Accelerated curing of adhesives using inductively heated Curie particles

From the department of production engineering

of the

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approved

Dissertation

by

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Beschleunigte Aushärtung von Klebstoffen mit induktiv erwärmten Curie Partikeln

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Dissertation

von

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Tag der mündlichen Prüfung: 02.02.2023

‘*A beautiful flight is turbulent.*’ – YKKE

For Anke, Bernd & Margret.

Declaration on oath

I – Morten Voß –

declare by my signature that I have prepared the present thesis independently and without unauthorised external help, that I have not used any sources or aids other than those indicated by me, and that I have marked any passages taken verbatim or in terms of content from the used works as such. The electronic version of the dissertation enclosed for examination purposes is identical to the version submitted.[1]

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erkläre durch meine Unterschrift, dass ich die vorliegende Arbeit selbstständig und ohne unerlaubte fremde Hilfe angefertigt habe, keine anderen als die von mir angegebenen Quellen und Hilfsmittel benutzt und die den benutzten Werken wörtlich oder inhaltlich entnommenen Stellen als solche kenntlich gemacht habe. Die zu Prüfungszwecken beigelegte elektronische Version der Dissertation ist identisch mit der abgegebenen gedruckten Version.[1]

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[1]Entnommen aus der Promotionsordnung der UNIVERSITÄT BREMEN für den Fachbereich 04 – Produktionstechnik vom 20.08.2020.

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Publication list and declarations of authorship

In the following, I have listed all publications that cumulatively form this doctoral thesis along with the associated declarations of authorship. For that, I commit myself to the official credit author statements published by ELSEVIER, which are accessible over the following link *https://www.elsevier.com/authors/policies-and-guidelines/credit-author-statement*, and include the terms and associated definitions listed in Table 1.

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1st publication

'Accelerated curing of G-FRP rods glued into timber by means of inductive heating – Influences of curing kinetics'
M. VOß & T. VALLÉE
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T. VALLÉE: Conceptualisation, Funding Acquisition, Project Administration, Supervision, Writing – Review & Editing.

8th publication

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A complete overview of my scientific contributions can be found in the curriculum vitæ included in part V of the thesis.

Abstract

The present thesis summarises my research on accelerated curing of adhesives, and adhesively bonded connections, using inductively heated Curie-particles (CP). Induction heating is used as a rapid and versatile heating technique. Curie-particles have been considered because of their property to generate heat up to their corresponding Curie temperature, if subjected to an electromagnetic field (EMF), but not beyond. The combination of induction heating and CP aimed at resulting in an accelerated curing process completely freed of the constraints of process control as usual for adhesively bonded joints.

In the first part of my thesis, I describe the conceptual and technical background necessary to understand the core issues of my research, and detail its scientific objectives. The second part includes all necessary information regarding the materials used and methods applied for the accelerated curing experiments. In the third part, my eight peer-reviewed accepted publications that constitute the core of my thesis are summarised. In the next, fourth, part, the achievements of the scientif work were assessed, a summary and an outlook conclude the thesis. Finally, the fifth part includes my curriculum vitæ and other personal information. During the research, I maintained a coherence between the individual chapters represented by the individual publications by systematically using the same adhesives and CP so to incrementally increase the level of investigation complexity based upon a solid background.

The scientific approach started, as outlined in my 1st paper, with characterising the curing kinetics so to identify changes thereof resulting from the addition of CP. For that, appropriate kinetic models were developed that allowed for the prediction of the curing degree in function of curing temperature and CP content for a series of two-component (2K) adhesives. The kinetic models were then applied to subsequently experimentally determined temperature profiles. This approach allowed for a much better interpretation of the heating behaviour during the induction processes, which proved to be influenced not only by the inductively generated heat, but also significantly by the exothermic polymerisation of the adhesives.

After the role of curing kinetics was successfully characterised, I wanted to know how the mechanical properties of the adhesives got altered by adding CP. I achieved this by performing a detailed thermo-mechanical characterisation of two exemplary 2K structural adhesives (EPX and PUR), which was split into two parts (2nd and 3rd publication). The 2nd paper focused on alterations of intrinsic adhesive properties like cohesive strength, stiffness or glass transition temperature. Overall, I found that induction curing with CP significantly influences the adhesive properties, leading to a fundamentally different, yet not necessarily degraded material behaviour. In principle the same conclusion can be drawn

for the second part of the thermo-mechanical characterisation, extensively presented in my 3rd publication, which focused on the compound characteristics on level of single lap shear specimens produced with different substrate materials. In addition to these contents, publication two and three contain further investigations regarding the heating behaviour of CP-cured adhesives, which revealed additional parameters influencing the curing process and its outcome.

Building on the achieved up this point, investigations were scaled up to the large component level: G-FRP rods glued into blocks of spruce, so-called Glued-in Rods (GiR), which constitute my 4th paper. The experiments proved the general applicability of the curing technique for large joints. They also allowed to validate appropriate temperature monitoring solutions. The results showed that CP-curing leads to joint strengths indistinguishable from those obtained for unfilled references, while significantly reducing curing time.

The research of publication 1–4 showed that the relationship between the complex heating behaviour and the progression of the degree of cure is an essential ingredient to describe the process. Up to this point, assessing whether a joint has been exposed sufficiently to the EMF for full cure, or not, was only possible in two ways: Firstly, by predicting the degree of cure based upon the kinetic models developed in my 1st paper, with temperature data recorded during the induction curing of a respective sample. Secondly, by comparing joint strengths experimentally determined on test series cured for different periods. Both options are associated with extensive experimental work, and remain empiric. To contribute to more predictable and efficient CP-curing operations, I thus developed a numerical model based on the Finite Element Method (FEM). This work was split into two parts, corresponding to my 5th and 6th publication, respectively. The modelling aimed to predict the adhesives curing behaviour using the exemplary application of GiR investigated experimentally in my 4th paper, relieving practitioners from extensive experimental approaches.

For the modelling, presented in my 5th paper, the kinetic models derived in my 1st publication were linked to a transient heat flow simulation in Ansys Academics 2019 R1®, which foot upon experimentally validated heat loads. As a result, it was made possible to predict the curing degree in dependency of curing temperature and CP content numerically. In detail, the publication presents the preliminary experimental work as well as all analytical methods implemented for the modelling. In addition, principal functionality of the simulation model was illustrated on temperature and curing degree profiles. The second part of the numerical modelling (6th publication) resulted in the validation of the FEA, which was then used to perform a series of parameter studies so to identify the most important influencers contributing to the heating behaviour of CP-cured joints. It was shown that, amongst others, adhesive layer thickness and CP content exert the greatest influence on arising curing temperatures.

Unlike mechanical joining, adhesive bonding requires some minimum temperatures in order for the adhesives to cure correctly. To circumvent this restriction, I validated in my 7th paper the use of inductive heating with CP for low temperature curing of adhesives. For that, the experiments described in my 4th publication were repeated at low temperatures (–10 °C and –30 °C). The results proved that the process

is well capable of curing adhesives in temperature ranges not yet explored in literature, with a fracture behaviour of the epoxy adhesives indistinguishable to that of RT-cured reference sets.

Finally, in my 8th publication, I took a step further towards industrial application of CP-curing. I was able to design an additional accelerated curing scenario based upon all previous studies, which was embodied in form of a series of single lap shear tubular joints (SLTJ) made of glass fibre reinforced plastic (G-FRP). Through this approach, validity and transferability of the previous results was successfully proven. Furthermore, the results of the paper made clear that a significant reduction in curing time can be achieved with comparatively few CP without deterioration of joint strengths.

Zusammenfassung

Die vorliegende Arbeit fasst meine Forschungsarbeiten über die beschleunigte Aushärtung von Klebstoffen und Klebverbindungen mit Hilfe induktiv erwärmter Curie Partikel (CP) zusammen. Die Induktionserwärmung wird als schnelle und vielseitige Erwärmungstechnik eingesetzt. CP wurden wegen ihrer Eigenschaften in Betracht gezogen, Wärme bis zu ihrer entsprechenden Curie-Temperatur zu erzeugen, wenn sie einem elektromagnetischen Feld (EMF) ausgesetzt werden, aber nicht darüber hinaus. Die Kombination von Induktionserwärmung und CP sollte zu einem beschleunigten Aushärteprozess führen, der völlig frei von den Zwängen der Prozesssteuerung abläuft, wie sie bei Klebverbindungen üblicherweise notwendig sind.

Im ersten Teil meiner Arbeit beschreibe ich den konzeptionellen und technischen Hintergrund, der für das Verständnis der Kernfragen meiner Forschung notwendig ist, und erläutere die wissenschaftlichen Ziele. Der zweite Teil enthält die notwendigen Informationen über die verwendeten Materialien und die für die Experimente zur beschleunigten Aushärtung angewandten Methoden. Im dritten Teil werden meine acht begutachteten und aktzeptierten Veröffentlichungen, die den Kern meiner Arbeit bilden, zusammengefasst. Im nächsten, vierten Teil werden die Ergebnisse der wissenschaftlichen Arbeit bewertet, eine Zusammenfassung und ein Ausblick schließen die Arbeit ab. Schließlich enthält der fünfte Teil meinen Lebenslauf und andere persönliche Informationen. Während der Forschung habe ich die Kohärenz zwischen den einzelnen Kapiteln der einzelnen Veröffentlichungen aufrechterhalten, indem ich systematisch dieselben Klebstoffe und CP verwendet habe, um den Komplexitätsgrad der Untersuchungen auf der Grundlage eines soliden Hintergrunds schrittweise zu erhöhen.

Der wissenschaftliche Ansatz begann, wie in meiner ersten Veröffentlichung beschrieben, mit der Charakterisierung der Aushärtekinetik, um deren Veränderungen durch die Zugabe von CP zu identifizieren. Dazu wurden geeignete kinetische Modelle entwickelt, die die Vorhersage des Aushärtungsgrads in Abhängigkeit der Aushärtungstemperatur und des CP-Gehalts für eine Reihe von zweikomponentigen (2K) Klebstoffen ermöglichten. Die kinetischen Modelle wurden dann auf anschließend experimentell ermittelte Temperaturprofile angewandt. Dieser Ansatz ermöglichte eine wesentlich bessere Interpretation des Erwärmungsverhaltens während der Induktionsprozesse, das nicht nur durch die induktiv erzeugte Wärme, sondern auch wesentlich durch die exotherme Polymerisation der Klebstoffe beeinflusst wurde.

Nachdem die Rolle der Aushärtekinetik erfolgreich charakterisiert worden war, wollte ich wissen, wie sich die mechanischen Eigenschaften der Klebstoffe durch die Zugabe von CP verändern. Zu diesem Zweck führte ich eine detaillierte thermo-mechanische Charakterisierung von zwei beispielhaften 2K-

Strukturklebstoffen (EPX und PUR) durch, die in zwei Teile aufgeteilt wurde (zweite und dritte Veröffentlichung). Die zweite Veröffentlichung konzentrierte sich auf Veränderungen der intrinsischen Klebstoffeigenschaften wie Kohäsionsfestigkeit, Steifigkeit oder Glasübergangstemperatur. Insgesamt stellte ich fest, dass die Induktionshärtung mit CP die Klebstoffeigenschaften erheblich beeinflusst und zu einem grundlegend anderen, aber nicht unbedingt schlechteren Materialverhalten führt. Im Prinzip kann dieselbe Schlussfolgerung für den zweiten Teil der thermo-mechanischen Charakterisierung gezogen werden, der in meiner dritten Veröffentlichung ausführlich dargestellt wurde und sich auf die Verbindungseigenschaften auf Ebene von einfach überlappenden Zugscherproben aus verschiedenen Substraten konzentriert. Zusätzlich zu diesen Inhalten enthalten die Publikationen zwei und drei weitere Untersuchungen zum Erwärmungsverhalten von CP-gehärteten Klebstoffen, die zusätzliche Parameter aufzeigten, die den Aushärtungsprozess und sein Ergebnis beeinflussen.

Aufbauend auf dem bis dahin Erreichten wurden die Untersuchungen auf die Ebene großer Bauteile hochskaliert: In Fichtenholzblöcke eingeklebte GFK-Stäbe, so genannte Glued in Rods (GiR), die meine vierte Publikation darstellen. Die Versuche bewiesen die allgemeine Anwendbarkeit der Härtungstechnik für große Verbindungen und ermöglichten auch die Validierung geeigneter Lösungen zur Temperaturmessung. Weiterhin konnte gezeigt werden, dass die Aushärtung mit CP zu Verbindungsfestigkeiten führt, die sich nicht von denen ungefüllter Referenzen unterscheiden, während die Aushärtungszeit erheblich verkürzt wurde.

Die Untersuchungen in den Veröffentlichungen 1–4 legten offen, dass die Beziehung zwischen komplexem Erwärmungsverhalten und dem Verlauf des Aushärtegrades ein wesentlicher Bestandteil zur Beschreibung des Prozesses ist. Bisher war es nur auf zwei Arten möglich, zu beurteilen, ob eine Verbindung dem EMF ausreichend ausgesetzt war, um vollständig auszuhärten oder nicht: Erstens durch die Vorhersage des Aushärtegrades auf der Grundlage der in meiner ersten Publikation entwickelten kinetischen Modelle und der während der Induktionsaushärtung einer entsprechenden Probe aufgezeichneten Temperaturdaten. Zweitens durch den Vergleich von experimentell ermittelten Verbindungsfestigkeiten an Versuchsreihen, die über unterschiedliche Zeiträume ausgehärtet wurden. Beide Möglichkeiten sind mit umfangreichen experimentellen Arbeiten verbunden und repräsentieren empirische Forschungsansätze. Um zu einer besseren Vorhersagbarkeit und Effizienz von CP-Härtungsprozessen beizutragen, habe ich daher ein numerisches Modell auf der Grundlage der Finite-Elemente-Methode (FEM) entwickelt. Diese Arbeit wurde in zwei Teile aufgeteilt, die meiner fünften und sechsten Veröffentlichung entsprechen. Ziel der Modellierung war die Vorhersage des Aushärteverhaltens von Klebstoffen anhand der in meiner vierten Publikation experimentell untersuchten GiR-Anwendung, um Anwender von umfangreichen experimentellen Ansätzen zu entlasten.

Für die Modellierung, die ich in meiner fünften Veröffentlichung vorstelle, wurden die in meiner ersten Veröffentlichung abgeleiteten kinetischen Modelle mit einer instationären Wärmestromsimulation in Ansys Academics 2019 R1® verknüpft, die sich auf validierte Wärmelasten stützt. Dadurch wurde es möglich, den Aushärtungsgrad in Abhängigkeit von der Aushärtungstemperatur und dem CP-Gehalt

numerisch vorherzusagen. Im Detail werden in der Publikation die experimentellen Vorarbeiten sowie alle für die Modellierung implementierten analytischen Methoden vorgestellt. Darüber hinaus wurde die prinzipielle Funktionsweise des Simulationsmodells anhand von Temperatur- und Aushärtungsgradprofilen illustriert. Der zweite Teil der numerischen Modellierung (sechste Veröffentlichung) führte zur Validierung der FEA, die dann zur Durchführung einer Reihe von Parameterstudien verwendet wurde, um die wichtigsten Einflussfaktoren zu identifizieren, die zum Erwärmungsverhalten von CP-gehärteten Verbindungen beitragen. Es hat sich gezeigt, dass unter anderem die Klebschichtdicke und der CP-Gehalt den größten Einfluss auf die entstehenden Aushärtungstemperaturen haben.

Anders als beim mechanischen Fügen sind beim Kleben bestimmte Mindesttemperaturen erforderlich, damit die Klebstoffe korrekt aushärten. Um diese Einschränkung zu umgehen, habe ich in meiner siebten Veröffentlichung die Verwendung der induktiven Erwärmung mit CP zur Aushärtung von Klebstoffen bei niedrigen Temperaturen validiert. Zu diesem Zweck wurden die in meiner vierten Veröffentlichung beschriebenen Versuche bei niedrigen Temperaturen (–10 °C und –30 °C) wiederholt. Die Ergebnisse zeigten, dass das Verfahren sehr gut geeignet ist, Klebstoffe in Temperaturbereichen auszuhärten, die in der Literatur noch nicht untersucht wurden, wobei das Bruchverhalten der Epoxidklebstoffe nicht von dem RT-gehärteter Referenzserien zu unterscheiden ist.

In meiner achten Veröffentlichung bin ich schließlich einen Schritt weiter in Richtung industrielle Anwendung der CP-Härtung gegangen. Es gelang mir, auf Basis aller vorheriger Veröffentlichungen ein zusätzliches beschleunigtes Aushärtungsszenario zu entwerfen, welches in Form von verschiedenen Prüfserien aus einfach überlappenden Zugscher-Rohrverbindungen aus glasfaserverstärktem Kunststoff (GFK) ausgeführt wurde. Durch diesen Ansatz konnte die Gültigkeit und Übertragbarkeit der bisherigen Ergebnisse erfolgreich nachgewiesen werden. Darüber hinaus verdeutlichten die Ergebnisse der Veröffentlichung, dass mit vergleichsweise wenigen CP eine deutliche Verkürzung der Aushärtezeit ohne Verschlechterung der Verbindungsfestigkeit erreicht werden kann.

Contents

List of abbreviations

Abbreviation	Meaning
2K	Two-component
AB	Adhesive bulk specimen
APDL	Ansys parametric design language
B-FRP	Basalt-fibre reinforced plastic / polymer
Cc / C-c	Cold cured at room temperature (23 ± 2 °C)
C-FRP	Carbon-fibre reinforced plastic / polymer
CP	Curie particles
DEA	Dielectric analysis
DLS	Double lap shear joint
DMA	Dynamic mechanical analysis
DSC	Differential scanning calorimetry
EaB	Elongation at break
EMF	Electromagnetic field
EPX	Epoxy adhesive
FE	Finite element
FEA	Finite element analysis
FEM	Finite element method
FRP	Fibre-reinforced plastic / polymer
FTIR	Fourier-transform infrared spectroscopy
G-FRP	Glass-fibre reinforced plastic / polymer
GiR	Glued-in Rod
GLT	Glued laminated timber
Ic / Ind-c	Inductively cured
MoE	Modulus of elasticity
Ov-c	Oven cured
PUR	Polyurethane adhesive
LVL	Laminated veneer lumber
Ref	Reference
RH	Relative humidity
RT	Room temperature (23 ± 2 °C)
SEM	Scanning electron microscopy
SLJ	Single lap shear joint
SLTJ	Single lap shear tubular joint
TDS	Technical data sheet
TGA	Thermogravimetric analysis
TS	Tensile strength
UTM	Universal testing machine
UV	Ultraviolet
XPS	X-ray photoelectron spectroscopy
vol-%	Volume percentage
w/w-%	Mass percentage

Part I: Introduction, state of the art, motivation, objectives & structure of the thesis

1 Introduction

Imagine a world in which a reliable method for accelerated curing of adhesives would have been available much earlier throughout human history. Would Hellenism have spread that far to the east if Darius disposed, at Gaugamela, of adhesively bonded scythed chariots built in large numbers at much faster pace? Would GENGHIS KHAN's Mongols have conquered an even larger empire thanks to much faster and cheaper production of their composite bows? Would wooden furniture of CHIPPENDALE not be considered as precious antiques, but rather be available in abundance nowadays? It can be assumed that some of these scenarios may have occurred while others may not – as History has not told us, we will never know for sure. However, what History has told us is that new technologies and innovations are always the consequence of social and economic circumstances, rearrangement of existing knowledge and, finally–yet importantly, new minds bringing fresh perspectives.

Although the origins of adhesive bonding technology reach far back into history of mankind[1,2], it remained for most of its time purely empiric. Analytical techniques for the characterisation of polymers such as dynamic mechanical analysis (DMA), differential scanning calorimetry (DSC) or thermogravimetric analyses (TGA) only became available in the course of the 20th century[3,4]. In principle, these measurement devices enabled scientists to analyse the relation between curing temperature and polymerisation speed[5,6]. However, the core topic of this thesis, the acceleration of polymeric cross-linking, was first not considered to be of primary interest – especially since the fundamental material behaviour of the comparatively novel material class of polymers was in the centre of research. In addition, corresponding advances in production technology were needed in order to get the most benefit out of the shortened curing cycles. For these reasons, initial attempts to accelerate polymerisation aimed at varying existing adhesive formulations, e.g. by adding catalysts. In this context, a remarkable achievement was the discovery of catalytically converted olefins, such as polyethylene or polypropylene, made by ZIEGLER as well as NATTA in 1953[7,8]. The so-called 'ZIEGLER-NATTA catalysts' paved the way for new application fields of polymers and gained ZIEGLER and NATTA the Nobel Prize for chemistry in 1963.

Figure 1: The first 'Talon Minute' Bar for quick repair of women's shoes using adhesive, Brussels 1957[9]

About the same time, in 1962, polymerisation speed was turned into a marketing argument, and the '*Fast-Curing Epoxy Adhesive*'[10] '*Minit-Cure*' promised to '*cure at room temperature in about 60 sec.*' , as shown in Figure 1. Obviously, slow curing was already regarded as a disadvantage. Since then, many different options to speed up polymeric cross-linking arose, resulting in the development of new curing techniques like e.g. microwave[11–17], induction[18–20], or radiation curing[21–23] as well as further advancements on the chemical level[24–26]. Among other aspects that were essential for this thesis, the essence of these techniques was sketched in the following sections.

2 State of the art

2.1 Adhesive bonding in civil engineering

As the accelerated curing experiments of the thesis were set in a timber-engineering context, the present section shortly adresses the associated challenges and conditions. In timber engineering, adhesive bonding has long been an established method for joining individual assemblies of structures. In recent years, research has concentrated not only on joining wood to wood but also with typical civil engineering materials such as steel[27,28], concrete[29,30], glass[31,32], or fibre-reinforced polymers (FRP)[33–35]. The bonded assemblies offer decisive advantages over other – mostly mechanical – joining techniques, such as a more homogeneous state of stress and joining of multi-material connections. As the topic of this thesis is the accelerated curing of structural adhesives, detailed information regarding adhesively bonded joints in general, for which dedicated literature is available[36], are not presented herein. In the following sections, two well-known civil engineering applications are presented, which served as exemplary components for the accelerated curing experiments.

2.1.1 Glued-in Rods

For many decades[37], a widely used connection type in timber engineering consists of rebars or threaded rods adhesively bonded into load-bearing wooden elements: Glued-in Rods (GiR). GiR have already been used in many different applications, such as beam-to-beam[38] or beam-to-column connections[39] as well as a reinforcement agent for timber structures[40]. Several recent publications detail their state-of-the-art[41,42], with two examples being illustrated in Figure 2.

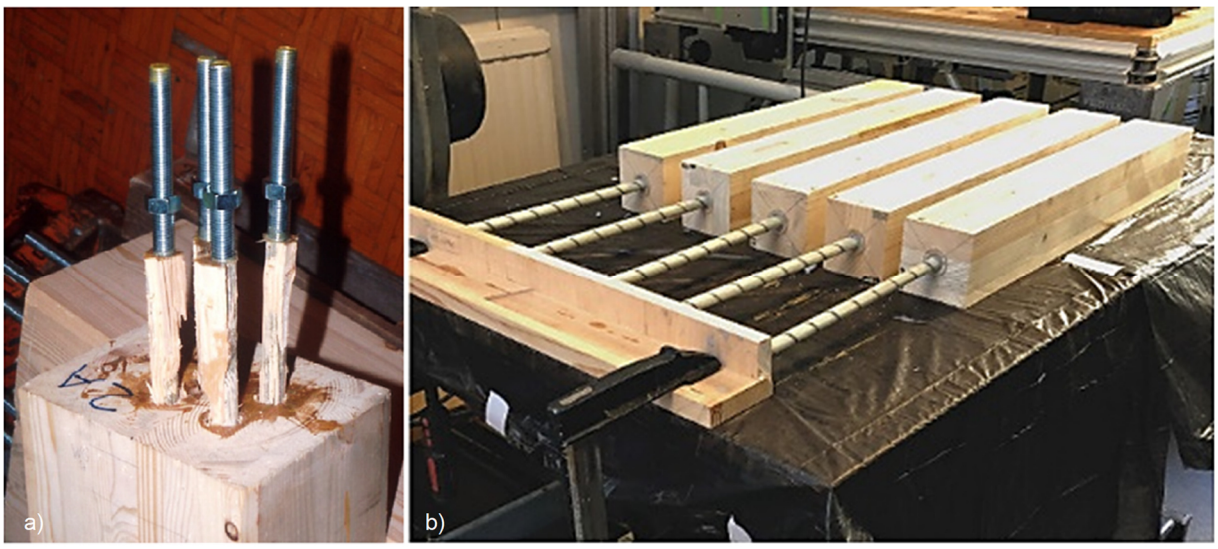


Figure 2: Exemplary Glued-in Rod (GiR) applications from literature herein produced with a) multiple threaded steel[43] and b) single G-FRP rods[44]

For a long time, research in the field of GiR focused on adhesive bonding of steel rods into wood. However, due to the increased availability of composite materials, rods made of FRP became an attractive

option for timber engineering[45–48]. FRP bars have excellent mechanical properties while exhibiting a very good resistance against thermal influences[49], corrosion[50] and chemical media[51]. Different types of FRP rods reinforced by fibres made of carbon (C-FRP)[52], glass (G-FRP)[40] or basalt (B-FRP)[53], are currently available on the market, and have already been used for GiR applications.

Many researchers investigated the mechanical behaviour of G-FRP rods bonded into wood under different load cases experimentally[38,54] and numerically[55,56]. Recently, TITIRLA et al.[57] investigated the influence of rod material (B-FRP and G-FRP), rod diameter, as well as the direction of wood grain, on the pull-out load capacity of GiR. They found that all parameters have a significant influence on failure loads, and on observed fracture patterns. RAFTERY et al.[40] investigated the reinforcement of low-grade glulam timber by bonded-in G-FRP rods and found that an enhanced stiffness and ultimate moment capacity of the connection can be achieved. In contrast, MADHOUSHI et al.[38] reported a decrease in static and fatigue strength of in-line beam-to-beam connections of pultruded G-FRP rods bonded into laminated veneer lumber (LVL) with epoxy resin.

The adhesives typically used for the bonding of GiR are usually high-strength thermosets, mostly two-component (2K) polyurethanes (2K-PUR) and epoxy resins (2K-EPX)[46]. Although adhesives uphold a very high potential in timber engineering[58], the actual bonding process is very time-consuming, as the usually used thermosets require several hours to days for curing under construction or production conditions. Therefore, components must be fixed, and cannot be moved for further processing, until sufficient handling strength has been achieved. In addition, full loading can only be applied once a high degree of cure, α, is reached. A reliable method for accelerated curing could minimise these restrictions for practitioners, and develop the bonding process towards shorter process times, including on site. In addition, the glass transition temperature, T_g, of RT-cured adhesives (typically in the range of 50–65 °C[59]) lies below the maximum temperatures to account for on site, +67 °C, as prescribed by relevant codes and standards[60] — even without fire exposure.

2.1.2 Single lap shear tubular joints

FRP are on the market for decades[61,62]. Nowadays, they are used in almost all industries where low-weight, high performance, and good durability are required, as for example in the automotive[63,64], aeronautical[65] or aerospace industry[66] etc. In recent decades, FRP components have also been considered for civil engineering applications, either to structurally reinforce existing structures[67], or to design them from scratch[68,69]. Unlike in other industries, where the gain in component weight and strength usually outbalances the higher material costs, civil engineering applications require much more economical composites — save for retrofitting, strengthening and rehabilitation, where urgency justifies the use of materials as carbon. Accordingly, most of the composites used in civil engineering are of pultruded shapes. More detailed information about the pultrusion process can be found in the state of the art of my 8th publication.

Although joining of fibrous composite materials is in principle possible by means of mechanical fasteners, for which a huge corpus of dedicated literature exists[70–74], it is generally agreed upon that adhesive bonding is by far a much more material adapted joining process[75–79]. For the sake of completeness it is reminded that hybrid joints, in which mechanical fasteners are combined with adhesive bonding, aim at combining the best of the two joining worlds[80–82].

The vast majority of experimental and numerical work related to adhesively bonded FRP joints has been carried out on the arguably simplest forms of bonded joints, either single lap shear (SLJ) or double lap shear joints (DLJ), for which this thesis does not ambition to give an overview. At this point, it will suffice to remind that failure of adhesively bonded SLJ is highly dependent upon sharp stress peaks generated at the ends of the overlaps[83,84]. In addition, load capacity increases with the overlap length[85,86] and transverse tensile strength of the laminate is at least as significant as its shear strength[87]. For much more details regarding the mechanics of bonded joints in general, the material properties that influence joint capacity, and methods of predicting thereof – in particular for pultruded G-FRP – dedicated literature is available[84,88,89].

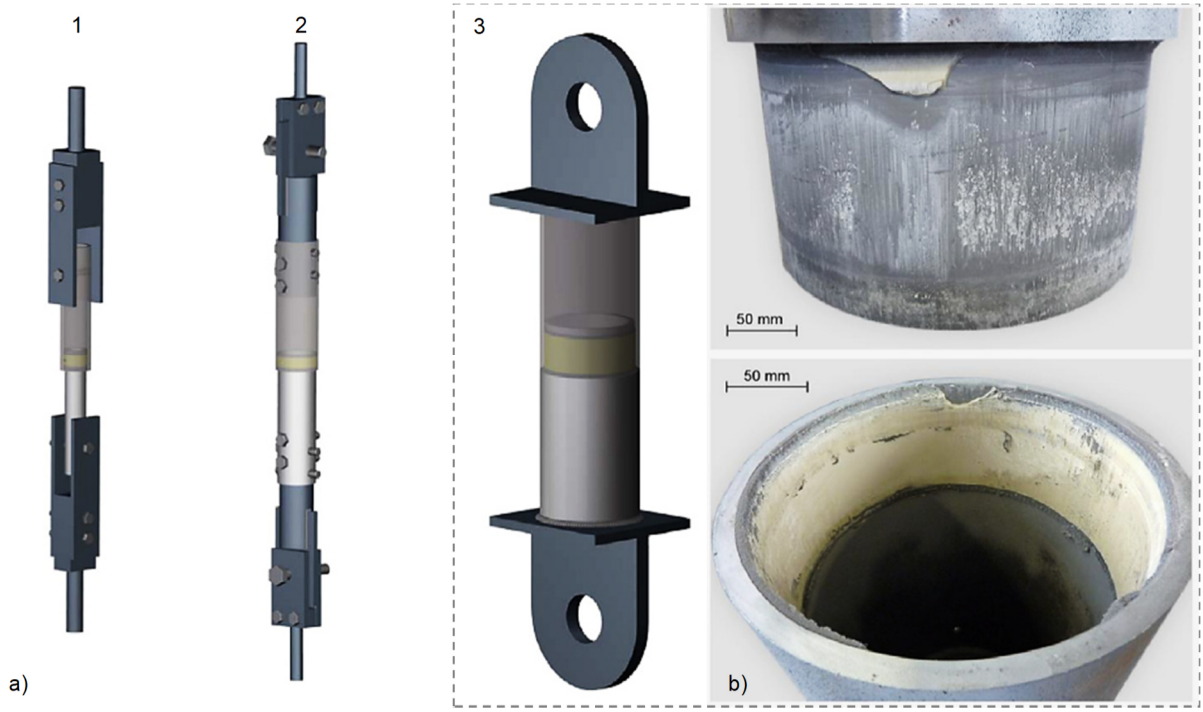


Figure 3: Example for an adhesively bonded single lap shear tubular joint (SLTJ) from literature[90] (herein made of steel) with a) different test configurations (1–3) and b) exemplary fracture pattern for test configuration 3

Besides SLJ and DLJ, adhesively bonded single lap shear tubular joints (SLTJ), which were used as an exemplary joint type for the accelerated curing experiments of this thesis, were less studied. An example of a SLTJ can be seen in Figure 3. The review article of PARASHAR & MERTINY'S[91] is a valuable resource that compiles many references. Amongst those who investigated their behaviour are ADAMS & PEPIATT[92] as well as LEE & LEE[93] whom developed a failure model for adhesively bonded SLTJ and subsequently presented an optimal design thereof[94]. In addition, CHOI et al.[95] considered not only tubular single but also double lap joints. ALBIEZ et al.[90,96] investigated adhesively bonded joints composed

of steel at both the experimental and numerical level and highlighted some of the dominant parameters influencing joint strength, as well as latter's robustness of the technique with regard to geometrical imperfections. These studies show that SLTJ represent a frequently used connection type with practical relevance.

2.2 Accelerated curing of polymers

While the sole focus on faster manufacturing processes is certainly an advantage in many industrial sectors, it falls far shortened of the mark in light of the manifold possibilities offered by modern adhesive bonding technology. Without any claim to completeness, the following positive effects may arise for fast curing:

- Exploitation of new application fields for adhesively bonded joints.
- Greater variety of design for bonding processes.
- Faster manufacturing processes and shorter cycle times leading to time and money savings.
- Superior adhesive properties such as T_g or bond strength (both adhesive as well as cohesive).
- Improved resistance to fatigue, moisture diffusion and elevated application temperatures.
- Manufacturing independent of ambient temperatures.

The choice of a reliable method for accelerated curing is largely determined by the manufacturing process, component geometry and the type of adhesive. Many of the available options foot upon heat generation within the polymer to be cured. During this process, the curing kinetics plays a decisive role, as some adhesives react poorly to heat supply, while others react well. To provide a better overview of the existing approaches, the most common methods have been summarised in Figure 4.

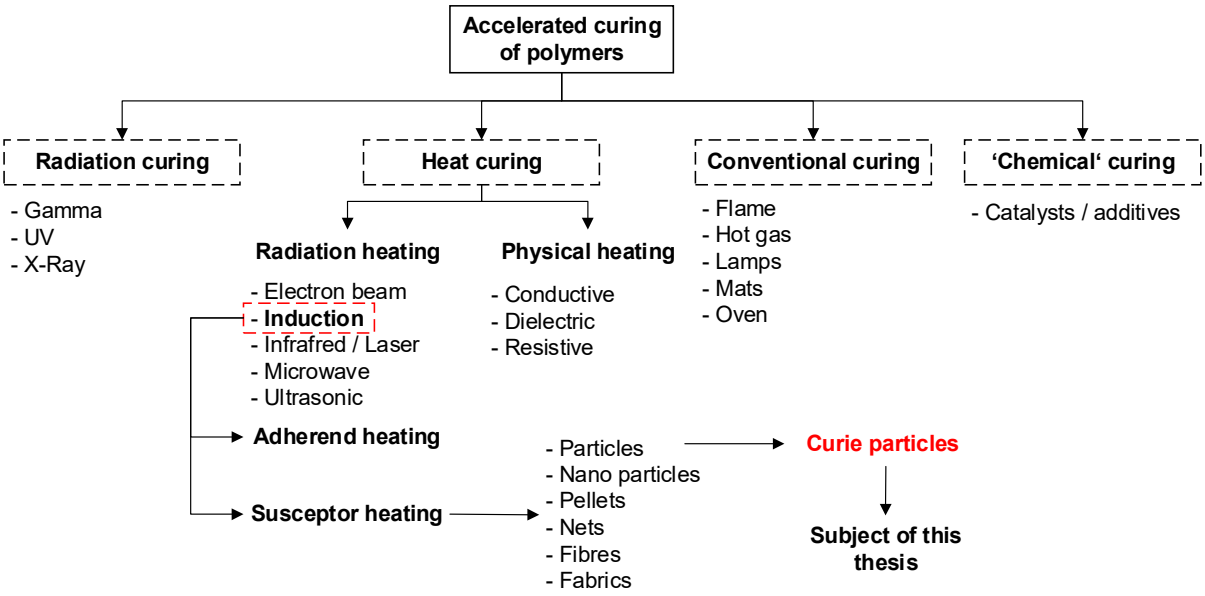


Figure 4: Overview of existing approaches for accelerated curing of polymers including disclosure of the thesis' subject (own representation)

Although there is planty of literature available on accelerated curing processes, there are no clear guidelines or standards for their implementation as for adhesive bonding in general[97]. For practitioners, this results in a relatively confusing situation as process conditions to be considered during practical application are usually not properly worked out. In addition, some of the available techniques are much better studied than others: For UV-curing adhesives, several review articles can be found[98–101], whereas for induction[102], microwave[103] or resistive curing there are hardly any available. Furthermore, these review articles often focus on accelerated manufacturing of FRP rather than on fast curing in general.

At this point, I do not want to dive deeper into the literature for the numerous methods available for accelerated curing, as it would only repeat the detailed survey within each of my publications. As this thesis focuses on a particle-induced accelerated curing process using heat generated by electromagnetic fields (EMF), the following sections highlight the developments in this particular field of research and draw attention to the basic ideas that lie underneath this thesis.

2.3 Curing of polymers by inductive means

2.3.1 Manufacturing concepts s & history

How can induction heating be used to accelerate the curing of adhesives? On the one hand, heat may be introduced from the outside of the bond by heating the adherend(s), which in turn transmits the heat to the adhesive by thermal conduction. On the other hand, heat can directly be brougth into the adhesive, which is achieved by adding EMF-sensitive materials to the polymer to be cured. Such added materials are called susceptors[1]. For a better understanding, I have illustrated these two inductive heating principles in Figure 5. The choice about which setup to be used is thus determined by the EMF-sensitivity of the parts to be joined.

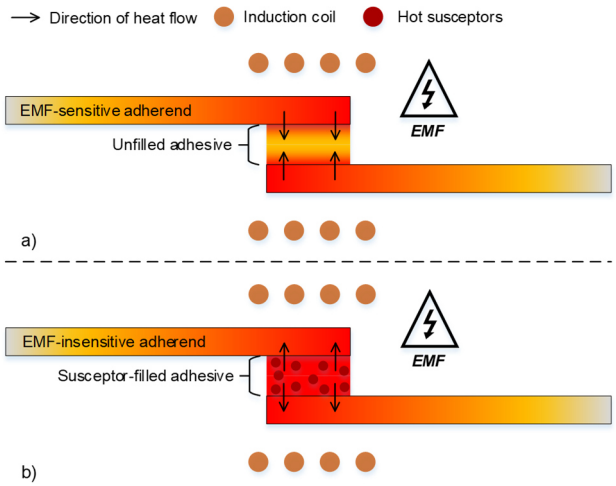


Figure 5: Existing approaches for accelerated curing by inductive means using SLJ as an exemplary connection, drawings not to scale; a) Indirect heating of adhesive over adherend material, b) direct heating of adhesive through addition of EMF-sensitive susceptors (own representation)

[1] Latin: – '*suscipo*' – to take upon / over.

Now that the basic approaches of induction curing have been outlined, I would like to take a step back in time and explain the origins of these techniques. Without claiming to be completely exhaustive, first records of induction curing date back to the 1980's, when NASA[104,105] reported on the development of new '*Rapid Adhesive Bonding concepts*', through which '*bonding times for specimens can be cut by a factor of 10 to 100 compared to standard press or autoclave bonding*'[106]. With a few isolated exceptions[107,108], research in the field remained quiet for about 10–15 years, perhaps because UV-curable adhesives experienced a boom during the 1980's and the 1990's, as numerous studies from this period indicate[109–112]. Around the turn of the millennium, the number of publications dealing with induction curing picked up again, with the process being applied both to FRP with[113,114] and without[20,115] susceptors, as well as to adhesives filled with metal meshes, fibres or particles[116,117]. Due to changing material- or geometry-related properties, the available susceptor classes have various advantages and disadvantages, some of which I have described in my publications. Since I focused on the bonding-related aspects of a particle-induced curing technique, I would like to elaborate on the current state of the art in this field – also to make it easier to understand why my results have significantly enriched it.

