# ADHESIVE JOINING TECHNOLOGIES ACTIVATED BY EXTERNAL TRIMS FOR AUTOMOTIVE APPLICATIONS

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## Abstract
Joining is a key and fundamental aspect of vehicle design and manufacturing process. The development of efficient, simple, inexpensive and reversible adhesive bonding technologies offer low cost and improved life cycle and recycling of a vehicle. Different typologies of susceptors, embedded in thermoplastic hot-melt adhesives, have been tested to investigate the thermal behavior of the innovative materials exposed to electromagnetic fields. These innovative technologies will optimize the bonding process offering new opportunities connected with cost reduction, resistance to applied load, easy and rapid dismantling and smart recycling. Experimental procedures to optimize the electromagnetic process have been performed. For more conclusive evaluation, both shear strength and sliding temperature of the modified adhesives have been investigated.

## 1 Introduction - Electromagnetic welding process
The process of electromagnetic bonding is based on the principle of electromagnetic (or induction) heating: magnetic field sensitive materials increase their temperature when subjected to a high frequency, alternating current field [1].
Electromagnetic radiation absorbed by ferromagnetic particles embedded in the adhesive matrix causes the susceptor particles to rapidly heat. Conductive heat transfer from the ferromagnetic particles into the polymer matrix material causes creeping flow of the implanted material [2].
The physics of electromagnetic induction applies to both Joule heating (eddy current losses) and magnetic heating (hysteresis losses). Benatar [3] has described the heating of ferromagnetic susceptor materials as the combination of both of these effect. He also described that magnetic and eddy current intensity decreases rapidly with increasing distance of the penetration depth. At each cycle of the applied radio frequency (RF) field, the susceptor responds by completing the full cycle. The area bounded by the hysteresis curve is proportional to the energy converted into heat. High frequencies are required because the incremental temperature risen for each hysteresis cycle is very small [4].

It is not within this present technical capability to directly measure the proportion to which these heating effects contribute to the total heating of the susceptor material, nor were we able to measure effective penetration depth. In this work, we explore certain fundamental relationships that regulate the electromagnetic welding process. The considered parameters associated with the modified adhesive and with the process are: typology and amount of nanoparticles in the adhesive, intensity and frequency of the electromagnetic field. There are many other parameters worthy of investigation, such as the shape of the coil, the coupling distance, but they are not evaluated within this present investigation.

## 2 Materials and testing methods

### *2.1 Materials*

This work employed five types of iron based nanoparticles: magnetite $Fe_3O_4$ (Sigma-Aldrich), hematite $\alpha$-$Fe_2O_3$ (Nanophase), cobalt-ferrite $CoFe_2O_4$ (Nanotesla), manganese-zinc ferrite $(Mn,Zn)Fe_2O_4$ (Nanotesla), and maghemite $\gamma$-$Fe_2O_3$ (Nanotesla).
These ferrites were selected to evaluate their effects when they are embedded into a hot-melt adhesive, characterized by the formulation listed in Table 1.

| **Components** | **% (w/w)** |
|---|---|
| Amorphous –poly-alpha-olefin (*APOA*) | 53.20 |
| Styrene-isoprene-styrene block polymer (SIS) | 6.60 |
| Ester of Hydrogenated Rosin | 38.13 |
| Polybutene tackifier | 1.73 |
| Antioxidants | 0.34 |

**Table 1.** Qualitative and quantitative composition of the hot-melt adhesive.

In order to investigate mechanical properties of modified and unmodified adhesives, joints in homopolymer polypropylene (PP) 10% talc filled were realized. This compound is largely used in automotive internal and external applications.

### *2.2 Testing methods*

Tapping mode-atomic force microscopy (AFM) was used to study the size and shape of the particles. Each typology of iron powder was ultrasonically dispersed in isopropyl alcohol prior to depositing it onto a plate. A Veeco Digital Instrument atomic force microscope was used for these studies (scan area 1 µm x 1 µm).
The geometry and dimensions of the Single Lap Joints (SLJ) specimens used to test the mechanical properties of modified and unmodified adhesives are presented in Figure 1.

