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Nicolas Perry, Stéphane Pompidou, Olivier Mantaux, Arnaud Gillet. Composite Fiber Recovery: Integration into a Design for Recycling Approach. Technology and Manufacturing Process Selection, Springer, pp.281-296, 2014, 10.1007/978-1-4471-5544-7_14 . hal-01066975

HAL Id: hal-01066975

<https://hal.science/hal-01066975>

Submitted on 6 Oct 2017

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Nicolas PERRY, Stéphane POMPIDOU, Olivier MANTAUX, Arnaud GILLET - Composite Fiber Recovery: Integration into a Design for Recycling Approach - 2014

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Composite Fiber Recovery: Integration into a Design for Recycling Approach

Nicolas Perry, Stéphane Pompidou, Olivier Mantaux
and Arnaud Gillet

Abstract In industry, the use of composites, and more specially carbon fiber/thermoset matrix ones, is ever increasing. However, end-of-life solutions for these materials are still under development. In this chapter, a solution linking design strategies with a recycling process based on the solvolysis of the matrix by water under supercritical conditions is proposed. The needs and multi-disciplinary skills required for (i) taking recycling possibilities into account from the early stages of the product design, and (ii) the necessity to standardize its recycling capabilities with design requirements, will both be discussed. The present chapter highlights the need for designers to take a functional approach into consideration, including material characterization, limits of the recycling process, constraints and opportunities. The first lessons learned from experiments using this technique will be shown.

1 Introduction

Today, reducing greenhouse gases and pollution is one of our society's main challenges as it strives for sustainable development. In this way, the key focus for transport industry is to make lighter vehicles in order to decrease both energy consumption and CO₂ emissions. Thus, composites provide good opportunities for combining high material properties with an increased freedom for defining parts' geometry; as a consequence, their use is ever increasing. Aerospace and aeronautics industries have integrated composites for long, and at different levels

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(e.g. organic matrix based composites for cold applications, and metallic or ceramic matrix based ones for high temperature parts). However today, composites' low potential of recyclability limits their use in the automotive industry. Indeed, regulations in this sector impose a 95 % recycling ratio for an out-of-use vehicle. Moreover in a global and eco-friendly approach, one must analyze and take into account end-of-life solutions for systems, at an early stage of their development process.

The term *de-manufacture* has become more and more common, especially in the electronics industry. This is a recycling process for materials and products that includes end-of-life strategies and logistics in product development (Berry 1996; Gaustad et al. 2010): design engineers have to balance safety, energy efficiency and cost. Unfortunately, they rarely get to the point of thinking about what will happen to the product at the end of its useful life (Kriwet et al. 1995; Vallet et al. 2010). However, as new materials and technologies are developed, the challenge that recyclers face in, safely and economically, by recycling those products, grows ever more difficult (Calcott and Walls 2005). Recycling processes have to balance the technical, economic and environmental aspects of the end-of-life proposal. Recycling a product means:

- to have a recycling technology available,
- to get a dismantling solution and an access to the product,
- to have a disposal for life-ending composites (i.e. used parts, eventually polluted), clean parts (e.g. offcuts from machining) or even raw materials (e.g. unused carbon fabric, preregs, etc.).

Composite applications have opened up a new field of research and development in the domain of recycling processes. For example, water under supercritical conditions gives the opportunity to recover thermoset matrix based composites' reinforcement, like carbon fibers (certainly one of the most interesting reinforcement to recycle, both environmentally and economically; Duflou et al. 2009). This process will also open up new opportunities for second-life composites. In this way, we are focusing on a research area that aims at integrating recycling constraints in the design stage of composite parts. At the same time, we hope to promote discussion between designers and recyclers for innovating in the definition of new recycled composite products. This means that information and skills from both sectors will be shared. However, it also implies that materials and mechanical knowledge have to be developed, for both designers and recyclers. Therefore, it is necessary to include a third party in the discussion: experts in material and mechanical characterization.

The first section of this chapter presents an overview of composite recycling possibilities and the technical and economic reasons for their development. The second part will focus on the design for recovery issue and the specificities related to composite design. The third section will explain our understanding in terms of skills, needs and know-how required for addressing this issue. Before concluding, the last section will illustrate some feedback and the first lessons learned regarding eco-design for composites.