2.3.2 Induction curing with particles

By reviewing the existing literature, it quickly becomes clear that the field of particle-induced curing by induction is yet quite open, and very young—with most of the available literature originating from the current decade. The studies mostly focus on single aspects of the complex topic. Examples are the works of BAE et al.[118,119] and MAURER & LAMMEL[120], who almost exclusively investigated locally recorded heating behaviour of particle-cured adhesives using different filler types, among others magnetite, iron or SiO_2-coated iron-oxide (formally known under its market name Magsilica©). Other authors extend their heating-related investigations to selected mechanical aspects, such as lap shear[121–126] or peel strength[127], T_g[128,129] as well as stiffness and tensile strength[126,128]. Unfortunately, systematic experimental campaigns, including extensive mechanical testing on the level of joints, are missing. The mechanical aspects are only reported, but not discussed in the broader context of thermo-mechanical adhesive properties. In addition to the aforementioned studies, there is a large number of works that – detached from accelerated curing – attempt to influence material properties of polymers by adding fillers, which I detailed in the original article of my 2nd publication.

In 2017, SEVERIJNS et al.[124] investigated accelerated curing of a 2K epoxy adhesive by inductive heating of admixed iron particles considering volume contents from 0.5 to 7.5 vol-%. The heating behaviour during inductive heating of G-FRP SLJ was monitored using a thermocouple and an IR camera. Even for the lowest Fe content investigated (0.5 vol-%), inductively cured specimens achieved lap shear strengths 15–20 % lower in comparison to cold cured reference sets. Curing of SLJ by electromagnetic induction was also studied by VATTATHURVALAPPIL & HAQ[126] using ferromagnetic nanoparticles (FMNP), which were added to a thermoplastic adhesive (ABS) with FMNP contents ranging from 4–20 w/w-%. Punctual adhesive temperatures during inductive heating of SLJ made of G-FRP were monitored using a fibre optic sensor embedded directly inside the polymer, and resulting thermo-mechanical

properties were analysed. It was found that ABS-filled SLJ with 16 w/w-% FMNP showed a good balance between stiffness and strength relative to other FMNP contents, and inductively cured bonds achieved 15 % higher peak loads in comparison to joints cured in the oven. These results proved the functionality of particle-induced accelerated curing by electromagnetic induction. However, in [124] (Fe particles, T_c = 768 °C) and in [126] (Fe_3O_4 particles, T_c = 578 °C) – along with other studies[123,130] – particles were used which can lead to irreversible adhesive damage and consequently deterioration of mechanical properties even if a tight control of induction power is adhered, as described in more detail further below.

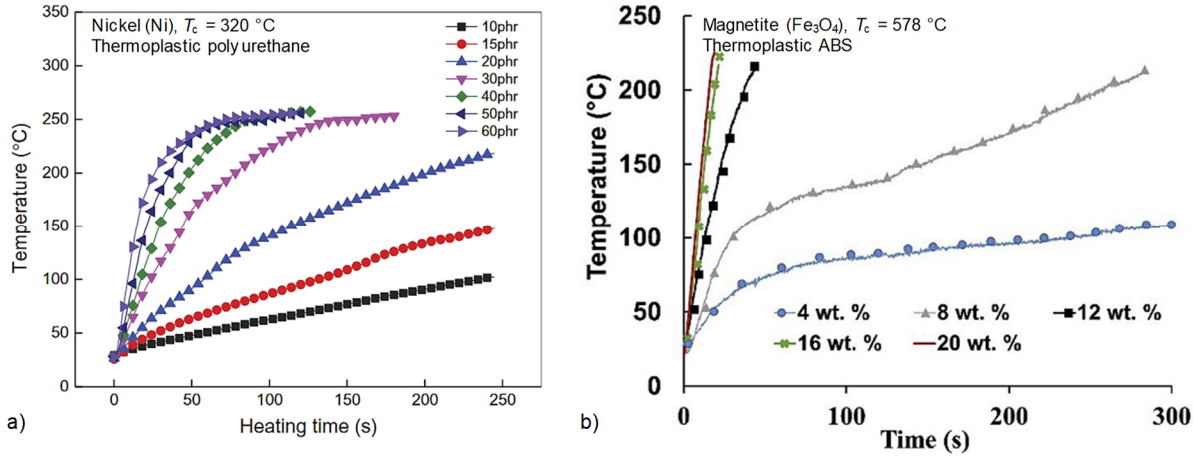


Figure 6: Exemplary parameter studies of a) LM[131] and b) VATTATHURVALAPPIL & HAQ[126] both published in 2019; Punctual temperature development for different contents of inductively heated a) nickel and b) magnetite particles

Although each of aforementioned studies dealt with induction heating, experiments were mostly presented as parameter studies, e.g. for particle type[121,123,128], content[124–127] or size[118,119,124,125], with two examples for different particle contents illustrated in Figure 6. On this basis, it is not possible to reasonably work out essential process conditions such as those needed to achieve homogeneous heating of particle-cured adhesives. A good example represents the role of polymerisation exothermy, which – as I have demonstrated in my publications – has a fundamental impact on the heating behaviour. While this topic has proven to be of fundamental importance, only one (short) article of MAURER & LAMMEL[120] addresses the subject of exothermy without revealing deep insights.

The literature survey points out that particle curing was mostly investigated at the level of bulk adhesives[118–120,129] or relatively basic standardised SLJ[126,128], restraining the research to a fundamental level. Exceptions of this represent the works of ADAM et al.[132] and VALLÉE & ADAM[130], both published in 2016, who investigated the heating behaviour and tensile strengths of small-scale G-FRP rods bonded into wood. However, both studies used magnetite (Fe_3O_4) as filler material, which may be heated very fast to a temperature range not compatible for adhesives, risking thermal degradation of the polymer. Therefore, induction power, and thus heat development, had to be controlled by means of thermocouples placed inside the considered joints as illustrated in Figure 7-a. Aforementioned problematic highlights the need for a simple and user-friendly method to control arising temperatures during particle-induced accelerated curing. The intention behind this thesis was thus to significantly enhance the state of

the art in the field by investigating self-regulated heating with inductively heated Curie particles (CP) on various dimensional joint scales both on the experimental as well as on the numerical level.

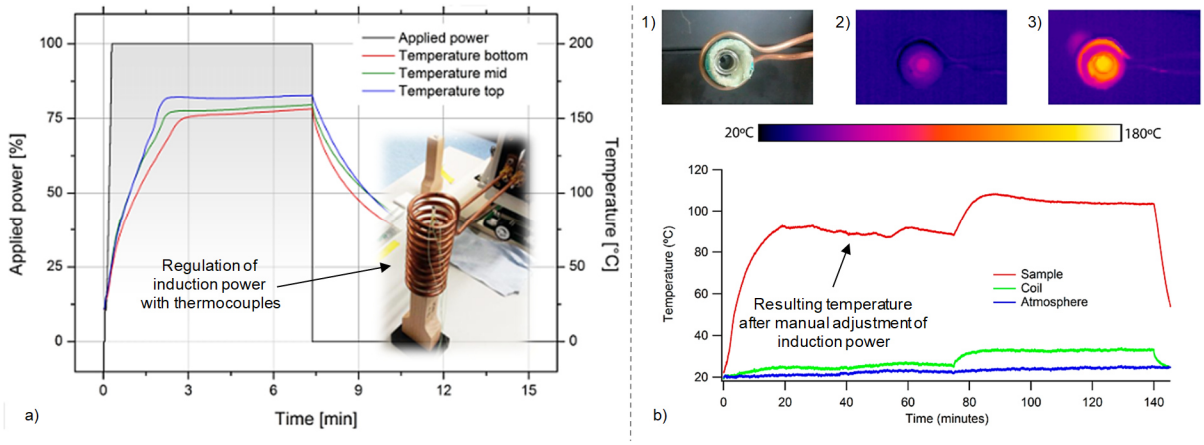


Figure 7: Examples of induction curing from literature using a) magnetite (Fe_3O_4, T_c = 578 °C) and b) iron oxide (e.g. Fe_2O_3, T_c = 585–675 °C) particles, for which curing temperatures have to be controlled by regulating induction power over a) embedded thermocouples[130] or b) manual adjustment[129]

2.3.3 Induction curing with Curie particles

The basic principle of Curie particles (CP) is to make use of the materials Curie temperature, T_c, discovered in 1894 by CURIE[133]. Without immersing too deeply into the physical principles, it is important to keep in mind that ferro- or ferrimagnetic materials carry intrinsic magnetic moments[134]. These moments may cancel each other out, if all electrons within the considered material are paired, so that the material has no spontaneous external magnetisation – the material is paramagnetic. In ferromagnetic materials, such as iron or nickel, the magnetic moments are aligned parallel so to exhibit a relatively strong magnetisation. Ferrimagnetic materials, such as magnetite or ferrites, also experience an external magnetisation, however, it is less pronounced, as the magnetic moments are aligned antiparallel and thus partly cancel each other out. Aforementioned relationships have been schematically illustrated in Figure 8-left.

If a ferro- or ferrimagnetic material is exposed to an external EMF, heat is generated within the material due to different physical effects; for particle-induced curing mainly as a consequence of hysteresis losses[135]. During the process, heat generation continues until the material temperature reaches T_c. At this point, the magnetic moments within the material lose their order, and the material becomes paramagnetic. In other words, beyond T_c, such materials do no longer react to the applied EMF, and no further heat is developed. Because of this self-regulated heating, no external temperature control is required during the curing process. Figure 8-right shows a typical temperature curve resulting from aforedescribed relationships.

I do not want to conceal that the Curie-principle has been known for quite some time in context of accelerated adhesive bonding. In addition to surgery, radiation and chemotherapy, the Curie effect also

established a separate method for treating cancer, the so-called hyperthermia[136]. Coming back to adhesive bonding, WETZEL & FINK[113] tried, in 2001, to utilise CP for accelerated and controlled curing of FRP. However, they initially focused on the selection of suitable particle types showing adequate heat generation rather than on the bonding-related aspects of the topic. They concluded that soft magnetic ferrites with sizes ranging from 1–10 µm may achieve sufficiently high heating rates if exposed to EMF's at frequencies between 100 kHz – 10 MHz. One year later, WETZEL et al.[116] extended their findings towards induction bonding of structural composite tubes using a 2K urethane elastomer. Their investigations aimed at identifying optimal conditions for their tube application, i.e. coil geometry, induction parameters etc., all of which determined by extensive preliminary investigations. Although suitable conditions were identified, and functionality was proven, final bond strength was not assessed by mechanical tests, and therefore disclosure of process-related risks and challenges was not realised. WETZEL'S study is a valuable source and a good example of an early attempt to implement CP-curing on an industrial scale. However, the findings are presented essentially in the form of extensive, application-specific 'trial-and-error' experiments. This was presumably due to the absence of appropriate numerical approaches, highlighting the need for further investigations aiming to establish more practitioner-friendly implementation of CP-curing processes.

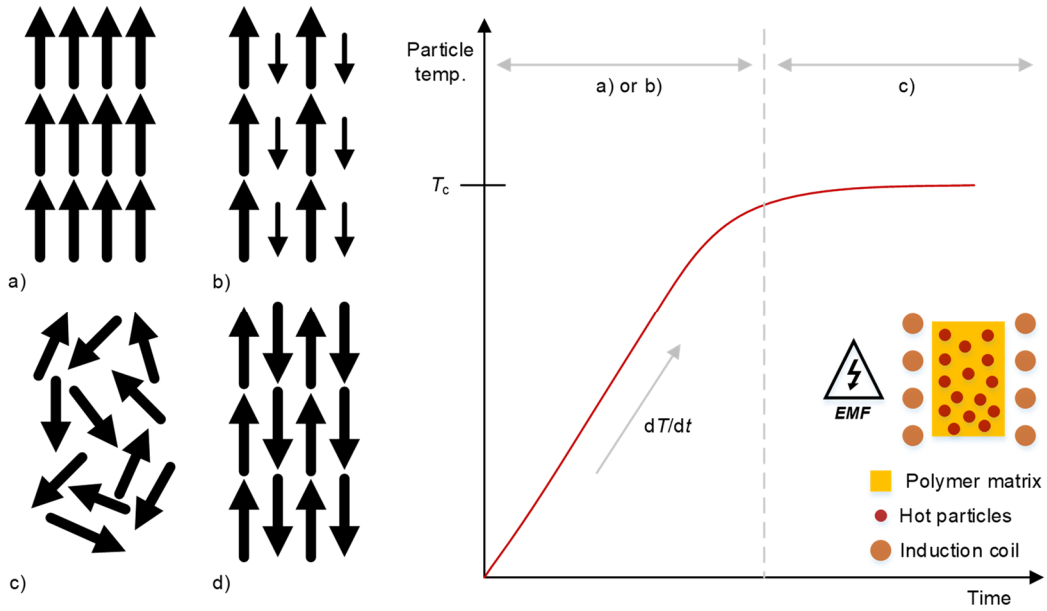


Figure 8: Left: Illustration of magnetic moment orientations in a) ferromagnetic, b) ferrimagnetic, c) paramagnetic and d) antiferromagnetic material, arrow thickness indicates strength of contribution; Right: Typical 'Curie-heating curve' for ferro- or ferrimagnetic material exposed to an alternating EMF using the exemplary application of particles embedded in a fully cured polymer (own representation)

As already described throughout the previous sections, the concept of WETZEL et al. was surprisingly not taken up during the following years. Thus, many of the available studies ignore the fact that used particles can be heated significantly above acceptable curing temperatures of polymers (~80–120 °C, for 2K adhesives, ~150–200 °C, for 1K adhesives) if tight control of induction power is disregarded. These include e.g. magnetite (Fe_3O_4, T_c = 576 °C[119,120,126,130,132]), iron (Fe, T_c = 770 °C[118,120,129]), nickel (Ni, T_c = 358 °C[127,137]) or ferrites of different types (T_c = 60-460 °C, depending on composition[121]). Thus,

ambitious efforts to control adhesive temperatures via embedded particles are predominantely missing. As already described in section 2.3.3, some researchers tried to control bond temperatures by using external monitoring techniques and adjustment of induction power[121,129,130] to prevent overheating. However, this involves additional experimental effort and does not prevent local adhesive damages, as measured temperatures always represent temperatures of the adhesive-particle mix and not the susceptors temperature. Moreover, the approach represents no satisfactory solution for practical application, as it entails additional technical as well as human resources.

The outlined circumstances reveal the strong need for an in-depth analysation, which considers all aspects of CP-curing. In the first step, this goal can be achieved by selection of a suitable CP class and associated material properties. To achieve this, it is possible to adapt a materials T_c to the process requirements by means of various techniques[138–141]. In addition, different approaches exist to vary shape, size or surface properties – keyword coatings – of particles.

2.3.4 Numerical modelling of particle-induced temperatures within adhesives

Apart from the experimental shortcomings concerning utilised particles and particle-induced temperature control, there are practically no numerical techniques available in literature that may contribute to a more predictable and efficient production planning of particulate curing processes[1]. Only SEVERIJNS et al.[124], in 2017, numerically investigated the heating behaviour of iron particles embedded within a fully cured 2K structural epoxy at the level of the adhesive bulk. The authors utilised the JILES-ATHERTON theory[142] to describe EMF-generated hysteresis losses within the iron particles, with the heating behaviour being significantly better reproduced when hysteresis heating was considered in the simulations, as shown in Figure 9-b. Overall, however, numerically calculated temperatures were about 15 % lower if compared to those recorded during the experiment, which was attributed to material properties taken from literature, and not experimentally determined.

While the approach by SEVERIJNS et al.[124] represents an important contribution, it still lacks the embedment in a much larger conceptual framework, e.g. by integrating the adhesives curing kinetics on the numerical level. The latter is, however, essential in order to answer application-related questions, such as the time needed to achieve full adhesive cure. For that, novel numerical methods have to be developed, which combine the individual physical processes, i.e. magnetic field, heat development, curing kinetics and mechanical aspects, and implement them on the level of whole joints. A first approach of such a model was developed and presented in my 5th and 6th publication, which opened up the way for a much more predictable design of induction curing with CP as well as to identify conditions important for practical implementation.

[1] There are also very few numerical models for induction curing of unfilled adhesives using adherend heating available (cf. principle illustrated in Figure 5-a). The existing approaches are discussed more deeply in the original articles of my 5th and 6th publication.

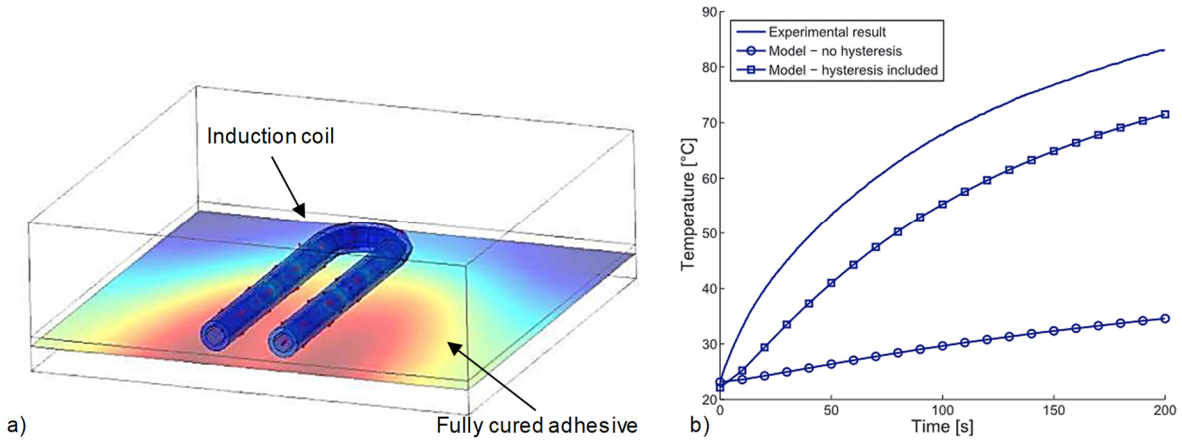


Figure 9: Numerical model for mapping particle-induced temperatures by electromagnetic induction (herein Fe particles) within a fully cured 2K structural epoxy adhesive of SEVERIJNS et al. from 2017; a) model geometry and b) comparison between numerically calculated temperatures in comparison to experimental results

2.4 Influences of particles on adhesives and adhesively bonded connections

Detached from particle-induced curing by electromagnetic induction, efforts have been devoted by different researchers to characterise the influence of filler materials, including particles, on adhesive and joint properties. Depending on the application, different objectives were pursued, which mostly aimed to improve resulting adhesive properties after complete cure, such as strength[143,144], stiffness[145], toughness[146–148] and thermal conductivity[149,150], or even to implement specific properties like electrical conductivity[151–157]. However, even if particle addition lead to the sought additional properties of the adhesive, the latter always represent anomalies, which can have a fundamental impact on other essential adhesive properties, in particular cohesive[158] or adhesive[126] strength.

In case of CP-induced accelerated curing by electromagnetic induction, the scientific problem is even more complex as used particles must fulfil various conditions. At first, a good EMF-sensitivity is needed in order to guarantee sufficiently high heating rates. Then, however, heating rates shall not be too high in order to prevent the adhesive from overheating, thus local damages. In addition, complex influences on the mechanical adhesive and joint properties, most significantly strength and stiffness, have to be considered, as outlined in the following two sections. Aforedescribed as well as further aspects to be considered when accelerated curing with particles is targeted have been illustrated in Figure 10. For CP-cured adhesives, the effects on mechanical and thermo-mechanical adhesive and joint properties have never been clearly summarised and quantified using systematic experimental campaigns. Therefore, one of the objectives of this thesis was to fill this knowledge gap.

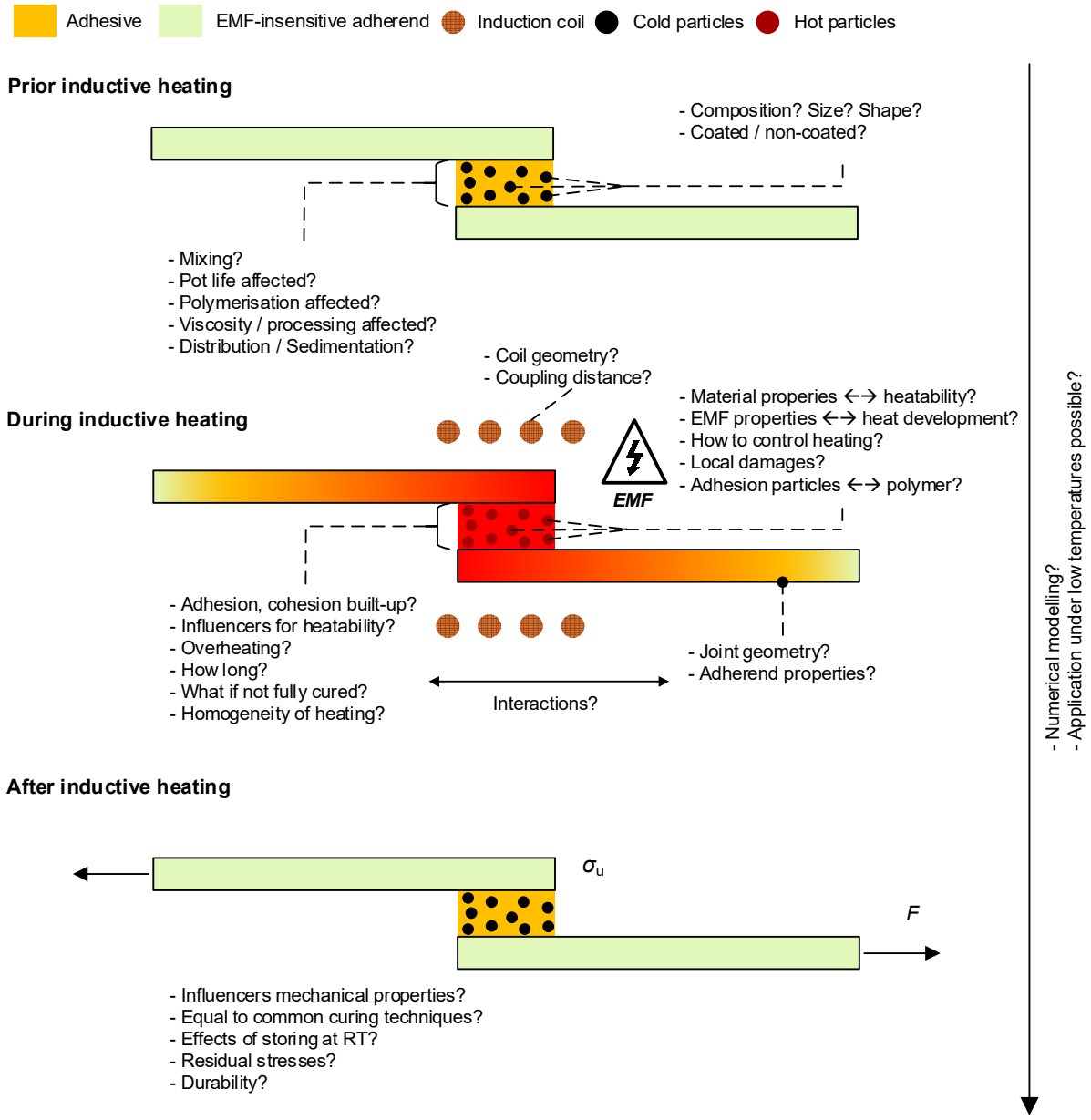


Figure 10: Scientific complexity and relationships to be considered during particle-induced accelerated curing with CP (own representation)

2.4.1 Influences of particles on bulk characteristics

BANEA et al.[159] investigated the effects of different contents of thermally expandable particles (TEPs, Ø10–16 µm, added to allow for subsequent debonding) on adhesive bulk properties of a 2K structural polyurethane adhesive. They found that achieved fracture toughness of TEP-modified specimens increased, while a significant decrease in tensile strength was observed (cf. Figure 11); both in comparison to specimens bonded with unfilled adhesive. In contrast, PARK et al.[160] used carbon black nanoparticles to improve the mechanical properties of an epoxy adhesive with particle contents ranging from 0.5–3.0 w/w-%. Best results were obtained for a particle content of 1.5 w/w-%, for which lap shear strength increased by 48 % and tensile strength by 22 %, if compared to unfilled reference samples. In 2014, the effects of adding carbon nanotubes (CNT) as reinforcing agent for two epoxy adhesives were pointed

out by WERNIK & MEGUID[161]. Experiments were carried out with four different specimen types, whereby the adhesive filled with CNT and polyvinylpyrrolidone (PVP) resulted in an increase of tensile strength by 90 %, compared to the adhesive only filled with CNT.

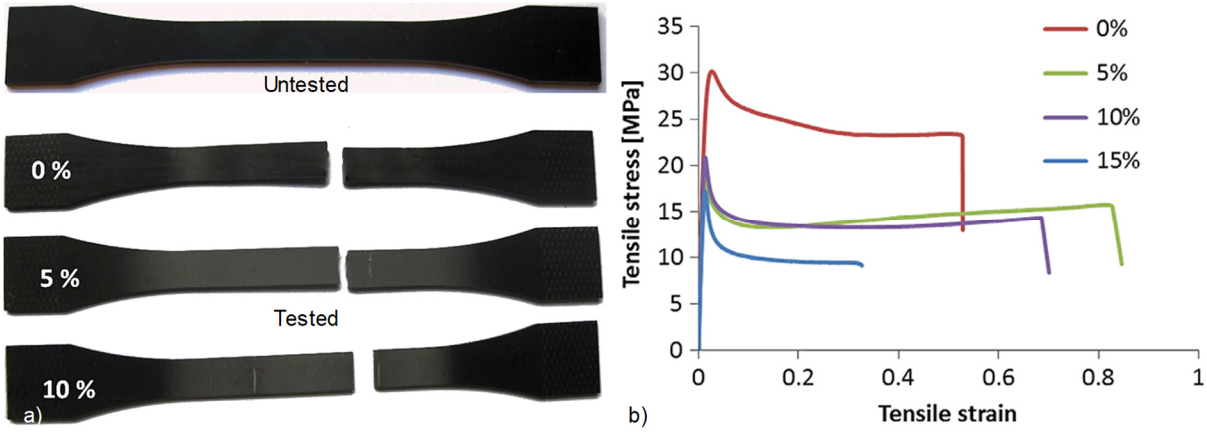


Figure 11: Mechanical results of BANEA et al.[159] from 2014 for RT-cured 2K structural polyurethane filled with different contents of thermally expandable particles (TEP's); a) Produced dog-bone specimens acc. to DIN EN ISO 527[162] and b) resulting stress-strain curves after mechanical testing at RT and v = 1 mm/min in function of TEP content

Aforementioned results make clear that particles have a decisive influence on resulting adhesive properties, which have to be considered when they are mixed in. The influences of CP-induced accelerated curing on bulk adhesive characteristics have been investigated in my 2nd publication.

2.4.2 Influences of particles on compound characteristics

Besides changes in bulk adhesive characteristics, added particles can have a significant impact on resulting joint properties as already shown in several studies for common RT-curing[163–165]. However, the identification of effects originating from added particles on adhesively bonded joints turns out to be complex, as several simultaneously interacting phenomena have to be considered. On the one hand, intrinsic adhesive properties (in particular strength and stiffness) are significantly changed depending on material type and composition of admixed particles[158,166]. On the other hand, particles may influence adhesion performance on the considered substrate[167], which additionally depends on adhesive type[168], joint geometry[169], surface pre-treatment of the substrate[170], the curing conditions[171] as well as the type of applied load[172].

The vast majority of studies investigated the effects of adding particles at the level of RT-cured SLJ. In 2018, QUAN et al.[173] added graphene nanoplatelets (GNP) to a rubber-modified epoxy adhesive in order to improve resulting fracture toughness and lap shear strength. They observed an increase in fracture energy by 21 % after adding 0.1 w/w-% of GNP, however, a gradual decrease of achieved lap shear strength from 21.7 MPa (unfilled reference) to 17.2 MPa after increasing the GNP content from 0.1 to 0.5 w/w-% was discovered. Recently, AKHAVAN-SAFAR et al.[172] highlighted influences resulting from adding micro cork particles to an epoxy adhesive. Experiments were carried out on SLJ subjected to quasi-static loading, at –20 and +75 °C, as well as fatigue loading at RT. The authors reported an increase

in lap shear strength at –20 °C after adding 1 vol-% of micro cork particles (cf. Figure 12-b), as well as an up to 4.6 times better performance under fatigue loading, all in comparison to reference sets.

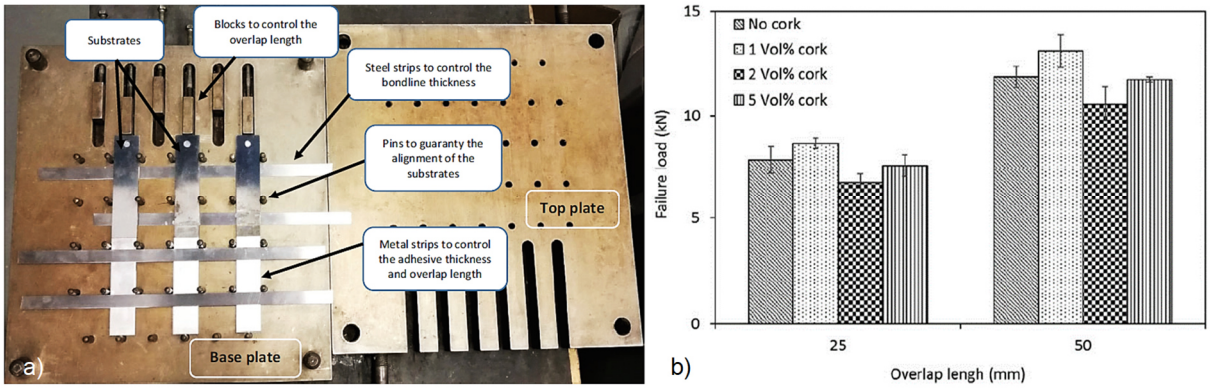


Figure 12: Mechanical results of AKHAVAN-SAFAR et al.[172] from 2020 for SLJ bonded with an RT-cured 2K epoxy adhesive filled with different contents of micro cork particles (Ø125–250 µm); a) Utilised mould for joint manufacturing and b) resulting failure loads in function of cork content and overlap length after testing at –20 °C and v = 1 mm/min

Aforementioned results make clear that even if curing of particle-filled adhesives is performed under the manufacturer's recommended conditions, addition of particles can lead to significant changes of resulting compound properties. It is thus of significant importance to consider not only influences caused by the mere addition of particles but also effects originating from curing at elevated temperatures when inductively heated particles are used to achieve faster curing. These relationships have been investigated in my 3rd publication.

2.5 Curing kinetics and modelling

2.5.1 Aims & structuring

In this thesis, curing kinetics and modelling was combined with accelerated curing by inductively heated CP for the first time, both on the experimental (e.g. 1st publication) as well as on the numerical level (e.g. 5th publication). The following sections are thus intended to provide a general overview in the field. Kinetic modelling, i.e. the determination of the relationship between time, temperature and level of adhesive cure has been extensively investigated in the past by many researchers[174–177] and addressed different classes of adhesives, such as e.g. epoxies[178,179], polyurethanes[180,181] or silicones[182,183]. A detailed overview of existing modelling approaches has been given by YOUSEFI et al.[184]. In order to understand the polymerisation mechanisms, two main types of models can be distinguished in literature: mechanistic[185] and phenomenological models[186], as principally illustrated in Figure 13.

Mechanistic models rely upon some presupposed model and reaction process, derive curing kinetics *ab initio* and can thus be performed at the molecular level[187], based upon mathematical models[188], or any other. They set a strong focus on qualitative understanding the process rather than on a precise quantification. However, reaction mechanisms of polymers may become complex, with several superimposed kinetic equations at a time. Consequently, such approach often requires detailed measurements of initial,

and intermediate, reactant's concentrations for validation, and are therefore more complex than phenomenological models[189]. Exemplary studies of aforementioned mechanistic modelling approach represent the works of e.g. MIJOVIC et al.[190], BLANCO et al.[191] or AMIROVA et al.[192]

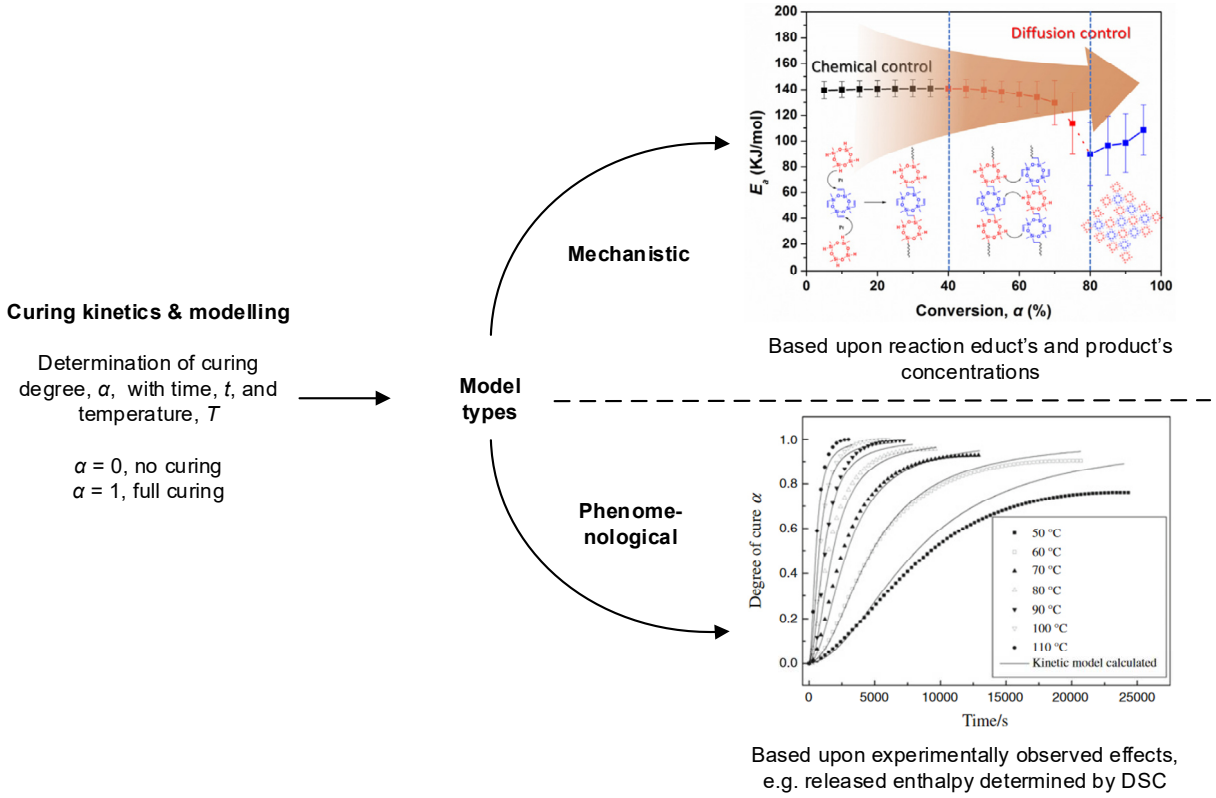


Figure 13: Principal illustration of available model types for kinetic modelling (own representation filled with exemplary graphs from [193], top, and [186], bottom)

At the opposite side, phenomenological models focus on the quantitative description of experimentally determined metrics[194], such as enthalpy change or mass loss, which are related to curing over time, without necessarily understanding them. Subsequently, the obtained data is fitted by an appropriate equation, through which the curing progress can be described as a function of temperature[189]. Due to their way simpler applicability, phenomenological models were frequently used for the description of adhesive cure, whereby determination of aforementioned metrics is in the centre of attention for the kinetic modelling. These can be derived by different techniques such as Dielectric Analysis (DEA)[195–198], Differential Scanning Calorimetry (DSC)[176,179,199–201], Fourier-transform infrared (FTIR)[202–204], Infrared[205] or Raman Spectroscopy[195,206,207] – and many more[208].

The curing progress is most often quantified via the degree of cure, α, which assumes values between 0 (no curing) and 1 (fully cured). During polymerisation, reaction educts are converted into one or more reaction products. In this process, the reaction rate $d\alpha/dt$ represents an essential parameter that describes the temporal change of the educts and products concentrations. Instead of individual reactant concentration changes, α is thus used for the description of the curing progress. For α, the reaction speed v_r can be written as in Equation 1, cf. [194]:

$$v_r = \frac{d\alpha}{dt} \tag{1}$$

The determination of the reaction educts and products concentration is, however, difficult. Mostly for practical reasons, it is thus assumed that the released reaction heat during the polymerisation correlates directly with the reaction progress. Therefore, the ratio between heat released up to any step t of the polymerisation, $H(t)$, and the maximum releasable heat, H_{Total}, after complete curing ($\alpha = 1$) is posited to equal the curing degree α, which is expressed by Equation 2:

$$\alpha(t) = \frac{H(t)}{H_{Total}} \tag{2}$$

The heat flow, H, can be determined directly in the DSC. Consequently, the measured curve for H can be used to calculate the temporal progression of α by integrating Equation 2.

2.5.2 An important phenomenological approach

Generally, two main types of phenomenological models can be distinguished: n^{th} order, and autocatalytic models. The former foot upon the assumption that the curing rate, $d\alpha/dt$, reaches its maximum when no conversion has yet occurred ($\alpha = 0$). In contrast, autocatalytic models surmise $d\alpha/dt$ being highest at a finite conversion value ($\alpha > 0$)[209]. As n^{th} order models often are not capable of achieving a satisfactory description of autocatalytic polymerisations, KAMAL & SOUROUR[210,211] introduced a phenomenological model, which was used, for example, in [175,212–216]. This model represents an extension of a kinetic model developed by HORIE et al.[217], who described an autocatalytic polymerisation of primary amines with epoxy resin through hydroxyl groups. KAMAL & SOUROUR generalised it to Equation 3, cf. [210]:

$$\frac{d\alpha}{dt} = (k_1 + k_2 \cdot \alpha^m) \cdot (1 - \alpha)^n \text{ with } k_{1,2}(T) = A_{1,2} \cdot e^{-\frac{E_{1,2}}{RT}} \tag{3}$$

Here, m and n represent the reaction orders and the temperature-dependent parameters k_1 and k_2 follow an Arrhenius law. The parameter k_1 represents the initial reactivity of the resin for $\alpha = 0$, while k_2 takes into account the autocatalytic acceleration of the polymerisation. Thus, in a purely autocatalytic behaviour $k_1 = 0$, while for $k_2 = 0$ the reaction has an n^{th}-order kinetics. A detailed derivation of the KAMAL & SOUROUR model was given for example in [194] or [218], and shall thus not be repeatedly discussed herein. KAMAL & SOUROUR'S model, along with many other kinetic models, is nowadays implemented in dedicated kinetic software. With the help of these programs, an evaluation of experimentally determined metrics, e.g. dynamic or isothermal DSC data, can be carried out more easily. In this thesis, KAMAL & SOUROUR'S model served to model the 2K adhesives curing behaviour during the accelerated curing operations both on the experimental (1st publication) as well as on the numerical level (5th and 6th publication).

2.5.3 Curing kinetics and numerical modelling

The numerical modelling of polymeric curing reactions and associated effects on adhesively bonded components represents a relatively new field of research. Accoringly, past studies on kinetic modelling were often carried out on adhesive bulk level using different approaches, e.g. [174,175,218]. Although a satisfactory description of the curing process could often be achieved, developed techniques were in many cases not transferred to the level of bonded joints. However, as resulting adhesive properties like strength[219] or stiffness[220] are strongly dependent upon applied curing conditions, implementation of kinetic models into appropriate simulation software based upon the Finite Element Method (FEM) represents a highly desirable target. In this way much more predictable, thus more efficient, curing processes may be designed. Besides these advantages, integration of curing kinetics into numerical models is still far from being state of the art, as proven by only a few approaches being available for curing of bulk adhesive[221,222] as well as FRP[223–226]. In addition to these knowledge gaps, there is currently no finite element (FE) software with an integrated kinetic package available on the market (except for LS-Dyna, for which the KAMAL & SOUROUR model can be adapted).

