Multi-level Circular
Process Chain for Carbon
and Glass Fibre Composites

MC4 101057394

Multi-level Circular
Process Chain for Carbon
and Glass Fibre Composites

Deliverable D5.6:
Sample materials for parts manufacturing

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Contents

1. OBJECTIVE

The aim of this document is to present the assessment of the recycled carbon fibres (rCF) materials manufacturing process and the set of samples materials which have been developed for part production in WP6. This development enters in the long-term loop of the MC4 project (Multi-level Circular Process Chain for Carbon and Glass Fibre Composites).

STFI and CHOMARAT have been the project partners involved in the sample production as well as the elaboration of D5.3 and D5.4. The partner PROFACTOR is responsible for the quality control and grading methods during the stages of the production process. Concerning the raw material, two different waste streams for the further processing are considered:

1. Uncured carbon fibre waste coming directly from a non-crimp fabric (NCF) production process.
   Project partner CHOMARAT delivered the waste material to STFI.
2. Cured carbon fibre waste from carbon fibre reinforced plastics (CFRP) recycled by a chemical process (solvolysis).
   Project partner GAIKER delivered the solvolyzed fibres to STFI.

The main development of the recycled carbon fibres reinforcements has been based on the uncured carbon fibre waste, as this source was available immediately at the beginning of the project. STFI and CHOMARAT worked in straight collaboration for the selection of the best fibres for the manufacturing process of each company. The selected fibres waste had been sorted by CHOMARAT on the carbon NCF line: 100% pure carbon fibres (12K, 24K or 50K), with a tow length of 80mm and still with the initial sizing from the CF manufacturer. A technical opportunity was also evaluated with the same kind of fibres, glass fibres from glass NCF line, for working on the cutting and laying with such fibres, in order to spread the range of recycled composites reinforcements with hybrid structure (glass and carbon) or pure Glass.

The second loop with solvolyzed carbon fibre from GAIKER was conducted as soon as the first samples of these recycled fibres were available. GAIKER managed its processing development in the Work Package n°3 (WP3).

The final recycled products which were delivered for the part demonstration (WP6), are stitched 0/90deg structures of 300 and 400gsm in 125cm width. This construction was chosen for the replacement of 12K woven fabric used today by the partner Amura for the Boat roof demonstrator of MC4 project.

2. PROCESSING OF RCF REINFORCEMENTS MATERIALS

2.1 Introduction

CHOMARAT is an industrial group specialized in 4 business units (Coatings & Films, Composites reinforcements, Construction and Garment). The composites Reinforcements field was developed in the 1960s, and grown along the decades following the boom of new molding solutions: hand lay-up, RTM light, vacuum infusion, prepreg, RTM, pultrusion… The variety of technical fibers in the reinforcements (glass fiber from 5,5tex to 4800tex, carbon fiber from 3K to 50K, Aramid fibers, …) is as wide as the technologies we use for creating products: weaving, knitting, tape, NCF… In the process, diverse amounts of fibre waste are generated and can be divided according to the fibre format. On the carbon NCF line, the waste is 100% pure carbon fibre (12K, 24K, 50K), with a tow length between 40 mm and 80 mm length. On the weaving loom the waste are short length carbon fibres (3K, 6K, 12K) from selvedges cutting (finished edges that prevent cloth from raveling) and includes a polyester yarn (2%). It exists also other waste from textile processes like the end of bobbins, short rolls, rolls with defects, etc., which can be a source of material for recycling solutions. In the MC4 project, in collaboration with the partner STFI, the focus was on the ‘’pure’’ waste from the NCF line as this is the biggest volume of waste in CF for the company and because such type of fibres is optimal for the nonwoven technology. Moreover, it gives also an opportunity to evaluate sorted fibres, which was not the case of the solvolyzed fibres from the partner GAIKER.
Concerning the processing of rCF reinforcements materials, based on the STFI oriented nonwoven, the most suitable technology for creating new dry fibres reinforcements is also the Non Crimp Fabric (NCF). This processing offers plenty of possibilities of various angles laying and has got a stitching device for improving the permeability properties which is always a high requirement for textile structures with discontinuous fibres. The collaboration with STFI, is detailed in the previous deliverable D5.3 of the WP5.

2.2 Development of semi-finished woven by project partner STFI

The first step for the processing is the preparation of the oriented nonwovens. This activity at STFI was operated until M30 of the project for managing two types of waste materials:

1. Uncured CF waste from CHOMARAT (virgin fibres coming from the cut edges on a carbon NCF line).
2. CF chemically recycled by solvolysis from composite materials (separation of fibres and matrix) from GAIKER.

The aim of the textile processing steps at STFI is to generate semi-finished textile products in the form of oriented nonwovens which can be transformed at CHOMARAT into specific NCF structures. The general process can be summarized in three steps:

- Opening the waste material by means of cutting, tearing and to separate the material into single carbon fibres
- Web forming out of the single carbon fibres via textile nonwoven processes (carding or airlay)
- Consolidation of the textile structures (mechanical bonding via needle-punching or thermal treatment)

2.2.1 Fibre materials delivered by project partner CHOMARAT

CHOMARAT selected a range of high strength carbon fibres which represent a panel of global applications among its customers. The uncured CF waste fibres out of the NCF production line was supplied to STFI in a sorted batch of different CF types during all the period of the development:

- Two CF 50K type from Zoltek (with two different sizings), and another 50K type from Mitsubishi (Pyrofil TRW40 50L). Both are used for high-performance applications: the first one is more used in the wind and civil engineering industry, and the second one is more used in the boat and automotive sector.
- Two CF from Toray group (T700S 12K grade which is the reference in prepreg industry and the T620S 24K grade which is very often used in the automotive sector).

With this 80mm waste fibre, free of any chemical residues, STFI assessed and demonstrated a good processability to produce nonwovens. Table 1 shows the fibre parameters of the used CF.