2 Composites Recycling: Motivations and Solutions

2.1 Motivations and Interests

Future regulatory constraints are driving industries to develop efficient end-of-life alternatives, based on technical and economic constraints. For carbon and aramid based products, high prices (e.g. carbon prepreg: approx. € 180/kg; Kevlar[®]: approx. € 150/kg) and the world shortage in raw materials production, are the leitmotiv for finding technical and cost-effective recycling solutions. In such cases, second use of composite fibers will be dedicated to the manufacture of medium or low loaded parts (non-structural in many cases). Indeed, the recycled fibers and reprocessed semi-products have to achieve full acceptance and gain the trust of users (i.e. designers), regarding their material qualities and performances. Actually upon seeing the “recycled” label, many stakeholders still tend to think of low quality.

However, as far as recycled carbon fibers are concerned, this is far from the reality. Methods exist today for recycling carbon fibers and pre-impregnated (prepregs), and the resulting recyclate retains up to 90 % of a new fiber’s mechanical properties. In some cases, this method even enhances the electrical properties of the carbon recyclate which can deliver a performance superior to the initial material’s (Perry et al. 2010a, b). So it is necessary to create demand for recycled reinforcement, by packaging it in a useful or attractive form to end-users. Besides, by retaining good material properties, this constituent can be reused as raw, in a so called *second generation (2G) composite*.

2G-composites (i.e. materials based on a 2G-reinforcement) are obviously more environmentally friendly. Indeed, they mainly ensure to decrease the use of petroleum for their production, and they also keep the potential of a next recycling loop. Lastly, 2G-composites are cheaper, which can lead to broaden the composites materials’ applications field.

2.2 Trails and Solutions

To make such a recycled product cycle viable, the key factors are the amount of waste deposit and its availability. In order to ensure the efficiency of the recycling path, that is to say, to guarantee and improve the flux regularity, infrastructures for both collection and identification must be established. Lastly, end-users must have confidence in the quality of the product in terms of robustness and value. Thus, mechanical, physical and chemical studies must be led in order to enrich the recycled material data, and comfort the properties of recycled fibers, semi-products and structures. This has been done successfully in the plastics industry; for example, identification labels were added to plastic parts to facilitate collecting and sorting. Most composite manufacturers already carry out waste management

procedures, with impetus from the REACH regulation; as a result, they recycle waste materials in the very workshop, when collection and processing solutions are proposed.

Otherwise, thermoset matrix-based composites' designers and manufacturers are currently taking two different directions. On the one hand, they are trying to increase the use of green or bio-composites; on the other hand, they are developing or improving recycling technologies. In the first place, the advantage of using bio-composites lies in their small environmental impact. They are made of:

- a bio-polymer matrix reinforced by synthetic fibers (e.g. glass, carbon, Kevlar[®]);
- a petroleum-derived matrix reinforced by natural fibers (e.g. kenaf, flax, hemp, bamboo, coconut stems);
- biopolymers (e.g. PLA or PHA) reinforced with natural fibers.

Natural fibers are currently tested to estimate their reinforcement properties. They promise to be the future solution for organic matrix composite parts (Feng 2010; Mohamad 2010). For example, regarding mechanical properties, some can easily compete with glass fibers, as their specific density ranges from 20 to 45, and their tensile stress from 400 to 1,500 MPa. Furthermore, solutions already exist for high-performances composites, e.g. by improving weaving processes (Weager 2010). Besides, biopolymers or bio-compounds (a combination of bio-polymer and petroleum derived polymer) have also been studied (Bourmaud and Baley 2009). For example, they come from PLA derived from cornstarch, or PA11 from castor seeds and its ricin protein. On the one hand, their carbon footprint is reduced by using a bio-renewable material, and on the other hand their recyclability potential is increased.

In addition, end-of-life impact awareness helps develop recycling technology. Thermoset matrix can be removed either by burning or grinding techniques. This is cheap but very aggressive for the carbon fibers (Mantaux et al. 2004, 2009). Complex thermal, chemical and mechanical processes are needed to obtain high quality recycled carbon fibers. Pyrolysis and solvolysis are two of these very promising solutions, as summarized in Table 1 (Pimenta and Pinho 2011). However, there are some limitations; for example, it is impossible to recycle different categories of matrix simultaneously. At the same time, specific coatings (e.g. metallic cladding for electric behavior) are not compatible with some processes. Thus, specific requirements must come from the recycling stages if they are to be efficient. The most obvious (but absolutely necessary) thing is to extract and free all the metallic inserts, even before grinding. Moreover before recycling, products have to be dismantled and adapted to the recycling process reactor. These reactors are mainly cylindrical; cutting operations are therefore compulsory.

