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Influence of magnetization on thermal and debonding properties of TEPs

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Bonding join

Its recyclability is not easy since it is an irreversible process and adhesive joints cannot be dissembled without damage the substrates.
The addition of these nanofillers change the adhesive properties and it is interesting to create a tailored particle distribution by magnetizing the particles and moving it applying a magnetic field.

Thermally expandable particles (TEPs) are formed by a thermoplastic shell filled with liquid hydrocarbon. When they are subjected to heat, the shell is softened, and the gasification of the hydrocarbon occurs. Therefore, they expand producing the debonding of the joint

Magnetic cork particles
P201730993 and PCT/ES2018/070519

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To manufacture functionally graded joints using magnetized cork microparticles (PAT354/2019).

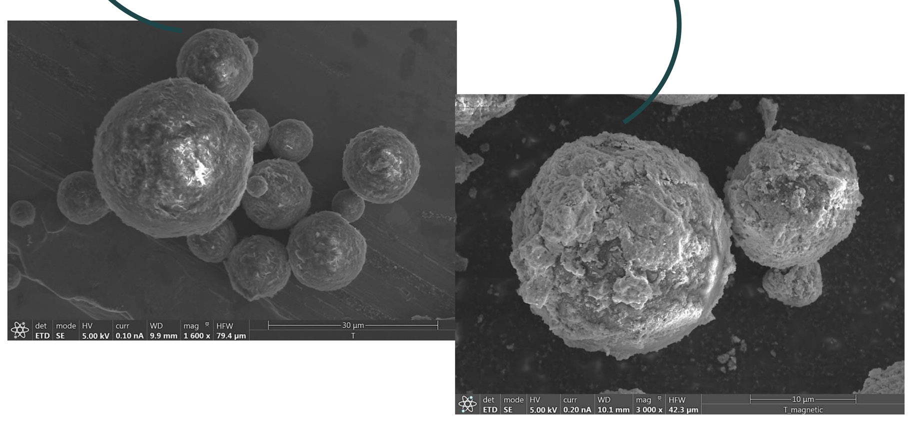
Objectives

(1)Magnetize TEP’s. (2) Study their influence on the curing of the resin.
(3) Check their mobility for the manufacture of graduated joints. (4) Study the debonding process that occurs with temperature

EXPERIMENTAL PROCEDURE

TEP’s or magnetic TEP’s + Araldite 2012

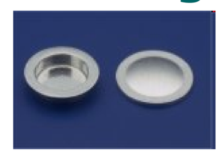
(25 wt. %)

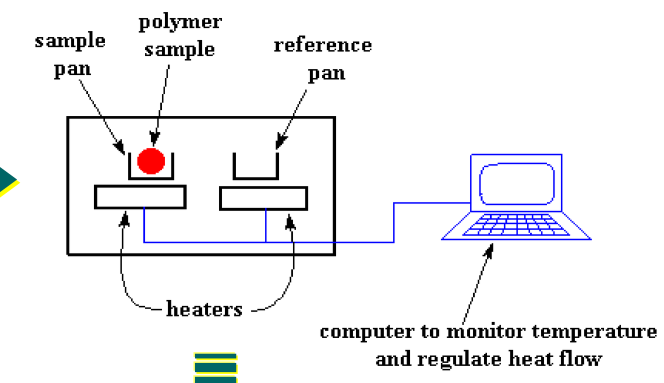


Expancel 031 DU 40
from 10 to 16 µm

Manual mix processes

8 - 8.5 mg mix





Tensile tests

Grit blasted Aluminium
SLJ (single lap joints)
Breaking before applying temperature
Breaking after applying temperature