Name of carbon fibre	Fibre fineness [dtex]	Fibre diameter [µm]	Tensile strength [MPa]	Tensile modulus [GPa]	Elongation at break [%]	Density [g/cm]
Zoltek Panex 35 50K W61 (PA sizing) W13 (Epoxy Sizing)	0.80	7.50	4100	239.01	1.45	1.81
Toray T620S 24K 50C	0.89	7.94	4300	190.09	1.63	1.77
Toray T700S 12K 50C	0.70	7.00	4900	218.21	1.92	1.80
Mitsubishi Pyrofil TRW40 50L	0.75	7.00	4100	240.00	1.70	1.81

Table 1: Characteristics of carbon fibres from CHOMARAT (dry production waste)

The state of the uncured CF waste material after arrival at STFI is shown in Fig 1. According to the need of STFI, CHOMARAT provided all the quantities per sorted pallet.

Figure 1: Waste material delivered by CHOMARAT (CF tow of 80mm length)

2.2.2 Fibre materials delivered by project partner GAIKER

The second source of raw materials is developed by partner GAIKER. In contrast to the CF material from CHOMARAT, the waste material provided by GAIKER is chemically treated by a solvolysis process, that means the fibres are separated from the resin and come directly from the stacking of the part. Different textile structures can be inside: woven fabric, braid, unidirectional, non-crimp fabric, veil, …. Consequently, it could be also a package of different fibres in terms of type, size, … and from different supplier. With this operation, the carbon fibres are ''cleaned' but no longer have their original size. Initial prototype batches of a few kilograms arrived during M18, and the bigger quantities were delivered during the period M27-M30, due to the set-up of the reactor at GAIKER.

Figure 2: Waste material delivered by GAIKER

In the MC4 project, the composite materials which are used by the partner GAIKER, come from a Spanish industrial partner but without detailed information on the fibres' type. STFI and CHOMARAT observed in the solvolyzed textile structures, that the used fibres in these reinforcements are in a thinner tow range (3K / 6K) than the selected fibres from the NCF waste. Moreover, other materials like aramid fibres are also inside; it comes classically from the edge yarns, of aeronautical woven fabrics. The quantity is low (<1%) but this is a characteristic to consider on such fibre materials.
With a high heterogeneity (fiber types, length, …), the STFI processing of nonwoven with the GAIKER's fibres is more difficult to develop. This scenario could also occur in the later establishment of this recycling cycle. Depending on the product group, it cannot always be assumed that recyclers have all the material information of the input material at their disposal. Thus, the material currently poses a major challenge for processing at STFI, but at the same time holds the potential to develop a cycle that can later be transferred to industrial processes.
To evaluate the state of the fibres and the residual amount of sizing after the chemical treatment, a surface inspection has been done by means of a Scanning Electron Microscope (SEM) which is very suitable due to its high resolutions to make single fibres and structural properties visible. This equipment is the same as laboratories used for virgin carbon fiber analysis. The SEM images show that the fibres are quite clean and only some residues (sizing or matrix material) are left on the fibres surface.
For the further processing of chemically recycled fibres, the same is valid as for the uncured waste: the waste material has to be free of chemical residues from processing steps and the fibre length should preferably be in the range of 70 to 80 mm.

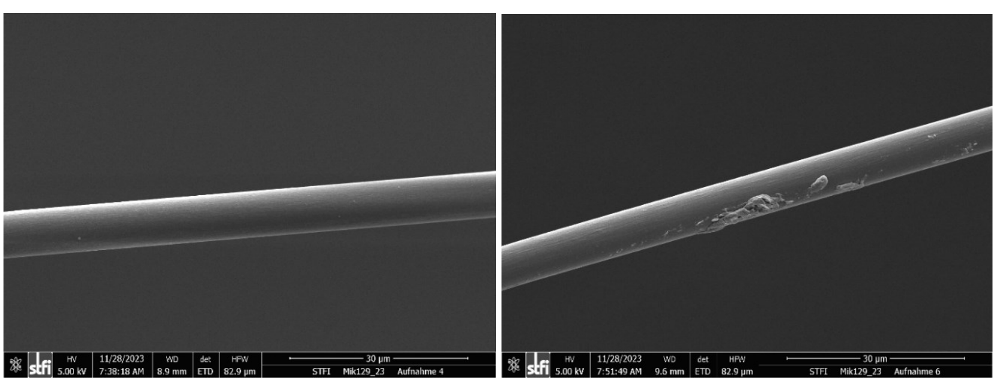


Figure 3: Carbon fibre surface after solvolysis

2.2.3 Description of technology – nonwoven production

STFI is a research institute that produces nonwovens and tapes out of recycled carbon fibres by using special technologies, such as carding or airlay together with different bonding technologies (needling, stitch-bonding, thermal). The processing with fibres of MC4 has been evaluated by all the different steps:

- Opening of the waste material by means of cutting and tearing into single carbon fibres.
- Web formation out of the single carbon fibres via textile processes (carding, airlay, tape).
- Entanglement of the textile structures (mechanical bonding via needle-punching or thermal consolidation) into semi-finished roll goods.

The target is to generate textile semi-finished products in the form of nonwovens or tapes made from the recycled carbon fibre material from CHOMARAT and which will be re-usable on an NCF line at CHOMARAT. The quality of the nonwoven is controlled by the area weight homogeneity across the width and the length, a visual homogeneity and the dry tensile properties. During the first period of the MC4 project until M18, STFI evaluated the different nonwoven possibilities by using the CHOMARAT sorted CF waste (80mm long, with sizing) to simplify the technical understanding. With this choice, the opening step is easy to realize as the homogeneity and quality of the sorted waste fibres is totally suitable to the limits of the machinery. It demonstrates that technical solution could be also found for specific products if the recycled waste is sorted upstream. A specific supply chain of dry textile waste (from textile companies like CHOMARAT or from customers with their dry cutting waste), can be also imagined in the future. The two figures below, illustrate the two types of nonwoven line at STFI (carded and needle-punching, and narrow tape).

