Accelerated curing of Glued-in-Rods by using Curie-Particles

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Résumé :

Les adhésifs couramment utilisés pour le collage dans les tiges (GiR) dans l'ingénierie du bois, les époxydes à deux composants (2K-EPX) ou les polyuréthanes (2K-PUR) durcissant à froid, ne durcissent que relativement lentement (généralement en heures ou en jours), ce qui est des magnitudes de fois plus long que la fixation mécanique. Des contraintes supplémentaires découlent du fait qu'elles nécessitent généralement une température minimale pour que la polymérisation puisse avoir lieu. Le chauffage par induction a été étudié pour deux applications de la technique du bois, à savoir les tiges en composite collées dans le bois et les structures en bois-verre.

Abstract :

Commonly used adhesives for Glued-in-rods (GiR) in timber engineering, cold curing two-component (2K) epoxies or polyurethanes, only harden relatively slowly (usually in hours to days), which is magnitudes of times longer than mechanical fastening. Additional constraints arise from the fact that they usually necessitate some minimum temperature so that polymerisation can take place. Induction heating was investigated in the light of two timber engineering applications, glued-in glass fibre reinforced rods in timber, and timber-glass structures.

Mots clefs : Glued-in rods, accelerated, curing, adhesives, FRP-rods

1 Introduction

Commonly used adhesives for glued-in rods in timber engineering, cold curing two-components (2K) epoxies (EPX) or polyurethanes(PUR), only harden relatively slowly (usually in hours to days). Additional constraints associated the fact that they usually necessitate some minimum temperature so that polymerisation can take place. Thus, depending on the location, bonding onsite can only occur in a limited number of months of a year. Adhesive curing can be accelerated by different methods. However, the most widely used method is increasing the temperature. One of the used techniques to generate heat is electromagnetic induction. If metallic adherends are considered, induction heating acts on them; if inductive heating is to be used on non-conductive adherends, it is necessary to ensure that the adhesive reacts to electro-magnetic fields; this is mostly achieved by adding appropriate particles or electrically conductive meshes [1,2]. For example, Curie particles, they are made of material that interacts with electromagnetic fields below a certain temperature (Curie temperature, TC), but are

(almost) insensitive to it beyond T_C ; these properties of Curie particle makes them an effective tool for controlling induction processes. Previous research pointed out that Curie particles have the potential to lift the constraints associated with temperature and induction power control. However, a series of complementary questions have to be answered, among them quantifying the effect of relatively high mass percentage of metallic particles on the mechanical properties and the workability, respectively the durability of the particles embedded within the adhesive.

2 Materials and Methods

Four adhesives were considered for this study: CR412, a 2K-polyurethane, and FIS EM, EP32 and Jo692.30 which were 2K-epoxies. All adhesives are commercially available and commonly used in timber engineering, where they are expected to cure under room temperature (RT, 23 °C) for relatively long time durations (from 18 hours to 10 days), as indicated by Table 1. For all experimental investigations presented herein, spruce was used in form of GLT. Quality was 24GL. Glass-Fibre Reinforced Polymer (FRP) textured bars Ø16 mm were delivered by the company Schöck/Germany, which markets them under the name ComBAR. If loaded in traction, they fail in a brittle manner, however, at tensile strengths beyond 1'000 MPa. Curie-particles, in a nutshell, consist of material that is susceptible to electromagnetic fields below a certain temperature labelled Curie-temperature (T_c), but loses this property beyond T_c . The particles used herein were supposed, to exhibit a TC of 110 °C according to their manufacturer.

Adhesive	Curing time at RT	Shear strength [MPa]	Heating rate	Curing temp.
CR412	10d	12.5	2 K/s	100 °C
FIS EM	18h	10.0	2 K/s	100 °C
EP32	10d	11.4	2 K/s	90 °C
Jo692.30	2d	9.7	2 K/s	80 °C

Table 1: Adhesives used in this study, lap shear strengths obtained on aluminium for cold curing, and accelerated curing, respectively

Addition of particles to an adhesive impacts the rheological behaviour of the resulting mixture. The corresponding increase of viscosity, in particular, might severely impact the filling process of the GiR. For these tests, a generic 1K epoxy was used, with mass fractions of 10 %, 20 %, 30 % and 40 %.

The Curie particles were characterized with regard to their potential to generate thermal energy is exposed to electromagnetic fields. All three particle types were mixed with mass fractions of 10 %, 20 %, 30 % and 40 % with a 1K-epoxy, and cast into small cylindrical probes Ø20 mm x 20 mm height. The samples were hollow, and exhibited a thickness of 2 mm. These dimensions were selected to reflect the adhesive layer in larger-scale probes. All 12 resulting specimens were then cured in an oven at a temperature of 120 °C for over 4 hours, such to ensure their complete curing; thus no exothermic was expected to be released in any test that would follow up.