By going more into detail, GHOREISHY & NADERI[227] combined an FE simulation with the curing kinetics of rubber cured by vulcanisation in a mould, with the kinetic model of KAMAL & SOUROUR[210,211] used for the description of the curing progress. Validation of the developed model was carried out using an experimentally determined temperature curve, which showed good agreement with the numerical calculation. Aforementioned results have since then been extended towards contact between rubber and mould, modified kinetic models, as well as a more robust computer code using Abaqus by RAFEI et al.[228] in 2009.

More recent studies in the field represent the works of CEBRIÁN et al.[229], CASSANO et al[230]. or DEVAUX et al[221], who tried to link curing kinetics of adhesives to experimentally determined thermal loads within appropriate numerical models. CEBRIÁN et al.[229], developed a simulation tool allowing for a prediction of the degree of cure, α, of a paste adhesive as a function of adherend (C-FRP) temperature heated by electromagnetic induction. The model was validated by comparing adherend temperatures recorded with a pyrometer at five different locations on the C-FRP surface to the numerical predictions. CASSANO et al.[230] investigated curing kinetics of a 2K epoxy paste adhesive using isothermal DSC measurements as a basis for the kinetic modelling. KAMAL & SOUROUR's[210,211] autocatalytic model was applied and integrated into a 2D cross-sectional finite element analysis (FEA) of an oven-cured trailing edge of a wind turbine that was simulated in Abaqus, as shown in Figure 14. Validation of the numerical model was successfully carried out using temperature profiles recorded with thermocouples embedded directly inside the adhesive layer of G-FRP sandwich panels, with adhesive layer thicknesses of 10, 20 and 30 mm being investigated. In 2015, DEVAUX et al.[221] used the kinetic model of KAMAL & SOUROUR[210,211] with consideration of diffusion control for the description of isothermal and dynamic DSC data of an epoxy adhesive. Curing kinetics was coupled to a transient thermal simulation performed in Abaqus using

different subroutines for the description of cure-dependent phenomena like e.g. release of polymerisation heat. The developed FEA was validated by experimentally determined curing temperature profiles, which showed very good agreement with the numerical predictions.

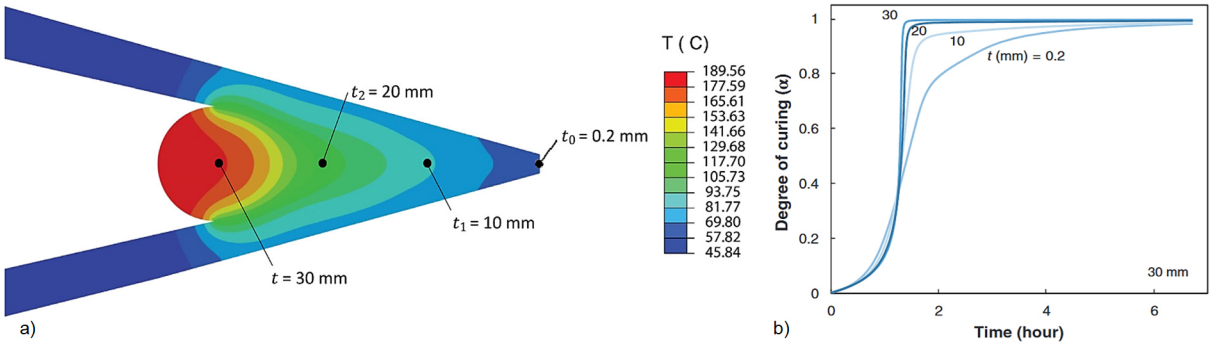


Figure 14: The numerical model of CASSANO et al. [230] from 2020; a) Numerical temperature condition throughout 2D trailing edge of wind turbine trailing edge at t = 82 min resulting from oven heating, b) Numerically calculated progression of curing degree, α, in dependency of temperature history at different points of the numerical model for standard cure cycle

Aforementioned results prove general feasibility of linking curing kinetics to experimentally determined heat loads within a transient thermal FEA. However, this has not yet been done in the context of accelerated curing by inductively heated CP. The method of CP-induced curing significantly differs from curing methods applied in aforementioned studies as heating is not introduced externally, e.g. by inductively heated adherends[229] or an oven[230], but from the inside of the polymer by the homogeneously distributed CP. In this context, curing has to be described not only as a function of developing curing temperature profiles resulting from CP-induced heating, but also in dependency of the influences on the adhesive's curing kinetics attributable to the introduced CP. Furthermore, the amount of heat introduced by the CP has to be described by the model. In this thesis, such a numerical model was developed, which was the topic of the 5th and 6th incorporated publication.

2.6 Low-temperature curing of adhesives

Since the CP-curing technique was utilised for low-temperature curing in the 7th publication, this section shortly sketches the state of the art in the field. Up to now, low-temperature curing of adhesives is not yet a clearly defined field of research, as two different curing scenarios may be distinguished. On the one hand, a specific bond may be manufactured at low temperatures and subsequently cured under the very same environmental conditions – an approach, most certainly resulting in prolonged curing times, which in many cases may not be tolerable for practitioners. On the other hand, it is possible to apply the adhesive at low temperatures while accelerating the polymerisation in the following, e.g. by introducing heating, as schematically shown in Figure 15. So far, both manufacturing approaches have been very little studied, which is why the content of my 7th publication represents a kind of pioneering work in the field.

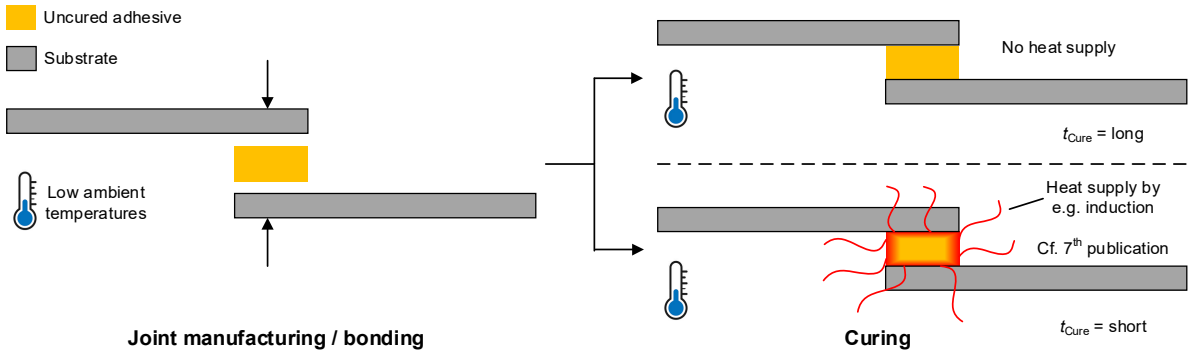


Figure 15: Principal illustration of possible low-temperature curing scenarios using the example of an SLJ manufactured under low ambient temperatures (own representation)

One of the few studies dealing with the former low-temperature curing scenario is that of MOUSSA et al.[231] almost ten years ago, who investigated curing of an epoxy adhesive at 5–10 °C for bridge construction during winter. Their investigations primarily focused on the effects on the glass transition temperature, T_g, as well as kinetic modelling of the curing process based upon isothermal and dynamic DSC data, i.e. development of T_g and curing degree, α, with temperature and time. Unsurprisingly, the authors found that curing is significantly slowed down in the considered temperature range, with several days being needed until curing degrees above $\alpha = 0.8$ could be reached. In comparison to the curing degree, the development of T_g was slowed even more, which the authors assumed to be connected to vitrification of the polymer at low temperatures. MOUSSA et al. concluded that '*the long curing periods at low temperatures required in winter are in most cases not acceptable in bridge construction. For adhesives to be applied however, joints must be designed in such a way that they are heatable in order to accelerate curing.*'

Some years later, in 2020, the suggestion of MOUSSA et al. was picked up by RATSCH et al.[232], who focused on the second conceivable approach for low-temperature curing – the use of thermal energy. In short, they investigated the accelerated low-temperature curing of threaded steel rods (Ø16 mm) glued into beech LVL using different 2K structural adhesives (three epoxies and one polyurethane). The joints were subjected to an EMF, with the induction power, however, being regulated by a thermocouple attached to the rods in order to prevent overheating. In total, three starting temperatures were chosen for the induction processes: –10 °C, +5 °C and +23 °C. The results proved that epoxy-bonded joints cured with the proposed method achieve bond strengths equivalent to reference series cured conventionally at RT. In contrast, strengths of the polyurethane cured under low temperatures were significantly lower, which was related to interference of moisture during cross-linking. These results highlight that adhesive curing may be achieved by targeted heat introduction, even if their initial curing temperature is below +10 °C. However, the CP-curing technique has never been utilised for this purpose, which is why the thesis aimed to close this knowledge gap and thus pave the way for new application fields of adhesively bonded joints.

3 Motivation

Particle-induced accelerated curing by electromagnetic induction represents a complex multiphysical engineering problem, in which different sub-disciplines interact to result in specific properties of the cured joints. This overriding cognition illustrates the profound shortcomings of currently available studies in the field: They do not describe the process as a whole, but rather consist in isolated and unlinked 'scientific islands' such as heating behaviour or thermo-mechanical joint properties. In the relevant literature, the various sub-topics are thus only considered isolated, in some cases not even investigated at all and connections between them are predominantly not established. In order to provide practitioners with targeted assistance to implement the curing technique into a specific manufacturing environment, it is paramount to model the process as a whole and to connect all individual aspects.

Besides these shortcomings concerning structuring, experiments in the field have often been performed as simple parameter studies, e.g. by varying particle type or size. If bonded joints were considered, the latter were almost exclusively carried out on the level of small-scale SLJ. There is thus a clear lack in the performance of experiments on the level of whole joints close to application and studies that consider inductively heated particles for low-temperature curing thereof are missing completely.

In addition, appropriate numerical models for accurate design of particle-induced curing processes are currently not available. Such models are needed to provide answers for essential scientific and technical questions, such as estimation of induction times and required energy to achieve full adhesive cure under specific production conditions. Furthermore, factors that determine the heating behaviour of particle-cured adhesives are largely unknown and may thus be identified and evaluated way more efficiently by using holistic simulation models based on the FEM.

4 Scientific goal and objectives

Based upon the extensively addressed shortcomings, I formulated the overarching goal of the thesis, which is the following:

'Design of the accelerated curing by inductively heated Curie particles as a process'

As a result, a process chart will be created, which includes a clear description of influencing factors to be considered as well as recommendations with regard to practical application.

In addition, I pursued the following secondary objectives:

- *1st objective*: Providing an inductive heating process without external temperature control by using CP.
- *2nd objective*: Quantification of possible reductions in curing time.
- *3rd objective*: Validation that the adhesives remain undamaged.
- *4th objective*: Identification of changes in mechanical and thermo-mechanical properties of the adhesives and joints caused by accelerated curing.
- *5th objective*: Outline influencing factors of the heating behaviour of CP-cured adhesives and joints as well as evaluation of their contribution.
- *6th objective*: Up-scaling to large components.
- *7th objective*: Numerical modelling of the induction heating process.
- *8th objective*: Extension towards low-temperature curing.

If considered individually, most of aforelisted objectives already represent an extension of the current state of the art. Consequently, investigations that combine all aspects under the roof of the proposed '*process design*' are most evidently not available at all. The thesis thus offers an approach on how to describe a curing method in its entirety and can be understood as a guideline on how these kinds of processes may be structured or even standardised in the future.

5 Strucutre of the thesis and methodology

To provide a clear overview of the thesis' contents, I visualised most important research objectives of each publication in Figure 16, which has to be read from left to right. In accordance with the thesis' main objective (cf. chapter 4), the papers were sorted according to the suggested '*process design*', with the investigations starting at the level of bulk adhesive (1st publication) and ending at the level of exemplary bonded connections (4th to 8th publication). The general structure of the thesis is as follows:

- The state of the art included in each of the original papers has been removed, shortened and re-structured into a unified state of the art in part I of the thesis.
- In order to avoid repetitions, the 'Materials & methods' sections of all puplications have been merged and shortened to the most relevant aspects. These form part II of the thesis.
- Part III of the manuscript includes a short description of the scientific content and most important findings of each incorporated publication.
- In part IV of the thesis, all findings were brought in line with the scientific objectives formulated in chapter 4 and the achievements of the dissertation were properly worked out. In addition, this part includes the summary of the thesis as well as an outlook.
- My curriculum vitæ is is included in part V of the thesis.

In order to achieve the scientific objectives set and presented in chapter 4, I adhered to the following methodological guidelines:

- **1st guideline:** As the thesis is settled in a civil engineering context, the investigations were focused on 2K structural adhesives, three 2K epoxy resins and two 2K polyurethanes. All selected polymers are commercially available and widely used in the construction sector so to guarantee the practical relevance of all findings.
- **2nd guideline:** In order to create optimal thermal conditions during the accelerated curing experiments, the CP were adapted to the requirements of the 2K adhesives. A material composition, i.e. Curie temperature, T_c, was chosen that matched the desired temperature range and CP size was adjusted to guarantee sufficient heating.
- **3rd guideline:** The very same validated mixing procedure was maintained across all investigations. This aimed at excluding any influence thereof on the results.
- **4th guideline:** Investigations were scaled up from the level of bulk adhesive to that of large bonded components. In this way, it was possible to understand the CP-curing technique as a process that influences the adhesive on different levels, starting from the polymerisation itself, over the formation of adhesive and cohesive strength up to the connections' mechanical behaviour.

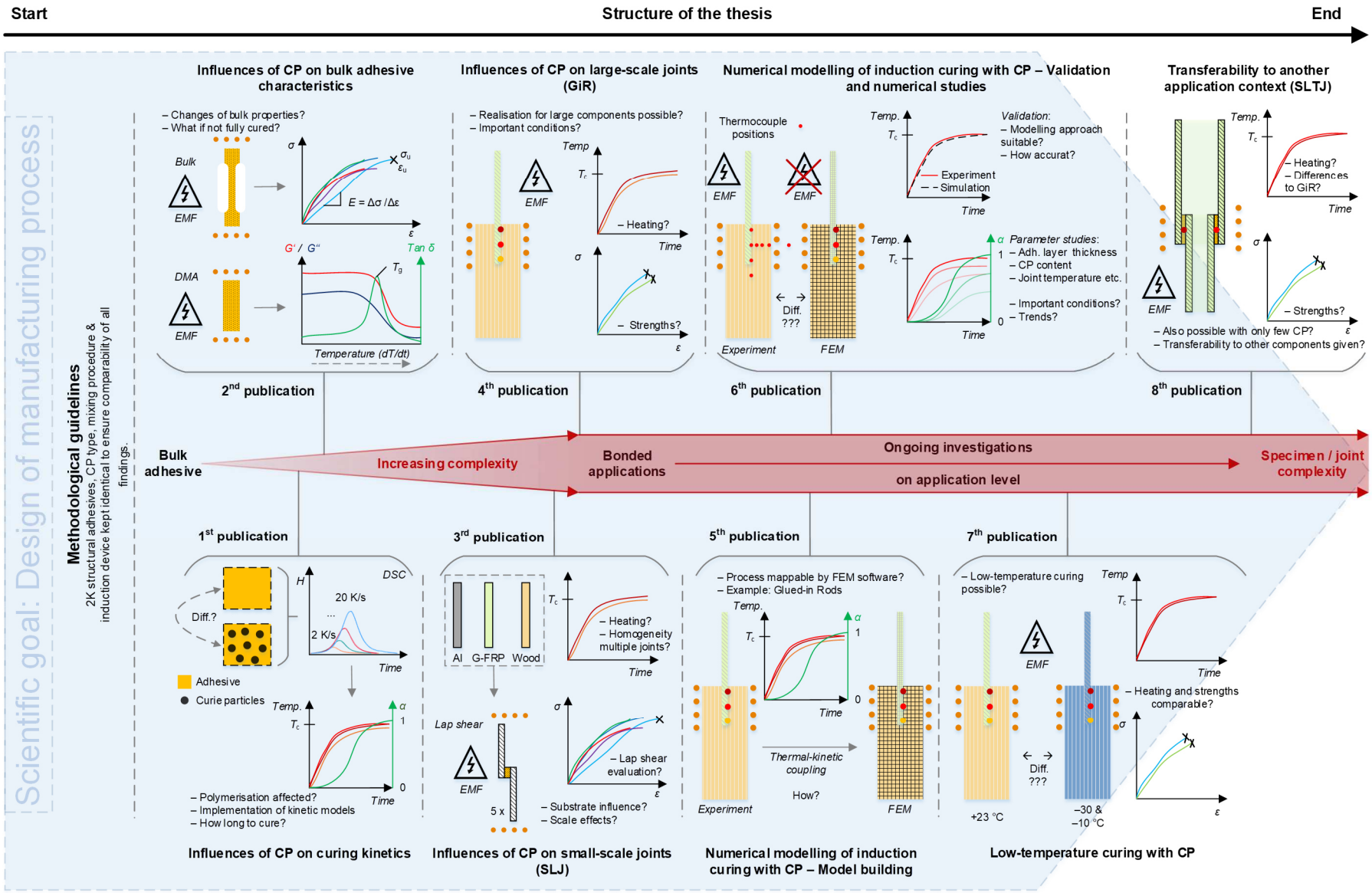


Figure 16: Content of the thesis with breakdown of individual subject areas according to the attached publications (own representation)

Part II: Materials & methods

6 Materials & methods

6.1 Adhesives

6.1.1 Material properties

In total, five commercially available 2K thermosetting structural adhesives of different adhesive manufacturers were investigated. For the adhesive class of 2K epoxy (subsequently referred to as 2K-EPX), the following polymers have been selected: Fischer EM390S (abbreviated from here on as Fi390, Fischerwerke GmbH & Co. KG, Germany), Jowat 692.30 (Jo692, Jowat SE, Germany) and Wevo EP32S (We32, Wevo-CHEMIE GmbH, Germany). To extend the findings towards another polymer class, two 2K polyurethanes (2K-PUR), Loctite Purbond CR421 (LP421) and its successor product CR821 (LP821, Henkel AG & Co. KGaA, Germany) were chosen. In the further course of the thesis, the abbreviations defined above are used for the designation of the adhesives. Essential material properties for the non-CP-filled polymers, i.e. commercially available formulations, have been summarised in Table 2.

Table 2: Essential material characteristics of all considered adhesives taken from manufacturer's technical data sheet (TDS) as well as values marked with * that have been measured. Curing times marked with ** calculated based upon Arrhenius law[233]. All values valid for non-CP-filled and RT-cured adhesive formulations. Information concerning the substrate materials used for the determination of lap shear strengths can be found in section 6.4.3.

Adhesive characteristic	Fi390	Jo692	LP421	LP821	We32
General					
Adhesive type	2K-EPX	2K-EPX	2K-PUR	2K-PUR	2K-EPX
Density, ρ [g/cm³]	1.50	1.46	1.35	~1.32	1.13
Curing properties					
Pot life for curing at RT[min]	14–30	25–35	10	30	30–40
Curing time at RT [h]	18	>24	240	240	20
Curing time at +5 °C [h]	40	48**	480**	480**	40**
Curing time at –10 °C [h]	160**	192**	1920**	1920**	160**
Initial viscosity at RT, η [Pa·s]	100 / 114*	50 / –	9 / 10.4*	1.5–4.5 / –	8–9 / –
Polymerisation enthalpy H_{Total}[1] [J/g]	306 ± 11.3*	208.6 ± 8.9*	114.2 ± 2.4*	149.5 ± 5.5	459 ± 16.9*
Thermo-mechanical properties					
Modulus of elasticity[2], E [MPa]	– / 5230*	6320 / 7020*	1560 / 2760*	~1000 / –	– / 3330*
Tensile strength[2], σ_u [MPa]	– / 40*	24.6 / 32.4*	25–30 / 28.6*	~45 / –	– / 43.7*
Elongation at break[2], ε_u [%]	– / 1.2*	– / 0.54*	2 / 1.6*	~5 / –	– / 1.6*
T_g for curing at RT[3] [°C]	– / 73.4*	– / 71.1*	– / 81.3*	– / –	– / 59.6*
Lap shear strength[4], τ_u, on:					
Aluminium[4-a] [MPa]	10.5 ± 0.2*	10.1 ± 1.6*	10.4 ± 1*	11.0 ± 0.8*	11.2 ± 1.4*
G-FRP[4-b] [MPa]	12.0 ± 1.0*	10.6 ± 0.6*	11.1 ± 1*	9.3 ± 2.0*	–
Wood (spruce, *picea abies*)[4-b] [MPa]	8.0 ± 0.4*	8.1 ± 0.4*	8.4 ± 0.8*	8.1 ± 0.6*	–

[1]DIN 53765[234]; Arithmetic mean out of dynamic DSC meausrements with ΔT = 2, 5, 10, 15 and 20 K/min, T_{range} = 0 to +250 °C; Measured values taken from 1st publication

[2]DIN EN ISO 527[162]; For values marked with *, v = 1 mm/min applies; Measured values taken from 2nd publication

[3]DIN EN ISO 6721[235]; T_{range} = –40 to +150 °C, ΔT = 2K/min, A = 20 µm, f = 1 Hz; Measured values taken from 2nd publication

[4]DIN EN 1465[236]; [4-a]t_a= 1 mm, v = 2 mm/min; [4-b]t_a= 0.3 mm, v = 2 mm/min; Measured values taken from 3rd publication

The adhesive selection foot upon the following two major reasons: Firstly, all adhesives are widely used for civil engineering applications – especially for bonding threaded rods (DIN 976-1[237]), or rebars, into wooden construction elements made of glued laminated timber (GLT) or LVL. The latter are known as so-called Glued-in Rods (GiR, see section 6.4.4) and served as an example to investigate the influences of accelerated curing with CP on a large component level, both experimentally (4th publication) as well as numerically (5th and 6th publication). For GiR applications, the adhesives LP421, and We32 carry national technical approval, which was granted by the *Deutsches Institut für Bautechnik* (DIBt, the technical authority in the German construction sector). Secondly, RT-curing of all selected adhesives normally lasts very long (1–10 days), which is why they represent a good example to illustrate the advantages of the accelerated curing process. Much more detailed information regarding specific application cases of each adhesive can be found in the original articles and will thus not be repeated herein. The selected polymers cover a representative spectrum of commercially available adhesives with – in addition – significant practical relevance, ensuring optimal result exploitation with regard to practical implementation.

In common construction practice, application of all considered adhesives is usually performed directly from the original cartridge into the holes or gaps to be filled using standardised mixer nozzles. Consequently, ambient conditions as temperature and relative humidity (RH) must always be considered. Attention has thus to be paid to the fact that processing at temperatures below about +10 °C is currently not allowed by codes and standards. The reason for that can be traced back to the fact that full cure, i.e. sufficient adhesion / cohesion build-up, cannot be guaranteed for low temperatures. In the worst scenario, the respective polymer will not be able to cross-link at all or, more specifically, its curing is extended over an unreasonable period. To overcome these severe limitations, the potential of the CP-curing technique to enable polymerisation that starts from low temperatures (+5 and –10 °C) was investigated in the 7th publication of the thesis. Due to the relations outlined above, curing times for low temperature ranges are not provided by adhesive manufacturers. Accordingly, these have been estimated following Arrhenius law[233] based on curing times needed at RT and were added to Table 2.

6.1.2 Determination of maximum heating rates and temperatures

Intention & assumptions

In context of any heat curing operation, excessive heating rates, dT/dt (ΔT), or curing temperatures, T_{cure}, can provoke polymer damage, which in turn may lead to deteriorated bond properties. In context of CP-curing, maximum permissible heating regimes had an even more important role, as the CP's Curie temperature, T_c, had to be adjusted to the thermal limits of the selected 2K polymers so to guarantee safe and CP-controlled heat introduction (see section 6.2). As a result, maximum tolerable curing temperatures represented an important polymer characteristic, which had to be determined prior to any CP-curing operation. For that, preliminary inductive heating experiments using aluminium SLJ according to DIN EN 1465[236] were carried out, with all information concerning the substrate material provided in

section 6.4.3. The investigations presented in the present section can thus be considered a prerequisite for all subsequently performed CP-curing operations.

To support the design of the preliminary experimental campain, tolerable heating conditions were determined in advance by each adhesive manufacturer, with the following thermal benchmarks being defined: ΔT_{max} = 2 K/s, $T_{cure,\,max}$ = 120 °C. Since heatability limits represent a characteristic of the polymers, the majority of SLJ experiments was performed with the non-CP-filled polymers. However, to investigate the influence of CP, one additional series was produced for each adhesive (cf. Table 3, rightmost column). Furthermore, it was assumed that overheating-induced polymer damages exert a direct negative impact on the adhesives lap shear properties, i.e. they result in lowered lap shear strengths or changing fracture patterns in comparison to RT-cured, unfilled references. Conversely, an equal or better fracture behaviour was considered to be an indicator for an intact joint.

Experimental procedure

The scope of the preliminary experimental campaign has been summarised in Table 3. Different aluminium SLJ series were EMF-heated using the induction device described in section 6.5.1 with mounted induction coil A (cf. Figure 26). In order to statistically validate the findings, each series consisted of three SLJ, with Figure 17 illustrating their manufacture. In detail, each adhesive was first applied from the original cartridge into a small sample beaker using the manufacturer's mixer nozzles, thus ensuring correct mixing ratio of resin and hardener. Afterwards, the adhesive was applied on the aluminium adherends using a spatula and joined in a PTFE mould (see Figure 17-a). The adhesive layer thickness of all SLJ was set to 0.3 mm using appropriate spacers. Immediately after joining, the PTFE mould was placed on a sample holder and the adhesive layers were positioned centrically within the induction coil (see Figure 17-b). The entire preparation process for each series, starting from the moment resin and hardener were in first contact to the point in time the induction device was switched on, took about 5 min, which was well within the pot life of all adhesives (cf. Table 2).

Table 3: Experimental program of inductively cured aluminium SLJ (DIN EN 1465[236]) to proof rapid curability of the 2K adhesives using lap shear strength as comparative metric, dT/dt = 2K/min, series marked with * produced with 30 w/w-% CP following the procedure outlined in section 6.3

Adhesive	Polymer class	Reference set	Curing time at 110 °C, t_{Cure} [s]									
			15	23	30	45	60	90	120	150	180	300
Fi390	2K-EPX	5	3	3	3	3	3	–	3	–	3	3 / 3*
Jo692		5	3	3	3	–	3	3	3	–	–	3 / 3*
We32		5	–	–	–	–	3	3	3	3	–	3 / 3*
LP421	2K-PUR	5	–	–	3	–	3	3	3	–	3	3 / 3*
LP821		5	–	–	–	–	3	3	3	–	3	3 / 3*

After positioning, the induction device was switched on, with the heat being generated within the aluminium adherends and conductively dissipated into the adjacent adhesive layer. The relatively thin adhesive layer (0.3 mm) was expected to result in a homogeneous temperature distribution in the area of

the overlap. More detailed explanations regarding this topic can be found in the original article of the 3rd publication. In the following, induction power was regulated in function of a pre-defined temperature profile measured with a thermocouple in order to control and to avoid adhesive overheating (cf. section 6.5.4). The thermocouple was attached to the rear side of the middle SLJ at the height of the adhesive layer using a temperature resistant polyamide adhesive tape (see Figure 17-b).

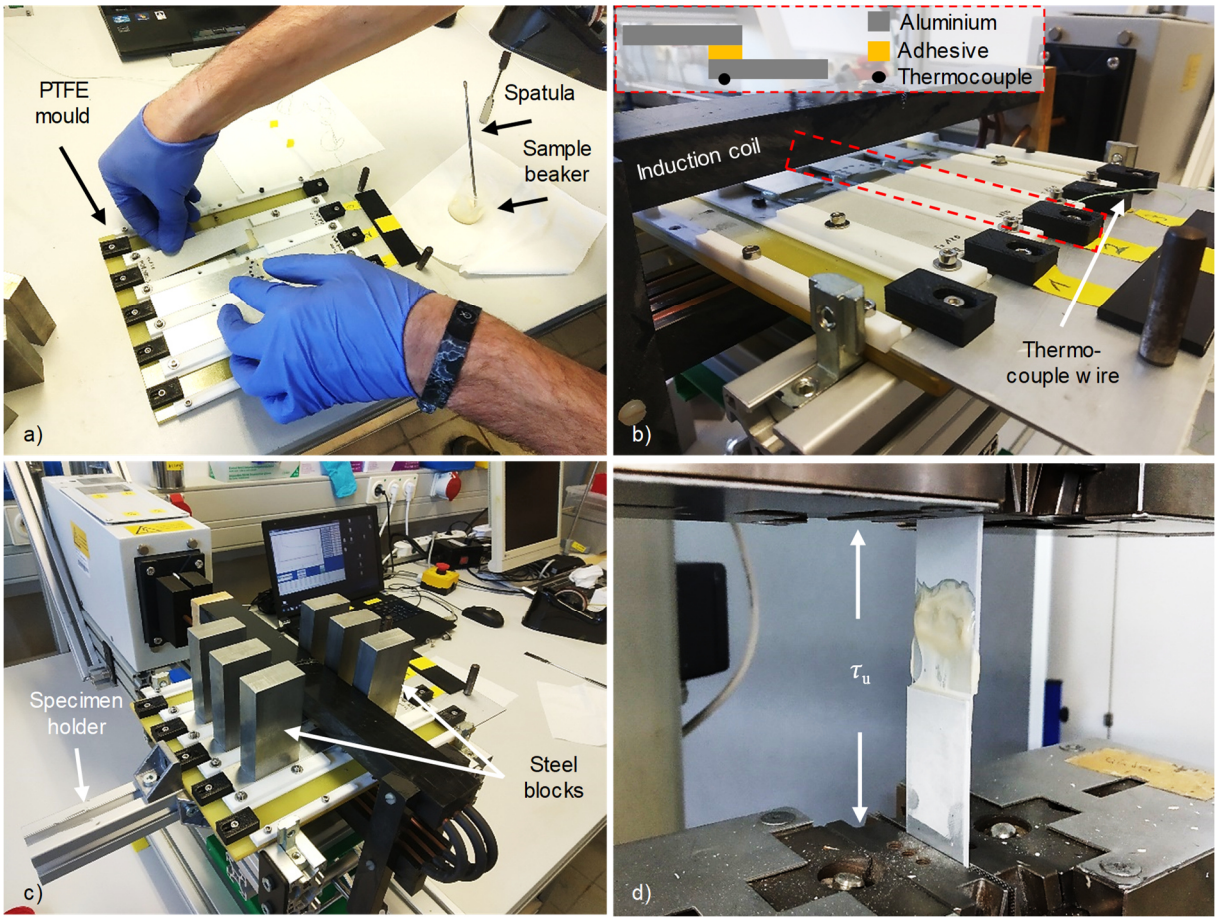


Figure 17: Applied experimental procedure to prove capability of the 2K adhesives for accelerated curing, herein exemplarily illustrated for the adhesive LP421; a) Joining of aluminium SLJ in PTFE mould, b) positioning in specimen holder and position of thermocouple used to regulate induction power, c) cooling of SLJ with steel blocks after inductive heating and d) mechanical testing of SLJ immediately after inductive heating (herein insufficiently cured); Zwick|Roell UTM (20 kN), v = 5 mm/min, all tests performed at RT

During the induction processes, each series was heated at a constant heating rate of 2 K/s (limit of adhesive manufacturers) to a target temperature of 110 °C (T_c of CP, less than 120 °C, cf. section 6.2). Depending on the respective series, the curing temperature of 110 °C was then held for different periods, with curing times, t_{Cure}, of 15, 23, 30, 45, 60, 90, 120, 180 and 300 s being investigated. After the curing time had elapsed, the induction device was switched off and the joints were quickly cooled to RT. For that, cold steel blocks were placed on each joint to avoid significant post-curing beyond the pre-defined temperature regime, i.e. alterations in resulting lap shear strengths (see Figure 17-d). An exemplary temperature profile measured for Fi390 and t_{Cure} = 300 s was added to Figure 18-b. Since strength buil-up is dependent upon underlying curing kinetics (cf. 1st paper), investigated curing times per adhesive differed

in order to fill gaps in the obtained lap shear data sets. Mechanical testing was performed immediately (~5–10 min) after the joints had reached RT.

Results

Lap shear strengths, τ_u, for each SLJ series were sorted by adhesive, plotted in function of t_{Cure} and fitted in OriginPro 2020© using a Hill function (see Figure 18-a, green curves). Furthermore, mean strengths and associated standard deviations of the RT-cured references were added to the graphs (see Figure 18-a, purple lines). All fracture patterns have been added to Table 4. The following presentation and discussion of the SLJ data was limited towards relevant aspects for proof of rapid curability.

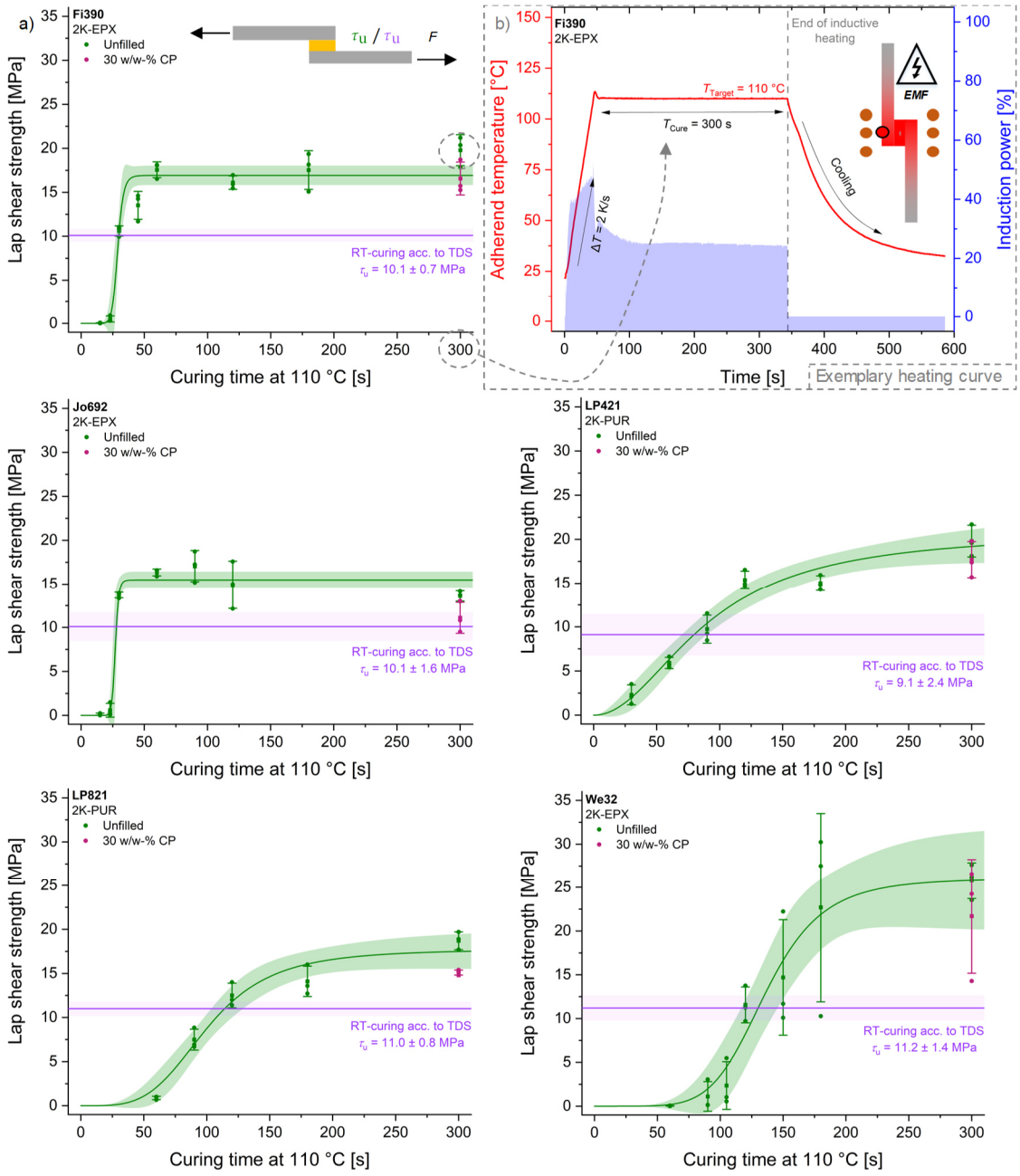
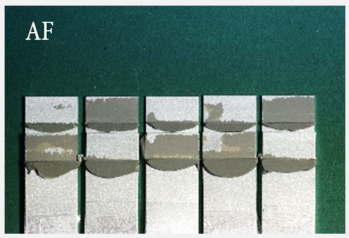
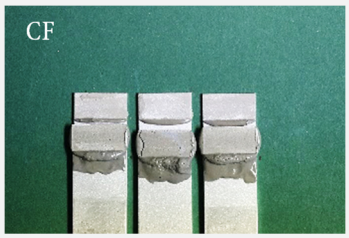
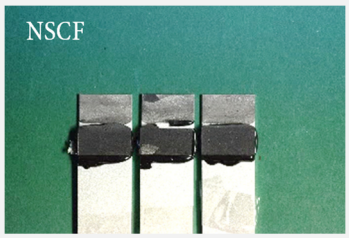
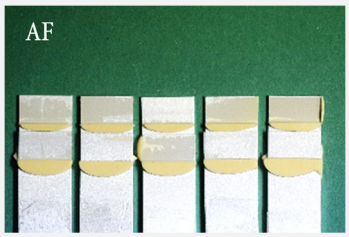
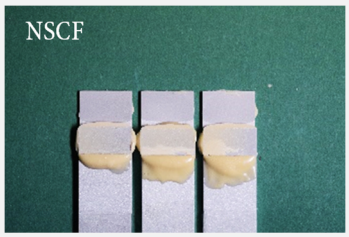
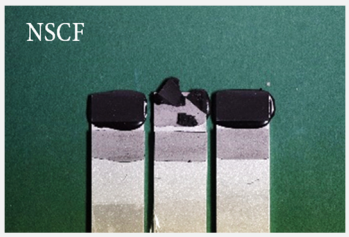
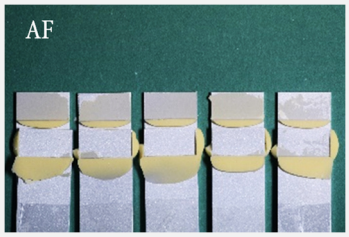
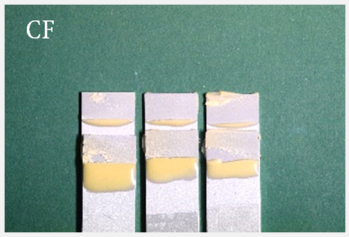
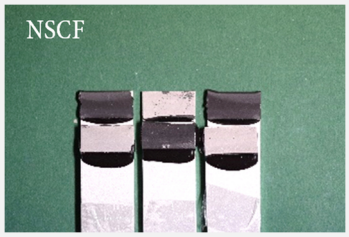
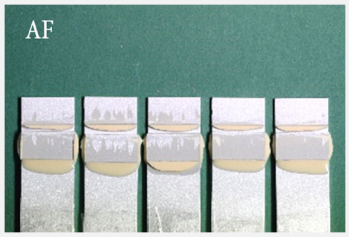
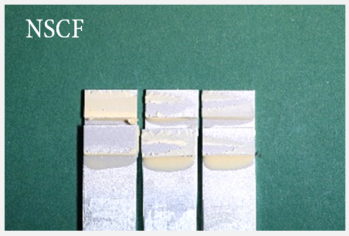
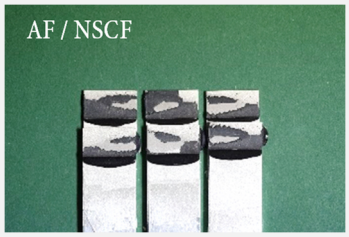
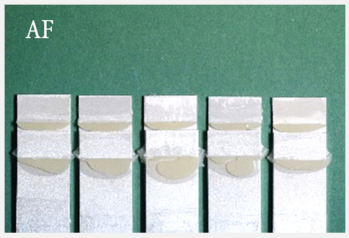
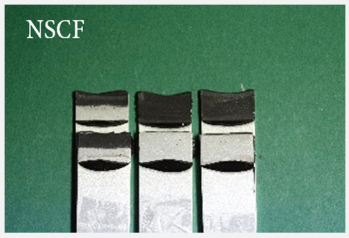


Figure 18: a) Relationship between curing time, t_{Cure}, at 110 °C (T_c of CP) and attainable lap shear strengths of unfilled (green curves, Hill fit with 95 % confidence intervals) and CP-filled adhesives (dark purple bars, t_{Cure} = 300 s) in comparison to RT-cured references (bright purple lines); Zwick|Roell UTM (20 KN), v = 5 mm/min, all tests performed at RT; b) Exemplary temperature curve recorded during inductive heating using thermocouple attached to rear side of middle SLJ

Table 4: Fracture patterns of representative inductively cured SLJ series produced to prove capability for accelerated curing of the 2K adhesives, Zwick|Roell UTM (20 kN), v = 5 mm/min, all tests performed at RT; *AF* = Adhesive / adhesion failure, *CF* = Cohesive failure, *NSCF* = Near-surface cohesive failure

Adhesive	**Series**		
	Reference set	t_{Cure} = 300 s, unfilled	t_{Cure} = 300 s, 30 w/w-% CP
Fi390			
Jo692			
LP421			
LP821			
We32			

The lap shear data shows that strength build-up under the selected experimental conditions is significantly dependent upon the adhesive. Thus, the adhesives Fi390 and Jo692 already attain adequate strengths of τ_u = 10.6 ± 0.6 MPa (Fi390) and 13.7 ± 0.3 MPa (Jo692) after being cured at 110 °C for only 30 s. In contrast, LP421(t_{Cure} = 90 s, τ_u = 9.7 ± 1.6 MPa), LP821 (t_{Cure} = 120 s, τ_u = 12.5 ± 1.4 MPa) and We32 (t_{Cure} = 120 s, τ_u = 11.6 ± 2.1 MPa) require considerable more time until similar strengths are reached. Changes in strength for LP421, LP821 and We32 are observable over the whole data range, whereas an abrupt increase of τ_u (t_{Cure} = 23–60 s) with almost constant strength values in the following (t_{Cure} > 60 s) can be seen for both Fi390 as well as for Jo692. With regard to SLJ series produced with CP, the data behaves similarly when all five adhesives are compared. Thus at t_{Cure} = 300 s, mean lap shear

strengths of CP-filled SLJ always lie about 10 % below the strengths of the respective unfilled and inductively cured series (see Figure 18-a, .dark purple bars). Aforedescribed CP-related results comply with the findings of my 3rd publication, in which different SLJ substrate materials were considered.

