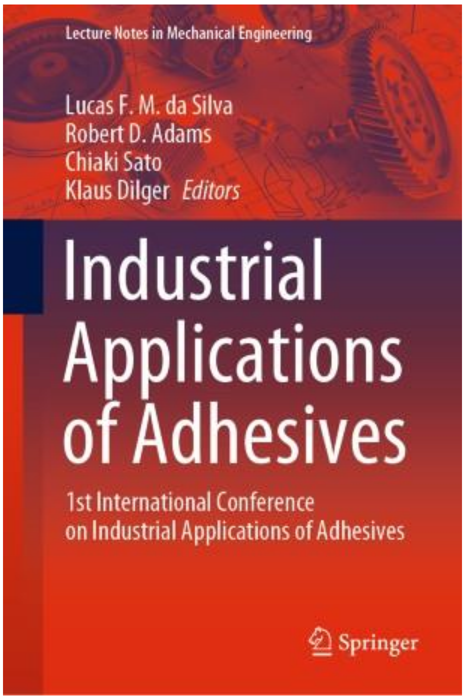

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Some Industrial Examples of Accelerated Curing Using Curie Particles

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Abstract

The aim of this paper is to assess the potential of accelerating the curing of epoxy adhesives by means of induction and ferromagnetic curie particles in the electronic and the automotive industries. Commercially available Araldite 2011 and Jowat 690 epoxy adhesives were considered, while Mn-Zn-Ferrite particles were considered for the modification of the adhesives. Induction experiments were performed at frequencies ranging from 188 kHz to 262 kHz, and with particle weight content of 16.7% and 30%. The effects of the induction frequency and the particle content on the temperature in the adhesive were investigated. In addition, the effect of the particle content on the lap shear strength of bonded joints was also studied. Moreover, a comparison between the lap shear strength of inductively cured joints and reference oven cured joints was performed. Results showed that the temperature of the adhesive increased with increasing both the induction frequency and the particle content. However, the lap shear strength of joints with Araldite 2011 was reduced by 17% when increasing the particle content to 30%. No impact on the strength was observed in the case of the Jowat 690 at 16.7% and 30% particle contents. This suggests that increasing the induction frequency is more efficient in increasing the adhesive temperature without compromising the lap shear strength of the brittle adhesive. Furthermore, the inductively cured samples of the Araldite 2011 with 30% particles achieved a substantial 120% of the reference lap shear strength of oven cured samples in 8% of the curing time. For samples with Jowat 690 with 30% particles, the inductively cured samples achieved 43% of the reference lap shear strength of oven cured samples in 8% of the curing time. This shows a high potential of accelerating the curing time for industrial applications by induction and ferromagnetic curie particles, to reach either high strength in case of Araldite 2011 or the handling strength in case of Jowat 690.

Keywords

Accelerated curing, Ferromagnetic particles, Epoxy adhesives, Lap shear strength, Induction heating

1. Introduction

Adhesive bonding using epoxy is currently considered a key technology in assembling multi-material parts and structures. Compared to classical bolted and riveted joints, adhesively bonded joints using structural epoxy adhesives offer a higher mechanical performance and a better weight reduction. Additionally, adhesive bonding offer the possibility to assemble dissimilar materials which are typically difficult to assemble mechanically, such as glass and composite materials. Despite the many advantages of epoxy adhesives, their relatively long curing time increase the overall manufacturing time and

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introduces a bottleneck in assembly lines. Some epoxy adhesives cure either at elevated temperatures or at room temperature. Depending on the type of the epoxy adhesive used, the whole assembly has to be either cured inside an oven, or stored for long times in an open inventory. This means that, besides the time requirement, unwanted thermal stresses in the structures can be introduced. In addition, extra costs of inventory could be added to the overall cost of the part. Consequently, it is important to reduce the curing times of structural adhesives.

Curing by induction is one of the techniques which is recently used to reduce the curing time of structural adhesives. Induction heating itself is a well-known technique used to heat and melt metallic materials. In this technique, metallic materials (either electrically conductive or magnetic) are exposed to a high intensity alternating magnetic field. The heat is generated either by the joule effect (resulting from the flow of the alternating eddy currents in the material), or by hysteresis losses (resulting from the internal friction of the molecules due to the change of the magnetic polarity). The induction technique is used to cure adhesives in component assembly of electric motors [1], and in several automotive applications [2]. In such applications, the metallic adherent material (either ferroelectric or ferromagnetic) heats up by means of induction, and the heat is transferred to the adhesive via thermal conduction. However, if the adherent material is nonconductive or non-ferromagnetic, the adhesive has to be modified with some susceptor particles in order to generate heat within the adhesive.

