

Increasing Strength of Recycled Materials by Fiberglass Pulverization and Powder Direct Molding

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Abstract. Direct molding is a technology where thermoset powders are agglomerated by compression molding without adding any additional substance or linking agent. It has been applied to powders from composite recycling, such as fiberglass. Agglomeration depends on residual reactivity of powders, the intrinsic re-activation of the particle surfaces because of the broken chemical links, and an incipient degradation mechanism during molding. In the case of continuous fiber laminates, the mechanical properties of the virgin item cannot be recovered as the recycled composite is made by particles. Nevertheless, high values may be reached, potentially interesting for such applications, depending on some precautions during the molding phase. Powders from grinding of fiberglass have been recovered from industry. They have been compression molded, and samples have been extracted from different parts of the molded plate to evaluate the distribution of the mechanical properties by bending tests. Results show that a bending strength up to 27 MPa can be achieved, without using any virgin material or additional substance, and a bending modulus over 3.5 GPa. However, pressure distribution during molding is not uniform and mechanical properties strongly vary from the periphery of the plate to the inner zones.

Introduction

Fiber Reinforced Plastics (FRPs) are acquiring an even more increasing demand in different fields of application. They represent 3% of the total plastic market volume and account for 16-20% of the total plastic market value. Construction (25%), transportation (24%), electronics (17%) and energy (13%) are the largest segments where both continuous and discontinuous fibers are used. Currently, the mentioned FRP market is highly dominated by glass fibers representing about 90% of the global volume mainly because of their notable strength-to-price ratio [1]. This increasing demand is the driving force for new research studies in developing other FRP products [2] which can also be applied in advanced sectors such as aeronautics, where fiber reinforced composites (FRCs) with thermoplastic matrix are used in Aircraft radome [3]. Simultaneously, sustainability issues led to the development of green composites with natural fiber reinforcements, also in the field of maritime engineering [4]. In this last field polymeric matrix composites (PMCs) with glass fibers reinforcements first found applications in marine structures such as recreational boats, working vessels and boat components [5].

The widespread use of glass fibers is also due to their lightness, high strength, thermal stability, electrical insulation properties and low costs. Glass fibers in combination with polymers, develop composite materials in the form of fiberglass with excellent thermal and electrical insulation properties; moreover, they are chemical inert under many conditions, dimensionally stable and non-magnetic. Fiberglass usually consists of a thermoplastic matrix, commonly, polyamide, poly(phenylene sulfide), polypropylene and polybutylene terephthalate) [6]. Polyurethane based composites are also acquiring great resonance because of their mechanical performance and durability

[7]. Polyester and vinylester based composites are their thermoset matrix counterparts; moreover, epoxy-based glass FRCs are also used and they find application among others, in high-voltage insulator core rods [8]. Appropriate combinations of resin matrix and glass fiber configuration (i.e., fabric or short fibers) influence mechanical performances [9]; the integration of carbon-based fillers such as carbon nanotubes or reduced graphene oxides into glass fiber reinforced plastics (GFRPs) influences mechanical performances as well; in fact, they are weakened if adhesion among fillers, glass fibers and matrix is not optimal [10]. For the mentioned applications, water absorption is also critical for mechanical performance, and in this view, epoxy matrixes are more susceptible to moisture absorption compared to vinyl-ester ones mainly due to differences in resin susceptibility to hydrolysis and matrix plasticization [11].

The wide diffusion of GFRPs opens the urgent need to introduce recycling strategies, transferable at an industrial level to avoid the commonly adopted habit of dismissal to landfills. In fact, to date there are ever increasing landfill restrictions. In recycling it is important to develop re-processing solutions of recovered composite constituents to obtain second life products with limited reduction in mechanical and functional properties. The current adopted strategies are based on mechanical, thermal and chemical processes but advanced recycling methods such as electrochemical recycling, micro-wave assisted recycling and sono-chemical methods have also been emerging [12].

Mechanical recycling methods based on pulverization, grinding and shredding are the most spread ones because of their ease of process. They are commonly applied with the end of life (EoL) of turbine blades, and the recycled products can be integrated in concrete and mortar to increase tensile and compression strength [13]; in this view the optimization of sieving process is crucial to reclaim useable fiberglass for second life products [14]. Mechanical performances of compression molded composites from chopped GFRPs made of a PA 6 matrix revealed that the size of chopped fibers causes complex damage mechanisms and variability in mechanical performance [15]. Unfortunately, mechanical recycling methods, differently from chemical-based ones, weaken the recovered fiberglass [16].