Overall, the preliminary experiments proved that curing at 110 °C leads to significantly increased lap shear strengths for all considered adhesives when the target temperature is held long enough. Assuming a curing time of 300 s, average strength enhancements range from 34.7 % (Jo692) to 129.4 % (We32). Aforedescribed trends are confirmed by the fracture patterns (see Table 4), with all RT-cured reference sets showing adhesive failure. In contrast, fracture patterns change to cohesive or near-surface cohesive failure when induction curing at 110 °C for 300 s is pursued. With regard to CP-filled series, slight strength deteriorations compared to unfilled SLJ (~5–15 %) are observable, with, however, strengths still lying clearly above those of the references. This is also reflected by the fracture patterns, with no clear cohesive but only near-surface cohesive failure being achieved. All these observations clearly indicate that both unfilled as well as CP-filled adhesives are well suited for the purpose of acceleterated curing, if considering the investigated heating rate and end temperatures. As shown by each of my publications, heating rates and maximum curing temperatures during the CP-induced accelerated curing experiments were always below the presented thermal benchmarks used during the preliminary experiments.

6.1.3 Curing kinetics and modelling

For this thesis, the adhesives curing kinetics plays a major role as it contributed to the temperature development during the accelerated curing operations (cf. section 6.5.6). In order to quantify this effect, kinetic models were developed for all adhesives, which foot upon dynamic DSC measurements and allowed to predict the curing degree, α, in dependency of curing temperature, T_{cure}, and CP content, $c_{particle}$ (cf. 1st publication). Subsequently, the kinetic models were integrated into a numerical GiR model (cf. 5th publication), which opened up the possibility to predict induction times needed to achieve full cure for various boundary conditions of the induction process such as adhesive layer thickness, joint temperature, $c_{particle}$ etc. (cf. 6th publication). For all kinetic modelling works presented in this thesis, the software package Netzsch Kinetics Neo® (Netzsch-Gerätebau GmbH, Germany) was used. A detailed description of the applied modelling approach as well as underlying assumptions can be found in each of the mentioned original articles.

6.2 Curie particles

6.2.1 General characteristics

Soft magnetic Manganese-Zinc-Ferrite (Mn-Zn-Fe) particles (Hengdian Group Magnetics Co. Ltd., China), hereinafter referred to as Curie particles (CP), served as susceptors throughout the entire thesis. The CP had the shape of small flakes with sizes thereof ranging from 0.5–25.0 µm ($\overline{Ø}$ = 11.8 µm) and 90 % of all CP being smaller than 21.3 µm (see Figure 19). To determine the material composition of the

CP, an EDX analysis was carried out, which revealed an Mn and Zn content of 16.67 mass percentage (w/w-%) each as well as an Fe content of 66.6 w/w-%.

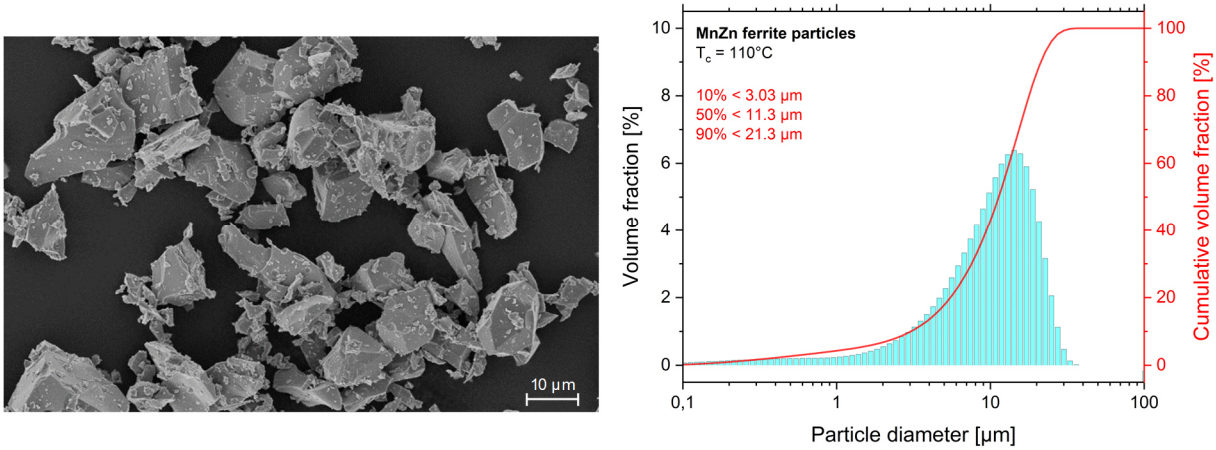


Figure 19: Left: SEM image of flake-shaped CP; Right: Size distribution of CP measured with particle size analyser (LS 13 320 XR, Beckman Coulter, USA)

The CP were selected because their Curie temperature, T_c, of 110 °C was closest to the range of temperatures relevant for the accelerated curing of the selected 2K adhesives (80–120 °C, see section 6.1.2). The properties of the CP, according to the manufacturer's TDS, are summarised in Table 5. Since the focus of this thesis was on the bonding-related aspects the accelerated curing process, further optimisations regarding size, shape or composition of the CP were not carried out.

Table 5: Material properties of CP according to manufacturer's TDS (calculated from toroid core)

Property	Conditions		Value
Density, ρ [g/cm^3]			4.9
Initial permeability, μ_i	10 kHz, B<0.25 mT	25 °C	12000 ± 30 %
Relative loss factor, $tan\ \delta/\mu_i$	10 kHz, B<0.25 mT	25 °C	<0.7 x 10^{-6}
Saturation flux density, B_s [mT]		25 °C	380
Residual flux density, B_r [mT]	50 Hz, 1194 A/m	25 °C	100
Coercive force, H_c [A/m]		25 °C	6
Rel. temp. coefficient, α [1/°C]	10 kHz, 1.5 ~ 3 mT	20 °C ~ 60 °C	-0.5 ~ 2.0 x 10^{-6}
Hysteresis material constant, η_B [mT]		25 °C	<1.5 x10^{-6}
Resistivity, ρ [Ω·m]			0.15

6.2.2 Determination of Curie temperature and Curie effect

Since controlled heat introduction during accelerated curing was one of the central objectives of the thesis, further CP characterisations were carried out. These aimed at quantifying the susceptors 'switch-off behaviour' at T_c, which was of primary importance for the numerical modelling (cf. publication five and six). As T_c represents an essential material parameter to map the CP-induced temperatures numerically, experimental investigations using a modified TGA were carried out for its determination. For that, a small sample of CP (~20 mg) was placed in the TGA device and heated in a temperature range of 25–250 °C. Starting from RT, the temperature of the TGA was increased using a relatively low heating

rate of 1 K/min to ensure that the heat was distributed evenly within the CP sample. During the measurement, the attraction force exerted by a magnet on the heated CP was measured.

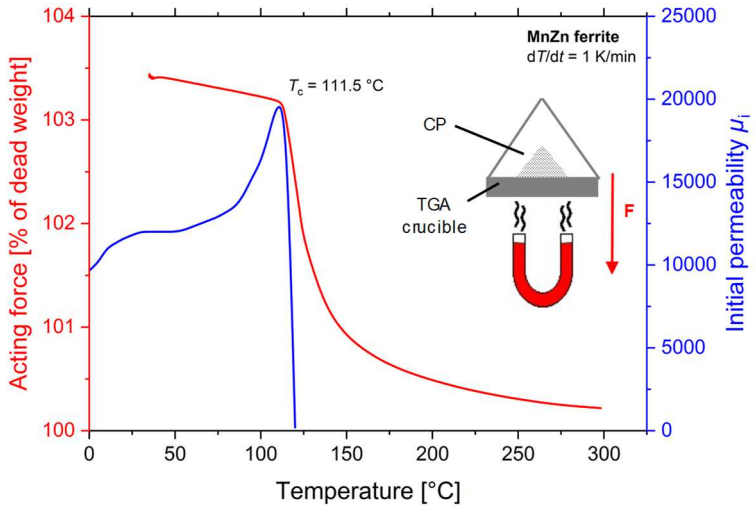


Figure 20: Comparison between modified TGA of CP (red curve) and initial magnetic permeability, μ_i, redrawn from TDS (blue curve); TGA device type Q5000 IR (TA Instruments Inc., USA)

The result of the modified TGA is illustrated by the red curve in Figure 20. At a temperature of ~25 °C, the attraction force between magnet and CP increases the "weight" of the crucible by about 3.5 %. When temperature is increased, the force first remains almost identical until the temperature reaches a value of approximately 111.5 °C, beyond which a significant decrease of the resulting force is visible. This is attributed to the fact that the susceptors begin to switch into the paramagnetic state beyond T_c. To support this observation, the initial magnetic permeability, μ_i, of the CP was taken from manufaturer's TDS and overlaid to Figure 20 as the blue curve. As it can be seen, both estimates of T_c agree well. These results were subsequently used to model the CP's 'switch-off' effect in the FEA tool, as shown in the 5th publication.

6.2.3 Determination of CP-induced heat

For the numerical modelling, thermal energy released by the CP exposed to EMF, labelled herein as H_{cp} expressed in W/cm^3, was determined in function of EMF frequency, f, and adhesive-CP temperature, T. For this purpose, small adhesive-CP toroidal cores were manufactured, which exhibited the same CP content (33.3 w/w-%) as the large-scale GiR joints used to validate the developed numerical model in publication six. In addition, the dimensions thereof matched those of the corresponding adhesive layer present during the GiR experiments (see paper four to six).

In detail, specimens manufacturing was carried out using annular silicone moulds with dimensions d_{out} = 20 mm, d_{in} = 18 mm, t_a = 1 mm and h = 3 mm. One demoulded, RT-cured specimen is exemplarily depicted in Figure 21-c. The experimental setup for the determination of H_{cp} is illustrated in Figure 21-a. Each adhesive-CP core was wrapped with a thin insulated copper wire (Ø0.3 mm), using 63 windings (see Figure 21-b). The two wire ends were attached to a circuit board, and connected to a wide band

power analyser, which was capable of generating AC signals in a frequency range up to 1 MHz. A frequency sweep was performed for each toroid at temperatures covering the temperature range of the inductive heating experiments used for the validation of the FE model (see publication six). An oven was used to heat up the samples to the target temperatures (see Figure 21-a, 3). Subsequently, a frequency sweep was performed for each toroidal core using frequencies of 25, 50, 75, 100 as well as 120 kHz. For all measurements a magnetic flux density, B, of 20 mT was applied over the cross-sectional area of the toroid specimens. Under the conditions relevant for most of this thesis, H_{cp} was determined to be 3.14 W/cm^3.

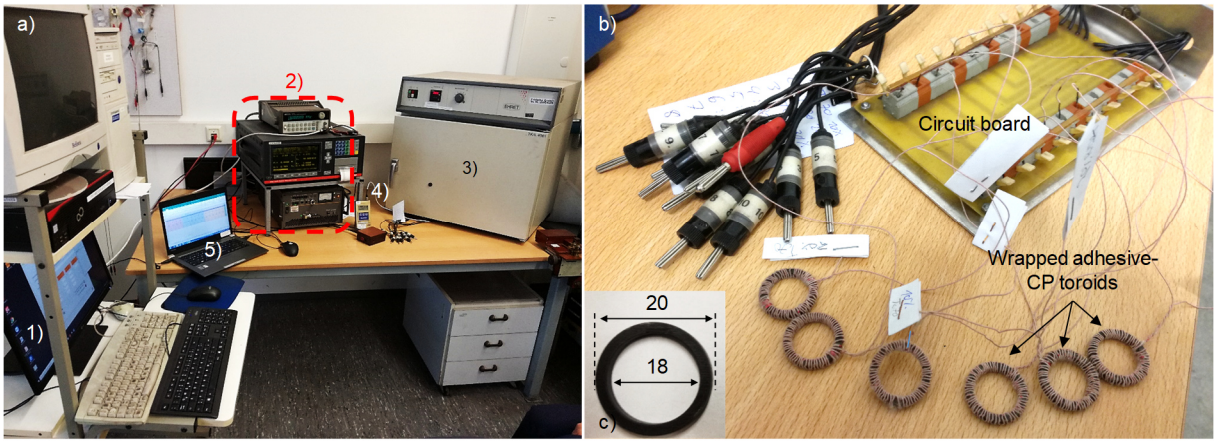


Figure 21: a) Experimental setup for determination of CP-induced heat, H_{cp}, with 1) software for measurement control and parameter supply, 2) wide band power analyser type D6100 (LEM Instruments GmbH, Germany) for generation of sinusoidal measuring signal, 3) oven for heating of adhesive-CP toroids, 4) thermocouple for external validation of oven temperature and 5) software for data evaluation; b) Different toroid cores wrapped in insulated copper wire (Ø0.3 mm, n = 63) attached to signal generator shortly before start of measurement; c) Exemplary dimensions of produced toroid core, herein consisting of RT-cured Fi390 mixed with 33.3 w/w-% of CP, dimensions in mm

6.3 Mixing of adhesives with CP

6.3.1 Intention & conditions

In order to guarantee an even mixing of the CP within the adhesives, an identical mixing programme was used for every CP-curing experiment included in this thesis. Irregular mixing would negatively affect the loaded joints mechanical performance due to inhomogeneously developing stresses. Air bubbles would represent a defect within the polymer matrix, which may provoke earlier failure of the connection.

Mixing was done under vacuum conditions (100 mbar) using a Planetary Centrifugal Vacuum Mixer illustrated in Figure 22-a-2. It aimed to achieve a high mixing quality while excessive heat development originating from frictional forces, i.e. shortenings in pot life, had to be avoided. For that, different mixing speeds within the permissible range of the utilised Speedmixer (500–2000 rev/min, respectively) were analysed. The following parameters proved suitable to meet these requirements: ν = 1000 rev/min, t_{mix} = 60 s.

Figure 22: Applied procedures for mixing (a) and processing (b) of the adhesive-CP mixes; a-1) Weighing of adhesive (herein Fi390) and CP, a-2) Speedmixer 800.2 VAC-P (Hauschild & Co. KG, Germany), a-3) adhesive-CP mixture after mixing for 60 s at 100 rev/min and 100 mbar; Aplication with b-1) syringe (herein for an AB specimens), b-2) spatula (herein for a G-FRP SLJ series) and b-3) 1K disposable cartridge (herein for a series of GiR)

6.3.2 Mixing procedure & mixture processing

All adhesive-CP mixtures were produced and expressed in terms of mass percentages (w/w-%). The investigations covered CP contents up to 40 w/w-%, beyond which the adhesive-CP mixtures proved not suitable. Firstly, the required amount of CP was weighed into a small sample beaker using a spatula. Subsequent adhesive processing was performed directly from the original cartridges using the manufacturer's mixer nozzles, and weighed into a second sample beaker so to achieve the correct mixing ratio of resin and hardener. Afterwards, the weighed CP were added to the adhesive (see Figure 22-a-1), the sample beaker was placed within the prepared Speedmixer and the raw materials were mixed at RT using the previously determined parameters. Mixing took about 3 min, which was well within the pot life of all considered adhesives (cf. Table 2). The subsequent application was then performed with a syringe, a spatula or a 1K disposable cartridge, as visualied in Figure 22-b.

6.4 Joint types & materials

6.4.1 Adhesive bulk specimens

Preparation of adhesive bulk (subsequently labelled as AB) specimens was performed according to DIN EN ISO 527-1[162]. All AB specimens were produced with the help of silicon moulds. Adhesive application was performed by decanting the unfilled adhesive or the adhesive-CP mixture into a syringe (see Figure 22-b-1). Afterwards, adhesive or adhesive-CP mixes were injected into the silicone mould.

6.4.2 Dynamic mechanical analysis specimens

Specimens preparation for dynamic mechanical analysis (subsequently referred to as DMA) measurements was done according to DIN EN ISO 6721[235] (35 x 10 x 3 mm^3). Similarly to the preparation of AB specimens, adhesive and adhesive-CP mixtures were transvased into syringes to be applied into aforementioned silicone moulds.

6.4.3 Single lap shear joints

The smallest joint type considered in this thesis were standardised single lap shear joints (SLJ) according to DIN EN 1465[236]. Two classes of substrate materials were used, EMF-sensitive and non-senstive. For the preliminary, and already presented, experiments to determine the heating limits (cf. section 6.1.2) as well as additional series presented in publication three, aluminium was used. For all experiments involving CP, materials not sensitive to the EMF were sought after: G-FRP and wood (herein spruce). All relevant properties of the three SLJ materials are listed in Table 6.

Table 6: Main material properties of adherend materials used for the production of SLJ, values taken from literature or manufacturer's TDS

Material property	Aluminium	G-FRP	Wood (spruce)
Material description	EN-AW 2024 (WS 3.1354)	Vetronit EGS 619	Picea abies
Density, ρ [g/cm³]	2.78	1.92	0.47
Modulus of elasticity, E [MPa]	73.100	23.000	10.000–12.000
Tensile strength, σ_u [MPa]	469	300	95
Shear strength, τ_u [MPa]	283	30	4–12
Thermal conductivity, λ [W/m·K]	121	0.3	0.1

6.4.4 Glued-in Rods

Large-scale Glued-in Rods (GiR) served as the first example to illustrate CP-curing on an application level. The timber blocks consisted of spruce, while the rods were made of G-FRP; their main properties are listed in Table 7. The geometry of the investigated samples is depicted in Figure 23-a along with a graphic visualisation of the applied manufacturing process, i.e. bonding, inductive heating and mechanical testing (see Figure 23-b). The detailed description of the GiR's manufacturing and accelerated curing procedure, can be found in the original articles of publications four to six.

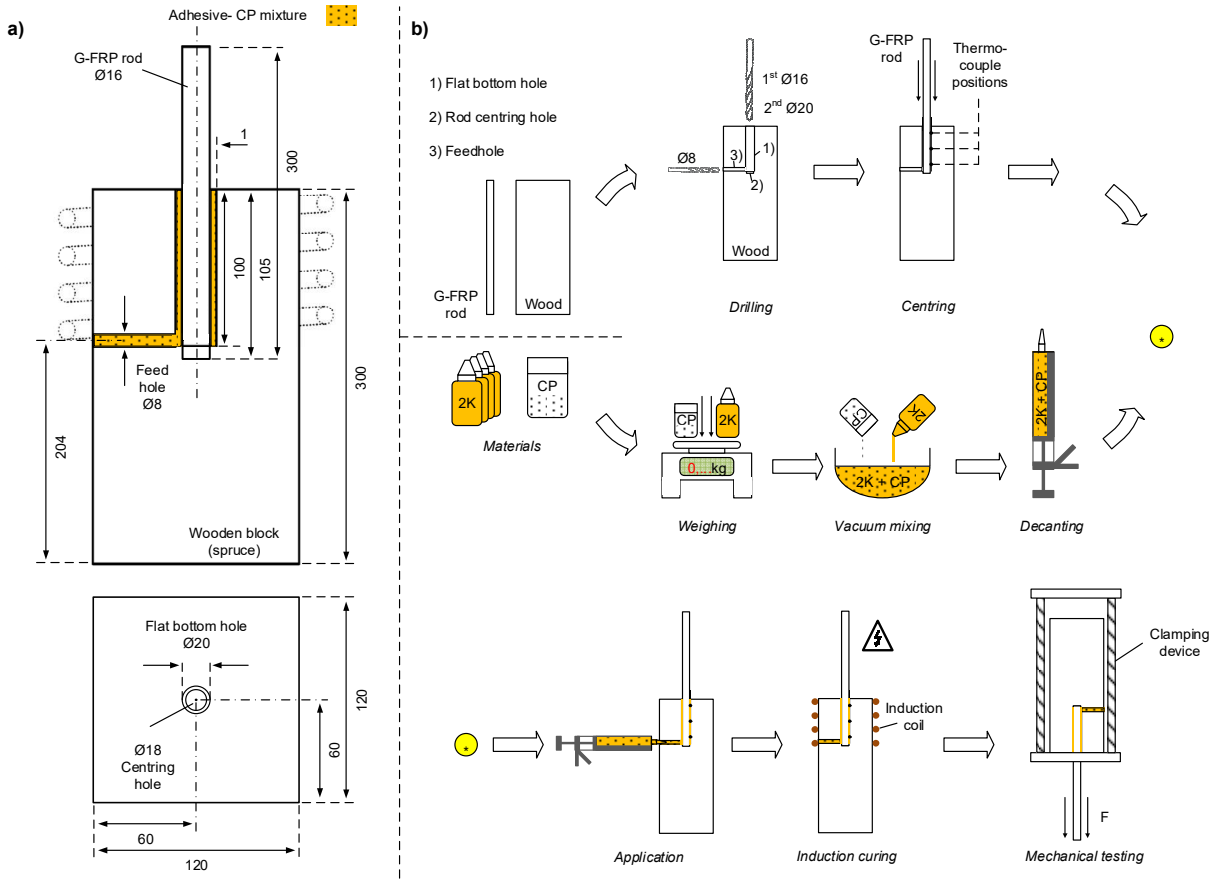


Figure 23: a) Schematic illustration of GiR geometry, dimensions in mm including tolerance of ± 0.1 mm (own representation), b) GiR manufacturing process for CP-cured and mechanically tested GiR at RT conditions (own representation)

Table 7: Substantial material properties of G-FRP reinforcement bars (ComBar®, Schöck Bauteile GmbH. Germany) according to manufacturer's TDS

Property according to EC2		Value
Char. value for tensile strength, f_{tk}[N/mm²]		>1000
Char. value for yield strength, f_{yk} [N/mm²]		No flowing
Rated value of yield strength, f_{yd} [N/mm²]		445
Strain under limit condition of load capacity [%]		7.4
Bending value for Pull-MoE [N/mm²]		60.000
Rated value of bond stress, f_{bc}	C20/25 [N/mm²]	2.03
	C30/37 [N/mm²]	2.33
Minimum concrete cover, c_y		d_r* + 10 mm
Density, γ (g/cm³)		2.2
Thermal conductivity, λ (W/mK)		0.7 (axial) / 0.5 (radial)
Thermal coefficient of linear expansion, α (1/K)		0.6×10^{-5} (axial) / 2.2×10^{-5} (radial)
Magnetism		No

* d_r = rod diameter

6.4.5 Single lap shear tubular joints

The second structural joint configuration investigated were G-FRP single lap shear tubular joints (SLTJ). These consisted of inner tubes with an external diameter of Ø40 mm, and outer tubes with an internal diameter of Ø42 mm; both exhibited a thickness of 3 mm. Discarding imperfections and tolerances, the combination of inner and outer tube leaves a gap of 1 mm to be filled by the adhesive. The SLTJ geometry

is sketched in Figure 24, while Table 8 summarises the main mechanical properties of the materials involved.

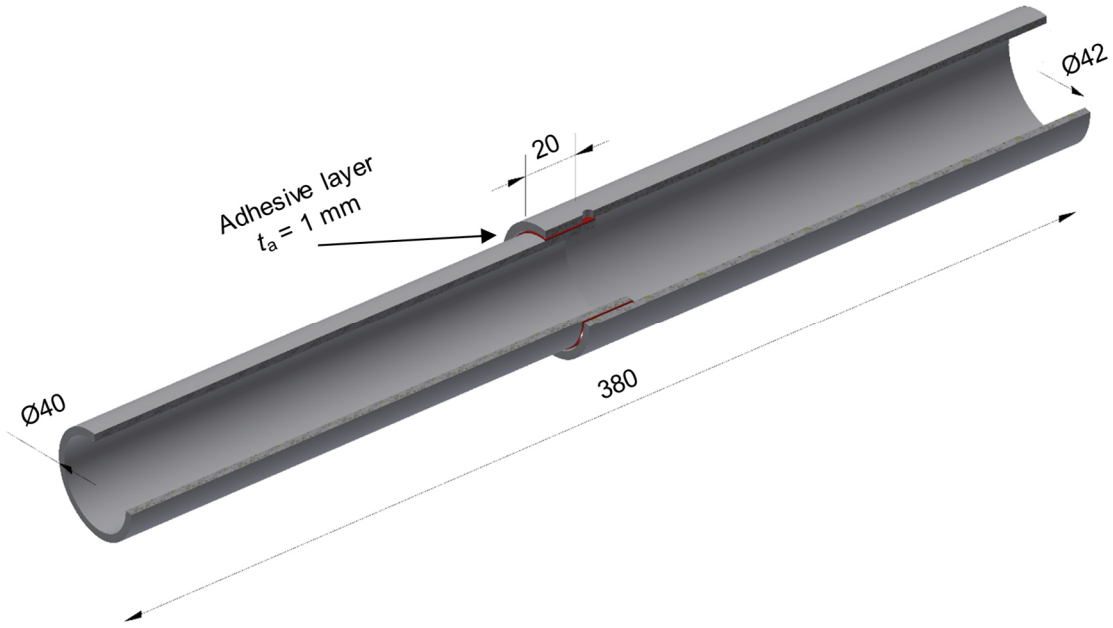


Figure 24: Sketch of SLTJ configuration made of pultruded G-FRP and investigated in publication eight (all dimensions in mm, own representation)

Table 8: Material properties of the G-FRP SLTJ according to manufacturer's TDS

Property	Glass weight [% by mass]	Tensile strength [MPa]		Tensile stiffness [GPa]		Poisson's ratio [-]		Thermal conductivity [W/m·K]		Coefficient of thermal expansion [x10^{-6} 1/K]	
		0°	90°	0°	90°	ν_{13}	ν_{23}	0°	90°	0°	90°
Value	60 ± 5	>250	>65	>22	>8	0.29	0.11	0.36	0.3	8–14	16–22

6.5 Inductive heating

6.5.1 Induction device

For all inductive heating experiments presented in this thesis, the high-frequency induction device Tru-Heat HF 5010 (Trumpf Hüttinger GmbH & Co. KG, Germany) was used. Depending on the experimental setup (coil geometry and mounted capacitors), the system is capable of providing an output up to 10 kW in a frequency range of 50–800 kHz. For all inductive heating experiments, four capacitors with a capacitance of 0.66 µF each were mounted in the system.

The complete experimental setup is illustrated in Figure 25, herein exemplarily arranged for inductive heating of SLJ. The induction system was regarded as a tool for heat generation, which influences the process on the quantitative level, i.e. how fast heating and consequently curing occurs. Accordingly, further technical developments of the utilised induction device were neglected for the time being, as these may be addressed by the induction equipment manufacturers much more efficiently.

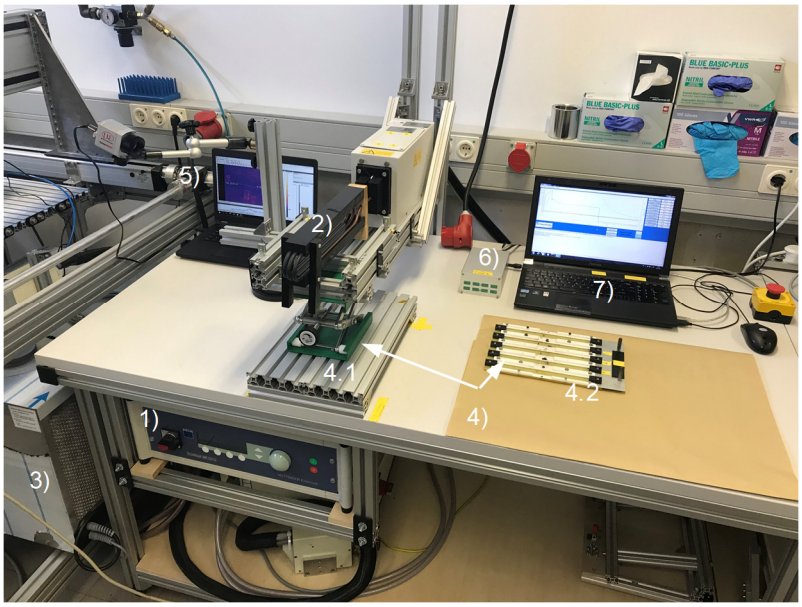


Figure 25: Experimental setup for inductive heating, herein arranged for SLJ with 1) induction device, 2) induction coil A, 3) cooling unit, 4) PTFE mould with specimen holder for positioning of SLJ, 5) IR camera with associated software, 6) temperature control unit for thermocouples and 7) associated software for temperature data evaluation

6.5.2 Induction coils, capacitors & induction parameters

Three cylindrical induction coils were used to heat the joints that were described in section 6.4. The geometric dimensions of all inductors have been illustrated in Figure 26. The coils were manufactured out of a hollow copper tube, which had an outer diameter of 8 mm and a wall thickness of 1 mm. The coil geometry was always adapted to the dimensions of the respective joint in order to achieve most homogeneous heating possible.

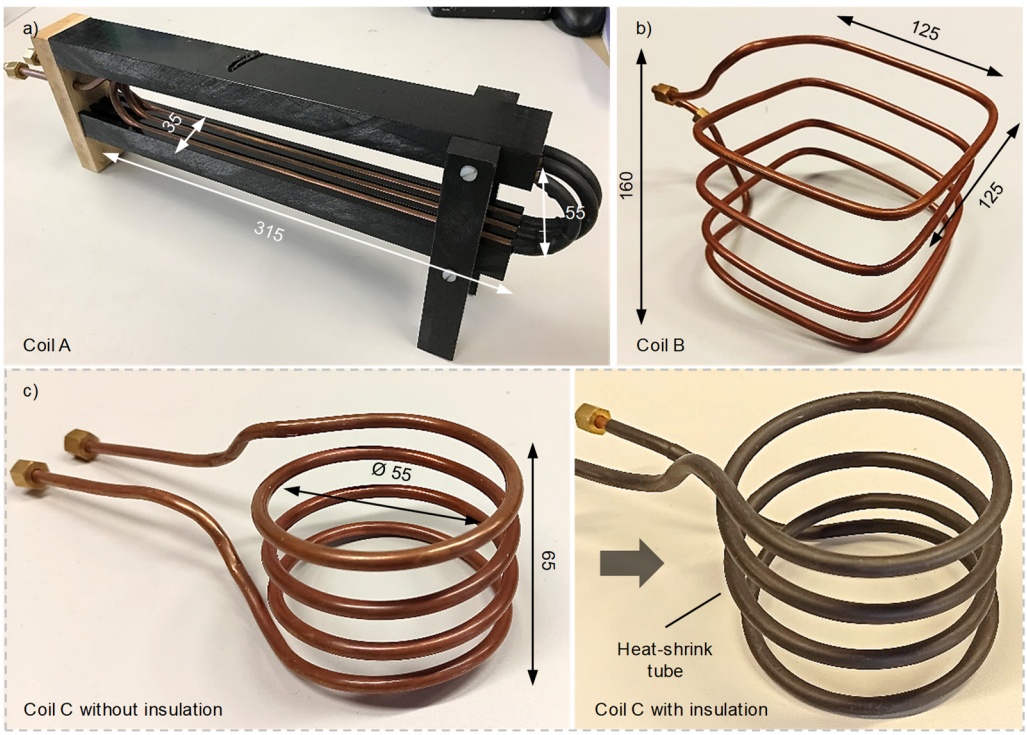


Figure 26: Hollow copper coils (Ø8 mm, wall thickness = 1 mm) utilised for inductive heating of a) AB, DMA as well as SLJ, b) GiR and c) SLTJ, dimensions in mm

During inductive heating, all coils were cooled from the inside with distilled water over an internal cooling unit (see Figure 25-3). If used under adverse environmental conditions, it is possible to insulate the copper coils by using heat-shrink tubes, thus guaranteeing safe application for operators. A representative example for an insulated coil is shown on the right of Figure 26-c. Induction parameters for a constant power level of 100 %, were listed in Table 9.

Table 9: Induction parameters of the coils shown in Figure 26 resulting for four mounted capacitors with a capacitance of 0.66 µF each, all values valid for full power (100 %)

Component	Coil*	Power *P* [kW]	Current *I* [A]	Voltage *U* [V]	Frequency *f* [kHz]
AB	A	4.8	26.3	640	134
DMA	A	4.8	26.3	640	134
GiR	B	3.8	29.4	795	120
SLJ	A	4.8	26.3	640	134
SLTJ	C	4.7	35.0	650	177

*see Figure 26

6.5.3 Temperature monitoring

Methods

Two methods for temperature monitoring were used: Type K thermocouples as well as an IR camera type thermoIMAGER TIM 160S (Micro-Epsilon Messtechnik GmbH & Co. KG, Germany). Both techniques have been exemplarily illustrated in Figure 27. The combination of a punctual (thermocouples) with surfacic (IR camera) monitoring technique opened up the possibility to draw much better conclusions about the specimens' thermal condition. The temperature data was recorded and stored in separate software tools for subsequent evaluation (see Figure 25-5, 7). Much more information can be found in the original articles.

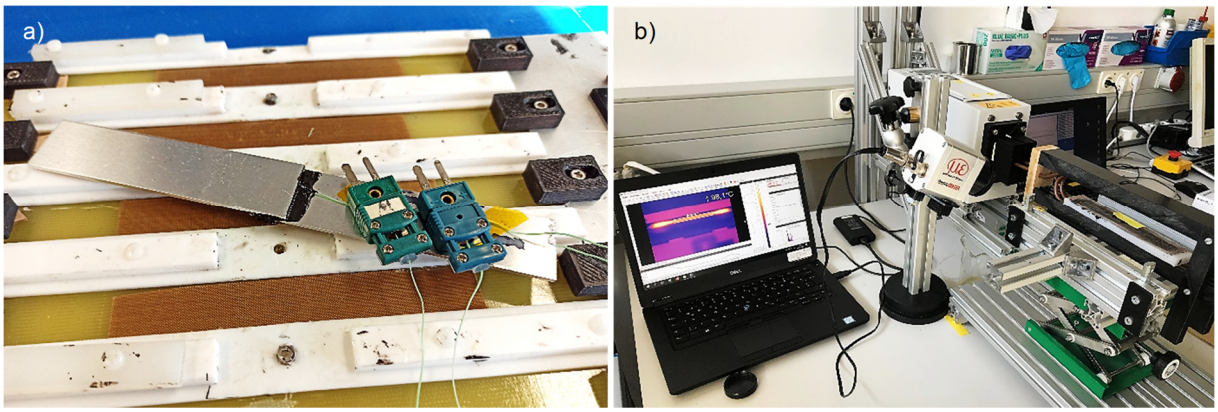


Figure 27: Applied methods for temperature monitoring with a) thermocouples of type K, herein attached to CP-bonded aluminium SLJ, and b) IR camera in operation during inductive heating of a CP-filled AB specimen

Preliminary experiments

Commercially available thermocouple wires are sensitive to EMF and thus it was not clear whether interference-free temperature monitoring could be achieved with them. Preliminary investigations on free thermocouples, and thermocpouples embedded in adhesive, were thus conducted, with the thermocouples being exposed to the EMF using coil B (Figure 28-a). The investigations revealed no interferences when the thermocouple is enclosed by adhesive, as shown by the results illustrated in Figure 28-b. In addition, numerous temperature measurements proved that the collected thermocouple readings agree very well with corresponding IR data (see e.g. 2nd or 3rd publication). In summary, it was thus concluded that the thermocouple recordings presented in the thesis can be trusted.

