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Effect of thermal postcuring on the micro- and macromechanical properties of polyurethane for wood bonding

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Abstract

The optimization of mechanical properties of adhesive bonds is of interest especially in structural applications. Besides transferring stresses, bondlines can also provide additional functionality, such as measuring deformations in structural timber applications by electrically conductive adhesives. This study investigates the influence of a thermal postcure treatment of polyurethane bonded wood joints. Bonded beech wooden samples were manufactured with three adhesives—a commercial one-component polyurethane for structural laminated timber and two modified ones, filled with electrically conductive particles. Adhesive bonds were subjected to a subsequent postcuring at 80 and 95 °C for 1 and 48 h, respectively. Mechanical properties of the bonds were studied on the macroscopic level by tensile shear tests and the properties of the cured adhesive on the microscopic level by nanoindentation. As a result, the tensile shear strength slightly dropped with addition of filler, while all specimens still fulfilled the requirement of EN 302-1 in dry condition. Nanoindentation revealed minor decreases in mechanical properties of the cured adhesive with postcuring time for two adhesives and a different reaction of carbon black filled polyurethane, as the creep factor decreases with the thermal postcure.

Keywords: Polyurethane prepolymer, Carbon black, Carbon nanofibers, Carbon nanotubes, Thermal postcure, Mechanical properties of adhesives, Nanoindentation, Wood adhesives

Introduction

The importance of mechanical properties of adhesives is evident, as the bond has to transfer mechanical stresses from one adherend to the other. When the adhesive is used for further functions like deformation measurements in structural timber, mechanical properties of the bondline will become even more important as the stiffness and creep of the adhesive polymer influences the sensor properties.

One approach to use the adhesive bondline as a sensor, is in providing the adhesive electrical conductivity [1, 2]. Such adhesives are produced by dispersing electrically conductive fillers into the electrically insulating adhesive matrix. With a sufficient dispersion quality they exhibit a strain-dependent electrical resistance change similar to a strain gauge.



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One-component polyurethane prepolymers (1C-PUR), such as those used frequently for structural wood bonding, consist of relatively high molecular weight polyols with a stoichiometric excess of isocyanates. The crosslinking to form the polymeric adhesive film is effected by the reaction of the isocyanate with water, which is supplied to the adhesive film as air and wood moisture [3]. In the cross-linked prepolymer urethane bonds (present in prepolymer macromolecules, as a result of the reaction with alcohol groups) are found in addition to urea bonds (reaction of isocyanate with water).

A change in mechanical properties can be achieved by further cross-linking of the polymeric structure, which leads to higher strength, elastic modulus and creep resistances [4]. For polyurethanes, the linear polyurethane macromolecules needs to be converted into a three-dimensional structure with an elastomeric or duromeric character [5, 6] by forming allophanate and biuret bonds from the reaction of urethane and urea bonds with free isocyanates. The occurrence of this chemical reaction depends mainly on the temperature, which ensures the necessary reaction energy of the active hydrogen at the nitrogen in the urethane bond.

Literature indicates a range of different temperatures necessary to start forming allophanates and biuretes between 80 and $140 \,^{\circ}\text{C}$ [4–8].

Additionally, the crosslinking by forming of allophanates and biuretes is sometimes reported as reversible [7] and increases with temperature and time [8]. Below 60 °C this crosslinking is very slow, but in bulk at high temperatures such as 145 °C it can also reach a conversion of 10% of all nitrogen-containing groups [8].

Thermal postcuring of samples bonded with polyurethane has been investigated by different authors and showed no obvious positive or negative effect: Bitomski et al. [9] reported of a 2-component polyurethane adhesive in which a thermal post-treatment at 80 °C resulted in increased peel strength. Additionally, a heat treatment at 60 °C after 7 days of curing at 20 °C indicated by IR spectroscopic that the converting of monomers to a certain state of equilibrium can be accelerated again. Richter et al. [10] investigated the temperature-dependent creep of seven different 1C-PUR by combining results from thermomechanical analysis (TMA), creep under temperature, load and analysis of chemical composition by carbon-13 nuclear magnetic resonance spectroscopy (¹³C-NMR). In addition, they applied a thermal postcuring of 80 °C for 4 h and showed that out of all the tested adhesives, only one showed an improvement in creep resistance after postcuring, while all others deteriorated. They concluded that the heat treatment has no positive effect on 1C-PUR in contrast to epoxy resins.