In southwest France, composite recycling will increase soon in terms of quantity due to the creation of two dismantling platforms:

- TARMAC platform (acronym for *Tarbes advanced recycling and maintenance aircraft Company*) is dedicated to civil aircraft applications in collaboration

Table 1 Summary analysis of different recycling processes (adapted from Pimenta and Pinho 2011)

Process	Thermal		Chemical
	Mechanical	Fluidized bed	
Advantages	Recovery of both fibers and resin	High retention of mechanical properties	Very high retention of mechanical properties and fiber length
	No use or production of hazardous materials	Potential to recover chemical feedstock from the resin	High potential for material-recovery from resin
Drawbacks	Significant degradation of mechanical properties	Possible deposition of char on fiber surface	Common reduced adhesion to polymeric resins
	Unstructured, coarse and non-consistent fiber architecture	Sensitivity of properties of recycled fibers to processing parameters	Low contamination tolerance
	Limited possibilities for re-manufacturing	Environmentally hazardous off-gases	Reduced scalability of most methods
		Unstructured (<i>fluffy</i>) fiber architecture	Possible environmental impact if hazardous solvents are used
		Impossibility of material-recovery from resin	

with Airbus, EADS Sogerma. TARMAC first focuses on the re-use and certification of replacement parts in aircraft maintenance;

- P2P platform, close to Bordeaux, deals with the disassembly of ballistic weapons.

Otherwise, in order to manage the end-of-life of structures, a consortium of aerospace manufacturers (EADS Astrium Space Transportation, Snecma Propulsion Solide, etc.) has been working on the RECCO project (acronym for *Recycling carbon fiber reinforced composites*). The solvolysis process has been chosen for removing the thermoset matrix. In this technological and industrial background, all the stakeholders involved are analyzing an early integration of the recycling constraints and possibilities, in the design process of carbon composite parts. The next sections will explain the integration levels we face into develop a *design for recovery* approach.

3 Composite Design for Recovery

In order to take the end-of-life information into consideration from the product design phase, we naturally opted for *design for X* approaches. In our case, we worked on design for *recovery* (rather than *recycling*); taking disassembly into consideration, it should lead designers to propose recovery solutions for products or composite parts. In this approach, all the recycling requirements are considered as input (data) that must be taken into account in the product's functional specifications. Consequently from a semantic point of view, we also encourage the use of the term *recovery* instead of *recycling*, to emphasize the second life or second use of the product or of its constituents, after the recycling phase.

Lastly, we shift from the *cradle to grave cycle* to the spirit of *cradle to cradle*. Thus, even if we are using a *design for recycling* methodology or using the term *recycling*, we consider it to be a dynamic state, like a *rebirth for future use*, and not as a static and final goal. This means to consider future product design or future second life material from the recycling level. In other words, we are in two dual areas of research:

- firstly, design for end-of-life can be summarized as *design for recovery*;
- secondly, from recycling to design, the research deals with *robust material recycled for design*, that is to say constituents for which mechanical properties remain intrinsically invariant.

The challenges in *design for recovery* are to protect the environment, and create sustainable means for preserving our resources and reducing energy loss and pollution. It has two very basic goals. The first is to eliminate or reduce the use of hazardous or toxic materials which may present a serious threat to the environment, or put a recycler's workforce in jeopardy. The second is to discourage the use of materials that are simply not recyclable, or manufacturing techniques that

make a product non-recyclable with current processes. The best time to address these issues is during the design stage (Ferro and Amaral 2006). Indeed, addressing a product's end-of-life is essential at the very beginning. Adopting this premise helps to ensure an efficient recycling chain, that goes far beyond the scrap processor in a mill, a smelter or an extruder, that would take the recycled materials to make new ones. Design for recycling is a mindset that all design engineers must embrace if they hope to have their products considered as environmentally friendly. As mentioned before, design for recycling is driven by governmental mandates like the European Union's waste electrical and electronic equipment directive (WEEE) or end-of-life vehicle (ELV) directive; for example in Europe, the rate of re-use and recovery should reach 95 % by 2015, and 85 % for re-use and recycling, in average weight per vehicle and per year.

