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Joining composite materials with reactive nickel-aluminium particles as an innovative additive in epoxy-based adhesives

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Abstract. Joints comprised of materials with different thermo-physical properties are often advantageous for lightweight components. However, the fabrication of such joints has become a key issue for joining technologies. An innovative approach to overcome the associated challenges is the use of reactive particles. Each reactive particle contains at least two reactants, which are able to undergo an exothermic, self-sustaining reaction after the ignition by an external energy input. Depending on the stoichiometric ratio of the reactants and the heating rate, temperatures up to 1500 K are reached within milliseconds for the nickel and aluminium system. In the automotive industry, bonding is state of the art for applications in electromobility. Yet, slow cross-linking of the adhesive increases cycle times and results in an initial low handling strength, which requires additional mechanical fixings. The benefits of using reactive particles in epoxy-based adhesives are shown in this paper. Varying quantities of reactive particles were integrated into two part epoxy adhesives and activated with microwave energy. Due to the promotion of the cross-linking of the epoxy resin and the reaction of the particles by the microwaves, curing times were significantly shortened. Joining aluminium and polypropylene specimens demonstrated the potential of the presented approach.

1. Introduction

Joining technologies play a key role in production engineering as they enable the use of composite materials for a resource-efficient lightweight design. A versatile and industrially established technology for joining components with different thermo-physical properties is adhesive bonding. However, processing reactive adhesives like epoxy resins, which are widely used due to their excellent temperature and environmental resistance, requires a certain period of time because of the curing reaction. In order to guarantee reasonable cycle times, additional mechanical fixings are often applied to overcome the initial low handling strength of the joint. Reactive particles, which were originally used in the field of combustion synthesis for fabricating high performance materials, represent a promising approach to reduce curing times. The exothermic reaction of each particle can be utilised as a tailored heat source to promote the cross-linking within the epoxy resin's network. By integrating reactive particles into adhesives, the advantageous characteristics of both constituents are combined in an innovative way.

1.1. Reactive particles in production engineering

Combustion synthesis, also denoted as self-propagating high temperature synthesis, has become an important field of research in academia and industry since the discovery of the solid flame phenomenon by A. G. Merzhanov [1]. The fundamental reason for this development is the unique



reaction characteristic of reactive systems. Two or more homogeneously mixed reactants undergo a self-sustaining, highly exothermic reaction after a defined energy input. As the term combustion synthesis implies, the main research focus is on the production of high performance materials, such as carbides, borides, nitrides, intermetallics, and composite powders [2]. The released energy allows the sublimation of impurities during the reaction and results in high-purity alloys. Another valuable approach is not to consider the reaction product, but the targeted use of the reaction heat. In propellants, the exothermic reaction of nickel and aluminium can be used to influence and stabilise the combustion reaction [3]. Furthermore, joining technologies, like welding, soldering, and bonding, require a controllable heat source, which can be provided by reactive systems.

In order to release the reaction heat in a defined way, the type of energy input to initiate the chemical reaction plays an essential role. Basically, two theoretical reaction modes need to be distinguished when studying the activation and reaction behaviour of reactive systems: the propagating and the simultaneous combustion mode [4]. A locally confined energy input into the reactive system results in the propagating mode and in the formation of a reaction front. Typical activation methods include for example a mechanical impact [5], a heated wire [6], or a laser beam [7]. The self-sustaining reaction front propagates through the reactive system until the reactants are fully converted into products. The simultaneous combustion mode occurs if the reactive sample is homogeneously heated to the ignition temperature, e. g. by a furnace [8] or electromagnetic radiation [9], and the combustion reaction is concurrently activated at each position within the reactive system.

Due to the high reaction rates and theoretical maximum adiabatic temperatures of up to 1910 K [10] for the nickel and aluminium system, an efficient energy transfer into the reactive system before and after the initiation is possible with electromagnetic radiation. Microwaves at a frequency of 2.45 GHz interact in an advantageous way with reactive systems and allow rapid and homogeneous heating [11].

Reactive powder materials, such as metallic particles, are commonly used in the field of combustion synthesis. For joining applications, a specific type of reactive systems has been developed. The so-called reactive multilayer systems consist of hundreds of alternating layers of the reactants [12]. However, due to their brittle nature as well as stability and handling issues [13] they are primarily suitable for joining planar or slightly curved surfaces. Reactive powder materials represent a more flexible alternative. As each particle contains at least two reactants, reactive particles can be used as an adaptable heat source. Figure 1 shows different intrinsic structures of reactive particles.

