Use Case 2: Thermal Recycling of Long Fibers

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Abstract This chapter describes the industrial demonstration of the reuse of recycled fibers obtained by a thermal process. Four demonstrators are described in which both recycled carbon fibers and recycled glass fibers have been incorporated into different matrices. The automotive sector proposes 3 demo cases (Pedal Bracket, Front-end carrier and Cowl top support) with demanding mechanical and thermal requirements. These components were manufactured by injection molding with thermoplastic matrices. The construction sector proposes 1 demo case (Light transmitting single skin profiled sheet.) with mechanical and light transmittance requirements that was manufactured by continuous lamination. It is demonstrated that the incorporation of recycled fiber for these applications is technically possible, fulfilling the requirements demanded by each sector.

Keywords Thermal recycling process · Pyrolysis · Recycled glass fiber · Recycled carbon fibers

1 Introduction

Glass fiber (GF) and carbon fiber (CF) reinforced polymer composites are widely used in sectors like transport (automotive, aircraft, railway, boats) and construction

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(building and infrastructures, plants, wind turbines), because of their attractive properties such as light weight, good corrosion resistance, high strength, high fatigue resistance and good dimensional stability [1]. Nevertheless, there are some disadvantages in the use of composite materials which include the high raw material and fabrication cost, susceptibility to impact damage and a greater difficulty in repairing a recycling.

Between the different available reinforcements, GF is the most used in several industrial applications, mainly due to its low cost but also because it's good thermal and electrical insulation. On the other hand, CF is characterized by better mechanical properties combined with a lower density, which makes possible to manufacture lightweight and very robust parts. The biggest disadvantage of CF is the raw material and manufacturing cost, which is much higher than GF.

In the case of the automotive sector, apart from using steel or aluminum to produce different parts, the main reinforcement used combined with polymers is GF. It is well known that in automotive sector, cost and weight reduction are imperative. Although GF is cheaper than CF, there is a growing interest for replacing virgin GF with recycled carbon fiber (rCF) in some automotive applications, due to its interesting properties mentioned above.

In the construction sector, some lightweight applications exist as high dimension roofs, where the low weight is an important requirement together with appropriate mechanical properties. In some applications, translucency is also important. Therefore, composite materials in this sector are widely used. The use of these laminates is especially high for industrial applications like pavilions, sports center, shelters, car park canopies, etc. These products at their end on life produce a high amount of waste, which is interesting to reuse; if possible, in the same application thus, closing the loop in a circular economy perspective [2].

The use cases which will be shown in this chapter, will be based on fibers recycled within the FiberEUse project. The recycling process used from the waste to the raw material that will be used industrially, goes through several intermediate stages of transformation. This value chain is essential to ensure the quality of the materials that will reach the industrial process, both in automotive and construction sectors. The key technologies exploited for the realization of demonstrators are:

- Pyrolysis or thermal recycling (Chap. 5): it consists of obtaining liberated clean glass or carbon fibers (rGF or rCF) by means of a thermal treatment which eliminates completely the matrix and other organic components of the composite residue.
- Re-sizing (Chap. 7): it consists of applying a chemical treatment on the surface of the recycled fibers, to increase the fiber-matrix interfacial adhesion for future applications.
- Compounding (Chap. 7): it consists of preparing a semi-finished product made of fibers and thermoplastic matrix in pellet form through an extrusion process.

In the following tables the demo-cases and the materials used for their realization are shown. Table 1 shows the materials used for automotive demo cases and that have

Table 1 Materials used for automotive demo cases

Partner	Demo case	Matrix	Fiber
BATZ	Pedal bracket	PA6	rCF short
	Front end	PP	rCF strand
MAIER	Cowl top support	PP	rGF short

PA6 Polyamide 6, *PP* Polypropylene

Table 2 Scheme of the materials used for construction demo case

Partner	Demo case	Matrix	Fiber
RIVIERASCA	Light transmitting single skin profiled GFRP sheet	UP	rGF mat

GFRP Glass fiber reinforced plastic, *UP* Unsaturated polyester

been previously transformed by a compounding process to obtain a suitable pellet for industrial injection molding process.

Table 2 shows the materials used for the construction demo case and which fibers have been previously processed to obtain a suitable mat for the industrial laminating process.

2 Definition of Demo Cases

The demo cases based on thermally liberated recycled fibers are characterized by demanding requirements, both mechanical and aesthetical, affected by the quality of the fiber. The recycled fiber must be clean and resized in order to be compatible with the new matrix, providing good properties [3, 4]. Automotive OEMs (Original Equipment Manufacturers) are looking for sustainable plastic materials (so called "green plastics") to decrease the carbon footprint during the production of vehicles.

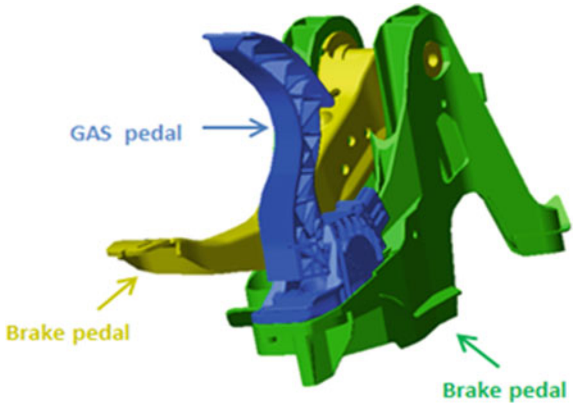
From the automotive sector parts manufactured by classic thermoplastic injection process or Injection Moulding Compound (IMC) process have been selected, based on the use of thermoplastics and fiber pellets. In the case of constructions, the laminating process will require a thermoset matrix which is deposited on the GF mat.

