# Functionalization of adhesives and composite matrix by micro and nanoparticle addition.

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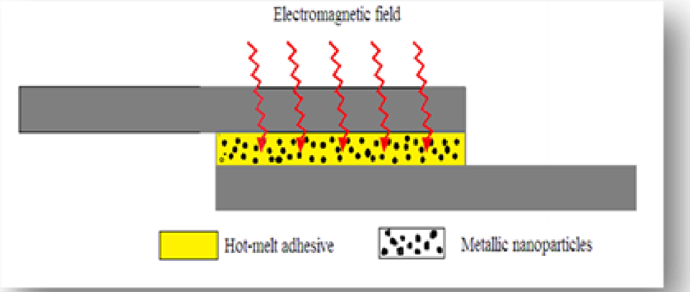
# Join plastic components with nanomodified adhesives and induction heating system. Background and motivations.




- Use of iron oxide coupled with electromagnetic induction

Where we are:

Challenge for the future:

- Switch on a smarter technology which the use of a robot arm.
- Use of lighter susceptors as graphene nano-platelets (GnP).
- Reduction of the assembly time and introduction of the possibility to separate when necessary.

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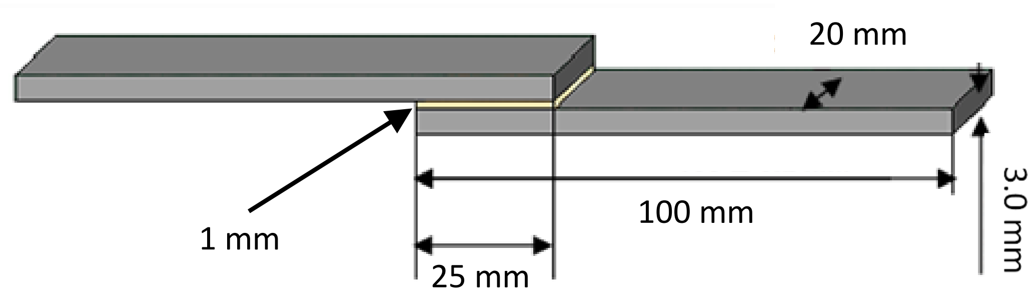
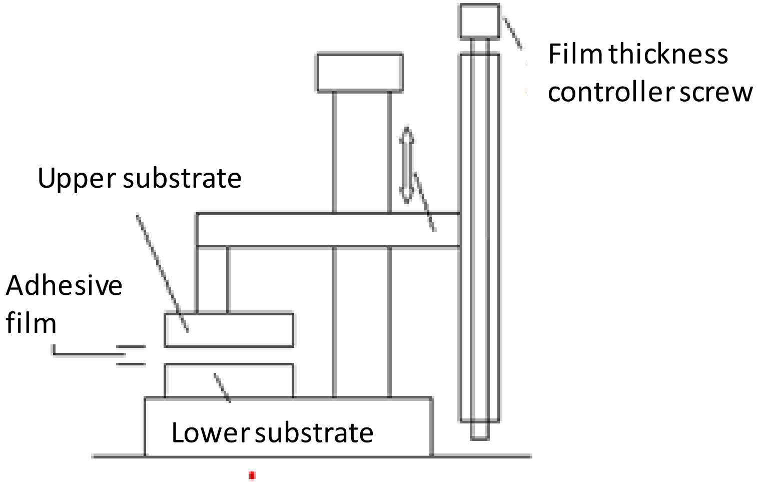
# Specimens preparation.

Mechanical properties were determined with the use of the single lap joint (SLJ) test. The main size of SLJ are shown in figure 1.




The overlap length was chosen after a set of preliminary tests in order to emphasize the adhesive behaviour and obtain mainly cohesive failures.

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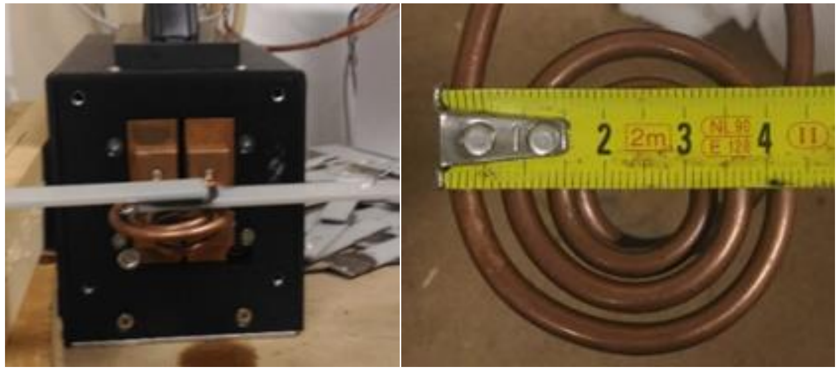
# Materials and methods

1. Polyolefin-based hot-melt adhesive (HMA).
2. Iron oxide (Sigma Aldrich) with a particles dimension less than 50 nm.
3. Iron particles (Goodfellows) with three different particle diameters: 450 µm, 60 µm and 1-6 µm.
4. Polypropylene copolymer (Lyondell Basel) with 10% of talc (Substrates of the Single Lap Joint specimens).
5. Twill, 2 x 2 type, E-Glass/Epoxy composite with a mass of 250 g/m2 and resin mass content of 36.5%.
6. Particle heating by means of electromagnetic field – inductor shape and excitation parameters.



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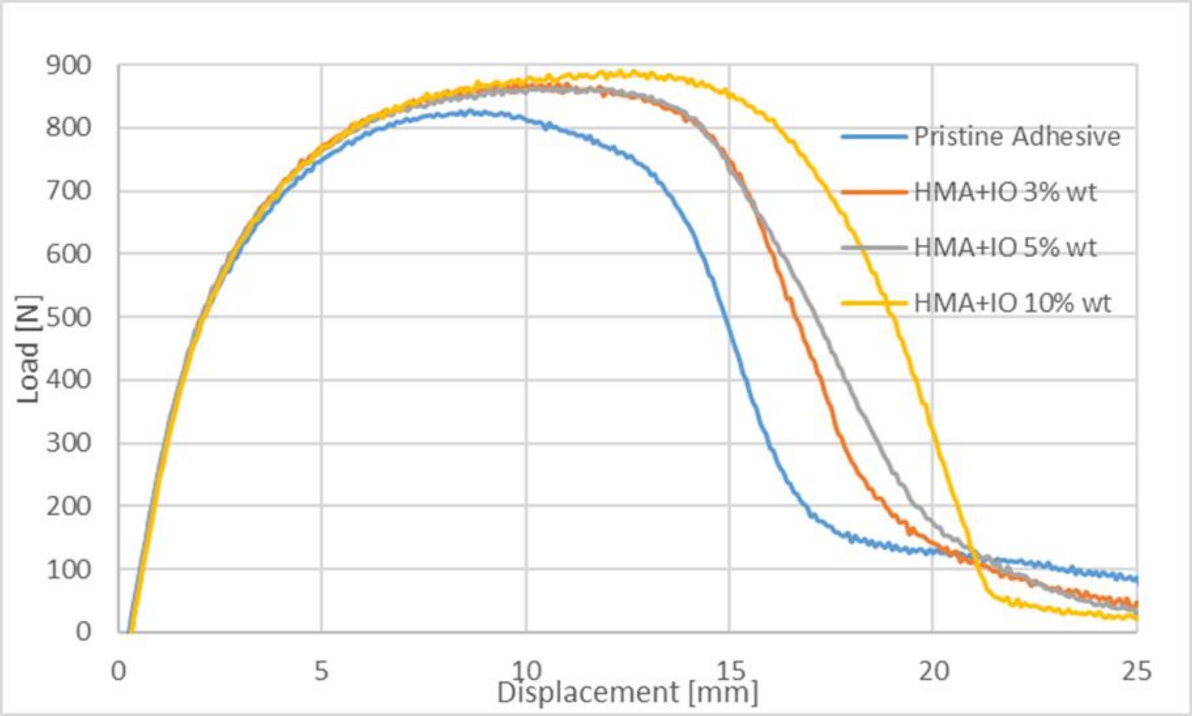
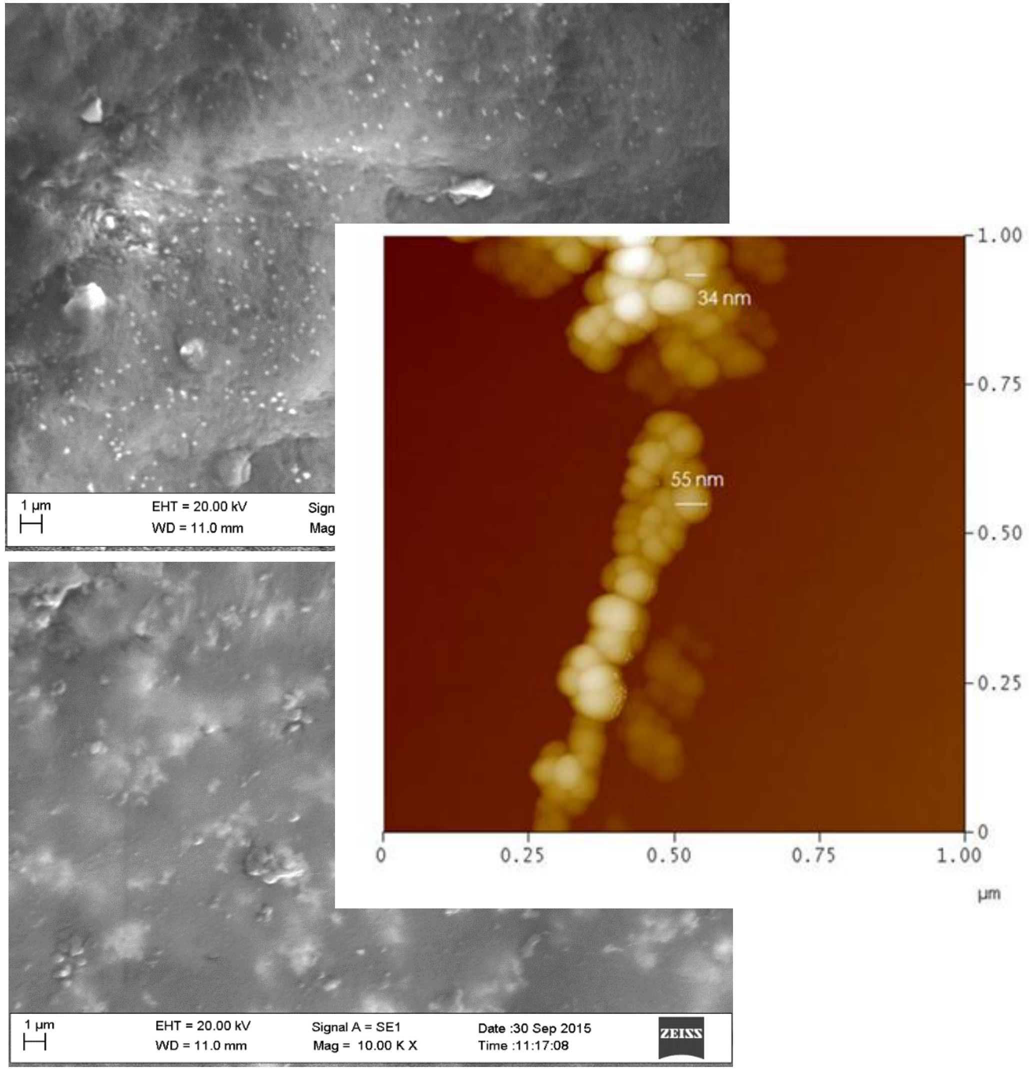
# Mechanical test of HMA modified with iron oxide (<50nm).

