# Fire resistant adhesive bonds for load bearing timber structures

## Development of a small-scale test method

Magdalena Sterley Joakim Norén

RISE



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# Sammanfattning


En småskalig testmetodik för att utvärdera limfogar vid brand har utvecklats. Syftet är att få ett enkelt verktyg för att kunna skilja mellan brandbeständiga och icke-brandbeständiga lim. CLT-prover exponerades vid värmeflödet 50 kW/m$^2$ som motsvarar verklig brandpåverkan. Brandprovningen utfördes i konkalorimetern enligt ISO 5660, som är ett mångsidigt och vetenskapligt erkänt instrument i liten skala. De stora fördelarna är snabb, enkel och kostnadseffektiv metodik att bestämma grundläggande materialegenskaper vid brand.

CLT limmade med 5 olika limsystem provades med solitt trä som referens. Densiteten för den yttre lamellen var ungefär samma i alla prov och 430-450 kg/m$^3$. Proverna brandexponerades och förkolning fram till limfogen tog 26 minuter. Förkolningshastigheten var 0,77 mm/min, vilket motsvarar hastigheten vid fullskalig brandprovning enligt standardbrand. Temperaturen i limskiktet var då 270°C. Direkt därefter utsattes proven för skjuvning vid 260°C. Brottslast och typ av brott bestämdes. Resultaten visar att brotten var av tre typer: i limskiktet, i kolskiktet och en blandning av dessa typer. Brotten skedde i den svagaste zonen. Brott i limfogen betyder att den var svagare än kolskiktet och brott i kolskiktet betyder att limfogen var starkare. Två av de  testade limmerna uppvisade högre styrka och brott i kolskiktet vilket tyder att dessa två limmer har en stark limfog även under förkolning i 260°C. Ett av limmerna uppvisade brott i limfogen med låg brottlast. Se bild nedan med de två olika brotttyperna.

Dessutom analyserades proverna enligt Dynamisk Mekanisk Analys (mekanisk provning av småskaliga prover under en dynamisk belastning och ökande temperatur), som visade att tre av de provade limmen hade konstant E-modul vid temperaturer 30-250°C. Ett av limmen hade låg E-modul, som dessutom sjönk med ca 50 % vid de högre temperaturerna. Detta lim hade lägst styrka och brott i limfogen vid brandprovningen, vilket visar att detta lim troligen har sämst egenskaper och störst risk för delaminering vid brandpåverkan. De två lim som uppvisade brottstyp i kolskiktet med stark limfog, hade också stabil E-modul i DMA analysen. Dessa resultat antyder att de två presenterade metoder kan vara korrelerade.

Inte alla testade lim var avsedda för bärande konstruktioner, men några av dem används i CLT-produktion och de visar olika brottbeteenden i denna studie, vilket kan indikera att de kommer att uppträda på olika sätt i brand. Om dessa slutsatser är kopplade till delaminering under brand kan bekräftas genom att jämföra nuvarande resultat med test större skala.

Denna nya småskaliga metodik måste korreleras med brandprovning i större skala, t ex i en kubikmeters modellugn eller i fullskaliga lägenhetsmoduler för att säkerställa relevansen. Sådana provningar pågår och några resultat finns tillgängliga, där samma lim använts. För några lim behöver fler prover utföras enligt den småskaliga metodiken. Den nya metodiken inkluderar många parametrar som möjliggör korrelation med prov i större skala.

Det är vår önskan och avsikt att fortsätta arbetet tillsammans med industrin för att få den nya småskaliga metoden verifierad och användbar i praktiken.

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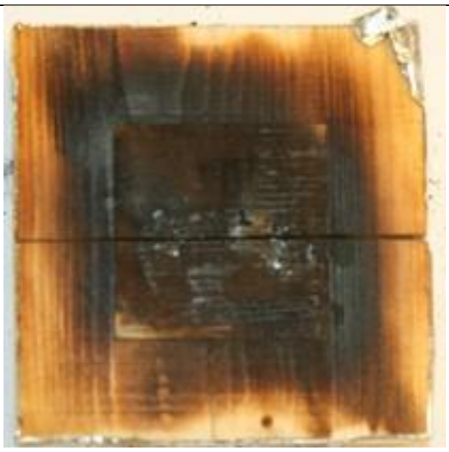
|  |  |
| Brott i kolskiktet | Brott i limfogen |

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# Abstract

A small scale fire test methodology for evaluation of adhesive bond performance in fire has been developed. The aim is to get an easy tool to distinguish between fire resistant adhesive bonds and non-fire resistant bonds. Glued CLT-specimens were exposed to constant heat flux at 50 kW/m2 in the cone calorimeter representing real fire exposure. The cone calorimeter in accordance with ISO 5660 is one of the most widely used bench-scale instrument in fire research. The original set-up of this device was to target the material properties. In the course of time, other potential uses have been studied and the cone calorimeter is finding an increasing implementation as a characterization tool for building products in research. This small-scale device has several advantages over larger-scale tests thanks to its fast, simple and cost-efficient manner to investigate basic material properties. Specimens glued with 5 different systems were tested and also solid wood as reference. Similar wood density was chosen of 430-450 kg/m3 for the outermost exposed lamella which was 20 mm thick. Specimens were exposed to heat flux heater and charred to the bond line, which took 26 min. The mean charring rate was 0,77 mm/min which is equivalent to fire testing in full scale. The temperature in the bond line was then 270°C. Directly thereafter specimens were placed in a mechanical testing machine for determination of shear strength at 260°C. The failure load and failure mode were determined. The most important result is that the bond lines failed in three separate failure modes: bond line failure, char failure and mixed bond line and char failure. The bond line failure could result at low char strength up to char strength. Failure in the char occurred at varying loads. Failure occurred in the weakest zone so when failure in the bond line occurred it means that bond line was weaker than char layer, when char layer failed it means that the bond line was stronger. Not all of the tested adhesives were aimed for load bearing constructions, but some of them are used in CLT production and they show different failure modes, which can indicate that they will behave in different way in fire. If those failure modes are connected to delamination during fire can be confirmed by comparing present results with larger scale testing.