The modification of polymers and adhesives using susceptor particles for induction heating was studied by several researchers. Bayerl et al. [3] studied the influence of induction frequencies, particle type, and particle content on the heating and the mechanical behavior of polyethylene and polyamide thermoplastics. Results showed that induction frequency of 2500 kHz was not sufficient to heat the thermoplastic/particle mix. Induction frequency of 450 kHz showed good heating effects, where the melting temperature of both thermoplastics could be reached in 3 minutes. In addition, the ferromagnetic particles (iron and magnetite) showed a better heating effect compared to carbon black particles which is only electrically conductive. Impact strength was also significantly reduced with the increase of particle content. Vallée et al. [4] investigated the effect of adding magnetite particles to Mn-Zn-ferrite particles in 1-component epoxy for induction heating at 373 kHz. Results revealed that the addition of 4% magnetite helped in increasing the adhesive temperature. However, subsequent increase in magnetite content up to 12% did not have additional influence on the adhesive temperature. Severijns et al. [5] studied the influence of iron particle content on the lap shear strength of bonded joints. Results showed that the lap shear strength was reduced by 15% with the addition of 0.5% volume content of particles compared to unfilled adhesive. In addition, the inductively cured joints showed 6% higher lap shear strength compared to oven cured joints.

This study aims to assess the potential of accelerating the curing of commercial epoxy adhesives using induction heating and curie particles, especially in electronics and automotive industries. Two industrial applications are considered in this study. The first application is potting of electronic sensors in a glass fiber reinforced polypropylene sulfide composite (PPS) housing. These sensors are typically used in monitoring the air quality in heat, ventilation, and air conditioning (HVAC) systems. Two-component epoxy adhesives are generally used for this process as they protect the sensors against humidity, high temperatures, and corrosion [6]. The potted assembly is typically cured in the oven at relatively low temperatures for longer hours (up to 80 ºC for 12 hours) so as not to damage the electronics and the housing materials. The second application is the bonding of a multifunctional bracket onto a glass windshield panel of passenger vehicles. The multifunctional bracket acts as an anchor point for the rearview mirror and additional driver assistance modules such as cameras, light sensors, rain sensors, etc. The bracket is typically made of glass fiber reinforced PBT/PET composite, while the surface of the laminated glass is treated with thin layer of enamel. Flexible epoxy grade adhesives and polyurethanes are generally used for this application to reduce stress concentrations and to damp vibrations [7]. The bonded assembly is generally cured at room temperature for 48 hours. In case of curing at elevated temperatures, the maximum allowable curing temperature is also 80 ºC in order not to damage the polyvinyl butyral (PVB) foil in the laminated windshield glass.

In this paper, the effect of the induction frequency and the weight percentage of the curie particles on the temperature in the adhesive is first investigated. Secondly, the effect of the weight percentage of curie particles on the mechanical performance of the adhesively bonded joints is studied using standard single lap shear samples. Finally, the potential of accelerating curing using induction is assessed and verified on single lap shear samples with regard to the abovementioned applications. The mechanical performance of the inductively cured joints is benchmarked against reference oven cured joints.

2. Materials and methods

2.1. Selection of adhesives and curie particles

Two epoxy based adhesives were considered in this study. For the sensor potting application, the Araldite 2011 two component epoxy (manufactured by Huntsman) was selected. This adhesive provides full electric insulation of the electronic sensors, high temperature resistance and good bonding with the adherent material. Additionally, no volatile contents are produced during curing. For the multifunctional bracket/windshield assembly, the Jowat 690 two component SE polymer was selected. This adhesive is a hybrid of epoxy resin and silane terminated polymer, which offers good flexibility, good bonding with glass, and solvent free curing. According to the Araldite 2011 datasheet, the recommended minimum curing time at 70 ºC is 50 min. For the Jowat 690, the minimum curing time recommended by the datasheet is 24 h to reach 50% of the final strength. Increasing the adhesive temperature could reduce the required curing time, however, no high temperature curing cycles were specified in the data sheet. Full curing is achieved after 7 days at room temperature. Given the selected curing temperature for both applications as mentioned in the introduction section, Dynamic Scanning Calorimetry (DSC) measurements were performed to determine the minimum curing time of both adhesives at 80 ºC. Figure 1 shows the evolution of the conversion fraction (α) at isothermal curing conditions at 80 ºC for 150 min for both adhesives, considering a heating rate of 50 ºC/min. As seen in Figure 1, the Araldite 2011 achieved a conversion of approx. 99% after 60 min, while the Jowat 690 achieved only 70% conversion after 150 min. By extrapolating the conversion curve of the Jowat 690, a conversion of 90% can be achieved within 5 to 6 hours at 80 ºC.