In this project, the challenge is to introduce rGF and rCF in structural components. The target output demonstrators are going to be a Pedal bracket, Front-end carrier, Cowl top support and industrial roof.

2.1 Demo Cases 4.1 & 4.2: Pedal Bracket (A) and Front-End Carrier (B)

The demo-cases selected by BATZ to work with rCF reinforcements are:

Fig. 1 Pedal bracket



A. The **pedal bracket** is a component in which different pedals are assembled (mainly gas and brake pedal). The pedal bracket is a security part with high structural requirements, both stiffness and strength due to driving conditions and emergency issues. The lightweight solutions are made of reinforced thermoplastic but there are also made of metal (Fig. 1).

B. The **front-end carrier** is the frontal structure of the vehicle where the front-end module system relied on. Different components such highlights, radiator, latches, cross members, etc. are assembled on it. The front-end carrier has to support different loads due to their weight as well as different usage scenarios. Adding to that, the front-end carrier contributes to the torsion performance of the car. Due to the high requirements that this kind of components have to withstand, the full plastic variants are made of high percentages of GF reinforcement (up to 40–50%) (Fig. 2).

2.2 Demo Case 5: Cowl Top Support

The demo-case selected by MAIER to implement rGF is a cowl top support. The cowl over is the part which is positioned between the windshield and the front hood, while the Cowl over support is the part that gives structure to the part. In Fig. 3 both components can be seen. The functionality of this part is to stand stiff and integer at static conditions, and break after absorbing a certain amount of energy at dynamic conditions. This part is produced by injection moulding process.

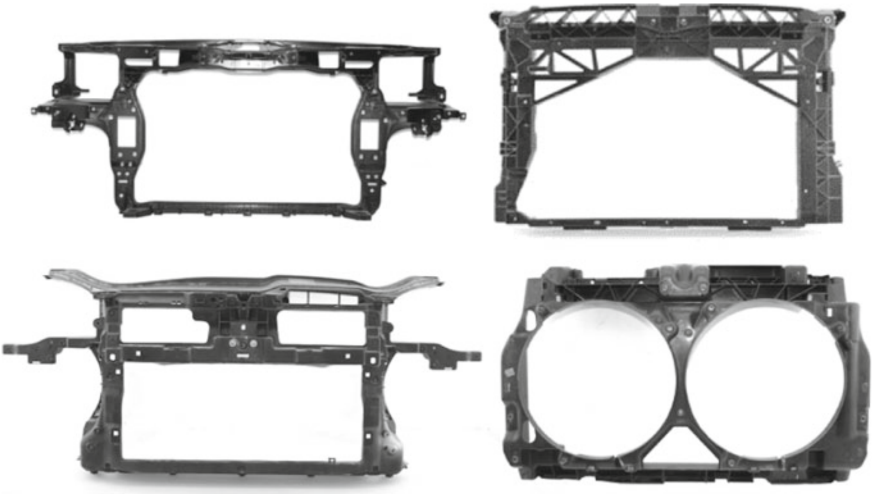


Fig. 2 Front-end carrier examples [5]

Fig. 3 Cowl over (left) and cowl over support (right)

2.3 Demo Case 6: Light Transmitting Single Skin Profiled GFRP Sheet

The Rivierasca's demo-case is a roof. It has to cover up any type of building but also to fit some requirements like, for example, a good acoustic or thermal insulation, good fire resistance or light transmission to indoor environments [6]. So, in this case, a light transmitting single skin profiled GFRP sheet has been developed (Fig. 4).

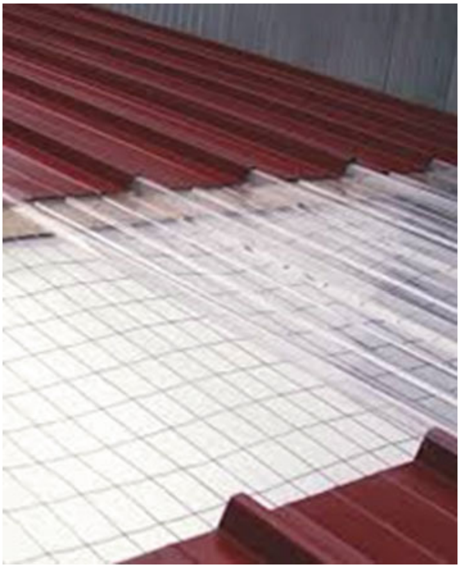


Fig. 4 Light transmitting single skin profiled GFRP

3 Pedal Bracket and Front-End Carrier Demo Cases

3.1 Pedal Bracket

Materials

The objective of this demo case is to replace a pedal bracket currently manufactured with PA6 + 40% of short GF by a new design based on PA6 + 20% of rCF (PArCF20). The pedal bracket must support the mechanical requirements defined for its use with the new material.

Manufacturing process

The pedal bracket demonstrator is processed with a conventional injection machine using pellets of PArCF20 developed in the FiberEUse project. The machine used is the same than used for usual GF reinforced plastics. The injection process is similar for both materials, but some parameters, such as melting temperature and injection velocity had to be adapted to the rheological characteristics of the new material (Figs. 5 and 6).

After its geometrical verification, the brake and gas pedal are assembled in the bracket for validation test (Fig. 7).