After curing at RT

DSC
- **Non-Isothermal method (20 ºC/min)**

from 0 to 80 ºC
T_g

DSC
- **Isothermal method (25, 40 and 60 ºC) 30 min**

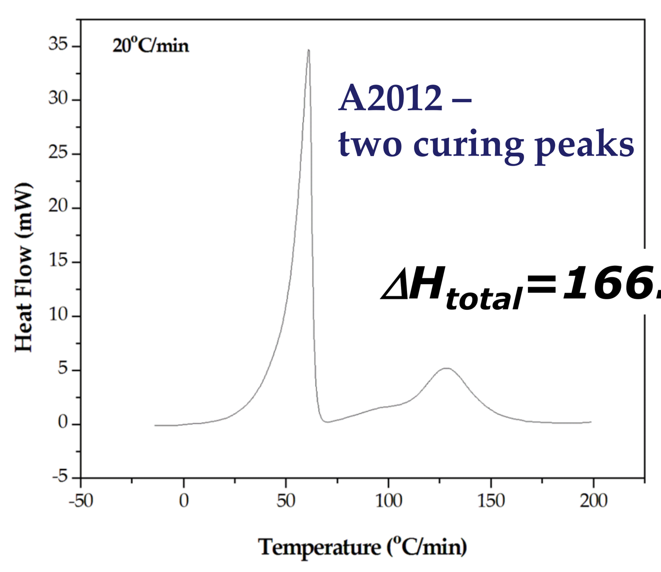
Analyse by Kamal method

Activation energy by Arrhenius equation

RESULTS

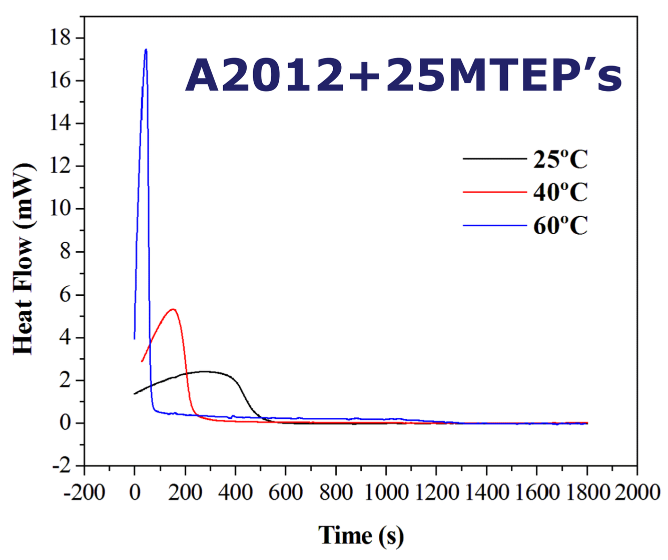
Kinetics

Non-isothermal scan



ΔH_{total}=166.79 J/g

Isothermal scans – first peak



$$\alpha = \frac{\Delta H_{at\ predetermined\ time}}{\Delta H_{Total}}$$

$$\frac{d\propto}{dt} = Kf(\propto)$$

$$\frac{d\propto}{dt} = (k1 + k2\propto^m)(1-\propto)^n$$

Kinetic Parameters

	T (ºC)	k1	k2	m	n
A2012	25	0.04	0.81	1.39	1.92
	40	0.16	9.86	2.36	2.9
	60	0.88	90.15	3.27	3.84
+25TEP	25	0.05	18.65	2.94	5.23
	40	0.32	65.7	3.92	4.25
	60	1.67	534.09	4.92	6.15
+25MTEP	25	0.11	90.14	3.56	7.15
	40	0.15	52.64	1.94	12.78
	60	0.036	3.13	3.72	5.67