In addition, Dynamical Mechanical Analysis (DMA) was used and showed that three of the tested adhesives exhibited constant modulus of elasticity (MOE) in the temperature scan from 30 to 250° C. One of adhesives showed low MOE and also that MOE decreased to about 50% during the temperature rise. This adhesive showed the lowest strength and bond line failure in the fire test. Those two results support the conclusion that this adhesive could possibly possess worst properties during fire with possible delamination.

Correlation of the present results with testing in larger scale, as testing in 1m$^3$ model furnace or full compartment testing, is needed to verify that the presented method is relevant for classifying adhesives with respect to delamination at fire exposure. Such tests are available and in some cases the same adhesives systems have been tested, and in other cases additional tests would be needed. Thus, some of such correlation can already be validated. It is our wish and ambition to continue our

work with industry as next step of this project. Another spinoff of this project is the complementary testing with our small scale method of adhesives tested already in full scale, contacts with industry was already established. The very cost efficient, small scale method, is useful for fast and reliable evaluation of adhesives system to determine their behaviour in fire with respect to delamination. The presented method is an example of such a method where many parameters are chosen and designed in such a way that the correlation with larger scale tests can be obtained.

# Background

Wood construction is growing rapidly and provides a substantial contribution to the development of a more sustainable construction sector. Several modern wood-based building systems are developed with focus on tall wooden houses and industrial production, where glued products are an important part. Fire safety is important, but the adhesive properties in fire conditions are not fully understood. This applies in particular to new adhesive systems, but also to existing ones which exhibit poor load carrying capacity in fire.

The problem has been noted by the FSUW (Fire Safe Use of Wood) global network, which formed a sub-group of "Glue-line failure of engineered wood products" with representatives from Australia, NZ, Canada, USA, France, Italy, Switzerland, Sweden and Germany. The global network has gathered knowledge and experience from known cases of fire testing of glued wood components (especially glulam, finger joints and CLT) and has defined research needs. The results highlighted by this group relate to delamination of glued bonds in fire which can cause increased charring of glued wood products, especially for CLT. The results show that the temperature during standard fire testing increases continuously without cooling phase and with delamination of CLT until a collapse of the structure occurs.

Thicker CLT may be required to reduce delamination risks or to protect the wood material. This can lead to increased costs and greater weight of the construction as well as reduced possibility of using visible wood.

Therefore, it is of the utmost importance to find methods for evaluating the adhesive bond properties in fire. The hypothesis is that different adhesive systems have different behaviour in fire, and especially that delamination behaviour can be avoided by choosing a suitable adhesive system. The best method for evaluation of fire delamination is a full scale test, but considering the high costs of such full scale tests, a smaller scale test needs to be developed. The intention is that such small-scale methods should give the same results as full-scale tests. A new, smaller scale method for classifying adhesives with respect to fire properties would also simplify the planning of full scale tests.

In parallel with the small scale fire tests, DMTA (Dynamic Mechanical Thermal Analysis) of the tested adhesive systems is planned. This method is used to characterize polymers with regard to their viscoelastic properties at different temperatures and loads. The combination of these two methods can probably provide better opportunities for classification of glue with regard to fire.

The aim of this preliminary study is to develop a small-scale method for evaluating the behaviour of various adhesive systems in fire. In this study adhesive bonds in CLT have been tested. Other investigations, in cooperation with adhesive manufacturing industry, and with small scale methods for finger joints and glulam were carried out at SP (now RISE) in 2015-2016 with promising results and a possible classification of adhesives. At RISE projects with fire safety in glued wood components are also underway, where full scale and meso-scale tests are planned. These provide an opportunity to test the same adhesive system in small scale as in the other projects and verify if results from small scale tests are correlated with full scale tests.

# Methods and materials

## Cone calorimeter

The cone calorimeter used in accordance with ISO 5660 is one of the most widely used bench-scale instrument in fire research, see Fig. 1. The original set-up of this device was to target the material properties. In the course of time, other potential uses of this equipment have been studied and the cone calorimeter is finding an increasing implementation as a characterization tool for building products in research. This small-scale device has a number of advantages over larger-scale tests thanks to its fast, simple and cost efficient manner to investigate the basic properties of materials.

When testing with the cone calorimeter a specimen is exposed to a heat by radiation from the heating source – a cone heater with a constant heat flux calibrated with the reference heat flux meter. The irradiance from the heater to the tested surface of a specimen is distributed uniformly within at least the central 50 mm x 50 mm area of the exposed surface area. A standard sample size surface area is 100 mm x 100 mm.

## Test specimen

CLT specimens were prepared by participating companies. The specimens were glued of spruce (Picea abies) with 5 different adhesive systems, both structural and non-structural adhesives. Among the different adhesive types there were also adhesives included that are presently used for CLT-production. Test specimens of dimensions 55x100x100 $mm^3$ were cut from the CLT. The thickness of the outermost fire exposed lamella was 20 mm (see Figure 2). The total glued surface was 100x100 $mm^2$. A notch was cut around the specimen, through the bond line, leaving the active glued area of approximately 50x50 $mm^2$. In order to measure the temperature during the fire test and estimate the temperature in the bond line during the shear loading test, thermocouples type K were inserted behind the bond line in the centre of the specimen, see Fig. 3 left. Thermocouples were placed parallel to the bond lines. The goal was to obtain temperatures around 270°C in the bond line before and during mechanical testing.

