Graphene/Epoxy Nanocomposites for Joule Heating Thermosets Curing and Repair of CFRPs

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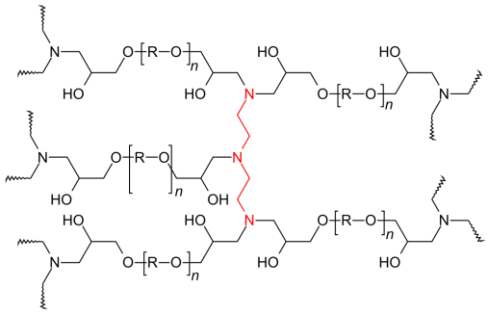
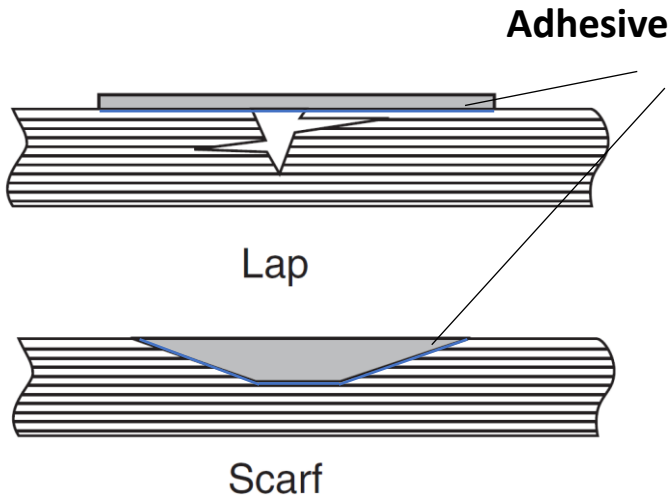
Need of out-of-autoclave curing methods

- CFRPs are used extensively in aerospace
- **Fabrication and Repair** are typically performed in an oven/autoclave

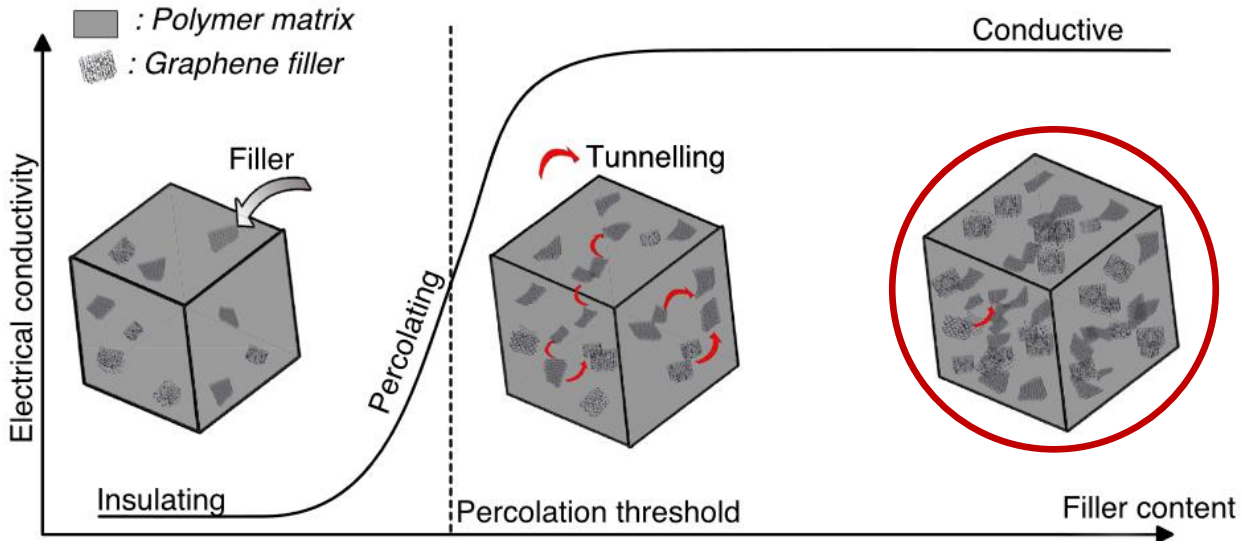




- Disadvantages typically associated with the use of oven/autoclave (size limitation, energy consumption)
- **Out-of-autoclave strategies to fabricate/repair these composites are strongly required.**

Composites Science and Engineering, 2012, 309-330

Electrically Conductive GNP/Epoxy Composites for Out-of-Autoclave Thermoset Curing through Joule Heating

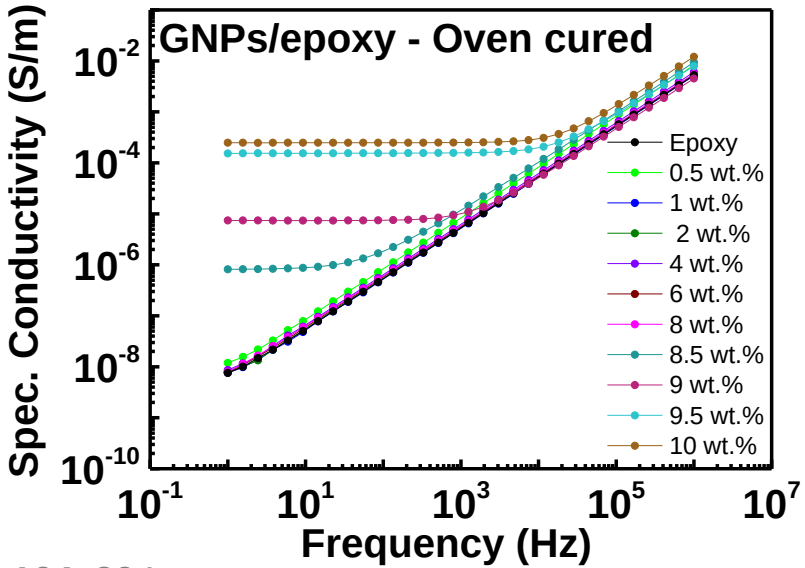


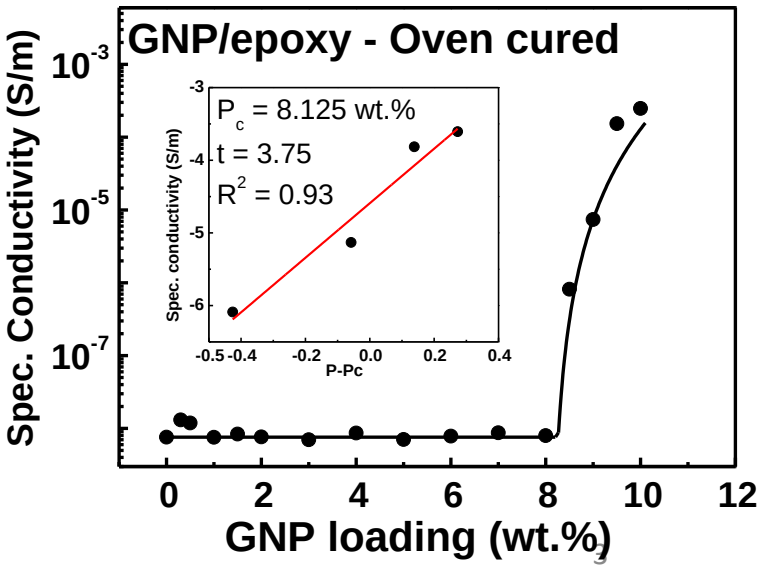
- **Percolated systems**
- To explore the potential of GNPs/Epoxy systems for **resistive thermoset curing (Joule heating)**
- **Out-of-autoclave curing** method