## Mechanical tests and SEM analysis




Dimensions of the specimen: 100x20x3 mm

Overlap: 25mm    Adhesive layer thichness: 1 mm

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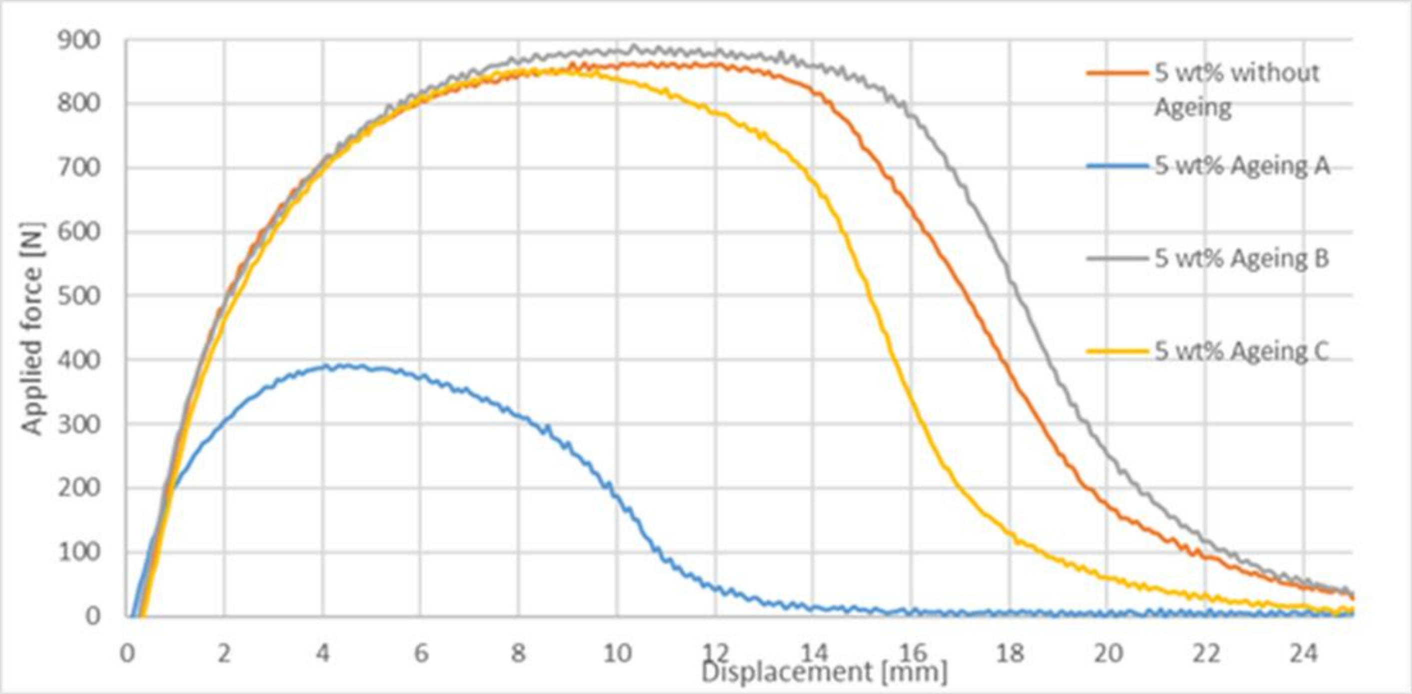
# Ageing cycles

**Cycle A**: Exposure at 90°C without the control of the Relative Humidity (RH) for 500 h.

**Cycle B**: Exposure at 40°C with RH set at 98% for 500 h.

**Cycle C**:Exposure at 80°C without RH for 24 hours,

Exposure at 40°C with RH set at 98% for 24 hours,  Exposure at - 40°C for 24 hours.

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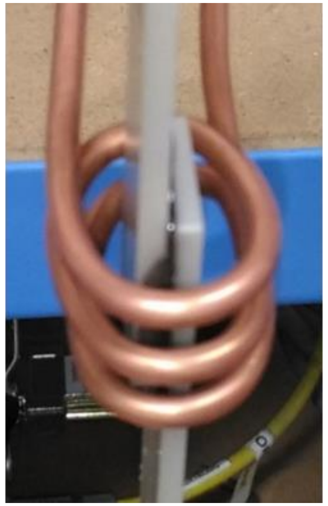
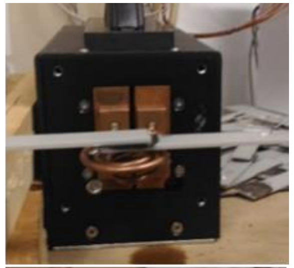
# Induction heating (shown in the second hearing presentation)

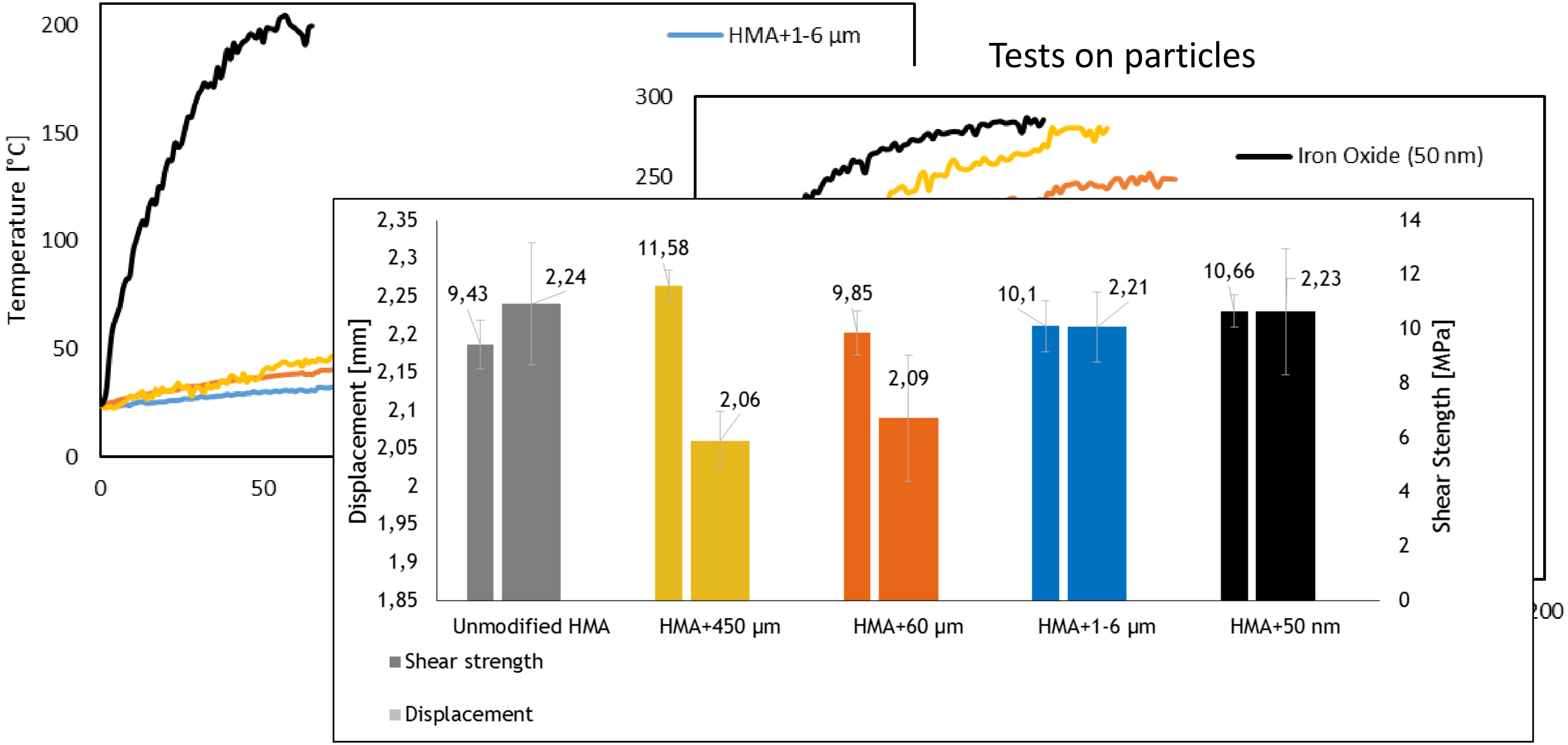
Studied parameters of the inductor:

1. Diameter of the coil pipe.
2. Frequency of the electro-magnetic field (given by the length and the shape of the coil).
3. Shape of the coil.
4. Applied current.