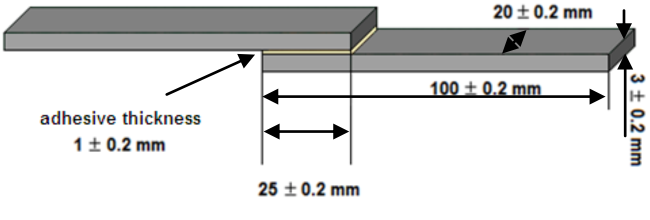


**Figure 1.** SLJs (geometry, boundary conditions, and dimensions in mm).

For surface preparation, the PP 10% talcum filled substrates were cleaned with isopropyl alcohol. The adhesive was dispensed by a Nordson Durablue adhesive melter and the excessive adhesive at the overlap edges was removed in all joints. A compressive force of 20 N was applied to the lap joints during the cooling period (two minutes). The single lap joints were assembled on specially manufactured apparatus , allowing the standardized joint preparation technique to be used repeatedly with constant adhesive film thickness. Then, the specimens were exposed at room temperature for 24 hours prior to testing.

Five tests were carried out for each adhesive Single-lap joint, SLJ. Specimens were subjected to tensile loading using a Instron 5544 Series dynamometer at room temperature under displacement control (100 mm/min); tabs at the ends of the SLJs were bonded to assure a correct alignment into the dynamometer cell. Throughout this work, the average shear strength was used to measure the joint strength, and was calculated as the maximum applied load of each test divided by the measured bonded area. The failure mode was determined by visual inspection.

To identify the slipping temperature, SLJs were loaded with the weight force given by a mass of 500 g and subjected to gradually increasing temperature (50°C/h) inside a temperature controlled chamber.

Modified adhesives were prepared by mixing the nanoparticles with hot melt adhesive on a PTFE sheet, placed on a hot-plate at a temperature of 180°C. Mechanical tests have been used to investigated samples.
SEM analysis was carried out on the surface and cross-section of modified adhesive film to verify the distribution of nanoparticles and obtain clear images of their morphology, respectively. The samples were coated with a thin film of Au in order to increase their conductivity.

For the electromagnetic test, a tape of modified adhesive (1,7 x 11 cm) was placed inside the inductive coil and AC current was supplied through the coil. Heating profile was measured using a opSens fiber optical sensor model OTG-M170, placed in the middle of the adhesive sample. Different work coils were used on the generator throughout the experimental study.

In order to realize joints by new assisted assembling technology, a new manufacturing methodology was set: modified adhesive was cut to obtain a tape with the proper size of the joint overlapping (20 x 25 cm). This sample was placed on a polypropylene substrate and the second substrate was laid upon that. Finally the joints were tacked on by a PTFE wire. For assembling tests by use of electromagnetic field, SLJs were hanged inside the coil, placing the overlapping area in the middle of the coil. In order to reveal the heating temperature, the fiber optical sensor was put inside the adhesive. For disassembling tests a similar setup was used: SLJs were loaded with the weight force given by a mass of 500 g, and inserted inside the coil. Placing the joint inside the coil, the RF field activates the modified adhesive allowing the joint dismantling.

## 3 Experimental results and discussion

### *3.1 Nanoparticles characterization*

Atomic Force Microscopy (AFM)

for each typology of iron particles, agglomerates of particles in the range from 10 to 60 nm, with an average radius smaller than the superparamagnetic limit. Figure 2 shows magnetite nanoparticles morphology. All the ferrite nanoparticles resulted typically spherical in shape.

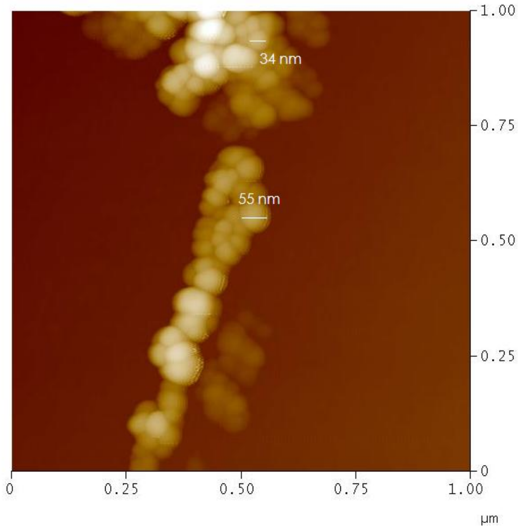


**Figure 2.** Atomic force image of magnetite $Fe_3O_4$ particles.

*3.2 Modified and unmodified adhesive characterization.*

Figure 3a shows SEM image, taken on the surface of adhesive modified adding 5 weight percent of magnetite nanoparticles. All the filled adhesives show that nanoparticles were aggregated into the adhesive matrix in a well dispersed way. Figure 3b was taken on the modified adhesive cross section and shows the morphology of nanoparticle clusters.