Difficult for the application of postcuring on wood adhesive bonds is that high temperatures can degrade the wood substrate, but for postcuring a relative high temperature is necessary. Small physical changes start with the emission of extractives at temperatures of around 40 °C [11]. Degradation starts with the hydrolysis of hemicellulosis in small amounts at 80 °C, followed by degradation of cellulose at 100 °C [11]. Thermal degradation of moisture-cured polyurethanes has also been reported to start at temperatures higher than 120 °C [12].

From the state of the art it is unclear whether an improvement of polyurethane by postcuring is possible and furthermore at which temperatures such a postcuring can effect the mechanical properties of the corresponding adhesive.

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The aim of the present study is to examine the hypothesis that subsequent heat treatment of polyurethane-based conductive wood adhesive joints (postcuring) improves the mechanical properties (strength, stiffness, hardness, creep resistance) of the bond, respectively the adhesive.

For this, lap joint tensile shear tests were used for investigating macromechanical properties and nanoindentation for micromechanical properties. As adhesives a commercial one-component polyurethane prepolymer was used and two variations from the same, each mixed with electrically conductive fillers.

Methods/experimental

Preparation

The process flow to produce adhesive bond specimens for micro- and macromechanical testing is illustrated in Fig. 1. Specimen preparation started by manufacturing different adhesives by dispersing electrically conductive fillers. Based on a commercial 1C-PUR adhesive (Jowapur 686.60, Jowat SE, Detmold, Germany), two additional modified 1C-PUR were produced by adding conductive particles into the adhesive, resulting in three different adhesives. The final modified adhesives are composed of the mentioned 1C-PUR with 4 wt% carbon black (Ketchenblack EC300J, Akzo Nobel Functional Chemicals B.V., Arnhem, The Netherlands), further named 1C-PUR-CB, and with a mixture of 2 wt% carbon nanofibers (PR-25-XT-HHT, Pyrograf Products Inc., Cedarville, USA) as well as 0.2 wt% carbon nanotubes (NC7000, Nanocyl S.A., Sambreville, Belgium), further named 1C-PUR-CNFT. The amount and composition of filler was determined in preliminary tests with the aim to find dispersions with sufficient processability and a low

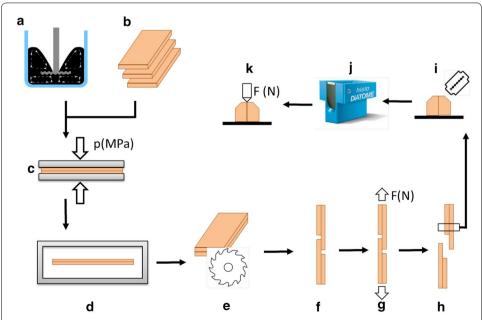


Fig. 1 Schematic process flow of specimen preparation: adhesive modification (**a**), wood lamella preparation (**b**) bonding by a laboratory press (**c**), thermal postcuring (**d**), formatting (**e**) preparation of lap joint tensile shear samples (**f**), tensile shear test (**g**), cutting of nanoindentation samples (**h**), cut to size by razor blade (**i**), surfaces preparation by Ultracut (**j**), testing by nanoindentation (**k**)

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electrical resistance of the bondline. Table 1 summarizes the relevant measurements of electrical resistance and the quantitative evaluation of the processability. The DC electrical resistance was measured with a digital multimeter (NI PXI 4071, National Instruments Germany GmbH, Munich, Germany) on bondlines with the dimensions of 20 mm (width), 55 mm (length) and around 0.1 mm (thickness). Due to the variability of wood bondlines (see also) the resistivity could be calculated reliable.