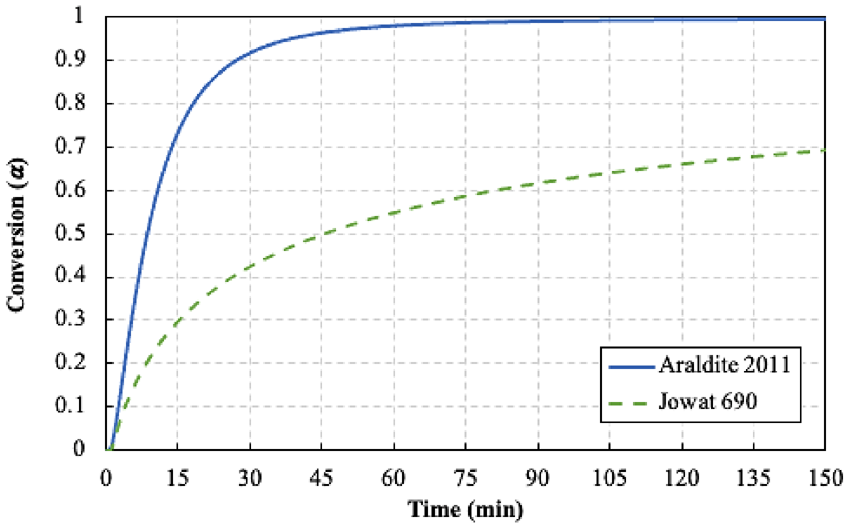

Figure 1. Evolution of the conversion fraction (α) with time for Araldite 2011 and Jowat 690 epoxy based adhesives at 80 ºC isothermal curing conditions

The selected curie particles were Mn-Zn-ferrite R12K particles (supplied by Hengdean group DMEGC Magnetics, China). The particles were supplied in one batch in a plate like morphology, with a mix of sub-micrometer and micrometer sizes having several sharp edges. An SEM analysis was performed to determine the average size and distribution of the R12K curie particles, as shown in Figure 2a. Approximately 50% of the particles had an average size of 0.5 µm, while the other 50% of the particles had an average size of 4.5 µm. According to the manufacturer's datasheet, the density and resistivity of the particles are 4.9 g/cm3 and 0.15 Ω m respectively. The curie temperature of the particles is 110 °C, which is below the softening temperature of the adherent materials of both applications. This curie temperature corresponds to the temperature at which the initial magnetic permeability of the particles drops to zero. Figure 2b shows the evolution of the initial permeability with temperature.

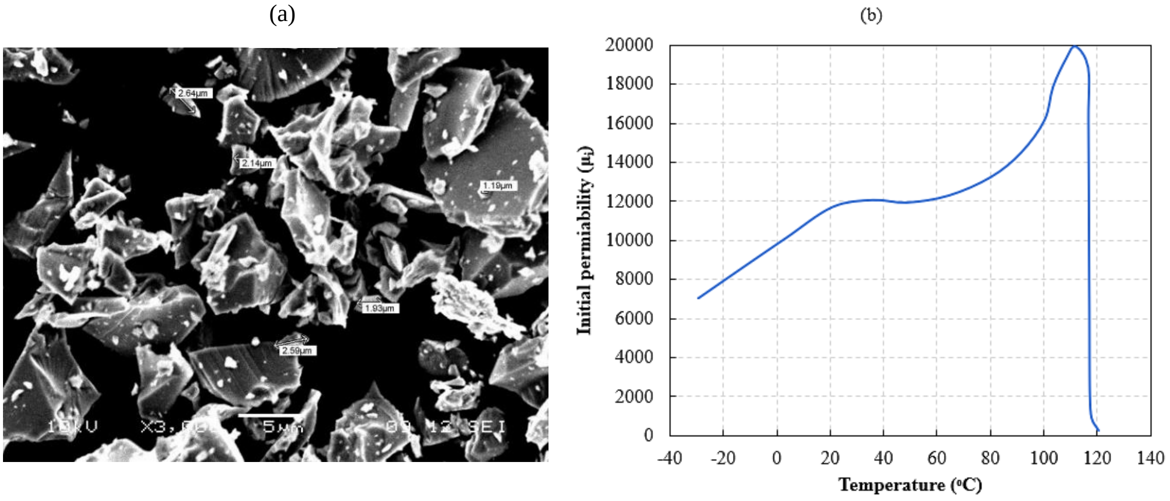
(a)
(b)

Figure 2. (a) SEM image of the R12K particles (magnification 3000x), (b) evolution of the initial permeability or the R12K particles with temperature [8]

2.2. Induction setup and temperature measurement

Induction experiments were performed on the TruHeat HF 1005-5010 induction machine (manufactured by TRUMPF Hüttinger GmbH) available at the Joining and Materials Lab at Flanders Make. It consists of 3 main units: a water cooling unit, a generator unit, and an inductance-capacitance circuit unit with the induction coil. The power generator is capable of supplying a maximum induction power of 11.2 kW and a maximum current of 35 A. Figure 3 shows the induction setup used. The main specifications of the induction machine are summarized in Table 1.