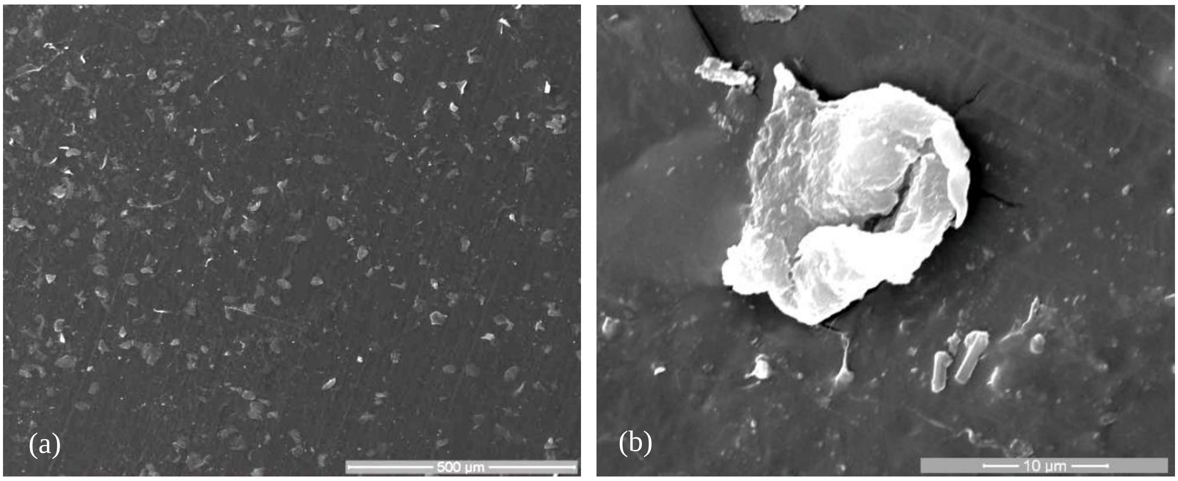


**Figure 3.** Scanning electron micrograph of the adhesive filled with 5% by weight of magnetite nanoparticles. (a) surface analysis 200x, (b) cross-section analysis 5000x.

As shown in Figure 4, modified adhesive SLJs (5-10% of magnetite) exhibited an increase in maximum shear strength against unmodified SLJs. The average shear strain at failure load increased by more than 10% for sample with 5 weight percent of magnetite, and more than 20% for sample with 10 weight percent of magnetite (Table 2). This implies that because of nanoparticles inclusions inside the adhesive matrix, the damage initiation is delayed and gives higher load carrying capacity of SLJ specimens. This phenomenon can be explained in other way: connecting to the presence of nanoparticles, the interfacial bond strength between adhesive and polypropylene substrate is so increased that leads to consistent cohesively dominated failures.

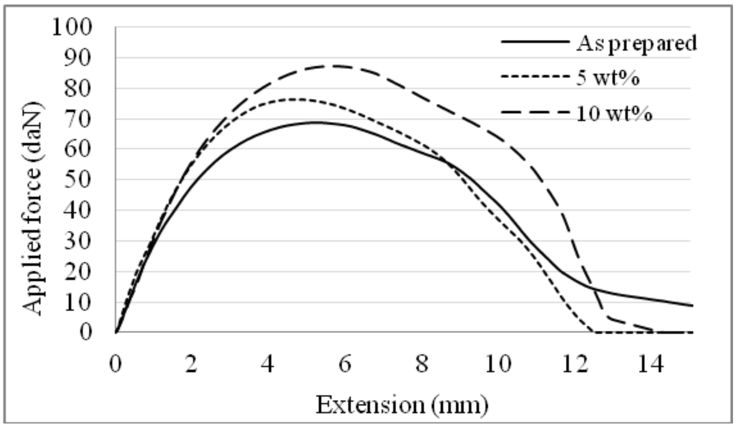


**Figure 4.** Applied force/extension curve for SLJ prepared with unmodified and modified adhesive (5 and 10% weight percent of magnetite).

| | Shear stenght ($daN/cm^2$) | Standard deviation ($daN/cm^2$) | Shear modulus 0.5-1% (MPa) | Standard deviation (MPa) |
|---|---|---|---|---|
| **As prepared** | 13.4 | 0.6 | 17.4 | 1.2 |
| **5 wt % magnetite** | 15.4 | 0.4 | 18.4 | 1.7 |
| **10 wt % magnetite** | 16.3 | 1.4 | 17.2 | 2.2 |

**Table 3.** SLJs results of unmodified and modified adhesive (5 and 10 weight percent of magnetite).

The introduction of ferrite nanoparticles into the adhesive matrix shows insignificant effect on SLJs slipping temperature, which remained almost the same of the unmodified adhesive SLJs (105°C).