Validation test

In the validation test, after an aging pre-treatment, the part is subjected to different loads applied in different areas of the brake and gas pedal. The applied load

Fig. 5 Raw material (left) and injection machine (right)

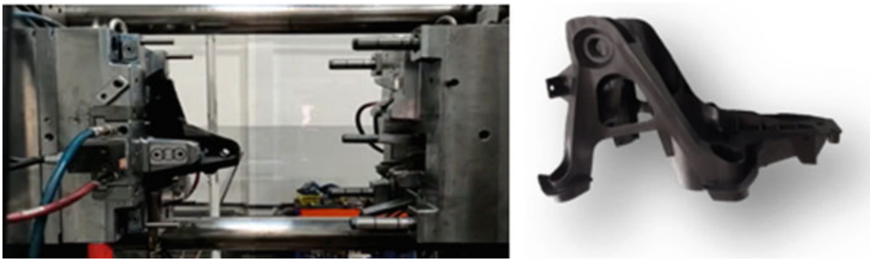


Fig. 6 Pedal mould (left) and prototype manufactured with PArCF20 (right)

Fig. 7 Pedal bracket prototype ready for validation test

must produce a component deformation less than a vehicle manufacturer defined requirement. In this case the component is validate for its use.

One of the more restrictive requirements is to withstand loads up to 3000 N, applied in the brake without any break exposed at extreme temperatures (80 °C and −35 °C). The Fig. 8 shows the test set-up. The new bracket manufactured with recycled fibers fulfils the requirements without suffering any damage. Table 3 shows the results of the validation test where the load values supported by the part are shown, demonstrating the technical viability of this prototype manufactured with rCF.

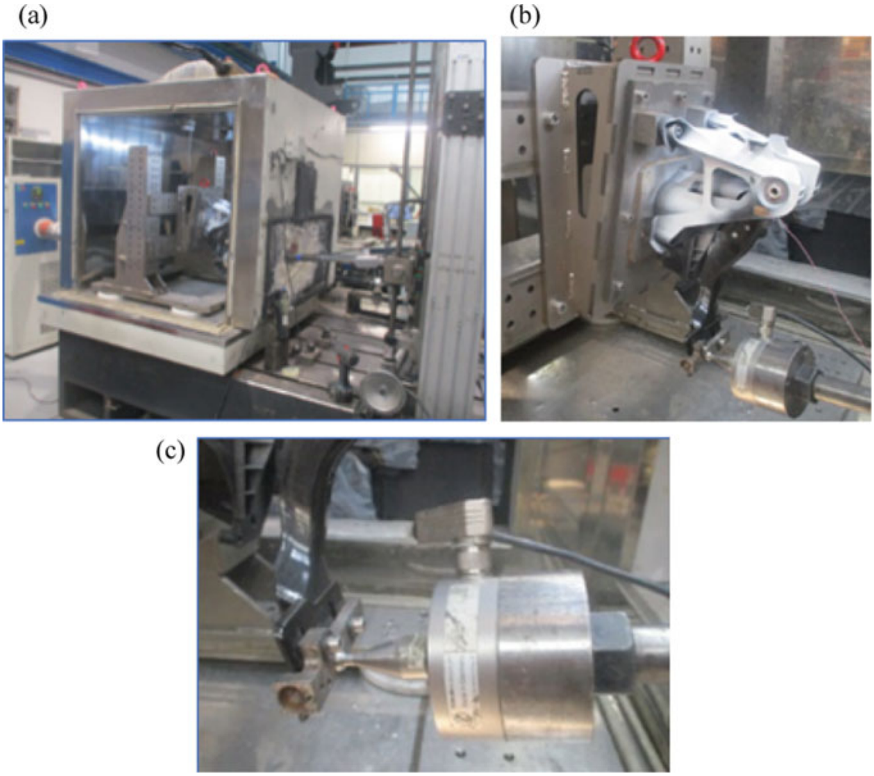


Fig. 8 **a** Validation test setup in climatic chamber **b** pedal bracket set-up **c** load applying on the frontal brake

Table 3 Testing results

Load case name	Load (N)	Requirement		Result	
		Elastic deformation	Plastic deformation	Elastic deformation	Plastic deformation
Frontal brake	2.300	< 17 mm	< 5 mm	OK	OK
	3.000	No breakage		No breakage	
Frontal accelerator	200	< 6 mm	< 2 mm	OK	OK
	1.000	No breakage		No breakage	

3.2 *Front-End Carrier*

Materials

The front-end carrier used to evaluate the performance of the new material is currently manufactured using PP + 40% of long GF (PPGF40) by IMC process, and it is replaced by PP + 20% of rCF (PPrCF20) in strand format. The prototype manufactured with the new material must fulfill the defined mechanical specifications. In addition, the change from GF to rCF will reduce the total component weight because the material has a lower density.

Manufacturing process

The process technology used for this demonstrator manufacturing was IMC. IMC is a direct inline-compounding that can be used to process highly filled and/or long fiber-reinforced thermoplastics in the injection-moulding process. IMC is characterized by the inclusion of a twin-screw extruder with a high plasticizing performance into the injection concept. The injection-moulding compounder allows feeding of endless fiber roving into the melt near the end of the twin-screw extruder. This configuration allows having longer fibers in the final part, achieving better mechanical properties compared to the commercial long fiber compounds available in the market in pellets.

The main elements of the IMC are gravimetric metering, the twin-screw extruder and the clamp unit. A co-rotating twin screw is used for plasticizing and a piston for injection, instead of a single screw for plasticizing and injection used in a conventional injection machine.

In the context of using rCF, where they have strand format, the endless fiber roving feeding system is replaced by a specific dispenser for feeding this kind of fibers (Fig. 9).