The test specimen was covered with 15 mm thick gypsum plasterboard Type F from four sides. Gypsum plasterboard of thickness 6 mm was also partially applied on the top surface area of the exposed specimen to protect the sides from charring. All joints of the gypsum boards were covered with aluminum tape. It was important to protect the timber and the notches around the specimen and maintain uncharred solid timber on the edges of the specimen to be able to expose the specimens later for shear load. At the same time, the size of the plaster board strips on the tested surface was designed and verified by testing to maintain a uniform heat distribution on the ~~c~~centre surface. The width of the gypsum strips was chosen to 2,5 cm. After the application of gypsum plasterboards on top of the specimen, the exposed surface area was 8 x 8 cm, see Fig. 3 right.

## Fire testing

Small size samples of CLT with different adhesives were tested with a cone heater of a cone calorimeter (ISO 5660). Solid wood specimens prepared in the same way as the glued specimens were also tested. The specimen was positioned in a horizontal orientation directly under the cone heater, see Fig. 4. The vertical distance between the cone heater and the surface of the specimen was fixed to 25 ± 1 mm. A constant predetermined heat flux level of 50 kW/m² was used in all tests.

At first, the fire tests were terminated when a temperature value of 270 degrees was reached in the bond line. However, this method showed somewhat inaccurate charring depths for various specimens which was most probably a result of an erroneous insertion of the thermocouples. The exposed surface area was also changed from 5 x 5 cm to final 8 x 8 cm in order to obtain even heat distribution over the whole active glued area. These first results were therefore disregarded and are not reported here. Then, another set of test specimens was tested with a fixed heat exposure time period of 26 minutes. This new approach yielded equal charring depths of specimens and ensured similar conditions for each specimen to be tested for shear load after fire testing.

The temperature behind the bond line was monitored through the whole test.

## Mechanical testing

Directly after exposing the specimens in the cone, the specimens were removed from the heater. The gypsum plasterboards were removed and the timber specimen was positioned under the shear loading device, see Fig. 5. The specimens were fixed on two lateral sides. The load was applied directly on the exposed side of the timber lamella from the thinner side and mounted in the Alwetron TC 50 testing machine for determination of the residual shear strength. The temperature in the bond line during mechanical testing was around 260 degrees and loading rate was 5 mm/min. The smouldering of the wood was visible during shear testing ensuring that bonds are tested while wood is charring. Two nominally equal tests were performed to achieve reliable test results.

The failure mode was analysed thereafter as well as the charring depth. Failure in the char layer, failure in the bond line and mixed failure, both in the bond line and char, were observed and assigned to each specimen (see Figure 6 and appendix).

Figure 1. Cone calorimeter (left) and test specimen during testing (right)

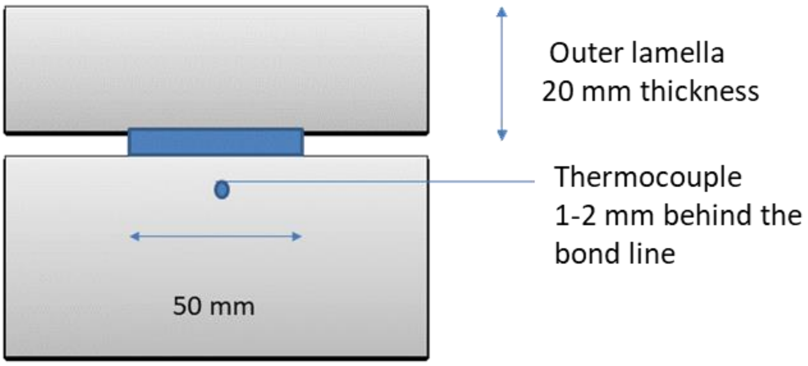


Figure 2. Small scale CLT specimen for fire testing without gypsum board protection

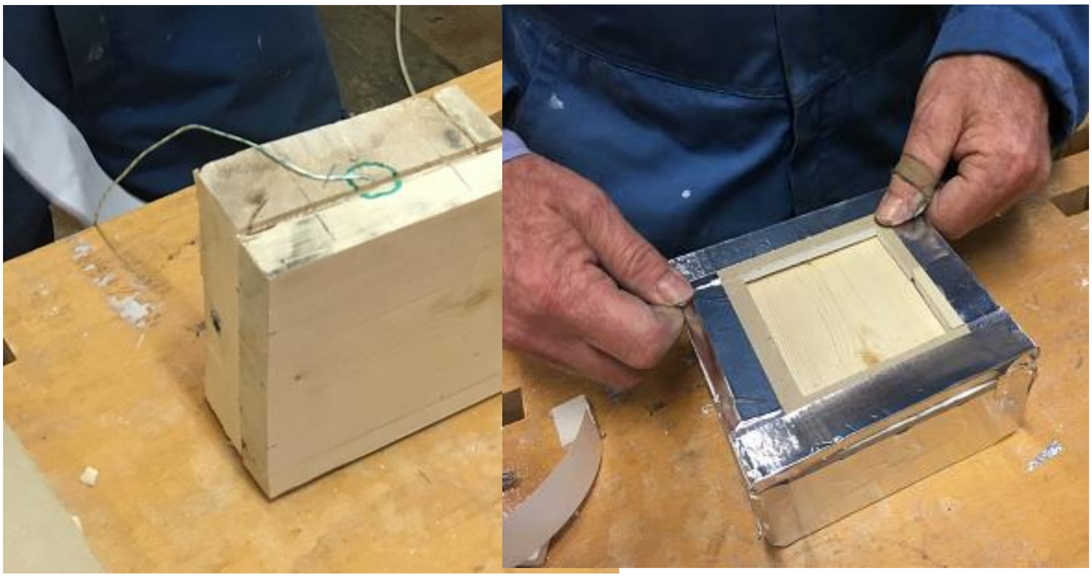

Figure 3. Left – Insertion of thermocouples to the bond line; Right – Application of gypsum plasterboards to the sides of timber specimen.