*3.3 Bonding process optimization.*

The temperature response as a function of time for each modified adhesive, 5 weight percent of different nanoparticles, is shown in Figure 6. Table 3 presents temperature response at a representative value of 40 seconds for the different modified adhesives.

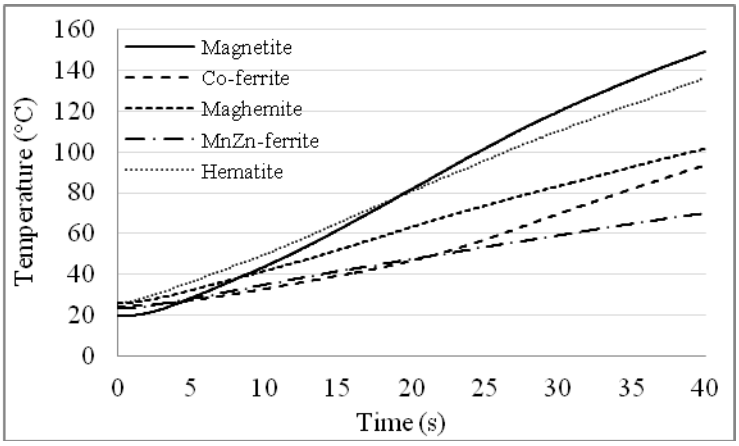


**Figure 5.** Thermal profile of modified adhesives exposed to electromagnetic fields (172 kHz, 3.3 kW).

| | magnetite | maghemite | Co-ferrite | ematite | MnZn-ferrite |
|---|---|---|---|---|---|
| **Temperature (°C)** | 151 | 138 | 98 | 86 | 67 |

**Table 3.** Adhesive heating results at 40 s – 172 kHz, 3.3 kW.

The adhesive which was modified by magnetite shows the best heating capability in electromagnetic field. This adhesive was able to reach in 40 seconds a temperature of 151°C (adhesive melting point).

Modified adhesive (5 weight percent of magnetite) was tested at different intensity of the electromagnetic field, in order to evaluate the temperature response as a function of time. Results are shown in Figure 6: the x-axis shows the power supplied by the voltage source, relatable to the intensity of the electromagnetic field, while y-axis is referred to the temperature reached within 40 s.

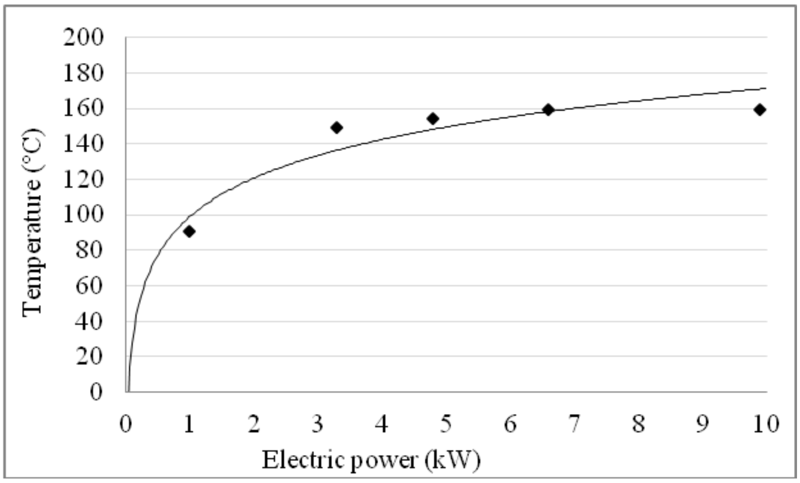


**Figure 6.** Temperature reached in 40 s vs electric power for magnetite modified adhesive (5 wt%, 172 kHz).

For tests realized at 1 kW the heating rate was quite lower than the other tested powers. After 40 seconds, the temperature was still below 100°C (below the softening temperature of the adhesive).
For remaining samples tested at elevated powers, the temperature reached after 40 seconds was much greater. The adhesive samples tested at 3.3, 4.8, 6.6 and 9.9 kW show a similar behavior and the reached temperature after 40 seconds didn't change a lot.
By analyzing these test results it could be reasonable the choice of 3.3 kW as optimized power to be used for future characterization. The temperature reached by this power was slightly lower than the temperature reached for higher power, but in any case satisfying for the innovative process. Therefore, the use of 3.3 kW could be advantageous in terms of spent energy and consequently cost.
Figure 6 shows that the modified adhesive increased its temperature logarithmically with the power supplied by the alternating voltage source.

From the electromagnetic equations, one can expect that the amount of heat generated by induction heating is directly proportional to the frequency of the electromagnetic field. Modified adhesive (5 weight percent of magnetite) were submitted to a 3 kW electromagnetic field, for different frequencies. Temperature increased with increasing frequency of the electromagnetic field in a linearly dependence manner.