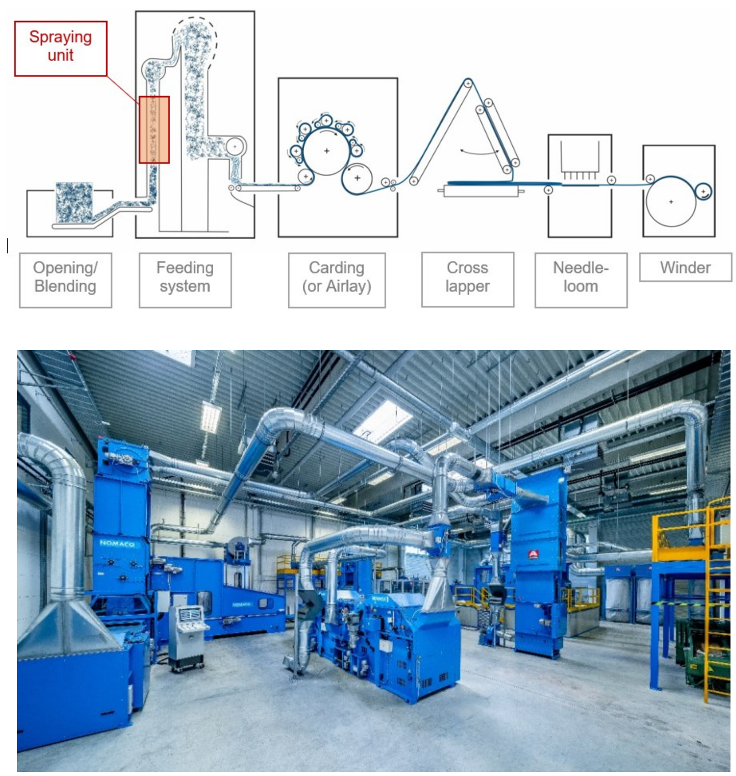


Figure 4: Scheme and photography of STFI carbon fibre nonwoven line

Figure 5: STFI nonwoven tape production from rCF

Several objectives had been tested for understanding the behaviour of each fibre and the minimal and maximal area weight. A matrix summarizes the results of trials of semi-finished nonwoven produced on the different technologies of STFI:

Nonwoven processing	**Airlaid (Isotropic)**	**Carded / Needle punched (Bi-directionnal 0/90)**	**Narrow Tape / Thermofixed (Oriented UD structure**
"Final Product" objective :	Technical evaluation for CF understanding	Promising design for 0/90 Non Crimp Fabric (NCF)	Promising design for +/-45deg Non Crimp Fabric (NCF)
Toray T700SC 12K 50C – 80mm	Not tested	OK, 80gsm to 150gsm	No tested
Toray T620SC 24K 50C – 80mm	OK if > 150gsm	OK, 80gsm to 150gsm	OK, 120gsm with 4% of binder
Zoltek Panex 35 50K W13 – 80mm	Not OK (fiber too fluffy with too low fibre friction)	Not OK (fiber too fluffy with too loo fibre friction)	No tested
Zoltek Panex 35 50K W61 – 80mm	Not OK (fiber too fluffy with too low fibre friction)	Not OK (fiber too fluffy with too low fibre friction)	No tested
Mitsubishi TRW40 50K – 80mm	No tested	OK, 200gsm	Not tested

*Table **2**: Summary of CF tested in semi-finished nonwoven at STFI*

This technical assessment had been concluded after regular trials sessions of feasibility testing.

Then, deep discussion took place with the partner in charge of the final part production (AMURA), because the ply area weight and the orientation are key parameters in the composites design. With these initial results, AMURA, STFI and CHOMARAT decided to select the carded needle punched nonwoven process, which is the technology the more reliable and provided oriented ply (0 and 90deg). The reasons for the non-selection of the other processing are that airlaid provide an isotropic repartition of fibres, which is not suitable with the stacking of Amura, and the lack of technology maturity for the thermofixed UD tape. This processing is still too much manual and couldn't be used for producing materials in sufficient quantities. In this technology evaluation screening, we observed also different behaviours of the CF grades (12K/24K/50K) which help us also to make the carbon fibres selection for the other steps of the processing. The Zoltek 50K presented a real issue for its transformation on the nonwoven line: a too high fluff volume of fibres happened during opening and carding. The lack of fibre friction between filaments produced a surface heterogeneity and no stability in the processing had been achieved. Zoltek 50K fibres were rejected for the next steps of the MC4 project. The technical reason could be the extreme softness of the Zoltek fibre, which is known and manageable today on a spread NCF line with continuous fibre, but not possible with a discontinuous type. This fibre is very specific in the CF market because its carbonisation level is at 95% and a consequence is a high variability of key textile properties (yield value tolerance, higher brittleness, …). Contrary to Zoltek fibres, Toray fibres with the 50C sizing give the best results and the Mitsubishi grade (50K, like the competitor Zoltek) demonstrates that heavy tow can work if the area weight is above 150gsm. To conclude, the table below gives an example of oriented nonwoven produced with the carbon fibre waste from CHOMARAT.

Carbon fibre from CHOMARAT (80mm length)	Fibre yield [Tex]	Area weight [gsm]	Width of nonwoven [cm]	Quantity [ml]
Toray T620S 24K 50C	1850	150	100	300
Toray T700S 12K 50C	800	120	100	300
Mitsubishi Pyrofil TRW40 50L	3750	200	100	300

Table 3: STFI products samples with CHOMARAT (dry production waste)

Concerning the technical quality controls measured by STFI during the trials, the homogeneity of the area weight and thickness are key characteristic. An example of capability study done at CHOMARAT lab show high variation on a recycled 80 gsm tape with T620S 24K 50C fibre (80mm long), coming from the carded cross-lapped processing and the needled-punched assembling at the end.

