World Conference on Timber Engineering Oslo 2023

LOW-TEMPERATURE BONDING OF TIMBER STRUCTURES

Morten Voß*, Tobias Evers* & Till Vallée*

ABSTRACT: Any bonding operation requires a tight control of environmental conditions (most importantly temperature and moisture) to ensure the desired mechanical performance during service life. To overcome these restrictions, the study shows how inductively heated Curie particles can be used to cure wood adhesives (epoxy and polyurethane) under low temperature conditions (+5 and –10 °C). The investigations were carried out using standardised specimens (dogbone / wooden lap shear joints) as well as large Glued-in Rods (GiR). The process is very well suited for the epoxy adhesive, which showed a mechanical performance equal to that of the references. In contrast, the technique is not applicable to polyurethane adhesives bonded on wooden adherends due to moisture diffusion, which interferes with the polymerisation – a problem, which might be solved by using a different particle type with lower Curie temperature, T_c.

KEYWORDS: Low-temperature curing, onsite bonding, induction heating, Curie particles

1 INTRODUCTION

A severe disadvantage of adhesively bonded joints compared to mechanical joining techniques represents the fact that the polymerisation progress is significantly influenced by ambient conditions, most importantly temperature and moisture [1,2]. As a result, practitioners have to costly adapt their production processes to the requirements of the respective adhesive in order to ensure durability and performance of the connections during service life. Furthermore, the bonding-related constraints exclude the use of adhesives for many applications from the start (e.g. onsite bonding in timber engineering).

Up to now, low-temperature curing of polymers is not a well-defined field of research, whereby generally two application approaches may be distinguished: Firstly, the bond may both be produced and cured at low temperatures, secondly, only adhesive application and joining is carried out at low temperatures while the polymerisation is accelerated in the following, e.g. by introducing thermal energy. Both approaches have yet been little studied, with investigations of Moussa et al.[3,4] and Ratsch et al.[5] being available. While Moussa et al. focused on changes in polymer characteristics due to low-temperature curing of a structural adhesive, Ratsch et al. concentrated on providing thermal energy by heating threaded steel bars in a Glued-in Rod (GiR) connection over an external electromagnetic field (EMF).

In order to overcome the temperature-related constraints, the authors investigated if inductively heated Curie particles (CP) can principally be used to cure structural wood adhesives under adverse temperature conditions (+5 and –10 °C). The particles are added to the adhesives and may be heated up to their material-specific Curie temperature, T_c, but not beyond. Thus, a curing process is designed, which automatically reduces the risk of overheating while the heat can be used to enable curing starting from low temperatures.

2 MATERIALS & METHODS

2.1 ADHESIVES

The investigations covered two thermosetting two-component adhesives (epoxy and polyurethane), which are frequently used for different structural applications throughout the German construction sector. In detail, the adhesives Fischer EM390S (abbreviated as Fi390, Fischerwerke GmbH & Co. KG, Germany) and Loctite Purbond CR421 (LP421, Henkel AG & Co. KGaA, Germany) have been selected. Their most important material properties have been listed in Table 1.

Table 1: *Material properties of the adhesives Fi390 and LP421 taken from technical data sheet (TDS) or * that have been measured, ** calculated based upon Arrhenius law* [6]*, tensile strengths measured with v = 1 mm/min and specimens type 1A according to DIN EN ISO 527[7]*

Property	**Adhesive**	
	Fi390	LP421
Adhesive type	2K-EPX	2K-PUR
Density, ρ [g/cm^3]	1.50	1.35
Pot life at +23 °C [min]	14-30	10
Curing time at +23 °C [h]	18	|240
Curing time at –10 °C [h]	160**	1920**
Young's modulus, E [MPa]	5230*	2760*
Tensile strength, σ_u [MPa]	40.0*	28.6*
Elongation at break, ε_u [%]	1.2*	1.6*
T_g for curing at +23°C [°C]	73.4*	81.3*

2.2 CURIE PARTICLES

Softmagnetic manganese-zinc (MnZn) ferrite particles (Ø0.5–25 µm, cf. Figure 1) with a T_c of 110 °C served as susceptors. The T_c was chosen so to match the heating requirements of the considered adhesives (permissible

**Fraunhofer Institute for Manufacturing Technology and Advanced Materials IFAM, Bremen, Germany*

https://doi.org/10.52202/069179-0046

heating rate / maximum curing temperature), which were determined by preliminary experiments. Moreover, the ceramic particles were selected, since they are corrosion resistant and have a comparatively low coercivity, which ultimately favours heat generation by hysteresis losses. In-depth information about the CP and their characteristics can be found in [8].