# Separation with iron.

Tests on particles




F= 275 kHz
I=550 A

Dimensions of the specimen: 100x25x2.5 mm
Overlap: 20mm    Adhesive layer thickness: 0.5 mm

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# Main findings

1. Configuration for the separation time: Highest frequency, current intensity between 400 A and 550 A, 10% wt of Iron oxide.

2. Pancake coil can be used to separate the nano-modified adhesive joint.

3. Micro iron particles are not suitable to separate adhesive.

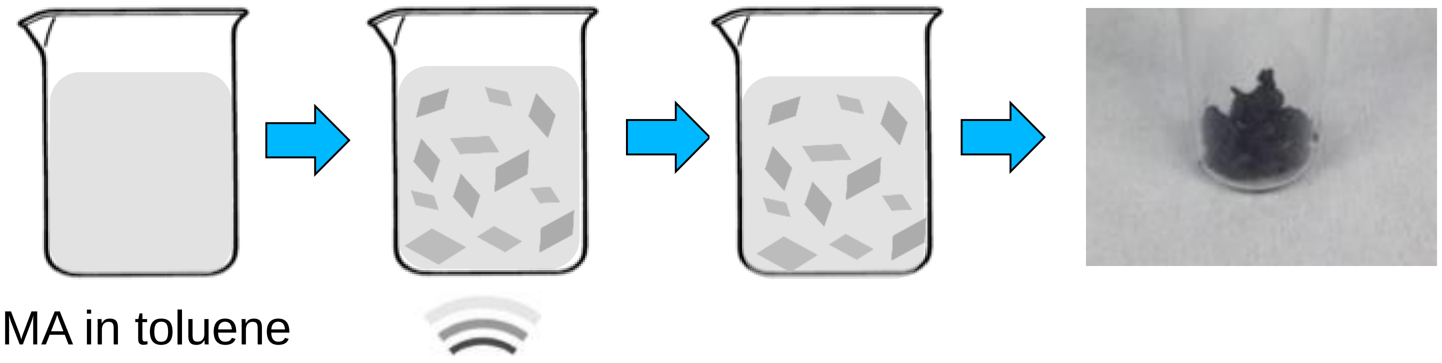
Other tests (shown in the previous hearing presentation):

1. Drop dart impact tests.

2. TGA, Fourier FT-IR, DSC

# Join plastic components with graphene nanomodified adhesives and microwave.

1. Use graphene nanoplatelets as microwave susceptors.
2. Cheaper solution.

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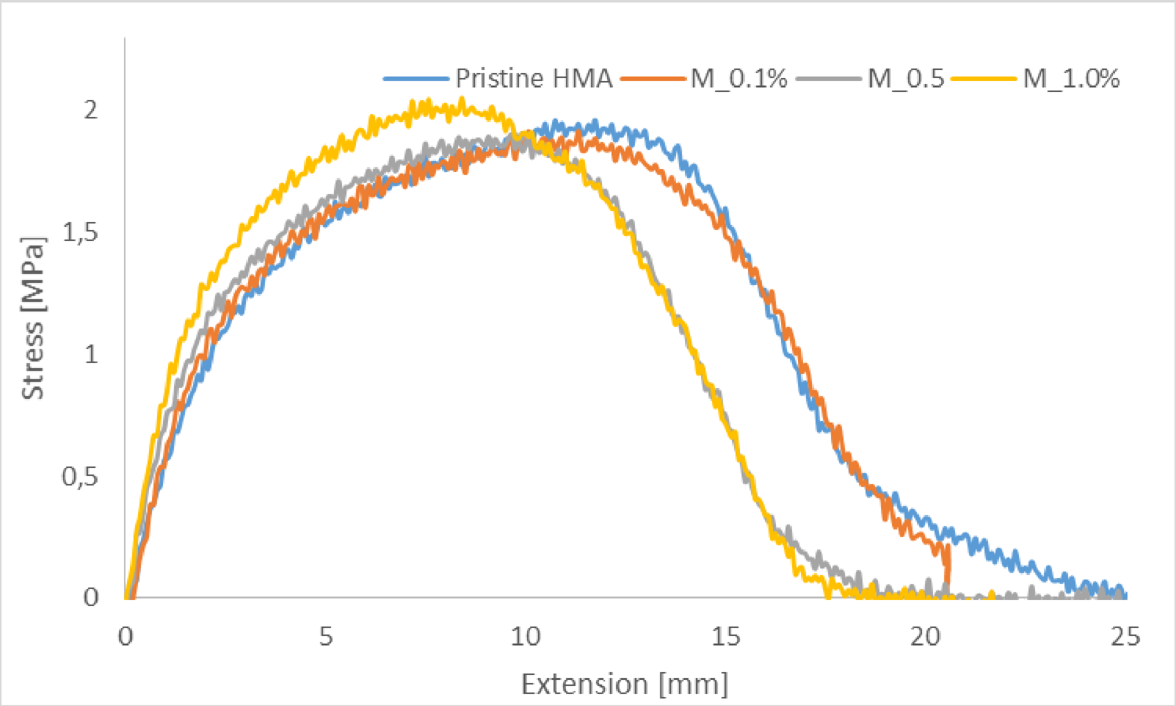
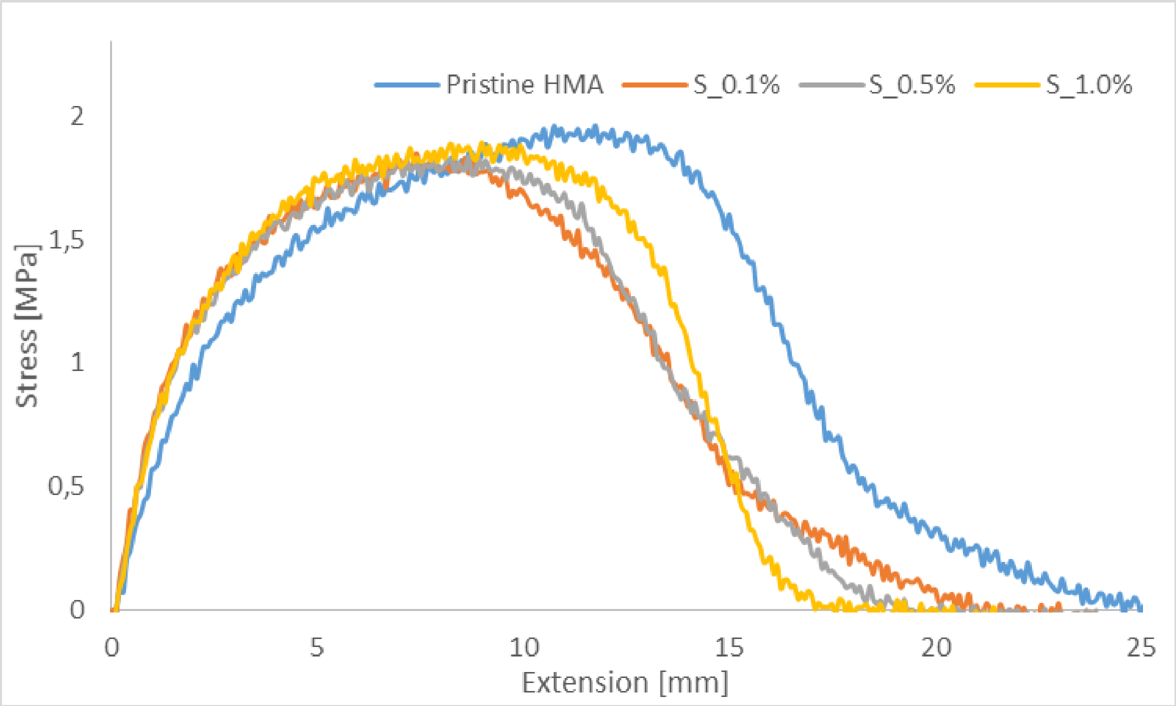
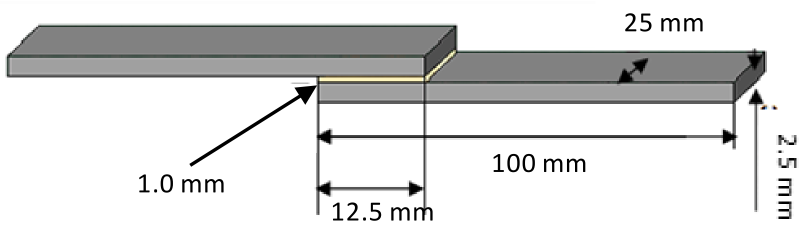
# Adhesive modified with graphene nanoplatelets. Mechanical performances: SLJ tests.

