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INNOVATIVE REVERSIBLE ASSEMBLING TECHNOLOGIES BASED ON ELECTROMAGNETICALLY-ACTIVE ADHESIVES

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ABSTRACT

The development of efficient and reversible bonding technologies offer cost reduction, resistance to applied load, easy and rapid dismantling and smart recycling processes. Different typologies of electromagnetically-active fillers (oxide nano-particles) were embedded in the thermoplastic matrix of provisional hot-melt adhesives, which have been further tested to assess their thermal behavior when exposed to broadband electromagnetic fields. The study started with a simulation of electromagnetic-thermal behavior of such composites under broadband electromagnetic fields, performed by CST Studio software. To appoint for the best transfer and conversion of electromagnetic to thermal energy for the innovative adhesives, an experimental broadband analysis of dielectric and electromagnetic magnetic properties vs. frequency of the field and temperature was performed on real samples. The procedure followed to optimize a high frequency assembling process has been based on the direct results of the experimental tests (heating rate vs. frequency, amount of nanoparticles and time) as a 3rd order polynomial function. Additionally, the heating rate vs. frequency of the electromagnetic field was modeled in a linearly dependence manner, as well as vs. electromagnetic field power - in a logarithmically dependence manner. Following the results obtained under different conditions as described above, a series of mechanical tests were performed in correlation with actual standards, on the way of prototyping the innovative reversible assembling technology for automotive industry.

Keywords: electromagnetically-active fillers, hot-melts, reversible joining technology

INTRODUCTION

The development of efficient and reversible adhesive bonding technologies is a must for many industries, to offer cost reduction, resistance to applied load, easy and rapid dismantling and smart recycling processes. Our paper is concerned with the development of electromagnetically-active hot-melt adhesives for innovative reversible assembling technologies, along with an appropriate control of electromagnetic power, to avoid material degradation because of high temperature. The proposed techniques have clear advantages in terms of maintaining, disassembling and recycling the end of life products. The process of electromagnetic bonding is based on the principle of electromagnetic heating: electromagnetically-active materials increase their temperature when subjected to a high frequency, alternating current field (J. Rotheiser, 2004). Tailored nanoparticles are currently used in automotive industries to foreshorten the curing time of thermosetting adhesives and ultimately to achieve comparatively minimum assembly time (R. Nichols, 2006). For example, the curing time of a commercially available epoxy based adhesive is about 9-hours;

however with embedded susceptible nanoparticles inside, when it is subjected to electromagnetic field, its curing time is reduced to around 17 minutes. But nowadays the use of thermosetting adhesives is considered obsolete. In light load application areas, hot-melt thermoplastic adhesives is used more often due to short curing time and reversibility at high temperature (J. Park, 2007). Positive conclusions of tests are expected on the way of prototyping the innovative joining technology for automotive industry.

HOT-MELT PRODUCT DESIGN AND SIMULATION

In our study, different typologies of electromagnetically-active fillers were embedded in the thermoplastic matrix of provisional hot-melt adhesives, which have been further tested to assess their thermal behaviour when effectively exposed to broadband electromagnetic fields. Five types of iron based nanoparticles: magnetite Fe_3O_4 (from Sigma-Aldrich), hematite $\alpha\text{-Fe}_2\text{O}_3$ (from Nanophase), cobalt-ferrite CoFe_2O_4 (from Nanotesla), manganese-zinc ferrite $(\text{Mn,Zn})\text{Fe}_2\text{O}_4$ (from Nanotesla), and maghemite $\gamma\text{-Fe}_2\text{O}_3$ (from Nanotesla) were used. The basic formulation of matrix is presented in Table 1. The experimental volume ratio of insertions was up to 2%, corresponding to a mass ratio up to 10%.

Table 1 **Formulation List**

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

Tapping mode-atomic force microscopy (AFM) was used to accurately study the size and shape of the particles, the particle clusters and dispersion technology. Scanning electron microscopy (SEM) was used to study the hot-melt surface and cross-section features. Figure 1 shows that nanoparticles were aggregated into the adhesive matrix in a well-dispersed quasi-homogenous way.

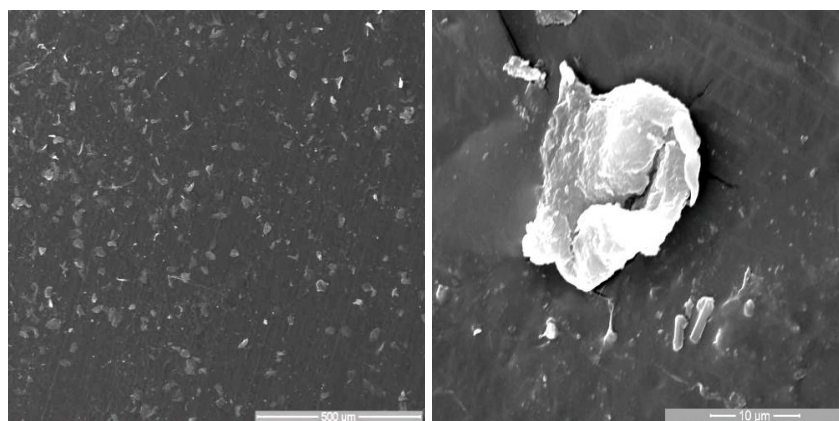


Fig. 1. Hot-melt with 5% by weight of magnetite nanoparticles.: surface analysis (200x), and cross-section analysis (5000x).