There is more than environmental compliance at stake here. As new materials are developed (such as carbon fiber-based composites), they bring about a new threat in terms of recycling. And as new constituents are introduced into products and are replacing some that have been recycled for generations, they negatively affect the products' recyclability (both practically and financially), and can have a devastating impact; like this, even recyclable materials can pose a problem when used in combination. Take for instance a product that is made from many different types of plastics; today's recycling technology can only sort two or three different types of polymer materials using a mechanical solution, at best. Composites effectively become non-recyclable, or at least the plastics fraction of that product will be non-recyclable (Seager and Theis 2004; Perry et al. 2010a, b).

Therefore, in order to address the global problem, we are working on different levels: (i) design teams, (ii) design methods, and (iii) design tools. In this way, we hope to reduce the gap between the existing recycling solutions or bio-composites possibilities, and designers' current solutions. Not only do engineers have new materials and new product design solutions for eco-responsible products, but there are also now different tools available to help modeling and evaluating the solutions and the impact of the product life. Lastly, this definition of the end-of-life requirements points out the needs in terms of maintenance or parts fixing.

Unfortunately, the results of these rating tools depend on the available information. Most of the time, little information can be found about the product's life and end-of-life at design stage. Furthermore, in the case of the recycling processes under development, it is necessary to anticipate the potential of the technologies and their applications. Uncertainty in decision-making will therefore increase. Indeed in terms of end-of-life consequences, design decisions will undoubtedly appear 5–20 years later.

Before giving further explanation, it is important to remember that composite design is complex: it is necessary to define simultaneously (i) geometry and shape, (ii) constituents (matrix, fibers and their orientation), and (iii) manufacturing process. These three topics are linked and interdependent in the design and optimization process. For example, the choice of laminate limits shape possibilities, and depends on manufacturing capability. Consequently, as illustrated in Fig. 1, the eco-designer has to juggle with constraints from various sources, in addition to

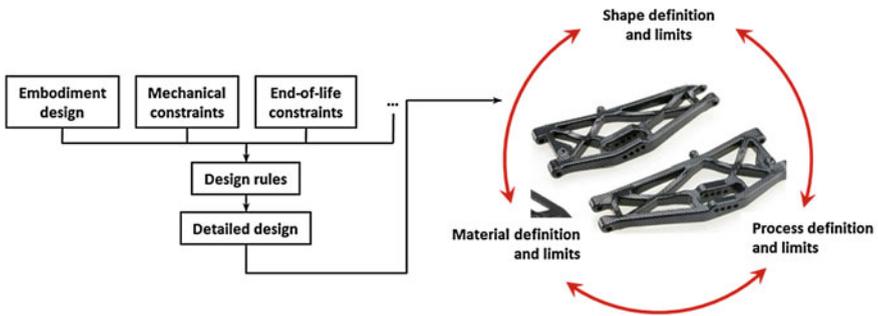


Fig. 1 Composite design constraints

internal constraints and relationships of the composite design. Up to now, these additional constraints have not been taken into consideration, except for the consequences of the new regulation (e.g. REACH) which focuses on manufacturing aspects.

4 Levels of Complexity for Integrated Design

Integrated design means addressing the recycling process development issue carefully, while at the same time proposing the possibility of including such evolutions in design methodology. We identified three main problems:

- the first is linked with recycling physics and scheduling to link and reach design methodologies;
- the second is dedicated to the uncertain and non-complete nature of the available information;
- the third problem has to do with competencies and skills needed for designing robust recovery problems.

4.1 Physics and Scheduling

This part deals with the knowledge of integrating the recycling process at the design stage.