M=Mechanical mixing
S=Sonicated mixing

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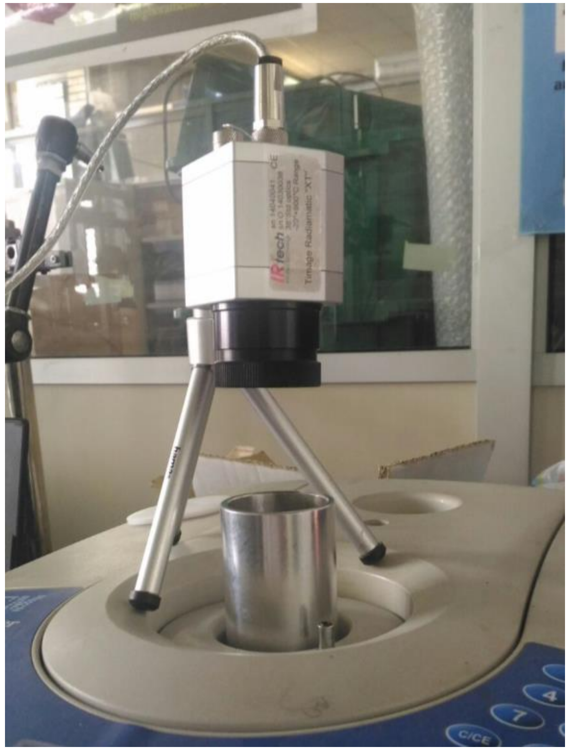
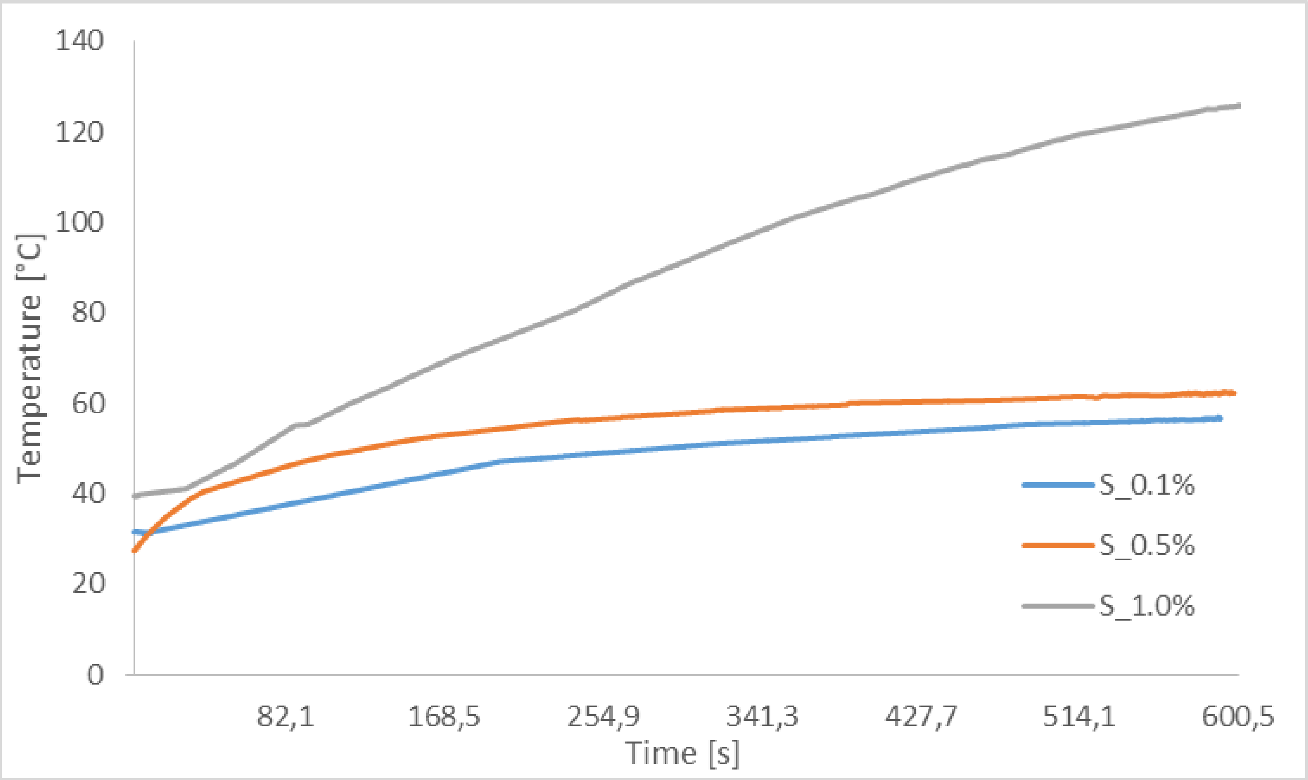
# Mechanical performances: SLJ tests.

| Maximum loads | Pristine | 0.1% | 0.5% | 1.0% |
|---|---|---|---|---|
| M_ [MPa] | 1.91 | 1.88 | 1.93 | 2.01 |
| Standard deviation | 0.058 | 0.109 | 0.099 | 0.045 |
| Increase/decrease | | -1.5% | +1.05% | +5.2 |
| S_ [MPa] | | 1.86 | 1.74 | 1.90 |
| Standard deviation | | 0.055 | 0.058 | 0.104 |
| Increase/decrease | | -2.5% | -8.6% | -0.6% |

# Microwave tests: Configuration of the test and Time-Temperatures curves.



The microwave parameters that can be controlled in this system are power and time. The power was set on the maximum values 300 W for 10 minutes for this preliminary analysis.




Microwave CEM Discover:
Magnetron Frequency: 2450 MHz
Power Output: 300 Watts

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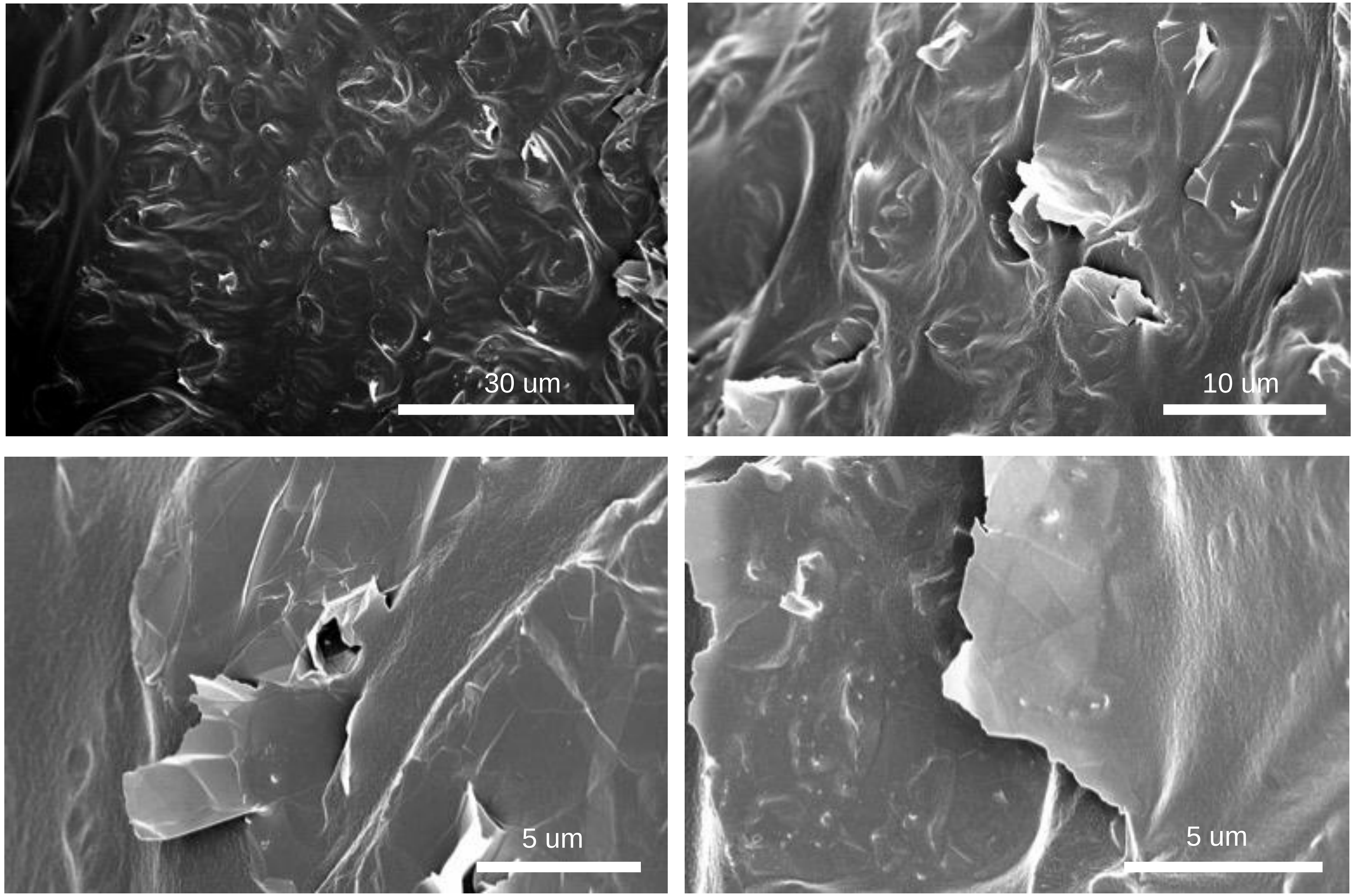
# SEM analysis of the sonicated adhesive.

SEM images show homogeneous distribution of the GNPs into the polymer matrix




Other performed tests:

1. TGA
2. DSC

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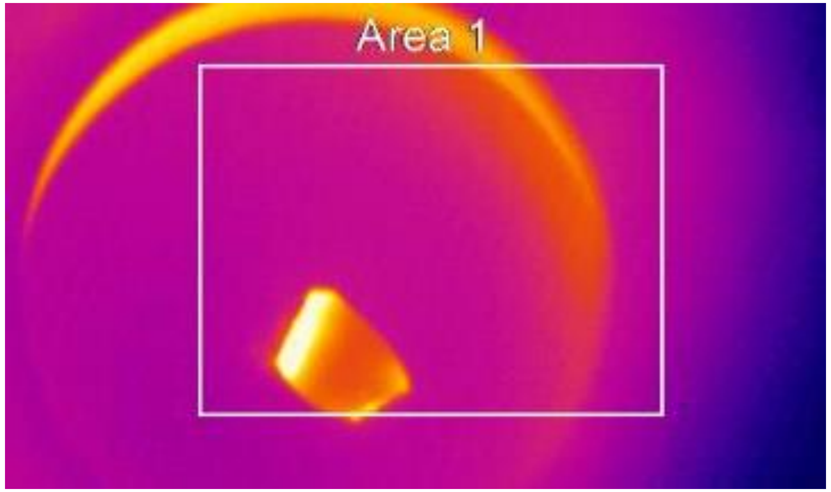
# Main findings.