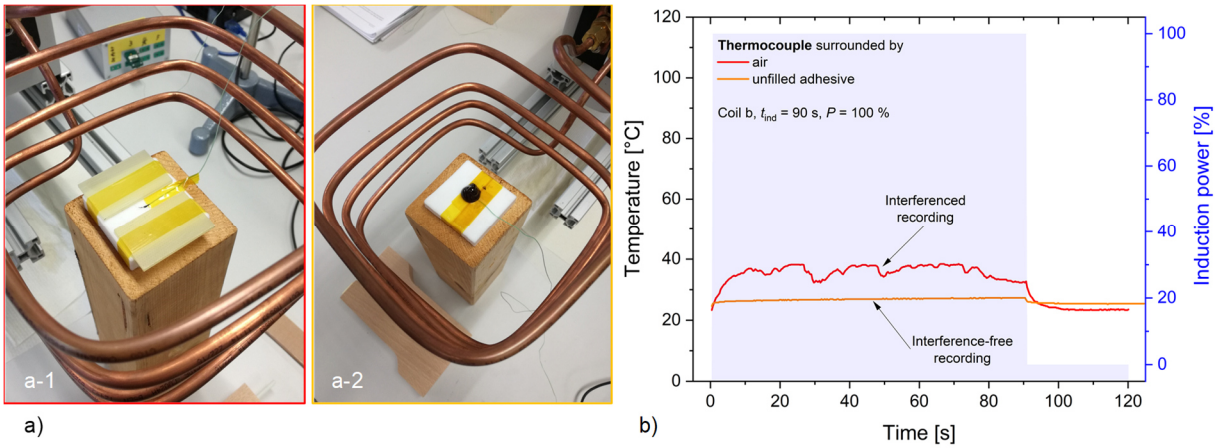


Figure 28: Representative result of the preliminary thermocouple experiment; a) Thermocouple positioned centrically within coil B and surrounded by a-1) air as well as a-2) unfilled adhesive, b) Associated thermocouple recordings (curves indicate arithmetic mean out of three repeated measurements), all experiments performed at RT

6.5.4 Inductive heating approaches

Two distinct approaches were available to inductively heat the joints. Firstly, induction power was maintained at a constant value expressed in % of the maximum power level, which itself was dependent upon coil geometry, i.e. its inductivity, as well as mounted capacitors. This approach was always used when the temperature was controlled by the embedded CP; examples thereof are the GiR experiments presented in e.g. my 4th publication. Secondly, induction power was regulated so to meet specified heating rates and temperatures, which were measured with thermocouples within the adhesive or attached to EMF-sensitive adherends (see e.g. aluminium SLJ experiments presented in the 3rd paper or preliminary experiments presented in section 6.1.2).

6.5.5 Inductive heating procedure

Save for the adhesive-CP application, the inductive heating procedure for the joint types described in section 6.4 followed a uniform procedure to maintain comparability of all findings. First, materials and experimental setup were prepared as far as possible in advance, so to minimise curing under RT conditions. The preparation phase included possible surface pre-treatments, adherend conditioning (e.g. at

low temperatures, see 8th paper), the applied measures for temperature monitoring (see section 6.5.3) as well as preparation of the induction system (coil attachment, software initialisation etc.) and the devices for mechanical testing (see section 6.6).

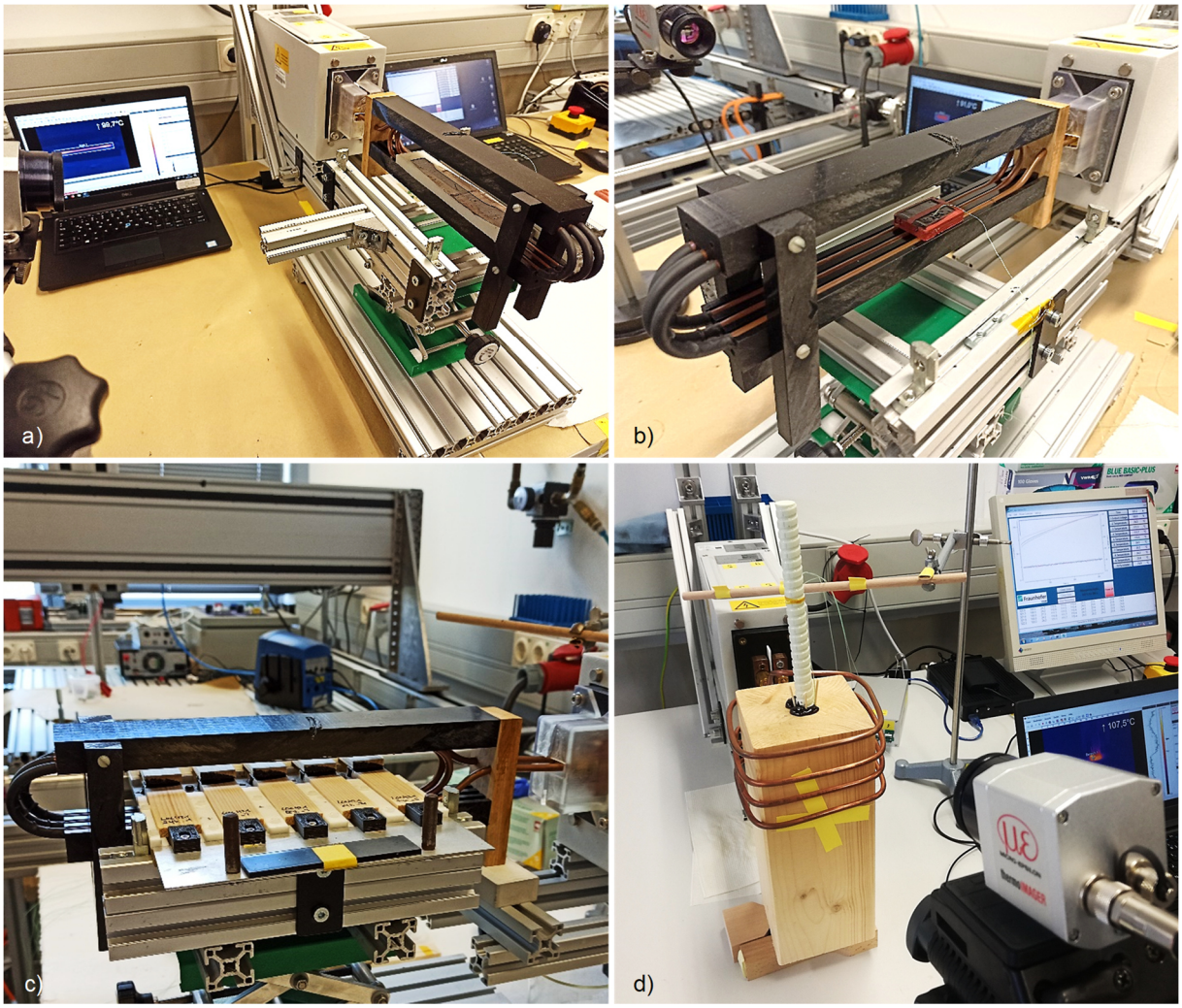


Figure 29: Examples for specimen positioning during inductive heating with CP for a) AB, b) DMA, c) wooden SLJ and d) GiR specimens, all experiments performed at RT

In detail, inductive heating was always carried out according to the following sequence: First, the adhesive-CP mixing and application procedure described in section 6.3.2 was applied. Then, the joined specimen was placed centrically within the induction coil as shown in Figure 29, the induction system was started and the specimen was cured using the CP-induced heat. Subsequently, the samples were left to cool to RT. Mechanical or thermo-mechanical testing was performed at different times after inductive heating ended (for the rationale behind this as well as the description of mechanical testing cf. section. 6.6). More detailed information regarding the inductive heating procedures can be found in the original articles included.

6.5.6 Influence of polymerisation enthalpy

Although not formally related to the presentation of materials and methods, it is important to keep in mind that two heat sources have to be considered during the induction operations. Firstly, and most evidently, the heat generated when the EMF acts on the CP; but secondly, and potentially overlooked, the reaction heat released by the exothermic polymerisation reaction.

In order to separate these distinct effects, many specimens were inductively heated twice. During the first heating cycle, both the CP-induced as well as enthalpy-induced heat contributed to monitored temperatures. After cooling, specimens were left to cure at RT for the times listed in the manufacturer's TDS, which ensured that no enthalpy remained. Subsequently, they were inductively heated a second time using the identical experimental procedure and conditions of the first cycle. Consequently, resulting temperature differences between the two heating runs can be attributed to the contribution of exothermy, respectively. Throughout the thesis, the runs are referred to as first (CP and enthalpy heating) and second heating cycle (only CP heating).

6.6 Thermo-mechanical testing

6.6.1 Differential scanning calorimetry

All differential scanning calorimetry (DSC) experiments were performed according to DIN 53765[234] using a Discovery DSC (TA Instruments, Inc., USA). Preparation of samples never exceeded a time window of 7.5 min, which was well within the pot life of all selected adhesives (cf. Table 2). Thus, almost all curing, i.e. release of polymerisation enthalpy, occurred within the DSC. For the exact measurement parameters, it is referred to the content of the original publications.

6.6.2 Dynamic mechanical analysis

All dynamic mechanical analysis (DMA) experiments were performed according to DIN EN ISO 6721[235] using a Dynamic Mechanical Analyser type 2980 (TA Instruments Inc., USA). To ensure a flat surface, each specimen was ground before being clamped into the DMA device. Furthermore, all measurements were performed using identical testing parameters: f = 1 Hz, A = 20 µm, $\mathrm{d}T/\mathrm{d}t$ = 2 K/min in a temperature range of –40 to +150 °C, thus covering usual application temperatures for civil engineering applications (roughly –20 to +60 °C). Further measurement characteristics of the DMA experiments can be found in the attached publications.

6.6.3 Quasi-static mechanical tests

Mechanical testing of the joint types presented in section 6.4 was carried out withuniversal testing machines (UTM's, Zwick|Roell GmbH & Co. KG, Germany, Figure 30). Prior to testing the SLJ overlap

length and width were measured using a caliper. For AB specimens, cross-section dimensions at gauge length (see Figure 30-a) were determined and entered in the software.

After curing was finished, each specimen was positioned centrically between the clamping jaws of the UTM. The mechanical tests were predominantely performed immediately after the specimens had cooled back to RT, which – depeding on the joints geometry – took between 15 (SLJ) and 90 min (GiR). To illustrate the evolution of the mechanical properties with longer RT conditioning, periods between the end of inductive heating and actual destructive testing were varied. The outcome of these tests is best illustrated by the experiments presented in my 3rd, for SLJ, and 8th publication, for SLTJ.

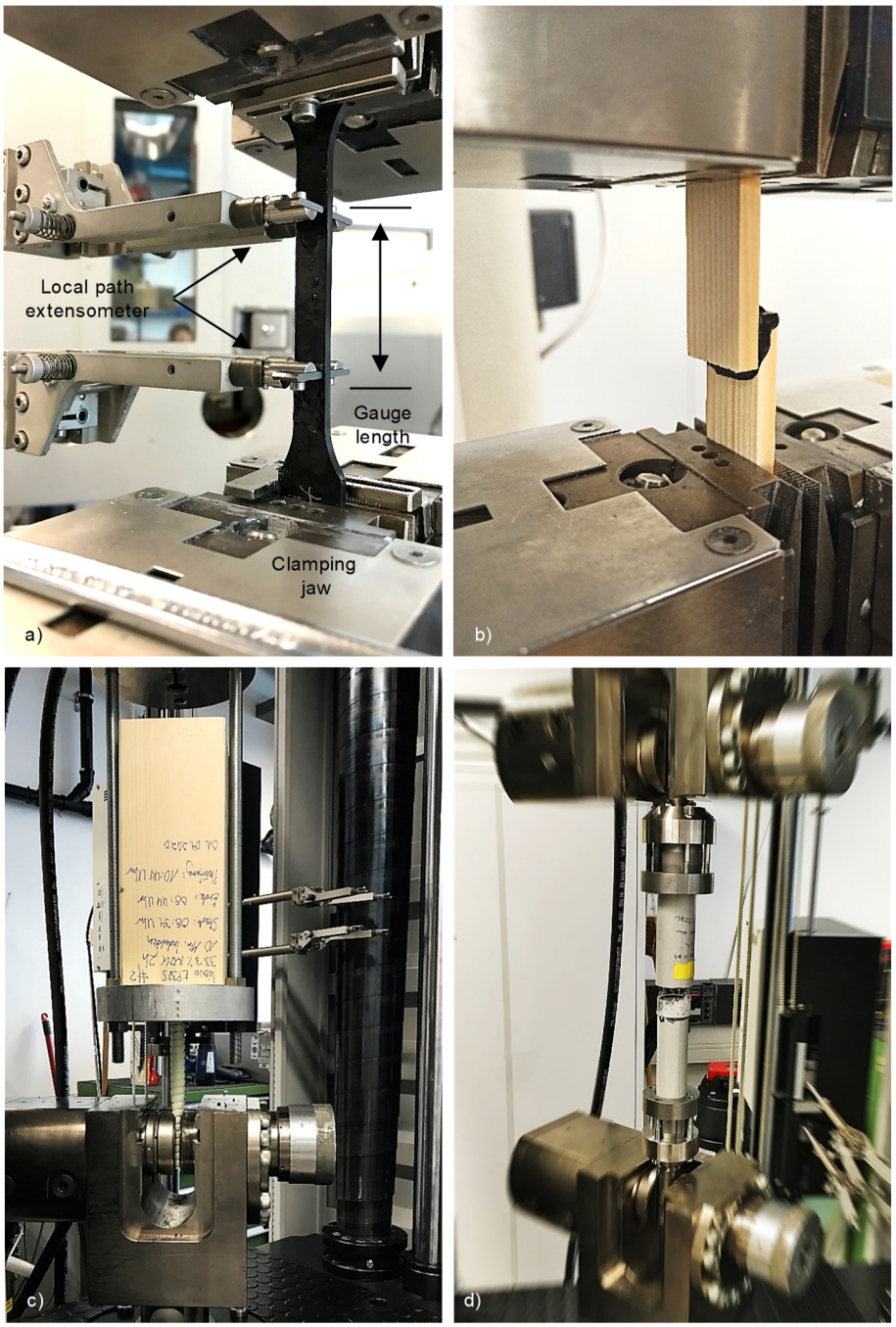


Figure 30: Exemplary illustration of setup for mechanical testing of a) AB (see section 6.4.1), b) wooden SLJ (see section 6.4.3), c) GiR (see section 6.4.4) and d) SLTJ (see section 6.4.5) herein bonded with Fi390, Zwick|Roell UTM's with 20 (SLJ), 50 (AB) or 100 kN (GiR and SLTJ) load cell, all tests performed at RT

To maintain comparability, testing speed for all series of a respective joint type to be compared was always kept identical. Depending on joint size, load cells in the range of 20–100 kN were utilised to record occurring forces. If strain data is provided, these were determined using local path extensometers as those depicted in Figure 30-a. Detailed information regarding applied testing procedures as well as testing times and speeds have been added to the respective Figures or can be found in each of the incorporated original articles of the thesis.

6.7 Statistical evaluation

Analysis of variance, or ANOVA[238], is a large group of data-analytical and structure-checking statistical procedures that allow for numerous different applications. These methods, that go essentially back to FISHER[239], have in common that they calculate variances and test variables in order to obtain information about the relationships behind data. The variance of one or more target variables is explained by the influence of one or more influencing variables (factors). The simplest form of ANOVA tests the influence of a single nominal scaled variable on an interval scaled variable by comparing the mean values of the dependent variable within the groups defined by the categories of the independent variable. Thus, ANOVA in its simplest form is an alternative to the t-test[240], which is suitable for comparisons between more than two groups.

For this thesis, ANOVA is essentially used to assert the significance of differences resulting from several experimental series, in which one parameter has been varied (one-way ANOVA). All computations were performed using the statistical software package Origin2020®, considering TUKEY'S test[241]. In essence, TUKEY'S test compares the means of a series and identifies pairwise differences greater than the expected standard error, which are then considered significant, or not. The decision being of statistical nature, its relevancy depends upon the confidence level set. For all subsequent results, a significance level of $p = 0.05$ is implied. Results are presented as plots comparing the means, from which significance, or not, can be deduced.

6.8 Numerical modelling

For all numerical modelling operations presented in this thesis, the FE software package Academic 2019 R1® was used. The numerical modelling was scripted in the Ansys Parametric Design Language (APDL) and aimed to predict the curing progress of two exemplary adhesives (Fi390 and We32) during accelerated curing with CP using the exemplary application of GiR (cf. section 6.4.4). For that, the curing kinetics of the two adhesives was linked to a direct transient thermal analysis, which foot upon experimentally determined heat loads. The model considers both heat released by exothermy, H_{cure}, as well as heat originating from the hot CP, H_{cp}. Furthermore, the Curie effect (switch-off at T_c, see section 6.2.2) was integrated on the numerical level. A detailed description of the modelling approach and the underlying analytical work can be found in the original article of my 5th publication, for which reason this section has been kept short.

Part III: Scientific content of the publications

7 Influences of Curie particles on curing kinetics

The following chapter summarises the findings extensively presented in VOß & VALLÉE, "*Accelerated curing of G-FRP rods glued into timber by means of inductive heating – Influences of curing kinetics*", published in THE JOURNAL OF ADHESION (2021):1-39. Any information not included in this summary, and not covered by the introduction of this thesis, can be found in the paper cited above.

Abstract > *This paper concentrated on the identification of CP-induced changes in the polymerisation course using several 2K structural adhesives. It significantly contributed to the predictable design of CP-curing through the quantification of the polymerisation progress so to develop design procedures for practical application. For that, kinetic models were developed considering four 2K adhesives, which allow for the prediction of the curing degree in dependency of CP content and curing temperature. For that, an in-depth DSC characterisation was performed on unfilled and CP-filled mixes. The scientific approach allowed for a much better interpretation of the heating behaviour, highlighting in particular the contribution of polymerisation enthalpy for adhesive curing, and by extension of the whole induction process.*

7.1 Objectives

To provide a clearer overview, the roadmap of the paper has been illustrated in Figure 31. Three main research objectives were defined:

- Identification of possible influences on the curing kinetics caused by the addition of the CP; this question has not yet been addressed in the literature.
- Development of suitable kinetic models for the description of the curing degree, α, as a function of curing temperature, T_{cure}, and CP content, $c_{particle}$; this has not yet been reported in literature.
- Separation of the proper induction heating resulting from the CP from that of the enthalpy release of the adhesives; this has not yet been reported in literature.

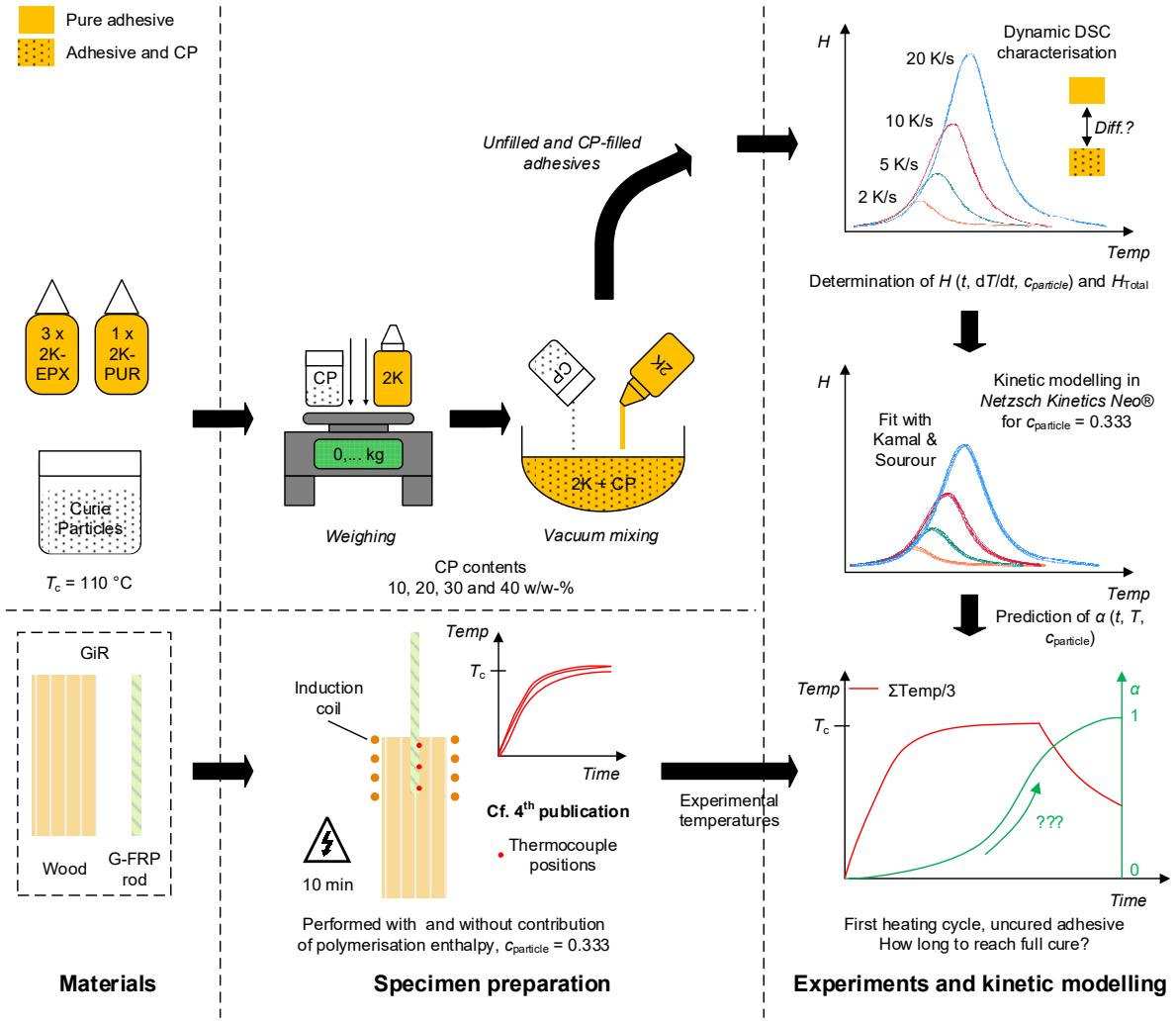


Figure 31: Roadmap of the 1st publication

7.2 Most important findings

The most essential findings of the paper were:

- An almost linear correlation between the CP content, $c_{particle}$, and the total amount of enthalpy, H_{Total}, was measured. No additional catalytic effects were identified after adding the CP, which

suggests that the polymerisation was not altered at the chemical level, if compared to that of the unfilled adhesives.

- Kinetic modelling, including the enthalpy-related effects described above, was combined with accelerating curing for the first time. The scientific approach is the basis to design efficient manufacturing processes, including the determination of process duration.
- The heating behaviour of the CP-bonded joint is largely determined by polymerisation enthalpy, rate of heat release during curing, polymerisation onset, as well as the adhesive's material properties, most importantly heat capacity and thermal conductivity.

8 Influences of Curie particles on bulk adhesive characteristics

The following chapter summarises the findings extensively presented in VOß & VALLÉE, "*Effects of Curie particle induced accelerated curing on thermo-mechanical performance of 2K structural adhesives – Part I: Bulk properties*", published in THE JOURNAL OF ADHESION (2021):1-42. Any information not included in this summary, and not covered by the introduction of this thesis, can be found in the paper cited above.

Abstract > *After CP-related effects on the polymerisation course had been uncovered, the investigation focus was raised towards thermo-mechanical adhesive bulk characteristics, most importantly cohesive strength, stiffness as well as glass transition temperature. For that, a detailed characterisation using RT-, oven- and inductively cured AB and DMA specimens with and without added CP was carried out for two exemplary adhesives (EPX and PUR). The paper contributed significantly to the understanding of the process as well as its capability to produce intact joints, as observed changes at the level of the bulk are unextricably linked to the mechanical behaviour of loaded joints. The results can therefore be understood as a prerequisite for all subsequently presented investigations on the level of joints.*

8.1 Objectives

The scientific approach of the publication has been illustrated in Figure 32. Three central research topics were defined:

- Upscaling the CP-supported accelerated curing with regard to size and shape of the samples, and ensuring the reproducibility of the resulting heating behaviour.
- Determination of influences resulting from the addition of the CP on the adhesive bulk properties after RT-curing; this has not yet been reported in literature.
- Determination of influences resulting from the addition of the CP on the adhesive bulk properties after induction-curing; this has not yet been reported in literature.

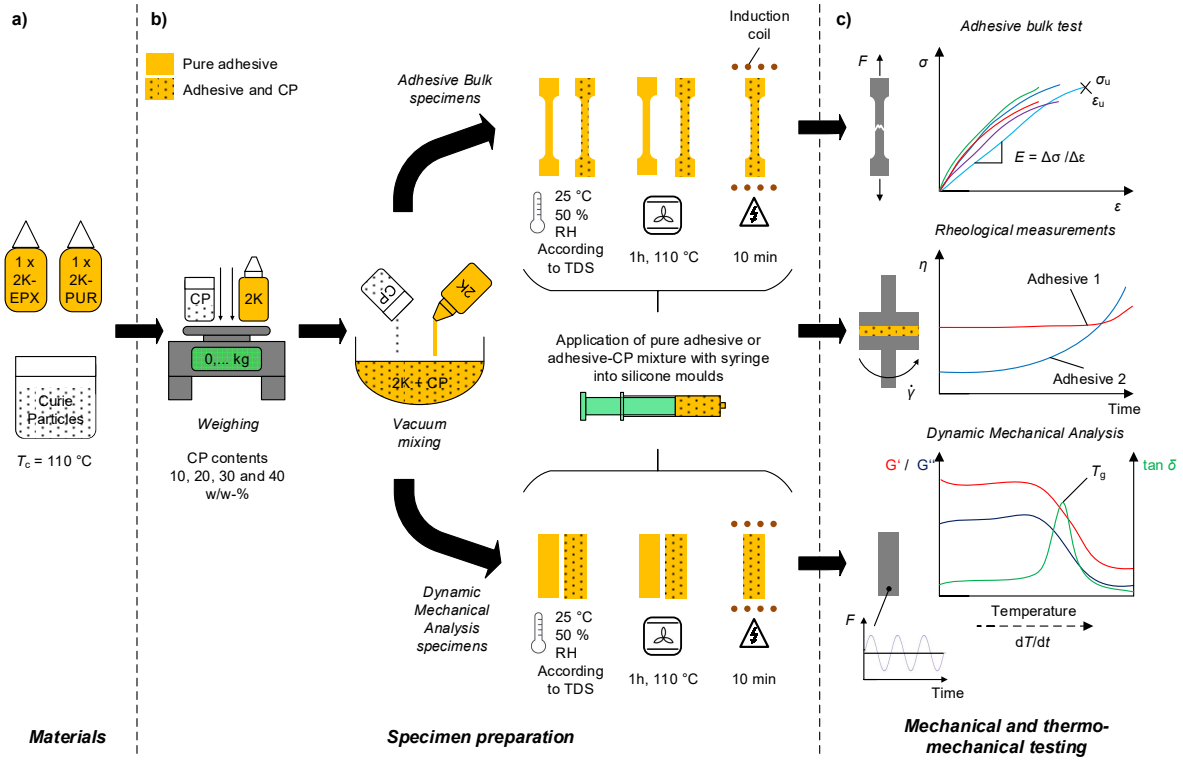


Figure 32: Roadmap of the 2nd publication

8.2 Most important findings

After evaluation of all data, the following main findings could be made:

- All CP-filled AB specimens showed a homogeneous CP distribution over the specimen's cross section and superficial damages, e.g. extensive bubble formation or burned parts in the polymer matrix, originating from CP-curing could not be identified.
- Even though it has been shown that CP-induced heat generation is limited by the Curie temperature, T_c, heat released from polymerisation can lead to an uncontrolled overheating of the adhesive. This effect gets more important the thicker the adhesive layer, the more enthalpy is contained and the earlier enthalpy is released.

- Mechanical properties of the adhesives are fundamentally altered by both the presence of CP as well as the induction curing at elevated temperatures. While the CP reduce tensile strength, curing at elevated temperatures compensates for that. In addition, the CP increase the adhesives stiffness, which was confirmed both by the AB as well as the DMA experiments.

- Curing with CP at elevated temperatures leads to a denser cross-linking and thus highered T_g, with observed increases of 15 °C, for the 2K-EPX, as well as 5 °C, for the 2K-PUR, in comparison to RT-cured references. With regard to practical implementation, this result can be considered as very positive, as CP-cured joints are most likely capable of tolerating higher application temperatures until significant reductions in strength occur.

- If the 2K adhesive is not fully cured within the induction process, e.g. when induction time is too short or curing temperatures are too low, thermo-mechanical properties equal to those of the references are reached due to 2K post-curing at RT. An exception of this represent resulting stiffnesses, which are highered due to the presence of stiff CP embedded within the way softer polymer matrix.

9 Influences of Curie particles on small-scale joints

The following chapter summarises the findings extensively presented in VOß & VALLÉE, "*Effects of Curie particle induced accelerated curing on thermo-mechanical performance of 2K structural adhesives – Part II: Lap shear properties*" published in THE JOURNAL OF ADHESION (2021):1-51. Any information not included in this summary, and not covered by the introduction of this thesis, can be found in the paper cited above.

Abstract > *In the 3rd paper of the thesis, investigations were scaled up to the level of joints for the first time. For this purpose, small-scale SLJ produced with different substrate materials and CP contents served as the exemplary joint type. To comply with the findings of the 2nd paper, the very same adhesives used during the bulk investigations (EPX and PUR) were considered on the lap shear scale. The experiments contributed significantly to the content of the thesis, as – for the first time – a systematic experimental campaign aiming to deliver insights into changes of compound characteristics originating from CP-curing was performed. In addition, further influencing factors concerning the heating of multiple joints during the same induction operation as well as the impact of subsequent RT-conditioning on the mechanical joint performance were demonstrated.*

9.1 Objectives

In compliance with the subsequent publications, the scientific content of the paper has been visualised in Figure 33. The following research goals were defined:

- Identification of conditions needed to achieve homogeneous heating between multiple joints cured within the same induction operation; this has not yet been reported in literature.
- Systematic determination of influences resulting from the addition of the CP on lap-shear properties after RT-curing; this has not yet been reported in literature.
- Systematic determination of influences resulting from the addition of the CP on lap-shear properties after inductive-curing; this has not yet been reported in literature.

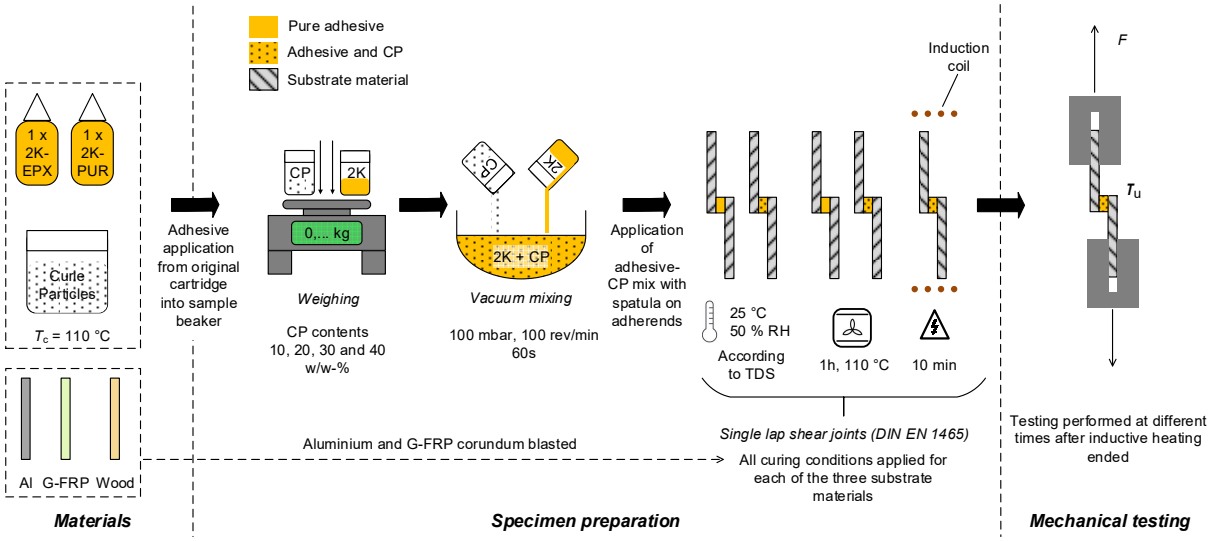


Figure 33: Roadmap of the 3rd publication

9.2 Most important findings

The most important findings of the publication are:

- The boundary conditions that have to be kept identical for most homogeneous heating between multiple joints have been identified: CP content, adhesive, coupling distance, joint geometry, adhesive layer thickness, heat dissipation from the adhesive layer towards other joint parts, i.e. adherend materials.
- Lap shear properties are changed fundamentally. Two counteracting effects were identified: Deterioration of lap shear characteristics attributable to the addition of CP, and improvements as a result of curing at elevated temperatures. These observations are consistent with the findings from the 2nd paper (bulk adhesive).
- It is strongly assumed that build-up of stiffness and strength does not occur simultaneously. It appears that stiffness development lags behind that of strength. This may be the reason for higher joint strengths right after the end of induction, if compared to samples additionally cured at RT after induction.

10 Influences of Curie particles on large-scale joints

The following chapter summarises the findings extensively presented in VOß & VALLÉE "*Accelerated curing of G-FRP rods glued into timber by means of inductive heating using Curie-particles – large-scale experiments at room temperature*", published in THE JOURNAL OF ADHESION (2021):1-29. Any information not included in this summary, and not covered by the introduction of this thesis, can be found in the paper cited above.

Abstract > *To complete the upscaling approach, investigations in the 4th paper were raised towards the application level using the example of inductively cured GiR bonded with four 2K structural adhesives. In order to enable cross-comparisons between the publications, the experimental campaign was designed in compliance with the testing plans on the bulk (2nd paper) and lap shear scale (3rd paper). The publication contributed significantly to the thesis since – for the first time – principal functionality for large joints close to application could be proven by systematic experiments with extensive mechanical testing. The GiR's fracture behaviour could be explained by the standardised experiments presented in the previos publications and scaling effects, especially with regard to the heating behaviour, were revealed.*

10.1 Objectives

This upscaling has been demonstrated for a series of GiR connections briefly presented in section 6.4.4. The objectives of this study were the following:

- Deliver a proof of feasibility of the CP-curing technique to enable curing of large components by a systematic and extensive experimental study; this has not yet been reported in literature.
- Demonstration of possible scaling effects, in particular with regard to the enthalpy originating from the adhesives and the thermal equilibrium defined by either the thermal properties of the joint's the boundary conditions (thermal capacity and transmissivity); this has not yet been reported in literature.

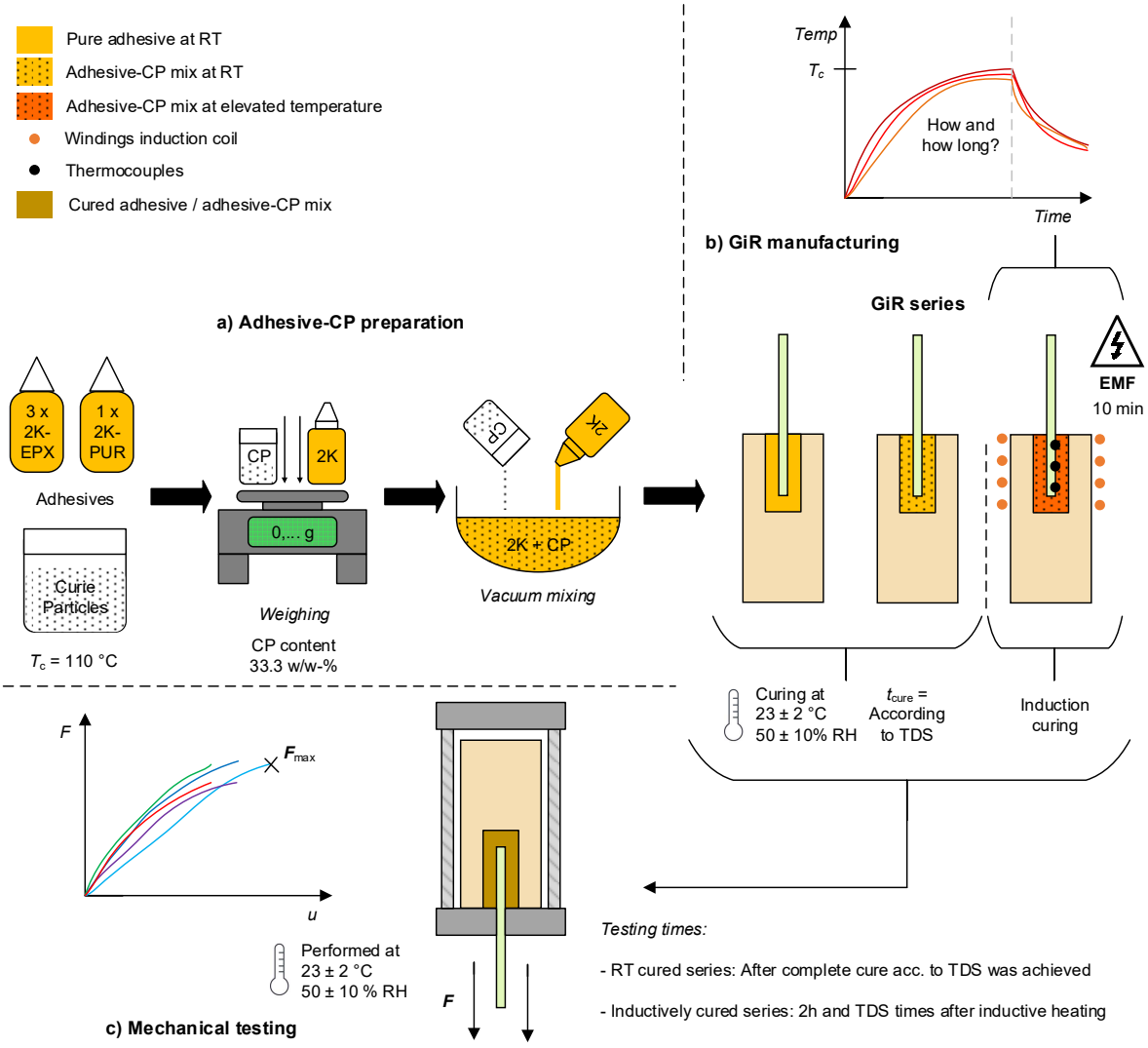


Figure 34: Roadmap of the 4th publication

10.2 Most important findings

Inductive curing with CP was successfully upscaled to the level of large-scale bonded components embodied by GiR using the very same four adhesive previously investigated and characterised in depth (cf.

1st paper). Upscaling revealed differences with regard to considerations at the smaller scale. Most importantly the significance of the thermal intertia provided by much larger joints; this allows the cooling phase to be used as an integral part of the curing process. This, however, may lead to further differentiation between the adhesives due to their inherent curing kinetics, with some being able to cure fully within the proper induction time, and others not. Additionally, potential interference of moisture entrapped in the wood and released by the generated heat have been identified, which affected the moisture-sensitive polyurethane-bonded GiR. Summing up, the most important findings were as follows:

- The experimental evidence that CP-curing can accelerate the cure of 2K adhesives at a large component level has been provided. A relatively homogeneous heating behaviour along the adhesive layer was achieved. For the epoxy adhesives, failure loads and fracture patterns were similar to the RT-cured reference series.
- The GiR experiments confirmed the prior observations and characterisation made at the level of bulk adhesive (2nd paper) and small-scale SLJ (3rd paper). Parameters that significantly influenced the large-scale induction heating were the curing kinetics of the adhesives, the joint geometry and material properties of their components. Compared to the small-scale investigations, thermal intertia played a much more significant role.
- The much larger GiR also allowed to identify issues related to moisture release, which negatively impacted the strength of the joints involving the moisture-senstitive 2K-PUR (LP421). Although already investigated at small-scale, the magnitude of the strength reduction, and its unfolding over longer times (10 days), was unexpected. It thus requires a differianted approach when using polyurethanes for heat-based accelerated curing of joints involving wood.

11 Numerical modelling of induction curing with Curie particles – Model building

The following chapter summarises the findings extensively presented in VOß, KAUFMANN & VALLÉE "*Curie-supported accelerated curing by means of inductive heating – Part I: Model building*", published in THE JOURNAL OF ADHESION (2021):1-43. Any information not included in this summary, and not covered by the introduction of this thesis, can be found in the paper cited above.