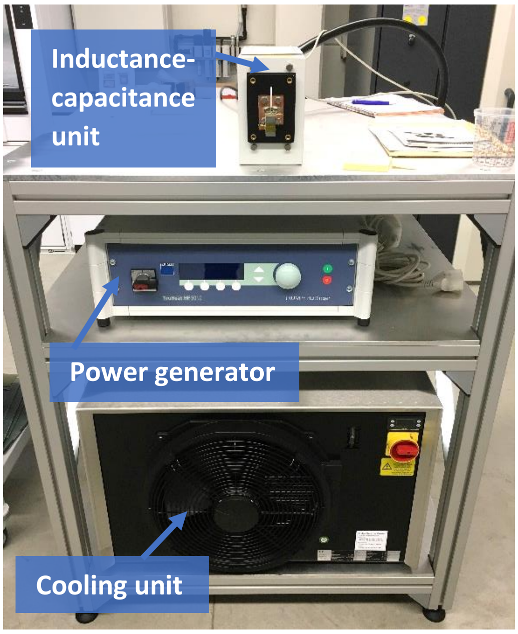

Figure 3. Induction setup used

Table 1. Specifications of the induction machine

Parameter	Value
Transformer ratio	16:1
Capacitors (nF)	660
Frequency range (kHz)	50 – 1000
Current range (A)	2.8 – 35
Maximum voltage (v)	1200
Maximum power (kW)	11.2

Three flat spiral coils (pancake coils) made up of 4 turns were used as shown in Figure 4. The coils were manufactured from hollow copper tubes (with outer diameters 8, 6, and 4 mm respectively), which allowed the flow of the cooling water inside the inductors. The frequency ranges achieved by the coils were 180 – 228 kHz for the large coil, 196 – 252 kHz for the medium coil, and 243 – 325 kHz for the small coil.

Figure 4. Flat spiral (pancake) coils used

Temperature inside the adhesive and in the bond line was measured using a partial immersion liquid-in-glass thermometer, having a measurement range of -10 to 250 ºC. The use of liquid-in-glass thermometer ensured the accurate measurement of the temperature in the bond line without any interferences due to the magnetic field.

2.3. Single lap shear test setup

Single lap shear tests were performed using a Shimadzu AGS-50NX universal testing machine shown in Figure 5. The load was measured using a 20 kN load cell. Special attention was paid to ensure the alignment of the lap shear samples with the grips in order to eliminate any bending moments. Samples were tested at cross head speeds of 2 mm/min for the joints with Araldite 2011 adhesives, and 5 mm/min for the joints with Jowat 690 adhesive, according to the recommendations of the ASTM D3163 standard [9].

Figure 5. Single lap shear testing setup

2.4. Specimen materials and geometries

Adhesive/particle mixtures were prepared by manually mixing the R12K particles with each adhesive separately with particle weight percentages of 16.7% and 30% respectively. Several single lap shear samples were prepared and cured using the adhesive/particle mixtures and neat adhesives for the different investigations, as summarized in Table 2. The adherent materials were Ryton® polypropylene sulfide (PPS) reinforced with 40% glass fibers, Ultradur® polybutylene terephthalate/polyethylene terephthalate (PBT/PET) reinforced with 30% glass fibers, and glass coated with thin enamel layer on the adhesion side. The length and the width of the adherent materials were 100 mm and 25 mm respectively. The thickness of the adherent materials were 2 mm for the PPS and PBT/PET, and 5 mm for the glass. The surfaces of the thermoplastic composite samples to be bonded were treated atmospheric plasma prior to the application of the adhesive, in order to improve the adhesive properties on the surface. All samples were prepared in special molds to maintain a bond line thickness of 0.1 mm, and an overlap distance of 12.5 mm, according to the ASTM D3163 standard [9]. The oven cured samples were left to cool in the oven for 16 hours after curing. Next to the lap shear samples, small cylindrical samples of diameter 18 mm and height of 10 mm were prepared to study the temperature in the adhesive at different induction frequencies and different particle weight content. The samples were prepared using the Araldite 2011/R12K mixtures and cured in oven for 3 hours at 60 ºC. A blind hole of diameter 5.5 mm and depth 8 mm was drilled in each sample to accommodate the liquid-in-glass thermometer. Additionally, thick adherent single lap shear specimens (PPS/PPS and PBT/PBT) of adherent thickness 4 mm were also prepared using both adhesive mixtures in order to monitor the temperature in the bond line during induction, as indicated in Table 2. Similar to the disk samples, a blind hole of depth 8 mm was also drilled in the center of the overlap distance to accommodate the thermometer. Figure 6 shows the cylindrical and the thick adherent samples used.