1. Maximum loads of the modified adhesives are close to the pristine HMA.

2. Good dispersion of particles in the HMA ensures that particles do not burn.

3. Higher concentrations should be tested in order to reach the melting point of the adhesive.

4. SEM analysis should be performed in order to verify if there are some areas that have a lower concentration of GnPs.

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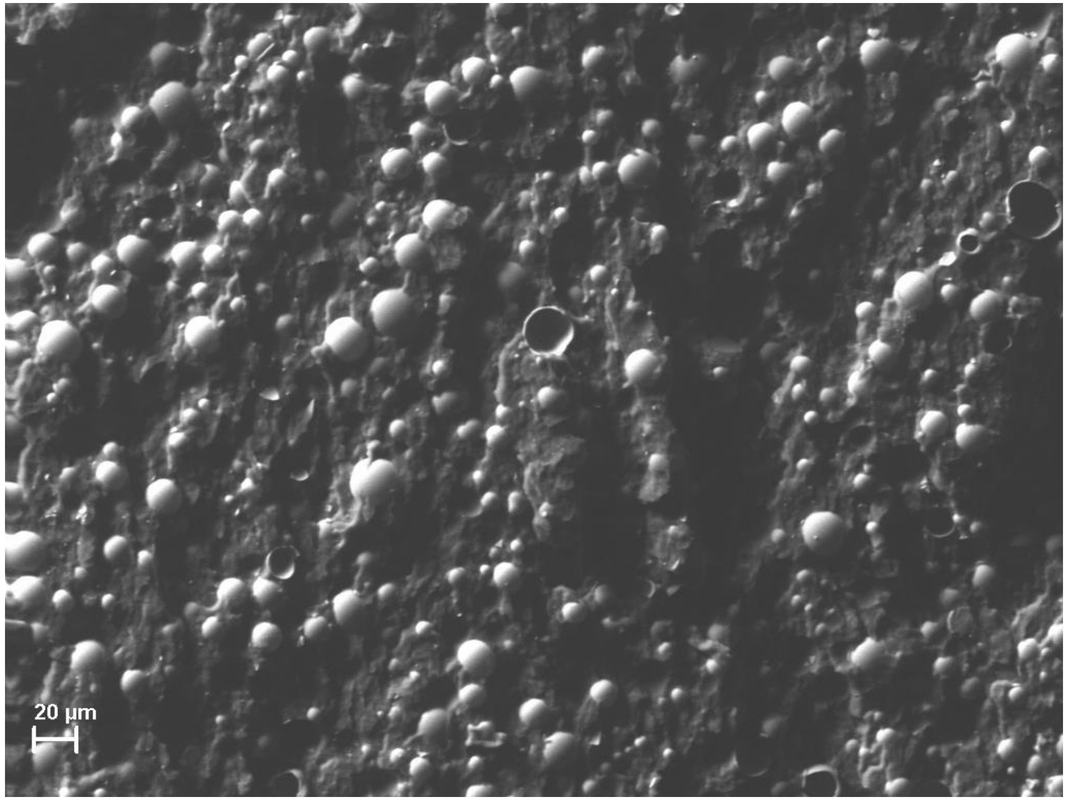
# Electrically conductive syntactic foam.




Background and motivations:

1. What is syntactic foam?
2. Aim of an eclectically conductive material.

Material used:

1. Hollow glass spheres.
2. Epoxy resin.
3. Graphene nanoplatelets.
4. Carbon black.

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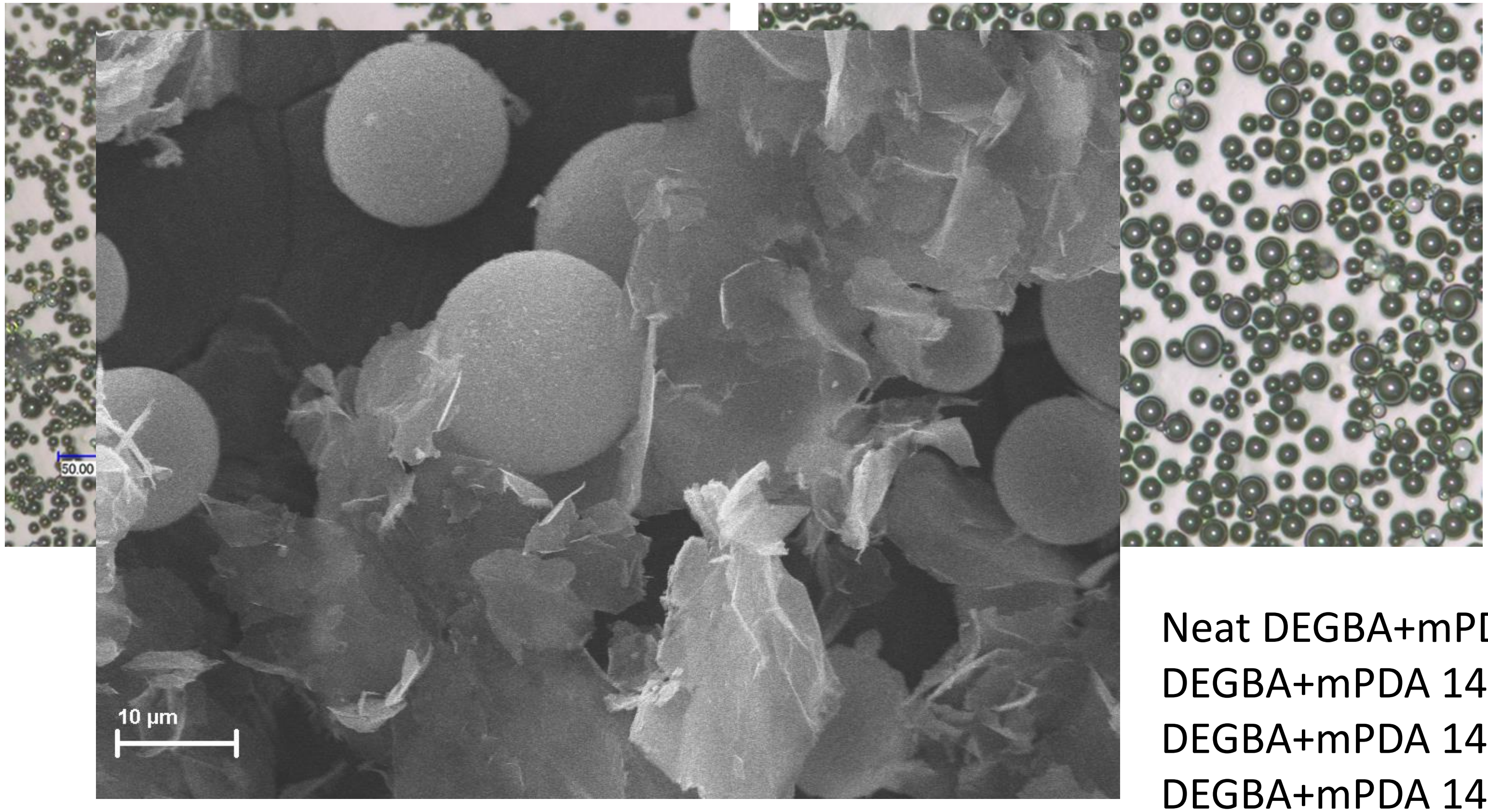
# Preliminary analysis.




Neat DEGBA+mPDA 14.5phr
DEGBA+mPDA 14.5phr+GB30%vol
DEGBA+mPDA 14.5phr+GB30%vol+1%volGnP-M25
DEGBA+mPDA 14.5phr+GB30%vol+1%vol Carbon black
DEGBA+mPDA 14.5phr+GB30%vol+1%volGnP-M25 4 (1-pyrenecarbaldehyde Treated)

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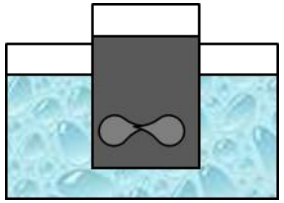
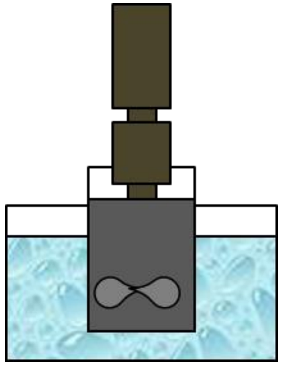
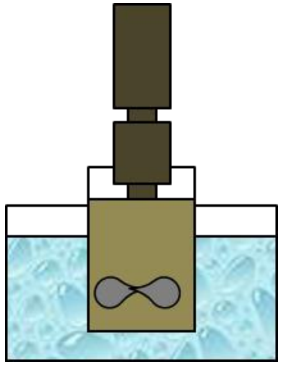
# Preparation of the materials.



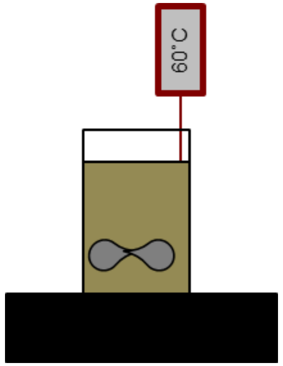
1: Add 1% wt of GnP to 500g of acetone and stir with magnetic stir bar



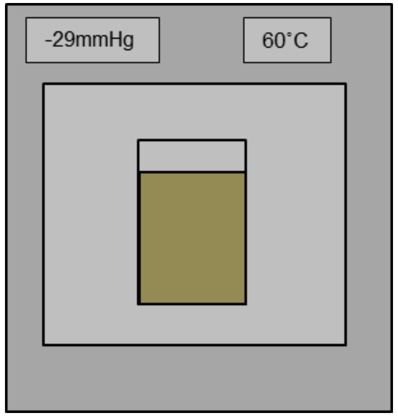
2: Sonicate GnP mixture for 5min at 70W in dry ice bath while stirring



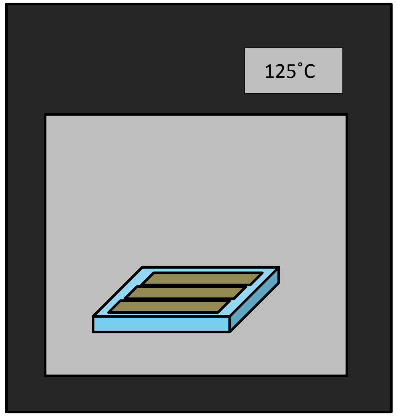
3: Add 180g of DGEBA and sonicate for 5min at 70W in dry ice bath while stirring