The preliminary reference dielectric parameters of hot-melt at 1 kHz were $\epsilon = 14.5$, $\tan \delta = 0.08$, obtained by measurements with broadband dielectric spectrometer. The simulated structure had a cubic shape with a dimension of 5552 μm , to allow the analysis of minimum

20 insertions vs. dimension. The simulation was made using CST Microwave Studio Software, based on a cubic model with equidistant nano-active insertions. The study was realized by the penetration of the field inside the structure, corresponding to the wave plane interaction. The entry ports were placed at a distance equal to the minimum between the 8th part of the wavelength and 5 mesh lines of the structure. The simulations were made between 100kHz and 10 MHz, because at lower frequencies the wavelength is too close to the dimension of the distributed nano-objects, giving errors. The simulation results of electromagnetic energy dissipation and electro-thermal effects are presented in Figure 2. For thermal simulation, an open thermal border around the cell unit was chosen, not to affect the accuracy of calculated limits. A very promising temperature gradient ($\Delta T \sim 83K$) was obtained due to dispersion of electromagnetic energy within the cell unit (the thermal open boundary was set at 273.1K).

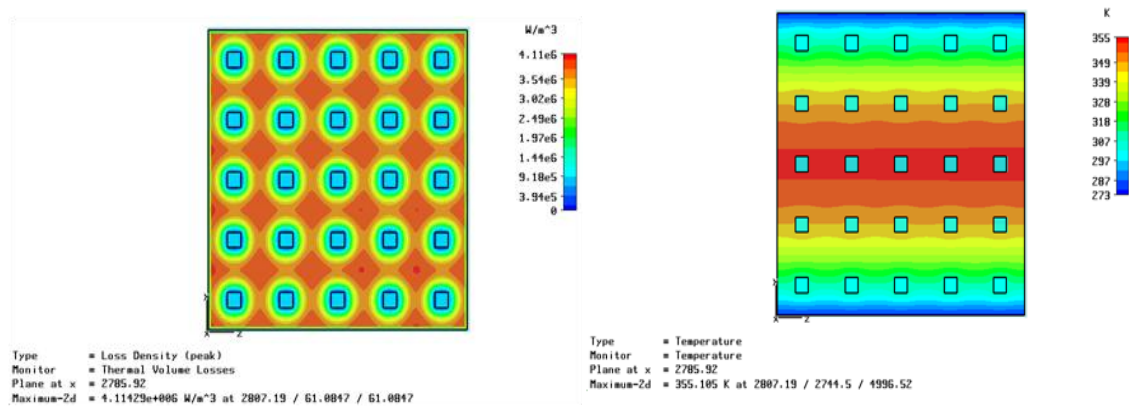


Fig. 2. Thermal energy and internal temperature at 1 MHz

ASSEMBLING TECHNOLOGY DEVELOPMENT AND TEST RESULTS

A model of single-lap-joint based on two superposed sheets was developed, with the dimensions presented in Figure 3. The experimental material to be bonded was a composite of polypropylene with 10% talc filler, commonly used in automotive internal and external applications. For surface preparation, the substrates were cleaned with isopropyl alcohol. The adhesives were 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 (about two minutes), on a specialized equipment, allowing a standardized joint preparation technique with constant adhesive film thickness. The specimens were exposed at room temperature for 24 hours prior to testing.

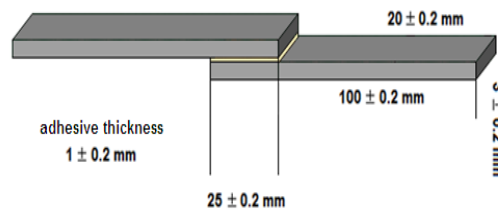


Fig. 3. Joint architecture and dimensions

To appoint for the best transfer and conversion of electromagnetic to thermal energy for the innovative adhesives, an experimental broadband analysis of electromagnetic properties vs. field frequency and internal temperature was performed on single-lap-joint based samples with different type and content of iron based nanoparticles. Both real and imaginary part of permittivity and permeability were considered, in order to obtain the most efficient domain of both dielectric and magnetic losses of developed adhesives. Some results are presented in Figure 4 for a content of 8% magnetite Fe₃O₄ nanofiller. The optimum energy transfer should be experimented at over 100 kHz, where e.g. both permittivity and dielectric losses are increasing vs. temperature, an ideal domain for hot-melt activation. All experimental results were in accordance with the theoretical simulation for the appointed frequency domain.