Recycling strategies based on thermal processes that are combustion, pyrolysis and fluidized bed [17] are also promising but different issues still must be addressed. Combustion involves energy recovery, in fact the material to be recovered is co-processed with another one. It develops via the cement kiln route where the polymer matrix is burned as fuel for the process whereas the glass fibers provide mineral feedstock to be used as part of the cement clinker. This way of co-processing reduces the carbon footprint up to 16% and provides valuable materials to the process. Pyrolysis allows conversion of organic material in an inert atmosphere at temperatures varying from 450°C to 800°C depending on the nature of the composite matrix. This process induces volatilization of the resin matrix whereas fibers and fillers are isolated and recovered. Unfortunately, the recovered fibers have lower mechanical properties than virgin ones, so they can be used as fillers or replacement in the production of new composite materials. Moreover, it does not make use of chemicals. Pyrolysis can be also used in combination with oxidization process to obtain glass fibers with carbon free surface [18]. In the fluidized bed process, solid particles are transformed into a fluid state through their suspension in a hot stream of air at temperatures ranging between 450°C and 550°C and streamed at speed between 0.4 and 1.0 m/s. Despite pyrolysis, in fluidized bed process the rich and high-flow oxygen atmosphere allows obtaining clean fibers with very little char surface contamination. As for pyrolysis, the recovered fibers show significantly reduction in mechanical performance, up to 75%.

New perspectives are offered by chemical methods as solvolysis which uses fluid in supercritical conditions to recover polymeric matrix. The use of H₂SO₄ allows reaction with the epoxy constituents to obtain in situ oxonium enabling for clean glass fibers with properties like virgin ones. Epoxy matrix can also be partially recovered [19]. A recent technique using ultrasonic power and sonotrode allows separation and reconsolidation of GFRP laminates for aerospace uses. It was demonstrated that reduction in mechanical performance was about 25% [20]. Alternative solutions consisting in tailoring epoxy resin with cleavable curing agents to allow successive separation by acid digestion from glass fibers maintaining a good surface quality compared with virgin ones are offered by the market of turbine blades [21].

Recycling GFRP constituents and fabricating second life products with good mechanical performances are severe matters of interest. In the current study a “direct molding process” based on compression molding, has been used to produce GFRP plates from pulverized fiberglass. This approach uses the residual reactivity of shredded GFRP powders to allow polymerization. In addition, broken bonds on particles’ external surface act as polymerization sites in further manufacturing steps. Moreover, during process any linking agents or virgin materials have been used. This manufacturing procedure has been previously used to prove the feasibility of obtaining second life products made from 100% recycled fiberglass [22] and its transfer to industries as well. Direct molding can be adopted to recycle several kinds of waste fiberglass and obtain recycled products with optimal mechanical performances [23, 24]. The further goal is to evaluate the distribution of the mechanical properties on large plates by direct molding of pulverized fiberglass waste. This study investigates this aspect in the case of waste fiberglass powder from industry, where grinding is used to provide the final shape to the manufactured items.

Materials and Methods

Fiberglass powder. Waste fiberglass in the form of powder was supplied by a factory of technical laminates. The collected powder was the result of a grinding procedure to resize the products which were laminates used as insulating refrigerated trucks. The polymeric matrix is a polyester resin.

Manufacturing. A recycled plate was produced by hot compression molding starting from the collected powder without any addition of binder, linking agent and virgin materials. This process is known as direct molding and takes advantage of the powder residual reactivity which develops high energy because of the high area to volume ratio. Moreover, the broken bonds due to the grinding process act as polymerization sites.

The plate was produced starting from a nominal mass content of the collected powder of 100 g. The amount of powder was poured into a $150 \times 150 \text{ mm}^2$ cavity of a steel mold without using a release film. The whole assembly was placed between the plates of a hot parallel press. A pressure of 2 bars (0.2 MPa) and a temperature of 250°C were applied for a time of 15 min during compression molding. The whole manufacturing procedure is shown in Fig. 1. The high temperature is responsible for increasing resin mobility once the transition temperature is overcome, whereas pressure is responsible for providing the contact among the particles during agglomeration. The molding parameters were chosen to limit degradation of the resin. At the end of molding, the assembly was left cooling down to room temperature before extraction.