4: Drive off acetone at 60˚C under stirring

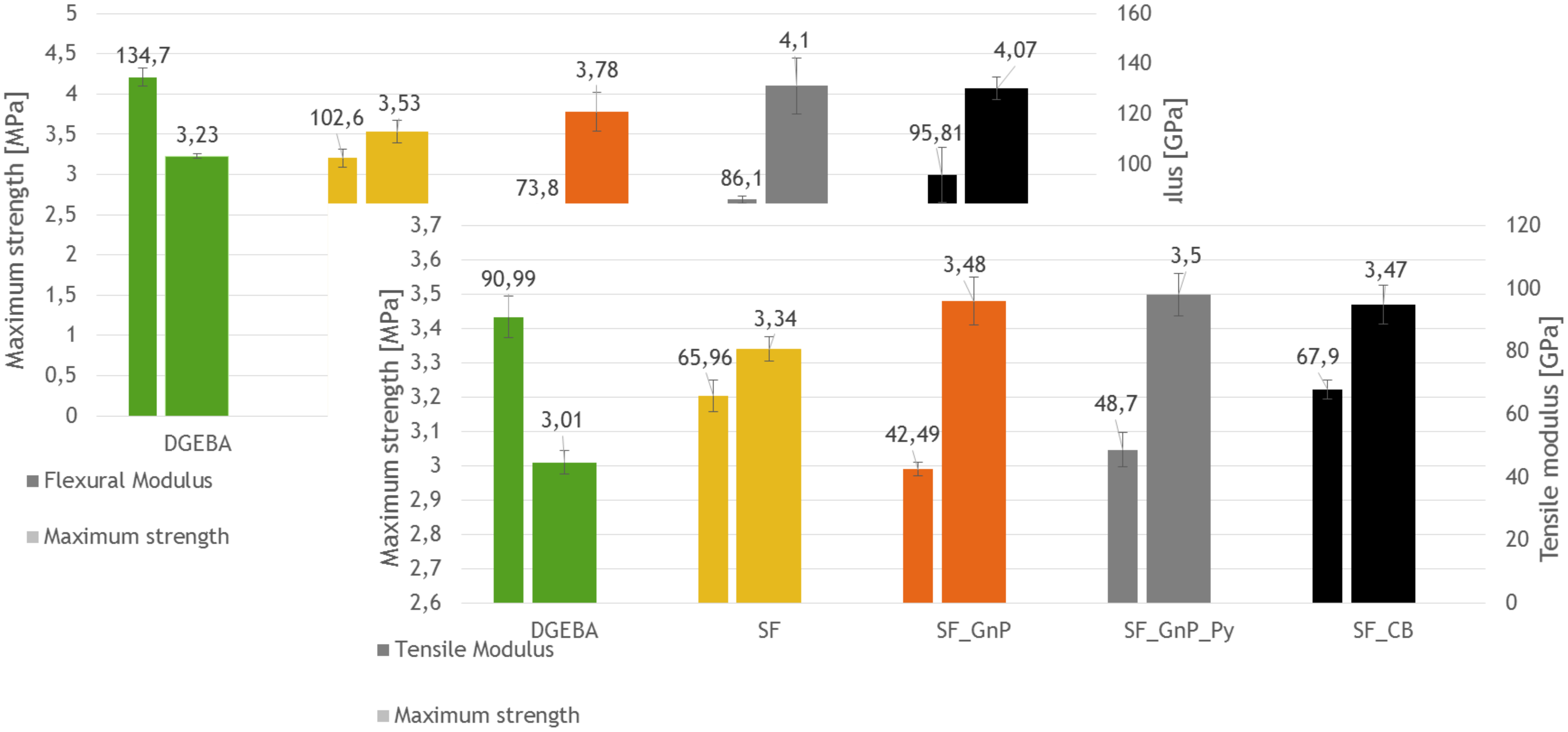



5: Add 14.5phr mPDA, hand mix and degas




6: Cast into silicone molds and cure at 75˚C & 125˚C for 2h each

# Mechanical tests.

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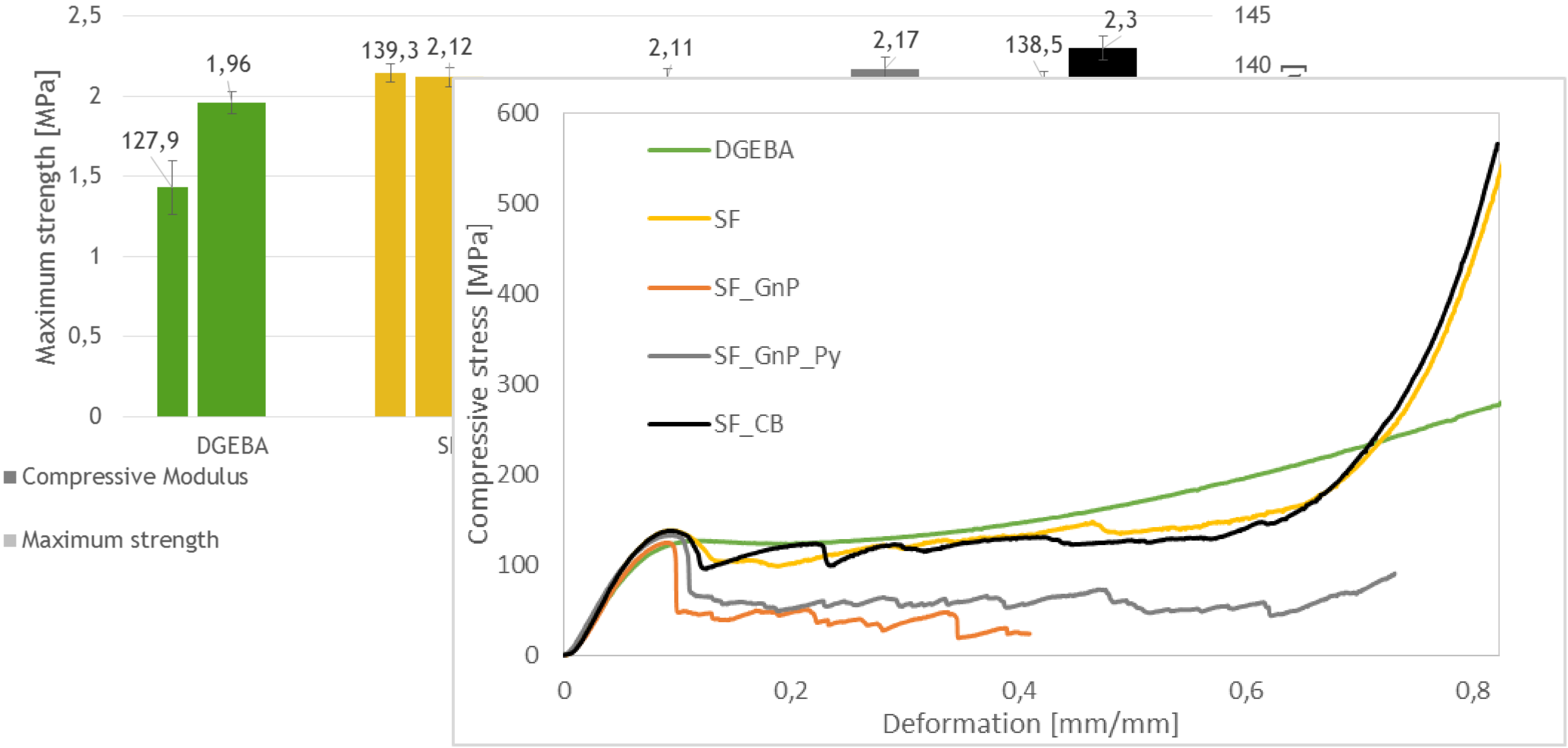
# Mechanical tests.

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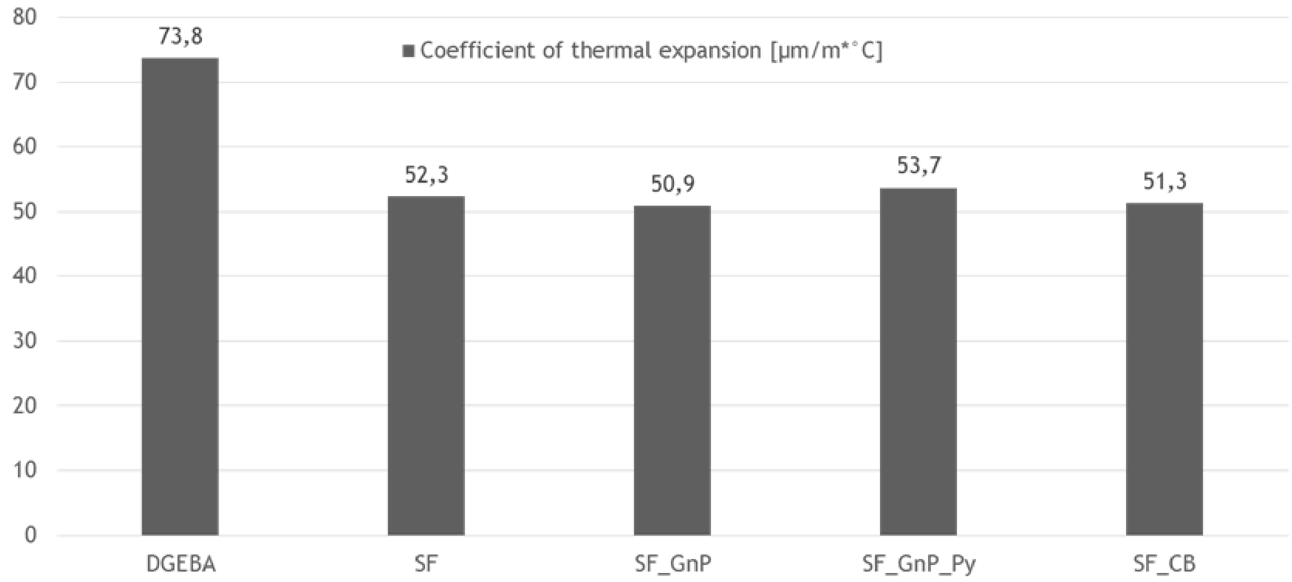
# Impact tests and TMA.

| | DGEBA | SF | SF_GnP | SF_GnP_Py | SF_CB |
|---|---|---|---|---|---|
| **Absorbed energy [J/m]** | 25,57 | 19,49 | 16,40 | 14,95 | 20,77 |
| **Standard deviation [J/m]** | 5,5 | 3,25 | 3,41 | 3,47 | 3,95 |
| **Gap DGEBA-SFs** | -- | -24% | -36% | -40% | -19% |