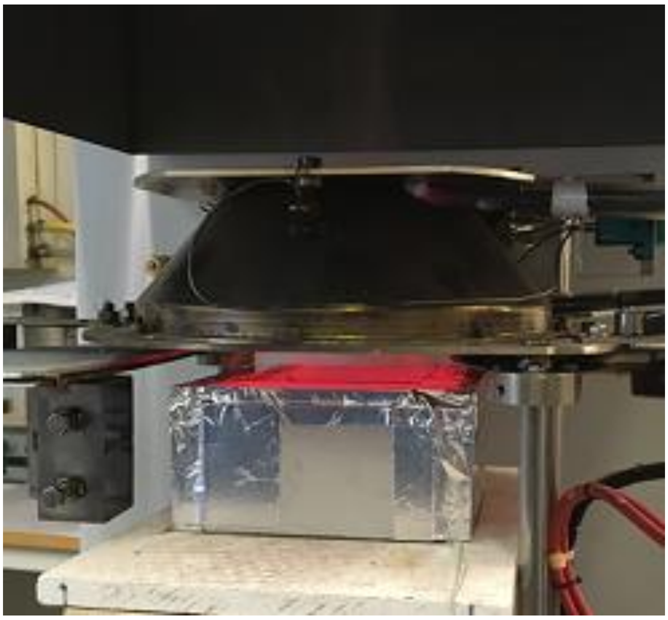

Figure 4. Fire testing with cone heater.

Figure 5. Load test: temperature monitoring (left)

Loading of specimen with visible smouldering (right)

Figure 6. Test specimen after load test.

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# Most important results

## Residual shear strength and failure mode of the bond line at 260°C

There are two main results from each test:

-residual shear strength

-failure mode.

Results are shown in Table 1. Type of adhesive is not revealed and is not important, only the correlation with full-scale tests is needed.

Table 1. Results from fire testing

| Adhesive no | Total test time | Failure load (N) | | Average failure load | Residual shear strength | Density of outermost lamella | Moisture content | Failure mode |
|---|---|---|---|---|---|---|---|---|
| | (min) | specimen 1 | specimen 2 | (N) | (MPa) | (kg/m$^3$) | (%) | |
| 1 | 26 | 202 | 227 | 215 | 0,08 | 430 | 11,5 | partial char failure/ bond line failure |
| 4 | 26 | 219 | 182 | 201 | 0,074 | 460 | 11,2 | bond line failure |
| 5 | 26 | 203 | 102 | 153 | 0,058 | 420 | 10,8 | char failure |
| 6 | 26 | 211 | 316 | 264 | 0,098 | 460 | 12,1 | char failure |
| 7 | 26 | 85 | 83 | 84 | 0,031 | 430 | 12,6 | bond line failure |
| Wood | 26 | 382 | 393 | 388 | 0,144 | 430 | 12,4 | char failure |

The strength of a char layer is normally assumed to be the same for any charred wood and close to zero, however various strengths were measured even when only the char layer failed.

Results show that charred wood of the solid wood specimens had the highest strength of 0,144 MPa at hot state compared to glued specimens. However, the glued specimens in the present study were glued with the layers oriented at 90 degrees of fiber direction to each other, and bond line in cross laminated timber have normally lower strength compared to parallel glued bonds. Moreover, the failed area of the char is not exactly 50x50 mm$^2$, but is not plane and even. Among the glued specimens, the best adhesives were no 6 and no 5 with respect of failure mode - both no 6 and no 5 failed only in the charred layer. No bond line failure could be observed for those adhesives. Adhesive 1 had relatively high strength but the failure mode was mixed, partially in the charred layer and partially in the bond line. Significantly lower strength and also bond line failure was observed for Adhesive 7. An interesting result was obtained for adhesive no 4 which possessed relatively high residual strength at the hot state but the failure mode was in the bond line. See appended pictures of specimens.

The presented method shows that different adhesives have different behaviour during charring especially with regard to failure mode. Some adhesives failed in charred layer some of them failed either to some extent in the bond line and some only in the bond line. The residual strength of the bond in the hot state during charring determined with the present method seems to vary a lot between different specimens and systems. However, the failure mode is showing the weakest link of the tested joint. The most important result outcoming from present method is the failure mode which shows if the adhesive can still maintain some strength even in charred zones. Failure in charred zone mean that bond line was stronger than char.  Not all of the tested adhesives were aimed for load bearing constructions, but some of them are used in CLT production and they show different failure modes. If those failure modes are connected to delamination during fire can be confirmed by comparing present results with full scale or meso-scale testing.

When failure occurs in the bond line the failure load is important representing adhesive strength. When the failure occurs in the char the load can vary because of the fact that failure do not occur in plane but in 3 dimensions. There is no failure plane (see specimen 5, 6 and wood in appendix).

## Dynamical Mechanical Analysis

Four of tested adhesives were tested with Dynamical Mechanical Analysis (DMA).

Results are shown in Figure 7.

Figure 7 illustrates and confirms the results from the fire testing. The best adhesive with highest stiffness was adhesive 6. The modulus of elasticity (MOE) of this adhesive is highest and constant during heating up to 250°C. The second and third ranked adhesives with respect to stiffness were in the same order as the ranking obtained in the fire tests: adhesives 1 and 5, respectively (there regarding residual shear strength). Both adhesives show stable MOE during the whole heating process. However, adhesive no 7, which showed to be the weakest one at fire testing, showed both the lowest MOE and, in addition, the MOE diminished while temperature was increasing, with a loss of approximately 50% of MOE at 250°C.