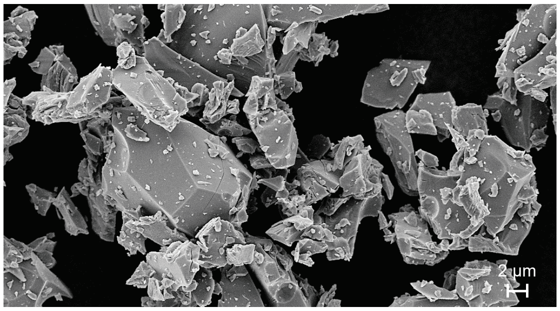

Figure 1: *SEM image of flake-shaped CP (Hengdian Group DMEGC Magnetics Co., Ltd., China)*

2.3 MIXING

All adhesive-CP mixes were prepared under vacuum conditions (100 mbar) using a Speedmixer of type DAC 800.2 VAC-P (Hauschild GmbH & Co. KG, Germany). Mixing was carried out for 60 s at 1.000 rev/min. To maintain comparability, a CP content of 33.3 w/w-% (~8–9 vol-%) was chosen for all produced specimens.

2.4 SPECIMEN TYPES

2.4.1 Dogbone

To analyse the impact of low-temperature curing on tensile properties, dogbone (DB) specimens acc. to DIN EN 527[7] type 1BA were produced, with the adhesive-CP mixes being applied into silicon moulds using a syringe.

2.4.2 Lap shear specimens

In order to characterise the performance of the low-temperature cured adhesives on a small joint scale, single lap shear specimens (SLS, 100 x 25 x 6 mm) acc. to DIN EN 1465[9] were produced using spruce adherends (*picea abies*) with a moisture content of 8.9 ± 0.6 %. The joints overlapped by 12.5 mm. For SLS series bonded with LP421, wood adherends were dried in an oven.

2.4.3 Glued-in Rods

Finally, the experiments were up-scaled to a large joint geometry, so-called Glued-in Rods (GiR). The connection consists of a spruce block (120 x 120 x 300 mm), equipped with bore holes so to embed a reinforcement bar made of glass-fibre reinforced polymer (G-FRP, Ø16 mm). Way more details regarding GiR production and the materials involved can be found in e.g. [10] and will not be repeatedly presented herein.

2.5 LOW-TEMPERATURE EXPERIMENTS

2.5.1 Pre-conditioning at low temperatures

Prior induction heating, all specimens for which low-temperature curing was targeted (cf. Table 2), were pre-conditioned within climate chambers at a temperature of –30 °C (cf. Figure 2). This was done to ensure adhesive temperatures of +5 and –10 °C at the start of inductive heating by letting the specimens re-warm under laboratory RT conditions until the starting temperatures were reached. Depending on the joint geometry, total duration for pre-conditioning differed to ensure that all parts of the joints were cooled to the target temperature.

Figure 2: *Pre-conditioning of wooden SLS joints equipped with thermocouple within climate chamber at –30 °C, adhesive-CP mix applied but not yet cured*

For DB and SLS specimens, the adherends and silicon moulds pre-cooled. Afterwards, adhesive-CP application was done within the climate chambers, which immediately cooled down the mixes and reduced polymerisation progress to a minimum. When the respective DB specimen or SLS series was cooled, they were transported to the induction device (~1 min). Finally, thermocouples were attached to the measurement electronics and the induction device was switched on when thermocouple temperature reached +5 or –10 °C.

2.5.2 Experimental setup & inductive heating

For all low-temperature inductive heating operations, the induction device TrueHeat HF 5010 (Trumpf Hüttinger GmbH & Co. KG, Germany) was used. As an example, Figure 3 shows the inductive heating of a set of five spruce SLS joints (thermocouple attached to middle specimen).

Figure 3: *Experimental setup for inductive heating using five SLS joints (herein bonded with LP421) as an example, P = 2.9 kW, I = 15.8 A, U = 384 V, f = 134 kHz*

All specimen were placed centrally within the induction coils (cf. Figure 3) and cured for 10 min with the EMF in

https://doi.org/10.52202/069179-0046

order to maintain comparability between the specimen types investigated. However, for some low-temperature GiR series, induction time had to be increased to 12.5 min in order to reach high temperatures and thus ensure full cure.