The demonstrator was injected in 2 materials: the current PPGF40, and PPrCF20 for performance evaluation. The process parameters were about the same for both materials, only the temperature of the barrel should be controlled better with the rCF compounding due to conductivity effect of the CF (Figs. 10 and 11).

Fig. 9 Configuration to dispense rCF strand

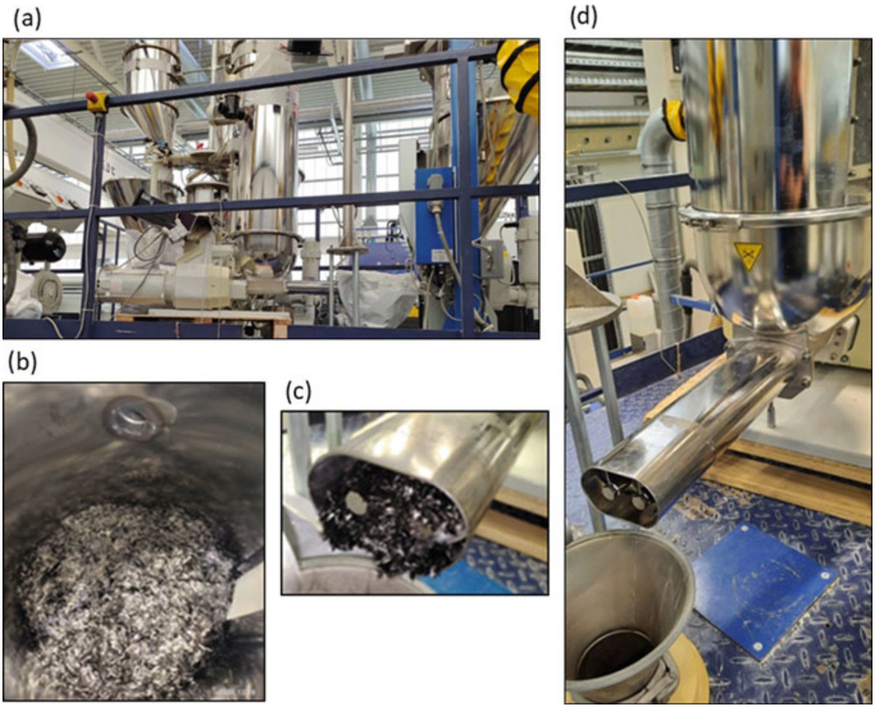


Fig. 10 **a** Specific dosing unit for fiber strand **b** rCF in the hopper **c** rCF being dispensed by the twin screws of the feeder **d** Detail of the dosing unit and the funnel of the IMC machine

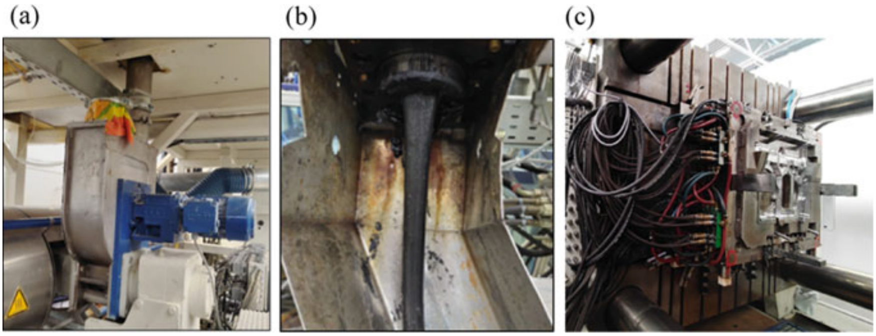


Fig. 11 IMC process during front end carrier manufacturing: **a** Lateral feeder where the recycled fiber is taken to the main extrusion screws of the IMC machine **b** melted PPrCF20 material flowing through the nozzle **c** front end carrier injection mould

Validation test

The validation test is focused on static load cases based on the use case of the bonnet latch overload. A compression and a traction load are applied in the latch area and the component must withstand a force of 2.300 N without any brake and achieving a specific stiffness.

With the aim to compare the component performance with different materials, a specific test was designed. This test is able to evaluate not only the component structural performance but also each material itself. Figure 12 shows the test set-up and Table 4 shows the loads on the component, the test conditions and the results obtained for the material developed PPrCF20.

The material developed in the FiberEUse project (PPrCF20) has been validated using the minimum value required for the component (Table 4), but it has also been compared with the results obtained in the same test for the original material that is proposed to be substituted (PPGF40). Table 5 shows the comparison between the two materials.

It can be observed that the experimental material has better physical properties than the current one in 3 of the 4 measured properties. In any of the cases, the values obtained always fit the specifications reported in Table 4.

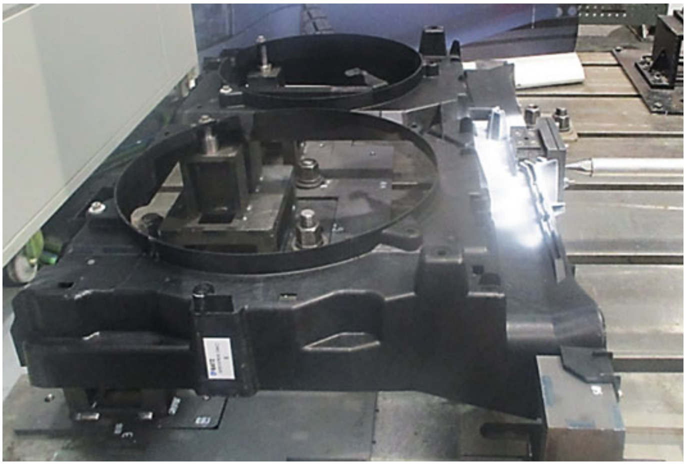


Fig. 12 Front-end carrier validation test

Table 4 Testing parameters and results

		Stiffness	Strength	Results
Static	Bonnet latch traction	420 N/mm	2.300 N	OK
	Bonnet latch compression	350 N/mm	1.500 N	OK