The manufacturing process of the conductive adhesives covered the following steps. First, the electrically conductive fillers have been dried at 103.5 °C for 24 h to reduce the overall moisture in the dispersion process of a moisture curing 1C-PUR. Afterwards, a dispersion container has been prefilled with argon to further reduce the amount of moisture in the dispersion process. Laboratory sized sample amounts of 40 g were produced by a miniaturized dispersing technique. The dispersion technique used a cooled laboratory container, a Ø25 mm dissolver disc (VMA Getzmann GmbH, Reichshof, Germany) and a rotational frequency of 15,000 rpm, which was adjusted with changing viscosity to maintain the doughnut effect according to manufacturer recommendations.

Clear, defect-free wood lamellas of beech wood (fagus sylvatica L.) were selected and prepared according to EN 302-1 [13]. The bulk density of the beech substrate was $648\pm11~{\rm kg~m^{-3}}$ at an equilibrium wood moisture content of $10.8\pm0.2\%$. The lamellas were bonded in pairs at 1.0 MPa and 30 °C in a laboratory press for 1.5 h according to the datasheet of Jowapur 686.60. After conditioning in standard climate (20 °C, 65% relative humidity) for 7 days, the two-layered lamellas were postcured at different conditions, as shown in Table 2.

Due to the expected thermal degradation of the wood adherend at excessive temperature, as outlined in the introduction, postcure temperatures were selected to be 80 °C and 95°. In order to prevent wood from dimensional changes as a result of loss or uptake of moisture, humidity has been selected according to the extended Keylwerth-chart of Böhner [14] to maintain the equilibrium wood moisture of beech constant (82% relative humidity at 80 °C; 85% at 95 °C). At these postcuring conditions no mechanical stress due to moisture induced swelling and shrinkage movements of the wood substrate in the bondline is expected.

Subsequent to the thermal treatment all lamellas were conditioned for 7 days at standard climate and lap-joint tensile shear specimens were manufactured according to EN 302-1 (Fig. 1f), resulting in 10–15 bonded specimens with dimensions of $150 \times 20 \times 10$ mm³ for each postcure parameter. From the tested tensile shear specimen nanoindentation samples were cut from intact parts, where no damage due to testing or clamping was visible. Bonded by Epoxy resin on metal disks, the samples were cut to size

Table 1 Results of prelimery tests to evaluate two different suitable dispersions of electrical conductive fillers with in component polyurethane (1C-PUR)

Adhesive Filler type	1C-PUR								
	CNF		CNT			CNF/CNT	СВ		
Filler in wt%	2	3	0.25	0.5	1	2/0.2	3	4	
Processability	Good	Good	Good	Moderate	Poor	Good	Good	Moderate	
Resistance $[\Omega]$	1.2M	200k	4M	50k	19k	20k	1M	34k	

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Table 2 Thermal postcure parameters and resulting terminology for the specimen series, made from 2-layered beech lamellas, bonded by one-component polyurethane (1C-PUR), mixed with carbon nanofibers/carbon nanotubes (CNFT) or carbon black (CB)

Adhesive	Temperature/relative humidity (°C/%)	Postcure time (h)	Terminology
1C-PUR	-	=	PU
	80/82	1	PU-80/1
		48	PU-80/48
	95/85	1	PU-95/1
		48	PU-95/48
1C-PUR-CNFT	_	-	CNFT
	80/82	1	CNFT-80/1
		48	CNFT-80/48
	95/85	1	CNFT-95/1
		48	CNFT-95/48
1C-PUR-CB	_	-	СВ
	80/82	1	CB-80/1
		48	CB-80/48
	95/85	1	CB-95/1
		48	CB-95/48

by a razor blade. Smooth surface was achieved by using a Leica Ultracut-R microtome (Leica, Ultracut R, Wetzlar, Germany) equipped with a Diatome Histo diamond knife (Diatome, Histo, Nidau, Switzerland).