K1 is less than K2, so it determines the reaction rate

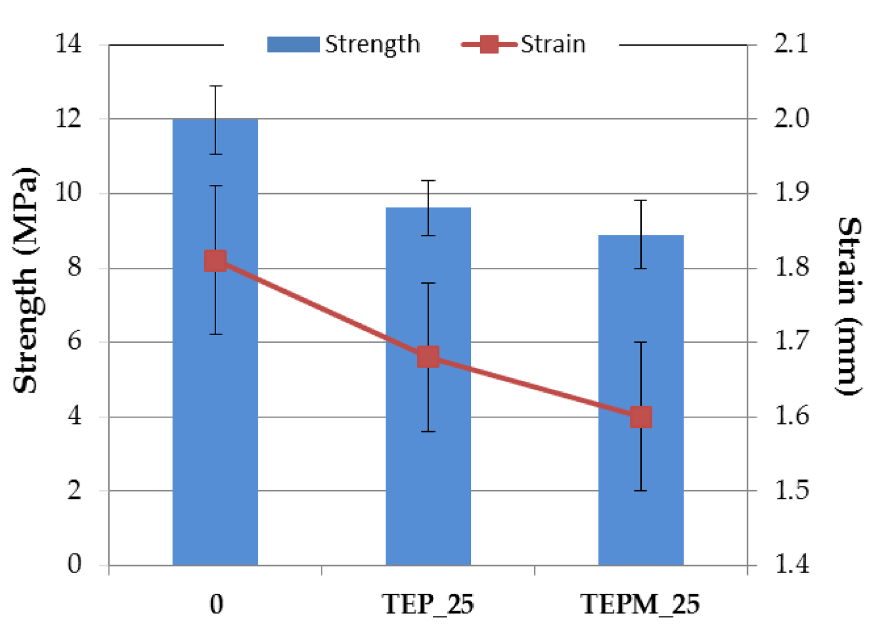
Activation energy

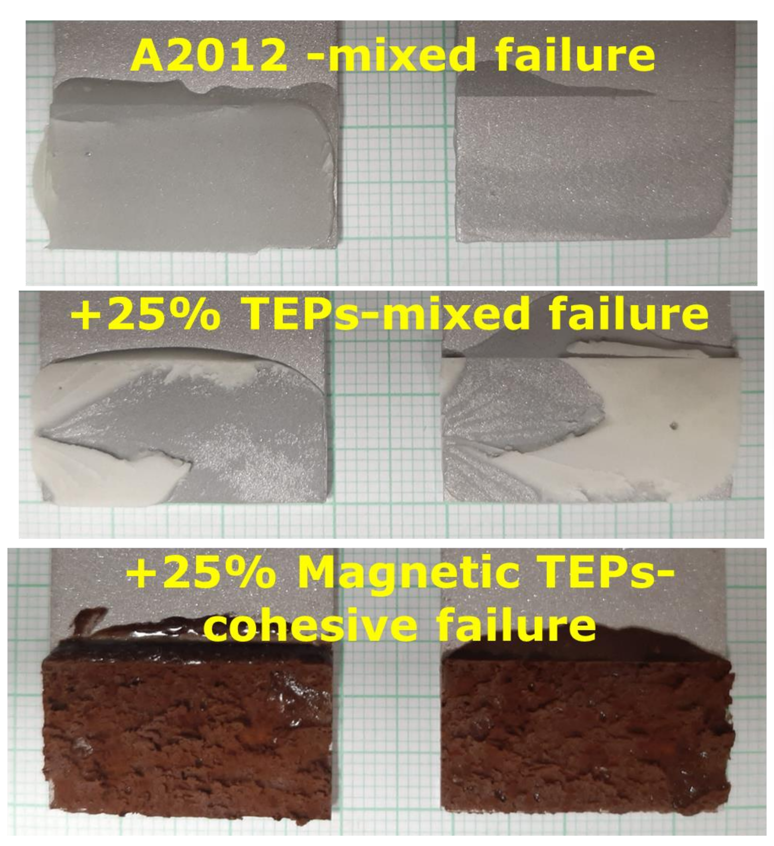
$$LnK = LnA - \frac{Ea}{RT}$$

	A	Ea (kJ/mol)
A2012	26	73
+25TEP	30	82
+25MTEP	9	28

TEP’s ↑ reaction rate and Ea
MTEP’s ↓ reaction rate and Ea
Their effect is stronger than for TEP’s

Tensile Strength tests





After warm-up (TT)

Samples with 25% TEPs have a remaining strength left (≈ 1.3 MPa), they do not break completely. 115-130 ºC / 4-5 min
However, with 25% magnetic TEPs break at less temperature and less time.
95-110 ºC / 2-2:30 min

CONCLUSIONS

- Preliminary tests have shown that the used adhesive cures in two stages. The first is very fast and the second requires a temperature above 100ºC.
- The used TEP’s opens from 90 ºC, therefore thermal curing is not possible. When curing at room temperature, T_g is low - around 35-37 ºC. Addition of TEP`s or magnetic TEP’s does not produces changes on T_g.
- TEP's particles influence the curing degree, it decreases 15% and 30% in the case of untreated TEP's and magnetic TEP’s, respectively.
- The curing rate changes with temperature, and with the addition of TEPs. Clear resin increases the curing rate, being the increase in k2 (autocatalytic reaction) very evident. Something similar can be observed for the resin with 25% of TEPs, with a greater increase in the autocatalytic rate. The addition of magnetic TEP's totally changes the curing process, although the reaction is also fast, the autocatalytic reaction rate decreases with temperature. However, the reaction at room temperature is faster than in clear resin or resin with TEP’s.
- Magnetic TEP's can be moved with a magnet when they are inside the resin, therefore they can be placed in a certain place of the joint.
- The SLJ samples were tested after one week. The presence of TEP's decreases the strength and strain of the adhesive bonding. Although there is no significant difference between clear resin and resin with TEP's, there is a significant difference with magnetic TEP's.
- When the samples are heated above 90°C, the adhesive bond begins to break. The samples with magnetic TEP's completely break in 2-3 min, and the samples with untreated TEP's have a residual strength of approximately 1.2 MPa after 5 min in the oven between 115 and 130 ºC.