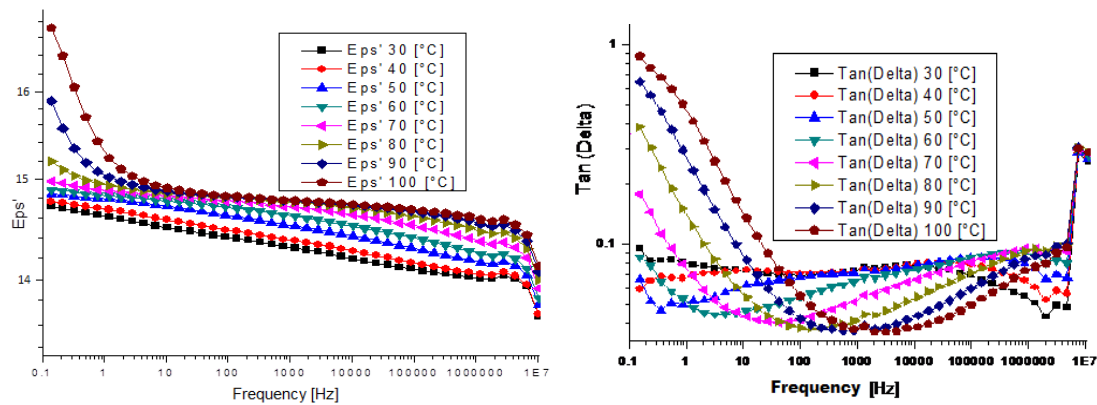


Fig. 4. The electromagnetic characteristics

The next step was related to the development of a power radiofrequency system, to effectively study the electro-thermally joining features of modified adhesives vs. exposure time. The optimization of the high frequency bonding process has been based on the direct results of the experimental tests (heating rate vs. frequency, amount of nanoparticles and time) as a 3rd order polynomial function. Additionally, the heating rate was modelled vs. both frequency of the electromagnetic field – in a linearly dependence manner, and vs. electromagnetic field power - in a logarithmically dependence manner. Some results are presented in Figures 5 – 6.

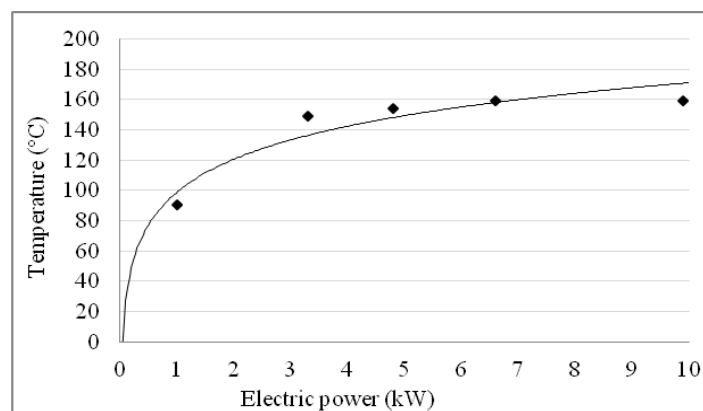


Fig. 5. Internal temperature reached in 40 s vs. electric power (5 % magnetite, 172 kHz).

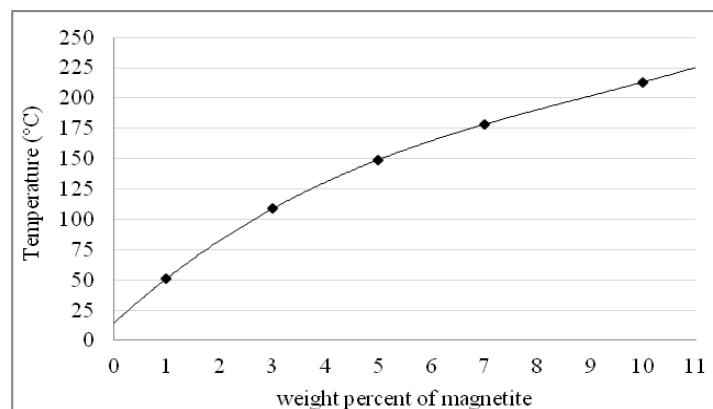


Fig. 6. Internal temperature reached in 40 s vs. magnetite percent (172 kHz, 3.3 kW).