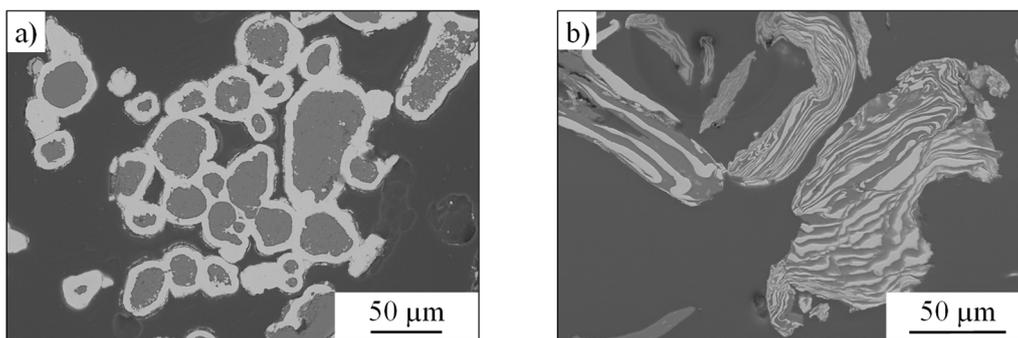


Figure 1. Scanning electron micrograph images of reactive particles with an intrinsic a) core-shell and b) lamellar structure.

Reactive particles with a lamellar structure are usually synthesised via ball milling [14]. A less common method is to shred reactive multilayer systems. Electroless [15] and electrochemical [16] plating processes of metal particles are efficient routes to obtain core-shell structures. As the size, shape, and stoichiometric ratio of the reactants are adjustable through the synthesis and significantly

influence the activation and reaction behaviour and thereby the released energy, reactive particles provide a customisable heat source for a wide range of joining applications.

1.2. Interactions between adhesives, additives, and microwaves

Epoxy resins are used in various ways in production engineering, e. g. as surface coating, fibre-reinforced composite, and as highly relevant adhesive [17]. In this paper, the term epoxy resin refers to both the prepolymer with the reactive epoxy groups as well as the cured resin. Reactive epoxy groups, also denoted as oxirane, are able to form three-dimensional molecular networks with good mechanical strength as well as high temperature and excellent environmental resistance [18]. The curing agent plays a decisive role as it does not only initiate the reaction, but also determines the reaction mechanism and the resulting characteristics of the cured resin. Depending on the formulation of the single or two part epoxy resin, heat curing is used to initiate or additionally promote the chemical reaction. The major challenges of processing epoxy resins are long curing times, non-uniform polymerisation, and supplementary post-curing in order to complete the cross-linking of uncured areas.

An alternative and industrially established approach is using microwaves for curing polymers. Reduced thermal gradients and a homogeneous fast energy transfer directly into the matter are major advantages of microwave processing compared to the conventional heat transfer modes via conduction, convection, and radiation in an oven [19]. However, effective microwave curing is a demanding challenge as it depends on the formulation of the resin, the uniformity of the electromagnetic field, the operation and dimensions of the microwave oven, and the arrangement of the sample [17]. Nevertheless, comparative studies on thermal and microwave curing with different curing agents demonstrated the enhancement of reaction rates and a higher degree of curing when using microwaves [20]. Moreover, reaction mechanisms strongly depend on the operation mode of the microwave (pulsed or continuous [21]) and deviate from conventional heating [22].

The formulation of the epoxy resin and the resulting dielectric properties, particularly their change during the polymerisation [19], are of great importance for microwave curing as they determine the interaction with the electromagnetic field. Furthermore, the integration of additives into the epoxy resin is a sophisticated possibility to further enhance the absorption of electromagnetic energy. Microwave curing of epoxy resins filled with nanoscaled ferrites [23], silicon carbide nanoparticles [24], multi-wall carbon nanotubes [25], and fly ash [26] confirmed an efficient energy transfer into the composite and resulted in a considerable shortening of the curing time compared to conventional heating. In addition, energy can be directly transferred into the joint without damaging temperature-sensitive joining partners. Consequently, electromagnetic waves as well as additives have a positive effect on the curing process. As the use of reactive particles as an innovative additive in epoxy resins and the curing with microwaves have not yet been studied, this research aimed at investigating the resulting interactions.