Table 5 Comparison between current material and new material for Front-end carrier application

		Stiffness (%)	Strength (%)
PPrCF20 increment over PPGF40	Bonnet latch traction	5.3	−34.6
	Bonnet latch compression	18.2	7

Table 6 Material properties for prototype manufacturing

Physical properties	Test method	Unit	Optimum	Minimum
Melt flow rate (230 °C, 2.16 kg)	ISO 1133	g/10 min	14	10
Melt volume rate (230 °C, 2.16 kg)	ISO 1133	cm^3/10 min	15	10
Density (23 °C)	ISO 1183-1/A	g/cm^3	1.14	

4 Cowl Top Support Demo Case

In this case, the innovation is to introduce recycled material as a reinforcement. Currently, to produce this type of parts, PP + 30% of virgin short GF (PPGF30) is used. The objective of this development is to substitute the reinforcement, at least partially, with thermally recycled GF.

4.1 Materials

The cowl support manufactured by Maier is made of PPGF30 with conventional injection moulding and in the FiberEUse project, it has been replaced with PP + 30% of rGF (PPrGF30). In table 6 it can be seen the performances that the material must fulfil for the prototype manufacturing.

4.2 Manufacturing Process

The manufacturing process of this part is plastic injection moulding (conventional injection of plastics).

Plastic injection moulding is the process of melting plastic pellets, in this case pellets of PP + rGF, that once melted enough, are injected into a mould cavity (under pressure and thermal conditions), which fills and solidifies to produce the final product. Injection moulding machine is composed by parts: the injection unit, the mould and the clamp unit.

The Fig. 13 scheme explains the injection moulding process. PPrGF30 pellets are fed into a heated barrel (4) and mixed using a helicoidal screw (6). Then it is injected

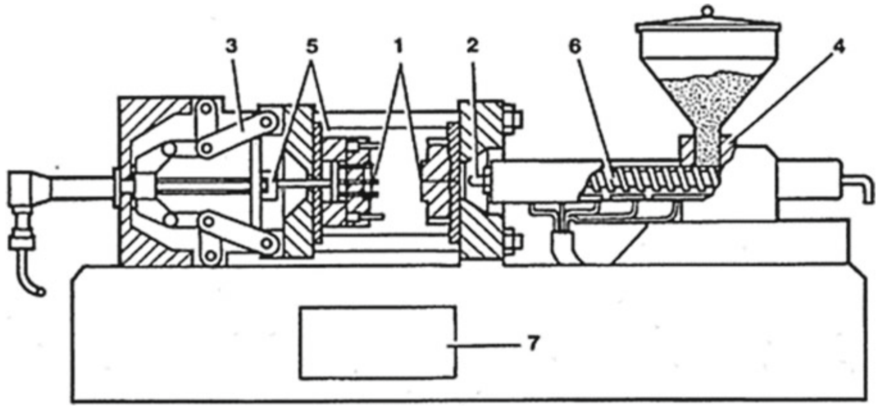


Fig. 13 Injection moulding machine scheme

into a mould cavity (1) though the nozzle (2). The other component of the injection machine is the closing group (3 and 5).

Parts to be injection-moulded must be very carefully designed to facilitate the moulding process. In this context, there are some aspects that have to be taken into account: material, desired shape and features of the part, mould material and the properties of the injection machine.

After the compounding of the PP material with the recycled glass fiber, there has been a first industrial injection trials to manufacture testing plates in order to analyze the differences between the injection process parameters with PPGF30 and PPrGF30. The parameters that have been studied are the screw temperature, the different injection pressure and the time of dosing and cooling.

After different injection trials the conclusion was that the injection process parameters needed are quite similar in both cases.

The demonstrator was injected in 2 materials: the current PPGF30 and PPrGF30 for performance evaluation. The Fig. 14 shows the material, the Fig. 15 the injection process and the mould of the cowl top support. The prototype obtained is shown in Fig. 16.

4.3 Validation Test

As mentioned before, the cowl top support has to give the needed stiffness to the part but also it has to break at determinate conditions. This characteristic must be maintained in different environmental and operating conditions. For that reason, the demo case is tested under different thermal and cycle conditions. The part also is tested by impact test method. The Table 7 shows the summary of the validation test methods and the requirements after the test to assure the suitability of the cowl over support of rGF.