Traditional Oven curing method (percolation threshold)

Classic percolation theory

$$\sigma = \sigma_0 (P - P_c)^t$$





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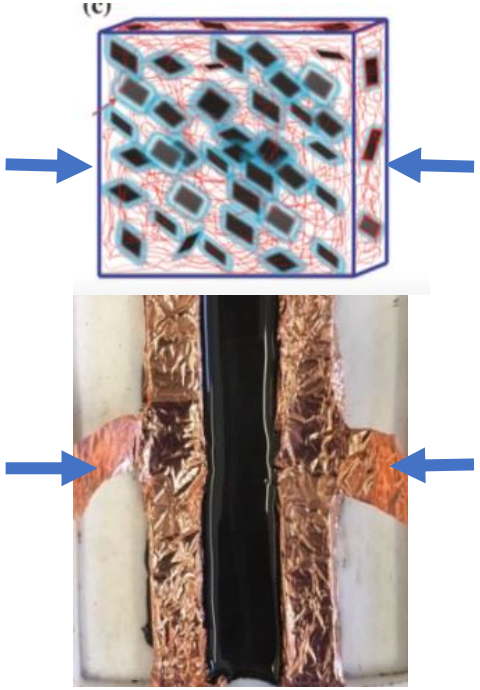
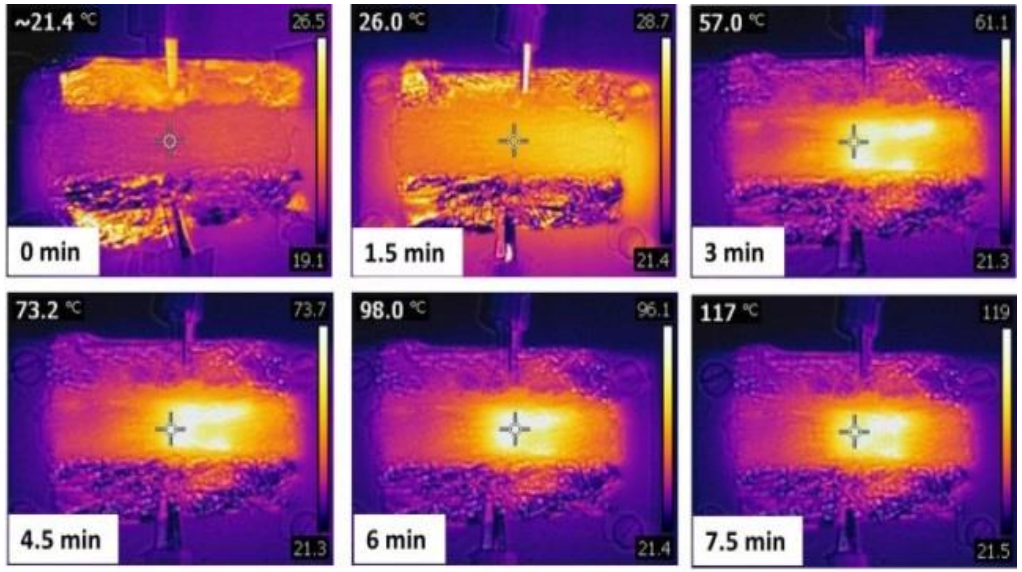
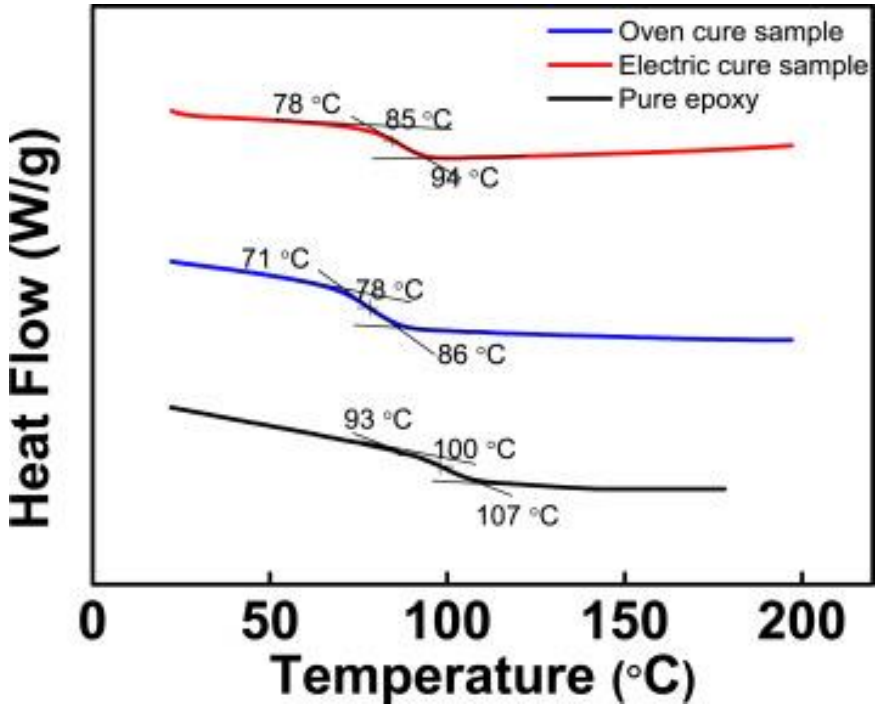
Joule Heating Induced Curing Method







Graphene flakes act as integrated **nanoheaters** when an electric current is passed through the uncured GNPs/epoxy mixture (above percolation) curing the composites **(Joule heating)**