2.6 MECHANICAL TESTING

All specimen types were destructively tested using Zwick||Roell universal testing machines (UTM's). Testing was performed at a speed of 2 mm/min under RT conditions (23 ± 2 °C, 50 ± 10 °C). Testing times were dependent upon the cooling rates of the specimens, with SLS and DB specimens tested ~0.5 h and GiR ~2 h after inductive heating ended. To get an impression of the mechanical tests, Figure 4 shows the setups used for GiR and DB specimens.

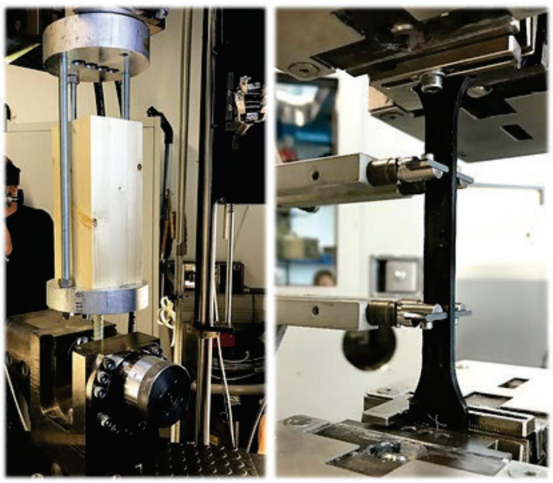

Figure 4: *Setups for mechanical testing of low-temperature cured GiR (left) and DB specimens (right), testing performed at RT*

2.7 EXPERIMENTAL PROGRAM

For a better overview, the complete experimental program has been summarised in Table 2. The experiments included references (unfilled and CP-filled) as well as inductively cured specimens starting from +23, +5 as well as –10 °C for both considered adhesives. The testing times can be found in section 2.6.

Table 2: *Experimental program produced for each Fi390 (2K-EPX) as well as LP421 (2K-PUR), CC = Cold cured at RT, IND = Inductively cured*

Type	Series				
	CC_ Ref	CC_ CP	IND_ +23	IND_ +5	IND_ -10
DB	-	-	5	5	5
SLS	5	5	5	5	5
GiR	5	5	4	4	4

3 RESULTS

3.1 HEATING BEHAVIOUR

In a first step, the heating behaviour for SLS and GiR joints bonded with Fi390 have been visualised in Figure 5. It can be seen that, depending upon specimen geometry, curing temperatures develop fundamentally different. Thus, for the way smaller SLS joints, curing temperatures of roughly 100–110 °C after ~4 min of continuous induction heating can be measured. In contrast, the bigger GiR do not reach the same temperature range although total induction time was 2.5 min longer (12.5 min) as for the SLS specimens (10 min). Consequently, the SLS temperature curve shows a clear flattening around T_c (110 °C), while mean GiR temperatures are still ~90 °C.

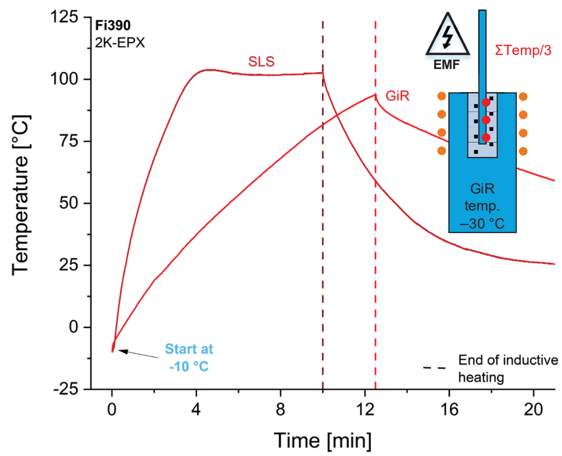

Figure 5: *Curing temperatures during inductive heating of joints bonded with Fi390 recorded with thermocouples embedded within the adhesive-CP mixes*

3.2 MECHANICAL PERFORMANCE

3.2.1 Dogbone

The mechanical results for the inductively cured DB specimens are shown in Figure 6.