Measurement on 250 specimen (0,01m²)	Average (gsm)	Min (gsm)	Max (gsm)	Standard deviation (gsm)
80gsm tape	88	59	127	15

Table 4: Area weight variation on 80gsm recycled nonwoven in T620SC 24K 50C

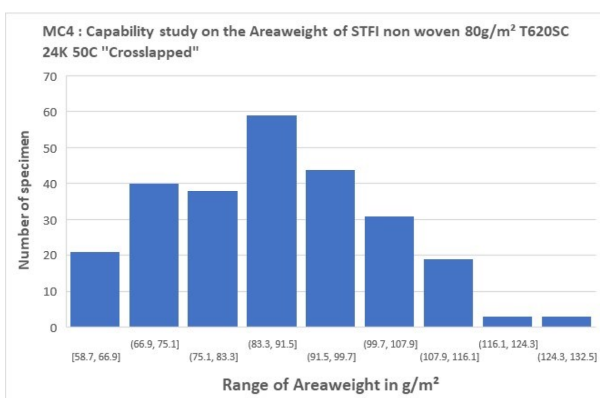


Figure 5: Area weight capability study on 80gsm recycled nonwoven in T620SC 24K 50C

Another important characteristic of the recycled oriented nonwoven for the CHOMARAT NCF processing, is that the stability and the tensile resistance is high as this semi-finished product will be cut, pulled and guided during mechanical cycle for which the speed is variable. On the NCF line, the tensile force management needs to be regulated as low as possible with a minimal limit. The stability of the nonwoven was observed during the NCF processing development to avoid any shrinkage or breakage during the cycle. A comparison was made between nonwovens manufactured with different fibres and different area weights. Table 5 shows tensile strength values measured at STFI laboratory with 150gsm nonwoven with T700SC 12K 50C, and 200gsm with Mitsubishi TRW40 50K. Both semi-finished products have been produced with carbon fibre waste from the CHOMARAT NCF line (80mm long, with the initial sizing from the carbon fibre manufacturer) and they have been processed at STFI through the same carded, cross lapped and needle punched operations. The result of T700SC 12K 50C is three times higher than Mitsubishi TRW40 50K. It demonstrates that even with a heavier area weight, Mitsubishi TRW40 50K fibre seems a lot of more fragile for processing it. The mechanical link coming from the sizing and the filaments friction, is a key point as both trials were produced in similar conditions: all fibres (CHOMARAT waste) or package of fibres (GAIKER) need to be considered individually. With these values, a first conclusion for the MC4 products developments is that recycled nonwoven structures with Toray T700SC 12K 50C (and T620SC 24K 50C which was demonstrated also), and Mitsubishi TRW40 50K, could be used for the AMURA demonstration. Zoltek fibres were rejected for the previous reasons of too fluffy fibres and non-homogeneous nonwoven processing.

STFI Nonwoven type (CHOMARAT fibres, 80mm length)	**Tensile strength MD [N]**	**Tensile strength CD [N]**
150gsm Carded, cross lapped Toray T700SC 12K 50C	8.83	25.05
200gsm Carded, cross lapped Mitsubishi TRW40 50K	2.97	5.60

*Table **5**: Initial study of dry tensile strength of STFI nonwoven*

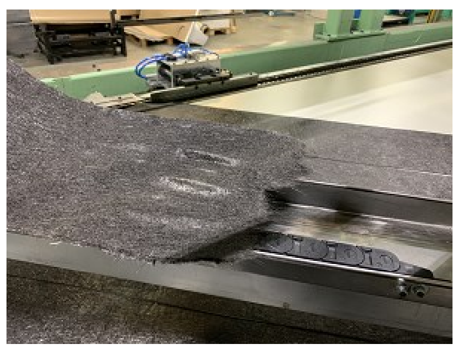


Figure 6: Example of a tape breakage during NCF processing

Concerning the assessment of nonwoven with solvolyzed fibres from partner GAIKER, only limited experiments at STFI were possible due to the low quantity of fibres. Initial prototype evaluations with 50mm cut length, with an area weight 150gsm, demonstrated that the fibre adhesion is insufficient and leads to a cracked nonwoven structure. The needle-punching to bond the nonwoven was not possible. In order to reduce with these technical problems, STFI re defined the processing parameters: longer tow length (80mm), reduced needle density (-30%), and higher mass per unit of the nonwoven (200gsm). With this configuration, positive observations appeared in the carding and web creation, as the figure 7 illustrates. The processing of the solvolyzed CF could be realized until the needle-punching step, even if the fibres remained quite brittle and stiff. The area weight of 200gsm was achieved as a minimal value for having a

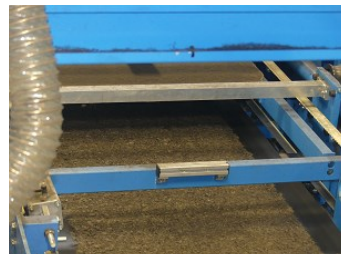
Figure 7: Initial evaluation of Gaiker fibres in STFI nonwoven processing (Failure with 50mm length)

fibre adhesion during the web formation but some wrinkles and material breaks occurred.
The ply weight limit value (200gsm) of the recycled nonwoven made of solvolyzed fibres, is a first constraint we discovered in MC4. Today, 200gsm corresponds to a standard ply in plenty of composite applications but this is a first information to consider in the processing assessment, as plenty of application requires 150gsm ply sequences.
The main remark is the surface quality of the nonwoven, as wrinkles remained present today on all nonwoven trials with 100% solvolyzed. Further trials with more fibres material to optimize the nonwoven process are necessary: a first conclusion we had is that until now, all the solvolyzed fibres correspond to a range of thin carbon fibres tow estimated 3K/6K.

Figure 8: Illustration of wrinkles on a 200gsm STFI nonwoven with Gaiker fibres

In MC4 project schedule, STFI realized complementary experimentations during M30 with the solvolyzed fibres (GAIKER), mixed with the pure waste from CHOMARAT. The principle was to understand if a mix can reduce the technical issues of too low fibre friction, wrinkles and reduce also the minimal ply weight limit below 200gsm. A trial plan was operated with an initial testing with a ratio of fibres mix of 50% / 50% and then 35% (Solvolyzed) / 65% (NCF waste) and then 20% (Solvolyzed) / 80% (NCF waste). The results of the trials plan are a failure for the first two trials and a positive processing of the last configuration. The trial with a configuration of 20% of solvolyzed fibres (3K/6K range, 80mm long, unsized) with 80% pure T620SC 24K 50C, 80mm long, original sizing from Toray was a success and achieved an area weight of 150gsm. This demonstration is an important result as it confirms that working with only solvolyzed fibres without any sizing is impossible. The solution of mixing with sized fibres is an intermediate solution and another conclusion is that the re-sizing of the recycled fibres coming from composites, is a new field to research in the future.