The last test was carried out in order to understand the influence of the amount of magnetite nanoparticles, on adhesive matrix heating.

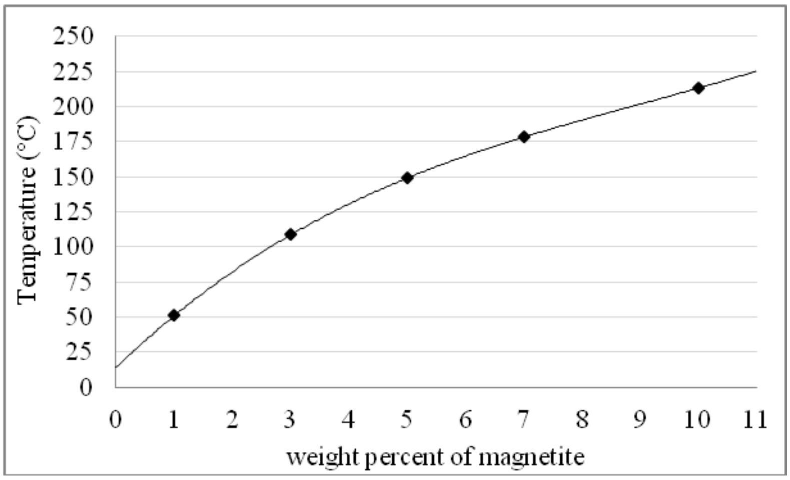


**Figure 7.** Temperature reached in 40 s vs weight percent of magnetite for modified adhesive (172 kHz, 3.3 kW).

Figure 7 demonstrates that heating rate varied with the increasing amount of magnetite nanoparticles into the adhesive as a 3rd order polynomial function.
Small amounts of nanoparticles (1, 3 weight percent) embedded into the adhesive matrix weren't enough to cause adhesive melting, while bigger amounts, from 5 to 10 % in weight, brought the modified adhesive to higher temperature and rapid heating.

*3.4 Disassembling of joints by innovative RF technology*
SLJs were tested by exposing them to electromagnetic field. In table 4 are summarized test results of modified adhesive based SLJ, with different amount of magnetite.

| | **Disassembling time (s)** |
|---|---|
| 5 wt% magnetite | 63 |
| 10 wt% magnetite | 20 |
| 30 wt% magnetite | 5 |

**Table 4.** Disassembling time for modified adhesive (5-10-30 weight percent of magnetite), exposed to electromagnetic field: 172 kHz, 15.8 kW

Increasing amount of embedded magnetite in the adhesive matrix, decreased time needed to reach the complete disassembling of the joints in electromagnetic field.

## Conclusions

Adhesive joint made with adhesives nanomodified by adding magnetic field sensitive particles have been studied. Different typologies of susceptors, embedded in thermoplastic hot-melt adhesives, have been tested in order to investigate the thermal behavior of the innovative materials exposed to electromagnetic fields.
An increase of magnetite content into the adhesive matrix affects mechanical properties: in particular the shear strength increases significantly.
This work is the first step in the study of bonding behavior of ferrite modified adhesives. The procedure followed to optimize a high frequency bonding process has been based on direct experimental tests: magnetite powder showed the best performance for induction bonding; heating rate varied with the increasing amount of nanoparticles dispersed into the adhesive as a 3rd order polynomial function.

Other variables influenced the bonding process: heating rate increased with increasing frequency of the electromagnetic field in a linearly dependence manner, while it increased logarithmically with the electromagnetic field power.
Increasing the amount of magnetite inserted into the adhesive matrix, also decreased the time it takes to reach the disassembling of the joints, when submitted to electromagnetic field.

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## References

[1] J. Rotheiser. *Induction/Electromagnetic Welding* in "Joining of plastics Handbook for Designers and Engineers", edited by Hanser Publishers, Munich, pp.331-332 (2004).
[2] R. Nichols, D. LaMarca, B. Agosto. *Performance of Susceptor Materials in High Frequency Magnetic Fields* in Proc. Of ANTEC, SPE (2006).
[3] D. A. Grewell, A. Benatar, J. B. Park, *Plastics and Composites Welding Handbook*, Hanser Publishers, Munich, pp. 110-112 (2003).
[4] J. Park, B. Au, J. Esposito, D. Lewis. *Weldability Evaluation on Effects of Filler Content of Polypropylene Copolymers for Electromagnetic Welding Process,* in Proc. of ANTEC, SPE (2007).