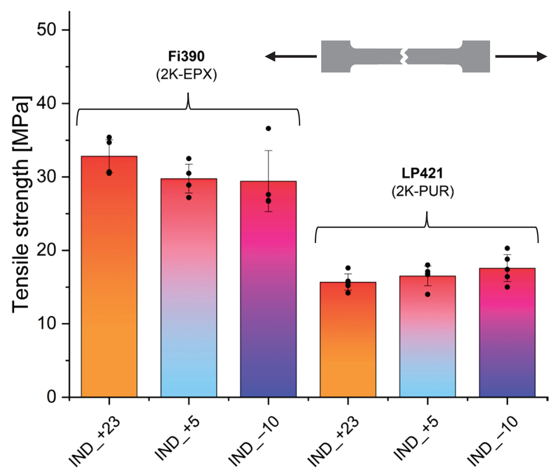

Figure 6: *Mechanical results for tensile strengths measured with DB specimens type 1BA for the two considered adhesives, v = 2 mm/min, tested at RT, Zwick|Roell UTM 50 kN*

The data reveals that the starting temperature of the induction process has no influence on measurable tensile strengths, which holds true for both considered polymers. At this point, the authors want to point out that a direct comparison with tensile strengths listed in Table 1 is not given since specimens geometry and testing conditions differed.

https://doi.org/10.52202/069179-0046

3.2.2 Lap shear specimens

In the next step, lap shear strengths have been visualised in Figure 7. It can be seen that generally all series result in a tear-off of the wooden fibres, with lap shear strengths of 7–9 MPa being reached. In addition, fracture patterns within the respective series are identical. These observations are valid for both the 2K-EPX as well as the 2K-PUR adhesive.

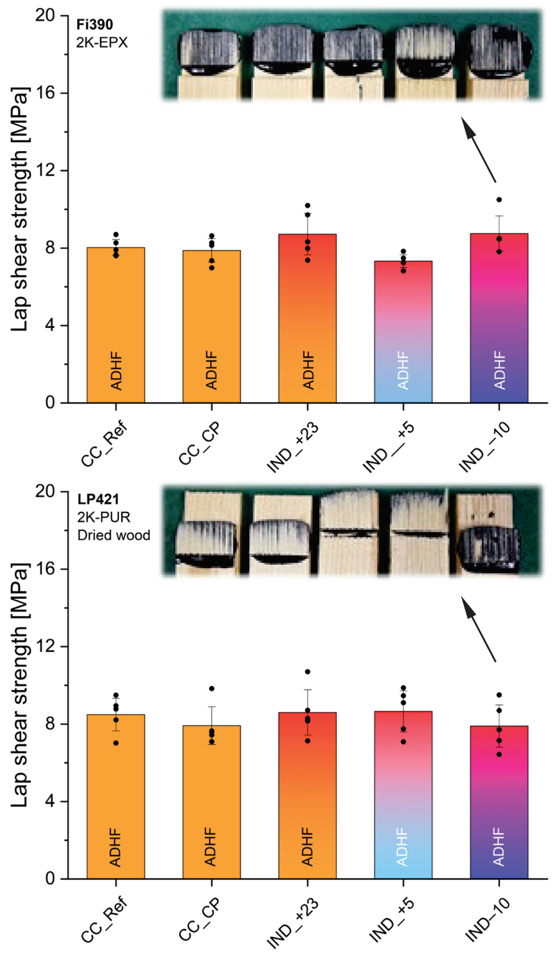

Figure 7: *Mechanical results for spruce SLS joints bonded with the two considered adhesives, v = 2 mm/min, tested at RT, Zwick|Roell UTM 20 kN; ADHF = Adherend failure; adherends for polyurethane dried in oven (moisture content of 0 %, measured immediately prior adhesive-CP application)*

3.2.3 Glued-in Rods

Finally, the mechanical results of the GiR-related mechanical tests have been illustrated in Figure 8. For the first adhesive under investigation, Fi390, all series – independently of the curing conditions considered – resulted in a pull-out of a wooden plug, indicating that the wooden adherends were the weakest part of the connections – a result, which corresponds to the findings on lap shear scale.