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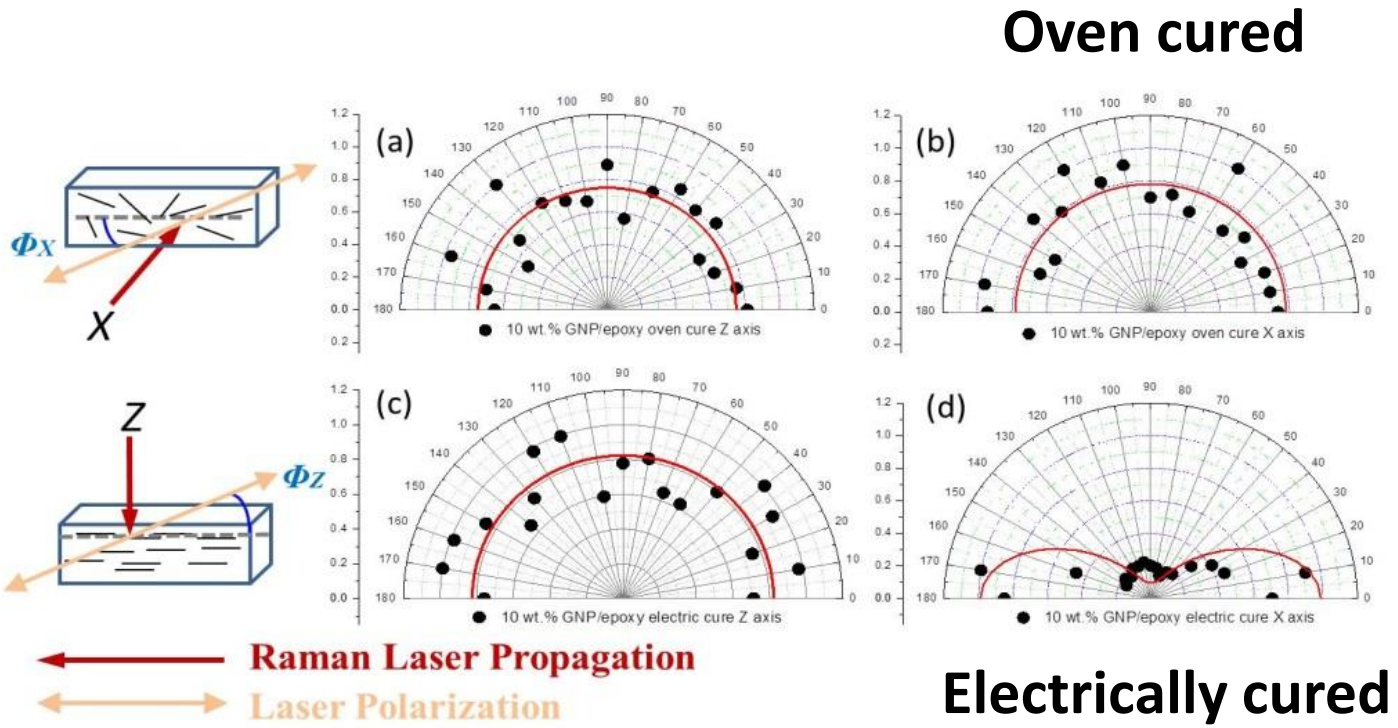
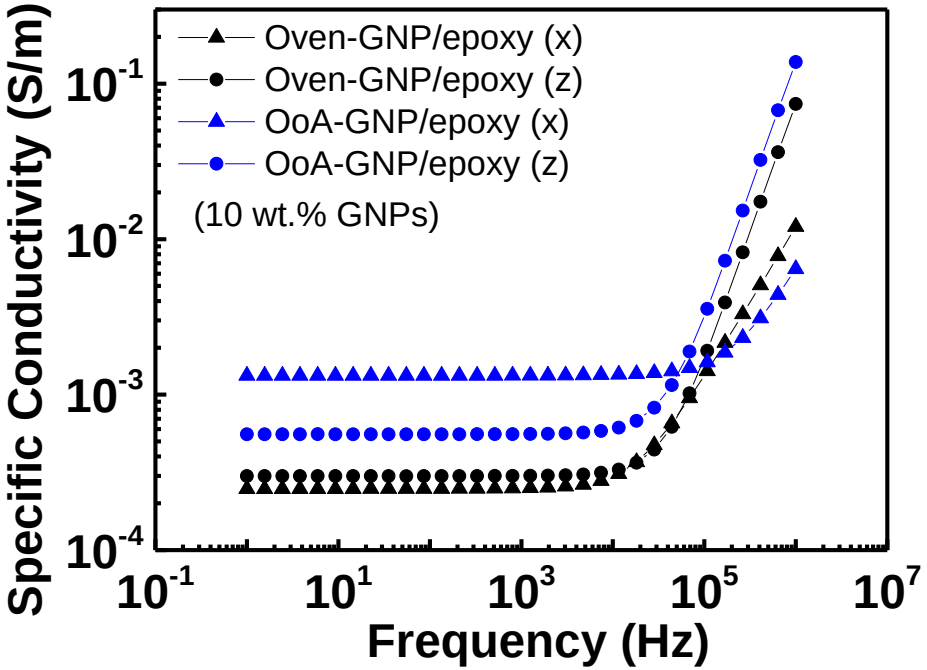
Joule Heating Induced Curing Method



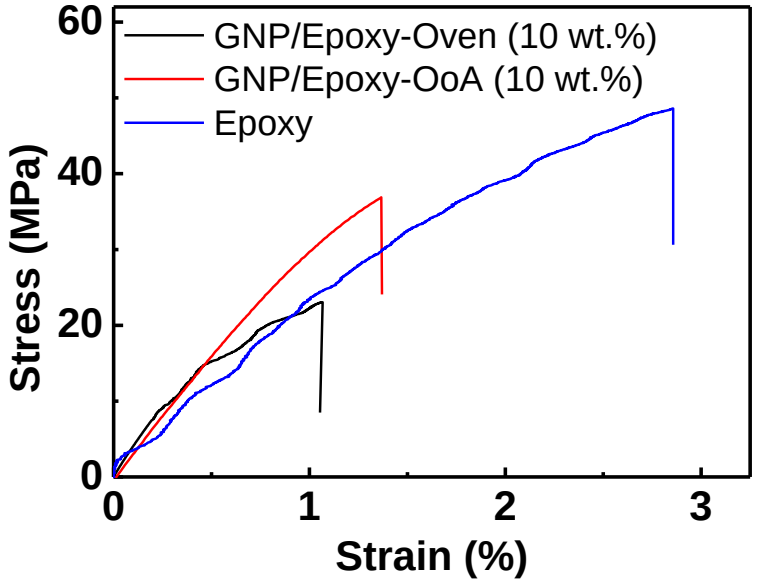


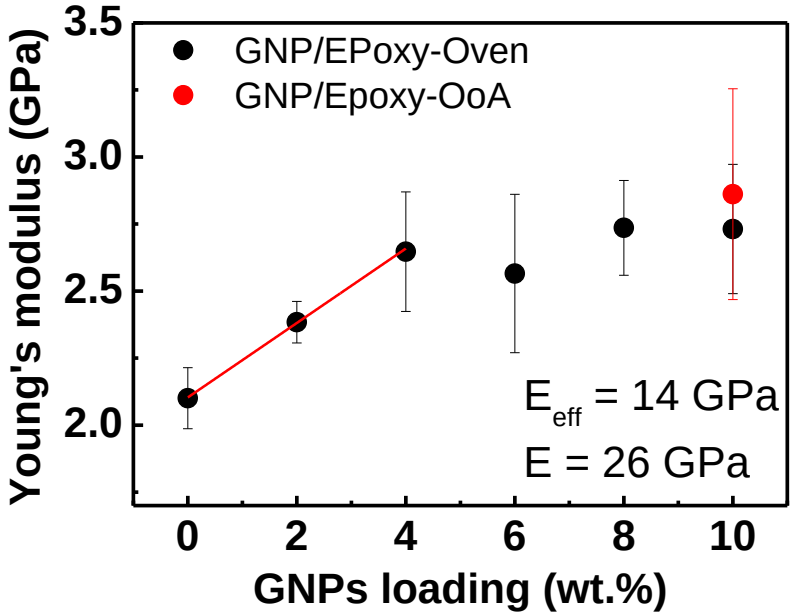
- **Orientation of GNPs promoted in the direction of the applied current**

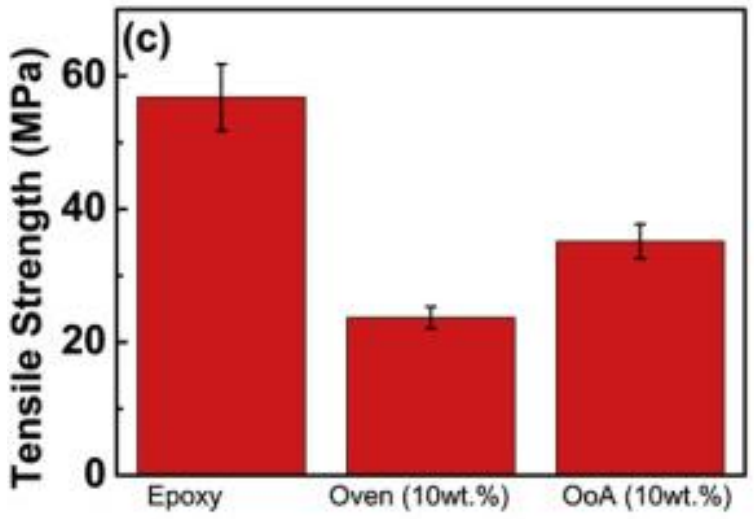
  (Polarized Raman to quantify the orientation factor).

