2.0 rCF based composite materials: manufacturing processes and mechanical properties

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Involved in these works
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Maxime Boulanghien (PhD, ICA, 2015)
Sabrine Jlassi (PhD, ICA, 2019)
Serge Da Silva (ARC)
IMT: Institut Mines-Télécom

7 engineering and 1 Business School

13,700 students
- 1,590 PhD
- 1,080 manager students

31% foreign students

1,650 Prof and Ass-Prof

4,885 graduated per year
10% by apprenticeship

64.2 M€ research turnover

1,900 rank A publications/year

45 industrial chairs

74 start-up created per year in school incubators

93% survival after 3 years
Joint research lab on Carbon fiber composite recycling

- **ICA - Albi**
  - UMR CNRS 5312

- **RAPSODEE**
  - UMR CNRS 5302

- **CGI**
  - Industrial engineering

- **Institut Clément Ader**
  - Research topic: Materials and structure Mechanics, Mechanical systems mainly for aeronautics and space
  - Staff ≈ 250 people
    - Permanent staff:
      - 85 Prof, ass. Prof(EC)
      - 34 Ing, tech and administrative
    - Non permanent staff:
      - 35 post doc, tech, research engineers
      - 90 PhD’s
  - Composite material and structure group
    - 75 people (40 PhD’s and post docs)
    - Biggest team in France working on composite materials and structures in a same lab
Properties of rCF: which test to state on the properties of recycled fibres

Properties of injected rCF-PA6.6 composites

Non-woven rCF 2.0 thermoplastic composite
  - Is fibre sorting required before recycling
  - What is the optimum length of fibres for Non-woven manufacturing
  - Effect of sizing or not (vrCF (virgin recycled Carbon fibres versus rCF)
  - Optimum non-woven architecture for thermo-compression manufacturing

Non-woven rCF 2.0 infused epoxy composites
  - Effect of stitching
  - Property comparison with rCF/PA6 and quadriaxial Glass fibre epoxy material
Recycled fiber mechanical properties

- Three test methodologies available
  - Impregnated tow test: test used by the fibre manufacturer (test affected by fibres, epoxy resin, and sizing)
  - Single fibre test: most used test (between 25 to 50 individual fibres)
  - Bundle test
rCF fiber rupture stresses

Hextow AS4C → Epoxy resin infusion → Composite sheets → Steam water Thermolysis

REFERENCE

400°C
95 % resin elimination

500°C
99 % resin elimination

rCF
Single fibre tensile test results (40 filaments)

Normal probability density function and frequency histogram of failure events (FR500)

Statistical analysis (normal distribution)

<table>
<thead>
<tr>
<th>Fibre samples</th>
<th>Mean of tensile strength (MPa)</th>
<th>Standard deviation (MPa)</th>
<th>95% confidence interval (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>VF</td>
<td>3776</td>
<td>547</td>
<td>146</td>
</tr>
<tr>
<td>RF400</td>
<td>3272</td>
<td>672</td>
<td>179</td>
</tr>
<tr>
<td>RF500</td>
<td>3610</td>
<td>540</td>
<td>144</td>
</tr>
</tbody>
</table>
Bundle Tensile Test (BTT)

Fibre bundle
Extensometer
Tensile grip

Slope of the tensile load-strain curve

Strain

\[ F(\varepsilon) = N_0 \cdot A_f \cdot E_f \cdot \varepsilon \cdot [1 - P(\varepsilon)] = \mathcal{R}_0 \varepsilon \cdot [1 - P(\varepsilon)] \]

M. Boulanghien & al, Advances in Materials Science and Engineering, 2018
Fibre Bundle Tensile Test results

- Mean strength 95% confidence interval drastically reduced with BTT tests
- Low dispersion between BTT results

Manufacturer data:
- 4327 Mpa
- Higher than SFTT and BTT
- Important to compare values obtained with the same methods