Regarding results both from fire testing and DMA adhesive 7 shows properties which are not favourable for fire conditions. The difference in terms of failure load, failure mode of the bond line and low and diminishing stiffness in heated condition was clear between adhesive 7 and the other tested adhesives.

It is important to underline here that the present study shows a possible methodology for classification of adhesives. More results and more different adhesives need to be investigated to be able to draw any final conclusion about possible delamination during fire.

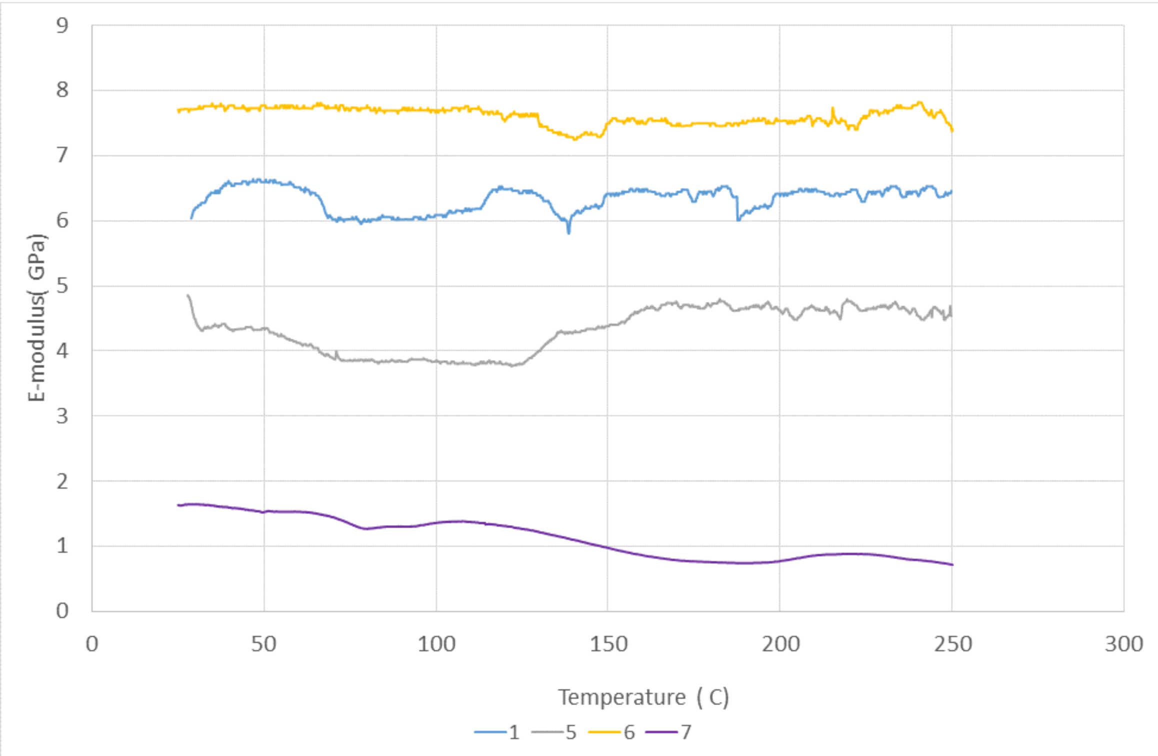


Figure 7 Modulus of elasticity in temperature scan measured by DMA.

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## Further work

The correlation of the present results with testing in larger scale, as testing in $1m^3$ model furnace or full compartment testing, is needed to be sure if the present method is relevant for classifying adhesives with respect to delamination. Such tests are available and in some cases the same adhesives systems have been tested, and in other cases additional tests would be needed. Thus, some of such correlation can already be validated. This is our wish and ambition to continue our work with industry as a continuation of this project. Another spinoff of this project is the complementary testing with our small scale method of adhesives tested already in full scale, contacts with industry was already established. The very cost efficient, small scale method, is useful for fast and reliable evaluation of adhesives system to determine their behaviour in fire with respect to delamination. The presented method is an example of such a method where many parameters are chosen and designed in such a way that the correlation with larger scale tests can be obtained.