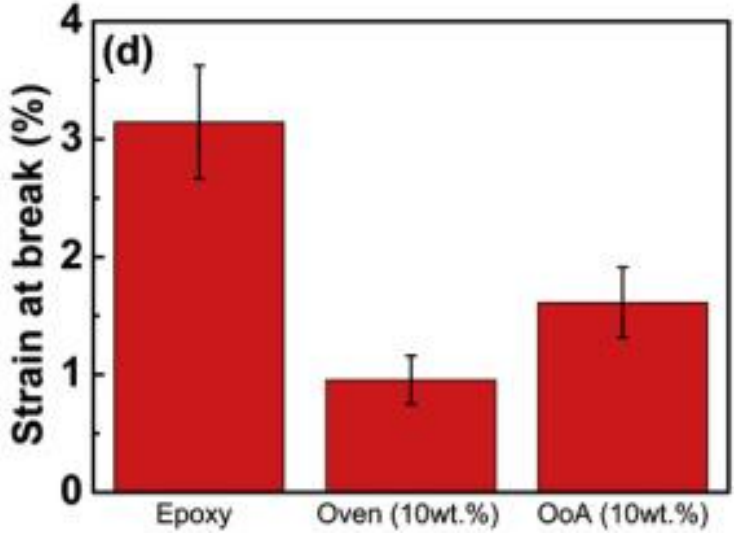
- Enhanced electrical properties found in the direction of alignment
- **Anisotropic materials**

Joule Heating Induced Curing Method









- The mechanical properties of the nanocomposite are not compromised

Potential for out-of-autoclave thermoset curing method for the aerospace sector

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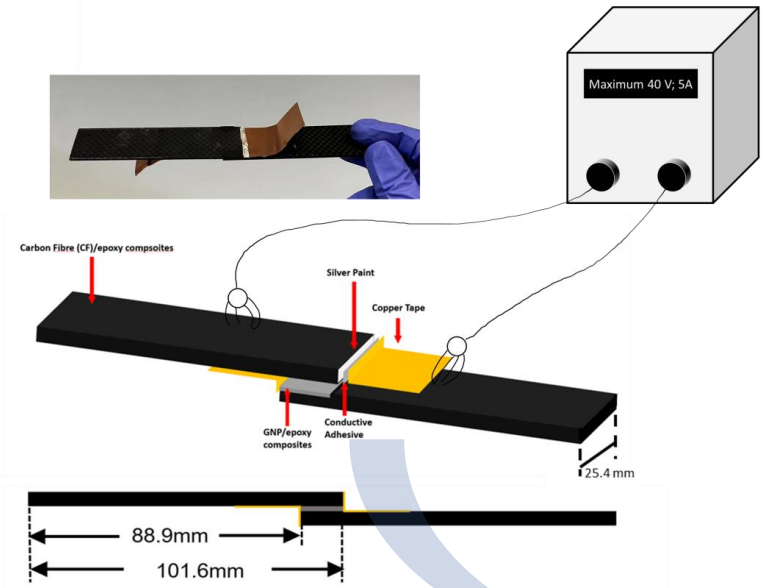
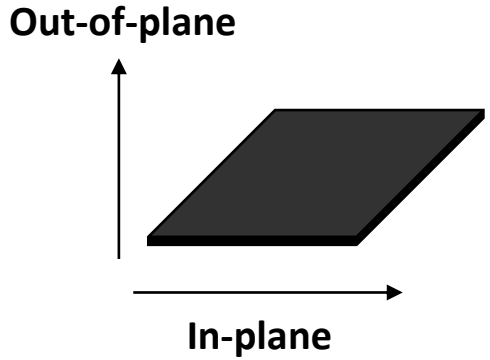
From there: CFRPs repair through Joule heat curing

Is it possible to use GNPs/epoxy composite mixtures as ‘conductive adhesives’ to repair CFRPs through Joule heating curing?



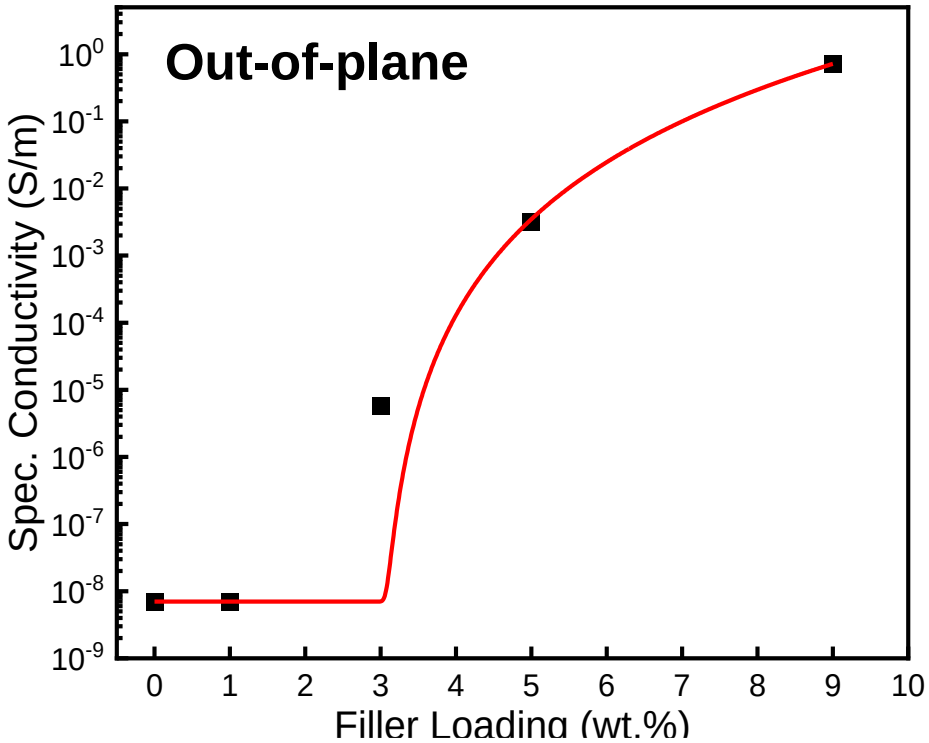


Sample Thickness: t = 0.5mm

Reducing sample thickness (from bulk to 0.5 mm films)