Tensile shear strength

Macromechanical properties of polyurethane bonded wood were characterised by tensile shear tests. Specimen dimensions are specified in EN 302-1 [13], the shear area was $20 \times 10 \text{ mm}^2$. A universal testing machine (Zwick/Roell Z020, load cell 20kN, Ulm, Germany) was used to carry out the tests according to EN 302-1 with a cross head speed of 1 mm/min. Tensile shear strength was calculated from the maximum force at shear failure divided by the overlapping area of the lap joint. The failure zone was analysed visually for percentage of wood failure. Corresponding wood failure amount was noted in 10% steps.

Nanoindentation

Characterisation of the micromechanical bondline properties was done by nanoindentation. All nanoindentation experiments were carried out by a Hysitron TriboIndenter system (Hysitron, Minneapolis, MN), equipped with a Berkovich type indenter (three-sided pyramid diamond). From each series, two to four specimens have been prepared for nanoindentation according to the described method in 2.1 and were clamped magnetically to the nanoindenter sample stage. Each specimen was characterized by at least 20 indents in the region of the bondline, giving 40–80 measurements for each series, which is known as representative from previous studies [15, 16]. Figure 2 shows the evenly distributed indents on one sample bondline. The experiments were performed in load controlled mode, using a preforce of 2 μ N and a three segment load ramp, which is shown in

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Fig. 2 Overview of an exemplary adhesive bondline between wooden adherends, prepared for nanoindentation. Red lines point to selected indent positions within the bondline

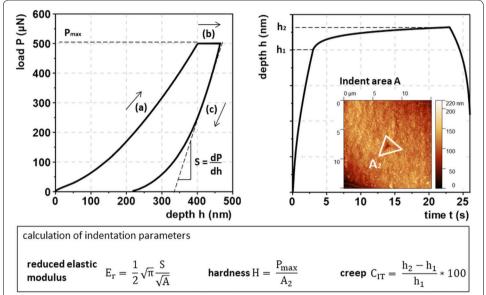


Fig. 3 Characterization by nanoindentation: TOP LEFT schematic load depth graph from nanoindentation with loading (a), holding (b) and unloading (c) sections as well as stiffness calculation from the unload section. TOP RIGHT schematic depth time response of the material and the contact area of the Berkovich tip (A2). BELOW calculated indentation parameters

Fig. 3. After reaching preforce, the maximum testing force of 500 μN has been reached in 3 s, was held for 20 s and was linearly released within 3 s.

The load-depth curve was analysed using the Oliver and Pharr method [17], resulting in reduced elastic modulus and hardness of the bondline. The reduced elastic modulus E_r was calculated from the measured unloading stiffness (S) (see Fig. 3). Hardness ($H_{Bondline}$) was calculated by dividing the maximum load by the contact area at the end of the holding segment (see Fig. 3). Indentation creep $C_{\rm IT}$ was calculated according to the test bench of CSM Instruments from the change in indentation depth during the holding segment, where the applied load remains constant (see Fig. 3) as described in a previous study [18].

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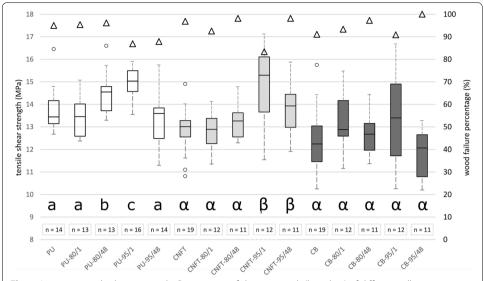