Abstract > *The experiments on the different joint scales proved that CP-induced accelerated curing represents a highly complex process with various conditions influencing the adhesives heating and consequently curing behaviour. Up to now, induction times needed to achieve full cure can thus only be determined through costly preliminary experiments. To contribute for a more efficient and controllable bonding process, the 5th paper of the thesis aimed at developing a numerical model based on the Finite Element Method (FEM) and capable of predicting the curing degree in dependency of curing temperature profiles and CP content. For that, curing kinetics of two kinetically different 2K epoxy adhesives were linked to a transient heat flow simulation, which was based upon experimentally determined heat loads. The paper focused on presenting preliminary experimental work as well as analytical methods implemented for the numerical modelling.*

11.1 Objectives

The proposed curing method has, to the knowledge of the author, never been modelled numerically as a whole. Accordingly, the paper aimed at providing a first approach by coupling the adhesives' curing kinetics (cf. 1st paper) with a transient heat flow analysis. In this paper, preliminary experimental work as well as analytical and numerical modelling approaches were presented, and implemented into a functional FEA code. For that, the procedure illustrated in Figure 35 has been applied. The following objectives were defined:

- Development of a numerical model capable of predicting the curing degree, α, in dependency of curing temperature, T_{cure}, and CP content, $c_{particle}$, using the exemplary application of GiR; this has not yet been reported in literature.
- Integration of the Curie effect as well as release of polymerisation heat into the FEA; this has not yet been reported in literature.

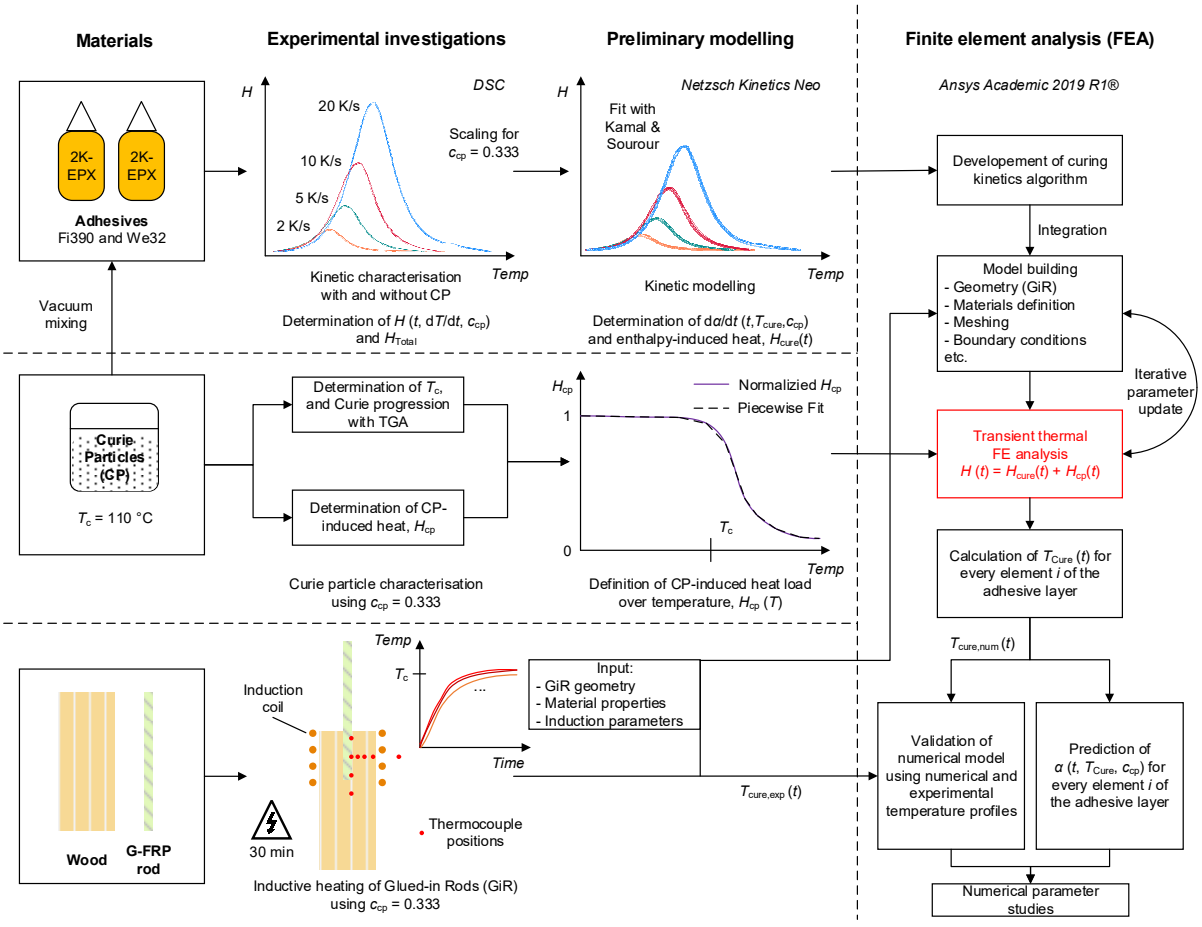


Figure 35: Roadmap of the 5th publication

11.2 Conclusion

The paper presented an approach to describe the multiphysics problem of CP-curing in a numerical model based upon the Finite Element Method (FEM). The FEA was used to link curing kinetics with a

transient thermal simulation. Temperature development in the adhesive-CP mixture was attributed to two effects: Firstly, heat introduced by inductively heated CP—including the Curie effect ('switch-off' at T_c). Secondly, heat originating from the release of polymerisation enthalpy. The general functionality of the FEA was demonstrated using large-scale GiR previously experimentally investigated in the 4th publication. The following important conclusions can be drawn:

- The developed algorithm delivers a realistic impression of temperatures originating from CP-curing. In addition, the Curie effect ('switch-off' at T_c) was successfully integrated into the FEA.
- The numerical model allows for better insights regarding the relationship between curing kinetics and heating behaviour, including delayed curing, on the level of large joints.
- In addition, the curing progress, $\alpha(t)$, can be predicted in dependency of adhesive-CP temperatures, opening up the possibility to determine induction times needed to achieve full cure for different boundary conditions numerically.

12 Numerical modelling of induction curing with Curie particles – Validation and numerical studies

The following chapter summarises the findings extensively presented in VOß, KAUFMANN & VALLÉE "*Curie–supported accelerated curing by means of inductive heating – Part II: Validation and numerical studies*", published in THE JOURNAL OF ADHESION (2021):1-30. Any information not included in this summary, and not covered by the introduction of this thesis, can be found in the paper cited above.

Abstract > *In the second part of the numerical modelling, the developed FEA presented in the 5th paper was first validated using extended GiR inductive heating experiments bonded with the adhesives, which were kinetically integrated into the simulation script in the previous paper. Afterwards, the validated model was used to perform various numerical parameter studies, highlighting capabilities of the FEA for the design of more predictable CP-curing operations. The paper significantly contributed to the content of the thesis as it offered the opportunity to sort the boundary conditions that contribute to the heating behaviour of CP-cured joints according to their importance. In addition, many effects previously observed on the experimental level, including enthalpy-induced overheating, could be confirmed by the simulations. Based on these promising results, practical implementation of the process is much easier to realise.*

12.1 Objectives

Since the model building was presented in the previous paper (see section 11), the present one focused on validation and application of the newly developed FEA. The scope of the publication has been visualised in Figure 36. The following research goals were defined:

- Firstly, a suitable experimental setup for CP-curing of GiR was derived, so to validate the numerical model; it is the first time that a numerical model of this kind has been validated that deeply.
- Secondly, the validated model was used to identify parameters important for the heating behaviour of CP-cured joints, and highlights its potential for the design of more efficient CP-bonding processes; this has not yet been reported in literature.

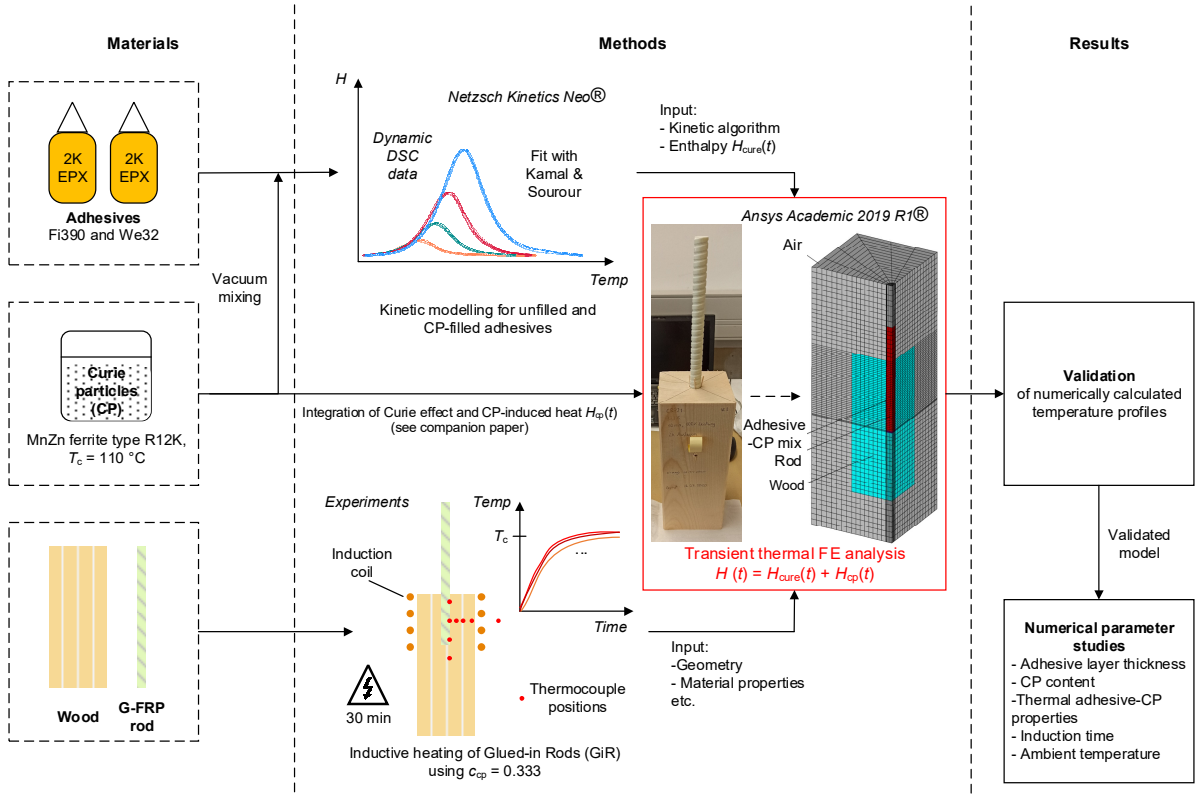


Figure 36: Roadmap of the 6th publication

12.2 Most important findings

After evaluation of all numerical data, and comparison to the experimental ones, the following important findings could be outlined from the results:

- The model validation was successfully carried out using thermocouple data recorded during CP-heating at various locations of the GiR, which showed very good qualitative – and also mostly also quantitative – agreement with the numerical predictions.

- It can thus be concluded that underlying experimental and analytical approaches for segregating the contributions resulting from the induction, H_{cp}, and exothermy, H_{cure}, were correctly implemented, and proved to be suitable for a realistic representation of the process; to the knowledge of the author, this is the first time that related effects have been illustrated that clearly.

- The numerical parameter studies made clear that almost all investigated parameters exhibit an influence on resulting temperatures. While adhesive layer thickness, CP content, induction time, and GiR starting temperature have a strong influence, heat capacity and thermal conductivity of adhesive-CP mixture have a minor one; this has not yet been reported in literature.

- The influence of the polymerisation enthalpy proved to be very important, its significance increases with the thickness of the adhesive layer, duration of the induction time and magnitude of the curing temperature of the GiR; to the knowledge of the author, this is the first time that related effects have been illustrated that clearly.

13 Low-temperature curing with Curie particles

The following chapter summarises the findings extensively presented in VOß, EVERS & VALLÉE "*Low-temperature curing of adhesives – Large-scale experiments*", published in THE JOURNAL OF ADHESION (2022):1-36; Any information not included in this summary, and not covered by the introduction of this thesis, can be found in the paper cited above.

Abstract > *In contrast to mechanical joining techniques, which can be manufactured under any ambient conditions, adhesively bonded joints always require some minimum temperatures, typically above +10 °C, in order to cure safely. To overcome these restrictions, the 7th paper of the thesis extended the inductive heating operations towards low-temperature curing using three 2K adhesives. For that, the GiR experiments performed at +23 °C and presented in the 4th publication were supplemented by additional GiR series conditioned at low temperatures (–10 and –30 °C) prior inductive heating. In accordance with the testing plan of the 4th paper, thermal and mechanical aspects of the low-temperature experiments were in the centre of research to enable cross-comparisons between the publications. The paper contributed significantly to the thesis' content since – for the first time – capability of CP-curing to enable curing under thermal conditions currently not intended by construction authorities could successfully be proven. In addition, changing boundary conditions in comparison to induction curing starting at RT were identified and guidelines for their practical consideration have been formulated.*

13.1 Objectives

The roadmap of this 7th paper has been illustrated in Figure 37, with the following scientific objectives being defined:

- Firstly, evidence about general suitability of the CP-curing technique to enable curing of 2K adhesives under low temperatures should be provided; this has not yet been documented in literature.
- Secondly, conditions that have to be observed by practitioneers for proper application of the process should be revealed and summarised; these have not yet been reported in literature.

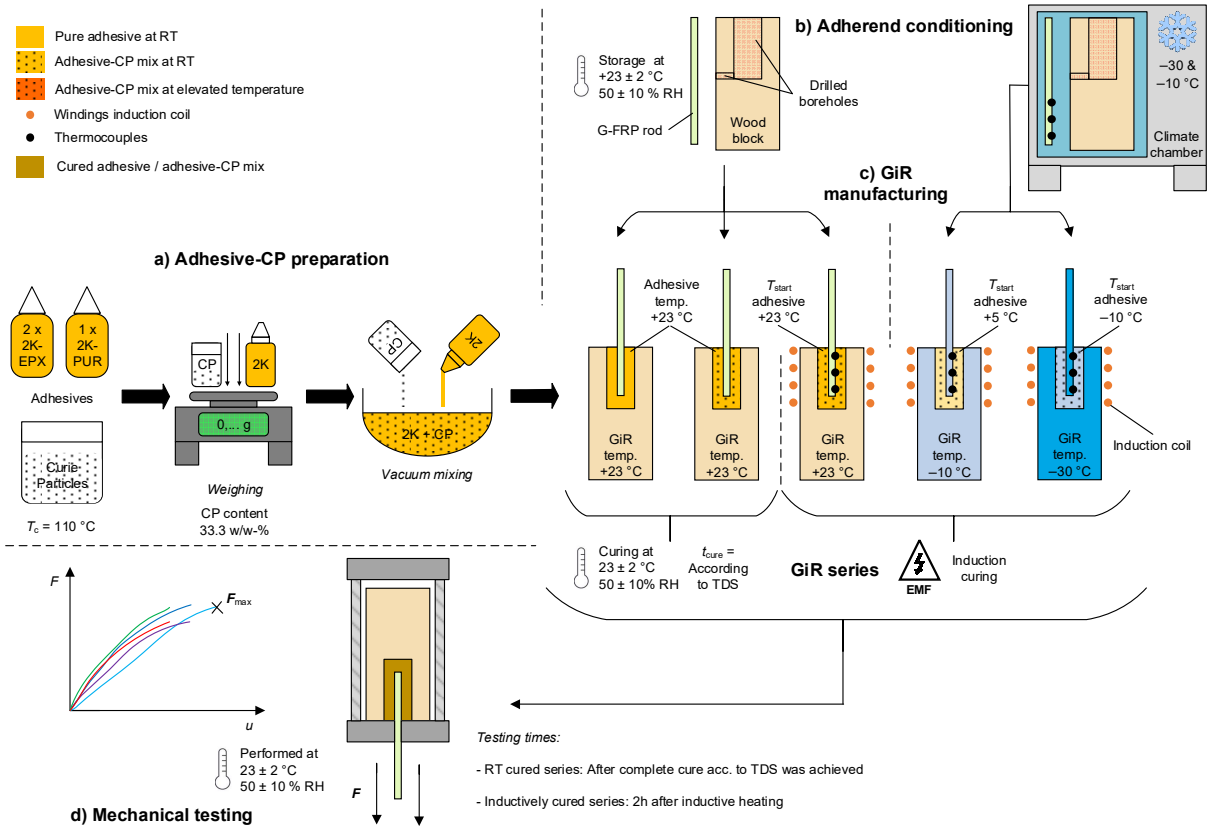


Figure 37: Roadmap of the 7th publication

13.2 Most important findings

The publication revealed the following important relations:

- Inductive CP-based curing of structural adhesives can be achieved under adverse environmental conditions, which shortens curing times from many days to minutes.
- Under lower temperatures, longer induction times may be needed. However, the required extension of the induction time only amounted to 2.5 min (from 10 to 12.5 min) and was only necessary for very low GiR temperatures of –30 °C.
- All epoxy-bonded GiR exhibited fracture patterns and failure loads indistinguishable to the unfilled reference sets cured at RT for many days – a very promising result.

- In contrast to the epoxy adhesives, failure loads of polyurethane-bonded GiR starting from low temperatures were reduced to ~50–75 % of the reference values. This was accompanied by a change of the observable fracture pattern from wood tear-out (references and CP-curing starting at RT) to mixed adhesion / cohesion failure (CP-cured GiR conditioned at –10 °C) as well as mixed adhesion / substrate failure (CP-cured GiR conditioned at –30 °C). It is strongly suspected that the deteriorated mechanical performance can be traced back to moisture settlement on the cold adherends.

14 Tranferability to another application context

The following chapter summarises the findings extensively presented in in VOß & HAUPT "*Accelerated curing of adhesively bonded G-FRP tube connections — Part I: Experiments*", published in COMPOSITE STRUCTURES (2021):1-16; Any information not included in this summary, and not covered by the introduction of this thesis, can be found in the paper cited above.

Abstract > *To further increase practical relevance of the thesis, the inductive heating operations were extended towards a second, independent, practical application: SLTJ made of G-FRP and bonded with two exemplary adhesives (EPX and PUR). These were assembled, cured following different protocols, and subjected to tensile tests. During the process, curing temperatures were monitored, and mechanical testing resulted in equal or higher shear strengths, if compared to reference sets. These were accompanied by a reduction in curing time by the factor of 100 for the EPX and 1400 for the PUR. The paper contributed significantly to the content of the thesis since transferability of the process to another application context could successfully be achieved by using the results of all previously presented papers. The study thus provided indirect validation of many previous findings and delivered extended insights into various conditions such as e.g. the impact of subsequent RT-conditioning on the mechanical joint performance.*

14.1 Objectives

In accordance with the previously presented papers, the roadmap of the publication has been illustrated in Figure 38. The objectives of this paper, as highlighted by the abstract, were the following:

- The design procedure developed throughout the previous papers was validated; this has not yet been done in literature.
- Based upon the previous experience, a novel application case could be planned in advance, including the preliminary steps needed.
- The previous findings obtained on the GiR, including on the specific substrates, have been confirmed on another geometry involving other materials, and much lower CP contents (e.g. the effect of induction curing on mechanical properties, heating behaviour, and subsequent post-curing).

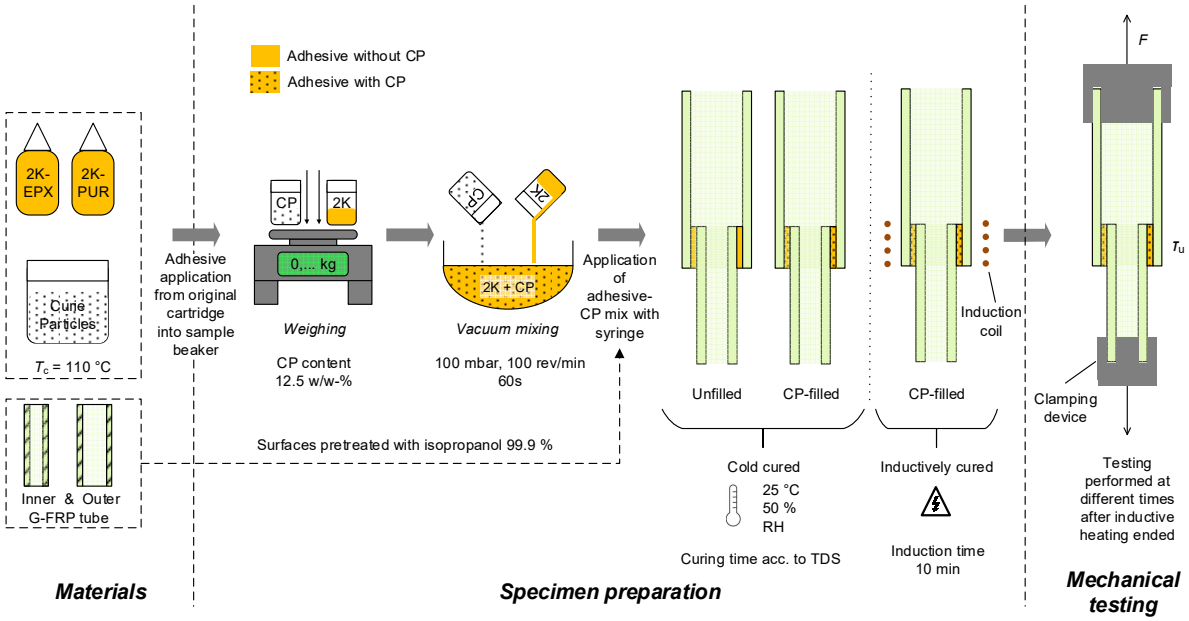


Figure 38: Roadmap of the 8th publication

14.2 Most important findings

After evaluation of all data, the following important conclusions were drawn:

- It proved possible to plan an accelerated curing process beforehand, based upon the experience gathered throughout all previous publications.
- Manufacturing tolerances have to be observed properly as temperature differences of 5–7 °C along the tube perimeter could be traced back to missalignement of the tubes, i.e. differences in adhesive layer thickness / enthalpy release.
- Failure occurred by tearing-off the G-FRP. The adhesives did thus not limit joint strength, but rather the substrate. Accordingly, joint capacity proved almost independent on the adhesive type, and the condition under which curing occurred.

- The influence of subsequent RT-conditioning on mechanical joint performance, which were already observed on level of SLJ (3rd publication) could be confirmed on a more complex joint geometry.
- The experimental results presented show that thanks to induction heating, significant reductions in curing times of SLTJ are possible even when comparatively few CP are used (~3 vol-%).

Part IV: Discussion, assessment of achievements, summary & outlook

15 Discussion

Building on the previous part of the thesis, the scientific outcome of the dissertation is discussed more deeply in the present part. For that, the discussion was guided along the overarching scientific goal and the secondary objectives formulated in chapter 4, which were:

Scientific goal:

'Design of the accelerated curing by inductively heated Curie particles as a process'

Secondary objectives:

- *1st objective*: Providing an inductive heating process without external temperature control by using CP.
- *2nd objective*: Quantification of possible reductions in curing time.
- *3rd objective*: Validation that the adhesives remain undamaged.
- *4th objective*: Identification of changes in mechanical and thermo-mechanical properties of the adhesives and joints caused by accelerated curing.
- *5th objective*: Outline influencing factors of the heating behaviour of CP-cured adhesives and joints as well as evaluation of their contribution.
- *6th objective*: Up-scaling to large components.
- *7th objective*: Numerical modelling of the induction heating process.
- *8th objective*: Extension towards low-temperature curing.

A summarised assessment of the aforelisted objectives can be found in section 15.10. The following discussion has been shortened to the most relevant aspects. In addition, the discussed points are formulated as quantified guidelines for practitioners to enable much easier practical implementation of the process. Way more detailed discussions can be found in each of the original articles.

15.1 Design of the accelerated curing by inductively heated Curie particles as a process

In this thesis, I succeeded to identify all important boundary conditions to be considered, while I focused on the aspects that are directly related to bonding; adhesive was thus the central theme of the thesis. The process chart with the associated conditions to be observed has been illustrated in Figure 39.

I showed that induction curing with CP is a highly complex multiphysics problem. It is influenced by various electromagnetic, thermic, chemical-kinetic, material engineering, mechanical as well as process-related effects. All these disciplines interact and determine the properties of the final bonded joint. The mechanical joint properties are influenced by the mechanical adhesive and CP properties, which are additionally influenced by the curing history of the polymer. The latter, in turn, depends strongly upon the boundary conditions of the induction process (coil geometry, EMF intensity, induction time

etc.), joint configuration (geometry, adherends etc.) as well as the electromagnetic, thermal and kinetic properties of the adhesive and the CP – just to name a few uncovered interrelations. From these observations, it comes to no surprise that derivation of generally valid recommendations to design and predict the outcome of the CP-curing technique is arduous. As a consequence, isolation of single influencers out of the complex 'induction jigsaw' was difficult to achieve, since alteration of one boundary condition, e.g. the CP content, ultimately affected several other process conditions.

Aforedescribed interrelated process concept has never been formulated in literature before, it thus represents an essential gain in knowledge. The process design enabled me to raise the curing method to a qualitatively much higher level, which represents the first step towards standardisation and controllability. For industrial applications, quantitative data, such as the exact magnitude of curing temperatures or resulting joint strengths, are necessary. Only on this basis, an efficient and target-oriented process integration into the production of a specific company can be achieved.

In the following, I will explain each of the points illustrated in Figure 39 in context of my experimental investigations, and formulated recommendations for practical application. For that, I refer to the publications that contain the complete information.

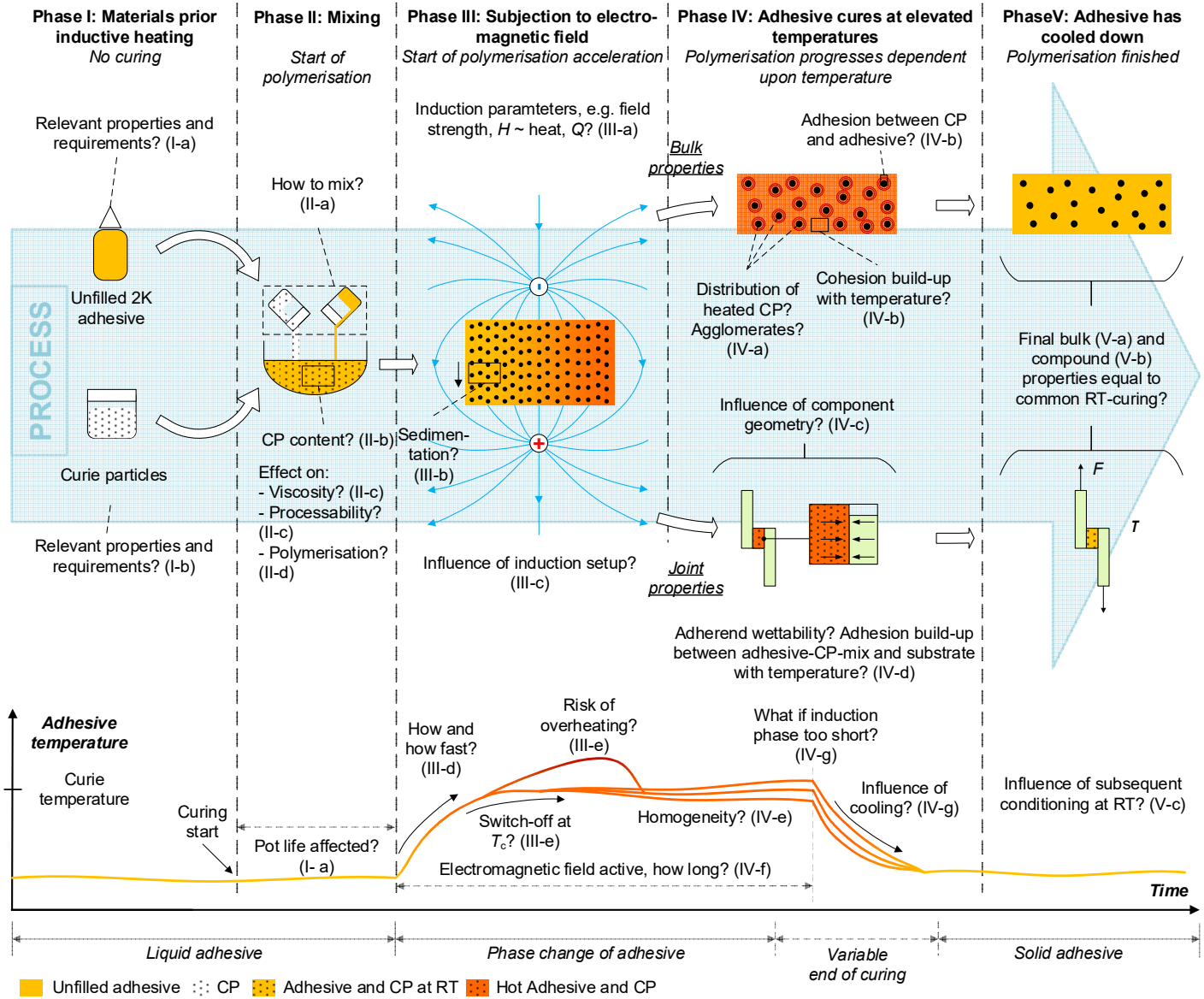


Figure 39: Process chart of the CP-curing technique along with associated scientific challenges and its heating principle (own representation)

Phase I: Materials prior inductive heating

I-a – Relevant adhesive properties

- **Curing kinetics and polymerisation enthalpy:** The curing kinetics of the adhesive must be suitable for elevated temperature curing, and no fundamental changes due to CP addition of the polymerisation (e.g. through catalysis) shall occur. Appropriate thermo-analytical measurements, presented in my 1st paper, have to be conducted so to acquire the necessary fundamental knowledge about the underlying cross-linking reaction – in particular in presence of CP. Based upon these investigations, adhesives with comparatively high enthalpy and fast polymerisation mechanism (like e.g. Fi390, H_{Total} = 306 ± 11.3 J/g) can be identified. Appropriate measures to prevent enthalpy-induced overheating, may then be taken, as e.g. reducing the CP content.
- **Adhesive class:** Both the 2K-EPX and 2K-PUR adhesives are very well suited for the process. For all adhesives investigated, significant reductions in curing time (cf. section 15.3) with – in most cases – similar or higher joint strengths were achieved. However, a restriction has to be formulated for 2K-PUR adhesives when moist wood adherends are to be bonded. In this case, moisture diffusion from the wood is facilitated by elevated temperatures, which alters the cross-linking reaction and reduces joint strengths to ~50–75 % of the unfilled RT-cured reference. This was accompanied by a change in the fracture pattern from adherend to adhesion or cohesion failure and observed for both SLJ as well as GiR. For industrial applications these relations have thus to be verified and accounted for.
- **Pot life:** For the formulation of advices concerning pot life, a distinction must be made between two different scenarios: Firstly, when mixing is performed with a Speedmixer as presented in this thesis, pot life should be sufficiently long as additional time is needed for mixing the CP into the adhesive after resin and hardener were in first contact. This requirement stems from the 2K nature of the adhesives considered, which already start curing at RT. Accordingly, pot life has to be long enough so to prevent significant curing to happen before the adhesive-CP mix is actually applied to the joint. For this mixing scenario, I recommend a pot life of 10 min minimum. Secondly, when other mixing scenarios are developed (e.g. adding the CP to one component within the original cartridges), pot life may be lower than 10 min as resin, hardener and CP get into first contact when the adhesive-CP mixture is applied.
- **Heat capacity and thermal conductivity:** Heat capacity, c_p, and thermal conductivity, λ, exert an influence on the heat development throughout the joint. Both thermal material parameters impact the ability to confine the generated heat within the vicinity of the adhesive. Accordingly, a lower thermal capacitiy, c_p, increases the temperature gradient, as less heat is required to warm the adhesive and the adherends. In typical c_p ranges for adhesives (500–2000 J/kg·K), temperature fluctuations of 5–15 °C can be expected when all remaining induction conditions are kept identical. While not formally shown, low thermal conductivity of the adhesive is expected to promote faster heating as heat dissipation away from the adhesive is prevented. These relationships are best illustrated by the numerical parameter studies presented in my 6th publication.

I-b – Relevant CP properties

- **Curie temperature:** The CP's Curie temperature, T_c, should match the temperature range needed for accelerated curing of the respective adhesive. However, this should follow the motto 'as high as possible, as low as necessary'. A safety margin, for which I recommend 10 °C, should be taken into account to ensure that the adhesive does not exceed maximum tolerable temperatures by the released polymerisation enthalpy.
- **Size:** It is strongly suspected that both the magnitude of T_c and the heat generated by the CP, are dependent upon their size. Accordingly, if the CP are not homogeneous in their dimensions, T_c is not a well defined characteristic, but spread over a temperature range. In my thesis, CP size was in the range of ~0.5–25 µm, with most of the CP (~90 %) being smaller than 21.3 µm. Retrospectly, the variability in sizes may have been disadvantageous as it may provoke a more inhomogeneous state of stress, i.e. lowered cohesive strength, within the polymers (cf. 2nd paper). For future works, I thus recommend to keep CP sizes as homogeneous as possible in order to avoid unnecessary divergences of developing stresses. I strongly suspect that this approach will cause the material to behave more 'isotropically', which may ultimately be favourable for the mechanical joint behaviour. Furthermore, it is conceivable that smaller CP will lead to a better joint heatability, as these offer a larger surface area for heat exchange with the polymer matrix in relation to their diameter. As a result, CP contents may potentially be further lowered, which in turn is ultimately associated with less impact on the mechanical joint behaviour, as extensively discussed.

Phase II: Mixing

II-a – Execution of mixing and associated challenges

Issue addressed in: All publications

During all investigations, the adhesives were vacuum mixed with the CP in a Speedmixer, corresponding to a mixing scenario on the laboratory scale. I adhered to a uniform mixing program, which proved suitable to produce homogeneous adhesive-CP mixes. The following mixing parameters were used: v = 1000 rev/min, p = 100 mbar, t = 60 s. Mixing speed must be sufficiently high to ensure homogeneity of the resulting mixture. However, it shall not be too high in order to avoid excessive heat development during mixing, which would reduce pot life. These considerations become more critical with larger adhesive masses as these promote faster heat generation. With regard to practical implementation, various mixing scenarios are conceivable to achieve a similarly high degree of homogeneity as in my experiments. The latter is a prerequisite for all uncovered relations in this thesis, however, appropriate mixing techniques may only be developed based upon application-specific boundary conditions, most importantly the adhesive properties (pot life, enthalpy, viscosity etc.).

II-b – CP content

Issue addressed in: All publications

Due to the complex interrelations between induction setup / joint geometry, curing kinetics, type of CP and resulting heating behaviour, general advices on how much CP should be admixed are unfortunately difficult to formulate. However, my experiments proved that good mechanical results with significant reductions in curing time can be achieved when comparatively few CP (~3 vol-% / 12.5 w/w-%) are contained, which is best illustrated by the SLTJ experiments presented in my 8th publication. Based upon my experience, the following general considerations regarding the CP content can be formulated:

- The bigger the joint, the more CP are needed to achieve sufficient heating rates.
- The stronger the heat dissipation from the adhesive layer (e.g. when glass adherends are to be bonded, λ = 0.8–1 W/m·K, c_p = 0.6–0.8 J/g·K), the more CP are needed to heat the joint to the relevant temperature range.
- The more CP are admixed, the more significant changes in thermo-mechanical adhesive and joint properties become (increased stiffnesses, viscosities etc.).
- The more CP are contained, the less polymerisation enthalpy is principally available to contribute to the heating.
- The risk for enthalpy-induced overheating is highest for medium CP contents in the range of 20–30 w/w-% as these result in comparatively fast CP-heating with much enthalpy being available. This aspect is best illustrated by the numerical parameter studies included in my 6th publication.

As a rough guideline, I advise practitioners to target CP contents of 10–30 w/w-%, since this range represents a good compromise between the multifarous effects outlined above. However, attention has to be paid to enthalpy-induced overheating, which might be favoured by other process conditions suchs as a comparatively thick adhesive layer (>0.5 mm) in combination with a good insulation (e.g. wooden adherends, λ = 0.13 W/m·K) and small coupling distance (<25 mm).

II-c – Effect on adhesive processabiliy and viscosity

Issue addressed in: All publications

Processability of all investigated polymers could be ensured up to a CP content of 40 w/w-%, which, depending on density of the respective polymer, corresponds to ~10–15 vol-%. Starting from 50 w/w-%, flowability of the highest viscosity adhesive under investigation (Fi390, η = 100 Pa·s according to TDS) was severely limited, which became noticeable by a more difficult workability. Practitioners have thus to keep in mind that mixed-in CP lead to higher initial viscosities η, which tend to increase non-proportionally the more CP are added. These relationships have been quantified by rheological measurements presented in my 2nd publication, with a rough doubling of η being measured for both considered adhesives (η = 231.7 Pa·s, Fi390, η = 19.7 Pa·s, LP421) when 30 w/w-% of CP were mixed in.

General advises regarding viscosity, and its alteration through addition of CP, can only be formulated in combination with application-related boundary conditions (e.g. joint geometry, type of CP, adhesive, adhesive layer thickness etc.). If seen from a 'viscosity-exclusive' perspective, I recommend to keep the CP content as low as possible in order to avoid unnecessary changes in flowability / processability.

II-d – Effect on curing kinetics

Issue addressed in: 1st publication

If practitioners aim to apply the CP-curing technique, they have to consider that added CP represent a filler material, which – depending on CP composition and curing kinetics – may alter the polymerisation mechanisms through catalytic effects. While such effects were not identified in my thesis, alternative adhesives and/or CP should be checked for that. Unrelated to that is the reduced total enthalpy, H_{Total}, which decreases proportionally to the CP content due to the fact that CP act as a 'chemically inert' material generating no reaction enthalpy.

Phase III: Exposion to the electromagnetic field

III-a – Relationship between induction parameters and generated heat

Issue addressed in: 6th publication

Since induction frequency is always determined by coil geometry and mounted capacitors, it was not possible to change frequency without altering other process conditions. Accordingly, I was not able to quantify the effect of frequency on heat development, e.g. by a parameter study. However, I posit that the frequency has a significant influence on heat generated within the CP, and might lead to a shifting of the Curie temperature. However, even if the induction parameters or type of CP would be changed, the principles developed in this thesis would remain valid.

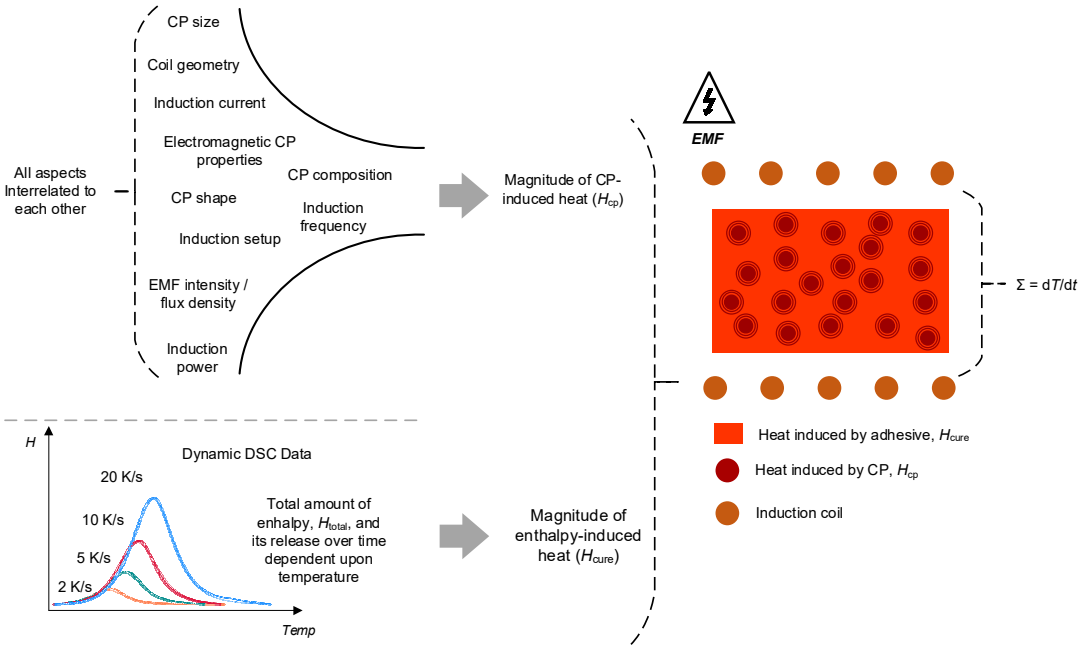


Figure 40: Heat sources to be considered during application of the CP-curing technique (own representation)

Independently on the previously said, I remind that two heat sources have to be considered. Firstly, heat induced within the CP, H_{cp}; and secondly, heat originating from the exothermic polymerisation reaction, H_{cure}. The first one depends on various superimposing conditions (see Figure 40, top-left), the second only from the curing kinetics of the adhesive. Both, however, interact more globally with the specifics of the joint, most prominently those parameters that may contain the heat within the vicinity of the adhesive, or dissipate it away therefrom.