Figures 9 below, illustrates three phases of the processing of the technical solution of recycled nonwoven with the hybrid recycled fibres (GAIKER: 20% of solvolyzed fibres, 3K/6K range, 80mm long, unsized with 80% CHOMARAT NCF waste: pure T620SC 24K 50C, 80mm long, original sizing from Toray). The first photograph is the mixing of the fibres, the second one is a view of the carded web before the cross lapping and the third one is the view of the nonwoven after the needle punching operation. Homogeneity of the product is evident and comparable to the nonwoven made with only CHOMARAT NCF waste.



2.3 Assessment of the processability

In the MC4 project, the final demonstration by the partner Amura is a carbon boat roof with two pillars made by vacuum infusion. This non-structural part is designed obtained by a stacking of a 400gsm woven product made in high strength 12K fibre. A roof version with a visual carbon look exists also with the addition of a 245gsm woven product in high strength 3K fibre on the exterior side. Both products are balanced (50% of area weight in warp and in weft) and their width is 1250mm. The first operating step of Amura is the cutting in layers and the draping according to the 0deg and 90deg axis of the part, with some possibilities of direction change. In agreement with AMURA, the process development at CHOMARAT was focused on a rCF stitched 0/90deg structure in 125cm width, to be closed to the woven fabric (Warp/Weft structure) used today in the design. Objective is the using of STFI nonwoven products on a Non Crimp Fabric (NCF) line, for orientating them in two layers (0deg and 90deg), and assembling them by stitching for a manipulatable reinforcement. NCF processing with recycled nonwoven is a lot of different from standard material, as with continuous fibres (spools like 2500lm for 50K grade or 7500lm for 12K grade), NCF handle flat tows with high pulling force resistance. The STFI semi-finished products are thicker and more fragile layers. The assessment of the processability concern the sequences below:

- Cutting the nonwoven at the right width according to their use on the NCF line, and winding.
- Laying with angles on the NCF line.
- Assembling the layers by stitching and final winding.

2.3.1 Processing development

The first step is tape preparation with a cutting and winding operation. On the NCF line, the guidance of the recycled nonwoven needs to be different from continuous yarn spools: processing is not with an online creel and dedicated guidance for tows, but with a specific offline unwinding station. STFI delivered to CHOMARAT the selected nonwoven type (carded crosslapped and needle punched) on soft rolls in 100cm width with no straight edges. Such rolls are not usable directly. A preparation at Chomarat is necessary to redefine the formats of the nonwovens, in order to be transferred on specific beams required for the Karl Meyer NCF line. The technical requirement is to create tapes in specific width for 0deg unwinding and 90deg laying. The operation is done by an offline cutting step which consists of unwinding and cutting the STFI nonwovens at constant speed with a circular knife. Several tapes are cut in parallel and rewound with a high accuracy of +/-1mm on the biggest possible roll.

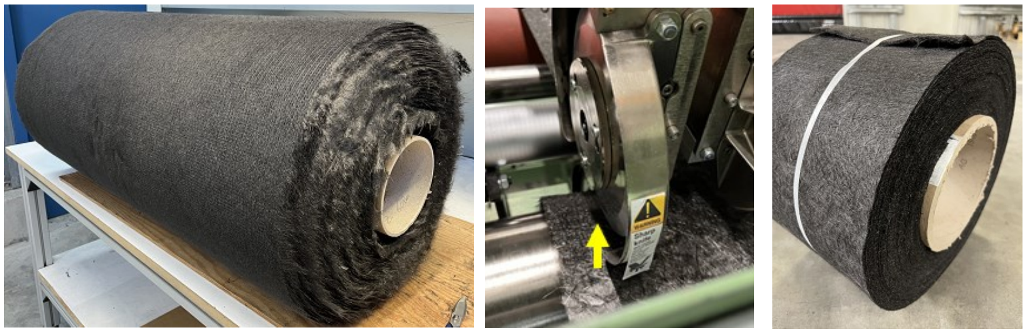


Figure 10: STFI rolls, to straight cut narrow tapes at CHOMARAT

Then, the second step is the core of the NCF processing technology: the laying of the fibres at the right direction on a conveyor belt, with a fixing system on the edges for maintaining the angle of the fibres with the right tension. At first, the tape roll is unrolled under regulated tension and then tape is pulled in the right direction. In the 0/90deg products development for MC4, we use unwinding stations at 0deg direction and at 90deg direction. Clamping force and pulling force are the key parameters for the settings. With nonwoven tapes, the tensile force management needs to be regulated as lower as possible according to the diameter size, for avoiding a shrinkage or a breakage of the tape in the cycle.

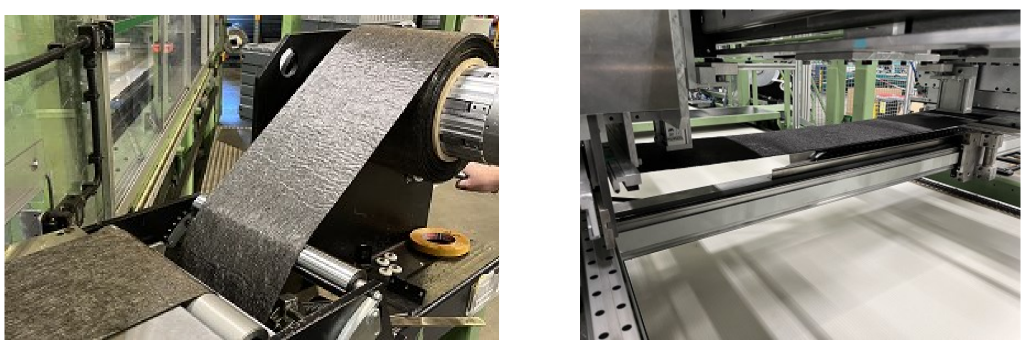


Figure 11: Offline station for unwinding and view of 90deg laying on the conveyor belt

After this unwinding and pulling step, the tape is cut at the right length for being caught by two robotic arms and inserted in the towing chains on both extremities of the width. The movements of the kinematics need to be synchronized with the advancement of the chains which feed the stitching at a constant speed. Due to the higher thickness of the recycled nonwoven, a specific fixing method in the chains was found. The cycle of mechanical movements was modified by adjusting different movements of the pneumatic cylinders.