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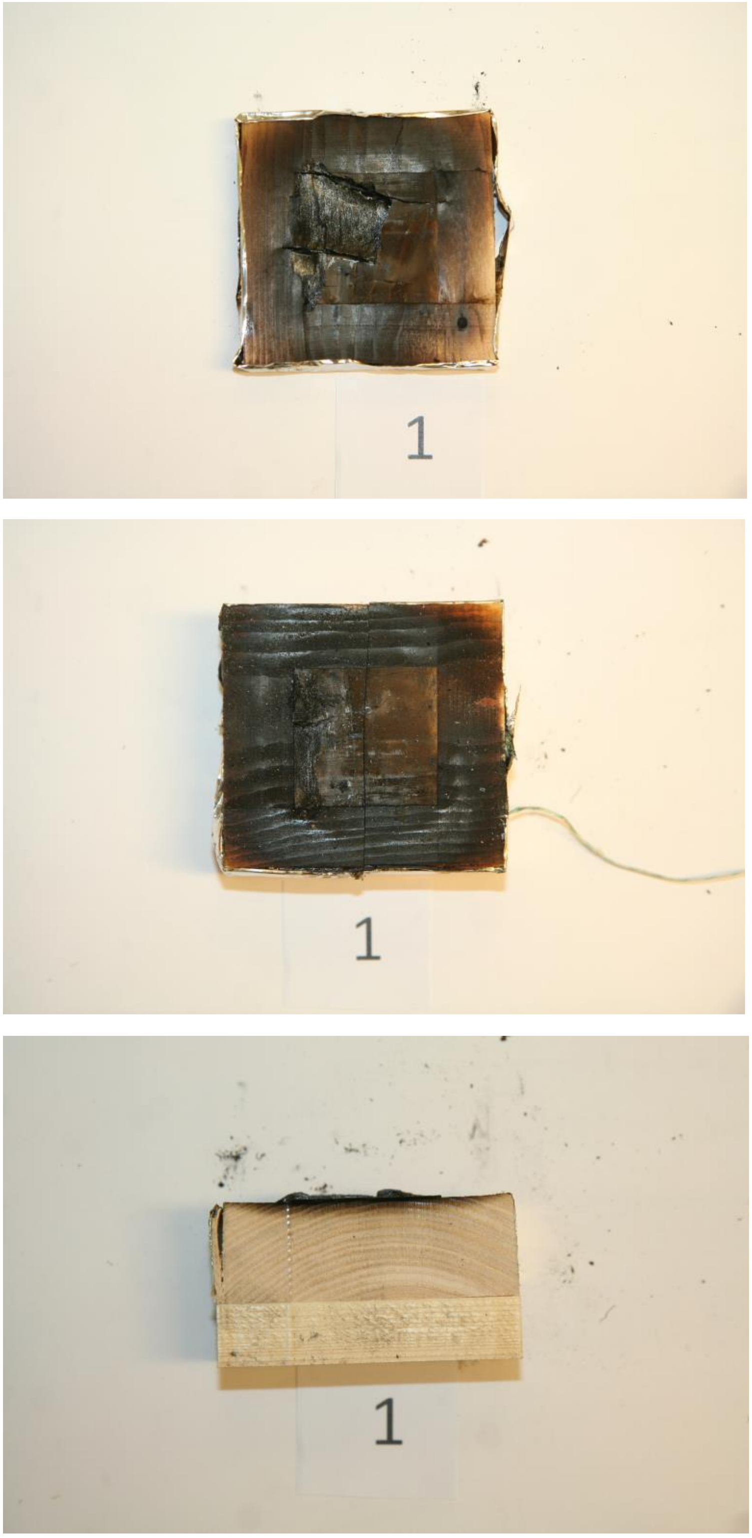
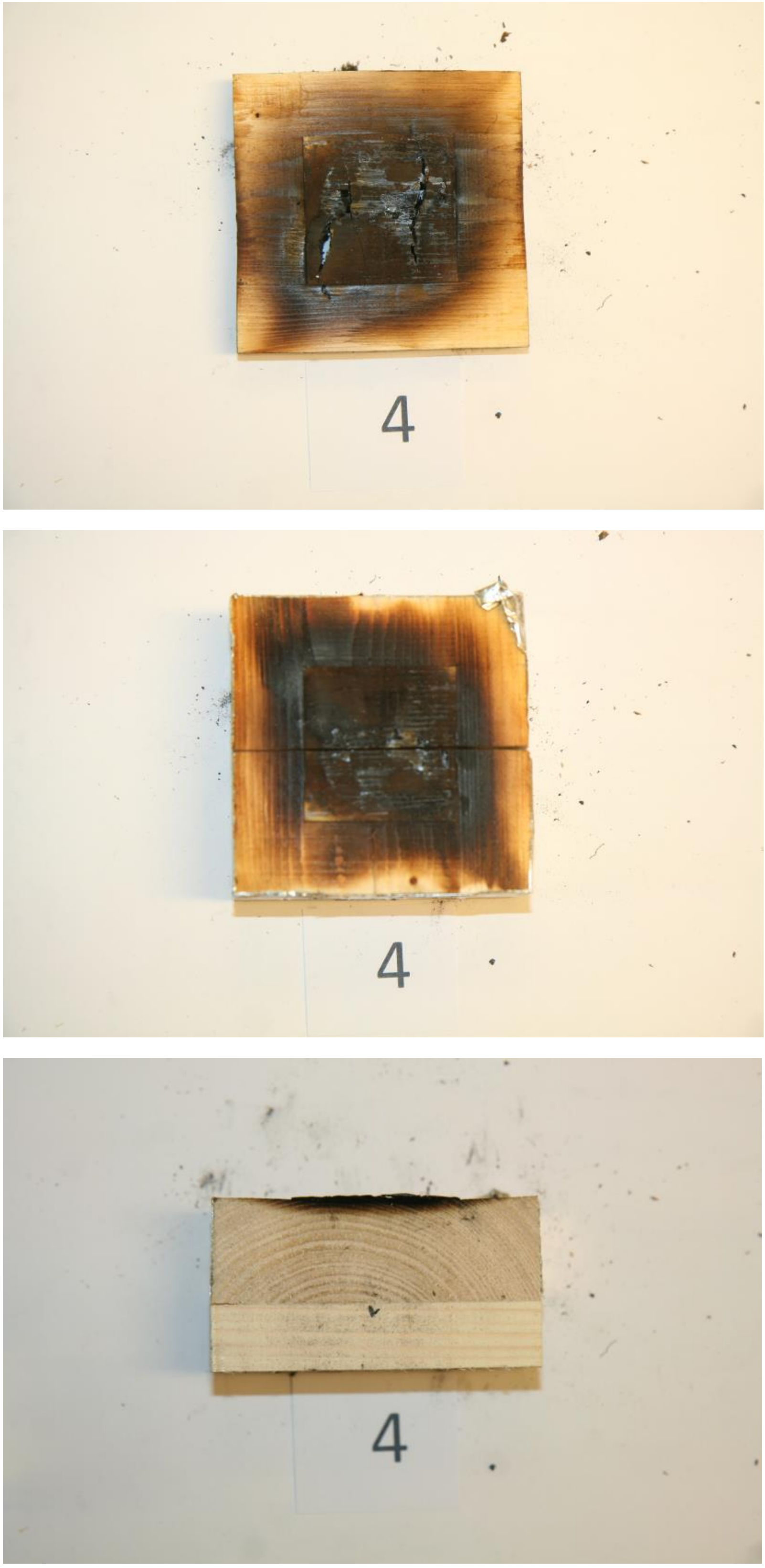
# Appendix 1 Test specimens after failure




Test specimen no.1 after failure. Partial char and bond line failure.