Fig. 14 Recycled material in pellets

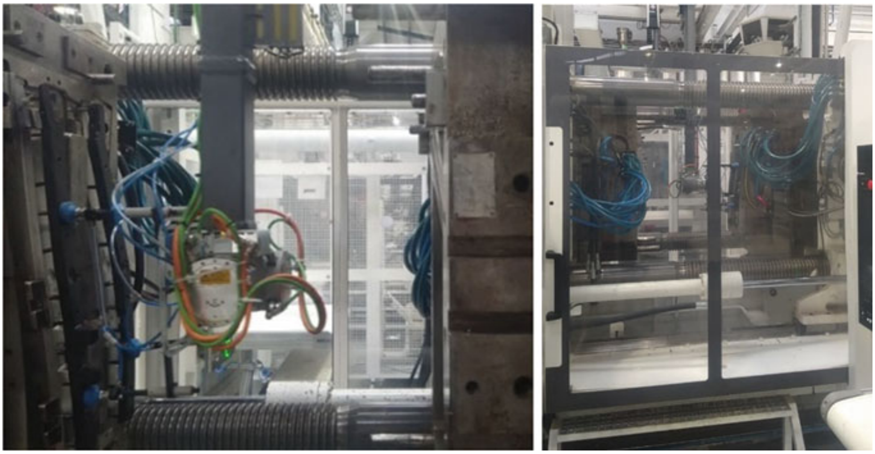


Fig. 15 Cowl top support injection process

Fig. 16 Prototype cowl top support with rGF

Table 7 Validation test conditions

Functionality	Test method	Requirement
Impact resistance	(1) 200 g/50 cm/−20 °C	Without break and without cracks
	(2) 1000 g/50 cm/23 °C	Without break and without cracks
Weathering	(1) 4 cycles. One cycle being: 16 h at 40 °C and 95%HR + 3 h at −20 °C + 6 h at 100 °C	Without deformation. Without peeling off
	(2) 3 cycles. One cycle being: 22 h at −40 °C + 2 h at 25 °C + 22 h at 95 °C + 2 h at 25 °C + 22 h humidity + 2 h at 25 °C	Without deformation. Without peeling off
Thermal stability	(1) 30 min at 110 °C	Without deformation. Without peeling off
	(2) 22 h at 90 °C	Dimensions between tolerances
	(3) 7 days at 70 °C	Dimensions between tolerances
	(4) 24 h at −40 °C	Dimensions between tolerances

HR Humidity relative

Table 8 Summary of the results of the tests performed

	PPGF30	PPrGF30
Weathering	OK	OK
Thermal stability	OK	OK
Impact resistance	OK	OK

The summary of the results is shown in Table 8.

5 Light Transmitting Single Skin Profiled GFRP Sheet Demo Case

The main objective to be reached with this demonstrator is to substitute virgin glass fibers (chopped roving) with a CSM (Chopped Strand Mat) made with rGF (so called TrGF Mat—Thermal recycled Glass Fibers Mat) to produce a light transmitting single skin profiled GFRP sheet. A Chopped Strand Mat is basically a non-woven fabric where a "binder" keeps the chopped fibers together.

Fig. 17 Cormatex industrial production of rGF mat

5.1 Materials

To manufacture this demo case, rGF from pyrolysis/depolymerization process have been used, combined with UP. The objective was to maintain the properties when the 25% of virgin GF coming from roving would be substituted totally by rGF coming in mat format. The rGF mat required in this demo case has been produced by an external company (Cormatex S.r.l.) in its facilities. The company developed an air-lay technology based on an aerodynamic fiber batt formation system, capable of replacing traditional mechanical processes using carding machine and cross lapper, in the nonwoven sector (Fig. 17).

5.2 Manufacturing Process

A light transmitting profiled GFRP sheet is usually produced with continuous lamination plant as shown in Fig. 18.

On a polyethylene terephthalate (PET) film (from 20 to 125 μm usually) catalyzed resin is spread with different systems as reciprocator, hopper, squeegee or rotating shaft. Over this first resin layer, a second glass fiber layer is added. Typical glass fiber layers are made directly by an in line cutting roving machine (so called chopped roving) or adding glass fiber mat as a chopped strand mat (CSM), continuous filament mat, woven roving or fabrics. These two layers goes under a calendar where a second PET film is coupled. As an option a thin layer of resin can be spread on the upper film, so called gelcoat finishing (Fig. 19).

Test specimens were manufactured by a continuous lamination plant at standard conditions (lamination speed) validate the use case.

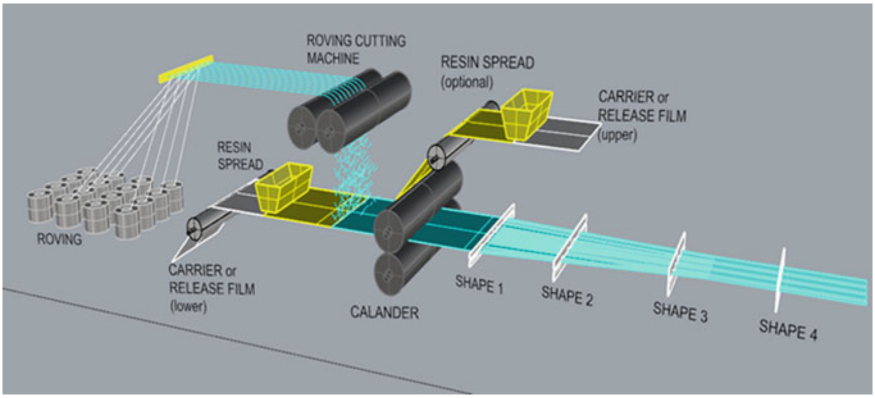


Fig. 18 Basic scheme of a continuous lamination plant

Fig. 19 rGF Mat processing

5.3 Validation Test

The objective of these tests has been to evaluate the processability in a continuous production line. The description of the test carried out are the following:

- A correct resin/GF ratio allows the surfaces to be regular and smooth. rGF mat has a resin/GF ratio 3.1:1 very similar to used virgin GF for Rivierasca application, that is 3.0:1. This means that a rGF mat can easily substitute a virgin CSM in standard application without varying raw material consumption.
- rGF mat wet out time: Wet out time is the time the resin needs to wet the GF. The GF is considered completely wet when it changes its colour from white to translucent. This test is carried out to verify the processability of the rGF mat in an industrial lamination plant without bubbles. The GF must be completely wet before entering the calander, so the wet-out time is closely related to plant speed. At Rivierasca plant the speed is 7.5 m/min and the area where the resin wets the GF is 8 m long. It has been verified that the wetting time of rGF is less than 63 s.
- rGF mat stability in continuous lamination (unbending and linear flow): The objective of this test is to verify that the rGF mat remains flat during lamination thanks to

smooth unwinding and linear flow under the calander without bending or moving aside. The folds will cause a non-homogeneous thickness of the laminate and aesthetical issues. Again, this test verifies the processability of the rGF mat in an industrial lamination plant.