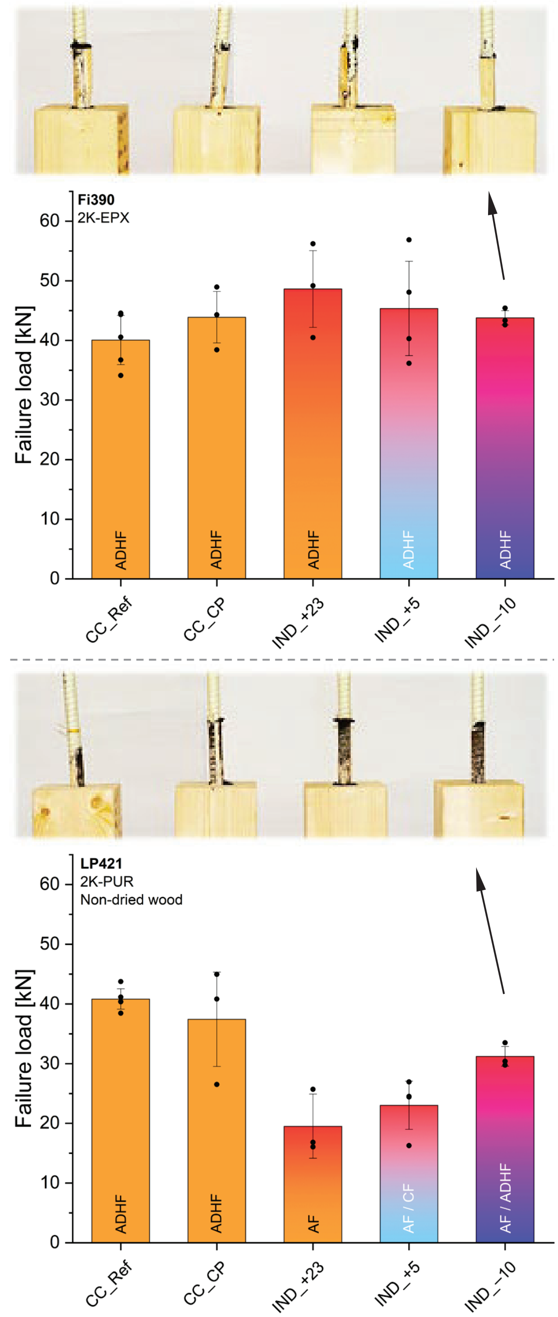

Figure 8: *Mechanical results for GiR joints bonded with the two considered adhesives, v = 1 mm/min, tested at RT, Zwick|Roell UTM 100 kN; ADHF = Adherend failure, all adherends non-dried with moisture content of 8.7 ± 0.9 %*

In contrast, GiR bonded with the 2K-PUR show another behaviour. Thus, for those series that were cured without the use of thermal energy, mechanical tests resulted in a failure of the wooden adherends (wood tear-out). However, when heat is introduced over the particles, failure loads decrease by ~60 % (inductive heating starting from +23 °C), ~50 % (+5 °C) as well as ~25 % (–10 °C), indicating that mechanical performance increases the lower the starting temperature of the joints is chosen. Aforementioned behaviour is accompanied by changes in fracture patterns from adhesion failure on side of the wood (+23 °C) to mixed adherend / adhesion failure (–10 °C).

4 DISCUSSION

4.1 HEATING BEHAVIOUR

With regard to the curing temperatures (cf. Figure 5), the results revealed a strong influence of joint geometry on the heating behaviour. Compared to the small SLS joints, the way bigger GiR (120 x 120 x 300 mm) needed significantly more time (~10 min) to be heated to the necessary temperature range (> 80 °C) to cure the joints in a decent amount of time. These observations can be traced back to the fact that the comparatively long adhesive layer of the GiR (100 mm) promotes heat dissipation from the adhesive towards the remaining parts of the joints, resulting in lower heating rates in the following. In contrast, the way smaller SLS joints require less heat due to the smaller ratio between adhesive layer thickness and area available for heat transfer. Furthermore, a significantly lower mass of material has to be heated.

A further aspect to be considered is the heat that is released by the exothermic polymerisation, which can be expected to be different depending on the kinetics of the respective polymer. It is already known that this effect may lead to strong inhomogeneity's in heating depending upon joint geometry and adhesive layer thickness[11]. It can thus be concluded that any practical application will require a numerical simulation model, which covers heating-related effects and enables identification of best boundary conditions for optimal and most homogeneous heating. The aforementioned relations have already been presented and discussed in many publications of the authors and will thus not be repeatedly described herein [8,12].