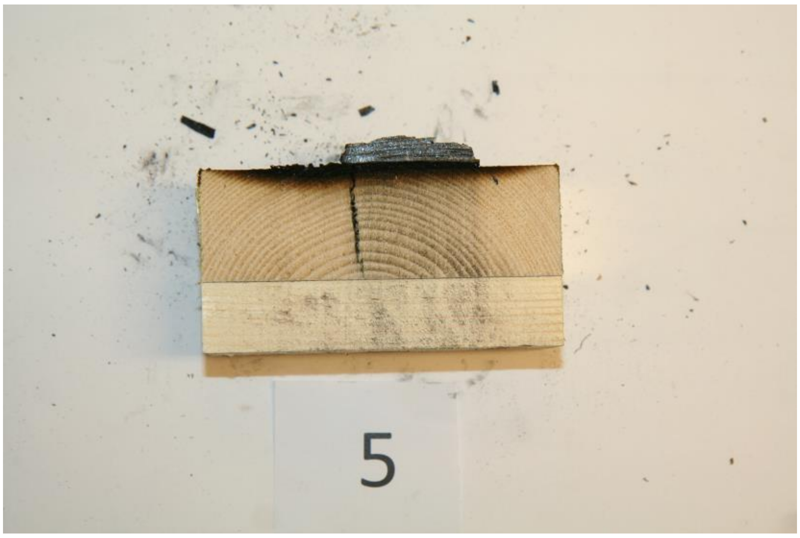
Appendix 1. Test specimens



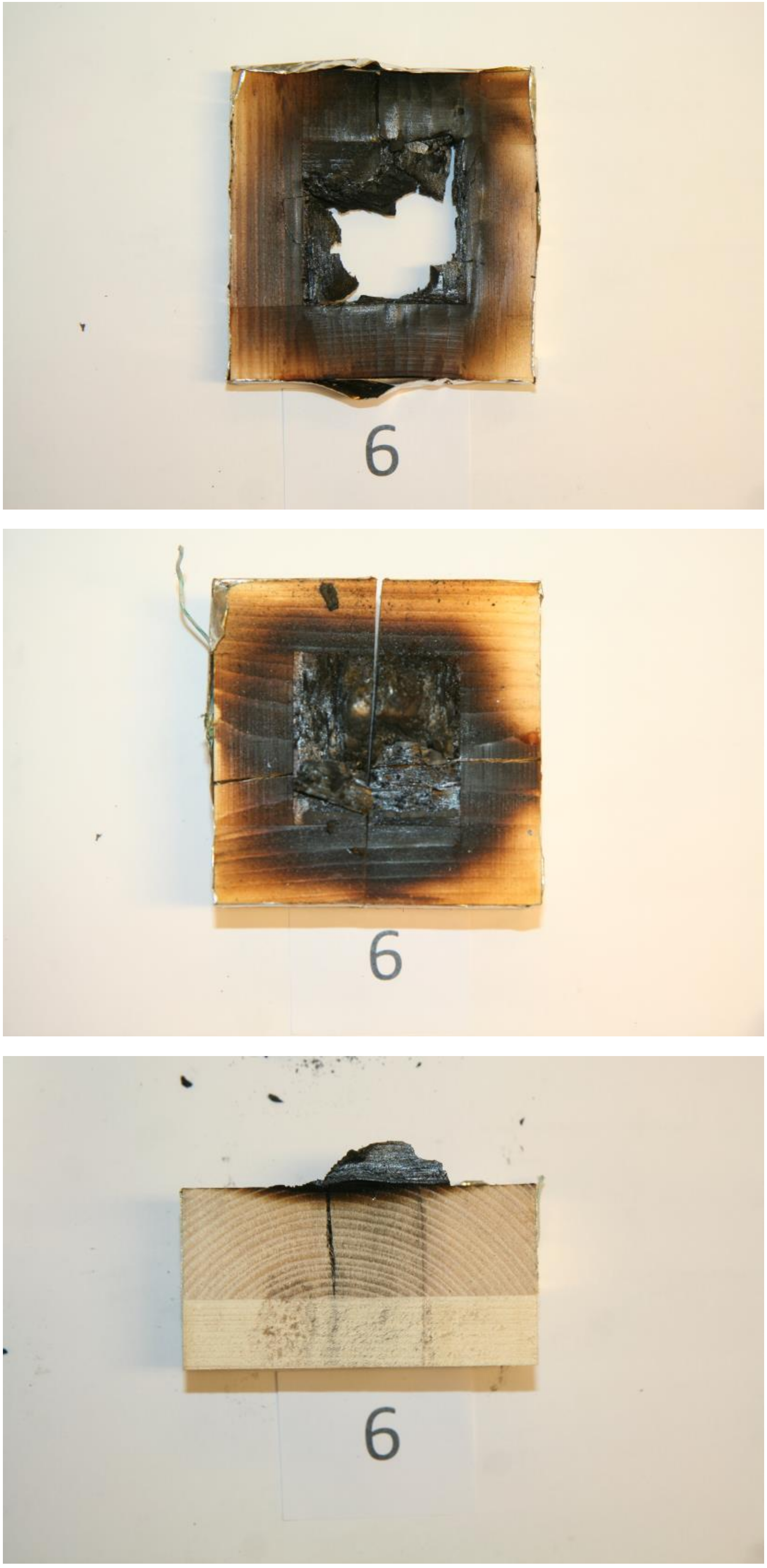

Test specimen no.4 after failure. Bond line failure

Appendix 1. Test specimens

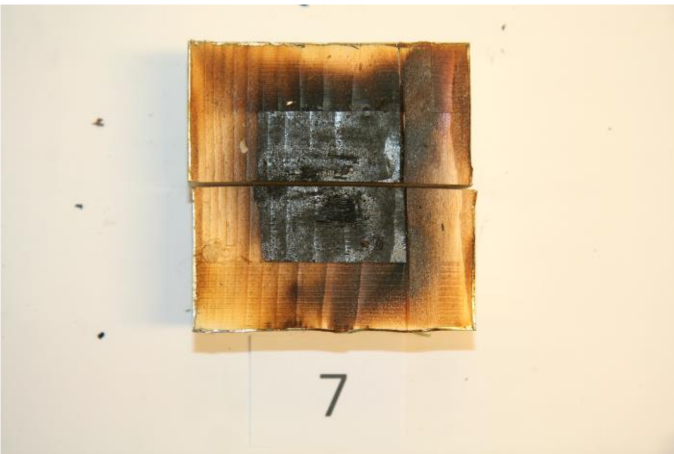





Test specimen no.5 after failure. Char failure.