2. Experimental setup and materials

In order to evaluate the cause-effect relationships between the reactive particles, the adhesive, and the microwave energy, various studies were conducted. First, curing times of different two part epoxy resins with varying quantities of reactive particles were investigated. Further knowledge about the influence of different quantities of other additives in two part epoxy resins and microwave power was gained by using differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). The findings obtained by that formed the basis for the preparation and the analysis of lap joints.

2.1. Preparation and analysis of the epoxy resin

The two part epoxy resin LOCTITE EA 3425 (Henkel AG & Co. KGaA) was applied with a manual dispensing gun and mixing tubes. Additives included reactive nickel-aluminium particles with a lamellar structure (Indium Corporation), manufactured by cold rolling and subsequent shredding of the

multilayer system, aluminium powder (New Materials Development GmbH, 6.9 μm , 99.7 % purity), and carbon black powder (P-1500, PENTACARBON GmbH). All materials were used as received.

The thermal analyses were carried out with instruments and crucibles by TA Instruments. DSC measurements with the thermal analyser Q2000 required Tzero aluminium pans and Tzero hermetic aluminium lids as sample holders. The heating rate was adjusted to 10 K/min and the maximum temperature to 433.15 K. For TGA measurements, platinum sample pans (100 μl) were used as crucibles in the thermal analyser Q5000SA. The heating rate was again set to 10 K/min and the maximum temperature to 873.15 K. Argon was used for both analysis methods.

2.2. Experimental setup for microwave curing

Initial curing experiments with reactive particles and two part epoxy resins were performed in a domestic microwave oven (HMT75M421, Robert Bosch Hausgeräte GmbH), which is widely used in scientific research [17] and is hereafter referred to as configuration I. For further studies, an experimental setup, which is denoted as configuration II, with a magnetron operating at a frequency of 2.45 GHz (MH2000S-215BB, Muegge GmbH) and a continuous output power was used. All samples were placed on the rectangular waveguide as illustrated in Figure 2.

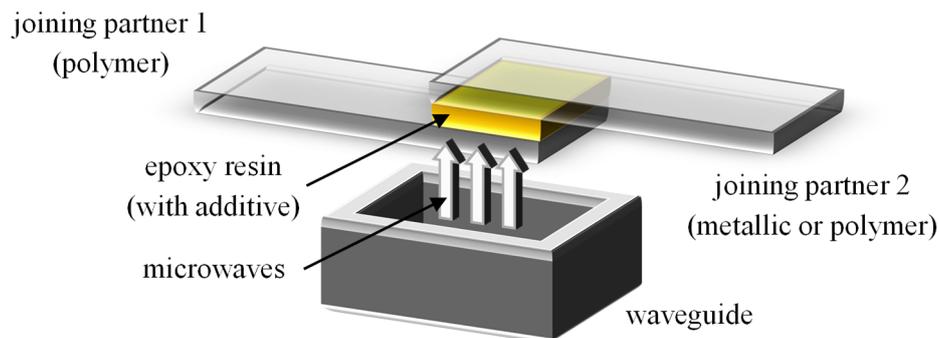


Figure 2. Scheme of the microwave curing process in configuration II.

In order to guarantee a homogenous microwave energy input into the adhesive and the additives, the joining partner facing the rectangular waveguide should not significantly interact with the electromagnetic field as this may result in unintended absorption or thermal damage. Therefore, lap joints with an overlap length of 12.5 mm consisting of a metallic and a polymer sheet as well as of two polymer sheets were studied. Dimensions of all test specimens for joining experiments were 100 x 25 x 2 mm according to DIN EN 1465.

2.3. Preparation and analysis of lap joints

Polypropylene (PP-H, Technoplast v. Treskow GmbH) was chosen as the polymer joining partner due to its bondability with two part epoxy resins and a relatively low interaction with microwaves. Moreover, the surface treatment of polypropylene is relatively simple compared to chemical etching processes. After rinsing with isopropyl alcohol and ultrasonic cleaning, an atmospheric-pressure plasma, generated by the Openair FG5001 plasma generator (PlasmaTreat GmbH), was used for the surface treatment of the polypropylene samples. Suitable settings of the plasma generator were determined by contact angle measurements (Contact Angle System, DataPhysics Instruments GmbH) and are listed in Table 1.