One cycle of the NCF machine corresponds to the deposition of one narrow tape on the conveyor belt, and the next cycle will juxtapose a second tape, with an accurate overlap. The overall coverage of the conveyor belt is done by this individual 90deg tapes laying, cycle after cycle. The right quality of rCF material was achieved as soon as the accuracy of the tape cutting was improved to within a tolerance of +/-1mm.

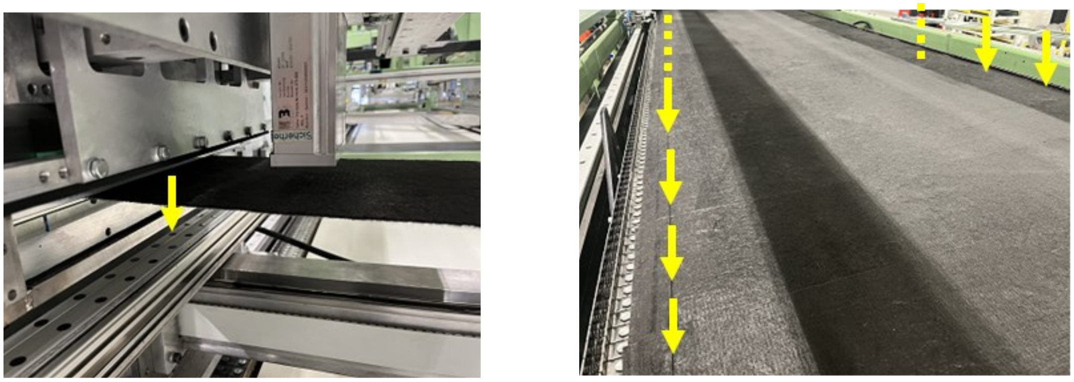


Figure 12: Detailed views of tape insertion cycle with 90deg orientation

Then, the 0deg layer is continuously unwound on the conveyor belt (on the top of the 90deg layer) just before the knitting device. The last step on the NCF processing line for the 0/90deg manufacturing is the assembling of the two layers by stitching and final product winding: Figure 13 shows the local area with the stitching needles and a view on both side of the product on the winder. The main critical processing parameter is the tension of 0deg and 90deg layers for keeping the right fibres orientation on the entire length on the conveyor belt and in the stitching are device. A second monitoring point is the total thickness of the two stacked layers which need to be regular and not too high. The technical reason is that stitching needles are in vertical position and penetrate the fibres stacking from the bottom to the top with a maximum vertical space, not possible to exceed. The two combined layers must be able to slide relative to each other in the knitting area. During all trials, the maximal acceptable thickness for the assembling of a 0/90deg NCF construction was with carded and cross lapped nonwoven of 200gsm, so 400gsm total. The parameters for stitching of the recycled 0/90deg prototypes products are the same as standard NCF with continuous fibres: E5 gauge with a chain-tricot point, and polyester yarn. Only the stitching tension was adapted to the thickness of the different produced materials.

Figure 13: Detailed views of a rCF 0/90deg material
(In the stitching area and on the winding device)

A complementary topic of new recycled products with glass fibre and hybrid (carbon fibre / glass fibre) was worked on in parallel by CHOMARAT. This work had been added in the development for opening all the new potential for recycled materials. With the failure trials of nonwoven made with 100% of the solvolyzed fibres, it added an opportunity to complete the study. Carbon fibres and glass fibres are in competition in the composites design of many applications and can also be used together according to the parts requirements. Based on the knowhow of CHOMARAT in composites business, possible intermediates industrial solutions can find a place like hybrid material with CF and GF inside. In the GF composites industry, the reinforcements products with discontinuous fibres like CSM (chopped strand mat) or veils or stitched products (CSM with NCF continuous fibre) are well spread. With the nonwoven technologies of STFI and CHOMARAT, similar textiles constructions can be created by taking advantage of the benefits of each fibre type. Due to a bigger filament size (from 17µm to 24µm) than carbon fibre and with specific sizing, glass fibre works well in the cutting and opening operations. Mixing and carding are the processing steps to develop. Glass fibre has also got the advantage of having a very low-cost which is 10 to 20 times less carbon fibre. Hybrid nonwoven made with carbon and glass fibres are interesting to consider as complementary works in the WP5.
The first experiments were done at the STFI plant on a Menningen carding machine. This dedicated line for short fibres, was tested at first for checking the feasibility. GF roving from Owens Corning 600 and 1200tex 111A (from Chomarat waste) was cut in 50mm length segments and then inserted in the carding. The beginning of the trials was quite interesting as the nonwoven was very regular, but the process failed after a few minutes. The too high strength and abrasion of glass filaments damaged the pins of the card. During the rotation of the carding device, a certain quantity of fibres accumulated inside the pins. Working cylinders were performing correctly as a homogeneous web was extracted from the end of the carding but fibres were not enough combed by the pins. The technical reason was the unsuitable pins for glass fibres. The conclusion of this machine evaluation is negative but an improved process with another design of the carding's pins seems realistic.