4.2 MECHANICAL PERFORMANCE

4.2.1 Bulk level – Dogbone specimens

The tensile tests on the level of inductively cured DB specimens with different curing conditions resulted in indistinguishable results for both adhesives when the starting temperature of the induction process was varied between +23, +5 as well as –10 °C. These promising results suggest that the general build-up of cohesion is neither hindered nor prevented by the presence of the particles, the inductive heating process as well as the low starting temperatures of the polymerisation. However, from previous investigations it is already known that the mere presence of the much stiffer particles may lead to stress concentrations and earlier failure when unfilled reference and CP-filled DB specimens cured at RT are compared. These negative impacts are, however, counterbalanced when thermal energy is introduced over the susceptors – a process that brings tensile strengths back to the level of the unfilled references [13].

4.2.2 Compound level – Lap shear and Glued-in Rods

The mechanical tests on both the level of SLS as well as GiR joints resulted in promising findings, whereby all EPX-bonded specimens showed a clear adherend failure of the wooden substrates. It can thus be concluded that the general capability of the adhesive to build up significant adhesive strength is not hindered by both the curing process as well as the cold adherend temperatures. The process is thus very well suited for the 2K-EPX adhesive Fi390.

With regard to the LP421-bonded joints (2K-PUR), results behaved differently. In detail, the GiR cured at elevated temperatures showed significantly lower failure loads as well as mixed failure modes (adhesion / cohesion failure). The reason for the deterioration in mechanical performance can be found in the moisture contrained within the wood adherends, which diffuses from the adherends towards the adhesive layer when temperature is applied. In the following, the authors assume that the isocyanate groups of the polyurethane react with the released moisture, leading to both lowered cohesive as well as adhesive strength on side of the wood in the following. These assumptions are supported by the results on lap shear scale (cf. Figure 7-bottom) for which dried adherends were used and thus clear substrate failure could be achieved. In addition, the authors suppose that mechanical performance for low-temperature cured GiR is better due to slower heat development, i.e. slower moisture diffusion (cf. Figure 5, curve for GiR). However, since targeted drying of wooden structures may not represent a realistic solution with regard to practical application, these circumstances might exclude the use of polyurethane adhesives for the technique when temperatures. As a workaround, particles with a lower T_c might offer a way out of the wood-polyurethane-related problems discussed and thus enable their safe CP-curing.

In summary it has been shown that curing of adhesively bonded wooden structures starting in temperature ranges currently not intended by building authorities can be achieved by application of induction heating. Since onsite polymerisation under adverse temperature conditions like those presented would last weeks or not occur at all, the findings might pave the way for additional application fields for bonded timber connections in the future.

5 CONCLUSION

The study focuses on low-temperature curing of structural wood-adhesives (epoxy and polyurethane) using Curie particles (CP), which are added to the polymers and can be heated by an electromagnetic field (EMF) up to T_c but not beyond. For that, experimental investigations were carried out considering three different starting temperatures for the polymerisation (+23, +5 and –10 °C). In a first step, standardised specimen types (dogbone and single lap shear) were produced, after which the investigations were scaled up to the level of large wooden) Glued-in Rods (GiR). The following important findings were made:

- The heating behaviour of low-temperature cured wooden joints using inductively heated particles represents complex due to multifarious conditions such as joint geometry, the materials involved, joint starting temperature,

https://doi.org/10.52202/069179-0046

polymerisation enthalpy as well as the particle type determining the temperature distribution throughout the joint. For better process control, a numerical model would thus be necessary to identify optimal heating conditions and thus avoid cumbersome preliminary experiments.

- The mechanical results showed that tensile strength is identical for different starting temperatures of the induction process (+23, +5, -10 °C). The development of significant cohesive strength takes place, which was observed for both adhesives. In addition, it is known that two counteracting effects have to be considered: Firstly, deterioration of mechanical performance by the mere presence of the particles embedded within the polymers, secondly, counterbalance of negative impact by heat introduction to or above the level of the references[13].
- On the level of small (SLS) and large joints (GiR), a substrate failure could be attained for all EPX-bonded joints, suggesting that the technique is very reliable for EPX adhesives. For the 2K-PUR adhesive, a restriction has to be formulated: When wooden adherends are used and heat is introduced over the particles, moisture diffuses from the wooden adherends and interferes in the cross-linking reaction of the polyurethane. In the following, both adhesive as well as cohesive strength is deteriorated. In order to solve this problem, the authors assume that a lower T_c of the particles will lead to better mechanical results. On the level of SLS joints, polyurethane-bonded dried wood adherends showed a fracture behaviour indistinguishable to those of the references.
- Above all it can be summarised that low-temperature curing of wood structures has been made possible with the presented technique. In detail, the two considered structural adhesives would need weeks to cure at a temperature of –10 °C, which has been reduced to an induction time of ~10 min – a time window, which can be further reduced by e.g. inclusion of the cooling phase for curing or adapting the CP.