M. Boulanghien & al, Advances in Materials Science and Engineering, 2018
2.0 rCF /PA6.6 pellet injection

Fiber length:
1 to 2 cm

- vCF
- rCF (FR500)

Polyamide 6,6 (Latamid 66)

Temperature:
270 °C

Extrusion – IPREM EPCP Pau

INJECTION ICA - Albi

Temperature:
290 °C

Commercial CF reinforced Pelets

Fibre not known

Thermoplastic composite materials reinforced with 10% of carbon fiber Vf

Length: 0.3 mm
2.0 rCF /PA6.6 pellet injection

- rCF/PA6.6 injected material properties are as high as commercial grades
- rCF injected properties are as close to the vCF ones
2.0 rCF non-woven composites manufacturing

Nonwoven processing: carding/needle punching

rCF composites manufactured by thermocompression film stacking or comingled non-woven
Impact of fibre grade and fibre length on non-woven vrCF PA6 composites

- Design of experiment approach to respond to question if fibre sorting is important or not before recycling

- 3 fibre grades
- 3 fibre lengths: 50mm, 80mm and 110mm
- PA6 matrix

S. Jlassi & al, ICCM18, Athens, 2018
The higher the fibre properties, the higher the composite properties: C1(T300), C2(T700), C3 (IM7)

Material not isotropic: Cross Direction > Machine Direction

Rupture stress is significantly affected if T300 (lowest fibre rupture stress)
Vf is > than 50% (C1,C4,C6,C8)

Detailed analysis (see PhD) shows that 80mm is the optimal length
2.0 Non-woven PA6 composites

Comparison between:

vrCF and rCF,

100% CF (200g/m²) or comingled PA6 (510g/m²)

- Carding of rCF damages fibres → loss of fibres → lower areal mass of non-woven
- Higher loss when carding comingled non-woven due to increase of carding speed
2.0 Non-woven PA6 composites

- Comingling is favorable for composite processing: higher properties due to better microstructure
- rCF non-woven are more isotropic than vrCF ones
- CD direction properties drop for rCF composites

- Comingled non-woven are well adapted for thermo-compression forming
<table>
<thead>
<tr>
<th>Non woven vrCF (T700) (STFI)</th>
<th>Stiched non woven vrCF (STFI)</th>
<th>Glass quadriax (Saertex)</th>
</tr>
</thead>
<tbody>
<tr>
<td>225 g/m²</td>
<td>210 g/m²</td>
<td>990 g/m²</td>
</tr>
</tbody>
</table>
Non-woven: Fibrous architecture compressibility

- Maximum possible fibre volume fraction depends on composite manufacturing process and fibrous architecture

Compressibility test results

- Non-woven vrCF
- Stitched non-woven vrCF
- Glass quadriaxial NCF

Thermocompression pressure

Infusion pressure
Non-woven Infusion process

- Vf after infusion is close to that expected from compressibility tests during first load increase
For rCF materials, there is an increase in stiffness with respect to fibre volume fraction, less evident for rupture stresses.
2.0 rCF epoxy infused materials: specific properties

- rCF infused materials are good candidates for replacement of glass reinforced materials in naval or automotive applications even if they have a lower fibre volume fraction.
Conclusions

- rCF fibre mechanical property level statement requires tests on a great number of filaments: bundle tensile tests are best suited.

- 2.0 injected rCF/PA6.6 materials are at the same level of properties as commercial ones. No influence of fibre desizing induced by steam-thermolysis.

- Non-woven rCF materials
  - Optimum rCF length of 80 mm for non-woven carding
  - Mixing of fibres: significant impact only if volume fraction of the less performance fibre is higher than 50%
  - Commingled rCF/PA6 non-woven well adapted to thermocompression manufacturing

- Non-woven rCF epoxy infused materials are candidates for replacement of quadriaxial NCF glass fibre composites used in naval or automotive applications.
Thank you for your attention

Team involved in these works:

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Maxime Boulanghien (PhD, ICA, 2015)
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