Table 2. Summary of the prepared single lap shear samples

Set number	Adhesive mixture	Adherent materials	Curing conditions	Required study
1	Neat Araldite 2011	PPS/PPS	Oven cured for 3 hours at 80 ºC	Effect of particle content on the lap shear strength
2	Araldite 2011+16.7% R12K	PPS/PPS	Oven cured for 3 hours at 80 ºC	
3	Araldite 2011+30% R12K	PPS/PPS	Oven cured for 3 hours at 80 ºC	
4	Neat Jowat 690	PBT/Glass	RT cured for 48 hours	
5	Jowat 690 + 16.7% R12K	PBT/Glass	RT cured for 48 hours	
6	Jowat 690 + 16.7% R12K	PBT/Glass	RT cured for 48 hours	
7	Araldite 2011 + 30% R12K	PPS/PPS	Oven cured for 3 hours at 80 ºC	Thick adherent samples for temperature monitoring in bond line during induction
8	Jowat 690 + 30% R12K	PBT/PBT	RT cured for 48 hours	
9	Araldite 2011+ 30% R12K	PPS/PPS	Induction curing	Application assessment
10	Jowat 690 + 30%R12K	PBT/Glass	Induction curing	
11	Jowat 690 + 30% R12K	PBT/Glass	Oven cured for 3 hours at 80 ºC	

(a) (b)

Figure 6. Samples for temperature measurement: (a) thick adherent lap shear samples with PBT/PBT sample at the top and PPS/PPS sample at the bottom), (b) cylindrical samples

2.5. Experimental conditions

In order to study the effect of the frequency and the particle weight content on the adhesive temperature, the small cylindrical samples were inductively heated for 15 minutes with the 3 pancake coils. For all the coils, the induction power was kept constant at 0.5 kW. The induction frequencies used were 188 kHz for the large coil, 207 kHz for the medium coil, and 262 kHz for the small coil. The coupling distance between the coils and the samples were 5 mm. The temperature was manually recorded every 0.5 min using the liquid-in-glass thermometer.

The effect of particle content on the lap shear strength was studied using oven cured samples of the Araldite 2011 adhesives, and room temperature cured samples for the Jowat 690, as detailed in Table 2. Three particle weight percentages of 0%, 16.7%, and 30% were tested. At least 3 samples were tested for each testing condition. The tests were conducted at ambient temperature of approx. 22 ºC and relative humidity of approx. 34%

For the induction curing tests, the lap shear samples where inductively cured using the small pancake coil for 15 minutes, at 5 kW induction power and 245 kHz induction frequency. All samples were fixed in a special fixture during induction, where the distance between the coil and the centerline of the bond line was 7 mm. Figure 7 shows the fixture used in the induction curing tests. The temperature in the bond line during induction was monitored using the thick adherent samples. The fixture of these sample were adjusted such that the distance between the coil and the bond line was also 7 mm. The selected induction parameters achieved a curing temperatures of approx. 80 ºC after 15 minutes. Figure 8 shows the induction power cycle and the temperature developed in the bond line during the induction curing tests. The lap shear strength of the inductively cured samples for both adhesive mixtures were compared against the oven cured samples of the same adhesive mixture. The oven curing conditions were 3 hours at 80 ºC, which is the same curing temperature achieved by the induction curing. All induction samples were left to cool for 1 hour before lap shear testing. The tests were also conducted at ambient temperature of approx. 22 ºC and relative humidity of approx. 34%.