As expected, the heating rate increased continuously with the increasing of the amount of nanoparticles content, especially in 5%-10% domain. Due to technological conditions, the limit of 10% is strongly recommended. On the other hand, the heating rate increased continuously with the electric power of source, but after 3 kW a saturation regime was noticed. It seems that the choice of 3.3 kW as optimized power is reasonable due to equipment cost and reached internal temperature, satisfying the technical conditions of the innovative assembling process. Accordingly, the optimized power radiofrequency system for hot-melts activation was developed for 172 KHz and 3.3 kW. The thermal profiles of hot-melts were finally analysed, emphasizing the superiority of magnetite and hematite fillers, as presented in Figure 7.

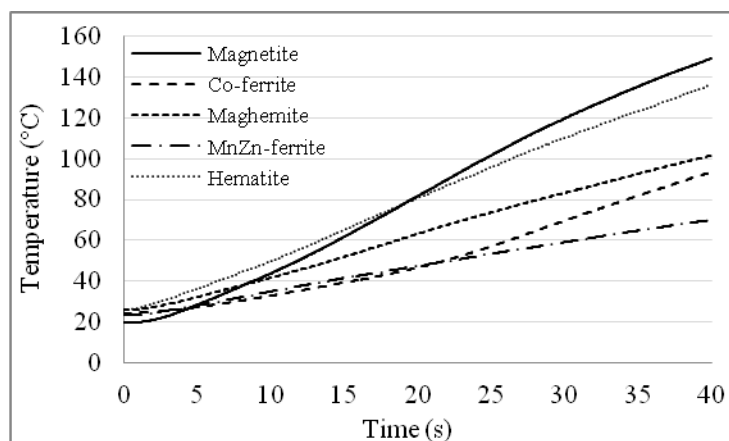


Fig.7 Thermal profiles of adhesives exposed to electromagnetic field (172 kHz, 3.3 kW, 5% w/w filler)

The specimens were submitted to tensile tests using an Instron 5544 dynamometer at room temperature under displacement control (100 mm/min). The failure mechanism was determined by visual inspection. Some results are presented in Figure 8, being in line with homologue characteristics offered by classic hot-melts, actually on market. The advantage of novel technology is given by the behavior of magnetite nano-insertions, acting as activated internal heat sources, generating an impressive heat within short time and offering a homogeneously distributed thermal energy in the adhesive mass, comparing to actual hot-melts which are heated externally in an inhomogeneous way, and may present creeping flows. The use of magnetite nano-insertions allows also the achievement of critical melting temperatures

of hot-melts at lower energy levels, comparing to classical external heating by contact, and preventing the destruction of surrounding material, i.e. the polymeric matrix.

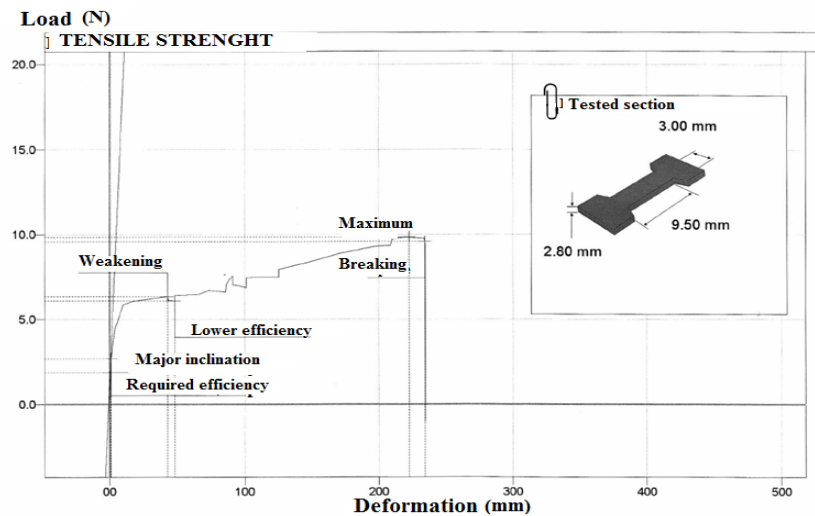


Fig. 8. Results of mechanical tests

The final study was related to assembling process reversibility. The disbonding process is similar to the one of bonding, due to a similar behavior of hot-melts under electromagnetic field. Effective disbonding operation takes up to 50% of the bonding time, but an extra time to clean the surfaces from the residual holt-melt is obviously needed.

CONCLUSION

The paper describes the R&D steps for the technology of electromagnetically-active self-heating hot-melts, to compete with the actual products, which are heated externally. The advantage is given by the homogenous distribution of thermal energy in the bonding area, preventing the creeping and destructions phenomena. The electromagnetic bonding process requires lower energy levels, comparing to classical bonding, and is reversible, offering rapid dismantling and smart recycling. Following the results obtained under different conditions, a series of successful mechanical tests were performed in correlation with actual standards, on the way of prototyping the innovative reversible assembling technology for automotive industry.

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