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Rapid bonding of timber structures

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ABSTRACT: Adhesively bonded timber-based elements are often manufactured with two-component (2K) adhesives (mostly epoxies and polyurethanes), which usually need many days until full cure, i.e. mechanical performance, has developed. The study shows how wooden components can be bonded in significantly less time by using inductively heated Curie particles (CP), which are added to the polymers and heated by an electromagnetic field (EMF) up to the particle's Curie temperature, T_c , but not beyond. The investigations were carried out using small single lap shear (SLS) as well as medium- and large-sized Glued-in Rods (GiR), all of which produced with spruce adherends. Furthermore, two frequently used 2K adhesives were analysed (epoxy and polyurethane). It could be shown that curing times can be reduced from 1/10 days down to 10 min of inductive heating by application of the technique.

KEYWORDS: Accelerated curing, wood adhesives, induction heating, Curie susceptors

1 INTRODUCTION

Depending on the respective application scenario, polymerisation can be a very slow process. Especially in many structural applications, the usually applied high-strength thermosets – mostly two-component (2K) epoxies (EPX) and polyurethanes (PUR) – require a lot of time (up to many days) until full cure has been achieved and the connections can be stressed or moved for further processing. The reason of these circumstances can be traced back to the comparatively long pot life, which is regarded as a prerequisite to be able to apply the adhesives onto the substrates in time.

		Accelerated curing of polymers		
Radiation curing - Gamma - UV - X-Ray	Radiation heating - Electron beam - Infutction - Infrarfed / Laser - Microwave - Ultrasonic	t curing Physical heating - Conductive - Dielectric - Resistive	Conventional curing - Flame - Hot gas - Lamps - Mats - Oven	Chemical' curing

Figure 1: Methods for accelerated curing of adhesives (no claim to completeness)

Due to these (and many more) application-related limitations, many techniques for accelerated curing emerged, which can be achieved by different means (cf. Figure 1). The methods range from UV-curing [1], over electron beam[2] or microwave curing [3], to more conventional methods such as ovens. Many of them use heat that is introduced into the adhesive to increase monomer mobility and thus speed up the curing progress. Probably one of the fastest ways for heat generation represents the use of electromagnetic induction[4] when metallic materials are subjected to an external electromagnetic field (EMF). In context of adhesive bonding technology, induction-based heating can be either used for direct heating of metallic adherends[5] or (when the joint consists of materials that are non-sensitive to EMF) for the heating of susceptors materials (e.g. fibres[6], particles[7], meshes[8] etc.) that are added to the polymers to be cured. In the following, the adhesives are cured 'from the inside'.

In this study, a special type of susceptors, Curie particles (CP), were investigated, which make use of the material-specific Curie temperature, T_c , to control the heating process (no more heat development above T_c) and thus prevent overheating – aspects, which many publications in the field do not consider[9,10]. It was thus targeted to create a self-regulating accelerated curing process, which eliminates the need to store timber-based elements for many days until they can be used.

2 Materials & methods

2.1 Adhesives

The investigations covered two thermosetting 2K adhesives (epoxy and polyurethane), which are frequently used in the German construction sector for wooden applications.

Table 1: Material properties of the adhesives Fi390 and LP4821 taken from technical data sheet (TDS) or * that have been measured

Bronorty	Adhesive		
Property	Fi390	LP821	
Adhesive type	2K-EPX	2K-PUR	
Density, ρ [g/cm ³]	1.50	1.32	
Pot life at +23 °C [min]	14-30	30	
Curing time at +23 °C [h]	18	240	
Young's modulus, E [MPa]	5230*	1.000	
Tensile strength, σ_u [MPa]	40.0*	45.0	
Elongation at break, ε_u [%]	1.2*	5.0	
$T_{\rm g}$ for curing at +23°C [°C]	73.4*	71.5*	

In detail, Fischer EM390S (abbreviated as Fi390, Fischerwerke GmbH & Co. KG, Germany) and Loctite Purbond CR821 (LP821, Henkel AG & Co. KGaA, Germany) were chosen. Most relevant material properties are listed in Table 1.