Fig. 4 Lap joint tensile shear strength. Comparison of shear strength (boxplots) of different adhesives (PU = one-component polyurethane, CNFT = PU, mixed with carbon nanofibers/nanotubes, CB = PU, mixed with carbon black), four different postcuring conditions (e.g. 80/1 = 80 °C for 1 h) and arithmethic mean of the wood failure percentage (triangle). ANOVA (p ≤ 0.05) indicates statistical significant differences in tensile shear strength, if letters are different (a ≠ b or a ≠ a). n = number of specimen tested. Boxplots: boxes = quartile of distribution, whiskers = 1.5 of interquartile range, circles = outliers

Results and discussion

Lap joint tensile shear strength

On the macroscopic level tensile shear strength of lap joint specimens was measured to characterise probable changes due to thermal postcuring as shown in Figure 4. A one-way analysis of variance (ANOVA, $p \le 0.05$) was used to statistically evaluate the postcuring effect on the tensile shear strength.

All specimens, untreated and thermal postcured, show higher tensile shear strength values then 10 MPa thus fulfilling the requirements for structural wood bonding according to EN 302-1 in dry condition (A1 treatment). Additionally, all specimens show a high mean wood failure percentage of 80–100%, implicating a good bonding quality.

Comparing the different non-treated adhesives (PU vs. CNFT vs. CB, see Table 2), both adhesives filled with particles show significantly lower tensile shear strength than the unfilled polyurethane. The type of filler did not show significant differences.

Within the group of PU the thermal postcure leads to no differences for 80/1 (80 °C for 1 h) and 95/48, but at a longer treatment of 48 h at 80 °C the tensile shear strength increases significantly (ANOVA, $p \le 0.05$). The highest value of tensile shear strength is reached at 95/1.

Within the group of CNFT significant increases in tensile shear strength were only measured for treatments at 95 °C.

Within the group of CB differences in tensile shear strength can be recognized, but they seem to be neither systematic nor statistically significant.

Overall, the presented dataset indicates significant higher tensile shear strength values for PU-80/48, PU-95/1, CNFT-95/1 and CNFT-95/48. Therefore, no systematic

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effect of thermal postcure on the lap joint tensile shear strength is evident. As no significant decrease for any postcure treatment for all three adhesives can be measured, it can be stated that this range of postcuring parameters didn't damage the wood adhesive bond. Based on the high wood failure percentage, the data set mainly reflects the shear strength of the wood adherend [18]. Thus no distinct conclusion can be drawn regarding the thermal postcuring effect on the adhesive.

The decrease in tensile shear strength with filler concentration has been reported in literature [19] already and is regarded as one of the drawbacks of electrically conductive fillers. As the wood failure percentage indicates that failure happens mostly in the wood adherend and in the wood-adhesive interphase, it's indicated that the incorporation of the filler doesn't seem to impact the bond strength of glued wooden samples according to EN 302-1 like in other applications.

Micromechanical properties of adhesive by nanoindentation

Nanoindentation experiments were used to characterise the mechanical properties of the bondline on the microscopic level. Figure 5 shows the reduced elastic modulus (E_r), Fig. 6 the hardness ($H_{Bondline}$) and Fig. 7 the creep factor (C_{IT}).

The non-treated adhesives (PU, CNFT and CB) show measured values of 2GPa (E_r) and 0.11Gpa ($H_{Bondline}$) and are therefore in the range of usual one-component polyure-thane prepolymers [20]. The investigated filler contents reveal no statistical significant difference (ANOVA, $p \le 0.05$) of E_r and $H_{Bondline}$, only the creep factor (C_{IT}) increases slightly, but significant.

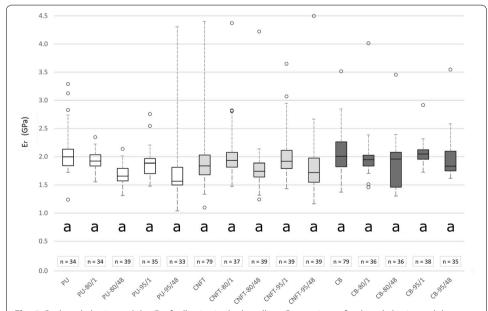


Fig. 5 Reduced elastic modulus E_r of adhesive in the bondline. Comparison of reduced elastic modulus of different adhesives (PU = one-component polyurethane, CNFT = PU, mixed with carbon nanofibers/ nanotubes, CB = PU, mixed with carbon black) and four different postcuring conditions (e.g. 80/1 = 80 °C for 1 h). ANOVA (p ≤ 0.05) indicates no statistical significant differences of reduced elastic modulus (only different letters indicate differences). n = number of specimen tested. Boxplots: boxes = quartile of distribution, whiskers = 1.5 of interquartile range, circles = outliers