These processes are for the most part under development, searching for breakthroughs, innovations and applications. The recycling rules that must be included in the design process are still under formalization, while designers must take decisions. Furthermore, decisions taken today will affect the product much later. Figure 2 shows the core elements that make up the skeleton of a product's end-of-life. Each phase of this process has its own limits and constraints to be

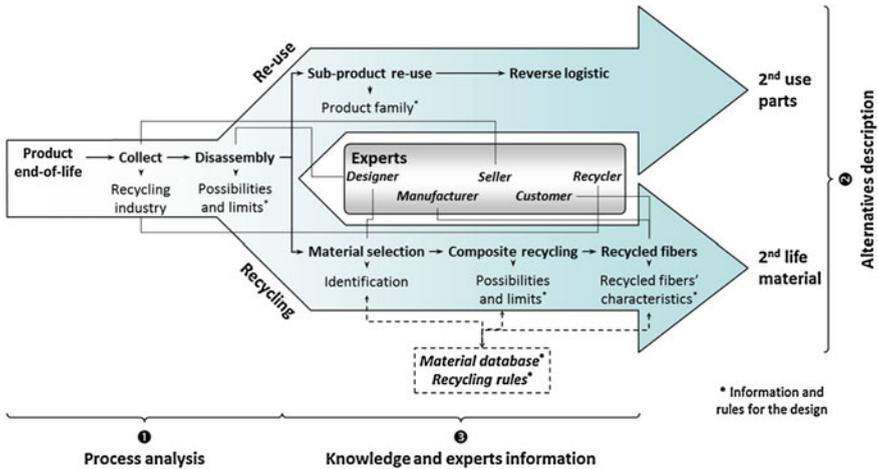


Fig. 2 Analysis of the recycling process, in the perspective of the product's design specifications and recycling process information

integrated or overshoot. Different stakeholders are included in the loop and should point out the information, expected data and decision rules they apply to switch from one stage to another. The different time frames between the real recycling process and the parts' design phase, increases the integration difficulties. For existing and robust end-of-life paths, constraints and material re-processing and re-use are well known. For new recycling processes, robust validation can take time, yet designers have to take decisions now. An extreme example is nuclear plants; built 40 years ago, there are still no efficient end-of-life solutions as their dismantling is about to begin.

Research and development teams are banking on some kind of technological breakthrough to guide developments in recycling. Innovation is needed not only in the recycling phase, but also for all the key stages. It is important to improve disassembly techniques (see *design for assembly/disassembly* approaches) or selection efficiency (Boothroyd and Altling 1992; Aymonier et al. 2006). At the end of the process, it is essential to develop innovative and valuable uses to compete with virgin raw materials (for similar characteristics), or to find new opportunities, at the very early design of the recycling path.

As far as composites are concerned, new processes enable fibers to be recovered with very little distortion and fracture, compared to the initial reinforcement used in the original composite part; supercritical fluids can provide such opportunities (Loppinet-Serani et al. 2010; Kromm et al. 2003). However, the problem of misalignment and realignment of the recycled fibers still remains. Competences (knowledge and know-how) and fiber spinning and weaving skills have been integrated by recycling teams.

Other alternatives consist in reprocessing medium-sized flat rectangular pieces of pseudo-unidirectional (1D) or woven (2D) recovered carbon fabric. The innovation

consists in proposing a patchwork approach for designing parts. Specific studies must identify the mechanical characteristics and efficient strategy for material characterization according to the product design development phase, from the recycled fibers to the final structure (Laurin 2005; Rollet 2007). This pyramidal testing problematic, at all stages of the product life and at all scales (i.e. from fiber to structure) must integrate this uncertainty, but in real case tests (cf. hereinafter) (Dennison 2010; Ladevèze et al. 2006).

4.2 Information Access and Trustworthiness of Results

Figure 2 can also be the starting point for the identification of the available information at each stage of the process. Integrating the different stakeholders helps identifying which kind of information is really available or needed, and who gets or requires it (Bernard et al. 2007).

In a classical product development V-cycle, two elements arise. Firstly, the kinds of data and their accuracy depend on the chosen end-of-life solution. As illustrated in Fig. 3 for a product re-use, the product life information might need to be certified and guaranteed for compliance. Thus, a material database will store all this information, structured for each recycling stage; then as an example, it will become available for the designer. Nevertheless in many cases, the kind of data required is known, but its real value is either unknown, or fuzzy, or ranges beyond the limits. As a result, design evaluation becomes uncertain. Therefore, end-of-life solutions are not fully defined at an early design stage, and these initial decisions will impact the environmental footprint.