2.2 Curie particles

Since no EMF-sensitive materials were involved in all three joint types (cf. section 2.4), particulate Curie susceptors were added to the polymers to be able to heat the adhesives inductively. The particles had a Curie temperature, T_c , of 110 °C, which was selected based upon permissible heating requirements of the polymers. An in-depth characterisation of the CP type has e.g. been presented in [11], which is why it will not be repeatedly described in the present publication.

2.3 Mixing

All adhesive-CP mixes were prepared using a Speedmixer of type DAC 800.2 VAC-P (Hauschild GmbH & Co. KG, Germany). To ensure most homogeneous particle distributions, mixing was carried out under vacuum (100 mbar) using identical parameters for all adhesive-CP mixes prepared (v = 1.000 rev/min, $t_{mix} = 60$ s). Two different particle contents were used: 33.3 w/-% (~11–13 vol-%) as well as 7.5 w/w-% (2–3 vol-%). Corresponding results have been marked in the result section.

2.4 Specimen types

2.4.1 Lap shear joints

To analyse the performance of the inductively cured adhesives on a small joint scale, single lap shear (SLS) specimens acc. to DIN EN 1465[12] were produced using spruce (*picea abies*) adherends with dimensions 100 x 25 x 6 mm. Their overlap length was 12.5 mm and adhesive layer thickness was selected to be 0.3 mm. The moisture content of the adherends was measured for five adherends at five locations each and amounted to $9.8 \pm 0.8 \%$.

2.4.2 Glued-in Rods

Besides the small SLS joints, experiments were up-scaled in the following to the level of medium- and large-scaled GiR connections, which represent a frequently used timber engineering application[13,14]. The latter consisted of blocks of spruce (*picea abies*, moisture content 8.1 ± 0.3 %) as well as rebars made of glass-fibre reinforced polymer (G-FRP), which were bonded into the wooden blocks. The pultruded rebars are marketed under the name Schöck ComBar[®] (Schöck Bauteile GmbH, Germany) and have a high resistance against corrosion. The joint geometry of the medium-scaled GiR can be seen in Figure 2.

The complete production approach of the GiR has been described in detail in e.g. [15], which is why detailed information regarding their manufacture will not be repeatedly presented in the present paper. However, the applied steps for induction heating are described in section 2.5.2 of the manuscript.



Figure 2: Geometry of medium-sized wooden Glued-in Rods (GiR) used as an exemplary application for the inductive heating experiments, dimensions for large-scale GiR correspond to a doubling of all dimensions shown, all dimensions in mm

2.5 Inductive heating

2.5.1 Experimental setup

For all inductive heating operations, the commercially available induction device TrueHeat 5010 (Trumpf Hüttinger GmbH & Co. KG, Germany) was utilised to cure the wooden joints. Depending on coil geometry and mounted capacitors, the system operates in a frequency range of ~50–500 kHz. Essential induction parameters have been summarised in Table 2. For validative purposes, curing temperatures were monitored with thermocouples embedded within the adhesive layers. Figure 3 shows the experimental setup for a medium-sized GiR as an example.



Figure 3: Experimental setup used for inductive heating with 1) medium-sized GiR (exemplary joint, herein bonded with LP821), 2) induction coil, 3) induction device and 4) measurement electronics / software for thermocouple data evaluation

Table 2: Induction parameters for the different joint geometries

Joint type	<i>P</i> [kW]	<i>I</i> [A]	U[V]	f[kHz]
SLS	4.8	26.3	640	134
GiR_M	4.7	35	650	177
GiR_L	3.8	29.4	795	120

2.5.2 Heating procedure

After mixing was complete, the adhesive-CP mixes were decanted into syringes or 1K disposable cartridges to be able to apply them onto the substrates – a process that has been visualised in Figure 4. All G-FRP rods were degreased with isopropanol 99.9 % and the wood blocks were freed of bore durst using compressed air- If temperature sensors were involved, these were fixed on the substrates using temperature resistant adhesive tape prior adhesive-CP application was performed. To minimise the possibility for air entrapments within the adhesive layer, all GiR joints were filled from bottom to top using a feedhole, which was drilled in the wooden blocks and ended flush with the hole that was used to centre the rods vertically.