III-b – Sedimentation

Issue addressed in: 2nd, 3rd, 4th and 7th publication

In my experiments, no evidence of significant CP-sedimentation or inhomogeneously distributed CP were observed. Thus, neither the microscopic measurements on AB specimens (see section 2nd paper) nor the extensive studies regarding the heating behaviour delivered evidence on the existence of significant 'CP-free' areas within the polymers. Depending on joint geometry, temperature deviations along the IR- and thermocouple-monitored adhesive layers were in the range of 5–20 °C, which could be attributed to specific boundary conditions influencing the heating behaviour, like e.g. thermal conduction or insulation by utilised adherend materials.

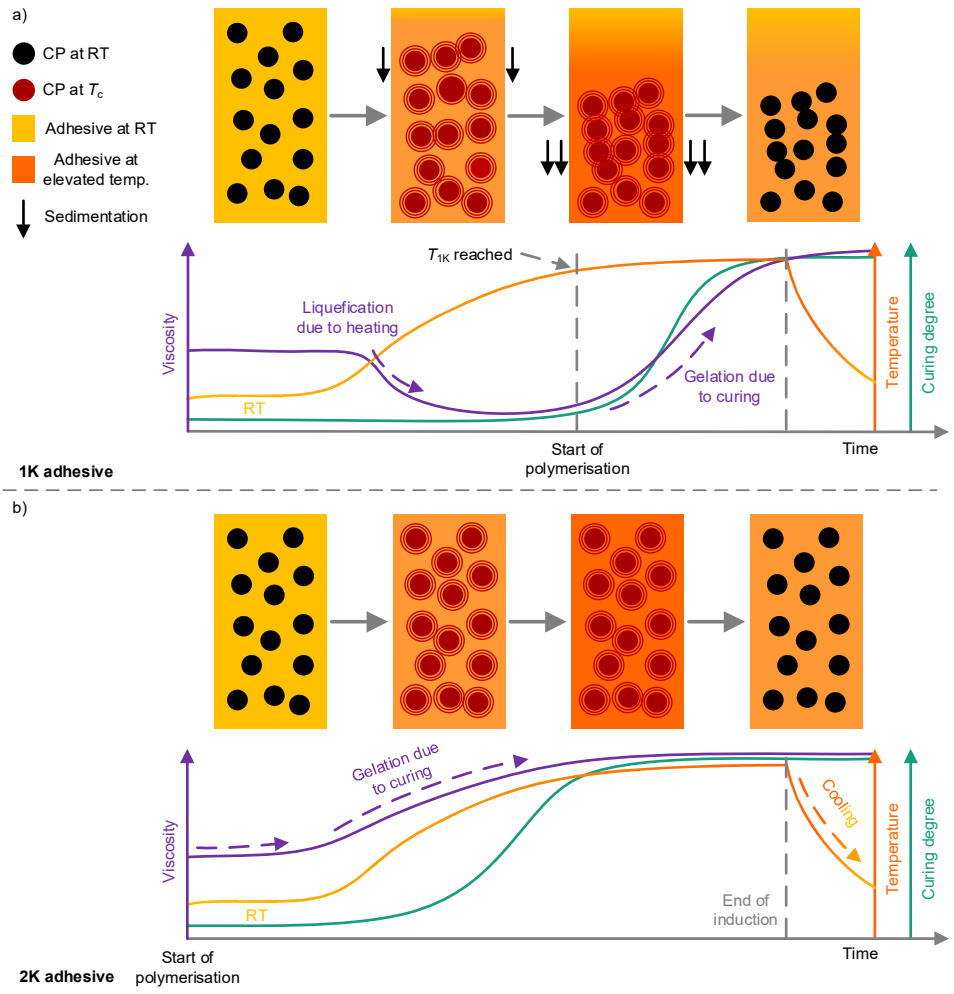


Figure 41: Relationship between curing temperature, curing degree, viscosity and CP sedimentation for a) 1K as well as b) 2K adhesive (own representation)

In addition, the induction processes were always started immediately (~5–10 min) after resin, hardener and CP had been vacuum mixed to keep the time for possible sedimentation as short as possible. These results suggest that early 2K gelation may prevent the CP from sinking down (see Figure 41-b). Other adhesive classes, such as 1K-EPX (see Figure 41-a), for which gelation starts at comparatively high curing temperatures (>140 °C), thus much later, may thus favour sedimentation.

Overall, practitioners must be aware of the fact that sedimentation might occur, especially if large differences in density between polymer matrix and CP are on hand. I thus recommend that, if applicable, density of the two materials should be kept as similar as possible and the induction curing should always be carried out immediately after the CP had been added in order to prevent sedimentation. Otherwise, significant inhomogeneities concerning the heating and curing behaviour, i.e. the mechanical joint performance, are to be expected.

III-c – Influence of the induction setup

Issues addressed in: All publications

III-c1 – Geometry of induction coil

Within this thesis, I exclusively used helicoidal coils. Inductive heating of all specimens was thus performed by placing the respective joint within the coil and aligning the adhesive layer centrically (see Figure 42-a). It is known that the EMF is more homogeneously distributed within helicoidal, or cylindrical, coils compared to one-sided inductors like e.g. so-called pancake coils (see Figure 42-b). If cylindrical coils are used for practical application, I thus recommend to centre the joint to lie within the coil, whereby the component should be maintained in a fixed position to avoid irregular heating as a consequence of possible EMF intensity deviations. While I did not use one-sided inductors, it can be assumed that using them to heat long bondlines is difficult, since the EMF is distributed more inhomogeneous along the coil in contrast to cylindrical coils (see Figure 42-b).

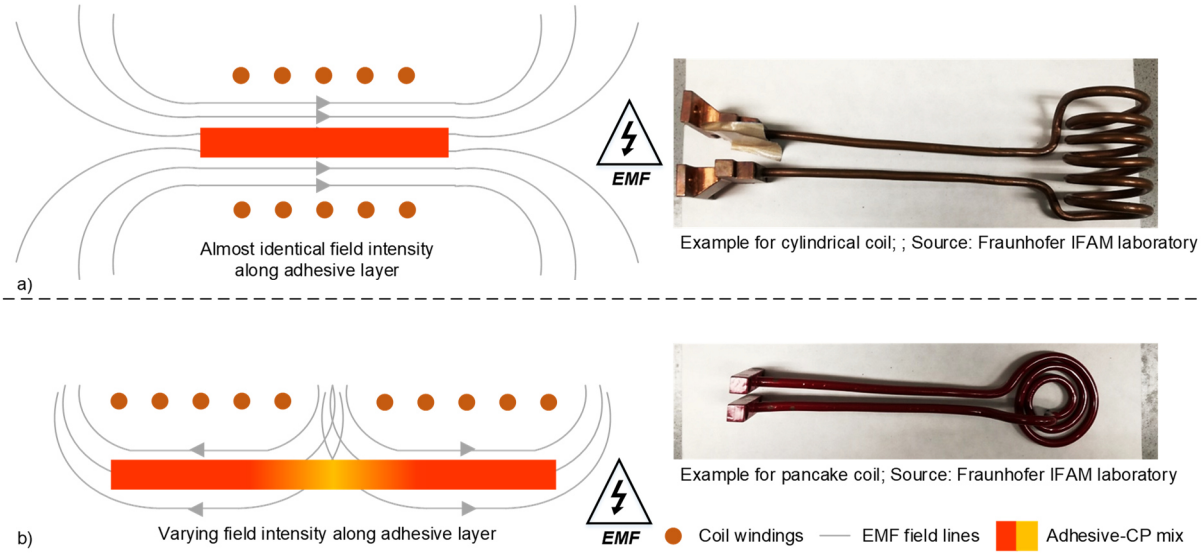


Figure 42: Shape of developing EMF in dependency of induction coil geometry for a) cylindrical coil and b) pancake coil (own representation)

III-c2 – Coupling distance

Homogeneous heating can only be ensured when the coupling distance remains constant along the bond length. In addition, if multiple specimens are to be cured within the same induction process (cf. 3rd publication), I suggest to keep coupling distance for all specimens identical (see Figure 43-a) so to avoid possible differences in resulting curing temperatures (see Figure 43-b). Furthermore, if component geometry allows for it, I advise to keep coupling distance as short as possible in order to achieve faster heating of the bond.

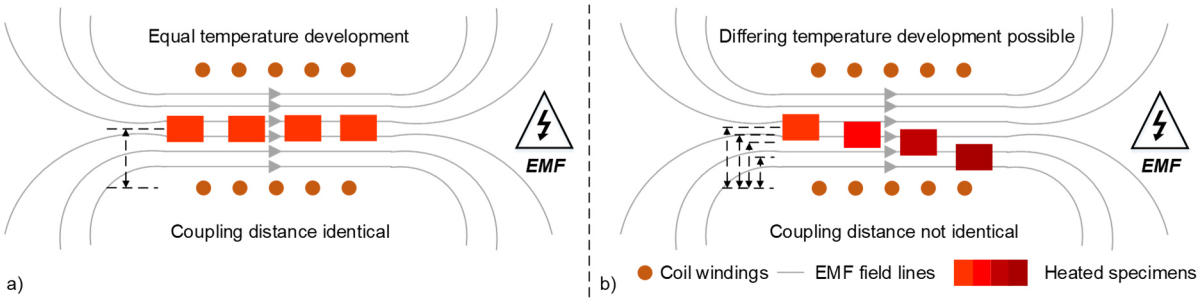


Figure 43: Heating behaviour to be expected for several CP-bonded joints cured within the very same induction process with a) identical as well as b) differing coupling distance (own representation)

III-c3 – Accessibility of adhesive layer

The applicability of CP-curing is significantly influenced by the joints size. Particularly critical is the distance of the adhesive-CP layer from the induction coil. My investigations revealed that it is more difficult to heat large joints (e.g. GiR, see 4th publication, coupling distance of ~60 mm) compared to small components (e.g. SLJ, see 3rd publication, coupling distance of ~25 mm), as bigger induction coils, thus more induction power, has to be supplied to achieve equal EMF strength, i.e. CP-heating, along the respective coil cross-section. The experience gathered so far allows me to conclude that CP-curing is very well suited for small- (SLJ) and medium-sized components (SLTJ). However, a final statement whether the method can cost-efficiently be applied to component sizes larger than the GiR presented in the 4th paper (120 x 120 x 300 mm, wooden blocks; Ø18 mm, l = 300 mm, G-FRP rods) cannot be made. To enable the induction curing of even larger joints, further process optimisations like EMF concentrators or CP optimisations may open up new production opportunities.

III-d – Influencers for heat generation and attainable heating rates

Issue addressed in: All publications

The explanations concerning the heating influencers can be found in section 15.6.

III-e – Curie effect at T_c and overheating

Issue addressed in: All publications

Temperature control over the introduced type of CP was successfully achieved. It effectively eliminated the need to apply external measuring techniques, e.g. thermocouples or IR camera, during the process. However, the Curie effect manifested way more complicated than initially expected. While the switch-off

behaviour at T_c can be guaranteed by choosing an appropriate CP composition, a thermal equilibrium between immited and emitted heat will always establish, its magnitude being dependent upon the respective application / joint geometry. Consequently, adhesive temperatures may not reach T_c, either because the EMF is too weak, the CP content too low, or because of excessive heat dissipation in the joint (see Figure 44-a). An example for aforedescribed curing scenario represent the SLTJ-related inductive heating experiments presented in my 8th publication. Using my numerical model (cf. 5th publication), and considering all material properties and boundary conditions, it is possible to estimate how much CP have to be added in order to reach T_c in a given time.

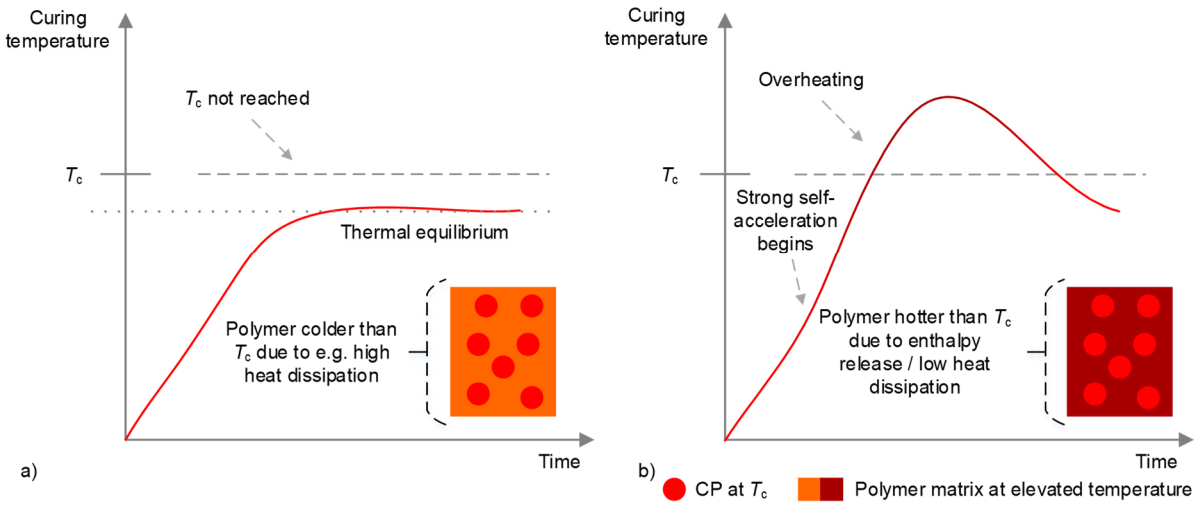


Figure 44: Extreme heating scenarios for the appearance of the Curie effect for a) too low as well as b) too high heating rate, dT/dt, resulting from application-related boundary conditions like heat dissipation, component geometry, CP content etc. (own representation)

The FEA also allows for estimates of the cases where the heating rates are 'too high' (see Figure 44-b), e.g. when a high CP content in combination with a strong EMF and a thick adhesive layer is considered. In such cases, overheating of the bond due to the exothermic self-accelerating polymerisation reaction may occur. This behaviour has been observed on the experimental level in my 2nd publication (DMA and AB specimens) and can be mapped by the simulations as shown by the parameter studies presented the 6th paper. In addition, uncontrolled runaway heating will not occur directly at the beginning of the heating phase, but rather in the middle of the induction process, as proven by the simulations. The reason for that, amply discussed in my 6th publication, is that in the beginning heating first results from the induction process. The release of exothermy is kicked-off only after the heating phase has advanced significantly, and higher temperatures have been generated. With this in mind, excessive heating rates will lead to a very fast release of enthalpy in relatively short induction time, resulting in an uncontrolled chain reaction and possible irreversible damages of the adhesive. Practitioners have thus not only to consider the absolute amount of enthalpy, H_{Total}, but also its rate of release for the heating, which can only be determined using appropriate characterisations as those presented in my 1st publication.

Phase IV: Adhesive cures at elevated temperatures

IV-a – Distribution of heated CP and agglomerates

IV-a1 – CP distribution during induction heating

Issue addressed in: 3rd and 4th publication

Although I did not perform any in-depth microscopic analyses by using e.g. SEM or EDX (save for microscopic imaging presented in the 3rd paper), I delivered an indirect proof for the homogeneity of the CP distribution. Various IR measurements on different joint geometries showed homogeneous temperatures with little deviations along the visible surfaces (see Figure 45). I therefore conclude that excellent intermixing has been achieved during the induction processes.

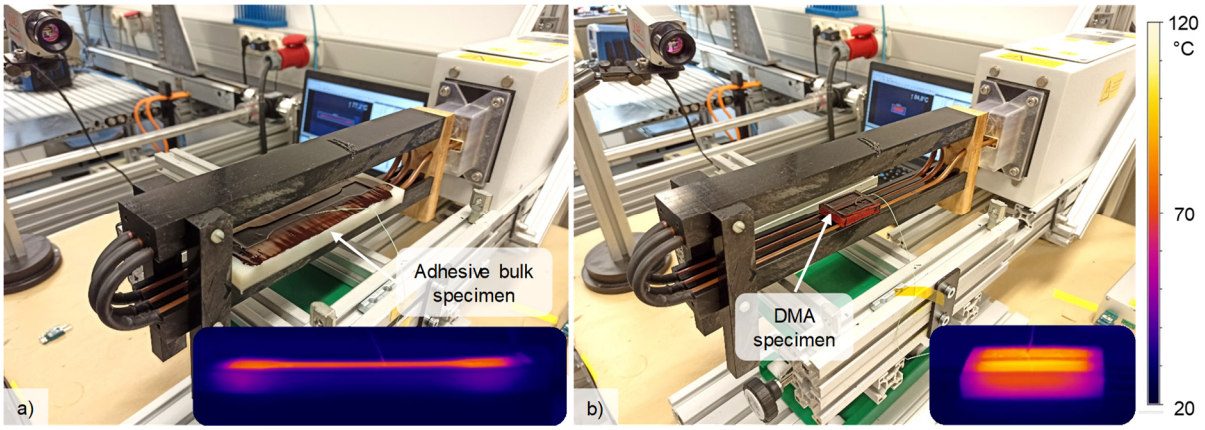


Figure 45: Temperature measurements using IR camera for CP-filled a) AB and b) DMA specimens taken and re-structured from my 3rd publication

IV-a2 – Agglomeration

Issue addressed in: 2nd publication

Regarding CP agglomerations, I did not perform specific measures to prevent the CP from agglomerating. However, the almost perfect homogeneity in CP distribution (discussed above) did not hint at such problems. If seen from the perspective of joint strength, my experiments demonstrated that inductively cured specimens were equivalent in comparison to unfilled references, which proved true across all joint geometries studied (SLJ, GiR and SLTJ). In contrast, RT-curing with added CP lead to a deterioration of cohesive strength (see 2nd paper) as well as adhesive strength (see 3rd paper) when the CP content was increased. Based on these observations I strongly suspect that CP agglomerates may have a negative impact on mechanical strengths due to intrinsic stress peaks originating from stiff CP agglomerates embedded in the way softer polymer matrix (see Figure 46).

Aforementioned agglomerate-related negative effect, however, plays a subordinate role, since induction curing at elevated temperatures counteracts the reduced strengths since a denser polymer network gets formed and flowability / adherend wettability is increased at high temperatures. This is particularly evident at the level of joints, where substrate strength often represents the limiting factor of the connection.

Especially for non-metallic materials like wood or FRP, the bonding of which is targeted by application of CP-curing, adherend strength is often not very pronounced, resulting in an early substrate failure although cohesive strength may be slightly reduced by CP agglomerates.

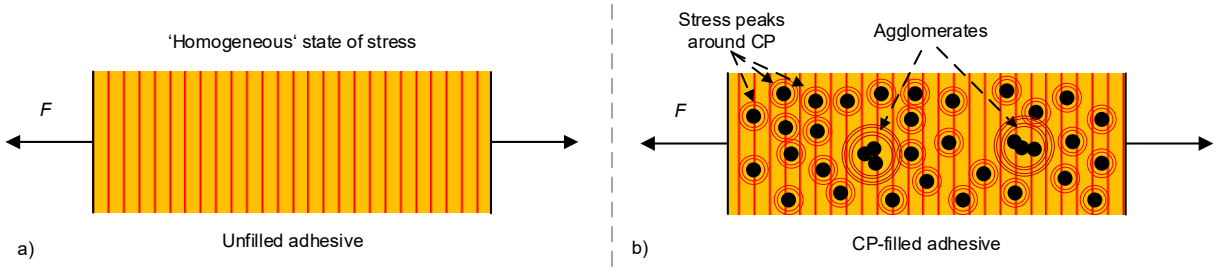


Figure 46: Schematic illustration of intrinsic state of stress for a) unfilled and b) CP-filled completely cured polymer (own representation), complementary numerical 2D data can be found in the original article of my 2nd publication

In summary, it can thus be concluded that agglomerates most likely exert a negative influence on the mechanical behaviour, however, the negative effects associated dwarf under induction-bonded joint conditions. That said, the topic of agglomeration should be investigated in future studies, e.g. by in-depth experimental analyses on the microscopic level as well as appropriate CP optimisations, in order to avoid them.

IV-b –Cohesion build-up with temperature

Due to the very good mechanical performance on different joint geometries, I conclude that strong matrix cohesion must have been developed, which in many cases was sufficient to result in adherend failure (see e.g. G-FRP /spruce SLJ in 3rd publication or SLTJ in 8th publication). Cohesive strength was thus more dominant than adherend strength originating from the associated stress-condition of the respective joint geometry. In addition, obtained mechanical strengths for inductively cured AB specimens (2nd publication), were equivalent or slightly lower (~10 %) compared to the reference sets, delivering supplementary proof for significant cohesive strength after CP-curing. These macroscopic results can be further complemented by the DSC measurements performed on CP-filled adhesives (1st paper), which revealed that the polymerisation, i.e. formation of cohesive strength, is not hindered by the presence of CP. Based on the available data, a final statement whether matrix cohesion or adhesion between CP and polymer represents the weakest intrinsic part of the adhesives cannot be made. For a deeper consideration of these relationships, in-depth microscopic investigations would be necessary.

IV-c – Influence of component geometry

Issue addressed in: All publications except 1st publication

The geometry of a CP-cured component is a decisive parameter. As already discussed, the joint to be inductively cured must not be too distant from the inductor, as otherwise EMF intensity may be too low to achieve sufficiently high heating rates. Additionally, the EMF should be designed so to favour homogeneity along the adhesive layer, as otherwise not all parts experience similar heating. In my experiments,

only moderate temperature inhomogeneities (~10–15 °C) were observed. However, homogeneous exposure to EMF alone does not guarantee homogeneity of heating, as aspects such as different thermal insulaton may interfere. Typically, the GiR in my investigations, despite being subjected to a homogeneous EMF, usually exhibited slightly lower temperatures at the top (~15 °C, which was exposed to the air), if compared to the (much better insulated) bottom (see 4th publication).

Additionally, manufacturing accuracy in terms of geometrical imperfections also plays a significant role. The effect thereof exerts a direct influence on adhesive layer thickness, i.e. enthalpy release / extent of polymeric self-acceleration. It is best illustrated by the SLTJ experiments (cf. 8th paper), in which temperature deviations along the tube perimeter of 6–7 °C were attributed to misalignment. For practical application, manufacturing tolerances must therefore either be low in order to avoid unnecessary deviations in adhesive layer thickness, i.e. curing temperatures, or accounted for. How much adhesive layer thickness fundamentally impacts temperature development was also illustrated by the numerical data presented in my 6th publication.

IV-d – Adherend wettability and adhesion build-up to substrate

All adhesives investigated in my thesis developed significant adhesive strengths on the respective adherend materials for various substrates and joint geometries, which – as already mentioned – provoked adherend failure in most cases. Nevertheless, it can be expected that the adhesives capability to wet the adherends gets reduced, especially when high CP contents are added, since viscosity increases non-proportionally with the CP content (see 2nd publication). However, elevated temperature curing compensates for reduced adhesion, which I attributed to liquification, thus increased wettability.

IV-e – Homogeneity of heating

Issue addressed in: 2nd, 3rd, 4th and 8th publication

The explanations concerning heating homogeneity can be found in section 15.6.

IV-f – How long to maintain the EMF

Issue addressed in: 5th and 6th publication

Prior the development of my thesis, it was not possible to reaslitically estimate induction times needed to fully cure CP-bonded joints. Practitioners were only able to evaluate the curing progression by comparing mechanical strengths at different times, and to estimate minimum or optimum curing times. By implementation of my numerical model (cf. 5th and 6th paper), I thus took an important step towards more efficient design of CP-curing processes, as I succeeded in predicting the curing degree, α, in dependency of various boundary conditions of the process like adhesive layer thickness, CP content and – above all – induction time.

Actual needed induction time is always dependent upon curing temperatures developing within the adhesive, which, as amply discussed, depend on multifarious application-related conditions. In my exepriments, an induction time of 10 min was chosen to maintain comparability between the different joint geometries, which proved sufficient to fully cure all considered adhesives. However, it is already known that induction time can be further reduced to ~2–5 min when process conditions are favourable, e.g. for adhesives with comparatively fast kinetics (e.g. Fi390, Jo692) used to bond small- to medium-sized joints (e.g. SLJ, SLTJ).

IV-g – Influences of a too short induction phase and the cooling phase

Issue addressed in: All publications

If inductive heating ends before full cure has been reached, post-curing will take place during cooling of the joint as well as subsequent conditioning at RT due to underlying 2K kinetics. This has been proven on the experimental (see 2nd paper) as well as on the numerical level (see 6th paper). For reasons related to thermal intertia, large joints (e.g. GiR, 4th paper) are much slower in cooling down, if compared to small ones (e.g. SLJ, 3rd paper). For practical application, it is thus of significant importance to consider cooling times in the production process so to get the most benefit out of the energy used for curing the joints.

Furthermore, practitioners can further reduce induction times by only targeting handling strength of the component instead of fully cure them —if 2K-adhesives are used. 2K adhesives will still continue to cure under 'normal' conditions, as at RT. If this approach is pursued, my DMA experiments (2nd paper) proved that – if seen from the thermo-mechanical perspective (G', G'' and T_g) – the adhesive will reach a T_g that is at least equivalent or most likely higher compared to TDS curing. However, it can be expected that G' and G'' get shifted towards higher stiffnesses over the whole operating temperature range, whereby this effect gets more pronounced the more CP are added to the respective polymer. Except aforedescribed stiffness-related alterations and their accompanied challenges for the mechanical joint behaviour, i.e. changed state of stress, there are thus no negative influences to be anticipated regarding T_g.

Phase V: Adhesive / joint has cooled down to RT

Applied experimental approach

My experiments proved that CP-curing fundamentally changes the properties of adhesives. The reason for that lies in the fact that two mechanically differing materials (polymer and CP) are combined, but also in the curing at elevated temperatures. By comparing unfilled RT-cured specimens (see Figure 47-a) with CP-filled RT-cured polymers (see Figure 47-b), it was possible to identify changes in adhesive properties resulting from the mere addition of the CP. In contrast, juxtaposing CP-filled RT-cured polymers (see Figure 47-b) with inductively cured specimens (see Figure 47-c) provided information about the impact of CP-curing at elevated temperatures on the adhesive characteristics.

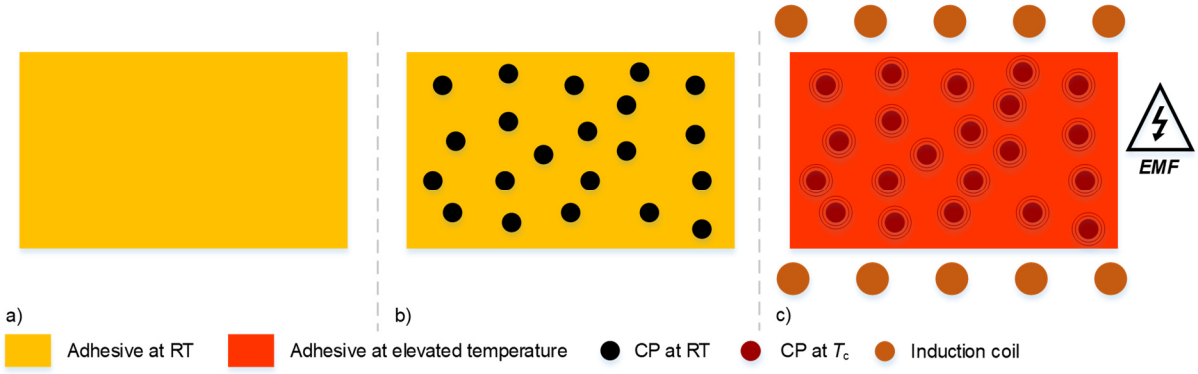


Figure 47: Curing scenarios applied for proper traceability of changes in adhesive properties as a consequence of CP-curing; a) RT curing according to TDS, b) RT curing according to TDS with added CP and c) induction curing over embedded CP (own representation)

V-a – Changes at the level of bulk adhesive

Issue addressed in: 2nd publication

Aforedescribed scientific approach revealed that the addition of CP mainly exerts influences on the adhesives stiffness – independently of the curing method applied. These result from much stiffer alloys being mixed into the comparatively soft polymer matrix. In addition, CP act as stress concentrators if the adhesive is set under tension, leading to lowered cohesive strengths due to inhomogeneously developing stresses. The higher the CP content, the more detrimental are these effects. In detail, increases in moulus of elasticity of ~40 % and reductions in cohesive strength by ~10–20 % could be measured for two adhesives (Fi390, LP421) when 30 w/w-% of CP were added, if compared to the unfilled references. However, at least the negative impact on cohesion is counterbalanced if CP-filled specimens are subjected to EMF and a denser polymer network is formed due to elevated temperature curing. These results indicate that thermal degradation, e.g. by local overheating, has been successfully prevented. The outlined relations are best illustrated by the AB and DMA experiments presented in my 2nd publication.

V-b – Changes at the level of joints

Issue addressed in: 3rd, 4th and 8th publication

At level of large joints (GiR, SLTJ), adding CP in combination with RT-curing did not result in any deterioration of the mechanical joint performance – a very encouraging result. However, the small-scale SLJ experiments (cf. 3rd publication) showed a degradation of adhesive strength with identical fracture patterns when the CP content was increased. This indicates that adhesive strength gets lowered by the mere presence of CP, which can also be attributed to the stress-related effects discussed above. In essence, the observations regarding induction curing at the bulk level are confirmed by those at the level of joints, with equal or higher (~10–20 %) joint strengths being attained.

V-c – Influence of subsequent RT conditioning

Issue addressed in: 3rd and 8th publication

Conditioning times at RT exert a non-negigible influence on the mechanical behaviour of CP-cured joints, with the observed effects tending to increase with longer conditioning times. Depending upon the adhesive class (2K-EPX or 2K-PUR), joint strengths either improved, or deteriorated, over time. However, even if a deterioration was observed over time, strength, in most cases, still exceeded that of the unfilled reference sets. The reasons for this behaviour are not yet fully understood but I strongly assume that strength and stiffness do not increase proportionally. While strength may already be fully developed at the end of inductive heating, stiffness may not yet, which leads to the formation of a more or less favourable state of stress, which in turn lowers or highers resulting strengths. Other potential reasons may be locked-up residual stresses that require some time to creep away.

15.2 Providing an inductive heating process without external temperature control by using CP

One of the core objectives of the thesis was to achieve an independent temperature control over the utilised type of CP, which would eliminate the need to control induction. For that, CP with a T_c of ~110 °C were selected. To illustrate the Curie effect, I exemplarily summarised all heating curves in dependency of joint geometry for the two adhesives Fi390 and LP421 in Figure 48. In all these experiments, the same induction time of 10 min was applied, although the induction power was reduced for most of the smaller samples.

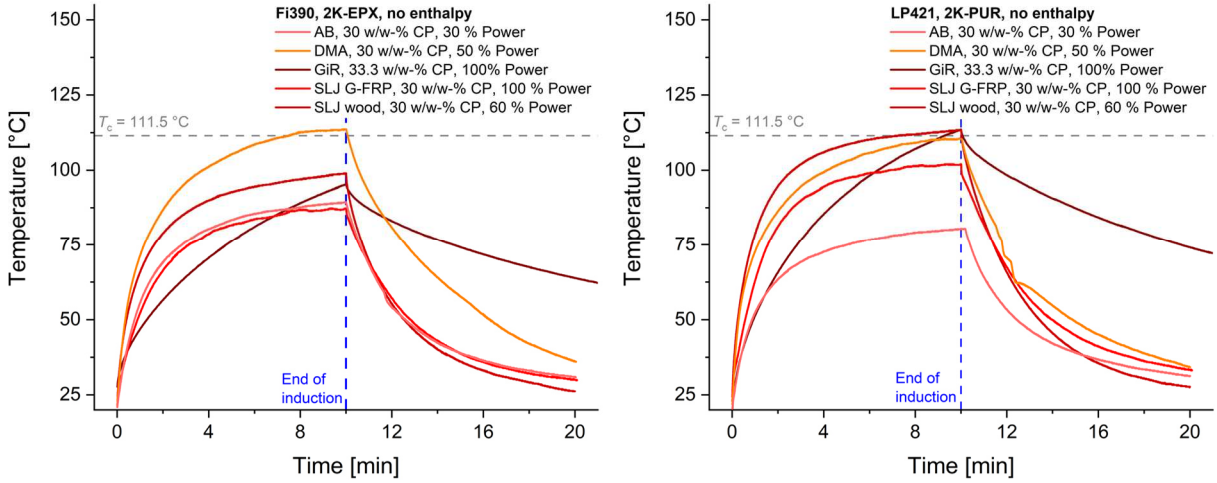


Figure 48: Summary of heating cycles for fully cured Fi390 (left) and LP421 (right) in dependency of joint geometry, temperature curves for GiR represent arithmetic mean out of three measuring points along the adhesive layer

All temperature curves show an arc-shaped / Curie-controlled progression, with a clear flattening being visible for all specimen types except the large-scale GiR joints (dark red curves). These needed significantly more time to achieve the target temperature range because of their thermal inertia, which is also reflected in the way slower cooling behaviour compared to the smaller samples. The temperature curves also reflect the thermal equilibrium between introduced and dissipated heat. All this strongly indicates

that the considered adhesives did not undergo overheating and that principal applicability of the Curie mechanism has thus been proven. The Curie effect also manifested in the numerical models, which were presented in my 6th publication.

15.3 Quantification of possible reductions in curing time

The achieved reductions in curing time in comparison to common RT-curing have been summarised in Table 10. Since the CP-curing technique was also considered for low-temperature curing (see 7th publication), different temperature ranges are included in Table 10 (leftmost column). Curing times for low ambient temperatures were calculated based upon Arrhenius law, as these are typically not provided by the adhesive manufacturers. For the understanding of Table 10, the following has to be clarified: The curing reduction factor, *crf*, is used to quantify the savings in curing time. In detail, *crf* = 60 indicates that induction curing with CP is 60 times as fast as common RT-curing of the unfilled adhesive. Since an induction time of 10 min was predominantly used throughout the thesis, t_{ind} = 10 min served as a basis for the calculation of the *crf* values included in Table 10.

Table 10: Needed curing time in dependency of curing temperature of the 2K adhesives (values before slash) juxtaposed with 'curing reduction factor' (*crf*) after application of CP-curing based on t_{ind} = 10 min (values after slash).

Temperature range for curing [°C]	**Adhesive**				
	2K-EPX			2K-PUR	
	Fi390 [h / crf]	Jo692 [h / crf]	We32[h / crf]	LP421 [h / crf]	LP821 [h / crf]
+20 to +30	10* / 60	12 / 72	8* / 48	120 / 720	120 / 720
+10 to +20	18* / 108	24* / 144	20* / 120	240* / 1440	240* / 1440
+10 to 0	40* / 240	48 / 288	40 / 240	480 / 2880	480 / 2880
0 to −10	80* / 480	96 / 576	80 / 480	960 / 5760	960 / 5760
−10 to −20	160 / 960	192 / 1152	160 / 960	1920 / 11520	1920 / 11520

* Curing times taken from TDS. All remaining values before slash calculated based upon Arrhenius law[233].

Although curing times at RT can also be reduced significantly, it quickly becomes clear that the benefit of the CP-curing technique drastically increases when a specific application has to be manufactured under temperatures lower than RT. Thus e.g. for Jo692, curing at RT takes 12 h, which can be reduced by induction heating to 10 min, corresponding to a 72-times faster curing scenario. In contrast, when lower curing temperatures of e.g. +5 °C are on hand, RT-curing is significantly prolonged to 48 h and the advantage of CP-curing becomes more pronounced (*crf* = 288). The results clearly show that significant reductions in curing time could be achieved, which holds especially true for the polyurethanes. Depending on the application, practitioners have to decide whether additional experimental effort and changing joint properties associated with the process are tolerable or not.

15.4 Validation that the adhesives remain undamaged

Based upon the good mechanical properties of the investigated adhesives I posit that irreversible polymeric damages have been prevented. As already discussed, resulting deteriorations as e.g. reduction of

cohesive strength could be traced back to the altered mechanical material behaviour, i.e. stiffness deviations between polymer matrix and CP as well as resulting stress concentrations. Possible damages like extensive bubble formation or burned polymer parts could not be identified by microscopic imaging presented in my 3rd paper. In addition, external damages or conspicuities as for example adhesive foaming during CP-heating were not observed by visual inspection. Finally, DSC analysis with added CP (1st paper) – excluding observed changes in enthalpy – revealed no fundamental alterations of the polymerisation progress. I thus strongly assume that thermal degradation has been successfully prevented and that the objective has been achieved. However, to finally assess the adhesives material condition, further experiments using SEM, EDX or XPS should be performed to analyse the bonding results on a deeper level.

15.5 Identification of changes in mechanical and thermo-mechanical properties of the adhesives and joints caused by accelerated curing

The objective was successfully achieved. The relations have been explained in section 15.1 and are best illustrated by the content of my 2nd and 3rd publication.

15.6 Outline influencing factors of the heating behaviour of CP-cured adhesives and joints as well as evaluation of their contribution

This thesis allowed to identify various contributions to the heating behaviour of CP-cured adhesives and joints. While determined on a subset of five adhesives (epoxies and polyurethanes), the conclusions drawn apply in principle to other adhesive classes with exothermic reaction mechanisms. Assuming that the CP are homogeneously distributed within the adhesive, the following aspects proved paramount:

- Curing kinetics of the adhesives, i.e. absolute amount of polymerisation enthalpy, H_{cure}, and rate at which it is released.
- Thermal material properties of CP, adhesive and adherends, most importantly c_p and λ.
- CP content, $c_{particle}$, with heating rates tending to be higher the more CP are added.
- Experimental setup, i.e. coil geometry, coupling distance, induction parameters and – depending on aforementioned conditions – intensity and distribution of EMF.
- Adhesive layer thickness, t_a, i.e. polymerisation enthalpy. Thicker adhesive layers typically result in substantial increases of maximum curing temperatures, due to stronger self-acceleration of the polymerisation.
- Component geometry, i.e. heat accumulation originating from insulation effects (e.g. wooden GiR) or faster heat dissipation from the bondline (e.g. for glass adherends).
- Starting temperature of the joint to be heated, T_{start}, which influences the heating most importantly on a quantitative rather than on the qualitative level. Dependent upon T_{start}, curing temperatures are almost linearly shifted.

- Induction time, with temperatures increasing with longer induction operations.