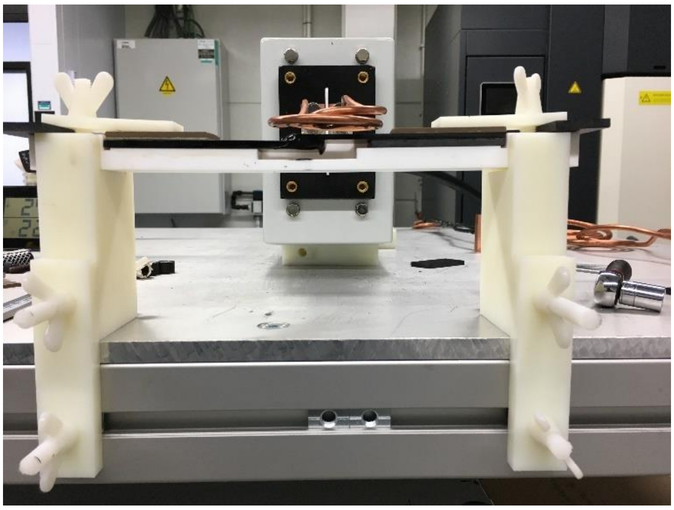

Figure 7. Sample fixture used during induction

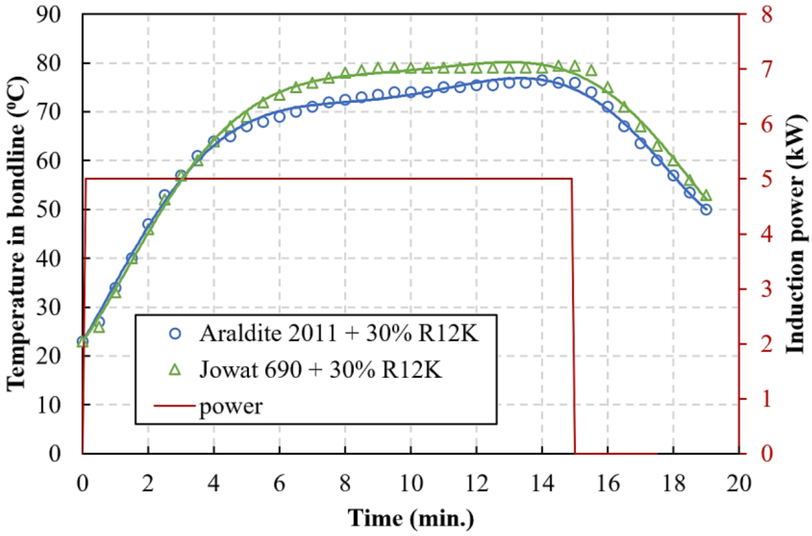

Figure 8. Induction power cycle and temperature in the bond line during induction

3. Results and discussion

3.1. Effect of frequency and particle content on the adhesive temperature during induction

Figure 9 shows the evolution of the temperature in the araldite 2011 adhesive at different induction frequencies and particle contents for 15 minutes. It can be seen that the temperature increased gradually for all induction frequencies and particle contents, and reached a stable level after approx. 13 minutes. The temperature generally increased with the increase of both the induction frequency and the particle content in the adhesive.

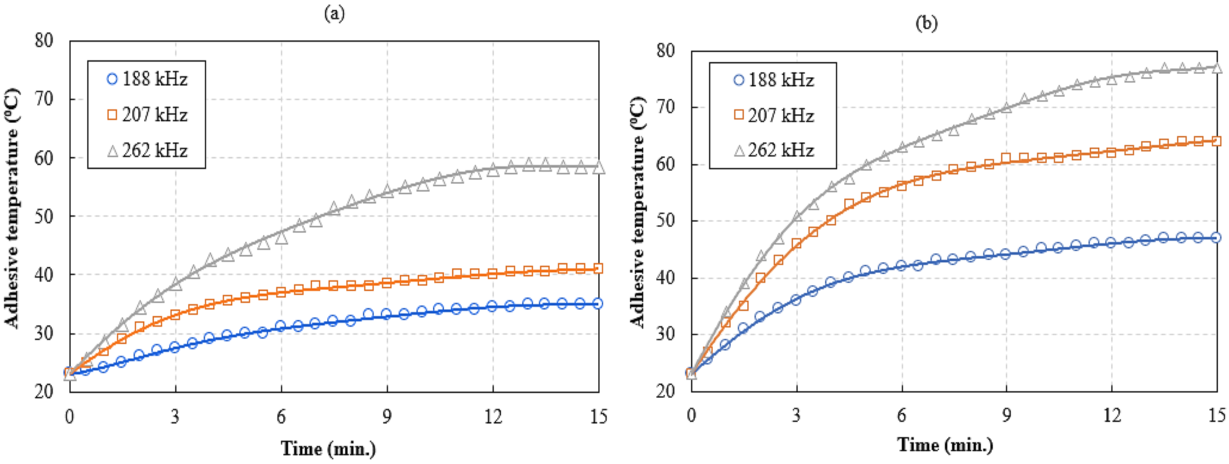

Figure 9. Evolution of the temperature of the Araldite 2011 adhesive during induction at different frequencies: (a) 16.7% R12K particles, (b) 30% R12K particles

Figure 10 shows the temperature of the adhesive mixtures as a function of the induction frequency after 15 minute induction. For the adhesive with 16.7% particle content, the temperature increased from 34 ºC at 188 kHz to 56 ºC at 262 kHz, which corresponds to an increase of approx. 65%. For the adhesive with 30% particle content, the temperature increased from 45 ºC at 188 kHz to 79 ºC at 262 kHz, which corresponds to an increase of approx. 75%. On the one hand, increasing the particle content by approx. 80% at the same induction frequency only increased the temperature in the adhesive by percentages of 34% to 52%. On the other hand, increasing the induction frequency by just 39% at the same particle content can increase the temperature by percentages of 65% to 75%. This suggests that increasing the induction frequency is more efficient in raising the adhesive temperature compared to increasing the particle content. This is attributed to the pronounced skin effect during induction heating. By using a least square power fit, the increase in adhesive temperature with the increase in induction frequency can be represented by the following relation:

$$T_A = Kf^m \tag{1}$$

Where T_A is the adhesive temperature, K is the temperature coefficient in ºC, and m is the frequency exponent. For the adhesive with 16.7% particle content, the temperature coefficient and frequency exponent were 0.0147 ºC and 1.4808 respectively. For the adhesive with 30% particle content, the temperature coefficient and the frequency exponent were 0.0118 ºC and 1.5886 respectively.