Figure 4: Application of the adhesive-CP mixes to the substrates using syringe for SLS joints (left) as well as 1K disposable cartridge for large-scale GiR (right), application for medium-sized GiR corresponds to right scenario but with syringe

After adhesive-CP application was complete, the joints were placed within the induction coil (cf. Figure 3), so to ensure that all parts of the adhesive layer were tightly enclosed by the coil. Independently of the different joint geometries, the preparation never exceeded a time window of 7.5 min, which was well within pot life (cf. Table 1).

2.6 Mechanical testing

In order to check the capability of the technique for accelerated curing, the joints were destructively tested at different times after inductive heating ended. However, to exclude any influence of the elevated temperatures on the mechanical results, testing could earliest be performed when the connections had cooled down to RT, which took ~15 min for SLS specimens as well as ~1.5 h, for the GiR. Furthermore, additional time for preparation of the testing devices (~15–30 min) was needed. In detail, testing times

of 2, 24, 240 as well as 720 h were investigated, with the GiR setups being illustrated in Figure 5.



Figure 5: Configurations for mechanical testing of medium- (left) and large-sized GiR (right), v = 2 mm/min, Zwick|Roell UTM 100 kN, all tests performed at RT

2.7 Experimental program

The investigations covered different test series, which have been summarised in Table 3. Besides the inductively cured joints (labelled as IND), reference sets with and without the addition of CP were produced. The latter were 'cold cured' (CC) at RT $(23 \pm 2 \text{ °C}, 50 \pm 10 \text{ \% RH})$ following the protocols that were provided by the adhesive manufacturers. In addition, the expressions GiR_M and GiR_L stand for medium- as well as large-scaled GiR series, respectively.

Table 3: Experimental program produced for each Fi390 (2K-EPX) as well as LP821 (2K-PUR), CC = Cold cured at RT, IND = Inductively cured, 2h = mechanical testing performed two hours after induction device was switched off

	Series					
Туре	CC_	CC_	IND_	IND_	IND_	IND_
	Ref	CP	2h	24h	240h	720h
SLS	5	5	5	5/3	5	5
GiR_M	3	3	3	3	3	-
GiR:L	5	3	3	3	3	-

3 RESULTS

3.1 HEATING BEHAVIOUR

The first step of the evaluation of the results circumvented the analysis of the heating behaviour of the inductively cured joints, as shown in Figure 6. It can be seen that the two adhesives behave fundamentally different during the heating phases of 10 min. Thus, all joints bonded with Fi390 reach the T_c of the particles (110 °C) after different times, whereby ~3 min (SLS), ~5–6 min (GiR_M), as well as 10 min (GiR_L) are needed depending on the respective joint geometry.

In contrast, joints bonded with the 2K-PUR reach slower heating rates, with temperatures recorded for the SLS joints showing a clear flattening at a curing temperature of approximately 70 °C. Furthermore, both GiR sizes reach temperatures of ~100–110 °C at the end of the inductive heating phase (t = 10 min).



Figure 6: Heating behaviour recorded with thermocouples embedded within the adhesive-CP mixes for the different joint geometries bonded with the two investigated adhesives, temperatures for medium- and large-sized GiR correspond to arithmetic mean out of three temperature sensors along the rod axis (top, middle, bottom)

A particularity of the temperature data visualised in Figure 6 can be seen for SLS joints bonded with Fi390 between an induction time of 3-6 min. In this time window, the data reveals a slight temperature overshoot to ~120 °C after which the curve flattens again towards the Curie temperature of 110 °C.

Finally, it can be observed that cooling progresses largely different for the three specimen types, whereby SLS joints – independently of the adhesive – reach RT \sim 15–20 min after the induction device has been switched off. In contrast, medium- (\sim 30–40 min) and large-sized GiR (\sim 90 min) require significantly more time for cooling.