- rGF mat visibility result (translucency) (Fig. 20): translucency is measured according EN ISO 13468-2 (light transmittance). The defined target for the "Light transmitting single skin profiled" was $\geq 60\%$.

Sample manufacturing and testing to avoid resizing of the rGF mat are shown in Table 9. Resizing was done through adding an organic functional silane to the UP resin system before laminating with the aim to improve the fibers-resin compatibility. The result from testing campaign was that re-sizing of fibers is mandatory to recover mechanical properties to standards values.

Besides a thermal demanufacturing rGF mat, a mat from directly shredded GF composite (Sh-rGF) has been also produced and tested. Such Sh-rGF mat shows an interesting solution for the composite industry especially for non-transparent applications (Fig. 21).

Samples of the results of the industrial lamination tests are shown in the Figs. 22 and 23. Sheet "A" is a test reference. It is a basic corrugated sheet made with virgin

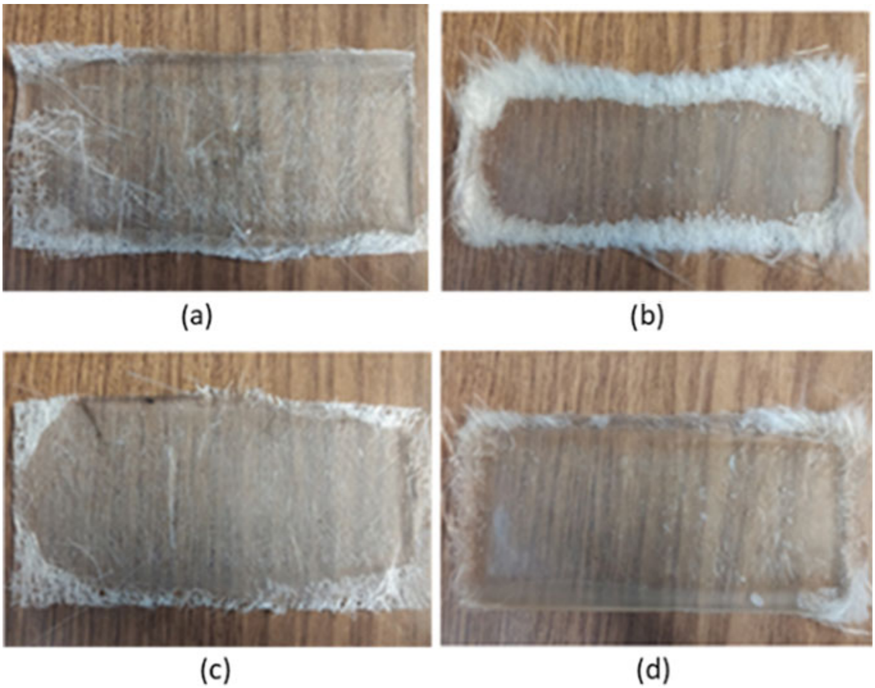


Fig. 20 Preliminary samples without sizing for testing translucency properties. **a** Test reference sample **b** test 1 sample **c** test 2 sample **d** test 3 sample

Table 9 Mechanical properties of specimens

Sample manufacturing				
	Test reference	Test 1	Test 2	Test 3
Kind of fiber	Virgin GF	rGF	rGF	rGF
Sample thickness (mm)	0.66–0.74	1.2–1.23	0.73–0.79	0.62–0.78
GF content wt%	25	25	25	12.50%
Additive	Styrene 3%	Styrene 3%	Styrene 3%	Silane 3%
Tensile test				
Modulus (MPa)	8310	6449	8378	6429
Stress yield (MPa)	112	34	45	29
Strain (%)	1.7	0.62	0.59	0.47

Fig. 21 rGF Mat (on the left) and Sh-rGF Mat (on the right)

roving and UP resin. Sheet "B" was produced by substituting the virgin glass fibers (roving) with the rGF mat (the rGF mat substituted the 100% of virgin roving). Sheet "C" was produced by substituting the virgin glass fibers (roving) with the Sh-rGF mat (the Sh-rGF mat substituted the 100% of virgin roving).

Sheet "D" (Fig. 24) is another test reference of a standard product from Rivierasca. It is a basic corrugated sheet made with virgin roving enclosed by two layer of virgin woven roving (on top and bottom side) and UP resin. This kind of multilayer laminate aims to provide higher mechanical performance of the sheets. Sheet "E" (Fig. 24) was produced by substituting only the virgin roving in the middle of the laminate with the rGF mat (the rGF mat substituted the 100% virgin roving). Sheet "F" (Fig. 25) was produced with the same layering of sheet "E" but using Sh-rGF mat instead of rGF mat with the same 100% virgin roving substitution.

The summary of the results is shown in Table 10.