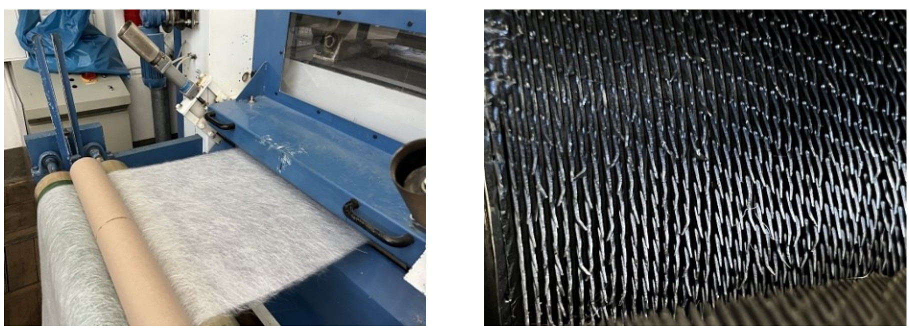


Figure 14: Trial of glass fibre carding at STFI

Another possibility is to use the CF dedicated nonwoven line for inserting glass fibres inside the opening operation, for a more advanced preparation of the fibres before carding. A highly homogeneous mix of glass fibre and carbon fibre can bring an advantage for carding as well as the mix of solvolyzed fibres with dry fibres waste did. This feasibility trial was operated on the Autefa line with success and opened another research field. The exact product was a 150gsm nonwoven (carded, crosslapped, needle punched) was achieved with 50% CF (Toray T700SC 12K 50C – 80mm) and 50% glass fibre (E-CR 1200tex 80mm).

With the failed experience on 100% glass fibre carding, Chomarat decided to continue a technical focus on this topic with another way than standard nonwoven processing: with dedicated cutting fibers and deposit processing by air. Several technologies exist like Finn & Fram or Spyke industrial devices and worked already at Chomarat with dedicated virgin glass fibres. They have a specific sizing able to split the roving in a regular number of fibres bundles during cutting. The technical development was concentrated on the fibres 'length influence (from waste) and their distribution on a belt or on combined structures with another reinforcement to assemble with. The way the roving of glass fibre is separated in filaments and the way this volume of fibres lay down, is the key point for the homogeneity of the final recycled product.

If the glass fibre is in the same family of carbon fibre for the composites industry, they have a different molecular structure and two main characteristics are very different: filament diameter size (17µm versus 7µm for the CF), density (2,6 versus 1,8 for the CF). Several feasibility studies had been done to understand the fibre length. An offline intermediate step was added in the processing for the feeding of the cutting system: on a Pierret machine (Guillotine technology), the NCF fibres waste is cut in short length in order to create more thin bundles and to have a material with more opened filaments. Some experiments have been tested up to 18mm fibre lengths and then have been laid on a belt and stitched. These initial works require deeper research and should offer other opportunities to a new recycling cycle.

Figure 15: Example of a CSM structure with 18mm cut glass fibres from NCF waste

2.3.2 Recycled reinforcement materials and characterization

Globally, with STFI carbon nonwoven semi-finished materials, the processability for the cutting and the laying on the Karl Meyer Max5 machinery works well if the quality was homogeneous inside the rolls. A critical point is the dry tensile resistance, as for cutting or laying, a minimal strength is necessary during pulling and winding operations. During the CHOMARAT research development phase, until the milestone M30 of MC4 project, all the nonwovens had been manufactured by STFI with the CHOMARAT waste, and not from the GAIKER fibres. Consequently, the semi-finished materials were with 80mm sized fibres, and a specific fibres friction linked to the properties of the original supplier.

A high variety of prototype products had been manufactured by STFI and CHOMARAT for advancing progressively to more defined recycled reinforcement materials. In the WP5 materials characterizations, a study on the permeability was realized for checking how these new textiles reinforcements will be impregnated by the customer resins. The laboratory device that CHOMARAT used for the tests is the EASYPERM machinery (Figure 16). This study was to compare the NCF with recycled tape (feasibility trials) with standard NCF (C-PLY range) that CHOMARAT had. No thermoset resin is used in this method, but an oil, with the same viscosity as composites resin, is used in two methods: in plane for X and Y values, and through the thickness of the stacking for the Z-permeability. Values for X, Y and Z are individual coefficients to understand. The result of this study is summarized in the table 7 below, which shows that the stitched recycled materials enter in the same range of permeability than standard products with continuous fibres. The main difference is linked to the high thickness of recycled products: the fibre volume fraction (FVF) is only at 20%.

Permeability of MC4 prototypes product (20% FVF)					Data from C-PLY (classical carbon NCF with continuous fibre, 4 plies at 51% FVF)	
Product	**Ply number**	**X Permeability**	**Y Permeability**	**Z Permeability**	**X and Y Permeability**	**Z Permeability**
0/90deg 160gsm	4	3.83 E-11	3.70 E-11	1.93 E-12	From 1.05 E-11 to 7.40 E-11	From 4.80 E-13 to 1.93 E-12
+/-45deg 160gsm	4	3.83 E-11	4.13 E-11	1.77 E-12	From 1.78 E-11 to 3.48 E-11	From 2.26 E-12 to 1.03 E-13
+/-45deg 240gsm	2	4.23 E-11	5.20 E-11	1.57 E-12		
+/-45deg 300gsm	2	3.20 E-11	3.90 E-11	1.80 E-12		

*Table **6**: Permeability study on recycled NCF structures (CHOMARAT)*

Figure 16: EASYPERM device at CHOMARAT laboratory

The partner STFI manufactured several composites panels all along the processing development (with the RTM compression molding method), for checking the level of mechanical properties of the recycled reinforcements. Characterizations of these panels were done with the semi-finished STFI nonwoven products, or on the final 0/90deg stitched reinforcements. Here below, the results of tensile testing of two kinds of recycled nonwoven are shown: 150gsm with 100% CF waste from Chomarat, and the same with 50% CF and 50% glass fibre from Chomarat waste. Both nonwovens have got the cross lapping with 1/3 of fibre quantity in 0deg direction and 2/3 in 90deg direction.