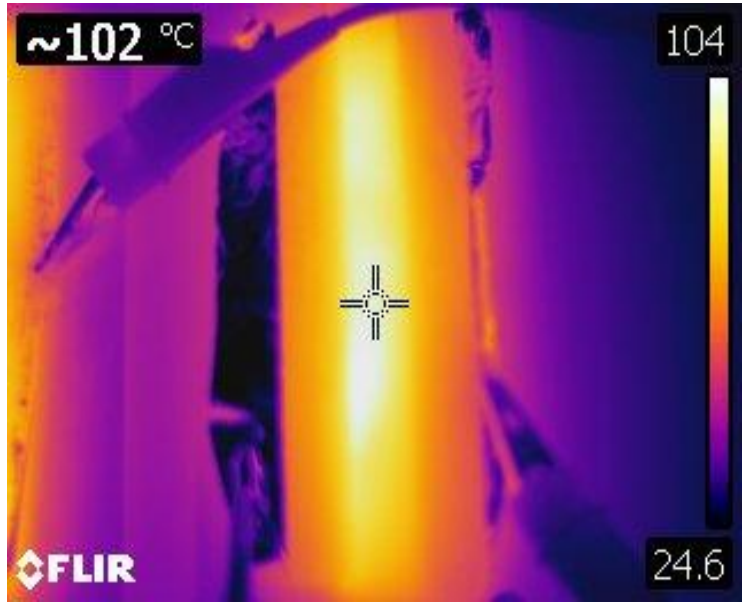
Classic percolation theory

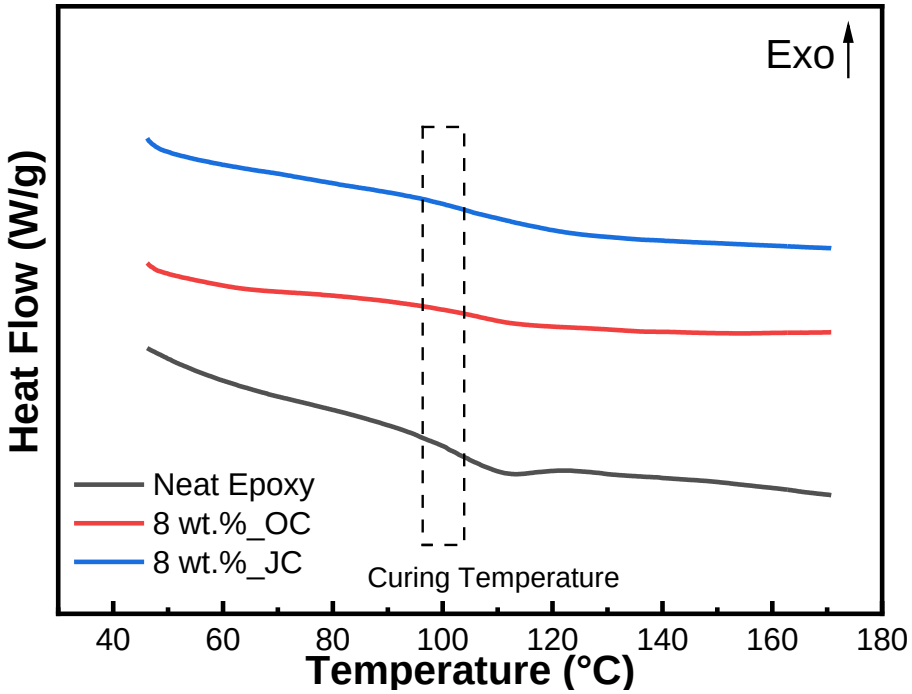
$$\sigma = \sigma_0 (P - P_c)^t$$

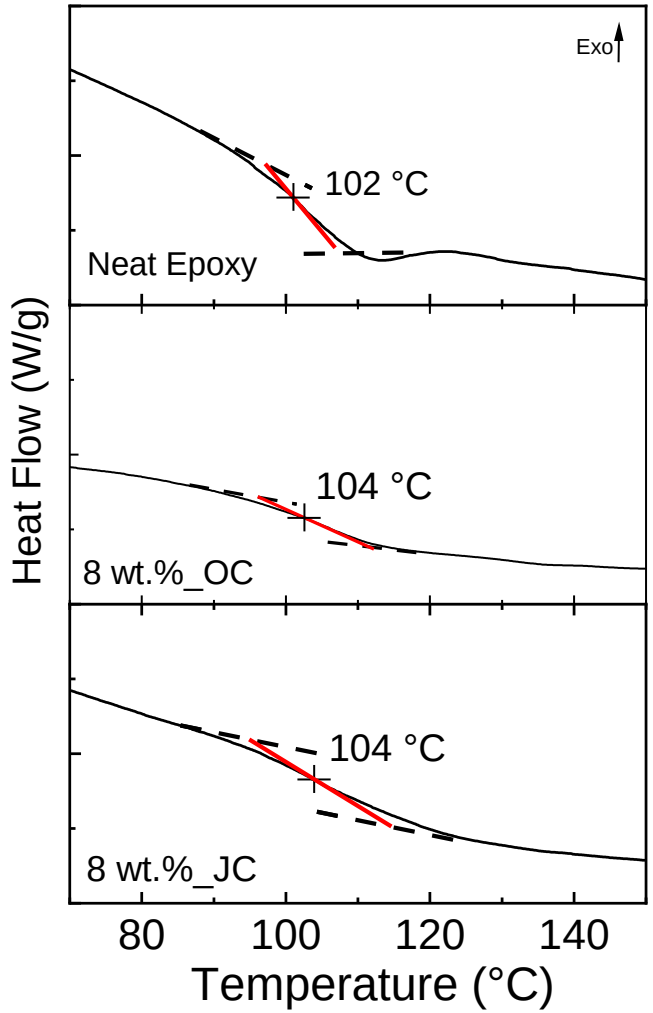


P_c = 3 wt.% (t = 4.9)

Joule heat curing of GNPs/epoxy composites films



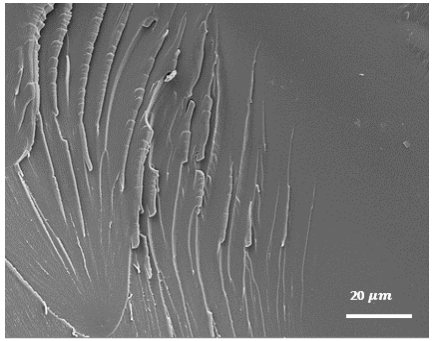




- GNPs/epoxy composite films successfully cured through Joule heating
- Similar degrees of curing achieved for oven cured and Joule heat cured samples (similar T_g revealed by DSC)

Structure of the GNPs/epoxy composites films

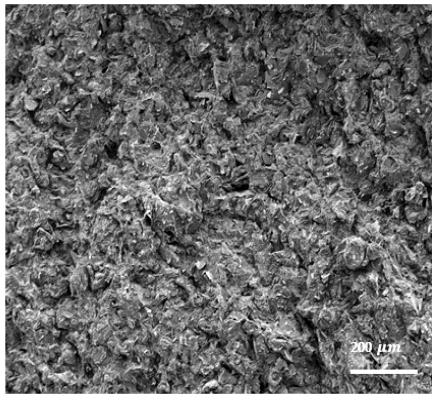
Epoxy

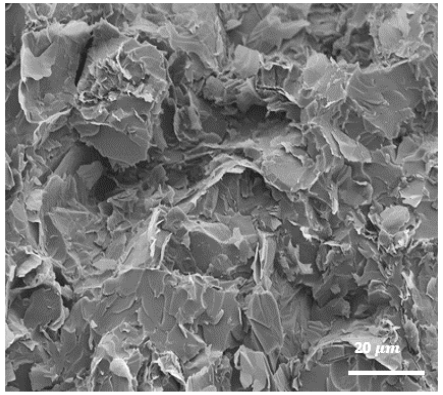


SEM:

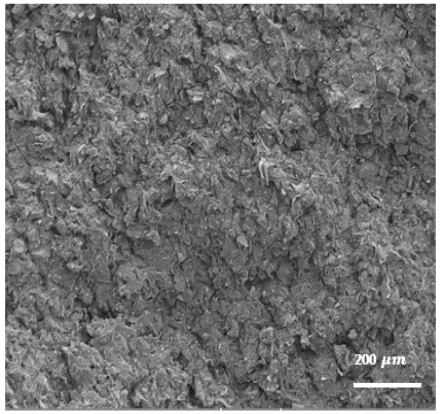
Structure of polymer and nanocomposites (OC, JC)

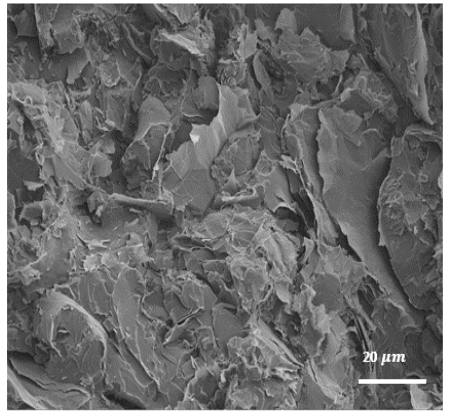
8 wt.% - OC



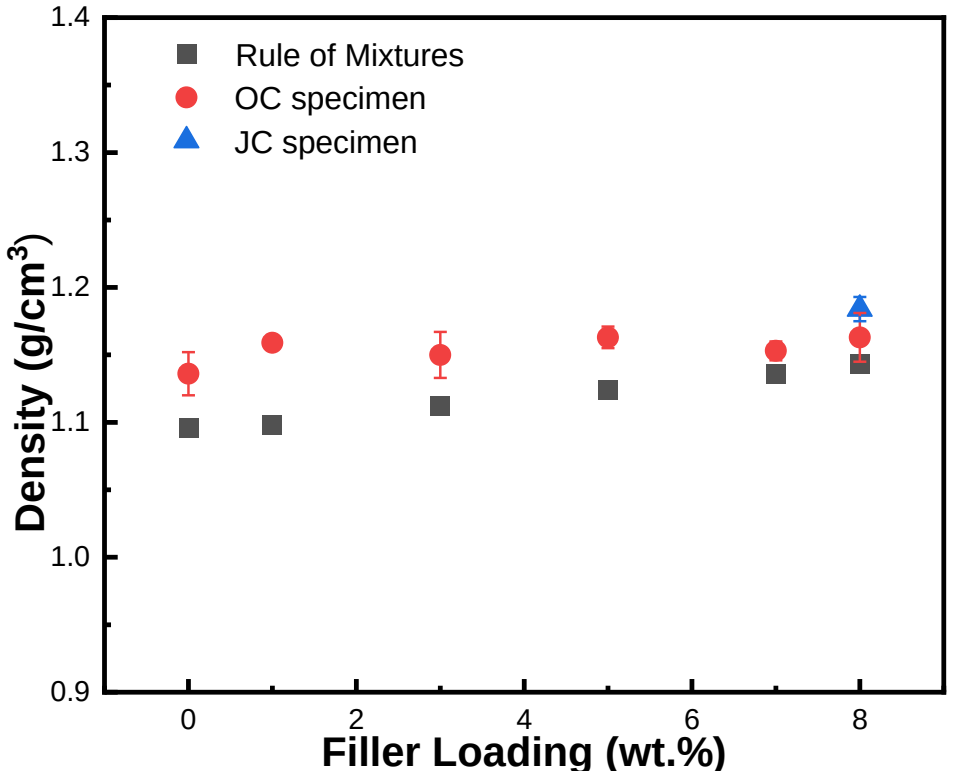


8 wt.% - JC





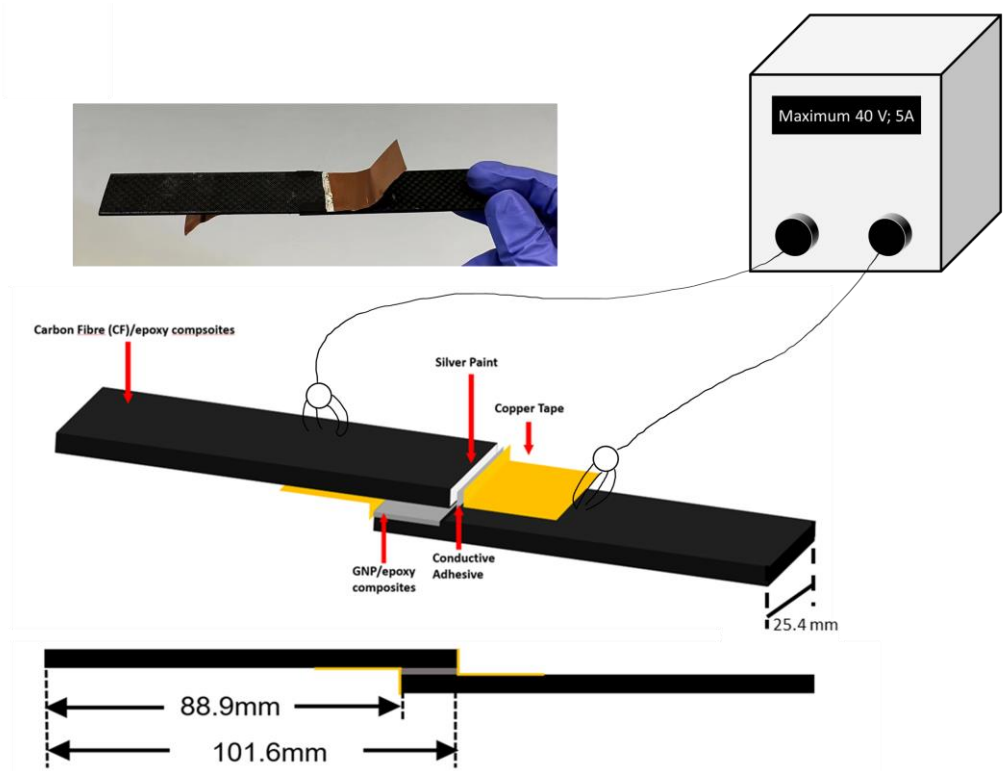
Density (Archimedes method)