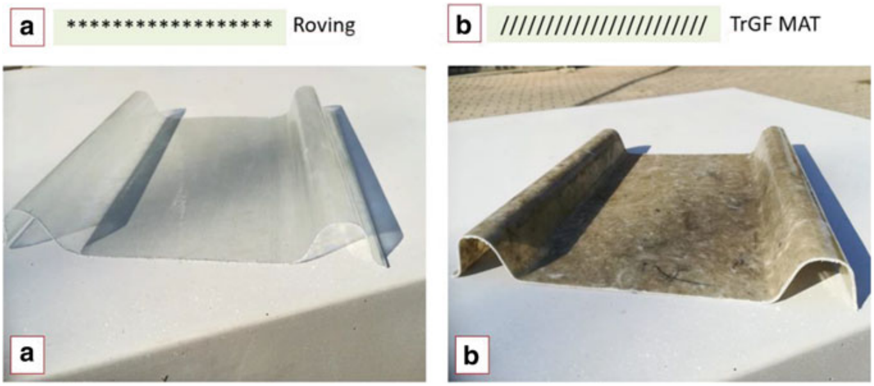


Fig. 22 Standard sheet and its equivalent made with rGF mat

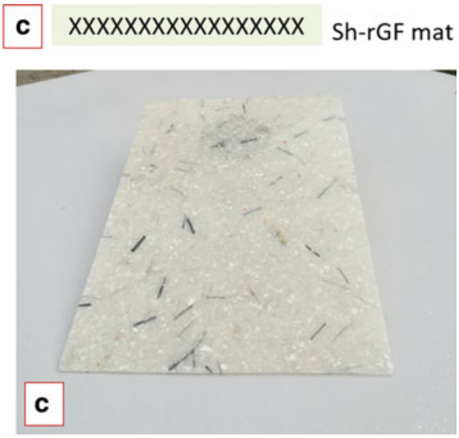


Fig. 23 Sample made with Sh-rGF mat

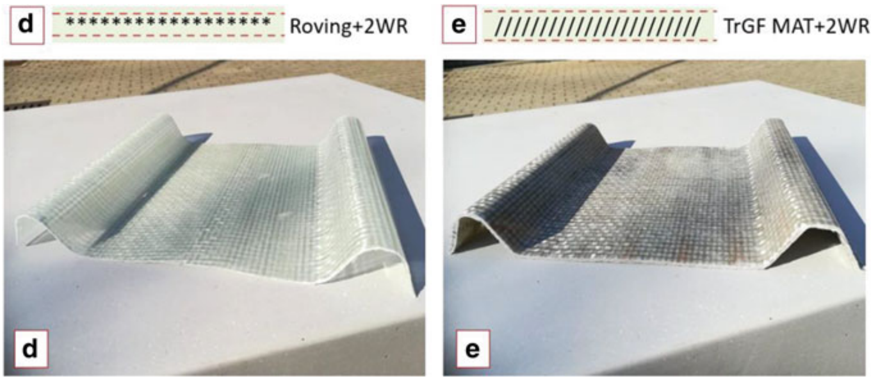


Fig. 24 Standard sheet (3 GF layers) and its equivalent made with rGF mat

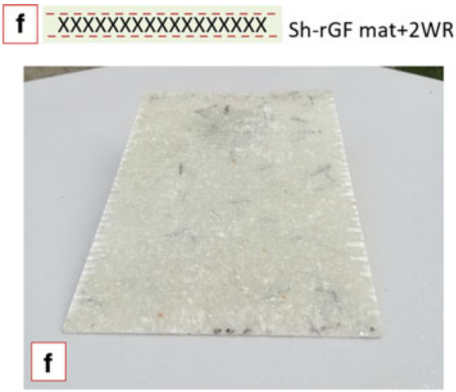


Fig. 25 Sample made with Sh-rGF mat and two woven roving

Table 10 Mechanical resistance of sheets

	Test ref. A	Test B	Test C	Test ref. D	Test E	Test F
Kind of fiber	Virgin GF	rGF	Sh-rGF	Virgin GF	rGF	Sh-rGF
Sample thickness (mm)	0.79–0.87	1.69–1.79	2.58–2.86	1.27–1.32	2.33–2.45	2.76–2.79
GF content wt%	25	25	17	14	13	13
Tensile test						
Modulus (MPa)	7765	5196	3302	16,018	15,317	7361
Stress yield (MPa)	81	41	20	216	73	97
Flexural test						
Modulus (MPa)	9994	6343	3378	14,651	14,802	14,257
Stress yield (MPa)	277	102	52	443	259	353

6 Conclusion

As a final technical conclusion after analysing the validation test of the different demo cases, it is summarized that:

- Pedal Bracket manufactured by BATZ passed the validation test, which consisted on an aging pre-treatment of the part, to subject later to different loads applied in different areas of the brake and gas pedal. Thus, it gives rise to the use of new materials in the automotive sector.
- Front-end carrier also manufactured by BATZ, has also been technically validated, fulfilling above the mechanical requirements defined for bonnet latch traction and compression tests. In some of the cases, the results obtained for the new material with recycled fiber overcome the values of the original material.
- Cowl top support also passes the validation test. After the results of the validation carried out on the component, it is proven that the weathering and thermal stability

tests have been passed. In addition, the impact resistance test, being the most critical due to the use of recycled glass fiber, has also been successfully overpassed. These results confirm the use of recycled glass fiber instead of virgin glass fiber for this application.

- The results obtained during the production of a Light transmitting single skin profiled GFRP plates demonstrated the technical feasibility of glass fiber recycling and reprocessing through a process chain from waste or EoL products to new semi-finished products such as rGF mat and Sh-rGF mat. These results are very promising for future industrial uptake and business.
- The reduction of the mechanical properties of rGF can be improved by using a mix of recycled fibers and virgin fibers.

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