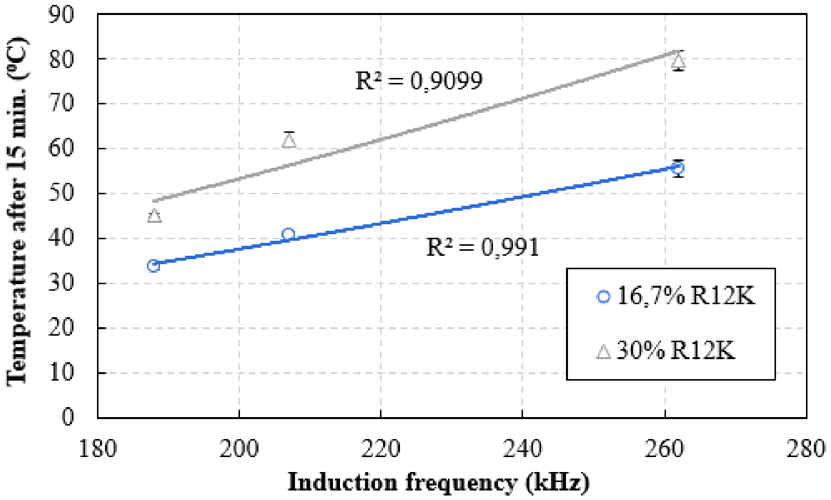

Figure 10. Temperature of the Araldite adhesive with different R12K particle content at different induction frequencies after 15 minutes induction

3.2. Effect of particle content on the lap shear strength of both adhesives

Figure 11 shows the effect of particle content on the lap shear strength using both Araldite 2011 and Jowat 690 adhesives. It can be seen that for the Araldite 2011, the increase in the particle content decreased the lap shear strength. At 30% particle content, the lap shear strength was reduced to approx. 8.3 MPa compared to 10 MPa for the neat adhesive. This corresponds to a percentage reduction of approx. 17%. For the Jowat 690, the lap shear strength was almost constant at all particle contents. The reduction in the lap shear strength of the Araldite 2011 is attributed to the effect of stress concentrations caused by the particles in the brittle adhesive. Whereas in the case of the flexible Jowat 690, the stress concentration effects caused by the particles are much less pronounced.

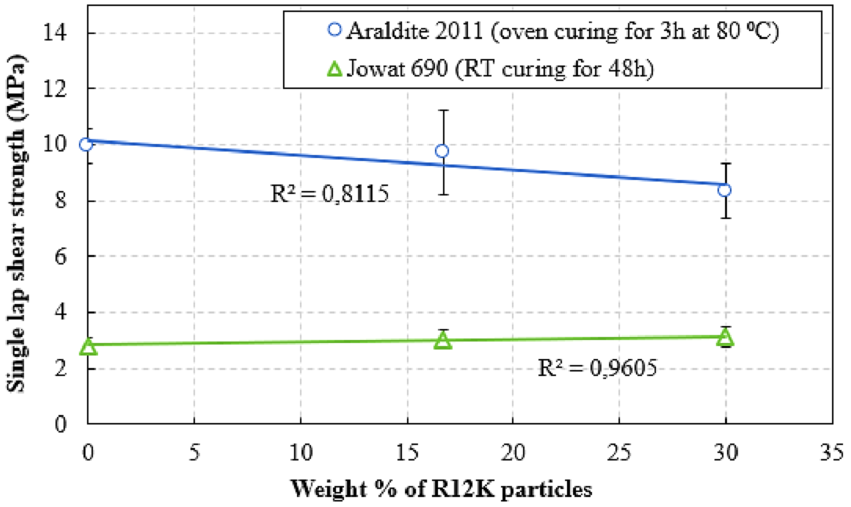

Figure 11. Effect of particle content on the lap shear strength using Araldite 2011 and Jowat 690 adhesives

3.3. Assessment of the induction curing technique for the selected applications

Figure 12 (a) shows a comparison between the lap shear strength of the inductively cured PPS/PPS lap shear sample for 15 minutes and a reference oven cured sample (3h at 80 ºC) using the araldite 2011/30% R12k adhesive mixture. It can be seen that the inductively cured samples achieved a lap shear strength of approx. 12 MPa compared to approx. 8 MPa for the oven cured sample in just 15 minutes. This corresponds to a percentage of 150% of the reference lap shear strength in only 8% of the curing time. Similar results were reported by Severijns et al. [5], however, further research is still required to understand why inductively cured lap shear samples with the araldite 2011 adhesive increased their lap shear strength compared to oven curing at the same curing temperature.