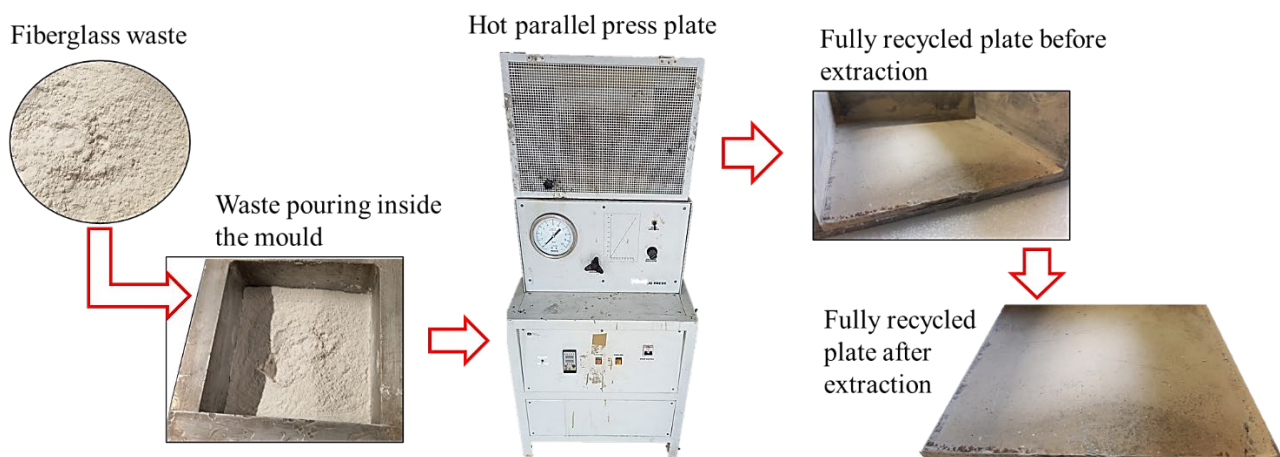


Fig. 1. Compression molding of a recycled fiberglass plate.

A sum of seven specimens with nominal sizes of $80 \times 10 \text{ mm}^2$ were extracted from the molded plate through a metallographic cutting machine. Specifically, specimens were extracted, one parallel to the other, from one edge to the opposite one, to evaluate the trend of physical and mechanical properties along this direction. In Fig. 2, the concept of experimentation is shown from extraction to mechanical testing.

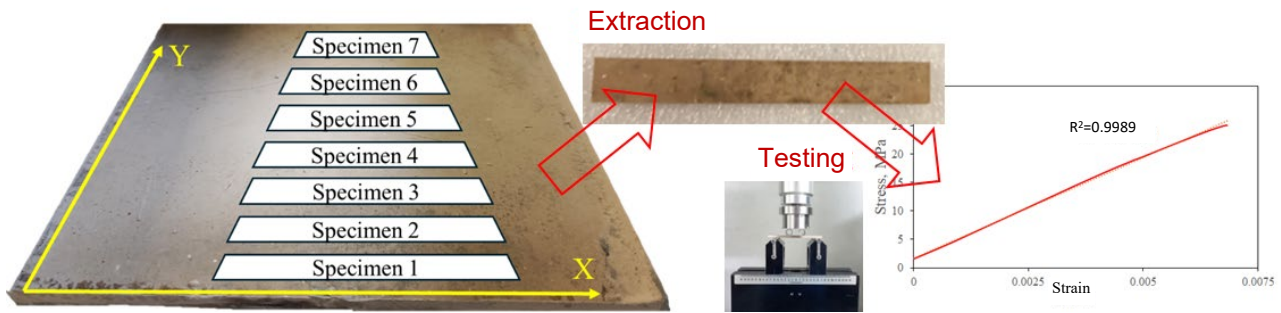


Fig. 2. Sample extraction and testing.

Testing. Specimen density was evaluated by weighing the samples and dividing by their bulk volume, approximated to a parallelepiped. Table 1 reports physical data for all the extracted samples. An average density of 1.26 g/cm^3 is inferred for the full plate. The mechanical behavior was evaluated through bending tests, carried out up to failure. Specifically, the 4-point bending test was performed according to the ASTM D6272-02 standard because of the brittle behavior of the samples. A support span of 60 mm and a load span of 20 mm, 1/3 of the support span, were selected. All tests were performed with a speed rate of 1 mm/min through an Insight 5 Universal testing machine by MTS.