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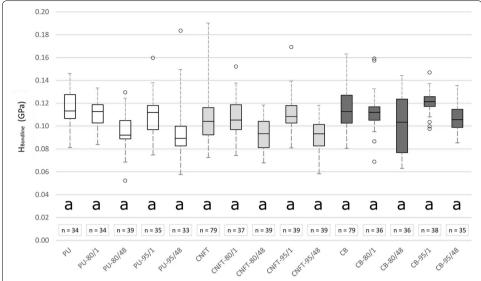


Fig. 6 Hardness of adhesive in the bondline ($H_{Bondline}$). Comparison of $H_{Bondline}$ of different adhesives (PU = one-component polyurethane, CNFT = PU, mixed with carbon nanofibers/nanotubes, CB = PU, mixed with carbon black) and four different postcuring conditions (e.g. 80/1 = 80 °C for 1 h). ANOVA ($p \le 0.05$) indicates no statistical significant differences of reduced elastic modulus (only different letters indicate differences). n = number of specimen tested. Boxplots: boxes = quartile of distribution, whiskers = 1.5 of interquartile range, circles = outliers

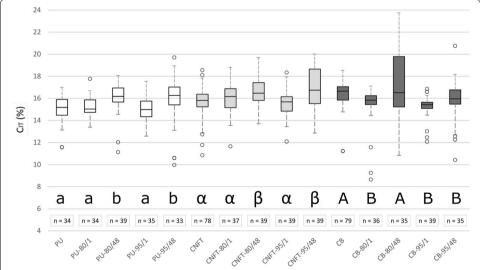


Fig. 7 Creep factor C_{Π} of adhesive in the bondline. Comparison of creep factor of different adhesives (PU = one-component polyurethane, CNFT = PU, mixed with carbon nanofibers/nanotubes, CB = PU, mixed with carbon black), four different postcuring conditions (e.g. 80/1 = 80 °C for 1 h) and arithmethic mean of the wood failure percentage (triangle). ANOVA (p ≤ 0.05) indicates statistical significant differences of creep factor, if letters are different (a ≠ b or a ≠ a) or a ≠ A). n = number of specimen tested. Boxplots: boxes = quartile of distribution, whiskers = 1.5 of interquartile range, circles = outliers

The measurements reveal a different effect of the thermal postcure on PU and CNFT versus CB. A systematic effect of postcuring time was found for PU and CNFT, deteriorating the mechanical properties (decrease of E_r and $H_{Bondline}$, increase of C_{IT}). In

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Postcuring conditions 95°C for 48 hours 80°C for 48 hours untreated

Fig. 8 Colour changes of leaking one component polyurethane adhesive (1C-PUR) indicating degradation as a result of thermal postcuring treatments. Postcuring conditions are indicated in the figure

contrast CB shows no systematic effect and partly improves his mechanical properties (decrease of $C_{\rm IT}$).

Within the groups of PU and CNFT all measured properties (E_r , $H_{Bondline}$ and C_{IT}) change systematically with thermal postcuring time, but differences are only significant for the creep factor (ANOVA, p \leq 0.05). While no difference is visible when thermally postcured for 1 h, a longer postcuring of 48 h results in a decrease of E_r and $H_{Bondline}$ (-10 to -20.3%) and in an increase of C_{IT} (+2 to +8.5%).