Aluminium sheets (EN AW-2017-A, Bikar-Metalle GmbH) were prepared by rinsing with isopropyl alcohol and ultrasonic cleaning. The distance between the joining partners was ensured by a stainless steel wire (0.20 mm, Advent Research Materials Ltd.) and a bracket. This device was made out of Teflon (PTFE virginal unfilled, KTK Kunststofftechnik Vertriebs GmbH) to avoid unintentional interaction with the electromagnetic field and adhesion of the epoxy resin.

Table 1. Parameters for the plasma treatment of polypropylene sheets.

parameter	value
travel speed of the nozzle	15 m/min
distance	10 mm
number of passes	5

The analysis of the manufactured lap joints was carried out on the basis of tensile strength tests with the single-screw tensile testing machine Z100 (ZwickRoell GmbH & Co.KG) and a speed of the crosshead displacement of 60 MPa/s.

3. Results and discussion

In the following sections, three different studies are presented. In the first test series, the potential of reactive particles as an advantageous additive in two part epoxy resins was evaluated. Second, the influence of different additives, of varying quantities of the former, and of microwave power is discussed on the basis of the thermal analysis results. Finally, suitable parameter settings were derived from the previously generated knowledge and used for joining.

3.1. Influence of reactive particles on curing times

The initial feasibility study was carried out in configuration I with varying quantities (0 wt.-%, 5 wt.-%, 10 wt.-%, 15 wt.-%) of reactive particles. The microwave power was set to 90 W, 180 W, and 360 W. Curing times were determined on the basis of five samples by indentation tests performed at regular intervals at the edges and in the centre of the sample. The results are shown in Figure 3.

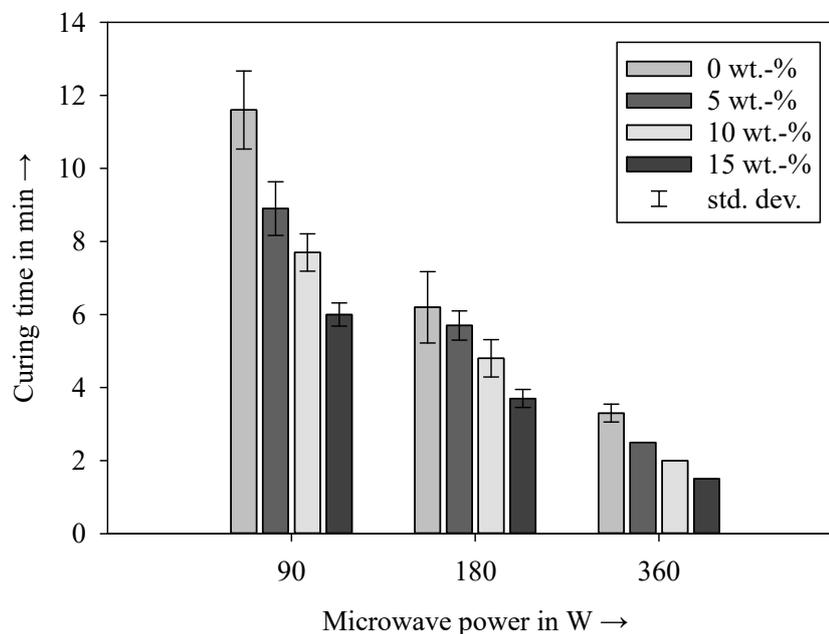


Figure 3. Curing times depending on the microwave power and the quantity of reactive particles.

It becomes obvious that both increasing microwave power and the presence of reactive particles significantly reduced curing times. As a reference, curing the two part epoxy resin LOCTITE EA 3425 at room temperature takes 24 hours according to the technical data sheet of the manufacturer. The values of the standard deviations demonstrate the good repeatability and range between 1.1 and 0. Due

to the inverse temperature profile [27], curing always started in the centre of the sample. A higher microwave power than 360 W resulted in a change of colour of the adhesive and in a significant thermal damage. Furthermore, partial sedimentation of the reactive particles could be observed at longer curing times, which should be avoided for homogeneous cross-linking. This might have also been the reason for reacted and unreacted particles within the epoxy resin, which were detected with scanning electron microscopy as illustrated in Figure 4. However, it is important to note that it is not intended to release the maximum possible energy of the reactive particles since this would severely damage the epoxy resin. Therefore, the energy input and the heating rate, which are determined by the microwave power, should enhance exothermic intermetallic reactions, such as solid state diffusion, and phase transformations only to a limited extent.