Table 1. Size and density of the extracted samples.

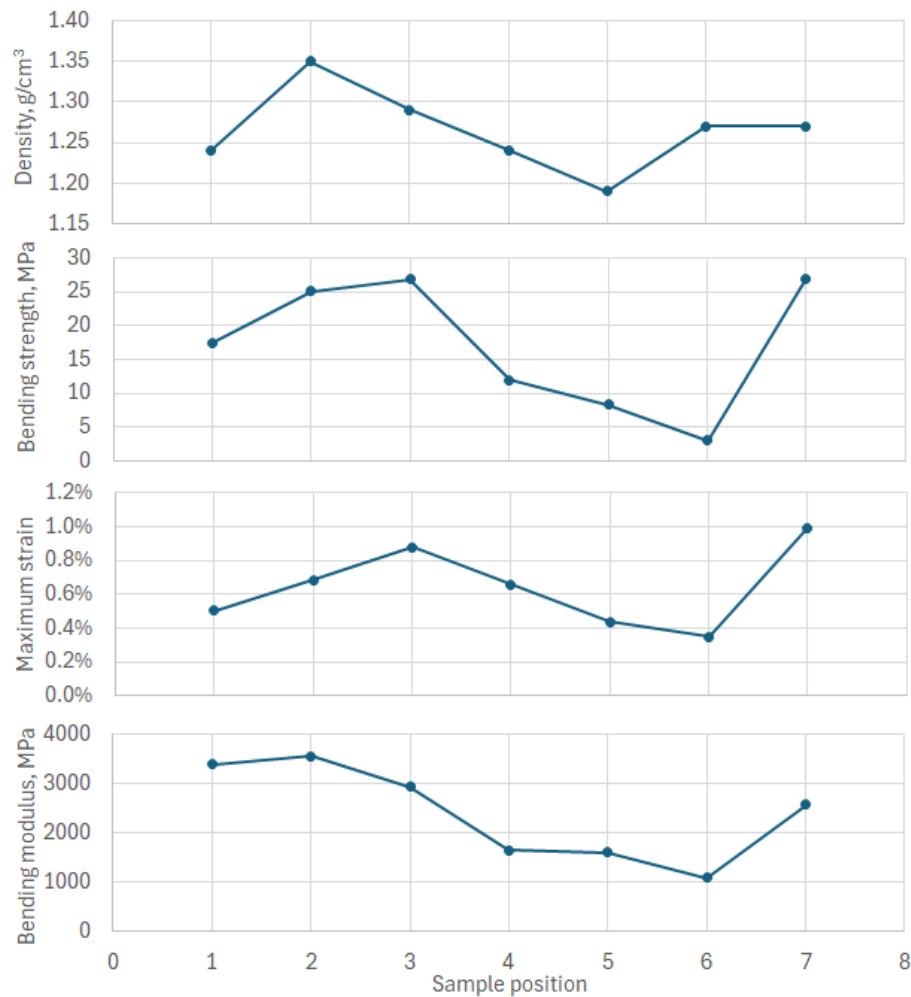
Sample ID	Thickness, mm	Density, g/cm^3
Specimen 1	3.09 ± 0.09	1.24
Specimen 2	3.03 ± 0.04	1.35
Specimen 3	2.98 ± 0.03	1.29
Specimen 4	2.94 ± 0.06	1.24
Specimen 5	3.01 ± 0.18	1.19
Specimen 6	2.79 ± 0.07	1.27
Specimen 7	2.76 ± 0.02	1.27

Results and Discussion

Results from mechanical testing are reported in Table 2. Samples exhibited a brittle behavior under testing, as shown in Fig. 2. The bending curve is linear for all the samples, with very good correlation factors, always higher than 0.98. This behavior is typical for agglomerated samples, mainly in the case of particle agglomeration. The average maximum strain of the plate is 0.64%, whereas the mean values for bending strength and modulus are 17.1 MPa and 2391 MPa, respectively. These values seem to be acceptable for such technical applications, where low strength is required, and it must be noticed that they have been achieved without using any additive or virgin substance.