In addition, as illustrated in the *Physics and timetable* part, an efficient end-of-life solution might not have been developed yet. Consequently, end-of-life

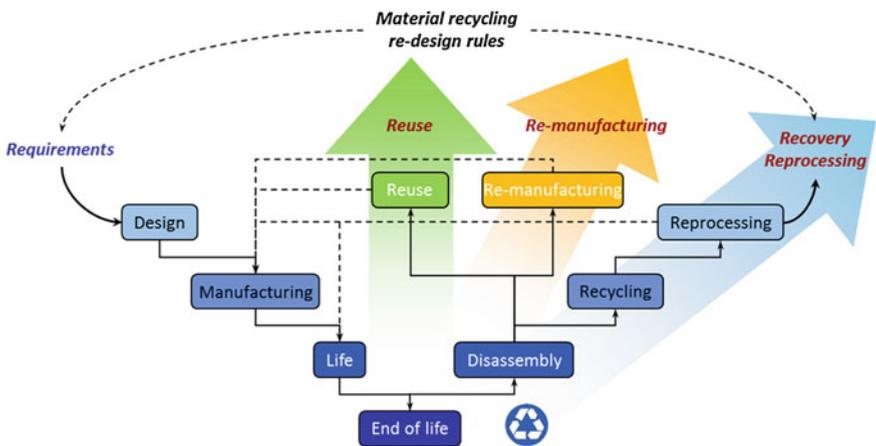


Fig. 3 V-cycle for information exchange

evaluations must be used with care, dividing the reliable results from the uncertain ones. Similar levels of information completeness can compare different solutions. Otherwise, the results should be taken as trends or qualitative comparisons.

So then, we firstly plan to map the design process cycle, with each key decision concerning life and end-of-life impact. These choices will need data, decision rules, etc. In addition, as previously shown in Fig. 2, all the information (requirements and constraints) will be captured in detail, in order to generate recycling rules. The connecting link between these two aspects, identified by using or generating the same data, will link designer to recycler.

4.3 Multidisciplinary Needs

The previous paragraph explained how we create the link between recyclers and designers. However in many cases, designers need data regarding a specific characteristic (e.g. maximum tensile stress) which recyclers are not able to give. Conversely, recyclers have to know about life damage, but designers can only inform on the use cases considered at design stage. Consequently, complementary information arises in this dual relationship. Material and product characterization is compulsory at different levels. People from material, chemical and mechanical fields will be able to provide a way of translating requests or requirements into real data. Indeed, many different characterizations must be carried out before and after the recycling process. For example, assembly consequences on the material end-of-life, and on the possible disassembly damage, must be identified. All these data guide the recycling process in order to minimize the variability incidence. In a second stage, the recycled material or re-processed semi-product has to be tested to

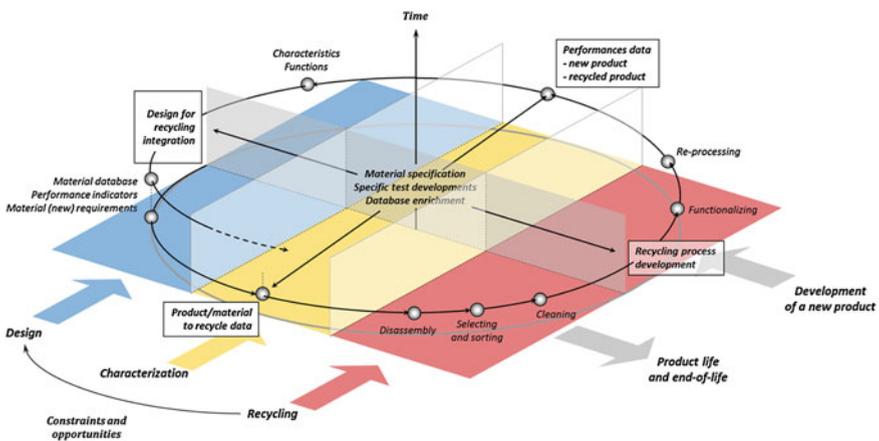


Fig. 4 Summary of the interrelation of the three skills

assess its quality and enrich design space for the designer. Figure 4 sums up the interaction between these three required skills.

From the characterization point of view, the key problem remains the adaptation of scale. The testing pyramid strategy (previously mentioned) helps to identify sufficient and necessary tests from the elementary sample level to the full system one (cf. Fig. 5). In most cases, specific tests must be developed in order to guarantee relevant and reliable results. This multi-level approach is also applied to the development of new recycling processes, and to their industrialization.