Within the group of CB no significant change of E_r and $H_{Bondline}$ is indicated, but a significant decrease of C_{IT} (-5 to -7.5%) for three of four thermal postcuring conditions (ANOVA, $p \le 0.05$). Only the treatment of 80 °C for 48 h shows no significant difference.

Additionally, a significant difference between the creep factor of all adhesives (PU vs. CNFT vs. CB) was found. The creep factor changes with the filler material, with an arithmetic mean of 15% for PU, 15.7% for CNFT and 16.4% for CB.

Comparing the results of PU and CNFT, the decrease in E_r and $H_{Bondline}$ together with the significant increase in creep factor indicates that the polyurethane bondline became softer, even after reconditioning to 20 °C for 7 days. While other studies showed a softening of polyurethane adhesive bondlines with increasing testing temperature [21, 22], the remaining decrease of properties after reconditioning is obvious. A possible reason could be the postcuring condition, which also included high humidity. While the equilibrium wood moisture of beech was hold constant, the high humidity could have supported the polyurethane degradation by oxidation or hydrolysation [23]. Indication for degradation by oxidation is a darker color, which was visible at the foamed polyurethane in the bondline of the series PU-80/48 and PU-95/48 (Fig. 8). As the oxidative degradation enhances with the amount of surface, the color of foamed polyurethane can't be alone used to prove this assumption.

As carbon black filled polyurethane (CB) didn't decrease in E_r and $H_{Bondline}$, but partly increases in C_{IT} , the filler seems to have a stabilizing effect on the adhesive.

The stabilizing effect of carbon black could be attributed to chemical interaction between carbon black with polyurethane or other mechanisms. However, the type of Winkler et al. Appl Adhes Sci (2018) 6:5 Page 11 of 12

interaction was not addressed within the frame of this study, but will be a topic of further work.

Conclusion

Several conclusions can be drawn from the results:

- One-component polyurethane prepolymers (1C-PUR) with different filler was successfully produced and reached tensile shear strength values between 10.2 and 16.4 MPa. Through addition of electrically conductive filler the tensile shear strength of the 1C-PUR significantly decreased without breaking the requirements of EN 302-1 in dry (A1) condition.
- 2. The applied postcuring leads to higher tensile shear strength for single treatment conditions, but in no case to a decrease. However, a systematic trend was not measurable.
- 3. Based on nanoindentation measurements of the bondline, a slight, but insignificant decrease of $E_{\rm r}$ and $H_{\rm Bondline}$ for two adhesives (PU and CNFT) was measurable for thermal postcuring times of 48 h. The creep factor of these adhesives increases significantly for thermal postcuring times of 48 h.
- 4. Carbon black filled polyurethane prepolymers (CB) differs from PU and CNFT in their response to the thermal postcuring and exhibit a stabilizing effect.

Abbreviations

1C-PUR: one-component polyurethane prepolymer; TMA: thermomechanical analysis; 13 C-NMR: carbon-13 nuclear magnetic resonance; 1C-PUR-CB: one-component polyurethane prepolymer, filled with 4 wt% carbon black; 1C-PUR-CNFT: one-component polyurethane prepolymer, filled with 2 wt% carbon nanofibers and 0.2 wt% carbon nanotubes; ANOVA: analysis of variance; PU: further abbreviation of 1C-PUR to include process parameters in the abbreviation; CB: further abbreviation of 1C-PUR-CB to include process parameters in the abbreviation; CNFT: further abbreviation of 1C-PUR-CNFT to include process parameters in the abbreviation; C_{Π} : creep factor; E_{r} : reduced elastic modulus; $H_{Bondline}$: hardness of the bondline.

Authors' contributions

CW, US and JK jointly designed the experiment. CW prepared all adhesives, samples and tested lap-joints, CW and JK performed nanoindentation experiments. All authors jointly evaluated the results, discussed them and contributed to the manuscript. All authors read and approved the final manuscript.

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Competing interests

The authors declare that they have no competing interests.

Availability of data and materials

All relevant data are presented in the manuscript, if needed additional information may be made available upon request.

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