always limited. Here, the GRG non-linear solver option of Excel was used for the regression analysis. Determining the regression constants by determining the least squares value using the GRG method indicated that multiple solutions are possible, giving almost the same minimum, making it difficult to determine some of the underlying physical principles. This is subject of further research. In addition, the sensitivity of the adhesive to load and temperature has been shown to be considerable when increasing the temperature from 60° to 90°. The effects should also be studied for more practice-oriented temperature ranges between 10° and 60°. Especially for applications with relatively thick gluelines, such as block-gluing, the glueline itself might become relevant.

6 Conclusions

It has been shown that a reaction kinetics based approach for adhesively bonded joints can be used to describe the test results from data that shows a clear non-linear time to failure effect. The study was performed at 60°C and 90°C, and time to failure was reduced considerably for the 90° specimens as compared to the 60°C. It can be concluded that the long term strength of adhesives can be described with a reaction kinetics model for bond breaking and reformation, and that this can be done in a similar manner as is done for time-to-failure effects in wood. The MUF adhesive used in this study, is a standard adhesive and approved for structural applications in accordance with EN 301/302 standards and requirements. Consequently, a long term strength analysis for adhesives under the influence of combined effects of mechanical loads and influences of temperature and/or moisture might become necessary and could be introduced in design codes for timber structures. Especially for applications with relatively thick gluelines, such as those that occur in block-gluing of glued laminated or cross laminated timber, the influence of long term strength of the glueline itself might become relevant for structural safety.

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