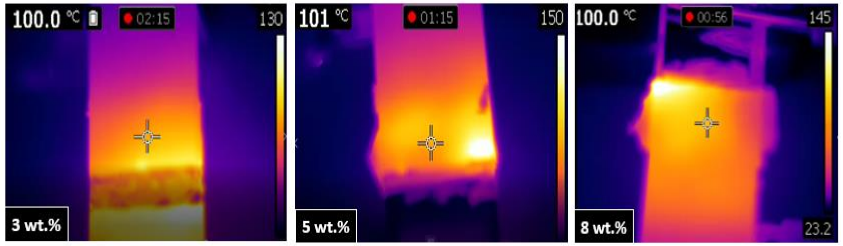


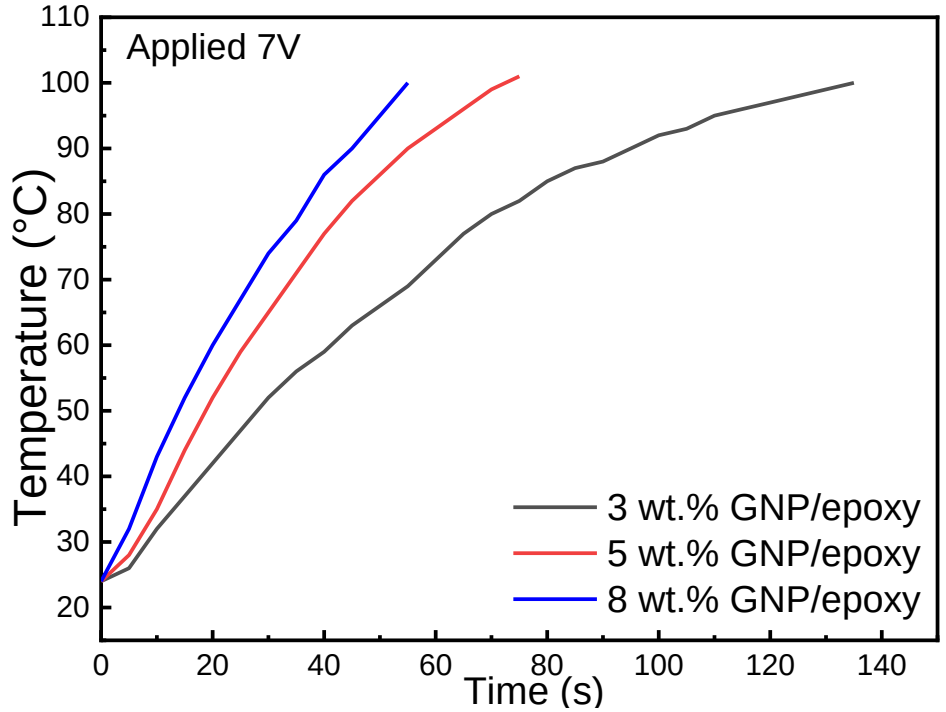
- Compact structures found for the composites independently of the curing method used
- No voids, similar densities

Repair of CFRPs through Joule heating of GNPs/epoxy

GNPs/epoxy composite mixtures as ‘conductive adhesives’ to repair CFRPs through Joule heat curing



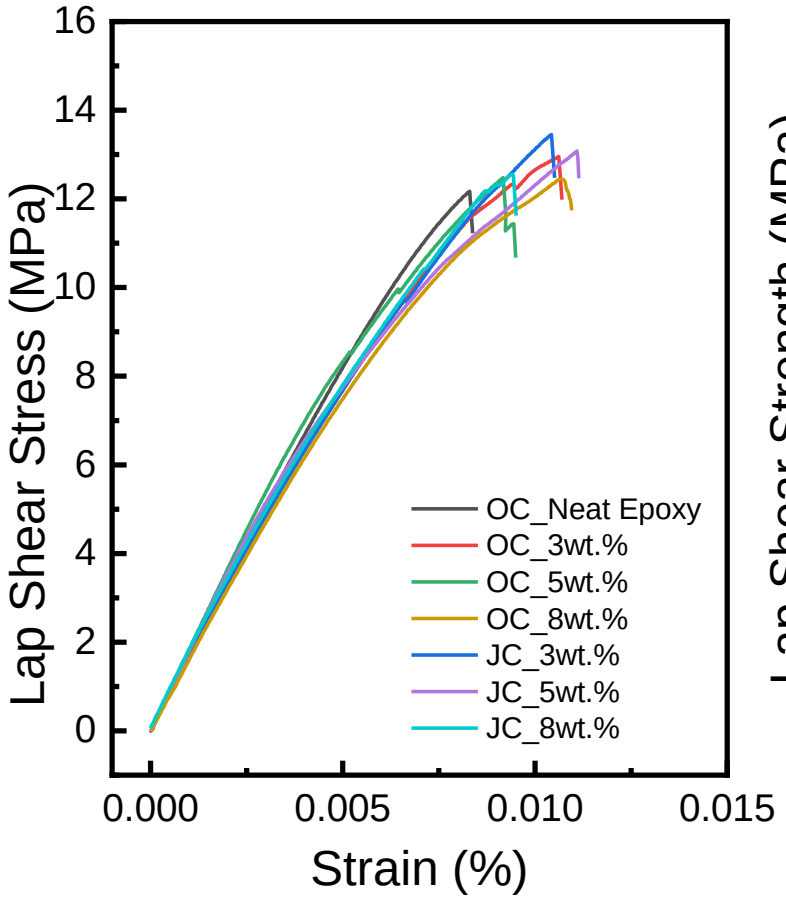


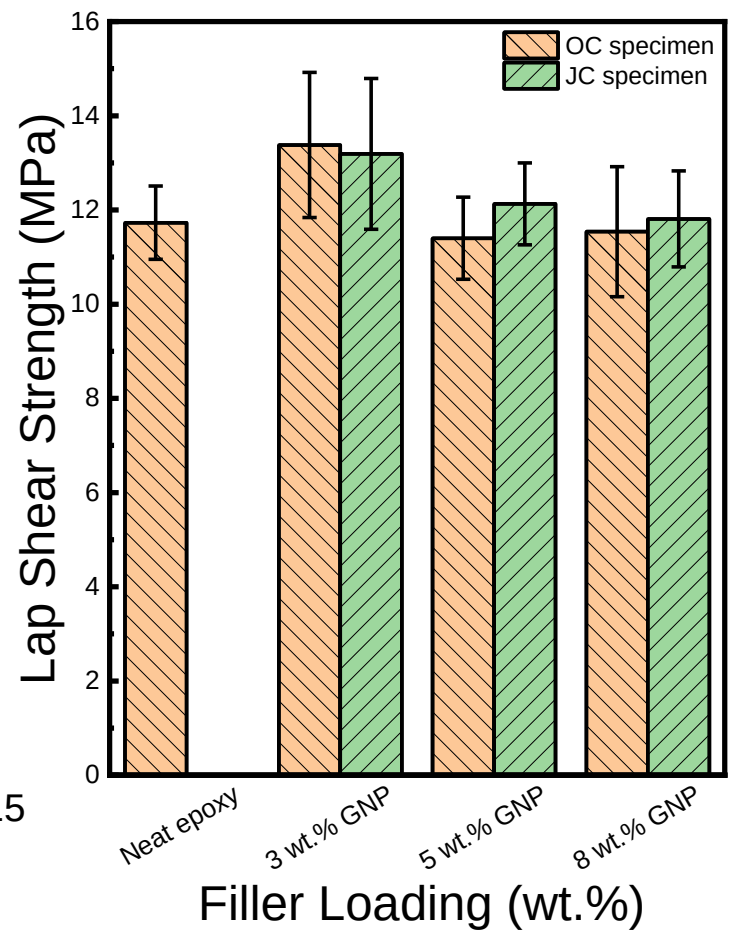


- Homogeneous distribution of the generated heat
- Influence of the GNPs loading on the heating rate (control of the curing)

Mechanical Properties (Shear Lap Test)

Single lap shear test for oven-cured and resistive-cured composites





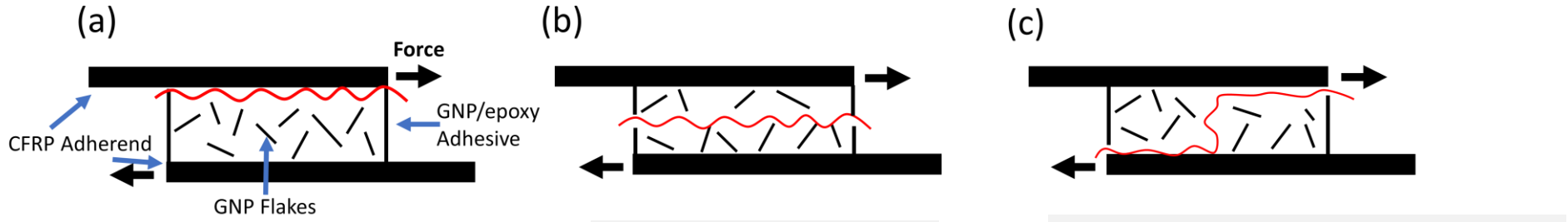
- LSS not affected by the curing method

Instron 3365, 5 kN load cell.
crosshead displacement rate: 1.3 mm/min
(ASTM D1002)

GNPs/epoxy mixtures seem effective as 'conductive adhesives' to repair CFRPs through Joule heat curing without compromising the mechanical properties.