3.2 MECHANICAL PERFORMANCE

3.2.1 Lap shear specimens

The results for the mechanical SLS tests have been summarised in Figure 7, with the produced series for each adhesive being illustrated as a bar chart. The data shows that both adhesives behave fundamentally different in terms of their lap shear characteristics, i.e. attained lap shear strengths and fracture patterns. In detail, all SLS series produced with Fi390 resulted in a failure of the wooden adherends, indicated by the torn off wood fibres that can be seen in the exemplary failure modes in Figure 7-top. Furthermore, lap shear strengths across all series ranged between $\sim 6-10$ MPa independently of the curing conditions considered.



Figure 7: Mechanical results for wooden SLS joints bonded with the two investigated adhesives, v = 2 mm/min, Zwick|Roell UTM 20 kN, all tests performed at RT

When the results of the second adhesive (LP821) are compared to those of Fi390, clear differences can be observed. In detail, both series that were cured at RT (with and without added CP) resulted in substrate failure. However, when heat is introduced over the CP, lap shear strengths are lowered to ~3–5 MPa, which is accompanied by a change in fracture patterns from substrate to cohesive failure (cf. Figure 7-bottom, rightmost pattern). This observation holds true for all inductively cured series of LP821, which were tested at different times (2–720 h) after inductive heating ended.

3.2.2 Glued-in Rods

In the next step, mechanical results for medium- and large-sized GiR were analysed and plotted in the form of bar charts in Figure 8 and Figure 9. Above all, the mechanical tests on GiR level confirmed the findings made on the standardised lap shear scale. Thus, for the 2K-EPX (Fi390) all produced series attained a failure of the wooden adherends, with wood plugs being pulled out of the blocks. Excluding outliners for the medium GiR, shear strengths mainly lie in the range of ~12–19 MPa, whereas large-scale GiR showed failure loads between ~30–55 kN (~4.8–8.8 MPa).



Figure 8: Mechanical results for medium-sized GiR bonded with the two investigated adhesives; v = 2 mm/min, Zwick|Roell UTM 100 kN, all tests performed at RT

Focusing on the LP821-related GiR results, the very same trend as on SLS scale became visible, with all CP-cured series cured at elevated temperatures showing a mixed cohesive / adhesive failure (wood side) – a result that is not changed by subsequent conditioning at RT. Furthermore, the mere addition of the susceptors did not impact the fracture behaviour on GiR level, proved by the RT-cured series with added CP.



Figure 9: Mechanical results for large-sized GiR bonded with the two investigated adhesives; v = 2 mm/min, Zwick|Roell UTM 100 kN, all tests performed at RT

DISCUSSION

3.1 HEATING BEHAVIOUR

In general, the investigations regarding the heating of the different joint geometries revealed slower heating rates for LP821 compared to Fi390 across the specimen types studied. Since all remaining experimental conditions (CP content, coils, induction time, substrates etc.) were kept identical, the authors strongly assume that the curing kinetics, i.e. polymerisation enthalpy and rate of its release, contributed to the temperature distribution throughout the joints. It is already known that the 2K-EPX (Fi390) has faster kinetics and contains more enthalpy [16], which is why the connections can be heated way faster to the relevant temperature ranges. Especially at the beginning of the induction processes, the self-accelerating character of the cross-linking reaction plays a decisive

role, since a lot of enthalpy is released in a relatively narrow time window – a conclusion, which is supported by the enthalpy-related temperature overshoot recorded for Fi390-bonded SLS joints after 3–6 min of inductive heating (cf. Figure 6-top, orange curve).

Besides these adhesive-related differences in heatability, the presented temperature data additionally reveals that the joint type, i.e. the geometry and materials involved, represent a decisive influencing factor for the development of curing temperatures. This becomes particularly clear when the results for Fi390-bonded large-scale GiR and SLS joints (both produced with 33.3w/w-% of CP) are compared (cf. Figure 6-top, dark red and orange curves). Thus for the GiR, a way slower heating can be seen than for the small SLS joints, although adhesive layer thickness, i.e. enthalpy and potential for self-acceleration, is higher. It can thus be surmised that large joints are more difficult to heat but not necessarily excluded for the technique.