5 Composite Design for Recovery: First Lessons

The RECCO project (in which we were involved from 2009 to 2011) sought industrial solutions to recover (i.e. recycle and re-process) wastes of carbon fiber based composites.

A matrix removal process has been developed for thermoset matrix based materials. It consists in the dissolution of the resin phase by water under super-critical conditions. At a pressure of about 200 bars and a temperature of 400 °C, water becomes a real solvent for the resin phase, which then can be easily and fully removed. After this, dry carbon reinforcement remains oriented according to the original composite sequence, with no major fiber degradation.

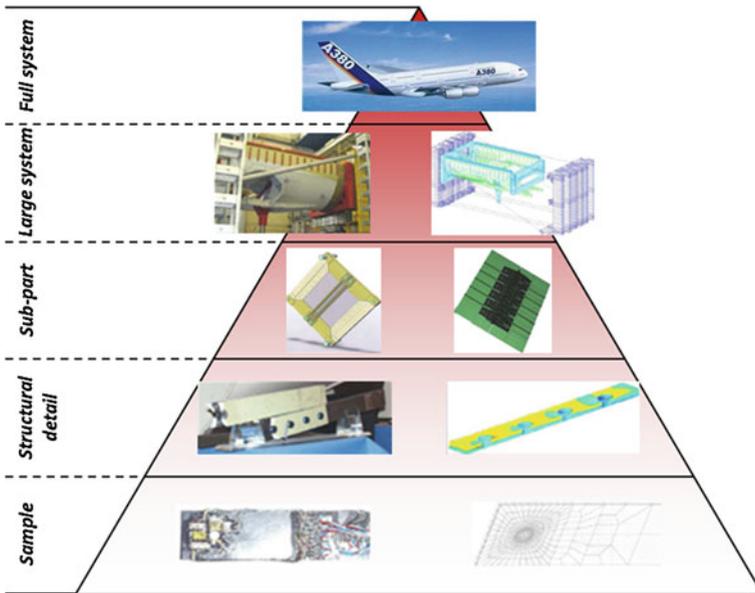


Fig. 5 Testing pyramid dimensions: from sample (micro scale) to structure tests (macro scale) (Laurin 2005)

The specificity of the solvolysis process is its maximal matrix removal, with a minimal water consumption; it also remains little polluting. Nevertheless, it is very sensitive to the presence of other non-organic material such as metal. For example, the solvolysis process oxidizes rivets, screws or inserts, and even parts of the process reactor in some cases. Then, it spreads small metallic particles in and on the carbon fibers, as illustrated in Fig. 6. Depending on the fibers orientation (unidirectional or woven), the solvolysis liquid flow concentrates this pollution on the outer edge of the fibers or where the tows cross.

This pollution has little influence on the dry fibers' mechanical strength, but it creates bridgings and local coatings that reduce the reprocessability (e.g. spinning or re-weaving). Nevertheless, when imbuing with matrix polymer, this eases mechanical grip (by limiting fibers sliding and tows misalignment) and has little effect on mechanical properties at part scale. Regarding the physical characteristics, this pollution only modifies the behavior locally. But more tests must be carried out to analyze these solvolysis pollution impacts.

Besides, these analyses have provided further feedback. It has been noticed that cutting processes (needed for adjusting parts to the recycling reactor) were burning matrix and changing its properties. This carbon-based layer (which can reach up to 200 μm with hard cutting conditions) locally glues fibers together, as illustrated in