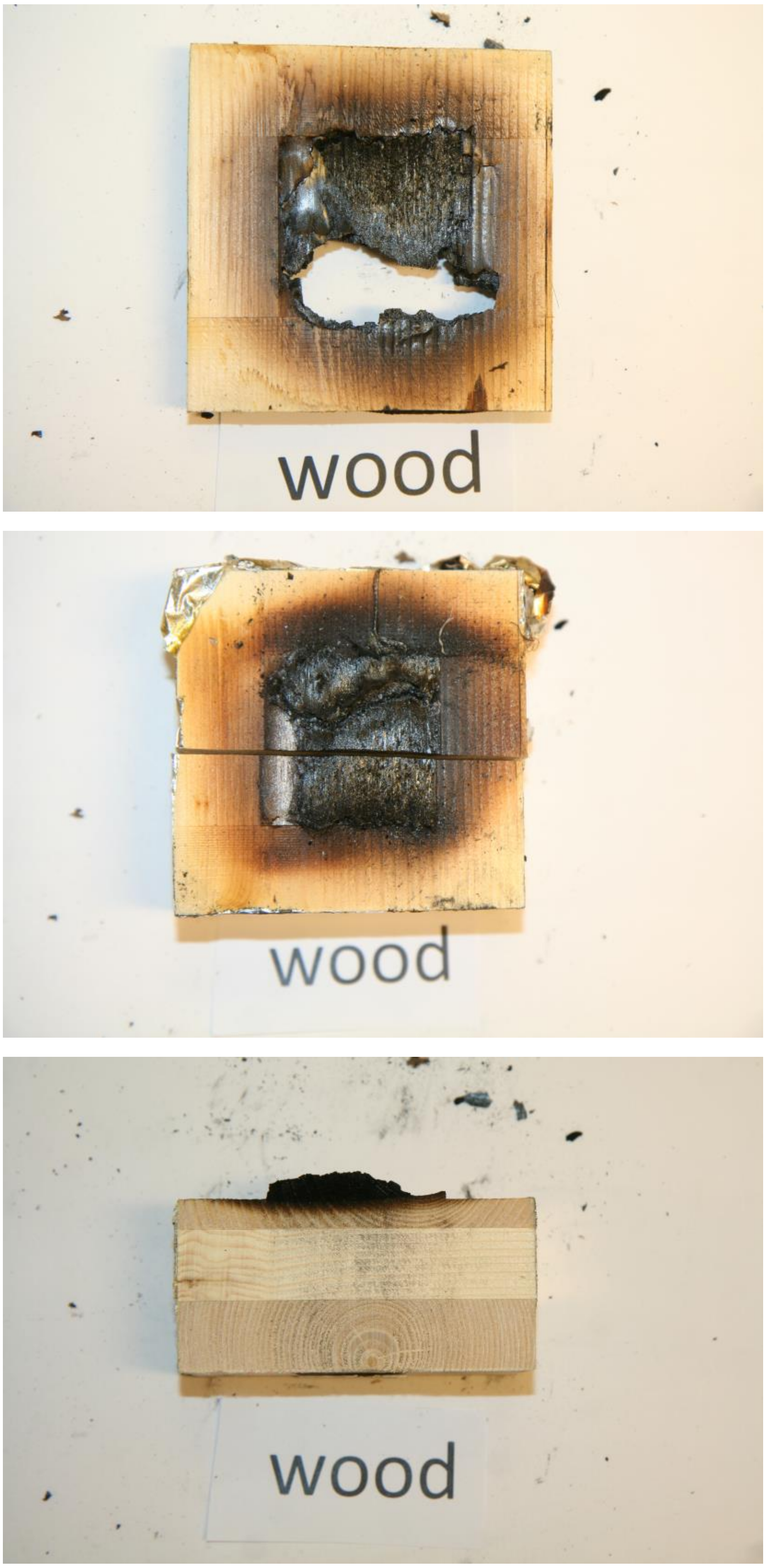

Test specimen no.6 after failure. Char failure.






Test specimen no.7 after failure. Bond line failure.

Appendix 1. Test specimens




Test specimen without glue line after failure. Char failure.