Failure mechanism of the bonded joints

Three different types of the failure behaviour depending on where the failure happens during the test:

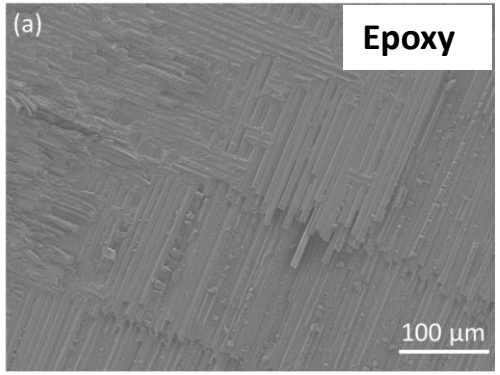


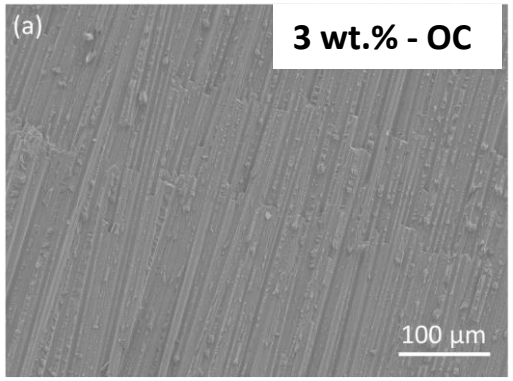
Adhesive/Interfacial Failure	Cohesive Failure	Interfacial/Cohesive Failure
The failure happens at the interface. (Weak interface between adhesive and adherend).	The failure happens within the adhesive. (Strong interface between adhesive and adherend).	Mixture of adhesive and cohesive failure. (Areas with strong/weak interface between adhesive and adherend)

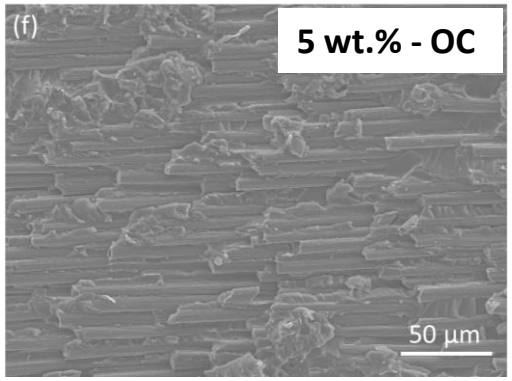
Failure mechanism of the bonded joints

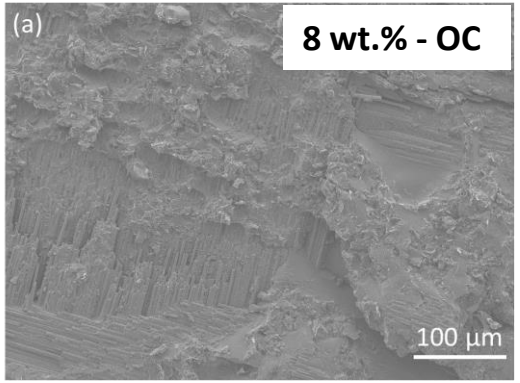
By adding GNPs: both **cohesive and interfacial mechanisms coexist**, with progressively increasing dominance of the cohesive over interfacial as the filler loading increases

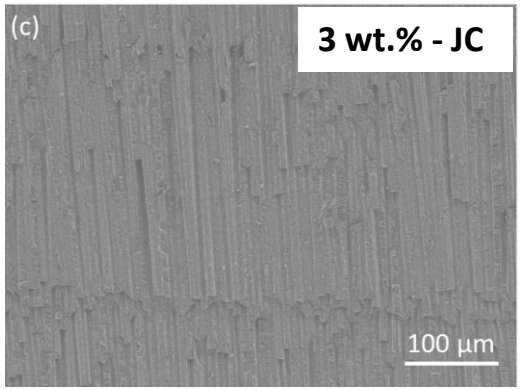
Epoxy: Interfacial failure dominates (weak adhesion)

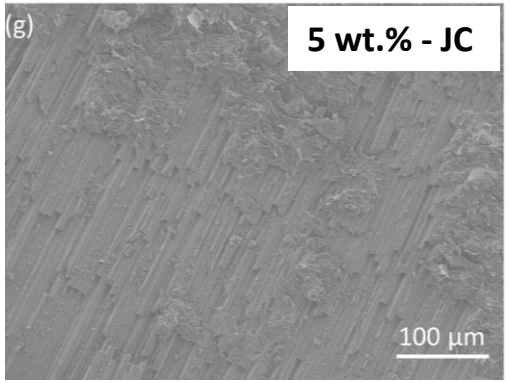


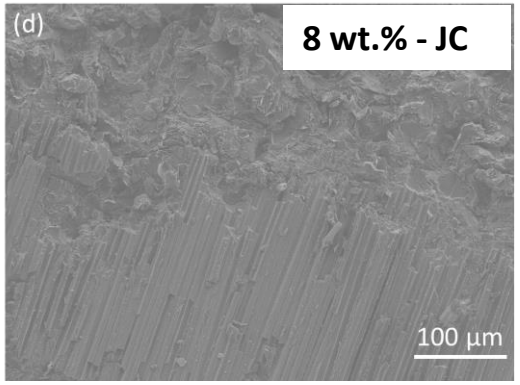












- The mechanism of failure did not seem to depend on the curing method
- Strategies to improve toughness of the nanocomposite need to be developed

Conclusions

- **GNPs/Epoxy nanocomposites have been probed to be successfully cured by Joule heating of the conductive network of flakes formed in the matrix above percolation:**
  - DSC reveals good level of curing (similar to those achieved for the oven cured samples)
  - Mechanical properties were not compromised using this curing method
  - Out-of-autoclave thermoset curing method with great potential to replace the use of the oven

- **GNPs/Epoxy mixtures as conductive adhesive to repair CFRPs through Joule heat:**
  - Lap shear tests reveal similar LSS for the same GNPs content independently of the curing method
  - Interfacial and cohesive failure mechanisms coexist, with increasing dominance of the cohesive as the GNPs loading increases
  - Promising out-of-autoclave method for CFRPs repair
  - Strategies to improve the toughness of the nanocomposite (adhesive) need to be developed

Acknowledgements

GRAPHENE FLAGSHIP

EPSRC
Pioneering research and skills

ROYAL ACADEMY OF ENGINEERING

Morgan Advanced Materials

Thank you for your attention