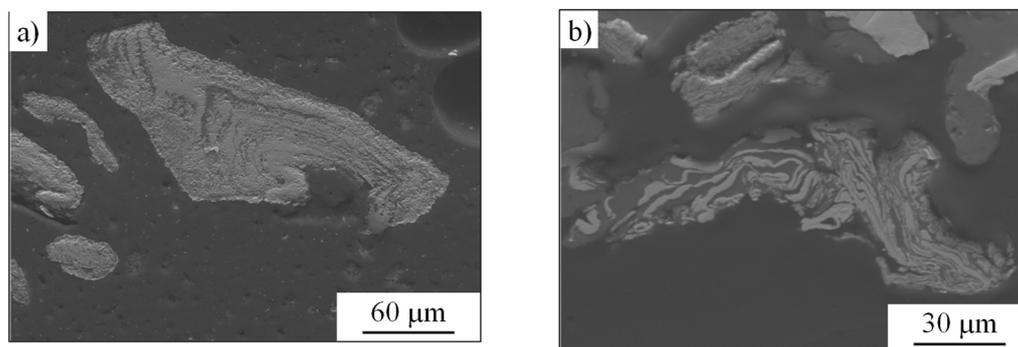


Figure 4. Scanning electron micrograph images of a) reacted particles with a relatively homogeneous nickel aluminide structure and b) unreacted particles with lamellas consisting of nickel (light grey) and aluminium (dark grey).

3.2. Results of thermal analyses

The second series of experiments included DSC and TGA measurements in order to investigate the polymerisation of the epoxy groups as well as the degree of cross-linking within the polymer network. Figure 5 shows the DSC curves for three different additives (10 wt.-%) compared to an unfilled reference on the basis of two samples.

The detected heat flow clearly indicates the exothermic polymerisation of the two part epoxy resin in all tested samples. Moreover, it can be derived that the three additives influenced the released enthalpy of the reaction. A comparison between the DSC curves reveals that the highest enthalpy of reaction is obtained for the unfilled reference sample, which does not contain any additional additive. The second highest enthalpy of reaction is observed for reactive particles. Apparently, these absorb a certain amount of energy, provided by the DSC oven, but do not significantly contribute to the overall enthalpy of the cross-linking reaction. Due to the relatively low heating rate, it is assumed that primarily solid state diffusion processes took place.

The samples for the TGA measurements were all cured in configuration II. In order to define the minimum and maximum microwave power, when operating with a continuous mode, curing times of unfilled reference samples were compared at first. By this means, thermal damage of the epoxy resin should be prevented. The microwave power was varied between 100 W and 200 W with 25 W intervals and curing times were tested as described before.

As in configuration I, curing times could be significantly reduced with increased microwave power. Curing at 100 W took approximately 16 min, while a microwave power of 200 W required 3 min for hardening. When comparing the results of configuration I (12 min at 90 W, 6 min at 180 W) with configuration II (16 min at 100 W, 3 min at 175 W), it can be assumed on the basis of the state of the art (see [17]) that the mode of operation had an influence on the polymerisation. However, curing at 360 W was not possible as significant thermal damage appeared at microwave powers above 200 W. Therefore, 200 W was chosen as maximum curing power for the epoxy resin LOCTITE EA 3425.