ACKNOWLEDGEMENT

The authors sincerely thank Dr. Joachim Schätzel of the Fischerwerke GmbH & Co. KG and Mr. Gordian Stapf from the Henkel AG & co. KGaA for providing the adhesives.

6 References

[1] Rudawska A. The influence of curing conditions on the strength of adhesive joints. The Journal of Adhesion 2020;96(1-4):402–22. https://doi.org/10.1080/00218464.2019.1656615.

[2] Benedetti A, Fernandes P, Granja JL, Sena-Cruz J, Azenha M. Influence of temperature on the curing of an epoxy adhesive and its influence on bond behaviour of NSM-CFRP systems. Composites Part B: Engineering 2016;89:219–29. https://doi.org/10.1016/j.compositesb.2015.11.034.

[3] Moussa, O. Vassilopoulos, A. P., Keller T. Effects of low-temperature curing on physical behavior of cold-curing epoxy adhesives in bridge construction. International Journal of Adhesion and Adhesives 2012;32:15–22. https://doi.org/10.1016/j.ijadhadh.2011.09.001.

[4] Moussa O, Vassilopoulos AP, Castro J de, Keller T. Early-age tensile properties of structural epoxy adhesives subjected to low-temperature curing. International Journal of Adhesion and Adhesives 2012;35:9–16. https://doi.org/10.1016/j.ijadhadh.2012.01.023.

[5] Ratsch N, Burnett-Barking M, Böhm S, Voß M, Adam M, Kaufmann M et al. Accelerated curing of glued-in threaded rods by means of inductive heating — part IV: curing under low temperatures. The Journal of Adhesion 2022;98(2):105–30. https://doi.org/10.1080/00218464.2020.1818562.

[6] Arrhenius S. Über die Dissociationswärme und den Einfluss der Temperatur auf den Dissociationsgrad der Elektrolyte. Zeitschrift für Physikalische Chemie 408(1):96–116.

[7] DIN EN 527-2:2019-07, Büromöbel_- Büro-Arbeitstische_- Teil_2: Anforderungen an die Sicherheit, Festigkeit und Dauerhaltbarkeit; Deutsche Fassung EN_527-2:2016+A1:2019. Berlin: Beuth Verlag GmbH. https://doi.org/10.31030/3030475.

[8] Voß M, Kaufmann M, Vallée T. Curie-supported accelerated curing by means of inductive heating – Part I: Model building. The Journal of Adhesion 2022;98(10):1394–437. https://doi.org/10.1080/00218464.2021.1902810.

[9] DIN EN 1465:2009-07, Klebstoffe_- Bestimmung der Zugscherfestigkeit von Überlappungsklebungen; Deutsche Fassung EN_1465:2009. Berlin: Beuth Verlag GmbH. https://doi.org/10.31030/1507601.

[10] Voß M, Vallée T. Accelerated curing of G-FRP rods glued into timber by means of inductive heating using Curie-particles – large-scale experiments at room temperature. The Journal of Adhesion 2021;97(16):1532–60. https://doi.org/10.1080/00218464.2020.1803067.

[11] Voß M, Vallée T. Accelerated curing of G-FRP rods glued into timber by means of inductive heating – Influences of curing kinetics. The Journal of Adhesion 2022;98(8):1037–75. https://doi.org/10.1080/00218464.2020.1870450.

[12] Voß M, Kaufmann M, Vallée T. Curie–supported accelerated curing by means of inductive heating – Part II Validation and numerical studies. The Journal of Adhesion 2022;98(13):2045–77. https://doi.org/10.1080/00218464.2021.1951712.

[13] Voß M, Vallée T. Effects of Curie particle induced accelerated curing on thermo mechanical performance of 2K structural adhesives – Part I: Bulk properties. The Journal of Adhesion 2022;98(9):1298–339. https://doi.org/10.1080/00218464.2021.1909482.