The cylindrical probes were placed in the middle of an induction coil (Figure 1). The same induction parameters as for the subsequent large scale tests, in particular the induction frequency of 120 kHz, were used. Temperatures were monitored over time with two thermocouples; data logger filtered any interference on the thermocouples resulting from the electromagnetic field.



Figure 1: (a) Inductive heating of small cylindrical probes of adhesive-particle mixtures, temperatures were measured with two thermocouples; (b) Small cylindrical probes of adhesive-particle mixtures (height: 20 mm, diameter: 20 mm, thickness: 2mm)

Large scale samples consisted in blocks of spruce GLT 120 x 120 mm² in cross-section, and a length of 300 mm. Holes Ø20 mm were drilled centrally up to a depth of 100 mm. For the determination of reference values, GRP rods were inserted into the holes and filled with adhesive through a lateral hole from below, and left to cure according to the TDS. In a second series, 33 % Curie particles were added to the adhesive. Correct mixing of the particles was ensured by using a speed mixer (at 1000 rpm) under vacuum for 45 s. The adhesive-particle mixtures were then injected in the GiR, and without any further delay subjected to the induction process, such that the 2K adhesives had no significant time to cold-cure.

The frequency of the electromagnetic field was 120 kHz. The induction process itself consisted in delivering the full power for a total duration of 10 minutes, without any external control by thermocouples. After the induction process, all probes were left to cool down for different times prior to testing. Both series of cold and induction cured GiR were tested in tension at a loading rate of 2 mm/min until failure.

3 Results

The induction tests performed on the cylindrical samples resulted in a series of temperature increases over time for one particle type with four different mass fractions thereof. The result shows that if subjected to a constant electromagnetic field, the adhesive-particle mixes' temperature increases. However, that temperature increase is not unbound, and that it converges towards an upper limit. The temperature increase proved to be dependent on the percentage of particles mixed to the adhesive, with higher mass fractions leading to faster increases. Independently on the mass fraction, however, the upper limit is the same for all mixtures, and this limit corresponds to the Curie-temperature.

Failure modes of the tested GiR proved similar to those observed for cold cured probes, as shown exemplarily in Figure 4 for CR 412 with particle. Resulting joint capacities, reported in Table 2 below, show that, if tested after 24 h of their inductively heating, GiR developed joint capacity lower by one quarter for the 2K-EPX, and only one half for the 2K-PUR, if compared to their cold cured counterparts. If tested after shorter waiting times, i.e. 2 h after inductive heating, with the adhesive still exhibiting temperatures of around 35 °C (cf. Figure 5 (a) for Jo692.30, a 2K-EPX, and Figure 5 (b) for CR 421, a 2K-PUR), significant handling strengths are obtained. These are roughly between half and two thirds of the strengths achieved through cold-curing.





Figure 3: Results of the inductive heating experiments on cylindrical adhesive particle mixtures

Figure 4 : Inductive heating and typical failure mode (CR421)

 Table 2: Experimentally determined joint capacities (in kN)

Adhesive	Cold cured		Inductive, after 24h		Diff [9/1
	Average	StdDev.	Average	StdDev.	— Dili. [/0]
CR421	60,4	3,6	31,7	5,1	-48%
EP32	62,0	12,2	46,1	4,7	-26%
Jo692.30	58,2	1,7	43,8	4,4	-25%
FIS EM	59,5	1,7	47,0	4,2	-21%



Figure 5: (a) Load-displacement curve of inductively heated GiR with Jo693 (a) and CR421 (b) tested after 2 h, compared to cold cured ones,

4 Discussion and conclusions

The Curie particles, which were added to the adhesives, were thermally characterized. It was shown that they were susceptible to the electromagnetic field, and that they increased the temperature. Depending upon the mass fraction added, temperatures could be raised at different paces. However, independently of the proportion of particles, temperatures converged towards an upper limit, which was the Curie-temperature of the considered material.

If compared to the cold cured GiR, inductively cured achieved strengths of roughly 75 %, except for the 2K-PUR, which reached only half the reference strength. If tested after 2 h, with adhesives still exhibiting temperatures of 35 $^{\circ}$ C, two thirds of final strength achieved.

The reduced joint capacities are attributed to the addition of 33 % of particles, which negatively impacted the capacity of the adhesives to develop their full potential. This conclusion is drawn because the unfilled 2K-adhesives, after accelerated curing, proved superior in mechanical terms to the cold cured ones.

Curing of glued-in rods was accelerated inductively by mixing Curie particles to commercial available and commonly used 2K-adhesives (PUR and EPX). Although preliminary investigations showed that 2K adhesives can be significantly cured, with reductions from hours to days down to minutes, resulting joint capacities of large scale GiR proved slightly lower, if compared to that of cold-cured probes. Additional investigations showed that the technique presented allows to obtain significant handling strengths within 2 h. The somewhat lower joint capacities were posited to result from the relatively high mass fraction of particles. Further studies might attenuate these issues.

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