From these relations, it becomes clear that an efficient production planning would require a numerical model in which heating-related conditions are mapped to predict the temperature development. Without a simulation model, enthalpy- and geometry-related impacts on the heating can only be determined based upon cumbersome preliminary experiments.

Excluding the slight temperature overshoot for Fi390-bonded SLS joints, it could also be shown that self-regulating heating was successful achieved. However, practitioners have to consider that enthalpy-related overheating can occur if joint and induction conditions favour fast heating – aspects that have to be considered if a numerical model is set up. Parts of such a model have already been presented by the authors [11,17].

3.2 MECHANICAL PERFORMANCE

The extensive experimental campaign revealed a uniform behaviour on the three joint types (SLS, medium and large GiR). For Fi390, all series attained a failure of the wooden substrates – a very promising result. The authors conclude that neither the adhesives capability to build up cohesive as well as adhesive strength is significantly deteriorated by application of the process. However, it is known that both cohesive [18] as well as adhesive strength [19] might be influenced / altered by both the presence of the CP as well as the inductive heating.

In contrast to the EPX adhesive, joints bonded with LP821 systematically showed a mixed failure (adhesive / cohesive) when heat is introduced over the CP. The authors know that this effect does not originate from the nature of the polyurethane adhesive considered, but rather from the moist substrates that were used. In detail, the moisture diffuses out of the wooden adherends when heat is applied and interferes in the polymerisation of the adhesive, i.e. the isocyanate groups react under the formation of CO_2 (indicated by the bubbles that can be seen in the associated fracture patterns). In the following,

adhesion / cohesion build-up is significantly deteriorated. It can be expected that the process is also applicable to the 2K-PUR when substrates are changed or the wood is dried before curing.

4 CONCLUSION

The study deals with the accelerated curing of adhesively bonded wooden joints for structural timber applications. For that, so-called Curie particles (CP) were added to two adhesives (2K-EPX and 2K-PUR), which are frequently used throughout the German construction industry. The particles can be heated by an electromagnetic field until their Curie temperature, T_c, is reached, above which the heating is capped by the nature of the particles. Based on these relations a self-regulating curing process is created, which eliminates the need for cumbersome temperature control and enables practitioners to bond under adverse conditions in significantly less time. For the experimental investigations, an up-scaling approach was chosen, with three different specimens types being investigated: standardised single lap shear (SLS) joints as well as medium- and large-scaled Glued-in Rod (GiR) connections - all of which produced with spruce adherends. In addition, the joints were tested at different times after the inductive heating to validate their mechanical performance immediately after and with increasing time after induction curing was complete. After evaluation of all data, the following impotant findings were made:

- The heating behaviour of CP-cured joints is largely determined by component geometry and the adhesive considered, in particular its curing kinetics and polymerisation enthalpy release, which contributes to the curing temperatures. Above all, larger joints are more difficult to heat, especially when long adhesive layers are heated, which offer a larger area for heat dissipation with the remaining parts of the joints.
- With regard to the mechanical performance of the adhesives, the 2K-EPX showed a fracture behaviour indistinguishable to the reference sets, which holds true for all specimen types considered. In contrast, the 2K-PUR showed strengths reductions of ~30-50 % across all joints when heat is applied over the added CP. This was attributed to moisture diffusion from the wooden substrates towards the adhesive laver, which interferes with the cross-linking reaction of the polyurethane. However, this problem might be solved by future investigations by e.g. using a CP type with a lower $T_{\rm c}$. In addition, it is already known that the technique works when the adherends are dried prior the induction curing.
- Above all, the findings have to be assessed as very positive, since curing times of both considered adhesives could be reduced from 1 (Fi390) or 10 days (LP821) to an induction time of only 10 min. For future studies, it should be

targeted to include temperatures during the cooling phase or adapt the CP regarding their size and shape to further reduce times needed to cure the joints.

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