Figure 12(b) shows a comparison between the lap shear strength of the inductively cured PBT/Glass lap shear sample for 15 minutes and a reference oven cured sample (3h at 80 ºC) using the Jowat 690/30% R12k adhesive mixture. Contrary to the Araldite 2011 case, the inductively cured samples achieved a lap shear strength of 1.34 MPa compared to 3.1 MPa for the oven cured samples. This corresponds to a percentage of only 43.2% of the reference lap shear strength in 8% of the time.

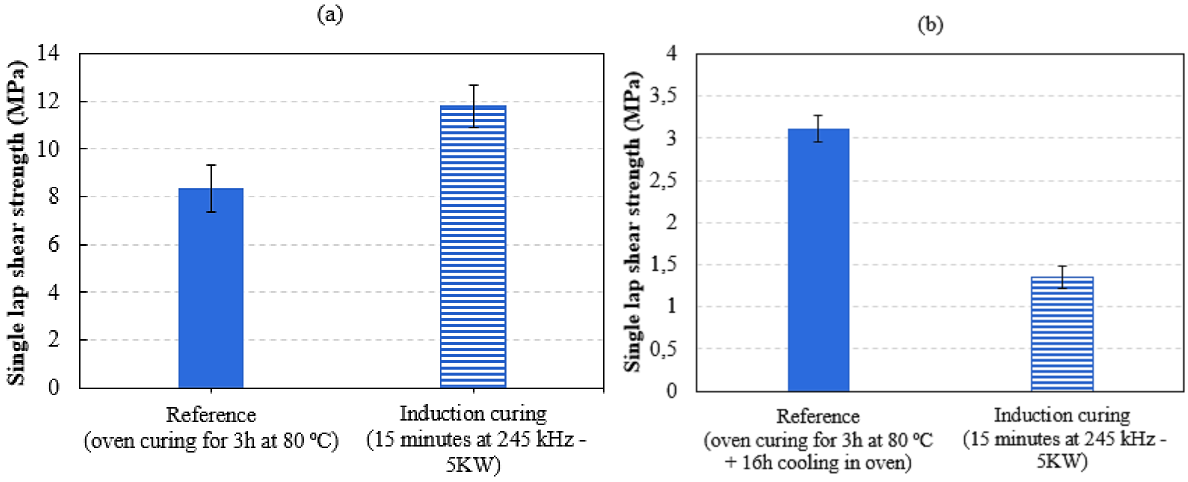

Figure 12. Comparison between the inductively cured lap shear samples and reference oven cured samples: (a) potting application with Araldite 2011, (b) automotive windshield application with Jowat 690

4. Conclusion

This study was performed in order to assess the potential of accelerating the curing of epoxy adhesives by induction and curie particles in two industrial applications. The first application was potting of electronic HVAC sensors in a thermoplastic composite housing. The second application was the assembly of a multifunctional thermoplastic bracket in glass windshields of passenger vehicles. Two adhesives were considered in this study: Araldite 2011 2-component epoxy for the electronic potting, and Jowat 690 flexible epoxy/SE polymer adhesive for the multifunctional bracket. The effect of the induction frequency and the particle content on the temperature in the Araldite 2011 adhesive was investigated using a liquid-in-glass thermometer and an industrial grade induction machine. The range of the induction frequencies studied was 188 kHz up to 262 kHz, and the weight percentages of the particles were 16.7% and 30%. In addition, the effect of the particle content on the lap shear strength of both adhesives was studied using a universal tensile testing machine. Moreover, and with regard to the selected industrial applications, the lap shear strength of inductively cured samples of both adhesives was tested and compared against oven cured samples. Considering the tested adhesives and samples, the induction conditions and the curie particles selected, the following can be concluded.

1. Increasing both the particle content and the induction frequency increases the temperature in the araldite 2011 adhesive.
2. Increasing the induction frequency by 39% at the same particle content can increase the temperature in the araldite 2011 adhesive by percentages of 65% to 75%, as compared to percentages of 34% to 52% if the particle content increased by 80%. This indicates that increasing the induction frequency is more efficient in raising the adhesive temperature compared to increasing the particle content.
3. The addition of 30% the particle content in the brittle Araldite 2011 adhesive reduces the joint strength by 17%.
4. The increase in the particle content does not have a significant effect on the joint strength of Jowat 2011 adhesive.
5. The inductively cured Araldite 2011/30% R12K joints achieved 150% of the reference oven cured strength in 8% of the oven curing time.
6. The inductively cured Jowat 690/30% R12K joints achieved 43.2% of the reference oven cured strength in 8% of the oven curing time.

Despite the lower strength achieved by induction for the Jowat 690/30% R12K, and given the fact that the Jowat 690 is a slow curing adhesive, it can still be considered that the handling strength was reached in 15 minutes. This handling strength is typically reached after 24 hours according to the data sheet. The induction technology, therefore, shows a high potential of accelerating the curing time for industrial applications by modifying the adhesive with ferromagnetic curie particles.

Acknowledgments

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