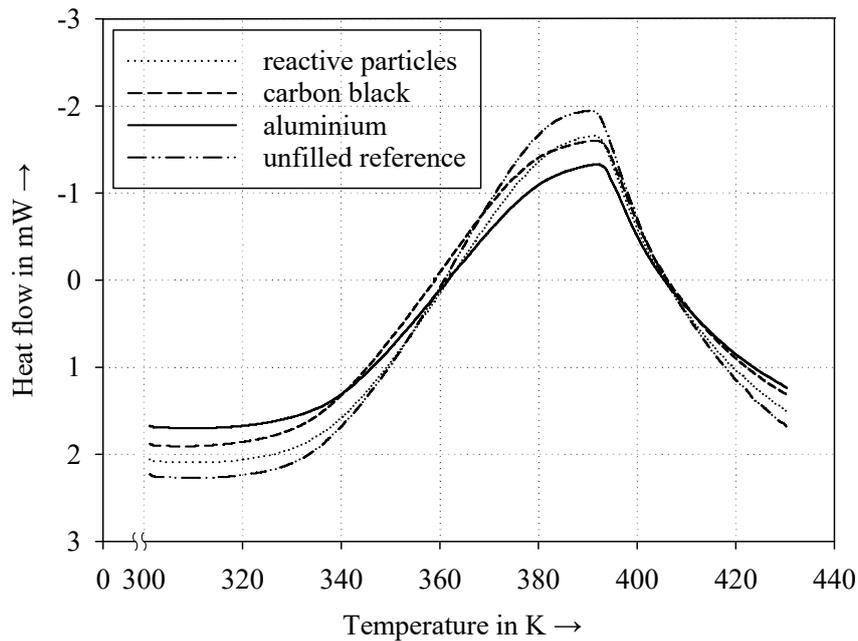


Figure 5. DSC curves of the two part epoxy resin with reactive particles, aluminium, and carbon black as additives and an unfilled reference.

TGA measurements of reference samples without additional additives were used to examine if post-curing at room temperature appeared a few minutes, 24 hours, and 48 hours after microwave processing in configuration II.

Almost identical TGA curves and degradation temperatures indicated that the samples were fully cured. The last set of experiments focussed on the interaction with the electromagnetic field and the quantity of the additive. Samples with 5 wt.-%, 10 wt.-%, and 15 wt.-% were cured at 200 W for 3 min. Figure 6 shows the corresponding TGA curves on the basis of two samples.

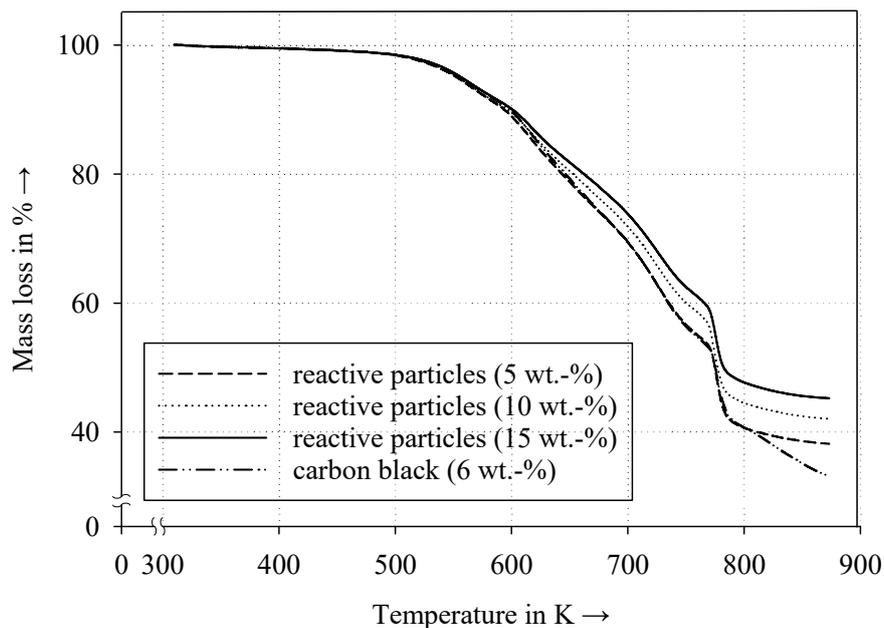


Figure 6. TGA curves of the two part epoxy resin with varying quantities of reactive particles and carbon black cured at 200 W for 3 min.

Due to the strong interaction of carbon black with the microwaves, samples with quantities above 10 wt.-% were significantly damaged. Therefore, the quantities for carbon black were reduced (3 wt.-%, 6 wt.-%, 10 wt.-%) in order to avoid evaporation of chemical components of the formulation. In configuration I, microwave processing with higher quantities of carbon black and microwave power was possible (see Figure 3). This result again highlights the significant influence of the microwave chamber and the operation mode. In contrast to reactive particles, TGA curves for carbon black were very similar to each other.

With aluminium or reactive particles as additive, no thermal damage was observed during the curing process, which could also be confirmed by the corresponding TGA curves. Identical quantities of the two metallic additives led to comparable TGA curves, which increasingly converged with reduced quantities within the epoxy resin. The comparison of the degree of cross-linking reveals that the samples with reactive particles exhibit lower degradation temperatures, which could be attributed to a too strong (pre-damage) or a too weak interaction with the electromagnetic field.

As a conclusion, it can be stated that the combination of microwave processing and reactive particles as additives enhances polymerisation and reduces curing times.