Table 2. Mechanical properties from testing.

Sample ID	Maximum strain	Bending strength, MPa	Bending modulus, MPa
Specimen 1	0.50%	17.5	3386
Specimen 2	0.68%	25.1	3551
Specimen 3	0.88%	26.8	2923
Specimen 4	0.66%	11.9	1644
Specimen 5	0.44%	8.3	1596
Specimen 6	0.35%	3.0	1076
Specimen 7	0.99%	27.0	2559

**Fig. 3.** Property distribution along the sample length.

Nevertheless, the highest value for the bending strength is 27 MPa, about 60% more than the average. Similarly, the bending modulus reached 3551 MPa, 50% more than the average. It is under discussion if these properties could be obtained in the full plate, instead of such points.

In Fig. 4, physical and mechanical properties of Table 1 and Table 2 are reported as a function of the sample position, which is related to the distance from one edge of the plate to the opposite other. It is visible that a trend is present for all of them, with the maximum of the properties toward the edges or close to them. The edges are the parts of the plate subjected to the highest amount of heat,

because of the presence of the lateral mold wall, whereas the internal part of the plate is heated only by the upper and lower face of the mold cavity. Nevertheless, because of the presence of the gap between the female and the male part of the mold, it is possible that the pressure is not perfectly transmitted. For this reason, if the maximum of agglomeration is not reached at the edge, it occurs a little bit far from it. Another aspect to consider is that it is difficult to apply a uniform pressure to the plate during compression molding as the powder does not flow under pressure. For this issue, it is fundamental to level perfectly the powder before closing the mold. This action becomes increasingly difficult by increasing the plate size. As expected, if one side of the plate reaches a good agglomeration, the other side of the plate receives less pressure, but a local maximum can occur at the edge because of the lateral wall. This mechanism seems to be very well represented from results of Fig. 4, as generally the left part of the plate is higher in performances than the right part, apart from anomalies at the edges.

By improving agglomeration, every property increases, comprising density, strength, stiffness and ductility. In fact, all these properties depend on the number of joined particles during the compression molding step. This fact is partially shown in Fig. 4 where the maximum strain and the bending modulus are reported for each sample as a function of the bending strength, after normalization by the maximum values among all the samples. Stiffness and ductility generally increase when the strength increases, even if data are scattered because of the brittle nature of the tested sample. This behavior is common for agglomerated samples.

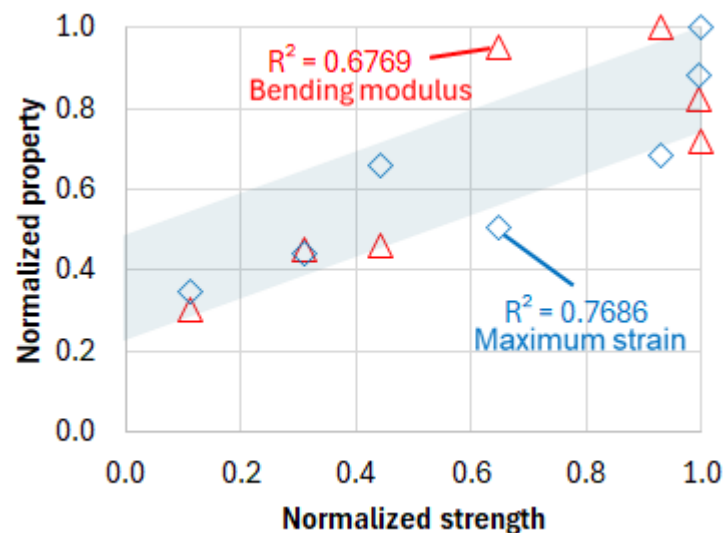


Fig. 4. Correlation between normalized mechanical properties.

Summary

The current study has obtained very high performances for recycled fiberglass in absence of any virgin substance or linking agent. In the best case, in the current experimentation, the bending strength has reached 27 MPa and the modulus has overcome 3.5 GPa. Nevertheless, it has been observed that there is a big variation in these properties in the molded plate. It is reasonable that such process limitations could have affected plate homogeneity. A possible cause is that the fiberglass powder cannot spread the applied pressure internally, and differences in powder bed thickness have led to more and less agglomerated zones. This aspect is critical to increase the size of the molded part and must be solved by adding some manufacturing steps to improve pressure distribution during molding.

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