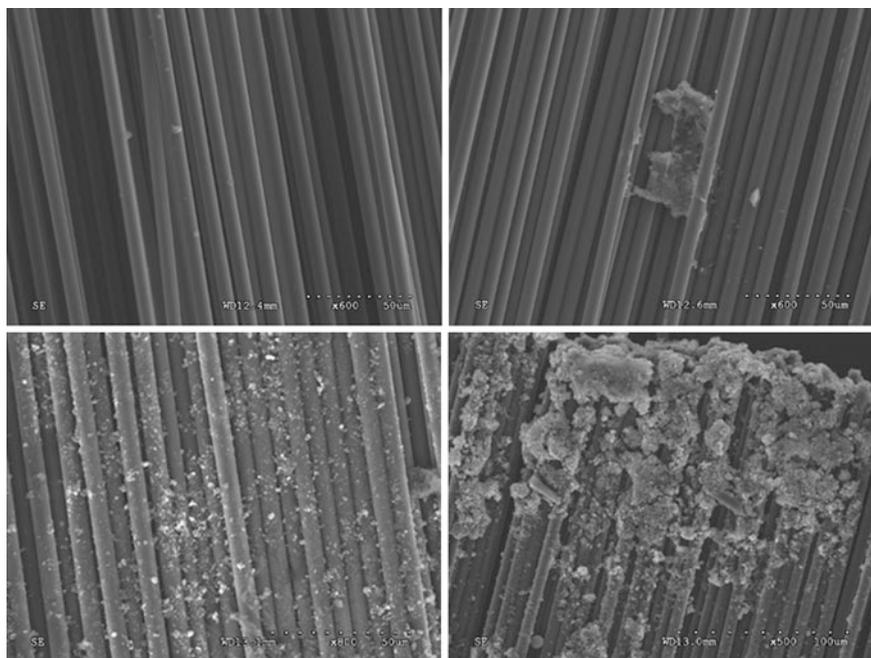


Fig. 6 SEM pictures of clean and polluted recycled carbon fibers: **a** Clean carbon fiber with small silicates, **b** Carbon fibers with slight nickel pollution, **c** Carbon fibers with considerable overall pollution from steel (Fe + Cr) and **d** Carbon fibers with edge pollution

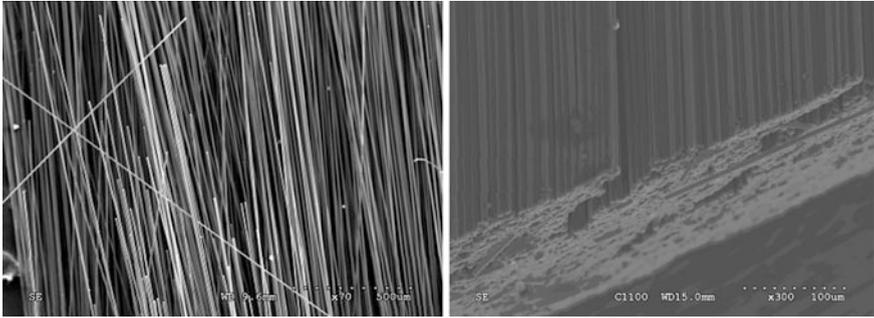


Fig. 7 SEM pictures of free (*left*) and glued carbon fibers at one edge, due to cutting process (*right*)

Fig. 7. This practical consequence can ease the handling of the recycle, by reducing the shreds when spinning a long tow with short or medium-sized fibers. Then on the one side, fibers are kept aligned; but on the other, the spinning efficiency of the inter-fiber grip can also be reduced.

Up to now, we have improved our competence in textile manufacturing; this will provide a help for implementing the end of the recovery loop (particularly the remanufacturing stage). The pilot demonstrator is under development, but we are already able to transform composite parts into second generation tows, or patches of recycled carbon fabric.

Mechanical properties of this new reinforcement are nearly the same as virgin one. Nonetheless, fibers are of course shorter than new ones due to the recycling process; this constraint leads to reprocess new semi-products that would be more easy to use, in order to close the recycling loop.

6 Conclusions

We are now able to recycle carbon fiber/thermoset matrix composites, and more precisely to recover carbon fibers. This development highlights the fact that recycling possibilities and constraints should be included from the early design stages. Skills and competences (knowledge and know-how) in materials and mechanical characterization fields are required to provide information about parts to be recycled, and about materials or semi-products derived from this recycling.

From our point of view, designers, recyclers and characterizers' skills must be interwoven to achieve design under recycling constraints, and to develop robust recycling processes for second design perspectives. In order to facilitate discussion and communication within these three areas of expertise, we have to work on defining requirements for and from designer, but we must also take the recycler's point of view into account. Systematic formalization of requirements and semantic alignment between the different (yet not so distant) communities should be implemented.

An environmental assessment of the solvolysis recycling process still has to be carried out in order to compare material and energy consumptions, pollution, cost, etc. for a same quantity of new or recycled composite.

The increasing use of composites in industry has brought about the development of end-of-life solutions. Regulatory constraints and financial perspectives (cost-cutting) will give rise to design for recovery or eco-design approaches.

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