3.3. Results of tensile strength tests

As it was expected that the aluminium sheets would absorb and reflect a certain amount of the introduced microwave energy, first experiments aimed at scrutinising the transferability of the previously obtained outcomes to joining.

With the same settings as in the aforementioned studies (200 W for 3 min, configuration II) it was not possible to cure a reference sample consisting of an aluminium sheet, a polypropylene sheet, and the epoxy resin. Increasing the curing time and the microwave power led to the curing of the adhesive. Though the heating of the aluminium specimen took place to such an extent that a clear distinction between the interaction of the epoxy resin with the electromagnetic field and curing through thermal conduction was not possible anymore. In order to unambiguously evaluate the influence of the microwaves on the epoxy resin, lap joints of two polypropylene sheets were prepared and analysed. Nevertheless, the microwave power had to be adjusted to 300 W to obtain a curing time of 20 min. Figure 7 displays the obtained results of the strength tests on the basis of three samples.

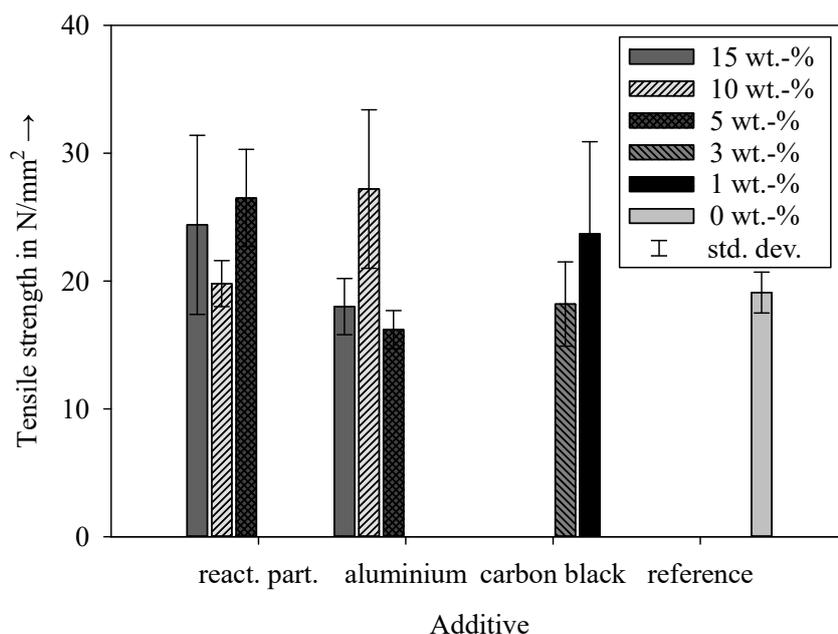


Figure 7. Tensile strength of polypropylene lap joints with varying quantities of reactive particles, aluminium powder, and carbon black as additives.

In contrary to the metallic particles, carbon black strongly interacted with the electromagnetic field above a quantity of 5 wt.-% and led to a relevant damage after a few minutes. For comparable results, the amount was reduced to 1 wt.-% and 3 wt.-%. The samples obtained by that were curable without defects.

Adhesive and cohesive fractures, hybrid forms, and failure of the base material were observed after the strength analyses. The high standard deviations impede a clear conclusion, which additive and which quantity is advantageous for joining. Additional studies should be carried out to investigate the cause-effect relationships and to further confirm the potential of reactive particles as additive in epoxy resins.

4. Conclusions

Epoxy resins are widely used in academia and industry due to their excellent thermo-mechanical characteristics. However, their long curing times represent a major disadvantage. Elaborated studies have shown that microwave processing and the use of additives are promising approaches to significantly reduce curing times and to influence the polymerisation in an advantageous way. Reactive particles, which undergo a self-sustaining exothermic reaction after an external energy input, represent an adaptable heat source for thermal joining applications. As they are also able to interact with electromagnetic fields at a frequency of 2.45 GHz, the use of reactive particles with a lamellar structure as a new, innovative additive in a two part epoxy resin was studied. The integration of reactive particles led to a considerable reduction of curing times. DSC and TGA measurements with no or further additives (aluminium and carbon black powder) revealed that the operation mode of the microwave oven strongly influenced the interaction between the epoxy resin, the additive, and the electromagnetic field. On the basis of these findings it was possible to fabricate composite joints. As the joining partners also interacted with the electromagnetic field to an essential extent, further studies on microwave processing of epoxy resins and reactive particles are required to enable an industrial usage.

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