STFI Nonwoven of 150gsm (Carded, crosslapped, needle punched)	Force H [N]	σm [MPa]	εm [%]	Emod [GPa]
100% CF (Toray T700SC 12K 50C – 80mm) 0° Direction	5724	264	1.46	19
100% CF (Toray T700SC 12K 50C – 80mm 90° Direction	9087	430	1.34	32
50% CF (Toray T700SC 12K 50C – 80mm) 50% Glass fibre (OC 111A 1200tex 80mm) 0° Direction	3831	181	1.59	12
50% CF (Toray T700SC 12K 50C – 80mm) 50% Glass fibre (E-CR 1200tex 80mm) 0° Direction	5315	252	1.65	16

*Table **7**: Example of mechanical data of STFI recycled nonwoven*

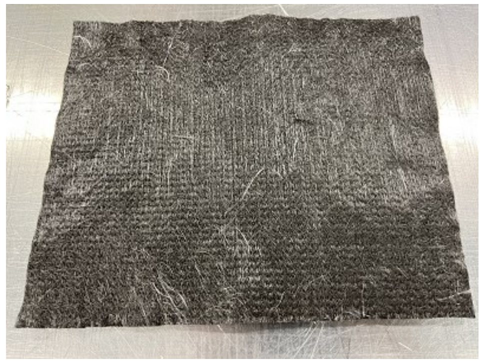


Figure 17: View of the hybrid recycled nonwoven
50% Carbon fibres + 50%glass fibres

As the direct supplier of the recycled reinforcements to partner Amura in MC4, CHOMARAT developed and delivered three products by M30:

•OCI1505 r BT240 CT3 12K x125cm: 0/90deg structure (2 layers of 120gsm at 0 and 90deg)
T700SC 12K 50C, 80mm length
Chain-Tricot Polyester stitching
•OCI1524 r BT400 CT3 50K x125cm: 0/90deg structure (2 layers of 200gsm at 0 and 90deg)
TRW40 50K K, 80mm length
Chain-Tricot Polyester stitching
•OCI1535 r BT300 CT3 24K x125cm: 0/90deg structure (2 layers of 150gsm at 0 and 90deg)
T620SC 24K 50C, 80mm length
Chain-Tricot Polyester stitching

A full mechanical characterization (tensile, bending, flexural and interlaminar properties) had been launched on these products at the STFI laboratory. At M30, only the study on product R BT240 CT3,4 12K/1 HS (80MM) with Toray T700SC 12K 50C, has been finalized: the two others are planned for M31. The CHOMARAT laboratory used resin transfer molding for making the composites plates. The mechanical property targets can be summarized with a rigidity and elasticity modulus from 20 to 40GPa (tensile strength from 200 to 300GPa and flexural strength from 400 to 500MPa). The curves and the values of strength obtained during the laboratory's analysis illustrate correct behavior but the modulus of 20GPa confirm that the recycled laminate has got a too low fibre volume content. Improvement needs to be done for fibre orientation and thickness reduction in the nonwoven manufacturing.

OCI1505 r BT240 CT3,4 12K/1 HS (80mm) with Toray T700SC 12K 50C			
Mass per unit area	240gsm		
Mechanical properties of Composite	Fibre volume content	20,92%	
	Part thickness	3,90mm	
	Tensile Strength	0deg: 279,1MPa	90deg: 292,9MPa
	Young's modulus	0deg: 19,3GPa	90deg: 19,7GPa
	Bending Strength	0deg: 367,2MPa	90deg: 395,4MPa
		45deg: 365,9MPa	-45deg: 355,4MPa
	Flexural modulus	0deg: 17,1GPa	90deg: 15,7GPa
		45deg: 15,7GPa	-45deg: 14,6MPa
	Interlaminar shear strength	0deg: 37,3MPa	90deg: 42,6MPa
		45deg: 38,1MPa	-45deg: 37,0MPa

*Table **8**: Initial mechanical data of composites with recycled carbon 0/90deg product*

The next steps will be to complete the data of the two other products, and of the latest developed nonwoven material from STFI: 150gsm made with a mix of 20% of solvolyzed fibres (3K/6K range, 80mm long, unsized) and 80% (pure T620SC 24K 50C, 80mm long, original sizing from Toray) which is planned during M31:

•OCI1540 r BT300 CT3 24K/1 x125cm: 0/90deg structure (2 layers of 200gsm at 0 and 90deg)
Fibres mix (80% T620SC 24K 50C, 80mm length with 20% solvolyzed fibre)
Chain-Tricot Polyester stitching

3. CONCLUSIONS

This work demonstrated the development of new recycled textile reinforcements suitable for composite parts. The close collaboration with the involved partners of WP5, has succeeded to manufacture several 0/90deg NCF structures on a serial NCF line at CHOMARAT with various types of fibres: TORAY T620SC 24K 50C and T700SC 12K 50C, MITSUBISHI TRW40 50K K. The scrap from the CHOMARAT NCF line (sorted fibres 80mm length) worked perfectly inside the nonwoven technologies of STFI, with a possible range of ply weight from 80gsm to 200gsm. The CHOMARAT NCF processing was adapted with success to the use of oriented nonwoven in 0/90deg configuration, after a definition of nonwoven limit characteristics (max 400gsm, homogeneity of the surface, minimal dry tensile resistance). Concerning the assessment of the nonwoven manufacturing process at STFI with the chemically recycled fibres by GAIKER, the conclusions are balanced. The nonwoven process failed when 100% of the batch of the CF is only solvolyzed fibres, linked to the unsized type of fibres and probably also from the mix of carbon fibres inside (3K/6K). A first configuration with a mix of solvolyzed fibres, with fibres from production waste having their sizing, was achieved and confirmed that such processing is possible.

Material characterization at the textile level (area weight, permeability, …) and at the composites level (mechanical properties) are in progress for finishing a database useful for the composites design of recycled solutions. The final report will provide information on the three reinforcements developed and supplied to Amura for the boat roof demonstrator.

During the project, also a complementary target for recycled composites reinforcements had been added with works on glass fibres waste. This research component needs to be explored deeper and opens a wider spectrum of applications, as glass fibre is a such well-established market and presents a cost advantage. New products such as hybrids could be designed to achieve topics that glass fibre or carbon fibre could not fill alone for various reasons.