In addition to these findings, numerical studies presented in my 6th publication proved that the influence of aforementioned conditions decreases according to the following sequence: adhesive layer thickness, CP content, induction time, GiR starting temperature, heat capacity and thermal conductivity of adhesive-CP mixture. A stronger influence of λ is suspected for thicker adhesive layers.

In order to heat the adhesive layer as homogeneously as possible, the following conditions must be met: identical coupling distance / EMF intensity along the adhesive layer, homogeneous CP distribution as well as identical heat dissipation / substrate material on both sides of the adhesive layer (see Figure 49). Nevertheless, slight temperature deviations (~5–15 °C, depending on process conditions) cannot completely be avoided as the centre of any adhesive experiencing an exothermic kinetics will always heat up faster compared to its edges where the CP-induced heat can be dissipated into the adjacent substrate materials.

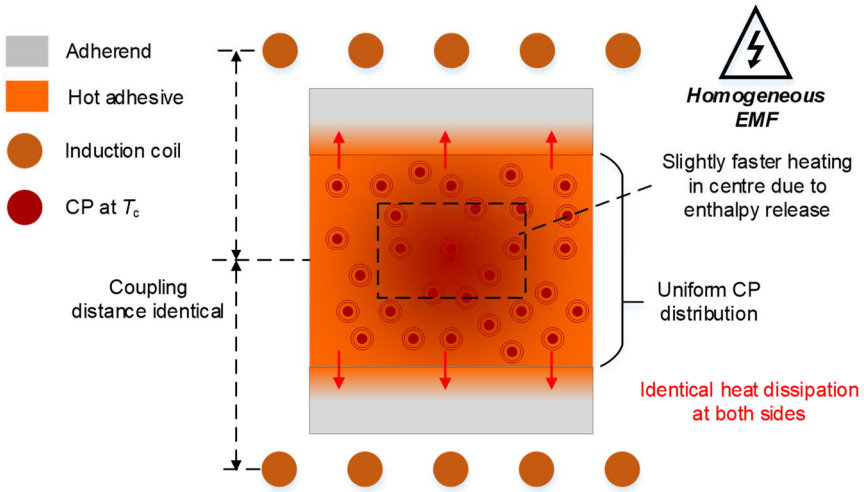


Figure 49: Necessary boundary conditions for most homogeneous heating of CP-cured adhesive layers (own representation)

15.7 Upscaling to large components

In this thesis, an in-depth implementation of CP-curing on the level of large bonded connections was carried out in my 4th (GiR) and 8th publication (SLTJ). For GiR, the experiments were additionally extended towards low-temperature curing (7th publication). This is the first time that systematic experimental campaigns including temperature monitoring and extensive mechanical tests have been performed on the level of joints. The state of the art has thus been significantly extended and the objective was achieved.

15.8 Numerical modelling of the induction heating process

In this thesis, an approach was developed on how to link thermal and kinetic aspects of CP-induced accelerated curing (see 5th publication). It was implemented numerically, so to open the possibility to

predict induction times needed to achieve full cure under differing curing conditions. In addition, the numerical model offers a way for targeted parameter variations, e.g. component dimensions or material properties, and thus deepens the understanding of factors influencing the curing processes. Validation of the numerical model was successfully carried out using temperature measurements (see 6th publication), which showed very good qualitative and also mostly quantitative agreement with the numerical predictions. Subsequently presented numerical parameter studies allowed for interesting insights into thermal and kinetic aspects of the CP-curing technique, which would not have been possible solely on the experimental level. A model of this kind cannot be found in the relevant literature, which is why the fulfilment of the objective represents a significant extension of the current state of the art in the field.

15.9 Extension towards low-temperature curing

The experiments presented in my 7th publication showed that the CP-curing technique can also be applied for low-temperature curing (starting from +5 and –10 °C), which was verified for three representative 2K adhesives (Fi390, Jo692 and LP421). Curing in these temperature ranges is currently not intended by construction authorities, as the polymerisation under these adverse thermal conditions normally lasts very long or would not occur at all. Sufficient strength development can thus not be guaranteed. Due to these reasons, the content of my 7th paper represents an important contribution and paves the way for new application fields of adhesively bonded joints. The objective has been successfully achieved.

15.10 Assessment of the achievements

1st objective

– Design of the accelerated curing by inductively heated Curie particles as a process –

The scientific approach of upscaling the investigations from small (adhesive bulk) to large (adhesively bonded connections) enabled me to holistically capture scientific sub-disciplines such as curing kinetics, heating behaviour or developing thermo-mechanical properties involved in the process. As a result, underlying trends and mechanisms, as well as their interrelations, could be worked out much more efficiently. The proposed ‘process design’ thus represents a useful methodological tool, which enables practitioners to integrate accelerated curing processes into their production processes much more efficiently. Summing up, the fulfilment of the objective represents an important step towards standardisation in the field.

2nd objective

– Providing an inductive heating process without external temperature control by using CP –

The research compiled in this thesis clearly indicated that induction heating with embedded Curie particles, and thus accelerated adhesive curing, can be freed from the constraints of external temperature control. The experimental investigations have repeatedly shown that the induced thermal energy is effectively capped once temperatures approach the Curie temperature (cf. section 15.2).

3rd objective

– Achievement of significant reductions in curing time –

Curing times have been significantly reduced, if compared to the times required for curing at RT. Depending upon the adhesive and ambient temperature range considered, the joints curing could be speed up by factors in the range of ~60–11.000 times (cf. section 15.3). The reductions in curing time are dependent upon many process conditions such as joint size, enthalpy or adherend material. For large-scale joints, more heat is needed to reach the desired temperature ranges and thermal inertia also requires significant cooling times, which proved longer than the proper curing times. The benefit of the technique increases drastically when bonding is to be carried out at low ambient temperatures, with reductions in curing time by factors of ~500 (epoxies) and ~6.000 (polyurethanes) being achieved for a temperature range of –10 to 0 °C.

4th objective

– Preventation of polymer damages as well as changes of the polymerisation course –

The adhesives, and adhesive-CP mixes (with various concentrations $c_{particle}$), were extensively investigated by means of differential scanning calorimetry (DSC, 1st publication). The results thereof clearly showed that the polymerisation process of the adhesives, thus their curing, was not altered, e.g. by catalytic effects. It was unambiguously shown that the CP acted as dead mass within the bulk. With that result, kinetic modelling of the adhesive-CP mixes, in essence, proved to be the same as those of the adhesive alone, save for a scaling factor $1-c_{particle}$. In addition to the kinetic-related findings, mechanical results across all joint geometries proved that CP-cured connections can achieve a fracture behaviour indistinguishable to that of unfilled RT-cured references. Based on these results, it is thus strongly assumed that fundamental polymer damages have been prevented.

5th objective

– Identification of changes in mechanical and thermo-mechanical properties of the adhesives and joints –

An extensive experimental campaign highlighted the influence of the CP content, and the curing conditions, on the mechanical properties of the adhesives, if compared to the standard case of curing at RT according to the technical datasheet. The addition of CP and subsequent curing at RT reduced tensile and shear strength (on various substrates), and increased the modulus of elasticity (MoE). However, curing at the elevated temperatures provided by the induction process, in most cases, resulted in an increase of both tensile and lap shear strength, beyond the respective standard values; the MoE, however, further increased as stiffness is mainly determined by the mere presence of the CP. The CP content, however, did not influence significantly the glass transitions temperatures, as shown by a series of dynamical mechanical analyses (DMA). In contrast, the resulting T_g gets highered due to elevated temperature curing, i.e. denser cross-linking, when induction heating is pursued.

6th objective

– Determination of influencing factors contributing to the heating behaviour of CP-cured adhesives and joints –

All significant factors influencing the heating behaviour of CP-cured adhesives and joints were identified (cf. section 15.6). Some influencing factor are obvious, as the CP content and the strength of the electromagnetic field (EMF, itself dependent upon a large variety of factors, most notably the coupling distance). Some others, although to some extent expectable to be important, appeared to be more significant than initially thought, as for examples the thermal boundary conditions of the joints, and their thermal inertia and the resulting capacity to effectively confine the generated heat in the vicinity of the adhesive layer. Similarly influencing are the thermal properties of the adherends, most prominently the thermal capacity and conductivity. Lastly, the exothermic heat released by the curing reaction, proved to be much more significant than initially thought of. The latter's influence being additionally very much modulated by the amount of adhesive, i.e. the adhesive layer thickness. Not taking it into account may result in sever underestimations of the developed temperatures, going so far as to damage the adhesive.

7th objective

– Extension and application to large components and low-temperature curing –

The large-scale low-temperature Glued-in Rod (GiR) experiments presented in my 7th publication, resulted in joint strengths comparable to RT-cured reference samples for the epoxy adhesives. This clearly indicates that the procedure developed in this thesis opens the door to controlled adhesive curing under adverse temperature conditions.

8th objective

– Numerical modelling of the induction heating process –

In my 5th and 6th publication, accelerated curing could be numerically modelled considering the real geometry, material properties, boundary conditions, Curie temperature, inherent thermal energy release of the CP, their content and curing kinetics of the adhesives including the time delayed release of enthalpy. This allowed for much deeper insights regarding the development of temperatures and curing degree. It also provided a meaningful tool for planning of accelerated curing procedures based upon Curie particles.

16 Summary

In this thesis, I investigated a novel approach for accelerating the cure of adhesives achieved by adding specially designed filler materials, so-called Curie particles (CP). The CP are susceptible to electromagnetic fields (EMF) and can be inductively heated up to their material-specific Curie temperature, T_c, above which heating stops. Aforedescribed curing method represents a self-regulating process, which – in addition – runs independently from ambient temperatures. In contrast, most literature describes the use of particulate susceptors consisting of e.g. iron (T_c = 768 °C) or magnetite (T_c = 578 °C) to heat the respective adhesives, which require an external temperature control, as otherwise excessive temperature degrades the polymer. To get most of the Curie temperature based technique, T_c has thus to be carefully adapted to the adhesives, and to joint geometry, so to optimise out curing time and mitigate risks of damaging the adhesive.

I showed that the CP-curing technique represents a multiphysics engineering problem, which results in fundamental alterations of the mechanical behaviour, from the adhesive bulk level to that of full-scale bonded joints. Various boundary conditions, most importantly joint geometry, curing kinetics and enthalpy release over time, have to be taken into account in order to fully understand the complex heating behaviour of CP-cured adhesives.

To illustrate effects and conditions associated with the novel CP-curing technique, I turned to 2K structural epoxies and polyurethanes. The adhesives selected being widely used in the European construction industry, thus ensuring the practical relevance of this research. Despite being illustrated on a subset of Curie particles, adhesives, adherends and joint geometries, the results are easely transferable to other bonding contexts.

The first and most important novelty offered by this thesis is the design of the CP-curing technique as a process. I worked out a comprehensive research structure, which opens up a way for a holistic understanding of the method. I subdivided the process into different working steps, all of which relevant to practitioners. These include the formulation of requirements for the adhesive and the CP, the mixing procedure, the curing progression during inductive heating and cooling, and finally the behaviour when the connection has cooled down to RT. Based upon this structuring, future particle-controlled accelerated curing processes can be implemented much more efficiently. This makes my thesis a useful contribution, as the presented 'process design' is directly implementable by practitioners.

The second innovation of my thesis represents the throughout thermo-analytical, mechanical as well as thermo-mechanical analysis of CP-cured 2K adhesives using susceptors with a T_c adapted to the temperature range optimal compatible for the selected polymers (80–120 °C) in order to specifically avoid overheating, i.e. irreversible bond damages.

I used the full potential of laboratory analytics to assess the influences of the CP-curing technique on the mechanical behaviour of the bulk and of bonded components. Investigations highlighted the effects of

adding CP on e.g. curing kinetics (1st publication) and changes of intrinsic adhesive properties such as stiffness and cohesive strength (2nd publication). Then, inductive heating experiments were scaled up to the level of joints. Firstly, on yet relatively small single lap shear joints (SLJ, 3rd publication), and secondly on large joint geometries such as Glued-in rods (GiR, 4th publication) or single lap shear tubular joints (SLTJ, 8th publication). For that, mechanical testing was performed for the first time to visualise effects of CP-curing on the joints fracture behaviour. The experiments revealed that CP-cured joints reach strengths equivalent to unfilled RT-cured reference sets, with curing times being significantly reduced. Based upon this extensive experimental work, it was possible to demonstrate size effects between the different joint geometries, especially with regard to the resulting heating behaviour.

With regard to the mechanical behaviour of the investigated 2K adhesive, two basic observations were additionally made: On the one hand, mechanical properties of the connections are deteriorated by the mere presence of the CP. This leads to higher viscosities, poorer adherend wettability, and stress peaks around the CP and their associated negative impacts on cohesive strength. On the other hand, curing at elevated temperatures improves the adhesives properties. In their combination, cohesive strength of CP-cured adhesive bulk specimens was equal to that of the reference sets.

Additionally, the influence of RT-conditioning after inductive heating ended was described for the first time (3rd and 8th publication). I showed that conditioning times at RT after inductive heating ended lead to subtle, yet not significantly altered joint strengths. I posit this to be related to strength and stiffness building up differently with temperature and time, with strength being already fully developed after the end of inductive heating and advancement of stiffness, i.e. state of stress throughout the respective joint, not yet completed.

The next major achievement of my work was to provide a clear summary of all boundary conditions to be observed for most homogeneous heating of CP-cured adhesives. Probably the most significant new finding is the role of exothermy, which has a decisive influence on temperature development within the adhesive layer, and consequently throughout the whole joint. My research highlighted that not only the absolute enthalpy is to be considered, but also the rate at which it is released. Aforementioned result is particularly important as overheating can occur despite CP-heating being capped by T_c. The adverse effects of the exothermic runaway reaction are also dependent upon the amount of adhesive present and the thermal insulation provided by the joint. The thermal insulation itself is dependent upon geometry and thermal properties of the adherends, most notoriously heat capacity and thermal conductivity.

I also made significant steps on the analytical and numerical level. The analytic kinetic models developed in my 1st paper allowed for a prediction of the curing degree, α, as a function of curing temperature and CP content, aspects yet not found in current literature. The complexity of these models was integrated into a numerical model based upon the Finite Element Method (FEM) capable of predicting the development of the curing degree, α. My extensive literature reviews clearly showed that such a simulation tool did not exist before. This advance opened up the possibility for quick and efficient identification of

critical boundary conditions. The developed numerical approach was presented in my 5th, and successfully validated and applied in my 6th publication.

In addition to these achievements, I applied the CP-curing technique at low temperatures, under which curing of 2K adhesives either would last many days, or not occur at all. The experiments were carried out on the level of large-scale GiR in my 7th publication and proved that the method is principally capable to accelerate curing even under these adverse conditions.

Based on these extensive findings, it must thus be concluded that my thesis represents a significant contribution both in context of particle-induced accelerated curing as well as in context of accelerated curing in general. In addition to the technical novelties outlined, the structural innovation of the thesis should also be emphasised, as it may be an example for future works focusing on accelerated curing, aiming to establish more clarity or even standardisation to these kind of processes.

17 Outlook

No research answers all questions, and usually leads to a plethora of new ones. In the following, I highlight some of the scientific issues that could not be answered in my thesis in the required depth, and should thus be addressed by future studies focusing on the CP-curing technique. Firstly, the method should be extended to other adhesive classes, aiming to identify chemical and kinetic conditions to be met for application of the process as well as to further validate important aspects identified in this thesis.

Optimisation of CP with regard to aspects as size, composition and shape. The optimisations should result in optimised CP-heatability and much steeper switch-off at T_c. It should also mitigate reduction of cohesive strength due to stress concentrations. Appropriate coatings of the CP so to optimise their embedment in the matrix, prevention of sedimentation and agglomeration are also worth of further investigations.

Extended investigations of the failure patterns to be carried out at the microscopic level, using e.g. scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX) or X-ray photoelectron spectroscopy (XPS). These should focus on evaluating the following issues: identification of possible adhesive damages, homogeneity of curing degree over specimen's width, characterisation of adhesion between CP and polymer matrix, identification of breaking edge progression (along CP or through matrix), in-depth validation of CP distribution as well as exclusion of significant sedimentation / agglomeration.

Extension of the numerical model towards inclusion of the EMF as well as mechanical aspects. Firstly, this would allow for the induction parameters (power, current, frequency) to be considered for modelling the heatability of CP. Further optimisation of the manufacturing setup (coil geometry, coupling distance, and field concentrators) can then be performed, leading to more efficient, thus less costly, CP-curing processes. Secondly, the development of stiffness, strength and chemical shrinkage resulting from CP addition and elevated temperature curing may be addressed. This might offer a way to gain fundamental knowledge about yet open questions such as the impact of RT-conditioning after induction curing on the mechanical joint performance.

Lastly, long-term effects of the embedded CP, including their behaviour under environmental loads, most notably moisture and temperature, is still to be experimentally investigated. The same applies to the assessment of their influence under repeated loads, as under fatigue.

Part V: Personal information

18 Curriculum vitæ

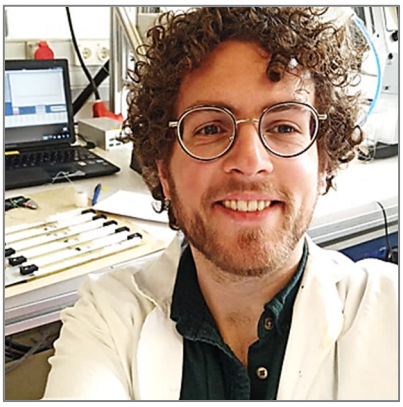
Personal data

Date of birth: 13.04.1990

Place of birth: Melle, Germany

Nationality: German

ResearchGate: https://www.researchgate.net/profile/Morten-Voss



Professional experience

03/2018 – 04/2023

Fraunhofer Institute for Manufacturing Technology and Advanced Materials (IFAM), Bremen

Position: RESEARCH ASSOCIATE / DOCTORAL STUDENT
Department: ADHESIVE BONDING TECHNOLOGY

Supervision of the following public research projects:

1. '**iCurie**' – *Site-optimised accelerated curing in wood construction* (AiF, IGF project no. 19499N)

   The research project investigated the potential of accelerated curing using inductively heated Curie particles added to two-component adhesives in context of structural timber engineering. The particles can only be heated up their Curie temperature, which eliminates the need to apply costly temperature monitoring during the accelerated curing process. Furthermore, the process allows for low-temperature curing.

2. '**Facido**' – *Process-reliable accelerated curing of adhesives with debonding option* (AiF, IGF project no. 201)

   The objective of the project was to enable accelerated curing of different adhesives by the use of inductively heated Curie alloys. The process was applied to several applications such as the joining of concrete, aircraft runways or the bonding of plastics with car windshields. Furthermore, the possibility of debonding via the introduced particles was investigated.

3. '**Hüttenzauber**' – *Year-round, quality-assured bonding of threaded rods in timber construction* (AiF, IGF project no. 19256N)

   In Hüttenzauber, a process concept based on the induction technique was developed that enables year-round bonding of threaded steel rods into load-bearing timber construction elements (regardless of ambient temperatures). For this purpose, practical processes for defined adhesive heating and temperature conditioning of the material were investigated.

4. '**NOISYC**' – *Non-surface contact inspection system for composites* (BMWi, funding code 20Q1520C)

   Non-destructive testing of FRP is currently very complicated and can often only be implemented with the help of ultrasonic sensors permanently connected to the structure. Therefore, the objective in NOISYC was to develop a non-destructive testing system for FRP components, which allows contact- and residue-free ultrasonic testing. In this context, extensive numerical models were developed that allow for a realistic prediction of the ultrasonic wave propagation in various FRP components.

5. '**HYPE**' – *Hybrid, non-contact ultrasonic testing method for complex fibre composite components with multiple actuators and virtual result alignment* (BMWi, funding code 20W1926C)

   The research project HYPE represents the follow-up project of NOISYC. The objective of the project is to transfer the numerical and experimental findings from NOISYC to more complex FRP components with curved surfaces and varying thicknesses. Ultimately, an intuitive, self-learning, simulation-based tool is to be developed which enables easier and efficient NDT of FRP components.

6. '**TACITUS FAQs**' – *Investigation and clarification of aspects for an economical application of hardwood constructions with Glued-in Rods* (AiF, IGF project no. 21550N)

   The core of the research project is to investigate practical aspects such as the durability as well as the swelling and shrinking behaviour of steel bars glued into hardwood under alternating climate conditions. The investigations are carried out on experimental as well numerical level, with the simulations aiming to realistically predict the mechanical behaviour of Glued-in Rods under the influence of humidity and temperature.

Tasks as research associate at the FRAUNHOFER IFAM:

- Development of analytical and experimental solutions to achieve the project-specific objectives.
- Execution of experimental investigations using standardised as well as self-developed test equipment in the field of bonded joints.
- Mechanical and thermo-mechanical characterisation of adhesives and bonded components.

- Performance of ageing experiments for bonded joints.
- Implementation of kinetic analysis for different adhesives using appropriate software aiming for a prediction of polymeric curing processes.
- Development of simulation models based on the Finite Element Method (FEM) in the field of hybrid bonded structures.
- Organisation and presentation of scientific results at the meetings of the project committees.
- Elaboration of project outlines for future public research projects.
- Elaboration of project outlines as well as processing of industrial projects in close coordination with partners from industry.
- Supervision of student assistants and student theses.
- Writing of scientific publications (see list further below).
- Presentation of scientific results at conferences in Germany and abroad (see list further below).

07/2016 – 02/2018 **Fraunhofer Institute for Manufacturing Technology and Advanced Materials (IFAM), Bremen**

Position: STUDENT ASSISTANT
Department: ADHESIVE BONDING TECHNOLOGY

- Supervision of the public research project NOISYC funded by the German BMWi for the development of a non-contact surface ultrasonic system for non-destructive testing of composite materials.
- FE-modelling of bonded joints in the field of hybrid as well as multifunctional structures.
- Fabrication and mechanical testing of bonded joints to support project-related work contents.
- Execution of manual activities in the laboratories.

02/2015 – 06/2016 **Bremen Institute for Mechanical Engineering (BIME), Bremen**

Position: STUDENT ASSISTANT
Field of activity: ASSEMBLY SYSTEMS

- Computer Aided Design (CAD) of an experimental assembly environment for large-scale assemblies.
- Preparation and implementation of a software-supported assembly laboratory in the field of industrial small appliance assembly.
- Support of research assistants by searching and structuring of scientific literature.
- Implementation of manual activities in the laboratories.

03/2013 – 04/2014 **Leibniz Institute for Materials Engineering (IWT), Bremen**

Position: STUDENT ASSISTANT
Department: PRODUCTION ENGINEERING

- Preparation and execution of experimental studies for the aerospace industry (Airbus Operations GmbH, Bremen) on a CNC orbital drilling unit.
- Application of innovative drilling methods such as clean drilling or vibration drilling on material composites.
- Preparation and evaluation of test data to support research assistants.
- Incorporation of new student assistants.

Scientific contributions

Peer-reviewed publications

03/2023 **'The contribution of numerical models to Lamb-wave-driven NDT processes – Part II: Experimental design and numerical studies'**

M. Voß, A. Szewieczek, W. Hillger, T. Vallée, F. von Dungern
Advanced Composite Materials
https://doi.org/10.1080/09243046.2023.2182532

11/2022 **'The contribution of numerical models to Lamb-wave-driven NDT processes – Part I: Model building'**

M. Voß, A. Szewieczek, W. Hillger, T. Vallée, F. von Dungern
Advanced Composite Materials
https://doi.org/10.1080/09243046.2022.2147042

07/2022 **'Controlling the mixing quality of 2K adhesives by means of electrical capacitance tomography – Part I: Necessary polymer characteristics'**

S. Voß & M. Voß
The Journal of Adhesion
https://doi.org/10.1080/00218464.2022.2102902

04/2022 **'Low-temperature curing of adhesives – Large-scale experiments'**

M. Voß, T. Evers & T. Vallée
The Journal of Adhesion
https://doi.org/10.1080/00218464.2022.2059353

12/2021 **'Experimental investigations on pre-tensioned hybrid joints for structural steel applications'**

C. Denkert, T. Gerke, R. Glienke, M. Dörre, K.-M. Henkel, H.Fricke, S. Myslicki, M. Kaufmann, M. Voß & T. Vallée
The Journal of Adhesion
https://doi.org/10.1080/00218464.2021.2003786

09/2021 **'Accelerated curing of glued-in rods: Influence of manufacturing defects'**

N. Ratsch, S. Böhm, M. Voß & T. Vallée
Construction and Building Materials
https://doi.org/10.1016/j.conbuildmat.2021.123665

07/2021 **'Curie-supported accelerated curing by means of inductive heating – Part I: Model building'**

M. Voß, T. Vallée & M. Kaufmann
The Journal of Adhesion
https://doi.org/10.1080/00218464.2021.1951712

07/2021 **'Curie-supported accelerated curing by means of inductive heating – Part II: Validation and numerical studies'**

M. Voß, T. Vallée & M. Kaufmann
The Journal of Adhesion
https://doi.org/10.1080/00218464.2021.1951712

07/2021 **'Accelerated curing of adhesively bonded G-FRP tube connections – Part III: Modelling of strength'**

M. Voß, T. Vallée & M. Kaufmann
Composite Structures
https://doi.org/10.1016/j.compstruct.2021.113900

07/2021 **'Accelerated curing of adhesively bonded G-FRP tube connections – Part I: Experiments'**

M. Voß & J. Haupt
Composite Structures
https://doi.org/10.1016/j.compstruct.2021.113999

05/2021 **'Modelling and strength prediction of pre-tensioned hybrid bonded joints for structural steel applications'**

T. Vallée, H. Fricke, S. Myslicki, M. Kaufmann, M. Voß, C. Denkert, R. Glienke, M. Dörre, M.-K. Henkel & T. Gerke
The Journal of Adhesion
https://doi.org/10.1080/00218464.2021.1928498

05/2021 **'Effects of Curie particle induced accelerated curing on thermo-mechanical performance of 2K structural adhesives – Part I: Bulk properties'**

M. Voß & T. Vallée
The Journal of Adhesion
https://doi.org/10.1080/00218464.2021.1909482

02/2021 **'Effects of Curie particle induced accelerated curing on thermo-mechanical performance of 2K structural adhesives – Part II: Lap shear properties'**

M. Voß & T. Vallée
The Journal of Adhesion
https://doi.org/10.1080/00218464.2021.1884551

02/2021 **'Fast inductive curing of adhesively bonded glass-timber joints'**

J. Wirries, M. Adam, C. Tornow, M. Noeske, T. Vallée & M. Voß
The Journal of Adhesion
https://doi.org/10.1080/00218464.2021.1880903

01/2021 **'Accelerated curing of G-FRP rods glued into timber by means of inductive heating – Influences of curing kinetics'**

M. Voß & T. Vallée
The Journal of Adhesion
https://doi.org/10.1080/00218464.2020.1870450

10/2020 **'Resistive curing of glued-in rods'**

N. Ratsch, M. Burnett-Barking, S. Böhm, S. Myslicki, M. Voß, M. Adam & T. Vallée
Construction and Building Materials
http://dx.doi.org/10.1016/j.conbuildmat.2020.121127

09/2020 **'Accelerated curing of glued-in threaded rods by means of inductive heating – Part IV: curing under low temperatures'**

N. Ratsch, M. Burnett-Barking, S. Böhm, M. Voß, M. Kaufmann & T. Vallée
The Journal of Adhesion
https://doi.org/10.1080/00218464.2020.1818562

08/2020 **'Accelerated curing of G-FRP rods glued into timber by means of inductive heating using Curie particles – large-scale experiments at room temperature'**

M. Voß & T. Vallée
The Journal of Adhesion
https://doi.org/10.1080/00218464.2020.1803067

12/2019 **'Accelerated curing of glued-in threaded rods by means of inductive heating – Part III: transient curing'**

N. Ratsch, S. Böhm, M. Voß, M. Adam, J. Wirries, N. Dreves & T. Vallée
The Journal of Adhesion
https://doi.org/10.1080/00218464.2019.1699071

11/2019 **'Influence of imperfections on the load capacity and stiffness of glued-in rod connections'**

N. Ratsch, S. Böhm, M. Voß, M. Kaufmann & T. Vallée

CONSTRUCTION AND BUILDING MATERIALS
http://dx.doi.org/10.1016/j.conbuildmat.2019.07.278

11/2019 **'Numerical simulation of the propagation of Lamb waves and their interaction with defects in C-FRP laminates for non-destructive testing'**

M. VOß, D. ILSE, W. HILLGER, T. VALLÉE, M. EPPMANN, J. DE WIT & F. VON DUNGERN
ADVANCED COMPOSITE MATERIALS
https://doi.org/10.1080/09243046.2019.1692273

08/2019 **'Accelerated curing of glued-in threaded rods by means of inductive heating – Part II: modelling'**

N. RATSCH, S. BÖHM, M. VOß, M. ADAM, J. WIRRIES & T. VALLÉE
THE JOURNAL OF ADHESION
https://doi.org/10.1080/00218464.2019.1654865

08/2019 **'Accelerated curing of glued-in threaded rods by means of inductive heating – Part I: experiments'**

N. RATSCH, S. BÖHM, M. VOß, M. ADAM, J. WIRRIES & T. VALLÉE
THE JOURNAL OF ADHESION
https://doi.org/10.1080/00218464.2019.1654864

09/2018 **'Influence of manufacturing methods and imperfections on the load capacity of glued-in rods'**

D. KOHL, N. RATSCH, S. BÖHM, M. VOß, M. KAUFMANN & T. VALLÉE
THE JOURNAL OF ADHESION
https://doi.org/10.1080/00218464.2018.1508351

Non-peer-reviewed publications

04/2021 **'Einfluss konstruktiver Details auf Klebungen pultrudierter GFK-Lamellen'**

A. KRUSE, C. GRUNWALD, M. KAUFMANN, M. VOß & T. VALLÉE
ADHÄSION KLEBEN & DICHTEN
https://doi.org/10.1007/s35145-021-0490-x

03/2021 **'Schnellhärtung geklebter FVK-Rohre durch Induktion (Teil 2)'**

J. HAUPT, M. VOß, M. KAUFMANN, C. GRUNWALD, T. VALLÉE & M. BODE
ADHÄSION KLEBEN & DICHTEN
https://doi.org/10.1007/s35145-021-0480-z

12/2020 **'Schnellhärtung geklebter GFK-Rohre durch Induktion (Teil 1)'**

J. HAUPT, M. VOß, T. VALLÉE & M. ALBIEZ
ADHÄSION KLEBEN & DICHTEN
https://doi.org/10.1007/s35145-020-0466-2

08/2020 **'Einkleben von Gewindestangen in Laubholz bei niedrigen Temperaturen'**

N. RATSCH, S. BÖHM, S. MYSLICKI & M. VOß
BAUTECHNIK
http://dx.doi.org/10.1002/bate.202000077

Conference contributions

07/2021 **'AB2021 – 6th International Conference on Adhesive Bonding', Porto, Portugal.**

Presentation: '*Load capacity prediction of bonded G-FRP tube connections*'

T. VALLÉE, M. VOß, M. KAUFMANN, H. FRICKE & M. ALBIEZ

Presentation: '*Enhancing the performance of glued-in rods at elevated temperatures*'

N. RATSCH, S. BÖHM, M. VOß, T. VALLÉE & T. TANNERT

Prensetation: '*Experimental investigations on pre-tensioned hybrid joints for structural steel applications*'

T. GERKE, C. DENKERT, M. DÖRRE, M-K. HENKEL, S. MYSLICKI, H. FRICKE, M. VOß, M. KAUFMANN & T. VALLÉE

Presentation: '*Accelerated curing of glued-in rods: Influence of manufacturing defects*'

N. RATSCH, S. BÖHM, M. VOß & T. VALLÉE

Presentation: '*Capacity prediction of pre-tensioned hybrid joints for structural steel applications*'

T. GERKE, R. GLIENKE, M. DÖRRE, M.-K. HENKEL, S. MYSLICKI, H. FRICKE, M. VOß, M. KAUFMANN & T. VALLÉE

Presentation: '*Numerical modelling of Curie-supported accelerated curing by means of inductive heating*'

M. KAUFMANN, M. VOß & T. VALLÉE

Presentation: '*Validation and numerical studies of Curie-supported accelerated curing by means of inductive heating*'

M. VOß, M. KAUFMANN & T. VALLÉE

Presentation: '*Influences of Curie-susceptors induced accelerated curing on thermo-mechanical performance of 2K structural adhesives*'

M. VOß, M. KAUFMANN & T. VALLÉE

Poster: '*Experimental investigations on accelerated curing of adhesively bonded GFRP tube connections*'

M. VOß, M. KAUFMANN, H. FRICKE, T. VALLÉE & H. FRICKE

07/2019 **'AB2019 – 5th International Conference on Adhesive Bonding', Porto, Portugal.**

Presentation: '*Resistively heated glued-in-rods in beech LVL – Part I: Experimental results*'

N. RATSCH, S. BÖHM, M. VOß, J. WIRRIES, M. ADAM, T. VALLÉE & S. MYSLICKI

Presentation: '*Glued-in rods resistively and inductively cured under low temperatures*'

N. RATSCH, S. BÖHM, M. VOß, M. ADAM, J. WIRRIES & T. VALLÉE

Presentation: '*Fast inductive curing of adhesively bonded glass-timber joints – Part II: In-depth interpretation of the results*'

M. VOß, J. WIRRIES, M. ADAM, T. VALLÉE, C. TORNOW, M. NOESKE, J. DERKSEN, K. THIEL & K. BRUNE

Poster: '*Influence of deviations and imperfections on glued-in rods cured by means of induction heating*'

N. RATSCH, S. BÖHM, M. VOß, M. ADAM, J. WIRRIES & T. VALLÉE

Poster: '*Accelerated curing of G-FRP rods glued into timber by means of inductive heating – Part I: Experimental results*'

M. VOß, M. ADAM, J. WIRRIES, T. VALLÉE, N. RATSCH & S. BÖHM

Poster: '*Accelerated curing of G-FRP rods glued into timber by means of inductive heating – Part II: Modelling*'

M. VOß, M. ADAM, J. WIRRIES, T. VALLÉE, N. RATSCH & S. BÖHM

05/2019 **'4SMARTS – Symposium for Smart Structures and Systems', Darmstadt, Germany.**

Presentation: '*Numerical simulation of the propagation of Lamb waves and their interactions with defects in C-FRP laminates for non-destructive testing*'

M. VOß, D. ILSE, W. HILLGER, T. VALLÉE, M. EPPMANN, J. DE WIT & F. VON DUNGERN

05/2019 **'2nd SAFE-FLY International Conference – SHM technologies for future European aircraft generation', Vitoria-Gasteiz, Spain.**

Presentation: '*Numerical simulation of the propagation of Lamb waves and their interactions with defects in C-FRP laminates for non-destructive testing*'

M. VOß, D. ILSE, W. HILLGER, T. VALLÉE, M. EPPMANN, J. DE WIT & F. VON DUNGERN

02/2019 **'19th Colloquium: Joint research in adhesive bonding technology', Cologne, Germany.**

Presentation: '*Site-optimised accelerated curing in timber construction*'

M. VOß, M. ADAM, T. VALLÉE, N. RATSCH, D. KOHL & S. BÖHM

09/2019 **'21st International Conference on Composite Structures (ICCS21)', Bologna, Italy.**

Presentation: '*Numerical simulation of the propagation of Lamb waves and their interactions with defects in C-FRP laminates for non-destructive testing*'

M. VOß, D. ILSE, W. HILLGER, T. VALLÉE, M. EPPMANN, J. DE WIT & F. VON DUNGERN

Academic and school education

Years of study

03/2018 – 03/2023 **Doctoral studies, University of Bremen**

Institute: FRAUNHOFER INSTITUTE FOR MANUFACTURING TECHNOLOGY AND ADVANCED MATERIALS (IFAM), Bremen

Institutes research focus: APPLIED ENGINEERING AND MATERIALS SCIENCES FOR METALS AND POLYMERS.

Dissertation topic: '*Accelerated curing of adhesives using inductively heated Curie particles*'

Intended degree: DOCTOR OF ENGINEERING (Dr.Eng. / Dr.-Ing.)

04/2015 – 02/2018 **Master's programme 'Industrial Engineering – Production Technology', University of Bremen**

Major field of study: SYSTEM DEVELOPMENT AND INNOVATION

Master thesis topic: '*Numerical simulation of the propagation of Lamb waves and their interaction with defects in C-FRP laminates for non-destructive testing*' (Grade = 1.0)

Obtained degree: MASTER OF SCIENCE (M.Sc., Grade: 1.59)

10/2010 – 03/2015 **Bachelor's programme 'Industrial Engineering – Production Technology', University of Bremen**

Major field of study: MANUFACTURING TECHNOLOGY

Bachelor thesis topic: '*Guidelines for the design of tools and cutting parameters for vibration drilling using Finite Element simulation*' (Grade: 1.3)

Obtained degree: BACHELOR OF SCIENCE (B.Sc., Grade: 2.33)

08/2009 – 07/2010 **Voluntary social year at the rescue and ambulance service in the district of Osnabrück (DRK), rescue station Melle**

Position: RESCUE ASSISTANT

Range of tasks: Support of the full-time rescue staff during emergency rescue and ambulance services.

School time

08/2007 – 04/2009 **Grammar school at the market, Bünde**

Obtained degree: GENERAL MATRICULATION STANDARD (Grade: 2.9)

08/2002 – 07/2007 **Grammar school, Melle**

08/2000 – 07/2002 **Orientation stage, Melle**

08/1996 – 07/2000 **Primary school Grönenberg, Melle**

Internships and sideline activities

05/2014 – 09/2014 and 11/2014 **Airbus Operations GmbH, Bremen**

Position: TRAINEE

Range of tasks:

- Preparation, execution and evaluation of corrosion tests on self-pierce riveted joints.
- Preparation of test specimens on various machine tools for experimental investigations.
- Execution and analysis of different riveting processes.
- Optimisation of production processes for landing flaps production.

08/2011 – 09/2011 **Cool it Isoliersysteme GmbH, Melle**

Position: PRODUCTION ASSISTANT

Range of tasks:

- Assembly and dispatch of door and gate systems for the food and chemical industry.
- Execution of various manual activities in the production.

08/2010 – 09/2010 **Schomäcker Federnwerk GmbH, Melle**

Position: TRAINEE

Range of tasks:

- Assembly, quality control and shipment of different spring models for commercial vehicles.
- Execution of various forming and riveting processes as well as shot peening.

- Processing of corrosion protection and paints.

as well as 05/2009 – 06/2009 and 03/2011

Position: PRODUCTION ASSISTANT

Range of tasks:

- Assembly, quality control and shipment of various spring models for commercial vehicles.
- Execution of various manual activities in the production.

Additional qualifications

EDP skills

Computer Aided Design (CAD)	Autodesk Inventor, Siemens NX10 (good knowledge), Catia V5, Pro Engineer (advanced knowledge)
Data processing	Microsoft Office, Origin Pro 2020 (expert knowledge)
Finite Element Analysis (FEA)	Ansys, Deform 10 (very good knowledge), PZFlex (expert knowledge)
Other	Netzsch Kinetics Neo (very good knowledge), Axiovision, LabVIEW (basic knowledge)

Trainings

10/2018 – 12/2018	Training as '*DVS/EWF – European Adhesive Specialist (EAS)*' at the Adhesive Bonding Technology Centre of the FRAUNHOFER IFAM in Bremen, Germany.
02/2017	Training as '*DSV/EWF – European Adhesive Bonder (EAB)*' at the Adhesive Bonding Technology Centre of the FRAUNHOFER IFAM in Bremen, Germany.

Languages

English	Very good knowledge (certificate B2)
French	Basic knowledge

Driving license Classes B and C1

References

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