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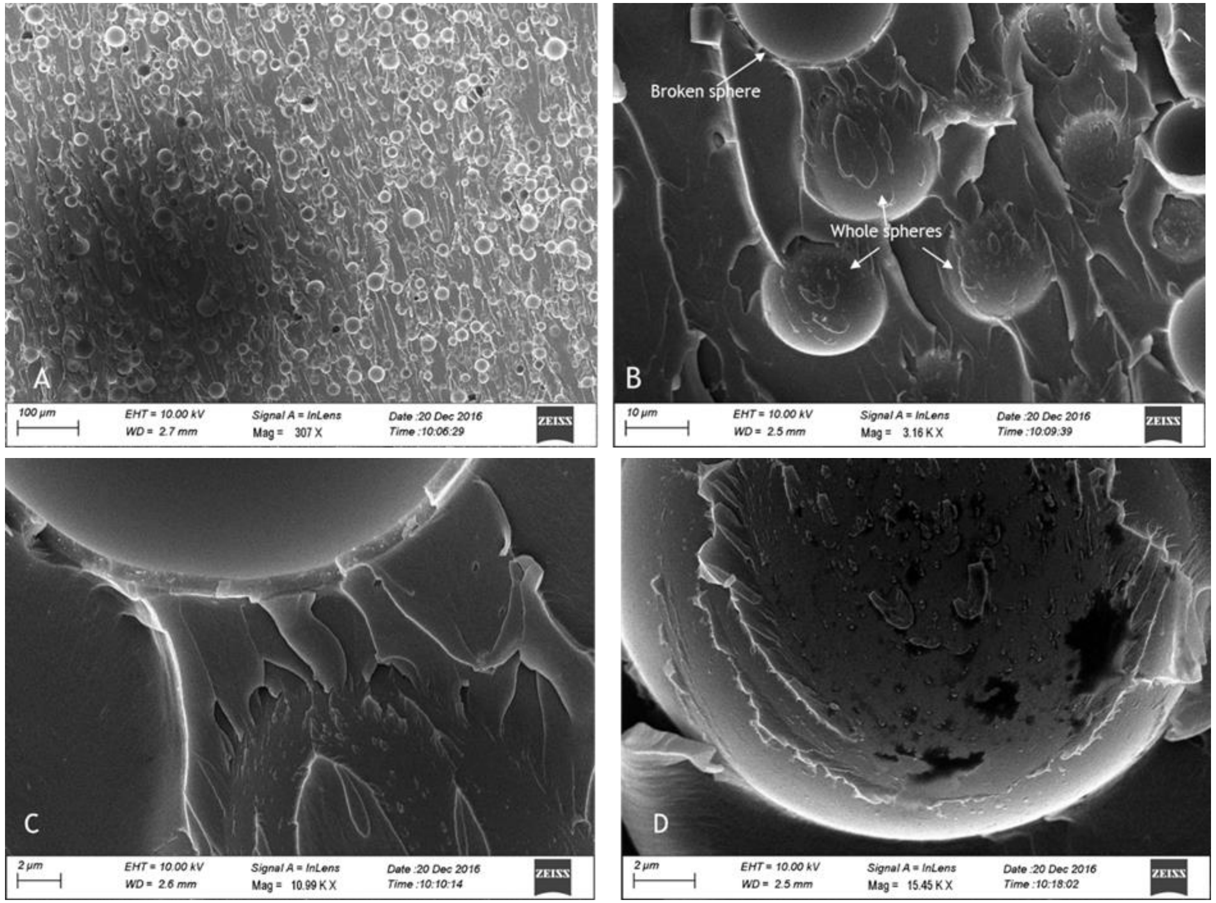
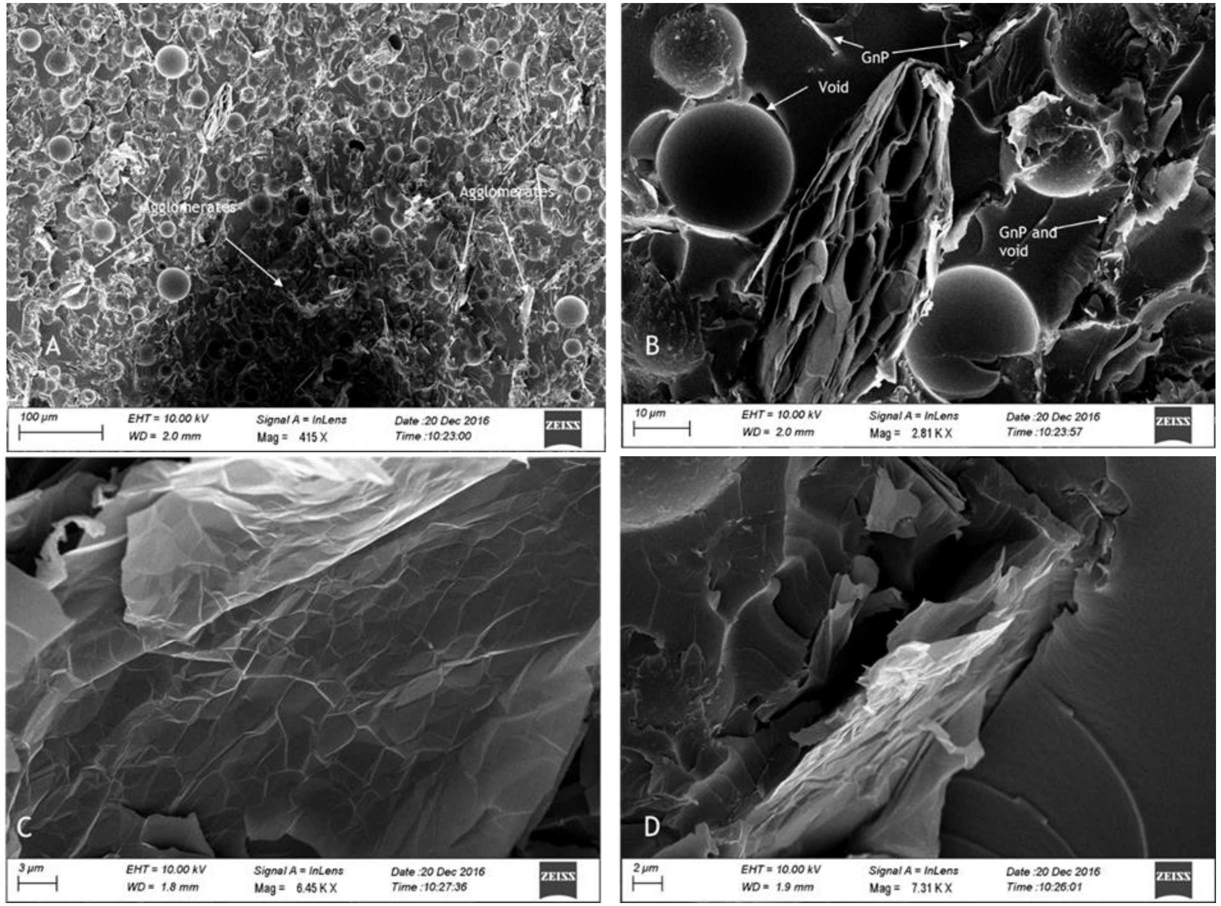
# Scanning electron microscope analysis.

GB

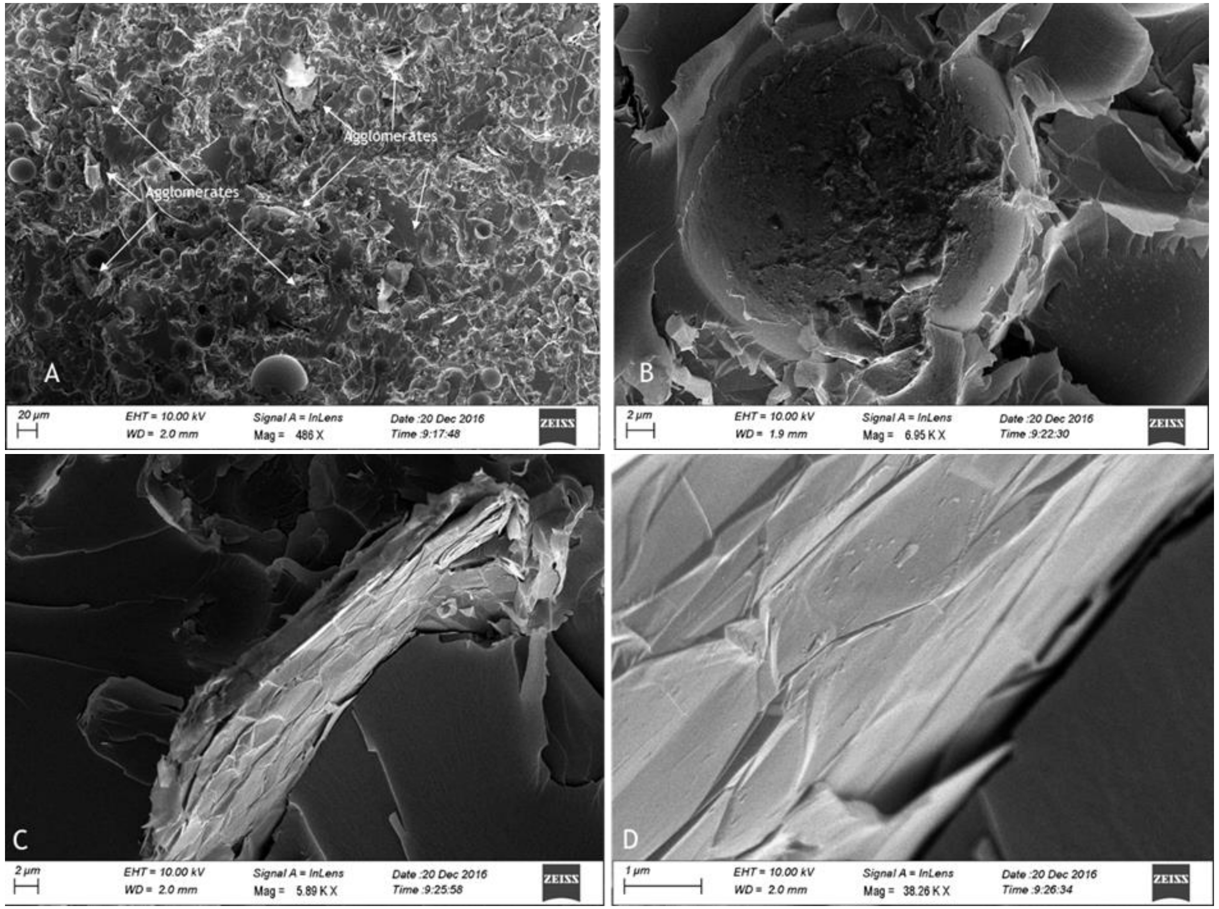



SF_GnP

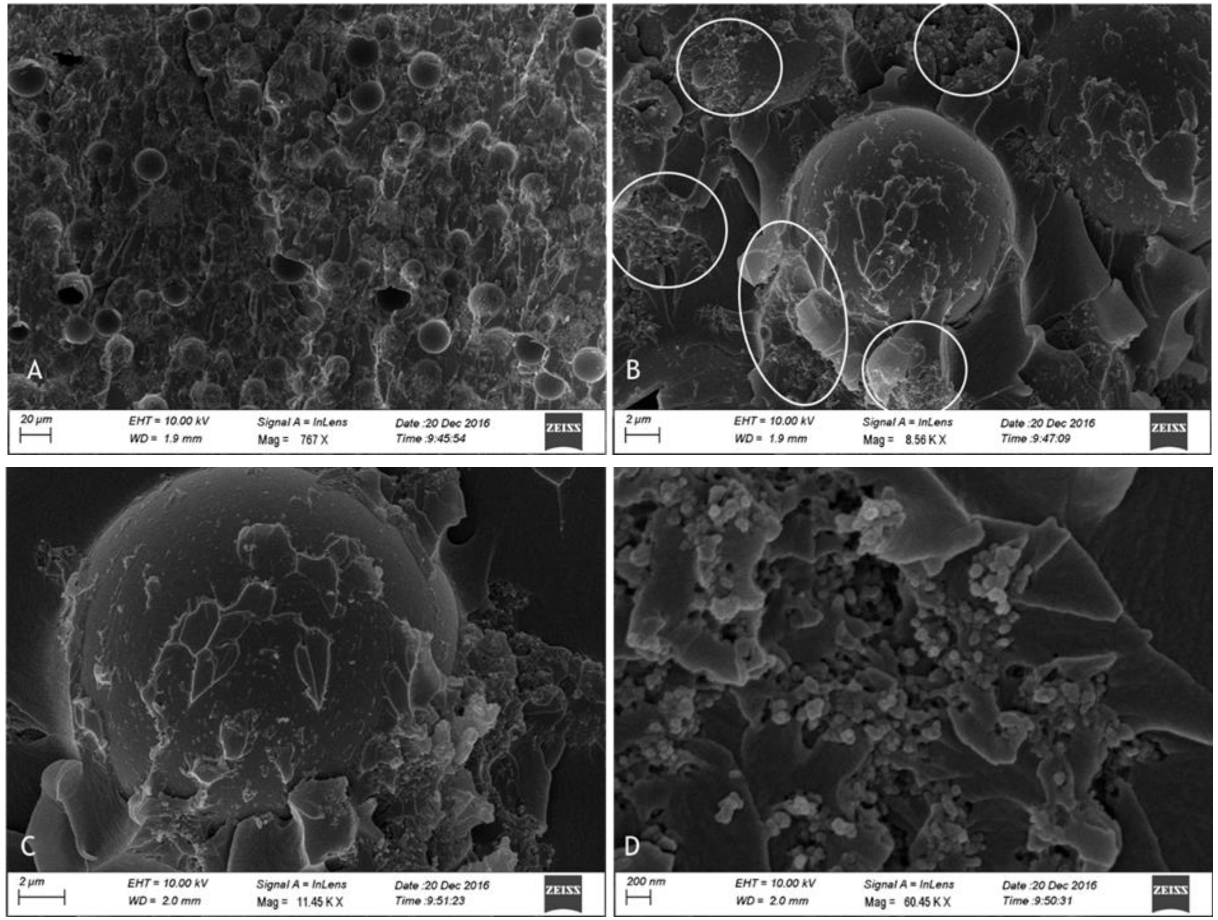


# Scanning electron microscope analysis.

SF_GnP_Py




SF_CB

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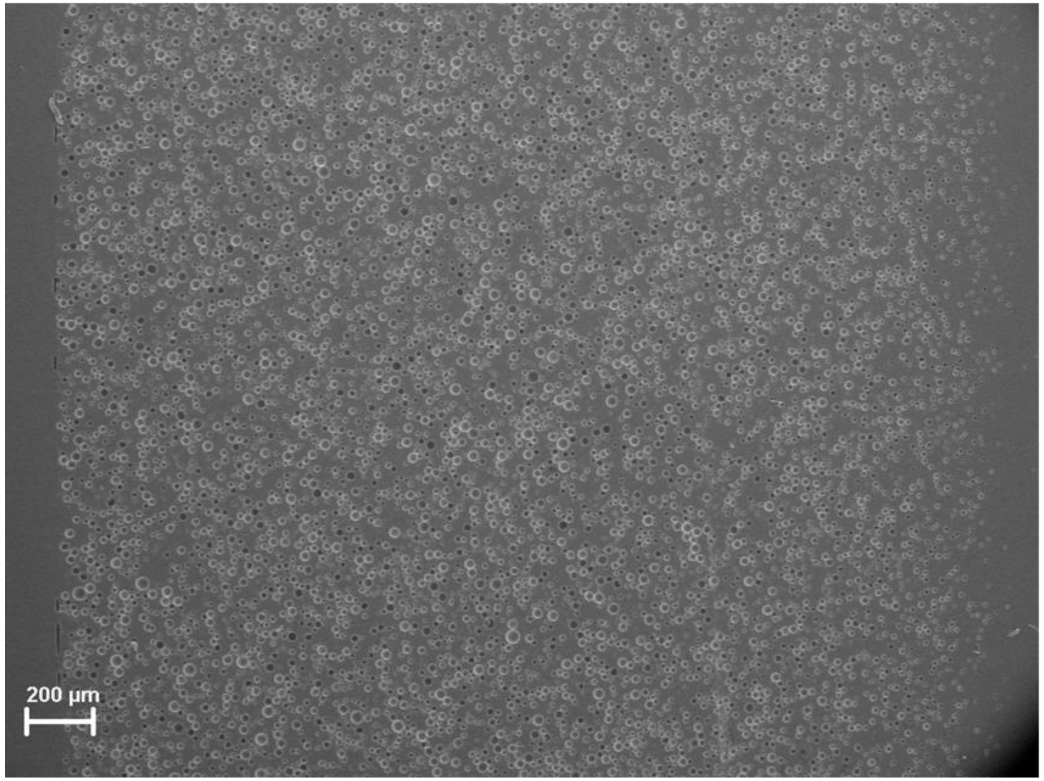
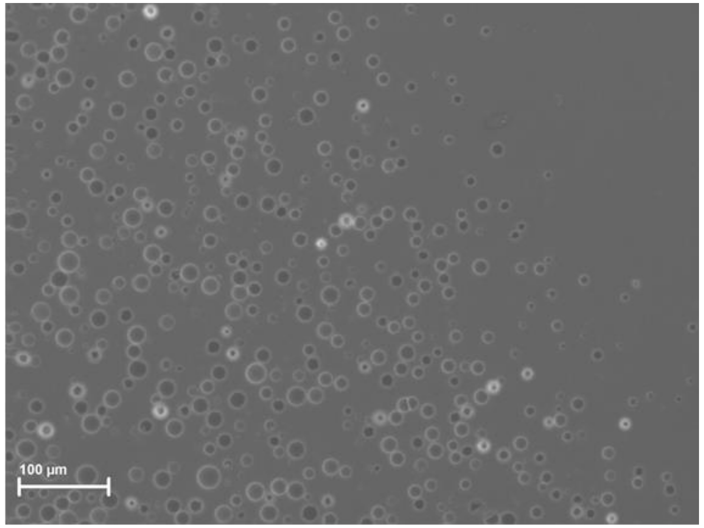
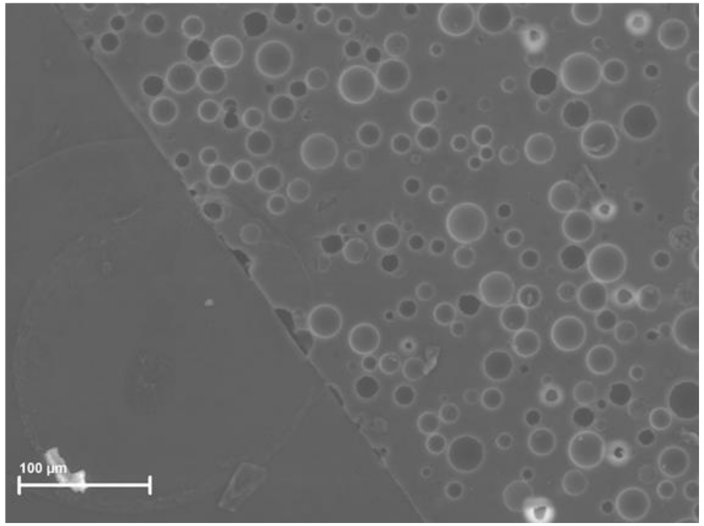
# Scanning electron microscope analysis.

SF




Hollow microspheres flows in the molds when carbon fillers are not present in the matrix.

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# Main findings.

1. Hollow microspheres can flow in the epoxy matrix before the curing process starts.

2. Carbon filler avoids this floating.

3. DGEBA is not able to guarantee a good mixing of GnPs, Carbon black and glass microsphere in high concentration.

4. A good electrical conductivity (around 1 ohm) was reached with a GnPs concentration of 4% wt., but the material presented many voids and low mechanical proprieties.

# Thank you for your attention!

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